STRUCTURAL AND OPTICAL PROPERTIES OF SILVER AND TITANIUM NANOPARTICLES CO-EMBEDDED MAGNESIUM-ZINC-SULFOPHOSPHATE GLASS WITH NEODYMIUM DOPING

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DEDICATION

This thesis is dedicated to Allah who strengthen me when I about to give up.

Greatly to my parents who financially support me and love me unconditionally.

To my brother who always accompanied me and supported me.

To my best friend who always cheers me up.

And to myself who keep moving forward even though I wish I could just quit.

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ABSTRACT

Demand of high emission cross–section laser amplifier is ever-increasing. The rare earth ions (REIs) doped magnesium zinc sulfophosphate (PMZ) glasses with noble metal nanoparticles (NPs) inclusion were shown potential as laser host. This study reported that the emission/absorption traits of these REIs inside the glass matrix could be improved using the NPs which enabled localized surface plasmon resonance (LSPR). The LSPR produces strong local electric field (LEF) in the vicinity of the REIs. The melt-quenching method was used to synthesize glass composition $58.5P_2O_5$-$20.0MgO$-$20.0ZnSO_4$-1.5 Nd_2O_3-*y*Ag NPs-*z*Ti NPs mol% where *y* and *z* could be in the range of 0.0-1.5 mol%. The co-embedment of two types of metal NPs inside the glass could provide flexibility to customize the generated LEF strength in the proximity of the REIs. The X-ray diffraction (XRD) analyses confirmed the amorphous nature of the as-quenched samples. The high-resolution transmission electron microscopy (HRTEM) images showed the nucleation of both Ag and Ti_3O_5 NPs inside the glass matrix. The nucleation of Ti into Ti_3O_5 NPs occurred via two-step Finke–Watzky mechanism. Both the Fourier transform infrared (FTIR) and Raman spectral analyses confirmed an insignificant structural change from the incorporation of the NPs into the glass matrix. The observed LSPR band of the Ag (≈442 nm) and Ti_3O_5 (≈588 and 774 nm) NPs verified their successful embedment into the host matrix. The ultraviolet-visible-near infrared (UV-Vis-NIR) absorption spectra of the glasses exhibited twelve characteristic absorption bands of Nd^{3+}. The photoluminescence (PL) emission spectra of the prepared glasses disclosed a prominent yellow band around 585 nm, corresponding to the transition of $^2G_{7/2}+^4G_{5/2}\rightarrow^4I_{9/2}$ in Nd^{3+}. The coupling of Ag and Ti NPs into the glass at a certain concentration could lead to PL intensity quenching. This ascribed to the local heating effect of Ti_3O_5 NPs. The Judd-Ofelt intensity and radiative parameters were calculated from the absorption and PL spectral data. The PMZ1.5Nd04Ti0.3Ag glass sample showed the highest stimulated emission cross–section of 4.78×10^{-24} cm^2. The results suggested that the co–embedment of metal NPs into glass could be an innovative strategy to tailor the spectroscopic characteristics of the glasses which is potential for photonic and solid-state laser applications.

