Spectroscopic analysis of magnesium-aluminum alloys by laser induced breakdown spectroscopy

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Abstract

In this work, the spectra of plasma glow produced by Nd:YAG laser operated at 1.064 μm on Al-Mg alloys with same molar ratio samples in air were analyzed by comparing the atomic lines of aluminum and magnesium with that of strong standard lines. The effect of laser energies on spectral lines, produced by laser ablation, were investigated using optical spectroscopy, the electron density was measured utilizing the Stark broadening of magnesium-aluminum lines and the electron temperature was calculated from the standard Boltzmann plot method. The results that show the electron temperature increase in magnesium and aluminum targets but decrease in magnesium:aluminum alloy target, also show the electron density increase all the aluminum, magnesium and mix both them. It was found that the lines intensities at different laser peak powers increase when the laser peak power increases then decreases when the power continues to increase.

Key words

Laser-Induced Breakdown Spectroscopy (LIBS), optical emission, plasma diagnostics.

Introduction

Laser-induced breakdown spectroscopy (LIBS) is a relatively new technique for elemental analysis and characterization of solids, liquids, gases, and aerosols. LIBS is an atomic emission spectroscopic technique where by a high energy pulsed laser is focused onto a small sample volume leading to a breakdown of analytical into ions and free electrons, resulting in a plasma characterized by both continuum and atomic emission [1, 2]. The intrinsic attributes of this technique, like quasi nondestructive character, rapidity or only optical access to the sample, are evaluated in remote analyses, in-situ analysis, field
or space applications, examination, cleaning or dating of archaeological materials and objects of art, forensic analysis and many other applications focused of material sciences, pharmaceutical products, medicine and biology [3].

Plasma diagnostics method can be done through the calculation of the plasma electron density \( n_e \) and temperature \( T_e \). The electron density in general, specifies the state of thermo-dynamical equilibrium of the plasma, while the temperature determines the strength of the different distribution functions describing the plasma state [4].

The electron temperature of plasma was calculated using Boltzmann plot relation [5]:

\[
\ln \left( \frac{\lambda_{ij} I_{ij}}{h c A_{ij} \gamma_{ij}} \right) = -\frac{1}{kT} E_j + \ln \left( \frac{N_i}{N_j \gamma(T)} \right)
\]

where \( I_{ij} \) is the relative intensity (in arbitrary units) of the emission line between the energy levels i and j, \( \lambda_{ij} \) its wavelength (in nanometres), \( g_i \) is the degeneracy or statistical weight of the emitting upper level i of the studied transition, and \( A_{ij} \) is the transition probability for spontaneous radiative emission from the level i to the lower level j. Finally, \( E_j \) is the excitation energy (in eV) of level i, \( k \) is the Boltzmann constant, \( N \) state population densities.

The calculation of the electron density \( n_e \) through Stark broadening effect requires a line which is free from self-absorption [6]:

\[
n_e = \frac{4\pi}{2\omega_s} N_r
\]

\( \omega_s \) is the theoretical line full width Stark broadening parameter, calculated at the same reference electron density \( N_r \approx 10^{17} \text{ cm}^{-3} \).

**Experimental part**

Magnesium, aluminum, and magnesium:aluminum pellets were prepared using equal molar quantities of magnesium and aluminum powder and mix them by mechanical motor with steel balls for 10 minutes. A piston with a pressure of 3.5 tons was used to make a disk of 5 gm of each Mg, Al and Mg:Al mix alloy with 1 cm diameter. The samples were exposed to Nd:YAG pulse laser (9 ns duration, 10 Hz frequency, wavelength 1064 nm) which was operated at various energies from 400 mJ to 900 mJ using LIBS experimental system shown in Fig.1. The emitted spectrum from the surface of samples was transferred by optical fiber to be analyzed using a spectrometer connected with a computer to study the effect of laser energy and wavelength on the properties of the produced plasma.

![Fig. 1: Schematic of the experimental setup for the laser induced breakdown spectroscopy.](image-url)
Results and discussion

We have produced Plasma by the laser interaction with (Al, Mg and mix Al:Mg) alloy targets using Q-switched Nd:YAG in air. A spectrum consists of a number of characteristic spectral lines of a particular atoms and ions. Figs. 2-4 are shown the emission spectra of laser induced plasma from (Al, Mg and mix Al:Mg) targets in air, by Q-switched pulse laser with fundamental wavelength.

