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TOPICAL REVIEW

Laser ablation assisted spark induced breakdown spectroscopy and its application

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Recently, laser ablation assisted spark induced breakdown spectroscopy (LA-SIBS) has been growing rapidly and continue to be extended to a broad range of materials analysis. Characterized by employing a specifically designed high voltage and pulse discharge circuit to generate a spark and used to enhance plasma emission produced by laser ablation, allows direct analysis of materials without prior sample preparation. This paper reviews recent development and application of laser ablation assisted spark induced breakdown spectroscopy for material analysis. Following a summary of fundamentals and instrumentation of the LA-SIBS analytical technique, the development and applications of laser ablation assisted spark induced breakdown spectroscopy for the analysis of conducting materials and insulating materials is described.

Keywords laser induced breakdown spectroscopy, laser ablation assisted spark induced breakdown spectroscopy, discharge circuit charactor

Contents

1 Introduction

Using a spark discharge to enhance laser induced plasma emission, firstly pronounced by Brech and Cross in 1962 [1] and described in detail by Rasberry [2] in 1967, is a plasma-based atomic emission analytical technique, which can be regarded as a "marriage" of two other pulsed plasma techniques for elemental analysis: traditional spark discharge optical emission spectroscopy (SD-OES) [3] and laser-induced breakdown spectroscopy (LIBS) [4, 5]. In LIBS technique, a tightly focused pulse laser is used to ablate material and generates plasma. LIBS is a recognized laser detection technique for analyzing a wide range of materials in the lab and in field applications, including environmental monitoring, industrial processing by materials analysis, biomedical studies, military and safety needs, space exploration, and artwork analysis, etc. [6, 7]. In traditional spark discharge optical emission spectroscopy (SD-OES) the signal detection and spectroscopy is similar to LIBS, however, the plasma is formed electrically [8]. The sample itself acts as the cathode, and an anode is brought very near to the surface. An electrical discharge is formed between the electrode and the sample and ablates some material while simultaneously creating plasma. It is an established technique and generally performed upon conductive samples (metals) and currently is predominantly used in alloy analysis.

Both laser-induced breakdown spectroscopy (LIBS) and electric spark discharge optical emission spectroscopy (SD-OES) had been used for detect most chemical elements with a limits of detection of about $\sim$ ppm range [3, 9, 10]. Dual pulse laser induced breakdown spectroscopy (DP-LIBS) technique has been established and proved to be a promising technique to enhance the plasma emission intensity and improve the analytical performance of LIBS [7, 11–13]. In DP-LIBS, an additional laser pulse was used as a secondary excitation source of first laser plasma, and it is especially helpful for increasing the plasma emission intensity and improving the elemental analysis sensitivity.

Based on the concept of secondary excitation by spark discharge in Brech *et al.*'s work [1] and laser triggered spark discharge enhance laser induced breakdown spectroscopy (SD-LIBS) developed by Nassef *et al.* in 2005 [14], we recently described an electric circuit which can produce a nanosecond or a microsecond discharge spark and used as a secondary excitation source to reheating the laser plasma, the laser ablation-spark induced breakdown spectroscopy (LA-SIBS) or laser ablation fast pulse discharge plasma spectroscopy (LA-FPDPS) [14–16]. In analogous to DP-LIBS, laser ablation assisted spark induced breakdown spectroscopy (LA-SIBS) has demonstrated a significant improvement in LIBS sensitivity [15, 17] and its spatial resolution for microanalysis [16], but using a spark discharge plasma generation instead of the second laser beam. In this brief review, the basic hardware required, pulse discharge process and plasma generation to perform laser ablation assisted spark induced breakdown spectroscopy is described. The development history and the typical analytical application of laser ablation assisted spark induced breakdown spectroscopy will be summarized.

