

# Laser ablation initiated fast discharge for spectrochemical applications

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## Abstract

The results of an experimental study of the optical emission enhancement possibilities during the single pulse laser induced breakdown spectroscopy of the aluminum alloy are presented. This study is performed in air, argon and helium at different pressures with and without the additional fast electric discharge. The discharge was initiated by plasma plume created by laser ablation of target. The influences of various capacitors and discharge voltages on enhancement of the studied spectral line intensities were also studied. The application of the fast discharge through optical emission enhancement enables lowering of detection limits thus making this spectrochemical method comparable with the other analytical techniques.

**Keywords:** spectrochemistry, analytical spectrometry, laser induced breakdown spectroscopy, optical emission spectroscopy.

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The application of lasers for analytical purposes started only three years after their introduction [1,2]. The laser radiation with energies around 100 mJ and duration of several nanoseconds was focused on a sub-millimeter spot on a solid, liquid or gaseous sample inducing breakdown. Namely, the free electrons created through multi-photon ionization and inverse bremsstrahlung processes absorb energy from the laser pulse, colliding and freeing yet more electrons in an ionization cascade until thermally-hot, charge-neutral laser-induced plasma is produced. The spectral recordings of this plasma showed lines of all elements present in the sample. This fact opened the possibility for a new analytical technique – laser-induced breakdown spectroscopy – LIBS. The main advantages of LIBS are: 1) simplicity, 2) lack of sample preparation for the analysis of the gases, liquids and solids, 3) simultaneous multi-element detection, 4) ability to detect high and low *z* elements, 5) only optical access to the target is required and 6) standoff analysis capabilities [2–4]. The unique capability of the laser plasma to sample materials and standoff possibility of LIBS enabled their use to monitor chemical, biological, radiological, nuclear and explosive threats from the safe distance. Besides the applications of LIBS in hazardous environment usually characteristic for chemical industry, other applications like rapid characterization of polymer type, composition of paints and varnishes or detection of impurities in petroleum, cosmetic or other chemical products are also numerous [2–4].

However, the detection limit was poor in comparison with other laboratory analytical techniques. The main reasons are low intensity of the spectral lines and very intense continuum radiation, which significantly lower signal to background – *S/B* ratio. The much faster decay of the background emission versus line emission opened the possibility for improvement of *S/B* ratio using gated photomultipliers or CCD cameras to record spectra. The development of the intensified CCD cameras enabled (besides gating) further gains in the recorded signal. Unfortunately, techniques such as inductively coupled plasma-mass spectrometry (ICP-MS) were still favorable.

These LIBS disadvantages were mainly overcome by enhancing optical emission with the use of the additional laser pulse in different double pulse technique configurations – DP-LIBS [2–6]. The first pulse is used to ablate the target and produce the plasma plume, while the second pulse is used to reheat the plasma and thus create a stronger emission. Currently, DP-LIBS technique is prevalent in broad range of applications such as analysis of samples under water [7] or in Mars conditions [8], detection of traces of various explosives [9] and many others.

In this work, single laser pulse techniques are tested as cheaper alternatives (to double pulse LIBS technique) for enhancing optical emission. For that purpose we performed analysis at different pressures in argon and helium in addition to atmospheric air. The use of the additional fast discharge initiated with a same laser pulse was also studied.

## EXPERIMENTAL

Typical single pulse stand-off LIBS equipment consists of a Nd-YAG laser with focusing optics, light col-

