UNIVERSITI PUTRA MALAYSIA

ELASTIC PROPERTIES OF EUROPIUM ZINC BOROTELLURITE GLASS SYSTEM DOPED WITH SILVER OXIDE

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By

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DEDICATION

This work is a tribute to my parents., Saharudin bin Mohd Ali and Siti Nor Afizah binti Nurruddin, who have always encouraged and taught me not to give up, as well as given me advice on how to improve myself and become a better person. I also dedicate this work to my family, who always accept and love me for who I am.

ABSTRACT

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Supervisor : Prof. Dr. Ts. Halimah Mohamed Kamari

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Melt quenching was employed to produce the chemical composition of a europium zinc borotellurite glass system series treated with silver oxide of $\{\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{0.96} (Eu_2O_3)_{0.04}\}_{(1-x)} (Ag_2O)_x$ where x = 0.0011, 0.0012, 0.0013, 0.0014, and 0.0015 molar fraction. Glass was distinguished using density, molar volume, X-ray diffraction (XRD), and elasticity studies. As the concentration of silver oxide increases, while the molar volume increases, the density will decrease. The glass sample's XRD spectra reveal that it is amorphous. Ultrasonic measurements was used to assess the elastic properties of glass samples of longitudinal and shear velocity. These values are then entered into the equation to determine the Young's modulus of the glass sample.

Elastic moduli such as longitudinal modulus, shear modulus, bulk modulus, and Young's modulus increase steadily with increasing silver oxide concentration. The presence of bridging oxygen causes this phenomenon, which improves the rigidity and strength of the glass system. Other physical properties, such as Poisson's ratio, microhardness, and fractal bond connectivity, exhibit a fluctuating trend as the compactness of the glass system changes, whereas softening and Debye temperature exhibit an increasing trend as the stiffness of the glass system increases. It is claimed that the effect of Ag_2O content on the elastic properties of glasses is beneficial for a variety of functional applications in the glass industry, including glass polishing, window glass, and optical lenses.

ABSTRAK

SIFAT–SIFAT ELASTIK SISTEM KACA EUROPIUM ZINK BOROTELURIT DOP DENGAN ARGENTUM OKSIDA

Oleh

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Lebur pelindapkejukan telah digunakan untuk menghasilkan komposisi kimia europium zink borotelurit siri sistem kaca dirawat dengan argentum oksida $\{\{ [(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{0.96} (Eu_2O_3)_{0.04} \}_{(1-x)} (Ag_2O)_x$ di mana x = 0.0011, 0.0012, 0.0013, 0.014 dan 0.0015 pecahan molar. Ketumpatan, isipadu molar, pembelauan sinar-X (XRD) dan ukuran elastik diukur untuk membezakan sampel kaca yang disediakan. Dengan peningkatan kepekatan argentum oksida, manakala jumlah molar meningkat, ketumpatan jatuh. Sampel kaca Spektra XRD menunjukkan bahawa mereka adalah sifat amorfus. Pengukuran ultrasonik digunakan untuk menilai sifat elastik sampel kaca halaju membujur dan ricih. Nilai – nilai ini kemudian dimasukkan ke dalam persamaan untuk menentukan modulus Young sampel kaca.

Moduli elastik, yang terdiri daripada modulus membujur, modulus ricih, modulus pukal, dan modulus Young, menunjukkan corak peningkatan yang mantap dengan peningkatan kepekatan argentum oksida. Fenomena ini disebabkan oleh kehadiran oksigen penyambungan, meningkatkan ketegaran dan pengukuhan sistem kaca. Ciri – ciri fizikal lain seperti nisbah Poisson, kekerasan mikro dan keterkaitan ikatan fraktal juga menunjukkan corak peningkatan atau penurunan akibat perubahan ketumpatan sistem kaca, manakala untuk melembutkan dan suhu Debye ia menunjukkan hasil corak pertambahan akibat peningkatan kekerasan kaca. sistem. Pengaruh kandungan Ag_2O pada ciri – ciri elastik kaca ditegaskan bermanfaat untuk pelbagai aplikasi berfungsi dalam industri kaca seperti kaca penggilap, kaca tingkap, kanta optik dan lain – lain.

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I'd like to express my heartfelt appreciation to everyone who helped and supported me with my final year project. This gratitude is especially dedicated to my supervisor, Prof. Dr. Ts. Halimah Mohamed Kamari, for her complete guidance, encouragement, and advice in order for me to complete my thesis project.

Aside from that, I would like to express my gratitude to all PhD and master students in the glass research laboratory, particularly Mr Sulaiman and Dr Nazirul Nazrin, for devoting their time to share knowledge, guidance, and unending assistance with material related to my topic, which I worked on for my final year project.

