GOLD NANOPARTICLES ASSISTED STRUCTURAL AND OPTICAL MODIFICATIONS OF SAMARIUM DOPED SODIUM-LITHIUM TELLURITE GLASS

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This thesis is especially dedicated to:

My beloved parents,

Zainuddin Mat Said, Asiah Mohamad

My Supportive Siblings,

Azidawati, Wazirudin, Razzi, Syikin

My nephew and niece,

Irsyad and Insyirah

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ABSTRACT

Modifying the physical and the optical properties of rare earth doped tellurite glasses by embedding metallic nanoparticles (NPs) with controlled size and optimized concentration is ever-demanding in solid state lasing materials. Thus, the influences of gold (Au) NPs concentration on the structural and the optical enhancements were examined. Two series of samarium (Sm^{3+}) doped sodium tellurite glasses, with and without Au NPs, which had compositions of $(80-x)TeO_2 + 10Li_2O + 10Na_2O + xSm_2O_3$ (where $0.0 \leq x \leq 3.0$ mol%) and $79TeO_2 + 10Li_2O + 10Na_2O + 1Sm_2O_3 + yAuCl_3$ (where $0.03 \leq y \leq 0.15$ mol% in excess), respectively, were prepared using melt-quenching technique. Subsequent annealing at 300°C for 3 hours was performed to control the growth of NPs. Structural and optical characterizations were determined using XRD, TEM, UV-Vis-NIR, FTIR, and PL measurements. XRD pattern confirmed the amorphous nature of the prepared glasses, whereas the TEM image manifested the growth of spherical NPs with mean size ~18 nm. Meanwhile, FTIR spectra displayed four broad absorption bands, which were assigned to TeO_4 and TeO_3 tetragonal pyramids, stretching vibration of hydroxyl group, and stretching vibration of strong metal bonding. In addition, the UV-Vis spectra exhibited six peaks that were centered at 471, 948, 1085, 1246, 1395 and 1494 nm, corresponding to the transition from ground state to different excited states of Sm^{3+} ion. Besides, significant enhancement in the absorption was observed for 0.09 mol% of Au NPs, while the Urbach energy showed an increase from 0.2374 to 0.2588 eV with the increase of NPs concentration. On top of that, the PL spectra displayed four enhanced peaks that were centered at 573, 613, 658 and 718 nm, which had been associated with the transition from the excited states to the ground state of Sm^{3+} ion. The enhancement was attributed to the surface plasmon resonance (SPR) effect and the strong local field of NPs positioned in the proximity of Sm^{3+} ion. Modifications in the physical, structural, and optical properties that originated from the alteration of glass network structure in the presence of Au NPs were analyzed and discussed. Furthermore, the mechanism of enhancement was comprehended and compared. The excellent features of the retrieved results suggest that these glasses may be nominated as potential candidates for solid state lasers, displays, and amplifiers.

