UNIVERSITI PUTRA MALAYSIA

DOPING OF ERBIUM AND SILVER IN ZINC TELLURITE GLASS SYSTEM FOR OPTICAL APPLICATION

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By

NAZIRUL NAZRIN BIN SHAHROL NIDZAM

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

September 2020

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DEDICATION

This thesis is especially dedicated to my parents, Shahrol Nidzam B. Shaari and Sa'adiah Bt. Abd Hadi, family members, lecturers, teachers and friends.

Not to forget, my late grandfather, Hj Shaari B. Majid, Uncle Azli B. Mat and Mdm Ambika Krishnasamy.

Thank you for encouraging me to accomplish this journey that full of cuts and thorns.

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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September 2020

Chairman : Professor Halimah Mohamed Kamari, PhD
Faculty : Science

The improvement of properties for glass material application in disparate range are open-ended challenges especially in materials science and technology. Although there are researches being executed on lanthanide elements with zinc tellurite glass as parent material, no researchers have tried to assimilate both erbium and silver in a glass system in order to investigate the changes made by the glass materials. To reveal the effect of erbium and silver oxides on structural, physical, elastic, thermal, optical properties as well as the execution of McCumber and Judd-Ofelt theory analysis, four glass series of zinc tellurite glasses that were doped with erbium and silver oxides were successfully synthesized by melt-quenching method. The glasses were prepared based on the chemical composition provided with variation concentration of dopants; 1) $[(TeO_2)_{0.7}(ZnO)_{0.3}]_{1-x}(Er_2O_3)_x$, 2) $[[(TeO_2)_{0.7}(ZnO)_{0.3}]_{0.96}(Er_2O_3)_{0.04}]]_{1-y}(Ag_2O)_y$, 3) $[(TeO_2)_{0.7}(ZnO)_{0.3}]_{1-x}(Ag_2O)_x$ and 4) $[[(TeO_2)_{0.7}(ZnO)_{0.3}]_{0.98}(Ag_2O)_{0.02}]]_{1-y}(Er_2O_3)_y$ where in first, second and fourth glass series, the molar fraction, x = 0.01, 0.02, 0.03, 0.04 and 0.05 whereas in third glass series, x = 0.005, 0.010, 0.015, 0.020 and 0.025. The different dopant's amount in third glass series were attributed to the less fragility of the glass if low concentration as low as 0.025 is employed. The structural, physical, elastic, thermal and optical properties of the glass samples were elucidated using equipment such as X-ray diffraction (XRD), Fourier transform infrared (FTIR), densimeter, Ultrasonic machine, Differential scanning calorimeter (DSC), UV-Visible (UV-Vis) and photoluminescence spectrometers. Meanwhile, Origin 6.0 had been used for deconvolution and McCumber analysis and spectragryph had been employed for Judd-Ofelt evaluation. The amorphous nature of the prepared glass samples is confirmed from the XRD analysis. In the study of FTIR spectra, the presence of TeO_4 was detected while TeO_3, ZnO, Er_2O_3 and Ag_2O functional groups were not detected. The non-existence of the remaining functional groups implies that these bonds have been broken down within the glass network. By implementing the deconvolution technique using Origin 6.0 software, the remaining functional groups were found. This phenomenon confirms that there is still a little amount of concentration of the remaining functional groups that can be observed. The density of

all glass series is found to increase approximately from 4.0 to 6.0 g/cm^3 due to the improvement in the compactness of the glass samples. Nevertheless, the molar volume (V_m) inclines for glasses that consists of erbium oxide as the main dopant while for the glasses that consist of silver oxide as the main dopant, the molar volume decreases. The inclination of molar volume is attributed to larger bond length of the dopant. On the other hand, the decrement of the molar volume can be theoretically related to the inverse relation between density and molar volume. The elastic moduli and some of the elastic parameters of the glasses are generally found to vary with the increase of erbium oxide and silver oxide while the Poisson's ratio lies in the expected range of 0.2 to 0.3. The presence of erbium oxide and silver oxide in the glass system make the glass sample becomes compact and rigid due to their larger atomic mass compared to the host glass materials. Besides that, the experimental study of elastic properties has shown the similarities in most of the theoretical elastic including Makishima and Mackenzie model and rocherulle model. The thermal properties were measured for all glass series. The glass stability for most of the glass samples with erbium oxide as the main dopant are been found to exceed 100°C, which is convenient in fiber optic application. The erbium oxide enhances the link of cation-anion in the glass system which will improve the strength of the glass. In terms of optical parameters, the optical band gap, refractive index and metallization criterion display some variation as the dopants were inserted into the glass samples. The band gap of the prepared glasses in this work ranges from 3.0 to 5.0 eV which is suitable for semiconductor device application. Meanwhile, refractive index values of the prepared glasses are found to be around 2.3 to 2.5, which are relatively high due to the presence of non-bridging oxygen, making them useful for laser operation. Metallization criterion with value of 0.30-0.45 guarantees the glasses ability for non-linear application such as optical switching and optical limiting. Blue, green and red emission are found in all glass series under 375 nm excitation wavelength. Intense and broad near infrared 1500 nm emission above 48 to 137 nm FWHM and gain bandwidth within the range of (97 to 625) x 10^{-28} cm^3 had been attained in all glass system except for third glass series. This result suggests that the fabricated glass samples have the potential to become a promising optical material for broadband telecommunication technology. In laser designation, the Judd-Ofelt analysis had been analyzed for all the glass system consisting of rare earth element. Large values of Ω_2, Ω_4 and Ω_6 are strongly related to the improvement of the asymmetrical behavior of erbium ions sites with the formation of higher electron density on the oxygen ligand ion. As a result, strong Er-O covalence is formed with the increasing erbium ions concentration.

