Laser Methods Development for In-situ ITER Walls Detritiation and Deposition Layers Characterisation

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Abstract. Laser methods for tokamak surface detritiation and characterisation have been under thorough multi-aspect experimental and theoretical study at LILM laboratory (CEA Saclay, France). The graphite surface characterisation and cleaning with nanosecond pulsed high repetition rate laser systems were successfully realised. Heating and ablation regimes were distinguished by ablation threshold fluence. Due to the significant difference in ablation thresholds for graphite (≈ 2.5 J/cm^2 for 100 ns laser pulses) and a deposited layer (≈ 0.4 J/cm^2) it was possible to clean the graphite surface without its damage, if the laser fluence is lower than the ablation threshold for graphite. The ablation efficiency of the TEXTOR deposited layer was 0.1 μm^3/J. The developed laser device is very flexible and can be fixed on a robot for in-situ surface characterisation and detritiation. It will be installed onto AIA (Articulated Inspection Arm) robot on TORE SUPRA. The ablated matter will be aspirated by the nozzle fixed on AIA and collected on filters. The integrated pyrometer system will be applied to record the surface temperature in laser heating regime (laser fluence below 0.4 J/cm^2) with a high repetition rate Nd-YAG laser. The flexibility of the developed laser system is an important advantage. The same laser system (by adjusting appropriately the laser beam energy and spot) may switch from heating regime (deposited layer depth estimation by pyrometer method) to ablation (where the layer depth is directly measured from the total ablation time) and to Laser Induced Breakdown Spectroscopy (LIBS) with laser plasma plume formation. A good agreement was demonstrated between the experimental results and the developed theoretical 3D model of surface heating (graphite + layer) that allowed to determine the deposited layer depth with micrometric accuracy. The preliminary studies have shown that LIBS might be suggested to estimate tritium concentration in the material. Further goals and tasks to satisfy ITER requirements are presented and discussed.

1. Introduction

The trapping of tritium in carbon-fiber composite (CFC) of plasma-facing components (PFC) is seen as a real problem for the efficient operation of a fusion device. For the ITER installation, PFC treatment is regarded as inevitable to satisfy safety requirements and tritium inventory control. Thus, in-situ characterisation of a deposition layer (tritium quantity and surface distribution, thickness, composition) and deposition layer detritiation are of major importance for tokamak installations with carbon as PFC component. The severe environmental conditions (vacuum, high magnetic field of some Tesla and high temperature of 450K) should be under special consideration. To find the appropriate solution for PFC treatment and tritium inventory control, a number of technological methods and techniques (deposited matter oxidation [1-3], flash lamp light/matter interaction [4], laser-based technologies [5-8] such as laser ablation (LA), laser heating, Laser Induced Breakdown Spectroscopy (LIBS) and combination of these methods) have been under investigation. The application of laser methods for future ITER surface treatment can offer a number of advantages for PFC treatment and tritium inventory control. Laser methods can provide a completely optical surface characterisation and cleaning. The optical fibers can ensure a flexible transport of the laser beam to the cleaning zone and of the optical diagnostic signal (laser plasma or thermal radiation) from the treated zone. Thus, it is possible to remove both the laser and detection systems (spectrometer or pyrometer) from the contaminated zone. The laser beam transport by the optical fibers can provide an easier access to gaps and hidden areas to control the trapped tritium. Being installed on a robot, the laser device can provide the remote handling that is seen as an additional advantage. The developed and commercially available powerful pulsed high repetition rate solid state lasers (where radiation transport is provided by the optical fibers) are seen as good candidates for decontamination of the vacuum chamber surfaces in tokamak thermonuclear installations.

Since 2003, thorough multi-aspect experimental and theoretical investigations of different laser methods for deposited layer detritiation and characterisations [6-8] have been made at LILM laboratory (CEA Saclay, France). LA, LIBS, laser heating and combination of these methods have been under special consideration. The investigations were made with TORE SUPRA, TEXTOR and JET graphite tiles without a deposited layer and with the layer of 5-300 µm thickness. A pulsed (100 ns duration) high repetition rate (10-20 kHz) solid state (Nd-YAG and Yb-doped fiber) laser systems were applied.

