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Abstract

The report discusses surface analysis and thermodesorption as methods for studying the gas content of inner wall components of fusion devices. The following problems are considered: (i) calibration of the thermodesorption, (ii) parasitic adsorption and reaction of released gases at inner walls of the analysing chamber, (iii) gas absorption during storage of the samples in air, (iv) influence of the surface morphology, and (v) influence of deposited layers of carbon and boron. A large number of tiles from the inner and outer divertor of ASDEX-Upgrade have been studied. The tiles consisted of pure graphite, massive tungsten, and graphite with sprayed tungsten coatings. Large regions of the inner divertor tiles show thick deposition layers of carbon, boron, and the hydrogenic isotopes containing up to 10^{23} deuterium atoms/m². In the regions of the outer divertor, where erosion processes prevail, the gas content is less by about one order of magnitude, depending in a complicated way on the surface structure and being laterally inhomogeneous. Codeposition trapping is favoured by the surface roughness even in the erosion zones.

1 Introduction

Considerable amounts of hydrogen, deuterium or tritium can be trapped and retained in wall materials after exposure to tokamak plasmas. At elevated temperatures these gases may be released, thus influencing the gas filling of the machines. The trapping mechanisms have been studied in great detail in the refs. [1-10]. As described in these papers there exist several mechanisms for hydrogen retention. For graphite the most important processes are the following:

- (i) Chemisorption of gas at the (geometric) surface (about 10^{18} - 10^{19} atoms/m² for a monolayer).
- (ii) Implantation of hydrogen ions. Above a minimum energy of 25 eV the irradiation of the surface leads to damage sites in the graphite lattice where the ions can be trapped with a binding energy of 2-3 eV. According to refs. [1,2] this process saturates at 0.4-0.6 H/C. The total amount trapped at saturation depends on the implantation depth and varies with the energy of the incident ions. According to ref. [11] at saturation the number of implanted D-atoms per m² amounts to about $3.5 \times 10^{18} E^{0.9}$, with E being the ion energy (in eV). For 100 eV saturation is reached at about 2×10^{20} atoms/m².
- (iii) Adsorption at inner surfaces enhanced by diffusion into pores. According to ref. [1] saturation occurs at about 10^{23} molecules/m² (related to the geometric surface). Hence, this process retains much more gas than adsorption at the geometric surface. The rapid surface diffusion may deposit the gas beyond the implantation zone to depths $> 1 \mu\text{m}$.

- (iv) Codeposition trapping (co-implantation). If at exposure new carbon layers are formed at the surface, hydrogen can be bound in these layers with about 0.4 H/C [1]. Since the layer thickness is virtually unlimited, amounts in excess of 10^{23}m^{-2} seem to be possible.
- (v) Intergranular diffusion. At temperatures $> 1000\text{ K}$ hydrogen can diffuse into the graphite lattice and be trapped at intrinsic trapping sites with a binding energy of about 4.3 eV [1, 2].

For massive tungsten without cracks diffusion into pores can be neglected. However, as shown in ref. [12] hydrogen can be dissolved deeply within the lattice with amounts considerably exceeding those expected within the implantation zone.

These processes have been studied mostly by ion beam implantation in small, well-defined samples. In practice the large graphite tiles in plasma machines are differently subjected to erosion and deposition processes that mainly influence the thickness of codeposited layers (process iv). For the divertor plates the contact to the plasma varies along the surface with the distance to the separatrix.

The deuterium inventory found in TFTR-tiles had an order of magnitude of 10^{21}m^{-2} , deposited a few μm below the surface [13]. Much higher values of $2.5 \times 10^{23}\text{m}^{-2}$ (deuterium) and $5.1 \times 10^{23}\text{m}^{-2}$ (hydrogen) were found by thermodesorption spectrometry (TDS) for small samples cut from ASDEX-Upgrade tiles [14]. In a second paper [15], the authors came to the conclusion that diffusion dominates the gas retention in graphite, which therefore is controlled by the temperature of the tiles. However, in order to conclude on processes involved in deuterium trapping at plasma exposure, additional information on the spatial distribution of the retained deuterium and a characterization of the surface with respect to roughness and impurity deposition is required.

