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# LIBS for monitoring of tritium and impurities in the first wall of fusion devices

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Laser Induced Breakdown Spectroscopy (LIBS) is the most promising method for quantitative in-situ determination of fuel retention in Plasma-Facing Components (PFCs) in magnetically confined plasma like JET, ITER etc. We present here the latest developments in view of ITER requirements since the review described in [1]. The studies focused on two main requirements: a) quantification of fuel from relevant surfaces with high sensitivity and b) the technical demonstration to perform LIBS with a remote handling system. (a) Composition depth profiles were measured in ITER-like coatings and deposits, with a typical depth resolution of 100 nm per ablation (accuracy 20%) at deuterium (D: substitute for tritium (T)) concentration levels below  $10^{16}$  D/cm<sup>2</sup>, confirming the required high sensitive. (b) A remote handling application was demonstrated inside the Frascati-Tokamak-Upgrade (FTU), where a compact, remotely controlled LIBS system was mounted on a multipurpose deployer providing an in-vessel retention monitor system. These achievements underline the capability of a LIBS based retention monitor, which complies with the requirements for JET and ITER operating in DT with Be wall and W divertor.

#### Introduction

Safe and successful operation of ITER and beyond requires accurate determination of the amount of plasma fuel retained in the PFCs and their deposits. In the case of Be PFCs, the majority of fuel is stored in Be deposits as shown in JET. During ITER shutdown phases e.g. for maintenance, the ITER vessel will be at dry air or nitrogen atmospheric pressure. In view of safety requirements, ITER will apply in maintenance phases LIBS on a remote handling system to identify the location of Be co-deposits in the vessel as well as to determine their T content. Moreover, the effectiveness of fuel removal activities like baking will be assessed by the LIBS system operating at elevated pressure. In support of these safety requirements for ITER, the EUROfusion research programme has a twofold aim: (a) the quantification (and research) of fuel release from W, Be and deposits during laser-induced ablation and b) the technological aspects of a remote handling application of LIBS inside a fusion reactor with neutrons.

In LIBS, a very small amount of material ( $\mu$ g) is ablated from the surface by a short (ns-ps) laser pulse, resulting in the formation of a plasma plume. Within the plume the ablated elements are excited and start emitting light at characteristic wavelengths. Depth-resolved composition information of the surface can be obtained by recording LIBS spectra after each successive laser pulse. The so-called calibration-free LIBS (CF-LIBS) method [1] is used to quantify the composition from the spectra; the analysis is essentially based on Boltzmann plots. Assuming (near) thermal equilibrium, it yields the concentration of each element.

For validation of the LIBS approach, samples from multiple devices have been analysed by LIBS and compared with results from other laboratory techniques suitable for elemental analysis and quantification (SEM,RBS, NRA,ERDA,TDS,SIMS etc.). For this purpose, coatings which mimic the expected ITER coatings have been produced in laboratory experiments either with a pre-determined concentration of plasma fuel (D or He) or with a fuel profile resulting from plasma loading in the linear plasma devices Magnum-PSI and PSI-2. Moreover, wall components from magnetically confined fusion devices (ASDEX Upgrade, W7X and JET) have been analysed with LIBS [2, 3, 4].

## Demonstration of quantitative D retention measurements of ITER-relevant coatings

- W-Al-D coatings (thickness 3  $\mu$ m, Al was used as proxy for Be) were investigated by CF-LIBS [5] at 100 mbar nitrogen pressure: the measured integrated D content (4.10<sup>17</sup> D/cm<sup>2</sup>, probing depth 400 nm) was within 25% equal to the D content given by the producer. The high H content limited the detection sensitivity to 2.10<sup>17</sup> D/cm<sup>2</sup>.
- The D content of 2  $\mu$ m thick Be-W-D coatings were determined by CF-LIBS in vacuum to be 5.10<sup>17</sup> D/cm<sup>2</sup> (probing depth 400 nm) (Fig. 1): accuracy 20% [6]. A similarly good correspondence was found for LIBS measurements on Be-O-C-D layers.

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Fig.1: LIBS spectrum corresponding to shot-numbers:  $1^{st}$ (surface),  $3^{th}$  (coating) and  $20^{th}$  (substrate) laser shot recorded for Be-W-D coating with  $5.10^{17} \text{ D/cm}^2$ , according to CF-LIBS and microscopy.

• The dynamic retention (outgassing) from tungsten samples, exposed to D plasma in PSI-2, was investigated by LIBS in vacuum. The D retention was reduced by a factor of 3-6 within 200 min after plasma switched-off, and stabilised on the level of  $5.10^{16} \text{ D/}cm^2$  (probing depth 200 nm, detection limit  $10^{15}$ D/ $cm^2$ ) [7]. Similar measurements were performed in Magnum-PSI.

In terms of sensitivity, LIBS at atmospheric pressure is more demanding, and therefore different signal enhancements techniques were investigated. The lifetime of LIBS plasma is short (100 ns-5  $\mu$ s). Concentrations of H-isotopes are measured in the afterglow of the plasma plume, i.e. then the Stark broadening of the spectral lines as well as the emission strength is reduced. To enhance the emission (and lifetime), the plume was re-excited, either by a second laser pulse (DP LIBS) which enhanced the plume emission by a factor ~6 [8], or by a spark discharge, which enhanced the plume brightness with more than 2 orders of magnitude [9].

#### Remote handling application of LIBS in the FTU tokamak

First D retention measurements were successfully carried out from outside on the FTU toroidal limiter [10]. Then a compact DP LIBS system  $(410 \times 300 \times 70 \text{ mm})$  (Fig. 2) was mounted on the FTU multipurpose deployer inside FTU. A broadband (210–800 nm) and narrowband (8-20 nm) spectrometer (fiber-based relay system) were used for collection of the light from the LIBS plume. On virtue of a pressure confinement tube, the system allows for measuring in vacuum (>10<sup>-3</sup> mbar) as well as at elevated pressure and flow (Ar, He, N2), i.e. adapting the operation conditions for maximum sensitivity.

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Fig.2: the compact LIBS device (Weight<8 kg).

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Fig. 3: LIBS device mounted on the FTU multipurpose deployer.

The LIBS system enabled to investigate large areas of the wall (Fig. 3) and the results found in [10] could be reproduced.

## Summary

With CF-LIBS low concentrations of fuel and other elements were succesfully measured in Be, W and associated deposits. LIBS performs accurately in vacuum as well as in elevated pressure, although in nitrogen pressure improvement of sensitivity is desirable. Furthermore, advanced spectral resolution techniques have to be applied for resolving D and T lines (Balmer- $\alpha$  line separation ~0.06 nm). A remote handling application of LIBS was succesfully realized in FTU.

In conclusion, a LIBS based system, as proposed as T monitor in ITER, will be capable to operate under the most demanding conditions. A further demonstration system is proposed for the remote handling arm of JET and shall demonstrate the capabilities to measure D and T after DT operation like in ITER.

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