UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, ELASTIC AND OPTICAL PROPERTIES OF ZINC TELLURITE GLASS SYSTEM DOPED WITH SAMARIUM, SAMARIUM NANOPARTICLES AND SILVER OXIDE

TAFIDA RABIU ABUBAKAR

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By

TAFIDA RABIU ABUBAKAR

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia in fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This work is dedicated to the Almighty God, for his love, care, protection in the course of writing this thesis and the entire Tafida's family Jalingo Taraba State Nigeria.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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Four series of zinc tellurite glasses doped with samarium, samarium nanoparticles and silver oxide were successfully fabricated using the conventional melt-quenching technique. The glasses were prepared based on the empirical formulas of $[(TeO_2)_{0.7} (ZnO)_{0.3}]_{1-x} [Sm_2O_3/ Sm_2O_3Nps]_x$, where x = 0.01 to 0.05 molar fraction and $[\{(TeO_2)_{0.7} (ZnO)_{0.3}\}_{0.99} (Sm_2O_3/ Sm_2O_3Nps)_{0.01}]_{1-y} (Ag_2O)_y$, with y = 0.005 to 0.025 molar fraction. The XRD tests confirmed that the glasses are amorphous. The Fourier Transform Infrared Spectrometer (FTIR) showed that all the glasses have a structural unit of TeO_4 and TeO_3. The TEM-images confirmed the existence of samarium nanoparticles with particle size of about 72.43 nm. The density of Sm_2O_3 and Sm_2O_3 NPs increased from 5.0419 to 5.3005 g/cm^3 and from 5.1095 to 5.3286 g/cm^3. Meanwhile, the molar volume increased from 27.4238 to 27.6901 cm^3/mol, and from 27.0610 to 27.5441 cm^3/mol. For Ag_2O(Sm) glasses the density increased from 5.1999 to 5.3747 g/cm^3 and decreased for Ag_2O (Sm NPs) from 5.3162 to 4.9163 g/cm^3.The molar volume decreased from 26.6788 to 26.1590 cm^3/mol for Ag_2O(Sm) and increased from 26.0952 to 28.5981 cm^3/mol for Ag_2O (Sm NPs) glasses. The elastic moduli and other elastic parameters increase with increase in dopant while the values of Poisson's ratio lie in the range of 0.2734 to 0.2902. The direct and indirect optical band gap increased from 2.7855 eV to 2.9867 eV and from 2.6714 eV to 3.0676 eV for Sm_2O_3 and Sm_2O_3 NPs doped series. Similarly, E_{opt} increased from 2.9691 eV to 3.1054 eV and from 2.7417 eV to 2.6278 eV for Ag_2O (Sm) and Ag_2O (Sm NPs). Photoluminescence investigation reveals that the glass samples can be used as a laser active medium for emission at 605 and 607 nm wavelength corresponding to the $4G_{5/2} \rightarrow 6H_{7/2}$ transitions. The Judd-Offelt analysis reveals that the large values of gain bandwidth (4.427×10^{-21}), optical gain (5.199×10^{-25}) and the radiative transition probabilities [(16195 S^{-1})/ (15894 S^{-1})] can be used for laser application. Furthermore, Ag_2O (Sm NPs) doped series having the largest value of gain bandwidth (9.975×10^{-21}), optical gain (9.061×10^{-25}) and the radiative transition probabilities [(119021 S^{-1})/ (16832 S^{-1})] made it the best candidate for laser materials.

