LUMINESCENCE OF ERBIUM DOPED TELLURITE GLASS CONTAINING SILVER NANOPARTICLES

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To my beloved Family

(Jamaludin Abd Rahman & Radhiah Md Noor)

“your advices, guides, and moral supports make me stronger”

“there is nothing in my life that makes me happy and cheerful

than your love and care”

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ABSTRACT

Enhancing the optical properties of rare-earth doped inorganic glasses by embedding metallic nanoparticles (NPs) of controlled sizes and examining the mechanism of enhancement are important and challenging. A series of Er^{3+} doped zinc tellurite glasses with and without silver NPs of molar composition $(74.5-x)TeO_2-25ZnO-0.5Er_2O_3-xAgCl$ with $0 \leq x \leq 1.0$ mol % were prepared by melt-quenching technique. They were characterized by X-ray diffraction, transmission electron microscopy, differential thermal analysis (DTA), and Fourier transform infrared (FTIR), ultraviolet-visible-near infrared and photoluminescence spectroscopy. The thermal, structural, and optical properties of these glasses were determined and analyzed. The XRD pattern confirms the amorphous nature of all samples. The high resolution TEM reveals the presence of silver NPs of average diameter 24 ± 2 nm with measured lattice constant of 2.09 Å and a Gaussian size distribution. The observed glass density ranges from 5.481 to 5.697 g cm^{-3} and the refractive index evaluated using Sellmeier's equation varies between 2.16 to 2.27. Thermal parameters such as glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m) and thermal stability ΔT (T_c-T_g) were determined from DTA analyses. A wide and stable glass formation range around 87°C-105°C was found. The structural properties measured by using FTIR show that as the AgCl content increases, the sharp infrared (IR) absorption peaks were consistently shifted from 675.13 cm^{-1} to 679.03 cm^{-1} which were assigned to the structural changes between the stretching vibration mode of TeO_4 (trigonal bipyramids) and TeO_3(trigonal pyramids). The absorption peaks around 1600 cm^{-1} and 3400 cm^{-1} are related to the stretching vibrations of the hydroxyl group participating in the strong metal and hydrogen bonding, respectively. The absorption spectra consist of eight absorption peaks centered at 1510, 975, 799, 655, 586, 523, 489 and 447 nm corresponding to transitions from ground state to $^4I_{13/2}$, $^4I_{11/2}$, $^4I_{9/2}$, $^4F_{9/2}$, $^4S_{3/2}$, $^2H_{11/2}$, $^4F_{7/2}$, and $^4F_{5/2}$ excited states, respectively. From PL spectra, excitation at 786 nm and 470 nm for up and down-conversion exhibits strong green and red emission comprised of three emissions band at 501, 548 and 634 nm. The significant enhancement in the luminescence with the increase of AgCl is ascribed to the local field enhancement induced by Ag^0 surface plasmon resonance and the energy transfer from fluorescent $Ag^0 \rightarrow Er^{3+}$ ions. The improved optical and structural properties suggest that the studied glasses are potential candidates for nanophotonic devices.

