INFLUENCE OF SILVER NANOPARTICLES CONCENTRATION ON SPECTROSCOPIC CHARACTERISTICS OF SAMARIUM DOPED ZINC TELLURITE GLASS

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To my beloved family and friends

“your advices, guidance for all this years”

“there is nothing I can achieve without your help”

“Love everybody”

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ABSTRACT

Tuning the enhanced optical properties of rare earth doped inorganic glasses by embedding metallic nanoparticles is ever-demanding in photonics. Optimized doping of NPs and subsequent detail characterizations for improved absorption and emission are the key issue. The effect of silver (Ag) nanoparticle on physical, structural and optical properties of Sm^{3+} doped zinc-sodium tellurite glass with composition $65TeO_2$-25ZnO-$10Na_2O$-0.15 gramSm_2O_3-(*y*)AgCl, where $y = 0$, 0.003, 0.075, 0.12 and 0.18 gram are determined. Glass samples are prepared using melt quenching technique method and characterized by ultraviolet visible near infrared (UV-Vis-NIR) absorption spectroscopy, photoluminescence (PL) spectroscopy, transmission electron microscopy (TEM), Raman spectroscopy and X-ray diffraction (XRD). XRD pattern confirms the amorphous nature of as-prepared glass. The incorporation of Ag NPs is evidenced to alter the structural arrangement and modifies the physical properties of glasses. The UV-Vis-NIR absorption spectra reveal six absorption peaks centered at 472 nm, 943 nm, 1089 nm, 1237 nm, 1392 nm, 1491 nm assigned to $^6H_{5/2}\rightarrow{}^4I_{11/2}$,$^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$ transitions, respectively. TEM micrograph displays the uniform size distribution of silver nanoparticle with average diameter of ~7.9 nm. PL spectra exhibit two emission bands located at 599 nm and 643 nm due to $^4G_{5/2}\rightarrow{}^6H_{7/2}$ and $^6H_{9/2}$ transitions, respectively. The observed enhancement in PL intensity is attributed to the highly localized electric field of Ag positioned in the vicinity of Sm^{3+} ion. Raman spectra shows the appearance of five peaks originates from 99.44 cm^{-1}, 179.88 cm^{-1}, 414.37 cm^{-1}, 673.44 cm^{-1} and 768.66 cm^{-1} which corresponds to boson peak, network connectivity represents the Te_2O_7 and TeO_4^{-4}, stretching and bending Te–O–Te linkages, antisymmetric vibration of Te_{eq}–O_{ax}–Te linkages and stretching of NBO with adjacent Te atoms respectively. It is asserted that the amplification in Raman signal is initiated by the contribution of surface plasmon. The mechanism of enhancement is identified, analyzed and understood. The admirable features of our results are highly beneficial for solid-state laser and optical device fabrication.

