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ZINC BOROTELLURITE GLASS DOPED WITH LANTHANUM NANOPARTICLES, LANTHANUM AND SILVER OXIDES AS SATURABLE ABSORBER FOR FIBER LASER APPLICATIONS

FAZNNY BINTI MOHD FUDZI

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By

FAZNNY BINTI MOHD FUDZI

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

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Chairman : Professor Halimah Mohamed Kamari, PhD
Faculty : Science

Improving and enhancing the properties of glasses for the sake of the application of the glass materials in various field are ongoing challenges in materials science and technology. Even though there are research being done on lanthanide elements doped borotellurite glass, but no researchers have tried to incorporate both lanthanum and silver in a glass system to study the changes that both of the element bring about to a glass material. In order to unveil the effect of lanthanum nanoparticles (La NPs), lanthanum oxide (La_2O_3) and silver oxide (Ag_2O) on structural, physical, linear as well as nonlinear optical properties of zinc borotellurite glass system, four series of zinc borotellurite glass doped with various concentration of La_2O_3, La NPs and Ag_2O were prepared by using melt quenching technique. Glass samples in the first and second series were doped with La_2O_3 and La NPs respectively. The compositions for both first and second glass series were $\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.70}(ZnO)_{0.30}\}_{1-x}(La)_x$ where x = 0.00, 0.01, 0.02, 0.03, 0.04 and 0.05 molar fraction. The third and fourth series were fabricated by introducing Ag_2O into the best glass sample from the first and second glass series. Thus, the chemical compositions for both the third and fourth series were $[\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.70}(ZnO)_{0.30}\}_{0.96}(La)_{0.04}]_{1-y}(Ag_2O)_y$ where y = 0.00, 0.02, 0.04, 0.06, 0.08 and 0.10 molar fraction. The structural, physical and linear optical properties of the fabricated glass samples were investigated by using X-Ray Diffraction (XRD) spectrometer, Fourier Transformation Infra-Red (FTIR) spectrometer, Transmission Electron Microscopy (TEM), densimeter and UV-Visible light (UV-Vis) spectrophotometer. The nonlinear optical properties of the glass samples were revealed through Z-scan technique while the ability for the prepared samples to become saturable absorber (SA) was tested in a fiber laser setup. The glassy state and amorphous nature of the prepared glasses had been proven via the presence of a broad hump in the XRD pattern.TeO_4, TeO_3, BO_3 and BO_4 structural units were detected in all samples as shown in the FTIR spectra. The existence of La NPs in the second and fourth glass series was verified through TEM with recorded particle size of 30.30 nm and 54.26 nm. The increasing trend of density for all glass samples were obtained because of the incorporation of silver and lanthanum with larger molecular mass in the zinc borotellurite

glass system. For glass series without the addition of Ag_2O, the indirect optical band gap was found to have increasing trend while the refractive index values decreases in the range of 2.20 to 3.90 eV and 2.19 to 2.50 respectively. On the other hand, the introduction of Ag_2O into the zinc borotellurite glass system doped with La_2O_3 and La NPs had improved the overall optical properties of the glass whereby the optical band gap decreases and refractive index increases drastically within the range of 1.94 to 4.16 eV and 2.14 to 2.76 respectively. Electronic polarizability that is affected by the overall polarizability of the glass system seems to reduce for glass series without the addition of Ag_2O while it increases for La_2O_3, La NPs and Ag_2O doped zinc borotellurite glasses. The reason behind the decrement in electronic polarizability is the formation of low polarizability bridging oxygen in La_2O_3 and La NPs doped zinc borotellurite glass. On the other hand, Ag_2O induced the creation of high polarizability nonbridging oxygen that contributes to the increasing electronic polarizability values. Zinc borotellurite glass doped with La_2O_3, La NPs and Ag_2O recorded nonlinear trend on nonlinear refractive index, nonlinear absorption coefficient, third order susceptibility and figure of merit. The fitted open aperture Z-scan plot had recorded both saturable absorber (SA) and reverse saturable absorber (RSA) trends implying the potential of the glass samples to be utilized as SA and nonlinear optical devices. The ability of the glass samples to become SA in erbium doped fiber laser (EDFL) were unveiled for the first time whereby the fabricated samples is able to initiate passively mode-locked and Q-switched pulse laser operation. Dual wavelength mode-locked pulsed laser with high pulse repetition rate of 5 GHz is recorded by utilizing zinc borotellurite glass as SA in the EDFL cavity. In contrast, the utilization of zinc borotellurite glass doped with 0.04 molar fraction of La NPs and 0.10 molar fraction of Ag_2O as SA in the same laser cavity, a stable Q-switching laser pulse with high pulse energy of 0.4 µJ is generated. The results from the pulsed laser analysis suggested that zinc borotellurite glass as well as La NPs and Ag_2O doped zinc borotellurite glass can be utilized as SA to generate laser pulse with high pulse repetition rate and high pulse energy respectively. This research is able to provide new knowledge on the effect of doping La_2O_3, La NPs and Ag_2O on various properties of zinc borotellurite glass system, the actual relation between result from Z-scan measurement and the ability of a glass material to become SA component as well as unveiling of the ability of glass material to be utilized as SA component in EDFL system.

