

## In-situ tokamak laser applications for detritiation and co-deposited layers studies

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### Abstract

Treatment of plasma facing components (PFCs) is a major issue for ITER operations and may be applied for different reasons. As a most important application, there is the control of tritium inventory to fulfil the safety requirements. In the following, a solution based on laser techniques is presented. Due to its flexibility, its capability to reach difficult access structures as voids or castellations and the possibility to be installed on remotely controlled devices, laser offers one of the most suitable solutions for detritiation of the PFCs via ablation process. In the following, the high ablation efficiency as well as the capability to detritiate co-deposited carbon layer without any interaction with bulk material will be discussed. Then laser induced breakdown spectroscopy (LIBS) will be described with a new diagnostic based on the study of the surface temperature response after a heating laser pulse. It will be shown that used together, the two techniques allow the determination both the concentration of tritium in co-deposited layer as well as the total quantity of carbon co-deposited on top of bulk material. These two bits of information are essential to determine when the detritiation process must take place and how long it should last. Finally, the still open issues will be presented that need to be developed in order to apply this global laser system in tokamaks.

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### 1. Introduction

Treatment of plasma facing components (PFCs) is a major issue for ITER operation. Wall conditioning after shutdown as well as during plasma

operation are mandatory to allow reproducible plasma start up, safe current ramp up and recycling control. Treatments are also needed to control tritium inventory and fulfil safety requirements. Several treatments have been proposed in the past decade in order to remove the carbon co-deposited layers observed in current tokamaks using graphite as part of their PFCs. In these layers, it is often observed a high concentration of tritium. One of

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the possible treatments is to oxidise this co-deposited material with oxygen or, better, to include oxygen in glow treatments [1–3]. These techniques have been already used in several test tokamaks and are presented at this conference. Interaction of light with material is another process which is used to recover the trapped tritium via ablation of carbon co-deposited layers. Flash lamp technique [4] has proven its capability to operate in a tokamak environment. Laser interaction with matter which was proposed to be used in tokamaks well before this flash lamp experiment, offers a very interesting alternative and/or complementary solution [5,6]. Laser devices are very flexible. They can be used by remote handling if embarked on a robot and if fibres are used to drive the laser light. At last they can treat areas which are difficult to access like voids or castellations thanks to the small size of the laser spot light. In the case of castellations, almost all the tritium is trapped in the side of the tile in an area which is less than 5 mm underneath the plan surface [21]. To treat these regions, an angle of incidence of  $11^\circ$  is needed which reduces the laser fluence by a factor 20–30. However, this reduction appears to be acceptable technically with the characteristics of the laser used in the laser ablation experiments.

Like in current machines where carbon is part of the PFCs, ITER in his start-up phase will use carbon in the divertor and will therefore experience high material erosion. Skinner and Federici [7] have estimated that the expected ITER deposition rate of tritium will be of the order of 5 g of tritium per pulse. This implies the necessity of an overnight removal rate of more than 100 g of tritium. If one supposes a ratio of tritium over carbon of 1, this quantity corresponds to 1000 g of carbon layer removal capability. From these assessments, it appears that an efficient technique able to treat this quantity is mandatory. We will show in the following that if, as observed in the JET tokamak [8], an accumulation of carbon layers is observed in limited area then ablation techniques and especially laser ones are sufficiently efficient to treat this supposed quantity.

However, during the ITER operation, it will be also of major importance to have in-situ tools to characterise, at least in between plasma pulses or during overnight period, the co-deposited materials in order to estimate their composition, the total quantity of tritium trapped in these layers and, of course, the total quantity of co-deposited material. This assessment of the quantity of tritium remaining

in the vessel is essential to close the fuel cycle as well as to determine if a fuel recovery action has to take place and how long would be needed to accomplish this duty.

Techniques based on laser can also be used to give answers to these questions. In the following, we will demonstrate how the chemical composition of the co-deposited material as well as the tritium concentration in this layers could be determined using laser induced breakdown spectroscopy (LIBS). Moreover, new diagnostic based on the study of the material thermal response after a laser heating excitation is under development and will be described. This technique makes possible to determine the presence of a co-deposited material. Several layer parameters can be addressed. In particular, the depth can be estimated with micrometer accuracy and this opens the way to an assessment of the total quantity of co-deposited material.

These two techniques coupled with laser ablation could provide a unique and global system based on interaction of laser light with matter, and capable of providing key information that would ensure ITER operations with an efficient fuel cycle control.

In conclusion, the remaining still open questions will be presented, which have to be addressed in the following years in a possible ITER accompanying work programme.