In particular, the influence of the surface roughness is poorly known. When analysing diffusion and codeposition, some authors tacitly assume smooth surfaces. Being variable by the interaction with the plasma, the roughness can have more influence on the gas trapping than anticipated thus far. The present paper describes experimental details of the thermodesorption method and focus on the combination of this method with surface analysis and studies of the surface morphology.

2 Experimental setup, methods and results

2.1 ASDEX-Upgrade divertor tiles

The analysed tiles stem from the lower divertor of the tokamak ASDEX-Upgrade, exposed to deuterium and hydrogen plasmas in two experimental campaigns. After the exposure they were brought through air to the surface analysis station and the device for thermodesorption (some of the tiles have been stored several months in air). The tiles consisted of pure graphite, graphite with tungsten coatings, and tiles with massive tungsten inlays. The graphite tiles were used from 1991 until 1995 for about 1900 discharges, while the tungsten tiles were exposed to about 800 discharges in 1996.

Figure 1: Schematic of tile arrangement at the inner and outer divertor of ASDEX-Upgrade. Shaded: strike point region

Toroidally the divertor consists of 16 segments (S1-S16). Fig. 1 shows the position of the tiles in a segment. In poloidal direction they are arranged in the rows A-E (inner divertor) and A-D (outer divertor), while the toroidal position is given by the numbers 1-6 and 1-8 for the inner and outer divertor, respectively. Most intensive plasma contact is expected to occur (and has been found by thermography) on tiles C of the outer divertor.

The tiles could be studied as a whole (with a size of up to $10 \times 10 \text{ cm}^2$) or by cutting smaller pieces out of them. Thermodesorption of the whole tiles has obvious advantages: the tiles can be reused, there is no contamination by the cutting procedure, and gas trapping by the sides of the tiles can be included. The disadvantages are poor spatial resolution and nonlinear heating rates for thermodesorption. In some cases large amounts of gas must be handled.

2.2 Thermodesorption (TDS) and related problems

2.2.1 The TDS-device

Fig. 2 shows a schematic of the apparatus used for the TDS-measurements. The main chamber (C_1) is a stainless steel vacuum vessel, volume $V=40 \text{ l}$, pumped by a turbomolecular pump with a pumping speed of $S \approx 200 \text{ l/s}$ for hydrogen. Base pressures $< 5 \times 10^{-8} \text{ mbar}$ are achieved. The samples (S) are horizontally put onto three molybdenum pins and heated by electron bombardment from an extended tungsten filament (F). Sample and filament are surrounded by a movable heat reflector (R) made from molybdenum.

The pressure p_1 in the chamber C_1 is measured by a Bayard-Alpert gauge. Via a slit in the reflector and a ZnSe window the sample temperature is monitored by an infrared thermometer (type Minolta/Land Cyclops 300 AF). A fraction of the desorbed gas reaches the analysing chamber (C_2) through a flow conductance of 1 l/s. This chamber ($V=5$ l) is pumped by another turbomolecular pump down to pressures $<10^{-8}$ mbar. It is equipped with a Balzers quadrupole spectrometer (type QMS 421) for analysing the desorbed gas. V1 and V2 are valves. Spectrally pure gases (deuterium, hydrogen) can be introduced via the leak valve V3.

The infrared thermometer covers the wave length range 0.6-20 μm . Its reading is checked by thermocouples at temperatures of 300-800 K, and by a pyrometer for temperatures above 900 K. The accuracy of the measured temperature is estimated to $\pm 5\%$. The samples are heated by electrons hitting the front side with currents of 0.1-0.5 A and energies of 1-3.5 keV, thus the heating power can be varied between 0.1-1.75 kW. For large tiles ($8 \times 8 \text{ cm}^2$) the maximum temperature amounts to 1500 K. The temperature of the samples is sufficiently uniform along the surface within ± 50 K. Figure 3 shows the temperature increase for a heated divertor tile (surface $8 \times 8 \text{ cm}^2$) at various heating powers. Here the heating time constant amounts to about 5 min., the temperature rise rate being not constant. With smaller samples (typically 1 cm^2) temperatures up to 2000 K could be reached in a nearly linear regime.

