OPTICAL AND PHYSICAL PROPERTIES OF EUROPIUM DOPED LITHIUM POTASSIUM BORATE GLASS

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I dedicate this work

To my dear parents

Whose love, kindness, patience and prayer have brought me this far

To my siblings

To my wife

For their endless laughs and tears

To my nieces and nephews

Whose presence fills my life with joy

To my friends

For their love, understanding and support through my endeavour

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ABSTRACT

Borate glass is widely used in many scientific studies. By using melt-quenching technique five samples of lithium potassium borate (LKB) doped with different concentration of europium oxide (Eu_2O_3) were prepared. To investigate the influence of dopant on the optical and physical characteristics of the glass, X-ray Diffraction and photoluminescence analyses were performed. The amorphous nature was confirmed by X-ray diffraction. The physical parameters of the glass which was doped by different oxidation state have been analyzed. These parameters involved are density, molar volume, ion concentration, inter-nuclear distance and Polaron radius. The exchange in the concentration of Eu^{+3} indicated the influence of Eu as a dopant on the photoluminescence emission of LKB glasses. The photoluminescence emission spectrum of LKB:Eu^{+3}were due to the transition of Eu^{3+} from$^5D_0$-^7F_r (r =1, 2, 3 and 4). The luminescence studies showed four peaks (590 nm, 613 nm, 650 nm, and 698 nm) for all samples excluding the pure sample. The glow curve exhibits single peak at 164 ºC. We establish that the proposed TL dosimeter at 0.5 mol% of Eu^{3+} has been observed to be 20 times less sensitive than TLD-100.

ABSTRAK

Kaca borat banyak digunakan dalam kajian saintifik. Dengan menggunakan teknik sepuh-lindap, 5 sampel litium potassium borat yang dengan europium oksida (Eu_2O_3) yang mempunyai kepekatan yang berbeza disediakan. Bagi mengkaji kesan dopan terhadap ciri-ciri optikal dan fizikal bagi kaca, analisis pembelauan sinar-X dan fotoluminesens telah dilakukan. Sifat amorfus bahan telah dibuktikan dengan pembelauan sinar-X. Parameter fizikal bagi kaca yang didop dengan aras pengoksidaan berbeza dianalisis. Parameter yang terlibat adalah ketumpatan, isipadu molar, kepekatan ion, jarak antara nukleus dan jejari Polaron. Perubahan kepekatan Eu^{3+} menunjukkan kesan Eu sebagai dopan dalam pancaran fotoluminesens kaca LKB. Pancaran spektra luminesens disebabkan peralihan Eu^{3+} dari $^5D_0$-^7F_r (r=1, 2, 3 dan 4). Kajian luminesens menunjukkan 4 puncak (590 nm, 613 nm, 650 nm dan 698 nm) untuk semua sampel kecuali sampel tulen. Satu lengkung berbara menunjukkan puncak tunggal pada 164°C. Dosimeter TL yang dicadangkan ini menunjukkan kepekaan 20 kali lebih rendah berbanding TLD-100 pada kepekatan 0.5 mol % Eu^{3+}.

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λ	Wavelength
n	Integer, Refractive index
d	Distance between atomic layers in a crystal
θ	Incedant plan wave
P	Probability of escaping from the trap
E	Energy
s	constant
k	Boltzman constant
ρ	Density
a	Weight of glass sample in the air
b	Weight of glass sample in the toluene
V_m	The molar volume
M	Molecular wiehgt
N	Ion concentration
r_p	Polaron radius
r_i	Inter-nuclear distance
E_g	Energy gap
f_{exp}	Oscillator strengths
ε	Molar absorption coefficient
Δv	The width of the band at half the peak intensity

LIST OF ABBREVIATIONS

TLD	Thermoluminescence dosimeters
TL	Thermoluminescence
PMT	Photomultiplier
ESR	Electron spin resonance
STE	Self-trapped exciton
DFT	Density functional theory
LINAC	Linear accelerator
RF	Radio frequency
PMMA	Polymethylmethacrylate
MU	Monitor Unit
SSD	Source to surface distance
ICRP	International Commission on RadiologicalProtection
SEM	Scanning electron microscope
NDT	Non-destructive

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CHAPTER 1

INTRODUCTION

1.1 Overview

The solids are subdivided into two groups depending on their network structure. The crystalline solids have reticular structure and atoms arranged in three dimensional periodic. The other kind of solids is amorphous (non-crystalline) which have no reticular structure and atoms packing fairly random. The common examples of amorphous are plastic and glass.

