STRUCTURAL AND OPTICAL CORRELATION OF EUROPIUM AND DYSPROSIUM CO-DOPED BORO-TELLURO-DOLOMITE GLASSES INCORPORATED WITH SILVER NANOPARTICLES

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DEDICATION

This thesis is dedicated to my beloved wife for her support and encouragement and also to the loving memory of my late mother.

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ABSTRACT

Rare earth ions doped glasses with tailored lasing and light emitting potency are active area of materials science research. In this view, a series of Eu^{3+} and of Dy^{3+} co-doped (at various concentrations) boro-telluro-dolomite (BTD) glasses included with silver nanoparticles (Ag NPs) were prepared by melt-quenching method and characterized for the first time. The role of co-dopants and Ag NPs contents on the optical and structural performance of the studied glasses was evaluated. X-Ray diffraction (XRD) patterns of the as-quenched samples affirmed their amorphous nature, and the energy dispersive X-ray (EDX) spectra showed the presence of actual chemical compositions of the glasses. The existence of Ag NPs with an average diameter of 25.50 nm in the glass matrix was verified using the high-resolution transmission electron microscopy (HRTEM) analyses. Ultrasonic and Vicker's micro-hardness analyses displayed high mechanical stability of these glasses. Fourier transformed infrared (FTIR) and Raman spectra of the glasses revealed various chemical functional units in their network structure. Ultraviolet-visible-near-infrared (UV-Vis-NIR) spectral data was used to estimate the optical band gap energies and refractive indices of the glasses using three different models. BTD1.0AgCl sample exhibited a distinct broad surface plasmon resonance (SPR) band at 479 nm. The photoluminescence spectra of the Eu^{3+}-doped glasses (under 464 nm excitation) displayed five significant emission bands at 577, 591, 611, 652 and 702 nm matching with ${}^5D_0 \rightarrow {}^7F_J$ transitions (with $J = 0$, 1, 2, 3, and 4) wherein the band intensities were quenched beyond 1 mol% of Eu^{3+} doping. The symmetry of the ligands in the vicinity of Eu^{3+} and Dy^{3+} in addition to their bonding nature of the glasses were evaluated from the Judd-Ofelt intensity parameters Ω_2, Ω_4, and Ω_6. The observed emission spectral overlap and change in the fluorescence lifetime indicated a substantial bi-directional energy transfer between Eu^{3+} and Dy^{3+} in the glass matrix, confirming the Forster-Dexter energy transfer process via the electric dipole–dipole interactions. Besides, the inclusion of Dy^{3+} altered the emission color of Eu^{3+} from red region with CIE coordinates of (0.638, 0.361, for BTD1.0Eu glass) to white light zone with CIE coordinates of (0.395, 0.317). The achieved hue was very close to the ideal red color phosphor value of (0.67, 0.33) and pure white light value of (0.33, 0.33). The calculated lasing parameters such as the transition probability, stimulated emission cross-section, luminescence branching ratio, optical gain, gain bandwidth, and radiative lifetime showed enhancement due to the incorporation of Dy^{3+} and Ag NPs. The produced glasses exhibited high color purity (ranged from 24 – 97.04%) and better quantum efficiency (ranged from 54.88 – 97.81%), wherein such improvements were mainly attributed to the efficient energy transfer between Eu^{3+} and Dy^{3+} as well as the Ag NPs SPR-induced local field effects. Overall, a correlation between the structural and optical features of the BTD glasses was determined. Based on the obtained results it can be concluded that the proposed glasses have great potential for the solid-state red laser and white light emitting devices applications.

