LASING AND THERMOLUMINESCENCE FEATURES OF SAMARIUM/DYSPROSIUM CO-DOPED BARIUM-SULFUR-TELLURO-BORATE GLASS EMBEDDED WITH PURE GOLD NANOPARTICLES

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Doctor of Philosophy

Faculty of Science
Universiti Teknologi Malaysia

OCTOBER 2020

ACKNOWLEDGEMENT

Alhamdulillah, All Praise due to Almighty ALLAH (S.W.T), the lord of mankind whose decree is “be” and there it is. His infinite peace and blessings be to our noble prophet Muhammad (S.A.W) the seal of the prophets, his household, and his entire companions.

I would like to express my heartfelt indebtedness and appreciation to my supervisor in the person of Professor Madya Dr. Suhairul Bin Hashim, for being an extraordinary mentor whose encouragement, support, and guidance made this doctoral research a reality. I would also like to extend my incredible honor to my co-supervisor, Professor Madya Dr. Sib Krishna Ghoshal for his wonderful advice, motivation, and guidance throughout the journey of my PhD. Indeed, Sir, your comments, critics and suggestions made me understand the ethics and core values of research.

My special gratitude goes to my parents for their moral and spiritual upbringing, support, help, and prayers from childhood to present. May Jannatul Firdaus be your final abode, Ameen. The role of my wives and kids towards actualizing this Doctoral research is highly acknowledged and Insha Allah will not go in vain. The support, guidance and prayers of M.C. Chiromawa and other colleagues will not be forgotten. May Allah reward you abundantly Ameen.

Finally, financial support from Tertiary Education Trust Fund (Tetfund), Abuja, Nigeria through the AST&D intervention (TETF/FED.UNIV/GUSAU/ASTD/2017) of Federal University, Gusau, Zamfara state, Nigeria was gratefully and duly acknowledged.

ABSTRACT

Contemporary technological advancements drive the need for optimized Rare Earth Ions (REIs) host combination for lasing and thermoluminescence (TL) applications whereas the weak absorption cross-section of the REIs remains a challenge. Herein, a series of Sm^{3+} doped Barium-Sulfur-Telluro-Borate (BSTB) glass was prepared by the melt-quenching method. The role of Sm^{3+}/Dy^{3+} co-doping and pure gold nanoparticles (AuNPs) embedment on the absorption cross-section of Sm^{3+} in the synthesized glasses was studied. The structural properties of the quenched glass samples were investigated using X-Ray Diffraction (XRD), Fourier Transform Infrared (FTIR) and Energy Dispersive X-ray (EDX) analyses. The Ultraviolet-Visible-Near-Infrared Spectroscopy (UV-Vis-NIR) spectra of the glasses exhibited characteristic absorption transitions of Sm^{3+}. The photoluminescence (PL) spectra of the Sm^{3+}-doped glasses showed four emission bands due 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 in Sm^{3+} with varying intensities. The glass made with Sm_2O_3 content of 1 mol% revealed maximum PL intensity and this composition was chosen for co-doping with Dy_2O_3. Moreover, the obtained lasing attributes were also considerably affected due to co-doping factor. High branching ratio and emission cross-section of 93.12% and 60.99×10^{-23} cm^2 were obtained, respectively. Energy transfer mechanism from the Dy^{3+} to Sm^{3+} was confirmed from PL decay analysis. This energy transfer induced the shift in the CIE coordinates of the glasses from the reddish-orange zone towards the white region. The obtained Transmission Electron Microscope (TEM) images of the AuNPs embedded glasses showed the presence of irregularly shaped AuNPs with an average diameter of 29 nm. The AuNPs Surface Plasmon Resonance (SPR) bands were found at 670 and 718 nm. The SPR mediation stimulated the shift in the chromaticity coordinates from white to yellowish-orange zone. While the Sm^{3+}-doped sample glasses did not show any TL response, the co-doped and AuNPs embedded glasses displayed TL response exhibiting a simple second-order glow curve with maximum intensity (I_m) at 272 ^oC. The appearance of I_m at high-temperature region indicated the stability of the glass against fading effect. The activation energies of the optimum glass obtained using the peak shape, initial rise, whole glow curve and computerized glow curve deconvolution methods were 1.021, 1.50, 1.537, and 1.369 eV, respectively. In conclusion, BSTBSmDyAu0.1 glass sample is best suited for lasing and thermoluminescence applications due to high branching ratio and emission cross-section approximately 55% and 111×10^{-23} cm^2, respectively, coupled with the exhibited simple TL glow curve.

