STRUCTURAL AND SPECTROSCOPIC PROPERTIES OF ZINC-TELLURITE DOPED SAMARIUM GLASS EMBEDDED WITH GOLD NANOPARTICLES

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To

The memory of my beloved father, Alhaji Aliyu Tanko who passed away while I am just graduating from junior secondary school;

My beloved mother, Hajiya Fadimatu Abdullahi;

My Uncle, Mohammad Tukur Tanko;

My Brothers and Sisters;

My wife, Rukayya Abubakar

And

My Children, Aliyu and Abubakar;

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ABSTRACT

Modification in the structural, thermal and optical properties of samarium ions (Sm^{3+}) doped zinc-tellurite glass with gold nanoparticles (Au NPs) embedment was studied. In this regard, two series of zinc-tellurite glass systems with composition (80-y)TeO_2-20ZnO-ySm_2O_3, where $0.0 \leq y \leq 2.0$ mol% and (79-x)TeO_2-20ZnO-1Sm_2O_3-x$AuCl_3$, where $0.00 \leq x \leq 0.10$ mol% were prepared using melt quenching technique. Sm^{3+} ions and Au NPs concentration dependent structural, thermal and optical properties of these glass samples were determined. Structural characterizations were made using X-ray diffraction (XRD), Fourier transform infra-red (FTIR) and Raman spectroscopy. Thermal properties were measured by differential thermal analyzer (DTA). The existence, size, and morphology of Au NPs in the glass matrix were examined via transmission electron microscopy (TEM). Optical properties of glass samples were determined using UV-Vis-NIR absorption and photoluminescence (PL) spectroscopy. The occurrence of broad hump in the XRD patterns verified the amorphous nature of glass samples. FTIR spectra exhibited three major bands which were allocated to the TeO_3, TeO_4 units and bending vibrations of Te-O-Te linkages or Zn-O vibrations. Raman spectra showed the bending vibrations mode of Te-O-Te and stretching vibration modes of non-bonding oxygen linked to the TeO_4 and TeO_3 units. TEM images manifested the existence of Au NPs of average diameter 17.12 nm. Both glass systems exhibited thermal stability and glass forming ability which was increased from 80 °C to 143 °C and from 0.37 to 1.03, respectively with the increase of Au NPs contents. The absorption spectra displayed ten prominent peaks corresponding to the transitions from the ground state ($^6H_{5/2}$) to various excited states of Sm^{3+} ions. Surface plasmon resonance (SPR) bands of Au NPs were detected at 652 and 715 nm. The bonding parameters of the prepared glass samples that were calculated from the absorption spectra revealed the covalent/ionic nature of the rare earth-ligand (Sm-O) bond. Optical band gap energies and the Urbach energy values were determined. The ligand field parameters estimation showed that the crystal field strength and the degree of covalency between ions were increased. Besides, the interelectronic f-f repulsion Racah parameters were decreased with increasing NPs contents. The room temperature PL spectra disclosed four prominent emission bands centered at 562 nm (green), 600 nm (orange), 644 nm (red) and 709 nm (red) which were assigned to the $^4G_{5/2} \rightarrow {}^6H_{5/2}$, $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$ and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ transitions, respectively. Overall, the structural, thermal and optical properties of the studied tellurite glass system were strongly influenced by the embedment of Au NPs in the glass matrix. The alteration in the overall properties was attributed to the mediation of Au NPs surface plasmon, radiative transitions and cross-relaxation effects. The improvement in the properties of these glasses may be beneficial for different optical applications.

