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Preliminary results of *in situ* laser-induced breakdown spectroscopy for the first wall diagnostics on EAST*

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Abstract

Post-mortem methods cannot fulfill the requirement of monitoring the lifetime of the plasma facing components (PFC) and measuring the tritium inventory for the safety evaluation. Laser-induced breakdown spectroscopy (LIBS) is proposed as a promising method for the *in situ* study of fuel retention and impurity deposition in a tokamak. In this study, an *in situ* LIBS system was successfully established on EAST to investigate fuel retention and impurity deposition on the first wall without the need of removal tiles between plasma discharges. Spectral lines of D, H and impurities (Mo, Li, Si, …) in laser-induced plasma were observed and identified within the wavelength range of 500–700 nm. Qualitative measurements such as thickness of the deposition layers, element depth profile and fuel retention on the wall are obtained by means of *in situ* LIBS. The results demonstrated the potential applications of LIBS for *in situ* characterization of fuel retention and co-deposition on the first wall of EAST.

Keywords: tokamak, laser-induced breakdown spectroscopy, impurity deposition, fuel retention, plasma wall interaction

(Some figures may appear in colour only in the online journal)

1. Introduction

Plasma-wall interaction (PWI) has become increasingly important in the International Thermonuclear Experimental Reactor (ITER) and future fusion devices because of the large high-temperature fluxes and particle loads on the wall [1]. The main issues are the induced wall erosion and the associated material deposition on the wall, which greatly deteriorate the performance and lifetime of plasma facing components (PFC) during long-pulse and high-performance operation. Furthermore, safety requirements with respect to fuel inventory in the vacuum vessel also determine the availability of the device [2].

Post-mortem methods are limited to after-campaign analysis and averaged over a variety of wall and plasma conditions. These methods cannot meet the requirements of continuous operation, as the removal of wall tiles from actively cooled structures in future fusion devices has become increasingly difficult [3]. Therefore, an effective method for *in situ* monitoring of the condition of wall materials is

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necessary [4], especially between plasma discharges, to determine the thickness of the co-deposition layers and impurity compositions on the first wall.

Laser-induced breakdown spectroscopy (LIBS), a multi-elemental analysis technique based on atomic emission spectroscopy, uses a high-intensity laser pulse as the vaporization or excitation source to create a high-temperature plasma on the material surface. Laser-induced ablation creates a hot plasma that generates the spectra of atomic and ionic characteristic emission lines, which are used to determine the elemental composition [5, 6]. LIBS can be applied between plasma discharges with high magnetic field and in the vacuum without any sample pre-treatment; therefore, LIBS is a promising method to fulfill the need for *in situ* diagnostic of fuel retention and impurities deposition on the first wall between plasma discharges in fusion reactors [7].

A number of investigations have been conducted on LIBS in the laboratory for impurity deposition composition and fuel retention in deposition layer analysis toward ITER application [8, 9]. In 2011, Huber *et al* studied the feasibility of applying LIBS for *in situ* characterization of deposited layers in TEXTOR [10]. In 2013, Grisolia and Semerok *et al* confirmed the feasibility of *in situ* LIBS on the Joint European Torus (JET) with the EDGE LIDAR Laser System [11, 12].

The Experimental Advanced Superconducting Tokamak (EAST) is a fully superconducting tokamak (1.7–1.9 m major radius, 0.45–0.5 m minor radius, 60 m^2 first wall surface area, 3.5 T toroidal magnetic field) with double divertor for long pulse operation [13] that will inevitably lead to strong plasma interactions with the first wall, resulting in increased impurity deposition, fuel retention, and material erosion. Therefore, EAST is in need of good wall diagnostics and is also highly suitable for R&D of PWI diagnostic for ITER and future fusion reactors [14].

In the present study, an *in situ* LIBS system was successfully established and implemented on EAST in the 2014 campaign. The main goals of this system were the detection of impurity composition and fuel retention in the deposited layers on the first wall of the EAST tokamak, especially the spatial distribution and dependence on plasma operation and wall conditioning methods. The preliminary results of *in situ* LIBS are discussed in this paper.

