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An upgraded LIBS system on linear plasma device PSI-2 for in situ diagnostics of plasma-facing materials

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Abstract

Laser induced breakdown spectroscopy (LIBS) is used to detect and monitor the D and H content on the W surface thanks to its capability of fast direct in situ measurement in extreme environment (e.g., vacuum, magnetic field, long distance, complex geometry). A new LIBS system has been set up on the linear plasma device PSI-2 to perform in situ, real-time and remote measurement of D outgassing on W samples. The new system is an upgrade of the previous LIBS system [1] and is improved in several ways including a newly designed and better constructed optical system, an improved timing and triggering system, a re-adjusted spectrometer with higher sensitivity and a number of remote control units to accomplish the in situ real-time measurement task. A number of key parameters including laser energy, the (de)focuing of the laser beam, the delay time of image intensifier (MCP) are studied and improved for the new system. Two different spectrometers with different configurations are used in the experiments. The measurement by the new LIBS system is then compared to other methods. The current work addresses the sensitivity and accuracy issues of the in situ measurement of D, H on PFC samples and for the first time, demonstrates the time evolution of D, H content on W samples before, during and after plasma-off point in the scales of seconds, minutes and hours.

Keywords: Laser induced breakdown spectroscopy, Plasma-wall interaction, Plasma facing component, in situ measurement, material diagnostic

1. Introduction

Laser induced breakdown spectroscopy (LIBS) is a powerful and versatile diagnostic tool. It has been considered as a strong candidate for plasma facing material analysis since the early days of material diagnostics for fusion [2]. Among the numerous researches dedicated to exploit the potential of LIBS in fusion material diagnostics, most of the works are done in an off-site laboratory [3] or merged with other techniques [4]. In our previous work [1], a LIBS system is installed on the linear plasma device PSI-2 for in situ diagnostics of the plasma facing component (PFC) materials after plasma exposure. The D content was measured in the range of the first 40 minutes to 2 hours after plasma shut down. The result showed the trend of D outgassing not long after plasma exposure. Similar works [5, 6] have proved the capability of LIBS for in situ measurement. However, due to the low D, H content on the target surface, the D, H signal usually appears only in the first couple of shots. These single shot spectra often have weak D, H line emission with strong background emission. To increase the signal, or signal-to-background ratio to be specific, effort has been made in many ways. For example, an adapted fibre bundle is used to replace the spectrometer slit to collect more light [5], long integration time is used for signal collection [7], and very frequently considered, the double pulse LIBS [8]. These methods all bring good improvement to the overall measurement but it is still challenging to achieve high intensity (counts number) and high resolution at the same time, i.e., good measurement sensitivity. In the current work, different instruments and methods are examined and evaluated in attempt to address the sensitivity issue and make LIBS a more practical tool for in situ diagnostics.

2. Experimental

The current LIBS system is an upgraded version of the previous setup [1]. A new set of optic unit with a

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focusing lens of f = 50 mm, F # = 1.4 is placed near the chamber window to collect the LIBS plasma light into the fibre. Two spectrometers were used for the experiments. One is a Czerny Turner spectrometer (Acton SpectroPro-500) with the grating of 2400 g/mm and the size of $68 \times 68mm$, focal length 500 mm and f/# = 6.5. The other one is a Littrow spectrometer equipped with a grating of 1200 grooves/mm, $160 \times 130mm$ and the blazing angle $57^o22^{"}$, focal length 750 mm and f/# = 5.8. The optics are arranged using the Littrow configuration and has the optimum intensity at the second order. Two sets of fibre coupling units are set up for each spectrometer. A 532 nm filter is used for the monochromatic spectrometer and a 630 nm filter is used for the Littrow spctrometer to filter out other orders. The camera is an image intensified CCD camera (Pike F032b with micro channel plate (MCP) image intensifier). W samples are mechanically polished and dehydrated in the oven, then exposed to deuterium plasma on PSI-2. The optical arrangement and experimental structure for LIBS on main chamber are shown in figure 1.

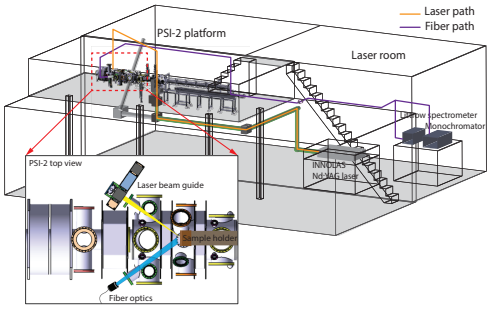

Figure 1: Experimental setup of LIBS on PSI-2.