ABSTRAK

Pengubahsuaian struktur, sifat terma dan optik kaca zink-tellurite dengan nanopartikel emas (Au NPs) didop ion samarium (Sm^{3+}) sentiasa diberi perhatian. Dalam hal ini, dua siri sistem kaca zink-tellurite dengan komposisi (80-y)TeO_2-20ZnO-ySm_2O_3 dengan $0.0 \leq y \leq 2.0$ mol% dan (79-x)TeO_2-20ZnO-1Sm_2O_3-x$AuCl_3$ dengan $0.0 \leq x \leq 0.10$ mol% telah disediakan menggunakan teknik pelindapan leburan. Kebergantungan struktur, terma dan sifat optik sampel kaca ini terhadap kepekatan ions Sm^{3+} dan Au NPs di tentukan. Pencirian struktur telah dibuat menggunakan pembelauan sinar-X (XRD), Spektoskopi infra-merah jelmaan Fourier (FTIR) dan spektroskopi Raman. Sifat terma telah diukur menggunakan penganalisa pembezaan terma (DTA). Kewujudan, saiz dan morfologi Au NPs dalam matrik kaca telah diukur menggunakan mikroskopi penghantaran elektron (TEM). Sifat optik sampel kaca telah ditentukan menggunakan penyerapan UV-Vis-NIR dan spektroskopi berpendaflor (PL). Kewujudan bonggol lebar pada corak XRD menunjukkan keadaan keamorfus semula jadi sampel kaca. Spektrum FTIR menunjuk tiga jalur utama yang sepadan dengan unit TeO_3, TeO_4 dan getaran membengkok Te-O-Te atau getaran Zn-O. Spektrum Raman menunjukkan mod getaran membengkok Te-O-Te dan mod getaran meregang oksigen tak-terikat yang terhubung kepada unit TeO_4 dan TeO_3. Imej TEM menunjukkan kewujudan Au Nps dengan diameter purata 17.12 nm. Kedua-dua sistem kaca mempunyai kestabilan terma dan keupayaan pembentukan kaca yang meningkat masing-masing daripada 80^{o}C kepada 143^{o}C dan daripada 0.37 kepada 1.03 dengan peningkatan kandungan Au NPs. Spektrum penyerapan memaparkan sepuluh puncak utama yang sepadan dengan transisi dari keadaan dasar ($^6H_{5/2}$) ke pelbagai keadaan teruja ion Sm^{3+}. Jalur resonans plasmon permukaan (SPR) bagi Au NPs telah dikesan pada 652 nm dan 715 nm. Parameter ikatan bagi sampel kaca yang disediakan telah dikira dari spektrum penyerapan untuk menunjukkan keadaan semulajadi ikatan kovalen/ionik atom ligan-nadir bumi (Sm-O). Tenaga jurang optik dan tenaga Urbach telah ditentukan. Anggaran parameter medan ligan menunjukkan bahawa kekuatan medan hablur dan darjah kekovalenan antara ion telah meningkat. Di samping itu, parameter Racah f-f tolakan interelektronik didapati mengurang dengan pertambahan kandungan NPs. Spektrum PL pada suhu bilik menunjukkan empat jalur pancaran utama pada 562 nm (hijau), 600 nm (oren), 644 nm (merah) dan 709 nm (merah) yang masing-masing mewakili transisi $^4G_{5/2} \rightarrow {}^6H_{7/2}$, $^4G_{5/2} \rightarrow {}^6H_{9/2}$ dan $^4G_{5/2} \rightarrow {}^6H_{11/2}$. Secara keseluruhannya, sifat struktur, terma dan optik dari sistem kaca tellurite yang dikaji dipengaruhi oleh penambahan NPs Au ke dalam matrik kaca. Perubahan pada keseluruhan sifat adalah disebabkan oleh plasmon permukaan NPs Au, transisi sinaran dan kesan santaian silang. Peningkatan sifat kaca ini mungkin berguna untuk aplikasi optik yang berbeza.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

at%	-	Atomic Percentage
Au	-	Gold
BO	-	Bridging Oxygen
CB	-	Conduction Band
CR	-	Cross Relaxation
DNA	-	Data Not Available
DTA	-	Differential Thermal Analyser
EDX	-	Energy Dispersive Electron Microscope
EM	-	Electromagnetic
ESA	-	Excited State Absorption
ET	-	Energy Transfer
eV	-	Electron Volt
FFT	-	Fast Fourier Transformation
FTIR	-	Fourier Transform Infrared
GSA	-	Ground State Absorption
HMO	-	Heavy Metal Oxide
HRTEM	-	High Resolution Transmission Electron Microscopy
IR	-	Infrared
KBr	-	Potassium Bromide
Ln	-	Lanthanide
LSPR	-	Localized Surface Plasmon Resonance
NBO	-	Non-Bridging Oxygen
NIR	-	Near Infrared
NMEF	-	Nanometal Enhanced Fluorescence
NPs	-	Nanoparticles
NR	-	Non-radiative
O_{ax}	-	Axial Oxygen

