HOLMIUM CONCENTRATION DEPENDENT STRUCTURAL AND OPTICAL PROPERTIES OF ZINC TELLURITE GLASS

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To my beloved family

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ABSTRACT

This thesis examined the effects of Ho_2O_3 dopant concentration variation on the physical, optical, thermal and structural properties of zinc tellurite glass system. Glass with composition of $(80 - x)$ $TeO_2 - 20ZnO - xHo_2O_3$, where $x = 0.0$, 0.5, 1.0, 1.5 and 2.0 mol% were prepared using melt quenching method. Samples were characterized by X-ray diffraction (XRD) measurement, Energy-dispersive X-ray (EDX) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, Raman spectroscopy, Differential Thermal Analysis (DTA), Archimedes Method, UV-Visible-NIR (UV-Vis-NIR) spectrometer and Photoluminescence (PL) spectrometer. XRD pattern verified the amorphous nature of prepared sample. EDX spectra detected the presence of appropriate elements in the glass. FTIR spectra showed all the functional group such as Zn−O, TeO_4 and TeO_3 in the range of 443 – 487, 654 – 675 and 760 – 763 cm^{-1}. Raman spectrum showed three bands at 463, 530 and 676 cm^{-1} of Te–O–Te linkages, TeO_4 and TeO_3 respectively. The DTA thermogram showed the peak for glass transition temperature, T_g (367 °C), melting temperature, T_m (632 °C) and crystallization temperature, T_c (458 °C). The density, ρ and molar volume, V_m of the prepared glass sample was varied respectively in the range of 5.554 − 5.580 g cm^{-3} and 26.088 − 27.270 cm^3 mol^{-1} with increase of Ho_2O_3 concentration. The absorption spectra showed seven bands centered at 418, 454, 480, 541, 646, 894 and 1155 nm which assigned to the transitions from the ground state to the excited $^5G_5$, $^5G_6$, $^5F_3$, $^5F_4$ + $^5S_2$, $^5F_5$, $^5I_5$ and $^5I_6$ levels of Ho^{3+}. The indirect band gap energy, direct band gap energy and Urbach energy were varied from 3.31 – 3.36, 3.21 – 3.30 and 0.17 – 0.21 eV respectively with the changes of holmium concentration. The PL spectra displayed two peaks at 657 nm (red region) and 753 nm (NIR region) for $^5F_5 \rightarrow {}^5I_8$ and $^5S_2 \rightarrow {}^5I_7$ transitions.

ABSTRAK

Tesis ini mengkaji kesan variasi kepekatan dopan Ho_2O_3 pada sifat fizikal, optik, terma dan struktur sistem kaca tellurite zink. Kaca dengan komposisi $(80 - x)$ TeO_2 $-$ 20ZnO $-$ $x Ho_2O_3$, dengan x = 0.0, 0.5, 1.0, 1.5 dan 2.0 mol% disediakan menggunakan kaedah pelindap-kejutan leburan. Sampel dicirikan oleh pengukuran pembelauan sinar-X (XRD), spektroskop penyebaran tenaga X-ray (EDX), spektroskop tranformasi infra merah (FTIR), spektroskop Raman, analisis perbezaan terma (DTA), cara Archimedes, spektrometer UV-Vis-NIR dan spektrometer fotoluminisan (PL). Corak XRD mengesahkan sifat amorfus pada sampel yang disediakan. Spektrum EDX mengesan kehadiran unsur-unsur yang sesuai di dalam kaca. Spektrum FTIR menunjukkan semua kumpulan berfungsi seperti Zn$-$O, TeO_4 dan TeO_3 dalam lingkungan 443 – 487, 654 – 675 dan 760 – 763 cm^{-1}. Spektrum Raman masing-masing menunjukkan dua kumpulan pada 463, 530 dan 676 cm^{-1} untuk hubungan Te$-$O$-$Te, TeO_4 dan TeO_3. DTA termogram menunjukkan puncak bagi suhu peralihan kaca, T_g (367 °C), suhu lebur, T_m (632 °C) dan suhu penghabluran, T_c (458 °C). Ketumpatan ρ, dan isi padu molar, V_m sampel kaca yang disediakan masing-masing dalam lingkungan 5.554 – 5.580 g cm^{-3} dan 6.154 – 6.258 x 10^{-24} cm^3 dengan peningkatan kepekatan Ho_2O_3. Spektrum UV-Vis menunjukkan tujuh jalur serapan berpusat pada 418, 454, 480, 541, 646, 894 dan 1155 nm yang ditugaskan untuk peralihan dari keadaan asal kepada teruja $^5G_5$, $^5G_6$, $^5F_3$, $^5F_4$ + $^5S_2$, $^5F_5$, $^5I_5$ dan $^5I_6$ tahap Ho^{3+}. Tenaga jurang band tak langsung, tenaga jurang jalur langsung dan tenaga Urbach berubah dari 3.31 – 3.36, 3.21 – 3.30 dan 0.17 – 0.21 eV masing-masing dengan perubahan kepekatan holmium. Spektra PL menunjukkan dua puncak di 657 nm (rantau merah) dan 753 nm (rantau NIR) untuk $^5F_5 \rightarrow {}^5I_8$ dan $^5S_2 \rightarrow {}^5I_7$ peralihan.