ABSTRAK

Permintaan terhadap *amplifier* laser dengan keratan rentas pancaran yang tinggi semakin meningkat. Kaca magnesium zink sulfofosfat (PMZ) terdop ion nadir bumi dengan pemasukan nano-zarah (NPs) logam adi menunjukkan potensi sebagai hos laser. Kajian ini melaporkan sifat pancaran/penyerapan REIs dalam matriks kaca boleh ditambahbaik menggunakan NPs yang membolehkan resonan permukaan plasmon setempat (LSPR). LSPR menghasilkan medan elektrik setempat (LFE) yang kuat berdekatan dengan REIs. Kaedah pelindap-kejut leburan telah digunakan untuk menghasilkan kaca berkomposisi 58.5P_2O_5-20.0MgO-20.0$ZnSO_4$-1.5 Nd_2O_3-*y*Ag NPs-*z*Ti NPs mol% dimana *y* dan *z* boleh berada dalam julat 0.0-1.5 mol%. Pembenaman bersama dua jenis logam NPS dalam kaca memberi fleksibiliti dalam menyuaikan kekuatan LFE yang terjana berhampiran REIs. Analisis pembelauan sinar-X (XRD) mengesahkan sifat amorfus sampel. Imej mikroskopi penghantaran elektron resolusi tinggi (HRTEM) menunjukkan penukleusan kedua-dua Ag dan Ti_3O_5 NPs dalam matriks kaca. Penukleusan Ti kepada Ti_3O_5 NPs berlaku melalui mekanisma dua-langkah Finke-Watzky. Kedua-dua analisis transformasi Fourier inframerah (FTIR) dan spektrum Raman yang dicerap mengesahkan tiada perubahan struktur kaca yang ketara dengan perangkuman NPs ke dalam matriks kaca. Jalur LSPR Ag (kira-kira 442 nm) dan Ti_3O_5 (kira-kira 588 dan 774 nm) mengesahkan kejayaan pembenaman NPs ke dalam matriks kaca. Spektrum penyerapan ultra lembayung-boleh nampak-infra merah hampir (UV-Vis-NIR) mempamerkan dua belas jalur penyerapan Nd^{3+}. Spektrum kefotopendarcahayaan pancaran (PL) kaca yang disediakan mendedahkan jalur kuning ketara di sekitar 585 nm sepadan dengan peralihan $^2G_{7/2}$+$^4G_{5/2}$→$^4I_{9/2}$ dalam Nd^{3+}. Gandingan Ag dan Ti NPs dalam kaca pada kepekatan tertentu boleh merendahkan keamatan PL. Ini disebabkan oleh pemanasan setempat yang dihasilkan oleh Ti_3O_5 NPs. Keamatan parameter Judd-Ofelt, sinaran kebarangkalian peralihan, nisbah bercabang, dan keratan rentas pancaran terangsang telah dihitung menggunakan data penyerapan dan spektrum PL. Sampel kaca PMZ1.5Nd0.4Ti0.3Ag menunjukkan keratan rentas pancaran terangsang tertinggi sebanyak 4.78 × 10^{-24} cm^2. Keputusan mencadangkan pembenaman bersama NPs logam ke dalam kaca boleh menjadi satu strategi inovatif untuk menyesuaikan ciri spektroskopi kaca yang berpotensi untuk aplikasi fotonik dan laser dalam keadaan pepejal.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

Au	Gold
Ag	Silver
BO	Bridging oxygen
Ce	Cerium
CR	Cross Relaxation
DC	Down-Conversion
Dy	Dysprosium
Er^{3+}	Erbium
EDX	Energy dispersive X-ray
ET	Energy transfer
Eu	Europium
ESA	Excited state absorption
ETU	Energy transfer up-conversion
eV	Electron volt
FTIR	Fourier transform infrared
FET	Field effect transistor
FFT	Fast Fourier transform
FWHM	Full Width at Half Maximum
Gd	Gadolinium
GSA	Ground-state absorption
HRTEM	High resolution transmission electron microscopy
HST	Hypersensitive transition
Ho	Holmium

ICSD	Inorganic Crystal Structure Database
IR	Infrared
IUPAC	International union of pure and applied chemistry
JO	Judd-Ofelt
KBr	Potassium bromide
LFE	Local field effect
La	Lanthanum
Lu	Lutetium
LPE	Lone pair electron
LSP	Localised surface plasmon
LSPR	Localised surface plasmon resonance
MCA	Multichannel analyzer
MEF	Metal enhanced fluorescence
MPR	Multiphonon relaxation
Mg	Magnesium
Na	Sodium
NPs	Nanoparticles
NIR	Near-Infrared
NBO	Non-bridging oxygen
Nd	Neodymium
NR	Non-radiative
Nd-YAG	Neodymium-doped yttrium aluminum garnet
OH-	Hydroxyl group
P	Phosphate
Pt	Platinum
PL	Photoluminescence
Pr	Praseodymium

