

**SPECTROSCOPIC PROPERTIES OF ERBIUM DOPED TELLURITE
GLASS**

SITI FATIMAH BINTI ISMAIL

UNIVERSITI TEKNOLOGI MALAYSIA

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SITI FATIMAH BINTI ISMAIL

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This thesis is specially dedicated to:

my beloved father (Ismail bin Mamat)
and my mother (Latipah binti Abdul Rahman),
my siblings (Adam bin Ismail, Zaini Arif bin Ismail, Sulaiman bin Ismail),
and my beloved friend (Allahyarham Nik Ahmad Irham bin Nik Rusli)

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ABSTRACT

Er³⁺ doped tellurite glasses of composition (75-x)TeO₂-25ZnO-5Na₂O-xEr₂O₃ with x = 0, 0.2, 0.4, 0.7, 0.9, 1.1 and 1.4 mol% were successfully prepared by melt quenching technique. No definite peaks were found from the X-ray diffraction pattern, thus verified the amorphous nature of glass. However, the X-ray diffraction pattern for heat treated glass showed a wide peak which indicated the occurrence of nanocrystallite particle in the sample. The crystallite size was determined by Scherrer equation and confirmed by the image obtained from transmission electron microscopy technique. The vibrational study was conducted using Fourier transform infrared (FTIR) spectroscopy in the range of 4000 cm⁻¹ – 400 cm⁻¹. It was observed that, four absorption peaks occur around 1600 cm⁻¹ – 3400 cm⁻¹, 666 cm⁻¹ – 755 cm⁻¹, 546 cm⁻¹ – 684 cm⁻¹ and 465 cm⁻¹ which were due to the stretching mode vibration of OH bond, TeO₄ trigonal bipyramids and TeO₃ trigonal pyramids, Er-O bond and Zn-O tetrahedral bond, respectively. The FTIR spectra for heat treated samples generated a higher intensity absorption band located at 564 cm⁻¹, 573 cm⁻¹, 669 cm⁻¹ and 688 cm⁻¹. The thermal stability of the glass was investigated using differential thermal analyzer. The thermal parameters, such as the glass transition temperature (T_g), crystalline temperature (T_c), melting temperature (T_m) and thermal stability (T_c-T_g) were determined. It was found that, this glass system has glass formation range around 51.47°C - 99.35°C. The emission spectrum was recorded using photoluminescence spectrometer at room temperature. From the luminescence spectra, blue and green emission bands associated with Er³⁺ transition at (382 nm) ⁴G_{11/2}→⁴I_{15/2}, (465 nm) ⁴F_{5/2}→⁴I_{15/2}, (501 nm) ²H_{11/2}→⁴I_{15/2} and (550 nm) ⁴S_{3/2}→⁴I_{15/2} with an excitation wavelength λ_{exci} = 786 nm were observed. Meanwhile, the luminescence intensity for heat treated samples was slightly higher. The absorption characteristic was determined using ultra violet-visible spectroscopy. It was observed that, the optical band gap (E_g) and the Urbach energy (ΔE) was in the range 2.89 eV - 2.18 eV and 0.59 eV - 0.15 eV, respectively depending on the Er₂O₃ concentration. For the heat treated samples, the optical band gap (E_g) and the Urbach energy (ΔE) was in the range of 2.72 eV - 2.68 eV and 0.54 eV - 0.21 eV, respectively.