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lection system (mostly fiber optics), detection system (like echelle spectrometer equipped with ICCD camera) and computer and electronics system for: a) synchronization of the laser, spectrometer and camera, b) detector gating and c) spectrum storage. In this experiment, see setup in Figure 1a, we used a Nd-YAG laser with energy of 50 mJ, pulse duration of 20 ns focused by use of a 5 cm diameter lens  $L_1$  having a focal length of 10 cm. The aluminum alloy target was positioned 2 mm in front of the focal point, so the 0.2 mm spot, *i.e.*, laser fluence of  $\approx 1.5 \text{ J/mm}^2$  was obtained. In order to perform the study in a controllable environment, the target was set inside a specially designed cross-shaped glass tube, see Figure 1b. The discharge part of the tube (having an internal diameter of 10 mm and tungsten electrodes 12 cm apart) was positioned perpendicular to the laser beam. This part of the tube has connectors for gas inlet and outlet and quartz windows for plasma observation. A laser beam entering through an upper glass window passes through the other branch of the tube and irradiates the target. A target surface was positioned in line with the bottom of the discharge tube, maximizing the recording volume of the plasma. In order to increase the optical signal reproducibility the target was rotated with a speed of  $\approx 2^\circ/\text{s}$ .

Appropriate gas pressure in the tube was established with the help of a vacuum pump and gas handling systems consisting of gas bottle, regulation valve, needle valve and manometer. The fast pulse discharge is driven by a set of capacitors – C, charged with high voltage power supply – HV PS fired by the same laser

pulse. By applying stabilized voltages up to 3 kV and using different C and current limiting resistor critically damped currents lasting several microseconds are realized.

A 1:1 plasma image was projected on the entrance slit of the 0.3 m imaging spectrometer Andor Schamrock 303, equipped with Andor ICCD iStar DH 720-18F-03 camera. The camera gating was performed by processing the Q switch signal from the laser with Stanford Research DG535 digital delay generator – DDG. The spectra were recorded at different delays between laser and current pulse (measured with Rogowski coil) and Tektronix TDS320 digital storage oscilloscope – DSO.

## RESULTS

The measurements in atmospheric air were used as reference data for comparing optical signal intensities in different experimental conditions. This measurement was conducted without the use of air flow, additional fast discharge and even without use of the end windows at the discharge part of the tube. Consequently, the conditions similar to the ordinary single pulse LIBS were obtained. The spectra emitted from the layer 1.5 mm apart from the target surface was recorded with an input slit width of 10 micrometers and camera full vertical binning – FVB. Thus, spatially integrated intensities, along this most intense part of the plasma plume, were recorded. In order to obtain the best signal to noise ratio, all spectra recordings

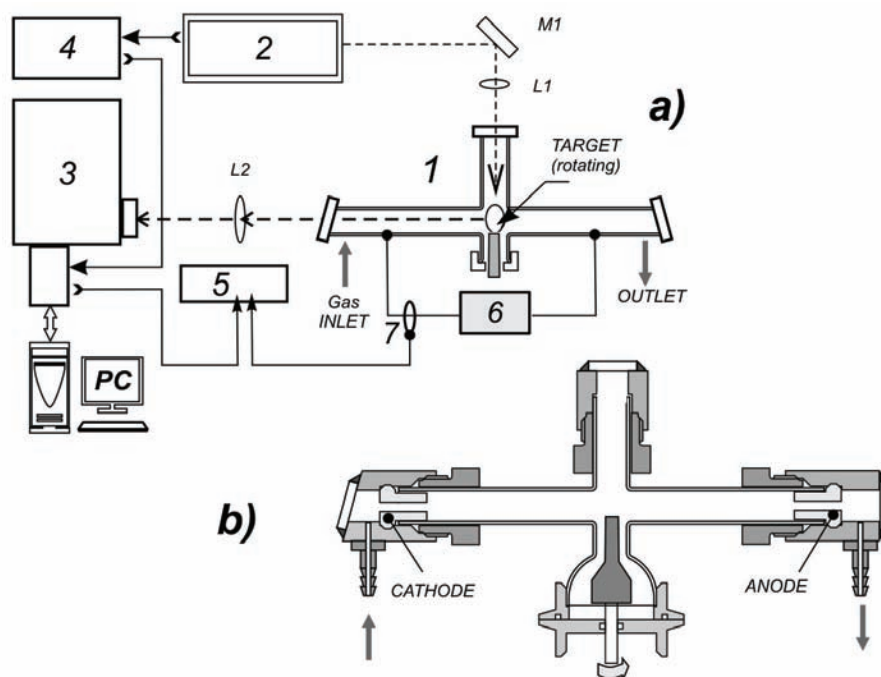


Figure 1. a) Experimental setup and b) detailed view of the glass tube (1) for laser-target interaction studies. 2 – Nd-YAG laser, 3 – imaging spectrometer, 4 – digital delay generator, 5 – digital storage oscilloscope, 6 – high voltage power supply, 7 – Rogowski coil.

