LIBS for tokamak plasma facing components characterisation: Perspectives on in situ tritium cartography

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ABSTRACT

Feasibility of in situ LIBS remote measurements with the plasma facing components (PFCs) from the European tokamaks (TORE SUPRA, CEA Cadarache, France and TEXTOR, Julich, Germany) has been studied in laboratory using Q-switched nanosecond Nd–YAG lasers. LIBS particular properties and optimal parameters were determined for in-depth PFCs characterisation. The LIBS method was in situ tested on the Joint European Torus (JET) in the UK with the EDGE LIDAR Laser System (Ruby laser, 3 J, 690 nm wavelength, 300 ps pulse duration, intensity up to 70 GW/cm^2). Several analytical spectral lines of H, CII, CrI, and BeII in plasma were observed and identified in 400–600 nm spectral range with the optimised LIBS and detection system. The LIBS in-depth cartography is in agreement with the surface properties of the tile under analysis, thus confirming feasibility of in situ LIBS. Further LIBS technique improvements required to provide tritium concentration measurements more accurately are discussed.

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1. Introduction

Tritium retention on plasma facing components (PFCs) of tokamak vacuum chamber is seen as a serious problem for a secure operation of modern nuclear fusion installations [1,2]. For consecutive detritiation of PFCs, tritium surface cartography with overall tritium contents determination is required. To achieve this goal, LIBS (laser-induced breakdown spectroscopy based on a spectroscopic analysis of the plasma produced by laser ablation of PFCs) is seen as a promising technique [3–6]. Our investigations were aimed to prove feasibility of in situ LIBS measurements. The laboratory studies were made with the graphite PFCs from the European tokamaks (TORE SUPRA, CEA Cadarache, France and TEXTOR, Julich, Germany) using Q-switched nanosecond Nd-YAG lasers, at 0.1–2 m from a target, under 1 bar, and reduced pressure. The in-depth spectral line emission profiles of hydrogen, carbon, and some trace elements (B, Ca, Cr, Fe, K, Li, Na, Ni, and Si) were obtained.

The LIBS method was in situ tested to analyse a JET tile (with eroded PFCs material deposited on top of a tungsten stripe) on the Joint European Torus (JET) in the UK with the EDGE LIDAR Laser System (Ruby laser, 3 J, 690 nm wavelength, 300 ps pulse duration, intensity up to 70 GW/cm^2) [6]. The LIBS in-depth cartography was in agreement with the surface properties of the analysed tile. Thus, the LIBS in situ proof of principle has been achieved.

This paper is organised as follows. Section 2 presents general properties of the LIBS method. Section 3 summarises some main nanosecond LIBS laboratory results. It also discusses both advantages and certain limitations of the method. LIBS test performances for JET PFCs characterisation are presented in Section 4. Discussion on possible improvements of the technique needed to measure in situ in-depth tritium concentration more accurately and also general conclusions are given in Section 5.

2. LIBS general features

In LIBS [7], a pulsed laser beam (nanosecond, picosecond or femtosecond) is focused to obtain high laser intensity ($I \geq$ GW/cm^2) on the analysed sample. The interaction of a powerful laser beam with a solid sample results in laser ablation (LA) with a crater formation on the sample surface and a near surface laser plasma plume creation. Laser plasma plume has typical dimensions of 1–10 mm and is composed of excited atoms and ions of the ablated matter. This plasma is of analytical interest for solid surface characterisation (elemental cartography or mapping) by spectral analysis of plasma emission. Spatial resolution of this method is associated with crater dimensions (diameter D_c and depth h_c). Crater diameter is mainly determined by laser beam diameter D_L, pulse energy E, and lateral distribution of intensity $I(r)$ and ranges from some micrometres (microanalysis) to some hundreds of micrometres (macroanalysis). Crater depth depends on sample properties, laser fluence, pulse duration, wavelength, environment gas composition, and pressure. Typically crater depth values ranged from some

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nanometres (femtosecond LIBS) to some micrometres (nanosecond micro-LIBS).