ABSTRAK

Kaca tellurite berdop Er^{3+} dengan komposisi $(75-x)\text{TeO}_2-25\text{ZnO}-5\text{Na}_2\text{O}-x\text{Er}_2\text{O}_3$ dengan $x = 0, 0.2, 0.4, 0.7, 0.9, 1.1$ dan 1.4 % mol telah berjaya disediakan menggunakan teknik pelindapan leburan. Corak pembelauan sinar- X tidak menemui puncak yang pasti, ini mengesahkan bahawa kaca tersebut adalah amorfus. Walau bagaimanapun, corak pembelauan sinar- X bagi kaca yang diberi rawatan haba menunjukkan puncak yang lebar menandakan adanya zarah nano kristalit dalam sampel. Saiz kristalit telah ditentukan daripada persamaan Scherrer dan ditentusahkan dari imej mikroskop transmisi elektron. Kajian getaran dijalankan menggunakan spektroskopi Fourier transformasi infra-merah (FTIR) dalam julat $4000 \text{ cm}^{-1} - 400 \text{ cm}^{-1}$. Empat puncak penyerapan berlaku di sekitar $1600 \text{ cm}^{-1} - 3400 \text{ cm}^{-1}$, $666 \text{ cm}^{-1} - 755 \text{ cm}^{-1}$, $546 \text{ cm}^{-1} - 684 \text{ cm}^{-1}$ dan 465 cm^{-1} yang masing-masing disebabkan oleh mod getaran regangan OH, TeO_4 *trigonal bipyramids* dan TeO_3 *trigonal pyramids*, Er-O and Zn-O *tetrahedral bond*. Spektrum FTIR bagi sampel yang terawat haba menghasilkan keamatan tinggi di jalur penyerapan pada 564 cm^{-1} , 573 cm^{-1} , 669 cm^{-1} and 688 cm^{-1} . Kestabilan terma kaca telah dikaji menggunakan analisis pembezaan terma. Parameter terma seperti suhu transisi (T_g), suhu penghabluran (T_c), suhu lebur (T_m) dan kestabilan terma (T_c-T_g) telah ditentukan. Sistem kaca ini didapati mempunyai julat pembentukan kaca di sekitar $51.47 \text{ }^\circ\text{C} - 99.35 \text{ }^\circ\text{C}$. Spektrum pancaran telah direkod menggunakan spektrometer fotoluminesen pada suhu bilik. Dari spektra fotoluminesen, jalur biru dan hijau yang dikaitkan dengan peralihan Er^{3+} di $(382 \text{ nm}) \text{ }^4\text{G}_{11/2} \rightarrow \text{}^4\text{I}_{15/2}$, $(465 \text{ nm}) \text{ }^4\text{F}_{5/2} \rightarrow \text{}^4\text{I}_{15/2}$, $(501 \text{ nm}) \text{ }^2\text{H}_{11/2} \rightarrow \text{}^4\text{I}_{15/2}$ dan $(550 \text{ nm}) \text{ }^4\text{S}_{3/2} \rightarrow \text{}^4\text{I}_{15/2}$ dengan jarak gelombang pengujaan $\lambda_{\text{exci}} = 786 \text{ nm}$ telah diperolehi. Sementara itu, keamatan untuk sampel terawat haba adalah lebih tinggi berbanding sampel asas. Ciri penyerapan telah ditentukan menggunakan spektroskopi ultralembayung cahaya nampak. Jurang jalur optik (E_g) dan tenaga Urbach (ΔE) masing-masing adalah dalam julat $2.89 \text{ eV} - 2.18 \text{ eV}$ dan $0.59 \text{ eV} - 0.15 \text{ eV}$, bergantung kepada kepekatan Er_2O_3 . Jurang jalur optik (E_g) dan tenaga Urbach (ΔE) bagi sampel terawat haba masing-masing adalah dalam julat $2.72 \text{ eV} - 2.68 \text{ eV}$ and $0.54 \text{ eV} - 0.21 \text{ eV}$.

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LIST OF SYMBOLS

4fn	-	Shell configuration belong to lanthanide series
4f	-	Orbital belong to lanthanide series
α	-	Absorption coefficient
A	-	Absorption intensity
CB	-	Conduction band
d	-	Sample thickness
E_g	-	Optical energy gap
Er^{3+}	-	Trivalent erbium ion
Er_2O_3	-	Erbium oxide
ESA	-	Excited state absorption
eV	-	Electron volt
FTIR	-	Fourier transformed infrared
ΔE	-	Width of the band tails
<i>hkl</i>	-	Crystal plane orientation index
IR	-	Infrared
Na_2O	-	Sodium oxide
NBO	-	Non-bridging oxygen
n	-	Integer number, called the order of reflection
nm	-	nanometer
RE	-	Rare earth
θ	-	Bragg angle
Δt	-	Differential temperature
T	-	Temperature
T_c	-	Crystallization temperature
T_g	-	Glass transition temperature

