



## In situ detection of hydrogen retention in TEXTOR by laser induced desorption

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### ABSTRACT

Long term tritium retention is one of the most critical issues for ITER and future fusion devices. While a global analysis of the T retention can be made by T accountancy in the activated phase of ITER, fuel retention and control must be already addressed in the non-activated phase, to identify the mechanism, location and amount of retention, its dependence on plasma and wall conditions and to qualify T retention mitigation and control techniques. For this purpose a new diagnostic, laser induced desorption spectroscopy of retained fuel has been developed and applied in TEXTOR. Hydrogen isotopes are desorbed from re-deposited layers on graphite plates by rapid heating with laser radiation. The released particles have been quantified in situ by spectroscopic measurements of hydrogen lines in a tokamak plasma. The results were validated by ex situ analysis of the hydrogen content of deposited a-C:H layers.

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### 1. Introduction

The operation of ITER will be substantially dependent on the interaction of plasma facing materials with the edge plasma. Erosion and deposition are the dominating effects that determine lifetime of the first wall as well as the main plasma parameter. Co-deposition of tritium in deposited layers of eroded material can lead to interruption of operation due to safety reasons [1]. One method to measure the retained tritium in ITER is the global gas balance. This method works only in the activated phase and measures the retention in a global way while it is expected that tritium in deposited layers inside the vessel is distributed inhomogeneously. Therefore the space resolved identification of material deposition and T retention are most important and necessary for effective cleaning procedures [2].

Laser induced thermal desorption spectroscopy (LIDS) is proposed as a diagnostic that in situ can monitor the inventory of hydrogen isotopes in deposited layers on plasma facing components [3,4]. A well defined spot at the surface is heated rapidly in a few ms by absorption of laser light that leads to thermal desorption of trapped hydrogen in thin layers. The released hydrogen enters the edge plasma where it is dissociated and excited. The intensity pulse of Balmer line radiation ( $H_{\alpha}$ ) above background is detected from which the fuel inventory is evaluated applying corresponding conversion factors. The S/XB factors are only weakly dependent on plasma parameters [5,6]. Thin layers are partly transparent for laser radiation and power absorption happens in the bulk. For a 1 ms laser pulse the heat wave penetrates about

100  $\mu\text{m}$  and deposited layers on bulk material must have good heat conduction to achieve the necessary temperatures for desorption.

The release process of hydrogen isotopes from amorphous hydrocarbon (a-C:H) layers by laser induced desorption and the conversion factors in dependence on plasma parameter were investigated and published in previous papers [3,4,7]. The measurement requires surface temperatures up to 1800 K that is achieved by absorption of laser radiation with power densities of 70  $\text{kW}/\text{cm}^2$  for about 1.5 ms. For the used laser spot diameter of about 2.5 mm at the surface the expansion of the desorption area by heat propagation during this short pulse duration is  $\leq 20\%$ . Under these conditions more than 95% of hydrogen in thin ( $< 1 \mu\text{m}$ ) amorphous hydrocarbon layers is released in a single laser pulse. It has been shown previously that the majority of desorbed particles are leaving the surface as molecules. With rising temperatures first hydrocarbon molecules are released followed by hydrogen molecules, which represent the majority ( $> 80\%$ ) of desorbed particles. Only a very small fraction of atomic hydrogen was observed at temperatures above 1500 K.

The majority of the molecules ( $\text{H}_2$ ,  $\text{D}_2$ , HD, hydrocarbons) that penetrate into the tokamak edge plasma are first ionized and then dissociated. In this process only the generated neutral atoms can be excited and emit Balmer lines before they will be ionized as well. The ionized fraction of all kind of dissociated molecules is not visible and considered in a correction factor determined experimentally.

This paper presents LIDS measurements of H isotopes inventory in amorphous hydrocarbon layers deposited on a graphite roof limiter inserted in the bottom vacuum lock at TEXTOR. Deposits were formed both by ohmic and neutral beam heated plasmas. First the experimental arrangement of the LIDS diagnostic is described.