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LIST OF SYMBOLS

θ	-	Angle
M	-	Molar mass
ρ	-	Density
V_m	-	Molar volume
T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Melting temperature
ΔT_s	-	Glass stability
H	-	Hubry
E_g^I	-	Indirect optical band gap energy
E_g^D	-	Direct optical band gap energy
ΔE	-	Urbach energy
n	-	Refractive index
R_m	-	Molar refractivity
α_e	-	Polarizability

LIST OF ABBREVIATIONS

TeO_2	-	Tellurium dioxide
ZnO	-	Zinc oxide
Ho	-	Holmium
XRD	-	X-ray diffraction
DTA	-	Differential thermal analysis
FTIR	-	Fourier transform infrared
EDX	-	Energy-dispersive X-ray
UV	-	Ultraviolet
Vis	-	Visible
NIR	-	Near Infrared
PL	-	Photoluminescence

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CHAPTER 1

INTRODUCTION

1.1 Overview

In this chapter, general information about the research background of study will be explained. This study is about holmium concentration dependent structural, thermal, physical and optical properties of zinc tellurite glass. The problem statement, objectives, scope, significance and motivation of study also will be explained in this chapter. Thesis outline also will be described in this chapter.

1.2 Research Background

Tellurium dioxide (TeO_2) based glasses were considered as promising materials for optical switching devices and laser hosts because of their unique properties. They possess high refractive index, high dielectric constant, good chemical durability, excellent infrared transmittance and can be prepared at low melting temperatures [1]. TeO_2 based glasses only can form glass when metal oxides such as ZnO, Na_2O, BaO, PbO, Nb_2O_5 and WO_3 are added to it or usual preparation conditions that are varied because TeO_2 belongs to the intermediate class of glass-forming oxides. These modifier molecules can improve the glass formation ability (GFA) in glass formers by increasing entropy and by breaking chains of structural units that can cause changes in structural units [1].

Many researchers reported that the $ZnO-TeO_2$ system is basic system that has good glass-forming ability because TeO_2 in combination with ZnO forms stable glasses. Tellurite zinc oxide glass is a promising host for photonic applications because the ZnO is a good material to use as modifier due to its excitation binding energy about 60 meV [2]. Besides, ZnO improve the ability of glass formation and reduce the crystallization rate of tellurite network [3]. $ZnO-TeO_2$ glasses are appropriate host for optically active rare earth ions because of the wide glass-formation range that close to the extremum for binary tellurite glasses. These glasses system also very useful medium for ultralow loss (1 dB 1000 m^{-1}) optical fibers for wavelengths in the 3.5 – 4 μm regions as a basis for multicomponent optical glass synthesis. So, tellurite glasses are very useful solid materials for many applications [4].

Researchers found that when tellurite glasses doped with heavy metal and rare earth oxides, it changes their density, optical and thermal properties [5]. Zinc tellurite glass appears to be excellent candidates for hosting rare earth ions because this glass provides a low phonon energy environment to minimize non-radiative loses. Rare earth ions can achieve optical emission from solid materials because of their sharp intra 4f−transitions and abundant energy level structures. Holmium allows multiple excited state absorptions which could trigger a wide emission spectrum and became important among rare earths [6].

Holmium ion displays several electronic transitions in the visible and infrared regions among various rare earth ions, so there are many laser transitions in its emission spectrum. Holmium ion also shows eye safe potential laser with a low threshold action even at room temperature that have very useful applications in atmospheric communication systems [7]. When the glasses are mixed with different network modifying ion, researchers expected that the structural modifications and local field variations around holmium ion with various luminescence transitions of holmium ions [8].