ABSTRAK

Kemajuan teknologi sezaman mendorong perlunya penggabungan perumah ion nadir bumi (REIs) yang dioptimumkan untuk aplikasi laser dan termopendarcahaya (TL) sedangkan keratan rentas penyerapan REIs yang lemah kekal menjadi cabaran. Di sini, satu siri kaca Sm^{3+} terdop Barium-Sulfur-Telluro-Borat (BSTB) telah disediakan dengan kaedah pelindapkejutan leburan. Peranan ko-dopan Sm^{3+}/Dy^{3+} dan nanopartikel emas tulen (AuNPs) pada keratan rentas penyerapan Sm^{3+} dalam kaca yang disintesis telah dikaji. Sifat struktur sampel kaca yang dilindapkan disiasat menggunakan analisis Pembelauan Sinar-X (XRD), InfraMerah Transformasi Fourier (FTIR) dan tenaga sinar-X terserak (EDX). Spektrum kaca spektroskopi Ultralembayung-Cahaya Nampak-Hampir-InfraMerah (UV-Vis-NIR) menunjukkan ciri peralihan penyerapan Sm^{3+}. Spektrum kefotopendarcahayaan (PL) dari kaca terdop Sm^{3+} menunjukkan empat jalur pancaran disebabkan oleh peralihan dalam Sm^{3+} daripada $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ dan $^4G_{5/2}\rightarrow{}^6H_{11/2}$ dengan keamatan yang berbeza. Kaca yang dibuat dengan kandungan 1 mol% Sm_2O_3 menunjukkan keamatan PL yang maksimum dan komposisi ini dipilih sebagai ko-dopan dengan Dy_2O_3. Tambahan pula, atribut laser yang diperoleh juga dipengaruhi oleh faktor ko-dopan. Nisbah mencabang dan keratan rentas pancaran yang tinggi masing-masing berjumlah 93.12% dan 60.99×10^{-23} cm^2 telah diperolehi. Mekanisme pemindahan tenaga dari Dy^{3+} ke Sm^{3+} disahkan dari analisis pereputan PL. Pemindahan tenaga ini mendorong anjakan koordinat CIE kaca dari zon jingga-kemerahan ke kawasan putih. Imej mikroskop elektron hantaran (TEM) yang diperoleh dari kaca terbenam AuNP menunjukkan kehadiran AuNP berbentuk tidak seragam dengan diameter purata 29 nm. Jalur Resonans Plasma Permukaan (SPR) AuNPs ditemui pada 670 dan 718 nm. Penengah SPR merangsang anjakan koordinat kromatisiti dari zon putih ke jingga-kekuningan. Walaupun sampel kaca terdop Sm^{3+} tidak menunjukkan sambutan TL, tetapi sampel kaca ko-dopan dan kaca AuNP terbenam menunjukkan sambutan TL yang mempamerkan lengkung bara aturan kedua yang ringkas dengan keamatan maksimum (I_m) pada suhu 272 ^oC. Penampilan I_m di kawasan suhu tinggi menunjukkan kestabilan kaca terhadap kesan pudaran. Tenaga pengaktifan kaca optimum yang diperoleh menggunakan kaedah bentuk puncak, kaedah kenaikan awal, kaedah lengkung bara penuh, dan kaedah penyahkonvolusi lengkung bara berkomputer masing-masing adalah 1.021, 1.50, 1.537, dan 1.369 eV. Kesimpulannya, sampel kaca BSTBSmDyAu0.1 paling sesuai untuk aplikasi laser dan termopendarcahaya disebabkan oleh nisbah mencabang yang tinggi dan keratan rentas pancaran masing-masing kira-kira 55% dan 111×10^{-23} cm^2, ditambah dengan lengkung bara TL ringkas yang dipamerkan.

