EFFECTS OF SILVER NANOPARTICLES DOPING ON PHYSICAL, STRUCTURAL, OPTICAL AND ELASTIC PROPERTIES OF SAMARIUM MAGNESIUM BOROTELLURITE GLASS

By

SITI NORUL FADHILAH BINTI ZALAMIN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

July 2022

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Dedicated to

To my beloved parents, Mr Zalamin Bin Noor and Mrs Samsiah Binti Yaacob
For their unconditional love and endless support

To my siblings and family
For all the love and care

To all my very wonderful friends
For making my life full of joy and happiness

To all my lecturers
For helping me a lot throughout this journey

Thank you all

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Chair : Mohd Hafiz Bin Mohd Zaid, PhD
Faculty : Science

Low energy absorption rates cause luminescence instability in magnesium borotellurite glass. Hence, a research was carried out to report the physical, structural, optical and elastic properties of new tellurite glass system with nominal composition of $(60\text{-}x)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}\ x(Sm_2O_3)$, where $0.0 \leq x \geq 3.0$ mol% and $(58\text{-}y)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}2Sm_2O_3\text{-}\ y$Ag NPs, where $0.0 \leq y \geq 2.0$ mol% synthesized via fast-cooling melt quenching method. To achieve the goals, the glasses are analyzed using densitometer, X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), UV-Visible Spectroscopy (UV-Vis), Photoluminescence (PL), CIE Chromaticity analysis, ultrasonic testing, and Makishima-Mackenzie modelling. The density of Sm^{3+} doped Magnesium Borotellurite (MBT) glass increased from 4.105 g/cm^3 to 4.403 g/cm^3 with increment of Sm^{3+} content meanwhile for Ag co-doped samarium MBT glass, the density is decreased from 4.162 to 4.310 g/cm^3 with incorporation of Ag NPs. No sharp peak appeared in the XRD region proved the amorphous state in both series of glasses. In TEM, the diameter size of Ag NPs is increasing from 40.94 nm to 367.70 nm with the rise of Ag NPs content from 0.1 mol% to 2.0 mol% in glass matrix. The optical absorption spectra were characterized to determine the energy band gap, Urbach energy, and refractive index of the glasses. The optical absorption spectra revealed eleven energy transitions bands centered at $^4G_{9/2}$, $^4I_{11/2}$, $^4F_{3/2}$, $^4G_{5/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^6H_{15/2}$, $^6F_{1/2}$ from ground state of $^6H_{5/2}$. The optical energy band gap of the Sm^{3+} doped MBT glass system is manifested to be in the range of 2.560 eV to 2.820 eV for indirect transitions and 2.830 eV to 2.958 eV for direct transitions. The refractive index lied in the range between 2.447 to 2.526 while the Urbach energy is measured to be in range of 0.299 to 0.345 eV. For Ag co-doped samarium MBT glass, the indirect transitions have an optical band gap of 2.618 to 2.800 eV, while direct transitions have an optical band gap of 2.839 to 2.897 eV. The refractive index is ranging between 2.453-2.508 while the Urbach energy, *ΔE,* is in the range of

0.338-0.391 eV. Four strong emission peaks occurred in photoluminescence for both glass series, with the highest red emission peak at 600 nm. Colour chromaticity analysis for both series presented colour coordinates lied down in the red-orange region. In elastic properties, the longitudinal, shear, Young's and Bulk modulus are increased from 60.57-73.24 GPa, 20.39-24.93 GPa, 50.83-61.92 GPa and 33.37-40.00 GPa, respectively in Sm^{3+} doped MBT glass system. Ag co-doped Samarium MBT glass showed a fluctuated trend in which the longitudinal, shear, Young's and Bulk modulus lied in the range 59.17-70.84 GPa, 20.70-24.21 GPa, 50.97-60.02 GPa and 31.57-41.77 GPa, respectively. Elastic properties for Makishima Mackenzie model of Sm doped MBT glass is increased from 44.58-48.55 GPa (Bulk moduli), 70.15-72.73GPa (Young's moduli) and 28.34-29.08 GPa (Shear moduli). Makishima Mackenzie model for Ag co-doped samarium MBT glass analyzed a decreasing trend of elastic moduli in which Bulk moduli (28.83-27.29 GPa), Young's moduli (47.21-42.37 GPa), Shear modulus (28.83-27.29 GPa).