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

PENDOPAN ERBIUM DAN ARGENTUM DALAM KACA ZINK TELURIT UNTUK APLIKASI OPTIK

Oleh

NAZIRUL NAZRIN BIN SHAHROL NIDZAM

September 2020

Pengerusi : Profesor Halimah Mohamed Kamari, PhD
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Peningkatan sifat untuk aplikasi bahan kaca dalam jurang yang berbeza adalah cabaran terbuka terutama dalam sains dan teknologi bahan. Walaupun terdapat penyelidikan yang dilakukan terhadap unsur lanthanid dengan kaca zink tellurit sebagai bahan induk, tidak ada penyelidik yang telah cuba mengasimilasikan kedua erbium dan argentum dalam sistem kaca untuk menyiasat perubahan yang dibuat oleh bahan kaca. Untuk mendedahkan kesan erbium dan argentum oksida pada sifat struktur, fizikal, elastik, terma, optik serta pelaksanaan analisis teori McCumber dan Judd-Ofelt, empat siri kaca zink telurit yang didop dengan erbium dan argentum oksida telah berjaya disintesis dengan kaedah lebur lindap. Kaca-kaca telah disediakan berdasarkan komposisi kimia yang disediakan dengan kepekatan variasi dopan; 1) $[(TeO_2)_{0.7}(ZnO)_{0.3}]_{1-x}(Er_2O_3)_x$, 2) $[[(TeO_2)_{0.7}(ZnO)_{0.3}]_{0.96}(Er_2O_3)_{0.04}]]_{1-y}(Ag_2O)y$, 3) $[(TeO_2)_{0.7}(ZnO)_{0.3}]_{1-x}(Ag_2O)_x$ dan 4) $[[(TeO_2)_{0.7}(ZnO)_{0.3}]_{0.98}(Ag_2O)_{0.02}]]_{1-y}(Er_2O_3)_y$ ialah pada siri kaca pertama, kedua dan keempat, pecahan molar, x = 0.01, 0.02, 0.03, 0.04 dan 0.05 sedangkan pada siri kaca ketiga, x = 0.005, 0.010, 0.015, 0.020 dan 0.025. Jumlah dopan yang berbeza dalam siri kaca ketiga telah dikaitkan dengan kerapuhan kaca yang kurang jika kepekatannya adalah serendah 0.025 digunakan. Sifat struktur, fizikal, elastik, terma dan optik sampel kaca dilaksanakan menggunakan peralatan seperti difraksi sinar-X (XRD), inframerah transformasi Fourier (FTIR), densimeter, mesin ultrasonik, kalorimeter pengimbasan pembeza (DSC), UV- Spektrometer cahaya (UV-Vis) dan fotoluminesen. Sementara itu, Origin 6.0 telah digunakan untuk dekonvolusi dan analisis McCumber dan spectragryph telah digunakan untuk penilaian Judd-Ofelt. Sifat amorfus sampel kaca yang disiapkan, disahkan daripada analisis XRD. Dalam kajian spektrum FTIR, kehadiran TeO_4 telah dikesan sementara kumpulan fungsian TeO_3, ZnO, Er_2O_3 dan Ag_2O tidak dapat dikesan. Ketiadaan kumpulan fungsi yang berbaki menunjukkan bahawa ikatan ini telah dipecah dalam rangkaian kaca. Dengan menerapkan teknik dekonvolusi menggunakan perisian Origin 6.0, kumpulan berfungsi yang masih berbaki telah dijumpai. Fenomena ini mengesahkan bahawa masih terdapat sedikit kepekatan kumpulan fungsi yang masih dapat diperhatikan. Ketumpatan semua siri kaca didapati meningkat kira-kira daripada 4.0 hingga 6.0 g/cm^3 disebabkan peningkatan padat sampel kaca. Walaupun begitu,