(from 300 to 800 nm) were recorded with a delay of a 600 ns after a laser pulse with a gate of 8  $\mu$ s with the accumulation of the ten pulses. Such gating is a standard LIBS procedure which prevents the recording of pronounced continuum radiation in first 600 ns and accumulation of optical or electrical noise after 8.6  $\mu$ s in the studied case, *i.e.*, after the moment when the optical signal falls below a few % from their maximum value.

### The effect of the surrounding atmosphere on optical signal intensity

The study of the surrounding atmosphere influence on the optical signal intensity spectra in argon and helium at 30 mbar was performed under the same experimental conditions. The part of the spectrum, presented in Figure 2a, clearly shows that the observed optical signals were higher for Ar and lower for He gas, compared to the signal obtained in an air atmosphere.

The enhancement was attributed to the lower ionization potential of Ar leading to the higher electron number density, as observed earlier by testing at atmospheric pressures [10]. The spectra in argon were also recorded at different pressures: 1, 5, 10, 20, 30, 50, 100, 150, 200, 300, 400, 700 and 1000 mbar. It is shown that the signal enhancement changes with the gas pressure, see Figure 2b. The optimum argon pressure in our case is 150 mbar. The enhancement is the same, not only for the most pronounced aluminum lines, but also for the lines of the different constituents of plasma. This is illustrated in Figure 2, where part of the spectra with characteristic lines of the aluminum, copper and magnesium are presented. Such enhancement is of fundamental importance for a more precise determination of alloy composition (in studied case), detection of impurity traces in pure metals or determination of various samples composition.