2. Experiment

2.1. Experimental setup

The schematic of *in situ* LIBS is presented in figure 1(a). As shown in the figure the *in situ* LIBS system is located at the H port, just under the Material and Plasma Evaluation System (MAPES) which is a comprehensive and flexible experiment platform on EAST [15]. In figure 1(b), a Gaussian profile Q-switched Nd:YAG laser (Brilliant Eazy, Quantel, France) with 5 ns pulse width and 10 Hz repetition rate, operating at its fundamental wavelength (1064 nm), was used as a source for material ablation [16]. A He-Ne laser in coaxial alignment with the Nd:YAG laser was used to determine the laser ablation spot on the sample surface. The laser beam was expanded from the initial 8 mm beam diameter to 30 mm by a beam expander which improved the beam divergence by a factor of ∼3. After reflection by two mirrors, the laser beam was perpendicularly focused onto the sample surface by a quartz lens with 3000 mm focal length. The focus lens was placed about 10 cm away from the H-port flange. In the 2014 campaign, the energy and power density of the laser pulse were 180 mJ and 4.5 J cm^{-2} with 80% transmission of opticals, respectively. The spot size of laser ablation was about 2 mm in diameter. Laser spatial movement steps were approximately 2.5 mm to avoid the overlap of ablation spots. The laser ablation area covered a region of 20 cm × 20 cm on the high field side of the first wall. Laser-induced plasma emission light ∼2 cm size diameter on the surface of the sample was collected perpendicular to the first wall of the high field side of EAST by a 300 mm focal length and 150 mm diameter lens to a bundle of optical fibers (600 μm core diameter, 1.88 m length) and then coupled to a spectrometer, the solid angle of collection was ∼0.002 sr. As shown in figure 1(c), the angle between the laser beam and the spectrometer's line of view was about 15°. The spectrometer used in the experiment was a LIBS2500 + (Ocean Optics Inc., USA) with seven linear silicon CCD array detectors, which allowed the analysis of the plasma emission within the wavelength from 200–980 nm with the spectral resolution of 0.1 nm. All spectra were acquired at the integration time of 1 ms. The spectrometer synchronized with the laser firing without delay.

2.2. Sample preparation

Several marker tiles (dimensions 10 mm × 10 mm × 3 mm) were prepared for the impurity deposition and fuel retention study by means of *in situ* LIBS. Marker tiles were composed of bulk molybdenum (Mo), bulk tungsten (W), Mo substrates with 1 μm W-coated layer, graphite substrates with 1 μm Mo coated layer, and graphite substrates with 1 μm W-coated layer materials. Bulk W(pure tungsten) and Mo(titanium-zirconium-molybdenum alloy, TZM) samples were produced by Advanced Technology and Materials Co., Ltd for the upper divertor and first wall, respectively. Graphite tile (1%B4C, 2.5%Si, 7.5%Ti) was obtained from EAST's lower graphite divertor [17]. Coated layers were deposited on the substrate by magnetron sputtering in the Lanzhou Institute of Chemical Physics [18]. The tiles were mounted on the first wall surface at the high field side. Substrate materials were selected according to the materials used on EAST [13]. In the top part of figure 2, the tile prior to plasma exposure is shown. In the bottom part of figure 2, the sample after exposure to EAST discharges and subsequent LIBS analysis is shown. On the surface of Mo tiles, two array spots were ablated by laser shots. The total irradiation time was 29 452 s from #44 327 to #52 804 of EAST discharge.

