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Laser based diagnostics of slaked lime plasma

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ABSTRACT

In the present work, we present the spatial evolution of the Slaked lime $Ca(OH)_2$ plasma generated by the first harmonic (1064 nm) and second (532 nm) harmonics of a Q-switched Nd:YAG laser. The experimentally observed line profiles of five neutral calcium (Ca I) lines at 601.59 nm correspond to 4p6s $^1P_1 \rightarrow$ 4s6d $^1D_2$ transition, 607.62 nm to 4p6s $^3P_1 \rightarrow$ 3d $^3P_0$, 634.46 nm to 3d9p $^3D_1 \rightarrow$ 4s6d $^3D_2$, 659.42 nm to 3d8p $^3P_1 \rightarrow$ 4s6d $^1D_2$ and at 712.17 nm to 4p4d $^1P_1 \rightarrow$ 3d $^3P_2$ respectively have been used to extract the electron temperature through Boltzmann plot method, whereas, the electron number density has been determined from the Stark broadening. Besides, we have studied the variation of electron temperature and electron number density as a function of laser irradiance at atmospheric pressure. The electron temperature and electron number density have also been calculated as a function of distance from the target surface.

1. Introduction

Laser-induced breakdown spectroscopy (LIBS), also known as laser-induced plasma spectroscopy (LIPS) has developed rapidly as an analytical technique over the past few decades. It employs a low-energy pulsed laser (typically tens to hundreds of mJ per pulse) and a focusing lens, to generate plasma on the surface of a target [1]. The spectrum of the plasma plume is the signature of the chemical species in the sample and its analysis yields their composition and relative abundance [2]. LIBS technique is unique that can be used to chemically analyze rocks, glasses, metals, sand, teeth, bones, powders, hazardous materials, liquids, plant, biological material, polymers and ceramics etc.[3]. The laser-produced plasma characteristics depend on several parameters including target features (physical and chemical properties), ambient medium properties (chemical composition and pressure) and laser pulse properties (pulse width, wavelength, spot size, and laser energy) [4]. Ohtsu et al. [5] observed calcium hydroxide slurry processing for bioactive calcium-titanate coating on titanium. López-Arce et al. [6] presented influence of relative humidity on the carbonation of calcium hydroxide nanoparticles and the formation of calcium carbonate polymorphs. Yang et al. [7] presented observations of pulpotomy in rats using *in vivo* Micro-CT—the changes after treatment of formocresol and calcium hydroxide pulpotomies or CO_2 laser irradiation. Philippen et al. [8] reported laser-heated pedestal growth of cerium doped calcium scandate crystal fibers. Garcimuño et al. [9] reported laser-induced breakdown spectroscopy for quantitative analysis of copper in algae. Garg et al. [10] reported Raman spectroscopic study of the evolution of sulfates and hydroxides in cement–fly ash pastes. Reig et al. [11] used influence of the activator concentration and calcium hydroxide addition on the properties of alkali-activated porcelain stoneware. Minh et al. [12] reported a novel one-step synthesis and characterization of bone-like carbonated apatite from calcium carbonate, calcium hydroxide and orthophosphoric acid as economical starting materials.

In the present work we have employed LIBS technique for the optical emission study of the $Ca(OH)_2$ plasma generated by the fundamental (1064 nm) and second (532 nm) harmonics of a Nd:YAG laser. We report the spatial evolution of the $Ca(OH)_2$ plasma in which, experimentally observed line profiles of neutral calcium (Ca I) have been used to extract the electron temperature (T_e) using the Boltzmann plot method. Whereas, the electron number density (N_e) has been determined from the Stark broadening parameter. Besides, we have studied the variation of electron temperature and electron number density as a function of laser irradiance.

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2. Experimental details

2.1. The sample

The sample under this study is Calcium hydroxide $Ca(OH)_2$. Calcium hydroxide, also called slaked lime, is a colorless crystalline powder. Slaked lime is useful as a cheap alkali for removal of acid gases in, for example, coal gas purification and recovery of ammonia in the Solvay process. It is used as a suspension in water, such as milk of lime. Calcium hydroxide has applications in leather tanning to remove hairs from hides, and is also used in the manufacture of bleaching powder, glass, caustic soda, mortar and cement. A small amount of it was used to prepare a pallet of 13 mm diameter and 3 mm thickness with the help of hydraulic press machine. The powder was pressed by a load of 10 t for time duration of 5 min. The Scanning Electron Microscope (SEM) photograph of the $Ca(OH)_2$ is shown in Fig. 1(a). The quantitative analysis of the sample conducted by JSM–6490A analytical scanning electron microscope (SEM) is shown in Fig. 1(b).

