OPTICAL ENHANCEMENT OF SAMARIUM ERBIUM CO-DOPED ZINC TELLURITE GLASS WITHOUT AND WITH SILVER NANOPARTICLES INCORPORATION

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To myself,

for the discipline and commitment that

I have put through this journey.

To my beloved parents, siblings and friends

For their endless love and supports

To religion, nation and country.

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ABSTRACT

This thesis reported the influence of pure silver (Ag) nanoparticles (NPs) on the improvement of optical properties of samarium (Sm^{3+}) and erbium (Er^{3+}) ion co-doped oxy-zinc-tellurite glass. To achieve this goal, a series of glass samples with composition 63.75 TeO_2 + 20 ZnO + 15 $ZnCl_2$ + 0.75 Sm_2O_3 + 0.5 Er_2O_3 + *y* Ag ($0.01 \leq y \leq 0.10$ g in excess) were prepared via melt quenching method. Physical properties such as density and molar volume were determined. Glass densities and molar volumes were ranged between 5.416 - 5.326 gcm^{-3} and 26.400 - 26.864 cm^3mol^{-1}, respectively. The synthesized transparent samples revealed good thermal stability over a wide glass formation region. The thermal parameters such as the glass transition temperature, the crystallization temperature, and the melting temperature were measured using Differential Thermal Analyzer (DTA). X-ray Diffraction (XRD) pattern verified the true amorphous nature of the prepared glass and the Energy Dispersive X-ray (EDX) spectra detected the presence of right elements in the composition. Transmission Electron Microscopic (TEM) images revealed the existence of spherical Ag NPs in glassy matrix with homogeneous distribution. Fourier Transform Infrared (FTIR) spectra were recorded in the range of 400 to 4000 cm^{-1}. Incorporation of Ag NPs in the glass host was found to shift the infrared bands slightly. It showed stretching of OH groups (3478 to 3453 cm^{-1}), hydrogen bonding as well as strong metal bonding (1668 to 1660 cm^{-1}), bending vibration of TeO_3 (786 to 762 cm^{-1}), stretching mode of TeO_4 (687 to 678 cm^{-1}) and bonding vibration of ZnO (460 to 446 cm^{-1}). The UV-Visible-NIR spectra displayed ten absorption peaks in which the first six peaks centered at 451, 485, 522, 653, 801 and 972 nm were assigned to the transition from the ground state ($^4I_{5/2}$) to the excited states ($^4F_{5/2}$, $^4F_{7/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, and $^4I_{11/2}$) of Er^{3+} ion respectively. The last four peaks centered at 1080, 1236, 1381 and 1493 nm were assigned to transition from the ground level ($^6H_{5/2}$) to the excited levels ($^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, and $^6F_{3/2}$) of Sm^{3+} ion respectively. Moreover, all the peak intensities were slightly enhanced due to the embedded Ag NPs into the glass systems. The indirect optical band gap energy was found to decrease from 2.63 to 2.52 eV due to the addition of Ag NPs. Conversely, the Urbach energy was found to increase from 0.20 to 0.27 eV because of Ag NPs inclusion. The calculated Judd-Ofelt intensity parameters (Ω_2, Ω_4 and Ω_6) showed $\Omega_4 > \Omega_2 > \Omega_6$ trend. The highest value of estimated quality factor is 6848.44 for the glass system containing 0.05 g of Ag NPs. The radiative parameters including average electric dipole, branching ratio and radiative lifetime were also computed to support the evidence of Ag NPs impact on optical properties. The values of branching ratios for the Sm^{3+} ions transitions of $^4F_{3/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$, $^4G_{5/2} \rightarrow {}^6H_{11/2}$, $^4G_{5/2} \rightarrow {}^6H_{13/2}$ and $^4G_{5/2} \rightarrow {}^4F_{1/2}$ were found to vary from 0.19 to 99.98%. The luminescence spectra under 488 nm excitation manifested six significant peaks located at 524, 550, 597, 640, 669 and 705 nm which are assigned to the transition of $^4F_{3/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$, $^4G_{5/2} \rightarrow {}^6H_{11/2}$, $^4G_{5/2} \rightarrow {}^6H_{13/2}$ and $^4G_{5/2} \rightarrow {}^4F_{1/2}$ respectively. The emission peak intensities revealed significant enhancement due to the embedded Ag NPs into the tellurite host. The results were analyzed, discussed and compared.