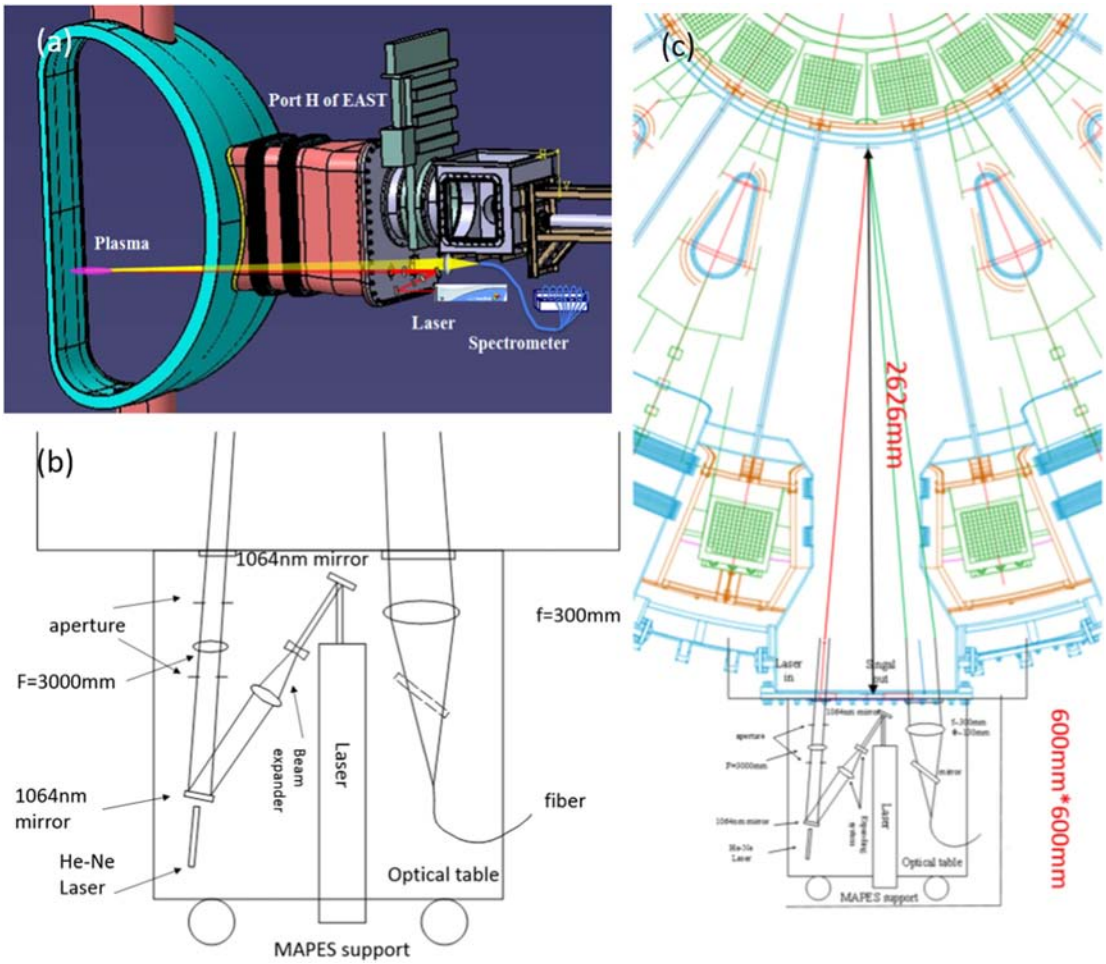

Figure 1. Schematic of the experimental setup. (a) H port of EAST. (b) Top view of *in situ* LIBS system on EAST.