O_{eq}	-	Equatorial Oxygen
PL	-	Photoluminescence
RE	-	Rare Earth
REIs		Rare Earth Ions
Ref	-	Reference
RT	-	Room Temperature
SEM	-	Scanning Electron Microscope
SPR	-	Surface Plasmon Resonance
TEC	-	Thermal Expansion Coefficient
tbp	-	Trigonal Bipyramid
TEC	-	Thermal Expansion Coefficient
TEM	-	Transmission Electron Microscopy
tp	-	Trigonal Pyramid
UC	-	Upconversion
UV	-	Ultraviolet
VB	-	Valence Band
Vis	-	Visible
wt%	-	Weight Percentage
XRD	-	X-Ray Diffraction
ZnO	-	Zinc Oxide

LIST OF SYMBOLS

2θ	-	Angle of Diffraction
σ	-	Spin of Metal ion
A_b	-	Absorbance
A	-	Activator
β	-	Nephelauxetic Ratio
D	-	Crystal Lattice Planar Spacing
Dq	-	Crystal Field Strength
$E°$	-	Reduction Potential
E_{opt}	-	Optical Band Gap
E_g	-	Direct Band Gap
E'_g	-	Indirect Band Gap
ρ	-	Density
δ	-	Bonding Parameter
ν	-	Band Position
υ	-	Wavenumber
$h\upsilon$	-	Photon Energy
ΔE	-	Urbach Energy
η	-	Nephelauxetic Function
h	-	Planck's Constant
Q	-	Charge of Electron
M	-	Mass of Electron
λ	-	Wavelength
H_r	-	Hruby Parameter
n	-	Refractive Index
n_t	-	Order of Reflection
M_{av}	-	Average Molecular Weight

α	-	Absorption Coefficient
α_m	-	Electronic Polarizability
χ-TeO_2	-	Mineral Tellurite
Ψ-TeO_2	-	Paratellurite
S	-	Sensitizer
S_0	-	Ground State
Sa	-	Angular Spin
S_1	-	Excited Singlet State
ΔT	-	Stability Factor
N_A	-	Avogadro's Number
V_i	-	Packing Density Factor
V_t	-	Ionic Packing Density
V_m	-	Molar Volume
R_m	-	Molar Refraction
T_c	-	Glass Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Glass Melting Temperature
W_a	-	Weight of Sample in Air
W_b	-	Weight of Sample When Immersed in an Immersion Liquid

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter provides a brief background on the development of glass research, in particular, the tellurite-based glass systems, aims towards potential application in solid state lasers and other optical devices. It also introduces the problem statement, objectives, scope of the study and significance of the research.

1.2 Research Background

Tellurium dioxide (TeO_2) host based glasses are of scientific and technological interest due to their unique properties. These properties include good rare earth (RE) solubility, better thermal stability, wide transparency window in UV-Vis-NIR region (0.4-6 μm), high refractive index (~2.0), low melting temperatures (~800 °C) and low cut off phonon energy (700 cm^{-1}) [1-4]. Tellurite glasses shows interesting and significant optical properties which have received significant attention in their potential applications over the last few years; several devices including optical amplifiers, planar waveguides, and nano wires have been fabricated using tellurite glasses. However, TeO_2 itself is a conditional glass-former, which requires a special fast-quenching procedure to vitrify [5]. Due to the difficulty of vitrifying TeO_2 alone by traditional method, the high transparent tellurite glasses are obtained by introducing other oxides such as transition metal oxides, alkaline oxides and alkaline-earth oxides without adding any conventional glass former [6].

