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Laser-Induced Breakdown Spectroscopy for W7-X Limiter Tile Analysis in OP1.1

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Abstract: Laser-Induced Breakdown Spectroscopy (LIBS) is a well-established elemental composition analysis method as well as one of the most promising candidates for in-situ first wall diagnosis of fusion devices. In this work, limiter graphite tiles which were exposed in the initial operational phase (OP1.1) of the Wendelstein 7-X stellarator to He and H plasmas are analyzed ex-situ by LIBS employing a picosecond pulsed laser for the first time and compared with post mortem analysis techniques. Depth profiles of each element and 2D profile of the ratio of H and C atoms on the surface are investigated. Both H content and retention depth on the deposition dominated zone are high than them on the erosion dominated zone due to the formation of C-H co-deposition layer. LIBS, cross-sectional scanning electron microscope image and electron dispersive X-Ray spectroscopy results on deposition layer are in agreement.

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

1. Introduction

Analysis and understanding of plasma wall interaction (PWI) such as material erosion, deposition, and fuel retention processes on plasma facing components (PFCs) are vital for the safe and reliable operation of long pulse nuclear fusion devices. For safety reason, the maximum quantity of tritium in the ITER vessel is limited to 700 g [1], which requires in-situ monitoring of tritium retention. The co-deposition formation on PFCs is accompanied with fuel retention [2,3] which is a key issue for fusion device, especially for long-pulsed operation fusion device such as ITER. Wendelstein 7-X (W7-X) [4] which is the biggest stellarator in the world has presented 3D plasma topology in its first operational

campaign in limiter configuration with graphite-based PFCs [5]. Therefore, investigation of the role of 3D divertor concepts in the physics and control of edge transport and stabilities, heat and particle exhausts is essential [6]. Study on fuel retention and material migration of PFCs in W7-X is vital to understand the 3D PWI in stellarator. Laser-induced breakdown spectroscopy (LIBS) is a well-established elemental composition analysis method as well as one of the most promising candidates for in-situ first wall diagnosis of fusion devices [7,8] which is essential for long-pulse operation as post-mortem analysis is inhibited. Its capability has been recently demonstrated on measuring and monitoring the fuel retention and the composition of deposition layers on PFCs of several fusion devices such as EAST [9-11], JET [12,13], ASDEX [14] and TEXTOR [15].

In the present study, limiter graphite tiles which were exposed in the initial operational phase OP1.1 of the W7-X stellarator to He and H plasmas are analyzed ex-situ by LIBS. Picosecond pulsed laser was employed to reduce the thermal effect and increase the ablated depth resolution. 2D mapping and depth analysis of H retention on the surface were provided by LIBS and compared with other post mortem analysis techniques such as scanning electron microscope (SEM), focused ion beam (FIB) electron dispersive X-Ray spectroscopy (EDX) and thermal desorption spectroscopy (TDS). The comparison allows both the quantification of fuel retention as well as depth-resolved information in absolute scale with LIBS.

2. Experimental Setup

2.1 Laboratory setup

Schematic of LIBS setup in the lab is shown in Fig.1. W7-X limiter tile samples were mounted on an XY translation stage inside a vacuum chamber which was equipped with quartz windows. The vacuum chamber was pumped down to a pressure of 1×10^{-5} Pa to reduce the effect from water in air. The light beam of a picosecond laser (EKSPLA, PL2241) with pulse duration of 35 ps were operated in 355 nm at 10 Hz. The laser beam was focused on the sample surface by a lens with a focal length of 300 mm. By focusing the laser beam to a diameter of 0.75 mm, the laser energy density of 4 J/cm^2 was achieved. Laser ablated depth of 100 nm/shot on sample was achieved by measuring cater profile by profilometer. The optical emission of plasma produced by laser was collected in a direction of 45° to the normal direction of sample surface by optical lens. The LIBS signal light was transported by an optical fiber into a USB spectrometer (Ocean Optics, HR2000). A spectral resolution of about 1 nm (FWHM) was obtained. SEM, FIB, EDX and TDS were also used for analysis.