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

KACA ZINK BOROTELLURIT TERDOP DENGAN NANOZARAH LANTANUM, LANTANUM DAN ARGENTUM OKSIDA SEBAGAI PENYERAP BOLEH TEPU BAGI APLIKASI SERABUT LASER

Oleh

FAZNNY BINTI MOHD FUDZI

Ogos 2018

Pengerusi : Profesor Halimah Mohamed Kamari, PhD
Fakulti : Sains

Pembaikan dan peningkatan sifat kaca demi kegunaan bahan kaca dalam pelbagai bidang adalah cabaran yang berterusan dalam bidang sains bahan dan teknologi. Walaupun sudah terdapat penyelidikan yang dilakukan terhadap kaca borotellurit yang telah didopkan dengan unsur lanthanid, tetapi tidak ada penyelidik yang pernah cuba mengabungkan kedua-dua lanthanum dan argentum oksida dalam satu sistem kaca yang sama bagi mengkaji perubahan yang dibawa oleh kedua-dua elemen tersebut terhadap sesuatu bahan kaca. Bagi memahami kesan nanozarah lantanum (La NPs), lanthanum oksida (La_2O_3) dan argentum oksida (Ag_2O) pada sifat struktur, fizikal, optik linear serta optik tidak linear kaca zink borotellurit, empat siri kaca zink borotellurit yang telah didopkan dengan pelbagai kepekatan La_2O_3, La NPs dan Ag_2O telah disediakan dengan menggunakan teknik lebur lindap. Sampel kaca dalam siri pertama dan kedua masing-masing telah didopkan dengan La_2O_3 dan La NPs. Komposisi bagi kaca siri pertama dan kedua ialah $\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.70}(ZnO)_{0.30}\}_{1-x}(La)_x$ di mana x = 0.00, 0.01, 0.02, 0.03, 0.04 dan 0.05 pecahan molar. Siri ketiga dan keempat telah disediakan dengan memperkenalkan Ag_2O ke dalam sampel kaca yang terbaik dari siri kaca pertama dan kedua. Oleh itu, komposisi kimia bagi siri ketiga dan keempat ialah $[\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.70}(ZnO)_{0.30}\}_{0.96}(La)_{0.04}]_{1-y}(Ag_2O)_y$ di mana y = 0.02, 0.04, 0.06, 0.08 dan 0.10 pecahan molar. Sifat struktur, fizikal dan optik linear bagi sampel kaca yang disediakan telah disiasat dengan menggunakan spektrometer pembelauan sinar-X (XRD), spektrometer Inframerah Jelmaan Fourier (FTIR), mikroskopi penghantaran elektron (TEM), densimeter dan spektrofotometer UV-nampak (UV-Vis). Sifat optik tak linear bagi sampel kaca telah didedahkan melalui teknik imbasan-Z manakala keupayaan bagi sampel yang disediakan untuk menjadi penyerap boleh tepu (SA) telah diuji dalam konfigurasi eksperimen serabut laser. Keadaan berkaca dan sifat amorfus bagi kaca yang disediakan telah dibuktikan melalui kehadiran bonggol yang luas dalam corak XRD. TeO_4, TeO_3, BO_3 dan BO_4 unit stuktur telah dikesan dalam semua sampel seperti yang ditunjukkan dalam spektrum FTIR. Kewujudan La NPs dalam siri kaca kedua dan keempat telah disahkan melalui TEM dengan di mana saiz nanozarah yang direkodkan ialah 30.30 nm dan 54.26 nm. Trend