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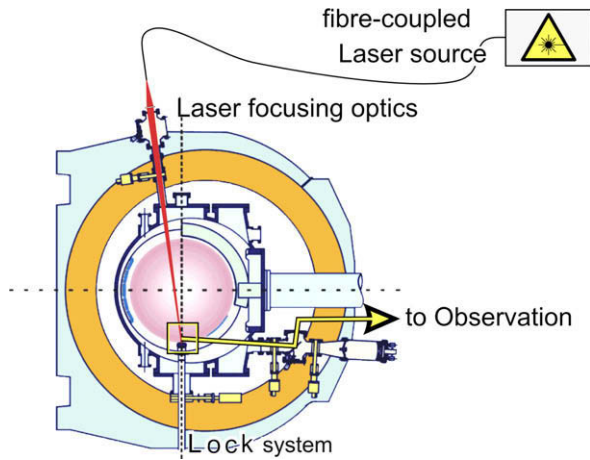


Fig. 1. Experimental arrangement of laser induced desorption spectroscopy measurement at TEXTOR.

After this, results obtained in situ at TEXTOR are compared with measurements performed ex situ after lock out from the discharge chamber. Finally the possible application of this diagnostic at ITER under its geometrical constraints and hazardous environment is described.

## 2. Experimental arrangement

The experimental arrangement for laser induced desorption spectroscopy at TEXTOR is shown in Fig. 1. The light of a pulsed Nd:YAG laser with 20 kW power at 1064 nm wavelength is transferred through the concrete shielding via a 400  $\mu\text{m}$  core diameter fibre over a distance 35 m to a port on top of the TEXTOR vessel and collimated by optical lenses in a distance of 1.75 m onto the surface of a graphite target, introduced by the TEXTOR material test facility from the bottom side. The targets were mounted on both sides of a roof limiter with 36° inclination of the surface in respect to the toroidal field lines. The tip of the limiter was located at radial plasma position of 47.5 cm, 15 mm outside the last close flux surface at 46 cm. A reduction of the inclination angle to 17° at the innermost 9 mm of the probe plates minimises overheating of the probe tip. The length of the investigated carbon plates are 63 mm that covers a radial range from 47.5 to 51 cm of the plasma radius. The spot size on the graphite surface exposed by the laser light was 7 mm<sup>2</sup>. Between discharges the laser beam could be shifted diagonally across the surface within  $\pm 0.3$  mm accuracy by moving the position of the fibre output window. Fig. 2 shows the limiter after

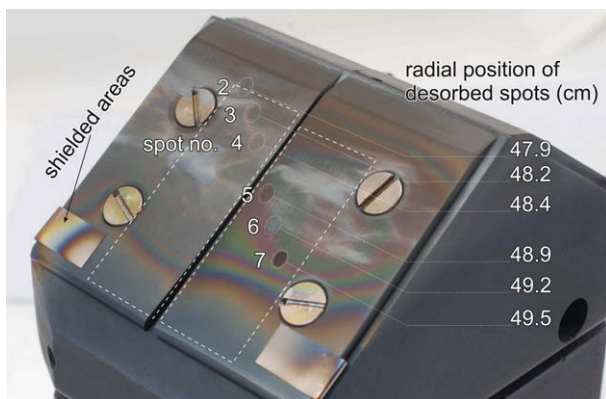


Fig. 2. Picture of plasma exposed roof limiter in TEXTOR.