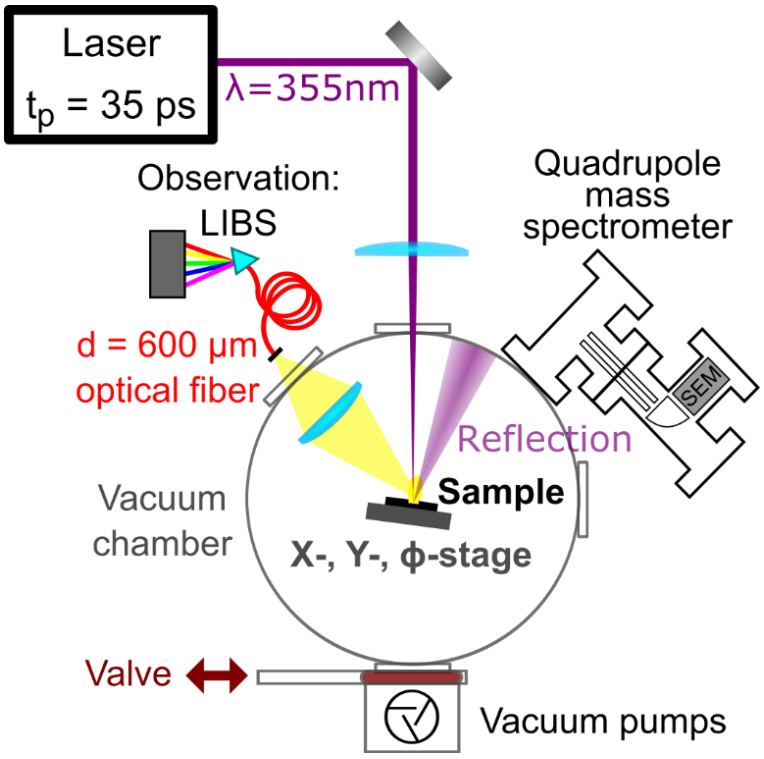

Fig.1 Schematic of ps LIBS setup with vacuum chamber.

2.2 Sample

During the first operational phase (OP1.1) of the W7-X, five specially shaped poloidal limiters, each consisting of nine discrete graphite tiles were used as the main PFCs [16]. The graphite tile with name of Limiter 4, Tile 6 was measured in this work. Dual erosion/deposition stripes were noteworthy on the surface of the tile (Fig.2). According to Winters's study [17], from the center to the edge of the tile along toroidal direction, four basic features were found: a smoothened center region, a rough deposit region, a pure erosion region and a smooth deposition region. In our study, we studied in erosion dominated zone near the center of the tile where tile received high flux plasma exposure and deposition dominated zone near the edge of the tile where C was re-deposited from erosion zone. LIBS was measured along lateral (x) from the middle of tile (x=0) to the edges of curved surface and compared to other analysis.

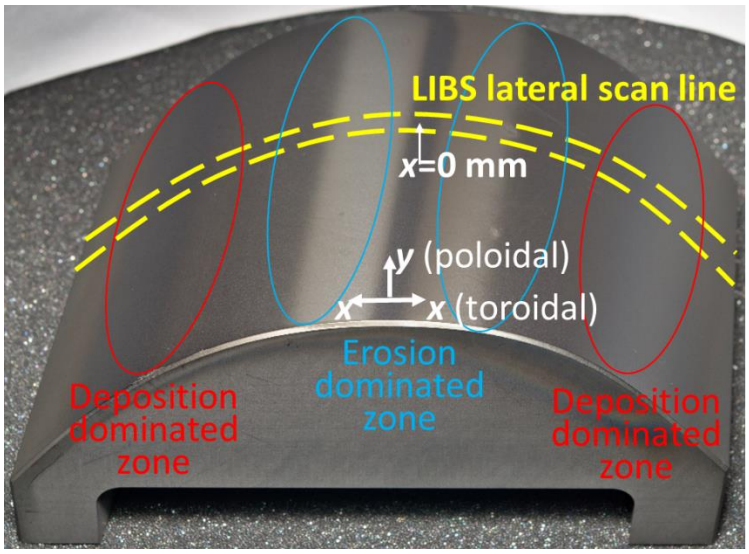

Fig.2 Photo of W7-X limiter tile exposed in OP.1.1.

3. Results

3.1 Cross-sectional on sample surface

Fig.3a shows the cross-sectional SEM image by FIB cutting on an erosion dominated zone of the sample on porous graphite. Due to high flux plasma exposure, there was no evidence of deposition on the erosion dominated zone. However, on a deposition dominated zone (Fig.3b), a smooth deposition layer with a thickness between 680 to 1100 nm on top of the graphite substrate was found.

