

Burst Mode Q-switched Laser Pulses for Plasma Excitation in LIBS Analysis

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1. Introduction

The Laser Induced Breakdown Spectroscopy (LIBS), is a technique that has been firmly established for the rapid determination of the elemental composition (Cremers, 2006). It relies on material ablation by using a short duration laser pulse with high density energy enough to produce plasma. By analyzing the light emitted by the plasma, it is possible to determine the elemental composition of practically any material. This technique has significant advantages over other conventional analytical techniques (Cremers, 2007; Rusak et al., 1998; Song et al., 1997). For example, requires no sample preparation, may be performed in solid, liquid or gas phase in samples with any shape or dimension. Moreover, it allows an in-depth study in order to characterize the composition of multi-component material (Adamson et al., 2007; Radziemski et al., 1983). It is especially suitable for field work by offering the possibility of real-time analysis with high portability. Thanks the above mentioned advantages, the LIBS technique has experienced strong growth, which is reflected in a large and growing number of publications.

LIBS is not a new technique: firsts laser-induced breakdown studies go back to the early 1960s and important application studies date from the 1980s with the work of Radziemski (Tognoni et al., 2002). A comprehensive review of LIBS development and applications through the mid-1990s was produced by Rusak *et al.* (Rusak et al., 1997).

The technique has many attributes that make it an attractive tool for chemical analysis, particularly as regards its potential as a field-portable sensor for geochemical analysis. LIBS is relatively simple and straightforward, so skilled analysts are not required. Little to no sample preparation is required, which eliminates the possibility of adulteration of the sample through improper handling or storage or cross-contamination during sample preparation. LIBS provides a real-time response and simultaneous multi-element detection and analysis. The laser plasma is formed over a very limited spatial area, so that only a very small amount of sample (picograms to nanograms) is engaged in each laser micro-plasma event. All components of the instrument can be made small and rugged for field use and LIBS sensors can be operated either as a point sensor or in a standoff detection mode. The

detection limits of LIBS are in the low hundreds to tens of ppm range for most common elements, so field-portable LIBS should be capable of field surveying and screening for the geochemical exploration and environmental remediation applications envisaged.

The existing equipment on the market used predominantly solid-state lasers based on Nd:YAG crystals. This is because this type of lasers, in addition to the advantages of robustness and compactness; allow obtaining very short pulses with high power density. To ensure the appropriate parameters, it works in the so-called Q: Switch regime. In this mode of operation, is introduced into the laser cavity an optical shutter based on a crystal whose transmittance is electrically switched, allowing the production of single-pulse of several nanoseconds in duration and very high intensity. In the last years several works have reported an improvement of plasma intensity, and therefore the limits and quality of detection, using systems that generate two consecutive pulses, delivered from one or two synchronized lasers (Tognoni et al., 2002). Recently, it was reported a system with dual crystal modulator by using two LiF crystals (Galbacs et al., 2005). While this system improved the signal-noise relation and the intensity of the plasma emission, has the disadvantage associated with the gradual degradation of these crystals (Zabello et al., 2003). In our study we propose a new low cost LIBS system, which uses passively Q:Switched, capable to produce pulse trains that enhances the plasma intensity. The employment of this regime is illustrated by LIBS characterization of art object. On the other hand, as an example of on-line monitoring of industrial process, and also the possibilities for different pulse regimes, we present the applications of free-running regime in the de-thorning process of cactus, a food Mexican product.

2. A low cost and portable LIBS device description

Figure 1 shows an outline of the experimental setup. A Nd:YAG laser emitting at 1064 nm, produces pulses with energy adjustable up to 0.9 J. The active element is a 6 x 60 mm Nd:YAG rod, pumped by a xenon lamp. It uses a Q:Switch consisting of a Cr:YAG crystal with 6 mm in diameter and 3 mm in thick and initial transmittance of 14%. The light emitted by laser is focused through a 5 mm focal length lens. The distance between the lens and the surface of the sample was 8.5 cm to achieve an area of 0.19 cm². For the experimental conditions used, this means a yield of 4.7 J/cm². The sample is placed on metallic base ensuring that the sample surface is located at the focal point.

An optical system coupled with the entry of optical quartz fiber, whose entry can be moved respect to the sample surface. By this way it is possible to capture the emission from a specific region of the plasma.

The spectrometer used in our configuration was an USB4000 Ocean Optics, with 350 nm to 900 nm spectral range and 0.2 nm resolution. The spectra were processed in computer by using Spectra Suite software.