pertambahan dalam kepadatan yang telah diperolehi bagi semua sampel kaca adalah kerana penambahan argentum dan lantanum yang mempunyai jisim molekul yang lebih besar ke dalam sistem kaca zink borotellurit. Bagi siri kaca tanpa pertambahan Ag_2O, jurang jalur optik tidak langsung didapati mempunyai trend yang meningkat manakala nilai indeks biasan semakin berkurangan masing-masing dalam lingkungan 2.20 hingga 3.90 eV dan 2.19 hingga 2.50. Sebaliknya, pengenalan Ag_2O ke dalam sistem kaca zink borotellurit yang didopkan dengan La_2O_3 dan La NPs telah meningkatkan keseluruhan sifat optik kaca di mana jurang jalur optik menurun dan indeks biasan meningkat secara mendadak masing-masing dalam lingkungan 1.94 hingga 4.16 eV dan 2.14 hingga 2.76. Kebolehkutuban elektronik yang dipengaruhi oleh kekutuban keseluruhan sistem kaca berkurang untuk siri kaca tanpa Ag_2O manakala kebolehkutuban elektronik meningkat untuk kaca zink borotellurit gelas didop dengan La_2O_3, La NPs dan Ag_2O. Alasan di sebalik pengurangan kebolehkutuban elektronik adalah pembentukan oksigen penitian yang mempunyai kebolehkutuban yang rendah dalam kaca zink borotellurit terdop La_2O_3 dan La NPs. Sebaliknya, Ag_2O yang mendorong penciptaan oksigen bukan penitian yang berkebolehkutuban yang tinggi telah menyumbang kepada peningkatan nilai kebolehkutuban elektronik. Kaca zink borotellurit yang didopkan dengan La_2O_3, La NPs dan Ag_2O telah mencatatkan trend tidak linear bagi indeks pembiasan tak linear, pekali penyerapan tidak linear, suseptibiliti urutan ketiga dan angka merit. Plot imbasan-Z dengan apertur terbuka telah mencatatkan penyerapan tepu (SA) dan penyerapan tepu songsang (RSA) yang menunjukkan potensi sampel kaca untuk digunakan sebagai SA dan alat optik tidak linear. Keupayaan sampel kaca untuk menjadi penyerap boleh tepu dalam serabut laser berdop erbium (EDFL) telah didedahkan buat kali pertama di mana sampel yang disediakan dapat memulakan operasi laser berdenyut mod terkunci dan Q-sius. Laser berdenyut dwi-gelombang dalam mod terkunci yang mempunyai kadar pengulangan laser berdenyut yang tinggi sebanyak 5 GHz telah dicatatkan dengan menggunakan kaca zink borotellurit sebagai SA dalam rongga EDFL. Sebaliknya, apabila kaca zink borotellurit yang telah didopkan dengan 0.04 pecahan molar La NPs dan 0.10 pecahan molar Ag_2O digunakan sebagai SA dalam rongga laser yang sama, laser berdenyut Q-suis yang stabil dengan tenaga laser berdenyut yang tinggi iaitu 0.4 µJ yang dihasilkan. Hasil daripada analisis laser berdenyut menunjukkan bahawa kaca zink borotellurit serta kaca zink borotellurit berdopkan dengan La NPs dan Ag_2O masing-masing boleh digunakan sebagai SA untuk menghasilkan laser berdenyut dengan kadar pengulangan laser berdenyut yang tinggi dan laser berdenyut yang bertenaga tinggi. Kajian ini dapat memberikan pengetahuan baru tentang kesan pendopan La_2O_3, La NPs dan Ag_2O pada pelbagai sifat sistem kaca zink borotellurit, hubungan sebenar antara hasil daripada pengukuran imbasan-Z dengan keupayaan bahan kaca untuk menjadi komponen penyerap boleh tepu serta menunjukkan keupayaan bahan kaca untuk digunakan sebagai komponen SA dalam sistem EDFL.

