THERMAL, STRUCTURAL AND OPTICAL PROPERTIES OF LITHIUM NIOBATE TELLURITE GLASS DOPED ERBIUM AND NEODYMIUM

NURHAFIZAH BINTI HASIM

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I dedicate this thesis to my lovely parents,
Hasim bin Mohd Said and Rositah binti Abdul Kadir;
my wonderful family members and
all my friends who's helping me throughout this thesis.
Thank you...

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ABSTRACT

A series of tellurite glasses of composition (70–x–y)TeO_2–15Li_2CO_3–15Nb_2O_5–$x$$Er_2O_3$–$y$$Nd_2O_3$ doped Er^{3+} and Nd^{3+}, with x=0, 1.0 mol%; $0 \leq y \leq 1.0$ mol% were prepared by using melt quenching technique. The glass phase and thermal behaviour were investigated using X-ray diffraction (XRD) and differential thermal analysis (DTA) while the structural and optical properties were investigated using Fourier transform infrared spectroscopy (FTIR), ultraviolet–visible–near infrared spectroscopy (UV–VIS–NIR) and photoluminescence spectroscopy. The XRD spectra confirmed that the glass was amorphous as no sharp peaks were observed. The thermal parameters particularly the thermal stability is around 291.5°C and Hruby criterion of around 0.82 to 0.93. The structural properties of the glass represented by the FTIR spectrum indicate that as Nd_2O_3 content increases, the sharp infrared absorption peaks shifted from 474.7 cm^{-1} to 499.4 cm^{-1}. These peaks are due to Nb–O, Te–O–Te and O–Te–O bond linkage bending vibration. For TeO_4 trigonal bipyramid, the peak occurred at 676.5 cm^{-1} whereas for TeO_3 trigonal pyramid, two infrared band peaks were observed at 787.5 cm^{-1} and 887.6 cm^{-1}. The absorption peaks around 1382.7 cm^{-1} is due to the Te–O–Nb stretching vibration while peaks at 1635.5 cm^{-1} and 3411.7 cm^{-1} are due to the stretching vibrations of the hydroxyl group participating in the strong metal bonding as well as in the hydrogen bonding, respectively. The UV–VIS–NIR spectrum exhibits absorption peaks corresponding to transitions from both ground state of Erbium, $^4I_{15/2}$ to the excited state of $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4I_{9/2}$, $^2H_{9/2}$, $^4I_{11/2}$, $^4I_{3/2}$ and $^4I_{13/2}$ and Neodymium, $^4I_{9/2}$ to the excited state of $^2G_{11/2}$, $^2G_{9/2}$, $^2G_{7/2}$, $^4F_{9/2}$, $^4I_{3/2}$ and $^4I_{15/2}$. The up conversion was observed in the luminescence spectra by the red emission at around 633 nm which is due to the transition from $^4F_{9/2} \rightarrow {}^4I_{9/2}$. The down conversion was represented by the green emission at 497 nm due to transition from $^2G_{9/2} \rightarrow {}^4I_{9/2}$.