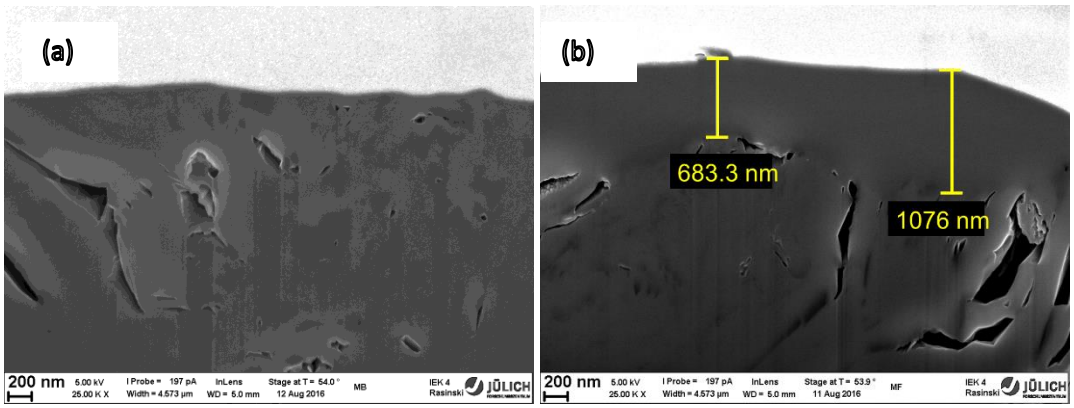

Fig. 3. Cross-sectional SEM images of (a) erosion dominated zone and (b) deposition layer on W7-X limiter tile.

3.2 LIBS results

Fig.4 shows the typical LIBS spectra of 1st and 10th laser shot on the same position (*x*=-96) on the sample. H (656.3 nm), Na (589.0 nm), Fe (396.9 nm) atom lines and C ion (657.8 nm) lines were used for LIBS analysis. All these elements signals were found on the 1st laser shot spectrum, however only C as substrate signal was found on the 10th shot spectrum. For depth profile analysis, the single shot LIBS spectra were recorded for 15 successive laser shots at the same position (*x*=-87). Fig.5 shows that the intensity of the H, Na and Fe peaks decreased with the number of laser shots and existed with few laser shots (few hundreds of nm). Impurity Na signal was much higher than it in the reference sample without plasma exposure. Fe element might come from the impurity transport from steel PFCs in W7-X during discharging. The C substrate was the only element whose intensity increased with the laser pulse and then plateaued.

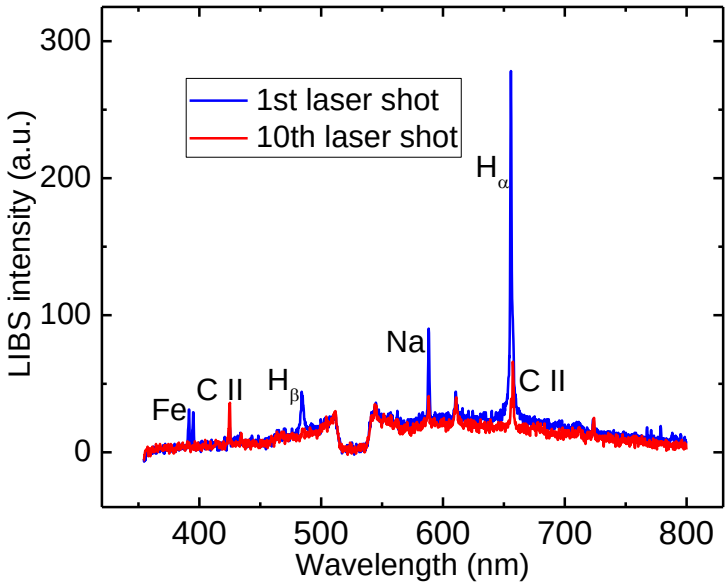

Fig.4 Typical LIBS spectra of 1st and 10th laser on the same position (*x*=-96 mm), H, Na, Fe atom lines and C ion lines were found.

