CHARACTERIZATION OF LITHIUM-MAGNESIUM-TELLURITE DOPED WITH ERBIUM AND NEODYMIUM GLASS

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CHARACTERIZATION OF LITHIUM-MAGNESIUM-TELLURITE DOPED WITH ERBIUM AND NEODYMIUM GLASS

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This thesis is specially dedicated to:

To my beloved daddy (Roslan Bin Paiman)
My mother (Jamiah Binti Supar),
my siblings,
and all my friends.

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ABSTRACT

Tellurite glass based on (78-x)TeO_2-10Li_2O-10MgO-2Nd_2O_3-xEr_2O_3, (where x = 0.4 to 2.0 mol %) has successfully been prepared by melt-quenching technique. The colour of glass is found to vary from light violet to dark violet as the Er_2O_3 content is increased. No definite peaks are found from the X-ray diffraction pattern, which shows that the glass is amorphous in nature. It also found that the densities and the molar volume of the glass increase as the Er_2O_3 content is increased. The glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m) and the temperature difference (T_c-T_g) are determined by means of Differential Thermal Analysis (DTA). It is found that the T_c, T_g and T_m are in the range of (419-430) ^oC, (300-345) ^oC and (885-890) ^oC respectively. Meanwhile, the vibrational study is conducted using the Infrared spectroscopy in the range of (4000-400) cm^{-1}. Two major absorption peaks are observed around (1600-3600) cm^{-1}, and (900-1200) cm^{-1} which are due to the stretching mode vibration of OH peak and Te-OH peak respectively. The optical absorption edge is studied using UV-Vis spectroscopy. The result shows that the optical band gap (E_{opt}) and Urbach Energy (ΔE) are in the range of (3.038-3.130) eV and (0.334-0.321) eV respectively, depending on the Er_2O_3 concentration. The refractive index is evaluated using the Sellmeier's equation and it is found that the value in the visible region is in the range of 1.724-1.781 depending on the Er_2O_3 content. The emission spectrum is recorded using the photoluminescence spectrometer excited at 582 nm at room temperature. The result shows that the emission spectrum of Er^{3+} and Nd^{3+} consist of five emission bands at ~457 nm, ~495 nm, ~556 nm, ~611 nm, and ~ 665 nm which can be assigned as a transition of $^4F_{7/2}\rightarrow{}^4F_{15/2}$, $^4S_{3/2}\rightarrow{}^4F_{15/2}$, $^4G_{11/2}\rightarrow{}^4I_{9/2}$, $^4G_{11/2}\rightarrow{}^4I_{15/2}$ and $^4G_{7/2}\rightarrow{}^4I_{13/2}$ respectively.

ABSTRAK

Kaca Tellurit berasaskan (78-x)TeO_2-10Li_2O-10MgO-2Nd_2O_3-xEr_2O_3, (dengan $0.4 \leq x \leq 2.0$ mol %) telah berjaya disediakan menggunakan teknik pelindapan leburan. Warna kaca didapati berubah dari ungu terang kepada ungu gelap apabila kandungan Er_2O_3 bertambah. Corak pembelauan sinar-X tidak menunjukkan puncak yang pasti dan ini mengesahkan bahawa kaca tersebut adalah amorfus. Didapati juga bahawa ketumpatan dan isipadu molar kaca bertambah apabila kandungan Er_2O_3 bertambah. Suhu peralihan kaca (T_g), suhu penghabluran (T_c), suhu leburan (T_m) dan perbezaan suhu (T_c-T_g) telah ditentukan menggunakan Penganalisis Pembezaan Terma. Didapati bahawa T_c, T_g dan T_m masing-masing berada dalam julat (419-430) ^oC, (300-345) ^oC and (885-890) ^oC. Sementara itu, kajian terhadap getaran telah dilakukan menggunakan spektroskopi inframerah dalam julat (4000-400) cm^{-1}. Dua puncak utama diperolehi disekitar (1600-3600) cm^{-1}, dan (900-1200) cm^{-1} yang masing-masing merujuk kepada puncak mod getaran regangan OH dan Te-OH . Pinggir serapan optik dikaji menggunakan spektroskopi ultraviolet cahaya nampak. Didapati bahawa jurang tenaga, E_g dan tenaga Urbach, ΔE masing-masing adalah di sekitar (3.038-3.130) eV dan (0.334-0.321) eV, bergantung kepada kandungan Er_2O_3. Indek biasan telah ditentukan menggunakan persamaan Sellmeier dan didapati bahawa nilainya dalam julat cahaya nampak adalah 1.724-1.781, bergantung kepada kandungan Er_2O_3. Spektrum pancaran telah direkod menggunakan spektrometer fotoluminesen yang diujakan pada 582 nm pada suhu bilik. Keputusan menunjukkan bahawa spektrum pancaran Er^{3+} dan Nd^{3+} terdiri daripada empat jalur pada ~457 nm, ~495 nm, ~556 nm, ~611 nm, dan ~665 nm dengan masing-masing mewakili transisi dari $^4F_{7/2} \rightarrow {}^4F_{15/2}$, $^4S_{3/2} \rightarrow {}^4F_{15/2}$, $^4G_{11/2} \rightarrow {}^4I_{9/2}$, $^4G_{11/2} \rightarrow {}^4I_{15/2}$ and $^4G_{7/2} \rightarrow {}^4I_{13/2}$.