isipadu molar (V_m) meningkat untuk kaca yang terdiri daripada erbium oksida sebagai dopan utama sementara bagi kaca yang terdiri daripada argentum oksida sebagai dopan utama, isipadu molar menurun. Kenaikan isipadu molar disebabkan oleh panjang ikatan dopan yang lebih besar. Sebaliknya, penurunan isipadu molar boleh dikaitkan secara teoritis dengan hubungan songsang antara ketumpatan dan isipadu molar. Moduli elastik dan beberapa parameter elastik untuk kaca-kaca didapati berbeza-beza dengan peningkatan erbium oksida dan argentum oksida sementara nisbah Poisson berada pada julat yang diharapkan daripada 0.2 hingga 0.3. Kehadiran erbium oksida dan argentum oksida dalam sistem kaca menjadikan sampel kaca menjadi padat dan tegar kerana jisim atomnya yang lebih besar berbanding dengan bahan kaca hos. Selain itu, kajian eksperimen sifat elastik telah menunjukkan persamaan pada kebanyakan teori elastik termasuk model Makishima dan Mackenzie dan model rocherulle. Sifat termal telah diukur untuk semua siri kaca. Kestabilan kaca bagi kebanyakan sampel kaca dengan erbium oksida sebagai dopan utama didapati melebihi 100°C, yang mudah digunakan dalam aplikasi optik gentian. Erbium oksida meningkatkan hubungan kation-anion dalam sistem kaca yang akan meningkatkan kekuatan kaca. Dari segi parameter optik, jurang jalur optik, indeks biasan dan kriteria metalisasi menunjukkan beberapa variasi apabila dopan dimasukkan ke dalam sampel kaca. Jurang jalur kaca-kaca yang telah disediakan dalam kerja ini berkisar antara 3.0 hingga 5.0 eV yang sesuai untuk aplikasi peranti semikonduktor. Sementara itu, nilai indeks biasan kaca yang disiapkan didapati sekitar 2.3 hingga 2.5, yang secara relatifnya tinggi kerana adanya oksigen bukan penghubung, menjadikannya berguna untuk operasi laser. Kriteria metalisasi dengan nilai 0.30 - 0.45 menjamin kemampuan kaca-kaca untuk aplikasi bukan linear seperti peralihan optik dan penghadan optik. Pencahayaan biru, hijau dan merah terdapat pada semua siri kaca dengan panjang gelombang pengujaan 375 nm. Pencahayaan 1500 nm berhampiran inframerah yang kuat dan luas melebihi 48 hingga 137 nm FWHM dan dapatan lebar jalur berada dalam julat (97 hingga 625) x 10-28 cm^3 telah dicapai pada semua sistem kaca kecuali untuk siri kaca ketiga. Hasil ini menunjukkan bahawa sampel kaca buatan berpotensi menjadi bahan optik yang menjanjikan untuk teknologi telekomunikasi jalur lebar. Dalam sebutan laser, analisis Judd-Ofelt telah dianalisis untuk semua sistem kaca yang terdiri daripada unsur nadir bumi. Nilai besar Ω_2, Ω_4 dan Ω_6 sangat berkaitan dengan peningkatan tingkah laku asimetri tapak ion erbium dengan pembentukan ketumpatan elektron yang lebih tinggi pada ion ligan oksigen. Akibatnya, kovalen Er-O yang kuat terbentuk dengan kepekatan ion erbium yang semakin meningkat.

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I certify that a Thesis Examination Committee has met on (date of viva voce) to conduct the final examination of Nazirul Nazrin bin Shahrol Nidzam on his thesis entitled 'Doping of Erbium and Silver in Zinc Tellurite Glass System for Optical Application' in accordance with the Universities and University Collages Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Date: 14 January 2021

Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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A	Area
D	Thickness
T_g	Glass transition temperature
NBO	Non-bridging oxygen
BO	Bridging oxygen
ΔE	Energy difference
N	Refractive index
Λ	wavelength
XRD	X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
E_{opt}	Optical energy gap
α	constant
$\hbar\nu$	Photon energy
Ec	Urbach energy
TeO_2	Tellurium oxide
ZnO	Zinc oxide
Er_2O_3	Erbium oxide
Ag_2O	Silver oxide
ρ	density
V_m	Molar volume
v_L	Longitudinal velocity
v_s	Shear velocity
L	Longitudinal modulus
G	Shear modulus

