COPPER DOPED AND STRONTIUM CO-DOPED LITHIUM MAGNESIUM BORATE GLASS AS THERMOLUMINESCENCE DOSIMETER IRRADIATED BY Co-60

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I dedicate this work specially

To my beloved parents

Salleh bin Muhamad

Azizah binti Jasman

To my lovely siblings

Muhammad Hilmi bin Salleh

Muhammad Syahmi bin Salleh

Muhammad Sadiy bin Salleh

Nurul Izzati binti Salleh

To my kind supervisors

Dr Abd Rahman bin Tamuri

Dr Abd Khamim bin Ismail

Dr Mohammad Alam Saeed

To my supportive friends

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Zaidatul Aslamiyah binti Tumijan

Hanin Athirah binti Harun

Siti Sarah binti Osman

Thank you very much for their love, support and prayers

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ABSTRACT

The present study has been conducted to investigate the thermoluminescence (TL) properties of copper (Cu)-doped and strontium (Sr) co-doped lithium magnesium borate (LMB) glass. Samples of undoped LMB, Cu-doped LMB, Cu-doped and Sr co-doped LMB were prepared using melt-quenching technique and irradiated with Co-60 gamma radiation at dose range of 1–100 Gy. X-Ray Diffraction (XRD) and Thermogravimetry-Differential Thermal Analysis (TG-DTA) showed all the samples are amorphous with good glass stability. All the samples were characterized using Thermoluminescence Dosimeter (TLD) reader and the TL properties were analyzed. In this study, it is found that the co-doped sample has better TL properties compared to the other samples and therefore was chosen for further analysis. The highest glow peak of the sample was observed at temperature of 190°C with the TL intensity of 4.14×10^5 nC g^{-1}. The best annealing procedure of the sample was determined at 100°C for 20 minutes with the heating rate of 7°C s^{-1}. The sample exhibits a good linearity with regression coefficient, R^2 of 0.990 at low dose range of 1–10 Gy and R^2 of 0.995 at high dose range of 10–100 Gy. The sensitivity of the sample is higher at 123 nC mg^{-1} Gy^{-1} in high dose range than that in a low dose range. The sample also shows low fading with the percentage signal loss of 48.2% at a low dose of 5 Gy and 47.7% at a high dose of 50 Gy after 60 days. The sample possesses good reproducibility with the percentage of standard deviation of 4% at 5 Gy and 6% at 50 Gy. Energy Dispersive X-Ray (EDX) analysis was used to calculate the effective atomic number (Z_{eff}) of the sample which is 8.33 close to Z_{eff} of human tissue. The minimum detectable dose of the sample is 2.44 mGy. In addition, the kinetic parameter analysis revealed that the type of glow curve of the sample is a second order kinetic. The activation energy of 1.2 eV and the frequency factor of 3.5×10^{11} s^{-1} were obtained using the initial rise method. Meanwhile, using the peak shape method, the calculated activation energy and frequency factor was 0.7 eV and 2.1×10^8 s^{-1}, respectively. Thus, from this study, it can be concluded that the co-doped sample satisfies most of the desirable TL characteristics as a good material and could be used for thermoluminescence dosimetry application.