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ABSTRAK

Penalaan terhadap peningkatan sifat-sifat optik kaca bukan organik berdop nadir bumi dengan pembenaman nanopartikel logam merupakan satu keperluan didalam bidang fotonik. Isu utama adalah nanopartikel terdop dioptimumkan dan seterusnya pencirian yang terperinci dibuat bagi menambah baik sifat penyerapan dan pemancaran. Kesan nanopartikel perak (AgCl) terhadap sifat fizikal, struktur dan optik bagi kaca tellurit zink- natrium didopkan dengan Sm^{3+} dengan komposisi $65TeO_2$-25ZnO-$10Na_2O$-0.15 gram Sm_2O_3-(*y*)AgCl dimana y = 0, 0.003, 0.075, 0.12 dan 0.18 gram telah ditentukan. Sampel telah disediakan melalui kaedah sepuh lindap leburan dan dikaji menggunakan teknik Ultra Lembayung Cahaya Nampak (UV-Vis-NIR), spektroskopi fotoluminesens (PL), Mikroskopi Pancaran Elektron (TEM), spektroskopi Raman dan serakan sinar-X (XRD). Corak XRD mengesahkan bahawa sifat amorfus kaca yang telah disediakan. Penggabungan nanopartikel Ag menunjukkan bukti perubahan kepada penyusunan struktur dan perubahan sifat fizikal kaca. Spektrum serapan UV-Vis-NIR menunjukkan enam puncak serapan berpusat pada 472 nm, 943 nm, 1089 nm, 1237 nm, 1392 nm, 1491 nm masing-masing merujuk kepada peralihan $^6H_{5/2}\rightarrow{}^4I_{11/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$. Mikroskopi TEM, menunjukkan taburan saiz partikel sekata dengan purata saiz ~7.9 nm. Spektrum PL, menunjukkan dua jalur pancaran terletak pada 599 nm dan 643 nm yang masing-masing merupakan transisi $^4G_{5/2}\rightarrow{}^6H_{7/2}$ dan $^6H_{9/2}$. Peningkatan keamatan PL yang dicerap merupakan sumbangan ketinggian medan elektrik setempat Ag yang terletak disekitar ion Sm^{3+}. Spektra Raman pula menunjukkan kemunculan lima puncak berasal daripada 99.44 cm^{-1}, 179.88 cm^{-1}, 414.37 cm^{-1}, 673.44 cm^{-1} and 768.66 cm^{-1} dimana masing-masing mewakili puncak boson, sambungan rangkaian mewakili Te_2O_7 dan TeO_4^{-4}, regangan dan lenturan sambungan Te-O-Te, getaran antisimetrik hubungan Te_{eq}-O_{ax}-Te dan regangan oleh NBO yang bersebelahan atom Te. Ianya telah ditegaskan bahawa penguatan dalam isyarat Raman dimulakan oleh sumbangan plasmon permukaan. Mekanisme peningkatan ini telah dikenalpasti, dianalisis dan difahami. Ciri-ciri yang berfaedah daripada hasil ujikaji ini adalah sangat bermunafaat untuk laser keadaan pepejal dan pembuatan peranti optik.

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LIST OF SYMBOLS/ ABBREVIATIONS

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
M_{av}	-	Molecular weight average
n,	-	Refractive Index
n^1	-	Density of Electron
$\vec{P}$	-	Polarization Density
χ	-	Molar fraction

P	-	Density
P_x	-	Toulene density
W_a	-	Weight in air
Wb		Weight in solution
R'	-	Refractivity
T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Melting Temperature
Ag	-	Silver
AgCl	-	Silver Chloride
Sm^{3+}	-	Trivalent Samarium Ion
TeO_2	-	Tellurium Oxide
Zn	-	Zinc
ZnO	-	Zinc Oxide
NaO	-	Sodium Oxide
CB	-	Conduction Band
CR	-	Cross-Relaxation
LPE	-	Local Field Effect
RE	-	Rare Earth
PL	-	Photoluminescence
GSA	-	Ground State Absorption
ET	-	Energy Transfer
SPR	-	Surface Plasmon Resonance
IR	-	Infrared
TEM	-	Transmission Electron Microscope
XRD	-	X-Ray Diffraction
NR	-	Non-Radiative
NBO	-	Non-Bridging Oxygen
Te-O	-	Tellurium-Oxygen Bond
Zn-O	-	Zinc-Oxygen Bond
Te-O-Te	-	Tellurium-Oxygen-Tellurium Bond
TP	-	Trigonal pyramidal
TBP	-	Trigonal Bipyramidal

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Lately, rare earth doped binary and ternary glasses became attractive for their unique and exotic properties suitable for assorted applications. Amongst all, tellurite glass exhibits superior properties such as high refractive index, high dielectric constants, a wide band infrared transmittance and large third order non-linear optical susceptibility. In addition, they posses relatively low transformation temperatures, high densities and non hygroscopic properties, which limit the application of phosphate and borate glasses. However, the TeO_2-ZnO glass system strongly depends on the cooling rate and the size of the melt, especially in the TeO_2-rich region (Sidek *et al*., 2009). Recently, glass technology has been developed immensely due to the presence of nanoparticles (NPs) and rare earth (RE) ions as alternative method in glass making. These two components are essential to alter the glass features for potential application in industry.