The incorporation of RE into oxide glasses have provided us with useful materials with several photonic applications such as optical fiber, laser emission and optical application [7, 8]. The trivalent samarium ion (Sm^{3+}) is preferred to other RE family due to its diligently lying energy level from the ground state to other excited states [9]. It also shows upconversion luminescence for ${}^4G_{5/2} \rightarrow {}^6H_{5/2}$ (green), ${}^4G_{5/2} \rightarrow {}^6H_{7/2}$ (orange), and ${}^4G_{5/2} \rightarrow {}^6H_{9/2}$ (red) anti-Stokes transitions [10], which is potentially used for obtaining upconverted solid state lasers [11]. In addition to the upconverted luminescence enhancement, luminescence quenching is also experienced at a higher concentration of the RE due to an increasing ion-ion interaction between the RE ions (REIs). Therefore, a new way to further enhance these hosts need to be applied which makes researchers to incorporate metallic nanoparticles (NPs) into these oxide glasses [12, 13].

Due to its enhancement in luminescence and non-linear optical properties arise from the NPs on the host material, RE-doped oxide glasses embedded with metallic NPs have attracted attention [14]. The luminescence efficiency could be affected as a result of energy transfer (ET) between the NPs and REIs and sometimes due to the local field improvement which acts on the REIs closely sited around the NPs. Malta et al. in 1985 describes the fluorescence enhancement of silver particles on Eu^{3+} doped glass [15]. Subsequently, the effect of the luminescence enhancement of gold (Au) or silver (Ag) NPs on various RE-doped glasses have been studied over the years [16, 17]. The known characteristics low phonon energy for TeO_2 glasses makes them outstanding materials to weaken the loss of non-radiative (NR) and multiphonon relaxation that enables an enhanced upconversion efficiency in the REIs-doped glasses [18]. In an REIs: metal NPs-doped tellurite glass, plasmon-enhanced fluorescence results from two contending processes: (1) increased excitation rate of the REIs due to local field modification and (2) quenching rate due to energy transfer by the excited REIs to the surface of metal NPs. Hence, to maximize the luminescence, the ratio between metal NPs and REIs concentrations must be less than unity. As the localized electric field produced by the metal NPs, plasmons has a distribution maximum in its vicinity, so there is a critical distance between the REIs and the metal NPs surface where the fluorescence enhancement is largest. Thus, at distances shorter than this REIs - GNP critical distance, the quantum efficiency diminishes due to the coupling to nonresonant higher-order plasmon modes [19].

Essentially, structure of anisotropic NPs possessing sharp edges form such as ellipsoids, nanorods, nanowires and concentric shells are more preferred candidates for nanometal enhanced fluorescence (NMEF) [20]. NMEF, as well called surface plasmon-enhanced luminescence is a phenomenon where plasmonic metal nanostructures in the area of the REIs enhance their free space spectroscopic features and enormously rise the production of their weak optical transitions through the generation of strong electric fields upon electromagnetic (EM) excitation [21]. Surface charges are induced on the NPs upon EM excitation. And the surface charge densities are significantly improved and confined close to the sharp edges of the anisotropic nanostructures that behaves like light-harvesting nano-optical antennas, transforming visible light to large localized electric field referred to as lightning-rod effect [22]. Consequently, NPs, if sited near REIs, RE's excitation rate is significantly enhanced. Therefore, intensive efforts are being dedicated to the study and exploring of surface plasmons and local field characteristics of non-spherical metal NPs. Some of the most predominant application of nanostructured materials at the surface are surface-enhanced fluorescence, surface-enhanced resonance imaging, and spectroscopy, surface enhanced Raman scattering.