ABSTRAK

Peningkatan sifat optik unsur bumi nadir yang didopkan pada kaca organik yang mengandugi logam nanopartikel (NPs) saiz terkawal dan menguji mekanisme peningkatan adalah penting dan mencabar. Satu siri kaca tellurit-zink yang didopkan dengan Er^{3+} dengan dan tanpa perak NPs bagi komposisi molar $(74.5\text{-}x)TeO_2$-$25ZnO$-$0.5Er_2O_3$-$xAgCl$ untuk $0 \leq x \leq 1.0$ mol% telah berjaya dihasilkan menggunakan teknik pelindapan leburan. Kaca ini dicirikan oleh pembelauan sinar-X, mikroskopi elektron penghantaran, analisis pembezaan terma, dan spektroskopi inframerah transformasi Fourier, ultraungu-cahaya nampak-inframerah dekat dan fotoluminesen. Sifat haba, struktur, dan optikal bagi kaca ini ditentukan, dan dianalisis. Corak XRD mengesahkan bahawa semua kaca tersebut adalah amorfus. TEM resolusi tinggi mendedahkan kehadiran perak NPs dengan purata diameter 24 ± 2 nm dengan kekisi malar 2.09 Å dan bertaburan saiz Gaussian. Ketumpatan kaca berubah dari 5.481 ke 5.697 g cm^{-3} dan indeks biasan ditentukan dengan menggunakan persamaan Sellmeier antara 2.16 ke 2.27. Parameter terma seperti suhu transisi kaca (T_g), suhu penghabluran (T_c), suhu leburan (T_m) dan kestabilan terma ΔT (T_c-T_g) ditentukan daripada analisis DTA. Pembentukan kaca yang luas dan stabil di sekitar 87ºC-105ºC diperoleh. Sifat struktur yang diukur dengan menggunakan FTIR menunjukkan bahawa dengan peningkatan kandungan AgCl, puncak penyerapan inframerah (IR) yang tajam beranjak secara selaras daripada 675.13 cm^{-1} ke 679.03 cm^{-1} yang memberikan perubahan struktur antara mod getaran regangan TeO_4 (bipiramid trigonal) dan TeO_3 (piramid trigonal). Puncak penyerapan masing-masing di sekitar 1600 cm^{-1} dan 3400 cm^{-1} adalah sebagai getaran regangan kumpulan hidroksil yang melibatkan bahagian dalam ikatan logam kuat dan ikatan hidrogen. Spektrum penyerapan terdiri daripada lapan puncak penyerapan berpusat di 1510, 975, 799, 655, 586, 523, 489 dan 447 nm masing-masing bersesuaian dengan peralihan dari keadaan asas ke keadaan teruja $^4I_{13/2}$, $^4I_{11/2}$, $^4I_{9/2}$, $^4F_{9/2}$, $^4S_{3/2}$, $^2H_{11/2}$, $^4F_{7/2}$, dan $^4F_{5/2}$. Daripada spektra PL, pengujaan pada 786 nm dan 470 nm untuk penukaran naik dan turun mempamerkan pelepasan cahaya hijau dan merah yang kuat terdiri daripada tiga kumpulan pancaran pada 501, 548 dan 634 nm. Peningkatan ketara dalam fotoluminesen dengan penambahan AgCl dianggap berpunca daripada penempatan medan peningkatan disebabkan oleh permukaan resonans plasmon dan pemindahan tenaga dari $Ag^0 \rightarrow Er^{3+}$ ions. Penambahbaikkan sifat optik dan struktur mencadangkan bahawa kaca yang dikaji berpotensi sebagai peranti nanofotonik.

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LIST OF SYMBOLS/ ABBREVIATIONS

θ - Angle of Incident
A - Absorbance
B - Constant
B_j - Constant containing the oscillation strength of the electron or oscillation transitions
$\alpha(\omega)$ - Absorption Coefficient
c , v - Speed of Light
d_2 - Thickness of the Sample
e - Electron
E - Electric Field
E_f - Energy of electron of final state at upper level
E_g - Energy Band Gap
E_i - Energy of electron at lower level
E_{opt} - Optical Energy Gap
E_{tail}, ΔE - Urbach Energy
f - Frequency
$\hbar\omega$ - Photon Energy
k - Wave Vector
λ - Wavelength
λ_{exc} - Excitation Wavelength
M - Molecular Weight
n, n' - Refractive Index
n - Density of Electron
P - Oscillator Strength Contain of Electric-Dipole
$\vec{P}$ - Polarization Density

ρ - Density
R' - Refractivity
T_c - Crystallization Temperature
T_g - Glass Transition Temperature
T_m - Melting Temperature
μ - Dipole Moment
μ_{ind} - Induced Dipole Moment
ω - Oscillation Frequency
v - Dispersion
Φ - Incident of Flux Photon
φ_a - Absorption
φ_r - Reflection
φ_s - Scattered
φ_t - Transmission
$\chi^{(3)}$ - Susceptibility
Ag - Silver
AgCl - Silver Chloride
Au - Gold
Er^{3+} - Trivalent Erbium Ion
Eu^{3+} - Trivalent Europium Ion
Tb^{3+} - Trivalent Terbium Ion
Sm^{3+} - Trivalent Samarium Ion
Ho^{3+} - Trivalent Holmium Ion
Cd - Cadmium
TeO_2 - Tellurium Oxide
Zn - Zinc
ZnO - Zinc Oxide
MgO - Magnesium Oxide
CB - Conduction Band
VB - Valence Band
RE - Rare Earth
REEs - Rare Earth Elements
Hg - Mercury

DSC	-	Differential Scanning Calorimetry
DTA	-	Differential Time Analyzer
PL	-	Photoluminescence
ESA	-	Excited State Absorption
GFA	-	Glass Formation Ability
GSA	-	Ground State Absorption
ET	-	Energy Transfer
ETU	-	Energy Transfer Up-conversion
UC	-	Up-conversion
SPR	-	Surface Plasmon Resonance
IR	-	Infrared
TEM	-	Transmission Electron Microscope
FTIR	-	Fourier Transform Infrared
XRD	-	X-Ray Diffraction
DL	-	Deuterium Lamp
H	-	Hruby Parameter
SAED	-	Selected Area Electron Diffraction
HRTEM	-	High Resolution Transmission Electron Microscope
NR	-	Non-Radiative
NBO	-	Non-Bridging Oxygen
Te-O	-	Tellurium-Oxygen Bond
Zn-O	-	Zinc-Oxygen Bond
Te-O-Zn	-	Tellurium-Oxygen-Zinc Bond