3. Results and discussions

3.1. Detectability of spectral lines

In figure 3, the typical LIBS measurement spectra using the fundamental wavelength of the Nd:YAG laser from the first and second laser pulse of the same place in the presence of 2.5 T magnetic field and a base pressure of 10^{-5} Pa vacuum are shown. In figure 3(a), several spectral lines in the laser-induced plasma were observed and identified within the wavelength range of 500–700 nm. Given that Mo was chosen as the first wall material of the EAST tokamak, three characteristic lines (550.65 nm, 553.30 nm and 557.04 nm) of Mo I with strong emission intensities were clearly identified. Simultaneously, many weak Mo peaks were detected within 400–500 nm in figure 3(b). Lithium (Li) coating was a routine method for wall conditioning in controlling impurities and hydrogen recycling during the 2014 EAST campaign [19]. Thus, three intensive Li emission lines were observed, namely Li I of 670.79 nm and 610.36 nm, and Li II at 548.428 nm. These Li lines were suitable for diagnostic purposes in the deposition on the substrate [19]. In figure 3(c), the Mo intensity of the first shot was weaker than that of the second shot. However, the Li I lines showed the opposite behavior, as shown in figure 3(d) the Li intensity was stronger in the first shot than in the second. These results demonstrate that there were less Mo impurities mixed with Li deposited on the substrate.

3.2. Elemental depth profiles

LIBS has been successfully applied for depth elemental profile by several subsequent laser pulses at the same point on the sample surface. The integrated intensities of spectral peak area were then plotted as functions of the laser pulse number to obtain the depth profile of elements in the laboratory [20]. In this study, the qualitative depth profiles of Li and Mo were measured. Typical results for Li coating and co-deposition layer are presented in figure 4. Figures 4(a) and (b) show the integrated intensity profiles of the atomic lines of Li I 610.365 nm and Mo I 550.634 nm lines, respectively, as functions of the number of the laser shots. The new position demonstrated that the deposited layers were cleaned by several laser shots and then deposited by lithium coating. The old position was not cleaned by laser shots initially, but with the same deposited lithium coating layer of the new position. In figure 4(a), data showed that the intensity of the Li I 610.365 nm line decreases to the baseline level by 20

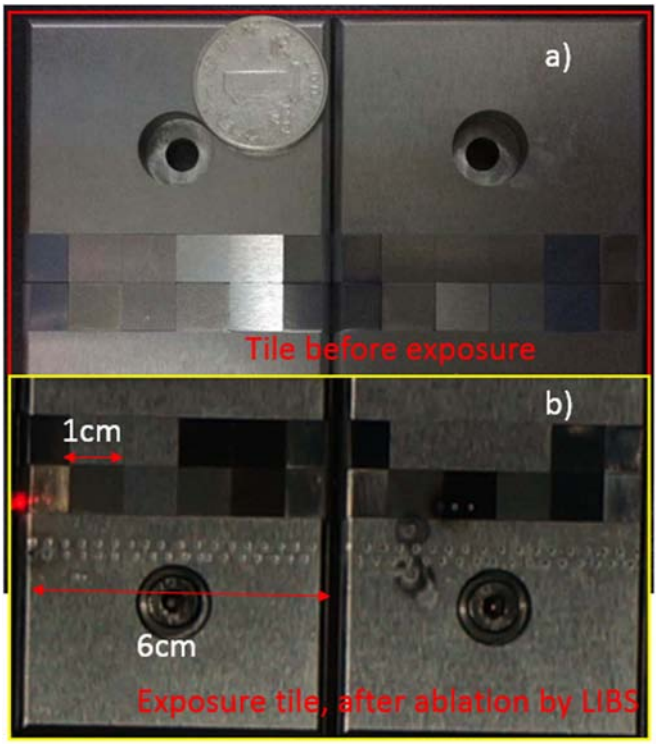

Figure 2. Molybdenum tile and sample tiles: (a) before exposure and (b) after plasma exposure and laser ablation.