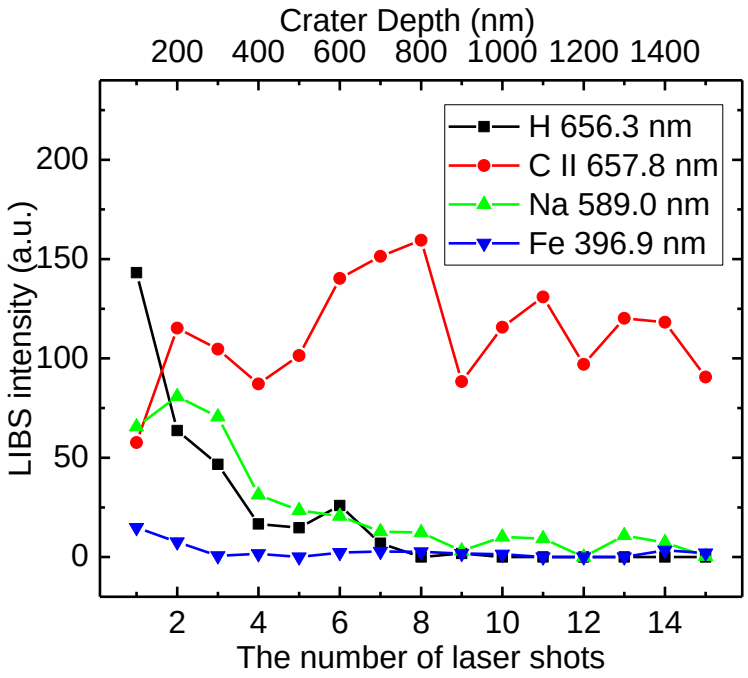

Fig.5 Depth profiles of H, C, Na, Fe elements on deposition dominated zone (x=-87 mm).

Fig.6 shows a lateral profile of the ratio of H and C atoms by integrating signal of 10 laser ablations. Total of 42 points were considered in two vertical (y direction) lines along the curved surface to get LIBS spectra for 2D elemental mapping analysis. The H signal was calibrated by TDS on reference samples. Assuming the density was the same in the samples, the C signal could be calibrated by ablated volume which was measured by a profilometer. Due to H plasma exposure, H contents were between 0.14 and 0.4 in the limiter tile. These were obviously higher than H content in the reference sample without plasma exposure. The lateral trend was almost symmetrical and consistent with stripes on the surface of the tile in Fig.2. Due to the formation of C-H co-deposition layer, H content in deposition dominated zone was higher than it in the middle part (erosion dominated zone) of the tile where H implanted due to high flux plasma exposure. H content at the edge of the tile where was far away from the plasma exposure zone was lower than other zone on the tile.

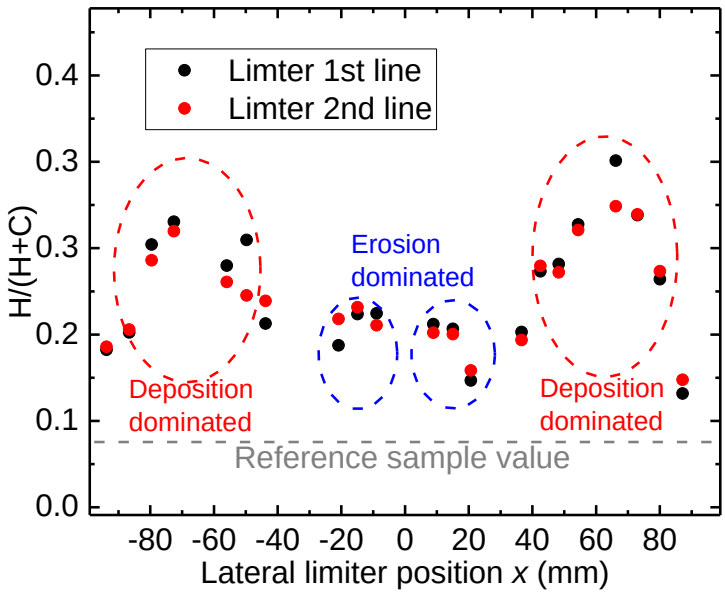

Fig.6 Lateral profile of the ratio of H and C atoms with different positions (x), about 0.08 H content was found in the reference sample without plasma exposure.

Fig.7 shows the depth profile of H content on different zones on samples. H content was always very high on the top of the surface by the first laser shot due to contamination from air. H retention in deposition dominated zone existed about 10 laser shots (1000 nm) which was much deeper than it in erosion dominated zone where H retention existed only few laser shots. The thickness of C-H co-deposition layer was around 1000 nm by LIBS measurement which consistent with the cross-section result in Fig.3. On the reference sample without plasma exposure, most of H retention existed on the top layer of surface and there was almost no H content from the second laser shot. EDX analysis with C and O intensity on different zones on the sample is shown in Fig.8. On erosion dominated zone, no O layer was found. There was clear O layer with thickness of about 900 nm on deposition dominated zone. However, O concentration in the sample was below the limit of detection of the LIBS setup. The thickness analyses of C by LIBS and O by EDX were in agreement.