The development of laser methods to characterise and detritiate the graphite deposited layers without damaging the substrate surface was the main aim of our investigations. The necessary experimental equipment was developed. Both graphite surface characterisation and cleaning were successfully realised. The results of our study may be considered very promising. Further goals and tasks to satisfy ITER operation and safety requirements will also be defined.

2. Laser Ablation

At present, LA is widely applied for matter vaporisation and controlled surface layer deposition, chemical analysis of the surface component composition, surface cleaning, and decontamination [9]. LA is very efficient for carbon deposition layer removal in modern tokamaks [6-8]. LA, in this case, was based on carbon layer evaporation/explosion obtained by a rapid (of the order of 100 ns) temperature increase (up to 4000 K) on the sample surface. For a number of passed years, laser methods for detritiation and characterisation have been under multi-aspect experimental and theoretical study in CEA, Saclay. Nd-YAG pulsed laser systems (5ns and 100 ns pulse duration, 532 nm) were applied to study TEXTOR tiles and TORE SUPRA PFC samples. The deposited energy fluence on the surface sample was adjusted either by the laser pulse energy (5 ns laser) or laser spot diameter (100 ns lasers).

Fig. 1 presents the results for LA of both bulk and deposited layer materials. The crater depth is plotted versus the laser fluence. As the surface roughness was of some micrometers, 100-1000 laser pulses were accumulated to obtain the crater depth per pulse with a sufficiently high accuracy. The environmental conditions (air or argon at 1 bar) did not affect the results of the experiments. For the graphite surface without a deposited layer, the ablation threshold was determined as $F_{th} = 1.0 \pm 0.3$ J/cm^2 for 5 ns laser and $F_{th} = 2.5 \pm 0.5$ J/cm^2 for 100 ns laser (**Fig. 1a**). For TEXTOR graphite tiles with a thick deposited carbon layer of 40±10 µm thickness, the ablation threshold was $F_{th} = 0.4 \pm 0.1$ J/cm^2 and was approximately the same for 5 ns and 100 ns pulses (**Fig. 1b**). Such a noticeable (5-fold) difference in the ablation threshold fluence F_{th} of the bulk and deposited materials for 100 ns LA is explained by their different thermal properties.

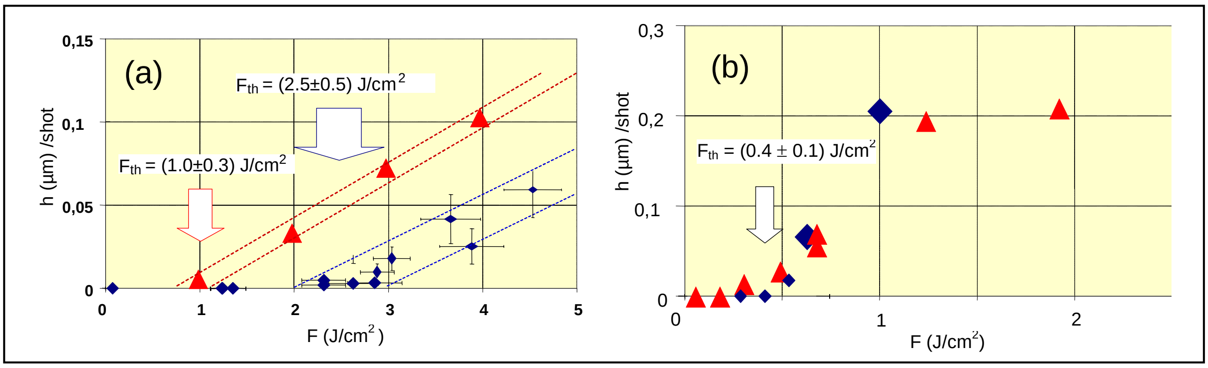
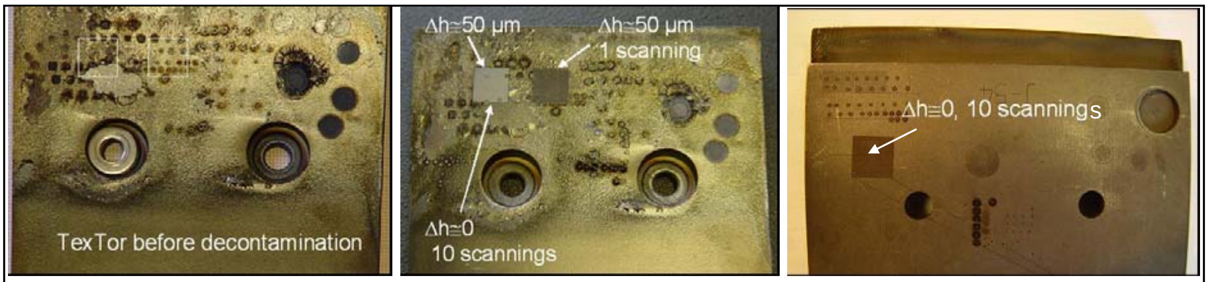