ABSTRAK

Kaca yang didopkan dengan nadir bumi dengan penyesuaian laser dan potensi pengeluaran cahaya adalah bidang aktif dalam kajian sains bahan. Dalam pandangan ini, satu siri kaca boro-telluro-dolomit (BTD) didopkan dengan Eu^{3+} dan Dy^{3+} dengan perangkuman zarah nanoperak (Ag NPs) disediakan menggunakan kaedah peleburan kaca pelindapkejutan dan dicirikan buat pertama kali. Peranan dopan bersama dan kandungan Ag NPs pada prestasi optik dan struktur kaca yang dikaji telah dinilai. Pola pembelauan sinar-X (XRD) sampel sepuhan lindap yang telah disediakan mengesahkan sifat amorfus kaca dan pola sinar-X sebaran tenaga (EDX) menunjukkan kewujudan komposisi kimia sebenar kaca. Kewujudan Ag NPs dengan diameter purata 25.50 nm di dalam matrik kaca telah disahkan menggunakan analisis mikroskop elektron penghantaran resolusi tinggi (HRTEM). Analisis ultrasonik dan Kekerasan-mikro Vicker’s menunjukkan kestabilan mekanikal kaca yang tinggi Spektroskopi inframerah *fourier* transformasi (FTIR) dan spektrum Raman mendedahkan pelbagai units imia fungsian di dalam struktur jaringan. Data spektrum ultra ungu-cahaya nampak-inframerah hampir (UV-Vis-NIR) telah digunakan untuk menganggar jurang tenaga optik dan indeks refraktif kaca menggunakan tiga model berbeza. Sampel BTD1.0AgCl mempamerkan satu bonggol lebar resonan plasmon permukaan (SPR) terbeza pada 479 nm. Spektrum kefotopendarcahayaan kaca yang didopkan dengan Eu^{3+} (diterujakan pada 464 nm) menunjukkan lima jalur pancaran penting pada 577, 591, 611, 652 dan 702 nm berpadanan dengan peralihan $^5D_0 \rightarrow {}^7F_J$ (dengan J = 0, 1, 2, 3, and 4) yang mana keamatan jalur melindap di luar 1 mol% pendopan Eu^{3+}. Simetri ligan kawasan sekitar Eu^{3+} dan Dy^{3+} ion dan sifat ikatan mereka telah dinilai daripada penilaian parameter keamatan Judd-Ofelt Ω_2, Ω_4, and Ω_6. Pemerhatian terhadap pertindihan spektrum dan perubahan pada tempoh hayat pendarfluor menunjukkan terdapat pemindahan tenaga dua arah yang besar di antara Eu^{3+} dan Dy^{3+} dalam matrik kaca, yang mengesahkan pemindahan tenaga Forster-Dexter melalui interaksi elektrik dwikutub-dwikutub. Tambahan pula, perangkuman Dy^{3+} merubah warna pancaran Eu^{3+} daripada kawasan cahaya merah dalam koordinat CIE (0.638, 0.361 untuk BTD1.0Eu) kepada kawasan cahaya putih dalam koordinat CIE (0.395, 0.317). Rona yang dicapai menghampiri dengan nilai fosfor warna merah ideal (0.67, 0.33) dan nilai cahaya putih unggul (0.33, 0.33). Nilai parameter yang telah dikira seperti kebarangkalian peralihan, keratan rentas pancaran terangsang, nisbah pencabang pendarcahaya, gandaan optik, gandaan lebar jalur dan tempoh hayat sinaran menunjukkan peningkatan kesan daripada perangkuman Dy^{3+} dan Ag NPs. Kaca yang dihasilkan mengeluarkan ketulenan cahaya yang tinggi (dalam julat antara 24 – 97.04 %) dan efikasi kuantum yang lebih baik (54.88 – 97.81 %) yang mana pembaikan ini dikaitkan dengan pemindahan tenaga efisien di antara Eu^{3+} dan Dy^{3+} dan kesan medan setempat yang dicetuskan oleh Ag NPs SPR. Secara keseluruhan, perkaitan antara sifat struktur dan optik kaca BTD telah ditentukan. Berdasarkan dapatan kajian, dapat disimpulkan bahawa kaca yang dicadangkan mempunyai potensi besar sebagai laser merah keadaan pepejal dan aplikasi peranti pemancar cahaya putih.

