STRUCTURAL AND OPTICAL PROPERTIES OF ERBIUM/ NEODYMIUM CO-DOPED MAGNESIUM PHOSPHATE GLASS

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Specially dedicated to my beloved mom, Pn. Zubedah Binti Setapa,
dad, Mazlan Bin Salleh,
sisters, Mazlina, Hidayah, and Asyikin,
and all my friends...
Thank you for your love and support.

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ABSTRACT

Two series of glass based on composition $50P_2O_5$ - 48MgO – (2-x)Er_2O_3 - $x$$Nd_2O_3$, where $0.5 \leq x \leq 1.5$ mole % and (50-y)P_2O_5 - 48MgO – $2Er_2O_3$ – $y$$Nd_2O_3$, where $0.5 \leq y \leq 2.0$ mole % have successfully been prepared by melt-quenching technique. The X-ray diffraction (XRD) analysis shows that all samples are in amorphous state and their density is found to be in the range of (2.568 - 2.686) gcm^{-3}. The vibrational study is conducted using the Infrared (IR) spectroscopy in the range of 400 – 4000 cm^{-1}. It is observed that the vibrational frequency occur around 3454 - 3564 cm^{-1}, 1298 - 1318 cm^{-1}, 1082 - 1085 cm^{-1}, 923 - 935 cm^{-1}, 727 - 776 cm^{-1}, and 468 - 498 cm^{-1} which is predominately due to the OH bending vibration, P=O asymmetric stretching vibration modes, symmetric stretch of PO_2, asymmetric stretch of P-O-P bridges (v_{as} (P-O-P)), symmetric stretching vibration of P-O-P bond (v_s (P-O-P)) and bending vibration of P=O unit, modes of PO_4^{3-} chain groups respectively. The UV-Vis-NIR spectroscopy analysis shows that the absorption characteristic consist of several significant absorption peaks around 872 nm, 801 nm, 743 nm, 580 nm, 1536 nm, 977 nm, 652 nm, 522 nm, 487 nm, 451 nm, and 406 nm which correspond to the transitions of both Nd^{3+} and Er^{3+} ions. The experimental and calculated oscillator strength is found to vary from 2.443 x 10^{-7} to 1.002 x 10^{-7} and from 1.801 x 10^{-7} to 0.745 x 10^{-7} respectively. The Judd-Ofelt intensity parameters Ω_2 varies from 1.968 x 10^{-22} cm^2 to 13.572 x 10^{-22} cm^2 while the value of Ω_4 and Ω_6 varies from 0.264 x 10^{-22} cm^2 to 11.007 x 10^{-22} cm^2 and from 0.775 x 10^{-22} cm^2 to 4.895 x 10^{-22} cm^2 respectively. It is also found that the energy gap, E_g and the Urbach energy, ΔE is in the range of 3.86-3.98 eV and 1.67 - 1.86 eV respectively, depending on the Nd_2O_3 concentration.

