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# The effect of pulse energy on plasma characteristics of femtosecondfilament assisted ablation of soil



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## ABSTRACT

In this paper, the effects of pulse energy on the plasma characteristics of femtosecond filament assisted ablation of soil were experimentally studied, and the space-and time-resolved evolution processes of soil plasma parameters such as emission intensity, electron temperature and electron density were obtained. The experimental results showed that the soil plasma parameters decreased rapidly in the range of 400 ns delay time, and then decreased slowly with increasing delay time. When increasing the distance away from the soil surface, the soil plasma parameters increased firstly, and then decreased. The soil plasma parameters all increased with the pulse energy that formed the femtosecond filament and the peak position of its spatial distribution shifted from 1.5 mm to 2.0 mm away from the soil surface when the pulsed energy was increased from 2.45 mJ to 3.23 mJ. Finally, the local thermal equilibrium condition was verified based on McWhirte criterion.

#### 1. Introduction

With the development of human society, there have emerged the industrial polluted water discharges of metallurgical and chemical companies, rain wash of mineral slags, agricultural polluted irrigation and deposition of powder and dust. These result in different degrees of heavy metal pollution of substantial cultivated land, which leads to the emergence of famous Japanese "cadmium-contained rice" and "toxic rice" in Hunan of China, and other food problems that endanger human diet security. Therefore, the detection of heavy metal pollution in soil has become a hot spot in environmental science research [1]. Laser-induced breakdown spectroscopy (LIBS), with some advantages of in situ detection, no sample preparation, and simultaneous analysis of multiple elements, is suited for the detection of heavy metal pollution in soil. Unnikrishnan et al. [2] utilized LIBS detecting copper, zinc, calcium and other heavy metal elements in soil, indicating that LIBS technology is feasible for detecting heavy metal in soil. However, it was found that the distance between focusing lens and sample surface has an enormous influence on the spectral intensity of LIBS [3]. The unevenness of sample surface affected the detection stability of LIBS, which made it difficult for practical application of LIBS. Femtosecond laser is induced to breakdown air and form long-distance plasma filament, and the laser intensity along the filament is about  $5 \times 10^{13}$  W/cm<sup>2</sup> due to "optical clamp" effect [4,5]. Femtosecond filament induced breakdown spectroscopy (FIBS) can effectively solve the effect of the distance between lens and

sample surface on spectrum intensity of LIBS [6]. For the elemental analysis of remote sample, the conventional ns-LIBS with two main limiting factors of diffraction on focusing optics and atmosphere optical fluctuation is limited to the distance of a hundred meters due to the difficulty of delivering sufficient high laser intensities to induce ablation and formation plasma. But the FIBS can overcome these limiting for the capability of filament to overcome the diffraction limit and deliver high laser intensity at long distance without focusing [7]. Therefore, FIBS-based analyses of ingredients such as metal [8], heavy metal polluted soil [9] and chemical biological pollutants [10] have been carried out, and showed higher sensitivity in spectrum.

The energy forming femtosecond filament is about one-tenth of the total fs-laser pulse energy, and the remaining fs-laser pulse energy is transmitted forward and surround the filament, supplementing energy loss of filament transmission by the way of "energy bank" [11]. The energy of "energy bank" around filament increases with the increasing fs-laser energy. When femtosecond filament is induced to breakdown solid material to form plasma, the laser energy inside "energy bank" around filament to assist ablating copper target. There exists copper plasma electron temperature damping along the filament, while both electron density and spectrum intensity firstly increase and then decrease along the filament. Isaac et al. [13] found that the spatial position of maximum spectral intensity of filament induced copper plasma was inside the filament, and the filament can enhance the ionization degree of plasma,

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so deviations of the spatial peak positions of the electron temperature and density along filament occur.

Under the condition of tight focus, the pulse energy has an effect on the time and space resolved evolutions of femtosecond laser induced plasma emission spectrum, plasma electron temperature, and electron density [14]. The pulse energy is crucial for the formation and transmission of filament, and influences the energy spatial distribution along the filament [15]. At present, more researches on FIBS application for solid material composition detection have been carried out [12,13,15],and the space resolved plasma characteristics ablated by filament were also studied. However, the effect of pulse energy on plasma characteristics of femtosecond filament assisted ablation of soil sample was not report. In this paper, the effect of pulse energy on the plasma characteristics of femtosecond filament assisted ablation of soil was studied, and the timeand space-resolved evolutions of the plasma emission spectrum, electron temperature and electron density under different femtosecond pulse energy were also obtained. The research results will provide theoretical support for the extensive application of femtosecond filament induced breakdown spectroscopy technology.