The laser/surface interaction is a complex process involving surface heating, melting, ablation, ablated atoms excitation and ionisation. It depends on a large number of laser parameters (laser pulse energy, spot size and duration, wavelength, laser beam angular divergence and polarisation), and on physical properties of solid sample. It depends also on environmental composition and pressure, and on the presence of magnetic or electrical field which can affect laser plasma plume spectroscopic properties. LIBS analytical signal (N_{ij}^a) for the given element (a) can be expressed by the relation:

$$N_{ij}^a \sim N_a A_{ij} \exp(-E_i/kT) u_a^{-1}(T) t_p K_d$$

where N_{ij}^a is the number of detected photons of the given element on wavelength λ_{ij}, N_a–the number of atoms of the given element in the plasma plume, A_{ij}–the Einstein coefficient of the $i \rightarrow j$ transition ($A_{ij} \approx 10^6 - 10^8 \, \text{s}^{-1}$), E_i–energy of the exited state i ($E_i \approx 2 - 10 \, \text{eV}$), $k = 1.38 \times 10^{-23} \, \text{m}^2 \, \text{kg} \, \text{s}^{-2} \, \text{K}^{-1}$–the Boltzmann constant, T–plasma plume excitation temperature ($T \approx 05$–$1.0 \, \text{eV}$), u_a–partition function for given element ($u_a \approx 0.1 - 0.01$), t_p–laser plasma plume lifetime ($t_p \approx 0.1 - 1.0 \, \mu\text{s}$), and K_d is the detection efficiency of the plasma photons ($K_d < 0.01$). It is supposed here that laser plasma is in local thermodynamic equilibrium (LTE) [8] that is generally satisfied for LIBS plasma at ≈ 1 bar pressure during its analytical lifetime (0.1–5.0 μs). In this case, excitation temperature equals electron temperature, and electron density is sufficiently high ($n_e \geq 10^{16} \, \text{cm}^{-3}$). In the first stage of plasma plume expansion, plasma continuum emission is very important. Gated ICCD cameras (0.1–1.0 μs delay, 1–10 μs gate time) are applied for LIBS analytical signal detection with improved signal/background ratio.

LIBS method sensitivity is low. Even for the most favourable case, $N_{ij}^a \approx 10^{-3} N_a$. Thus, in order to register one analytical signal (photon), more than 10^3 atoms in laser plasma plume are necessary. Taking into account that atomisation of ablated matter is only partial, an accurate optimisation of LIBS parameters is required to obtain LIBS signal with high amplitude. This consideration is of special importance, specifically for LIBS application in complex ITER-environment (vacuum, high magnetic field of 1–5 T, small angular aperture of the optical collection system, high excitation energy $E^* \approx 10 \, \text{eV}$ for hydrogen isotopes). To provide reliable LIBS measurements under these conditions, one may require optimisation of laser ablation system (laser, focalisation optics) and also of detection system (collection optics, spectrometer, optical detector) that were under consideration in our studies. The results of LIBS multi-parameter studies to solve the optimisation problems are presented in Section 3.

3. LIBS laboratory results

LIBS method in application to graphite tiles with deposited layers was under study in [3]. LIBS installation (6 ns laser pulses, 10–60 mJ energy, 532 nm wavelength) supplied with two spectrometers with gated ICCD cameras was developed. The application of two spectrometers (1000 mm focal length Cherny-Turner and Echell) allowed us to observe multi-element spectral lines. To avoid the effect of the atmospheric water hydrogen, a sealed cell was implemented to study LIBS spectral lines in controlled atmosphere (argon or air under 1 bar). Laser beam was focused on samples by lens of 100 or 250 mm focal length to a spot of 0.4–0.75 mm diameter.

For LIBS detection of hydrogen or carbon, sufficiently high laser fluence (10–20 J/cm²) was required. This requirement resulted from high excitation energy of the analytical transitions for hydrogen (H_α 12.088 eV) and carbon (7.685 eV). The experimental results demonstrated the necessity to "homogenise" laser beam. A relatively homogeneous laser beam was obtained with the "diaphragm aperture imaging" on the sample surface. The laser ablation rates were determined as 0.5 μm per pulse for the near Gaussian laser beam and as 0.25 μm per pulse for homogeneous laser beam with ≈ 20 J/cm² fluence. The crater depth was a linear function of the number of applied laser shots.