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CHAPTER 1

INTRODUCTION

1.1 General Introduction

Glass is an amorphous solid material of fusion which has cooled to a rigid condition without crystallization. The interesting thing about the glass is that it has characteristic properties such as shiny, transparent and non-corrosive. They are now under consideration in many applications. In recent years rare earth ion (REI) doped inorganic glasses (binary, ternary etc.) received great attention due to their wide range of applications in lasers, optical amplifiers and optical sensors, among other devices (El-Mallawany, 2001; Neto et al., 2002; Chen, 2007). The non-radiative energy transfer (ET) and multi-phonon relaxation are detrimental for devices unless inhibited. The ET processes may support particular applications such as the operation of anti-Stokes emitters and may not be beneficial as in case of RE based laser because contribution of interactions among the active ions for the increment of the laser threshold.

Tellurite dioxide-based glasses are superior attractive hosts and present large potential material because they offer low cutoff phonon energy, accept large concentration of RE ions (Taylor et al., 2002), exhibit large transmittance window (from the visible to the infrared region) and present high refractive index (~2.0) (Richards et al., 2008). Tellurite glasses matrices are fairly suitable host for rare-earth solid state laser because interactions among the active ions contribute for the increase of the laser threshold and have attracted a great deal of scientific and technological importance that generated renewed interest lately. They are special due to excellent optical and structural properties, chemical durability and thermal stability.

Tellurite glasses are demanding due to several advantages such as relatively low transformation temperatures, high densities and non-hygroscopic properties and the lack of such properties limit the application of phosphate and borate glasses (Jiménez et al., 2011). Tellurite glasses have large Raman shifts (up to 1200 cm^{-1}) in some compositions and large Raman gain coefficients of order of 60 times of silica. Therefore, these glasses are suitable host for active element doping; represent the main justification for their continuous technological interest in areas of optoelectronics such as laser technology, optical fibers, non-linear optical devices and sensor systems (Oubaha et al., 2010). The TeO_2-ZnO system shows excellently stable glass-forming ability with a broad region (Sahar et al., 2008; Sidebottom et al., 1997). The magnitude of the third-order nonlinear susceptibility $\chi^{(3)}$ (Ferreira et al 2003; Santos et al., 2009) and up-conversion luminescence intensity could be enhanced by modifying the local structure in tellurite glasses (Jha et al., 2000). These glass systems doped with modifier ZnO exhibits distinct optical bistability and become an excellent broadband saturable absorber when doped with cobalt or an optical power limiter while doped with erbium (Chen, 2007; Xiao et al., 2007; Babu et al., 2007). However, the glass formation strongly depends on the cooling rate and the size of the melt, especially in the TeO_2-rich region (El-Mallawany, 2002).

Recently, the enhancement of light energy up-conversion luminescence properties of metallic nanoparticles (NPs) embedded rare earth doped tellurite glasses has received special attention because of their prospective use in solar near infrared concentration for photovoltaic exploitation, infrared sensing and biological labeling (Jlassi et al., 2012). Glasses containing metallic NPs and rare earths ions are attracting renewed interest because the NPs may impart changes of the material’s luminescence characteristics as well as enhancement of the nonlinear optical properties either due to the ET mechanism from NPs to the RE ions or by the large induced local field on the RE ions. The quantum effect of NPs is an important factor to improve luminescence efficiency performance of NPs-embedded glasses, while precise control of the size distribution and content of NPs in the glass is the key to achieve good quality for optical performance. Accordingly, luminescence enhancement due to the presence of metallic NPs has been reported for Pr^{3+} (Kassab et al., 2007; Lakshminarayana et al., 2009) and Er^{3+} (Singh et al., 2010) in oxide glasses, but no systematic experimental studies are made so far, to explain the influence of the embedded NPs on structural and optical properties in the rare earth doped tellurite glasses.