Therefore, this research contributes to addressing the pressing challenge of developing new materials for laser.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SIFAT STRUKTUR ELASTIK DAN OPTIK SISTEM KACA ZINK TELURIT TERDOP DENGAN SAMARIUM, SAMARIUM NANOPARTIKEL DAN ARGENTUM OKSIDA

Oleh

TAFIDA RABIU ABUBAKAR

Januari 2021

Pengerusi : Profesor Halimah binti Mohamed Kamari, PhD
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Empat siri kaca zink telurit yang terdop dengan samarium, samarium nanopartikel dan argentum oksida telah berjaya difabrikasi menggunakan teknik pelindapkejutan lebur konvensional. Kaca tersebut telah disediakan berdasarkan formula empirikal, $[(TeO_2)_{0.7} (ZnO)_{0.3}]_{1-x} (Sm_2O_3)_x$, di mana x=0.01 hingga ke 0.05 pecahan mol dan $[\{(TeO_2)_{0.7} (ZnO)_{0.3}\}_{0.99} (Sm_2O_3/\ Sm_2O_3Nps)_{0.01}]_{1-y} (Ag_2O)_y$, dengan y = 0.005 hingga ke 0.025 pecahan mol. Ujian XRD mengesahkan bahawa kaca adalah amorfus. Spektrometer Inframerah Jelmaan Fourier (FTIR) menunjukkan bahawa semua kaca mempunyai unit struktural TeO_4 dan TeO_3. Imej TEM mengesahkan kewujudan samarium nanopartikel dengan saiz partikel lebih kurang 72.43 nm. Ketumpatan Sm_2O_3 dan Sm_2O_3 NPs meningkat daripada 5.0419 hingga ke 5.3005 g/cm^3 dan daripada 5.1095 hingga ke 5.3286 g/cm^3. Manakala, isi padu molar meningkat, masing-masing daripada 27.4238 hingga ke 27.6901 cm^3/mol, dan daripada 27.0610 kepada 27.5441 cm^3/mol. Ketumpatan Ag_2O(Sm) meningkat daripada 5.1999 hingga ke 5.3747 g/cm^3 dan menurun bagi Ag_2O (Sm NPs) daripada 5.3162 hingga ke 4.9163 g/cm^3. Isi padu molar menurun daripada 26.6788 hingga ke 26.1590 cm^3/mol bagi Ag_2O(Sm) dan meningkat daripada 26.0952 hingga ke 28.5981 cm^3/mol bagi kaca Ag_2O (Sm NPs). Modulus elastik dan parameter elastik kaca lain didapati meningkat dengan peningkatan dalam dopan manakala nilai nisbah Poisson berada dalam julat 0.2734 hingga ke 0.2902. Jurang jalur optik langsung dan tak langsung meningkat daripada 2.7855 eV hingga ke 2.9867 eV dan daripada 2.6714 eV hingga ke 3.0676 eV bagi siri terdop Sm_2O_3 dan Sm_2O_3 NPs. Begitu juga, E_{opt} meningkat daripada 2.9691 eV hingga ke 3.1054 eV dan daripada 2.7417 eV hingga ke 2.6278 eV bagi siri terdop Ag_2O (Sm) dan Ag_2O (Sm NPs). Penelitian kefotopendarcahayaan memperlihatkan bahawa sampel kaca dapat digunakan sebagai medium aktif laser bagi pelepasan pada 605 dan 607 nm jarak gelombang sepadan dengan $4G_{5/2} \rightarrow 6H_{7/2}$ transisi. Analisis Judd-Offelt memperlihatkan bahawa nilai gandaan lebar jalur yang besar (4.427×10^{-21}), gandaan optik (5.199×10^{-25}) dan

kebarangkalian transisi radiatif [(16195 S^{-1})/ (15894 S^{-1})] dapat digunakan bagi pengaplikasian laser. Di samping itu, siri terdop Ag_2O (Sm NPs) mempunyai nilai gandaan lebar jalur terbesar (9.975× 10^{-21}), gandaan optik (9.061 ×10^{-25}), dan kebarangkalian transisi radiatif [(119021 S^{-1})/ (16832 S^{-1})] menjadikannya calon terbaik untuk bahan laser. Oleh sebab itu, penyelidikan ini menyumbang kepada pengutaraan cabaran yang mendesak bagi membangunkan bahan baharu untuk laser.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF SYMBOLS AND ABBREVIATIONS