## 2. Laser ablation: fuel inventory control

### 2.1. Principles and laboratory achievements

This technique of laser ablation is used at industrial scale to vaporize material and obtain controlled layer deposition or chemical analysis of surface components. It is also used to clean surfaces or cleanse hot cells by removing contaminated paints [9].

Laser interaction with matter has been also successfully applied on carbon and carbon layer coming from tokamak. Two types of treatments were tested. The first one uses thermal material heating [5] which leads to gaseous desorption whereas the other ablates the material [6]. In the following, we will concentrate in the ablation process. Layer ablation is obtained by carbon sublimation, e.g. raising rapidly the temperature sample surface at 4000 K. Pulse heating has to be as short as possible in order to get ablation without any particle and/or heat diffusion from the surface to the bulk material. Sub microsecond laser pulses are well adapted to the ablation process and they have proved to be efficient

for ablation process. Therefore, femtosecond and picosecond lasers are not currently developed to ablation purpose. In the experiment presented hereafter, laboratory results obtained with tiles from TEXTOR tokamak (FZ-Jülich, Germany) are presented. These graphite tiles are covered with co-deposited carbon layers of 50  $\mu\text{m}$  thickness at the maximum. A Nd-YAG laser has been used, operating in its second harmonic at 532 nm with uniform laser intensity across the laser spot. The pulse length was 100 ns and the co-deposited energy on the surface sample was adjusted by varying the laser spot dimension.

In Fig. 1, the results obtained for ablation of bulk graphite and of co-deposited carbon layers are presented. In this figure, the crater depth per shot measured by mechanical profilometry is plotted versus laser fluence. To get good depth accuracy, 1000 laser pulses are accumulated; the laser pulses jitter in energy being less than 5%. Due to differences on the thermal properties, the two materials

show different ablation threshold fluences ( $F_{\text{th}}$ ). For the bare graphite material  $F_{\text{th}}$  is 2.5  $\text{J}/\text{cm}^2$  whereas is five times less for the co-deposited layer (0.5  $\text{J}/\text{cm}^2$ ). For given laser characteristics, the threshold energy for layer ablation remained the same in air and in inert gas (Ar) atmospheres (see Fig. 1(a)).

$F_{\text{th}}$  depends on the laser pulse duration and reduces to 1  $\text{J}/\text{cm}^2$  for bare Tore Supra graphite with the same laser but with a 5 ns pulse duration. This is mainly due to the differences in the heat propagation during the laser pulse and thus in the depth of heated material.

Thanks to the observed different ablation threshold fluences, the ablation process is an auto-limiting one. Indeed, if the laser fluency is set to a value that is above the layer  $E_{\text{th}}$  but lower than the bulk material  $E_{\text{th}}$ , the ablation process will take place only if the layer is still present and will stop when the bulk surface is reached. During this selective experiment in which only layers are removed, no bulk modification and/or destruction have been experienced.

## 2.2. Tokamak development and in-situ demonstration at JET

In order to develop a dedicated laser system which can be embarked on tokamak remote handling tools, several constraints have to be fulfilled.

The first one is the use of optical fibre in order to transmit the laser light. However, the in-situ robot positioning related to the PFC surfaces is a complicated issue. The distance between the laser device and the wall has not to be too short and an accuracy of several centimetres has to be assured. To achieve these constraints, an Ytterbium fibre laser has been chosen. This laser operated on a fundamental wavelength of 1060 nm, with 20 kHz pulse repetition rate, 120 ns of pulse duration and 20 W of average power. The beam divergence at the exit of the fibre is limited that allows a focalisation length of  $40 \pm 2$  cm. The laser fluence on the ablated surface was 2  $\text{J}/\text{cm}^2$  with a beam diameter of 250  $\mu\text{m}$ . During the ablation experiment, a galvanometric scan is used to move the laser spot on the treated sample with steps of 25  $\mu\text{m}$ . Results of this automatic ablation treatment on a TEXTOR tiles are presented on Fig. 2.