2.1 Nd:YAG laser with passive Q:Switch

In the laser head of our device the pump radiation is produced by xenon pulse lamp. The light from this lamp is concentrated on the active medium of Nd:YAG through an special reflector made from monolithic quartz with external metallic coating. The quartz block is doped with atomic Ce at 1 % doping level. This feature avoids the optical damage of the active medium due the ultraviolet light emitted by xenon lamp. On the other hand, the

conversion of UV to visible radiation contributes to enhance the absorption of pump light in laser crystal and, in consequence, the laser efficiency.

For obtaining a compact device, the resonator mirrors of 99,8% and 50 % reflectance respectively, were placed at 1.5 cm from the rod ends. The rod dimensions are 5 x 50 mm. As a Q-Switch element a YAG:Cr crystal with dimensions 6 mm in diameter and 3 mm thick was used. The initial transmittance of Q-Switch was 21 % for 1064 nm wavelength.

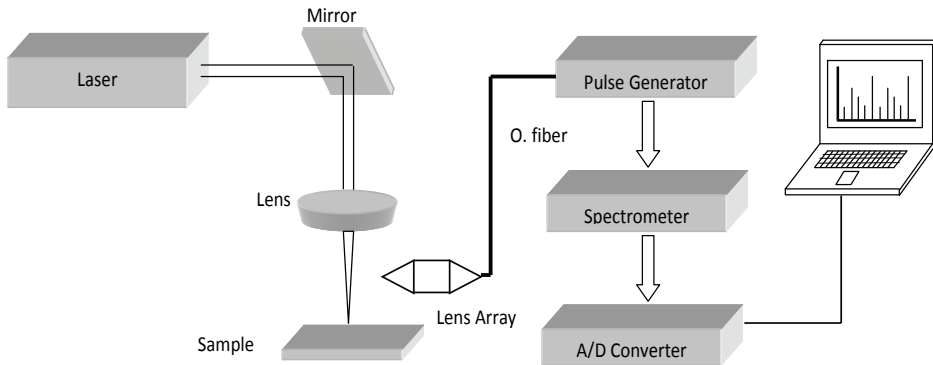


Fig. 1. Schematic of experimental setup

For this configuration, the laser can produce a train of pulses with duration for each pulse of about 20 ns, separated by 10 μ s interval, with a total duration of the pulse train about 700 μ s. In Figure 2 the acquired signal of laser pulse for passive Q-Switched laser is showed.

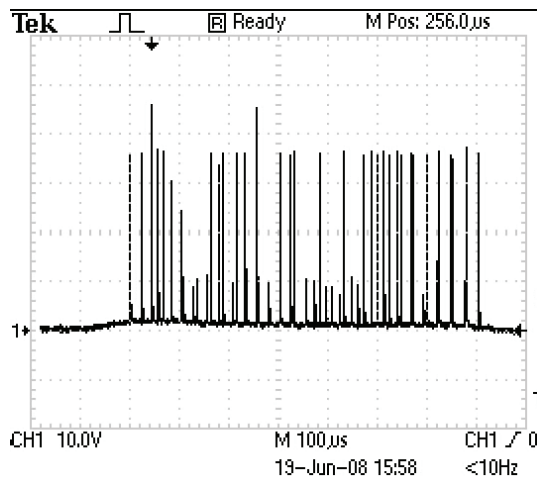


Fig. 2. Oscillogramme of pulse train obtained with passive Q-Switch.

All system elements are mounted on a stereo-microscope in order to obtain better facility for positioning and observation of samples as well as to follow the results of irradiation. The dimensions and weight of LIBS device is 40 x 20 x 15 cm and 5 kg respectively. Figure 3 shows a photograph of device, called MicroLIBS.

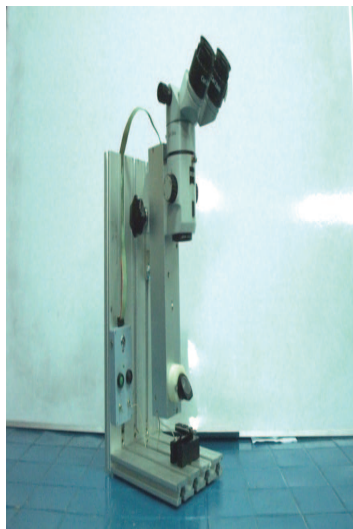


Fig. 3. Photograph of MicroLIBS system.

