THE STRUCTURAL AND LUMINESCENCE PROPERTIES OF STRONTIUM BOROTELLURITE GLASS DOPED WITH EUROPIUM AND DYSPROSIUM

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Science (Physics)

Faculty of Science
Universiti Teknologi Malaysia

APRIL 2014

Dedicated to my beloved parents and siblings,

for their endless love, supports and encouragements.

ACKNOWLEDGEMENT

I would like to express my deepest gratitude to Prof. Dr. Rosli Bin Hussin who have introduce me to glass science and also being friendly, supportive and inspiring supervisor throughout my research. I am thankful to my parents and siblings for their continuous love and hard work that allow me to pursue study.

I am also indebted to Ministry of Higher Education and Universiti Teknologi Malaysia for funding my studies through Mybrain15 and research grant. My special thanks go to Mr. Jaafar and Mrs. Anisah for helping in Material Science Lab (Physics, UTM) and also thanks to all my friends especially in Material Science Lab (Physics, UTM) and lecturers for sharing ideas. Last, but certainly not least, I thank Materials Science Lab (FKM, UTM), FT-IR Lab (Chemistry, UTM) and CRIM (UKM) for providing instruments for measurements.

ABSTRACT

A series of strontium borotellurite glasses with the general formula xSrO·(100-x)[0.5B_2O_3·0.5TeO_2], ($15 \leq x \leq 35$ mol%) have been prepared. In addition, glass samples with the composition 20SrO·40B_2O_3·40TeO_2 and 30SrO·35B_2O_3·35TeO_2 doped with Europium, Eu^{3+} and Dysprosium, Dy^{3+}, (1 mol% each) were also prepared. All the glass samples were prepared using melt quenching method, followed by annealing process at 400 ^{o}C for 6 hours. The structural property of the glass samples was characterized using X-ray diffractometer (XRD) and Fourier transform infrared spectrometer and the luminescence property using fluorescence spectrometer. The XRD diffraction patterns showed the amorphous phase of the glass sample. Infrared spectra reveals that increased of strontium in borotellurite glass significantly promote conversion of [BO_3] to [BO_4] and [TeO_4] to [TeO_3] in the structural units. This conversion indicated some disintegration of boroxol rings into network structures while strontium acted as network modifier. The emission spectra line of undoped and doped glasses is in the visible range. The emission spectra line of undoped glass at 513 nm originated from the glass host. The emission spectra lines from Eu^{3+} doped glass are due to transition of Eu^{3+} ion at $^5D_0 \rightarrow ^7F_0$ (580 nm), $^5D_0 \rightarrow ^7F_1$ (593 nm), $^5D_0 \rightarrow ^7F_2$ (613 nm) and $^5D_0 \rightarrow ^7F_3$ (652 nm). Meanwhile the emission spectra lines from Dy^{3+} doped glass are due to transition from $^4F_{9/2} \rightarrow ^6H_{15/2}$ (483 nm), $^4F_{9/2} \rightarrow ^6H_{13/2}$ (578 nm) and $^4F_{9/2} \rightarrow ^6H_{11/2}$ (660 nm). The emission spectra lines from Eu^{3+} and Dy^{3+} doped glass are shown at 483 nm, 513 nm, 578 nm, 613 nm and 660 nm. The results revealed that the emission intensity of Eu^{3+} and Dy^{3+} doped glass are dependent on host composition. Furthermore, the luminescence decay curve for Eu^{3+} doped glass showed multi-exponential decay with lifetimes for 20SBT:1Eu^{3+} glass are 1 ns and 22 ns, while for 30SBT:1Eu^{3+} glass, the lifetimes are 1 ns and 18 ns.

