DETERMINATION OF PLASMA PROPERTIES BY USING LASER INDUCED BREAKDOWN SPECTROSCOPY

SITI NUR AZILAH BINTI AZIZ

A thesis submitted in fulfilment of the requirements for the award of
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ABSTRACT

Laser-Induced Breakdown Spectroscopy (LIBS) is a growing technique in atomic spectroscopy due to its advantages with minimum sample preparation. LIBS use high-powered Nd-YAG laser that is focused on the surface of heavy metal samples. The samples choose were aluminium, brass and copper. This interaction caused the formation of plasma emission. This plasma emission is detected by a spectrometer and then analysed by using the application software, Origin 8.5 in the personal computer. This research was conducted in order to study the properties of the plasma such as the electron temperature and electron density. Electron temperature was calculated by using Boltzman plot method, whereas electron density was determined by using Saha-Boltzmann equation. Besides that, the effect of the time gate to the signal intensity also include in this project. Different delay time of 0 μs, 0.1 μs and 5.0 μs were used to compare the pattern of the spectral line. The results show the 5.0 μs delay time has a good pattern compared to 0 μs and 0.1 μs.

ABSTRAK

Laser-Induced Breakdown Spectroscopy (LIBS) ialah suatu teknik dalam spektroskopi atom yang mempunyai kelebihan dengan persediaan sampel yang minimum. LIBS menggunakan tenaga laser Nd:YAG yang tinggi iaitu yang difokuskan kepada sampel logam berat. Sampel yang dipilih adalah aluminium, pancalogam dan tembaga. Interaksi ini menyebabkan terjadinya pancaran plasma. Pancaran plasma tersebut di kesan oleh spektrometer dan di analisis menggunakan aplikasi Origin 8.5 dalam komputer peribadi. Kajian ini dijalankan adalah untuk mengetahui ciri-ciri plasma seperti suhu elektron dan ketumpatan elektron. Suhu elektron dikira menggunakan kaedah Boltzmann manakala ketumpatan elektron ditentukan dengan menggunakan persamaan Saha-Boltzmann. Disamping itu, kesan daripada masa integrasi keatas isyarat intensiti turut dilakukan dalam projek ini. Perbezaan dalam masa tangguh iaitu 0 μs, 0.1 μs dan 5.0 μs telah digunakan untuk membandingkan bentuk sesuatu garis spektra. Keputusan menunjukkan masa tangguh sebanyak 5.0 μs mempunyai bentuk garis spektra yang bagus berbanding masa tangguh sebanyak 0 μs dan 0.1 μs.

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LIST OF ABBREVIATION

LIBS	-	Laser-Induced Breakdown Spectroscopy
LIPS	-	Laser-Induced Plasma Spectroscopy
LSS	-	Laser Spark Spectroscopy
ICP	-	Inductively Coupled Plasma
DBD	-	Dielectric Barrier Discharge
LTE	-	Local Thermal Equilibrium
FWHM	-	Full Width Half Maximum
Nd:YAG	-	Neodymium-doped Yttrium Aluminium Garnet
CO_2	-	Carbon Dioxide
PMTs	-	Photomultiplier Tubes
CCD	-	Charged-Couple Detector
SNR	-	Signal to-noise-ratio

LIST OF SYMBOL

K	-	Kelvin
ΔE	-	Natural property of energy levels
h	-	Planck constant
T	-	Electron temperature
eV	-	Electron volt
I	-	Intensity
g	-	Statistical weight
A	-	Transition probability
λ	-	Wavelength
k	-	Boltzmann constant

CHAPTER 1

INTRODUCTION

1.1 Research background

Emission spectroscopy is the utilisation of electromagnetic radiation emitted by matter to study quantitatively and qualitatively, the matter or physical processes involved. Atoms, molecules, ions or solid are the examples of matter. In the process of optical emission, there involved a transition from higher energy level to a lower energy level, where the energy loss is converted into radiation. Non-radiative decay happened when there is no radiation emitted and the energy loss is converted into heat during transition.

Collision with other particles and absorbing electromagnetic radiation of particular frequencies are the two methods that can excite an atom to higher energy level. This is achieved by increasing temperature or simply by shining light on it. Optical emission is the case where atoms are excited via high temperature. This is also known as atomic emission. For the case where the atoms are excited via light source, this is called as fluorescence or phosphorescence which is distinguished from the lifetime of excited species. Based on wavelength and intensity of emission spectrum, it provides element or species identification as each elements has different emission spectrum (Hong, 2012).