ABSTRAK

Tesis ini melaporkan kesan nanopartikel (NP) perak tulen (Ag) terhadap peningkatan kaca sifat optik oksi-zink tellurit yang didopkan dengan ion samarium (Sm^{3+}) dan erbium (Er^{3+}). Untuk mencapai matlamat ini, sampel siri kaca dengan komposisi 63.75 TeO_2 + 20 ZnO + 15 $ZnCl_2$ + 0.75 Sm_2O_3 + 0.5 Er_2O_3 + y Ag ($0.01 \leq y \leq 0.10$ g lebihan) telah disediakan dengan menggunakan kaedah pelindapan peleburan. Sifat fizikal seperti ketumpatan dan isipadu molar kaca telah ditentukan. Ketumpatan kaca dan isipadu molar masing-masing adalah dalam julat antara 5.416 - 5.326 gcm^{-3} dan 26.400 - 26.864 cm^3mol^{-1}. Sampel tersintesis lutsinar menunjukkan kestabilan terma yang baik ke atas kawasan pembentukan kaca yang luas. Parameter terma seperti suhu transisi kaca, suhu penghabluran dan suhu lebur telah diukur menggunakan Penganalisa Terma Pembeza (DTA). Corak Pembelauan Sinar-X (XRD) mengesahkan keadaan amorfus sebenar kaca yang disediakan dan spektrum Serakan Tenaga Sinar-X (EDX) mengesan kehadiran unsur sebenar dalam komposisi. Imej Mikroskopi Elektron Penghantaran (TEM) menunjukkan kehadiran NP Ag dalam matriks kaca yang bertabur secara homogen. Spektrum Inframerah Jelmaan Fourier (FTIR) telah direkod dalam julat antara 400 hingga 4000 cm^{-1}. Penggabungan NP Ag dalam hos kaca didapati telah menganjakkan sedikit jalur inframerah. Ianya menunjukkan regangan kumpulan OH (3478 ke 3453 cm^{-1}), ikatan hidrogen seperti ikatan kukuh logam (1668 ke 1660 cm^{-1}), getaran membengkok TeO_3 (786 ke 762 cm^{-1}), mod regangan TeO_4 (687 ke 678 cm^{-1}), dan getaran ikatan ZnO (460 ke 446 cm^{-1}). Spektrum UV-Visible-NIR mempamerkan sepuluh puncak penyerapan yang mana enam puncak pertama berpusat di 451, 485, 522, 653, 801 dan 972 nm masing-masing berpadanan dengan transisi dari aras dasar ($^4I_{5/2}$) ke aras teruja ($^4F_{5/2}$, $^4F_{7/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, dan $^4I_{11/2}$) ion Er^{3+}. Empat puncak terakhir yang berpusat di 1080, 1236, 1381 dan 1493 nm masing-masing berpadanan untuk transisi dari aras dasar ($^6H_{5/2}$) ke aras teruja ($^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, dan $^6F_{3/2}$) ion Sm^{3+}. Tambahan pula, keamatan semua puncak didapati meningkat sedikit disebabkan penambahan NP Ag dalam sistem kaca. Tenaga jurang jalur optik tidak langsung didapati berkurang daripada 2.63 kepada 2.52 eV disebabkan penambahan NP Ag. Sebaliknya, tenaga Urbach didapati bertambah daripada 0.20 kepada 0.27 eV kerana penambahan NP Ag. Parameter keamatan Judd-Ofelt (Ω_2, Ω_4 dan Ω_6) yang dikira menunjukkan trend $\Omega_4 > \Omega_2 > \Omega_6$. Nilai tertinggi faktor kualiti anggaran ialah 6848.44 untuk sistem kaca yang mengandungi 0.05 g NP Ag. Parameter sinaran termasuk dwikutub elektrik purata, nisbah cabang dan jangka masa hayat sinaran juga dihitung untuk menyokong bukti impak NP Ag ke atas sifat optik. Nilai nisbah cabang untuk ion Sm^{3+} bagi transisi $^4F_{3/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$, $^4G_{5/2} \rightarrow {}^6H_{11/2}$, $^4G_{5/2} \rightarrow {}^6H_{13/2}$ dan $^4G_{5/2} \rightarrow {}^4F_{1/2}$ adalah berubah daripada 0.19 hingga 99.98 %. Spektrum luminesens dengan pengujaan 488 nm menunjukkan enam puncak penting yang terletak pada 524, 550, 597, 640, 669 dan 705 nm masing-masing berpadanan dengan transisi $^4F_{3/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$, $^4G_{5/2} \rightarrow {}^6H_{11/2}$, $^4G_{5/2} \rightarrow {}^6H_{13/2}$ dan $^4G_{5/2} \rightarrow {}^4F_{1/2}$. Keamatan puncak pancaran menunjukkan peningkatan yang ketara kesan pertambahan NP Ag ke dalam hos tellurit. Hasil kajian telah dianalisa, dibincangkan dan dibandingkan.