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LIST OF ABBREVIATIONS

A_{rad}	-	Radiative transition probability
AuNPs	-	Gold Nanoparticles
$BaSO_4$	-	Barium Sulfate
B_2O_3	-	Borate
CGCD	-	Computerized Glow Curve Deconvolution
DTA	-	Differential Thermal Analysis
Dy_2O_3	-	Dysprosium Oxide
Dy^{3+}	-	Dysprosium Ion
EDX	-	Energy Dispersive X-ray
E_g	-	Band Gap Energy
E_{urb}	-	Urbach’s Energy
F_{cal}	-	Calculated oscillator strengths
F_{exp}	-	Experimental oscillator strengths
FTIR	-	Fourier Transform Infrared
HR	-	Heating Rate
HRTEM	-	High-Resolution Transmission Electron Microscope
IR	-	Initial Rise
KBr	-	Potassium bromide
LSPR	-	Localised Surface Plasmon Resonance
NIR	-	Near Infra-red
PL	-	Photoluminescence
PS	-	Peak Shape
REIs	-	Rare Earth Ions
S_{ed}	-	Electric dipole line strength
Sm_2O_3	-	Samarium Oxide
Sm^{3+}	-	Samarium Ion
S_{md}	-	Magnetic dipole line strength
TeO_2	-	Tellurium Oxide
TL	-	Thermoluminescence
T_c	-	Critical Temperature

T_m	-	Maximum Temperature
UV-Vis-NIR	-	Ultraviolet-Visible-Near infra-red
WGC	-	Whole Glow Curve
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

b	-	Order of Kinetics
c	-	Speed of light
E	-	Activation energy
h	-	Planck's constant
J	-	Total angular momentum
k	-	Boltzmann's Constant
n	-	Refractive Index
s	-	Frequency factor
μ	-	Symmetry Factor
v	-	Wavenumber
$\bar{\bar{\beta}}$	-	Nephelauxetic Ratio
β	-	Heating rate
^oC	-	Degree Celsius
$\alpha(v)$	-	Absorption coefficient
Ω_2	-	Judd-Ofelt parameter
Ω_4	-	Judd-Ofelt parameter
Ω_6	-	Judd-Ofelt parameter
δ	-	Bonding Parameter
λ	-	Wavelength
η	-	Quantum Efficiency
β_r	-	Branching ratio
τ_{rad}	-	Radiative lifetime
λ_P	-	Emission band position
δ_e	-	Stimulated Emission Cross-section

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter describes the fundamental knowledge of the study. Thus, the chapter encompasses the research background, problem statement, research objectives, scope, the significance of the research, and the thesis outline.

1.2 Research Background

Rare-earth ions (REIs) doped glasses formed an integral part of the contemporary high technological advancements in the fields of solid-state lasers, radiation dosimetry, fibre optic transmission cables, astronomical science and quantum electronics[1–3]. Rare earth (RE) elements consist of the members of Lanthanides from Lanthanum to Lutetium. When embedded in a host matrix, RE usually exists in trivalent nature and also serves as a luminescence centre by substituting the host ions[4]. The luminescence property of REIs arises from the f-f electronic transition of the corresponding REIs[5] and is being exploited in lasers for example. Technological advancement drives the need for developing novel and strategic optical materials with an optimized performance aimed at meeting the ensuing needs. In this regard, the selection of a suitable REIs-host combination to match a specific requirement remains challenging[6, 7].

Crystals and glasses are found to be excellent gain materials with minimal scattering loss and thereby emerged as the dominant REIs host[8]. While crystal-based hosts are characterized by narrow absorption bandwidth due to the settlement of the active REIs in an only specific site of the crystal lattice, glasses due to their random atomic arrangements offer the advantage of having a broader absorption and emission bandwidths[8] thanks to the occupation of many divergent sites by the REIs. Furthermore, the simplicity of fabrication, low cost coupled with uniform spectroscopic features of glasses makes them good hosts for REIs.

