PLASMA PARAMETERS CHARACTERIZATION OF LASER INDUCED BREAKDOWN SPECTROSCOPY ON SOLID MATERIALS AT DIFFERENT WAVELENGTHS

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To my husband Mr. Khairil Roslan, the person that always stay by my side, gives help, support, guidance and everything, I hope you too can finish your thesis. My son Nuh Zulkarnain, thank you for all the love. My parents, Mr Tan Halid and Mrs Habsah, the person who's always pray for me. My sibling who's always looks up for me. I hope that you can follow your dream. My family and friends, thank you for your support.

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ABSTRACT

The purpose of this research is to investigate the influence of laser wavelength on the plasma parameters in Laser Induced Breakdown of three different types of samples. The samples used were carbon rod, iron rod and stainless steel which represent non-metal, metal and alloy sample respectively. The source used is Nd:YAG laser operating at 1064 nm and 532 nm with laser energy of 150 mJ and 110 mJ respectively. The experiment was conducted in air at atmospheric pressure. The samples were placed at 5 cm, approximately 45° from a detector and 12 cm from the laser source. The plasma emission spectra were recorded and analyzed. Plasma parameters that have been taken into consideration were plasma emission spectrum, area of ablated surface, plasma spark shape, plasma electron temperature and plasma electron density. The results show that each sample produced different emission spectra. In addition the carbon sample shows the largest circular ablated area with radius 1.313 mm when generated using 1064 nm laser. Each sample produces different shapes of plasma sparks when generated using both laser sources. The plasma electron temperatures of carbon and iron samples produced by 1064 nm laser were estimated at 36671 K and 28200 K respectively while those generated with 532 nm laser were at 27271 K and 21585 K respectively. The stainless steel is consisted of Fe, Ni, Cr and Mn elements. The plasma electron temperatures of these elements were 21005 K, 2901 K, 10676 K and 12649 K respectively when generated using 1064 nm laser and 15202 K, 2785 K, 9748 K and 11697 K respectively using 532 nm laser. The other result shows that carbon rod as a non-metal sample has the highest plasma electron density when generated using 532 nm laser while the stainless steel sample has the highest plasma electron density when used with 1064 nm laser. In conclusions, the plasma emission spectra in all samples have been captured. The study shows that both types of laser are suitable for elemental detection. The plasma electron temperature and the plasma electron density have been determined for both laser sources.

ABSTRAK

Tujuan penyelidikan ini adalah untuk menyiasat pengaruh panjang gelombang laser keatas parameter plasma dalam penghuraian teraruh laser bagi tiga sampel yang berlainan jenis. Sampel yang digunakan ialah rod karbon, rod besi dan keluli tahan karat yang masing-masing mewakili sampel bukan logam, logam dan aloi. Sumber yang digunakan ialah laser Nd:YAG beroperasi pada 1064 nm dan 532 nm masing-masing dengan tenaga laser 150 mJ dan 110 mJ. Kajian dijalankan di udara pada tekanan atmosfera. Sampel diletakkan pada 5 cm, anggaran 45º dari pengesan dan 12 cm dari sumber laser. Spektrum pancaran plasma direkod dan dianalisis. Parameter plasma yang dipertimbangkan ialah spektrum sinaran plasma, luas permukaan yang terkopek, bentuk pancaran plasma, suhu elektron plasma dan ketumpatan elektron plasma. Hasil kajian menunjukkan setiap sampel menghasilkan spectrum berbeza. Tambahan pula sampel karbon menunjukan luas kawasan terkopek membulat terbesar dengan jejari 1.313 mm apabila dijana menggunakan laser 1064 nm. Setiap sampel menghasilkan sinaran plasma berbeza apabila dijana menggunakan kedua-dua sumber laser. Suhu elektron plasma untuk sampel karbon dan besi dihasilkan pada 1064 nm masing-masing dianggarkan 36671 K dan 28200 K manakala suhu elektron plasma yang dijana oleh laser 532 nm masing-masing ialah 27271 K and 21585 K. Keluli tahan karat mengandungi elemen Fe, Ni, Cr dan Mn. Suhu elektron plasma bagi elemen tersebut masing-masing ialah 21005 K, 2901 K, 10676 K dan 12649 K apabila dijana menggunakan laser 1064 nm dan masing-masing 15202 K, 2785 K, 9748 K and 11697 K mengunakan laser 532 nm. Keputusan lain kajian menunjukkan karbon sebagai sampel bukan logam mempunyai ketumpatan elektron plasma yang paling tinggi apabila dijana menggunakan laser 532 nm manakala ketumpatan elektron plasma untuk sampel keluli tahan karat paling tinggi untuk laser 1064 nm. Kesimpulannya, spektrum sinaran plasma untuk semua sampel telah direkodkan. Kajian menunjukkan kedua-dua jenis laser sesuai untuk pengesan elemen. Suhu plasma elektron dan ketumpatan plasma elektron telah ditentukan untuk kedua-dua sumber laser.