The argon effect on the spectral line intensities resulted mainly in the line intensity increase. The H/C ratio and line intensity changes with the pulse number were practically the same both in air and argon. The argon effect manifested itself as a black re-deposition around the craters. The black circular zone was not detected with ablation in air, but was observed in Ar. It resulted from graphite powder re-deposition. The graphite powder appeared in the laser plasma plume after its cooling and carbon condensation into micro-particles. Ar was considered to prevent carbon atoms oxidation. In air, oxygen gave rise to CO_2 and, thus, suppressed the black circle formation around the crater.

The analytical transition wavelengths for hydrogen (H_α 656.281 nm), carbon (CI 247.856 nm), and other impurities (B, Fe, Si, and Cu in 246–254 nm spectral range) were determined. The impurity spectral lines were observed only for the initial 5–20 pulses for thin (1–5 μm) deposited layers and even with the 100th pulse application for thick (≈ 50 μm) layers. The impurity signal detection seems interesting for deposited layer thickness measurements.

Hydrogen was detected on the face and backside of TexTor graphite tile surfaces (with and without a deposited layer) and even in the bulk graphite. The H/C spectral line intensity ratio was different for the face and backside tile surfaces. The hydrogen spectral line intensity was higher on the face surface with the deposited layer. A high H/C ratio (H/C ≈ 5) on a thick deposited layer was observed up to 30 pulses. On backside tile surface and on a thin deposited layer, H/C ratio decreased just after 5 pulses.

LIBS in-depth resolved measurements were made for two ITER-like calibrated multi-layered samples (W/Mo and W/C layers on Ti-substrates) [9]. The samples were previously characterised by glow discharge optical emission spectroscopy. For LIBS measurements, a Nd:YAG laser (with 1064 nm, 532 nm, 355 nm, and 266 nm wavelengths) was used. The investigations were aimed to study laser beam shaping, fluence, wavelength, environmental conditions (air, Ar, He), and pressure effect on LIBS performances.

It was demonstrated that the ablation depth increased with laser wavelength (due to plasma screening and increase in laser absorption by the target with a shorter wavelength). The fluctuations of the line intensities were significantly reduced, when we applied a shorter wavelength (except for $\lambda_{las} = 266$ nm).

Spatial distribution of the laser beam was important for improved in-depth resolution. With the use of the Gaussian beam, ablation was not uniform, and the W–Mo layers still contributed to plasma emission, thus keeping the spectral lines visible even after deep ablation. In contrast, a "top-hat" beam profile allowed us to limit the effect of non-uniform ablation, thus resulting in disappearance of W/Mo spectral lines.

The LIBS signal and the ablation depth per pulse increased with laser fluence. Thus, one has to seek a compromise between ablation depth (which optimises the depth resolution) and a sufficient LIBS signal for detection. The results obtained in air, argon, and helium under atmospheric pressure were compared. It was found that argon is the most suitable environmental gas for these studies as it limits signal fluctuations and enhances *signal-to-noise* ratio.

The most accurate LIBS in-depth measurements were obtained with a "top-hat" shaped laser beam and the third harmonic of a Nd:YAG nanosecond laser ($\lambda_{las} = 355$ nm) in argon, under

1 atm (Fig. 1). The use of UV wavelength was beyond the frames of ITER investigations.

The mixing of different layers after melting by high power laser ablation is seen as a severe problem for hydrogen isotopes analysis (because of their high desorption at temperatures above 1000 K). It is quite possible that the isotopes would be lost by bubbling in the melted layer. Thus, to minimise heat diffusion, laser fluence should be lowered as much as possible, but be kept high enough to excite hydrogen for LIBS measurements. The use of picosecond or femtosecond lasers may significantly reduce heat diffusion and, thus, increase the depth resolution of LIBS measurements.