2 Overview of LA-SIBS (Principle of LA-SIBS)

Secondary excitation by applying a discharge spark after laser ablation allowed an increase of the LIBS signal intensity and overall quality [14, 16, 18]. Different geometries of the electrode positions relative to sample surface and to the incidence of the laser ablation pulse have been used in LA-SIBS. The most common used configuration is shown in Fig. 1. The electrodes are placed 1–3 mm above the sample horizontally [14, 15] or in the shape of "V" [2], and the electric field was applied perpendicularly to the incident laser beam. For conductive samples (metals) analysis, the metal sample usually serves as the cathode and only need an additional anode [16, 19–21] similar to that in traditional spark discharge optical emission spectroscopy [8]. The electrodes are commonly made of cylindrical high purity tungsten rods with a diameter of a few mm and in a hemisphere or conic shaped tip. The gap between the anode and cathode is usually set at about 2–6 mm, dependent on the discharge voltage preselected. The discharge current was usually in a damped oscillating [14, 15] or quasi squared wave [20, 22]. Based on the electronic circuit design and discharge trigger mode, there are two typical laser ablation assisted spark induced breakdown spectroscopy. Laser ablation spark induced breakdown spectrum (LA-SIBS) and laser ignition assisted spark-induced breakdown spectroscopy (LI-SIBS) [2, 16]. In LA-SIBS the discharge spark is initiated automatically between electrodes connected to a pre-charged high voltage capacitor by laser plasma. In LI-SIBS, the initiation of discharge spark can be actively controlled by using a trigger pulse to switch on a HV pulse power supply connected directly to the electrodes.

2.1 Laser ablation spark induced breakdown spectrum

Figure 1 is the schematic diagram of the laser ablation spark induced breakdown spectroscopy (LA-SIBS) [15] or spark discharge laser induced breakdown spectroscopy (SD-LIBS) [14], alternatively. In principle, the discharge circuit and plasma generation process in LA-SIBS and/or LA-FPDPS is almost identical to that of SD-LIBS, excepted that a current limiting resistor is replaced by a diode to prevent the reverse current. In addition, the capacitance used in the circuit is quite difference between the LA-SIBS and SD-LIBS, leading to a different discharge character, plasma emission enhancement and sampling process.

The main discharge circuit is composed of a high voltage DC power supply, high voltage capacitance, and a pair of electrodes for both LA-SIBS and SD-LIBS. In LA-SIBS/SD-LIBS technique, the electric energy is initially stored in the capacitor with a preselected high voltage, a laser pulse triggers an electric spark between two metal electrodes and a sample, usually in a "V" shaped spark, and the excited plasma is analyzed by OES. Without a laser pulse the spark is suppressed as the applied electric field strength is below the self-breakdown threshold in air. The electrons and ions in the laser plasma serve as a preionization source and grow exponentially due to an electron avalanche process [23]. The laser-triggered discharge

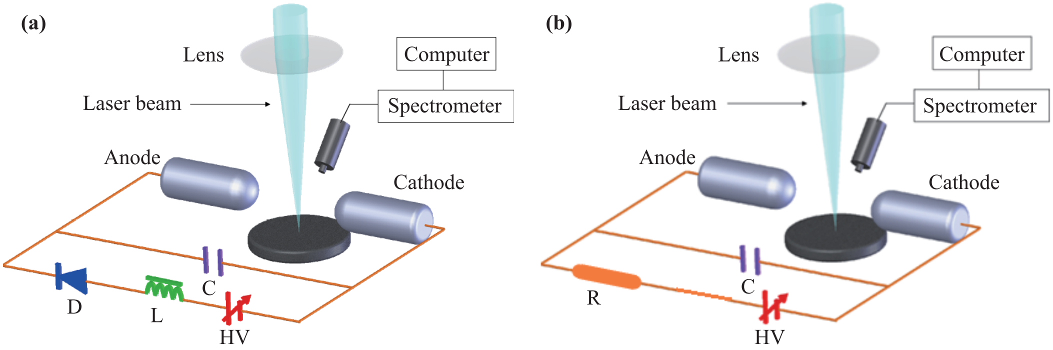

Fig. 1 Schematic diagram of LA-SIBS **(a)** and SD-LIBS **(b)** system. C, capacitor; HV, high voltage DC power supply; D, diode; L, distributed inductance; R, resistor.

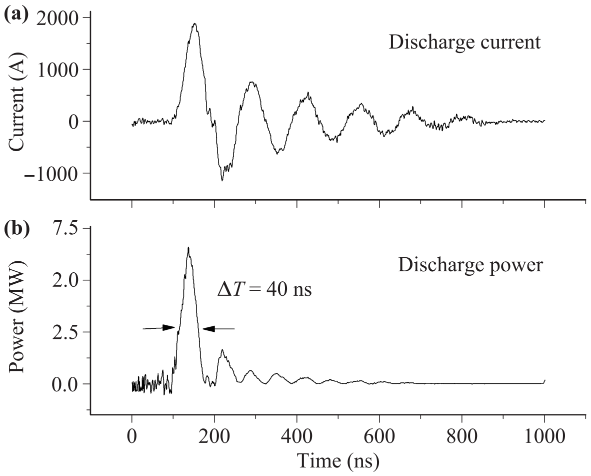

Fig. 2 The typical temporal profile of the discharge current **(a)** and discharge power **(b)**.