K	Bulk modulus
E	Young's modulus
σ	Poisson's ratio
H	Micro-hardness
Θ_D	Debye temperature
Z	Acoustic impedance
T_s	Softening temperature
M	Metallization criterion
R_m	Molar refraction
α_e	Electronic polarizability
α_o^{2-}	Oxide ion polarizability
Λ	Optical basicity
DSC	Differential scanning calorimeter
IR	Infrared
UV	Ultraviolet
Vis	Visible
A	Absorbance
T	Transmittance
3D	3 Dimensions
v_{mean}	Mean velocity
N_A	Avogadro constant
T_f	Fictive temperature
v	velocity
c	Speed of light in vacuum
h	Planck's constant
k	Boltzmann's constant
ed	Electric dipole
ET	Energy transfer
FWHM	Full width half maximum

GBW	Gain band width
H_r	Hruby parameter
JO	Judd-Ofelt
md	Magnetic dipole line strength
T_o	Onset crystallization temperature
T_c	Crystallization temperature
T_m	Melting temperature
TeO_3	Trigonal pyramid
TeO_4	Trigonal bipyramid
S_{meas}	Measured line strength
S_{ed}	Electric dipole line strength
S_{calc}	Calculated dipole line strength
Ω	JO intensity parameters
τ_{rad}	Radiative lifetimes
σ_e	Emission cross section
σ_a	Absorption cross section
ß	Branching ratio
S	Thermal stability
T_s	Glass stability
E_a	Activation energy
n_b	Number of bonds per unit volume
n_c	Average cross-link density
F	Average stretching force constant
V_t	Packing density
G	Dissociation energy

CHAPTER 1

INTRODUCTION

This chapter discusses the introduction of glass, tellurite glass and all the chemicals that are used in this research. This chapter also includes problem statements, scope of study and limitations, objectives of the study, hypothesis, significance of the study, and outline of the thesis.

1.1 Preamble

Glass has brought the biggest definition to the world which contributes a lot of benefits to mankind especially in the scientific development and also in industry (Oliver, 1975). Recently, the contribution of glass has enlarged the scope of study of certain material based on the engineering perspective. The comprehensive findings on glass science and technology which discover the recent research on the application of photonic and optical is unquestionable. The novelty in the research on the application of glass is investigated consistently due to the unique properties in this era. Further quest in the glass field regarding the novelty of glass application inspires the scientist all over the world. The useful requirement and concern especially on communication system improve the evolution of glass material that has a high potential to be implemented in optical communication. There are numerous types of glass material which are established in the application of optic.

To date, silicate-based glass is required as the crucial component of making good fibre optics. However, silicate-based glass has weak absorbance and weak nonlinearity as well as high melting point (Halimah *et al.*, 2010). A good quality of optical glass is necessary to enhance the on-going of optical application. For that reason, tellurite-based glass has been chosen to be one of the best successors which produce a great quality of glass materials (Halimah *et al.*, 2010).

Halimah *et al.*, (2010) had reported that the tellurite glasses are the promising materials for non-linear application especially in optic and also laser which is due to several aspects, for instance, low phonon maxima, low melting point, and high refractive index. TeO_2 is also known as a conditional glass former where it requires an addition of modifier ion in order to form the glassy state easily. This will eventually lead to the creation of new structural units such as trigonal pyramidal (TeO_3) and trigonal bipyramidal (TeO_4). TeO_2 has its own influence where in certain chemical composition, it tends to decrease the hygroscopic level which will enhance the quality of the glass, making the glass less brittle and free from bubbles as well as improving the IR transmission.

Tellurite-based glass is also highlighted to be one of the unique and highly recommendable components in glass materials due to its wide band infrared transmittance; high refractive index and high dielectric constant (Sidek *et al.*, 2009). It has been deeply promoted in several optical applications such as optoelectronics, light emitting diode (LED), glass sensor and fiber optics. Up until now, the improvement of tellurite-based glass in the optical application is still in progress. As this glass is adaptable to several types of oxide materials, different oxides have been added into the tellurite glass network with a purpose to investigate either is it preferable to enhance the optical properties or contrariwise.

According to Sidek *et al.*, (2009), tellurite glasses had been thoroughly investigated due to its scientific and technological relevance. Tellurite glasses are considered as a good candidate for recent usage in optical materials due to some desirable aspects such as a wideband infrared transmittance, high refractive index and high dielectric constant. In addition, they also exhibit high densities, low transformation temperature and non-hygroscopic properties which can overcome the application of phosphate and borate glasses. Hence, these glasses are appropriate as a host material for active element doping and serving as the major justification for their endless technological significance in certain areas of optoelectronics such as optical fibre, sensor system, and laser technology.