Our study has clearly demonstrated that ablation depth depends on a number of parameters and features, which are difficult to predict, especially for heterogeneous tokamak deposited layers with practically unknown physical properties. Thus, we may conclude that in-depth analysis is possible only with consecutive measurements of crater depths.

The results obtained with LIBS in-depth measurements (Fig. 1) and those obtained with glow discharge optical emission spectroscopy were compared and found to be in agreement. However, the layers mixing was observed and attributed to diffusion through the melted material and to non-homogeneity of the laser beam spatial distribution. The depth resolution was on the order of several thermal diffusion lengths, but it may be improved by using picosecond or femtosecond laser pulses. The results obtained may find their application for tritium concentration measurements with in-depth resolution for the deposited layers of tokamak first walls (the case of future ITER fusion reactor).

Time- and space-resolved LIBS analyses with H/D isotope resolution were performed in Refs. [9–12]. The expansion dynamics of hydrogen and deuterium was characterised by H_α and D_α spectral lines observation, respectively, in argon under low pressure. D_α intensity was decreasing more slowly than that of H_α. It was attributed to atomic mass difference of these isotopes, which resulted in a higher expansion velocity for a lighter hydrogen and, thus, in a faster line emission decay. As a consequence of plume segregation, LIBS measurements tend to over-estimate the relative concentration of heavier elements.

LIBS analyses of PFCs of the Tore Supra have demonstrated encouraging results for ITER tritium inventory. The use of adequate laboratory diagnostics of laser-induced plasmas allowed us to get a better understanding of plume expansion and its properties. As a result, the developed procedure (which consists of only two LIBS acquisitions) allowed us to extract both H+D/C and H/D ratio within microseconds. The H+D/C ratio is performed during the early stages of plasma lifetime (where the LTE condition is satisfied), while hydrogen isotope ratio is determined a few microseconds later (when H_α lines of the isotopes are distinguishable due to plasma relaxation and spectral lines narrowing).
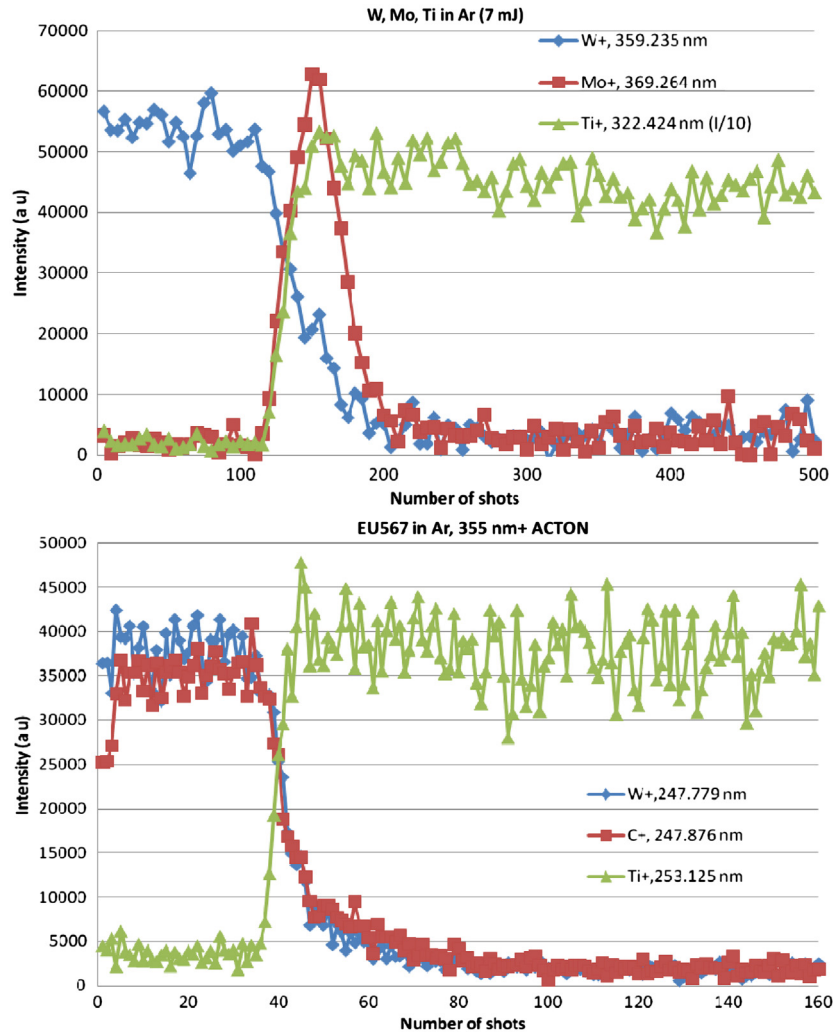
Fig. 1. Evolution of the spectral line intensities of ionic transitions of W, Mo, Ti and C during ablation of ITER-like samples $\lambda_{las}=355$ nm and 7 J cm^{-2} (a) and 18 J cm^{-2} (b). Acquisition gate width is 2 µs, and delay is 200 ns.