ABSTRAK

Satu siri kaca strontium borotellurit dengan persamaan umum xSrO·(100-x)[0.5B_2O_3·0.5TeO_2], ($15 \leq x \leq 35$ mol%) telah dihasilkan. Tambahan pula, sampel kaca dengan komposisi 20SrO·40B_2O_3·40TeO_2 dan 30SrO·35B_2O_3·35TeO_2 yang didop dengan Europium, Eu^{3+} dan Dysprosium, Dy^{3+} (1 mol%) juga telah dihasilkan. Kesemua sampel kaca telah dihasilkan menggunakan kaedah pelindapan lebur diikuti dengan proses sepuh lindap pada suhu 400 ^{o}C selama 6 jam. Struktur sampel kaca telah ditentukan melalui pembelauan sinar-X (XRD) dan spektroskopi inframerah transformasi Fourier dan sifat luminesen melalui spektrometer luminesen. Corak pembelauan XRD menunjukkan fasa amorfus. Spektra inframerah menunjukkan bahawa, pertambahan kandungan strontium di dalam kaca borotellurit, mengalakkan perubahan struktur unit daripada [BO_3] kepada [BO_4] dan [TeO_4] kepada [TeO_3]. Perubahan ini menunjukkan bahawa sebahagian daripada cincin boroksol bertukar menjadi struktur rangkaian sementara strontium bertindak sebagai pengubahsuai rangkaian. Spektra pancaran daripada kaca tak berdop dan berdop adalah dalam julat cahaya nampak. Garis spektra pancaran daripada kaca tak berdop pada 513 nm adalah dari perumah kaca itu sendiri. Garis spektra pancaran dari kaca berdop Eu^{3+} adalah daripada peralihan ion Eu^{3+} pada $^5D_0 \rightarrow {}^7F_0$ (580 nm), $^5D_0 \rightarrow {}^7F_1$ (593 nm), $^5D_0 \rightarrow {}^7F_2$ (613 nm) dan $^5D_0 \rightarrow {}^7F_3$ (652 nm). Sementara garis spektra pancaran kaca berdop Dy^{3+} adalah disebabkan oleh peralihan dari $^4F_{9/2} \rightarrow {}^6H_{15/2}$ (483 nm), $^4F_{9/2} \rightarrow {}^6H_{13/2}$ (578 nm) dan $^4F_{9/2} \rightarrow {}^6H_{11/2}$ (660 nm). Garis spektra pancaran dari kaca berdop Eu^{3+} dan Dy^{3+} adalah pada 483 nm, 513 nm, 578 nm, 613 nm dan 660 nm. Hasil kajian menunjukkan bahawa keamatan garis pancaran kaca berdop Eu^{3+} dan Dy^{3+} bergantung kepada komposisi perumah. Tambahan pula, lengkuk pereputan luminesen kaca berdop Eu^{3+} menunjukkan pereputan pelbagai eksponen dengan jangka hayat bagi kaca 20SBT:1Eu^{3+} ialah 1 ns dan 22 ns manakala bagi kaca 30SBT:1Eu^{3+} jangka hayat ialah 1 ns dan 18 ns.

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LIST OF SYMBOLS

c	-	Speed of Light
C	-	Celsius
d	-	Length
k	-	Force Constant
T_f	-	Freezing Temperature
T_g	-	Transformation Range Temperature
τ	-	Tau
^{o}	-	Degree
μ	-	Reduced Mass
v	-	Frequency
λ	-	Lambda
θ	-	Angle

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CHAPTER 1

INTRODUCTION

1.1 Research Background

At first, glass is widely utilized in the field of lighting and telecommunications. It has been define as a solid material lacking in long range, periodic atomic structure and exhibiting glass transformation range. Common methods to produce glass are melt quenching, vapor deposition and sol-gel processing solutions. However, the most conventional way is by melt quenching due to its simplicity and low cost compare to other methods.

In general, glass making oxides can be divided into glass former and conditional glass former oxides. Examples of glass former oxides are silicon oxide, boron oxide, germanium oxide and phosphorus oxide while tellurium oxide, aluminium oxide and vanadium oxide as conditional glass former oxides. Glass former readily form glasses and act as the backbone in glasses containing mixed of oxides. On the other hand, conditional glass former cannot form glass on their own but possible to form glass by mixing with other oxides at suitable quantity. Specifically, mixed conditional glass former with other oxides is to lower the crystallization effect and increase glass forming ability.

According to Lin *et al*. (2005), borate glasses highly transparent, low melting temperature, high thermal stability and good rare earth ions solubility. However, it have been less utilize due to high phonon energy (1500 cm^{-1}). On the other hand, tellurite glasses have been extensively studied due to good properties such as low melting temperature (733 ^{o}C), high chemical stability, good visible-infrared transmission, low phonon energy (700 cm^{-1}) and high refractive index ($\geq$ 2.00) (Ricardo *et al*., 2008). As a matter of fact, Kumar *et al.* (2012) state glass with low phonon energy provide less non-radiative relaxation rates and high quantum efficiencies thus suitable for producing high efficiency optical materials. As reported by Maheshvaran *et al*. (2013a), incorporation between borate and tellurite oxides possible to produce borotellurite glass of low phonon energy.