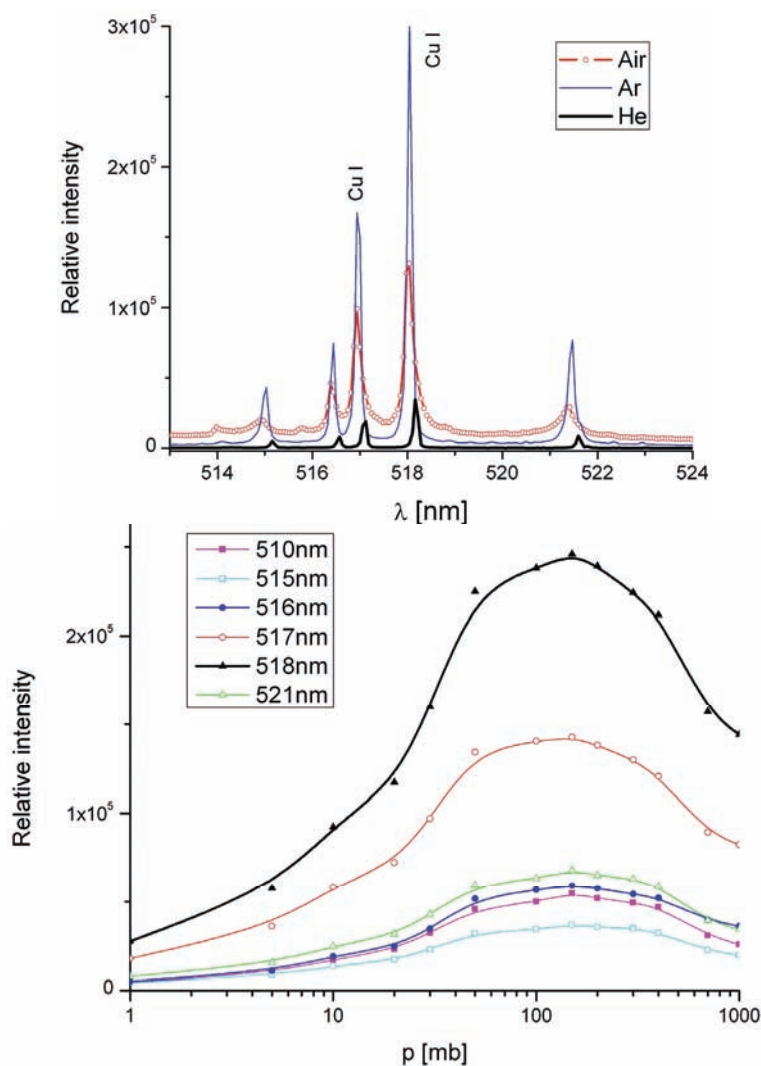


Figure 2. a) Part of the spectrum emitted from Al alloy target in air at 1 bar and in Ar or He at pressure of 30 mbar; b) relative line intensities for different argon pressures.

### The effect of additional fast electric discharge – FED on signal intensity

The investigations of the optical signal enhancement by the use of the additional fast discharge, earlier performed only in air at atmospheric pressure [11] were broadened to studies in air, Ar and He at different pressures. In order to find the conditions for the enhancement of analytical line intensities, it is important to ensure that fast pulse discharge will be initiated by laser pulse after a minimum delay. This condition arises from the requirement that most of the evaporated target material will still be present inside the observation volume during the establishing of the discharge. Since the propagation of laser produced aluminum plasma is very fast, see, *e.g.*, [12], this delay time must be as small as possible. The duration of the evaporated material presence between the electrodes was first increased by lowering the flow rate of the surrounding gas by use of a needle valve inserted between the discharge tube and the vacuum pump. Moreover, the delay between the laser and the current pulse decreases with the increase of the applied voltage, which is limited by self-breakdown. Hence the necessity to determine the dependence of the self-breakdown voltage on pressure of used gases for our inter-electrode distance, *i.e.*, so-called Paschen curves, exists, see Table 1.

The initial study with argon at a pressure of 30 mbar and with a capacitor  $C = 14.5 \mu\text{F}$  charged up to  $U = 2 \text{ kV}$  produced a significant but suboptimal increase of the line intensities, see Figure 2a. Although this voltage is almost half of the breakdown voltage (see Table 1), the delay between laser and current pulse is still smaller than the residence time of the target material in the discharge tube. Presence of target material during discharge is verified by measuring time evaluations of the emitted spectra. Measurements were performed with a fixed gate width of  $0.2 \mu\text{s}$ , at different delay times with a variable step:  $0.6 \mu\text{s}$ ;  $1\text{--}10 \mu\text{s}$  (step =  $1 \mu\text{s}$ );  $10\text{--}50 \mu\text{s}$  (step =  $5 \mu\text{s}$ );  $50\text{--}100 \mu\text{s}$  (step =  $10 \mu\text{s}$ ). Special attention was devoted to the characteristic lines of various plasma constituents: Mg I ( $\lambda = 383 \text{ nm}$ ); Cu I ( $\lambda = 515 \text{ nm}$ ); hydrogen  $\text{H}_\alpha$  ( $\lambda = 656 \text{ nm}$ ); Al II ( $\lambda = 705 \text{ nm}$ ); and in II diffraction order Mg I ( $\lambda = 766 \text{ nm}$ ); Al I ( $\lambda = 790 \text{ nm}$ ).

In order to confirm the value of the spectral line intensity enhancement induced by the application of high voltage only, the comparison was performed by recording spectra at the same Ar gas pressure with two