ABSTRAK

Sistem kaca tellurit dengan komposisi $(70\text{-}x\text{-}y)TeO_2$–$15Li_2CO_3$–$15Nb_2O_5$–$xEr_2O_3$–$yNd_2O_3$ dop Er^{3+} dan Nd^{3+}, dengan x=0, 1.0 mol%; $0 \leq y \leq 1.0$ mol% telah diperoleh menggunakan teknik pelindapan leburan. Fasa dan ciri terma kaca telah dikaji menggunakan teknik pembelauan sinar–X (XRD) dan penganalisa perbezaan terma (DTA). Struktur dan sifat optik sampel telah dikaji menggunakan spektroskopi inframerah transformasi Fourier (FTIR), spektroskopi ultraungu–nampak–inframerah dekat (UV–VIS–NIR) dan spektroskopi fotopendarcahaya. Spektrum XRD mengesahkan sampel kaca adalah amorfus kerana tiada sebarang puncak tajam dicerap. Parameter terma terutamanya kestabilan terma adalah 291.5°C dan kriteria Hruby berlaku dalam lingkungan 0.82 hingga 0.93. Selain itu, spektrum FTIR menunjukkan bahawa pertambahan kandungan Nd_2O_3 menyebabkan puncak penyerapan Inframerah akan beranjak dari 474.7 cm^{-1} kepada 499.4 cm^{-1}. Anjakan ini menunjukkan mod getaran lenturan ikatan Nb–O, Te–O–Te dan O–Te–O. Di samping itu, mod getaran trigonal bipiramid TeO_4 berlaku pada 676.5 cm^{-1} manakala dua getaran trigonal piramid TeO_3 masing-masing berlaku pada 787.5 cm^{-1} dan 887.6 cm^{-1}. Puncak penyerapan disekitar 1382.7 cm^{-1} terhasil disebabkan oleh getaran regangan Te–O–Nb dan puncak penyerapan sekitar 1635.5 cm^{-1} dan 3411.7 cm^{-1} adalah disebabkan oleh getaran regangan kumpulan hidroksil yang terlibat dalam ikatan logam dan ikatan hidrogen. Spektrum UV–VIS–NIR yang telah diperoleh menunjukkan puncak penyerapan dengan transisi dari keadaan asas Erbium, $^4I_{15/2}$ ke keadaan pengujaan $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4I_{9/2}$, $^2H_{9/2}$, $^4I_{11/2}$, $^4I_{3/2}$ dan $^4I_{13/2}$. Puncak penyerapan yang berlaku pada Neodymium melalui transisi dari keadaan asas, $^4I_{9/2}$ kepada keadaan pengujaan $^2G_{11/2}$, $^2G_{9/2}$, $^2G_{7/2}$, $^4F_{9/2}$, $^4I_{3/2}$ dan $^4I_{15/2}$. Transisi menaik dicerap dalam spectrum pendarcahaya oleh pancaran merah pada sekitar 633 nm yang disebabkan oleh transisi dari $^4F_{9/2} \rightarrow {}^4I_{9/2}$. Transisi menurun diwakili oleh pancaran hijau pada 497 nm yang disebabkan oleh transisi dari $^2G_{9/2} \rightarrow {}^4I_{9/2}$.

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$\alpha\ (v)$	- Urbach function
α	- absorption coefficient
$\alpha\omega$	- fundamental of absorption edge
ΔE	- width of band tails of localized states
λ	- wavelength
$\Phi/\ \Phi_1/\ \Phi_2$	- photon incident flux
ω	- frequency dependence
$\hbar\omega/\ hv$	- photon energy
μ	- reduced mass of cation – anion molecules
A	- absorbance
A	- constant
ax	- axial
B	- constant
BO	- bridging oxygen
c	- speed of light
CB	- conduction band
CR	- cross relaxation
d	- distance between atomic layers in crystal
E_f	- final state of energy
E_g	- energy gap

E_i	- initial state of energy
E_{opt}	- optical band gap
Er_2O_3	- Erbium (III) oxide
ESA	- excited state absorption
ET	- energy transfer
ETU	- energy transfer upconversion
eq	- equatorial
f	- force constant
FTIR	- Fourier Transform Infrared
H_R	- Hruby criterion
I	- anti – stokes emission intensity
IR	- infrared
k	- momentum
Li_2CO_3	- Lithium carbonate
m_o	- atomic weight of cation o
m_r	- atomic weight of cation r
n	- integer
Nb_2O_5	- Niobium pentoxide
NBO	- non – bridging oxygen
Nd_2O_3	- Neodymium (III) oxide
NIR	- near infrared
p	- index characterize of optical absorption process
PA	- photon avalanche
q	- phonon
R_1	- ground pumping rates
R_2	- excited pumping rates
SRO	- short – range order