ABSTRAK

Pengubahsuaian sifat-sifat fizikal dan optik kaca telurit berdop nadir bumi dengan memasukkan nanozarah logam (NPs) dengan saiz terkawal dan kepekatan yang dioptimumkan sentiasa diperlukan dalam bahan pelaseran berkeadaan pepejal. Oleh itu, pengaruh kepekatan NPs emas (Au) terhadap penambahbaikan struktur dan optik telah diteliti. Dua siri kaca natrium telurit berdop samarium (Sm^{3+}), tanpa dan dengan NPs Au yang mempunyai komposisi (80-x)TeO_2 + 10Li_2O + 10Na_2O + xSm_2O_3 (di mana $0.0 \leq x \leq 3.0$ mol%) dan 79TeO_2 + 10Li_2O + 10Na_2O + 1Sm_2O_3 + y$AuCl_3$ (di mana ≤ 0.03 $y \leq 0.15$ mol% lebihan), masing-masing, telah disediakan dengan menggunakan teknik pelindapan cair. Seterusnya, penyepuhlindapan pada suhu 300°C selama 3 jam dilakukan untuk mengawal pertumbuhan NPs. Pencirian struktur dan optik dibuat menggunakan kaedah XRD, TEM, UV-Vis-NIR, FTIR, dan PL. Corak XRD mengesahkan sifat amorfus bagi kaca yang disediakan, manakala imej TEM menunjukkan pertumbuhan NPs berbentuk sfera dengan purata saiz ~ 18 nm. Sementara itu, spektrum FTIR memaparkan empat jalur serapan yang luas yang menentukan piramid tetragon TeO_4 dan TeO_3, getaran regangan kumpulan hidroksil, dan getaran regangan ikatan logam yang kukuh. Spektrum UV-Vis mempamerkan enam puncak yang berpusat pada 471, 948, 1085, 1246, 1395 dan 1494 nm, yang sepadan dengan peralihan daripada keadaan asas kepada keadaan teruja ion Sm^{3+}. Selain itu, peningkatan besar dalam penyerapan diperhatikan untuk 0.09 mol% NPs Au, manakala tenaga Urbach menunjukkan peningkatan daripada 0.2374 kepada 0.2588 eV dengan peningkatan kepekatan NPs. Di samping itu, spektrum PL memaparkan empat puncak meningkat yang berpusat pada 573, 613, 658 dan 718 nm, yang berkaitan dengan peralihan daripada keadaan teruja kepada keadaan asas ion Sm^{3+}. Peningkatan ini adalah disebabkan oleh kesan resonans plasmon permukaan (SPR) dan medan setempat NPs yang kuat berhampiran ion Sm^{3+}. Pengubahsuaian dalam sifat-sifat fizikal, struktur, dan optik yang berasal daripada pengubahsuaian struktur rangkaian kaca dalam kewujudan NPs Au dianalisis dan dibincangkan. Mekanisme peningkatan telah difahami dan dibandingkan. Ciri-ciri yang sangat baik daripada keputusan yang diperoleh mencadangkan bahawa kaca-kaca ini boleh dicadangkan sebagai pilihan yang berpotensi untuk laser berkeadaan pepejal, paparan dan, penguat.

TABLE OF CONTENTS

4 RESULTS AND DISCUSSION

5 CONCLUSION AND SUGGESTIONS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BO - Bridging oxygen
CR - Cross relaxation
CW - Continuous wave
DC - Down-conversion
EM - Electromagnetic
ET - Energy transfer
ETU - Energy transfer Upconversion
FTIR - Fourier transform infra-red
GSA - Ground state absorption
IR - Infra-red
LSPR - Localize surface plasmon resonance
NBO - Non-bridging oxygen
NPs - Nanoparticles
PA - Photon Avalanche
PL - Photoluminescence
RE - Rare earth
REI - Rare earth ion
SPR - Surface plasmon resonance
tbp - Trigonal bypiramidal
TEM - Transmission electron microscope
tp - Trigonal pyramidal
UC - Upconversion
UV - Ultraviolet
UV-Vis - Ultraviolet visible

LIST OF SYMBOLS

$4f_n$ - Shell configuration belongs to lanthanide series
d_2 - Thickness of sample
θi - Incident angle
A - Einstein coefficient
As_2O_3 - Arsenic Trioxide
Au - Gold
$AuCl_3$ - Gold Chloride
B_2O_3 - Boron Trioxide
BeF_2 - Beryllium Floride
Cd - Cadmium
Cr_2O_3 - Chromium (III) Oxide
E_{Dir} - Direct bandgap
E_{eff} - Local Field
E_f - Final state energy
E_g - Energy bandgap
E_i - Initial state energy
E_{Ind} - Indirect bandgap
Er^{3+} - Trivalent Erbium Ion
Eu^{3+}, - Trivalent Europium Ion
E_{Urb} - Urbach energy
eV - Unit of energy
GeO_2 - Germanium Dioxide
Hg - Mercury
HO^{3+} - Trivalent Holmium Ion
I - Intensity