Two square zones ( $10 \times 10$  mm<sup>2</sup>) have been treated. In both cases layers have been completely removed. The best operational result is obtained for the right square, where the 50  $\mu\text{m}$  layer is removed in a single scan. The extrapolation of this

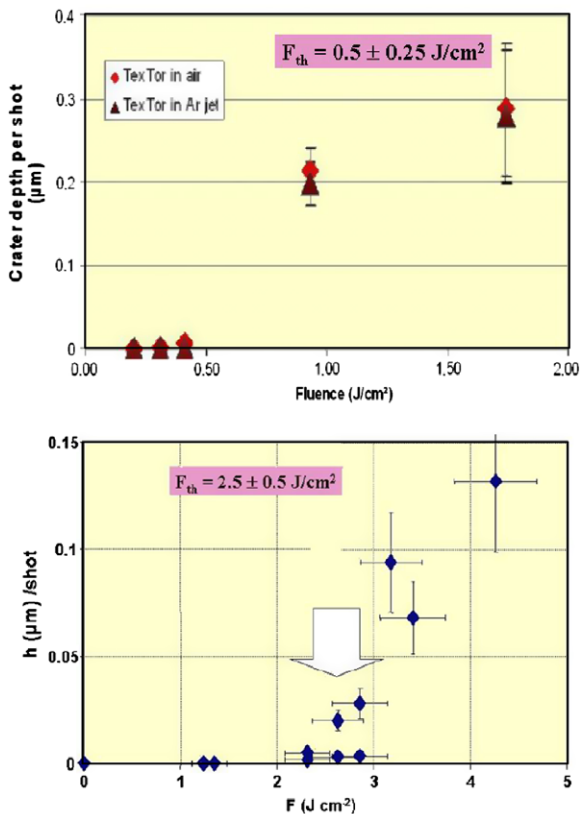


Fig. 1. Crater depth/shot vs laser fluence  $F(\text{J}/\text{cm}^2)$  for: (a) a co-deposited carbon layer on TEXTOR tile and (b) for a pure graphite surface from TORE SUPRA. Nd-YAG laser (532 nm, 100 ns pulse duration, 10 kHz, 1000 shot per crater).

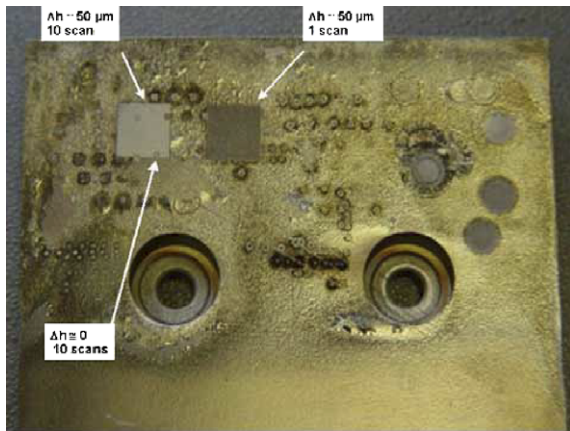


Fig. 2. Treatment of a TEXTOR tile covered by a thick layer of 50  $\mu\text{m}$ . Two zones treated shown.

result to 100 W laser gives a cleaning efficiency of about  $1 \text{ m}^2/\text{h}$  in a single scan for a deposit of the same thickness. The total quantity of graphite removed was then 100 g/h. This value has to be compared with the value needed for ITER overnight detritiation which is 400 g. As quoted in the introduction, if the ITER co-deposited layers are distributed in area as at JET, a laser ablation system installed on one (or more) robot could accomplish this task overnight.

This ablation device has to be tested in a real tokamak environment and a dedicated apparatus has been designed for trials at JET, using the available Remote Handling boom. It is thus also foreseen, to assess the capability of this dedicated apparatus in a mock-up of the JET divertor. The in-vessel tests may be possible in occasion of next long shutdown in 2009.

In the meanwhile, tests will be undertaken during 2006 spring in the JET Beryllium Handling Facility using the device shown on Fig. 3 and tritiated tiles from JET PFCs. Detritiation evaluation is foreseen with surface characterisation techniques such as ion beam analysis [10], calorimetry [11] and combustion of cored samples [12].

As it has been shown, the efficiency of the laser ablation technique is ITER compatible. The galvanometric scan has to be actively cooled in order to operate in the ITER environment and this is achievable. However, there are still some pending issues: this system is currently not compatible with operation in a permanent magnetic field.

Dust collection after laser ablation appears to be one of the major issues to be solved before implementing this technique in the tokamak environ-

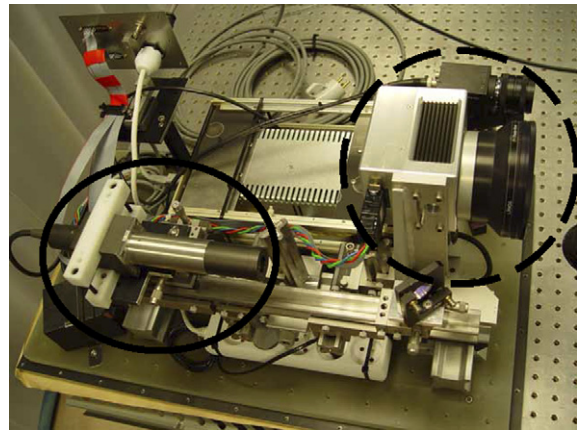


Fig. 3. Special device built in order to detritiate tritiated tiles in the JET BeHF. On this picture, the Ytterbium fiber laser output is surrounded by a solid line and the galvanometric scan by a dotted one.

ment. R&D studies are on going and nowadays there is no satisfactory technique. This dust collection could be done with an embarked aspirating system that will have also to work under magnetic environment. In case, the aspiration system is positioned out of vessel in order to avoid field effects, studies must be undertaken to check the dust recovery capability of this remote suction system.