Fig. 1. *Crater depth h(µm/shot) versus laser fluence F (J/cm ^{2}) in air (1 atm) for pure TORE SUPRA graphite (a) and TEXTOR thick co-deposited layer (b).*
(▲) – low (20 Hz) repetition rate laser, 4 ns pulse duration, 532 nm, homogenized beam;
(◆) - high (10 kHz) repetition rate Nd-YAG laser, 100 ns pulse duration, 532 nm, homogenized beam.

The experimental ablation thresholds for pure graphite (**Fig. 1a**) are in a good agreement with the theoretical values obtained with the numerical 3D-model of high repetition rate laser heating that was developed in our laboratory [10-11]. The ablation rates for graphite were described by the Stefan-like model for LA of sublimating/evaporating materials. This model is based on numerical solution of the heat equation in one dimension with the Stefan boundary condition [12-13] on graphite surface, since the one-dimensional approach on the time scale of 100 ns (laser pulse duration) is relevant to our experimental conditions. The three-dimensional effect of preheating (that can be important for high repetition rate and a large number of laser shots applied for crater formation) is incorporated in the model.

Laser ablation rate for TEXTOR deposited layer was ≈ 0.2 µm/pulse for laser fluence in the range of (1 - 2) J/cm^2. Thus, laser cleaning rate is $\mathbf{V(mm^3/sec) = \eta(mm^3/J) \times P(W)}$, where $\eta(mm^3/J) \approx 0.02\ mm^3/J$ is the ablation efficiency of the TEXTOR deposited layer and P(W) is the mean laser power. Such a high ablation rate is related with the explosive character of the deposited layer ablation with nanosecond high repetition rate laser beam. In this case, the main ablation mechanism is seen as micro particles ejection due to thermal stresses in a non homogeneous deposited layer. This particular feature of graphite deposited layer cleaning may be of a special importance for the development of ablated matter aspiration system, collection and tritium recycling (that is beyond the subject of this paper). In near future, such a system will be developed and tested on TORE SUPRA tokamak in Cadarache (France).