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LIST OF SYMBOLS

As_2O_5	-	Arsecin pentoxide
Al_2O_3	-	Aluminium oxide
B_2O_3	-	Boron oxide
Bi_2O_3	-	Bismuth oxide
Ga_2O_3	-	Gallium(III) oxide
GeO_2	-	Germanium dioxide
TeO_2	-	Tellurium oxide
TiO_2	-	Titanium dioxide
Li_2O	-	Lithium Dioxide
MgO	-	Magnesium Oxide
MoO_3	-	Molybdenum trioxide
P_2O_5	-	Phosphorus pentoxide
SeO_2	-	Selenium dioxide
SiO_2	-	Silicon dioxide
V_2O_5	-	Vanadium pentoxide
WO_2	-	Tungsten oxide
WO_3	-	Tungsten trioxide
ZnF_2	-	Zinc fluoride
Li^{3+}	-	Lithium trivalent ion
BOs	-	Bridging oxygen
ESA	-	Excited state absorption
NBO	-	Nob-bridging oxygen
SRO	-	Short range order
tbp	-	Trigonal bipyramid

tp	-	Trigonal pyramid
α-TeO_2	-	Paratellurite
RE	-	Rare earth
Er^{3+}	-	Trivalent erbium ion
Nd^{3+}	-	Trivalent neodymium ion
Yb^{3}	-	Trivalent Ytterbium ion
4f	-	Orbital belong to lanthanide series
4*fn*	-	Shell configuration belong to lanthanide series
DTA	-	Differential Thermal Analyzer
EDFAs	-	Erbium doped fiber amplifiers
FTIR	-	Fourier Transmission Infrared
IR	-	Infrared
NIR	-	Near infrared
UV-Vis	-	Ultraviolet Visible
PL	-	Photoluminescence
WDM	-	Wavelength division multiplexing
XRD	-	X-Ray Diffractometer
T_m	-	Melting temperature
T_c	-	Crystallization temperature
T_g	-	Glass formation temperature
$\alpha(\omega)$	-	Absorption coefficient
A	-	Absorbance
A_j	-	Sellmeier parameter
$A_{1,2,3}$; $B_{1,2,3}$	-	Sellmeier coefficients
c	-	Speed of light
d	-	Distance between each adjacent crystal planes
d_2	-	Thickness sample
D	-	Dispersion
E	-	Energy