different gate widths. The first one corresponds to the time period before starting the discharge, while the other one includes variable times after starting FED. In the case when  $C = 14.5 \mu\text{F}$  and  $U = 2 \text{ kV}$  the gates are  $4$  and  $8 \mu\text{s}$ , respectively. In both cases we used a delay of  $600 \text{ ns}$ . These measurements, see Figure 3a, showed that the number of the spectral lines and their intensities are significantly increased. The reasons are more pronounced excitation of the Ar, as well as elements present in the target, glass tube and electrodes. Multiple ionization of all elements also occurred. With such a rich spectra it is difficult to resolve analytical lines due to overlap with other lines. This fact limits the application of these spectra for precise detection of alloy composition. Here, we must mention that all measurements are performed in the wavelength range  $350\text{--}800 \text{ nm}$ , due to spectrometer limitations. However, the appearance and intensities of the ion lines in the studied range are a strong indication that the use of the ion lines below  $300 \text{ nm}$  or above  $800 \text{ nm}$  under this conditions can be the same or even favourable compared to the other spectroscopic techniques, as also stated in description of LA-FED applications in atmospheric air for rapid detection of carbon in soils [13]. So, conclusion of inapplicability of these LA-FED conditions must be restricted only to the visible part of the spectra. In order to enable the application of LA-FED in the visible part of the spectra it was necessary to reduce the energy dissipated to the discharge. The first attempt of reducing the energy was by lowering the voltage, which caused the start of the discharge with significant and variable delay after the laser pulse *i.e.* pronounced non-reproducibility. Another attempt, to use a small capacitor of  $0.1 \mu\text{F}$  lead to the establishment of the glow discharge due to the low RC constant of the charging circuit. Namely, after a first initiation of the discharge by a laser pulse, the capacitor was so rapidly recharged that transition to glow occurred. All spectral lines were very weak and synchronization of the camera with a laser pulse, *i.e.*, evaporation of the target material was impossible. On the contrary, the charging of a slightly bigger capacitor  $C = 0.33 \mu\text{F}$  up to  $2 \text{ kV}$  created a discharge with a delay of  $20 \mu\text{s}$ . The spectral lines recorded are broader, but with almost the same peak intensity, see Figure 3b. These lines are superimposed on a very pronounced background even tens microseconds after the laser pulse. This significantly reduces signal to noise ratio and makes it inadequate for analytical applications.

Table 1. Measured dependence of breakdown voltage ( $U / \text{V}$ ) in Ar and He

Atmosphere	$p / \text{mbar}$											
	0.75	1.5	2.25	3	4.5	6	7.5	11.25	15	22.5	30	37.5
Ar	600	950	1050	1200	1400	1600	1800	1950	2300	2700	3400	3800
He	2400	1000	600	800	950	1000	1050	1100	1250	1500	1650	1850