k - Extinction coefficient
KBr - Potassium Bromide
$KHSO_4$ - Potassium Bisulfate
Li_2O - Lithium Oxide
M - Mass sample
m - Index of optical transition
MgO - Magnesium Oxide
MoO_3 - Molybdenum (vi) Oxide
n - Refractive Index
Na_2O - Sodium Oxide
Nd^{3+} - Trivalent Neodymium Ion
nm - Unit of wavelength
P_2O_3 - Phosphorus Trioxide
Pr^{3+} - Trivalent Praseodymium Ion
q - Phonon
R - Radiative decay
R_M - Molar refractivity
SeO_2 - Selenium Dioxide
SiO_2 - Silicon Dioxide
Sm_2O_3 - Samarium Oxide
Sm^{3+} - Trivalent Samarium ion
Tb^{3+} - Trivalent Terbium ion
TeO_2 - Tellurite Oxide
TeO_3 - Tellurium Trioxide
TeO_4 - Tellurate
T_g - Transition temperature
V - Volume
V_m - Molar volume
w_a - Weight of glass sample in air
w_ι - Weight of glass sample in liquid
WO_3 - Tungsten Oxide
Yb^{3+} - Trivalent Ytterbium Ion
Zn - Zinc

$ZnCl_2$ - Zinc Chloride

ZnO - Zinc Oxide

α - Polarizibility

ΔE - Urbach energy

θ - Bragg angle

θ_r - Reflection angle

λ - Wavelength

ρ - Density

χ - Susceptibility

μ - Fixed Dipole Moment

ω - Oscillation Frequency

Φ - Flux

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Lately, rare earth (RE) doped tellurite glasses with embedded metallic nanoparticles (NPs) has received special attention due to their interesting properties like high REI solubility, higher refractive indices compared to silicates and fluoride glasses, huge amplification bandwidth, high infrared transmittance and good thermal and mechanical stability (Kassab et al., 2008; Ghoshal, et al., 2012; Liu, 2007). They are emerging as promising candidates for practical applications in photonics as optical amplifiers and recording, laser-active media, also laser up-converters for infrared to visible range. The interest of the present study is driven by its features that help in developing new solid-state short-wavelength laser.

Undoubtedly, there are renewed research interests on doped tellurite glasses containing metallic NPs that focus on their extraordinary third-order non-linear properties and enhanced luminescence characteristics (Jlassi, et al., 2010; Ghoshal, et al., 2011). Despite of many efforts the understanding on unusual structural, thermal, optical and electronic properties originate from the incorporation of NPs is still lacking. In optimization and applications, the characterizations of optical and also thermo-optical properties are very essential. These require careful synthesis, characterizations, and thorough analyses. Therefore, this research is attempted to make detailed investigations of such properties and compare with past studies, which would ultimately enable to understand their fundamental origin of improved properties in such glasses.

The relevance of the present study relates to the need of technology for producing efficient and better glasses with controlled dopants and NPs. This study is basically important for perceiving the mechanism responsible for the modifications in thermal, structural and optical properties in glassy materials. We hope that our systematic experimental methodology of sample preparation and spectroscopic studies would make accurate quantitative estimate regarding the optical and structural behavior of tellurite glass.

1.2 Problem Statement

Nowadays, novel efficient glass materials to be used as host are on demand in industry. Majority of research show that the tellurite glass based upon NPs embedded RE doped glass are interesting for various notable properties. There is a need to prepare tellurite glass with optimized concentration of RE and NPs having superior physical and optical properties (Hu et al., 2011). Only few studies are made on samarium doped sodium lithium tellurite glasses containing Au NPs.

In recent years, investigations are made to study the luminescence of crystal, optical fibers, and also glassy materials doped/co-doped with various REI. Among those researches, it is shown that TeO_2 based glasses capture special attention for photonic devices application due to their unique features (Selvaraju and Marimuthu, 2012). Trivalent REI doped TeO_2-based glasses presents large luminescence efficiency in various wavelengths ranges. Two major emission regions of REI in glasses need intensity and gain improvements (Som and Karmakar, 2011).However, at high concentration of RE dopants tellurite glass displays the luminescence quenching which is disadvantageous for applications and need to be minimized. Therefore, it is important to enhance their emission and absorption cross-section by embedding Au NPs and examine the role of SPR in improving them (Som and Karmakar, 2011). Modifications in optical responses and structural properties of Au NPs embedded glasses are the major issues.