The energy required to arrange atoms in irregular packing is larger than that of regular packing, so nature favours the crystalline state and most of the solids in the universe exist in this case. Nearly all material can form glass by changing a network structure form from regular to irregular packing by heating the material to its melting temperature then reduce the temperature sufficiently fast to ensure atoms don't have enough time to rearrange themselves, this is called random network theory (Turnbull, 1969). There are many types of glass that are known recently such metallic glass, halide glass, magnetic glass, commercial glass, oxyhalide glass, and oxide glass.

There are many techniques which can be used for producing glass. The most prevalent method called melt quenching technique which is based on melting a metal oxide then increase the viscosity very quickly, so the crystal growth could not occur.

The most important point in this technique is the ratio between melting and cooling temperature different from material to another.

Some minerals such as fluorite emit a transient glow when temperature increases in darkness, this phenomenon called thermoluminescence. In addition, this phenomenon becomes one of most important research topics especially in the dosimetry field (Oberhoffer *et al.*, 1981).

Denial and his-workers in the 1940s, were used this phenomena in order to measure the amount of radiation exposure and they concluded (LiF) was the most appropriate compound for evaluating ionizing radiation exposure (McKinlay, 1981).

Currently, there are extensive studies on a borate glass subject in industries to develop technological products based on thermoluminescence properties due to its lower cost compared to other compositions. It also owns high sensitivity and easy to prepare (Jiang *et al*, 2009).

Recently there are many studies about thermoluminescence for borate glass to be used in dosimeter in several fields such as industry and medical fields. Moreover, preliminary studies have shown the possibility of TL characteristic exploitation such as TLD.

Amendments in borate glass composition are going to be executed in our work by adding other oxide alkali on this composite to get the proposed network formed. Such network will be achieved by changing the structure of glass borate. The goal of this modified structure is for reinforcing the glass properties and consequently release some of undesirable properties like unstable alone, easily crystallized and got hygroscopic properties (Hussin *et al.*, 2009).

1.2 Background of the problem

Recently, different radiation source became important in various industrial and medical applications. In order to work in a safe environment and avoid or reduce radiation risk especially for who work in this environment and exposed to rays continually, for this point measure quantity of radiation become a requirement. For this purpose, badge was created from several materials that have got special properties carried out by radiation worker specialists. It is known as a film dosimeter and is calibrated by determine radiation dose, the first one was produced is Eggerd and Luft (1929).

The thermoluminescence dosimetry (TLD) becomes widespread in measurement radiation dose, due to its high sensitivity with small size. (Mayles *et al*, 2007). Most previous studies of thermoluminescence focused on developing and investigate optical fibre in the commercial uses as TLD material. Comparatively there are a few thermoluminescence researches on the glass being reported.

In radiation detector not all material can be used, it's depending on the thermoluminescence properties. The properties of borate glass such as effective atomic number (Z_{eff}= 7.42), chemical stability, capability to accept transition metal ions, and the attractive nonlinear optical properties encouraged researchers to study the possibility to use it as a TLD (Rao, 2002).

1.3 Problem statement

The researchers are using the borate system especially binary borate referring to its attractive properties. Unfortunately, lithium potassium borate is studied by a small number of researchers. Furthermore, they are narrowed specific features and doping with rare earth is not much studied. The current study aims to investigate the structure characteristic and optical properties of the undoped and doped lithium potassium borate glass as well as the effect of concentration europium ion on luminescence properties.

1.4 Objectives

This study embarks on the following goals:

i) To study the optical properties of lithium potassium borate doped with europium.

ii) To identify the physical properties of the new prepared glass.

iii) To determine the thermoluminescence glow curve and sensitivity of lithium potassium borate doped and un-doped samples.

1.5 Scope of the research

The current study subdivided into some scopes in order to achieve the stated objectives as follow:

i) Preparation of glass samples from lithium potassium borate doped with europium by using melt quenching technique.

ii) Use x-ray diffraction to identify the amorphous phase of the prepared glass samples.

iii) To investigate the absorption properties of the obtained glass by using UV-vis spectroscopy.

iv) Determination of excitation and emission spectra of the samples by using Photoluminescence Spectroscopy.

v) Evaluation some of physical parameters for doping and un-doping lithium potassium borate glass

vi) Investigate the thermoluminescence characteristics for the samples by exposing to photon radiation source.

1.6 Significant of the study

The current study has been done to promote the understanding of the luminescence and thermoluminescence characteristics of the glass. Furthermore, doping samples by Eu^{3+} may develop as a new material.

