PHYSICAL AND OPTICAL PROPERTIES OF LITHIUM POTASSIUM BORATE GLASS DOPED WITH NEODYMIUM

SITI NOOR HAFIZAH BT IBRAHIM

UNIVERSITI TEKNOLOGI MALAYSIA

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SITI NOOR HAFIZAH BT IBRAHIM

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To my beloved parent, brothers and sisters.

To all my dearest friends.

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ABSTRACT

A series of glass based on the composition of $20Li_2CO_3$: $10K_2CO_3$: $(70-x)B_2O_3$: xNd_2O_3, where $0 \leq x \leq 1.3$ mol % have been successfully prepared by conventional melt-quenching technique. The X-Ray Diffraction (XRD) spectra obtained confirmed the amorphous nature of the glass samples. The physical properties which includes density, ion concentrations, polaron radius and inter-nuclear distance have been determined. The glass transition temperature, crystallization temperature and melting temperature were determined by using Differential Thermal Analysis (DTA). The thermal stability of the glasses were increased with successive increase of Nd_2O_3 concentration. Infrared spectra were recorded at room temperature from 400 – 4000 cm^{-1}. The bands arising from the vibrations of the borate network were observed in the range of 500 – 1600 cm^{-1}. Since no peaks is observed at 806 cm^{-1}, there are no boroxol ring involved and the glass network structure were expected to consists of randomly connected BO_3 and BO_4 units. The absorption spectra obtained from UV-Vis-NIR spectroscopy revealed eight absorption peaks, with the highest intensity at 581 nm. The increment of dopant concentrations had increased the absorption intensity of the glass samples. Based on the absorption measurements, the optical band gap energy and refractive index have been determined. Energy band gap show a maximum for 0.7 mol % and a minimum for 0.3 mol % of Nd_2O_3 concentrations. While refractive index shows an opposite trend with energy band gap. Photoluminescence spectra of doped glass samples exhibits three emissions in visible region under 310 nm excitation wavelength. It is observed that a violet, blue and green emission occurred at 420 nm, 484 nm and 538 nm due to $^2P_{1/2} \rightarrow {}^4I_{9/2}$, $^4G_{9/2} \rightarrow {}^4I_{9/2}$ and $^4G_{7/2} \rightarrow {}^4I_{9/2}$ transitions of neodymium ions, respectively.

ABSTRAK

Satu siri kaca berasaskan $20Li_2CO_3 : 10K_2CO_3 : (70-x)B_2O_3 : xNd_2O_3$, dimana $0 \leq x \leq 1.3$ mol % telah berjaya dihasilkan dengan menggunakan teknik sepuh lindap. Analisis Pembelauan Sinar-X (XRD) membuktikan bahawa semua sampel kaca menunjukkan sifat amorfus. Sifat-sifat fizikal kaca seperti ketumpatan, isipadu ion, jejari polaron, jarak inter-nuklear juga telah ditentukan. Suhu transformasi, suhu penghabluran dan suhu lebur kaca ditentukan dengan menggunakan kaedah Penganalisis Pembeza Terma (DTA). Kestabilan terma kaca semakin meningkat dengan peningkatan isipadu Nd_2O_3. Spektrum inframerah bagi sampel kaca telah direkod pada suhu bilik dari 400 – 4000 cm^{-1}. Puncak-puncak daripada getaran rangkaian borat dapat dilihat pada julat 500 – 1600 cm^{-1}. Tiada puncak serapan diperhatikan pada 806 cm^{-1}, maka tiada cincin boroxol yang terlibat dan rangkaian kaca bagi sampel-sampel dijangka terdiri daripada unit BO_3 dan BO_4 yang disambungkan secara rawak. Spektrum serapan yang diperoleh daripada UV-Vis-NIR menunjukkan lapan puncak serapan, dengan keamatan tertinggi pada 581 nm. Penambahan isipadu dopan telah meningkatkan keamatan daripada sampel-sampel kaca. Berdasarkan pengukuran serapan, jurang tenaga optikal dan indeks biasan telah ditentukan. Jurang tenaga menunjukkan nilai maksimum untuk 0.7 mol % dan minimum untuk 0.3 mol % isipadu Nd_2O_3. Manakala indeks biasan menunjukkan trend yang bertentangan dengan jurang tenaga. Spektrum fotoluminesens pula menunjukkan tiga pancaran pada julat cahaya nampak di bawah pengujaan dengan panjang gelombang 310 nm. Diperhatikan bahawa pancaran sinar ungu, biru dan hijau berlaku pada 420 nm, 484 nm dan 538 nm, masing-masing disebabkan oleh transisi ion neodymium dari $^2P_{1/2} \rightarrow {}^4I_{9/2}$, $^4G_{9/2} \rightarrow {}^4I_{9/2}$ dan $^4G_{7/2} \rightarrow {}^4I_{9/2}$.