T	- temperature
t	- thickness of glass sample
tbp	- trigonal bipyramid
T_c	- crystallization temperature
TeO_2	- Tellurium dioxide
T_g	- glass transition temperature
T_I	- temperature interval
T_m	- melting point temperature
tp	- trigonal pyramid
UV	- ultraviolet
V	- specific volume
k	- wave number
VB	- valence band
Vis	- visible
XRD	- X – ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

Glass history is very long but yet interesting to know and understood. Moreover, glasses are known as the oldest as well as the newest materials in the world where it is used for variety applications in everyday life. Glass has been considered by researchers in many applications. Glass has no long-range order, when there is no regularity in the arrangement of its molecular constituents on a scale large than a few times the size of these groups (Doremus, 1973). Lack of precise experimental information on the properties and structure of glass or even amorphous solids are considered difficult in theoretical analysis for researcher. Therefore, research needs to fill this gap for future benefits where including fundamental bases on new optical glasses together with new application such as second- harmonic generation, optical switches, third-order-nonlinear optical materials, optical amplifiers, waveguide lasers, bulk lasers and up- conversion glasses (El- Mallawany, 2002; Hai Lin *et.al.*, 2005).

A solid lacking long range positional order is called non-crystalline solids (NCS) where is produced by melt-cooling generally referred to as glass (Prabhat, 1996). There are many techniques can be used to form glass material including cooling from the liquid state (Burger, 1992; El-Moneim, 2002; Hai Lin *et.al.*, 2003; Jianhu *et.al.*, 2003), pressure quenching (Holloway *et.al.*, 1992; Caprion *et.al.*, 2002), sol-gel formation, condensation from vapour, solution hydrolysis, anodization and bombardment of crystal by high energy particles or shock wave (Dehelean *et.al.*, 2009). The most important and widely used glass formation is cooling from liquid state. Primary reasons due to the research on glass and non-crystalline solids are divided into three where the first reason suggested all liquids could be rendered to glassy state if the crystal growth were suppressed where it was based on theoretical understanding twenty years ago. The experimental research is the second reason that shown many non- crystalline solids can be prepared by other process other than cooling from liquid state. Lastly the third reason is the increasing awareness in industry of fabrication viewpoint concludes that glasses are better than crystal where glass can play an important role in electronics sector (Sulhadi, 2007).

The most stable oxide of tellurium (Te) is tellurium dioxide (TeO_2) with the melting point of 773 ^{o}C. Based on fundamental of chemistry, transitional position of Te between metals and non-metals are long held special significance and tellurium oxide are stable which attract researchers (El-Mallawany, 2002). Furthermore, the valance characteristics of Te results in two sets of Te-O distance conclude that TeO_2 is a covalent with highly deformed octahedron structure. In addition, three tellurium atoms shared with oxygen atom where symmetry requirements force distortion of octahedral to accommodate them into a regular repeating lattice. Therefore, this distortion could produce a structure energetically similar to the vitreous state where only short- range orders appear (Sulhadi, 2007). Moreover, TeO_2-based glasses has scientific and technical interest because of their low melting temperature, high refractive index, high dielectric constant and high transmittance from ultraviolet (UV) to near infrared (NIR) (Burger *et.al.*, 1992; Annapurna *et.al.*, 2000; Ovcharenko *et.al.*, 2001; El-Moneim, 2002). TeO_2 have low phonon energies, high refractive indices, high rare earth solubility, infrared transmission up to 5 μm and high gain per unit length (Oermann *et.al.*, 2009) indicates a potential application in laser host and pressure sensors such as optical waveguide laser and amplifier (Hai

Lin *et.al.*, 2003). Zinc tellurite glasses is a suitable host for optically active rare-earth ions (Rosmawati *et.al.*, 2007) because it prove low-phonon-energy about $780cm^{-1}$ (Shaoxiong *et.al.*, 2008) environment which minimize nonradiative losses as well as possess good chemical durability and optical properties (Sulhadi *et.al.*, 2007).

