Development of Laser-Induced Breakdown Spectroscopy System with a Palm-top Nd:YAG Laser

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China loess attracts attention in East Asia. In the previous research of our group, an elemental detection method with LIBS has been developed. In this work, we tried to develop the element detection method of China loess with a LD pumped passively Q-switched Nd:YAG laser. We developed the system which can duplicate appearance of China loess in the air. The breakdown thereshold of China loess blown by the air was estimated. And the required sensitivity in the system was also estimated.

Key Words: LIBS, China loess, DPSSL, Detection method

1. Introduction

Recently, economic growth rate of China has been increased, and these evolutions make atmospheric pollutions in China. These pollutions can contaminate the other environmental species such as China loess that has been blown into the atmosphere from northwest area of China, and are transported over long distances to East Asia and Pacific regions by strong westerly winds. ¹⁾ And it was found by the chemical analysis that when China loess passes through polluted air, heavy metal elements and oxides, like nitrogen oxides and sulfur oxides, accrete to surface of China loess. ²⁾ However, chemical analysis costs much time on pre-treatment and has risk of contamination.

Previously, our research group has developed a trace elements detection method on China loess by LIBS (Laser-Induced Breakdown Spectroscopy) with a flash lamp pumped Nd:YAG laser. LIBS is a well known elemental detection method without general pre-treatment, so it is suitable for in-situ monitoring.

When the high power laser pulse was focused directly in gaseous or liquid sample, or on a solid-state sample, the intense electric field can evaporate the sample and generate plasma.

So, spectroscopy on the emission from the evaporated atoms or ions gives the information of the trace elements in the sample.

LIBS includes: (i) real-time response; (ii) in-situ analysis with no sample preparation required; (iii) point sensing or standoff detection; (iv) multi-elements detection. ³⁾ LIBS was applied to detection of small-scale sodium leak ⁴⁾ or measuring distribution of elements in aerosol particles at Pittsburgh ⁵⁾ etc. In every case, advantage of LIBS appeared.

Our research group cleared up what kind of element was contained in China loess by LIBS too. Fig.1 shows the spectrum. The wavelength of plasma emission from each

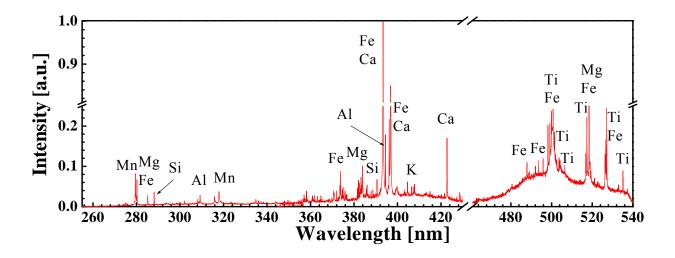


Fig.1 The spectrum of China loess obtained previously.

element is described at NIST Atomic Spectra Database.6)

But some facts were found from previous study.

- It took long detection time due to low repetition rate of the laser.
- · There was no need to breakdown air.
- Required fluence and intensity were 77J/cm² and 13GW² respectively.

From the above results, it is suggested that the lasers of high repetition, small size may be more suitable.

Recently many kinds of palm-top-sized lasers with Nd:YAG crystal or ceramic chips have been developed.

Though most of such lasers are cw lasers, it is easy to obtain passively Q-switching pulsed laser operations that can perform the peak power of the range of 100kW-1MW. Using longitudinally pumping with a single-emitter laser diode of high output power, the laser system can be simplified and miniaturized by reducing number of required optical elements.

In addition, the short pulse duration of 1ns and high repetition rate up to 3kpps can be expected. Our research group also have developed a passively Q-switched microchip laser with 4W laser diode. Peak power of 70kW and maximum pulse energy of 60μ J are obtained, respectively. It shows good beam quality of M² of 1.2.

Q-switched lasers fall into two types. One is the actively Q-switched laser and the other is passively Q-switched laser. Actively Q-switched laser contains an electro-optical crystal. Passively Q-switched laser contains a saturable absorber. High peak power can be obtained with actively passive Q-switched laser. And, actively Q-switched lasers can be controlled from outside. But they need voltage supply to control voltage applied voltage of electro-optical crystal. So, the device size can be very large. So, passive Q-switched laser was chosen.

In this work, passively Q-switched and palm-top-sized DPSS Nd:YAG laser is developed for real-portable LIBS system.

2. Laser system for micro LIBS

Figure 2 shows schematic of the palm-top laser system. A high power single emitter laser diode (Spectra Physics, SCT-200-808-Z6-01) is used as the pump source. And a Nd:YAG ceramic (5mm ϕ , 5mm at.) is used as the laser crystal. A Cr:GSGG crystal is used as a saturable absorber. LD

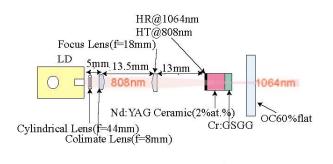


Fig.2 Scematic of the passively Q-switched Nd:YAG laser.