Figure 2: Schematic of the device for thermodesorption (TDS). C_1 - main chamber, C_2 - analysing chamber, S - sample, R - reflecting shield, F - filament for electron bombardment of the samples, QMS - quadrupole mass spectrometer, V_1 , V_2 , V_3 - valves.

Figure 3: Temperature increase of a heated divertor tile ($8 \times 8 \text{ cm}^2$) for various heating powers.

2.2.2 Measuring principle and calibration

The gas analysis is performed in a dynamical regime with open valves V_1 and V_2 . The pumping speed S_1 is high enough to guarantee proportionality between pressure change and gas desorption on the time scale of interest, the pumping time constant being $V/S_1=0.2$ s. So the desorption rate Γ_k of a mass species k is given by the partial pressure $p_{1,k}$ and the pumping speed $S_{1,k}$ for this mass

$$\Gamma_k = p_{1,k} S_{1,k} .$$

For the total pressures p_1 , p_2 in the chambers C_1 and C_2 , respectively, we have $p_1 \gg p_2$, thus the gas flow into chamber 2 does not depend on p_2 . Then the partial pressure $p_{1,k}$ is proportional to the ion current I_k of the mass spectrometer measured in chamber C_2 :

$$p_{1,k} = c_k I_k .$$

For calibration, gas is introduced into C_1 to reach constant equilibrium pressures $p_{1,k} = P_{1,k}/f_k$ with $P_{1,k}$ being the readings of the ionisation manometer and f_k the correction factor taking into account the mass-dependent ionisation probability. Table 1 shows the values of c_k for various gases, valid for a given resolution of the mass spectrometer.

Table 1: Calibration factors

GAS	H ₂	D ₂	CH ₄	H ₂ O	N ₂	Ar
mass number k	2	4	16	18	28	40
f_k	0.44	0.35	1.6	1.0	1.0	1.2
c_k ($\mu\text{A}/\text{mbar}$)	7.0	3.8	0.74	0.9	0.52	0.24

The strong decrease of c_k with increasing mass number is due to the mass-dependent conduction between chambers C_1 and C_2 , and to the mass sensitivity of the spectrometer as a function of the resolution.

Fig. 4 shows the relation

$$\int \Gamma_4 dt = c_4 S_4 \int I_4 dt$$

for deuterium ($k=4$), yielding a fairly straight line with $c_4 S_4 = (1.3 \pm 0.4) \times 10^{27}$ Molecules/As. For hydrogen the result is $c_4 S_4 = (5.2 \pm 2) \times 10^{26}$ Molecules/As. The main error is the uncertainty of the effective pumping speed.

Figure 4: Calibration of the deuterium signal of the mass spectrometer in chamber 2 against the deuterium content in chamber 1 when filling chamber 1 with deuterium.

The calibration has been checked by Nuclear Reaction Analysis (NRA) of small tungsten coated graphite samples (surface 2.5×2 cm²) containing deuterium. The samples are bombarded by 790 keV ions ^3He , and the resulting protons are counted. Basing on the nuclear reaction $^3\text{He} + \text{D} \rightarrow ^4\text{He} + \text{p}$, the number of trapped deuterium atoms can be obtained. After these ion beam measurements the samples are analysed by TDS, yielding $\int I_4 dt$. Fig. 5 shows the results. From this figure we obtain $c_4 S_4 = (1.2 \pm 0.4) \times 10^{27}$ molecules/As, in fair agreement with the above calibration. Finally, the calibration has been tested by a Round-Robin-Experiment together with different laboratories [16]. The results deviate from those of the other laboratories by a factor < 2 .