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LIST OF ABBREVIATIONS

AgCl	Silver Chloride
ASF	Absorption Spectral Fitting
BTD	Boro-Telluro-Dolomite
BO	Bridging Oxygen
CB	Conduction Band
CCT	Correlated Color Temperature
CIE	Commission International de IʹEclairage
CP	Color Purity
CR	Cross Relaxation
CRC	Cross Relaxation Chanel
DASF	Derivation of Absorption Spectra Fitting
DD	Dipole Dipole
Dm	Dolomite Marble
Dp	Dolomite Pebble
DQ	Dipole Quadrupole
ET	Energy Transfer
ETC	Energy Transfer Chanel
FTIR	Fourier Transform Infrared
FWHM	Full Width at Half Maximum
GSA	Ground State Absorption
IR	Infrared
JO	Judd-Ofelt
LSPR	Localized Surface Plasmon Resonance
LFE	Local Field Effect
NPs	Nanoparticles
NR	Non-Radiative
OBGEs	Optical Band Gap Energy
PL	Photoluminescence
QQ	Quadrupole Quadrupole
REIs	Rare Earth ions

RO	Luminescence intensity ratio
SPR	Surface Plasmon Resonance
TEM	Transmission Electron Microscope
UV	Ultraviolet
VB	Valence Band
VIS	Visible
XRD	X-Ray Diffraction

LIST OF SYMBOLS

2θ	Angle of Diffraction
$Arad$	Radiative Probability
A_{ed}	Electric-Dipole Transition Probability
A_{md}	Magnetic-Dipole Transition Probability
D	Fractal bond connectivity
E	Young modulus
E_{Drc}^{Tauc}	Direct Optical Band Gap Energy by Tauc’s model
E_{Indrc}^{Tauc}	Indirect Optical Band Gap Energy by Tauc’s model
E_{Drc}^{ASF}	Direct Optical Band Gap Energy by ASF model
E_{Indrc}^{ASF}	Indirect Optical Band Gap Energy by ASF model
E_{Drc}^{DASF}	Direct Optical Band Gap Energy by DASF model
F	Field Strength
f_{cal}	Experimental Oscillator Strength
f_{exp}	Experimental Oscillator Strength
G	Shear modulus
H_V	Hardness
I	Intensity
K	Bulk modulus
L	Longidunal modulus
M_{av}	Average Molecular Weight
n	Refractive index
N	Concentration
N_A	Avogadro’s number
r_p	Polaron Radius
r_i	Inter Nuclear Distance
R_m	Molar Refraction
$S_{ed}\,, S_{md}$	Electric and Magnetic Dipole Line Strengths
T	Temperature

T_c	Crystallization Temperature
T_g	Glass Transition Temperature
T_m	Melting Temperature
t	Time
$\|U^{(i)}\|^2$	Reduced Matrix Elements
V_M	Molar Volume
V_L	Longitudinal velocity
V_S	Shear velocity
W	Weight
α	Absorption Co-efficient
α_m	Molar Polarizability
β_r	Branching Ratio
σ	Poisson ratio
ε	Dielectric Function
ε_0	Permittivity of Volume
h	Plank’s Constant
ρ	Density of Glass
σ_{emi}	Emission Cross-Section
Ω_i	Judd-Ofelt Intensity Parameters
δ_{rms}	Root Mean Square Deviation between Experimental and Calculated Oscillator Strengths
λ	Wavelength
τ	Lifetime
ω	Wavenumber
$\|(S,L)J >$	Electronic State of an Element Defined by its Spin, Orbital and Total Momentums
σ_P^E	Stimulated Emission Cross-Section
$\Delta\lambda_{eff}$	Effective Band Width

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CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter recap the pertinent introduction covering the basic background knowledge of the study along with the most relevant literature survey from previous researched. Besides, it includes the problem statement which led to this research, objectives of the research, scope of the research, significance of the research and the thesis outline.

1.2 Background of the Research

The increasing demand for cost effective eye safe lasers and energy-saving light emitting devices has grab research attention in the hunt for novel optical materials that can be customize to meet the aforementioned need [1-4]. Of all materials, glassy ones by virtue of their easy preparation, good chemical stability and possibility to produce bulky size (discs/slabs/rods) to complex miniatures (graded index optical fibers) are widely preferred compared to crystalline [5, 6].