1.3 Problem Statement

Over the years, the linear and nonlinear optical properties of RE-doped glasses with metallic NPs embedment have been investigated. Dousti et al. [23] has investigated the enhanced upconversion emissions in Er^{3+} doped tellurite glass containing gold NPs. Mahraz et al. [24] have studied the effect of silver NPs enhanced luminescence of Er^{3+} ions in boro-tellurite glasses. Although there are some reports on the influence of Sm^{3+} ions on zinc tellurite glass [25], there is no report on the role of gold NPs on structural, thermal, optical and ligand field parameters of this glass systems. Despite many spectroscopic studies, the role of Au NPs and enhancement of optical properties on Sm^{3+} ions in tellurite glass is far from being understood. The effect of Au NPs on the thermal stability and thermal properties is not much studied. Additionally, the enhancement or quenching in the luminescence by adding NPs is not correlated to the ligand field parameters of Sm^{3+} doped zinc-tellurite glasses. It is proclaimed that the SPR wavelength depends on the host and metal-dielectric as well as on the dimensions and shape of the NPs [16]. The tunability of plasmon band positions at different wavelengths giving an effect to the plasmon resonance facilitates varieties of applications [26]. However, the correlation between SPR and ligands fields interaction due to the growth of NPs is not established yet.

It has been shown that glass formation in the ZnO-TeO_2 system depends strongly on the cooling rate, particularly in the TeO_2-rich region. Bürger et al. [27] shows that a glass system with composition $80TeO_2$-$20ZnO$ being melted at 1123K in platinum crucible and cooled with a rate of about ~10 K/s was found to be most stable. In this study, tellurite glass has been used as a host whereas samarium oxide as a dopant and Au NPs will be embedded into it. We prepared the glass systems using the conventional melt-quenching method. Therefore, we aim to explore, examine, identify and determine the effects of Au NPs on the structural and thermal characterizations of the prepared glass samples. Furthermore, optical absorption, emission, bonding between ligands and oxide glasses are determined.

1.4 Objectives of Study

i. To prepare samarium doped zinc-tellurite glass systems without and with gold nanoparticles embedment by melt quenching technique.
ii. To determine the structural characteristics of the synthesised glass systems using Infrared and Raman spectroscopy.
iii. To determine the influence of varying samarium and Au NPs concentration on the absorption and photoluminescence properties of the synthesised glass systems.
iv. To evaluate the mechanism of luminescence enhancement and quenching in the synthesised glass system.

1.5 Scope of Study

In this study, conventional melt quenching technique is used to prepare samarium doped zinc-tellurite glass systems without and with gold nanoparticles embedment having composition of (80-y)TeO_2-20ZnO-ySm_2O_3, where $0 \leq y \leq 2.0$ in mol% and (79-x)TeO_2-20ZnO-1Sm_2O_3-x$AuCl_3$, where $0 \leq x \leq 0.1$ in mol%. The amorphous nature of the prepared glass is confirmed by X-ray diffraction (XRD), the existence and the size distribution of the Au NPs inside the glass is verified by TEM and HRTEM imaging technique. Archimedes method is used in measuring the density of the prepared glass systems. And these measured densities are employed in determining the molar volume and ionic parking density. The glass transition temperature (T_g), crystallization temperature (T_c), and melting temperature (T_m) are determined using the thermogram from the Differential Thermal Analyser (DTA) as well as its stability. The vibrational Structural features are demonstrated using FTIR and Raman spectroscopy. The optical absorption parameters such as refractive index, optical band gap, Urbach energy, and electronic polarizability are measured by means of UV-Vis-NIR spectroscopy. Also, the bonding and ligand field parameters are calculated from the higher energy region of the UV-Vis optical absorption spectra. The emission spectra will be studied using photoluminescence spectrometer.

1.6 Significance of Study

In the realm of nanoglass (glass containing NPs), this research might be of much significance because it reached out to current knowledge which exist in that field. Tellurite glass is characterized by large refractive index and low maximum phonon energy which helps to improve fluorescence emission compared to another oxide host.

This study can provide great knowledge on the structural, optical and thermal behaviour of NPs in the vicinity of Sm^{3+} doped zinc tellurite glass. The results can establish the role of Au NPs on the optical, structural and thermal properties of Sm^{3+}

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