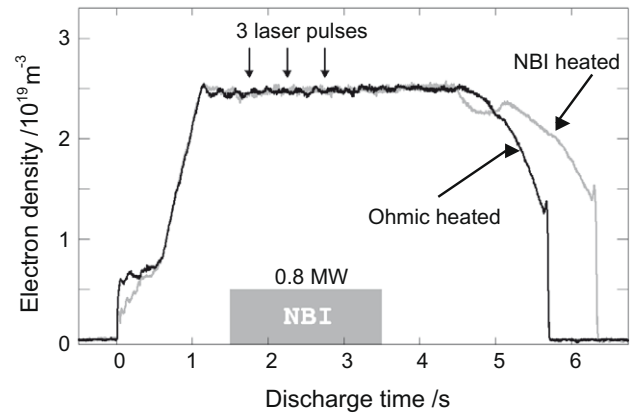


Fig. 3. Temporal development of line averaged density profile.

exposure with 42 identical ohmic and neutral beam heated discharges. Laser desorption measurements were performed every fifth discharge with ohmic heating only to reduce  $H_{\alpha}$  background light. The line averaged density profiles of the discharges are presented in Fig. 3. The NBI power was 0.8 MW.

With laser power densities up to 100 kW/cm<sup>2</sup> including reflection and transmission losses the target surface was heated up to 1800 K within typically 1.5 ms. The laser was fired three times with a frequency of 2 Hz. Measurements were performed consecutively at seven positions across the target plates with increasing plasma radii. The total times of plasma exposure for ohmic and NBI heated parts of the discharges are listed in Table 1.

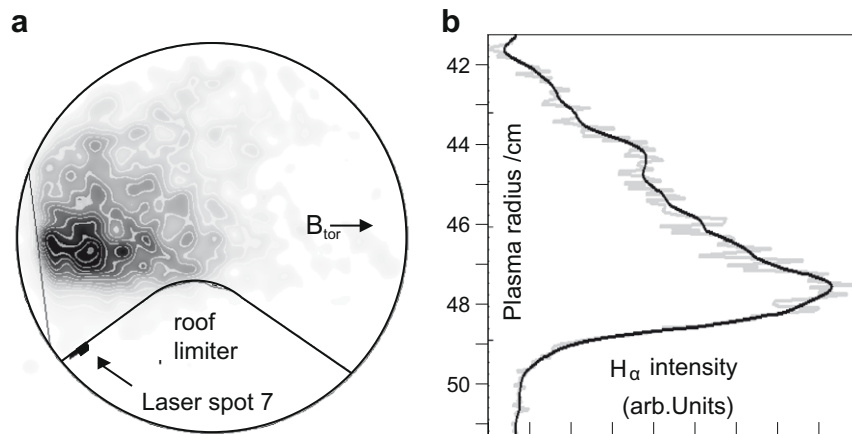
Neutral hydrogen beams were injected into a deuterium target plasma and therefore H and D was retained in the deposited hydrocarbon layers. For this measurement the H/D ratio of desorbed particles was not recorded spectroscopically but measured in post mortem analysis.

Before the experiment the graphite targets were heated for about 11 h at 1000 °C in a vacuum chamber to reduce the intrinsic hydrogen inventory in the bulk material. Inside the vacuum lock system the limiter head was heated to 400 °C for 12 h to remove water from the surface. Before plasma exposure the selected spot positions were additionally heated by several 5 ms laser pulses up to 2000 °C. By all these measures the contribution of residual hydrogen in the bulk material to the desorption signal was below the detection limit.

The limiter inserted through the material test facility was observed in poloidal direction by a CCD camera with  $H_{\alpha}$  interference filter and 20 ms time resolution covering an area of about 10 cm diameter around the limiter surface. The transmission of the interference filter with 1.5 nm FWHM is identical for all hydrogen isotopes. Fig. 4(a) presents the spatial distribution of a typical desorption signal after subtraction of background light measured before and after the laser exposure. The radial intensity profile of  $H_{\alpha}$  and  $D_{\alpha}$  was determined by integration in toroidal direction (Fig. 4(b)), converted into photon numbers after calibration with an Ulbricht sphere and multiplied by corresponding conversion factors. These are dependent on electron density and temperature in the edge, which were determined from the He beam diagnostic in the equatorial plane [8]. For the spatial range investigated here an average value of  $X/SB = 14.5$  was used, which is identical for all H isotopes. By integration of the converted  $H_{\alpha}$  and  $D_{\alpha}$  signals in radial direction the amount of desorbed hydrogen isotopes listed in Table 1 was evaluated. The edge plasma parameter delivered the radial profiles of flux particle density. By time integration from start of deposition until laser exposure the fluence of a selected spot was determined as shown in Table 1. The spectroscopic prop-