Interestingly, borate oxide (B_2O_3) among various glass hosts has been spotted as one of the principal glass formers with a very high glass forming ability, large concentration of rare earth ion solubility, high thermal stability and widespread range of transparency [7]. Indisputably, these unique characteristics of borate glasses make them promising for optical device construction [8]. In contrast, the imperfections of borate glass hosts such as hydroscopic nature and high phonon energy ($\sim$1300-1500 cm^{-1}) seriously hinder its practical applications. Aiming to circumvent these downsides, Aziz et al. [9] affirmed that inclusion of tellurium oxide (TeO_2) into B_2O_3 network forming boro-tellurite network (BTN) can powerfully conquer the weaknesses of borate thereby improving it optical performance. Indeed, this new glass system represents a perfect cooperation between the requirements of high chemical durability, higher refractive index, low phonon energy, ease fabrication, higher mechanical strength, higher thermal stability and low transmission loss in the IR region [10].

Divina et al. [11] further attest that the existence of an alkaline earth metals in BTN could significantly improve its optical qualities through $BO_3 \rightarrow BO_4$ and $TeO_3 \rightarrow TeO_4$ unit's transformations. The pioneered studied on the synthesis and characterization of synthetic calcium borotellurite (CBTe) glasses by Paz and his co-researchers [12] testified a very good thermal stability greater than 100℃ and wide optical transparency (350-2600 nm) when compared to phosphate and fused silica. Besides, Karthikeyan and his team mates [13] also affirmed that the inclusion of calcium oxides in B_2O_3-TeO_3 network is expected to overcome the hydroscopic nature and hence mechanically reinforce its network; forming a potential luminescent host in which Ca^{2+} cation can be replaced by rare earth ions. Furthermore, incorporating CaO into a glass network can cause a decline in the high phonon energy of the unadulterated oxide glasses and subsequently intensifies their optical values [14].

In spite of the renowned prospect of BT glasses containing calcium oxide, researchers have seldom used calcium rich natural mineral as modifier in BT glasses for potential applications. It is believed that the synergism between synthetic BT and dolomite mineral can form boro-telluro-dolomite (BTD) glasses of better quality [15]. Additionally, the inclusion of naturally stable and plentiful dolomite mineral in the BT network can overshoot the main shortcomings associated with artificial chemicals-based glasses such as hydroscopic nature and high production cost [15]. Stimulated by this rationale, first-ever synthesis and characterization of BTD glasses by using standard melt quenching method and various spectroscopic techniques, respectively is examined to ascertain their lasing and light emitting potency.

According to the literature survey, it has been well established that the effectiveness of optical glass host for a precise application strictly depend on the good correlations between physical, mechanical, structural and optical properties as a function of dopants; either singly, doubly doped with rare earth ions and or metallic nanoparticles co-embedment [16-19]. Among different dopants, Eu^{3+} ion has attracted special attention owning to its peculiar features like distinctive energy level's structure, valence fluctuation property and the optimized red emission allocated to $^5D_0 \rightarrow ^7F_2$ (611 nm) transition is identified as the most proper materials for making diverse optical devices [20, 21]. However, due to the parity forbidden character of 4f-4f transition, the absorption cross-section of Eu^{3+} ion is very low, leading to low emission efficiency under the ultraviolet (UV) excitation. To pay off this disadvantage, co-doping with Dy^{3+} can boost the excitation efficiencies and the luminescence of Eu^{3+} ions via co-excitation/energy transfer [19].

Undeniably, Eu^{3+} and Dy^{3+} co-activators present a good complementary which simultaneous emission of blue and greenish-yellow ($^5F_{9/2} \rightarrow ^6H_{15/2,}$ 13/2, transition of D^{y3+}) and orange-reddish ($^5D_0 \rightarrow ^7F_{2,}$ Eu^{3+} transition) light is achieved by UV laser excitation. This implies that by co-doping a glass host with suitable concentration of Eu^{3+} and Dy^{3+} ions, generation of white light become possible since the needed primary lights (blue, yellow and red) are emitted [22]. In spite the remarkable features of Eu^{3+} and Dy^{3+} co-embedment, only limited amount of pumping excitation is absorbed by these ions resulting to a very low converting efficiency, thanks to metallic nanoparticles (MNPs) for creating other excitation mechanism through energy transfer by sensitization from absorbing species in a wide spectral range. Incorporation of MNPs (as embedding agent) in a glass host along with REs (as dopants) induced sizeable enhancement in the absorption and cross-section of REs inside various disorder; thus, providing a lifeline to optical devices [23]. The renowned enrichment is attributed to the intense local electromagnetic field generated from the NPs assisted Surface Plasmon Resonance (SPR) effect.