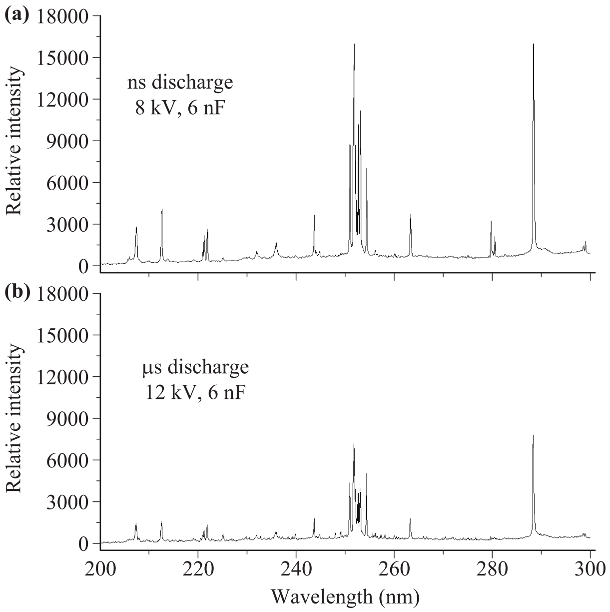

Fig. 3 Optical emission spectra of silicon plasma of LA-SIBS with nanosecond discharge technique **(a)**, microsecond discharge technique **(b)**.

is spatially confined to a plasma channel between two electrode tips and ablated spot on the sample. The expanded laser plume is then intercepted and heated by a discharge spark. In LA-SIBS and/or LA-FPDPS, a low inductance capacitor with a capacitance of a few nF is used and only a smaller electronic energy is needed in LA-FPDPS [17]. Figure 2 is the typical temporal profile of the discharge current, and electric power deposited into the plasma in LA-SIBS [17]. A damped oscillating discharge current was observed. And an intense main discharge peak power lasting for a shorter period of nanosecond range (FWHM) is obtained. An obvious advantage of the nanosecond discharge is that the line intensity of plasma emission was greatly increased along with an improved signal-to-noise ratio, in comparison with that using a microsecond pulse discharge circuit [24], as shown in Fig. 3. In addition, as the discharge energy in LA-SIBS/ LA-FPDPS is quite small, there is no obvious difference between the crators formed in LA-FPDPS and in LIBS with the same laser energy [18, 25, 26], indicating that only plasma reheating attributes to plasma emission enhancement in LA-SIBS. This character also will benefit applications where high analytical sensitively but low sample damage is require, such as high resolution chemical mapping, the minimally destructive analysis, depth profiling and cultural relic analysis.

If a capacitance of ∼ μF was used as in SD-LIBS [14], the period of oscillation discharge current will be larger than that in LA-SIBS [15]. And a main discharge peak power lasting for a longer time period of about 4–10 μs (FWHM) with a relative weak discharge peak intensity is observed, dependent on the energy stored in the capacitor, the electric circuit parameters, and the electrode geometry. The energy deposition rate to the plasma is smaller as well, leading to a weak signal enhancement [17, 24]. Furthermore, an electrical discharge is formed and generates a plasma while simultaneously ablating some material. A larger and deeper ablation crator on the sample surface was left compare to the ablation crator in LIBS with the same laser energy [14]. Therefore both plasma reheating and secondary sample ablation by spark discharge could attribute to plasma emission enhancement. In addition, high repetition rate (up to 30 kHz) laser [21, 27], specifically design discharge circuit including charged coaxial cable [22, 28] to generate a quasi square discharge wave, has been integrated into LA-SIBS technique.

2.2 Laser ignition assisted spark induced breakdown spectroscopy (LI-SIBS)

In LI-SIBS, the initiation of discharge spark is actively controlled by external trigger and switch on a HV pulse power supply [2, 16], in contract to that in LA-SIB, where the spark discharge is triggered by laser plasma. The schematic diagram for the LI-SIBS is shown in Fig. 4.

