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Deuterium trapping in divertor tiles of ASDEX-Upgrade

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Abstract

The deuterium inventory of divertor target tiles used in ASDEX-Upgrade for up to 2000 discharges has been analyzed by thermodesorption spectrometry. In addition, surface analysis techniques as auger electron spectrometry, secondary ion mass spectrometry, nuclear reaction analysis, electron microscopy and optical surface profilometry have been applied for investigating erosion and deposition phenomena. The original plasma facing surfaces were graphite (EK98) and plasma sprayed tungsten, respectively. The total deuterium inventories have been found to vary between 4×10^{21} D-atoms/m^2 and 3×10^{23} D-atoms/m^2. The deuterium is contained in a deposit on the surface of the graphite and tungsten tiles consisting mainly of carbon, boron and the hydrogen isotopes. There is strong indication that morphological effects influence impurity deposition, deuterium trapping and re-erosion of the contamination.

Keywords: ASDEX-Upgrade; Carbon-based materials; Deuterium inventory; Divertor targets; Erosion/deposition

1. Introduction

The uptake and release of hydrogen isotopes at the plasma–facing components in magnetic confinement fusion devices affects the working gas recycling, the plasma behavior and the tritium inventory [1]. This attracts considerable interest to the investigation of hydrogen trapping during plasma exposure.

The most intensive plasma material interaction occurs on limiters and divertor plates. Postmortem analysis of such components have shown that the hydrogen isotopes are mainly contained in deposits see e.g. [2–4]. In a recently published paper it was reported that diffusion dominates the gas retention in plasma exposed graphite divertor tiles of ASDEX-Upgrade [5].

The authors investigated the total deuterium inventory of small samples cut out from the graphite tiles. However, in order to conclude on processes involved in hydrogen 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 this paper further results on the deuterium inventory, the impurity analysis and the surface morphology of divertor tiles of ASDEX-Upgrade are presented.

The original plasma–facing material of the target tiles were graphite (EK98) and tungsten, respectively. The tungsten was plasma sprayed with a layer thickness of 500 μm on graphite.

2. Experimental details

2.1. Divertor target tiles

The analyzed tiles stem from the lower inner and outer divertor. The investigated graphite tiles (Plansee AG) were used from the beginning of the ASDEX-Upgrade experiment in 1991 until summer 1995. During this period about 2000 plasma discharges and 13 boronization procedures for wall conditioning were performed. In 1995 the graphite tiles of the inner and outer divertor near the strike point position were replaced by tungsten covered tiles. About 800 discharges and 6 bo-

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ronization procedures were performed in the successive period before all divertor tiles were dismounted in summer 1996. During the boronization procedures the wall of the main chamber was covered with an a-B : D layer of about 40 nm. The amount of boron deposited on the target tiles during boronization is not known.

Most of the plasma discharges had a deuterium fraction between 60 and 80%. About 50% (graphite tiles) or 60% (tungsten tiles) of the discharges were NBI-heated. Generally, the mean surface temperature of the tiles during neutral beam heated discharges did not exceed 500 K (see also [5]). During disruptions and ELM's the surface temperature exceeded 1000 K locally. However, such events were scarce.

The torus of ASDEX-Upgrade consists of 16 segments with poloidal divertors at top and bottom. Until now, all discharges were performed in single null configuration and lower divertor operation. The lower divertor tiles were poloidally arranged in rows A through D or E [5,6]. In the following the poloidal location is given precisely by the coordinate *s*. Most intensive plasma contact was found to occur by thermography at the expected strike point positions. This position varied between 280 mm and about 320 mm for the inner divertor and between 600 mm and 700 mm for the outer divertor. The densities of the particle fluxes hitting tiles near the strike points were some 10^{22} particles/m^{2}s, and the total fluences of the hydrogen isotopes to the graphite targets were of the order of 10^{26} atoms/m^2 at these positions.

2.2. Analysis methods

The following surface analysis techniques were applied to investigate the surface contamination of the target tiles: auger electron spectroscopy (AES) combined with sputter depth profiling, secondary ion mass spectrometry (SIMS) with a primary beam of 10 keV O_2-ions and nuclear reaction analysis (NRA) with the reactions ^{3}He(d,p)α at 790 keV and ^{11}B(p,α)^{8}Be at 690 keV. The diameter of the analyzing beam was l μm (AES), about 5 μm (SIMS) and 1 mm (NRA). This allowed to take 2D-images of the deuterium and impurity contamination with a lateral resolution in the μm-range by SIMS and local AES-analysis. The SIMS-investigations have been made to get information on the spatial distribution of the contamination (in depth and area). The other methods (AES and NRA) are suitable for obtaining quantitative results.