Due to the significant difference in ablation thresholds for graphite and a deposited layer, it is possible to decontaminate the graphite surface without its damage if the laser fluence is lower than the threshold fluence (F_{th}) for graphite. That is, if the laser fluence is higher than F_{th} of a layer (0.4 J/cm^2 for 100 ns pulses) but lower than the fluence F_{th} of the bulk material (2.5 J/cm^2 for 100 ns pulses), the ablation will take place to the total depth of the layer up to the bulk surface. LA, in this case, may be considered as auto-limiting. To test the auto-limiting LA and laser cleaning rate on TEXTOR tiles, the necessary experimental equipment has been developed. The experimental ablation device was based on an Ytterbium fiber laser (20 W mean power, 1060 nm wavelength, 20 kHz repetition rate, 120 ns pulse duration, the Gaussian beam). The low beam divergence ($M^2 \approx 1.5$) at the fiber laser exit provided laser beam diameter $2a_o \cong 250$ µm (full width at e^{-1} intensity) in the lens focal position (450 mm from the focusing lens). The laser fluence on the beam axis in the waist was chosen as 2 J/cm^2. The Rayleigh length $\mathbf{L_R = 2\cdot\pi\cdot a_o^2/\lambda\cdot M^2} \approx 5$ cm of the focused beam was very large. Thus, the problem of laser/surface positioning was avoided. Two zones on TEXTOR tile with a deposited layer of 40 ± 10 µm thickness were under LA treatment (**Fig. 2b**). A rapid zigzag scan was applied to move the laser spot with a 25 µm horizontal step and a 125 µm vertical step on the treated zones (10 mm x 10 mm). For the right zone on **Fig. 2b,** a single scanning of 2 seconds was applied. For the left zone, such scanning was applied ten times. A complete layer removal without the substrate damage was observed for both the zones. The best cleaning rate 2 mm^3/s was obtained with a single scanning. Such performance corresponds to the ablation efficiency $\eta \cong \mathbf{0.1\ mm^3/J}$. In comparison with the ablation efficiency of **Fig. 1b**, a 5-fold efficiency increase is observed. These results confirm also the explosive character of graphite layer ablation. Thus, the results obtained with an Ytterbium fiber laser of 20 W mean power may be regarded as very promising for LA application in a future ITER installation.



(a) (b) (c)

Fig. 2. *TEXTOR tile with deposited layer of 40 ± 10 µm thickness: tile before cleaning - (a); tile with the right and left cleaned zones - (b). TEXTOR tile backside surface - (c). Interaction parameters: laser fluence – 2 J/cm^2, laser mean power – 20 W, high repetition rate – 20 kHz, air at 1 bar, scanned zones of 10 mm × 10 mm for 2 sec.*

Further investigations are envisioned. The developed LA device should be thoroughly tested under real tokamak operating conditions and in JET vessel. The first tests with JET tiles with the developed laser device in JET Be-Handling Facility (BeHF) were successfully made in June, 2006. The preliminary results suggest that LA is efficient for a complete deposited layer (50-300 µm depth) removal without graphite (CFC) tile damaging. The estimated ablation efficiency with the JET deposited layers was 0.1 mm^3/J or higher. For future in-situ studies on surface cleaning and characterisation on TORE SUPRA, the developed laser system will be installed onto Articulated Inspection Arm (AIA) robot [14]. The ablated matter will be aspirated by the nozzle system fixed on AIA and collected on the aspirator filters. In-vessel tests in TORE SUPRA are scheduled for 2008.

3. Repetitive Pulsed Laser Heating

A new laser diagnostics based on the surface temperature response to the repetitive pulsed laser heating of the sample surface has been proposed and developed for high repetition rate laser heating of a graphite surface with a deposited layer. This diagnostics was evolved as a result of the development of a special pyrometer system and an adequate numerical model. The diagnostics was demonstrated experimentally as promising. The depth of the carbon deposited layer on the Tokamak tiles can be determined from the accurate analysis of the surface temperature time behaviour that resulted from the surface heating by repetitive laser pulses with the fluence lower than the ablation threshold. A high repetition rate Nd-YAG laser ($F=0.1\text{-}0.4\ J/cm^2 < F_{th}$, 10 kHz, 100 ns, 532 nm) was applied for the laser heating in our experiments. The surface temperature was measured by the integrated pyrometer system. The surface temperature evolution in time was compared with calculation results obtained with the developed laser heating model. The 3-D analytical model of material (graphite + layer) heating was developed on the basis of the data from the available literature, certain assumptions and deduced parameters. For laser heating, the laser pulse duration is very short (100 ns) and it is possible to consider the layer as a semi infinite medium on a single laser pulse scale. Thus, the surface temperature evolution in time just after one laser pulse provides the layer thermal properties and porosity. The minimum and maximum temperatures during the repetitive pulsed laser heating and the time required to reach these temperatures allow to estimate both the layer/surface adhesion coefficient and the layer thickness [11]. A good fit with the experimental data was obtained. Thus, the layer depth was determined as 7 µm with 1 µm accuracy. This value was very close to 4±2 µm layer thickness measured with an optical microscope after LA of the layer. Our investigations demonstrated that the repetitive pulsed laser heating can be considered as a suitable and reliable diagnostics for in-situ estimations of thickness of the carbon layers deposited on PFC. It can provide high accuracy estimations of the deposited layer thickness (>10 µm) in real Tokamak installations.