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LIST OF SYMBOLS

T_m	-	Melting temperature
T_g	-	Transition temperature
T_c	-	Crystallization temperature
W_a	-	Weight of the glass sample in air
W_L	-	Weight of the glass sample in immersion liquid
ρ_L	-	Density of immersion liquid
ρ_o	-	Density of toluene
V_m	-	Molar volume
N	-	Ion concentration
r_p	-	Polaron radius
r_i	-	Inter-nuclear distance
M	-	Average molecular weight of glass sample
d	-	Characteristic lattice spacing
λ	-	Wavelength
θ	-	Diffracted angle
n	-	Integer representing the order of diffraction

λ	-	Disintegration Constant
s	-	Glass thermal stability
H_R	-	Hurby criteria
T_{rg}	-	Glass forming ability
I_T	-	Intensity of transmitted light
I_A	-	Intensity of absorbed light
I_R	-	Intensity of reflected light
I_0	-	Intensity of incident light
I	-	Intensity
d	-	Thickness
α	-	Absorption coefficient
A	-	Absorbance
$\hbar\omega$	-	Photon energy
E_{opt}	-	Optical band gap energy
B'	-	Band tailing constant
n	-	Refractive index
E_g	-	Energy band gap
λ_g	-	Wavelength corresponds to the E_g

LIST OF ABBREVIATIONS

NIR	-	Near Infrared
IR	-	Infrared
Vis	-	Visible
UV	-	Ultraviolet
PL	-	Photoluminescence
DTA	-	Differential Thermal Analysis
FTIR	-	Fourier Transform Infrared
NBO	-	Non-Bridging Oxygen
XRD	-	X-ray Diffraction Analysis
NA	-	Avogadro Number
LKB	-	Lithium Potassium Borate
Li_2CO_3	-	Lithium Carbonate
K_2CO_3	-	Potassium Carbonate
B_2O_3	-	Borate Oxide
Nd_2O_3	-	Neodymium Oxide

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Various research and development of glass in science and technology have produced various type of glass that have been utilized and benefited in our daily life. For many years, rare earth elements in glasses have become an attractive area of research due to their applied and scientific importance, especially in development of optical devices such as solid state lasers and optical fibers. It is also well known that glasses is a convenient host material that offer a medium for the production of devices that are flexible in sizes and efficient. The knowledge of glass properties is very important, either concerning both industrial and research point of view since it offers various useful applications. Over the years, the properties of glasses doped rare earths have been widely investigated by many researchers.

There are many glass formers that can form a glass by itself which is included borates, silicates, phosphates and germanates. Owing attractive properties that grab many researchers' interest, borate has become one of the most important glass formers. Those attractive properties of borate glass included high transparency, radiation and thermal stability, non radiative relaxation states and low melting point. (Alajerami *et al*., 2012a). Borate glasses also have the advantageous photonic properties such as high

density, optimum band widths, good infrared transmission, high mechanical stability, corrosion resistance and low cost (Vijaya and Suresh, 2012).

However, despite of interesting and useful properties that possessed by borate, it's alone has a few disadvantages as described by Husin *et al*., (2009). Borate alone is relatively stable, easy to crystallize after melting and has hygroscopic properties. These disadvantages somehow will affect their optical properties and limit the practical use. These disadvantages can be reduced by adding modifiers and activator to the glass network (Alajerami *et al*., 2012a). Glass network modifiers can be alkali metal cations, alkaline earths and many of the transition metal cations (Nandyala and Jose, 2008).

Adding modifier to the glass network can disrupt the glass lattice, open up the network structure, weaken the bond strength, lower the viscosity of the glass and hence improve the glass stability. While, by adding dopant or activator such as magnesium, indium, nickel, cerium, europium, copper and manganese to the glass host can enhance the luminescence properties of lithium borate glass (Alajerami *et al*., 2012a). Among others commonly used modifiers, lithium oxide has been found to be one of the most preferable modifiers. It has been reported that adding modifier to the glass network can enhance the quality and optical performance of borate glass (Reddy *et al*., 2008).

Borate glass incorporated with various composition of modifiers and dopants have been widely investigated. A subsequent research for new borate compounds led to the continued discovery of new borate glass with improved in their properties. Glass mixture together with alkali oxide doped with rare earth elements is significance in photoluminescence studies due to its high chemical stability and good homogeneity (Nor Ezzaty, 2010). Works by Alajerami *et al*., (2012b) regarding the effect of phosphorus and copper oxide on the photoluminescence characteristic of lithium potassium borate glass shows around three times enhancement on the photoluminescence spectra. This is due to the increment of phosphorus impurity dopant in the glass mixture. The result of this study also shows a nonlinear optical

relation which is very useful for optical applications. Another study carried by Saddek (2004) also showed a nonlinear behavior with the increment of alkali oxides.