sequential laser shots, and the intensity of the Li 610.365 nm line of the old position was stronger than that of the new position. It demonstrated that Li deposited on the old position was much thicker than that on the new position. In figure 4(b), it was found that the signal of the Mo 550.634 nm line on the old position in the initial three or four shots increased quickly, and then decreased to the baseline after 15 shots. This change may be caused by the Li layer from the wall conditioning covering the Mo impurities. With the mixed layer removed, the intensity of Mo decreased to the baseline level. The same phenomenon was also found by Hai in the laboratory [20]. Normalized intensities of Li and Mo lines were presented in figure 4(c). From the intersection of the two lines, the interface of the Li deposited layer and Mo substrate was found at the fourth laser shot. At the same time, in order to obtain the thickness of deposited layer, the linear correlation analysis method, which has been described in detail by Hai [21], was carried out. It was found that the Li deposited layer and Mo substrate separated at the tenth laser shot in figure 4(d). The difference of these two methods was caused by the Gaussian energy distribution of the laser beam, which produced unfavorable features in the depth profile [11]. The depth profiles clearly showed the separation of the Mo substrate from the Li deposition layer. Ablation rates of the mixed layers were slightly higher than those of pure Mo, which suggested the weaker structure of the Li-Mo compound. A large variety of calibration measurements on the ablation rate will be required to resolve the absolute depth profiles of the mixed layers in future tokamak applications.

3.3. Detection of fuel retention

As shown in figure 5, the D_α line at 656.1 nm and the H_α line at 656.29 nm of the first laser pulse were visible. However, the intensity of D_α and H_α in the second and third laser pulses strongly decreased compared with the signal from the first laser pulse. In the second pulse, the intensity of D_α and H_α decreased to 50% of the value of the first pulse. In the third pulse, the D_α and H_α signals were at the background level. The fact for the majority of D_α and H_α light observed in the first pulse indicated that the hydrogen isotopes were distributed only on top of the deposited layer, as about 100 nm deposited layer was removed by one laser shot. The ablation depth was estimated from the thickness of lithium conditioning. In the experiment, the surface area of EAST tokamak is $\sim$60 m^2, from the density of Lithium (0.534 g cm^{-3} (300 K)), about 1.4 μm thick lithium film will be deposited on the wall by 45 g lithium. The lithium film was removed by about 10 laser shots.

As shown in figure 5(b), the D_α and H_α lines could not be fully resolved with the spectral resolution of 0.1 nm. To distinguish the D_α and H_α lines, the Voigt profile was used to fit the spectra and obtain the $H_\alpha/(H_\alpha + D_\alpha)$ ratio [22]. Figures 5(c)–(e) show the evolutions of the D_α and H_α signals in the deposited layer of the first laser shot for three consecutive days. The $H_\alpha/(H_\alpha + D_\alpha)$ ratio during the three days was estimated to be about 40% from the fitted spectra. However, the $H_\alpha/(H_\alpha + D_\alpha)$ ratio calculated from the edge plasma spectra during plasma discharge was less than 5%. We infer that H_α and D_α were absorbed by the co-deposition layers on the first wall. This result indicated that Li coating wall conditioning could significantly reduce $H_\alpha/(H_\alpha + D_\alpha)$ in the vacuum vessel and enhance long-pulse H-mode plasma operation and ICRH power coupling.

3.4. Detection of impurity deposition

In figure 6, the spectra measured by *in situ* LIBS on different material samples are shown. The emission lines of Mo, Li, Si, Ti, La, Na and Ca for the first laser shot on the surface of samples were observed. We assume that the Mo impurity was caused by sputtering of the first wall and subsequent material migration. Elemental Li and Si elements were attributed to the daily Li and Si wall conditioning. For Na, we deduced that it was from Li conditioning as its purity was about 99%. Two intense lines at 393.366 nm and 396.847 nm from Ca were detected on the surface of Mo tiles and samples. Ca was observed only on the initial several pulses on deposited layers. After several laser pulses, the Na and Ca lines disappeared. This finding showed that these trace elements may gather on the surface during Li and Si conditioning, together with co-deposition processes. The present study successfully demonstrated the capability of *in situ* LIBS to map the spatial distribution of impurities and study the composition of deposited layers on the surface of the first wall in the EAST tokamak.

