

Available online at www.sciencedirect.com



Journal of Nuclear Materials 337-339 (2005) 570-574



www.elsevier.com/locate/jnucmat

Laser desorption of deuterium retained in re-deposited carbon layers at TEXTOR and JET

B. Schweer ^{a,*}, A. Huber ^a, G. Sergienko ^a, V. Philipps ^a, F. Irrek ^a, H.G. Esser ^a, U. Samm ^a, M. Kempenaars ^b, M. Stamp ^c, C. Gowers ^c, D. Richards ^c

^a Institut für Plasmaphysik, FZ Jülich GmbH, EURATOM Association, Trilateral Euregio Cluster, 52425 Jülich, Germany ^b FOM-Instituut voor Plasmaphysica 'Rijnhuizen' EURATOM Association, Trilateral Euregio Cluster, P.O. Box 1207,

3430 BE Nieuwegein, The Netherlands

^c UKAEA, EURATOM Fusion Association, Culham Science Centre, Abingdon, Oxon OX14 3DB, UK

Abstract

ITER operation might be limited by the tritium inventory that will be retained in carbon tiles and re-deposited carbon layers. Means to determine in situ the tritium content of these layers and also the deuterium content in the nonactivated phase of ITER and tritium recovering techniques have to be developed. This paper describes an approach in which the thickness and deuterium (tritium) content of re-deposited carbon layers is determined by local laser heating. This leads to the release of the retained fuel by evaporation and, depending on the laser power density even to the removal of the layer itself. An essential element of this method is the quantitative determination of the released particles by local spectroscopy in the edge plasma. Results from this method from experiments in the laboratory, and on TEX-TOR and JET are described in this paper.

© 2004 Elsevier B.V. All rights reserved.

PACS: 52.70.Nc; 32.10.Bi; 78.70.-g; 79.20.Ds *Keywords:* Desorption; Retention; TEXTOR; Spectroscopy; Deuterium inventory

1. Introduction

The present choice of first wall materials in ITER foresees beryllium at the main chamber, tungsten at the baffles and graphite CFC at the divertor plates. In particular the use of graphite can lead to accumulation of tritium in hydrocarbon layers and is a serious concern for ITER operation. These layers are formed by codeposition of eroded wall materials with deuterium and tritium and develop in net deposition areas, such as the inner divertor area or the slits of the castellated tiles [1–3]. An estimation of the amount of retained T in ITER is highly uncertain because the prediction relies upon the knowledge of many parameters, such as the main chamber material erosion, the flows in the plasma edge, and the local transport of material inside the divertor. A diagnostic is urgently needed that monitors in situ the T retention at different representative locations and, in combination with other methods e.g. a global gas balance, delivers reliable data of the in vessel T content in ITER. This is of particular importance in the non-activated phase of ITER which should provide the information on this topic for the activated T phase.

^{*} Corresponding author. Tel.: +49 2461 615536; fax: +49 2461 613331.

E-mail address: b.schweer@fz-juelich.de (B. Schweer).

^{0022-3115/\$ -} see front matter @ 2004 Elsevier B.V. All rights reserved. doi:10.1016/j.jnucmat.2004.10.156

It is well known that a hydrocarbon layer releases the stored fuel gas upon heating [4]. Thus, laser radiation is proposed to heat any target surface (tokamak tiles) [5]. The temperature increase is mainly determined by the heat capacity and adhesion of the layer to the bulk material. However, the heat absorption can also be influenced by plasma that is created by interaction of released particles with high power densities of the laser radiation.