ABSTRAK

Dua siri kaca berasaskan $50P_2O_5$ - 48MgO – (2-x)Er_2O_3 - $x Nd_2O_3$, dengan $0.5 \leq x \leq 1.5$ % mol dan (50-y)P_2O_5 - 48MgO - $2Er_2O_3$ - (2-y)Nd_2O_3, dengan $0.5 \leq y \leq 2.0$ % mol telah berjaya dihasilkan menggunakan teknik pelindapan leburan. Analisis pembelauan sinar-X (XRD) membuktikan bahawa semua sampel menunjukkan sifat amorfus dan ketumpatan didapati berada dalam julat (2.568-2.686) gcm^{-3}. Kajian terhadap getaran telah dilakukan dengan menggunakan spektroskopi Inframerah (IR) dalam julat 400 – 4000 cm^{-1}. Didapati bahawa frekuensi penyerapan berada pada julat 3454 - 3564 cm^{-1}, 1298 - 1318 cm^{-1}, 1082 - 1085 cm^{-1}, 923 - 935 cm^{-1}, 727 - 776 cm^{-1}, dan 468 - 498 cm^{-1} yang masing-masing merujuk kepada mod getaran regangan OH, mod regangan bersimetri P=O, regangan bersimetri PO_2, regangan tidak bersimetri P-O-P, regangan bersimetri P-O-P, regangan P=O dan kumpulan rantaian PO_4^{3-} masing-masing. Keputusan daripada UV-Vis-NIR Spektroskopi menunjukkan bahawa terdapat beberapa puncak-puncak penyerapan utama telah dicerap sekitar 872 nm, 801 nm, 743 nm, 580 nm, 1536 nm, 977 nm, 652 nm, 522 nm, 487 nm, 451 nm, dan 406 nm akibat daripada transisi ion Nd^{3+} dan Er^{3+}. Kekuatan pengayun diuji dan didapati di antara 2.443×10^{-7} hingga 1.002×10^{-7} dan daripada 1.801×10^{-7} hingga 0.745×10^{-7}. Parameter keamatan, Ω_2, Ω_4, dan Ω_6 Judd-Ofelt masing-masing berada dalam julat 1.968×10^{-22} cm^2 hingga 13.572×10^{-22} cm^2, 0.264×10^{-22} cm^2 hingga 11.007×10^{-22} cm^2 dan daripada 0.775×10^{-22} cm^2 hingga 4.895×10^{-22} cm^2. Didapati bahawa jurang tenaga, E_g dan tenaga Urbach, ΔE adalah di sekitar 3.86 - 3.98 eV dan 1.67 - 1.86 eV masing-masing, bergantung pada kepekatan Nd_2O_3.

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LIST OF SYMBOLS

A	-	Surface area
A_{ed}	-	Electric-dipole spontaneous emission probability
A_{md}	-	Magnetic-dipole spontaneous emission probability
α	-	Absorption coefficient
$\alpha(\omega)$	-	Urbach rule
B, B'	-	Constant
c	-	Speed of light
cw	-	Continuous wave
d	-	Sample thickness
ΔE	-	Width of the band tails
E_g	-	Band gap energy
E_c	-	Conduction band energy
E_v	-	Valence band energy
E_{opt}	-	Optical energy gap
eV	-	Electron Volt
e	-	Charge of electron
f	-	Force constant of the bond
h	-	Planck constant
I	-	Anti-Stokes scattering intensity
$I(\omega)$	-	Stokes scattering intensity
k	-	Absorptive factor
λ	-	Wavelength
M	-	Molecular weight
m	-	Mass of electron
l	-	Length

t	-	Time
m_1, m_2	-	Mass of atoms in molecules
δ	-	Error
n	-	Refractive index of the incident medium
n'	-	Refractive index of the materials
A_j	-	Sellmeier parameter
Ω_q	-	Judd-Ofelt parameters
Θ	-	Angle
Q^n	-	Phosphate tetrahedral group
n	-	Number of bridging oxygen per tetrahedral
ω	-	Frequency
f	-	Oscillator strengths
f_{cal}	-	Theoretical oscillator strengths contains of magnetic-dipole
f_{exp}	-	Experimental oscillator strengths
f_{md}	-	Oscillator strengths contains of magnetic-dipole
ρ	-	Density
S_{ed}	-	Electric-dipole line strengths
S_{md}	-	Magnetic-dipole line strengths
T	-	Temperature
$\hat{u}$	-	Complex refractive index in vacuum
$\langle \lVert U^q \rVert \rangle$	-	Double reduce matrix elements
V	-	Volume
V_m	-	Molar volume
v	-	Vibration frequency
$\bar{v}$	-	Wavenumber
W_a	-	Weight of sample in air
W_t	-	Weight of sample in immersion fluid

LIST OF ABBREVIATIONS

CB	-	Conduction band
ESA	-	Excited state absorption
IR	-	Infrared
g	-	Gram
mm	-	Millimetre
XRD	-	X-ray diffraction
FT	-	Fourier transformed
FTIR	-	Fourier transformed infrared
UV	-	Ultraviolet
Vis	-	Visible
VB	-	Valence band
ASTM	-	American Society for Testing Material
NBO	-	Non-bridging oxygen
RE	-	Rare earth
NIR	-	Near infrared
P	-	Phosphorus atom
P_2O_5	-	Phosphorus pentoxide
Er_2O_3	-	Erbium oxide
Nd_2O_3	-	Neodymium oxide
MgO	-	Magnesium oxide

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Glass is one of the most common and versatile materials made by Man; millions of tons are produced annually. The word 'glass' is derived from an Indo-European root which mean shiny, and has also given the words glare, glow and glaze. It is known where, when or how glass was first manufactured, although there is little doubt that the industry was established since 1500 B.C. Nowadays, glass plays an important role in our daily life. The rapid developments of glass in science and technology have produced various types of glasses that can be used not only in our daily life, but has been extended to comprise in manufacturing and industrial needs.