The choice of a suitable REIs glass host for a particular application requires an in-depth analysis of the physical and spectroscopic properties aimed at obtaining vital information to be used in fabricating efficient materials for diverse applications[9, 10]. These properties are determined by the structure, composition, optical absorption, and optical emission of the REIs doped glass matrix. Among the different types of glasses, oxide glasses prove to be stable hosts for highly efficient spectral features of REIs[11]. Multi-component REIs doped oxide glass matrices such as telluro-borate are characterized by improved luminescence properties and are excellent optical materials for example with regards to low-loss solid-state lasers[12].

Borate being one of the best oxide glass formers[10] receives much attention from researchers worldwide due to its unique physical and optical properties such as low melting temperature, excellent heat stability, good glass-forming ability and high optical transparency[13, 14]. However, borate-based glasses have very low chemical durability, highly hygroscopic, and large phonon energy (~1400 cm^{-1}) which reduces the luminescence efficiency. Tellurite based glasses, on the other hand, have excellent REIs solubility, good chemical durability, wide optical transmission window, and low phonon energy (~700 cm^{-1}) which reduces the non-radiative decay loss and in turn, improves the luminescence efficiency of the matrix[15, 16]. Thus, multi-component telluro-borate glasses are promising host matrices for REIs due to enhanced photoluminescence (PL) and thermoluminescence (TL) efficiencies.

REIs doped tellurite and borate-based glasses are reported to have excellent thermoluminescence dosimetric features[17–20]. In a similar manner, $BaSO_4$ was reported to exhibits good TL response[21] and hence its introduction into telluroborate glass could improve the associated TL response. Thus, REIs doped barium-sulfur-telluro-borate glass matrix is a potential thermoluminescence dosimetric material. TL process is the emission of light from an irradiated defects containing semiconductor or insulator due to the influence of heat[18]. The emitted light is a function of both the trapping centers and the absorbed radiation. Among the diverse applications of TL is radiation dosimetry, which is used in monitoring the amount of radiation dose absorbed. The selection of dosimetric material depends on the quality of the derivable thermoluminescence kinetic (trapping) parameters which describe the nature of the trapping centers. Borate based glasses are characterized by good kinetic parameters and are, therefore, potential TL materials but certain drawbacks need to be addressed.

In the quest of overcoming some of the drawbacks such as luminescence quenching which results from the low absorption cross-section associated with the REIs and which affects both the lasing and thermoluminescence potentials of REIs doped glasses, the co-doping technique is being applied. Co-doping simply refers to the presence of two or more species of REIs at an appropriate proportion in a single host matrix. This creates an energy transfer channel between the two active REIs which in turn enhances the associated absorption cross-section. Alternatively, the incorporation of metallic nanoparticles also augments the absorption cross-section of the REIs[22]. Nanoparticles are inorganic particles with a size in the range of 1 to 100 nm. The luminescence enhancement by nanoparticles is as a result of their role in favouring localized surface plasmon resonance (LSPR) which is the resonant oscillation of the nanoparticles free electrons in the presence of light[23]. LSPR generates a strong electric field within the vicinity of the REIs and this results in the subsequent improvement in the spectroscopic properties of the glass matrix[24]. The enhancement may also be due to the energy transfer between the REIs and the nanoparticles. Nanoparticles possess the advantage of high surface area to volume ratio, large surface energy, and plasmon excitation[23].

1.3 Problem Statement

The quest for an optimized REIs-host combination for a particular scientific and/or technological application is continuous and never-ending task[25]. Telluroborate-based glasses are prominent REIs hosts[26, 27], However; the absorption cross-section of REIs is too weak because the partially filled 4f shell is well screened by the filled 5s and 5p orbits leading to a weak interaction between the REIs and the host lattice, hence weak absorption cross-section which in turn leads to fluorescence intensity quenching effect in the presence of a higher concentration of REIs[28]. Thus, novel multi-component telluro-borate glass design with an efficient energy transfer mechanism through co-doping compositions of REIs is still needed. It is, therefore, intended to establish the effects of Sm^{3+}/Dy^{3+} co-doping on the physical, structural, lasing, and TL properties of Barium-sulfur-telluro-borate glass in the present work.