1.3 Problem Statement

Tellurite glasses have drawn much attention as promising candidates for many optoelectronic applications due to their advantageous properties, such as high refractive index, relatively low phonon energy, good visible and infrared transmissivity, suitability for doping with rare earth elements in a wide range and good electrical properties [1]. In this study, tellurite glasses will be studied due to their advantages in lasing properties in photonic applications. Even though there are numbers of research on tellurite glass has been done, yet the structural, thermal, physical and optical properties of holmium doped zinc tellurite glass is still required. This research is needed because the best composition of holmium doped zinc tellurite glass for lasing properties is required for photonic applications in the future. Therefore, the present study will investigate the holmium concentration dependent structural, thermal, physical and optical properties of zinc tellurite glass by using X-ray diffraction (XRD), Energy-dispersive X-ray (EDX), Fourier Transform Infrared (FTIR), Raman, Differential Thermal Analysis (DTA), Archimedes method, UV-Visible (UV-Vis) and Photoluminescence (PL) spectroscopy.

1.4 Objective of Study

The objectives of study are:

i. To synthesize different holmium concentration doped zinc tellurite glass by melt quenching technique with glass compositions of $(80 - x)\ TeO_2 - 20ZnO - xHo_2O_3$ where x = 0.0, 0.5, 1.0, 1.5 and 2.0 mol%.
ii. To characterize different holmium concentration doped zinc tellurite glass for their structural, thermal, physical and optical properties.

1.5 Scope of Study

In order to achieve the above objectives, the study had been focused by several scopes. Different holmium concentration doped zinc tellurite glasses were prepared by melt quenching technique. The structural properties of glass would be characterized by using X-ray diffraction (XRD), Energy-dispersive X-ray (EDX), Fourier Transform Infrared (FTIR) and Raman spectroscopy. The thermal properties of glass also would be characterized by using Differential Thermal Analysis (DTA). Besides, the physical properties of glass would be characterized by using Archimedes method. Lastly, the optical properties of glass would be characterized by using UV-Visible (UV-Vis) and Photoluminescence (PL) Spectroscopy.

1.6 Significance of Study

Research on zinc tellurite glass doped with holmium is very important because of the possible optical and photonic applications. From this research, the information about structural, thermal, physical and optical properties of different holmium concentration doped zinc tellurite glass would be found out which would enhance the development of new optical technology in glass science and engineering in last century.

1.7 Motivation of Study

Tellurite glasses are promising glasses based on their good advantages in future photonic applications. The use of tellurite glasses may be more valuable than other glasses due to their large third-order nonlinear optical susceptibility, high refractive index and wideband infrared transmittance. Tellurite glasses also combine the qualities of good glass stability, a short wavelength UV edge, a slow corrosion rate, rare earth ion solubility and relatively low phonon energy among oxide glass formers. In addition, rare earth ions such as holmium doped zinc tellurite glass appear to be excellent among optical glasses. It is because holmium allows multiple excited state absorptions which could trigger a wide emission spectrum and displays several electronic transitions in the visible and infrared regions.

1.8 Thesis Outline

Firstly in chapter one will explain briefly about the research background, problem statement, objectives, scope, significance and motivation of this study about holmium concentration dependent structural, thermal, physical and optical properties of zinc tellurite glass.

In chapter two, a brief review of history of glass, definition of glass, preparation of glass, tellurite glasses and rare earth elements will be presented. Some literature reviews as structural, thermal, physical and optical properties holmium doped zinc tellurite glass will also be briefly given.

In chapter three, it will describe the experimental procedure of this study including glass preparation and techniques used to characterize the sample by using X-ray diffraction (XRD) spectroscopy, Energy-dispersive X-ray (EDX) spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, Raman spectroscopy, Differential Thermal Analysis (DTA), Archimedes method, UV-Visible (UV-Vis) spectroscopy and Photoluminescence (PL) spectroscopy.

In chapter four, all the result from the experiments will be presented. In all cases, data, spectra and graph will be interpreted and discussed based on the dependence of holmium concentration in zinc tellurite glass system.

Lastly in chapter five, from the results of this study, some conclusions can be draw and summarized in this chapter and future outlook of this study can be determined.

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