**Table 1**  
Measured properties of co-deposited a-C:H layers at spot positions selected for in situ measurements in TEXTOR. The values represent the sum of hydrogen and deuterium.

Spot #	4	5a	5b	5c	6	7	Remarks
Radial position/cm	48.4	48.9			49.2	49.5	LCFS at 46 cm
OH exposure time/s	59	60	19	17	118	132	147 total
NBI exposure time/s	42	42	15	12	84	93	96 total
H <sup>+</sup> fluence/(10 <sup>25</sup> /m <sup>2</sup> ) until LIDS	1.07	0.74		0.38	1.22	1.04	Spot 5c shifted 0.3 mm
desorbed inventory H <sup>0</sup> /(10 <sup>21</sup> /m <sup>2</sup> )	5.7	1.7		2.8	8.5	9.5	TEXTOR LIDS
H <sup>0</sup> /H <sup>+</sup> /(10 <sup>-4</sup> )	5.33	2.33		7.4	6.97	9.13	In situ TEXTOR
Total H <sup>+</sup> fluence/(10 <sup>25</sup> /m <sup>2</sup> )	2.56	1.75			1.36	1.10	
Retained H <sup>0</sup> 10 <sup>21</sup> /m <sup>2</sup>	8	8.8			8.2	9.3	LID-QMA Laboratory
H <sup>0</sup> /H <sup>+</sup> /(10 <sup>-4</sup> )	3.13	5.03			5.66	8.45	Ex situ Laboratory
Layer thickness/nm	450	460			450	420	Colorimetry



**Fig. 4.** Picture of H<sub>α</sub>/D<sub>α</sub> light in front of the roof limiter induced by LIDS on spot 7. a) camera picture in poloidal direction, b) radial profile of the toroidal integrated signals.

erties of all hydrogen isotopes are treated identical because the differences of the energy levels are very small. Also it is assumed that co-deposition and desorption have no isotope dependence.

Several methods were applied for post mortem analysis. The layer thickness was deduced from colorimetry [9], which also gives information about the hydrogen content assuming saturation [10]. The radial distribution of the hydrogen inventory was again obtained by laser induced desorption in laboratory but using a quadrupole mass analyser (QMA) for detection. For a single laser shot the mass spectra from  $2 \leq m \leq 48$  was recorded. Major contributions are observed at masses corresponding to D<sub>2</sub>, CD<sub>4</sub> and C<sub>2</sub>D<sub>4</sub> and mixtures with hydrogen.