Furthermore, the witnessed nano-technological advancements have paved a way for intensive research on the incorporation of metallic nanoparticles into REIs doped glasses for scientific applications. Nevertheless, the incorporation of pure gold into these glasses in general and telluro-borate glasses in particular is still lacking as in most of the reported researches gold salt ($AuCl_3$) was considered. The problem of using gold salts is that nanoparticles are formed by aggregation when heated, the heating process may lead to the growth of crystals within the glass matrix and thus causes unwanted light scattering which dampens the operational efficiency of gain materials such as laser[22]. Thus, in the present research, the influence of pure gold nanoparticles on the physical, structural, and lasing properties of the proposed glasses will be determined.

The knowledge of the thermoluminescence kinetic parameters is necessary for defining the overall efficiency of a TL process. Meanwhile, metallic nanoparticles embedded multi-component REIs co-doped telluro-borate glass has some excellent latent thermoluminescence (TL) features, however; the role of pure AuNPs embedment on the TL kinetic parameters of Sm^{3+}/Dy^{3+} co-doped systems are far from being explored. Thus, it is intended to determine the thermoluminescence kinetic parameters of pure gold embedded Barium-sulfur-telluro-borate glass co-doped with different concentrations of Samarium and Dysprosium ions.

The goal is to explore the lasing and thermoluminescence kinetic parameters of optimized multi-component REIs co-doped glasses embedded with pure gold nanoparticles. Thus, it is intended to explore the role of Sm^{3+}/Dy^{3+} co-doping and pure AuNPs embedment on structural, physical, optical, lasing, and thermoluminescence kinetic parameters of $BaSO_4$-TeO_2- B_2O_3-Sm_2O_3-Dy_2O_3-Au glass matrix. The research is an attempt to extend the existing knowledge of the principles of interaction between light and metals which is vital to the development of the glass industry.

1.4 Research Objectives

The main objective of the study is to synthesize a single material that will concurrently serve as efficient lasing and TL material. Thus, it is intended to explore the structural, physical, optical, lasing, and TL kinetic parameters of $BaSO_4$-TeO_2-B_2O_3-Sm_2O_3-Dy_2O_3-Au glass matrix. Specific objectives of the research are:

(a) To synthesize Sm^{3+}/Dy^{3+} co-doped barium-sulfur-telluro-borate glasses with and without pure gold nanoparticles at varying contents.

(b) To determine the influence of Sm^{3+}/Dy^{3+} co-doping on the structural, physical, optical, and lasing properties of the synthesized glasses.

(c) To evaluate the role of AuNPs embedment on the structural, physical, optical, and lasing parameters of barium-sulfur-telluro-borate glass systems based on the framework of Judd-Ofelt analysis.

(d) To evaluate the role of both Dy^{3+} co-doping and AuNPs embedment on the thermoluminescence kinetic parameters of the optimum glass samples upon exposure to ionizing radiation.