3. Results and discussion

The use of LIBS as an analytical tool depends on three underlying assumptions: (i) that material ablation is stoichiometric so that the composition of the plasma generated is fully representative of the sample composition; (ii) that an optically thin plasma is generated so that emission from the central high-temperature portion of the plasma plume is not reabsorbed in the colder plasma boundary region; and (iii) the plasma is in local thermodynamic equilibrium. These conditions have been verified by Chan & Russo (Chan & Russo, 1991) and Corsi *et al.* (Corsi *et al.*, 2000) and it is understood how to realize these conditions analytically.

The use of LIBS for elemental detection is simple and readily accomplished via peak matching against a spectral library constructed in advance for a specific application. For this type of application, which can be accomplished through statistical analysis of LIBS spectra (Anzano *et al.*, 2000), it is only relative peak intensities and overall spectral shape that is important, not absolute peak intensities. Quantitative LIBS analysis of specific elements in natural materials is significantly more difficult because of the broad issue known as “matrix effects”. This is an observed dependence of the intensity of the LIBS emission response to the nature of the material analysed (Eppler *et al.*, 1996), which manifests as variations in laser-target coupling and resultant plasma intensity variations. These two facets of the sample, which are generally lumped together as “matrix effects” (Aguilera *et al.*, 2009; Corsi *et al.*, 2000; Ctvrtnickova *et al.*; Lu *et al.*, 2010; Ma *et al.*; Mohamed, 2008; Shen *et al.*, 2009; Windom & Hahn, 2009), will determine LIBS signal reproducibility, namely sample composition and sample surface character.

3.1 Multi-pulse excitation

An important approach for enhance the detection limits and the signal-noise relation was reported early (Ponce *et al.*, 2008), and consist in the use of passively Q:Switched multi-pulse scheme in order to perform the plasma formation-excitation process. In order to

establish a comparison, it was used an electro-optically Q-Switched Nd:YAG laser with similar parameters with the passively Q-Switched laboratory-made device. In the first case, the laser pulse energy of 0.14 J was focused on an area of 1 mm diameter, thus obtaining a fluency of 4.6 J/cm², similar to that used in the passively Q-Switched laser. The only experimental difference: Pulse duration of 20 ns in single pulse configuration and 700 μ s total duration for the multi-pulse configuration. In Figure 4 are observed an example of spectra captured for both lasers. The spectra chosen to illustrate the comparison were collected from Prickly Pears spines. As can be seen, are clearly identified several peaks associated with the CaII, MgII and Cl. These peaks, like the rest of the spectrum, have higher intensity and better signal-noise relation for a train of pulses configuration.

These results can be explained through a several steps process (Zabello et al., 2003). Initially, the absorption occurs in the first laser pulse at the sample surface, with the consequent overheating above the melting point. This causes the explosive material ablation, his rupture and rapid heating of the surrounding atmosphere, and a strong electronic emission. The steam flow expands and produces a shock wave, which in its motion dragging behind an area of low pressure. The initial pressure is restored after about 100 μ s, as estimated experimentally (Kabashin et al., 1990).

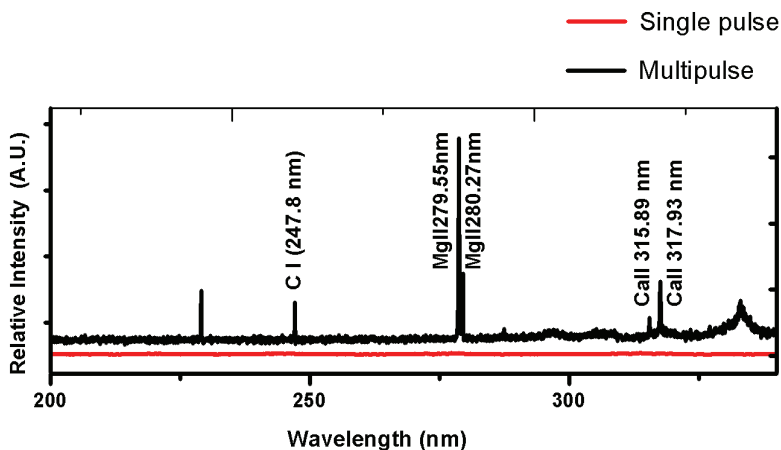


Fig. 4. LIBS spectra captured by using multipulse (above) and single-pulse (below) configurations.

By influencing subsequent pulses, separated from each other by a few tens of microseconds, the conditions for these interactions are the same as the first except for the presence of the above mentioned area of low pressure and a broadcast electronics (noise), much lower, because at that time the flow of electrons has disappeared. Moreover, the highest line intensity for multipulse regime can be explained by the additional excitation pulses produced in the steam of initially ejected material. Thus, the use of pulse trains by using passive Q-Switch, produce higher emission intensities and better signal noise rate.

