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Comparative Study of the Optical Emission Spectrum of TiO_2 Mixed with Al_2O_3 , and MgO , to Calculate the Plasma Parameters using the Boltzmann Plot Method

Zainab J. Jaffer, Alaa Nazar Abd Algaffar *

Department of Physics, College of Science for Women, University of Baghdad, Baghdad, Iraq

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Abstract

This research aims to apply laser-induced breakdown spectroscopy (LIBS) to characterize plasmas generated in air at atmospheric pressure. In this study, optical emission spectroscopy (OES) was used to analyze the plasma generated from $\text{TiO}_2\text{:MgO}$ and $\text{TiO}_2\text{:Al}_2\text{O}_3$ mixtures using a Q-switched (Nd: YAG) laser at different powers in air. The Boltzmann plot and Stark broadening methods were employed to investigate the plasma parameters of the prepared targets, including electron temperature (T_e), electron density (n_e), Debye length (λ_D), plasma frequency (f_p), and the number of particles in the Debye sphere (N_D). It was observed that the plasma parameter values of the prepared targets increased with increasing laser beam energy. The calculated electron temperature of the prepared samples were in the range (1.883 to 2.128) eV for $\text{TiO}_2\text{:MgO}$ and (1.654 to 1.701) eV for $\text{TiO}_2\text{:Al}_2\text{O}_3$, accompanied by electron densities ranging from $(7.647 \text{ to } 9.118) \times 10^{17} \text{ cm}^{-3}$ for ($\text{TiO}_2\text{:MgO}$) and $(2.632 \text{ to } 3.683) \times 10^{17} \text{ cm}^{-3}$ for ($\text{TiO}_2\text{:Al}_2\text{O}_3$) for laser energy range of (200– 400) mJ. Our findings indicate that laser energy influences all plasma parameters.

Keywords: OES, Optical properties, TiO_2 , Al_2O_3 , MgO , Effects of laser energy, Optical properties of mixed materials

دراسة مقارنة لطيف الانبعاث البصري لخلائط TiO_2 مع Al_2O_3 و MgO وحساب معاملات البلازما باستخدام طريقة مخطط بولتزمان

زينب جوهر جعفر , الاء نزار عبد الغفار *

قسم الفيزياء , كلية علوم للبنات , جامعة بغداد , بغداد , العراق

الخلاصة

تم في هذه الدراسة استخدام مطيافية الانبعاث البصري (OES) لتحليل البلازما المتولدة من الخلائط $\text{TiO}_2\text{:MgO}$ و $\text{TiO}_2\text{:Al}_2\text{O}_3$ باستخدام الليزر Q-Switched (Nd:YAG) بطاقات مختلفة في الهواء. تم إجراء توسيع Boltzmann Plot و Stark لدراسة معاملات البلازما للعينات المحضرة بما في ذلك درجة حرارة الإلكترون (T_e) وكثافة الإلكترون (n_e) و طول ديبي (λ_D) وتردد البلازما (f_p). نلاحظ ان قيم

*Email : alaana_phys@cs.w.uobaghdad.edu.iq

معلومات البلازما للعينات المحضرة تزداد مع زيادة طاقة شعاع الليزر وكذلك درجة حرارة الإلكترون المحسوبة للعينات المحضرة هي (1.883 إلى 2.128) إلكترون فولت لـ (TiO₂: MgO)، و (1.654 إلى 1.701) إلكترون فولت لـ (TiO₂: Al₂O₃) مصحوبة بكثافة إلكترونات التي تتراوح بين $(7.647-9.118) \times 10^{17} \text{cm}^{-3}$ (TiO₂:MgO) وتتراوح بين $(3.683-2.632) \times 10^{17} \text{cm}^{-3}$ لـ (TiO₂:Al₂O₃) لنطاق طاقة ليزر يتراوح بين (200 و 400) مللي جول. تشير نتائجنا إلى أن طاقة الليزر تؤثر على جميع معاملات البلازما.

1. Introduction

Laser-induced plasma spectroscopy (LIBS) is a type of atomic emission spectroscopy in which materials with optical properties are excited by high-energy laser pulses. Plasma, which is recognized as a source of exceptionally pure metal spectra, is generated when powerful laser pulses impact a metal surface (target) [1]. There is a significant amount of research in both the theoretical and practical aspects about plasmas formed by lasers as a result of interactions with solid objects. The features of these plasmas are influenced by a large variety of parameters that define the characteristics of the target. These parameters include the characteristics of the medium in the surrounding environment, the duration of the pulse, the wavelength of the laser, and more [2-3]. The production of plasma involves many stages. Each stage can be split down into its independent processes. An early stage of the process involves the interaction of laser light with a solid target, resulting in the production of plasma products, and surface ionization acceleration compared to the pulse period. The plasma absorbs the laser light in the second stage, which grows efficiently and consistently [4]. Plasma is formed after the third stage, which may occur as soon as the laser pulse energy heats the specific material (sample, target), causing the atoms of the target material to receive more energy and develop into plasma. The benefit of getting considerable samples of solids, liquids, and gases is something that can be accomplished [5]. The plasma and the concentration of the elements TiO₂ and MgO in the sample are responsible for determining the strength of the spectral emission lines of the spectrum produced by the created plasma. Electron temperature (T_e) and electron density (n_e) significantly affect the efficiency of LIBS [6]. LIBS is often used in biological studies for tissue samples, some bacterial types, gallstones, and aerosols. One of the primary applications of Laser-Induced Plasmas (LIP) is the utilization of plasma spectral sources to achieve a variety of benefits [7]. The plasma electron density (n_e) and electron temperature (T_e) can be calculated using a diagnostic plasma method. While the temperature regulates the intensity of the various distribution functions that characterize the state of the plasma, the electron density defines the state of thermodynamic equilibrium of the plasma. In general, the electron density is what characterizes the state of the plasma [8]. It is possible to determine the temperature of plasma using the ratio approach, a common method [9], using the following equation:

$$T_e = \frac{(E_2 - E_1)}{K \ln \left(\frac{I_1 \lambda_1 A_2 g_2}{I_2 \lambda_2 A_1 g_1} \right)} \quad (1)$$

where T_e is the electron temperature, E is the energy level of the spectral lines, I is the intensity of the emitted light at a particular wavelength in the plasma, λ is the laser wavelength, and g represents both the probability of a transition and its statistical weight.

The electron density is determined through diverse methodologies, one of which is the widely recognized Stark widening of spectral lines in a linear fashion. The Doppler width and the Stark effect are the primary factors that affect line broadening in LIBS plasmas. Doppler line broadening is determined by both the temperature of the emitting species and its atomic mass [10]. In this investigation, the Saha-Boltzmann equation was applied. Moreover, the spectra of the identical line-containing element and the intensity of ionization were

considered. The Saha-Boltzmann system can be described by the equation presented below [9]:

$$\eta_e = \frac{I_1}{I_2^*} 6.04 * 10^{21} (Te)^{3/2} e^{\frac{(E_1-E_2-X_Z)}{kTe}} (cm^{-3}) \quad (2)$$

$$\text{Where: } I_2^* = \frac{I_2 \lambda_2}{g_2 A_2} \quad (3)$$

Where X_Z represents the ionization energy of a species at the z level of ionization, measured in electron volts (eV), I_z denotes the line intensity, which is the strength of the associated transition, which represents the probability of a transition, g represents the statistical weight, which is a measure of the number of energy states available to the system, I is the intensity of the emitted light at a particular wavelength in the plasma, and T_e represents electron temperature.

The plasma frequency (f_p) is determined by the equation [9]:

$$f_p \approx 8.98 \sqrt{n_e} \quad (Hz) \quad (4)$$

A significantly high value for the plasma frequency is produced by the fact that the plasma density influences the plasma frequency, resulting in a relatively high value [11]. The phenomenon known as "Debye shielding" describes how charged particles react to localized electric fields by reducing their intensity. Plasma exhibits quasi-neutrality due to this shielding effect.

The Debye length (λ_D) (in cm), is defined as [12]:

$$\lambda_D = \left(\frac{\epsilon_0 k T_e}{n_e e^2} \right)^{1/2} = 743 * \left(\frac{T_e}{n_e} \right)^{1/2} \quad (5)$$

Where L is the system dimension in cm, n_e is the electron density, T_e is the electron temperature in eV, and K is the Boltzmann constant.

The number of particles in the Debye sphere (N_D) is determined by the density of electrons and temperature. The existence of plasma is definite since the N_D value is larger than 1, (N_D) also noted as D [13]:

$$N_{D= \frac{4\pi}{3} n_e \lambda_D^3} \quad (6)$$

The manufacturing of nanoparticles and their application areas have tremendous prospects in the fields of science and technology, as they exhibit unknown traits and behavior that depend on their size [14,15]. The versatile features of (TiO₂), (Al₂O₃), and (MgO) nanoparticles make them useful in many different contexts. Paint, pigments, cosmetics, and a host of other items rely on titanium dioxide (TiO₂) as an essential ingredient [16]. Optical coatings, beam splitters, and anti-reflective coatings also make use of it because of its high refractive index and dielectric constant. It has been employed as a hygrometer and a thermocouple for measuring oxygen levels at elevated temperatures [17-18]. Alumina (Al₂O₃), or aluminium oxide, can be synthesized using different strategies, resulting in various phases, including α -phase, β -phase, β - γ , and $\alpha\delta$. For each phase, different temperatures are employed during its fabrication. The characteristics of its various phases include chemical stability level, thermal expansion resistance, alternating relative strength condition, different types of wearability, high hardness, a hard tumbling point, high electrical resistivity, and a meltable magneto resistive property. Furthermore, it may serve as an intermediate form in certain types of chemical reactions [19-20]. It is anticipated that this will have a beneficial impact on the Al₂O₃ manufacturing sector in terms of cost of production (economic assessment), techniques of synthesis, and artificial goods. Alumina has been frequently employed in various applications due to its distinctive characteristics, which

include its use in microelectronics, catalysis, technical ceramics, and refractories [21]. Magnesium oxide (MgO) is a multifunctional compound employed in various scientific and technological contexts. In addition to its role as a buffer layer in magnetic tunnel junctions, it has various other applications [22]. MgO nanoparticles have prospective use in microelectronics, diagnostics, and biomolecular detection [23].