Pm	Promethium
PA	Photon avalanche
RE	Rare earth
R	Radiative decay
SEM	Scanning electron microscope
SPR	Surface plasmon resonance
SERS	Surface enhance Raman scattering
S	Sulfur
SO_4	Sulfate oxide
Sc	Scandium
Sm	Samarium
Te	Tellurite
Ti	Titanium
TiO_2	Titanium dioxide
TEM	Transmission electron microscope
Tbp	Trigonal bipyramid
Tp	Trigonal pyramid
Tb	Terbium
Tm	Thulium
UTM	Universiti Teknologi Malaysia
UV-Vis-NIR	Ultra violet-visible-near infrared
UC	Up-conversion
Vis	Visible
Xe	Xenon
XRD	X-ray diffraction
XRF	Fluorescence
XPS	X-Ray photoelectron spectroscopy

Y	Yttrium
Yb	Ytterbium
Zn	Zinc

LIST OF SYMBOLS

2θ	Angle of Diffraction
A_{ed}	Electric-Dipole Transition Probability
A_{md}	Magnetic-Dipole Transition Probability
A_{rad}	Radiative Transition Probability
E_{dir}	Direct Optical Band Gap
E_{ind}	Indirect Optical Band Gap
F	Field Strength
ΔE	Urbach Energy
f_{cal}	Calculated Oscillator Strength
$f_{\exp}$	Experimental Oscillator Strength
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
$\left\|U^{(i)}\right\|^2$	Reduced Matrix Elements
V_m	Molar Volume
W	Weight

α	Absorption Coefficient
α_m	Molar Polarizability
β	nephelauxetic ratio
β_R	Branching Ratio
ε	Dielectric Function
ε_0	Permittivity of Volume
ε_m	Medium dielectric
ε_1	Real region of the complex dielectric function
ε_2	Imaginary region of the complex dielectric function
$\hbar$	Plank's Constant
ρ	Density of Glass
σ_{emi}	Emission Cross-Section
$\eta(\nu)$	Molar Absorptivity
η	Enhancement Factor
η_q	Quantum Efficiency
Ω_i	Judd-Ofelt Intensity Parameters
δ	Bonding parameter
δ_{rms}	Root Mean Square Deviation between Experimental and
λ	Wavelength
$\lambda_{\max}$	Maxima of SPR absorption band
τ_{rad}	Radiative lifetime
ν	Wavenumber
$\left|(S,L)J\right|>$	Electronic State of an Element Defined by its Spin, Total Momentums
σ_P^E	Stimulated Emission Cross-Section
$\Delta\lambda_{eff}$	Effective Band Width
$d_{Ag-TiNPs}$	Spacing between silver and titanium nanoparticles
d_{QR}	the gap values in the quantum regime
ω_{SPR}	SPR frequency

m_e	The effective mass of free carrier
C_{ext}	extinction cross-section of the NPs
R_{NPs}	the average radius of plasmonic NPs
v_F	the electron velocity at the Fermi surface
$\Delta\omega$	FWHM of the LSPR absorption band

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Lately, rare earth (RE) ions doped materials received great attention due to their several potential applications including solid state laser, panel displays, waveguides, optical amplifier and optical fibres (Zhang *et al.*, 2015). Typically, RE ions have abundant excited energy states that enable them to absorb and emit light from the ultraviolet (UV) to near infrared (NIR) in the electromagnetic spectral range in solid hosts (Basavapoornima and Jayasankar, 2014; Filho *et al.*, 2016). Among amorphous materials, glass is the most attractive host for RE ions owing to its wide spectral transparency (280 nm to 3000 nm) (Brahmachary *et al.*, 2015), ease of fabrication and higher solubility of RE ions compared to crystals (Ahmadi *et al.*, 2016). Inspired by this fact, researchers have done extensive work to determine the best glass composition that gives the best optical performance to enhance present optical devices and materials.