2.2.3 Time constants of gas release

The gas release depends on temperature and heating time. If a given temperature level could be reached instantaneously, the desorption rate would increase with a time constant

Figure 5: Calibration of the deuterium signal of the mass spectrometer in chamber 2 against the deuterium desorption of heated samples with known deuterium content.

t_1 , reach a maximum, and then decrease with a time constant t_2 due to depletion of the trapping sites that deliver the gas at the selected temperature. In TDS-measurements one generally assumes that t_1 can be neglected against the heating time constant t_h , so the desorption curves are only determined by t_h and t_2 . In the present case with the large samples and slow heating rates this is not obvious, especially if the gas diffuses out from deeper zones in the bulk. Therefore, a special test has been carried out as depicted in fig. 6. Here the deuterium release of an 8×8 cm² graphite tile was measured at different heating powers (i. e. heating rates). With 100 W about 580 °C are reached. The heating is then stopped before the depletion becomes important (the latter being visible in the slight decrease of the desorption rate), and the sample is allowed to cool down. Next the sample is heated with 300 W, reaching an end temperature of 700 °C. This curve meets the preceding one at the same point where it had stopped (at 580 °C). After cooling down the experiment is continued in an analogous way with heating rates of 500, 700 and 1100 W, reaching end temperatures of 820, 950 and 1100 °C, respectively. Again, each curve meets the preceding one where these had been stopped. Thus, t_1 is smaller than the shortest heating time at 1100 W, the desorption follows the temperature nearly instantaneously.

2.2.4 Desorption maxima

Desorption maxima occurring at $dT/dt \geq 0$ indicate the depletion of trapping sites having binding energies smaller than a fixed value. With simple systems and with constant rise rate of the temperature, this behavior is used to measure binding energies E . The Redhead-theory [17] yields for the temperature T^* where the maximum occurs the relation

$$\frac{E}{kT^{*2}} = \frac{\nu}{\beta} \exp\left(-\frac{E}{kT^*}\right),$$

Figure 6: Deuterium signals vs. temperature with increasing heating rate. Between the runs the samples were allowed to cool down.

ν =constant being a characteristic frequency and β the rise rate of the temperature. At constant temperature, the time constant of depletion is

$$t_2 = \frac{1}{\nu} \exp\left(\frac{E}{kT}\right).$$

In the present work, the trapping system is rather complex, being controlled by deposition layers and variable surface roughness (see below). Therefore, no distinct values of E can be expected. Since also the rise rate β is not constant, no quantitative evaluation of desorption peaks is attempted. However, some qualitative conclusions can be drawn from the shape of the pressure-time records. Fig. 7 gives an example. Here the deuterium signal is shown for graphite and tungsten sprayed tiles from the inner and the outer divertor of ASDEX-Upgrade. The heating rate is the same for all curves. The graphite tiles produce the maximum at about 700 s where the temperature reaches 1000 K. Tungsten from the outer divertor has the maximum at 400 s, about 750 K, thus the deuterium is bound less strong. Interestingly, tungsten from the inner divertor exhibits a desorption shape analogous to the graphite, the maximum occurring at about 1000 K. This indicates that at these tiles trapping does not occur in the bulk but in carbon layers deposited on the surface.

2.2.3 Time constants of gas release

Above temperatures of 1300 K practically no more deuterium is released. This is shown in table 2 (tile S3 B2, outer divertor) for successive runs with increasing end temperature.

Figure 7: Deuterium signal vs. heating time at 600 W heating power for two graphite tiles and two tungsten coated graphite tiles, having different deuterium content. Sequence of materials from top to bottom: graphite (full circles) and tungsten coated tiles (open circles) having high deuterium content, graphite (full triangles) having medium deuterium content, and tungsten coated tiles (open triangles) having low deuterium content. Arrows: momentary temperature of the tiles.

Table 2: Released deuterium as a function of maximum temperature

	800 K (100 W)	950 K (300 W)	1100 K (500 W)	1200 K (700 W)	1300 K (900 W)	1400 K (1200 W)
% of total release	9	35	27	18	11.5	0.5

2.2.5 Deuterium compounds

To obtain the total deuterium inventory, not only released D_2 , but also compounds must be considered. Fig. 8 shows a spectrum of the gas desorbed from a graphite tile. It is rich of deuterium-hydrogen-carbon compounds that are difficult to decode. The main contributions are HD (mass 3) and CD_4 (mass 20). While the contribution of CD_4 is less than 10 %, HD cannot be neglected. The amount of released HD depends on the desorption of hydrogen. From numerous measurements the following relation has been deduced

$$\Gamma_3 = (1.0 \pm 0.4) \sqrt{\Gamma_2 \Gamma_4},$$

The relatively high standard deviation of this equation may be due to uncontrolled contribution of hydrogen molecules from internal surfaces of the vacuum system.