As is known, zinc oxide is a chemical compound that has a molecular formula of ZnO. According to Noorazlan *et al.*, (2013), zinc oxide has promoted the reduction of melting point and improved the capability of the glass forming during the glass production. It has been informed that the reaction of ZnO in the glass sample can reduce the optical energy band gap and enhance the refractive index. In addition, ZnO can also fill up both network former and network modifier position in the glass network. As a result, the physical properties of such glasses show non-continuous changes when the structural role of the cation changes. Zinc oxide can act as a glass stabilizer in order to produce suitable and good glass system as well.

The combination of zinc oxide and tellurium oxide will form a series of zinc tellurite glass system. Zinc tellurite glass system unveils the favourable and reliable glass-forming ability within a broad region. Nonetheless, this glass formation firmly relies on the cooling rate specifically in the tellurium dioxide rich region. This glass system is suitable to be used as a basis for multi-component optical glass synthesis and a good candidate of composition especially for super heavy optical flint glasses (Noorazlan *et al.*, 2013)

Currently, lanthanide or rare earth oxide group doped glasses have attracted a lot of attentions. The lanthanide doped glasses offers a huge, latest and crucial application especially in the field of optical communication. Different type of lanthanide doped glasses have enhanced towards linear and nonlinear optical properties which are applicable for the devices in the area of photonics in last decades. The refractive index, optical absorption and optical band gap of a material are the crucial optical properties that should be studied before the material is implemented to optical system in the area of photonics (Noorazlan *et al.*, 2013). Therefore, lanthanide or rare earth oxide group is the promising successor to improve the optical properties of tellurite-based glass. Currently, the finding on rare-earth oxide doped tellurite glass has also been widely explored due to their potential applications in several fields, specifically in optical communication.

One of the rare earth oxide groups that are highly recommendable is erbium oxide. Back in the days, a lot of efforts were devoted in order to discover the optical behaviour of the Er^{3+} ions due to its broadband emission at 1.53 µm, which is essential for the applications of optical data transmission (Mahraz *et al.*, 2014). Therefore, erbium-doped glasses have been investigated in a number of studies as it is also applicable in fiber amplifiers of long-range optical fiber communications (Azlan *et al.*, 2015). Mahraz *et al.*, (2014) had also discussed that the trivalent erbium ion is one of the most crucial and efficient ions, and its solubility could achieve up to 0.1 molar fractions. It was figured out that the refractive index of erbium-doped tellurite glasses had the largest value among the functional glasses (Azlan *et al.*, 2015). One of the impressive properties of erbium oxide is their capability of up conversion emission due to their low phonon energy (Sahar *et al.*, 2008). Energy up conversion takes place when infrared or visible radiation with a small energy of light is converted to visible or infrared radiation with larger energy light through multiple transfer or absorption of energy.

Rare earth doped fiber amplifier is used to improve the optical communication. One of the good and preferable candidates of fiber amplifiers is Er^{3+}-doped fiber amplifier (EDFA) devices. The photonic-based system is an attractive application for ultra-high speed transfer and processing. Glass is one of the examples to be utilized in the selected application because of its desirable properties of nonlinear optical (NLO) and low phonon energy (Duffy and Ingram, 1971).

Other than that, silver (Ag) is one of the heavy metal oxides which subsist of special properties such as high refractive index and optical enhancement that have shown its good and suitable element for the preparation of glass-based materials. The aforementioned properties of this element has achieved a peculiar position among the dopants in order to obtain the glassy materials. Hence, the doping of glasses with silver has brought more focus to the scientific society correlate with the materials science area such as photonics and optoelectronics. Silver-doped glasses are the solution for materials to expand its functions in every single aspect such as structural, physical and chemical properties. It forms a variety of attractive characteristics which contribute to branches of advantages. The advantages have included a lot of applications which enhance some phenomena to occur such as silver-glass interaction, diffusion of silver into the glass, and optical response (Gonella, 2015).

1.2 General introduction

In the study done by Alemi *et al.,* (2009), glass has been defined as a homogenous material with a random, liquid-like (non-crystalline) molecular structure. In this process, the raw materials are required to be heated at a sufficient temperature in order to form the entire fused melt. Then, the molten is cooled rapidly; turn to rigid solid without crystallization. Glass which is located at the fourth state of matter connects the rigidity of crystals with the random molecular structure of liquids. The arrangement of the atom in a glass is random but frozen in position. Therefore, the combination of glass can be explained in the aspects of crystalline solid and liquid which can be classified as rigid like a solid but random in the atomic structure of the liquid.