There are many type of glass that has been used but the most common used in the manufacturing and industrial fields is silicate glass. Other glass formers are phosphate, tellurite, borosilicate, and calcogenide glass. These glasses have received a great attention for the past few years especially the phosphate glass. The properties that make phosphate glass a potential candidates for so many different applications are related to their molecular-level structures.

There have been many excellent reviews on structural studies of phosphate glasses, including those of Van Wazer (1958), Abe (1983) and Martin (1991). Martin's (1991) review, marked the beginning of new structural information on phosphate glass. By studying such glass, Van Wazer (1958) has established the foundation and presents the understanding about the nature of phosphate glass. Since phosphate glasses are hygroscopic and react easily to the atmosphere, the use of phosphate glasses has been restricted to commercial industries only. At the same time, Kordes and Nieder (1968) have re-examined the alkali earth phosphate glasses, including UV-transmitting compositions, and found some anomalous trend in properties which they then suggested that these are compositional dependence especially on the coordination number of metal cation.

Over the years, interest in rare-earth doped phosphate glasses is stimulated by their used in variety of industrial applications including communications, medical and manufacturing (Hattori *et al.*, 1996; Doremus, 1994). The advent of solid state lasers in the 1960's heralded a new are of phosphate glass research. Certain compositions have large rare-earth stimulated emission cross-sections and low thermo-optical coefficients (compared with silicate glasses) and are candidates, particularly for high power laser applications as studied by Weber (1990) and Brow (2000). Metaphosphate glasses can incorporate large quantities of rare-earth ions and have interesting optical characteristics (Hunt and Speck, 1989), making them as important host materials for lasers. The addition of modifier cations in the glass compositions and the suitability of these modifiers and compositions for making a good glass laser have been discussed elsewhere by Campbell *et al.* (2000), Buyn *et al*. (1994), Hayden *et al.* (1990), and Tandon *et al.* (1997). Different phosphate glass systems have been developed for numerous applications, specially tailoring the properties of glasses. Phosphate glasses are technologically important materials due to their low melting point and relatively high thermal expansion coefficient and glass transition temperature, which is suitable for technological applications, such as in medical use on solid state electrolytes (El-Egili *et al*., 2003). In addition, the thermo-optic coefficient of phosphate also has been utilized for a number of specialty applications. Phosphate glass has also been found to be an excellent host for Er^{3+} ions in terms of the spectroscopic properties (Desirena *et al.*, 2006). These glasses also have considerable potential for applications in optical data transition and laser technology. For example, neodymium phosphate glasses have been widely used in laser applications (Higazy, 1995).

Melt quenching technique has been used to prepare the glass sample. This technique is also known as supercooled liquid, frozen in liquids which have progressively attained the characteristics of a solid without crystallizing (Zarzycki, 1982). Other techniques used in obtaining glass are vapour deposition method, solution method, and solid state transformation. The vapour deposition method fall into two categories, that is a reactive process that involve chemical reaction and non-reactive process (evaporation and sputtering). These processes are typically used for producing thin film glass for electronic and optical application. However, the technique of cooling from the liquid state is the most important and most widely used because it is relatively simple and inexpensive. Melt quenching technique has been used by many researcher such as Chowdari *et al*., (1995) and Winter *et al*., (1997) in developing the energy storage devices and solid state battery glass. Desirena *et al*., (2006) using this techniques to develop glass laser material based on rare earth dopant.

1.2 Problem Statement

Although there are numbers of research on phosphate based glass have been done, yet the behavioral characteristics of these glass Er^{3+}/Nd^{3+} co-doped has not been fully understood. Therefore it is the aim of this study to investigate the effect of Er^{3+}/Nd^{3+} as co-dopants on structural and optical properties of phosphate glass. This is very important in order to understand the lasing mechanism in phosphate glass.

