Hydrogen depth profiles using laser-induced breakdown spectroscopy (LIBS) on graphite target of divertor in LHD

N. Ashikawa[1], Dongye Zhao, Cong Li[2], Hongbin Ding[2] and LHD experimental group[1]

[1] National Institute for Fusion Science, Gifu, Japan

[2] Dalian University of Technology, Dalian, China

1. Introduction

Investigations on hydrogen isotope inventories in plasma facing walls are important with the view of controls of fuel recycling and in-vessel tritium inventories in fusion devices. But, removal processes of hydrogen isotopes have not been optimized yet and still serious problems in ITER and DEMO. In particular, retained hydrogen isotopes in deposition layers are higher than that in bulk materials. Depth profiles in target materials are different between the retained hydrogen isotopes originating from energetic hydrogen isotopes during plasma discharges and the molecular hydrogen isotopes.

Laser-induced breakdown spectroscopy (LIBS) is one of useful analytical methods for hydrogen isotopes with depth profiles on materials [1-2]. Advantages of LIBS as ex-situ measurements are (1) short time analysis, (2) elements can be detected including hydrogen isotopes and helium, and (3) intensities mappings at 2-D positions. For research topics of hydrogen removal experiments for ITER, analyses of hydrogen isotope depth profiles are important. In this study, a demonstration of hydrogen depth profile analyses on graphite target exposed to LHD divertor plasmas was done using LIBS. From a comparison with LIBS and other analyses, effective approaches are discussed.

2. Experimental setup

YAG laser that a wavelength of 1064 nm is injected to a target material under low pressure in a vacuum chamber as shown in Fig.1. A laser energy per pulse is 100 mJ and a pulse length is 5 ns. A spectrometer has seven silicon CCD array detectors. A gas extraction system was installed to create a controlled Ar atmosphere.

An isotropic graphite (IG-430U, Toyo Tanso Corp.) exposed to hydrogen divertor plasmas using the material probe system at the 4.5 lower

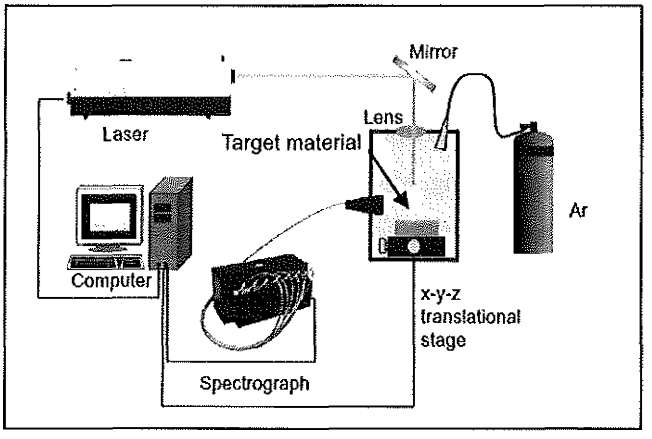

Figure 1. Systematic drawing about LIBS

port in LHD. A size of IG-430 target sample is 10 mm x 50 mm x 1 mm and three targets set on a sample holder made by molybdenum as shown in Fig.2 (a). A target "C" which located at right side of the holder in Fig. 2(a), was selected to LIBS measurement. Figure 2 (b) shows a picture of target "C" after LIBS analysis. A spot size of analyzed position is about 1 mm and each interval between analyzed points is about 1 mm. Analyzed points are written as circles in Fig.2 (b). Number of positions from 1 to 25 are written from an upper side to a lower side on this picture. Positions 4 or 5 (P4 or P5) are located at the divertor footprint of LHD and smaller numbers from P1 to P3 are located at private regions of the divertor configuration. For one position, total 20 pulses by Nd:YAG laser were injected and detected intensities at each pulse indicates different depth positions on the target.

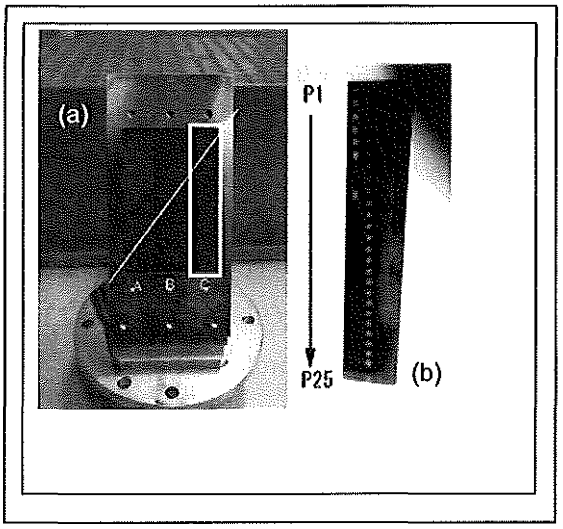

Figure 2 (a) A picture of a sample holder with IG-430U targets after plasma exposure in LHD. A line indicated the divertor footprint. (b) A picture of target "C" after LIBS analyses. Circles at 25 positions from P1 to P25 are analyzed traces by erosions of Nd:YAG laser.

3. Results

3-1. Hydrogen depth profile on graphite target exposed to LHD

Figure 3 shows spatial distributions of hydrogen, molybdenum, carbon and oxygen elements on the target sample C in Fig.2 (b). Figure 3 (a) is results at the first laser pulse and (b) is the second laser pulse by ex-situ LIBS. On the first layer such as in Fig.3 (a), deposited impurities and air contaminations are mixing. Higher hydrogen intensity is observed at private region of divertor plasma configuration. Positions from P1 to P13 show higher molybdenum intensities due to deposition from eroded sample holder made by molybdenum. Deposited molybdenum is observed at the first layer only and deeper regions does not show higher intensities such as the second layer as shown in Fig.3 (b).