Other than fluorophosphates glasses (Ratnakaram *et al.*, 2016), sulfophosphate glass is considered as potential glass host for solid state laser (Yang *et al.*, 2012). Sulfophosphate glass has low melting point, high chemical stability compared to some other phosphate glasses system and possess less hygroscopic behaviour together with high solubility of REs, transition and noble metal ions (Thieme *et al.*, 2015). The addition of Zn^{2+} cation and magnesium oxide into these sulfophoshpate glasses can further improve the glass properties. The usage of Zn^{2+} may improve the polarizability of the glass and altering the glass refractive index (Sahar *et al.*, 2015). In addition, the experimental results shows Zn^{2+} cation may increase the glass' chemical durability and decrease the glass transition making it more stable (Joshi *et al.*, 2011). The Zn^{2+} may also reduce crystallization rate of the glass and increase the glass forming ability (Gayathri Pavani *et al.*, 2011). The incorporation of magnesium oxide (MgO) may improve the glass ability over moisture attack, presumably due to the formation of more chemically resistant P–O–Mg bonds (Smith and Brow, 2014). Besides, the modifier MgO also has high ionic field strength values (~ 5 $Å^{-2}$) that may tighten the glass network and enhances the glass mechanical strength and density (Diba *et al.*, 2012). As luminescence centre, neodymium (Nd^{3+}) remains as one of the most efficient ions used for laser action, frequency up–conversion and optical fibre amplification application (Seshadri *et al.*, 2010). However, the small spectroscopic (absorption and emission) cross–section of most of the Nd^{3+} ions limit its application in solid–state laser and this drawback requires further improvement. Researchers would normally experiment with other type of REs or metallic nanoparticles (NPs) to overcome the low spectroscopic gain of Nd^{3+} ion. In-depth literature review revealed that very few studies are performed on Nd^{3+}-doped magnesium zinc sulfophosphate glasses to determine the effect of co-embedment of metallic NPs for improving the spectroscopic properties.

The present studies intend to oversee the influence of two type metallic NPs (silver and titanium) inclusion on the modification of optical properties of magnesium zinc sulfophosphate glasses doped with Nd^{3+} ions. Determining the mechanism of metal NPs that enabled localized surface plasmon resonance (LSPR) to assist optical enhancement of glass containing Nd^{3+} ion is the primary concern. Melt–quenching technique is used to incorporated NPs (Ag and Ti) into Nd^{3+} ions doped magnesium sulfophosphate glasses. Prepared glasses are systematically characterized using various analytical techniques (spectroscopy and imaging) and the data are analyzed to unravel emerging optical properties of such glasses. This work is expected to contribute towards the advancement of functional nano–glass for diverse optical and photonic applications.

1.2 Problem Statement

The cross–section of absorbance and emission of the Nd^{3+} ions inside the glasses need further enhancement for better optical properties towards the applications of solid–state laser material (Zamratul *et al.*, 2016). High doping of Nd^{3+} ions inside the glass would cause luminescence quenching and it is a drawback as solid–state laser material. In the past, RE–doped glass has been sensitized using a second dopant such as RE ions or plasmonic NPs to overcome the limitation. However, less work has been done to evaluate the prospect of Nd^{3+} ions photoluminescence when embedded with two types of metallic NPs (Ag and Ti) inside the glass. Detail studies on the mechanism of LSPR enhanced optical properties due to co-embedment of Ag and Ti NPs inside Nd^{3+}–doped magnesium zinc sulfo–phoshophate glass have not been explored. It is worth to mention that Ag and Ti NPs exhibit SPR band in the visible region that have a high probability to overlapped with the excitation or emission of the Nd^{3+} ions (Ahmadi *et al.*, 2020). Careful synthesis of such glass composition, their systematic characterization and in–depth data analysis using Judd–Ofelt theory has are not been performed earlier. The present attempt would allow the determination of the mechanism responsible for spectral modification of Nd^{3+} ions doped inside the magnesium zinc sulfo–phoshophate glass containing two types of metallic NPs which may have a good potential for solid–state laser advancement.