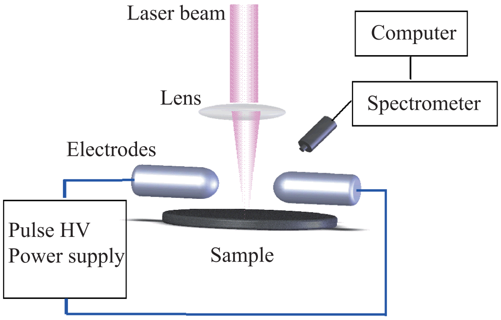

Fig. 4 Schematic diagram of LI-SIBS system.

The discharge circuit is mainly composed of a high voltage pulse power supply and a pair of electrodes. The switch of high-voltage pulse power supply was actively controlled by a TTL signal from a pulse delay generator and a high voltage pulse (usually in a quasi squared wave shape [20, 22]) was then delivered on to the electrodes, leading to spark induced plasma for analysis. The pulse laser, pulse power supply and the time delay between the HV pulse and laser pulse is controlled by pulse delay generator. Generally, the electrical discharge is formed between the electrode and the sample and forms a "V" shaped spark in LA-SIBS. To some extent, the "V" shaped discharge could ablate some sample and increase the diameters and depths of the craters [14]. Under suitable laser energy, configuration of electrodes, it was possible to change the V-shape discharge to a parallel discharge only between a pair of electrodes, if select a relatively lower discharge energy and long time delay between the HV pulse and laser pulse [29]. This selectable time delay ensures that the parallel discharge does not peel off the sample and ensure that the spark does not damage the spatial resolution of sample surface element analysis and possibly suitable for chemical mapping.

3 Applications

Several investigators have used laser ablation assisted spark induced breakdown spectroscopy (LA-SIBS/LI-SIBS) in a variety of samples and sample matrices [14–16, 19, 26, 30–32] with enhanced plasma emission and improved analytical sensitivity, signal to noise ratio and precision in terms of relative standard deviation. In this following section, the applications of LA-SIBA on the analysis of insulating materials and conducting materials are presented respectively.

3.1 Analysis of insulating materials

Although laser ablation assisted spark induced breakdown spectroscopy has been presented in the early 60^{th} of last century [2], and its electronics was quite similar to that in LI-SIBS described in Section 2.2. After that, the technology does not attract much attention, partially due to cost of the expensive laser at that time. With the development of laser technology and electronics, LA-SIBS have been widely used for trace elemental analysis in soil, fluorine and sulfur in metallurgical slag, phosphorous in fertilizer, and copper in plants, classification the origin of brown rice, and identify the neoplastic and nonneoplastic gastric tissues, and in some cases, with a modified discharge circuits [20]. In 2010, Zhou *et al.* developed a high-voltage, fast pulse discharge circuit and described how applying a carefully timed high voltage discharge could effectively reheat the laser-induced plasma thereby improving sensitivity, precision and S/N in the measurement of a wide range of major and trace elements in soil [15, 17, 25, 33] and Si lines in monocrystal silicon sample [24]. The electron temperature and electron number density of soil plasma generated by LA-SIBS at varying discharge voltages and capacitances have been studied [33]. The damping time constant, oscillation frequency, and intrinsical inductances of discharge circuits were estimated [34]. The technology has been used successfully to analysis trace elements in soil [35] in 2012, calibration curves for Pb, Mg, and Sn in soil were derived and the limits of detection were observed to be 1.5, 34, and 0.16 μg/g, respectively. In 2014, Hou *et al.* used LA-SIBS with cylindrical space confinement to study the coal samples [31]. By adjusting the expansion of plasma through space confinement, the stability of spark discharge was improved, and finally the plasma morphology was stabilized. In 2015, an electric arc with time decayed (∼ 1 ms FWHM), low-voltage (50 V) and high-current (peak value 250 A) was used by Eschlboeck-Fuchs *et al.* to enhance the laser plasma, and to analyze metallurgical slag [20]. The optical emission lasts up to a few milliseconds. The emission lines of fluorine and sulfur can be easily detected while that cannot be detected by conventional LIBS. In 2018, Vieira *et al.* used spark discharge assisted laser-induced breakdown spectroscopy to detect P element in fertilizer [36]. With a discharge voltage of 4.5 kV the obtained calibration curves showed correlation coefficient $\geq$0.993 and RSD $\leq$8%. In 2019, Seifarinezhad *et al.* evaluated the possibility of diagnosing gastric cancer by using LA-SIBS [37]. The normalized calcium (Ca) and magnesium (Mg) peaks of neoplastic and nonneoplastic gastric tissues could be viewed as a practical measure for tissue discrimination. In 2019, LA-SIBS has been used to designation of origin of brown rice by Pérez-Rodrígueza *et al.* [32]. A 84% of accuracy, 100% of sensitivity and 78% of specificity in classification has obtained. The LA-SIBS has also been used for determine the copper in onion leaves by Jabbar *et al.* in 2019, the limit of detection of copper in the samples has been improved to be 0.028 ppm [38].