2. Experimental part

TiO₂:Al₂O₃ (using 1.72 g TiO₂ and 0.29 g of MgO) and TiO₂:MgO (1.16 g of TiO₂ and 0.84 g of Al₂O₃) pellets were synthesized by combining TiO₂ with Al₂O₃ and TiO₂ with MgO in uneven molar ratios, according to Lorentz theory[24]. The powders were blended using a mechanical motor. A 3.5-ton piston was utilized to compress a mixture of 2 grams each of TiO₂:Al₂O₃ and TiO₂:MgO mixtures into a disk with a diameter of 1 cm. An Nd: YAG Q-switched pulsed laser (with a wavelength of 1064 nanometers, an oscillation frequency of 10 Hz, and a period of 9 nanoseconds) was used for examining the samples. The laser was operated at different energy levels ranging from 200 to 400 mJ using the LIBS experimental apparatus depicted in Figure 1. The optical emission spectroscopy (OES) was used to analyze the generated plasma. A spectrometer (type Surwit, S3000-UV-NIR) linked to a computer was used to analyze the spectrum of radiation that are sent from the samples' surfaces over an optical connection. This was done to find the effect of different laser intensities on the plasma's properties.

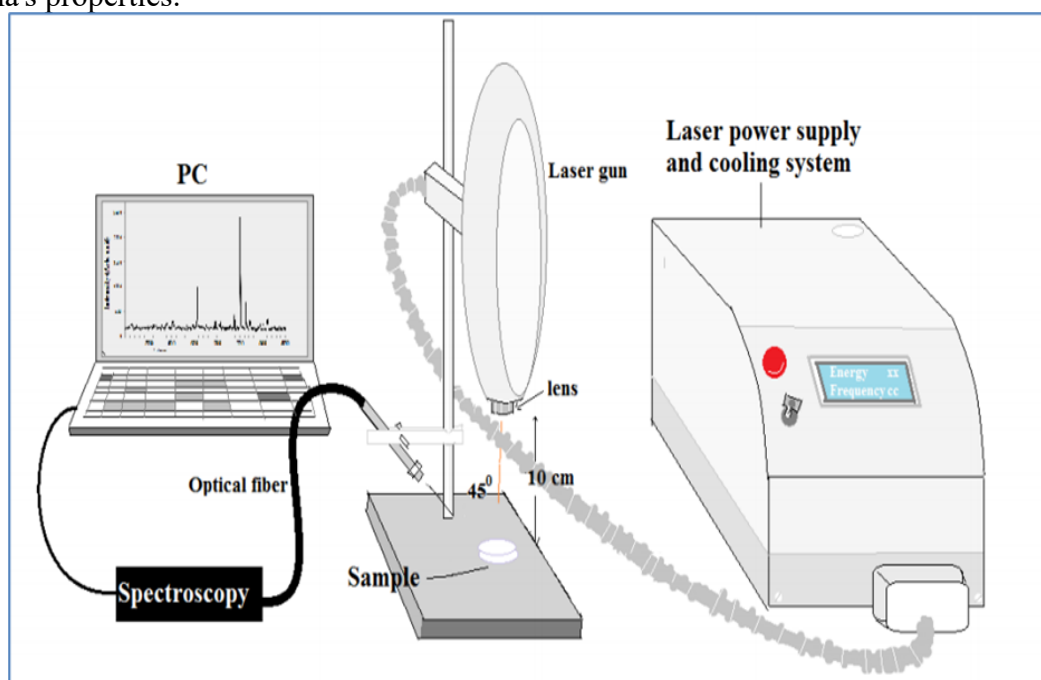


Figure 1: schematic diagram of the laser-induced breakdown spectroscopy experimental setup.

3. Discussion and Results

3.1 Impact of Laser Energy on the Emission Spectrum of Mixed Material Plasmas (TiO₂: Al₂O₃) and (TiO₂: MgO).

A material's composition is determined by simulating the gradual decomposition of exciting species in plasma through the use of spectroscopy. Optical emission via excitation of species can be used to identify whether a substance is a pure component. An excited electron is bound to relax and revert to a lower energy state, known as the ground state, according to this procedure. In this instance, photons with a specific wavelength are produced that are shown as lines in the emission spectrum. The plasma's emission spectra of (TiO₂: Al₂O₃) and

(TiO₂: MgO), in the spectral range of 250–750 nm for specific pulsed laser energies of 200, 300, and 400 mJ, are shown in Figure 2(a-b). The spectrum shown in Figure 2(a) displays multiple neutral (Ti I) peaks, which occur at wavelengths of 365.34, 388.28, 392.98, 397.77, 399.86, 407.48, 428.60, 444.91, 455.24, 466.30, 475.92, 487.01, 501.41, 506.46, 512.04, 512.03, 551.45, 586.64, and 589.92 nm. Additionally, there is an (Al I) peak throughout the spectrum at a specific wavelength of 308.21 nm. The ionic emission lines of titanium (Ti II) can be observed at wavelengths of 323.45, 334.94, 375.93, 390.05, and 391.34 nm. Similarly, aluminum (Al II) emits at wavelengths of 281.61, 559.33, and 624.33 nm. While the spectrum in Figure 2 (b) displays multiple peaks of neutrality (Ti I) element with designated wavelengths, 365.34, 388.28, 392.98, 397.77, 399.86, 407.48, 428.60, 444.91, 455.24, 466.30, 475.92, 487.01, 501.41, 506.46, 512.04, 512.03, 551.45, 586.64, and 589.92 nm. Additionally, there are (Mg I) peaks observed at wavelengths 285.21, 293.85, 382.83, 518.36, and 571.11 nm. An ionic emission of the lines of titanium (Ti II) is observed at wavelengths of 323.45, 334.94, 375.93, 390.05, and 391.34 nm. Additionally, the emission line of magnesium (Mg II) appears at a wavelength of 279.55 nm. From Figure 2(a-b), it can be noted that an increase in laser energy results in greater absorption of laser light by plasma, hence enhancing the intensity of spectral lines and the ablation rate [25].