ABSTRAK

Kajian ini telah dijalankan untuk mengkaji sifat termopendarcahaya (TL) kaca litium magnesium borat (LMB) berdop kuprum (Cu) dan berko-dop strontium (Sr). Sampel LMB yang tidak berdop, LMB berdop Cu, LMB berdop Cu dan berko-dop Sr telah disediakan dengan menggunakan teknik pelindapan lebur dan didedahkan dengan sinaran gama Co-60 pada julat dos 1–100 Gy. Belauan sinar-X (XRD) dan analisis terma pembezaan-termogravimetri (TG-DTA) menunjukkan semua sampel adalah amorfus dengan kestabilan kaca yang baik. Semua sampel telah dicirikan menggunakan pembaca dosimeter termopendarcahaya (TLD) dan sifat TL telah dianalisis. Dalam kajian ini, sampel berko-dop didapati mempunyai sifat TL yang lebih baik berbanding dengan sampel lain dan oleh itu dipilih untuk analisis selanjutnya. Puncak bara tertinggi sampel telah diperhatikan pada suhu 190^{o}C dengan keamatan TL 4.14×10^{5} nC g^{-1}. Prosedur penyepuhlindapan terbaik sampel telah ditentukan pada 100^{o}C selama 20 minit dengan kadar pemanasan 7^{o}C s^{-1}. Sampel menunjukkan kelinearan yang baik dengan pekali regresi, R^2 0.990 pada julat dos rendah 1–10 Gy dan R^2 0.995 pada julat dos tinggi 10–100 Gy. Kepekaan sampel adalah lebih tinggi iaitu 123 nC mg^{-1} Gy^{-1} pada julat dos yang tinggi berbanding kepekaan pada julat dos yang rendah. Sampel juga menunjukkan kepudaran yang rendah dengan peratusan isyarat hilang 48.2% pada dos rendah 5 Gy dan 47.7% pada dos tinggi 50 Gy selepas 60 hari. Sampel mempunyai kebolehulangan yang baik dengan peratusan sisihan piawai 4% pada 5 Gy dan 6% pada 50 Gy. Analisis sinar-X tenaga tersebar (EDX) digunakan untuk mengira nombor atom berkesan (Z_{eff}) sampel iaitu 8.33 berhampiran dengan Z_{eff} tisu manusia. Dos pengesanan minima sampel ialah 2.44 mGy. Di samping itu, analisis parameter kinetik menunjukkan jenis lengkung bara sampel ialah kinetik tertib kedua. Tenaga pengaktifan 1.2 eV dan faktor frekuensi 3.5×10^{11} s^{-1} telah diperolehi melalui kaedah kenaikan awal. Manakala menggunakan kaedah bentuk puncak, tenaga pengaktifan dan faktor frekuensi yang dikira masing-masing ialah 0.7 eV dan 2.1×10^{8} s^{-1}. Oleh itu, daripada kajian ini, dapat disimpulkan bahawa sampel berko-dop memenuhi kebanyakan daripada sifat TL yang diingini sebagai bahan yang baik dan boleh digunakan dalam penggunaan dosimetri termopendarcahaya.

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LIST OF SYMBOLS

A – Total activity
A_o – Initial activity of the gamma source
A – Area under the glow curve
a_n – Weight fraction of each element
A_W – Atomic weight
b – Order of kinetics
$\overline{B}$ – Mean TL background signal from non-irradiated samples
D – Absorbed dose
D_o – Minimum detectable dose
E – Activation energy
E_f – Energy level
$f(D)$ – Linearity index
F – Calibration factor
$F(D)$ – Dose response at dose D
$F(D_1)$ – Dose response at linear dose D_1
H_R – Hruby parameter
I – Intercept
I – TL Intensity
I_m – Maximum TL Intensity
k – Boltzmann’s constant
m – Concentration of recombination centers
m – Slope from linearity graph
n – Number of trapped electrons
n_o – Number of trapped electrons at $t_o = 0$
N – Trap concentration
N_A – Avogadro’s number

