

In-situ measurement of fuel retention by laser-induced thermal desorption of hydrocarbon layers in TEXTOR

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In ITER the divertor plates will be made of CFC. Carbon erosion, transport and redeposition lead to hydrogen retention in amorphous hydrocarbon layers (a-C:H) [1,2], which greatly determines the availability of ITER [3]. If the in-vessel inventory of tritium reaches the safety limit of 350 g, operation will have to be suspended to allow for cleaning procedures [4], in particular removal of these co-deposits. Thus it is essential to study already during the first phase of hydrogen operation the characteristics of fuel retention by co-deposition. This calls for a suitable diagnostic which is presently not planned for ITER but which needs to be developed urgently. A very promising method is a careful total gas balance supported by in-situ diagnostics for the fuel inventory of wall components and redeposited layers. Such a diagnostics is being developed at TEXTOR. It employs laser-induced thermal desorption of plasma facing components and the spectroscopic analysis of the released hydrogen during plasma operation.

Experimental Procedure

The experiment was performed in lock 1 at the bottom of the TEXTOR vessel [5]. The sample holder was positioned in the plasma boundary 2 to 4 cm outside the LCFS at 46 cm. The beam of a fibre-coupled Nd:YAG laser ($\lambda = 1064$ nm) is introduced from top of the vessel nearly perpendicular to the target surface and imaged on an area (spot size) of 4 mm^2 . With power densities of 50 to 80 kW/cm^2 for 1.5 ms target surface temperatures of 1000 to $1400 \text{ }^\circ\text{C}$ are obtained. The desorbed hydrogen molecules enter the plasma boundary where they are excited and dissociated by electron collisions. The resulting

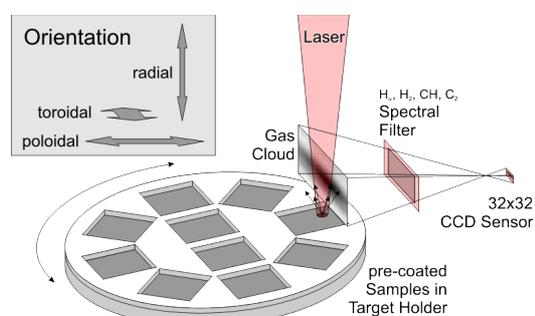


Fig. 1: Principle view of the experimental arrangement for laser desorption in TEXTOR. The holder contains 10 samples ($22 \times 22 \text{ mm}^2$) which are located 2 mm behind the sample holder surface.

radiation [6], is measured with a fast CCD camera using narrowband interference filters ($D_\alpha(656.3 \text{ nm})$ $\text{FWHM}_{\text{Filter}} = 1.5 \text{ nm}$, D_2 Fulcher band (strongest part, 601.5 nm) $\text{FWHM}_{\text{Filter}} = 3 \text{ nm}$). Figure 1 gives a view of the experimental arrangement. The results described in this paper are observed in a hydrogen plasma whereas the samples are coated with deuterated hydrocarbon layers produced in the laboratory [7]. This allows for the discrimination of released particles trapped in the layer (D) from those in the substrate (H) and at the surface (absorbed H monolayer) by the measurement of the H/D ratio of the desorbed gas cloud with a high resolution spectrometer [6].

To investigate the influence of the plasma onto the spectroscopic measurement the sample radial position was 2, 3, and 4 cm from the LCFS. The spectroscopic observation is tangential to the sample surface in poloidal direction, employing a Dalsa 32 x 32 pixel CCD camera which images an area of $4.5 \times 4.5 \text{ cm}^2$. A frame rate of 500 Hz is chosen to depict the emission cloud on a maximum of 2 frames while suppressing the integration of noise. The background light is measured before and after the laser pulse and subtracted from the image, which is then integrated in toroidal direction by applying line-by-line a Gauss fit to the signal, thus accounting for the cropped sides of the emission cloud. From the resulting radial emission profile that represents the known hydrogen inventory of the layer within the spot the conversion factors can be extracted using plasma parameters in the TEXTOR edge measured separately with an atomic He-beam [8].

For the experiments a-C:D layers (200...400 nm) were characterised in preceding laboratory measurements by means of laser desorption, using the same power density and pulse duration as later in TEXTOR and analysing the released gases with a mass spectrometer [7].

Results and discussion

The present work focuses on the calibration and the demonstration of feasibility of this method. Therefore, it constrains to the desorption of hard ($\text{H/C} = 0.35\text{--}0.4$, $n = 1.9\text{--}2.0$, $k = 0.05$) deuterated a-C:H layers of 200-400 nm thickness on rough ($R_a = 3\text{--}6 \mu\text{m}$). For these samples the necessary laser power density and pulse length are determined by the optical and thermodynamical properties of the substrate. The samples are contaminated by adsorption of atmospheric water, which is seen in a continuous release of H during

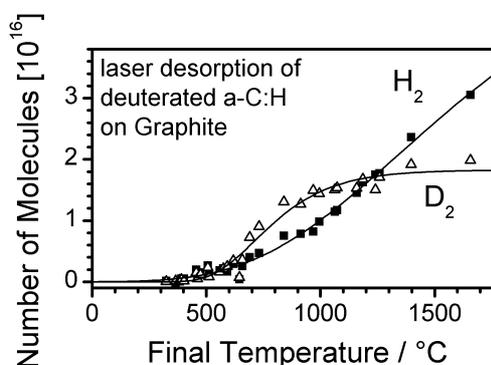


Fig. 2: Hydrogen release (H_2 and D_2) as a function of surface temperature for a 130 nm deuterated a-C:H layer on EK98 Graphite; the heating rate is 10^6 K/s (laboratory measurement)

the laser pulse, while the layer always releases the same amount of deuterium if the surface reaches a minimum temperature of 1200 °C [Fig. 2]. The desorption process of the layer is finished within $t < 0.2$ ms. For this reason, reproducible laser parameters are critical to allow for a detailed comparison with the data from the sample pre-characterisation.

