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In situ analysis of impurities deposited on the tokamak flange using laser induced breakdown spectroscopy

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HIGHLIGHTS

- Identification of deposited impurity material on the surface of the Aditya tokamak flange.
- Variation of spectral intensity of impurity elements with successive number of laser shots.
- Quantification of impurity elements present on the exposed surface of the flange using CF-LIBS.
- Potential of LIBS to study different matrices using successive number of laser shots.

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ABSTRACT

The impurity materials deposited on the plasma exposed surface of the flange in Aditya tokamak have been identified using laser induced breakdown spectroscopy. The spectral lines of Fe, Cr, Ni, Mn, Mo, Cu and C are detected in the coated layer of the flange. Intensity variation of spectral lines with successive number of laser shots has been studied. The concentrations of elements present in successive laser shot (1st to 12th) have been determined using CF-LIBS technique. Although, the intensity of the spectral lines in 1st and 12th laser shots are entirely different but relative concentration of constituents are similar. This anomalous behavior may be due to different nature of the matrices of 1st (coated layer) and 12th laser shot (flange material). Present study clearly demonstrates the capability of CF-LIBS to study the two different natures of the samples but having similar relative concentration of the constituents.

1. Introduction

Laser-Induced Breakdown Spectroscopy (LIBS) technique has emerged as a powerful analytical tool which is based on the atomic and ionic emission lines emitted from the plasma generated by the intense laser pulse-sample interaction [1]. In the last decades LIBS has become very popular owing to numerous advantages such as rapidity, multi-elemental analysis, remote material analysis, minimal sample preparation, minimal sample destruction, low cost, versatility of being applied to a wide range of materials without any preprocessing, and flexibility in experimental setup, with strong potential for in situ and real-time analysis [2]. In LIBS, sample material is vaporized, excited to higher energy levels and dissociated into ionic and atomic species which then relax to their ground state resulting in emission of characteristic wavelengths of elements present in the sample [3–6]. High-resolution spectrometer is used for spectral analysis of the emitted light from the plasma for identification and quantification of the elements present in the sample [7]. Many research groups have done the quantitative analysis using the calibration curve method which requires reference samples with similar matrix and having composition similar to the unknown sample. For quantitative elemental analysis, Ciucci et al. have developed calibration-free LIBS (CF-LIBS) approach, it is an alternative method to calibration curve, which require no reference sample. It is based on amount of constituent present in the plasma; it uses the plasma temperature and electron number density, assuming that the plasma composition reflects exactly same composition of the sample, i.e. stoichiometric ablation; provided the plasma is optically thin and in local thermodynamic equilibrium (LTE) [2]. CF-LIBS method is used for analyzing different metallic alloys, as well as nonmetallic samples such as soils, rocks, glasses, human hair and environmental samples [8–14].

LIBS can be used as a diagnostic tool for characterization of the wall conditioning/coating on wall of tokamak vacuum vessel. It can be performed under ultrahigh vacuum conditions and the existing toroidal magnetic field [15]. In magnetic confinement fusion devices, due to plasma-wall interactions during the plasma discharge, lead to the erosion of plasma facing

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components. These eroded impurities are transported to relatively cooler areas of the vacuum vessel and re-deposited as thin layers on their surfaces [16–19]. Eroded wall materials [20–21] migrate in the vacuum chamber and accumulate in the ducts equipped with the diagnostic optical elements and other parts like flange [18].

In the present study, surface of the flange exposed by the tokamak plasma is used to identify the impurities. LIBS can perform the qualitative and quantitative analysis of the material deposited in the different part of the vacuum vessel of tokamak, which is in line of sight of detector. An idea of the thickness of the deposited material by measuring the variations of their spectral intensities with successive number of laser shot was also employed. Elemental composition in the different layers of impurity and flange was evaluated using CF-LIBS approach.

2. Experimental setup

Fig. 1 shows the experimental arrangement to record the LIBS spectra of the flange of the tokamak at different depths and different locations. It consists of a laser source, a rotational stage (Sandvic Components, India) placed on jack (Sandvic Components, India). The laser source was a frequency-doubled Nd:YAG laser (532 nm) (Continuum Surelite III-10) capable of delivering a maximum energy of 425 mJ with a pulse duration of 4 ns (full-width at half-maximum) with a maximum pulse repetition rate of 10 Hz. In the present experiments, a lens of focal length 30 cm was used to focus the laser beam onto the flange surface. The emission from the plasma was collected at an angle of 45° to the incident laser beam by an optical fiber bundle and was fed into the spectrometer (Ocean Optics, LIBS 2000+) equipped with a charge-coupled device (CCD). The sample was rotated mechanically to get fresh spot of the sample for every laser shot. The LIBS spectra of different portions of the sample were recorded using a spectral resolution of 0.1 nm in the spectral range 200–500 nm. All the spectra have been recorded in open atmosphere. Software OOI LIBS 2000+ was used for analyzing LIBS spectra. To eliminate plasma continuum effects, the LIBS spectra were collected at a 1.5-μs gate delay. The laser pulse energy and pulse repetition rate were optimized and the best signal to background ratio and signal to noise ratio were observed with energy of 15 mJ. Laser pulse energy was measured with an energy meter (Genetec-e model UP19K-30 H-VM-DO) [4,6,14].