Feasibility of LIBS remote measurements under low pressure for in situ characterisation of the deposited layer in the JET environment was studied in Ref. [5]. The required remote measurements under low pressure were provided by the available ChemCam set-up developed for LIBS remote analysis under the Martian conditions within the frames of the ChemCam project [13–15]. Our investigations were aimed to study whether LIBS remote measurements (12 m distance) under low pressure (10^{-3} bar) obtained on the ChemCam set-up are reproducible on the JET site for future LIBS in-situ test.

Single-shot LIBS spectra were obtained, and depth profiles of the emission lines of H, C and impurity elements (B, Ca, Cr, Fe, K, Li, Na, Ni, and Si) were traced; 100 laser shots were applied onto the same sample position. The measurements were performed at 140 J/cm² and 18.4 J/cm² laser fluence, in air and Ar, under 1 bar and 10^{-3} bar.

Comparison of the spectra and depth profiles under 1 bar and 1 mbar revealed that the plasma confinement under atmospheric pressure contributes both to the chemical reactions in plasma and to formation of the dimers (like C_2 and CN). Thus, the atomic emission LIBS signal is lower, and the plasma energy is consumed mainly for molecules excitation.

The LIBS spectra of H and C were obtained under 1 mbar pressure with a single-shot time resolved detection (400 ns gate delay, 2 µs gate width) for backside and face tile surfaces. The carbon and hydrogen spectral line signals increased with fluence due to a high energy required to excite the observed lines at 247.856 nm (7.685 eV) and 656.3 nm (12 eV), respectively. However, the signal intensity remained very weak in the single-shot mode. Its fluctuations were dominated by the shot noise. The C and H lines intensity was the same both for the backside and face surfaces. It was constant with in-depth ablation. Thus, the layer thickness measurements with the H-line detection were not possible in this case. The monitoring of hydrogen seems to be difficult under these conditions. Measuring the plasma spectral line emission very shortly after laser pulse/surface interaction may be suggested as a solution. However, this suggestion should be verified in future studies.

Contrary to C and H, the lines intensity of impurities exhibited a transient regime in the first dozens of microns, thus allowing us to measure the layer thickness. At low fluence (18.4 J/cm²), the results were more accurate than at high fluence (140 J/cm²). This may be attributed to the signal fluctuations associated with the shot noise. Thus, to determine the layer thickness more accurately, one should increase the number of the collected photons. The depth profile measurements with accumulation of 10 spectra allowed us to estimate deposited layer thickness for both low and high fluences. Thus, the reliable LIBS analysis in the JET environment was clearly demonstrated in our investigations. The sensitivity of LIBS measurements for the detected impurity elements (B, Ca, Cr, Fe, K, Li, Na, Ni, and Si) was on the order of several dozens of ppm. This sensitivity may be improved by accumulation of the laser shots number (N). In this case, theoretically, the gain in the sensitivity may be expected to be on the order of $\times N^{0.5}$.