p – Probability escape of an electron

R – Recombination center

R^2 – Regression coefficient

s – Frequency factor

s' – Pre-exponential factor

$S(D)$ – Sensitivity

$S_r(D)$ – Relative sensitivity

t – Time

$t_{1/2}$ – Half-time

T – Temperature

T_c – Cut-off temperature

T_c – Crystallization temperature

T_g – Transition temperature

T_m – Melting temperature

T_{rg} – Glass forming ability

T_1 – The lower temperature of the T_m corresponding to the half peak intensity

T_2 – The higher temperature of the T_m corresponding to the half peak intensity

T_m – Peak temperature

W_i – Fractional weight

Z – Atomic number

Z_{eff} – Effective atomic number

Z_n – Atomic number of each element

β – Heating rate

τ – Mean lifetime

σ_B – Standard deviation from mean background signal

ω – Total half intensity width

δ – High temperature half width

τ – Low temperature half width

μ_g – Geometrical factor

LIST OF ABBREVIATIONS

CB	–	Conduction band
EDX	–	Energy Dispersive X-Ray
EPD	–	Electronic personal dosimeter
IEC	–	International Electrotechnical Commission
IR	–	Initial rise
LMB	–	Lithium magnesium borate
MDD	–	Minimum detectable dose
MOSFET	–	Metal-oxide semiconductor field-effect transistor
PC	–	Personal computer
PMT	–	Photomultiplier tube
RE	–	Rare Earth
SEM	–	Scanning Electron Microscope
STA	–	Simultaneous Thermal Analyzer
TG-DTA	–	Thermogravimetry-Differential Thermal Analysis
TL	–	Thermoluminescence
TLD	–	Thermoluminescence dosimeter
TTP	–	Time temperature setting
UKM	–	Universiti Kebangsaan Malaysia
UPMU	–	University Laboratory Management Unit
UTM	–	Universiti Teknologi Malaysia
VB	–	Valence band
WinREMS	–	Windows Radiation Evaluation and Management System
XRD	–	X-Ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of study

Ionizing radiation has been extensively utilized in several fields such as in medical and industrial applications. However, the risk of ionizing radiation exposure is very harmful to human life and environment, so that it is important to have a device that can monitor and measures the amount of ionizing radiations. There are many personal dosimeters for ionizing radiation such as electronic personal dosimeter (EPD), metal-oxide-semiconductor field-effect transistor (MOSFET) dosimeter, film badge dosimeter, quartz fiber dosimeter and thermoluminescence dosimeter (TLD). TLD is most widely used to measure the amount of ionizing radiation dose and it is known as the cost-effective technique of radiation dosimetry. It has been used for environmental and personnel radiation monitoring in the last three decades (Kortov, 2010). TLD is also used in many fields such as human radiation protection, radiotherapy clinic, industrial and environmental applications. In personnel dosimetry, the employees who work in the radiation area are provided with TLD to measure the amount of radiation. This measurement is important for occupational health so that the safety protection can be taken into considerations (Pekpak *et al.*, 2010).

The efficiency of TLD depends on the type of dosimeter materials. The preferable thermoluminescence (TL) materials are the one that possess good TL properties such as a simple glow curve peak in the range of 180–250 ^{o}C, simple annealing procedure, high sensitivity and stability, good linearity in wide dose range, low fading, good reproducibility and has effective atomic number (Z_{eff}) which is 7.42 close to that of the human tissue. Besides that, the materials should have a high precision, high accuracy and good resistance against environmental factors such as light, humidity, gases and moisture. The materials also should be strong, chemically inert and radiation resistant (Pradhan, 1981, Furetta, 2003, Kortov, 2007).

There are a few of commercially TL materials such as TLD–100 (LiF: Mg, Ti), TLD–700H (LiF: Mg, Cu, P), TLD–800 ($Li_2B_4O_7$: Cu, Ag, P), TLD–900 ($CaSO_4$: Dy) and TLD–500 (Al_2O_3: C) (Fox *et al.*, 1988; Noh *et al.*, 2001). Lithium fluoride (TLD–100) is a commercial TLD and used the most in radiation dosimetry due to its excellent TL performance with high reproducibility and good tissue equivalence (Khan, 2014). However, this material has many disadvantages such as a supralinearity at high doses rate, complicated annealing behavior, hygroscopic and storage instability (Juan *et al.*, 2008). Therefore, extensive researches have been carried out to improve the dosimetric properties of TL materials.