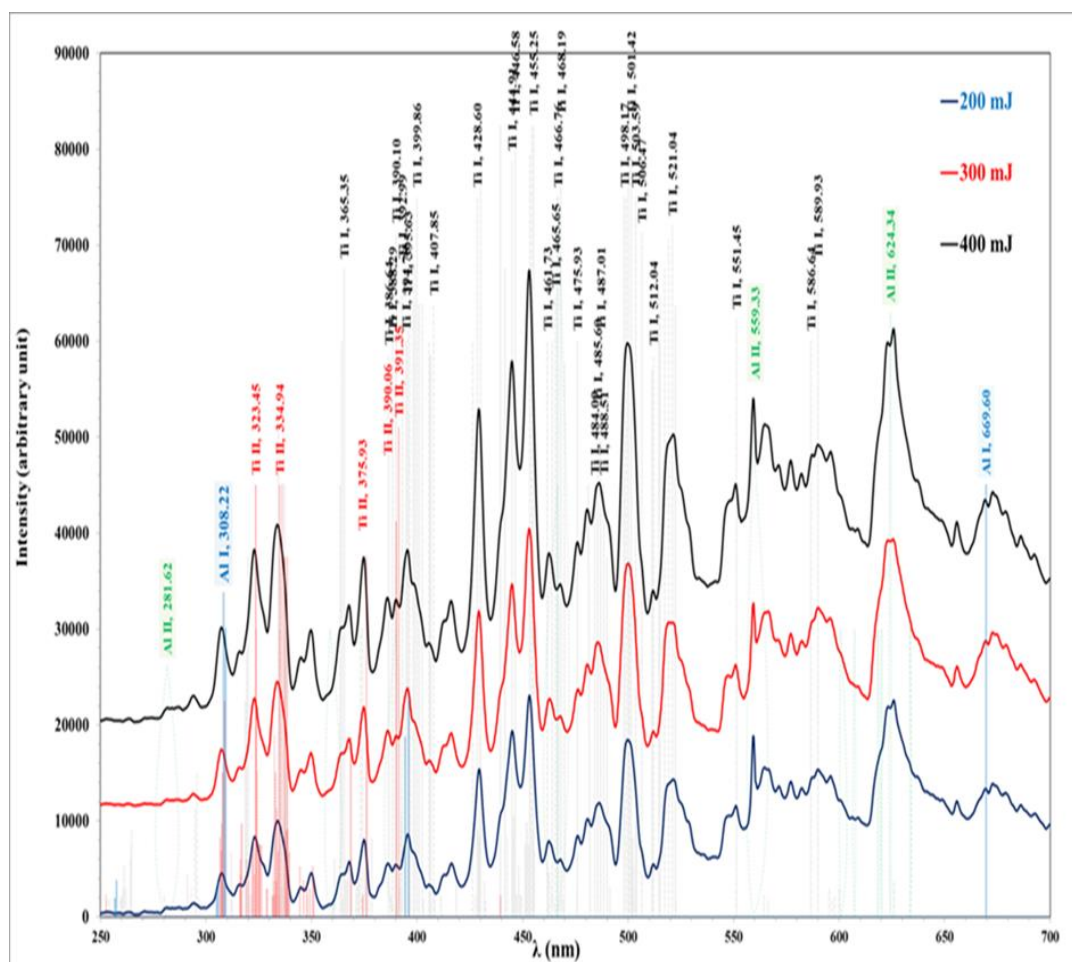


Figure 2 (a): Plasma emission spectra generated by Nd: YAG laser at different laser energies for (TiO₂:Al₂O₃) powder.