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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:

Halimah Mohamed Kamari, Ph.D.
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Amirah Abd Latif, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

Farah Diana Muhammad, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

____
ROBIAH BINTI YUNUS, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

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Signature: ______________________________

Name of Chairman of Supervisory Committee: Professor Halimah Mohamed Kamari

Signature: ______________________________

Name of Member of Supervisory Committee: Dr. Amirah Abd Latif

Signature: ______________________________

Name of Member of Supervisory Committee: Dr. Farah Diana Muhammad

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ASTM	American Society for Testing Materials
T_g	Glass transition temperature
T_m	Melting temperature
TeO_2	Tellurium oxide
B_2O_3	Boron oxide
ZnO	Zinc oxide
La_2O_3	Lanthanum oxide
La NPs	Lanthanum nanoparticles
Ag_2O	Silver oxide
P_2O_5	Phosphate oxide
GeS_2	Germanium disulfide
In_2S_3	Indium sulfide
KI	Potassium iodide
AgI	Silver iodide
Dy_2O_3	Dysprosium oxide
Gd^{3+}	Gadolinium ion
Er^{3+}	Erbium ion
RE	Rare earth
NBO	Nonbridging oxygen
BO	Bridging oxygen
FOM	Figure of merit
SA	Saturable absorber
BP	Black phosphorous
EDFL	Erbium doped fiber laser
Zn NPs	Zinc oxide nanoparticles
Ag NPs	Silver nanoparticles
THDF	Thulium holmium doped fiber
SMF	Single mode fiber
EDF	Erbium doped fiber
WDM	Wavelength division multiplexer
ISO1	Optical isolator 1
ISO2	Optical isolator 2
PC	Polarization controller
OC	Optical coupler
OSA	Optical spectrum analyzer
XRD	X-ray diffraction spectroscopy
FTIR	Fourier transform infrared spectroscopy
TEM	Transmission electron microscopy
UV-Vis	Ultra-violet visible light spectrophotometer
V_m	Molar volume
α	Absorption coefficient
E_{opt}	Energy band gap
ΔE	Urbach energy
n_0	Linear refractive index
α_m	Electronic polarizability
α_{O2-}	Oxide ion polarizability
Λ	Optical basicity
M	Metallization criterion

n_2	Nonlinear refractive index
β	Nonlinear absorption coefficient
TPA	Two photon absorption
$\chi^{(3)}$	Third order susceptibility

CHAPTER 1

INTRODUCTION

This chapter unveils the research background of this study that consists of a simple history as well as an introduction to glass, the formation of glass material and the chemical components that made up the glass. Crucial component of this study such as problem statement, scope of study, research objective, hypothesis and the outline of this thesis are also included in this chapter.

1.1 History of Glass

Glass is a material that plays an important role in the daily life of every human being. There are many things in the world today that are actually made from different types of glass material including drinking goblets, window glasses, chemical glassware, decorative vases, spectacles, electric bulbs and communication cables (Rao, 2002). Sometimes, the existence of glass material in human life is so common that at a certain level, humans tend to neglect and seldom notice the presence of glass material.

The first ever glass has been dated back to Mesopotamia era in the western Asia at 4000 B.C. Although its usage remains unknown, the glass seems to be coloured as the presence of copper compound is detected in the glass material. Later, the Egyptians learnt the technique of producing glass by melting raw materials such as sands which contain high percentage of silica in the process. The Egyptians developed glass blowing method and the produced glass materials were widely used as glass tableware and even for decorative purposes. Roman Empire is the most glorified era in glass making history whereby humans at that time able to create glasses with different and various colour along with their famous glass blowing technique. Around that time, the technique of creating coloured glass, for instance, the fabrication of red colored glass requires the inclusion of gold component in the glass, is considered as the family secret whereby it is passed on from generations to generations of glass maker (Shelby, 2005).

Over these few years, glass materials have once again intrigued the interest of glass researches from all over the world in owing to the ability of glass material having different properties when it is composed of different chemical compound. The evolution in the preparing method of glasses and the wish of having a deeper scientific understanding in glass structure also contribute to the peaking interest on glass materials among the researchers. The growing and improving technology nowadays provide various new application for many materials especially glass materials. Glass has been an important component in many field for examples in the telecommunication and medical field whereby glass started to replace copper wire in order to transmit data more efficiently across the world as well as generating laser beam used in the medical procedure. Recently, instead of manufacturing glass by using the silica element which exist in ordinary sand as the backbone of the glass, the chemical composition that made up the glass system is being chosen according to the function of the glass after it is manufactured. For instance, glasses that are being incorporated with rare earth ions will be employed as laser to generate high intensity light beam, glasses doped with halide oxide will have the ability to change colour upon exposure to light while large and thick lead oxide glasses doped with lead oxide are usually being utilized as radiation shielding glasses.