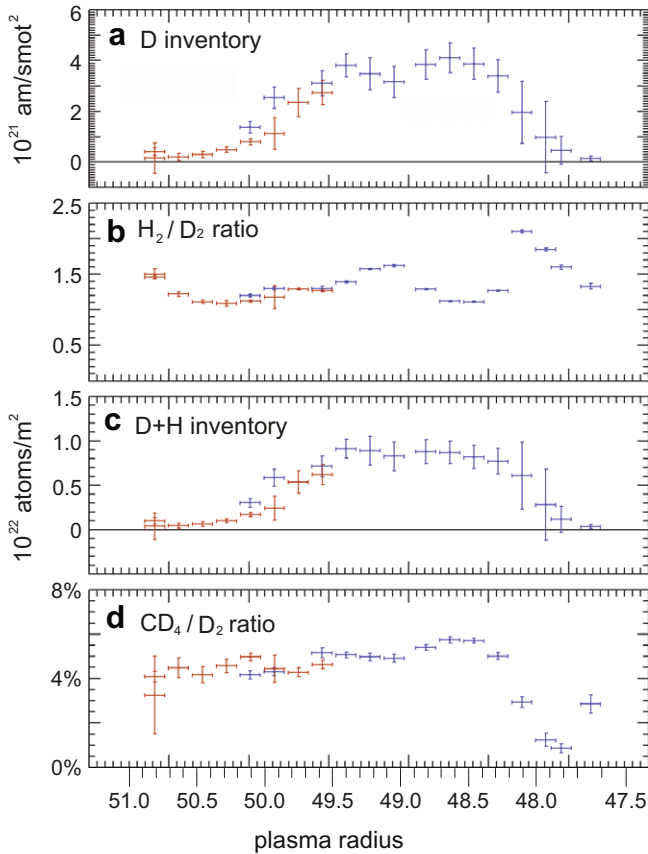
### 3. Results and discussion

The results obtained by LID in the laboratory are summarized in Fig. 5, all as a function of plasma radius. The distribution of the desorbed D atoms is given in Fig. 5(a), for which also the hydrocarbon (less than 10%, Fig. 5(d)) has been taken into account. The hydrocarbon layer contains hydrogen from NBI and deuterium from the target plasma and the deuterium signal multiplied by the factor  $1 + \text{H}_2/\text{D}_2$  (Fig. 5(b)) gives the radial profile of the retained hydrogen isotopes presented in Fig. 5(c). The first 9 mm of the probe tip, 2.2 mm in radial direction, is an erosion dominated area without any carbon deposition. The hydrocarbon layer thickness increases up to the radial position of 48.25 cm and stays about constant to 49.5 cm (Values for spots measured in situ at TEXTOR are listed in Table 1), followed by an exponential decay to nearly 0 at the end of the plate. The ratio of desorbed deuterated methane to deuterium molecules is given separately in Fig. 5(d). At positions

closer to the LCFS a significant drop of the ratio is observed. This behaviour indicates that elevated surface temperatures have been reached during the plasma exposure at which hydrocarbons are already thermally released [11].

The radial dependence of total inventory of hydrogen isotopes in the re-deposited hydrocarbon layer, measured by LID-QMA and normalized to the corresponding total ion fluence is shown in Fig. 6 and also listed in Table 1 for the selected spot radii. This can be interpreted as a deposition efficiency of hydrogen isotopes in hydrocarbon layers that increases nearly linearly from the region of net erosion ( $r \leq 47.8$  cm) until a maximum at 49.5 cm.

In TEXTOR LIDS has been applied at seven radial locations (see Fig. 2) on the roof limiter each with different times of plasma exposure. For the NBI heated discharges the erosion and deposition dominated regions could be clearly distinguished. In the net erosion zone, the H inventory is low but clearly measurable and dependent on flux density (spot 1) followed by a transition region (spot 2 and 3) where the deposition rate is still growing. The spots 4 to 7 cover a radial range of 1.6 cm, in which the carbon deposition rate is nearly constant. The inventory of desorbed hydrogen atoms isotopes and the impinging H<sup>+</sup> fluences at these spots until the measurement with LIDS are listed in Table 1. For each spot the ratio of these values, H<sup>0</sup>/H<sup>+</sup>, is added into Fig. 6. The error bars are determined by spot size and uncertainties in the absolute calibration of the light intensity and conversion factors and the fluence measurements. For discharges with same H<sup>+</sup> flux density profiles the results should be comparable with the values obtained in the laboratory measurements. The agreement of the data measured by LIDS in TEXTOR and LID-QMA in the laboratory experiment are within the error bars of the absolute calibration of the camera



**Fig. 5.** Laboratory results a) radial profile of desorbed D inventory, b) radial profile of the  $H_2/D_2$  ratio, c) radial profile of the hydrogen isotope inventory, d) radial profile of the  $CD_4/D_2$  ratio.

and QMA sensitivity, respectively. Only one value obtained in spot 5a significantly deviates from the trend line, which might be explained by a disruption occurred in a previous discharge. Due to a technical failure, the signals obtained in 5b were not recorded. In 5c the laser spot had to be realigned but was shifted by 0.3 mm in respect to the previous position. Therefore the inventory, representing 15% of the spot size was desorbed from an area with longer exposure times. This was considered in the evaluation of the deposition efficiency.