To study the elemental analysis of impurity material, we record the single laser shot LIBS spectra by focusing the laser beam on Aditya tokamak [22] flange surface. LIBS spectra have also been recorded by allowing successive number of laser shots at different points of the flange surface.

3. Result and discussion

3.1. Qualitative analysis of the flange

Fig. 2a–c represent the typical LIBS spectra of exposed surface of the flange. The wavelength of the atomic lines of the different elements appearing in the LIBS spectra of flange have been identified using the National Institute of Standards and Technology (NIST) atomic spectroscopic database [23] and Brode, Chemical Spectroscopy [24]. Spectral signatures of Cr, Fe, Ni, Mo, Mn, Cu, C, Ca and Mg are clearly seen in the LIBS spectra of exposed surface of the flange (Fig. 2a–c).

To study the different layers of the flange sample, successive laser beams were focused at a point of the exposed side of the flange and the LIBS spectrum of each laser shot was recorded. The intensity of the spectral lines of the elements present in the deposited layers (Cr, Fe, Ni, Mo, Mn, Cu and C) and matrix of the flange (Cr, Fe, Ni, Mo, Mn, etc.) were measured for every laser shots (i.e. one laser shot, two laser shot and so up to 12 laser shot at a point).

The tokamak wall and flange are constructed by the same material (SS316L) [25–28]. The impurity deposited layer is arising due to eroded material from the wall (SS316L) through the tokamak plasma-wall interaction. Therefore the constituents of deposited layer and the flange material are nearly same. Thus it is difficult to identify the deposited layer and the flange material whose compositions are similar. It is clear from Fig. 2a–c that the elements present in the LIBS spectrum of the deposited layer i.e. in the 1st laser shot are same as the constituents of the flange or wall materials. Fig. 3 shows the variation of intensity of the spectral lines of the elements Cr (428.9 nm) and Fe (438.3 nm) with number of laser shot at the same point of the flange sample. It is clear from the figure that the integrated intensity of these elements decreases with increasing number of laser shots. But, the intensity of chromium (Cr) and iron (Fe) spectral lines does not become zero on increasing laser shots; instead it becomes constant after 5th or 6th laser shot. One may think that in first 5–6 laser pulses, it modifies the surface of a homogenous substrate (change of roughness, optical properties) and only after these initial pulses equilibrium is reached. So to remove the above confusion, we have performed the experiment and recorded the LIBS spectra of the unexposed surface of the flange at different points with successive number of laser shots, but we do not see any considerable change in the spectral intensities of flange material. Thus, we came in a conclusion that the changes in the intensity of spectral line with successive laser shots occur because after 5th or 6th laser shot (Fig. 3a and b) the ablated material contains the flange constituents similar to the impurity material and for further laser shots the laser faces the same matrix. The color of the reflected light from the two

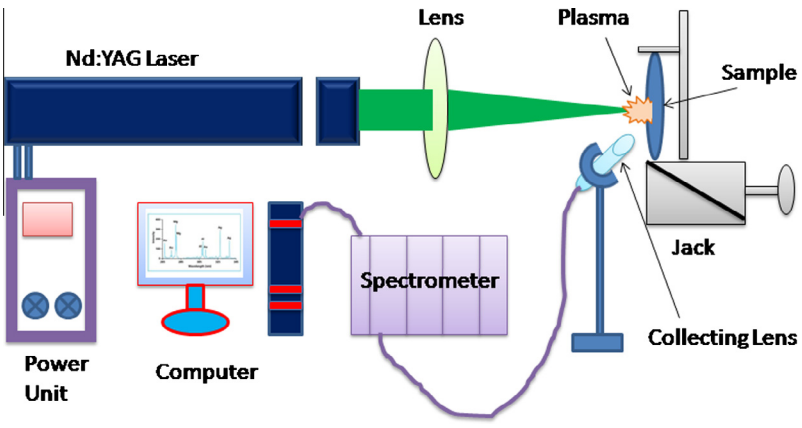

Fig. 1. Block diagram of experimental set up for recording the LIBS spectra.