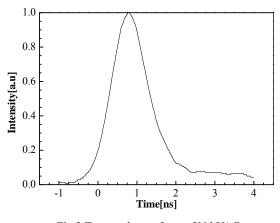


Fig.3 Temporal waveform of Nd:YAG



Fig.4 Photograph of the two lasers.

driving current is pulsed, and its current value and duration are 7.5A and 400 μ s, respectively. The pumping laser is collimated by the cylindrical lens (*f*=44 μ m) and the collimate lens (*f*=8mm) is focused by the focus lens (*f*=11mm) and illuminated to the Nd:YAG ceramic.

Temporal waveform of this passively Q-switched Nd:YAG laser output is shown in Fig.3. The pulse duration is 1.04ns. And the output energy is 80μ J. Horizontal M² and vertical M² are 1.73 and 1.22, respectively.

The length of the laser is 13cm. And the length of the laser system our research group used previously is 78cm. Fig. 4 shows difference on the size between the two lasers. The upper black and silver laser is the passively Q-switched laser developed in this work. The lower gray laser is the Nd:YAG laser pumped with flash lamp used for LIBS previously.

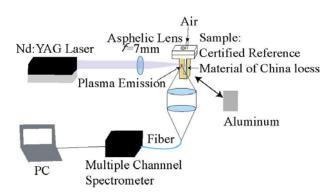


Fig.5 Experimental setup for LIBS of China loess.

3. Experiment

Figure 5 shows proposed setup of LIBS for China loess. Target is certified reference material of China loess. In this setup, the China loess is blown by air injection. The laser output is focused by focus lens (f=7mm) and illuminated to China loess samples, generating plasma. And the light of plasma is corrected to optical fiber and transferred to multi channel spectrometer.

The focused beam waist is calculated to be approximately $38 \ \mu \ m^2$. Figure 6 shows the gaussian fitting results with measured and calculated value. So, the intensity and fluence are estimated as approximately $210J/cm^2$ and $202GW/cm^2$ respectively. Breakdown on China loess is confirmed if the intensity is $17.25J/cm^2$ or more in this set up.

So, it is thought that LIBS detection with this passively Q-switched Nd:YAG laser is possible. But in fact, spectrum of China loess can be not obtained.

So, LIBS target is changed to aluminum as a reference. Figure 7 shows the spectrum of aluminum. Al $({}^{2}P^{0}{}^{-2}D)$ 308.2nm line and Al $({}^{2}P^{0}{}^{-2}D)$ 309.3nm line are measured. The plasma emission intensities of China loess and aluminum sheet are shown in Fig.7. Figure 8 shows scattering of the plasma emission intensities of China loess and aluminum. The average plasma emission intensities of China loess and of aluminum are 1.66 and 2.64, respectively. Standard deviation of plasma emission intensities of China loess and aluminum are 0.64 and 0.12, respectively.

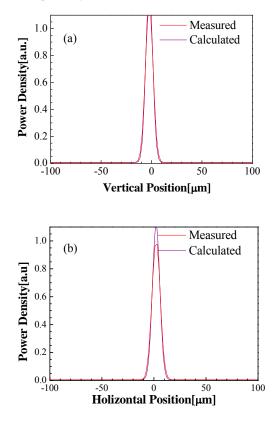
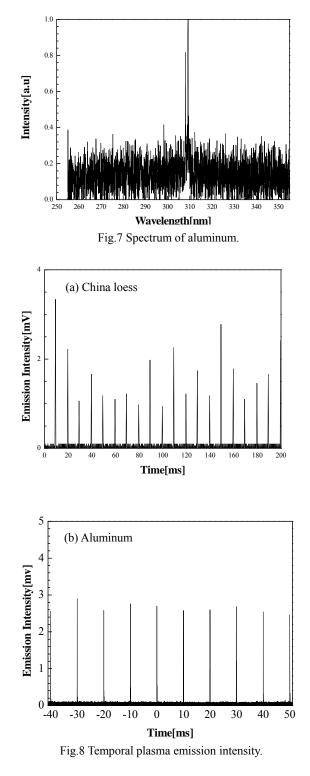


Fig.6 Focused laser spatial profile. (a) Vertical, (b) Holizontal.



The value of standard deviation of China loess may be ascribe to the size distribution of China loess particles and the place on China loess the focused laser output illuminated to.

And, the value of standard deviation of aluminum may be due to the output distribution of the laser.

The plasma emission intensity is scattered but due to high repetition of the laser, averaging signal can be obtained in a short time.

Al in China loess is 6 % of all. So, it is estimated that 3.0×10 times sensitivity is required to detect Al element in China loess. And it is found from Fig. 1 that LIBS signal intensity of Zn, one kind of heavy metal, the main target element, is 0.3 times that of Al. So, it is estimated that to obtain signal of Zn, 9.0×10 times sensitivity is required in the setup.

Repetiton of the laser was 100Hz. So, if repetition of the laser can be 9kHz, signal of Zn can be measured. But, it is difficult to perform repetition of 9kHz for the laser.

So, we will try to obtain signal of heavy metal with narrowband interfered filter and photoelectron multiplier.

4. Conclusion

We tried to obtain whole spectrum of China loess with passively Q-switched Nd:YAG laser pumped with LD. But it was found that 9.0×10 times plasma emission intensity is required to obtain signal of Zn in China loess in the setup. So,

we will try to obtain Zn signal with narrowband interfered filter and photoelectron multiplier.

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