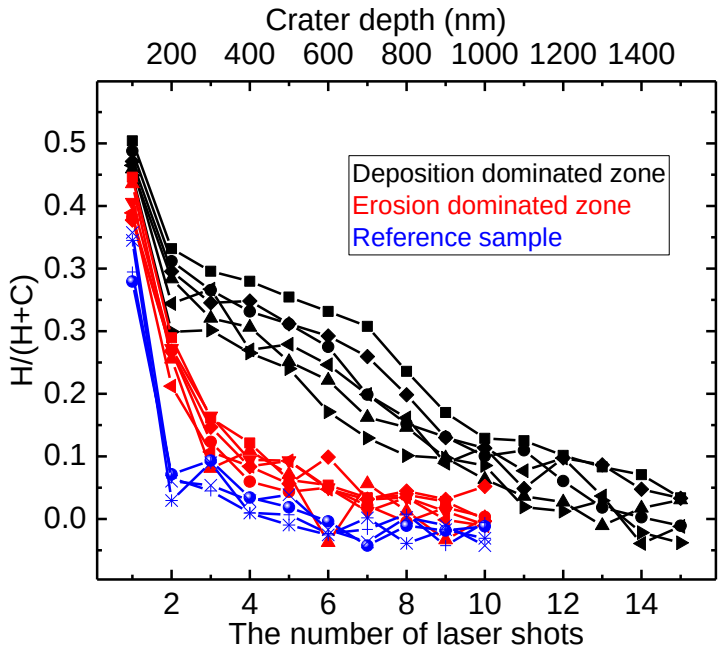

Fig.7 Depth profile of the ratio of H and C atoms on deposition dominated zone, erosion dominated zone and reference sample without plasma exposure.

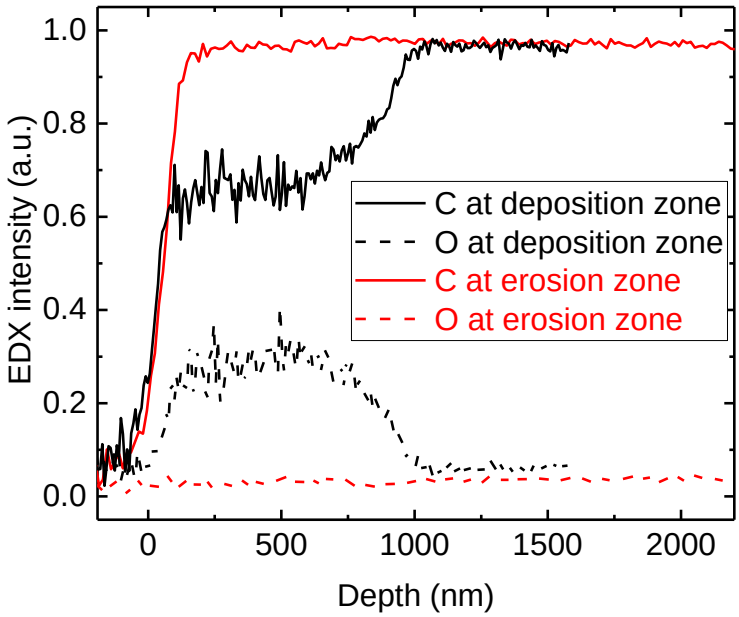

Fig.8 Depth profile of EDX intensity of C and O on deposition dominated zone and erosion dominated zone.

4. Conclusion

In this work, limiter graphite tiles which were exposed in the OP1.1 of the W7-X to He and H plasmas were analyzed ex-situ by LIBS employing a ps pulsed laser and compared with post mortem analysis techniques. A smooth deposition layer with thickness between 680-1100 nm could be obvious found on the deposition dominated zone on the sample by cross-sectional SEM images. Elemental depth analysis shows that H, Na and Fe peaks intensities decreased with the number of laser shots and existed with few laser shot (few hundreds of nm). Impurity Na signal was much higher than it in the reference sample without plasma exposure. Fe element might come from the impurity transport from steel PFCs in W7-X during discharging. 2D profile of the ratio of H and C atoms shows that H content on the used tile was higher than it on reference sample without plasma exposure. Duo to the formation of C-H co-deposition layer, the H content on deposition dominated zone was much higher than it in erosion dominated zone. A maximum ratio of H and C atoms could reach to 0.4 on the deposition dominated zone. The H content was only around 0.08 was found on the reference sample without plasma exposure. LIBS depth analysis shows the deposition layer was around 1000 nm on the deposition dominated zone. The H retention on the erosion dominated zone was only few hundreds of nm. EDX depth analysis shows deposition layer with thickness of about 900 nm was found and consistent with LIBS and SEM results. In-situ study on the mapping of the PFCs surface by LIBS will be crucial to understand the 3D plasma on the plasma wall interaction in the stellarator geometry for further operation phases.

Acknowledgements

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