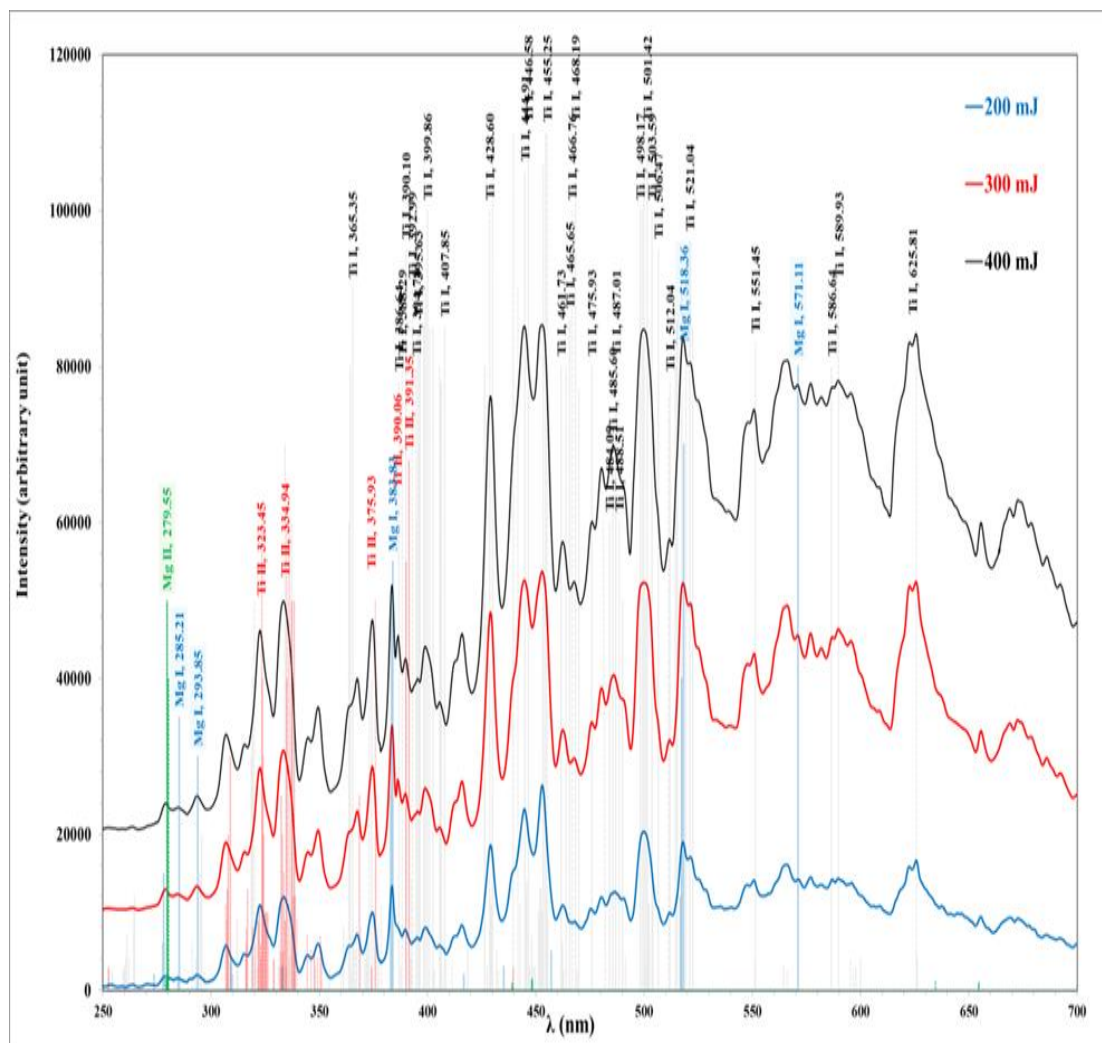
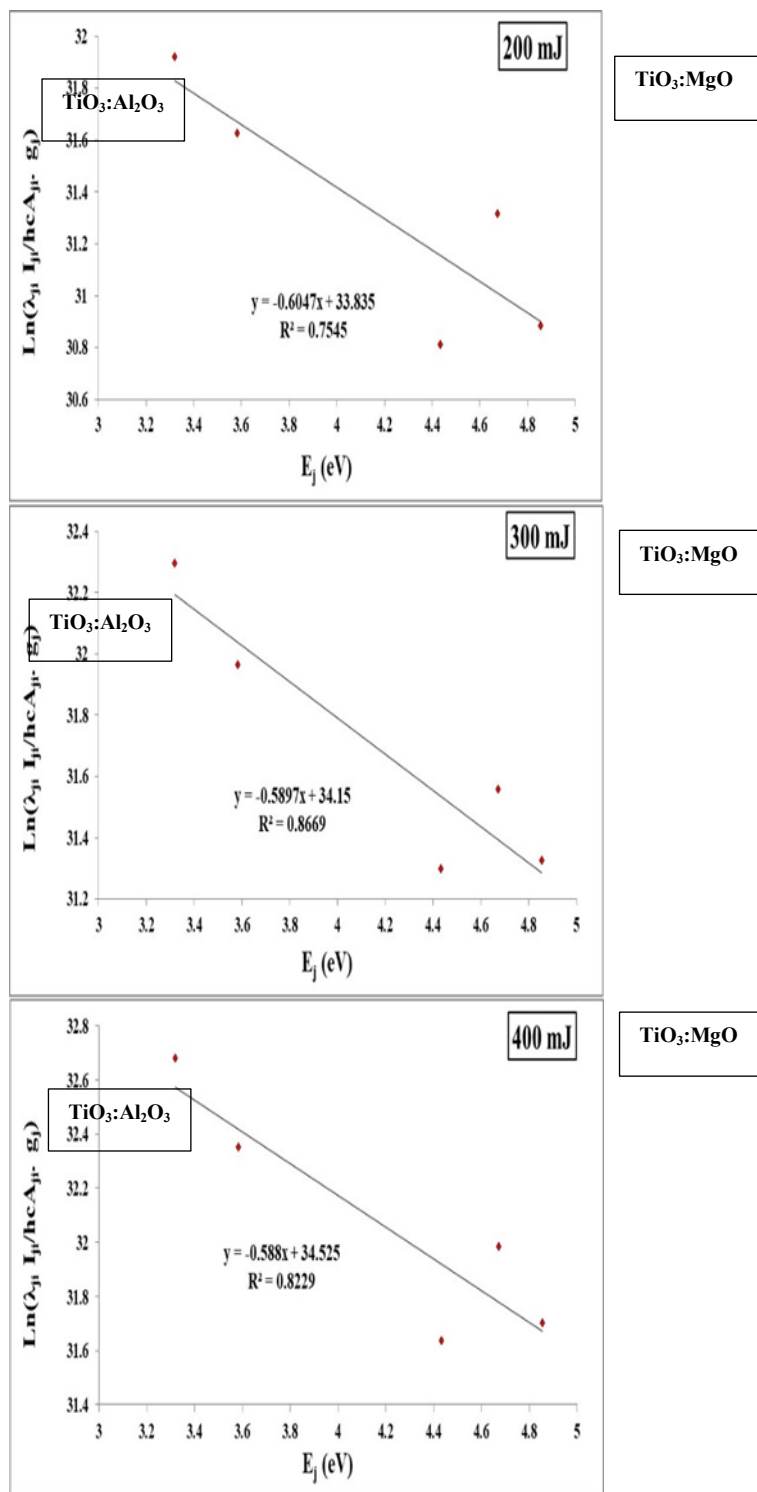