Rapid developments in laser technology and construction of magneto-optic device in the past decade have lead to an extensive study on rare-earth glasses. More important the understanding of structures of the materials, together with the preparation of high-quality glasses and the development of new preparative techniques are far behind the needs of today's technology. Incorporation of rare-earth into various glass oxides is a key to the optical devices development such as infrared lasers, infrared-visible converters, fibre and waveguide amplifiers for the use of optical transmission network (Azman *et.al.*, 2010; Azman, 2012). In addition, trivalent rare-earth ions (RE^{3+}) are attractive as active elements in materials because of many fluorescence state to choose from 4f electron configuration where most of it located in visible range which contributes to easy pumping, tunable dye laser. Therefore, these glasses usually called as activated glasses where they are capable of emitting radiation (luminescence) upon excitation (Azman, 2012). Hence, rare-earth ion will have capability of colouring glass through absorptive transitions together with a set of states capable of emitting light (Sulhadi, 2007).

An addition of Erbium (Er^{3+}) and Neodymium (Nd^{3+}) into tellurite glass will lead to a high possibility of active media for a solid-state lasers operating in three different regions which are the visible, near infrared (NIR) and infrared region (IR) spectral region (Azman, 2012). Energy transfer between both of these rare-earth ions exist as energy absorbed by one of the rare earth, Er^{3+} ion which appear as fluorescence may be lost to the lattice through the intermediary of second rare-earth, Nd^{3+} ion where energy gained by one of rare-earth ion via transfer from a second could produce an enhancement of its emission (Shen *et.al.*, 2008).

Lithium Niobate based tellurite glass codoped with rare earth has been receiving special interest due to their ability to enhance the performance of tellurite glass for their applications especially in laser and photonics. From the previous research, there is less research on the photoluminescence investigation of the optical up-conversion in rare earth, Erbium and Neodymium co-doped with lithium niobate tellurite glass although both of the elements given rise to various luminescence band in many host materials (Longjun *et.al.,* 2007; Shiqing *et.al.,* 2008).

Luminescence is the emission of light by a substance which occurs when an electron returns to the electronic ground state from an excited state and loses its excess energy as a photon. There are energy gap for conducting electrons; therefore, there is an energy gap between the conduction and valence electron states. Under normal conditions electrons are forbidden to have energies between the valence and conduction bands. If a light particle (photon) has energy greater than the band gap energy, then it can be absorbed and thereby raise an electron from the valence band up to the conduction band across the forbidden energy gap. Moreover, the up-conversion process happens when the excitation with lower energy radiation gives rise to higher energy luminescence (Ruan *et.al.*, 1998). The up-conversion in rare-earth doped materials caused by excited- state absorption or energy transfer from different rare-earth ions (Huang *et.al.*, 2005). In addition, Er^{3+} and Nd^{3+} doping are important to obtain efficient up-conversion because of the host material, in this case is tellurite which has low-phonon-energy can reduce the non-radiative loss due to multiphonon relaxation to achieve strong up-conversion luminescence (Shiqing *et.al.*, 2005) and it is proved to be the most stable host material (Azman *et.al.*, 2010). As for the excitation used for up conversion are 585nm excitation (Azman, 2012) and 980nm laser diode (LD) excitation (Shiqing *et.al.,* 2008)