It was shown for thin layers that the primarily released species is molecular hydrogen (75%), with parts of methane (15%) and ethene (10%) as is seen in figure 3. Atomic release was not found.

An example of a TEXTOR measurement is shown in fig. 4. The image is fit line-by-line with a gauss function, which gives the radial emission profile on the right. This profile is then integrated in radial direction yielding the value of I_{tot} shown for various sample positions and plasma densities in fig. 5. The data measured in TEXTOR shows no variation in the number of emitted photons from laser desorption at 2 and 3 cm distance to the LCFS. The signal significantly drops at 4 cm. This effect is less clear for higher density, indicating that this low signal might be a systematic error introduced by the data evaluation. With desorption farther from the LCFS the signal becomes fainter and spatially more extended exceeding the camera image. The camera image was not recorded down to the background level, so the I_{tot} of faint signals

(H_α at 4 cm, H_2) is determined with an uncertainty of 50%. The background level used is founded on earlier measurements with this camera. H_α data measured at the other distances is more peaked and less susceptible to such errors. The data is only

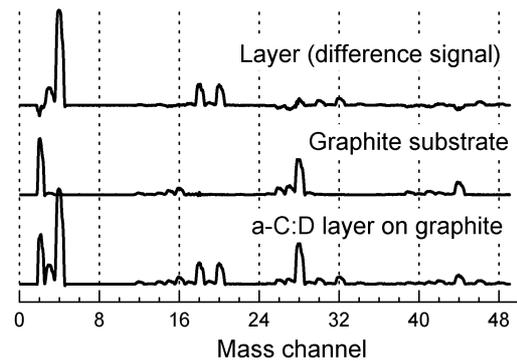


Fig. 3: Mass spectra of the gas released during laser desorption ($70 \text{ kW/cm}^2 \times 1.5 \text{ ms}$) of a 130 nm a-C:D laser on Graphite: D_2 , CD_4 and C_2D_4 emanate from the layer, the substrate releases H_2 , CO , and traces of CO_2 and CH_4 .

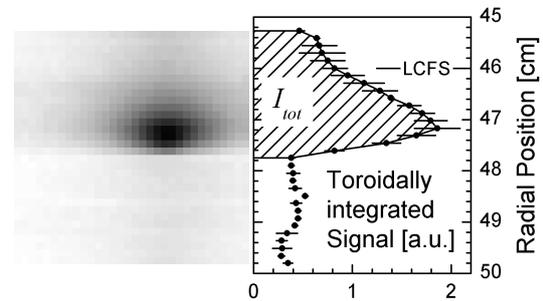


Fig. 4: typical CCD-camera image (D_2) and toroidally integrated signal.

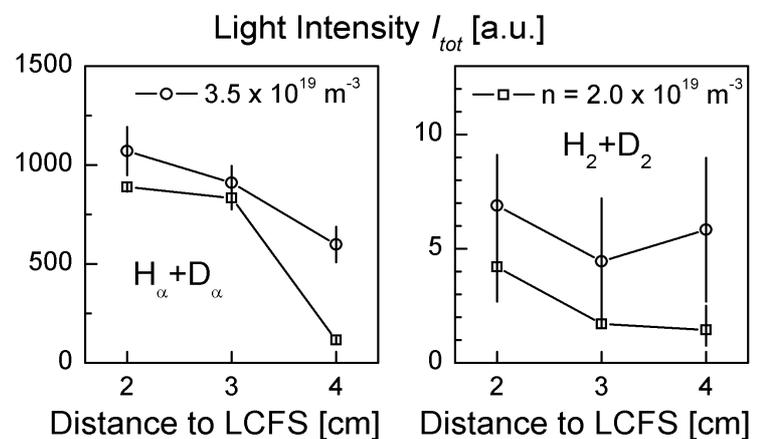


Fig. 5: Integrated signals for samples desorbed at different radii and central plasma densities. H_2 and H_α are calibrated relatively

calibrated relatively (H_{α} and H_2 intensity), so a comparison with literature data is still to be done.

Conclusion and Summary

Laser-induced thermal desorption spectroscopy is a flexible tool for determining the hydrogen inventory of redeposited layers in a tokamak with an accuracy of 10% to 30%. The sensitivity of the present design is below 10^{16} hydrogen atoms, which corresponds to the inventory of approximately 10 nm a-C:H layer on a spot area of 0.20 cm². Giving up spatial resolution of the emitted light will lead to even higher sensitivity. The maximum thickness which can be desorbed in one laser shot is set by the laser light absorption length of the layer. Extrapolating the properties of thin hard layers gives a rough estimate of 20 to 50 μm for the 1064 nm laser radiation absorption length. At these thicknesses the thermal properties of the substrate will play a minor role. Longer laser wavelengths may penetrate deeper into the layer, while technically this will be limited by available lasers and light guides. Furthermore, thick layers are mechanically less stable and might delaminate from the wall in macroscopic flakes as they do in laser ablation experiments. More studies of the desorption of thick layers are therefore necessary before a well-founded maximum thickness may be claimed.

For a future implementation the diagnostics may be set up coaxially, with the laser and the spectroscopic observation using the same optics and being decoupled by dichroic mirrors. The adjustment then reduces to choosing the focal length of the system, while the observation may record all relevant spectral lines and bands simultaneously using photo diodes.

Acknowledgments

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