3. Results and Discussion

3.1. Optimisation of key parameters

First of all, the system is tested on D, H loaded C samples to obtain an initial range for further optimisation. C I 657 nm is chosen as test line because the wavelength is very close to D_α and H_α lines. The optimum laser energy was tested to be between 280 mJ to 350 mJ, to ensure the ablation efficiency and avoid raising the background significantly. The (de)focusing position was above the sample surface which is a common defocusing position for many LIBS applications [9, 10]. The background (continuum) rises immediately after the laser shot, the characteristic emission starts to merge at about 100 ns later and they both start to decrease at about 350 ns. Thus the time window for characteristic emission is only about 200 ns. The continuum peaks at about 200 ns and the line emission peaks at 250 ns. Higher laser energy gives a moderate increase in the emission intensity and has little or no impact on the optimum delay time. Further tests show that under the same focusing condition, higher laser energy is preferred though its influence is not as significant as the MCP delay and exposure time.

MCP delay (ns)	expo. time (ns)	D, H visibility	intensities rel.
365	500	H and D	3.23+2.62
370	600	H and D	3.3+2.3
370	800	only H	3.2
375	600	H and D	3.2+2.6
380	600	only H	3.1
385	1000	only H	3.45

Table 1: Measurement result of D on W surface.

Unlike the measurements on C samples, D_α line only appears in the first (one or two) shot(s) on each fresh surface and there is limited fresh surface for a thorough scan. With the estimated delay range from the results on C samples, we have managed to test and find the parameters that are closest to the optimum. Table 3.1 shows the settings that have clear visibility of D and H or H signals. The visibility is shown in the second last column and the relative intensity is shown in the last column. The closest optimum delay and exposure time we have found are 365-380 ns and 500-600 ns.

3.2. Czerny Turner spectrometer 10 μm vs. 200 μm slit

In order to measure D and H content at a lower level on W surface with the Czerny Turner spectrometer, we use two methods with two different slit widths. The first method reduces the slit width aiming for a better resolution. The second method uses a wider slit to increase the light input as much as the slit width is just enough to resolve D_α and H_α spectral lines.

The upper figure in figure 2 shows the spectrum taken with 10 μm slit and the line fit with Voigt profile. When a narrow slit is used the two lines are well separated. The absolute counts (intensity) number peaks at 500 - 600 and the background is flat and close to 0, thus a very good signal-to-background ratio is achieved. The intensity ratio of the two lines is $R = I(D_\alpha)/I(H_\alpha) \approx 0.24$. Hydrogen is the dominant element in this measurement. This specific W sample was exposed to D plasma about 8 months ago and left in clean atmospheric environment. The high H content is most likely due to the water

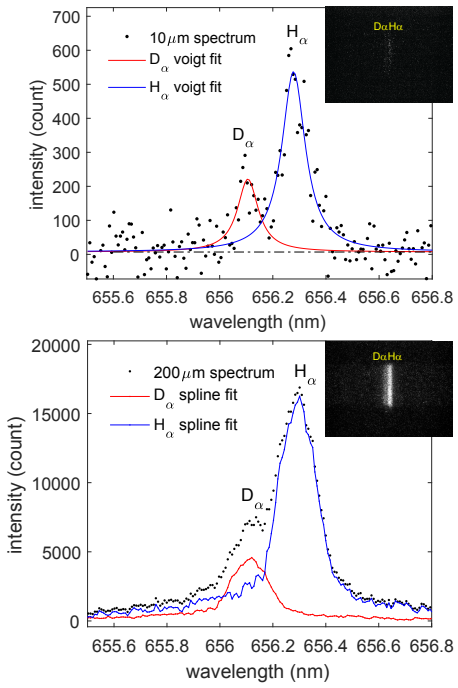

Figure 2: D, H spectrum from Czerny Turner spectrometer with voigt and spline fitting.

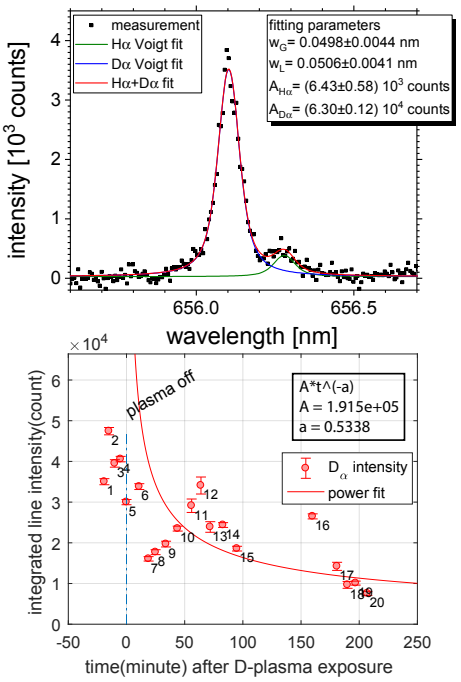

Figure 3: Littrow spectrum (top) and the time evolution of the D content on W in the first 3 hours after plasma exposure (bottom).

vapour and the D content is the residual from the previous plasma exposure.