Because the analysing system is not in line of sight to the samples, the desorbed gas molecules must undergo many reflections at inner walls before reaching the mass spectrometer.

Figure 8: Spectrum of gas desorbed from a graphite tile.

These processes can enhance the fraction of the compounds, especially formed at the heat reflection shield around the samples. Fig. 9 indicates the effect. Here a small graphite sample is heated to about 2000 K. The deuterium signal decreases rapidly ($t_2 < 10$ s), thus the sample is almost completely exhausted. However, the HD-signal remains well over the initial value. Enhancing the heating power, there is a second increase of H_2 and HD that must be ascribed to a secondary source other than the sample. Probably at the shield some part of the desorbed deuterium is transformed to HD and then released with relatively high time constants. At temperatures > 1000 K deuterium can be desorbed in atomic form [18]. In this case compounds can be formed also at cold surfaces as found for CH_4 with atomic hydrogen [19].

Because of the dynamic measuring principle, the deuterium originating from such compounds will be counted twice, leading to overestimate the inventory. Therefore, the uncertainty of estimates including the compounds is higher than of conclusions based on the deuterium signal alone.

Another source of error, viz. memory and recycling effects in the mass spectrometer, is minimized in the present work because of the reduced pressure in the analysing chamber C_2 . Such effects will play a role at deuterium pressures $> 10^{-5}$ mbar.

2.2.6 Hydrogen

Most of the released gas is hydrogen (compare figs. 8 and 9). The H_2 released from the tiles is not necessarily characteristic for the gas trapping during plasma exposure, since it

Figure 9: Gas desorption when heating a small graphite sample.

can be absorbed during fabrication and handling prior to exposure in the tokamak, and even after exposure by handling and transport to the TDS-device, or simply by storing. Evidence for this is given in table 3, which shows the gas release from a sample at the first and second degassing, and then after storage at atmospheric pressure in a clean container for about three months. Relative signals are given (current-time integrals of the signals from the mass spectrometer).

Table 3: Gas release from a repeatedly degassed carbon tile (S6 C7 outer)

	H ₂	D ₂	H ₂ O
after plasma exposure	3.2×10^{-7} As	1.9×10^{-8} As	7.0×10^{-8} As
second degassing			
without exposure to air	2×10^{-8} As	3×10^{-10} As	4×10^{-10} As
after storing in air	8.7×10^{-8} As	3.6×10^{-10} As	2.3×10^{-8} As

Thus, storing the degassed sample at atmospheric pressure considerably increases the release of H₂, but not of D₂. Since H₂O behaves analogously to H₂, the main reason for the increase will be the absorption of water vapor from the atmosphere. In a second case, after five months a degassed graphite sample showed 1.5×10^{-7} As hydrogen and 3.3×10^{-8} As water vapor. Finally, a degassed graphite tile was weighed beginning after 10 minutes exposure to air. After four days the weight had increased by 3 mg. Interpreting this as absorption of water vapor, the corresponding number of hydrogen molecules would be 10^{20} .

Analysing numerous tiles the amount of released hydrogen molecules is found to be nearly constant: $(3.8 \pm 0.3) \times 10^{22}$ hydrogen molecules/m² (if one takes only the front side of the tile), whereas D₂ varies by more than two orders of magnitude. This is also shown in fig. 10 where for a number of tiles the desorption of hydrogen molecules is compared with the release of deuterium molecules. The constancy of hydrogen desorption indicates that this gas is dominated by absorption processes other than plasma exposure. Hence, the hydrogen signals are not well suited for analysing gas trapping in the tokamak.

Figure 10: Desorption of H₂-molecules vs. D₂-desorption.