1.2 Definition of Glass

American Society for Testing Materials (ASTM) in 1945 had defined glass can be defined as an inorganic product of fusion which is cooled to a rigid condition without undergoing the crystallizing process. Meanwhile, Doremus (1994) had stated that glass is a material which can be prepared by the cooling of normal liquid state matter whereby the material will either become more or less rigid as there is an increment in the viscosity of the material. In other words, glass is a type of solid that can be obtained by rapidly cooling a liquid in which it will possess an amorphous nature that can be verified via x-ray diffraction spectroscopy (Rao, 2002). Generally, a material can be classified as crystalline, polycrystalline, amorphous, non-crystalline or glassy which depends upon the extent of the periodic atomic or molecule arrangement in the material (Kothiyal et al., 2012). The terms amorphous, non-crystalline and glassy can be used interchangeably to describe glass material since glass material usually have a short range of periodic atomic arrangement. Usually, the periodic order of atoms or molecules in amorphous materials such as glasses are usually limited to only a few atomic distance around 10 Å.

The traditional and most common technique employed to prepare a glass material is by melt quenching technique whereby the molten glass will be rapidly cooled in order to achieve the required glassy state. However, there are also many other methods such as vapour deposition and sol-gel processing of solution that can be used to manufacture glass depending on the chemical composition of the glass system. In general, no matter what glass fabrication process is used in manufacturing the glass, the common characteristics that can be found in all glass materials include the lack of long range periodic atomic arrangement in their structure as well as the time dependent glass transformation behavior of the glass material (Karmakar et al., 2016).

1.3 Formation of Glass

The most famous theory on the glass formation is proposed by Zachariasen in 1932. After considering the glass forming ability of oxides, Zachariasen (1932) had came out with the rules which one must follow in order to fabricate a glass successfully:

1. Each oxygen atom must not linked to more than two glass forming atoms or two cations.
2. The coordination number for the glass forming atoms or cation must be small (3 or 4)

3. The oxygen polyhedral should share corners with each other instead of sharing edges or faces
4. The 3D glass network should have at least three corners shared with each other

According to Doremus (1994), oxides with chemical formula of A_2O or AO where A represents metal atoms does not satisfy the rules for forming a glass system. On the other hand, oxides in A_2O_3 form perfectly satisfy the proposed rules since the oxygen atoms are able to form triangles around each A atoms and A atoms also have the ability to support three oxygen atoms. During Zachiariasen time, he listed the possible glass former oxides that include boron oxide, silicon dioxide, germanium oxide, phosphorus oxide and arsenic oxide which have been fabricated successfully into glass. It has been discovered later that the requirement for the oxides to form three dimensional network is to allow the breaking of the primary bond when modifier is introduced into the system.

The glass transition diagram that explains the glass forming process is as presented in Figure 1.1. According to Zanotto and Mauro (2017), after the chemical oxide are heated as well as melted above the melting temperature, T_m, it exists in a stable liquid form which did not encounter any crystallization. When the liquid is cooled between T_m and the glass transition temperature, T_g, the liquid is in its metastable state. At this point, if the liquid is slowly cooled, the liquid will have the time to rearrange themselves in a periodic order to allow crystal nucleation to occur and finally after a certain time, the liquid will crystallize. On the other hand, when the liquid is supercooled at a rapid rate, the atoms in the liquid will be frozen in an irregular arrangement since it does not have enough time to rearrange themselves. If a glass is maintained at a temperature around T_g for a sufficiently long times, the glass will have the ability to relax, rearrange its atomic arrangement to undergoes crystallization.

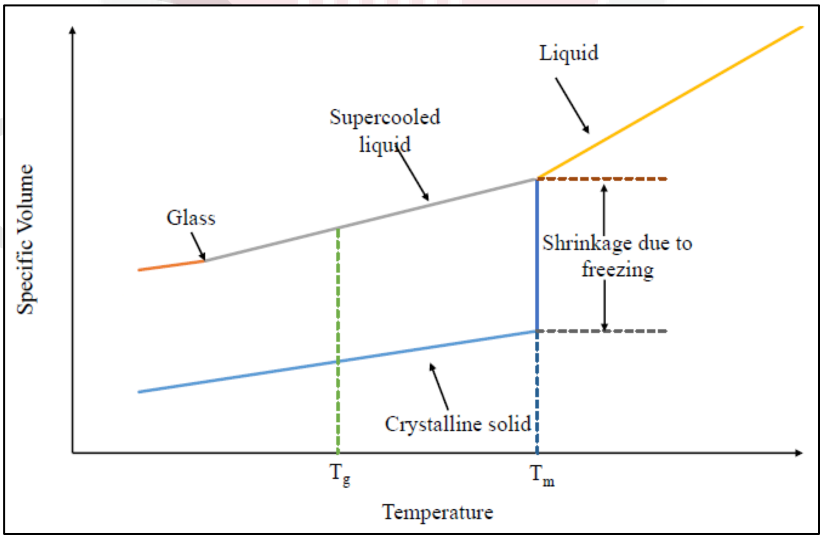

Figure 1.1: Glass transition diagram (Stachurski, 2011)