Optical emission spectrum can even be applied on the study of plasma properties such as plasma temperature and electron density. This principle is used in one of the well established spectroscopic techniques today, the Laser Induced Breakdown Spectroscopy (LIBS) which serves as an essential tool especially in material analysis and analytical chemistry.

LIBS is a system that can do a sampling technique for solids, liquids and gases in research area and industrial applications (Cowpe et al., 2011). Sometimes, it has been called as Laser-Induced Plasma Spectroscopy (LIPS) or Laser Spark Spectroscopy (LSS) but nowadays, the use of LIBS is very common. LIBS has been known in the industrial and scientific applications for the determination of trace component (Aguirre et al., 2012). Increasing the effectiveness of the LIBS technique as a tool for materials analysis implies increasing or maximizing the intensity of the desired atomic emission lines, while decreasing or minimizing the intensity of the measured background emission (Hohreiter et al., 2005).

The setting up of an apparatus to perform a LIBS measurement is very simple compared with many other types of elemental analysis (Cremers et al., 2006). Like other spectroscopic techniques, one peculiarity of LIBS is that material sampling, atomization and excitation occur in the same step. In the specific case of LIBS, the plasma expansion and emission add still more complexity to the analysis, particularly because of light collection issues and the temporal dimension (Sirven et al., 2008).

When the high intensity of pulsed laser beam is focused on the surface of the samples, it will produce plasma if it has a sufficient irradiance (Sarkar, 2010). When the plasma cools down, the atomic species present in the focal volume emit light at their characteristic wavelengths. The plasma emission is collected by the optical fibre and a spectrometer disperses the light emitted by excited atomic and ionic species in the plasma, then the emission signals will be recorded by the detector and it will be digitized and the results are displayed through a computer.

The choice of the operational parameters can affect the analytical performance of LIBS. There are a lot of variables that can affect the measurements such as laser intrinsic properties which include the wavelength, energy, pulse duration and shot-to-shot energy fluctuation. In measurement strategies there are delay time, integration time gate, emission lines selection and amplification detector gain. Besides, the optical design, ambient atmosphere and test sample presentation are also important variables to increase the efficiency of the LIBS system (Jr et al., 2012).

More efforts are still required to increase the efficiency of the LIBS system so that it can produced very quality plasma of any kind of samples. Not all the parameters must be measured in the same time. That is why in this research, quantitative and qualitative analysis is chosen to review the plasma properties of the certain metals.

1.2 Problem statement

A spectroscopic technique based on the relative intensities of lines must be used to measure the properties in plasma. The currently available spectrometer today comes with software which enables users to analyse the spectrum of any samples. For this project, SPECTRASUITE software will be used for data acquisition and it will be analysed by using Origin 8.5 application software.

However, the relationship between the measure of line intensity and the plasma temperature is complex and a number of issues must be examined for the diagnostic. Also, the quantitative and qualitative analysis for each individual peaks from the spectrum is not explained in detail for most researches.

1.3 Objectives of the research

This study embarks on the following objectives:

1) To resemble the Laser Induced Breakdown Spectroscopy (LIBS) system by studying the spectrum lines.
2) To determine the technique of calculating the electron temperature and electron density from the spectra.
3) To identify the atomic species from emission spectra.
4) To compare the pattern of the spectra due to different gate delay.

1.4 Scope of the study

The experimental was set-up to build the LIBS system. The purpose is to get the spectroscopy signal with good signal-to-noise ratios. Second is the plasma generated on the samples where the plasma emits a continuum of radiation which does not contain any useful information about the species present, but within a very small time frame the plasma expands at supersonic velocities and cools. At this point the characteristic atomic emission lines of the elements can be observed. Lastly is the analytical technique studied can be applied to determine the properties of plasma generated on the metals after laser discharged.

1.5 Significance of study

The outcome of this study is important in giving a better understanding on fundamental science knowledge and the properties of atomic spectra. This studied can be applied to various laboratory generated plasma such as inductively coupled plasma (ICP), dielectric barrier discharge (DBD) or thin film deposition. Besides, determination of element’s abundance plays a significant role in astrophysics for instance, stellar plasma diagnostic.

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