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LIST OF ABBREVATIONS

LIBS	Laser induced breakdown spectroscopy
Nd:YAG	neodymium-doped yttrium aluminum garnet
NIST	National Institute of Standard and Technology
Fe	Iron
Cr	Chromium
Ni	Nickel
Mn	Manganese
T_e	Plasma electron temperature
N_e	Plasma electron density
C	Carbon
eV	Electron volt
K	Kelvin
a.u.	Arbitrary unit
LTE	Local thermodynamic equilibrium
T_{ext}	Excitation temperature
T_H	Heavy particle temperature
T_ν	Photon distribution
UV	Ultra violet
h	Planks constant
m_e	Mass of electron
E_{ion}	Ionization energy

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CHAPTER 1

INTRODUCTION

1.1 Introduction

The development of laser has started since invention of laser in May 1960. The first clear instruction of LIBS was already provided (Maxwell, 1963). LIBS were used as a medium to investigate the element that contained in any types of substance at any states, solid, liquid, or gas. There are so many advantages when the element was detected. Harmful substance in the sample can be detected, the quality and the status of the metal can be determined also the properties of the substance can be analysed. Therefore, any improvements for the substance depend on the suitable composition.

Adding certain elements can effects the properties of the material, for example, to increase the strength of the stainless steel, element carbon will be added with suitable amount. Even with low concentration, it still changes the properties of the material. LIBS have been utilized at the micro scale in order to provide chemical mapping for given sample (Mateo *et. al,* 2003; Menut *et. al* 2003; Fabre *et. al,* 2007). Each material has different composition, LIBS gives unique spectrum for each sample. LIBs has wide application in various field for example, metal analysis, material processing, thin film deposition, and detection of explosive residue, also the analysis in medical, forensics and art fields. In addition, LIBS also can be used to detect the hazardous substance in the product. LIBS method requires minimal preparation procedures and only small amount of the sample is required to do analysis.

LIBS are one of the effective methods to analyse all types of samples starting solid until gas state, or in air or vacuum condition. The pulsed lasers are demonstrated to ablate the sample, as the result, the sample vaporized and ionized in hot plasma. Finally, it will be detected by the spectrometer. The analysis of the spectroscopic data emitting from the laser induced plasma when the irradiance of the laser focused on the material exceed its threshold (Fabre *et. al*, 2007). This technique frequently use on solid sample rather than liquid sample (Cramers and Radziemskii, 2006) since hot plasma are hardly form on the liquid samples.

The light emitting from laser induced plasma information that provide the qualitative and quantitative analysis about the sample. The plasma properties that can be obtained from the plasma spectrum were temperature, electron density, and the number densities of the emitting species (Cramers and Radziemskii, 2006). It is important to understand the entire process occur, such as dissociation, atomization, ionization and excitation, so that the improvement can be made for LIBS application (Dacey, 1962).

Recent studies shows that the there are no research about the comparison between non-metal, metal and alloy by using two different wavelengths. Some of the research did study about the alloy, for example Aguilera *et. al*., (2007), they study alloy by using 1064 nm laser wavelength. The plasma electron temperature was increase when the radial length decreases. They also concluded that the plasma electron temperature of neutral atom was different with single ionized atom.

On the other hand, Messoud Aberkane, (2015) study the plasma electron temperature of Fe-V-C metallic alloy in 1064 nm, 532 nm and 355 nm to calculate the plasma electron temperature and the relationship with surface hardness, unfortunately the T_e was not clearly stated. They concluded that T_e can be used to calculate the surface hardness. They also study the crater depth of the target spot. It shows that it increase when the laser wavelength decrease. It shows that the 1064 nm laser wavelength more suitable where it more sensitive and less destructive. Hanif, (2012) did compare the laser wavelength, but they only focus on one element that is nickel. Majority of the research only focus on pure sample, for example carbon, aluminium, and copper. There is research by Zhang *et.al*, (2014) where they study detail about the laser-induced plasma temperature but did not cover the plasma electron density.