1.4 Chemical Compounds in Zinc Borotellurite Glass doped with Lanthanum Oxide, Lanthanum Nanoparticles and Silver Oxide

According to Elliot (1991), the short range order of atoms or molecules arrangement that affect the properties of a glass is influenced by the nature of the glass melts which controlled by its composition, its manufacturing process and the type of dopants incorporated into the glass system. Nowadays, various type of glass are studied and investigated by scientist around the world in owing to their unique and distinct properties. The reason behind the unique properties of each glass system is the chemical composition of the glass whereby the properties of a glass system will reflect the intrinsic nature of the component that has been added into the glass mixture.

Tellurium oxide (TeO_2) has been chosen to be the main component in this glass system because of its splendid characteristic for optical application. Tellurium oxide has always been the candidate for being the best host matrix for linear and nonlinear optical glass in owing to its high polarizability tellurium ion, low melting point, good infrared transmission and large third order susceptibility (Suresh et al., 2006). Tellurium oxide is a conditional glass former whereby it requires the addition of a modifier oxide such as alkali, alkaline earth and transition metal oxide or other glass former in order to form glassy state (Nazrin et al., 2018). The special characteristic of tellurium oxide that allows the experimentation with a wider selection of elements in its glass composition leads to the creation of tellurite based glasses with various properties and function. Boron oxide is a well-known superior host matrix for rare earth metal oxide due to its ability to form glass over a wide range of composition without the addition of any network modifiers or other glass former. This excellent glass forming ability of borate glass is contributed by the BO_3 triangles and BO_4 tetrahedra units in borate matrix that to allow it to form stable borate groups such as diborate, triborate and tetraborate after being fabricated into glass (Maheshvaran et al., 2013). The reason of choosing boron oxide to take part as glass former along with tellurium oxide is because boron is able to assist tellurium oxide in manufacturing stable tellurite based glass with an enhancement in its optical and electrical properties. Other than that, tellurium oxide is able to reduce the hygroscopic nature of borate glass and at the same time widen the infrared transmittance of the overall glass system (Azlan et al., 2016). Zinc oxide is an intermediate oxide with dual nature in which it can act as network former and network modifier after being incorporated into a glass system. Zinc oxide is introduced into the zinc borotellurite in order to help in reducing the crystallization rate in the glass system as well as to increase the glass forming ability of the glass (Pavani et al., 2011a). When zinc oxide is added into a glass system, it would also be able to stabilize and lower the overall melting point of the glass network.

It has been a global trend for glass researches all over the world for studying the properties of glasses that have been doped with rare earth element. The doping of rare earth ions into glass system is able to enhance and improve the optical properties of the glass materials in owing to its f shell valence electrons (Shakeri and Rezvani, 2011). Nowadays, rare earth elements are usually added into certain glass host to emit high intensity laser light. Light emitting materials also have been doped with rare earth element for allowing it to emit energy saving light with good optical efficiency as well as a long life span. In contrast with the recent trend of exploring the most famous rare-earth element, erbium, lanthanum is chosen to be doped into the zinc borotellurite glass system to unveil the effect of lanthanum on various properties of the glass system. Lanthanum is the first element in the lanthanide group and one of the major rare earth elements, which occupies the 14.1% of total rare earth resources. In previous researches done by Doweidar and Saddeek (2010), lanthanum seems to play a role as network modifier oxide when it is introduced into a glass system. According to Terashima et al. (1997), lanthanide ions (Ln^{3+}) in its ground state have an electronic structure of [Xe] $4f^{n}$ $5s^2$ $5p^6$ (n = 0-14), whereby the nonbonding f electrons are shielded by $5s^2$ $5p^6$ orbital. Unfortunately, lanthanum with electron configuration of $1s^2$ $2s^2$ $2p^6$ $3s^2$ $3p^6$ $3d^{10}$ $4s^2$ $4p^6$ $4d^{10}$ $5s^2$ $5p^6$ $5d^1$ $6s^2$ is the only lanthanide that does not possess any f electron that has been well-known to have a dominant influence on the optical properties (Lerner and Trigg, 1991). As reported by Gedam and Ramteke (2013), the inclusion of lanthanum oxide in their glass material seems to bring about enhancement in both optical and electrical properties. Nanomaterials that have been studied extensively nowadays are also included in this research. The nanoparticles with much smaller particles size should be able to reveal interesting and unique properties after they are doped into the glass system. Silver oxide along with copper and gold oxide are among the oxides that are able to enhance and improve the optical properties of glass material. Halimah et al. (2010) reported that silver oxide has the ability to modify the borotellurite glass network by altering the structural units of the borate network from (BO_3) to (BO_4) and tellurite network from (TeO_4) to (TeO_3).