For the morphological investigations electron microscopy and optical surface profilometry were used. The surface profiles were taken with laser beam positioning and diameter of 1 μm.

The thermal desorption measurements have been performed in a vacuum system consisting of two separately pumped vessels. Samples of different size can be heated in a 40 l-chamber by electron bombardment with a power up to 1.75 kW. This results in heating rates between 1 and 100 K/s depending on the size of the samples. The largest samples (80×80 mm^2) could be heated up to temperatures of 1500 K and smaller ones to even higher temperatures. Even with the largest samples the temperature distribution was uniform within 50 K.

A fraction of the desorbed gas reaches the analysis chamber through a flow conduction of 1 l/s. This separated chamber is equipped with a quadrupole mass spectrometer for gas analysis. Further information on experimental aspects, the calibration procedure and detailed results are described elsewhere [6]. Here, data on the total amount of the released deuterium are presented which is expected to be accurate within a factor of 2–3. This accuracy is not well defined but takes into account that gas recycling at the chamber walls is somewhat uncertain. In a separate experiment with thin deuterium containing contamination layers (thickness <0.3 μm) on polished graphite surfaces (EK 98) it was demonstrated that the deuterium amounts derived from TDS- and NRA-measurements agree within 20%. The TDS-investigations on ASDEX-Upgrade divertor tiles have been done with complete tiles and cut pieces. Measurements with complete tiles have the advantage that there is no contamination by cutting procedures and the total gas inventory of the tiles including the sides of the tiles can be determined. However, in order to increase the local resolution in these measurements some graphite tiles were cut.

3. Results

Visual inspection of the target tiles reveals strong morphological modifications of the graphite surface after plasma exposure. In particular, some fresh and many older arc tracks were visible. Prior to the plasma exposure the graphite tiles had a mean surface roughness of 2–3 μm measured by laser profilometry. After the plasma exposure this value of surface roughness was hardly changed for large regions of the inner divertor and for regions far from the strike point of the outer divertor. In contrast, graphite target tiles exposed near the strike point of the outer divertor showed an enhanced surface roughness in the order of 10 μm (see below). Fig. 1 shows a photograph of the tile surface from the outer divertor taken by electron microscopy. Apparently, plasma erosion by arcing and sputtering produced microcraters, microcracks and open microcavities at the graphite surface.

The applied surface analysis methods indicate that the surface of the graphite and tungsten tiles is covered with a deposit of varying amount. The deposited material has a similar composition for both target materials and consists mainly of carbon, boron and the hydro-

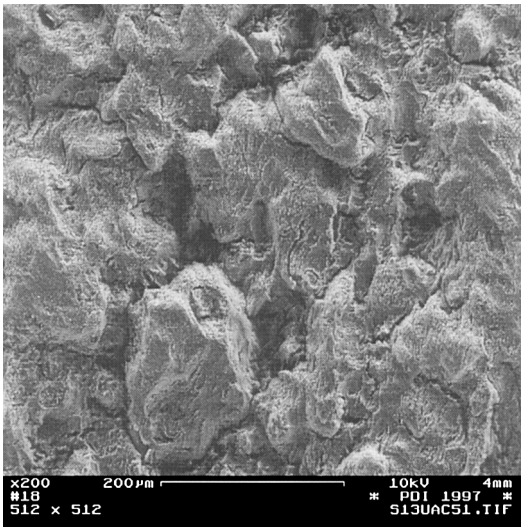

Fig. 1. SEM photograph of the graphite surface from target tiles of ASDEX-Upgrade near the strike point (outer divertor).

genic isotopes as measured by AES and SIMS. Fig. 2 shows as an example the depth distribution of the deposited species measured by SIMS for a tungsten target tile of the inner divertor. Similar depth distributions of boron, hydrogen and deuterium were observed by SIMS at the surface of graphite tiles of the inner divertor. In contrast to the deposits found on C^{13}-collector probes exposed in the SOL-plasma of the main chamber and the divertor plasma containing boron with a concentration of about 1 percent [7] the relative boron concentration in the deposit on the tungsten and graphite target tiles measured by AES is about 30%. Fig. 3 shows the deposited boron amount on the graphite and tungsten target tiles of the inner and outer divertor in dependence on the poloidal coordinate. These values have been obtained by integrating AES-depth profiles (up to depths