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sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN-KESAN DOP ZARAH NANO PERAK KEPADA SIFAT-SIFAT FIZIKAL, STRUKTUR, OPTIK DAN ELASTIK KACA SAMARIUM MAGNESIUM BOROTELLURIT

Oleh

SITI NORUL FADHILAH BINTI ZALAMIN

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Kadar penyerapan tenaga yang rendah menyebabkan ketidakstabilan pendarkilau dalam kaca borotellurit magnesium. Oleh itu, sebuah penyelidikan dijalankan untuk menghuraikan sifat-sifat fizikal, struktur, optik dan kekenyalan sistem kaca tellurit baharu dengan komposisi nominal $(60\text{-}x)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}\ x(Sm_2O_3)$, di mana $0.0 \leq x \geq 3.0$ mol% dan $(58\text{-}y)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}2Sm_2O_3\text{-}\ y$Ag NPs di mana $0.0 \leq y \geq 2.0$ mol% disintesis melalui kaedah lindapan leburan. Untuk mencapai matlamat ini, kaca dianalisis menggunakan densitometer, Belauan Sinar X (XRD), Mikroskopi Elektron Penghantaran (TEM), Spektroskopi UV-Visible (UV-Vis), Fotopendarcahayaan (PL), analisis Kekromaan CIE, ujian ultrasonik dan model Makishima-Mackenzie. Ketumpatan kaca Magnesium Borotelurit terdop Sm^{3+} (MBT) meningkat dari 4.105 g/cm^3 kepada 4.403 g/cm^3 dengan pertambahan kandungan Sm^{3+} manakala untuk kaca samarium MBT terdop bersama Ag, ketumpatan berkurangan daripada 4.162 kepada 4.310 g/cm^3 dengan pencampuran Ag NPs. Tiada puncak tirus yang muncul di kawasan XRD membuktikan keadaan amorf dalam kedua-dua siri kaca. Dalam TEM, saiz diameter Ag NPs meningkat daripada 40.94 nm kepada 367.70 nm dengan peningkatan kandungan Ag NPs daripada 0.1 mol% kepada 2.0 mol% dalam matriks kaca. Spektrum penyerapan optik telah di ukur untuk menentukan jurang jalur tenaga, tenaga Urbach, dan indeks biasan kaca. Spektrum penyerapan optik mendedahkan sebelas jalur peralihan tenaga berpusat pada $^4G_{9/2}$, $^4I_{11/2}$, $^4F_{3/2}$, $^4G_{5/2}$, $^6F_{11/2}$, $^6F_{9/2}$, $^6F_{7/2}$, $^6F_{5/2}$, $^6F_{3/2}$, $^6H_{15/2}$, $^6F_{1/2}$ daripada asas bawah $^6H_{5/2}$. Jurang jalur tenaga optik sistem kaca MBT terdop Sm^{3+} ditunjukkan dalam julat 2.560 eV hingga 2.820 eV untuk peralihan tidak langsung dan 2.830 eV hingga 2.958 eV untuk peralihan terus. Indeks biasan terletak dalam julat antara 2.447 hingga 2.526 manakala tenaga Urbach diukur berada dalam julat 0.299 hingga 0.345 eV. Untuk kaca samarium MBT terdop bersama Ag, jurang jalur optik peralihan tidak langsung ialah 2.618 hingga 2.800 eV, manakala peralihan terus mempunyai jurang jalur optik 2.839 hingga 2.897

eV. Indeks biasan adalah antara 2.453 – 2.508 manakala tenaga Urbach, ΔE, berada dalam julat 0.338-0.391 eV. Empat puncak sinaran keluar yang kuat berlaku dalam fotopendarcahayaan untuk kedua-dua siri kaca, dengan puncak pelepasan merah tertinggi pada 600 nm. Analisis kekromatan warna untuk kedua-dua siri menunjukkan koordinat warna terletak di kawasan merah-oren. Dalam sifat kekenyalan, modulus membujur, ricih, Young dan Bulk meningkat masing-masing daripada 60.57-73.24 GPa, 20.39-24.93 GPa, 50.83-61.92 GPa and 33.37-40.00 GPa dalam system kaca MBT terdop Sm^{3+}. Kaca Samarium MBT yang didop bersama Ag menunjukkan aliran turun naik di mana modulus membujur, ricih, Young dan Bulk masing-masing terletak dalam julat 59.17-70.84 GPa, 20.70-24.21 GPa, 50.97-60.02 GPa and 31.57-41.77 GPa. Sifat kekenyalan untuk model Makishima Mackenzie bagi kaca MBT terdop Sm meningkat daripada 44.58-48.55 GPa (modul Bulk), 70.15-72.73GPa (modul Young) dan 28.34-29.08 GPa (modul ricih). Model Makishima Mackenzie menganalisa satu aliran penurunan untuk modul-modul kekenyalan kaca samarium MBT terdop Ag di mana modul Bulk (28.83-27.29 GPa), modul Young (47.21-42.37 GPa), modul ricih (28.83-27.29 GPa).