TeO_2	Tellurium oxide
ZnO	Zinc oxide
Sm_2O_3	Samarium (III) oxide
Sm_2O_3 NPs	Samarium oxide nanoparticles
Ag_2O	Silver oxide
BOs	Bridging oxygens
NBOs	Non-bridging oxygens
XRD	X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
TEM	Transmission electron microscopy
tbp	Trigonal bipyramids
tp	Trigonal pyramids
OPD	Oxygen packing density
V_o	Oxygen molar volume
ρ	Density
R_i	Inter-ionic distance
N	Ionic concentration
V_m	Molar volume
v_L	Longitudinal velocity
v_s	Shear velocity
L	Longitudinal modulus
E	Young modulus
K	Bulk modulus

G	Shear modulus
σ	Poisson's ratio
v_m	Mean ultrasonic velocity
θ_D	Debye temperature
T_s	Softening temperature
H	Microhardness
f_g	Fugacity
d	Fractal bond connectivity
Z	Acoustic impedance
V_t	Packing density
G_t	Dissociation energy
n_b	Number of bonds per unit volume
$\bar{n}_c$	Average cross-link density
l	Atomic ring size
X_c	Peak position
α	Absorption coefficient
E_{opt}	Optical band gap energy
ΔE	Urbach energy
n	Refractive index
R_m	Molar refraction
α_m	Molar electronic polarizability
α_e	Electronic Polarizability
M	Metallization criterion
ϵ	Dielectric constant

$\alpha_{O^{2-}}$	Oxide ion polarizability
Λ	Optical basicity
λ	Wavelength
$\hbar\nu$	Photon energy
Ω	Judd Offelt parameters
f_{exp}	Experimental oscillator strength
Δ_{rms}	Root mean square deviation
β_R	Branching ratio
τ	Lifetime
F	Average Stretching Force Constant
A_{MD}	Magnetic dipole radiative transition probability
A_{ED}	Electric dipole radiative transition probability
$\Delta\lambda eff \times \sigma p$	Gain band width
$\sigma p \times \tau$	Optical gain

CHAPTER 1

INTRODUCTION

The chapter reveals the background of the research which consists of the simple history of glass materials, definition of glass materials and the chemical components that made up the glass. The important element of this study such as problem statement, scope of the study, research objective, hypothesis as well as outline of thesis are also included in this chapter respectively.

1.1 History of glass

Glass materials play an important role in the life of every human being. Many things in the world today are originated or manufactured from different types of glass materials such as the normal window glasses in our homes, spectacles, electric bulbs and communication cables. The existence of glass material in our daily life is so common, especially the use of spectacles among our general populace has increased drastically that at a certain level, some individuals will hardly read or write without the use of glasses. Roman empire is the regular old period of glass making history, whereby people were able to make glasses of different colors at that time not forgetting the popular technique of glass blowing. The method of making colored glass, for example, involves incorporation of a golden component into the glass, is considered to be the family secret by which glassmakers are transferred from generation to generation (Shelby, 2005).

1.1.1 Definition of glass

Glass materials are defined as a solid which is found through the rapid cooling of liquid and takes the nature of amorphous which are revealed by the X-ray diffraction analysis (XRD) (Rao, 2002). In the meantime, Doremus (1973) further said glass is a substance that is formed by cooling normal liquid matter and that it becomes either more or less rigid as its viscosity increases. The most commonly used techniques to synthesized glassy materials remain the conventional melt quenching technique where the molten liquid will be cooled rapidly to have the required glassy state. Furthermore, the terms amorphous and glassy can be used to describe the glass material, where glass is typically arranged regularly in a short-range order.