1.5 Problem Statements

The quest of improving the optical properties of glass material for their application in various field including the enhancement in materials science and technology is a non-stop challenges for researches all over the world. Up to this day, many researches had investigated various glass system with different chemical composition that has the potential to be utilized as all optical switching devices, optical limiting devices, fuel cells, radiation shielding glass, chemical glassware and as laser host (Eevon et al., 2016; Sasmal et al., 2014).

It is a well-known fact that tellurite based glass is the best glass material to be employed as photonic devices. Tellurite glass has been suggested as the glass with high potential to be utilized in photonic field in owing to exceptional chemical durability, good thermal stability, good optical switching response, low optical loss as well as great optical nonlinearity (Yousef et al., 2012). High polarizable tellurium ions in tellurite based glass contribute to the high polarizability of the overall glass that eventually able to reduce the energy band gap and at the same time increase the linear and nonlinear refractive index of respective glass system. Reddy et al. (2008) stated that the addition of tellurium element in a particular glass system along with the introduction of suitable dopants ions is able to improve the overall transmission capability of the glass as well as increasing the moisture resistance and transparency of the glass in the ultraviolet and infrared wavelength regions In owing to the various advantages of tellurite based glass, researchers tried and succeed in doping rare earth element into the tellurite glass system by using melt-quenching technique. Surprisingly, the incorporation of rare-earth element into the tellurite system seems to further enhance the optical properties and expends the possible application of the doped glass.

Nowadays, the glasses that are doped or co-doped with rare-earth ions have the potential for promising applications including as sensors, fiber amplifier, laser, optical data storage and optical communication devices (Akmar Roslan et al., 2012).

However, there are very few information and knowledge on the effect that lanthanum oxide and lanthanum nanoparticles bring about to the zinc borotellurite glass system. Although zinc borotellurite glass system doped with lanthanide element with 4f electron such as erbium, samarium, dysprosium and gadolinium has been studied recently, exceptional lanthanum with the absence of 4f electron might influence the overall characteristic as well as properties of the zinc borotellurite glass system. In addition, the co-doping of lanthanum and silver element in the same glass system have never been studied by any researchers up until now. The physical, structural, linear and nonlinear optical properties of lanthanum oxide, lanthanum nanoparticles and silver oxide doped zinc borotellurite glass system have never been investigated to allow their possible application as all optical devices or optical limiters. Moreover, the application of glass material as saturable absorber has never been discovered while its potential to be utilized as saturable absorber can be unveiled through z-scan open aperture measurement. Thus, the aim of this research is to study the influence of lanthanum oxide, lanthanum nanoparticles and silver oxide on physical, structural, linear and nonlinear optical properties of zinc borotellurite glass as well as its possible application as saturable absorber in erbium doped fiber laser system.

1.6 Scope of Study

The scope of this study is limited to the physical properties (density and molar volume), structural properties (TEM, XRD and FTIR), linear optical properties (optical absorption, energy band gap, Urbach energy, refractive index, electronic polarizability, oxide ion polarizability, optical basicity, and metallization criterion), nonlinear optical properties (nonlinear absorption coefficient, nonlinear refractive index, third order susceptibility and figure of merit) and pulsed operation of erbium doped fiber laser (mode of pulse laser operation, pump power, central wavelength, pulse repetition rate, pulse energy and pulse width).