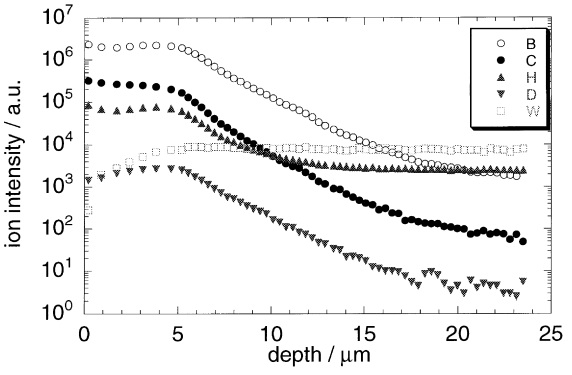

Fig. 2. SIMS-depth profiles of the contamination on tungsten sprayed target tiles from the inner divertor ($s = 250$ mm).

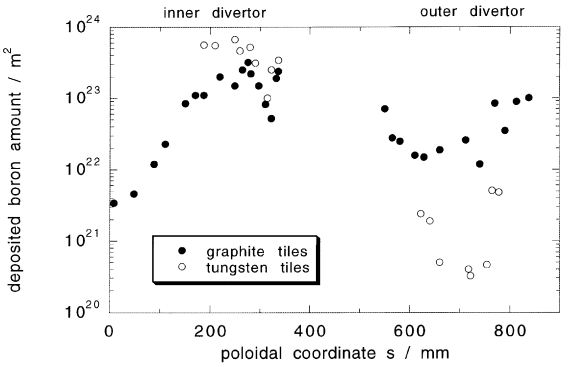

Fig. 3. The poloidal distribution of the boron amount on graphite and tungsten tiles obtained by integrating depth profiles measured by AES. The values of the tungsten tiles were observed for the unshadowed region.

of 25 μm) measured with a defocused electron beam to reduce lateral effects (see below). It has to be noted that B-and D-amounts derived from NRA-measurements are up to a factor of 10 lower than the values of Fig. 3. This is due to the limited analysis depth of the NRA-method of about l μm in view of the thickness of the deposits and the morphology of the graphite surface in eroded regions with its high roughness.

Fig. 3 shows a thick contamination on inner divertor targets (up to 4×10^{23} B-atoms/m^2 at $s = 280$ mm) with the exception of a small zone at the strike point at s of about 310 mm and regions far from this position. On inner tungsten target tiles up to 8×10^{23} C-atoms/m^2 were found. The tungsten tiles showed an additional toroidal dependence of the contamination on each tile. These tiles were somewhat tilted in toroidal direction to avoid leading edges. Hence, shadowing effects by adjacent tiles are expected [8] and caused a continuos increase of the contamination towards the shadowed edge.

Large regions of the outer divertor tiles show less contamination with strong differences for both target materials (see Fig. 3). In near strike points region with s between 550 mm and 750 mm the deposited boron amount on the surface of the tungsten tiles is about 10^{21} B-atoms/m^2 while it is found at least to be one order of magnitude larger for the graphite tiles. The higher contamination of the graphite tiles is due to the surface morphology (see below).

The observed high surface roughness of the graphite tiles has strong influence on the lateral distribution of the contamination. In fact, 2D-images taken by SIMS show only a nearly uniform lateral distribution of the deposited species at contamination higher than 5×10^{22} B-atoms/m^2 (inner divertor tiles). For thinner contamination (outer divertor tiles) a strongly inhomogeneous distribution for boron and deuterium on the graphite surface is found. The contamination is found in surface

depressions like microcraters, but can also occur on surfaces of cracks and pores. At these locations the deposits are protected against re-erosion. Using AES with a focused electron beam (diameter about 1 μm) it is found that the material deposited in these craters consists also of carbon and boron with a relative concentration ratio of 2/1. The lateral distribution of boron is maintained after heating the samples up to 1500 K indicating that diffusion of boron is not important at this temperature.