Recently, silver nanoparticles (Ag NPs) were used by many researchers to improve the luminescence properties of REI's in glass matrices [18, 24-26]. The synergic combination between Ag NPs and REs could provide benefits like energy transfer from Ag NPs to REs and induce strong electric field in the vicinity of the REs due to SPR which in turn increase the absorption cross-section. Saad and his co-researchers [27] reported the excellent optical performance of $Eu^{3+}/Dy^{3+}/Ag$

nanoparticle co-doped phosphate glasses. Their major finding explored that a dual mode energy transfer from Ag NPs and Dy^{3+}ions to Eu^{3+} ions lead to the augmentation of the emission bands of Eu^{3+} ions. Also, the effect of silver co-doping on the Sm^{3+} luminescence upgrade in lithium tetraborate glasses was investigated by kindrat et al [28]. Truthfully, the luminescence of Sm^{3+}ions was greatly enhanced with about 1.43 times due to Ag embedment. The observed enhancement was principally credited to the excitation energy transfer from Ag^{+} and ion molecule-like nanoclusters to the Sm^{3+}ions. Despite the noted potential of Ag NPs in rare earth doped glasses, the mechanism of tailoring the localized surface Plasmon resonance (LSPR) band of Ag NPs co-embedded inside a glass host that are responsible for luminescence enhancement need further clarifications.

1.3 Problem Statement

The search for an optimized rare earth ions (REIs)-doped glasses as essential futuristic lasing and lighting emitting host is an endless mission. The glasses derived from synergetic combination of synthetic and natural minerals have been proven to be excellent host for REIs [15, 29]. However, selection of abundant minerals that can suitably be incorporated into existing synthetic based glass formers to form a new class of glass matrix remains the key issue. Besides, doping the aforesaid glass system with high concentration of single REI’s result to weak absorption cross section and luminescence quenching which greatly hinder their practical applications. In this sense, fabrication of boro-telluro-dolomite (BTD) glasses with low contents and co-doping of REIs is a necessity. Moreover, report on the energy transfer mechanism in Eu^{3+}/Dy^{3+} co-doped glasses is deficient. Thus, further research is required to comprehend the role of Eu^{3+}/Dy^{3+} co-doping on the physical, mechanical and structural features; and the possibility of enhancing optical performance in BTD glasses via energy transfer process. In addition, to explore the lasing and light emitting potency from these glasses, Judd-Ofelt, radiative and Commission International de I’Eclairage (CIE) 1931 analyses need to be performed.

Furthermore, nano-technological uprising demands the synthesis and characterization of new nanostructured materials, preferably by a simple technique but with outstanding properties and beneficial applications [30]. From literature review, coupling REIs with metallic nanoparticles became a precious strategy to improve the absorption cross section and the luminescence yield of REIs [18, 31]. Nevertheless, incorporation of AgCl NPs into Eu^{3+}/Dy^{3+} co-doped BTD glasses has not been studied yet. Hence, determining the mechanism of optical enhancement; and improved physical, mechanical and structural qualities in Eu^{3+}/Dy^{3+} co-doped BTD glasses containing AgCl NPs is the motivation and novelty behind this study.

1.4 Research Objectives

The design and fabrication of optimized glass host with lasing and light emitting potentials is the core objective of this study. In this regard, the specific research objectives are:

i. To optimize the composition of Eu^{3+}/Dy^{3+} co-doped boro-telluro-dolomite (BTD) glasses without and with varying contents of AgCl NPs

ii. To determine the role of both Eu^{3+}/Dy^{3+} co-doped and AgCl NPs embedment on the physical, mechanical, structural and optical features in BTD glasses

iii. To analyse the effect of surface plasmon resonance (SPR) and energy transfer mechanism on luminescence enhancement in BTD glasses due to Eu^{3+}/Dy^{3+} co-doped and AgCl NPs inclusion

iv. To evaluate the lasing and light emitting performance of the glasses through Judd-Ofelt, radiative and Commission International de I'Eclairage (CIE) 1931 analysis

v. To establish a structure-optical correlation responsible for the improvement in the optical properties of BTD glasses