Figure 11: Decrease of deuterium signal due to gas diffusion when heating a Ti-sample.

2.2.7 Deuterium adsorption without plasma discharges

To estimate the contribution of deuterium absorption by exposure to the gas alone (without plasma), graphite and, for comparison, titanium samples were exposed to deuterium at elevated temperatures (800-1300 K). Fig. 11 shows deuterium absorption by a massive titanium tile with a front surface of 150 cm². The previously degassed sample is heated at a constant flow of deuterium gas (equilibrium level 10⁻⁴ mbar). This causes a decrease of the deuterium pressure corresponding to the absorption of (3-4)×10²⁰ deuterium molecules. The hot tungsten filament alone absorbs about (5-7)×10¹⁸ deuterium molecules, and can, therefore, be neglected. When heating the reflecting molybdenum shield separately (i.e. with hot filament and hot shield) the same absorption is found as with the filament alone. When heating previously degassed graphite tiles in deuterium, the absorption amounts to < 10¹⁹ deuterium molecules. Hence, diffusion of deuterium into hot graphite is < 5×10²⁰ deuterium molecules/m².

Table 4: Hydrogen and deuterium released from divertor tiles of ASDEX-Upgrade

TILE	MATERIAL	PERIOD	Released H ₂ - molecules/m ²	Released D ₂ - molecules/m ²	Total number of D-atoms/m ²
S5 C5 inner	C	1995	5.0×10 ²²	1.8×10 ²²	7.2×10 ²²
S16 C6 outer	C+W (vacuum)	1995	3.6×10 ²²	6.1×10 ²⁰	6.8×10 ²¹
S8 C2 inner	C+W (argon)	1995	5.7×10 ²²	1.2×10 ²²	5.5×10 ²²
S7 C5 outer	C+W (argon)	1995	4.4×10 ²²	2.2×10 ²¹	1.6×10 ²²
S7 B5 outer	C+W (argon)	1995	3.9×10 ²²	4.1×10 ²¹	2.3×10 ²²
S8 D2 inner	C+W (argon)	1995	4.2×10 ²²	2.4×10 ²²	8.6×10 ²²
B3 outer	C, polished	1995	3.2×10 ²²	5.0×10 ²¹	2.5×10 ²²
B4 outer	C, polished and degassed	1995	3.3×10 ²²	2.4×10 ²¹	1.5×10 ²²
S7 D5 inner	C+W (vacuum)	1995	5.0×10 ²²	2.4×10 ²²	9.0×10 ²²
S16 B6 outer	C+W(vacuum)	1995	3.0×10 ²²	1.1×10 ²¹	9.1×10 ²¹
S6 A7 outer	C	1995	4.1×10 ²²	1.3×10 ²¹	1.1×10 ²²
S6 B7 outer	C	1995	2.7×10 ²²	3.2×10 ²¹	1.8×10 ²²
S1 B6 outer	C+Ti	1995	4.7×10 ²²	1.7×10 ²¹	1.4×10 ²²
S6 C7 outer	C	1995	2.8×10 ²²	5.0×10 ²¹	2.4×10 ²²
S14 B8 outer	C+W(vacuum)	1996	2.8×10 ²²	2.6×10 ²⁰	3.8×10 ²¹
S14 C8 outer	C+W(vacuum)	1996	2.7×10 ²²	1.3×10 ²¹	9.7×10 ²¹
D6 inner	C	1996	4.0×10 ²²	2.8×10 ²²	9.6×10 ²²
B outer	C+W (vacuum)	1996	2.8×10 ²²	2.2×10 ²⁰	3.4×10 ²¹
C6 inner	C	1996	5.4×10 ²²	2.4×10 ²²	9.1×10 ²²
S3 A2 outer	C	1996	3.4×10 ²²	1.8×10 ²¹	1.3×10 ²²
S3 B2 outer	C	1996	5.0×10 ²²	6.3×10 ²¹	3.4×10 ²²
S3 C2 outer	C	1996	2.7×10 ²²	3.0×10 ²¹	1.7×10 ²²
S3 D2 outer	C	1996	3.6×10 ²²	7.8×10 ²¹	3.6×10 ²²
S3 A5 inner	C	1996	3.2×10 ²²	8.7×10 ²⁰	8.1×10 ²¹
S3 B5 inner	C	1996	5.0×10 ²²	5.4×10 ²¹	3.1×10 ²²
S3 C5 inner	C	1996	4.9×10 ²²	2.1×10 ²²	8.0×10 ²²
S3 D5 inner	C	1996	4.2×10 ²²	2.4×10 ²²	8.6×10 ²²
S7 C5 inner	C	1996	3.4×10 ²²	4.6×10 ²⁰	5.7×10 ²¹