E_g	-	Optical energy gap
E_i	-	Energy lower band
E_f	-	Energy upper band
e	-	Electron charge
eV	-	Electron Volt
ΔE	-	Urbach energy
ε_o	-	Electric permittivity
f	-	Vibration frequency
ik	-	Imaginary part
k	-	Extinction coefficient
k	-	Force constant
μ	-	Reduce mass
m	-	Mass of atom
m	-	index transition
M	-	Molar mass
n	-	Refractive index
n^*	-	Complex refractive index
OH	-	Hydroxyl
ρ	-	Density
ρ_l	-	Toluene density
ρ_a	-	Air density
Q	-	Quality factor
q	-	Phonon
R	-	Reflactance
v	-	Speed
v_{eq}^{s}	-	Symmetric stretching vibration
v_{ax}^{as}	-	Asymmetric stretching vibration
V	-	Volume

V_m	-	Molar Volume
W_a	-	Weight of sample in air
W_l	-	Weight of sample in immersion fluid
M_i	-	Molar mass of substance mol
Z	-	Atomic number
χ_i	-	Percentage of substance mol
$\hbar\omega$	-	Photon Energy
θ	-	Angle
λ	-	Wavelength
λ_j	-	Resonance wavelengths of the transitions
ΔT	-	Glass stability

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

Glasses are materials in the world that find a variety of applications in everyday human life. The characteristics of glasses are known to be sensitive to even very minor changes in the glass composition (Emad and Richard, 2012) which is important in developing new modern glasses. Ironically, although the physical properties and crystalline solids are now understood in essence, but this is not the case for glass. A glass has no long-range order, that is, when there is no regularity in the arrangement of its molecular constituents on a scale larger than a few times the times of these groups (Doremus, 1973). By new science and technology approach the application of basic scientific understanding to the improvement of glass manufacture and new applications of glass has vigorously occurred. The benefit will include the providing of the fundamental bases of new optical properties glasses with new applications. Recently, tellurite based glasses has been developed for various applications such as optical switches, laser second harmonic generation, third-order nonlinear optical materials, up-conversion glasses and optical amplifiers (El-Mallawany, 2002).

Technically, there are a variety of techniques can be used in order to formed a glass samples. The most conventional way is by melt-quenching method. On the other hand due to the research in glass, many techniques of glass had been used. One of the most popular technique nowadays is sol-gel technique because it deals with low temperature preparation and homogenized composition compared to the conventional method. However, sol-gel method preparation is quite difficult, time consuming and the material was used are very expensive.

The stability of tellurium oxide is one of the characteristic that has attracted researcher especially for the formation of tellurite glasses (El-Mallawany, 2002). Tellurium oxide (TeO_2) is the most stable oxide of tellurium (Te) with a low melting point of 773 ˚C (El-Mallawany, 2000; Eranna, 2011). The basic structural units of tellurite glasses (TeO_2-based glasses) is a TeO_4 trigonal bipyramid (tbp) by which each oxygen atom shared by two units, bonded in the equatorial position to one tellurium atom and in the axial position to another (John *et al.*, 2006; Zhian *et al.*, 2010). As reported by Rosmawati et al. (2008), there is four coordination of Te in the tetragonal form, the nearest-neighbour being arranged at four of the vertices of the trigonal bipyramid which suggesting considerable covalent character of the Te-O bonds. In paratellurite all the vertices of TeO_4 groups are shared in a 3-dimensional configuration by which the oxygen bond angle is 140˚, the coordination polyhedron there are two equatorial ($Te\text{-}O_{eq}$ = 1.90 A) and two axial ($Te\text{-}O_{ax}$ = 2.08 A) bonds (Lambson *et al.*, 1984).

Tellurite glass has received attention as new oxide glasses in technologically and scientifically due their outstanding properties, such as in remarkable optical properties (high refractive index, high dielectric constant, a wide band infrared transmittance), thermal stability, chemical durability, high homogeneity, and low melting temperature (El-Mallawany, 2002; Khattak *et al.*, 2004; Raffaella *et al.*, 2001). TeO_2 not only interesting in terms of practically use, but also showing interesting properties in the structure of glass and glass forming ability.