### 3. Tritium content in co-deposited layers: the LIBS technique

#### 3.1. Principles and laboratory assessment

Laser Induced Breakdown spectroscopy (LIBS) is a laser-based technique which provides non-intrusive measurements for qualitative and quantitative analysis of a material surface [13]. LIBS is based on the detection of the atomic lines observed in the plasma induced after a nanosecond (or less) laser pulse interaction with the material surface [14]. In the LIBS experiment, a small part of the surface material is ablated using laser pulses with fluence higher than the ablation threshold fluence of the material studied. Laboratory tests have been undertaken at the CEA Saclay in order to check the feasibility of such a technique to analyse tokamak characteristic materials. The experiment arrangement is shown on Fig. 4. The same TEXTOR tile used for ablation development was studied in this LIBS test.

The laser used is a Nd-YAG laser operating in its second harmonic at 532 nm. The pulse duration is

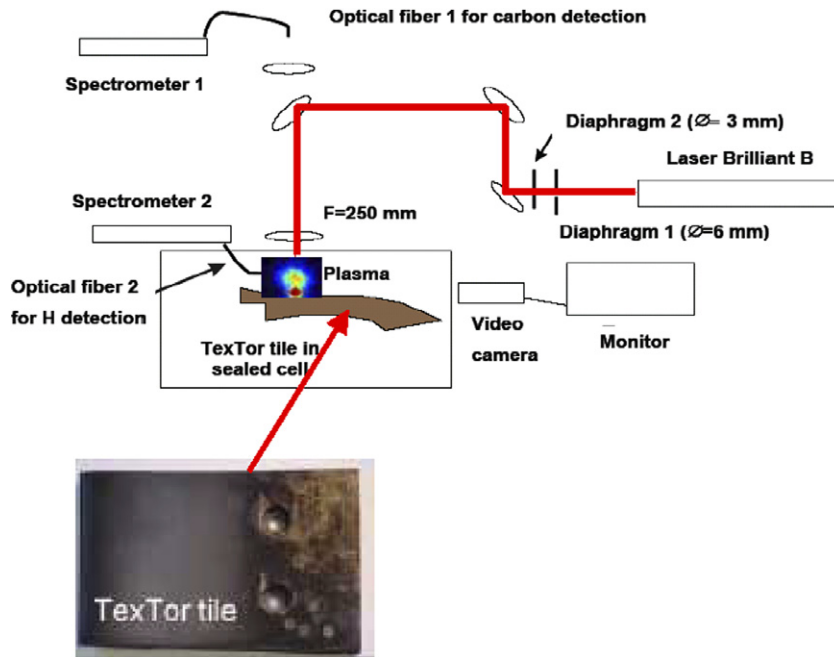


Fig. 4. Experimental arrangement for LIBS laboratory measurements on TEXTOR tile.

now 5 ns and, the energy fluency fluence is 10–20 J/cm<sup>2</sup> and the repetition rate is 10 Hz.

The spectral analysis of the plasma plume obtained after the material ablation is done with two spectrometers which are adjusted to detect the carbon line at 247.856 nm and the hydrogen line at 656.285 nm. The two spectrometers are equipped with intensified gated CCD camera (ICCD) in order to detect the time resolved spectral line intensity.

In Fig. 5, the spectral lines of hydrogen and carbon are shown together with several other elements present on the TEXTOR tiles (e.g. boron or silicon).

From these measurements, the ratio of the intensities of hydrogen and carbon lines is analysed for different areas of the treated tile. The first one (see Fig. 6(a)) corresponds to the front side (facing the plasma) of the tile. The second one (Fig. 6(b)) corresponds to the backside of the same tile. These signals are presented versus the number of laser pulses. The total duration of the experiment is 60 s, i.e. 600 pulses. The laser beam was Gaussian and the crater profile obtained at the end of the experiment is conical (depth ~ 190 μm, diameter ~ 350 μm and cone angle ~ 70°).