Recently, various studies have been conducted to find out the suitable and better TL dosimeter materials. Glasses have advantage over other forms of TL materials such as chips, powder, disks, pellets, rods or Teflon form because of its easy preparation, low cost, long term stability, high sensitivity, chemically inert and good absorption among others (Depci *et al.*, 2008; Engin *et al.*, 2010). The borate glass is one of the excellent TL materials compared to other glasses due to its effective atomic number that is very close to the atomic number of soft tissue (Z_{eff}=7.42) where it is used in radiation dosimetry and clinical applications (Tekin *et al.*, 2010). Also, following properties of borate glass with high sensitivity, good transparency, high chemical durability and thermal stability, low cost, and easy preparation make it suitable TL material (Elkholy, 2010; Jiang *et al.*, 2010). However, pure borate glass is limited to be used as radiation dosimetry due to its hygroscopic nature (Santiago *et al.*, 2001). Therefore, to overcome this drawback, modifiers or activators should be added to the glass network.

The addition of different modifiers such as transition metals, rare earths, alkali and alkaline earth metals in glass could improve the intensity and enhance the chemical durability of the borate glass. It can also interrupt the glass network and reduce the viscosity of the glass (Hussin *et al*., 2010; Jiang *et al*., 2010). Besides that, the addition of modifiers can reduce the hygroscopic nature of borate glass. This is very important for TL dosimeters because water can cause an adverse effect on the TL efficiency of the material associated with non-radioactive relaxations during thermal stimulation (McKeever, 1995). The borate glasses are relatively chemically stable compound and show no problems to be doped with impurities and can performed better if modified by metal oxides (Aboud *et al*., 2012).

Lithium borate is mostly used due to its low effective atomic number (Z_{eff}=7.3) that is near to human tissue, low cost and easy annealing procedure (Furetta *et al*., 2001). The addition of magnesium oxide (MgO) as a second modifier can reduce the hygroscopic nature of the borate glass and increase the strength of the glass (Mhareb *et al*., 2014). Copper is the most efficient transition metal as an activator in borate glass that shows excellent TL properties with high sensitivity, low fading and linearity in wide dose range (Singh *et al*., 2011; Alajerami *et al*., 2014). The presence of co-dopant in borate glass is used to overcome the quenching state, improve the stability and enhance the TL sensitivity of borate glass. It is also can create deeper and stable traps on the host of borate glass (Alajerami *et al*., 2013d).

In this study, the thermoluminescence properties of undoped, copper-doped and strontium co-doped lithium magnesium borate glass samples were investigated. The thermoluminescence response of lithium magnesium borate glass (LMB) doped with copper (Cu) and co-doped with strontium (Sr) is expected to enhance the TL sensitivity and improve the TL performance of studied samples. This newly proposed TL material could be a potential candidate to be used in TLD in the future.

1.2 Problem statement

The studies on the borate glass dosimeters become more interesting nowadays due to its high sensitivity, good thermal stability and mechanical properties. Nowadays, many studies have been conducted to find a better dosimeter material to improve the properties of the borate dosimeters. Several impurities such as lithium, magnesium, potassium, sodium and zinc were used to improve the physical and TL properties of borate glass. LMB glass was considered as a good TL material because of their tissue equivalent near to human tissue, good linearity, low fading and good reproducibility.

There are various studies that made modification on lithium magnesium borate glass with the addition of modifiers or activators such as alkali and alkaline earth metals, transition metals and rare earths for better results. So far, from the previous work, the studies on the physical, optical and thermoluminescence properties concerned on lithium magnesium borate doped rare earths such as Tb, Gd, Dy, Pr, Mn, Ce, Eu, Sm and Nd (Anishia *et al*., 2011; Alajerami *et al*., 2012b; Reduan *et al*., 2014; Mhareb *et al*., 2014; Hashim *et al*., 2015; Mhareb *et al*., 2015).