2.2. The experimental setup

The experimental setup is shown in Fig. 2, and is same as that described in our previous work [13–17]. Briefly we used a Q-switched Nd:YAG (Quantel Brilliant) pulsed laser having pulse duration of 5 ns and 10 Hz repetition rate which is capable of delivering 400 mJ at 1064 nm, and 200 mJ at 532 nm respectively. The laser pulse energy was varied by the flash lamp Q-switch delay through the laser controller, and the pulse energy was measured by a Joule meter (Nova-Quantel 01507). The laser beam was focused on the target using convex lens of 20 cm focal length. The $Ca(OH)_2$ sample was mounted on a three dimensional sample stage, which was rotated to avoid the non-uniform pitting of the target. The distance between the focusing lens and the sample was kept less than the focal length of the lens to prevent any breakdown of the ambient air in front of the target. The spectra were obtained by averaging 10 data of single shot under identical experimental conditions. The radiation emitted by the plasma were collected by a fiber optics

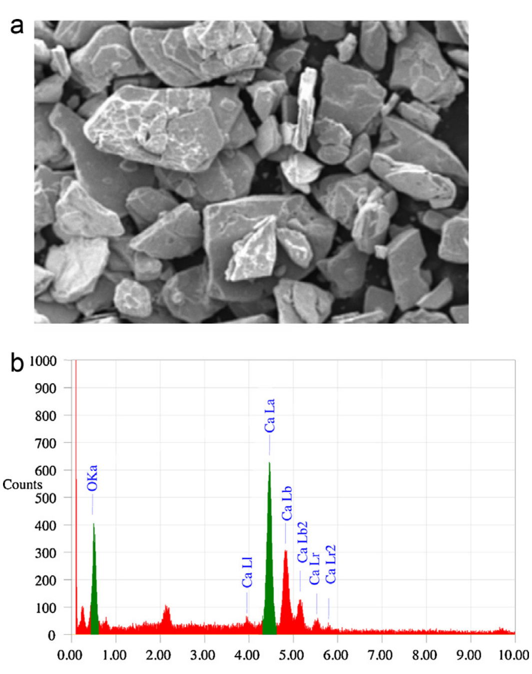

Fig. 1. (a) SEM photograph of slaked lime sample showing its morphology. (b) SEM photograph of slaked lime sample showing its morphology.

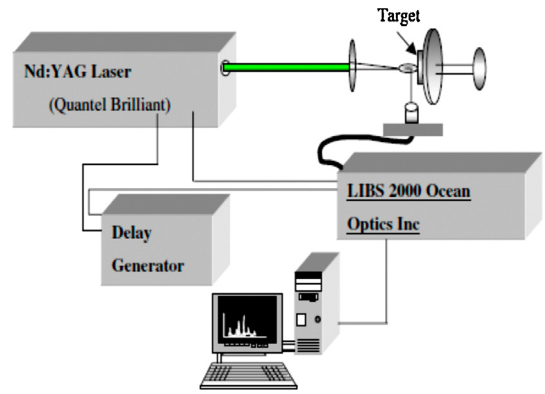

Fig. 2. Block diagram of the experimental setup.

(high-OH, core diameter: 600 μm) having a collimating lens (0–45° field of view) placed at right angle to the direction of the laser beam. The optical fiber was connected with the LIBS-2000 detection system (Ocean Optics Inc.), to measure the plasma emission. The emission signal was corrected by subtracting the dark signal of the detector through the LIBS software. The LIBS-2000 detection system is equipped with five spectrometers each having slit width of 5 μm, covering the range between 220 and 720 nm. Each spectrometer has 2048 element linear CCD array and an optical resolution of ≈0.05 nm by scanning a narrow bandwidth dye laser. In the experiments, the time delay between the laser pulses and the start of the data acquisition is about 3 μs, whereas the integration time is about 2 ms. In order to record the emission spectrum, the LIBS-2000 detection system was synchronized with the Q-switch of the Nd:YAG laser. The flash lamp out of the Nd:YAG laser triggered a detection system through a four-channel digital delay/Pulse generator (SRS DG 535). The LIBS-2000 detection system triggered the Q-switch of the Nd:YAG laser.

3. Results and discussions

3.1. Emission studies

In the first set of experiments, we have recorded the plasma emission generated by the fundamental (1064 nm) and second (532 nm) harmonics of an Nd:YAG laser. The laser was focused by a quartz lens with a focal length of 20 cm. Ca plasma was recorded at different positions along the direction of propagation of the plasma. Fig. 3(a) shows the emission spectrum recorded at 532 nm laser, covering the spectral region from 200 to 720 nm. Both neutral as well as singly ionized calcium lines are present in this region. Fig. 3(b) shows the emission spectrum recorded at 1064 nm laser covering the spectral region from 600 to 720 nm. This portion of the spectrum predominantly shows the spectral lines of singly ionized calcium (Ca I) lines. The calcium spectral line at 601.59 nm correspond to 4p6s $^1P_1 \rightarrow$ 4s6d $^1D_2$ transition, 407.62 nm to 4p6s $^3P_1 \rightarrow$ 3d $^3P_0$, 634.46 nm to 3d9p $^3D_1 \rightarrow$ 4s6d $^3D_2$, 659.42 nm to 3d8p $^3P_1 \rightarrow$ 4s6d $^1D_2$ and at 712.17 nm to 4p4d $^1P_1 \rightarrow$ 3d $^3P_2$ respectively. The assignment of these spectral lines is straightforward as the levels belonging to the lower and upper state configurations are well known and are tabulated in the database [18] and their transitions are shown in the Fig. 4.

3.2. Determination of electron temperature

Having observed the well-resolved multiplet structure from a number of excited levels and decaying to a common lower level, it is tempting to extract the plasma parameters from the observed spectra; in particular, the electron density and the plasma temperature.