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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 Master of Science. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

3 METHODOLOGY

4 RESULTS AND DISCUSSION

5 SUMMARY, CONCLUSION AND RECOMMENDATIONS FOR FUTURE RESEARCH

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Sm^{3+}	Samarium
Er^{3+}	Erbium
Ag	Silver
Te	Tellurium
Zn	Zinc
Mg	Magnesium
Ca	Calsium
Pb	Lead
Eu^{3+}	Europium
Dy^{3+}	Dysprosium
La^{3+}	Lanthanum
Au	Gold
Mn^{3+}	Manganese
Tm^{3+}	Thulium
NP	Nanoparticle
ZnO	Zinc Oxide
Ag_2O	Silver Oxide
PbO	Lead (II) Oxide
TeO_2	Tellurium Oxide
B_2O_3	Boron Oxide
Sm_2O_3	Samarium Oxide
Al_2O_3	Aluminium Oxide
σ	Poisson's ratio
H	Microhardness

ΔE	Urbach energy
E_{opt}	Optical Band gap
n	Refractive index
V_L	Longitudinal velocity
V_S	Shear Velocity
L	Longitudinal modulus
G	Shear modulus
E	Young's modulus
K	Bulk modulus
V_t	Packing density
G_t	Dissociation energy
ρ	Density
V_m	Molar Volume
XRD	X-Ray Diffraction
TEM	Transmission Electron Microscopy
UV Vis	Ultraviolet-Visible
PL	Photoluminescence
CIE	Commission Internationale de l'Elcairage

CHAPTER 1

INTRODUCTION

1.1 Research background

The glass industry has been one of the most economic industries nowadays. Glass is a common material that people encounter and use on a daily basis. The most frequent ancient applications of glass materials are drinking vessels, medical implants, windows, spectacles, and storage containers (Musgraves et al., 2019). Glasses have been known for their good properties and wide potential applications. Even so, many more extensive studies and detailed information on the various properties of glass materials have been done. Glass is a very transparent material and is amorphous (Brinker & Scherer, 2013).

Researchers have focussed on the glass industry owing to the overwhelming need for glass applications such as optics, photonics and solid-state laser (Qi et al., 2014; Halimah et al., 2019; Hossain et al., 2021). This intrigued people to figure out more about physical, structural, optical, elastic, or even thermal properties (Mondal et al., 2020; Shen et al., 2021). The contributions of glass are not limited to personal use only but can serve as worldwide application such as medical devices, construction materials, radiation protection materials, and telecommunication systems. Glass with ability to adapt with very extreme conditions and corrosive liquid would be a great advantage to form a superior glass (Halimah, 2005).

The types of glass can be fabricated through the variation of chemical composition and glass melting procedures. Glass can be classified into borate, silicate, phosphate, germinate, and tellurite. As compared to other glasses, tellurite glass is promising to have a captivating ability in physical and optical properties (Azlan et al., 2019). Tellurite-based glass is a new-fangled glass system and has been notorious among the glass industry. Borotellurite glass system is an extensive study apart from the tellurite glass. Borotellurite glass is avowed for its ability to be a saturable absorber, fibre glass, amplifiers, photonic and optoelectronic applications (Halimah, et al., 2010). Some would explain that borotellurite glass has the more extraordinary ability in lasing, sensors telecommunication, and display devices (Mahraz et al., 2013).

To date, many have recognized borotellurite glass for its potential and ability in the glass industry. In virtue of outstanding properties combination such as low photon energy, high refractive index, low acoustic losses by tellurium oxides (Leena, 2013) and high melting point, low preparation cost by borate oxides (Hazlin et al., 2017), a brighter discovery of future glass has been governed.

Plus, the combination of these two elements contributed to a non-hygroscopic material as the final product which helps more in manufacturing the glass. The linkage network of the borotellurite glass is more stable due to the number of atoms increased, and the bridging oxygen (BO) bond created (Hashim et al., 2013).