1.5 Scope of the Study

In this research, three series of barium-sulfur-telluro-borate glasses at varying constituent compositions were synthesized using the melt-quenching technique. The optimum un-doped composition was determined and analyzed through a differential thermal analyzer (DTA) before being doped with the REIs. Using the obtained optimum composition, three series of barium-sulfur-telluro-borate glasses doped with different concentrations of Sm/Dy REIs and embedded with pure gold nanoparticles were then prepared by the melt-quenching technique. The densities of the prepared samples were calculated using the Archimedes principle with toluene as the standard liquid. The phases of the synthesized glasses were confirmed utilizing XRD measurements. Fourier Transform Infra-red (FTIR) analysis was employed in probing the structural adjustments in the prepared glass systems. The refractive indices of the samples were measured by an Abbe refractometer. To analyze the morphological structure of the samples, Energy Dispersive X-ray (EDX) mapping and High-Resolution Transmission Electron Microscope (HRTEM) analyses were employed. To determine the spectroscopic properties such as Bandgap energy, Urbach's energy, molar refractivity, refractive index, and the dielectric constant of the samples, Uv-Vis-NIR absorption analysis was employed. The LSPR peak of the pure gold nanoparticles was obtained using Uv-Vis-NIR spectroscopy. Photoluminescence analysis was used in investigating the emission pattern of the glass samples. While the energy transfer processes were discussed using both photoluminescence and decay curve analyses, the luminescence colour adaptations of the synthesized glass samples were explored using CIE 1931 guidelines. Furthermore, the lasing parameters such as stimulated emission cross-section, branching ratio and optical gain were determined based on the framework of Judd-Ofelt analysis. Irradiation of the samples was done using a 6 MV photons set up linear accelerator (LINAC) in the dose range of 0 to 4 Gy. The Thermoluminescence response were recorded using Harshaw 4500 thermoluminescence dosimeter (TLD) reader in the temperature range of 0 to 400 ^{o}C and optimum heating rate of 7 ^{o}C/s.

1.6 Significance of the Study

This research is an attempt to produce new and optimized REIs glass host in the form of barium-sulfur-telluro-borate with enhanced lasing and TL kinetic parameters suitable for applications in laser and radiation dosimetry. Samarium and Dysprosium ions were adopted for co-doping the newly developed glass host whereas pure gold nanoparticles were chosen for embedment. The inquisition will establish the role of pure gold nanoparticles embedment in enhancing the structural, physical optical, lasing, and thermoluminescence properties of synthesized glasses. Briefly, the study aims to produce a single material capable of serving as both efficient lasing and TL material and thus pave way for several applications.

1.7 Thesis Outlines

This thesis is divided into five different chapters. Chapter 1 outlines the background of the research, problem statement, objective of the research, the significance of the research, and the thesis outlines. Chapter 2 presents the literature review of the spectroscopic and thermoluminescence behaviours of REIs in glasses in general and in telluro-borate glasses in particular which is our proposed material. Samarium and Dysprosium elements were reviewed; gold nanoparticles were also reviewed and linked to the proposed material. Different thermal, structural, spectroscopic, and thermoluminescence characterization techniques were also discussed. Chapter 3 highlights the adopted experimental techniques. This includes sample preparations, optimization of the base sample composition, and the details of the various sample characterization methodologies. Chapter 4 consists of two parts; Part I presents the structural, physical, optical, and lasing features of the prepared series I, II, and III glass systems. The lasing features were derived from the radiative properties based on the Judd-Ofelt analysis. Part II presents the thermoluminescence kinetic parameters obtained from different methods of glow curve analysis. These parameters were discussed and analyzed. Chapter 5 contains the final summary and conclusion of the findings of the study of Sm/Dy ions co-doped barium-sulfur-telluro-borate glass system embedded with AuNPs. Suggestions for further study were also included.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. Abdullahi. I., Hashim. S., Ghoshal. S.K., Sa'adu. L. Modified structure and spectroscopic characteristics of Sm^{3+}/Dy^{3+} co-activated barium-sulfur-telluro-borate glass host: role of plasmonic gold nanoparticles inclusion. *Optics and Laser Technology*, 2020. 132(2020): 106486 **(Q1, IF: 3.233)**

2. Abdullahi. I., Hashim. S., Ghoshal. S.K. Waveguide laser potency of samarium doped $BaSO_4$-TeO_2-B_2O_3 glasses : Evaluation of structural and optical qualities. *Journal of Luminescence,* 2019. 216(2019): 116686 **(Q1, IF: 3.280)**

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4. Abdullahi. I., Hashim. S., Ghoshal. S.K., A.U. Ahmad. Structures and spectroscopic characteristics of barium-sulfur-telluro-borate glasses : Role of Sm^{3+} and Dy^{3+} Co-activation, Mater. Chem. Phys. 247 (2020) 122862 **(Q2, IF: 3.408)**