1.7 Research Objective

This study is done based on four clear and precise objectives. The objectives are as follow:

1. To synthesize zinc borotellurite glasses doped with lanthanum nanoparticles, lanthanum and silver oxides.
2. To determine the effect of lanthanum nanoparticles, lanthanum and silver oxides on the physical and structural properties of zinc borotellurite glass system through transmission electron microscope (TEM), X-ray diffraction (XRD) as well as fourier transform infrared (FTIR) spectroscopy.

3. To study the effect of lanthanum nanoparticles, lanthanum and silver oxides on the linear and nonlinear optical properties of zinc borotellurite glass system via Ultraviolet-visible light spectrophotoscopy as well as Z-scan technique.
4. To investigate the ability of zinc borotellurite glass system doped with lanthanum nanoparticles, lanthanum and silver oxides to function as saturable absorber as well as to generate pulse operation in erbium doped fiber laser cavity.

1.8 Hypothesis

Based on the four concise objectives, the hypotheses for this research include:

1. All the glass samples are successfully fabricated via melt quenching technique whereby the prepared glasses are stable and non-hygroscopic.
2. The incorporation of lanthanum oxide, lanthanum nanoparticles and silver oxide into zinc borotellurite glass system should produce glasses with amorphous nature that can be verified through XRD and the breaking of glass structure which will form TeO_4, TeO_3, BO_4 and BO_3 in the glass system which should be detected by the FTIR. The presence of lanthanum nanoparticles in the glass system even after the glass formation process is expected to be observed in the TEM image. The density values are expected to be increased as heavier dopant are added into the glass matrix.
3. The crucial parameter for linear and nonlinear optical properties such as linear refractive index and nonlinear refractive index of zinc borotellurite glass doped with lanthanum oxide and lanthanum nanoparticles are expected to have decrement trend since lanthanum does not have f shell electron that is able to improve the linear and nonlinear optical properties. Meanwhile, it is expected that the linear and nonlinear optical properties for zinc borotellurite glass doped with lanthanum oxide, lanthanum nanoparticles and silver oxide will be improved. This is might be due to the presence of silver oxide in the glass system that is able to create more highly polarizable nonbridging oxygen and contributes to the reduction in energy band gap as well as increment in both linear and nonlinear refractive index.
4. Passive Q-switched and passive mode-locked pulse laser operation should be achieved since zinc oxide and silver oxide that has the potential to induce pulse laser operation are part of the glass composition of the studied glasses.

1.9 Outline of Thesis

This thesis is divided into six chapter whereby the first chapter explains the background of this research, brief introduction on the history of glass material, glass formation process as well as chemical component chosen and used in the studied glass. Chapter 1 also focuses on the problem statement, objective and possible outcome of this study. Chapter 2 gives a brief review from previous reports that is related to this study. The brief literature includes some information about zinc borotellurite based glass, lanthanum doped glass, silver doped glass, structural properties, physical properties, linear optical properties, nonlinear optical properties and the pulse laser operation that had been achieved using various material while chapter 3 highlights the theory and equations that are used in this research. The derivation for some equations are also shown in this chapter. Chapter 4 describes the process and technique employed in fabricating the glass samples. The setup for z-scan measurement and saturable absorber testing are also presented and explained in this chapter. On the other hand, chapter 5 discusses the results obtained for the required parameters that covers the structural, physical, linear and nonlinear optical properties of all glass samples. The comparative studies between some parameters of the fabricated glass with the previous studies are also covered in this chapter. The summarization of the important outcome from this research and some suggestion for future studies are included in chapter 6.

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BIODATA OF STUDENT

Faznny Binti Mohd Fudzi was born in Hospital Besar Ipoh, Perak, Malaysia. She received her primary education in Sekolah Jenis Kebangsaan (Cina) Mambang Di Awan and then continued her secondary education in Sekolah Menengah Kebangsaan Pei Yuan. She went to Kolej Matrikulasi Perak before pursuing her degree in Bachelor of Science (Honors) Majoring Physics at Universiti Putra Malaysia and graduated in 2015. She further her study on the degree of Doctor of Philosophy in Material Science at Universiti Putra Malaysia. Her current research focuses on the zinc borotellurite glass doped with lanthanum and silver oxide as saturable absorber for fiber laser applications.

LIST OF PUBLICATIONS AND CONFERENCE ATTENDED

List of Publication:

Faznny, M. F., Halimah, M. K., & Azlan, M. N. (2016). Effect of lanthanum oxide on optical properties of zinc borotellurite glass system. *Journal of Optoelectronics and Biomedical Materials*, *8*(2), 49-59.

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