Based on the results obtained, it is possible to formulate some technical suggestions on LIBS instrumental equipment for in situ measurements on JET with JET EDGE LIDAR Laser System:

(a) The laser energy per pulse and spatial quality should provide ~20 J/cm² at 5.36 m. In fact, the irradiance (3–4 GW/cm²) rather than fluence should be specified. This irradiance may be provided at 5.36 m with commercially available nanosecond or sub-nanosecond lasers.

(b) A broadband Echelle spectrometer applicability depends on measurement requirements. In our studies with one or two elements under analysis, it seems quite reasonable to use a Cherny Turner spectrometer to collect more LIBS signal. The resolving power $\lambda/\Delta\lambda = 5000$ is sufficient, as carbon matrix is not complex (in terms of spectral line interference and $H_\alpha/D_\alpha + T_\alpha$ lines resolution). For D_α (656.104 nm)/T_α (656.045 nm) spectral lines resolution, a spectrometer with the resolving power of $\geq 10,000$ should be applied.

(c) Angular aperture of the optical collection system should be as maximal as possible.

4. LIBS JET-test results

LIBS feasibility for in situ analysis of the deposited layers with the JET EDGE LIDAR Laser System was under study in Ref. [6]. High vacuum environment (10^{-8} bar) in the zone of the analysis and a long distance (5 m) from the laser plasma to the JET optical window were the distinguishing features of the in situ LIBS measurements on the JET installation. With the EDGE LIDAR optical scheme, the Ruby laser beam (3 J, 690 nm wavelength, 300 ps pulse duration) was focused onto the centre of the JET D-T plasma column. Optical scheme of the EDGE LIDAR System was modified. An additional divergent lens was used to fix the laser beam focal point on the divertor tile surface, thus resulting in the laser intensity increase up to 70 GW/cm² (laser fluence of 20 J/cm²). Such intensity was sufficient to ablate the deposited layer and to create bright laser plasma. A number of analytical spectral lines of the laser produced plasma in 400–600 nm spectral range (Fig. 2) were observed during our experiments on the JET (November, 2008) performed with the optimised LIBS and the detection system features (optical collection system, spectrometer, ICCD-detector).

The spectral lines of H, CII, CrI, and BeII were identified [6,16]. With the increase of the number of laser shots applied on the
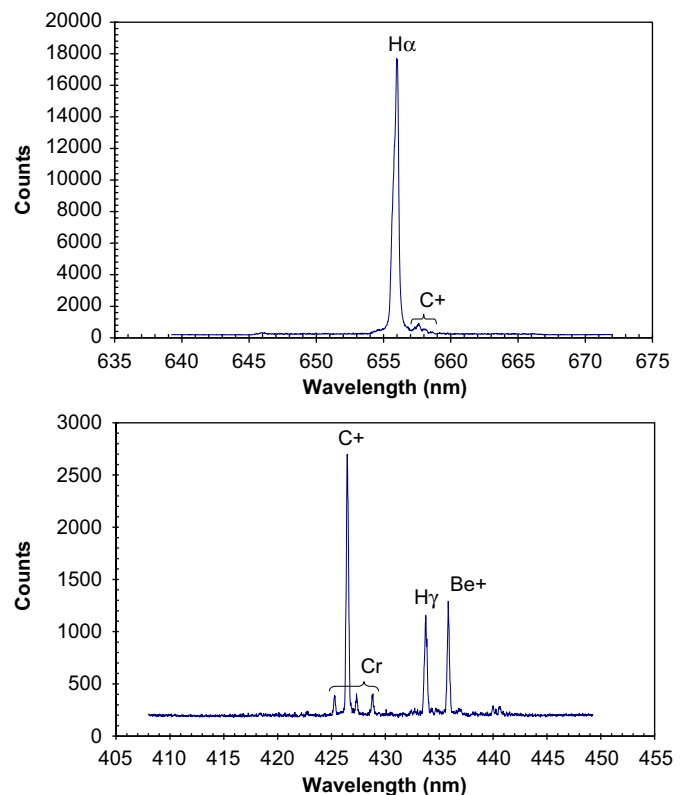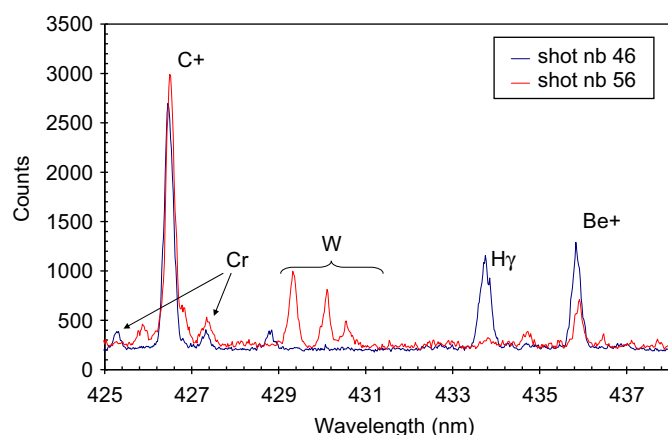