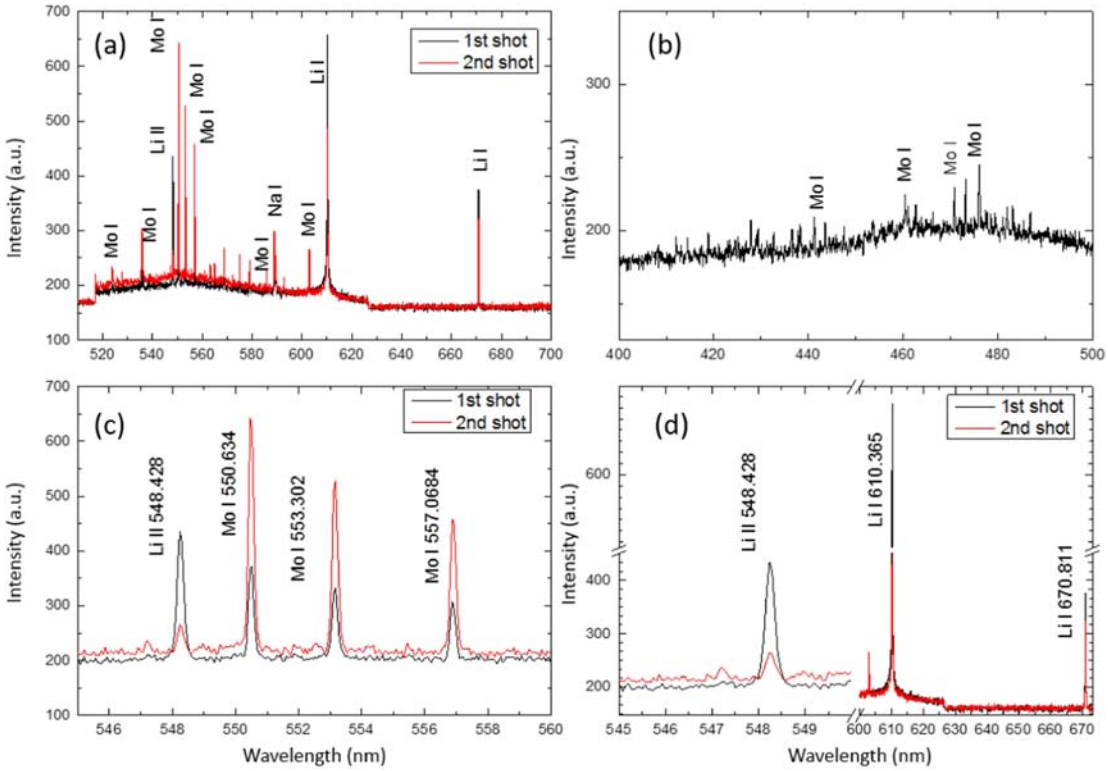

Figure 3. Typical LIBS spectra obtained from first wall ablation in the EAST.