Researchers have been demonstrated a huge experimental studies to raise the new achievement in glass technology. It is acknowledged that some electronic devices utilize the rare earth for glass making(Mohan Babu *et al*. 2011). However, the luminescence will be quenched at high concentration of RE due to the energy transfer processes (Dai *et al*., 2006). In order to enhance some features, several components or elements are incorporated into the host glass. Materials in nanosize such as silver NPs exhibit different feature from the bulk. Glass doped with RE and NPs have been attracts more attention due to their particular optical properties. Recently, RE ions coupled with plasmonic metal nanoclusters have been developed as a novel tool to enhance the luminescence intensity of RE ions (Widanarto *et al*. 2013).

Obtaining high up-conversion (UC) efficiency of RE ions is probable when the electromagnetic excitation induces the noble metal NPs to exhibit surface plasmon resonance (SPR). SPR is the collective oscillation of the free conduction band (CB) electrons in the visible region (Som *et al*., 2009). Accommodating the RE ions the vicinity of metallic NPs leads to the luminescence efficiency when the optical frequency of the excitation beam or luminescence frequency near the SPR of NPs (Osorio *et al*., 2012). However, the controlled production of NPs with desired ranges of sizes and shapes embedded in glass matrix is a current challenge with promising applications (Rivera *et al*., 2011).

Generally, the optical properties of glass embedded with metallic NPs is strongly depend on the NPs size, shape, density and spatial distribution as well as the surrounding environment of the host medium. It is of an utmost importance to achieve a necessary an accurate understanding of the optical interaction and structural particularities of the constituents which build the glass network (Baia *et al*., 2007). Therefore, the aim of this study is to examine and determine the effects of varying concentration of AgCl on the optical and structural properties of glass.

1.2 Problem Statement

The effect of NPs in modifying the characteristic of glass is emerged as an important issue for most researchers. The enhancement in optical properties of glass can be achieved by interactions of RE and NPs. However, most of the previous studies reported the interaction between the RE ions, which is well known as the co-doped glass. The optical and structural properties of glass is strongly depends on the constituents which build the glass network. Nevertheless, the study of Sm^{3+} doped zinc-sodium tellurite glass containing Ag NPs has not reported widely. Further experimental details regarding the effect of AgCl to the Sm^{3+} doped zinc-sodium tellurite glass is not reported apparently. Therefore, the careful sample preparation and characterization are required to identify the interaction and mechanism associates with this glass system. Enhancing the luminescence of Sm^{3+} ions by adding Ag NPs is worth to be understood due to their potential in solid state laser. In this study, the behaviour of Sm^{3+} ions with presence of Ag NPs will be interpreted by investigate the physical, optical and structural properties of glass.

1.3 Objectives

1.3.1 General Objectives

The general objectives of this study are:

i. To synthesize a series of Sm^{3+} doped sodium tellurite glass with varying concentration of AgCl.
ii. To identify the presence of Ag NPs in the glass sample.
iii. To determine the influence of AgCl concentration on optical absorption of glass.

iv. To determine the effect of Ag NPs on the luminescence properties of the prepared sample.

1.3.2 Specific Objectives

The specific objectives of this study are:

i. To prepare series of glass with melt quenching technique.
ii. To calculate the density and molar volume.
iii. To determine the absorption spectra by UV-VIS-NIR spectroscopy.
iv. To determine the emission spectra by photoluminescence spectroscopy.
v. To quantify structural behaviour in term of vibration band of these glasses by using Raman spectroscopy.
vi. To identify the topological structure of the glass with help of transmission electron microscope.