Extensive studies of rare-earth glasses started in the 1960s, when the unique characteristics of rare-earth ions in the amorphous matrices were discovered. The study of rare-earth doped glasses have received great attention for optical applications, such as lasers, display devices, fiber amplifier, optical communication, and sensors (Zhang *et al.*, 2007, Neeraj Kumar Giri *et al.*, 2007; Kaushal and Rai, 2007; Chen *et al.*, 2008). Enhancing the linear and nonlinear optical effects in rare-earth doped tellurite glasses are amongst the most important subjects of present day materials science and technology. Meanwhile, tellurite glass co-doped with two or more rare earth ions inspire intense interest in functionalizing it for widespread applications (Vineet and Rai, 2004; Dai Shi Xun *et al.*, 2003). This is because the rare earth ions have very high solubility which that is allows the material to be co-doped with several rare earths ions together (Hiroki *et al.*, 2005, Wenbin and Chun , 2010). Rare earth is good candidates for active ions in laser materials because they show many absorption and fluorescence transitions in almost every region of the visible and the near infrared range (Deva and Madhukar, 2012; Hotan, 2007).

Recently, energy transfer between Er^{3+} and other rare earth ions have been discovered by many researchers. Erbium-doped tellurite glasses have optical and chemical properties appropriate for optical applications (Jaba *et al.*, 2005; Marjanovic *et al.*, 2003). Moreover, the low loss tellurite-based Er^{3+} doped fiber amplifiers (EDFAs) from 1528 to 1611 nm is beneficial in upgrading the design wavelength division multiplexing (WDM) network applications (Mori *et al.*, 1998). The other lanthanide ions also attract a lot of consideration such as thulium, praseodymium, neodymium, or dysprosium, which can increase the wavelength domain of a transmission towards higher energy, up to 1.3 μm (Jacquier *et al.*, 2005). Neodymium (Nd^{3+}) has been known as one of the most efficient rare earth ions for solid-state lasers in a variety of hosts because of its intense emission at about

1.06 μm (Chen, 2008). Moreover, the absorption of Nd^{3+} is useful in solar cell (Jacek *et al.,* 2009) and good applicant for improving the pumping efficiency (Lakshminarayana *et al.,* 2008). In addition, Nd^{3+}-doped tellurite single-mode fibre laser has been carried out recently (Wang *et al.,* 1994).

In this work, tellurite has been used as a glass host due to their potential as a laser host matrix while erbium oxide as a dopant. Therefore, three modifiers ions namely Lithium Dioxide (Li_2O also known as Lithia), Magnesium Oxide (MgO), and Neodymium oxide (Nd_2O_3) will be added to the glass host as modifier by modifying the glass structure in certain reaction during melting process. Conventional melt quenching technique has been applied throughout the glass preparation. The work represents a part of continuing effort to characterize the influence of Er^{3+} ions doped Li_2O-MgO-Nd_2O_3 with respect to density, molar volume, refractive index, IR spectroscopy, optical absorption in ultraviolet and visible range and photoluminescence respectively.

1.2 Problem Statement

Research on tellurite based glass system has been study by many researchers. Unfortunately, there is lacking the behavioural characteristics of these glass Er^{3+}/Nd^{3+} co-doped with modifier (MgO, Li_2O) has not been fully investigated. Few studied had been done in this system but are limited to certain properties and doping with rare-earth ions is not study. Therefore, the present study is done in order to know the optical and structural behaviour of the Er^{3+}/Nd^{3+} co-dopant glasses besides the effect of doping rare-earth ions on luminescence properties are presented in this thesis.

1.3 Research Objective

In order to provide more information on the glass properties, the objectives of this research are:

i. To prepare a new glass system of Erbium doping Lithia-magnesium-Neodymium-tellurite glass in order to identify optical properties in the glass network.

ii. To determine the physical properties of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 in order to develop basic structure of glass network.

iii. To investigate the thermal behaviour of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass to see the forming glass ability in the glass.

iv. To examine the structural change as the dopant Er^{3+} concentration added in the network Li_2O-MgO-Nd_2O_3-TeO_2.

v. To study the variation of optical properties in function of the Er^{3+} composition in Li_2O-MgO-Nd_2O_3-TeO_2glass.

vi. To study the fluorescence emission for understanding the upconversion phenomena of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass.