1.1.2 Chemical compounds in zinc tellurite glass doped with samarium oxide, samarium nanoparticles and silver oxide

Tellurium oxide-based glasses (TeO_2) are of scientific and technological concern on the justification of their exceptional properties such as high refractive indices, low melting point, high dielectric constant, slow crystallization rate and good infrared transmission

(Hasnimulyati et al., 2016). As reported in many works of literature, pure TeO_2 alone cannot form glassy material under normal condition and therefore it requires the addition of various oxides to enhance its glass-forming ability (Manikandan et al., 2012). When tellurite is combined with zinc, zinc tellurite glasses are formed which are studied intensively for their everyday applications and enhancement of glass properties. Hence the presence of zinc oxide (ZnO) in the glass network produces the following: Low rates of crystallization, decreases the melting point because it serve as a glass modifier and former at the same time, increases the glass-forming ability, decreases the optical energy band gap and increase the refractive index of the glass system (Hajer et al., 2014). Zinc oxide is used in the glass network in the form of either glass modifier, glass former or both of them. Currently, inorganic glasses are doped with rare-earth ions to enhance the physical and optical properties of the glass host because of their exceptional spectroscopic properties arising from their optical transitions in the 4f shells (Nandi et al., 2009).

Among the rare earth ions (RE), samarium ion (Sm^{3+}) received much attention from lots of researchers as a result of its spectroscopic applications (Tanko et al., 2016a). Samarium oxide is one of the rare earth families that are used as a dopant to create the lasing character of tellurium oxide-based glasses TeO_2 (Eraiah, 2014). Also, the certain composition of Sm_2O_3 has been suggested for use in lasers and photonics devices (Ravi et al., 2012). Generally, it is expected that glasses doped with Sm^{3+} ions are classified as a possible candidate for lasers and photonics application due to their spectroscopic properties (Selvaraju and Marimuthu, 2013). The addition of silver oxide (Ag_2O) in the glass network plays a vital role of a modifier by enhancing most of the glass properties because silver oxide acts as a suitable and convenient element in the preparation of glassy materials (Nazrin et al., 2019). Silver oxide is among the oxides that enhance and improve the optical properties of glassy materials. Silver oxide can modify the zinc tellurite glass network by transforming the structural units of the tellurite network from (TeO_4) structural units to (TeO_3) structural units respectively (Nazrin et al., 2019). In the present study, undoped zinc tellurite glass system is not considered because it is regarded as immaterial when it comes to the glass applications since the laser application of the synthesized glass system is as a result of the doping with rare earth samarium oxide (Sm_2O_3).

1.2 Problem statement

The search for improving the applications of optical properties of glass materials in several fields and the enhancement of the materials cannot be overemphasized. This is considered as a non-stop challenge for researchers around the world. A lot of researches is conducted on the glass of various compositions to obtain the most suitable glasses for various applications. Tellurite glasses possess unique and exceptional properties which draw the attention of most researchers around the world. Some of these properties include good thermal stability, exceptional chemical durability, high refractive indices and low melting point (Yousef, 2013). Tellurium oxide-based glasses have the potentials to be applied as a new laser host and hence are now under consideration in various applications. Recently, there are few data to support the advancement of optical applications of rare earth oxide and rare earth nanoparticles based glasses. Additionally, investigation has shown that there is still lack of research that presented on samarium, samarium nanoparticles incoporated silver oxide doped with zinc tellurite glass system. The nanoparticles are known as the promising materials to improve the optical properties of tellurite-based glass. Therefore, the investigations on samarium, samarium nanoparticles, and silver oxide doped zinc tellurite-based glass system are still needed since there are limited data to support their future optical applications. Hence, this study was conducted in order to investigate the structural, elastic and optical properties of the glass system. Furthermore, the influences of samarium, samarium nanoparticles and silver oxide addition to the properties of the prepared glasses were also studied as well as its possible application as a laser-active medium.

1.3 Scope and limitations of the study

The objectives of this study are achieved by the following scope of the study stated below;

1. The scope of the present work is restricted to physical and structural properties which include molar volume, density, Transmission Electron Microscopy (TEM), X-ray diffraction analysis (XRD) and Fourier Transform infrared spectroscopy (FTIR).