For this research, many trials have been conducted to find suitable material to be co-doped into Cu-doped LMB glass such as Ce, Nd, Sm, Eu, Dy, Er. However, these rare earths material did not produce good TL response. Therefore, Sr co-doped was chosen after many trials that has good TL response. The studies of copper-doped and strontium co-doped lithium magnesium borate glass as TL material was limited and it is still lacking. The investigations on the effect of copper and strontium into lithium magnesium borate glass are expected to enhance the TL response and improve the TL properties of the borate glass.

Thus, newly proposed TL materials of Cu-doped and Sr co-doped LMB glass subjected to Co-60 gamma irradiation in this study is an attempt to provide new fundamental knowledge of the TL properties such as glow curve, linearity, sensitivity, fading, reproducibility, minimum detectable dose, kinetic parameters and effective atomic number that can be used for personnel and environmental applications.

1.3 Objectives of study

The objectives of this study:

i. To determine the optimum concentration of undoped, Cu-doped and Sr co-doped LMB glass.

ii. To determine the structure, samples composition and glass stability of undoped, Cu-doped and Sr co-doped LMB glass.

iii. To determine the optimum annealing temperature, annealing time and heating rate of undoped, Cu-doped and Sr co-doped LMB glass.

iv. To determine the TL dosimetric properties of undoped, Cu-doped and Sr co-doped LMB glass subjected to Co-60 gamma irradiation.

1.4 Scope of study

In this study, the undoped LMB, Cu-doped and Sr co-doped LMB glass was prepared using the melt-quenching technique. The optimum annealing temperature (100, 200, 300 and 400 ^{o}C), annealing time (10, 20, 30, 40, 50 and 60 minutes), heating rates (1, 3, 5, 7 and 10 ^{o}C s^{-1}) and concentration of the prepared samples were determined. All the samples were irradiated with Co-60 gamma ray at a dose range of 1–100 Gy at Universiti Kebangsaan Malaysia (UKM). The characterizations of the samples were studied to determine the structure of the glass samples by using X-Ray Diffraction (XRD) technique, the elemental composition of the samples was identified by Energy Dispersive X-Ray (EDX) technique and the glass stability was determined using Thermogravimetry-Differential Thermal Analysis (TG-DTA). The TL response of irradiated samples was read out using TLD reader model Harshaw 4500 at Nuclear Laboratory, Universiti Teknologi Malaysia (UTM). The TL properties such as glow curve characteristics, annealing procedure, linearity, sensitivity, fading, reproducibility, minimum detectable dose, effective atomic number and kinetic parameters were investigated in this study.

1.5 Significance of study

The present study on Cu-doped and Sr co-doped LMB may exploit the new knowledge and additional information for better understanding about the TL properties of borate glass. The presence of activators with the addition of Cu as dopant and Sr as co-dopant into the LMB glass may enhance the TL response and improve the properties of borate glass. The development of new TL material reveals to have better performance that can be used as radiation dosimetry.

1.6 Thesis outline

Chapter 1 is an introduction of the study that consists of a background of the study, problem statement, objectives of the study, the scope of study, the significance of study and thesis outline. Chapter 2 describe the definition of glass and component of oxide glass. This chapter also briefly discuss the definition of thermoluminescence and TL phenomena. The glow curve theory, thermoluminescence properties and overview of TL materials are also explained in details. Chapter 3 describes the experimental methods and procedures used in this work. It also includes the material identification, preparations of glass and sample characterizations using X-Ray Diffraction (XRD), Energy Dispersive X-Ray (EDX) and Thermogravimetry-Differential Thermal (TG-DTA). The thermoluminescence properties measurements are also explained in this chapter. Chapter 4 presents the discussion and results obtained from the undoped, copper-doped and strontium co-doped lithium magnesium borate glass systems. Lastly, Chapter 5 provides the conclusion of the study with the recommendations and suggestions about the future works.

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