1.4 Scope of Study

In order to achieve the objectives, the study has been divided into several scopes which are:

a) Preparation of co-doped glass in the composition of (78-x)TeO_2-10Li_2O-10MgO-2Nd_2O_3-xEr_2O_3 with $0.4 \leq x \leq 2.0$ mol%.

b) Determination of the amorphous phase of the obtained glass using X-ray diffraction (XRD).

c) Identification of the physical properties of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glasses in term of density and molar volume.

d) Determination the thermal stability of the Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass in term of melting temperature T_m, crystallization temperature T_c and transition glass temperature T_g using Differential Thermal Analyzer (DTA).

e) Determination the structural properties of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass band using Infrared Spectroscopy.

f) Determination the optical properties of Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass in term of refractive index, energy band gap, Urbach energy and refractive index using Ultraviolet-Visible Spectroscopy.

g) Determination of the luminescence spectra using Photoluminescence Spectroscopy.

1.5 Glass System Chosen

In order to achieve the aims of these studies, one series of glass samples has been prepared based on constant lithium oxide, magnesium oxide and neodymium oxide with a variation of erbium oxide. This series is based on composition (78-x)TeO_2-10Li_2O-10MgO-2Nd_2O_3-xEr_2O_3 with $0.4 \leq x \leq 2.0$ mol%. Five samples of glass have been prepared.

Tellurite glasses are chosen because owing high density, chemical durability and wide transparency which is a suitable host for rare earth (Dhiraj *et al.*, 2012). It also has lowest phonon energy of ~590 cm^{-1} among oxide glasses and the largest refractive index values, both of which are useful for high radiative transition rates of rare-earth ions. Then, tellurite glass has the ability to dissolve high concentration of lanthanide ions without clustering and thereby increasing the fluorescence lifetime and quantum efficiency, which are important spectroscopic requirements for a good luminescence material.

The choice of erbium oxide (Er_2O_3) as dopant because it is relatively stable in air and are not quickly oxidizing. Additional Li_2O into tellurite glass will increase the ionic conductivity (Muruganandam and Seshasayee, 1997). There have also been literature reports on Li^{3+} ions transport in tellurite glasses (Harish *et al.*, 2004; Marcio and Shigueo, 2006; Jayasinghe *et al.*, 1999; Rodrigues *et al.*, 2000; Patrick *et al.*, 2002; Lee *et al.*, 2002). MgO has no notable influence upon the strength of the network, but having an effect on the optical properties of glass.

1.6 Significant of the study

Due to the limited of the study based on Er_2O_3 doping Li_2O-MgO-Nd_2O_3-TeO_2 glass, this present study has been done to understand further the optical features of the glass. By adding doping to the system, new materials can be developed as new luminescence materials. These materials can emit light in the visible range and have colourful glasses.

1.7 Summary of Thesis

This thesis contains of five chapters. Chapter 1 gives a brief overview of the introduction of the study in the band, which previous studies on related glass materials development undertaken by other researchers and the discussion about the problem statement, the objective, the scope of this research and the choice of system.

Chapter 2 comprises the literature review of this research. This chapter consists of the theoretical background of physical properties of tellurite based glasses and the properties of the lanthanide elements. This chapter also provides some theoretical review on the characterization method of x-ray diffraction, infrared spectroscopy, absorption, refractive index, transition mechanism and density.

Chapter 3 focuses on the experimental techniques and equipments used in the research. Details on the sample preparation, design of the experiment and the measurement techniques employed are outlined. This is followed by the characterization of the samples by using X-Ray Diffractometer (XRD), densitometer, Differential Thermal Analyzer (DTA), Infrared (IR) spectrometer, UV-visible spectrometer (UV-Vis) and Photoluminescence (PL).

Chapter 4 deals with the discussion on the experimental results. The result on density, molar volume, XRD pattern, thermal parameters, IR vibrational spectra, absorption spectra, refractive index, and luminescence properties will be discussed in this chapter. Chapter 5 concludes this thesis with a brief summary on the achievement of the objectives. This chapter also consists of some suggestions for further studies.

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