2.2.8 TDS-results for complete tiles

Table 4 lists the gas release from complete divertor tiles. C means pure graphite, C+W (vacuum) means W-coated graphite tiles, the coating being made by plasma spraying in vacuum, and C+W (argon) means fabrication of W-coatings by plasma spraying in argon. As desorbing area the front surface of the tiles has been taken (somewhat questionable for hydrogen). The total number of D-atoms is calculated from the contributions of D₂ and HD. It is subjected to the uncertainties discussed in section 2.2.5.

2.3 Surface morphology

To find the dominating trapping mechanisms, the depth distribution of the deuterium must be analysed. In [15] it is concluded that in the region of intensive plasma contact most trapping is due to diffusion up to a depth of 100 μm . This analysis, however, does not consider the surface roughness. Fig. 12 shows scanning electron micrographs from graphite tiles, left: outer divertor, row C, being subjected to prevailing erosion; right: inner divertor, row D, being subjected to prevailing deposition. In the erosion zone, the roughness is greatly enhanced, exhibiting structures with a size of about 100 μm . At the bottom of the holes and valleys gas can be adsorbed or buried by codeposition in a virtual depth of 100 μm without necessitating diffusion. The rough structure screens these sites, so contaminants can survive the contact with the plasma. The comparison of both tiles demonstrates that the roughness is variable in dependence on the location of the exposure. Thus, to analyse trapping mechanisms, the surface morphology must be taken into account.

Figure 12: SEM-pictures of graphite tiles from the outer divertor, row C (a) and the inner divertor, row D (b).

2.4 Surface Analysis

A commercial surface analysis station (PERKIN ELMER Φ 5600) is used that allows Auger-Energy-Spectroscopy (AES), Secondary-Ion-Mass-Spectroscopy (SIMS) and ion

beam sputtering of the large tiles. AES allows a local resolution of $1\ \mu\text{m}$, and SIMS uses a $10\ \text{keV}\ \text{O}_2$ beam of about $5\ \mu\text{m}$ diameter for imaging. With these methods, for tungsten coated tiles the deposition of carbon and boron can be studied. For graphite tiles, the distribution of boron can be used as indicator for deposited layers. Fig. 13 shows the boron amount obtained by integrating AES-depth profiles as a function of the poloidal coordinate s from the inner divertor to the outer divertor for graphite tiles. For comparison, the figure also presents the poloidal distribution of deuterium released by TDS for complete and cut graphite tiles (long and small horizontal bars, respectively). The correlation to the boron deposition is evident. Figs. 14 and 15 show depth profiles of tungsten and graphite tiles taken by SIMS. Tungsten tiles from the inner divertor exhibit thick contamination layers, having a width of about $5\ \mu\text{m}$ (fig. 14 a). These layers consist of boron, carbon, and deuterium with a nearly constant ratio along the depth profile. The same holds for boron and deuterium at the graphite tile, fig. 15 (a). In this case, the bulk carbon dominates at depths $> 5\ \mu\text{m}$, but at the surface the carbon contamination seems to have a similar ratio to boron as with the tungsten tiles. This is even more obvious from the AES-measurements that allow quantitative estimates.

Figure 13: Poloidal scans of boron deposition and deuterium trapping in graphite tiles, left: integrated AES-depth profile of boron, right: TDS of deuterium for complete and cut tiles (long and short horizontal bars, respectively).