Figure 4 shows depth profiles of H, Mo, C and O elements at the position 11 on the target sample C in Fig.2 (b). A sputtering rate by a laser pulse is not calibrated yet, but it is estimated about 100 nm / a laser pulse. Higher hydrogen intensities are detected from the top surface until 500 nm. In this analytical setup, a counting of 150 (a.u) is background signal level and sufficient counts were detected near top surface regions. This result of a depth distribution is consistent with effective areas of interactions from divertor geometry. Carbon intensities are uniform on this depth distribution.

3-2. A comparison with ex-situ LIBS and other analyzers

A detection limit of hydrogen is lower than that of glow discharge optical emission spectroscopy (GD-OES). For depth regions less than 100 nm, GD-OES data supports such as sensitive depth profiles near the top surfaces. For bulk target materials, such as ferritic steel alloys, thicknesses of iron/chromium oxide layers measured by GD-OES and XPS were compared and both thicknesses were about 30 nm. Hence, a good agreement based on an analytical result is shown. XPS can analyze elements with atomic number more than 3 and can not detect hydrogen and helium. On the other hand, GD-OES can detect elements with all atomic numbers due to a detection using s spectroscopy. A depth resolution of GE-OES is about 1 nm and it is good advantage of this analyzer. However, signal intensities depend on glow discharge parameters, and a stability of glow discharges is required.

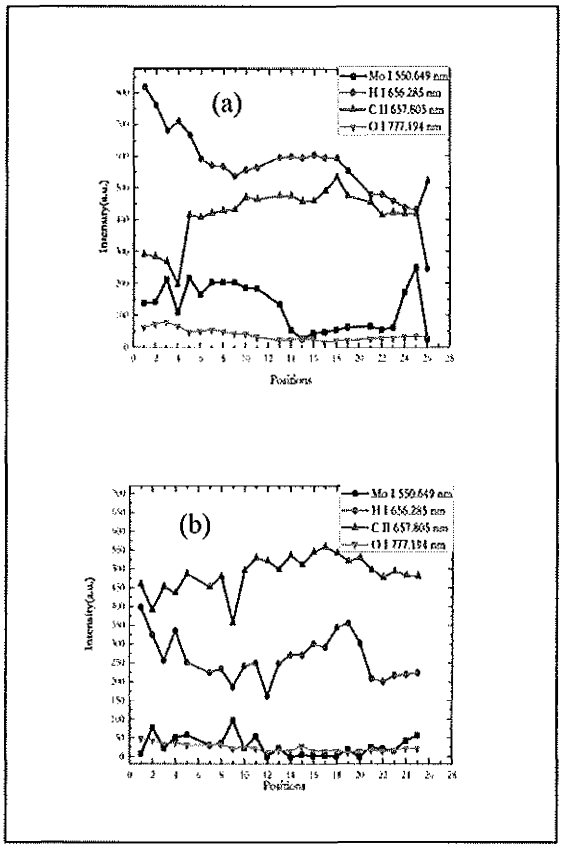

Figure 3. Spatial distributions of H, Mo, C, and O elements on the target sample C in Fig.2 (b).

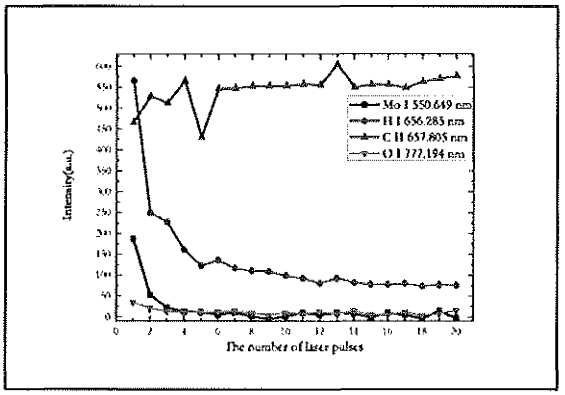

Figure 4. Depth distributions of H, Mo, C, and O elements at position 11 on the target sample C in Fig.2 (b).

Advantages of ex-situ LIBS are a good detection sensitivity, a small spot size of a detection area and detected elements with all atomic numbers. A detection sensitivity of LIBS is two order of magnitude higher than that of GD-OES. Detection areas have erosions in both analyzers, and then smaller detection area by LIBS is better. A minimum depth resolution by LIBS is about 50 nm and it is depending on materials, and it is difficult to observe an order of 1 nm.

Two analyzers, LIBS and GD-OES, have different characterizations, and good advantage for hydrogen and helium detections. A comparison between GD-OES and LIBS data is planned near future.

4. Summary

Hydrogen and impurities depth profiles on the IG-430U target exposed to hydrogen divertor plasmas of LHD were measured by LIBS and a proper results is obtained. At present, quantitative values cannot be analyzed using LIBS, but this results using the graphite target show one of an advantage of LIBS, such as good sensitivity with low detection limit of analyzers. In future work, a comparison with LIBS and other analyzers, such as GD-OES are planned.

Acknowledgments

This work was supported by the NIFS budgets ULFF004, KUHR010 and. the JSPS-NRF-NSFC A3 Foresight Program in the field of Plasma Physics (NSFC: No.11261140328, NRF: No.2012K2A2A6000443).

References

[1] Cong Li, et al., J. Nucl. Mater. (2015) 915.

[2] Ran Hai, Cong Li, et al., J. Nucl. Mater. (2013) S1168.