In addition, the development of natural glass is due to the cooling of molten in different compounds including alkaline, alkaline earth, and transition metal oxide. The order of glass formation had been reported by Zachariasen (1932) where a few outlines came out in line with the formation of glass such as:

1) Each oxygen atom is linked to not more than two cations
2) The cation coordination number is small which is 3 or 4
3) Oxygen polyhedral share corners, not edges or faces
4) For 3D networks, at least three corners must be shared

Besides that, the molten must also be cooled under appropriate conditions to allow the production of the glass. Basically, the glass structure consists of random network and amorphous structural arrangement as shown in Figure 1.1.

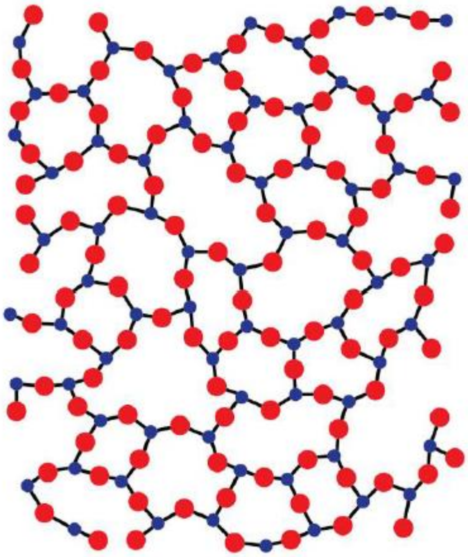

Figure 1.1 : Basic glass structure (Stachurski, 2011)

Although glass does not consist of any crystalline phase, it still has a strong rigidity while holding to the liquid structure. The atoms are connected to one another and form a random structure. In the heating process of liquid, there are two mechanisms exists which are:

1) The atoms' vibration
2) The atoms' and molecules' movement in a random motion

The atoms' vibration or atoms' and molecules' vibration are mainly related to the absorption of energy which is corresponding to the vibrations' frequency. A fundamental vibration is aroused when one such quantum of energy is absorbed by the molecule in its ground state. However, when multiple quanta are absorbed, the first or any higher frequency that is greater than the frequency of a sound are excited.

As the temperature is lowered, the heat energy is slowly reduced until freezing process takes place. The liquid is changed to solid where the structure turns into regular order. However, when the liquid is cooled down from high temperature to room temperature rapidly, the structure becomes rigid but still able to keep the internal structure of a liquid in control. This process is basically known as "supercooled" liquid.

The supercooled liquid state can be defined as a metastable state. At freezing point, the liquid is turned to a solid state where at this point, the internal energy is reduced until it achieves a minimum point and reaches a stable state. However, the supercooled liquid is not in a stable state. Therefore, in order to obtain the stable state, it has to underg o the intermediate state at higher energy. Hence, the supercooled liquid can produce glass whenever the internal energy is provided in which the crystalline state is obtained. The supercooled liquid which has a potential to perform glass (glass-forming liquid) shows speedy increment in viscosity or stiffness as the temperature decreases at below than melting point.

In the area positioned below than melting point, the glass-forming liquids are required to be cooled down swiftly to ensure that the glass-forming liquid does not achieve crystalline phase. Withal, the slow cooling process might give an effect in the production of crystalline phase and yet the glass system formed will not be purely amorphous.

In the process of transition of glass, it is crucial to plot the behavior of the amorphous materials from the supercooled state to glass in a V-T diagram as shown in Figure 1.2. The temperature is conspired in the x-axis and the volume enthalpy employed by the material is conspired along the y-axis, where the T_m is the melting point and T_g is the glass transition temperature. The glass transition temperature arises when the supercooled liquid freezes into an amorphous solid with no hasty discontinuity in volume, nearby the glass transition temperature (El-Mallawany, 2002; Adler, 1971; Vogel, 1985; Yamane and Asahara, 2000; Varshneya, 1994).

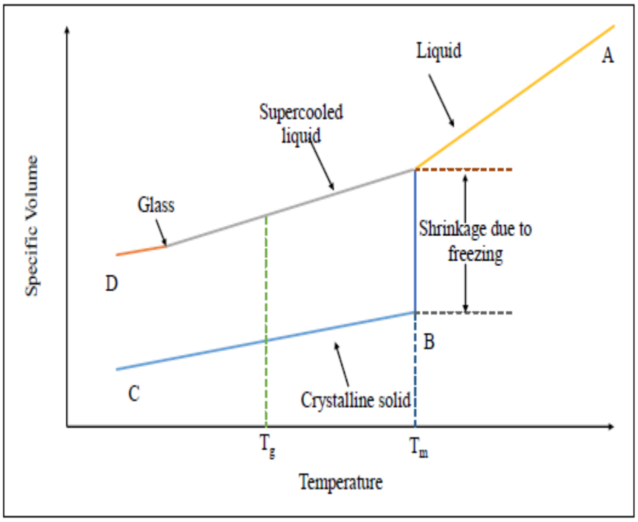

Figure 1.2 : Diagram of glass transition (Stachurski, 2011)