The observed mass spectra showed that released deuterium was dominantly detected as HD-molecules and D_2-molecules with a maximum of the desorption rate at 1000 K for graphite. The number of D-atoms detected as HD-molecule exceeded that detected as D_2-molecule by a factor up to 5. The contribution of CD_4 and other deuterium containing molecules to the desorbed deuterium amount was about 10% [6] and was neglected. Unfortunately, the inventory of the hydrogen isotope caused by the plasma exposure could not be determined due to water adsorption at air exposure [6]. Fig. 4 shows the poloidal distribution of the total deuterium amounts from the inner and outer graphite divertor tiles. Values obtained at outgassing of complete divertor tiles are given with a broad interval corresponding to their poloidal extent.

In order to increase the local poloidal resolution some tiles were cut into pieces of 10 mm width. The D-amount released from the cut samples are also shown with a shorter poloidal interval. The results from complete tiles and cut samples agree well indicating that contamination during sample preparation is not important. Comparing the D-inventories of the cut samples with and without tile edges, those containing the tile edges with sides shadowed by adjacent tiles show slightly enhanced values indicating deuterium trapping down the sides of the tiles. Deposits into gaps between the tiles were also observed in JET [3].

As it can be seen from Fig. 4 the deuterium inventories of complete graphite tiles near the strike points are 2×10^{22} D-atoms/m^2 (outer divertor) and 10^{23} D-atoms/m^2 (inner divertor). TDS measurements on small samples cut out from graphite tiles made in an other laboratory [5] gave somewhat higher deuterium inventories (factor 2–4). This discrepancy is not clear but can be partly attributed to different sample locations. For a given poloidal position complete tiles showed a toroidal variation of the deuterium inventory and the thickness of the deposit by a factor of 3.

In order to investigate the areal and the depth distribution of the trapped deuterium in more detail surface layers of graphite tiles were locally removed by milling and then heated for degassing. Fig. 5 shows a surface profile of an outer graphite divertor tile after milling of three grooves with different depths. These results indicate that the surface roughness of the plasma exposed surface is of the order of 10 μm and the cracks and pores occur even at larger depths. Fig. 6 shows results from the TDS-measurements on graphite samples from areas of

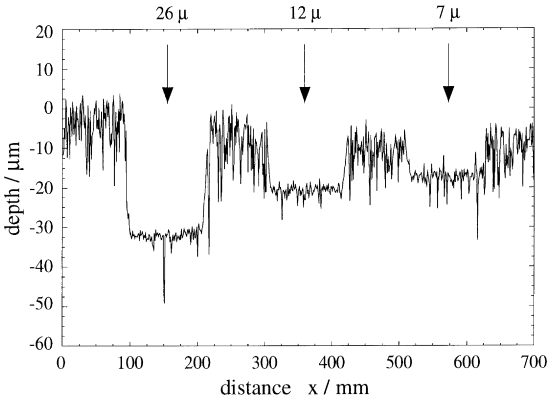

Fig. 5. Surface profile of an outer divertor graphite tile after milling of three grooves with different depth.

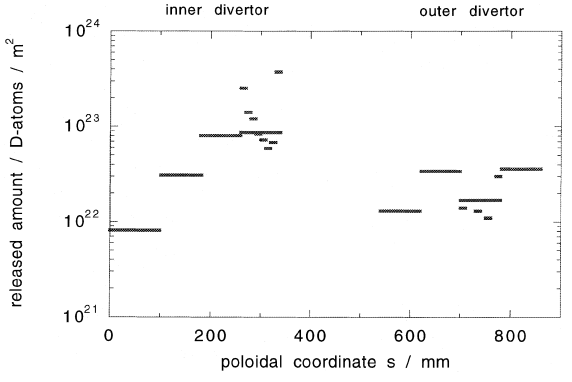

Fig. 4. The poloidal distribution of the deuterium inventory in graphite tiles measured by TDS.