#### 2. Experimental setup

The schematic diagram of the experimental setup for femtosecond filament assisted ablation of soil is shown in Fig. 1. The femtosecond laser (Libra-Usp-He, USA, Coherent) with center wavelength of 800 nm, repetition rates of 1 kHz, maximum pulsed energy of 4 mJ, energy stability of 0.5% and pulse width of 50 fs was focused by fused quartz lens L1 (500 mm focal distance) to breakdown air to form long length femtosecond filament. Soil sample was placed into the femtosecond filament routine. The plasma spectrum of soil produced by femtosecond filament assisted ablation was 1:1 imaged by fused quartz lens L2 (75 mm focal length) and coupled into a grating spectrometer (Spectra Pro500i, American PI) equipped with ICCD detector (PI MAXII,  $1024 \times 256$  pixel) with slit width of 30 µm, spectral resolution of 0.05 nm @ 1200 cm<sup>-1</sup> at 500 nm blaze wavelength. ICCD was triggered by femtosecond laser pumping system, and gate width was set as 30 ns. To avoid excessive ablation, soil sample was placed on XYZ (PI company) translation stage, which made filament always ablate a new position of soil surface and ensured the same experimental condition. The experiment was carried out under the environmental condition of one standard atmospheric pressure, 25° C room temperature and 22% relative humidity.

The soil sample used for the experiment, which are standard soil specimens (serial number GBW07458(ASA-7)) bought from China standard material website, are adopted as benchmark. 2.5 g of 99% purity lead nitrate  $Pb(NO_3)_2$  (Echo reagent) was put in 10 mL distilled water solvent. After complete dissolution, 5 g weighed standard soil specimen was poured in. After mixing evenly, place it in a vacuum oven at 60 °C for 2 h for drying. After vibration grinding, it was grinded into soil powder. Then press it under 25 MPa pressure for 30 min to prepare a Pb-containing soil specimen with diameter of 30 mm and thickness of 4 mm.

#### 3. Experimental result & discussion

#### 3.1. Plasma emission spectroscopy

Femtosecond pulsed laser with 3.32 mJ is focused by L1 to form a filament with long length about 40 mm. Soil sample is placed at the position 20 mm from the starting point of filament. The obtained plasma emission spectroscopy of soil sample is shown in Fig. 2. The detection delay time and gate width of ICCD are 300 ns and 30 ns, respectively, and the collection position of fiber probe is 1.5 mm from the surface of soil specimen. In the range of 385 ~ 425 nm, detection spectrum is mainly plasma spectrum of Fe and Pb elements. We selected Pb I 405.78 nm, Fe I 387.85 nm, Fe I 399.74 nm, Fe I 404.58 nm, and Fe I



Fig. 1. The experiment setup of femtosecond filament assisted ablation of soil sample.



Fig. 2. FIBS spectrum of soil sample.

407.66 nm as the analytical spectral lines for plasma characteristics of plasma filament assisted ablation of soil sample.

The position of spectrometer fiber probe is fixed at the point 1.5 mm from soil sample surface. The time-resolved evolutions of spectral intensities of Pb I 405.78 nm and Fe I 404.58 nm of soil plasma produced by femtosecond filament assisted ablation are shown in Fig. 3, the femtosecond filaments of which were formed by 2.45 mJ and 3.23 mJ laser pulses. It can be seen in Fig. 3 that the spectral intensities of Pb I 405.78 nm and Fe I 404.58 nm generally decreases with the increase of delay time. At the early stage of the plasma expansion, the soil plasma expanded rapidly in the form of adiabatic expansion process [16], and the plasma emission spectrum intensity decreased rapidly due to the cooling effect of the surrounding air. As the delay time increased, the atom density generation rate of three-body recombination of electronion-atom in the soil plasma was balanced with the reduction rate caused by the plasma expansion. Therefore, the atom spectrum intensity changed slowly with the increasing delay time. With the increase of the femtosecond laser pulse from 2.45 mJ to 3.23 mJ, the plasma spectrum intensity produced by filament assisted ablation increases significantly at the beginning of plasma expansion, while the change of plasma spectrum intensity is small at the later stage of plasma expansion.

The spatial evolution of spectral intensity of Pb I 405.78 nm and Fe I 404.58 nm was shown in Fig. 4, and the time delay was fixed at 150 ns. It can be seen that the spectral intensities of Pb I 405.78 nm and Fe I 404.58 nm increased firstly and then decreased with the distance away from soil sample surface along the axial expansion direction of soil plasma plume. The plasma spectral intensities were maximized in the range of  $1\sim2.5$  mm from soil sample surface at delay time of 150 ns. During the soil plasma plume expansion process, the particle population

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