Previous research showed a tremendous improvement of glass when doping with transition metals, alkaline earth metal, and alkali metal (Salman et al, 2007; Babu et al, 2009; Aziz et al, 2018). These network modifiers played their parts respectively in the glass network. Some usually used zinc oxide (ZnO) and magnesium oxide (MgO). Both MgO and ZnO can act as stabilizers and lower the rate of crystallization, which helps much in the glass industry (Ahmad et al., 2014). Besides, lead oxide (PbO) insertion is also reported to provide a good accomplishment by enhancing thermal stability (Iskandar et al., 2012).

Nowadays, attention has been focused on rare earth ions (REI) doping in glass networks to enhance glass performance. REI characterizations are intensively experimented due to their benefits in manufacturing glasses (Azlan et al., 2016, Azlan et al., 2017; Halimah, et al., 2017). An excellent result was established by a previous study on REI doping in glass systems, such as increased stiffness by creating more NBO (Aziz et al., 2017a). Trivalent samarium ion, Sm^{3+}, as dopants in the glass systems assisted in generating intense emission, hence able to produce luminescence materials. Besides, the ability of Sm^{3+} to develop a high refractive index tends to fabricate an optical switching device (Mondal et al., 2020).

On top of that, introducing metallic nanoparticles has also become a trend today. Nanoparticles have had a big impact on the performance of the glasses. Metallic nanoparticles such as silver and gold are the most being used. This can help improve the bonding and structural characteristic of glass hosts (Yusoff & Sahar, 2015a). The addition of silver oxide (Ag_2O) can intensify the rigidity of glass samples by reconstructing the atom arrangement in glass matrix (Halimah, 2010). Besides, luminescence properties of glass are enhanced with incorporation of Ag NPs. This is attributed by strong local electric field around NPs which led radiative transition probability of energy level of REI to increment (Meng et al, 2020; Yun et al., 2021).

In this study, two series of glass with composition of $(60\text{-}x)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}x(Sm_2O_3)$, where $0.0 \leq x \geq 3.0$ mol% and $(58\text{-}y)TeO_2\text{-}30B_2O_3\text{-}10MgO\text{-}2Sm_2O_3\text{-}y\mathrm{Ag}$, where $0.0 \leq y \geq 2.0$ mol% are prepared by using melt quenching method. The precursor of the glass encountered weighing, mixing, grinding, melting, and annealing process to fabricate a glass structure. After that, the glass samples underwent few characterizations such as density measurement, molar volume, X-ray diffraction (XRD), Transmission Electron Microscopy (TEM), UV-Visible spectroscopy (UV-Vis), Photoluminescence (PL) spectroscopy, colour chromaticity, and ultrasonic testing in corresponding to probe the physical, structural, optical and elastic properties of the glasses.

1.2 Problem statement

Over the years, it can be seen the glass industry has developed tremendously. Magnesium borotellurite glass diverted the attention of researchers to dig into the topics deeper. Glass doped with transition metal and REI are intrigued many researchers to have a close-up on their improvements as well (Babu et al., 2009; Aziz et al., 2018). Currently, researchers are focusing on developing an exemplary process for fabricating glass as well as improving its performance. Another alternative, such as the Sol-gel process, would be far more expensive and challenging to implement. As a result, to overcome this issue, magnesium borotellurite glass was prepared using the melt quenching method, which is very practical, effortless to do, and saves time.

Tellurium oxide is believed to be unable to form glass by itself. Incorporating heavy transition metal oxide and alkaline earth into glass matrices is a current trend due to the benefits far outweigh its drawbacks (Rajendran et al., 2003). Magnesium oxide (MgO) and boron trioxide, B_2O_3 can serve as network modifiers due to its ability to lower the rate of crystallization and enhance the stability of glass (Salman et al., 2007). Nonetheless, luminescence instability induced by low energy absorption rates has hindered the utilisation of magnesium borotellurite (MBT) glass. Hence, insertion of rare earth ions (REI) into the host glasses would be effective way to overcome this problem.

Rare earth ion (REI)-doped glasses have developed as active media for a wide range of photonic applications, including solid-state lasers, optical fibers, waveguides, and optical amplifiers (Jackson, 2003; Biju et al., 2004). Trivalent REI-doped glasses have gained interest in fabricating visible and infrared optical devices due to their efficient luminescence properties (Dantas et al., 2002). Initially, Sm^{3+} ions were added to glass to produce a prominent orange-red colour or unique optical properties to manufacture lasers for specific applications (Lim et al., 2013). Due to its lowest emission level, $^4G_{5/2}$ has a better quantum efficiency and distinct quenching pathways, Sm^{3+} ion is considered suitable to probe energy transfer processes (Carnall et al., 1968).