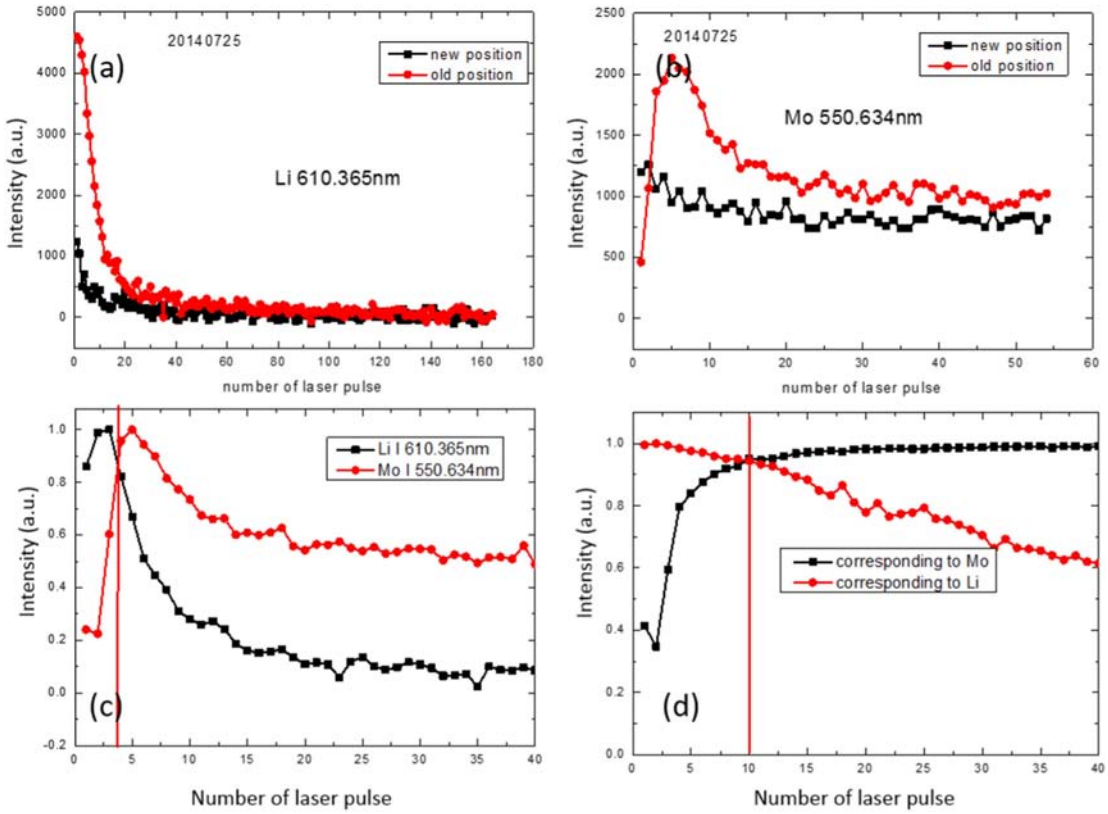

Figure 4. Depth profiles of Li and Mo as functions of the number of laser shots in the deposited layer of EAST first wall. (a) Intensity of the Li 610.365 nm line. (b) Intensity of the Li 550.634 nm line. (c) Normalized intensity of Li and Mo lines. (d) The correlation coefficient of Mo and Li lines.