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A	-	Spontaneous emission probabilities
A_{ed}	-	Electric-dipole
A_{md}	-	Magnetic-dipole
α	-	Absorption coefficient
aJ	-	Ground state
B	-	Constant
β	-	Branching ratio
bJ	-	Excited state
C	-	Speed of light
C_{RE}	-	Concentration of the rare-earth
C	-	Light velocity
D	-	Density of air
E_u	-	Urbach energy
E_f	-	Energy of electron of final state
Ei	-	Eenergy of an electron at lower band
E_g	-	Optical energy bandgap
$\varepsilon_a(v)$	-	Molar extinction coefficient
e	-	Electron charge
F	-	Force constant
f_{exp}	-	Experimental oscillator strength
f_{cal}	-	Oscillator strength
hv	-	Photon energy

H_R	-	Hruby parameter
Λ	-	Wavelength
Ω_λ	-	Judd-Ofelt parameters
M	-	Molecular weight
M	-	Electron mass
m_r	-	Atomic weights in kg of cation
m_o	-	Atomic weights in kg of anion
N	-	Integer
θ	-	Angle
ρ	-	Density
ρ_o	-	Density of distilled water
P	-	Number of transitions
Q	-	Quality factor
rms	-	Root-mean-square
S_{ed}	-	Line-strength for electric
T_c	-	Glass crystallization temperature
T_m	-	Glass melting temperature
T_g	-	Glass transition temperature
T_{rad}	-	Radiative lifetime
ΔT	-	Glass thermal stability
T	-	Thickness of the sample
U_K	-	Values of reduced matrix elements
μ	-	Reduced mass
V	-	Wave number
V_m	-	Molar volume
w_A	-	Weight of sample in air
w_1	-	Weight in distilled water
Ω	-	Frequency dependence

LIST OF ABBREVIATIONS

TeO_2	-	Tellurite
Ag	-	Silver
Au	-	Gold
BO	-	Bridging oxygen
CB	-	Conduction band
DTA	-	Differential thermal analysis
EDX	-	Energy dispersive X-ray
Er	-	Erbium
FTIR	-	Fourier transform infrared
H	-	Hydrogen
IR	-	Infrared
J-O	-	Judd-Ofelt
NBOs	-	Non-bridging oxygens
NPs	-	Nanoparticles
O	-	Oxygen
PL	-	Photoluminescence
RE	-	Rare earth
SPR	-	Surface plasmon resonance
Sm	-	Samarium
TEM	-	Transmission electron microscopy
UV-Vis	-	Ultraviolet visible
VB	-	Valence band
XRD	-	X-ray diffraction
ZnO	-	Zinc oxide
$ZnCl_2$	-	Zinc chloride

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter explains the purpose of this research including a background of research, problem statement, objectives of research, scope of research, significance and contribution of this research and thesis outline.

1.2 Background of the Research

Study of tellurite based glass received attentions of researchers in recent years is due to interesting optical, electrical and magnetic properties (El-Mallawany *et. al.*, 2004; Chillcce *et. al.*, 2011; Yusoff and Sahar, 2015). In glassy materials, tellurite based glass is very noticeable. This is because of their exclusive properties such as excellent transmission in visible as well as IR wavelength regions, good in mechanical strength and chemical durability also high in electrical conductivity (Jaba *et.al.*, 2000; Mohamad *et. al.*, 2006; Sidek *et. al.*, 2009; Sayed *et.al.*, 2016). Despite that, these glasses also possess good physical properties which is higher refractive index (in range 2.0 - 2.5), low cut-off phonon energy (~700 $cm^{-1)}$ and low melting temperature (733 ºC) that contributes to high possibility of stable glass forming using conventional melt quenching method.