1.3 Objectives of the Study

The objectives of this study are:

i. To prepare two series of magnesium phosphate glass with nominal composition of $50P_2O_5$ - 48MgO – (2-x)Er_2O_3 - xNd_2O_3 and (50-y)P_2O_5 - 48MgO – 2Er_2O_3 – yNd_2O_3 by melt-quenching technique.

ii. To determine the physical properties of glass system by means of their density and refractive index.

iii. To characterize the transmission behavior by using FTIR spectroscopy.

iv. To characterize the absorption behavior by using UV-VIS spectroscopy.

1.4 Scope of Study

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

i. Preparation of two glass series using melt quenching technique.

ii. Determination of glass density by Archimedes method.

iii. Determination of refractive index by Sellmeier fitting method.

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

v. Characterization of absorption spectra in the UV and Visible region using UV-Vis spectrophotometer.

vi. Calculation of the Judd-Ofelt parameters, Ω_2, Ω_4, and Ω_6 from the absorption spectra.

1.5 Choice of the System

Two series of glass systems are chosen. First series known as S1, with the nominal composition of $50P_2O_5$ - 48MgO – (2-x)Er_2O_3 - xNd_2O_3, where $0.5 \leq x \leq 1.5$ mol % and second series known as S2 with the nominal composition of (50-y)P_2O_5 - 48MgO – 2Er_2O_3 – yNd_2O_3, where $0.5 \leq y \leq 2.0$ mol %.

Phosphate glasses are chosen because they possess large glass formation regions that are good host for fluorescence ions. Although, compare with other glasses, phosphate rarely exhibit the extreme property values possible, they are usually offer the best overall combination of properties for many different laser applications. The modifier that has been use in this study is MgO from group II. This modifier is acted as network modifier where it modifies the glass network during the melting process. This modifier is commonly used because when alkali or alkaline oxides are added to the phosphate, the network is presumably broken up into chain or ring network. The additional of other glass forming oxides or modifiers can contribute in the change of the physical and chemical characteristic of glass. Hence, it can ensure the stability of the phosphate glass.

Erbium oxide (Er_2O_3) and Neodymium (Nd_2O_3) are used as a dopant since it is relatively stable in air and are not quickly oxidizing. They exhibit two valence states of +3 and +5 which are very important for the electronic excitation and the emission of laser. It can also be applied as a glass coloring, as an amplifier in fiber optics and in laser for medical and dental use. The ion has a very narrow absorption band for coloring erbium salts pink mentioned by Speghini *et al.*, (2001). Erbium oxide (Er_2O_3) itself is a good first solid-state laser glass project. Erbium is occasionally used in infra-red absorbing glass. It is also called eye-safe laser, since the longer wavelength does not penetrate the eye to retina. Most of the rare-earth oxides exhibit a sharp absorption bands in the visible, ultraviolet, near and infrared region (Lide, 2004).

1.6 Thesis Plan

This thesis contains five chapters. The first chapter briefly explains the general overview of the glass, continued by the problem statement, scope and the objectives of this study. The reasons in choosing the glass system also discussed in this chapter followed by the thesis planning. In the second chapter, reviews of a current knowledge and background of phosphate glass doped rare-earth ion, erbium oxide (Er_2O_3) and Neodymium (Nd_2O_3) are presented. It will include the general theory on glassy state, phosphate glass fundamental structure, a special survey on the phosphate glass formation region, density, Infrared absorption spectroscopy, Photoluminescence spectroscopy, UV-Visible spectroscopy and refractive index measurement.

In the third chapter, all the methodology of the experimental aspect is described. These including the preparation of the glass, X-ray diffraction (XRD) technique, evaluation of density, Infrared absorption spectroscopy (FTIR), photoluminescence spectroscopy, and UV-Visible spectroscopy. At the end of this chapter, the experimental works and measurement of the glass refractive index using a Sellmeier equation will be presented.

The fourth chapter presents all the results and discussion. The results discussed including the glass formation range, X-ray diffraction analysis and the density of the glass samples. While, the vibrational bondings from FTIR spectroscopy, optical band gap, E_{opt} and refractive index from UV-Visible spectroscopy also will be discussed in detail.

In the fifth chapter, the conclusions based on the results and discussions from the previous chapter are made. The suggestion and recommendation for future investigation is also included.

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