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Double-Pulse Remote Laser-Induced Breakdown Spectroscopy Analysis of Magnesium Alloys

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ABSTRACT

A self-built double-pulse remote Laser-Induced Breakdown Spectroscopy system in a collinear configuration was used to investigate the magnesium alloys. The enhancement of the intensity was observed, about 4.7 times compared with single pulse LIBS. The peak intensities of line Y II 366.4 nm and Zr I 468.7 nm were used in the calibration curves, and the correlation coefficients were 0.9998 and 0.9547 respectively.

Keywords: Double Pulse; LIBS; Remote; Magnesium Alloys

1. Introduction

The chemical elementary component analysis of metallic alloys is very important for process control and quality assessment in metallurgical processing. The dominant analytical tools applied nowadays are based on spectroscopic techniques such as inductively coupled plasma-atomic emission spectroscopy (ICP-AES), X-ray fluorescence (XRF), etc. These techniques are mostly used in off-site laboratories and require sample preparation which is time consuming. Laser-induced breakdown spectroscopy (LIBS) is a useful technique known as a spectrochemical tool for detecting the chemical composition of a wide range of materials such as metals, minerals, chemical substances, and trace species without the need of sample preparation and the analysis procedure is simple and fast [1]. It is a potential powerful technique for the metal smelting in real-time and online chemical analysisor monitoring. However, the lower sensitivity and precision than the other elemental analysis methods is one of the major drawbacks especially in remote detection [2]. To improve LIBS, a lot of approaches have been proposed to enhance the analytical performance [3-6], and the double pulse LIBS (DP-LIBS) has been demonstrated by a number of studies [7-11], which is a very efficient approach for enhancing the intensity of plasma emission and improvement of the analytical capabilities of LIBS.

In this work, a self-built remote collinear DP-LIBS is used to analyze the chemical component of magnesium alloys and compare with the single pulse LIBS about the intensity of plasma light. The goal of this study is to confirm the effect of the intensity enhancement in remote DP-LIBS and the probability of the remote DP-LIBS technique applied in metal smelting process.

2. Experimental

The experimental system in this study consisted of two Nd:YAG lasers, which were characterized by a maximum repetition rate of 10Hz, a maximum energy of 200 mJ per pulse at 1064 nm, and a full width half maximum (FWHM) of about 10 ns. A combination of half wave plate and polarization beam splitter (PBS) was used to adjustment the energy of the laser beams and alignment the two laser beams in collinear. The collinear laser beams were focused on the targets at 2.5 m by a combination of four lenses. The emitted plasma radiation was collected by a commercially available 12 inch Schmidt-Cassegrain telescope, then focused into an optic fiber and guided to a spectrometer (LIBS 2500) developed by Ocean Optics, Inc. The delay time between the two lasers and the gate delay time of the spectrometer was set and controlled by a versatile digital delay/pulse generator (DG645). A schematic of the experimental setup is shown in **Figure 1**. In order to reduce the influence of Bremsstrahlung and free-bound electronic recombination continuum radiation, the gate delay time after the second laser pulse and the integrate time of the spectrometer were respectively set 3 μs and 1ms in all the measurements. For all the samples, 500 spectra were acquired and averaged into a single spectrum to reduce the spectral fluctuations.

All the magnesium alloy samples in this work were

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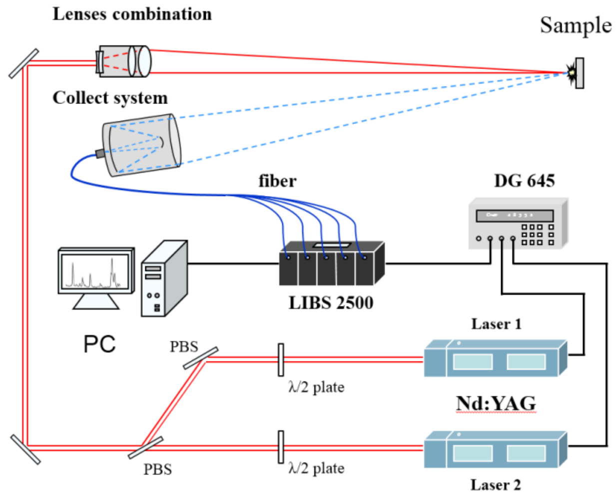

Figure 1. Theschematic of the experimental setup.

provided by the institute of metal research, Chinese academy of sciences. The average chemical compositions of the samples are listed in **Table 1**.