Tellurium oxide and zinc oxide (TeO_2-ZnO) glass forming range has been reported by previous researchers (Burger *et.al.,* 1992, Jaba *et.al.,* 2000; Rafaella *et.al.,* 2001; Marjanovic *et.al.,* 2003; Mohamad *et.al.,* 2006; Surendra *et.al.,* 2007; Rosmawati *et.al.,* 2007; Sahar *et.al.,* 2008; Sidek *et.al.,* 2009; Sidek *et.al.,* 2013). It is reported that the combination of host and modifier of TeO_2-ZnO form a good and stable glass (Burger *et.al.*, 1992; Sidek *et.al.*, 2009) and suitable host for optically rare earth (RE) ions (Rosmawati *et.al.*,. 2007; Sahar *et.al.*, 2008).

As mentioned above, the TeO_2-ZnO binary systems provide broad glass forming region. However, this glass formation depends on cooling rate and size of melt especially in TeO_2 rich region. The TeO_2-$ZnCl_2$ binary systems was found to exhibit a continuous glass forming region, yielding optically attractive stable and easy to prepare glass. Introducing metal halides which is zinc chloride ($ZnCl_2$) into TeO_2-ZnO glass systems could improve glass properties (Sahar *et.al.,* 1997). However, $ZnCl_2$ itself is a bit tricky because of its properties which is hygroscopic, unstable and difficult to handle. Despite of its hygroscopic properties that can limits many applications of pure halide glasses (Sahar and Noordin, 1995), the combination of $ZnCl_2$ with ZnO (called zinc oxyhalide) as a modifier of TeO_2 glass are reported has desirable glass forming ability, long fluorescence lifetime and high emission cross sections which can be acted as suitable materials for laser applications (Guonian *et.al.,* 2005).

Presently, glasses containing rare earth (RE) and metallic nanoparticles (NPs) have been reported (Kassab *et.al.*, 2008; Fauzia *et.al.*, 2014; Yusoff and Sahar, 2015; Hssen *et.al.*, 2014). It is reported that introducing RE and NPs into the tellurite based glass will change the structure of glass and improve the optical properties (Luciana *et.al.,* 2011; Yusoff and Sahar, 2015). RE doped glass is important in their potential applications in optical devices such as lasers, sensors, and telecommunications. However, the absorption cross-section of most RE ions in tellurite is small (Raja *et.al.,* 2014). This will be disadvantages for the applications that need higher optical performances. The effort to increase this efficiency by increasing RE concentration is unsuccessful (Fauzia *et.al.*, 2014). Therefore, by introduce the metallic NPs into glass system with RE dopants is found to be successful (Giehl *et.al.,* 2011; Reza *et.al.,* 2013; Asmahani *et.al.,* 2014). This is because the existence of energy transfers from species with large absorption cross-section to RE ions and will enhance fluorescence emission (Luciana *et.al.,* 2011; Reza *et.al.,* 2013; Sazali *et.al.,* 2015).

Reza *et.al.,* (2013) shown an enhancement of intensity of the erbium doped zinc tellurite glass with the presence of Ag NPs. They also observed the zinc tellurite based glass showed good capability accepting Re and metallic NPs. Yusoff and Sahar (2015) reported the decreasing of energy bandgap, E_g as an increasing of Ag NPs in their samarium doped magnesium tellurite glass systems. This is due to structural changes caused by the existence of Ag in glass systems. Hssen *et.al.,* (2014) found an enhancement of photoluminescence intensity and photoluminescence lifetime in glass systems of erbium doped tellurite glass embedded with Ag NPs. Other than that, Tripathi *et.al.*, (2008) investigated the energy transfer between Sm^{3+}: Er^{3+} in tellurite glass and Bahadur *et.al.,* (2010) studied spectroscopic properties on Sm^{3+}: Er^{3+} doped barium fluorotellurite glass. Therefore, it would be interesting to study the effect of metallic NPs on optical properties of Sm^{3+}: Er^{3+} zinc oxyhalide tellurite glass as well as their thermal and structural properties.

Therefore, the aim of this research is to investigate the effect of silver NPs on optical properties of samarium co-doped erbium zinc oxyhalide tellurite glass. The combination of Sm^{3+} and Er^{3+} is supposed to present an energy transfer and the addition of metallic NPs is an alternative ways to improve the absorption and emission cross-sections of the RE ions. In this present research, samarium co-doped erbium zinc oxyhalide tellurite glasses are prepared with and without NPs by melt quenching technique. Additionally, physical, thermal and structural characterizations will also be carried out to support the results in optical properties of prepared samples. Besides the experimental approach, the Judd-Ofelt (J-O) intensity and other relevant optical parameters such as average electric dipole, branching ratio and radioactive lifetime also will be determined for optical properties.