T_m	-	Glass melting temperature
TeO_2	-	Tellurium oxide
UV	-	Ultraviolet
Vis	-	Visible
$\bar{\nu}$	-	Wavenumber
ω	-	Frequency
θ	-	Angle
λ	-	Wavelength
XRD	-	X-ray diffraction
ZnO	-	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Glass is an amorphous material and has a degree of short range. Glass also known as a hard, brittle, corrosion resistant, inert and transparent material. Glass is defined as the mixture of inorganic material that has been cooled to a rigid condition without be a crystal. Glass also defined as a material produced as a result of cooling of the material from the liquid state to a condition in which the mass is rigid and the viscosity increases. Glass is generally defined as a product of inorganic material produced by the combustion process (melting), then cooled to a rigid solid state without crystallization (Sahar,1998). Glass has excellent features in a wide range of fields such as optically homogenous, transmits light well, excellent solid solvent into which nearly all elements can be melted to produce diverse materials and various shapes can be easily formed.

There a several types of glass found such as silicate glass, borate, phosphate and telluride. Tellurite glasses contain tellurium oxide (TeO_2) as the main component. Tellurite glasses have numerous favorable optical properties such as a wide transmission range from ultraviolet to mid-infrared (0.35-5.0 μm), lower

phonon energy ($700\text{-}750\text{ cm}^{-1}$) than other known glass hosts such as borates or silicates, high linear and nonlinear refractive index, good stability and resistance to moisture. Tellurite glasses are of particular interest because of not only their low transition temperature but also their excellent infrared transmission (Sahar,1995).

In this production of tellurite based glass, zinc oxide (ZnO) and erbium (III) oxide (Er_2O_3) added to strengthen the glass structure. Zinc telluride is one of the more common semiconducting materials used in optoelectronics. Zinc telluride is one of the II-VI compound and which have special electrical and optical properties. This special properties make ZnTe is important for development of various semiconductor devices such as photodetectors, light emitting diode, laser diodes, and solar cells. Zinc telluride is a direct band gap material with band gap of 2.23-2.25 eV. It is usually a p-type semiconductor. Zinc telluride typically has a cubic crystal structure but can be also prepared as hexagonal crystals. Zinc telluride glasses are reported to be suitable host for optically active rare earth ions. The structure of TeO_2 -based glasses is also of interest because there are two types of basic structure unit, namely TeO_4 trigonal bipyramids (tbp) and TeO_3 trigonal pyramid (tp) (Aida *et al.*, 2000). Water in glass influences a variety of glass properties such as reduces the viscosity and increases the rates of structural relaxation and phase separation of glass. It has been reported elsewhere that IR absorption in TeO_2 -ZnO glasses is very much depend on the ZnO content. As the amount of ZnO content became higher, the absorption peak due to the Te-O bond vibration decreases, while the position absorption peak due to the Te-O bonds increases. Structural measurements in telluride glasses are very essential to interpret their physical and chemical properties.

Erbium (III) oxide is one of the rare earth in lanthanide series and it pink solid. Erbium sometimes used as a colouring for glasses and a dopant for optical fibers and optical amplifiers. Melting and boiling points of the Erbium (III) oxide is very high at $2344\text{ }^\circ\text{C}$ and $3290\text{ }^\circ\text{C}$. Erbium (III) oxide has high density of 8.64 gcm^{-3} and molar weight 382.56 g/mol . It has a strong ionic bond and can increase mechanical properties of glass. The main application of erbium is as an activator in

the manufacture of laser glass because the glass containing erbium laser will emit radiation when given the appropriate energy.