From these curves, it appears that the lines of carbon and hydrogen are seen during all the experiment. During the 30 first pulses and for the side which is facing the plasma and which is apparently

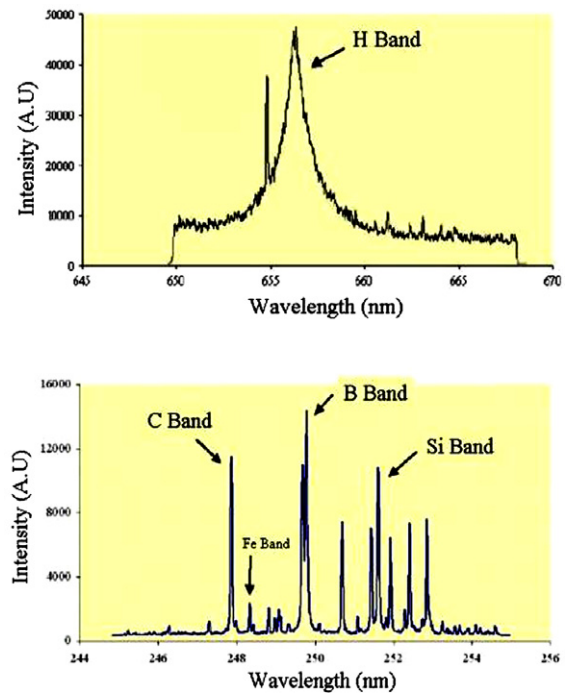


Fig. 5. Spectra obtained for Hydrogen and for Carbon, Boron, Silicon and Iron for a TEXTOR tile. 1200 spectra accumulated.

covered by a co-deposited layer, the H/C ratio is 3–6 times higher than for the ratio observed of the back

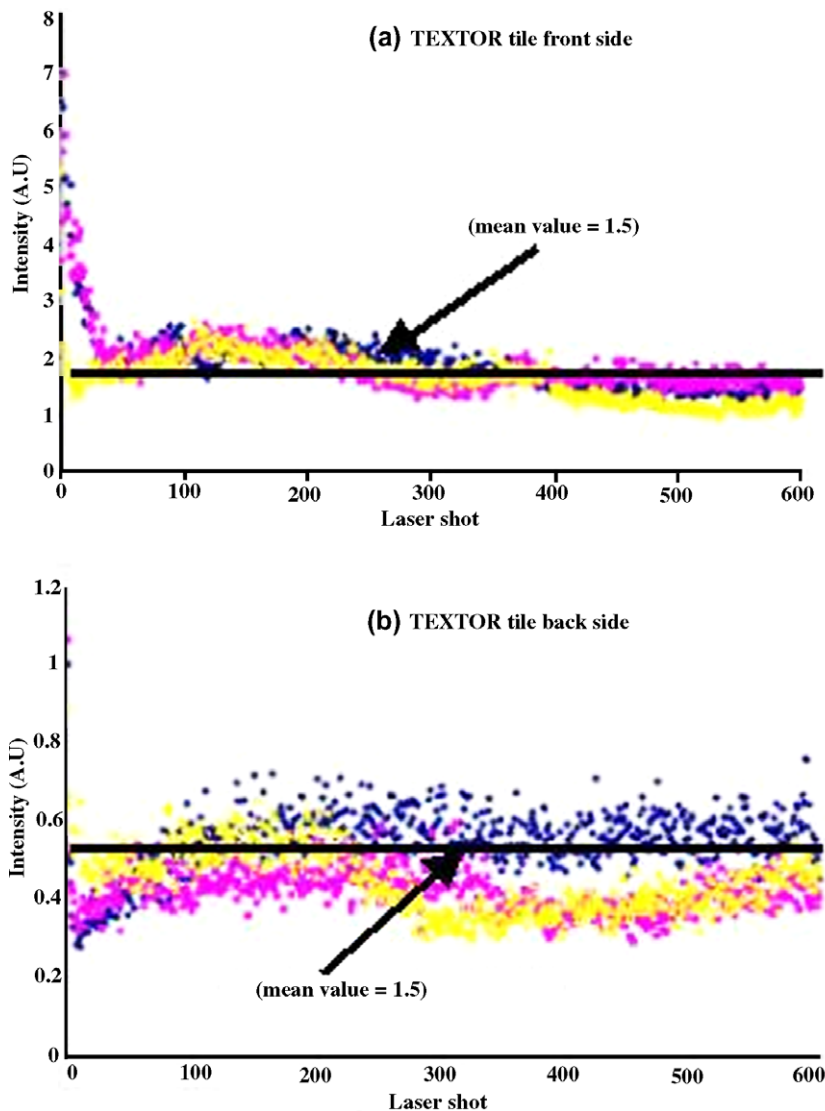


Fig. 6. Ratio of the H/C line intensity during LIBS experiment on TEXTOR tile for: (a) the side facing the plasma and (b) the back side. These results are presented versus the number of laser shots.