Silver NPs embedded in zinc-tellurite glass exhibit surface enhanced Raman scattering and Plasmon enhanced Er^{3+} fluorescence. However, less exposure of nanoparticles as doping agent to enhance glass system is detected. The large local electric field generated by the silver NPs was found to be responsible for a significant increase in Raman and photoluminescence intensities (Amjad et al., 2013; Meng et al., 2020). Moreover, elastic properties of Makishima Mackenzie model for both samarium and silver nanoparticles doped magnesium borotellurite glass is less reported.

For all that reasons, a comprehensive study of the samarium REI doped magnesium borotellurite glass incorporating silver nanoparticles has been investigated to measure all the potentials.

1.3 Research objective

The main objective of this study is to synthesis magnesium borotellurite glass doping with Sm^{3+} and co-doped with silver (Ag) nanoparticles. This study includes the election of compositions, chemicals used, melt-quenching process, annealing, and doping process to fabricate a suitable glass. This study was carried out to achieve few objectives as in the following:

1. To study the influence of samarium ions doping on the physical, structural, optical, and elastic properties of the magnesium borotellurite glasses.
2. To study the effect of silver nanoparticles as co-doping on the physical, structural, optical, and elastic properties of the samarium magnesium borotellurite glasses.
3. To evaluate the elastic properties of samarium co-doped silver nanoparticles magnesium borotellurite glass using Makishima Mackenzie model.

1.4 Scope of the study

To achieve the objectives of the study, the scopes of the study are as follows:

1. Two series of glass with nominal composition of $(60-x)TeO_2-30B_2O_3-10MgO-x(Sm_2O_3)$, where $0.0 \leq x \geq 3.0$ mol% and $(58-y)TeO_2-30B_2O_3-10MgO-2(Sm_2O_3)-yAg$, where $0.0 \leq y \geq 2.0$ mol% has been prepared using melt quenching method.
2. The physical, structural, optical and elastic properties of samarium doped magnesium borotellurite glass incorporated with silver nanoparticles have been analyzed using densitometer, X-Ray Diffraction, Transmission Electron Microscopy, UV-Visible spectroscopy, Photoluminescence spectroscopy, Colour chromaticity analysis and Ultrasonic testing.
3. The experimental data of the elastic moduli for investigating glasses were compared with theoretical values from Makishima-Mackenzie Model.
4. TEM analysis was carried out to determine the occurrence and size of nanoparticles in glass system.

1.5 Significant of the study

Compared to silicate, borate, and phosphate glass, tellurite glass is dominated in high electronic polarizability denoted by strong lone pair in the valence shell and high Te^{4+} ions polarizability (Ersundu & Ersundu, 2016). Moreover, high transparency and high refractive index would make tellurite the best candidate for the host glass (Pereira et al., 2016). The incorporation of alkaline earth or heavy metal oxide in tellurite glass has greatly promises a brighter future for glass. It is believed that magnesium oxide can lessen the crystallization rate and raise the stiffness of the glass (Salman et al., 2007).

REI doped tellurite glass are getting high interest from many aspects due to comprehensive coverage in applications. REI doped materials could be industrialized in infrared optical devices, visible optical devices, and telecommunication systems (Devi et al., 2016). Silver (Ag) nanoparticles' insertion into glass matrix could enhance the luminescence properties of glass by its strong local field (Xu et al., 2004; Yun et al., 2021).

To the best of our knowledge, the reports on physical, structural, optical and elastic properties of samarium doped magnesium borotellurite glass are significantly less. In the present research, the preparation of samarium doped magnesium borotellurite glass incorporated with silver nanoparticles by conventional melt-quenching is reported. Subsequently, the incorporation of trivalent Sm^{3+} and silver nanoparticles (Ag) is done to increase the improvement and quality of the final products.

1.6 Outline of thesis

The thesis arrangement is organized as follows. Chapter 1 is about the introduction of borotellurite glass, the doping agent, problem statements, and significance of study, the scope and objectives of the projects. Chapter 2 gives out the literature review about previous studies by other researches on the related topics. All the apparatus, chemicals used, methodology and characterization of the borotellurite glass are discussed in Chapter 3. The results and justification of the samarium doped magnesium borotellurite glass incorporating metal nanoparticles are explained in Chapter 4. Finally, the conclusion and recommendations are suggested for future in Chapter 5.

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