Experiments with laser heating have been performed already at JET and TEXTOR. At JET the Edge LIDAR beam always hits a carbon tile at the inner divertor with power densities of about 10 GW/cm² during 300 ps pulses. This generates ablation of deposited amorphous hydrocarbon layers (a-C:H) but might also damage the bulk material. The released D and C atoms were observed spectroscopically in the edge plasma [6]. In order to avoid the removal of bulk material a ruby laser pulse with comparable energy but much lower power density $(40 \text{ kW/cm}^2 \text{ for } 300 \text{ }\mu\text{s})$ was fired onto a plasma exposed C test limiter at TEXTOR between discharges. The desorbed deuterium was detected with a Quadrupole Mass Analyser (QMA) [7]. Because of the large volume of ITER the sensitivity for the detection of the released particles might not be sufficient. Therefore the application of emission spectroscopy in combination with laser induced desorption might be another possibility for an in situ diagnostic for the determination of the tritium content in co-deposited layers. The 'long' laser heating leads to the release of D(T) atoms and molecules. The evaporated particles enter the plasma and their $D_{\alpha}(T_{\alpha})$ radiation is observed. With known plasma parameters the emission light can be converted to absolute particle fluxes using appropriate conversion factors [8,9].

In this paper results are presented where 'free running' ruby lasers are fired onto graphite targets with hydrocarbon layers. In the first section, the laser induced desorption on well-defined hydrocarbon layers during exposure is investigated in a laboratory experiment. The spectroscopic detection of laser desorbed particles in the plasma edge and conversion into total fluxes is studied at carbon test limiters in TEXTOR, results of which are presented in the second section. For the same purpose, the Edge LIDAR at JET system was changed to long pulse operation. The evaluation of the D_{α} emission signals of desorbed D in three consecutive laser pulses (1 s delay) was measured during the limiter and divertor phase. This is described in the last section.

2. Laboratory experiment

The target is located at the centre of an UHV chamber with 300 mm diameter. The base pressure generated by a turbomolecular pump is below 10^{-6} mbar. A target manipulator allows a vertical and horizontal movement over the whole area of a 5×5 cm² target. The radiation of a ruby laser is focussed onto the target. The spot size and correspondingly the power density can be varied from 1 mm to 10 mm diameter by retraction of the focussing lens. A further variation of the beam power without changing the spot size is performed by the introduction of neutral filters into the beam line. The maximum laser energy is about 1.5 J for pulse duration of 350 μ s. The laser power is monitored by a fast diode.

A QMA and an IR array camera are attached at the equatorial plane of the vacuum chamber each under 45° in respect to the laser beam. For the investigation of a-C:H layers deposited on carbon the atomic masses 2 (D), 4 (D₂), 24 (C₂) and 36 (C₃) were selected at the QMA. A 128 element linear array camera was operated with 100 kHz frame rate and 36 µm/pixel spatial resolution. Only IR light above 850 nm was selected by an optical filter and the intensities were compared with Planck's law to determine the surface temperature. The lowest detectable temperature at the highest time resolution was 700 °C.

The a-C:H layer on a graphite target was produced in a DC glow discharge with a bias of 600 V. The deuterium content of this layer with a thickness of 141 nm was determined by thermal desorption [10]. A D/C ratio of 0.31 was measured that corresponds to 1.7×10^{17} D atoms/cm² and was used for calibration of the QMA signals. The ratio of released atomic and molecular D might depend on the surface temperature [11] that yet has not be considered for the calibration of the QMA signals.

The time traces of a single laser pulse for power density, absorbed energy and surface temperature rise at the centre of the exposed area $(1.1 \times 0.036 \text{ mm}^2)$ are presented in Fig. 1. A temperature of about 700 °C is reached after 120 µs that is in reasonable agreement with calculations made for carbon, assuming a spiking laser radiation of 1 µs spike length with 100 kHz repetition rate. With the experimental data of 0.3 J energy over 350 µs focussed on 2 mm², a power density of 430 kW/ cm² is found in a single spike. To a certain extent the temperature rises are coupled to the spike structure of the ruby laser.



Fig. 1. Temporal development of the (a) laser power and laser energy, (b) surface temperature at the spot centre.