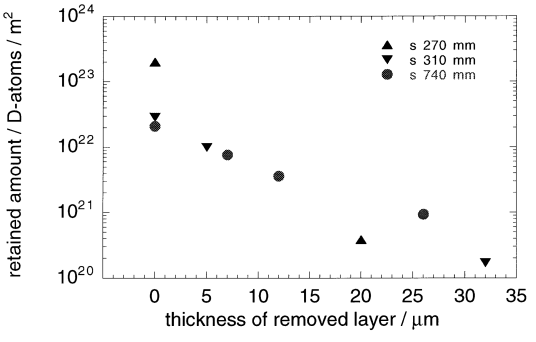

Fig. 6. Deuterium inventory measured by TDS in samples from graphite divertor tiles after removing surface layers by milling.

different grooves for inner and outer divertor tiles. This figure demonstrates clearly that the dominant amount of deuterium is trapped close to the surface. Only about 10^{21} D-atoms/m^2 were found after removing surface layers with a depth larger than 20 μm. This amount is less than 1% (inner divertor, $s = 270$ mm) or less than 10% (outer divertor, $s = 740$ mm) of the total deuterium inventory found in the neighboring unmilled material.

4. Discussion

The deuterium inventories in the graphite target tiles of ASDEX-Upgrade measured by TDS (Fig. 4) are much higher than expected for this material by implantation [9], all the more, if it is taken into account that due to the plasma isotopic composition at least the same amount of hydrogen is additionally trapped. In fact, exposing this material in a low temperature pure deuterium plasma with T_e of about 5 eV representing the divertor plasma of ASDEX-Upgrade without significant impurity contamination results in trapped deuterium amounts up to 1.7×10^{21} D-atoms/m^2 at an incident fluence of 5×10^{25} D/m^2.

A comparison of the deuterium inventories (Fig. 4) and the deposited boron amount (Fig. 3) shows a strong correlation between both quantities. In particular, a thick contamination layer on inner divertor tiles with boron and deuterium amounts in the order of 10^{23} atoms/m^2 has been found. Moreover, this contamination also contains carbon and hydrogen as measured by AES and SIMS (Section 3). The relative high boron concentration in the deposit on the surfaces of the target tiles (compared to the collector probe deposit) may result from boron deposition during boronization procedures or from boron transport from the vessel wall to the target plates during plasma discharges. Due to preferred erosion of carbon by chemical effects the composition of the deposit on the target tiles is not necessarily representative for the relative concentrations of the deposited plasma contaminants even if the contamination is caused during the plasma discharges.

Less contamination with boron and deuterium is found in a narrow region at the strike point position of the inner divertor indicating stronger re-erosion of deposited material locally.

The differences in deuterium trapping and impurity deposition between the inner and outer divertor target plates of ASDEX-Upgrade are significant and were also found on other tokamaks [3,4]. This can be explained by higher plasma temperatures and stronger erosion in the outer divertor. Such poloidal asymmetries in plasma temperature and heat fluxes have been observed by visible spectroscopy and thermography [10,11]. In addition, the change of the strike point position on the outer divertor caused erosion in a broader region (s between 550 mm and 750 mm) with less contamination. Remarkably, even in this eroded region boron and deuterium amounts larger than 10^{22} atoms/m^2 were found on the graphite tiles (Figs. 3 and 4). This contamination is not a closed surface layer but is found at deeper locations of the graphite surface where it is more protected against re-erosion. Protruding parts of the surface are predominantly eroded. In this way the surface roughness has a significant influence on erosion, deposition and deuterium trapping during plasma exposure. Simultaneous nonuniform carbon deposition and gradual erosion of a boron layer on graphite were also observed in impurity collection experiments on TEXTOR [12].

5. Conclusion

Applying thermodesorption in combination with surface analysis methods we find clear indications that deuterium trapping in divertor target tiles of ASDEX-Upgrade is dominated by codeposition with carbon and boron. Dominant deuterium inventories (>10^{23} D-atoms/m^2) are found in several μm thick contamination layers on inner divertor tiles consisting mainly of carbon, boron and the hydrogenic isotopes. However, contamination with boron and deuterium excess of 10^{22} atoms/m^2 has also been found on the surface of graphite tiles of the outer divertor with prevailing erosion. This contamination is located in surface depressions of μm-scale and is protected against re-erosion by the surface morphology. Plasma erosion processes like arcing and sputtering enhanced the surface roughness of the graphite material.

Removing surface layers of several μm by milling it could be demonstrated that the dominant deuterium amount is trapped close to the surface. The contribution of diffusion effects along grain boundaries to the deuterium inventory in graphite is estimated to be smaller than 10% in eroded regions of the outer divertor and less than 1% in deposition zones of the inner divertor.

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