side which is free of co-deposited layer. However, this value decreases rapidly and before 100 laser pulses are performed, the H/C ratio obtained is very close to the corresponding ratio of the backside. As seen on Fig. 5, several impurities are also observed during this experiment. B, Fe, Si and Cu traces are detected during 300 pulses for the front side and only three pulses for the backside of the tile. The results presented above correspond to experiments performed in air at atmospheric pressure. However, same trends are obtained if they are done under an Argon atmosphere.

As a conclusion of these laboratory tests, it appears that LIBS is a potential tool for co-depos-

ited layer analysis. Several optical lines can be used for the estimation of the upper limit of the tritium concentration in the layers.

However, these preliminary results, even if they appear to be suitable for tritium concentration evaluation, are at present only qualitative. Quantitative values could be obtained using a calibration process in which the results obtained for tokamak co-deposited layers have to be compared with results obtained with well characterised layer which could be obtained from in-situ tokamak samples chemically analysed or from laboratory samples prepared with known T/D/H concentration. It has to be pointed out that, even if calibration with dedicated

sample could be easily implemented, studies are ongoing to avoid calibration by using model calculations which, from the line intensity, could give absolute value of material components.

Another important issue that needs to be studied is the influence of the pulse duration on the observed results and, in fact, on possibly induced changes in the material composition. Indeed, with a too long laser pulse, the heat pulse could propagate through the material and modify the composition via particle diffusion. This is usually an important process impacting tritium trapping in CFC [15]. Studies have to be undertaken in order to determine the needed duration for the laser pulse (nanosecond versus picosecond or less) and the compatibility of the proposed laser technique with remote handling facilities.

### 3.2. First JET proof of principle

In order to test if LIBS experiments could be considered in tokamak environments, a series of tests has been undertaken at JET using the ex-situ

LIDAR laser [16]. This laser has been fired to the inner divertor leg as shown in Fig. 7. The intensity of the light emitted by the plasma created by the laser/matter interaction is recovered with a photoelectric electron-multiplier tube (PM) during all the experiment. During these preliminary tests, the light observed was not spectroscopically analysed, only its intensity was monitored.

It is observed that the PM intensity is decreasing by a factor of 5. This corresponds with the trends observed in laboratory even if the composition of the different ablated materials during each laser pulse is not available.

Several new activities are expected in 2006–2007 in order to confirm these preliminary observations by implementing the spectral resolution of the recovered light. These positive JET tests have shown that LIBS could be implemented in tokamak with a laser firing from outside the vessel via optical windows. However, in order to get more ITER relevant information (e.g. homogeneity, tritium concentration on hidden surfaces), this technique must use optical fibres for laser light transfer.

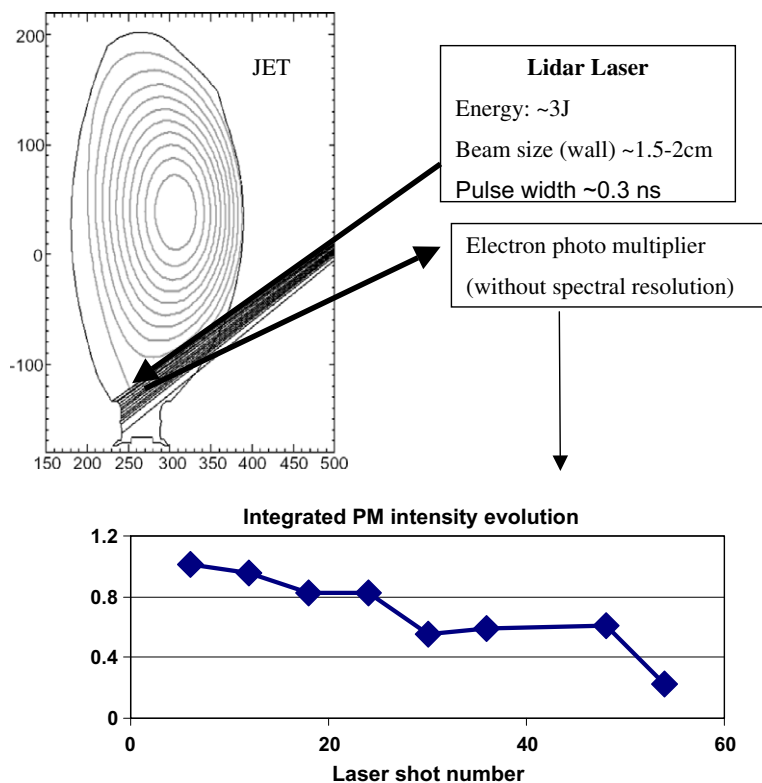


Fig. 7. Schematic of the LIBS preliminary tests done using the JET LIDAR laser. The intensity of the PM signal is also presented versus the number of lasers pulses.