Thus, the dominant amount of deuterium is trapped by codeposition with carbon and boron. On the inner divertor tiles, contamination layers are present with a thickness of several μm , whereas on the outer divertor tiles (erosion zone) most of the contaminants are located in surface depressions with amounts up to several 10^{22} atoms/ m^2 . SIMS-imaging reveals that the lateral distribution of the contaminants is not uniform. Fig. 16 gives an example. Here the lateral distribution of boron and deuterium on a graphite tile is shown after removing $12\ \mu\text{m}$ by milling. Obviously the contaminants are concentrated in pores, holes and cracks determined by the surface morphology (compare the roughness structure in fig. 12).

Group II refers to the erosion zone of the outer divertor with amounts up to $(2-3) \times 10^{22}$ deuterium atoms/ m^2 . Group III is a transition zone, and in group IV deposited layers prevail. Here we have $(7-8) \times 10^{22}$ deuterium atoms/ m^2 , independently of the material.

Figure 14: SIMS-depth profiles for tungsten tiles. Left: inner divertor, right: outer divertor. The depth scale holds for tungsten. For the contamination layers it must be multiplied by a factor of about two due to the higher sputter rate.

Figure 15: SIMS-depth profiles for graphite tiles. Left: inner divertor, right: outer divertor. The depth scale holds for the bulk graphite. For the contamination layers it must be multiplied by a factor of about two due to the higher sputter rate.

Figure 16: SIMS-image of the lateral distribution of boron and deuterium in graphite at a depth of $12\ \mu\text{m}$. Field of view $1\times 1\ \text{mm}^2$. Left: boron image, right: deuterium image.

3 Discussion

Table 5 lists average values of deuterium desorption according to the position of the tiles. Here the dominant process of plasma-target interaction (third column) was deduced from the surface analysis.

Table 5: D_2 - release summarized from table 4 (mean values)

GROUP	POSITION	CHARACTERIZED BY	MATERIAL	D_2/m^2	D-atoms/ m^2	No. of TILES
I	A inner, outer	minor deposition	C	1×10^{21}	9.4×10^{21}	3
II	C outer	prevailing erosion	W (vacuum)	1×10^{21}	9.4×10^{21}	2
			W (argon)	2×10^{21}	1.4×10^{22}	1
			C	4×10^{21}	2.3×10^{22}	2
III	B, D outer	partial deposition	W (vacuum)	5×10^{21}	2.7×10^{22}	3
			W (argon)	4×10^{21}	2.3×10^{22}	1
			C	5×10^{21}	2.7×10^{22}	5
IV	B, C, D inner	deposition	C	1.7×10^{22}	6.4×10^{22}	7
			W (vacuum)	2.4×10^{22}	8.4×10^{22}	1
			W (argon)	1.8×10^{22}	6.7×10^{22}	2

In table 5, the measured values are arranged in four groups. Group I, distinctly above the detectable limit with about 10^{22} deuterium atoms/ m^2 , indicates the possibility of gas retention without much plasma contact. The experiments with hot graphite tiles in deuterium have shown that temperature controlled gas diffusion into the interior amounted to $< 5\times 10^{20}$ deuterium molecules/ m^2 . Weak contact with energetic plasma particles could have some larger effect.

Group II refers to the erosion zone of the outer divertor with amounts up to $(2-3)\times 10^{22}$ deuterium atoms/ m^2 . Group III is a transition zone, and in group IV deposited layers prevail. Here we have $(7-8)\times 10^{22}$ deuterium atoms/ m^2 , independently of the material.

Thus, the deposition zone yields high deuterium release. In group II the deuterium release increases in the sequence *vacuum sprayed coatings* - *argon sprayed coatings* - *graphite without coatings*. This can be attributed to the different surface morphology (mainly roughness) that influence surface contamination. The influence of the material entirely disappears in groups III and IV, as well as in the desorption-time curves and in the absolute amount of desorbed deuterium. This indicates that here the deposition layers dominate the deuterium retention. This conclusion is supported by the surface analysis (figs. 13-16). More details on the results of the surface analysis will be published in a forthcoming paper.

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