3.2 Free-running excitation

Typically, the LIBS technique is performed using Q-Switched lasers with high power pulses (megawatt range), produced with time durations in nanosecond range. As far as we know,

no LIBS experiments were performed by using free-running regime until the recent reports of LIBS in Prickly Pear (Flores et al., 2009), because the low energy density for this mode of laser operation. However, in the mentioned report, due to the strong absorption in areoles, an intense plasma emission takes place even for microsecond range in pulse duration with kilowatts/cm² range of laser fluency. As we demonstrated, this allows to use in experiments not only a Q-Switched laser but also a free running low cost Nd:YAG laser regime. The experiments in free-running regime have a great practical interest because at this regime is possible to obtain a higher productivity in spines elimination. On the other hand, the laser operating at this regime is less expensive and the operation cost is lower.

At present, our research group is working of high energy and long pulse LIBS excitation, performed at millisecond pulse time duration. The special interest of this exotic conditions in plasma generation, is explained by two reasons: The possibility of simulation of some natural events like interstellar jets or atmospheric thunders from one side, and, the on-line monitoring of several industrial applications in millisecond range as hole perforation or pulse welding from the other side.

In principle, as lower pulse duration them better resolution can be obtained, if we taking in to account the fact that typically with shorter pulse duration avoid higher power densities and better plasma ionization level. I also important the fact that the plasma life is extended no more than several microseconds, and, in consequence, if the pulse duration is longer, them the interaction with subsequent excitation must be taken in consideration in the LIBS spectra interpretation. As an example of line-width behaviour for different pulse duration, LIBS spectra captured on metal sample are showed in figure 5.

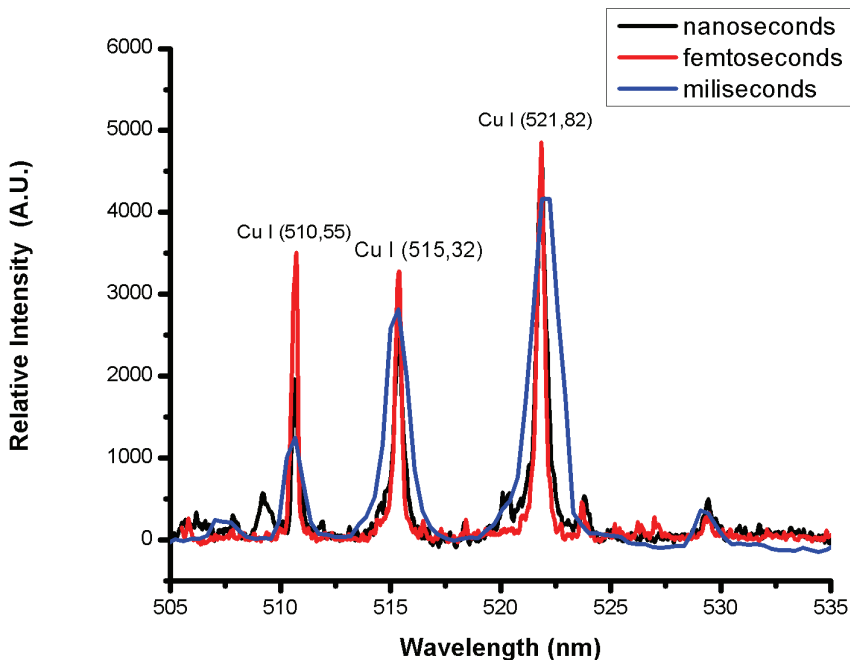


Fig. 5. LIBS spectra obtained from Cu sample.

3.3 Field applications: Free-running excitation for Cactus de-thorning

An exciting example of our LIBS device is the on-line elemental determination for laser de-thorning process. In this new application a Nd:YAG (Flores et al., 2009) laser is used for selective ablation of thorns of cactus called opuntia. In figure 6, is showed a deep-profile LIBS measurement of thorn-cortex systems illustrating the behavior of Na(I) 589.5 nm line and C-H band.

These curves shows that LIBS technique contribute to control the spine elimination process: For C-H line the relative intensity reach the maximal value after two or three pulses and decrease gradually until the full thorn elimination reaching in this moment a fixed value that correspond to lower percent of C-H concentration in cortex. In opposite direction, the Na line intensity grows gradually reaching a higher level simultaneously with C-H decrease. The high concentration of Na in cortex is reached after spine elimination precisely when the inner part of cactus is to be ablated.