Host material, lithium niobate tellurite is also an important factor for obtaining high efficient up-conversion emission (Lakshminarayana *et.al.,* 2008) where the low-phonon-energy can reduce the nonradiative loss due to multiphonon relaxation and thus yields a strong up-conversion signal (Zhe Jin *et.al.,* 2007). In addition, when the dopant, Er and Nd concentration is increase; the results in lower fluorescence are higher (Eakins *et.al.*, 2004). The excitation with lower energy radiation happened will gives rise to higher energy luminescence, then the up-conversion process took place (Ruan *et.al.*, 1998). Furthermore, there is no exact excitation spectrums used to find the up-conversion of this glass. Moreover, the up-conversion phenomenon usually used in explaining about laser processes where the emission in Er^{3+}:Nd^{3+} tellurite glass always occurs at 4f electron level (Cherif *et.al.*, 2010). In this study, the interaction among optically excited ions of Er^{3+}:Nd^{3+} systems in tellurite glass which leads to the discovery of new laser based on the energy transfer in neither single ions or multi ions material are presented together with a suitable excitation used to find the emission of this glass which cause an enhancement of luminescence emission (Xujie *et.al.*, 2008).

1.2 Research Problem Statement

Rare-earth doped tellurite glasses doped with two rare-earths, erbium and neodymium has been receiving special interest due to their ability to enhance the performance of tellurite glass for their applications. Even though there are numbers of research on tellurite glass has been done, yet the characteristics of Er^{3+} and Nd^{3+} ions doped tellurite glass has not been fully investigated. In addition, in spite of few experiments on tellurite doped with Er^{3+} and Nd^{3+} ions, the clear explanation about the role played are still lacking. Few studies have been done in this system but are limited to certain properties. Therefore, in the present study we will investigate the role played by the neodymium dopant on optical properties by spectroscopic techniques which are UV-Vis, Photoluminescence and FTIR spectrometer.

1.3 Research Objectives

In this research, a stable with wide formation ranges of tellurite glasses are prepared to full fill these objectives which are:

i. To prepare $Li_2CO_3 – Nb_2O_5 – TeO_2$ (LNT) where $(70\text{-x-y})TeO_2 – 15Li_2CO_3 – 15Nb_2O_5 – (x)Er_2O_3 – (y)Nd_2O_3$ system (x = 0 mol % and 1.0 mol %; 0 mol % $\leq$ y $\leq$ 1.0 mol %).

ii. To characterize thermally, optically and structurally of Er^{3+} and Nd^{3+} doped tellurite glasses.

iii. To analyze the emission of the glass system – the role of Nd^{3+} doped.

1.4 Scope of Research

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

- Preparation of Erbium and Neodymium co- doped with lithium niobate tellurite by using the melt quenching technique.
- Determination of thermal stability of glass system using Differential Thermal Analysis.
- Determination of the structural and optical properties of glass system using UV-VIS-NIR Spectroscopy, Photoluminescence Spectroscopy and Infra-Red Spectroscopy.

- Determination of the emission and excitation spectra of the glass and determine the optical emission based on the results gain on experimental.

1.5 Significance of Research

This study is fundamentally important to explain the role played neodymium dopant inside tellurite glass by analyzing the absorption, emission, and FTIR spectra of the glass. Hence, from this systematic experimental of fabrication and spectroscopic studies of this sample it would give better suggestions about how to increase the efficiency of tellurite glass for their applications.

1.6 Thesis Outline

This thesis describes the preparation and characterization of Erbium and Neodymium doped tellurite glasses prepared by melt quenching technique. This thesis has been divided into five main chapters. Chapter 1 is the introduction part where the research studies reviewed. In addition, it will also describe the problem statement, objectives, scope of study, choice of glass system and significance of the research as well as the thesis outline. Chapter 2 will describe some theories related to glass such as glass formation, general glass structure as well as structure of tellurite glass. Theory on thermal analysis, XRD, FTIR, UV–Vis–NIR and luminescence spectroscopy will be elucidated in details in this chapter. Chapter 3 will focus on glass preparation, research methodology and experimental techniques. In Chapter 4, all the experimental results and discussion will be given in detail. A special attention will be given on the effect of Er^{3+} and Nd^{3+} doped on thermal, structural and optical properties of tellurite glass. Finally Chapter 5 will present the major conclusions that can be derive from discussion mentioned in Chapter 4 and future outlook will be presented in this chapter.

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