Figure 2 (b): Plasma emission spectra generated by Nd: YAG laser at different laser energies for (TiO₂: MgO) powder

The value of temperature is determined using the Boltzmann plot technique, as delineated in Eq. (1) and shown in Figure 3. To fulfil this criterion, the peaks must originate from the same atomic species and ionization state. The necessary data may be acquired from the NIST website. The electron temperature is ascertainable by the inverse slope of the fitting line, with the slope represented as $(-1/T)$. The R^2 value of the fitting equations is a statistical factor that indicates the quality of the linear regression. The ideal selection is the one with an R^2 value closest to 1 [26].



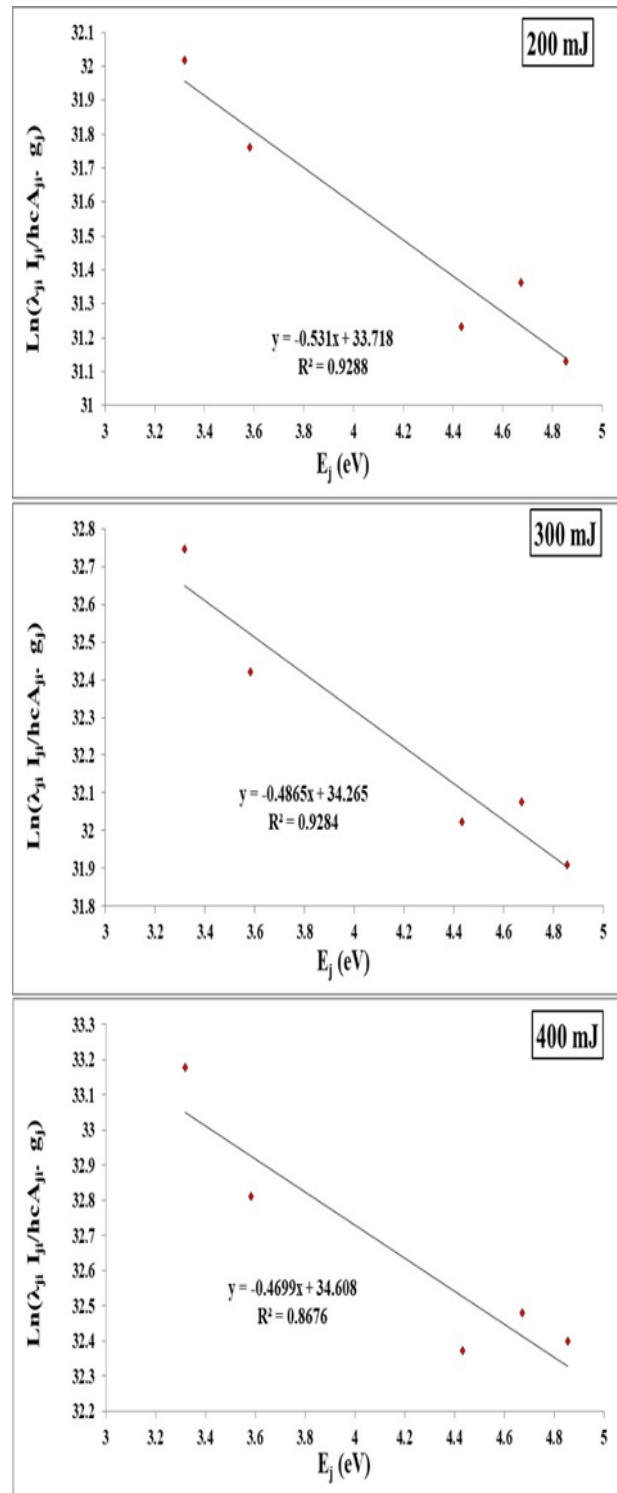


Figure 3: Boltzmann plots for (TiO₂:Al₂O₃) and (TiO₂: MgO) mixtures using Nd: YAG laser at different laser energies

Collisions between charged particles result in pronounced spectral line broadening in plasmas, which is related to electron density. These lines shown in Figure 4 are Lorentzian fits of the Al-II and Mg-II lines, and the change in FWHM is the Al-II Stark broadening at 559.33 nm and the Mg-II Stark broadening at 383.83 nm at different laser energies.