4. Laser Induced Breakdown Spectroscopy

Laser Induced Breakdown Spectroscopy (LIBS) is a laser method for almost non-destructive qualitative and quantitative analysis of surface matter composition [15]. It is based on atomic lines detection in plasma induced by nanosecond (or shorter) laser pulse interaction with the surface material. The method may schematically be presented as follows. Laser radiation being focused onto the target surface results in surface ablation and laser plasma formation. Laser plasma composed of the excited particles (atoms, ions) of the ablated matter provides a source of radiation that might be used to characterise the matter under study. The plasma spectral analysis allows to determine the elemental composition of the analysed sample.

Our laboratory investigations were aimed to study LIBS for TEXTOR tile characterisation [16-17]. A high repetition rate Nd-YAG laser (532 nm wavelength, 5 ns pulse duration, 10 – 20 J/cm^2 energy fluence, 10 Hz repetition rate) was applied. The experiments were made in air and argon environment at 1 atm. 600 pulses of the laser beam (the Gaussian distribution) were applied to obtain LA on the TEXTOR tile. The obtained crater profile was conical with ≈ 190 µm depth and ≈ 350 µm diameter. High power ($F = 10 – 20\ J/cm^2$) laser beam interaction with the sample surface resulted in the laser plasma plume formation that was studied by the spectral analysis. Two 1-meter focal length spectrometers (Jobin-Yvon) were adjusted to detect carbon (247.856 nm) and hydrogen (656.285 nm) atomic lines that have been observed in the TEXTOR tiles under investigation. The spectrometers were equipped with a gated ICCD camera (Hamamatsu C4346-01, 200-800 nm spectral range) to detect the time resolved spectral line intensity.

The ratio of H/C spectral line intensity (plotted versus the number of applied laser pulses) for the front (facing the plasma) and back side of the same tile revealed the following [16]. Both C and H lines were observed during the whole experiment (60 sec, 600 pulses). For the first 30 laser pulses, the front side with a deposited layer demonstrated a 3 - 6 fold H/C ratio if compared with the ratio on the back side (without a layer). This ratio decreased very rapidly. Even up to 100 laser pulses application, the H/C ratio was almost the same as the one on the back side of the tile. B, Fe, Si, and Cu impurity traces were observed during 300 laser pulses for the front side and, in contrast, during only 3 pulses for the back side. The experiments were performed in air (1 atm). Argon environment did not affect the results for the H/C ratio.

Our laboratory investigations on TEXTOR graphite tiles have demonstrated LIBS as a potential method for the optical analysis of the spectral lines for H, C, and other impurities in a deposition layer. Thus, we can conclude that LIBS can be applied to estimate tritium concentration in a deposited layer in a future ITER. However, the obtained preliminary results should be considered only qualitative. A number of problems should be resolved till this method is applied for in-situ measurements: a) homogeneous and controllable in-depth LA; b) possible hydrogen concentration changes in a near-surface zone due to its high diffusivity at high temperature; c) feasibility of LIBS with 100 ns pulses on a frail deposited layer or with ultra short (sub nanosecond) pulses transported by an optical fiber. These problems are under intensive studies in our laboratory.

5. Laser Heating/Ablation Coupled with LIBS

The combination of various laser techniques can offer a number of promising applications. Laser heating/ablation coupled with LIBS can be suggested as the combined technique to obtain reliable results on the deposited layer parameters (deposited layer thickness, tritium concentration in a deposited layer and total carbon quantity). Thus, the possibility to obtain the exact parameters allows to reduce the number of inevitable theoretical assumptions and required deductions of the layer porosity and laser light absorption coefficient in the applied 3-D analytical heating model.