1.5 Scope of the Research

Herein, three series of boro-telluro-dolomite (BTD) glasses with varying content of dopants (Eu^{3+} and Dy^{3+}) and AgCl NPs were prepared by melt quenching method. The densities of the prepared glass samples were determined using the Archimedes principle with distilled water as the standard liquid. The phases of the fabricated glasses were verified through XRD measurements. Meanwhile, the structural morphology of the studied glasses was analyzed by using Energy Dispersive X-ray (EDX) mapping, Scanning Electron Microscopy (SEM) and High-Resolution Transmission Electron Microscope (HRTEM). The mechanical properties were evaluated to ascertain the glass stability. Besides, Fourier Transform Infra-red (FTIR) alongside Raman analysis was employed in probing the structural changes in the prepared glass network. Ultraviolet-Visible-Near Infrared (UV-Vis-NIR), Photoluminescence (PL), and Fluorescence spectrophotometers was performed to describes the optical features. Using PL emission data, Commission International de I’Eclairage (CIE) 1931 was utilized to assess the color emission and purity of the glasses. Additionally, the energy transfer processes were discussed using both photoluminescence and decay profile. Finally, the lasing parameters such as stimulated emission cross-section, branching ratio and optical gain were determined based on the context of JuddOfelt analysis.

1.6 Significance of the Research

Studies on the optical glass material become significant due to their great potential in Nano-glass technology. In this research, the role of AgCL NPs embedded in Eu^{3+}/Dy^{3+} co-doped BTD glasses is explained by suitable control and optimizing the content of REs and NPs. However, understanding the physical, mechanical, structural and optical properties of the glass matrix is important to determine the optimum composition of the glass. Besides, the energy transfer mechanism and the SPR effect by NPs responsible for luminescence enhancement is significant in applications point of view. Furthermore, optical parameters analysis such as energy band gap, quantum efficiency, stimulated emission cross-section, branching ratio, optical band gain, emission color and purity are highly beneficial for the development of the active solid-state lasers and light emitting devices.

1.7 Thesis Outline

The content of this thesis is divided into five chapters describing the comprehensive work carried out to achieve the aims set out and meeting the objectives given: chapter one begins with a brief introduction, background of the research, problem statement, research objectives, significance of the study and the thesis outline. Chapter two contains important theories and the review of pertinent literature. The detail methodologies are described in chapter three where glass sample preparation procedures and characterization techniques are presented. Major results, analyses, discussion and comparison with existing data are summarized in chapter four. Finally, chapter five renders summary and conclusion based on the results and also offer recommendations for the future study. The calculation of glass composition, some optical properties and list of publications are appended in the Appendix A and B, respectively.

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LIST OF PUBLICATIONS

1. **Ibrahim bulus**, R. Hussin, S.K Ghoshal, Abd Rahman Tamuri, S.A Jupri. Enhanced elastic and optical attributes of boro-telluro-dolomite glasses: Role of CeO_2 doping, Ceramic International.45(15) (2019)18648-18658 (**Q1 with IF of 3.83)**

2. **Ibrahim bulus**, H Bhaktiar, R Hussin, I M Danmallam, S K Ghoshal. Realization of efficient red laser using europium doped new boro-telluro-dolomite glass hosts: Ag nanoparticles functionality. Presented at International Laser Technology and Optics Symposium held from 3-4 September 2019 at Le Grandeur Palm Resort Johor.

3. **Ibrahim bulus**, R. Hussin, S. K Ghoshal, Abd Rahman Tamuri, I. M Danmallam, Y.A Yamusa. Europium doped boro – telluro – dolomite glasses for red laser applications: A basic insight on spectroscopic traits. Under review at Journal of Non-Crystalline Solids. 534(2020)119949 (**Q1 with IF of 3.531)**
4. **Ibrahim bulus**, S. K Ghoshal, Abd Rahman Tamuri, R. Hussin, I. M Danmallam, A. S Alqarni. Customized structural and mechanical traits of boro-telluro-dolomite glasses for mobile screen protector: Rejuvenation of Eu^{3+}/Dy^{3+} co-doping. Submitted to the journal of materials chemistry and physis (**Q2 with IF of 4.094)**