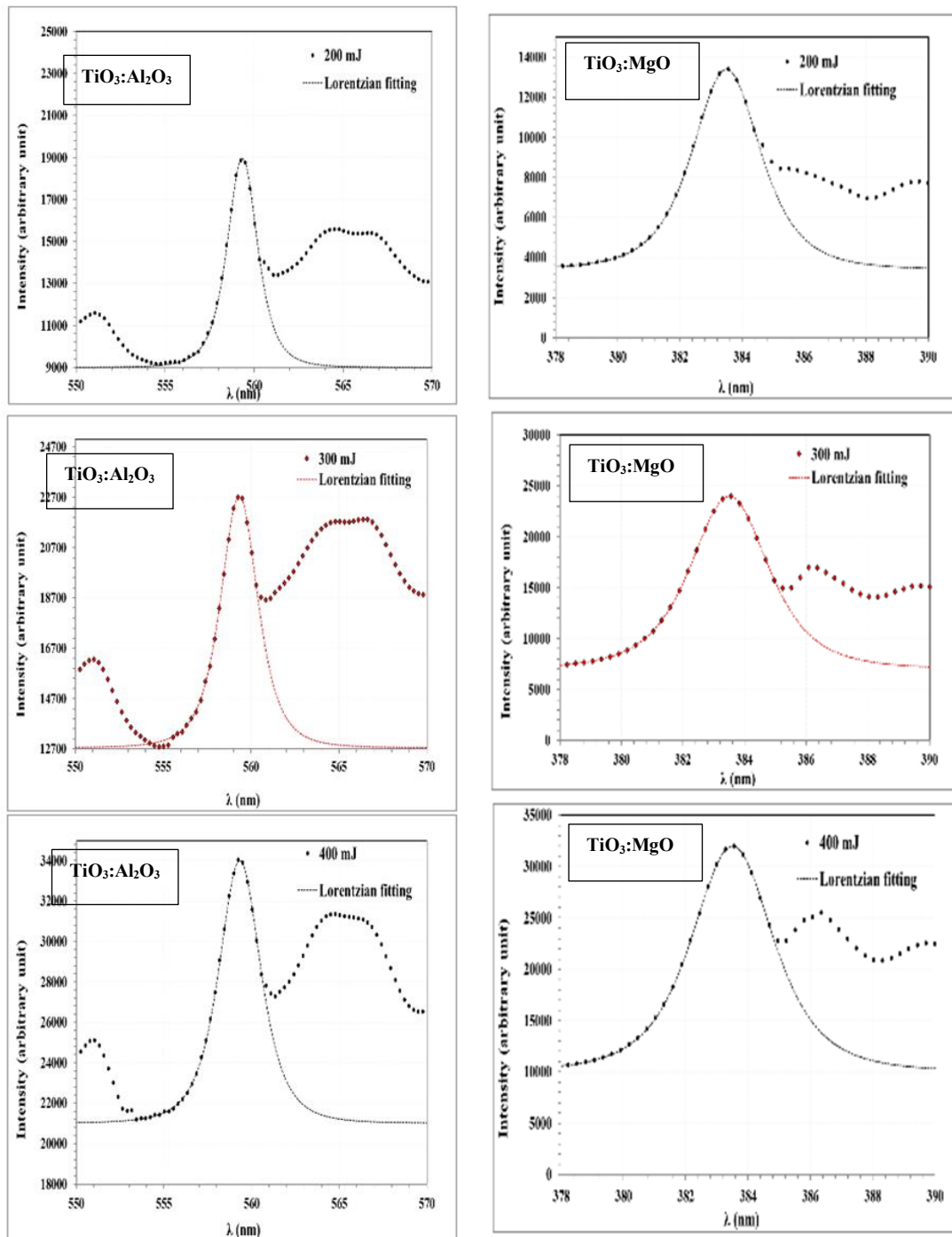


Figure 4: The Stark broadening for Al and Mg plasma at different laser energies

3.2 The Effect of the Target Metal on the Electron Density and Temperature.

Emission features specific to atomic and ionic species of $(\text{TiO}_2:\text{Al}_2\text{O}_3)$ and $(\text{TiO}_2:\text{MgO})$ spectrum intensities and line widths are used to infer two crucial plasma parameters: the electron temperature and density, which are calculated as a function of laser energy. The two-line ratio was utilized in Equation (1). The electron temperature was calculated using the atomic lines of titanium (Ti I) and aluminum (Al I) elements, as well as the ionic lines of titanium (Ti I) and magnesium (Mg I) elements. The laser energy utilized for this calculation was 200, 300, and 400 mJ. Table 1 presents the parameters of the spectral lines for $(\text{TiO}_2:\text{Al}_2\text{O}_3)$ and $(\text{TiO}_2:\text{MgO})$ mixtures. Stark broadening refers to the width of a spectral line of an atom or ion, is one of the most precise ways to determine the electron density. This

broadening occurs when charged particles collide with released atoms. The relationship below can be used to compute electron density [27]:

$$n_e = \left(\frac{\lambda_{FWHM}}{2\omega} \right) \times 10^{16} \quad (7)$$

where ω is the electron impact parameter, n_e is the electron density (cm^{-3}), and λ_{FWHM} stands for the Stark full-width at half-maximum. Figure 5(a-b) shows that both the electron temperature and electron density rise with increasing laser energy. The rationale for this is that the phenomenon known as electron-neutral inverse Bremsstrahlung, which is the process that is accountable for the absorption of laser photons in plasma, provides an explanation for the correlation between the rise in laser intensity and the increase in Stark widening [28].

