FEASIBILITY STUDY OF USING A PORTABLE, HIGH REPETITION RATE FIBER LASER FOR LASER-INDUCED BREAKDOWN SPECTROSCOPY

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Abstract

Laser-induced breakdown spectroscopy (LIBS) is a dynamically evolving elemental analytical method, which has a great array of unique and advantageous features such as quickness, multi elemental detection, virtually none destructiveness and requires little to no sample preparation. The quick advancement of LIBS is partly due to the ever improving properties of pulsed laser sources. One laser type which went through a significant improvement in recent years are the fiber lasers. In this current study we investigate the performance characteristics and feasibility of using a fiber laser for the ignition of microplasmas.

Introduction

LIBS measurements require the focusing of high energy pulsed laser light onto the sample surface. Due to the high irradiance the sample in the focal spot first melts, then evaporates, and from the evaporated sample a microplasma is formed. The analytical information is gained from the spectroscopic observation of this short lived light source.

Due to their robust nature, fiber lasers have recently been more and more widely used in the industry for cutting, welding, marking. Industrial fiber lasers usually employ a fused silica optical fiber doped with rare earth metals as an active medium (amplifier) and the end-on pumping is achieved by a fast semiconductor laser. This construction provides very good cooling, thus enabling stable, continuous operation and high energy/power and also allows a change of the pulse profile (waveform) [1].



Figure 1. Time resolved intensity profile (on the right) and intensity profile of consecutive laser pulses released from the fiber laser at a high pulse repetition rate (inter-pulse delay is $1 \mu s$)

These sources can provide continuous or pulsed emission, and their pulse repetition frequency (PRF) is much higher (kHz-MHz range) compared to that of typical solid state lasers (usually 1-20 Hz). They are robust, have a long life span (tens of years) and require little

maintenance. Due to the recent developments in this field, the pulse energy of these lasers reaches several mJ, accompanied by ns duration pulses, which brings up the possibility of their use in LIBS [2].

Experimental

During our experiments the laser pulses were provided by a fairly compact (473 mm x 417 mm x 133 mm) TRUMPF Trupulse nano 5020 fiber laser source, which has emission at 1060 nm and has 63 different pre-programmed waveforms. The maximum pulse energy and the length of the waveforms changed between 10 and 2000 ns, 0.35 and 4.95 mJ respectively. The light exiting the fiber laser was collimated into a 10 mm diameter beam and then focused onto the sample surface by a 100 mm focal length lens (Thorlabs, LA4380). Spectra were recorded by either an Avantes FT 2048 CCD spectrometer or an LTB Aryelle 200 Echelle spectrometer with an iCCD detector. The synchronization of the laser source and the spectrometer was achieved by a TTI TG5011 signal generator. Stainless steel and other iron alloys were used as samples.

Crater depths were measured by a Veeco Dektak 8 contact profilometer (Veeco, USA). To measure their depths, the craters were fully mapped, 40 parallel linescans were executed on all of them, then the line with the deepest line was chosen to identify the crater depth. The temporal profile of the waveforms was recorded by the usage of DET10A/M photodetector (Thorlabs, USA).

Results and discussion

As expected, different laser waveforms, even if they have the same energy, have different ablation and laser-induced breakdown spectroscopy properties. Although longer laser pulses have higher ablation rates, but the LIBS signal recorded from them is significantly smaller. It is probably due to the shorter lifetime of the plasma, which suggest that the initial temperature of these plasmas is lower as well.



Figure 2. The depths of craters created by laser pulses with the same pulse energy, but different pulse durations (on the left), LIBS signal recorded from microplasmas created by laser pulses with the same pulse energy, but different pulse durations (on the right)

It is known that consecutive laser pulses with small interpulse delays are able to dramatically increase the performance of LIBS [3,4]. These signal enhancing methods are known as double-pulse (DP-LIBS) if only two pulses are used, and multi-pulse LIBS (MP-LIBS), if three or more pulses are used. On the left hand side of the Figure 3. we show the cumulative plasma intensities (t_{int} = 2 ms), and as it can be seen the DP-LIBS is advantageous in the case of elongated (270 ns

long) pulses as well. Although the timing of the second laser pulse is known to have significant effect on the signal enhancement, but seeing the maximum enhancement at an inter pulse delay, when the plasma emission is already over was somewhat surprising. Thus we investigated the pulse energy of the second pulses as well, and found that, the energy of the second laser pulse is quite dependent on the interpulse delay, (right hand side of the Figure 3.). This means that the signal enhancement, what we realized is at least partly due to the increased energy of the second laser pulse.



Figure 3. LIBS signal intensities recorded by using 270 ns long consecutive laser pulses of the fiber laser as a function of the inter-pulse delay (on the left), and energy of the second laser pulse in function of the inter-pulse delay time (on the right)



Figure 4. Signal generated by the first second and third laser pulses, when these pulses had roughly the same energy (on the left), Plasma temperatures (using the Boltzmann plot method) of the plasma created by the first and second laser pulses (on the right)

This, however, does not mean that the signal enhancement is only due to the second, higher intensity laser pulse. We investigated the case, when the second and third laser pulses had roughly the same pulse energy, and still a significant increase in the intensity could be realised. In this case the increased signal cannot came from the reheating of the already existing plasma, since it has been already extinguished, but from the sample heating effect of the previous plasma (laser pulse). If not more than a few tens of μ s interpulse delay is used, then the sample surface is still warm when the next laser pulse arrives, thus it requires less energy for the ablation, and more is energy can be used to heat the plasma. This way the temperature of the plasmas created by the second and third laser pulses is elevated, as it was observed.

To demonstrate the usefulness of fiber lasers in the elemental analysis, LIBS spectra of stainless steel calibration standards were recorded using both single pulses and double pulses with the optimized waveform and interpulse delay. As it can be seen the DP-LIBS provide much better sensitivity, and the standard deviation of the signal does not grow significantly, hence the limit of detection also improves.



Figure 5. Comparison of calibration curves for cromium in stainless steel in SP and DP mode LIBS

Conclusion

In conclusion we managed to produce good quality LIBS spectra using a conventional fiber laser. The effects of the elongated pulses and high pulse repetition rates were investigated in detail. We also proved that this type of laser source is suitable for quantitative analytical measurements as well.

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