Fig. 2. Decay of desorbed D_2 (integrated QMA signal) for consecutive laser pulses (average power density 20 kW/cm²).

A nearly complete desorption of deuterium out of the a-C:H layer was performed in a single laser shot exposure with an energy of 1.5 J. Two consecutive laser shots with same energy were focussed onto the same spot of 3 mm² (mean power density 150 kW/cm²). The target was at room temperature. The QMA signal of the second laser pulse was significantly lower (<20%) than in the first pulse. In further laser pulses with the same energy the C₂ QMA signal was observed that remained nearly constant at values obtained from a pure carbon surface. This behaviour can be explained by carbon evaporation induced by the high power densities (about 1.5 MW/cm²) in single spikes.

With reduced laser energy of 0.2 J about 10 laser shots were necessary to reach the same reduction of the deuterium signal as in the previous experiment. Even with doubled laser energies the release of D_2 could not increased significantly as seen in Fig. 2. The strong signals obtained in laser shot 1 and 17 might be related to diffusion and adsorption of deuterium at the exposed surface. The C₂ signal measured at the end of this series was more than a factor of 10 lower compared to the previous experiment. This behaviour supports the assumption that only carbon from the amorphous hydrocarbon layers is removed at these power densities while the power of a single spike is not sufficient to cause sublimation of the bulk carbon. The temperature integrated over 10 µs never increased above 750 °C in this case.

3. TEXTOR experiments

The experimental set-up of the laser desorption system at TEXTOR is shown in Fig. 3. A carbon limiter was inserted into a limiter lock system on top of the vessel and the tip was positioned at the last closed flux surface (LCFS) at a plasma radius of 460 mm. The laser beam with energies up to 5 J entered the vessel from the bottom. Results presented in this paper were obtained from a laser spot 1 cm in the shadow of the LCFS located 35 mm from tip of the limiter on the electron drift side. A polychromator equipped with interference filters detected simultaneously the CD, D_{α} , D_{β} , D_{γ} and



Fig. 3. Experimental set-up for laser desorption on test limiters at TEXTOR.

CII light in front of the limiter surface with a time resolution of 5 μ s. Additionally a spectrum of the plasma background was measured with a spectrometer. The initial temperature distribution at the limiter surface was monitored with a CCD camera collecting the IR light above 850 nm. The fast temperature rise averaged over the laser-exposed area was measured by a pyrometer with 15 μ s time resolution. The edge plasma parameters needed for the determinations of the conversion factors were measured with He beam diagnostic [12]. Also the local density was deduced from the ratio of D_{α}/D_{β} . A fast photodiode recorded the laser power.

In Fig. 4 time traces of $H_{\beta}(D_{\beta})$ and CII lines are presented which were obtained by laser induced desorption in Ohmic heated TEXTOR discharges ($I_p = 350 \text{ kA}$, $n_e = 2 \times 10^{19} \text{ m}^{-3}$; $B_t = 2.25 \text{ T}$) in deuterium. The total fluxes for H/D and C given in Fig. 5 were deduced from H_{β} and CII line intensities using the appropriate conversion factors (S/XB) of 67.5 and 6.97, respectively as given in the literature [8,9] for the measured electron density and temperature. About $(6-10) \times 10^{15} \text{ cm}^{-2}$ H(D) atoms and $1 \times 10^{15} \text{ cm}^{-2}$ C atoms are released from a spot of about 8 mm diameter with an initial



Fig. 4. (a) Temporal behaviour of the ruby laser radiation and (b) the corresponding line intensities (H_{β} and CII) of desorbed particles in front of a carbon test limiter during a TEXTOR discharge.