3.2 Analysis of conducting materials

Except for these investigations on insulating materials, LA-SIBS has also been widely used for analysis of conducting materials, including aluminum alloy, copper alloy, some metals and stainless still. The effect of ambient gas on the performance of LA-SIBS was investigated [39, 40]. In 2005, Nassef *et al.*developed a laser trigged spark technique to reheating the laser induced plasma [14] and to study the laser-induced breakdown spectrum of Al and Cu targets. The experimental results show that with the increase of applied voltage, the intensity of Al 358.56 nm line increases, and with the a 3.5 kV spark discharge, the signal to background ratio increases 6 times. In 2010, Chen *et al.* used LI-SIBS technique to analyze trace mercury ions in aqueous solutions [16]. The trace mercury ions in aqueous solutions were electrically deposited on the surface of a high purity copper plate. A 30 times analysis sensitivity of mercury in LI-SIBS was obtained in comparison with that in laser-induced breakdown spectroscopy. 1n 2014, LA-SIBS was used by Ivković *et al.* to analyze aluminum alloy in ambient gas of air, argon and helium and at different pressures [39]. The influences of various capacitors and discharge voltages on enhancement of the studied spectral line intensities were also studied. In 2016, an aluminum plate was analyzed by Sobral *et al.* using a fast, high voltage square-shaped electrical pulse to enhance the laser ablated plasma spectrum [22]. Under the voltage of 12 kV, the atomic spectrum of Al was increased by 5 times, and the ion spectrum of Mg was increased by 15 times. An enhancement of up to one order of magnitude on the emission signal-to-noise ratio can be achieved. In 2018, a spark discharge was used to reheating high repetition rate (1 kHz) laser plasma, either nanosecond [21] or femtosecond [41] laser plasma by He *et al.* Chromium, manganese, magnesium and copper in aluminum alloys were analyzed with better analytical sensitivity and analytical reproducibility than that in LIBS with the same laser pulse energy. With a discharge spark to enhance a high repetition fiber laser plasma, a 3–9 fold improvement of the detection limits of Cr, Cu, Mn, Mg, and Zn in aluminum alloys [30] and Pb, Fe, and Al in copper alloy [27] were determined. In 2018, Stainless still was investigated using LA-SIBS under air and Ar gases respectively by Liu *et al.* [40]. The enhancement effect is more apparent in the presence of Ar ambient. In 2018, Robledo-Martinez *et al.* used a quasi square discharge pulse produced by the discharge of a charged coaxial cable to enhance laser plasma. A similar effect on signal enhancement was observed with the square wave discharge spark [28]. In 2018, a signal enhancement theoretical model of spark discharge assisted laser-induced breakdown spectral was proposed by Hassanimatin *et al.* The experimental study on the ablation amount, plasma volume, life and temperature of granite and aluminum samples was carried out, and the theoretical calculation model was in good agreement with the experimental results [26]. In addition, at different sample temperatures, the theoretical enhancement ratio was in good agreement with the experimental one as well in another report in 2019 of the same group [42].

4 Conclusion

There are a great number of researches investigating the application and on the modification of the configuration and discharge circuit of laser ablation assisted spark induced breakdown spectroscopy, especially in the near decade. In analogous to DP-LIBS, using a spark discharge plasma generation instead of the second laser beam, LA-SIBS has a significant improvement over LIBS in signal quality and analytical sensitivity. And it has been demonstrated that the LA-SIBS is a suitable and valuable technique in elemental analysis for both conducting materials and insulating materials. Due to the intrinsic instability of laser plasma and spark discharge, and complicated interactions of particle with light and electric field, the discharge character, enhancement on laser plasma emission and its analytical performance is restrictedly dependent on the experimental configuration of discharge circuit and sample, and most of these studies are still limited at the laboratory research stage. Some preliminary researches of LA-SIBS technology on the application of tumor diagnostic and food source identification were presented and the results initially proved the feasibility of this approach, but still leave a long way to real application. Therefore, further investigation on improving instrumentation, data analysis, optimization of laser and discharge parameters, and even mechanism of LA-SIBS should be carried on in the future.

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