Table 1: Characteristics of plasma for ($\text{TiO}_2:\text{Al}_2\text{O}_3$) and ($\text{TiO}_2:\text{MgO}$) mixtures

Samples	E (mJ)	T_e (eV)	$n_e \times 10^{17} (\text{cm}^{-3})$	$f_p (\text{Hz}) \times 10^{10}$	$\lambda_D \times 10^{-5} (\text{cm})$	N_d
$(\text{TiO}_2:\text{Al}_2\text{O}_3)$	200	1.654	2.632	460.664	1.863	7122
	300	1.696	3.289	515.038	1.687	6615
	400	1.701	3.684	545.065	1.596	6279
$(\text{TiO}_2:\text{MgO})$	200	1.883	7.647	785.278	1.166	5078
	300	2.056	8.824	843.524	1.134	5391
	400	2.128	9.118	857.468	1.135	5586

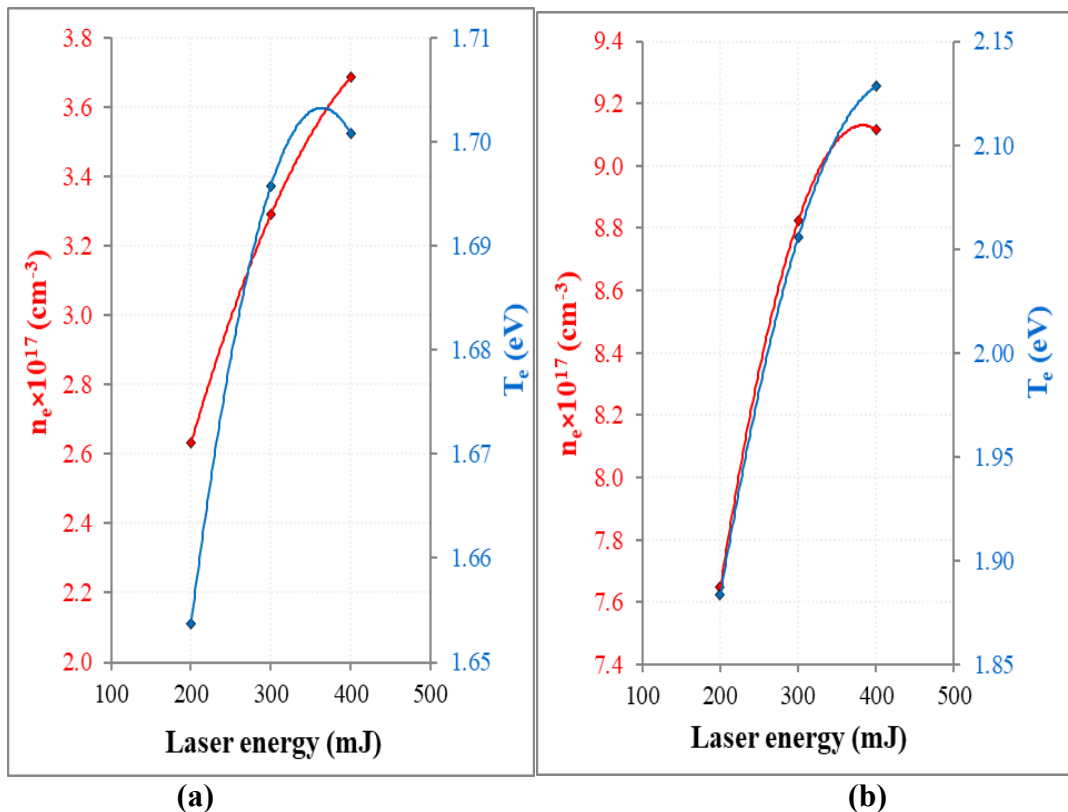


Figure 5: the relation between the electron temperature and density and different laser energies in the mixtures of ($\text{TiO}_2:\text{Al}_2\text{O}_3$) and ($\text{TiO}_2:\text{MgO}$) at atmospheric pressure.

Conclusions

The properties of $\text{TiO}_2\text{:MgO}$ and $\text{TiO}_2\text{:Al}_2\text{O}_3$ spectral lines in plasma were shown to be greatly impacted by the laser energy. It was shown that many important plasma properties, such as electron density, electron temperature, Debye length, plasma frequency, and the number of particles in the Debye sphere, are influenced by the laser energy. To make measurements, optical emission spectroscopy was used to ascertain the correlations between plasma properties, including electron density and electron temperature. Their sensitivity to laser energy was used to calculate the plasma properties. As the laser energy increased, the results showed that T_e , n_e , and f_p values increased while λ_D values decreased.

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Author contributions

Zainab J. Jaffer contributed towards conceptualization, methodology, data curation, investigation, and writing- original draft preparation. Alaa Nazar contributed to methodology, investigation, validation, resources, and supervision.

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Data availability

No data or material is provided in this manuscript.

Declarations

Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Consent for publication

The authors hereby consent to publication of the present research in this journal if selected for publication.

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