Fig. 5. Total amount of D and C released from a carbon test limiter at TEXTOR at different limiter temperatures (plasma parameter $n_e = 2 \times 10^{18}$ /cm³, $T_e = 50$ eV).

temperature of 450 °C (Fig. 5(a)). The fast pyrometer shows a temperature rise of about 300 K at the surface during laser radiation. As shown in the laboratory a mean power density of about 40 kW/cm² is not sufficient to desorb all D atoms of an a-C:H layer that should be a saturation fluence of $1-2 \times 10^{17}$ cm⁻². The amount of released carbon atoms was strongly dependent on the initial temperature of the test limiter before laser exposure. The carbon signals rose by a factor of 10 when the initial surface temperature was increased from 450 °C to 770 °C (Fig. 5(b)). This effect indicates that the spiking power intensity of the ruby laser radiation surpasses the sublimation temperature of carbon for the later case.

4. JET experiments

Without Q-switches the edge LIDAR system [13] delivered an average power density of about 10 kW/ cm² for about 350 μ s. The beam hitted the upper inner divertor tile above the strike point of the plasma (see [6]). A series of three laser pulses with 1 Hz repetition rate was fired onto the same spot during the limiter and divertor phases. The D_{α} signal of the desorbed particles was measured with a fast wide-angle observation system (250 kHz sampling rate). Laser induced desorption measurements were applied for a variety of different plasma conditions. The D_{α} signals as shown in Fig. 6(a) showed always a significant drop after the first laser shot and a weaker decay for the next two laser shots. In series of consecutive discharges the amplitude of D_{α} signals



Fig. 6. Integrated (1.5 ms) D_{α} line intensity for three consecutive laser pulses at JET during the (a) divertor and (b) limiter phase.

varied within about a factor of 3. This might be explained mainly by changes of plasma parameter but more analysis is needed here. This behaviour although less pronounced was also found if the laser was fired during limiter mode for which the D_{α} signals were much lower (Fig. 6(b)). However, to determine the total amount of desorbed D, the higher S/XB factor for lower electron densities and temperatures and the lower amount of particles exited in the observation volume must be taken into account.

The retention of deuterium on these areas occurs mainly during the divertor phase since only a very weak signal was observed in the first shot during the limiter phase if the laser was fired once before the discharge. However laser induced desorption during the divertor phase did not show a clear influence on any change of discharge conditions. An explanation for this behaviour can be the high particle flux to the divertor tile that leads to saturation within a few seconds or more likely that the laser power is too weak to desorb all the deuterium from deeper zones. The measured deuterium signal would thus only correspond to a shallow surface layer.

5. Conclusion

The content of deuterium and carbon in co-deposited hydrocarbon layers has been measured by means of laser desorption using 'free running' ruby lasers with pulse durations of 350 µs. In a laboratory experiment released deuterium and carbon was detected using a QMA system. At lower mean power densities (50-80 kW/cm²) sublimation of carbon during laser spikes (about 1 µs) is nearly avoided if the target was at room temperature. The threshold for significant carbon sublimation decreases with increasing initial temperature of the target as expected for a thermodynamic process. Laser desorption on a-C:H layers produced in the laboratory shows that more than 80% of the deuterium was desorbed in a single laser shot with an average power density of 150 kW/cm^2 . However, in such case the significant higher power densities in the spikes have almost removed the layer. It is a disadvantage of this type of free running ruby laser, that the desorption of D and C is largely governed by the spiking structure of the ruby laser pulse. The mean surface temperature is determined by the absorbed energy but a spike with peak power density of 1 MW/cm² for 1 µs increases the surface temperature on carbon by more than 600 K.