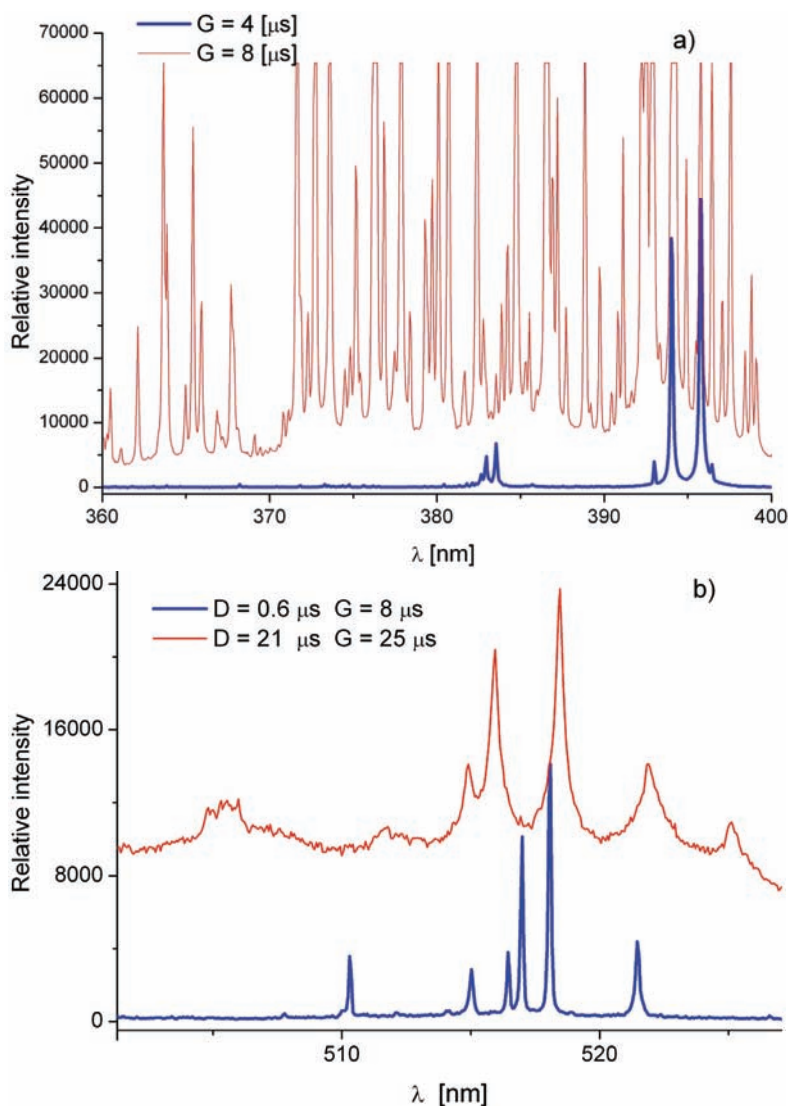


Figure 3. Example of the comparison of optical signals in 30 mbar of Ar with  $U = 2$  kV at different delays –  $D$  and gates –  $G$ ; a)  $D = 600$  nm,  $C = 14.5$  μF and b)  $C = 0.33$  μF.

A series of measurements in air at a pressure of 10 mbars were also conducted, but the results were the same or worse than in argon. Namely, the obtained spectra are even more complicated, with lots of molecular band overlapping with the spectral lines of the target constituents.

In order to reduce the number of interfering spectral lines the investigation was extended to the use of the helium at pressure of 30 mbar. This pressure was chosen in order to facilitate comparison with measurements in argon. Given the influence of the voltage on the delay between the laser pulse and the discharge and the dependence of the self-breakdown voltage on pressure presented in Table 1, the capacitor voltage ( $C = 0.33$  μF) did not exceed 1 kV. So, measurements of the spectra were performed in the same way as in argon but with different  $D$  and  $G$  values. Namely, the spectra characteristics for the ordinary SP-LIBS disap-

pear 20 μs after a laser pulse, while after a 45 μs discharge in helium with the evaporated target material started.

Comparison of the spectra presented in Figure 4 shows that line intensities with application of fast discharge are more than two times greater than in SP-LIBS for the same gate  $G = 20$  μs. Also, the prolonged duration of the plasma with discharge enables use of a larger gate width. Under these conditions, the order of magnitude increase of the line intensities is presented in Figure 4.

The enhancement of signals, shown in Figure 4, for He at 30 mbar and  $C = 0.33$  μF, may be attributed to the increase of plasma length as well as to its prolonged duration. It should be stressed that significant enlargement of the radiation volume in the other direction also exists, whenever voltage is applied. It is clearly visible with the naked eye that the discharge fills