On the other hand, it is another important difference between the two curves showed in figure 6. While the Na pike dependence versus number of pulses growth continuously, the C-H dependence shows periodical changes. It's remarkable that similar variations were detected in previous work (Arronte et al., 2010), by photo-acoustic technique when the signal intensity versus number of pulses was measured. In principle, these changes can be attributed to periodical character of combustion process in laser de-thorning, consisting in

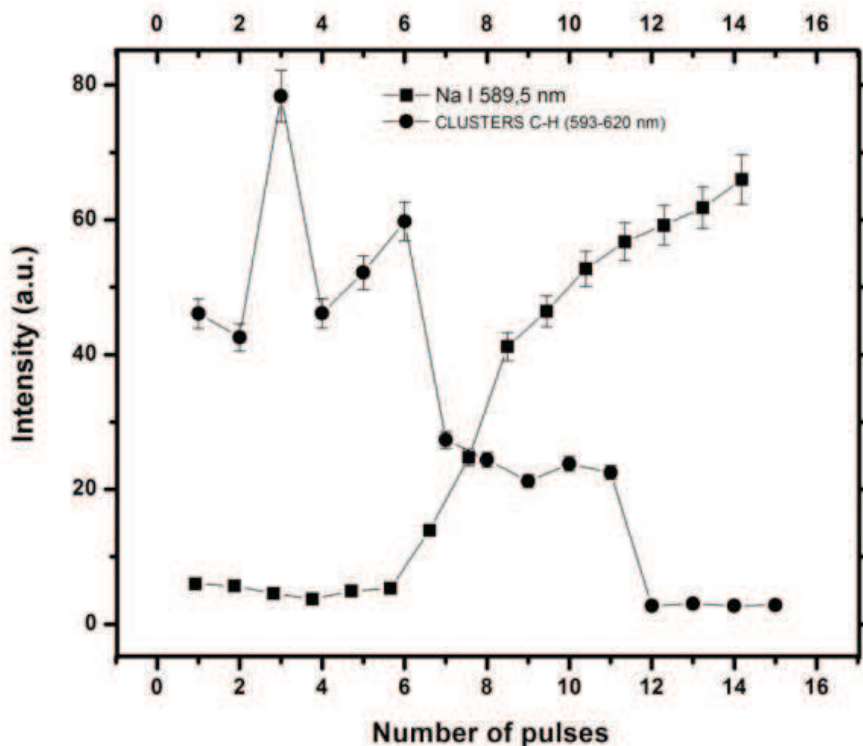


Fig. 6. Intensity profile curves Na I (589.5 nm) y emission band of the C-H clusters

several stages combustion-ablation: While the first pulse prepare the surface making it darker by combustion, the second pulse irradiate a surface with strong absorption and ablate it at very high rate.

3.4 Field applications: Bust mode Q:Switched excitation for LIBS analysis of art metal object

LIBS offers a potential alternative to other techniques used in art conservation, works of art, archaeology, etc., being a rapid elemental analysis technique, performed in situ and virtually non-destructive (Fotakis 2006). A LIBS's particularly interesting application is the analysis of metal objects with different compositional layers, particularly when trying to get in-depth composition profiles to distinguish the different elements present in a metal object and can develop a proper restoration process, among other uses, see Fig. 7a. Thus, the determination of the elemental composition, so far unknown, of the different layers present in a 20th Century Metal Jug, of Japanese origin, belonging to a private collection and the only one of its kind (see Figure 7b), is performed using a prototype LIBS equipment. For this purpose, successive burst mode laser pulses are applied to a well-localized area of the sample. The determination of the components avoid to the better selection of the restoration method to be used.

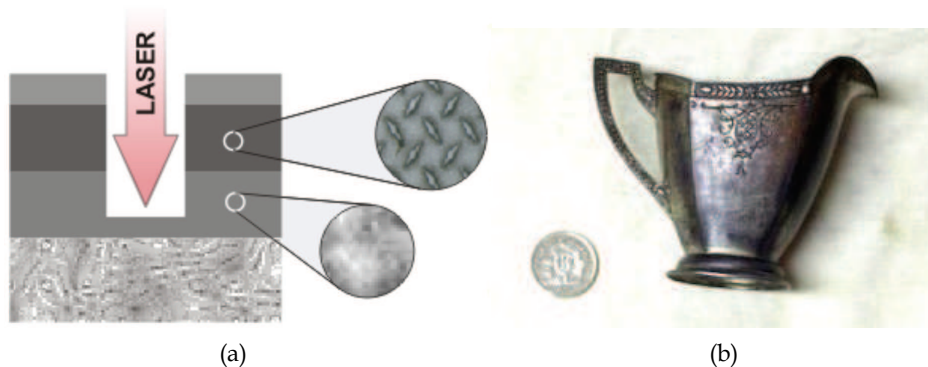


Fig. 7. (a) An schematic of in-depth laser microanalysis performed by LIBS technique, (b) The picture of Japanese Jug.