In TEXTOR and JET it has been demonstrated that the desorbed particles can be determined by in situ emission spectroscopy although in both cases the available power densities were to low to heat the whole a-C:H layer. In TEXTOR, the amount of deuterium and carbon released from limiter by a ruby laser pulse was measured by quantitative emission spectroscopy on a shot by shot basis and found in the order of 5- 10×10^{15} D/cm². This amount is understood in terms of the retention of fuel in a shallow surface layer due to direct implantation from the edge plasma, while a fraction of fuel is desorbed also in form of hydrocarbons, causing the carbon light emission. However this value is small compared with values expected from post mortem surface analysis of erosion dominated graphite surfaces. This indicates either a non-complete desorption, which might be due to the spiky structure of the power input, or uncertainties in the conversion of the spectroscopically obtained signals. More work is needed here in the future to qualify this technique better. In JET, the deuterium release from an inner divertor tile has been measured in a similar way, but without absolute calibration so far. The fuel retention at this location occurs mainly during the divertor operation phase. No clear dependence of the amount of desorbed fuel on the number of accumulated discharges before the laser pulse could be found. This indicates that the laser power in JET is insufficient to desorb the fuel from thicker co-deposits.

For application at ITER further improvements of this diagnostic are necessary. A constant power density distribution over the beam cross-section [14] could reduce the uncertainty of the measurements. An essential element of the diagnostic for the application at ITER is the development of a powerful laser with constant temporal output to avoid the sublimation effects of the spikes.

Acknowledgments

This work is being carried out under the research programme activities of the EU-PWI Task Force under EFDA technology task TW3-TPP-ERDIAG.

References

- G. Federici, R. Causey, P.L. Andrew, C.H. Wu, Fus. Eng. Des. 28 (1995) 136.
- [2] G. Federici, R. Anderl, J.N. Brooks, R. Causey, J.P. Coad, D. Cowgill, R. Doerner, A.A. Haasz, G. Longhurst, S. Luckhardt, D. Mueller, A. Peacock, M. Pick, C. Skinner, W. Wampler, K. Wilson, C. Wong, C. Wu, D. Youchison, Fus. Eng. Des. 39&40 (1998) 445.
- [3] P. Wienhold, V. Philipps, A. Kirschner, A. Huber, J. von Seggern, H.G. Esser, D. Hildebrandt, M. Mayer, M. Rubel, W. Schneider, J. Nucl. Mater. 313–316 (March) (2003) 311.
- [4] V. Philipps, E. Vietzke, M. Erdweg, K. Flaskamp, J. Nucl. Mater. 145–147 (1987) 292.
- [5] J.W. Davies, A.A. Haasz, J. Nucl. Mater. 266–269 (1999) 478.
- [6] D.D.R. Summers, M.N.A. Beurskens, J.P. Coad, G. Counsell, W. Fundamenski, G.F. Matthews, M.F. Stamp, J. Nucl. Mater. 290&291 (2001) 496.
- [7] A. Huber, M. Mayer, V. Philipps, A. Pospieszczyk, T. Ohgo, M. Rubel, B. Schweer, G. Sergienko, T. Tanabe, Phys. Scr. T 94 (2001) 102.
- [8] A. Pospieszczyk, in: R.V. Janev, H. W. (Eds.), Drawin Atomic and Plasma-Material Interaction Processes in Controlled Thermonuclear Fusion, Elsevier Science, 1993, p. 213.
- [9] http://adas.phys.strath.ac.uk.
- [10] J. Winter, H.G. Esser, P. Wienhold, V. Philipps, E. Vietzke, K.H. Besocke, W. Möller, B. Emmoth, Nucl. Instrum. and Meth. B 23 (1987) 538.
- [11] S. Brezinsek et al. (2001) 28th EPS Conf. on Contr. Fusion and Plasma Phys. Funchal ECA, Vol. 25A, 2077.
- [12] B. Schweer, G. Mank, A. Pospieszczyk, B. Brosda, B. Pohlmeyer, J. Nucl. Mater. 196–198 (1992) 174.
- [13] P. Nielsen, S. A. Arshad, C. Gowers, 26th EPS Conf. on Contr. Fusion and Plasma Physics, ECA 23J (1999) 281.
- [14] F. Le Guern, Laser ablation and heating tests performed on Tore Supra and TEXTOR graphite samples, FZJ-IPP colloquium talk, June 26, 2003.