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Original research article

Optimization of experimental parameters about laser induced breakdown and measurement of soil elements

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ABSTRACT

The experimental parameters for observing the characteristic of laser induced breakdown (LIB) plasma are optimized in the atmospheric environment. It shows that when the laser pulse energy is 21 mJ, the focal length of lens is 15 cm and the sample is 2 mm in front of the lens focus, the spectral intensity and the ratio of signal to noise (SBR) of LIB plasma are highest. It is helpful to improve the detection accuracy of the technique of laser induced breakdown spectroscopy (LIBS). The LIB spectra of a special soil samples are observed with the optimized parameters. The elements Fe, Mn, Ca, W, Co, Ti, Si, Cr and V contained in the soil sample are determined based on the spectral peaks. The electron temperature in plasma and the quantities of Mn, Ca in the soil are obtained further.

1. Introduction

Laser induced breakdown spectroscopy (LIBS), which has been developed with the development of laser technology and spectrometer, is a kind of spectral technique for qualitative and quantitative analysis of elements. It was first proposed by Brech at the 10th international conference on spectroscopy in 1962 [1]. Since 1980s, the technique has been developed more and more mature and has been widely used in various fields, such as on-line analysis of material composition, environmental monitoring, industrial process control, biomedicine, cultural relic identification and space detection, etc [2–8]. It not only has the advantages of fast, in situ and convenient operation, but also can realize multi-element analysis simultaneously.

In recent years, the application of LIBS technique on the detection of nutrition and heavy metal pollution in soil has received extensive attention [9]. As with the rapid development of industrialization and urbanization, the problem of soil pollution is becoming more and more serious. The detection of soil elements has gradually become one of the key contents of environmental science research. Y. C. Jiang et al. detected the content of magnesium in soil with LIBS technique [10]. A mobile LIBS system, which is used to detect heavy metal elements in soil has been studied by D. Meng et al. [11]. Martin et al. [12] have obtained the contents of total carbon, inorganic and organic carbon in soil with the help of LIBS. Owing to that the composition of soil is complex, the matrix effect is serious [13]. The stability of LIBS in the quantitative analysis of soil elements is still worse than the methods of mature spectrochemistry [14]. X. Fu et al. proposed a fast variable selection method that combining interval partial least squares and modified iterative predictor weighting-partial least squares. They applied it to the LIBS quantitative analysis of soils and obtained the detection limits of 11.4, 4.3, 3.6, 529.5 and 307.6 mg kg^{-1} for Cu, Ba, Cr, Mg and Ca [15]. The reliability of LIBS analysis is relate with many factors, such as laser energy, laser repetition rate, the focal length of used lens, the method for spectral data processing and so on. The main point of this paper is to seek

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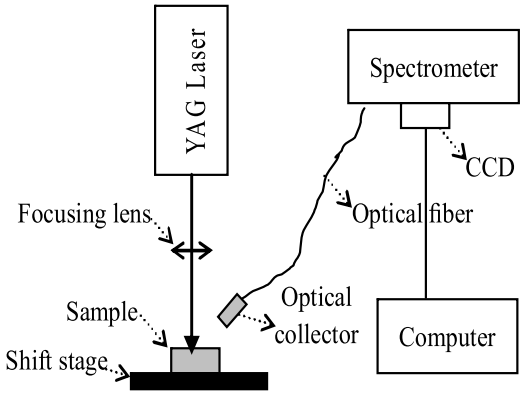

Fig. 1. Schematic diagram for LIBS.

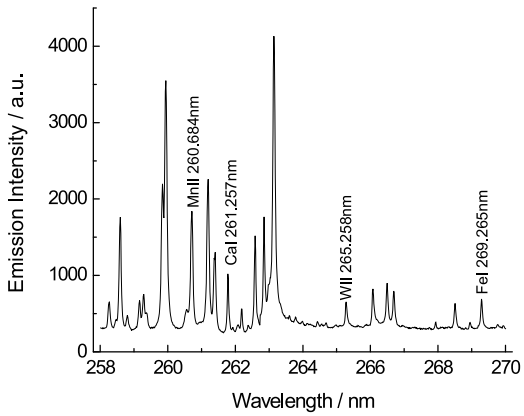

Fig. 2. Emission spectrum of soil plasma.

the optimal value of the experimental parameters. With the optimal parameters, the LIB spectra of a special soil sample are collected and the quantities of Mn and Ca in the sample are calculated by the method of free calibration

2. Experimental configurations

The experimental device for performing LIBS is shown in Fig. 1. It mainly contains excitation source, sample table, spectrometer and computer. The excitation source is the harmonic output 532 nm of an Nd:YAG laser (Powerlite DLS 8000). The repetition rate is 10 Hz and with 8 ns pulse duration. The output energy of the laser is adjustable. The grating spectrometer (Acton SP2750) has a spectral resolution of 0.03 nm. It is calibrated with a sodium lamp. The accuracy for wavelength measuring is ±0.1 nm. Laser can be focused on the surface of soil sample to generate high temperature plasma. The emission of the plasma is coupled into the spectrometer by a fiber array, which consists of 19 fibers with a core diameter of 200 μm. The dispersed light is converted into electric signal by CCD which is interfaced to the spectrometer. Then the signal from the CCD is fed into a computer for later analysis. The angle of the fiber head to the surface of the sample and the distance between the fiber head and the sample are adjusted to ensure that the spectrometer can not only obtain strong spectral signals, but also protect the fiber head from being contaminated by sputtering impurity particles.

The soil sample is collected from the mountain area of Fuping County, Hebei Province, China. After natural air drying, the collected soil is crushed, sifts out the impurity, and then is pressed into slice for later use.

3. Results and discussions

The LIB spectrum in the wavelength region of 258–270 nm of the soil is shown in Fig. 2. It is shown that the spectral peaks of CaI 261.754 nm, WII 265.258 nm and FeI 269.265 nm are independent of others, They can be selected to observe the influence of experimental parameters on the character of LIBS.