In this research, LIBS's capabilities were tested on solid samples at different wavelength of 1064 nm and 532 nm of Nd:YAG laser, both of laser wavelength has different laser energy. 1064 nm has 150 mJ while the 532 nm has 110 mJ. Plasma emission spectrum pattern of stainless steel, iron and carbon samples were collected. The spectrum for each sample was analysed and characterize. Identifying the element exists in the samples was the preliminary result. The electron temperature and electron density of plasma for each sample were calculated for both laser wavelength. The T_e was estimated by the Boltzmann plot method and the n_e by Saha-Boltzmann equation. The results will be comparing on how the laser wavelengths influence the plasma spark, ablated area, the T_e and the n_e. of the three types of the samples which were non-metal, metal and alloy.

1.2 Problem Statement

Plasma is an ionized particle form results from high temperature heating in instant. Some of the important properties of plasma parameter are electron temperature and electron density. Each element has different electron temperature and electron density due to many factors. The laser wavelength affects the formation of plasma, such as the laser plasma interaction and the plasma ignition threshold, the sample ablated area and the plasma electron temperature and plasma electron density. Thus, this study was conducted to investigate the effect of wavelength of Nd:YAG laser at 1064 nm and 532 nm on the LIBS plasma parameter for several solid samples. The samples were categorized as non-metal, metal and alloy.

1.3 Objectives

This study focus on the following objectives:

i. To study the plasma spectrum of three different samples which were stainless steel, iron rod and carbon rod by using LIBS technique with two different types of Nd:YAG laser wavelength at 1064 nm and 532 nm in air at atmospheric pressure.

ii. To calculate the plasma electron temperature and electron density of those three samples for both wavelength of Nd:YAG laser.

iii. To verified the effect of laser wavelength on plasma parameters for non-metal, metal and alloy.

1.4 Scope of Study

In this study, a Q-Switched Nd:YAG laser with wavelength 1064 nm and 532 nm was used as laser sources. The laser output of 1064 nm was 150 mJ, while at 532 nm the output energy was 110 mJ with pulse duration of 10 ns. The research works was to obtain the spectroscopy signal from three different samples, which were stainless steel, iron rod and carbon rod. The laser was shot the sample and the sample produced plasma spark which contain the information about elements on the sample. The plasma plume was captured out by using spectrometer which was connected to the computer. The analytical technique was applied to determine the properties of plasma emission.

There were two methods used in this study for estimating the electron temperature and electron density of the plasma. The calculation of plasma electron temperature was done by Boltzmann plot method and the plasma electron density by Saha-Boltzmann equation. Furthermore, the LIBS plasma was assumed in LTE condition.

1.5 Significances and Original Contributions of This Study

This research has some significant. First, it was able to conduct the element identification by using simple and low cost methods. Secondly, it able to compare the plasma parameter for three different types of sample which was non-metal, metal and alloy. Lastly, able to compare the effect of different types of laser wavelength on plasma parameter such as plasma plume formation, image of ablated area, plasma temperature and electron density. This study implies a great importance to the development of laser induced breakdown spectroscopy application. It can contribute to the development of system application and human capital in this field. Hopefully it is used as the catalyst and reference point for further research.

1.6 Thesis Outline

This thesis consisted of five chapters and describes the qualitative and the quantitative analysis of determining the plasma parameters by using spectroscopic technique. This study offers a systematic way of analysing the laser induced breakdown spectroscopic plasma.

Chapter 1 provides brief introduction of this research. Problem statement and objective are included to show the difficulty and ways to solve the problem. The scope and significance of this research are also written in this chapter.

Chapter 2 offers general information about laser induced breakdown spectroscopy, nonlinear optics of second generation, plasma theory that related to this work and the role of wavelength in plasma formation. Some overview of previous studies also attached to show the validity of this research.

Chapter 3 was the methodology of research preparation and procedure carried out to obtain results from device used. The schematic diagram of all experimental set up. In addition, the information regarding analysis in terms of mathematical equation also explained roughly in this chapter.

Chapter 4 presents all the results of plasma analysis include the discussion on the data analyses towards the proposed objectives. The result divided into three sub chapter that represent three different samples. This part covers the plasma parameters which include the plasma plume, ablated area images on the sample, plasma electron temperature and plasma electron density.

Chapter 5 concludes the entire research to ensure research objectives are fulfilled. The information gather from this research may beneficial for future directions. Certain important calculations involving the results and tables that are not included in Chapter 4 are attached in the appendix.

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