3. Results

The delay time between the two laser pluses plays an important role in the signal intensity of DP-LIBS [7,8,11]. **Figure 2** shows the emission intensity for the Zr I line at 468.7 nm and Y II line at 366.4 nm of sample #56 as a function of the delay time in DP-LIBS from 0 to 10 μs. It can be seen that the signal intensity increases by the delay time, maxima at about 6 μs. After 6 μs an almost constant intensity is observed. Consequently, the delay time between the laser pulses in DP-LIBS was fixed at 6 μs in this research.

Turned off the laser 1, just the laser 2 was on work for a single pulse LIBS in this work. **Figure 3** shows significant enhancement in LIBS signal for collinear DP-LIBS compared with single pulse LIBS. The energy of the both laser pulses were fixed at 100 mJ in DP-LIBS, and the energy in single pulse LIBS was fixed at 200 mJ for keeping consistent for both single pulse and double pulse LIBS. Sample #56 was chosen as sample for this study. Observed from the data, it can be clearly seen that the enhancement in signal intensity is about 4.7 times for collinear DP-LIBS compared with the single pulse LIBS of the Y II line at 366.4 nm. In the remote LIBS system, the higher laser energies are required for an effective detection due to the larger propagating consumption of the laser pulses and the plasma light in the free space [12]. Using the DP-LIBS, which allows lower laser energy with enhancement of the LIBS signal is an available technique to improve the intensity and sensitivity in remote LIBS.

According to the standard concentrations of the magnesium alloy samples in **Table 1**, the peak intensity of line Y II 366.4 nm and Zr I 468.7 nm were selected for

Table 1. Average chemical compositions of the Mg-RE samples.

C No.	Content			w/%	
	Zn	Y	Zr	Gd	Mg
#56	6.12	1.14	0.58		residue
#138	3.79	2.22	0.5		residue
#139	4.75	1.41	0.53		residue
#118		2.1	0.32	9.32	residue
#132	0.48	2.7	0.4	9.44	residue

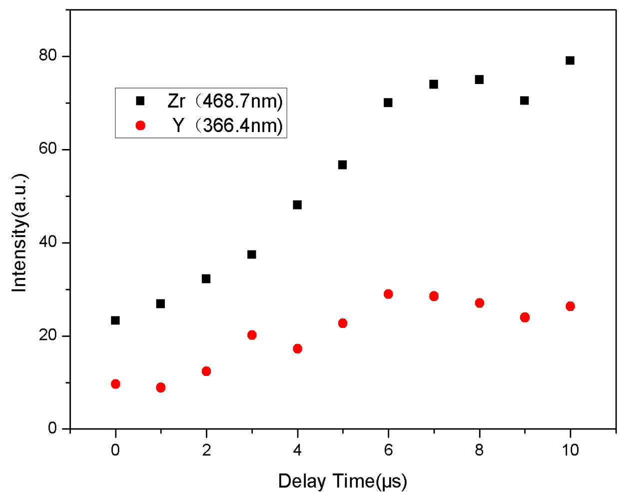

Figure 2. Intensity vs. gate delay time for the Y II 366.4nm and Zr I 468.7nm (#56).