Laser heating/ablation in combination with LIBS can be presented as a multi-step procedure. Laser heating is first applied to a certain zone on the tile to estimate the co-deposited layer thickness with the 3-D analytical heating model with the assumption of some layer parameters. Then, with the laser spot diameter decrease, the laser intensity is increased to obtain laser plasma. LIBS is applied to observe the atomic lines in the plasma plume and to register the analytical lines until the deposited layer disappears from the surface. The total number of laser pulses required to remove the layer is recorded. Supposing that each laser pulse removes a known part of the layer thickness, it is possible to obtain the total layer thickness. This layer thickness is compared with the one obtained during the heating. Based on this comparison, the 3-D model parameters can be adjusted if necessary. Then, the 3-D model parameters are confirmed with LA and LIBS measurements. With the supposition that the layer properties are homogeneous on the whole mapping, one can make a deposition map of the tile surface under investigation. Nevertheless, during laser heating and LA, the additional verification of the estimations obtained with the 3-D heating model might be required in some tile zones to reduce the non-homogeneity of the deposited layer properties.

The technological aspects of the combined laser heating/ablation/LIBS method are of a special interest. It should be stressed that switching from laser heating (deposition layer depth estimation by pyrometer method, $F < 0.4$ J/cm^2) to LA (where the layer depth is directly measured from the total ablation time, $F = 1 - 3$ J/cm^2) and, finally, to LIBS with the laser plasma plume formation (laser intensity $I \approx 1 - 5$ GW/cm^2) can be realised with the same laser system by adjusting appropriately the laser beam energy and spot diameter. Laser heating/ablation in combination with LIBS can provide reliable data on the total carbon quantity in the deposited material and tritium concentration in the layer. Thus, the results obtained should be regarded as very important when considering the appropriate method for detritiation and its duration for a future ITER. For further investigations on the possible application of the combined method described above, the laser devices will be installed on a robot and tested in some EU tokamaks. The study on these techniques with mixed materials and metal surfaces is also envisioned.

6. Conclusions

Multi-aspect thorough experimental and theoretical investigations of different laser methods have been undertaken at LILM laboratory (CEA Saclay, France). LA, laser heating, LIBS and combination of these methods were under special consideration. The application of these methods for surface detritiation and characterisation was the main goal of our investigations. New experimental equipment has been developed and tested. TEXTOR and TORE SUPRA PFC samples were under study with high repetition rate, 100 ns duration laser pulses. For graphite samples, the ablation thresholds were determined both for graphite without a deposited layer (F_{th} =2.5 ± 0.5 J/cm^2) and TEXTOR deposition layer of 40 ± 10 µm thickness (F_{th} = 0.4 ± 0.1 J/cm^2). Due to the significant difference in ablation thresholds for graphite and a deposition layer, it is possible to decontaminate the graphite surface without its damaging if the laser fluence is lower than the ablation threshold fluence for graphite. For pulses with F = 1 - 2 J/cm^2, the efficiency of the TEXTOR deposition layer ablation is $\eta \cong 0.1$ mm^3/J. Thus, with the pulsed repetition rate lasers of 100 W mean power, a deposited layer of 40 µm thickness can be removed with the rate of one square meter per hour.

Laser facilities are very flexible and can provide access to shadowed narrow and hidden structures. This is seen as an important advantage for ITER. For in-situ studies on surface cleaning and characterisation of TORE SUPRA, the developed laser system will be installed onto AIA (Articulated Inspection Arm) robot. The ablated matter will be aspirated by the nozzle fixed on AIA and collected on the aspirator filters. The work to adapt the system to JET remote handling facilities is in full progress.

Our investigations have demonstrated that laser methods can offer an efficient surface cleaning and provide important data for the surface material characterisation. The results obtained may be regarded as very promising. However, one should mention a number of problems that should find the appropriate solution:

- robot and laser device operation in a very strong permanent magnetic field (of some Tesla) and at high temperature (450K) ;
- precise localisation of the deposited areas and the access to them in a future ITER;
- assessment of in-vessel tritium inventory and safety limit, the appropriate time and duration of detritiation;
- tritium diffusion and fuel lost assessment;
- operation of a dust aspiration system in a strong magnetic field during LA.

Further investigations are required. The studies with complex and mixed materials (Be/C/W) are also envisioned.

7. References

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