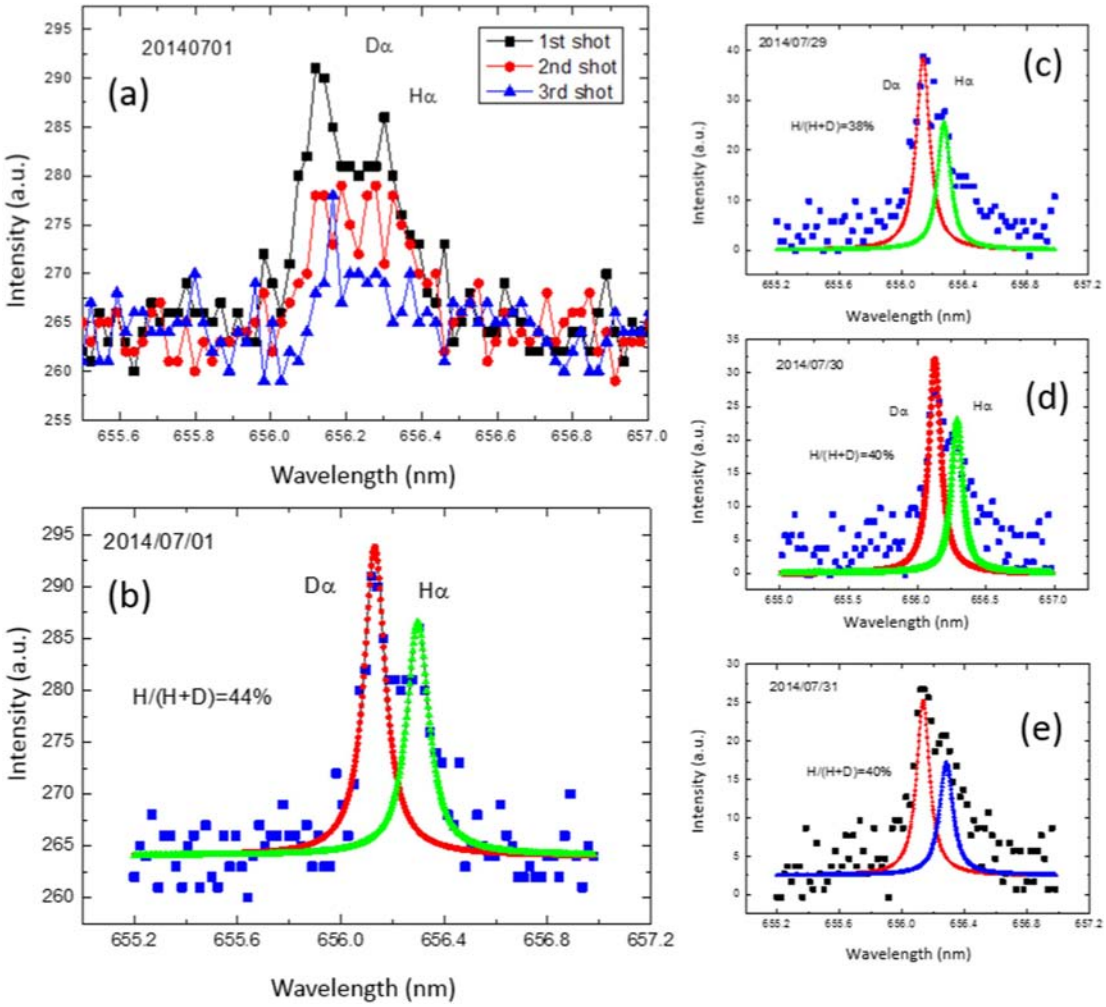

Figure 5. Spectral signals of Dα and Hα in the deposition layer.

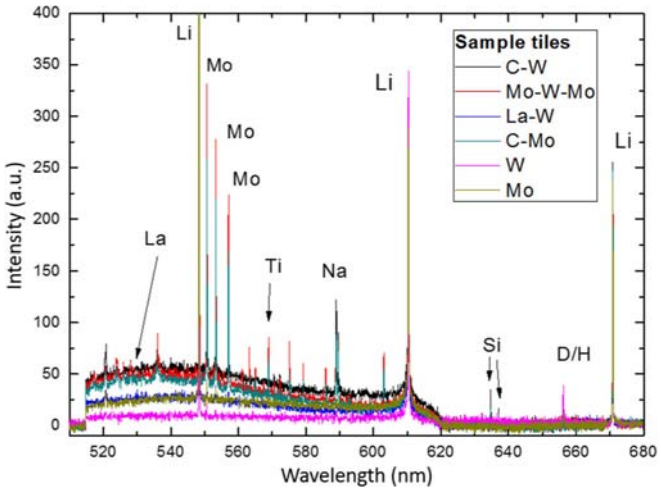

Figure 6. LIBS spectra of impurities on the first wall.

4. Conclusion

In this study, an *in situ* LIBS system was successfully established on EAST and operated as a daily diagnostic for the first wall before and after machine operation. The measurements were performed in the presence of the 2.5 T magnetic field and a base pressure of 10^{-5} Pa vacuum. The thickness of the deposition layers, element depth profiles and fuel retention were studied by performing LIBS measurements on a set of samples that were mounted on the high field side of the first wall. The results demonstrated the potential applications of LIBS for *in situ* characterization of fuel retention and co-deposition on the first wall of EAST. Hence, LIBS is a promising *in situ* method to replace the post-mortem methods for monitoring fuel retention and impurity deposition in the future. This work is valuable for the application of LIBS as a wall diagnostic technique for ITER and future fusion devices. In further studies, the main goals are to perform radiometric calibration of the spectroscopic system, to monitor a large area of the first wall and to upgrade the diagnostic to cover the divertor region.

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