In conclusion, the hydrogen inventory was measured in TEXTOR in situ by LIDS reliably if the retention exceeds about  $2 \times 10^{20}/m^2$  that is about the value detected in the erosion deposited area (spot 1). This lower limit is set by the natural fluctuations of the hydrogen background light and corresponds to a layer thickness of 10 nm assuming a density of  $2 \times 10^{19}/m^2$  nm H atoms in hydrocarbon layers. LIDS was performed on thin layers only with good heat conductance to the bulk material, which mainly absorbs the laser power. The control of temperature development might not be possible in thick deposited layers, which loose contact to the substrate.

**4. ITER application**

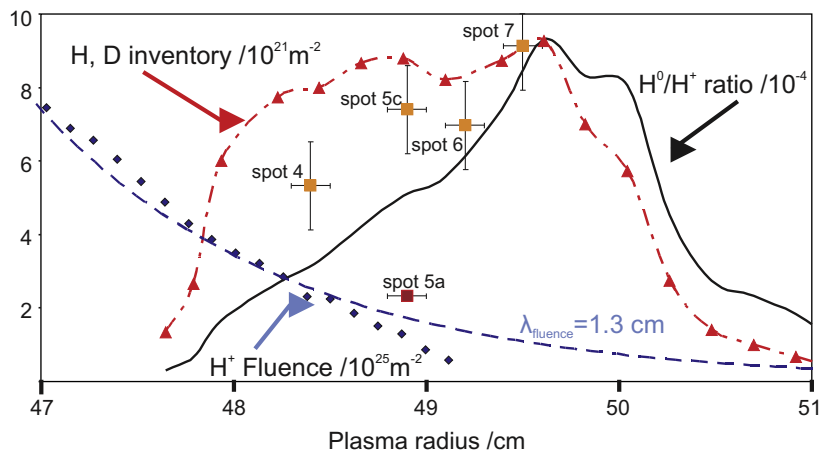
The results obtained by LIDS on co-deposited hydrocarbon layers in TEXTOR are encouraging and motivate to propose this diagnostic for the in situ measurement of tritium inventory at preselected locations of PFC in ITER. A preliminary study shows that a Nd:YAG laser beam with 50 kW single pulse power must be guided by mirrors into the discharge chamber and focussed on a spot of about 1 cm $^2$  in order to heat the surface up to 1800 K within 2–3 ms. If the same spot is exposed repetitively by the laser beam the temporal development of hydrogen retention can be monitored. The  $H_\alpha$  intensity peak can be recorded by a single detector that must collect the full range of enhanced  $H_\alpha$  radiation above the background light, which increases with the distance of the spot to the edge plasma and will determine the sensitivity limit. For tritium operation the T/D ratio in the plasma should be identical in deposited hydrocarbon layers. Considering this ratio the local tritium retention can be determined.

**Acknowledgement**

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**Fig. 6.** In situ measurement (squares, spot 4–7) of deposition efficiencies of hydrogen isotopes  $(H^0/H^+)/10^{-4}$  during similar TEXTOR discharges and comparison with results (solid line) obtained ex situ in laboratory. Hydrogen isotopes inventory  $H^0/10^{21} m^{-2}$  in hydrocarbon layers measured in laboratory (triangles), integrated hydrogen isotopes ion flux density  $H^+/10^{25} m^{-2}$  determined from  $T_e$  and  $n_e$  measurements in TEXTOR (diamonds) where the dashed line represents an exponential fit with  $\lambda_{decay} = 1.3$  cm.

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