#### 4. Total quantity of co-deposited carbon estimation: the laser heating technique

The determination of the total quantity of carbon co-deposited on the tokamak tiles and the depth of the co-deposited layers can be obtained from the careful study and interpretation of the surface temperature increase after interacting with repetitive laser pulses having fluence lower than the ablation threshold.

The surface temperature of a tile covered with a layer and submitted to a repetitive laser heating pulse is presented in Fig. 8.

The observed time evolution is fitted with a 3D analytical model which is detailed elsewhere [17].

In this model, thermal properties of the bulk graphite are taken from the literature. The thermal properties of the layer are deduced from the bulk properties and from an estimated value of the layer porosity. In order to reduce the number of unknown parameters of the model, the layer porosity of  $\sim 25\%$  has been deduced from the comparison of the flakes density evaluated at JET [18] ( $\sim 1.69 \text{ g/cm}^3$ ) and the mono-crystalline graphite density ( $\sim 2.24 \text{ g/cm}^3$ ).

In this 3D model, several unknown parameters are used to determine the depth of the layer:

- The mean absorption coefficient of the laser light by the co-deposited layer. It is obtained from the value of the ablation threshold fluence of a comparable material interacting with the same laser as used in the heating process. As expected, the obtained value is very close to the mean absorption coefficient for manufactured graphite at 532 nm.

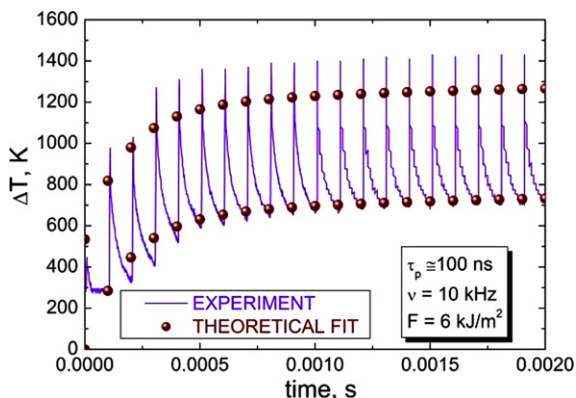


Fig. 8. Temperature evolution of the surface of a tile submitted to a laser light interaction (Nd Yag laser, 10 ns, 10 kHz and  $0.6 \text{ J/cm}^2$ ). Points represent the model fit. For sake of clarity, only few points of the theoretical fit are plotted.

- The adhesion of the layer to the substrate.
- The thermal conductivity of the layer.

Due to the small laser pulse duration, the layer can be considered in the model as a semi-infinite medium during the time of the laser heating process. Therefore, the time evolution of the surface temperature after one laser pulse gives information on the layer thermal properties and on its porosity.

The value of minimum and maximum temperature at the equilibrium as well as the time needed to get this min/max temperature gives the possibility to estimate the adhesion coefficient and the layer thickness.

The value obtained for the co-deposited layer depth is  $7 \mu\text{m}$  with an accuracy of  $1 \mu\text{m}$ . This estimation is very close to the thickness of the layer measured with an optical microscope after the laser tests:  $4 \pm 2 \mu\text{m}$ . This heating technique is clearly a suitable diagnostic which could be used in-situ to estimate the co-deposited layer thickness. It has to be kept in mind that actual co-deposited layer thicknesses are much larger than  $10 \mu\text{m}$ . In that case, laser heating diagnostic could be very well adapted due to its high accuracy.

#### 5. Depth deposition map diagnostic: LIBS and laser heating technique

The heating technique described above gives access to the layer thickness value. However, to get reliable results without using any assumption as the value of layer porosity or the coefficient of absorption of the laser light, the heating technique has to be used in combination with the LIBS analysis. This will be done in several steps.

The first one is based on heating and permits to get in one point of the tile the estimation of the layer thickness using the 3D calculation and assuming several layer parameters.

Then, for the same spot light, the laser pulse fluence is increased to get the layer ablation and the atomic lines observed in the plasma are analysed with the LIBS technique. As long as the layer is present, ablation is seen on the LIBS measurements. The total number of laser pulses necessary to get rid of the layer is recorded. Supposing that each laser pulse remove a known part of the layer thickness (this value is deduced from the  $E_{\text{th}}$  which is observed when ablation starts), the total thickness of the layer is obtained. It is then possible to compare it with the



value obtained during the heating step and to adjust if necessary the model parameters.