![Fig.2: Plasma emission spectra induced by 1064 nm laser, with different laser energies for aluminum powder in air.](image1)

![Fig.3: Plasma emission spectra induced by 1064 nm laser, with different laser energies for magnesium powder in air.](image2)
Fig. 4: Plasma emission spectra induced by 1064 nm laser, with different laser energies for magnesium and aluminum powder in air.

Where the intensities of the spectral lines increase with increasing in the laser peak energy because the mass ablation rate of the target also increases, the increase in laser power will increase its absorption in the plasma resulting in more ablation which results in the increase of the spectral line intensities of plasma emission.

The value of $T_e$ is obtained using the Boltzmann plot method by Eq.(1), as shown in Figs. 5-7 this requires peaks that originated from the same atomic species and the same ionization stage with data from NIST site, where the electron temperature equal to the invert of slope of fitting line (the slope of fitted line equals $-1/k_B T$). The fitting equations with the $R^2$ is (a statistical coefficient indicating the goodness of the linear fit) The best one has $R^2$ value closer to 1 [7].
Fig. 5: Boltzmann plot method for aluminum target using 1064 nm laser, with different laser energies.
Fig. 6: Boltzmann plot method for magnesium using 1064 nm laser, with different laser energies.
Fig. 7: Boltzmann plot method for magnesium and aluminum mix using 1064 nm laser, with different laser energies.

Electron temperature ($T_e$) was calculated from the slope of fitting line using Eq. (2) and electron density ($n_e$) using stark broadening as shown in fig.8. Stark broadening of spectral lines in lasmas results from collisions with charged species resulting in both a broadening of the line and a shift in the peak wavelength. Typical Stark broadened line profile is approximately Lorentzian and the experimental results shown here in Fig.8.
Fig. 8: Variation in the signal intensity and width of the Mg (II) lines at 279.553 nm and Mg I 285.2 nm at different values of 1064 nm laser.

Fig. 9 shows the variation of $T_e$ and $n_e$ as function of laser energy. This figure shows that the $T_e$ increase with increasing laser energy because collisions, and $n_e$ increase with increasing laser energy when varies from 400 to 900 mJ.

Fig. 10 shows the variation of $T_e$ and $n_e$ with laser energy. This figure shows that the $T_e$ increase with increasing laser energy because collisions, while $n_e$ increase with increasing laser energy when varies from 400 to 900 mJ.
Fig. 10: The variation of $T_e$ and $n_e$ with laser energy for plasma induced on Mg target.

Fig. 11 shows the variation of $T_e$ and $n_e$ with laser energy for mix alloy. This figure shows that the $T_e$ decrease with increasing laser energy, while $n_e$ increasing with increasing laser energy when varies from 400 to 900 mJ.

Fig. 11: The variation of $T_e$ and $n_e$ with laser energy for plasma induced on Mg:Al target.

At higher laser peak energy $T_e$ being near stable and doesn’t increase, because the plasma becomes opaque to the laser beam which shields the target. from Figs.9 and 10, we can see that the electron temperatures show a slow linear increase as the laser peak power increased; This is due to the absorption of laser photon by the plasma [8].

Conclusions

We can summarize ours conclusions as follows:

1- The interaction of laser irradiation with metallic targets is a very helpful method to produce plasma plumes consisting of highly concentrated electrons
2- The spectral lines intensities of the laser induced plasma emission exhibited a strong dependence on the ambient conditions. It is found that the lines intensities at different laser peak powers increase when the laser peak power increases and then decreases when the power continues to increase.
3- The plasma parameters such as temperature and number density are found to increase with the laser irradiance. It is inferred that at first stage the laser vapor interaction is largely due to the inverse bremsstrahlung (IB) process, and their values is different because of the competing effects of the target surface reflectivity and the laser plasma absorption, while the plasma the temperature begins to decrease more slowly in mix alloys due to the energy released by the recombination which compensate the cooling due expansion processes.

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