A wide spectral window was selected, covering approximately 450 nm and that was centered at 575 nm, so that any elements contained in the metal object, including those elements that can arise by the influence of environmental factors such as Ca, Mg, Si, Al, Na and K, may be measured simultaneously.

The Jug was analyzed superficially in four different and distant points to determine the surface composition and ensure the minimum possible sampling and thereby minimize damage as a result of ablation. The normalized characteristic spectrum for each of the samples is shown in the Figure 8. As shown, certain elements are represented in each of the measurements performed. The Cu constitutes the fundamental component and in turn the one that shows major proportion in relation to the other materials. Other elements were found to a lesser extent such as Ca, Si, Na and K, including marked relative differences between their intensities, showing changes in their proportions. These elements are attributed to deposition of dirt and impurities on the surface of the sample. Another element

determined is the Pb which, similarly to the other aforementioned elements, has lower intensity values as compared to Cu. Finally, O and N were determined essentially using the same behavior in all measurements, and their presence is due to mainly the combustion process during the laser-matter interaction with the atmosphere, among others. Each time a laser pulse is applied on the surface of the Jug, a portion of the material that makes up the surface is removed and plasma is generated. If the laser is applied all the time in one place, it will penetrate the material (see Fig. 7a). In order to obtain the composition profile and identify different elements and layers present in the Jug, a multispectral analysis of the emission of the plasma generated by each laser shot was performed. The procedure to determine each layer consisted of recording pulse-to-pulse variations in the intensity of emission lines characteristics of maximum amplitude of the obtained constituent elements of the surface: Ca (II) 392.83 nm, Pb (I) 405.56 nm, Cu (I) 521.24 nm, Si (II) 546.04 nm, Na (I) 588.67 nm and K (I) 765.93 nm. The intensity of these elements depending on the number of pulses is shown in the Fig. 9.

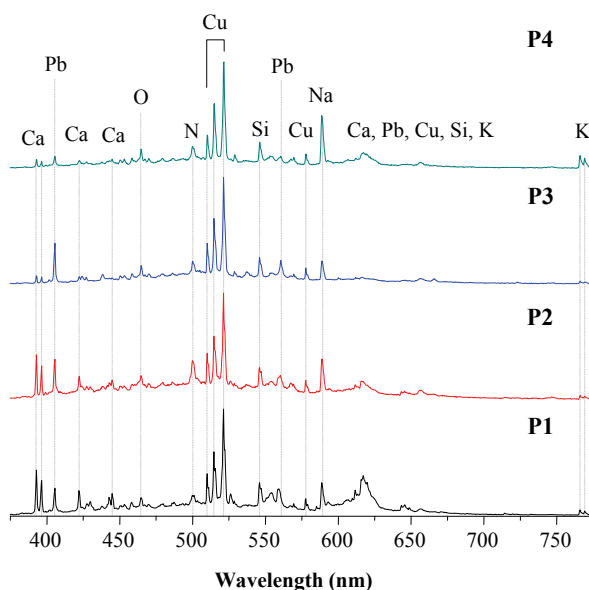


Fig. 8. LIBS spectra obtained in four different zones of metal Jug.

By increasing the number of pulses (greater penetration in the sample), the elements Ca, Si, Na, and K had a decreasing behavior. The little variation of intensity from pulse 4 indicates that the elements have virtually disappeared. The lack of a zero value is justified by the presence of background signal, which will have a greater or lesser value depending on the spectral region in which they are found. Low levels in the intensities of these elements, compared to Cu, involve small concentration values in the object. This supports the idea of the presence of elements resulting from the deposition of dirt and impurities on the Jug's surface. Metal objects often show heterogeneity and surface oxidation caused by heat treatment during construction and environmental degradation. These factors can cause variation in the morphology and elemental composition of ancient metal alloys, which results in pollution of surface and global object.