1.3 Problem Statement

Luminescence of RE ions has been widely exploited due to their potential of converting light from infrared to visible range. However, most of the RE ions in tellurite glasses have small absorption cross-section. In purpose for enhancing the luminescence; the absorption cross-section has to be increased. As reported by Giehl *et.al.*, (2011) and Fauzia *et.al.*, (2014), the use of higher concentration of RE in glass to increase the absorption cross-section efficiency is a failure. This is because the emission intensity easily gets quenched due to the losses which are stimulated by de-excitation of different energy levels (Sazali *et.al.*, 2015). Surprisingly, introducing the coupling of RE ions with metal NPs into tellurite glass systems were reported could enhance luminescence efficiency and absorption cross section of RE ions (Luciana *et.al.*, 2011; Asmahani *et.al.*, 2014). Therefore, tellurite glasses co-doped with RE and NPs are particularly interested. Besides, the results on J-O intensity and radiative parameters containing Sm^{3+} and Ag NPs do not extensively reported. Therefore, the aim of this present research is providing information of thermal, structural and optical characterization including J-O parameters of samarium co-doped erbium zinc oxyhalide tellurite glasses samples with and without Ag NPs.

1.4 Objectives

The objectives of this research are:

i. To synthesize a series of samarium co-doped erbium zinc oxyhalide tellurite glass samples without and with Ag NPs by melt quenching technique.
ii. To characterize the physical, thermal, structural and optical properties of samarium co-doped erbium zinc oxyhalide tellurite glasses embedded with Ag NPs.
iii. To analyze the effect of embedding Ag NPs on enhancement luminescence of samarium.
iv. To calculate the oscillator strength and radiative properties using Judd-Ofelt theory.

1.5 Scope of Research

The scope of this research is including the preparation of 4 series of tellurite glass with melt quenching technique. The first 3 series of glass composition of TeO_2 + ZnO + $ZnCl_2$ + Sm_2O_3 + Er_2O_3 are prepared without Ag NPs and the final series of glass is with Ag NPs. The first 3 series will be prepared to find optimum compositions of the modifier and RE for these zinc oxyhalide tellurite glass systems and that optimum composition will be embedded with Ag NPs in the final series (4^{th} series).

The physical properties including density and molar volume of glass will be determined by Archimedes principle. Thermal properties including glass stability will be determined by Differential Thermal Analyzer (DTA) and amorphous nature of glass will be determined by X-ray diffractometer (XRD). Energy Dispersive X-ray (EDX) will be analyzing to know the actual composition of glass. The existence of Ag NPs will be confirmed by Transmission Electron Microscopy (TEM). The structural behaviour of glass is studied by Fourier Transform Infrared Spectrometer (FTIR). Optical properties of glass will be determined by using UV-Vis and photoluminescence spectroscopy including energy bandgap, Urbach energy and energy level diagram to observe the luminescence enhancement. The experimental approach will be completed with the theoretical calculations of J-O intensity and radiative parameter. All research experiment provided are highly relevant for applied technology for preparing efficient glasses.

1.6 Significance of Research

The characterization of the optical properties of samarium co-doped erbium zinc oxyhalide tellurite embedded with silver NPs glasses are extremely important for the optimization in applications. Consequently, the main interest in this research is to enhance the optical properties by introducing the proposed glass compositions with metallic NPs. The relevance of this present research conveys the technology needed for preparing efficient glasses with controlled dopants and NPs. This research can provide knowledge on thermal, structural and optical behavior of NPs with samarium co-doped erbium in zinc oxyhalide tellurite glass.

1.7 Thesis Outline

This thesis will describe the glass preparation and characterization of samarium co-doped erbium zinc oxyhalide tellurite embedded with silver NPs glasses. This thesis is divided into five chapters. Chapter 1 will introduce the purpose of this research including a background of research, problem statement, objectives, scope of research, and significance of research. Chapter 2 will describe related theories of this glass which is the definition of glass, the glass formation, the glass networks, physical, thermal, structural and optical properties.

Chapter 3 contained detailed on glass preparation which is the procedure and experimental techniques. Chapter 4 will discuss the results of the experimental details. Lastly, chapter 5 entitled the conclusion clarification concerning the work presented in this thesis and future suggestion.

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