1.3 Problem statements

The crusade of enhancing the properties of glass material for their application in miscellaneous field including the improvement in materials science and technology is an irrepressible challenge for researches all over the world. Until now, many researchers had researched on numerous glass systems with different chemical compositions that have the potency to be employed as fiber optic execution and laser designation, semiconductor devices such as light-emitting diodes (LEDs), non-linear application including optical switching and optical limiting devices, conducting materials, super-capacitor, microwave materials as well as the theoretical approach which is the artificial neural network (ANN) (Eevon *et al*., 2016; Sasmal *et al*., 2014).

It is a notable fact that tellurite based glass is the outstanding glass material to be employed as photonic devices. Tellurite glass has been proposed as the glass with high potential to be exploited in the photonic field, owing to its remarkable 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 are liable to the high polarizability of the overall glass that eventually able to dwindle the energy band gap and deepen the linear and nonlinear refractive index of the respective glass system. Reddy *et al*., (2008) declared that the inclusion of tellurium element in a particular glass system along with the introduction of suitable dopants ions can upgrade the overall transmission capability of the glass as well as expanding the moisture resistance and transparency of the glass in the ultraviolet and infrared wavelength regions.

Various approaches of oxide glass components for instance zinc oxide, lead oxide and tungsten oxide are among the potential candidates for this objective. Hence, the option of choosing a suitable composition is very important since it will affect the chemistry, physical and optical properties of the glass host. Zinc oxide is an element that has a superior glass modifier which will make the glass formation in parent with tellurite glass as a host glass matrix to become smoother (Sidebottom *et al*., 1997).

By having advantages of the tellurite glass system incorporated with zinc oxide, researchers had tried and succeeded in doping rare earth elements into the host glass material by utilizing the conventional melt-quenching method. Unusually, the inclusion of rare earth elements into the host glass material seems to farther improve the optical properties and broaden the promising application of the doped glass such as fiber amplifier, laser, optical data storage, optical communication device and sensor (Akmar Roslan *et al*., 2012).

Notwithstanding, there are little facts on the response of erbium oxide micro-particles to the zinc tellurite glass system. Although zinc tellurite glass system doped with lanthanide element with 4f electrons such as holmium and neodymium has been investigated newly, unprecedented erbium might predispose the comprehensive characteristics as well as properties of the zinc tellurite glass system. Furthermore, the co-doping of erbium and silver element in the same glass system has never been explored by any researchers up until now.

The structural, physical, elastic, thermal, and optical properties of erbium oxide and silver oxide zinc tellurite glass system have been investigated to permit their attainable application categorically in fiber optic and laser implementation. Moreover, the application of glass as potential material for the aforementioned application has never been examined while it's potential to be explored as fiber optic and laser can be prevailed through the photoluminescence effect as well as McCumber and Judd-Ofelt theories. Therefore, the purpose of this research is to study the effect of erbium oxide and silver oxide on structural, physical, elastic (experimentally and theoretically), thermal and optical properties along with McCumber and Judd-Ofelt analysis of zinc tellurite glass system to its possible application as fiber optic and laser designation.

1.4 Scope of study and limitations

The scope of this study is limited to the structural properties (XRD, FTIR and deconvolution), physical properties (density and molar volume), elastic experimental (longitudinal velocity, shear velocity, longitudinal modulus, shear modulus, bulk modulus, Young modulus, Poisson's ratio, hardness, Debye temperature, mean velocity, acoustic impedance, softening temperature and fractal bond connectivity), elastic theoretical (Makishima and Mackenzie model, bulk compression model, ring deformation model and rocherulle model), thermal properties (glass transition temperature, onset crystallization temperature, crystallization temperature, melting temperature, glass stability, thermal stability, reduced glass transition temperature, Hruby parameter, fragility and activation energy), optical properties (optical absorption, energy band gap, Urbach energy, refractive index, molar or electronic polarizability, oxide ion polarizability, optical basicity, and metallization criterion), photoluminescence, McCumber theory (absorption and emission cross-section and gain), and Judd Ofelt analysis.

1.5 Research objectives

The introduction of erbium oxide and silver oxide with variation composition are important to understand the investigated properties in the glass system. Moreover, in this current study, the use of different composition of dopant is necessary to determine the most satisfactory properties for the optical application. Therefore, four objectives have been included in this research work:

1) To determine the effect of erbium oxide and silver oxide on structural and physical properties of zinc tellurite glass system.

2) To study the repercussion of erbium oxide and silver oxide on elastic properties experimentally and theoretically of zinc tellurite glass system.