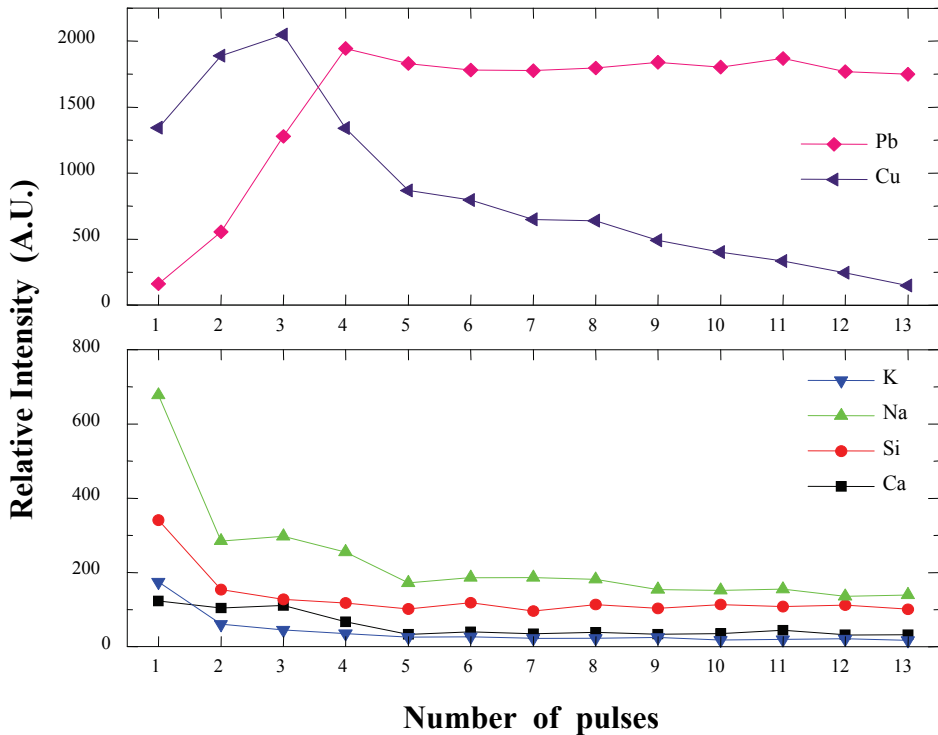


Fig. 9. In-depth profiles for Ca(II) 392.83 nm, Pb(I) 405.56 nm, Cu(I) 521.24 nm, Si(II) 546.04 nm, Na(I) 588.67 nm and K(I) 765.93 nm as function of number of pulses.

The Cu, during the first pulses (pulse 1 - pulse 3), shows increasingly higher intensity values, constituting the main element on the jug's surface (pulse 1) and, turn on, a small interior portion of the jug. The Pb, initially (pulse 1) at very small intensity levels, has a linear increase until coming to its maximum value after pulse 4. After this pulse, the intensity of Cu starts to decrease until reaching a practically zero value. On the other hand, the Pb maintains a stable behavior during the following pulses. After pulse 13, virtually all elements have disappeared being the Pb the majority element in the jug's composition. Figure 10 shows the superposition of the Jug's spectrum, after pulse 13, with a spectrum pattern of Pb showing a perfect match between these spectrums. It is, therefore, evident the presence of two phases or layers in the Jug: phase 1 with Cu as the majority element, and phase 2 for Pb.

On the other hand, the Cu-Pb interface, through the behavior between the intensity ratios of Cu/Pb and Pb, precisely on the pulse 4 is observed clearly in the Figure 11.

Precisely, the Pb reaches its maximum value and the ratio Cu/Pb becomes lesser than 1 which, from the following pulses, tends linearly to zero. While the existence of two layers in the depth profile of the Jug was determined, from Figure 11 can be seen that the Cu layer, in addition to Ca, Si, Na and K, there is the presence of Pb. This result comes from the thickness of Cu layer and the laser energy. While energy was selected in an optimal work area; nonetheless, given the thickness of Cu layer, this energy level remains relatively

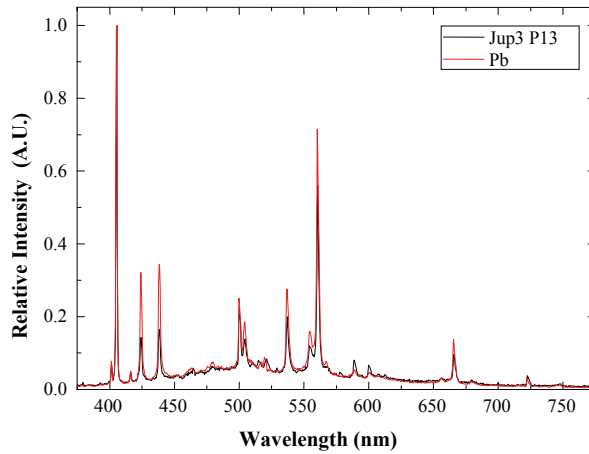


Fig. 10. Comparison between the spectra of metal Jug and Pb standard.