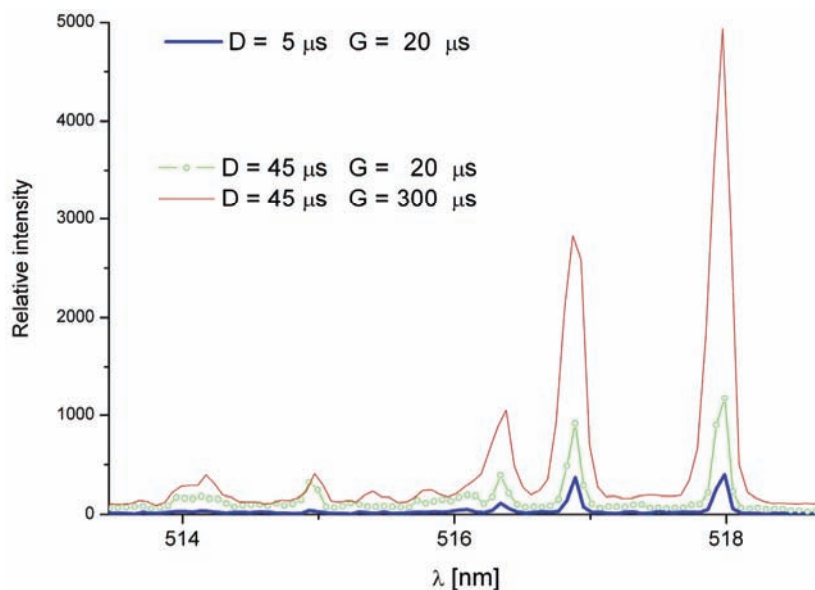


Figure 4. Example of the comparison of optical signals in He at  $p = 30$  mbar,  $C = 0.33 \mu\text{F}$  and  $U = 1$  kV at different delays –  $D$  and gate widths –  $G$ .

the whole discharge tube, while without applying voltage there is only a small plasma plume in front of the target. As stated before, all comparisons are performed for measurements at 1.5 mm with a slit width of  $10 \mu\text{m}$ . This plasma volume increase additionally enhances the spectral line intensities in comparison to SP-LIBS, thus making this method comparable to other analytical techniques.

## SUMMARY AND CONCLUSION

A laser initiated fast discharge, as an alternative method for enhancing the line intensities in a single pulse laser induced breakdown spectroscopy for the spectrochemical applications, was studied. Studies in various surrounding atmospheres with and without application of the fast electric discharge were enabled by constructing a cross shaped discharge tube with a 10 mm inner diameter and tungsten electrodes positioned 12 cm apart, see Figure 1b. The investigations were performed by measuring the time evaluation of the spectra from 350 to 800 nm with different delays and different gate widths. In all studied cases, the spectra were recorded by an imaging spectrometer equipped with a ICCD camera, only in the periods when a pronounced continuum radiation extinguish. The special attention was devoted to the recording of the characteristic lines of various elements present in targets like Mg I ( $\lambda = 383$  nm); Cu I ( $\lambda = 515$  nm); hydrogen  $H_{\alpha}$  ( $\lambda = 656$  nm); Al II ( $\lambda = 705$  nm); and in II diffraction order Mg I ( $\lambda = 766$  nm); Al I ( $\lambda = 790$  nm).

The measurements in different surrounding atmospheres without applying any voltage clearly show that the observed optical signals were higher for Ar and

lower for He gas, compared to the signal obtained in an atmospheric air, see Figure 2a. The enhancement was attributed to the lower ionization potential of Ar leading to the higher electron number density, as observed earlier by testing at atmospheric pressures [10]. It is shown that the signal enhancement changes with the gas pressures, see Figure 2b. The optimum argon pressure in our case is 150 mbar.

Spectral line intensities enhancement with applications of the fast electrical discharge initiated by the same laser pulse was studied by charging capacitors of 14.5; 0.33 and  $0.1 \mu\text{F}$  to different voltages between 1 and 3 kV. The voltage was determined from conditions: a) to be lower than self-breakdown voltage whose dependence on gas type and pressure was previously determined and b) high enough to minimize delay between the laser pulse and discharge starting. The second condition arises from the requirement that the most of evaporated target material would still be present within the observation volume during establishing of the discharge. The capacitance, gas type and pressure were determined from conditions: a) prevention of glow discharge development after laser initiation, b) lowering energy losses, due to the multiple ionizations of elements present in plasma, c) lowering of the background caused by overlapping of broad closely spaced spectral lines and, of course, d) enhancement of signal/background ratio by realization of more intense, longer lasting and larger volume plasma. It is shown (see Figure 3a) that discharging of the  $14.5 \mu\text{F}$  capacitor previously charged up to 2 kV in 30 mbar of argon leads to intense spectra inappropriate for analytical application. This conclusion holds only for the recorded part of the spectra between 350 and 800 nm, while obser-

vation and intensities of the ion lines range are a strong indication that the use of the ion lines below 300 nm or above 800 nm under these conditions can be the same or even favourable to the other spectroscopic techniques.