3) To evaluate the imprint of erbium oxide and silver oxide on thermal and optical properties of zinc tellurite glass system.

4) To investigate the ability of erbium-doped and silver-doped zinc tellurite glass system on producing efficient device for laser application.

1.6 Hypothesis

According to the four specific objectives, the hypotheses for this research are:

1) The inclusion of the dopants into zinc tellurite glass system form glasses with amorphous nature that can be validated via XRD and the formation of glass structure which will form TeO_4 and TeO_3 in the glass system should be detected by the FTIR spectroscopy. However, the presence of other elements such as zinc oxide, erbium oxide and silver oxide can also be discovered by doing deconvolution process via Origin 6.0 software, if only the elements cannot be detected by the instrument due to low amount of concentration.

2) The density values are expected to be increased as heavier dopants are added into the glass matrix. Nevertheless, there is still a possibility for the molar volume not to obey the theoretical relationship which is inversely proportional to each other due to several reasons such as longer bond length and larger interstices space within the glass structure.

3) The addition of different dopants into the glass samples are expected to enhance the elastic properties of the glasses by increasing the ultrasonic velocity, elastic moduli and other elastic parameters. This is because rare earth and heavy metal ion can cause the formation of bridging oxygen that will increase the rigidity of the glass samples. The inconsistency of the trend might be due to the concurrent presence of bridging oxygen and non-bridging oxygen that can be proven from the FTIR and deconvolution analysis.

4) The thermal properties such as glass transition temperature, glass stability, thermal stability, reduced glass transition temperature and Hruby parameter are predicted to increase but opposely to the fragility value. The effect of dopants in the glass system can enhance the glass sample to become either stiffer or brittle.

5) The linear optical properties parameters for zinc tellurite glass system with various dopants can be constant in trend either increase or decrease. This might be due to the presence of dopants in the glass system that might act as a glass modifier or glass former. When the dopants play a role as a glass modifier, there is a possibility for the creation of more polarizable non-bridging oxygen which contributes to the reduction in energy band gap as well as the increment in linear refractive index. The results can also be distinguished if the dopants act as a glass former in the glass system.

6) The emission of photoluminescence from the dopants of erbium oxide and silver are anticipated to exist at certain transition levels of the zinc tellurite glasses.

7) The absorption and emission cross-section is investigated based on different types of dopants and concentration. High emission cross-section has a tendency to produce more gain in a laser application.

8) The values of Ω_2 and Ω_4 parameters are expected to correspond to the asymmetry of the local environment of Er^{3+} ions sites which depend on the covalency between Er^{3+} ions and ligand anions. Meanwhile, the value of Ω_6

parameter is linked to the local basicity of the Er^{3+} ions and inversely proportional to the covalency of the Er-O bond.

1.7 Significance of study

In these days, the usage of glass is not only restricted for the things that can be seen by naked eyes such as windows, building and furniture instead it is comprehensively can also be applied in optoelectronic, laser and fiber optic. A few studies on the rare earth and heavy metal elements are investigated in order to encourage the properties of the glasses, for instance the optical properties. Since then, huge interests and passions of the researchers of exploring more in order to make good glass with better properties is very encouraging.

In determining good parameters for laser application, some major properties such as elastic, thermal and optical properties are investigated as well. The investigation of the respective properties would determine the best glass sample to be used in laser application. Therefore, the outcome of this study may provide deeper knowledge about the properties of the new composition of glasses towards the laser application. This research can be a reference for further research on elastic, thermal and optical properties of both rare earth and heavy metal doped glasses in the educational field and also in industrial applications.

1.8 Outline of thesis

Chapters are divided into few sections as well as the sub-sections. Chapter 1 is the overview in the field of research work and brief introduction of glass materials. It also concentrates on the finding of the research and highlights the objective of the investigation. The relevant points are also listed in the problem statements. Chapter 2 reports the earlier information regarding the related field of research. It also mentions about various views and some approaches from previous researchers which are related in this study. Chapter 3 outlines the theory that being implied in this investigation. The derivations of equations are also included in this chapter. Chapter 4 explains deeply about the method of fabrication and collection of data. The overview of the instrument used is also described in this section. Chapter 5 discusses in details regarding the results obtained in this study and the comparison among the glass series. The discussions cover a few types of measurement such as X-ray diffraction (XRD), Fourier transform infrared (FTIR), deconvolution analysis, density, molar volume, ultrasonic technique, Differential scanning calorimeter (DSC), UV-Visible spectroscopy (UV-Vis), photoluminescence spectroscopy, McCumber theory and Judd Ofelt analysis. Chapter 6 summarizes the crucial result of the finding and recommendation for the future works.

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