The aim of this thesis is to investigate the effect of silver NPs on up-conversion (UC) luminescence processes of erbium doped tellurite glass. Among rare-earth ions that efficiently generate visible up-conversion and infrared fluorescence, where erbium is the most widely studied rare earth dopant. The erbium ions excited-states $^2H_{11/2}$, $^4S_{3/2}$, and $^4F_{9/2}$ are populated via stepwise ground state and excited state absorption, followed by multi-phonon non-radiative relaxation and cross-relaxation processes (Babu et al., 2011; Yang et al., 2009: Xiao et al., 2007). Most of the recent results revealed that blue, green and red up-conversion emission signals are strongly influenced by the concentration of Er^{3+} ion and temperature. It is important to study the frequency up-conversion mechanism in alternative hosts and identify the major relaxation and interaction mechanisms of rare-earth ions implanted into the material and the influence of NPs. On the other hand, the influence of metallic NPs on the up-conversion (UC) luminescence of rare-earth ions is not much exploited. The incorporation of metallic NPs is considered to be an alternative route to improve the absorption and emission cross-sections of the rare earth ions. Our work represents a part of continuing effort to characterize Er_2O_3 doped TeO_2-ZnO glasses containing silver (Ag) nanoparticles for their density, refractive index, DTA measurement, IR spectroscopy, optical absorption in ultraviolet and visible region, photoluminescence and TEM spectroscopy respectively.

1.2 Problem Statement

Despite some reports on tellurite based glasses the controlled incorporation of metallic NPs in these Er^{3+}doped glasses to examine the role played by silver NPs in influencing their physical properties are not fully investigated. Considerable efforts are dedicated to enhance the intensity and gain of these glasses without understanding the microscopic mechanism of enhancement. First, it was reported that increment of concentration of REs in the system could intensify the up- and or down-conversion luminescence. However, a quench is observed often, after the introduction of 1~2 mol% of the fluorophores (Jlassi et al., 2010). Due to the limitation of increasing the RE ions concentration the alternative path to get efficient emission and enlarge in the emission intensity by localizing a large electric field in the dielectric host needs further studies. Therefore, the present study is dedicated to determine the structural features of glasses with and without silver NPs besides the effect of silver NPs on luminescence properties and host composition. Optimizing the NPs and REI concentrations in the glass to enrich optical response is lacking. New results on optical, thermal and structural properties are expected that has not been explored so far.

1.3 Objectives

The objectives of this research are:

a) To prepare a series of Er^{3+}doped zinc tellurite glasses samples with and without silver NPs using melt quenching method.
b) To determine the physical properties of glass system in terms of their density and refractive index.
c) To determine the thermal stability of glasses samples.
d) To characterize the transmission behavior including bonding by using Fourier Transformed Infrared (FTIR) spectroscopy.
e) To determine the absorption behavior of glasses samples UV-Vis.

f) To determine the up-conversion and down-conversion emission properties of glasses samples.
g) To determine the distribution and size of the Ag in glass sample using TEM.

1.4 Scope of Study

In order to achieve the listed objectives the focused perspectives are:

a) Preparation of Er^{3+} doped zinc tellurite glasses embedded with and without AgCl based on (74.5-x) TeO_2-25ZnO-(0.5)Er_2O_3-(x)AgCl ($0.0 \leq x \leq 1.0$ mol%) with different composition by melt quenching technique.
b) Determination of glass density by Archimedes method.
c) Confirmation of the amorphous phase of obtained the glass using X-ray diffraction.
d) Determination of thermal stability of samples using DTA measurement.
e) Characterization of transmission spectra by using Fourier Transform infrared (FTIR) spectrometer.
f) Determination of the absorption properties of samples using UV-VIS spectroscopy.
g) Determination of the emission properties of the samples using photoluminescence measurement.
h) Determination of distribution and size of silver nanoparticles using TEM.

1.5 Significance of Study

Due to limited studies performed on TeO_2-ZnO-Er_2O_3 glasses, this present study will examine and determine the structural, thermal and optical features of these glasses. The primary focus is to enhance the optical properties of these newly developed materials by embedding silver NPs that would contribute towards the design of new short-wavelength solid-state laser and other nanophotonic devices. This is very important to understand the mechanism behind the enhancement of optical properties and the role played by the Ag NPs in vicinity of Er^{3+}ion doped in tellurite glass. Such up-converted ideal system can be used in lasing mechanism. The study has both fundamental and applied interests. Increasing the absorption and emission cross-section due to the incorporation of Ag NPs in glasses have many technological implications. Our detail experimental analyses and quantification may constitute a basis for developing nanoglass with superior optical properties.

Now in the next chapter we turn our attention to present a brief research background to justify our research interests.

5) Presence of other metallic NPs such as gold certainly will change the optical properties and the coupling with other RE ions will also adjust optical absorption and emission spectra.

6) The embeddment of magnetic NPs such as Mn, Co, Ni, Fe are important for spintronics and magneto-optic devices.

7) Designing plasmonic solar cells using this glass has tremendous interests.

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