Insertion of metallic nanostructures is considered as an alternative route to enhance the optical response via SPR effects. Further studies on the mechanism of SPR enhanced optical properties due to gold NPs of varying concentration in the host are still lacking. The determination of the influence of various metallic NPs and their controlled growth at small concentrations needs to be done. However, the incorporation of Au NPs in the tellurite glass and their influences on optical and structural properties are not extensively studied. Despite some effort, the mechanisms behind the variation in transitions intensities which strongly depend on the environment of the REI in presence of metal NPs are not understood. Furthermore, the possible influence by metallic NPs which leads to the existence of luminescence from $4f$–$4f$ electronic transitions remain unclear due to the lack of experimental data and theoretical explanations.

Nevertheless, the definite mechanism on how the embedded metallic NPs improves overall properties and make structural arrangements of constituent atoms in glasses is far from being understood. The enhancements in structural and optical properties in the presence of Au NPs of different contents are not reported yet. The current research will provide new information about the absorption and emission modifications of lower concentration of Sm^{3+} in presence of Au NPs at different concentration. Detail characterization, careful synthesis of tellurite glass with optimized samarium and Au doping are not explored yet. The intention is to relate the significant enhancement of optical properties and structural properties of the glass with NPs stimulated SPR effects. In view of these, we aim to study and determine the structural and optical behavior of TeO_2-LiO_2-NaO_2-Sm_2O_3-$AuCl_3$ glasses. New knowledge will be generated which will be useful for the development of glass physics.

1.3 Objectives

The objectives of this research are:

i. to synthesize a series of Au NPs embedded samarium doped sodium-lithium tellurite glasses with composition of 79 TeO_2 + 10 Li_2O + 10 Na_2O + 1 Sm_2O_3 + y $AuCl_3$ (where $0.03 \leq y \leq 0.15$ mol% in excess).

ii. to identify the presence of Au NPs in the glass matrix and determine the modifications in structural and optical properties due to the incorporation of Au NPs.

iii. to study the mechanism of enhanced absorption and luminescence assisted by Au NPs of different concentrations.

iv. to analyze the effect of localized surface plasmon resonance responsible for enhanced luminescence.

1.4 Scope of Study

In this research, the preparation of Au NPs embedded Samarium doped Sodium-Lithium tellurite glasses by using melt-quenching technique is the main focus. The main objective is to characterize the structural and optical properties of the glass series using several spectroscopic techniques; UV-Vis, PL, TEM, XRD and FTIR. The UV-Vis is used to determine the absorption spectra which are useful for Urbach energy determination and other physical properties. The UV-Vis data also provides information regarding the location of SPR band. PL analysis is done in order to study the effect of SPR while TEM can be used to determine the growth (morphology) of Au NPs with their average size. Besides, XRD analysis is very essential to help us in conformity the amorphous nature of glass. By studying FTIR spectra, the bonding in the glass can be determined and analyzed. Last but not least, the determination of glass density is done by using Archimedes method.

1.5 Significance of Study

In this research, the optical and structural interaction mediated by the presence of different constituents in glasses will be determined. Optical performances can be enhanced by optimizing the incorporation of NPs and REI in the glass system. This study will help to produce new variations in structural and optical properties stimulated by the growth of NPs. Furthermore, a clearer understanding on glass modification by inserting NPs will be achieved as it is expected to give improvement towards properties of tellurite glasses. Thus, this research is performed to create more fundamental understanding on the nano-glass and to develop nano-structured amorphous materials in parallel with the development of nanotechnology. The correlation between the SPR effects and alterations in overall properties can be established through these systematic experimental investigations.

1.6 Thesis Outline

This thesis is divided into five chapters. Chapter 1 provides the rationale of research by emphasizing why this study is important. It also highlights the problem statement, objectives, research significances, and scope of the study. Chapter 2 deals with the literature review on different experiments and theory related to the tellurite glass in general and NPs embedded tellurite glasses in particular. The detailed methodology of our research is documented in Chapter 3. Chapter 4 presents the results, discussion, and comparison with detailed sample analyses. Chapter 5 concludes the thesis by underscoring the fulfillment of stated objectives. Some suggestions for future study are also cited in this chapter.

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