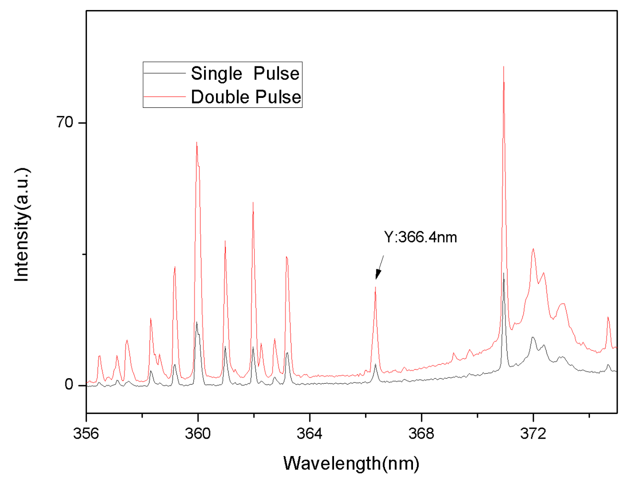

Figure 3. Comparison of DP-LIBS signalintensity with single pulse LIBS for Y (#56).

the calibration curves, plotted as a function of the relative concentration in a linear scale. In **Figure 4**, the calibration curve of Y II shows a near straight line, it was calculated that the correlation coefficients of the ratio (R^2) is 0.9998. Average relative error of calibration is less than

10% and the calibration curves that allow quantitative analysis in unknown samples [13]. In **Figure 5**, the calibration curve of Zr I is shown. The correlation coefficients of the ratio (R^2) is 0.9547, slightly poor compared with Y II at 366.4 nm, which probably influenced by the spectral peak overlapped with other elements such as Y, Gd, etc.

4. Conclusion

In this experiment, 5 magnesium alloy samples have been studied by a self-built remote DP-LIBS system. Compared with single pulse LIBS, the emission line intensities was enhanced about 4.7 times in DP-LIBS. The correlation coefficients of the calibration curves of Y II and Zr I were 0.9998 and 0.9547 respectively. The results of this study provide a potential DP-LIBS technique for metal smelting in real-time online chemical analysis and monitoring that used less energy to achieve enhanced spectra, and the distance between the system and targets about 2.5 m. The long-range detection distance of the LIBS system can be flexibility applied to the complex high-temperature environment of metal smelting process.

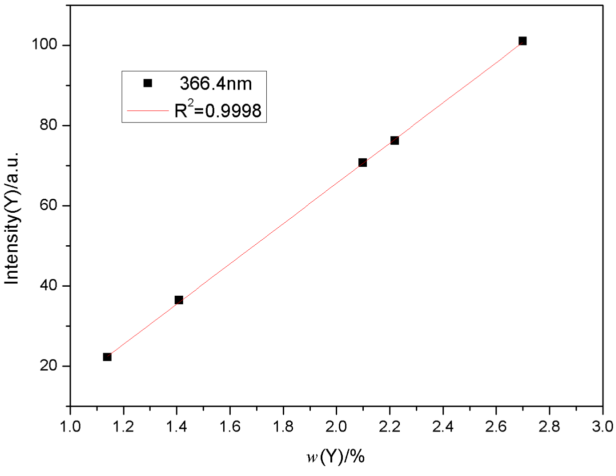

Figure 4. Calibration curve for Y II 366.4 nm.

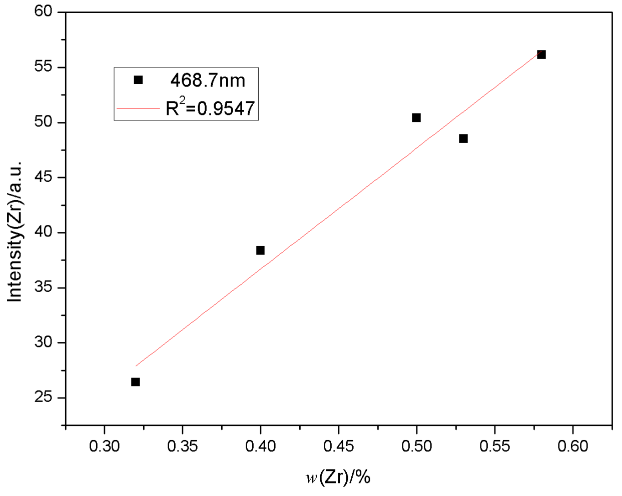

Figure 5. Calibration curve for Zr I 468.7 nm.

5. Acknowledgements

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