Furthermore, many studies have been done on the incorporation between alkali and alkaline earth metals with borotellurite glass. Some of the reported studies are by Maheshvaran and Marimuthu (2011) on magnesium potassium borotellurite glass doped with dysprosium while Selvaraju *et al.* (2011) worked on magnesium potassium borotellurite glass doped with europium. The evidence suggests, this glass is highly potential for laser application. Furthermore, strontium of alkaline earth metals appeared to be part of many phosphors. As illustration of, works by Zhang *et al*. (2007) has shown strontium borate glass doped with europium resulting long lasting phosphorescence in the range of second's.

In addition, lanthanide elements namely europium and dysprosium have been doped into many different systems to obtain desired optical characteristics. In general, glass doped with europium and dysprosium resulting combination emission of blue, yellow and reddish orange (Joshi and Dhondiyal, 2005). On the other hand, glass doped with europium shows intense and narrow emission of reddish orange (Jamalaiah *et al*., 2009), meanwhile glass doped with dysprosium resulting combination of blue and yellow emission (Kumar *et al*., 2012). Complementary to this, europium and dysprosium widely used as spectroscopic probe to study the local structure around the ions in the surrounding ligand in numerous glasses (Lidia, 2013).

1.2 Problem Statement

In the past, combination between borate and tellurite were able to form borotellurite glass (Rada *et al.*, 2008b). Recently, there were studies incorporating different metals into borotellurite glass system. For example, Maheshvaran and Marimuthu (2011) and Selvaraju *et al*. (2011) have reported studies on alkali and alkaline earth borotellurite glass. Both studies shows alkali and alkaline earth metals were part of the system producing borotellurite glasses that suitable for solid state lighting and laser application. However, there was limited structural information regarding alkali and alkaline earth metals effect in the glasses. Therefore, it is interesting to study the aspect of alkali or alkaline earth metal in borotellurite glass system. More importantly, literature survey shows there were limited studies on strontium borotellurite system. Thus, in this research, an attempt is made to investigate strontium borotellurite glass in order to establish the local network structure changes induced by addition of strontium. In order to throw more light on this investigation, the ratio of borate to tellurite fixed as 1:1 and luminescent properties of glass undoped and doped with europium and dysprosium also characterized.

1.3 Research Objectives

- To prepare undoped and doped with europium and dysprosium strontium borotellurite glasses by melt quenching method.
- To determine structural nature and local network structure through X-ray diffraction and FT-IR spectroscopy.
- To determine luminescence emission and decay curve of undoped and doped with europium and dysprosium strontium borotellurite glasses.
- To determine elemental composition of undoped and doped with europium and dysprosium strontium borotellurite glasses.

1.4 Scope of Study

Glass system of strontium gradually replacing borate and tellurite were formulated. Five compositions consist of strontium oxide, boron oxide and tellurite oxide were mixed through solid state reaction while luminescent strontium borotellurite glasses was prepared based on two compositions and doped with 1 mol% of europium oxide and dysprosium oxide. By using melt quenching method, firing temperature for melting oxides mixtures were varies between 900 and 1100 ^{o}C. Apart from that, annealing temperature was set at 400 ^{o}C for 6 hours.

The structural nature, local network structure, luminescence emission and decay curve, and elemental composition of the undoped and doped strontium borotellurite glasses were evaluated by X-ray diffractometer (XRD), Fourier transform infrared spectrometer (FT-IR), fluorescence spectrometer and energy dispersive X-ray spectrometer (EDX).

1.5 Significance of Study

The study of alkaline earth effect in borotellurite glasses is important to obtain information from the local network structure. The information is useful in glass technology to develop new material.

Undoped and doped strontium borotellurite glass of low phonon energy, highly transparent and chemically stable were prepared through melt quenching method. Melt quenching is simple and cost effective which is suitable for mass production. Low phonon energy was the major advantage in producing luminescent glass of higher efficiency for technological application.

1.6 Outline of Study

This thesis subdivided into five chapters, Chapter 1 is introduction for present studies. Chapter 2 specifically for literature reviews then Chapter 3 focuses on methodology. Coming are Chapter 4 which is the most important chapter covering results and discussion. Finally are Chapter 5 which is conclusions and recommendation for future work.

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