Fig. 2. Example of two parts of JET tile spectra. Cr, C^+, H_α, Be^+, and H_β spectral lines are observed.

Fig. 3. LIBS spectrum after the first (shot number 46) and 10th laser shot (shot number 56) on the W-stripe zone with the deposited layer. Laser pulse energy $E=3.0$ J, spectrometer slit width=200 μm, ICCD camera delay=180 ns, and gate width=2 μs, vacuum.

same divertor surface (without the laser beam displacement), the hydrogen line intensity decreased with the appearance of the tungsten (WI) spectral lines (Fig. 3). The spectral line intensities of the impurities (Cr, Be) decreased somewhat slower. With the analysis of the spectral line behaviour with respect to the applied laser shots, it was possible to analyse the deposited layers on the JET divertor tiles.

Thus, based on the performed studies and results obtained, one may with all the reasons conclude that LIBS is applicable for in situ real-time analysis of the JET divertor deposited layers with the available JET EDGE LIDAR System.

5. Discussions and conclusions

The presented qualitative LIBS results on hydrogen content measurements with tokamak tiles may be regarded as promising. Adequate understanding of the LIBS processes was obtained, revealing the associated problems and difficulties. To improve some LIBS analytical performances for hydrogen contents measurements, the method optimisation (LIBS with ultra-short pulses [17], double pulse plasma re-heating [18,19], appropriate choice of the detection scheme, etc.) should be considered as important.

A numerical code for simulations of optical emission spectra generated during LIBS measurements on the JET chamber walls may be useful for LIBS spectra treatment. The developed code is meant to predict LIBS spectra for the design of an optimised LIBS system and to analyse the experimental spectra to obtain the chemical composition without any preliminary calibration ("calibration-free LIBS"). For absolute quantitative tritium determination with "calibration-free LIBS", special attention should be given to hydrogen isotopes and matrix elements separation in laser plasma plume due to plasma expansion in vacuum.

The reliability of LIBS method for hydrogen isotope contents measurements should be confirmed with the samples with a known hydrogen concentration and in-depth distribution. Thus, further investigations on ITER-like calibrated samples with deuterium implantation are required to characterise hydrogen isotope concentrations in the deposited layers. The difficulty of hydrogen content measurements is associated with hydrogen

release or diffusion from the laser heated zone [20,21]. For further in-depth LIBS measurements and quantitative analysis validation, the investigations with ITER-like samples (tungsten layer on tungsten substrate with deuterium implantation) are necessary.

LIBS system integration in tokamak environment and in situ validation in European tokamaks with a mobile version of LIBS device is seen as a final step in LIBS method validation. LIBS data-bank of tokamak plasma facing surfaces needs to be created for reliable quantitative LIBS characterisation of PFCs.

This review is based on the results obtained in CEA (DEN/DANS/DPC and DSM/IRFM, France) during the last decade of LIBS studies. LIBS method development for PFCs characterisation is under study in numerous scientific laboratories in the European community [22–30]. For efficient and successful LIBS method implantation for fusion technology, future coordination and cooperation of the involved scientific laboratories and teams would be very important.

Acknowledgements

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