1.7 Organization of the dissertation

Chapter 1, borate glass system studied by many researchers, overview about the results are carried out in this chapter to establish a fundamental knowledge for present research. Lithium potassium borate glass doped with europium is proposed to become new thermoluminescence material. Three main objectives are listed in this chapter to be accomplished.

Chapter 2 describes briefly the basic theory of glass preparation. The attractive properties for host, modifier, and activators are briefly presented. Furthermore, this explains the thermoluminescence phenomena and presented the thermoluminescence dosimetry system.

In chapter 3, the glass preparation process and the methodology used to investigate the optical and physical properties are described in details. The techniques that being used for the characterization are PL, UV, XRD, ion concentration, and molar volume.

Chapter 4, the results of the current study were presented in this chapter. PL, UV, and thermoluminescence were carried out and discussed. The amorphous phase was confirmed by XRD.

Finally chapter 5 presents the concluding remarks and suggests some recommendation for future study.

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APPENDIX

CALCULATION OF GLASS COMPOSITION

$Li_2CO_3 + B_2O_3 + K_2CO_3 \longrightarrow B_2O_3 + K_2O + Li_2O + 2CO_2$

The quantity needed to prepare glasses sample is calculated as below:

Molar mass of Li_2CO_3	= 73.8910 g/mol
Molar mass of Li_2O	= 29.8800 g/mol
Molar mass of B_2O_3	= 69.6202 g/mol
Molar mass of K_2O	= 94.20 g/mol
Molar mass of K_2CO_3	= 138.2055 g/mol

Weight system, Wsys	= (0.7 x 69.6202) + (0.1 x 94.20) + (0.2 x 29.8800)
	= 48.7341 + 9.420 + 5.976
	= 64.1301 g/mol
Total mass	= 15g

For B_2O_3, Mass = $\frac{48.7341}{64.1301} \times 15g = 11.3989$ g

For K_2O, Mass = $\frac{9.420}{64.1301} \times 15g = 2.2033$ g

For Li_2O, Mass = $\frac{5.976}{64.1301} \times 15g = 1.3984$ g

In this project, potassium oxide and lithium oxide are made by heating K_2CO_3 and Li_2CO_3 to above 825 °C and liberate a molecule of carbon dioxide (CO_2); leaving potassium oxide.

$$K_2CO_3\ (p) \xrightarrow[\Delta]{} K_2O\ (p) + CO_2\ (g)$$

$$Li_2CO_3\ (p) \xrightarrow[\Delta]{} Li_2O\ (p) + CO_2\ (g)$$

To get the mass for K_2CO_3 and Li_2CO_3,

$$\frac{100}{\frac{Molar\ mass\ of\ \mathrm{Li2O}}{Molar\ mass\ of\ \mathrm{Li2CO3}}\ x\ 100\%} \times \frac{100}{Grade\ Purity\ of\ \mathrm{Li2CO3}} \qquad \text{Eq.1}$$

$$\frac{100}{\frac{Molar\ mass\ of\ K2O}{Molar\ mass\ of\ K2CO3}\ x\ 100\%} \times \frac{100}{Grade\ Purity\ of\ K2CO3} \qquad \text{Eq.2}$$

Li_2CO_3 = Mass of Li_2O × Eq.1

K_2CO_3 = Mass of K_2O × Eq.2

Molar mass of Li_2CO_3 = 73.8910 g/mol

Hence, the mass Li_2CO_3 = $1.3984 \times \frac{100}{40.4379} \times \frac{100}{99} = 3.4930$ g

Molar mass of K_2CO_3 = 138.2055 g/mol

Hence, the mass K_2CO_3 = $2.2033\ x\ \frac{100}{68.1593} \times \frac{100}{99} = 3.2652$ g

Doped 0.3

$$Li_2CO_3+B_2O_3+K_2CO_3+Eu_2O_3 \longrightarrow B_2O_3+K_2O+Li_2O+2CO_2+Eu_2O_3$$

0.2 + 0. 7 + 0.095 + 0.005

The quantity needed to prepare glasses sample is calculated as below:

Molar mass of Li_2CO_3	= 73.8910 g/mol
Molar mass of Li_2O	= 29.8800 g/mol
Molar mass of B_2O_3	= 69.6202 g/mol
Molar mass of K_2O	= 94.2000 g/mol
Molar mass of K_2CO_3	= 138.2055 g/mol
Molar mass of Eu_2O_3	= 351.926 g/mol
Weight system, Wsys	$= (0.7 \times 69.6202) + (0.097 \times 94.20) + (0.2 \times 29.8800)$
	$+ (0.003 \times 351.926)$
	= 48.7341 + 9.1374+5.976+1.0558
	= 64.9033 g/mol
Total mass	= 15g