2. linear optical properties which include: The optical absorption spectra, bandgap energy, Urbach energy, index of refraction. Elastic properties which include the longitudinal and shear ultrasonic velocity, elastic moduli, Poisson's ratio, microhardness, Softening and Debye temperature.

3. The four theoretical models of Makishima and Mackenzie, Rocherulle model, Bond compression model and Ring deformation model are studied and compared with the experimental results.

4. The Judd-Offelt parameters of the synthesized glasses are analysed and used to calculate the radiative parameters of the studied glass samples.

1.4 Objective of the study

The study was conducted based on four clear and precise objectives which include the following:

1. To investigate the effect of samarium, samarium nanoparticles and silver oxide on the physical and structural properties of zinc tellurite glass system.

2. To study the influence of samarium, samarium nanoparticles and silver oxide on the experimental elastic and linear optical properties of the synthesized glass samples.

3. To verify the area of validity of the four theoretical elastic models on the zinc tellurite glass system and use it to compare with the experimental values.

4. To study the Judd-Offelt parameters of the synthesized glasses and use it to calculate the radiative parameters of the studied glass samples.

1.5 Hypothesis

The following hypothesis is anticipated based on the research objectives:

1. The addition of Sm_2O_3, Sm_2O_3 NPs and Ag_2O into the zinc tellurite glass system will bring about changes mostly in the physical and structural properties of the synthesized glasses by producing glasses with amorphous nature that can be verified through XRD and the breaking of glass structure that form TeO4 structural unit which can be detected using FTIR. The samarium nanoparticles are expected to be present in the glass system following the glass forming process in the TEM photo. The density values can be increased by incorporating heavier dopant in the glass matrix.

2. It is projected that the linear optical and elastic properties of zinc tellurite glass doped with samarium oxide, samarium nanoparticles and silver oxide will experience a lot of modifications. This will happen for elastic properties as a result of the increase in the strength and rigidity of the studied glasses. This is anticipated because rare-earth ions encourage the formation of bridging oxygen's (BOs) and hence the rigidity of the glass samples increases as well. For the optical properties of the glass system, it is expected that the bandgap energy will decrease while the refractive index, molar refraction and electronic polarisabilities are expected to increase with an increase in dopants due to the 4f transitions of rare-earth ions in the glass network.

3. The theoretical and experimental elastic moduli results are expected to be very close to one another in ranges if the theoretical elastic models remain valid with the multi-component glass system.

4. It is anticipated that the analysis of Jud-Offelt parameters in the four glass series will explore the possible application of the synthesized glasses. This is expected based on the following parameters: spectroscopic quality factor, radiative transition probabilities, branching ratio, gain bandwidth and the optical gain of the studied glasses.

1.6 Thesis outline

The arrangement of this thesis is outlined in the following approach:

Chapter 1 consist of the history of glass, a brief definition of glass material by different authors, the chemical constituent selected and used in synthesizing the glass samples. The chapter also consists of the following: scope of the study, the problem statement, objective of the study, the hypothesis and the likely result of the research work.

Chapter 2 of the present research work provides information in respect of the previous research that is connected to the study. The review of related literature comprises of brief information about the zinc tellurite doped glasses, glasses doped with rare earth oxide, silver oxide doped zinc tellurite glasses, physical and structural properties, elastic properties and linear optical properties in the four-glass series under study.

Chapter 3 discuss the existing theory as well as models and equations that are used in the research work. The derivation of various equations is also presented in the chapter.

Chapter 4 provides procedures and method used in synthesizing the sample glasses using conventional melt quenching technique. The chapter went further to highlights some basics of characterization techniques employed in the study.

Chapter 5 of this thesis analysed and discussed the trends and various results for the four-glass series which contain the structural and physical analysis of the four-glass series, the linear optical properties of the four glass samples, experimental and theoretical elastic properties and the Judd-Offelt theory for the four-glass series under study.

Chapter 6 provides a summary concerning the important findings of the research work as well as recommendations for future studies.

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