Decreasing of a number of interfering lines in the visible part of the spectra by use of the He at  $p = 30$  mbar and capacitor of  $0.33 \mu\text{F}$  charged up to only 1 kV showed, see Figure 4, that S/B ratio can thus be increased by one order of magnitude. Recording of spectra from the whole plasma volume, which is also significantly increased, will lead to the further enhancement of optical signals thus making LA-FED comparable with other analytical techniques.

The optimization of the working conditions (gas type and pressure), as well as the use of small nF capacitance and modifications of power supply in order to obtain voltages above 10 kV and periodical nanosecond current pulses which most probably lead to even greater optical signal enhancement, is in progress.

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## IZVOD

## LASERSKOM ABLACIJOM INICIRANO BRZO ELEKTRIČNO PRAŽNENJE ZA SPEKTROHEMIJSKE PRIMENE

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(Naučni rad)

U ovom radu ispitivane su različite metode za povećavanje intenziteta spektralnih linija elemenata uzorka pri interakciji laserskog impulsa sa metom od legure aluminijuma. Povećanje optičke emisije osnovni je preduslov za snižavanje praga detekcije spektroskopije laserski indukovanoj probnoj sa jednim laserskim impulsom. Ispitivanja su vršena pri različitim pritiscima argona, helijuma ili vazduha, sa ili bez dodatnog brzog električnog pražnjenja. Konstruisana je i korišćena cev za pražnjenje u obliku krsta, unutrašnjeg prečnika 10 mm, sa rastojanjem između elektroda 12 cm. Snimana je vremenska evaluacija spektara od 350 do 800 nm sa različitim vremenima kašnjenja i različitim širinama vremenskih prozora. Merenja su vršena pomoću spektrometra opremljenog ICCD kamerom, samo u vremenima posle gašenja intenzivnog kontinualnog zračenja. Merenja u različitim okolnim gasovima na istom pritisku bez električnog pražnjenja pokazala su da su optički signali u Ar veći, a u He manji od signala dobijenih u vazduhu. Maksimalni intenziteti dobijeni su pri pritisku Ar od 150 mbar. Ovo povećanje je najverovatnije prouzrokovano povećanjem gustine elektrona usled nižeg jonizacionog potencijala argona. Istraživanja uticaja brzog električnog pražnjenja na intenzitet zračenja vršena su pražnjenjem kondenzatora kapaciteta 14,5; 0,33 ili 0,1  $\mu\text{F}$ , prethodno napunjenih do napona između 1 i 3 kV. Napon kondenzatora određivan je iz uslova da njegova vrednost bude: a) manja od napona samookidanja i b) dovoljno velika radi minimiziranja kašnjenja između laserskog impulsa i pojave pražnjenja. Drugi uslov proizilazi iz zahteva da ispareni materijal mete bude još uvek prisutan u posmatranoj zapremini tokom uspostavljanja pražnjenja. Kapacitet kondenzatora, kao i vrste i pritisci okolnog gasa birani su kako bi se: a) sprečilo uspostavljanje tinjavog pražnjenja, b) smanjili gubici energije usled višestrukih jonizacija elemenata u plazmi, c) smanjilo pozadinsko zračenje nastalo usled preklapanja brojnih spektralno bliskih linija, d) produžilo vreme trajanja i e) povećala zapremina plazme. Optimalno povećanje integralnog intenziteta analitičkih linija od preko dva reda veličine dobijeno je u He pri  $p = 30$  mbar sa kondenzatorom od 0,33  $\mu\text{F}$  napunjenim do 1 kV, vidi sliku 4. Treba napomenuti da je iniciranjem pražnjenja u argonu ( $p = 30$  mbar;  $C = 14.5 \mu\text{F}$ ;  $U = 1$  kV) dodatno povećana brojnost i intenzitet linija. Zbog preklapanja linija, analitička primena pražnjenja pri ovim uslovima onemogućena je u posmatranoj oblasti spektra, što ne mora biti slučaj u ostalim oblastima.

*Ključne reči:* Spektrohemijska • Analitička spektrometrija • Spektroskopija laserski indukovanoj probnoj • Optička emisiona spektroskopija