Glass doped rare earth, mostly trivalent lanthanide ions have been extensively studied due their promising properties for production of many optical devices. Lanthanide series such as Ce, Pr, Nd, Sm, Eu, Dy, Er, Tm and Tb can be easily doped into glasses without inducing crystallization to produce an optical devices in different shapes and sizes. One of the most distinctive properties of lanthanide ions in solid state host is the sharpness of many absorption and emission spectral lines, as well as good light emitters in the spectral region form visible, NIR and IR up to 3μm. The study and knowledge regarding optical properties of rare earth in glass is very important so that the best ion-host configuration can be optimized (Souza *et al*., 2000).

1.2 Problem Statement

Borate glass has been studied intensively due to its efficiency in various features as mentioned before. Becoming a good host for different metals (transitions and rare earth) make borate glass become one of the most preferable material for various technological applications. Although there are numbers of research on borate based glass have been done, yet it is seems limited to certain properties and dopants. Due to desirable properties of borate glass for various applications, therefore, this present work aims to study the physical and optical properties of lithium potassium borate glass undoped and doped with different concentrations of neodymium.

1.3 Objectives of the Study

The objectives of this study are:

1. To prepare the glass samples based on the composition of $20Li_2CO_3 : 10K_2CO_3 : (70 - x)B_2O_3 : xNd_2O_3$, where $0 \leq x \leq 1.3$ mol %.

2. To determine the physical and structural properties of glass system.

3. To determine the optical and photoluminescence properties of the samples by means of UV-Vis-NIR and PL spectroscopy.

1.4 Scope of the Study

In order to achieve the objectives, the study have been focus on the given scope:

1. Preparation of lithium potassium borate glass samples undoped and doped with neodymium using melt quenching technique.

2. Determination glass density, molar volume, ion concentration, polaron radius and inter-nuclear distance of neodymium ions.

3. Determination of thermal properties by using Differential Thermal Analysis (DTA).

4. Characterization of infrared transmission spectra by using Fourier Transform Infrared (FTIR) spectrometer.

5. Characterization of absorption spectra by using UV-Vis-NIR spectrophotometer and thus determination of optical band gap energy and refractive index.

6. Characterization of photoluminescence spectra by using Photoluminescence spectrometer.

1.5 Significance of the Study

Recently, borate glass are chosen because they are known for a long time as a good glass former that can be formed at low melting point. Furthermore, melts of compositions that are rich in B_2O_3 have higher viscosity and are easier to form glasses compared to those rich in other oxides (Nandyala and Jose, 2008). The results of this study may provide a basis for employing this glass system in related applications. For instance, the study on the optical properties in this work might be significant in providing a baseline data that can be used for the further research and development of borate based optical devices.

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APPENDIX A

The Nominal Composition of Glass and Calculations.

Based on the glass system of $20Li_2CO_3 : 10K_2CO_3 : (70 - x)B_2O_3 : xNd_2O_3$ where $0 \leq x \leq 1.3$ mol %, a series of glass sample has been successfully prepared. The total weight of each prepared sample was 15 grams.

To prepare S1 with nominal compositions of 20 mol % Li_2CO_3, 10 mol % K_2CO_3, 70 mol % B_2O_3 and 0 mol % Nd_2O_3, the required mass for each compound is calculated as below. Note that the other glass samples also follows the same calculations and procedures.

Given that molecular weight of each compound:

$Li_2CO_3 = 73.89\ gmol^{-1}$

$K_2CO_3 = 138.21 gmol^{-1}$

$B_2O_3 = 69.62\ gmol^{-1}$

$Nd_2O_3 = 336.48\ gmol^{-1}$

Thus, molecular weight of S1 glass:

$$W = (0.2 \times 73.89) + (0.1 \times 73.89) + (0.7 \times 73.89) + (0 \times 73.89)$$
$$= 77.33 \text{ gmol}^{-1}$$

The required mass of each compound for 15 g of S1 glass:

$$Li_2CO_3 = \left(\frac{0.2 \times 73.89 \text{ gmol}^{-1}}{77.33 \text{ gmol}^{-1}} \right) \times 15 \text{ g} = 2.8665 \text{ g}$$

$$K_2CO_3 = \left(\frac{0.1 \times 138.21 \text{ gmol}^{-1}}{77.33 \text{ gmol}^{-1}} \right) \times 15 \text{ g} = 2.6809 \text{ g}$$

$$B_2O_3 = \left(\frac{0.7 \times 69.62 \text{ gmol}^{-1}}{77.33 \text{ gmol}^{-1}} \right) \times 15 \text{ g} = 9.4531 \text{ g}$$

$$Nd_2O_3 = \left(\frac{0 \times 336.48 \text{ gmol}^{-1}}{77.33 \text{ gmol}^{-1}} \right) \times 15 \text{ g} = 0 \text{ g}$$