With this robust 3D model in which the parameters are now confirmed by ablation and the LIBS measurements, a deposition map can be obtained on the analysed tile supposing that the layer properties are homogeneous along the mapping. However, the crosscheck of the 3D model estimation during heating phase and the ablation process could be done in several locations of the tile in order to overcome the in-homogeneity of the layer properties.

With these coupled techniques, the total quantity of the co-deposited material is known as well as the concentration of tritium in the layer. In the course of the ITER operation, the area of deposition could be determined with its carbon total quantity and its tritium concentration. This will give the possibility to know when detritiation has to take place and how long this process will last.

## 6. Conclusions and open questions which still to be addressed

### 6.1. Heating coupled with LIBS/Ablation

In this paper, the capability of the heating diagnostic coupled to the LIBS analysis to address the co-deposited layer parameters as the layer thickness has been presented. The estimation of the upper limit of the tritium concentration in the layer has been demonstrated. These techniques, based on laser interaction with matter, are under development in CEA laboratory and investigations on real tokamak material have been undertaken. These results are of major importance for the detritiation process since the total quantity of carbon to be removed can be determined. Moreover, the quantity of tritium trapped in these layers can be also obtained and this information will help to close the fuel cycle.

These techniques have now to be experienced in a tokamak, embarked on a robot, to check if they are compatible with this specific environment. These tests will be included in the work programme of several EU tokamaks.

Present tokamaks are operating in a carbon environment. In the future of JET, it is foreseen to turn to an all-metal machine. In ITER a mixture of Carbon, Beryllium and Tungsten is expected for the PFCs. These new techniques must be checked with mixed materials and metal surfaces. Some work could be envisaged with beryllium tiles coming

from the old JET beryllium divertor and these studies have to be undertaken in the immediate future.

### 6.2. Ablation

The high efficiency of the laser ablation technique has been shown. Treatment efficiency of more than  $1 \text{ m}^2/\text{h}$  for a layer of  $50 \text{ }\mu\text{m}$  has been already achieved with an industrially available laser. This quantity removed is not so far from what is supposed to be treated overnight in ITER. It is also comparable with the treated quantity achieved using flash lamp but higher flexibility is foreseen with laser that can have access to narrow and hidden structures.

The adaptation to JET remote handling capabilities is under development but already the lasers seem easy to embark on robots. The galvanometric scan currently used cannot operate under magnetic field but robot operation experiences the same difficulty. Developments are needed in order to overcome this constraint and studies are ongoing to replace the galvanometric scan by a system compatible with magnetic field.

However, several open issues need to be addressed before the ITER start-up or during the course of its operation before the DT phase:

- Can the ablation characteristics observed for pure carbon material be extrapolated to more complex materials as mixed one (Be/C/W)?
- It is easier to detritiate a tokamak by ablation if the deposits are concentrated in small areas like in JET, for instance, where they are located under the inner leg of divertor. Therefore, it is of major importance to know where the deposition areas are located in the vessel. Are the current codes used for particle erosion and transport able to predict where the co-deposited area will be in ITER? Are they capable to predict when the in-vessel T tritium inventory will exceed the safety limit and what is the periodicity needed for the ablation treatment? Unfortunately, at present, the answer seems to be both negative. A strong effort is needed in order to get reliable answers to these two important points.
- Dust recovery during ablation is a complex problem and has to be addressed. If dust recovery is based on suction system, is this process compatible with the ITER gas management system?

In several machine, almost 50% of the injected fuel is lost in the wall during plasma operation

[19]. The process of trapping seems to be unclear. It is supposed that the co-deposited layers trap the majority of the fuel. However, it cannot be excluded that deuterium/tritium is trapped by diffusion inside the CFC itself. This hypothesis seems to be corroborated by some studies [15] but also by Bekris' results [11] who has observed deep diffusion of tritium in JET tiles. Substantial tritium amounts were even measured into the bulk of the tiles several centimetres below the plasma exposed surface. This phenomenon is more important in CFC than in pyrolytic graphite probably due to surface diffusion within the voids of this highly porous material. If it turns that tritium diffusion is the source of the fuel lost, laser ablation will be completely inefficient to control the ITER fuel cycle as well as all the other treatments that are at present proposed. Graphite and especially CFC will have to be removed from fusion machine or diffusion transport barrier implemented as proposed in [20] at this conference.

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