For B_2O_3, Mass $= \frac{48.7341}{64.9033} \times 15\text{g} = 11.2630$ g

For K_2O, Mass $= \frac{9.1374}{64.9033} \times 15\text{g} = 2.1118$ g

For Li_2O, Mass $= \frac{5.976}{64.9033} \times 15\text{g} = 1.3811$ g

For Eu_2O_3, Mass $= \frac{1.0558}{64.9033} \times 15\text{g} = 0.2440$ g

In this project, potassium oxide and lithium oxide are made by heating K_2CO_3 and Li_2CO_3 to above 825 °C and liberate a molecule of carbon dioxide (CO_2); leaving potassium oxide.

$$K_2CO_3\ (p) \xrightarrow[\Delta]{} K_2O\ (p) + CO_2\ (g)$$

$$Li_2CO_3\ (p) \xrightarrow[\Delta]{} Li_2O\ (p) + CO_2\ (g)$$

To get the mass for K_2CO_3 and Li_2CO_3,

Li_2CO_3 = Mass of Li_2O × Eq. 1

K_2CO_3 = Mass of K_2O × Eq. 2

Molar mass of Li_2CO_3 = 73.8910 g/mol

Hence, the mass $Li_2CO_3 = 1.3811 \times \frac{100}{40.4379} \times \frac{100}{99} = 3.4498$ g

Molar mass of K_2CO_3 = 138.2055 g/mol

Hence, the mass $K_2CO_3 = 2.1118 \times \frac{100}{68.1593} \times \frac{100}{99} = 3.1296$ g

Doped 0.5

$$Li_2CO_3+B_2O_3+K_2CO_3+Eu_2O_3 \longrightarrow B_2O_3+K_2O+Li_2O+2CO_2+Eu_2O_3$$

0.2 + 0. 7 + 0.095 + 0.005

The quantity needed to prepare glasses sample is calculated as below:

Molar mass of Li_2CO_3	= 73.8910 g/mol
Molar mass of Li_2O	= 29.8800 g/mol
Molar mass of B_2O_3	= 69.6202 g/mol
Molar mass of K_2O	= 94.20 g/mol
Molar mass of K_2CO_3	= 138.2055 g/mol
Molar mass of Eu_2O_3	= 351.926 g/mol

Weight system, Wsys = (0.7 x 69.6202) + (0.095 x 94.20) + (0.2 x 29.8800) + (0.005 x 351.926)

= 48.7341 + 8.9490 +5.976+1.7596

= 65.4187 g/mol

Total mass = 15g

For B_2O_3, Mass = $\frac{48.7341}{65.4187} \times 15g = 11.1743$ g

For k_2O, Mass = $\frac{9.1374}{64.9033} \times 15g = 2.0519$ g

For Li_2O, Mass = $\frac{5.976}{64.9033} \times 15g = 1.3703$ g

For Eu_2O_3, Mass = $\frac{1.0558}{64.9033} \times 15g = 0.4035$ g

In this project, potassium oxide and lithium oxide are made by heating K_2CO_3 and Li_2CO_3 to above 825 °C and liberate a molecule of carbon dioxide (CO_2); leaving potassium oxide.

$$K_2CO_3\ (p) \xrightarrow[\Delta]{} K_2O\ (p) + CO_2\ (g)$$

$$Li_2CO_3\ (p) \xrightarrow[\Delta]{} Li_2O\ (p) + CO_2\ (g)$$

To get the mass for K_2CO_3 and Li_2CO_3,,

Li_2CO_3 = Mass of Li_2O × Eq. 1

K_2CO_3 = Mass of K_2O × Eq. 1

Molar mass of Li_2CO_3 = 73.8910 g/mol

Hence, the mass $Li_2CO_3 = 1.3703 \times \frac{100}{40.4379} \times \frac{100}{99} = 3.4224$ g

Molar mass of K_2CO_3 = 138.2055 g/mol

Hence, the mass K_2CO_3 = 2.0519 × $\frac{100}{68.1593}$ × $\frac{100}{99}$= 3.0408 g

Doped 0.7

$Li_2CO_3+B_2O_3+K_2CO_3+Eu_2O_3 \longrightarrow B_2O_3+K_2O+Li_2O+2CO_2+Eu_2O_3$

0.2 + 0. 7 + 0.093 + 0.007

The quantity needed to prepare glasses sample is calculated as below:

Molar mass of Li_2CO_3	= 73.8910 g/mol
Molar mass of Li_2O	= 29.8800 g/mol
Molar mass of B_2O_3	= 69.6202 g/mol
Molar mass of K_2O	= 94.20 g/mol
Molar mass of K_2CO_3	= 138.2055 g/mol
Molar mass of Eu_2O_3	= 351.926 g/mol

Weight system, Wsys	= (0.7 x 69.6202) + (0.095 x 94.20) + (0.2 x 29.8800) + (0.007 x 351.926)
	= 48.7341 + 8.7606+5.976+2.4635
	= 65.9342 g/mol
Total mass	= 15g

For B_2O_3, Mass = $\frac{48.7341}{65.9342}$ × 15g = 11.0869 g

For k_2O, Mass = $\frac{8.7606}{65.9342}$ × 15g = 1.9930 g

For Li_2O, Mass = $\frac{5.976}{65.9342}$ × 15g = 1.3595 g

For Eu_2O_3, Mass = $\frac{2.4635}{65.9342}$ × 15g = 0.5604 g

In this project, potassium oxide and lithium oxide are made by heating K_2CO_3 and Li_2CO_3 to above 825 °C and liberate a molecule of carbon dioxide (CO_2); leaving potassium oxide.

$$K_2CO_3\ (p) \xrightarrow[\Delta]{} K_2O\ (p) + CO_2\ (g)$$

$$Li_2CO_3\ (p) \xrightarrow[\Delta]{} Li_2O\ (p) + CO_2\ (g)$$

To get the mass for K_2CO_3 and Li_2CO_3,,

Li_2CO_3 = Mass of Li_2O × Eq. 1

K_2CO_3 = Mass of K_2O × Eq. 2

Molar mass of Li_2CO_3 = 73.8910 g/mol

Hence, the mass Li_2CO_3 = $1.3595 \text{ x } \frac{100}{40.4379} \text{ x } \frac{100}{99}$ = 3.3960 g

Molar mass of K_2CO_3 = 138.2055 g/mol

Hence, the mass K_2CO_3 = $1.993 \text{ x } \frac{100}{68.1593} \text{ x } \frac{100}{99}$ = 2.9535 g

Doped 1.0

$$Li_2CO_3+B_2O_3+K_2CO_3+Eu_2O_3 \longrightarrow B_2O_3+K_2O+Li_2O+2CO_2+Eu_2O_3$$

0.2 + 0. 7 + 0.09 + 0.01

The quantity needed to prepare glasses sample is calculated as below:

Molar mass of Li_2CO_3 = 73.8910 g/mol

Molar mass of Li_2O = 29.8800 g/mol

Molar mass of B_2O_3 = 69.6202 g/mol

Molar mass of K_2O = 94.20 g/mol

Molar mass of K_2CO_3 = 138.2055 g/mol

Molar mass of Eu_2O_3 = 351.926 g/mol

Weight system, Wsys = (0.7 x 69.6202) + (0.090 x 94.20) + (0.2 x 29.8800) + (0.010 x 351.926)

= 48.7341 + 8.4780+5.976+3.5192

= 66.7073 g/mol

Total mass = 15g

For B_2O_3, Mass = $\frac{48.7341}{66.7073}$ x 15g = 10.9584 g

For k_2O, Mass = $\frac{9.1374}{66.7073}$ x 15g = 1.9063 g

For Li_2O, Mass = $\frac{5.976}{66.7073}$ x 15g = 1.3437 g

For Eu_2O_3, Mass = $\frac{1.0558}{66.7073}$ x 15g = 0.7913 g

In this project, potassium oxide and lithium oxide are made by heating K_2CO_3 and Li_2CO_3 to above 825 °C and liberate a molecule of carbon dioxide (CO_2); leaving potassium oxide.

$$K_2CO_3\ (p) \xrightarrow[\Delta]{} K_2O\ (p) + CO_2\ (g)$$

$$Li_2CO_3\ (p) \xrightarrow[\Delta]{} Li_2O\ (p) + CO_2\ (g)$$

To get the mass for K_2CO_3 and Li_2CO_3,

Li_2CO_3 = Mass of Li_2O × Eq. 1

K_2CO_3 = Mass of K_2O × Eq. 2

Molar mass of Li_2CO_3 = 73.8910 g/mol

Hence, the mass Li_2CO_3 = 1.3437 × $\frac{100}{40.4379}$ × $\frac{100}{99}$= 3.3564 g

Molar mass of K_2CO_3 = 138.2055 g/mol

Hence, the mass K_2CO_3 = 1.9063 × $\frac{100}{68.1593}$ × $\frac{100}{99}$= 2.8250 g