1.3 Research Objectives

The objectives of this study are as follows:

i. To synthesize a series of Nd^{3+}–doped magnesium–zinc-sulfophosphate glasses with different concentration of Silver (Ag) and Titanium (Ti) Nanoparticles (NPs).
ii. To evaluate the structural properties of the prepared samples containing Ag and Ti NPs.
iii. To determine the optical properties of the glasses co–embedded with Ag and Ti NPs containing Nd^{3+}.
iv. To determine the Judd Ofelt (JO) parameters, radiative transition probability (A_{rad}), branching ratio (β_R) and radiative lifetime (τ_{rad}) of the prepared glass system.

1.4 Scope of the Study

Few series of glasses with the composition of $58.5P_2O_5$–$20.0MgO$–$20.0ZnSO_4$–$1.5\ Nd_2O_3$–yAg NPs–zTi NPs mol% (where y and z were in the range 0.0–1.5 mol%) were prepared using the melt–quenching technique. The optimum doping content of Nd^{3+} kept fixed at 1.5 mol% for the second to fifth glass series. The optimum contents of Ag NPs and Ti NPs were kept constant in third and fifth glass series with a value of 0.5 mol% and 0.4 mol%, respectively. The optimum concentration of Nd^{3+} and metal NPs were based on the highest PL intensity exhibited around 585 nm corresponding to the transition of $^2G_{7/2}+^4G_{5/2}\rightarrow^4I_{9/2}$. The amorphous state of the glass is determined by X-Ray diffraction (XRD) spectroscopy. The lattice spacing of Ag and Ti NPs inside the samples is observed by High-Resolution Transmission Electron Microscope (HRTEM) to confirm its existence inside the glass. Meanwhile, Fourier Transformed Infrared (FTIR) and Raman measurement used to determine the structure and vibration bonding inside the glass. The optical properties are characterized using Ultraviolet–Visible–Near Infrared (UV–Vis–NIR) and photoluminescence (PL) spectrometer. Judd-Ofelt model was used to study the radiative nature of Nd^{3+} ions inside the glass and thus determine the glass capability as solid–state laser material.

1.5 Significance of the Research

The understanding of the fundamental physics behind properties modification of disorder network materials like the glass is important to invert a better optical material such as LED displays, solid–state laser glass and fibre optic amplifier. Improvement of REI doped magnesium–zinc sulfophosphate glass with a silver (Ag) and titanium (Ti) NPs might reveal new knowledge and insight regarding the origins of optical alteration due to overlapped SPR mediation. This means that the SPR induced by both metal NPs would create a junction where a strong local field could be generated and promote Nd^{3+} ion excitation (Hazra *et al.*, 2016). Deep analyses of spectroscopic behaviour of proposed glass using JO formalism provide easiest comparison method to overlook its capability as laser material. Furthermore, the optimization of the two metallic NPs inside the glass could contribute a basis for their large–scale synthesis useful for sundry of applications.

1.6 Outline of the Thesis

This thesis is composed of five chapters. The summaries of the chapters are as follows:

Chapter 1 presents the introduction, problem statements, research objectives, scope of the study and significance of studies.

Chapter 2 explains briefly the definition of glass and its structure, phosphate glass, rare earth (RE) ions; particularly Nd^{3+} ions, theory of plasmonic nanoparticles (Ag and Ti NPs) and some findings from previous studies.

Chapter 3 reveals the procedure in the synthesis of the glass and the instrumentations used for glass characterization in this study.

Chapter 4 presents the result of proposed glass in term of physical, structure, absorption, emission properties and Judd-Ofelt (JO) analysis.

Chapter 5 presents the conclusion of these studies and some recommendation for the future work.

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[3] **Yusof, N.N.,** Ghoshal, Jupri, S.A, Azlan, M.N., Nd^{3+} Doped Magnesium Zinc Sulfophosphate Glass: New Candidate For Up-Conversion Solid State Laser Host. *Journal of Optical Material*, 2020. **(Q2, IF: 2.78)**

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