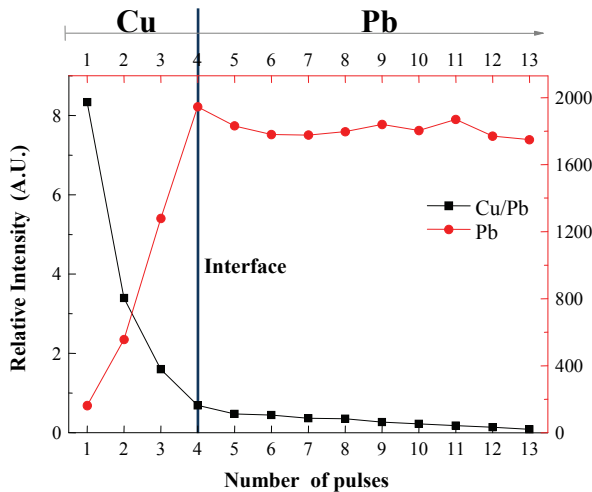


Fig. 11. In-depth behavior of Cu/Pb rate and Pb relative intensity.

sufficient to interact with the next layer (Pb) and generate also laser ablation. Similarly, the presence of Cu in the Pb layer can be seen in the Figure 9. In this case, the ratio is given in the form of how laser energy is distributed. The distribution of energy of the pulse laser is not exactly homogeneous (at the edges it is lower than in the center); so the amount of material removed from the irradiated region will be lower in edges than in the center, so that debris of the previous layer may be always remained as it moves in depth.²⁰ From the crater produced by 13 laser pulses in interaction with the jug, the crater’s wall can be seen, see Figure 12.

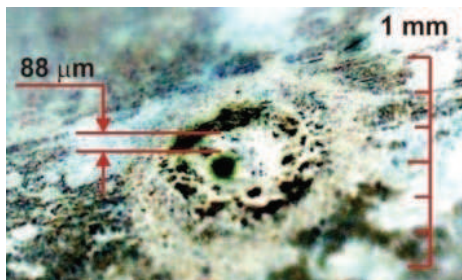


Fig. 12. Contrast image of laser treated zone captured by optical microscopy (magnification 60x).

Referencing the image scale, the approximate depth of the crater can be determined. Therefore, knowing the depth of the crater and the number of pulses, it is possible to determine how much material is extracted in every interaction of laser with the Jug and, in turn, determine the thickness of Cu layer. Making the necessary calculations, we find that the thickness of the crater's wall is approximately $88\ \mu\text{m}$, which implies, for a total of 13 laser pulses, a penetration factor of $6.8\ \mu\text{m}/\text{pulse}$. Therefore, as estimated, the thickness of Cu layer is $27\ \mu\text{m}$ knowing that it ends after pulse 4.

7. Conclusions

Recent technological advances are leading to the development of fully field-portable LIBS systems as show MicroLIBS, the compact, portable and low cost LIBS device developed by IMRE-Havana University. The use of Cr:YAG crystal as Q:Switch element allows simplifying the design and reducing costs as compared to electro-optical Q:Switch and also to guarantee more duration that LiF elements. The application of pulse trains as an excitation source helps to achieve a higher intensity of plasma emission and a substantial improvement in the signal-to-noise ratio compared with single-pulse systems.

The advances in modification of laser parameters open new applications no limited to elemental determination but also covering the laser material processing on-line monitoring or the simulation of specific plasma events in wide range of energies or pulse durations.

Regarding the fields applications, two examples were given: On one side, the real-time monitoring of de-thorning process for one food product, and, on the other side, the compositional characterization of heritage object.

For the first field application, the burst-mode excited LIBS technique is shown to be a suitable method for new industrial processes. Additionally, the approach of complex band emission pattern recognition can be used for determining of contamination problems in such vegetable food.

For the case of metal Jug analysis, the multi-pulse regime shows the possibilities of the technique for heritage objects characterization. The presence of key elements (Cu and Pb) was determined. They are distributed in layers, being the Cu the surface element with an estimated thickness of $27\ \mu\text{m}$. The elements Ca, Si, Na and K are impurities caused by dirt and the environment that influence the object's surface.

In resume, the multi-pulse excited LIBS technique serves as an in situ, low-cost and virtually non-destructive analytical tool, in the identification of elements and environmental monitoring or characterizing a wide spectrum of process and objects.

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