After using the narrow slit for better resolution, we use a much wider slit (200 μm) to allow more light input. D and H lines will be resolved later during signal processing. A pre-scan of the spectra with different slit widths shows that with the current monochromator spectrometer and the lens set, the maximum slit width to resolve D_α and H_α lines is 240 μm. The slit width of 200μm is used to record the spectrum. The wide slit maximises the light input and is sufficient to separate the two isotope lines. When the slit is much wider, the line shape is not a Voigt profile anymore, either Gaussian or Lorentz fit will not be sufficient to describe the intensity distribution. We use a self-developed spline fit to deconvolute D_α and H_α lines. The lower figure in figure 2 shows the spline fitting that separates D_α and H_α line. The intensity ratio of the two lines is $R = I(D_\alpha)/I(H_\alpha) \approx 0.25$, which is comparable to that with 10 μm slit and Voigt fitting ($R = 0.24$). The measurements done with 10 μm slit and 200 μm slit are on the same patch of W samples and so the two methods coincide.

3.3. Time evolution of D content and D outgassing

Apart from the monochromatic spectrometer used in the above sections, a Littrow spectrometer is also used for the measurement. The Littrow spectrometer has a larger grating size of 160 × 130 mm and a larger etendue than the monochromator. Second order emission in the Littrow configuration is used and the two lines are sufficiently resolved at 200 μm slit width. A series of tests have been carried out to optimise the system parameters with this spectrometer, similar to the previous procedure. The Littrow spectrometer is used for the in situ measurement and the time evolution of D, H on W is presented in figure 3. The spectrum is shown in the upper figure and the time scan of D content is shown in the lower figure. D_α is the dominant emission line. The background is close to 0 and the two lines are well fitted by voigt profile. A power fit is performed on the D content measurement (using only the data points after plasma is off) and the result (fitting coefficient a = 0.52) is comparable to the calculation result [].

The measurement starts at about 50 minutes before (-50 on the time scale) plasma-off point (0). A few more pulses are taken in every 5 to 8 minutes. One measurement at the time of plasma being shut off (0 ± 2 seconds) and the time scan proceeds at roughly the same pace until about 200 minutes (more than 3 hours) after

the plasma is off. It is the first time that the D (and H) content on W surface is thoroughly scanned over time by in situ measurement. The time window from 100 minutes before to 200 minutes after plasma-off point is the most critical time period to study the fuel content in PFC materials.

3.4. Comparison with TDS and NRA result

Two of the W samples from the same exposure patch were examined by TDS and NRA for a comparison study. TDS heats up the sample and measure the gas that comes out of the sample. It gives a total measure of about 5.5×10^{16} D/cm^2 on the W sample. NRA produces the depth profile of D atoms on W sample. The result gives about 4.9×10^{16} D/cm^2 in the first 1.5 μm. This gives the general information that the D content on W surface is in the range of 10^{16} D/cm^2. With the crater size of LIBS, we can estimate that there is about 2×10^{15} D atoms in one LIBS shot. The LIBS signal of D_α line per spectrum was 500 - 1000 counts in a crater of $\varnothing$ 1 mm (radius) $\times$ 25 nm (depth). This gives an estimation of 10^{14} to 10^{15} D per LIBS measurement.

4. Conclusions and Future Work

An upgraded LIBS system on linear plasma device PSI-2 is used for D, H content measurement on W samples. A number of key parameters are optimised for the purpose of in situ measurement of fuel content. The optimum values for MCP delay and exposure time are both a few hundred nanoseconds with a precision range of less than 20 ns, which means the in situ measurement in this specific case requires significantly high sensitivity from the detecting system. In order to improve the measurement sensitivity, series of tests and experiments are carried out. The optic units have been re-designed and re-constructed. New spectrometer is introduced to the system. With this system, the time evolution of D, H content on W samples is revealed for the first time within the critical time window of plasma-off point. The corresponding dynamics of D outgassing is studied. The new LIBS system with upgraded parts, optimised parameters and measuring method, set a solid foundation for in situ real-time measurement and monitoring of D, H (and T, in future) on plasma facing components such as W tiles.

5. Acknowledgement

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