1.4 Significance of the Study

The purpose of doing this research is to study the absorption and luminescence properties due to interaction of metallic NPs and RE in glass matrix. It is necessary to have a clear understanding on the mechanism associate with the optical interaction and structural changes in glass. In addition, the effect of RE and metallic NPs in enhancing the particular properties of glass is significant in this study. Despite many studies, the interaction of metallic NPs and RE in alter the physical, optical and structural properties of glass are not fully investigated. Therefore, we aim to communicate the information and findings from this research to the other researchers and community. The collected information and findings is vital for civilisation, science and technology. Further, the optimal features of our glass samples are beneficial for application in photonic devices and solid state laser.

1.5 Scope of Study

It is important to verify the potential host glass for glass formation. TeO_2-based glass is chosen as a glass former due to their characteristics as the most stable oxide of tellurium with melting point of 733^oC (El-mallawany 2002). TeO_2 also possesses good optical properties in visible and infrared region. This feature attracts lots of attention due to its good stability of Te between metal and non-metal. However, tellurite glasses possess weaker Te–O bonds, which can be easily broken and this is advantageous to accommodate RE ions and metal oxides. Thereby, TeO_2-ZnO glass system is chosen due to their capability as a host for optically active RE ions. Among lanthanides, the Sm^{3+} ion is one of the most studied ions due to its interesting photoluminescence properties because of different quenching channels exhibited by the $^4G_{5/2}$ emitting level (Amjad *et al*., 2012). Furthermore, the RE ions (Sm^{3+} in this study) are able to perform the lasing action in the glassy hosts (Sahar *et al*., 2008). In addition, the incorporation of Ag NPs in glass matrix contributes to the structural and optical modification. Therefore, the luminescence can be intensified by energy transfer from the Ag NPs or due to enhancement of the local field that acts on the Sm^{3+} ion located in the proximity of the NPs (de Araujo *et al*., 2013).

The glass samples are prepared by melt quenching technique. The melt quenching technique also called a rapid cooling. This method is likely avoiding the materials from becoming crystalline which include the slow rate of cooling. The absorption and emission analysis is the main part in this study. From absorption spectra, the energy band gap and Urbach energy can be determined. In addition, the luminescence characteristic from the Photoluminescence spectroscopy is performed to analyze the lasing properties of Sm^{3+} ions with the presence of Ag NPs. X-ray diffraction (XRD) is performed for structural determination. This technique employs the mechanism in which the X-ray can be diffracted by lattice spacing. TEM technique involves the interaction of beams of electron transmitting through the sample. Thus, the presence of Ag NPs can be verified. The IR and Raman spectra of glasses provide significant information of various bonds present between different ions in glass network.

1.6 Thesis Outline

This thesis describes the preparation, optimization the concentration of AgCl and characterization of various properties of Sm^{3+} doped zinc-sodium tellurite glass embedded with Ag NPs. The simple conventional melt quenching technique is used to prepare the glass samples.

Chapter 1 presents a brief introduction of the current study. In this chapter, emphasis is given to problem statement, objectives, significance and the scope of the study.

Chapter 2 signifies the detailed information regarding main concept underlying the glassy state. The definition and general process involved in glass formation will be discussed. This is followed by a discussion of tellurite glass as a glass former. An emphasized is given to the Sm^{3+} ions as dopants and role of Ag NPs to mediate the enhancement and modify the structural properties. The optical interaction experienced by Ag NPs is discussed briefly.

Chapter 3 describes the methodology of sample preparation and experimental procedure. In addition, the detailed information regarding characterization in terms of physical properties, structural and optical properties of samples are underlined. The fundamentals and basic concept of respective instruments are rendered.

Chapter 4 exemplify all the results in terms of different characterizations and measurements of prepared glass samples. The findings in modification of structural properties which leads to the improvement in optical properties of glass will be discussed. Findings which are attributed to different mechanisms will be compared with literatures and assertions are made.

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