1.2 Research background

Nowadays, rare earth doped tellurite nanoglass had become more explore in the research due great application on the optical telecommunication. Nanoglass or nanocrystalline materials are characterized by a microstructure length or grain size of up to about 100 nm. Method of fabricating useful nanoglass through the controlled nucleation of glass has stimulated a broad research effort in the field of glass nucleation in 1957 by Stookey.

Glasses would crystallize spontaneously if they were brought to thermodynamics equilibrium at room temperature. Why glasses remain amorphous when they are undercooled below the equilibrium melting temperatures of their crystalline phases and this situation is necessary to understand the principles of catalyzed crystallization. There are two possible reasons either the nucleation rate or the growth rate is negligibly small at all temperatures. The extremely slow nucleation rate is the critical factor preventing crystallization of glasses. Once growth has started, crystals grow sizable rates in many glasses at suitable temperatures. Evidently, there are no nucleation in the conventional glass melt and prevent from homogenous nucleation. Solution to induce nucleation and crystal growth is by using concept of heat treatment.

A lot of work have been done on rare-earth doped glass ceramic materials with active ions embedded in the crystalline phase (Mattarelli *et al.*,2004). The rare-earths, especially Er^{3+} which are optically active ions commonly embedded in tellurite crystalline phase and several extraordinary advantages have been observed

for optical properties of rare earth ions in tellurite nanocrystals. It was also found that the intensity of the frequency up-conversion luminescence of transparent Er^{3+} -doped tellurite based crystallized glasses at around 550 nm is strong compared with their precursor glass (Sakai *et al.*, 2000; Hirano *et al.* 2001) .

In this study, Er^{3+} doped tellurite glass will be prepared by using conventional melt quenching technique with various concentration of erbium. In characterizing the samples, the spectroscopic studies of the tellurite glass will be measured. As a result, more information will be collected about erbium doped tellurite glass.

1.3 Statement of problem

Tellurite glasses have excellent optical and chemical properties. Tellurite glasses are promising candidates for optical fiber laser and optical amplifier application. It has been reported that tellurite glasses are good for hosting rare earth ions since they provide a low phonon energy environment to minimize the nonradiative losses. Erbium doped tellurite glass exhibit more good optical quality, stability and also have been used in many optical applications. However, to our knowledge not many spectroscopic properties of tellurite glass doped Er^{3+} have been reported. It is therefore the aims of this study to prepare and characterizes the spectroscopic properties in term of absorption, transmission, structural and emission in order to obtain clear understanding of the relationship between structural and optical properties of Er^{3+} doped tellurite glass.

1.4 Objectives

The objectives of this research are:

- i. To prepare $\text{TeO}_2\text{-ZnO-Na}_2\text{O-Er}_2\text{O}_3$ with different Er_2O_3 content by using melt quenching technique.
- ii. To characterize the structural properties by using x-ray diffraction, transmission electron microscopy (TEM) and fourier transform infra red (FTIR) spectroscopy.
- iii. To characterize the thermal properties by using differential thermal analyzer (DTA).
- iv. To characterize the optical properties by using photoluminescence and UV-Visible spectroscopy.

1.5 Scope of study

In order to achieve the above objectives the works have been focused on the given scope:

- i. The preparation of the glass based on $(75-x) \text{TeO}_2\text{-}25\text{ZnO-}5\text{Na}_2\text{O-}x\text{Er}_2\text{O}_3$ ($x = 0, 0.2, 0.4, 0.7, 0.9, 1.1, 1.4$ mol %) glass system will be provided through the melt quenching technique.
- ii. Determination of crystallite particles size by Scherrer equation.
- iii. Characterization of transmission spectra by using Fourier Transform Infra Red (FTIR) spectroscopy.
- iv. Characterization of absorption in the UV and visible region using UV-VIS spectrophotometer.

- v. Characterization of emission using photoluminescence spectroscopy.

1.6 Significant of research

Research on tellurite glass doped erbium is very important because of the possible photonic applications. From this research the information about spectroscopic properties of $\text{TeO}_2\text{-ZnO-Er}_2\text{O}_3$ will be found out and as well as glass will be enhanced the development of new optical technology in glass science and engineering in last century.

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