Most importantly, I want to give special thanks to my family for always encouraging me to complete my thesis even when I was going through a difficult time. They continue to encourage me and love me unconditionally. This would not have been possible without them. Thank you very much.

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Thank you to all of you.

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LIST OF ABBREVIATIONS

BO_4	Tetraborate
TeO_2	Tellurium oxide
Te	Tellurium
B_2O_3	Boron oxide
ZnO	Zinc oxide
Zn	Zinc
Eu_2O_3	Europium oxide
Eu	Europium
B	Boron
Ag_2O	Silver oxide
Ag	Silver
Bi_2O_3	Bismuth (III) oxide
TeO_4	Trigonal bipyramid
NaCl	Sodium chloride
CaO	Calcium oxide
SiO_2	Silicon dioxide
ASTM	American Society for Testing and Material
NBO	Non-bridging oxygen
BO	Bridging oxygen
FTIR	Fourier Transform Infrared
XRD	X-ray Diffraction
PbO	Lead oxide

BO_3	Triborate
ρ	Density
V_L	Longitudinal velocity
V_S	Shear velocity
L	Longitudinal modulus
G	Shear modulus
K	Bulk modulus
E	Young's modulus
$\boldsymbol{\sigma}$	Poisson's ratio
H	Microhardness
d	Fractal bond connectivity
T_s	Softening temperature
Θ_d	Debye temperature

CHAPTER 1

INTRODUCTION

1.1 Background of study

Among the different type of glasses, borosilicate glass is one of the most cost- effective to manufacture. Window mirrors, glass ceramics, optical and optoelectronic devices, and mechanical and thermal sensors are among the Bi_2O_3 and Eu_2O_3 glasses applications that have piqued curiosity. Tellurite oxide is recognized as the closest contender to heavy metal compounds due to its unique optical and electrical features. Tellurite-based glass has the best compatibility and solubility with other oxide glasses of all the heavy metals. Meanwhile, in borotellurite glasses, the presence of two network formers, such as TeO_2 and B_2O_3, can cause complex speciation in the glass structure and the development of mixed structural units (Kaur and Khanna, 2014). Europium oxide dopant was used for this study to evaluate its impact on the elastic characteristics of zinc borotellurite glass and silver oxide co-dopant for zinc properties improvement.

Therefore, the advantages of adding europium oxide into the zinc borotellurite glass system are electronic polarizability, refractive index, reflection loss, and optical basicity may all be improved. Then, for the silver oxide, it can assist in the dissolution of a weak link. Furthermore, elastic characteristics have shown the material’s stress-strain relationship. El-Mallawany *et al.*, (2006) mentioned the elastic properties of a material are very helpful in understanding its structure as they can be related directly to the interatomic potential. The longitudinal and shear velocities, as well as the calculation of additional elastic constants such as the elastic moduli, Poisson's ratio, microhardness, softening temperature, and Debye temperature, will be the emphasis of this research.

1.2 Glass definition

Glass is a term that frequently heard in everyday conversation. It is usually used to describe a clear substance with qualities such as hardness, rigidity, or even brittleness. Many other interpretations of glass have been presented over the years, including one from the US National Research Council, which glass is described as an x-ray amorphous substance that may display the glass transition. This phenomenon can be determined in which the temperature is visible in the solid amorphous state changes upon heating and changes the heat capacity and expansion coefficient are examples of derivative thermodynamic characteristics. Glass has a structure that is similar to the parent supercooled liquid (Zanotto and Mauro, 2017). Apart from that, the glasses appear to be solid on a human time scale, yet they will flow at any temperature when subjected to gravity. Aside from that, glass is an out-of-balance substance that is constantly relaxing towards a supercooled liquid state (Welch *et al.,* 2013).

On the other hand, Kolb *et al.,* (2000), asserted that glass is depicted as a physical condition rather than a specific composition. Glass is a stiff substance, according to them, since it has the extrinsic qualities of a solid despite being in a less organised structure, making it more similar to the attributes of a liquid. Glass in solid form exhibits non-crystalline, liquid- like behavior and lacks long-range periodicity. Welch *et al.,* (2013) also reported that the properties of the glass are affected by the entire thermal, composition, and pressure histories. Traditionally, glasses are formed by cooling them from the melt, and they are inorganic and non- metallic. According to Hasanuzzaman *et al.,* (2016), the American Society for Testing and Materials (ASTM) defined glass is as an inorganic fusion product that has cooled to a hard state without crystal formation.

Powderless non-fusion glass processing has recently been introduced as a result of the oxide materials may now be synthesized using a modern solution-based sol-gel method, which can occur at substantially lower temperatures traditional solid-state fusion methods operate as demonstrated by several X-ray and electronic diffraction studies. As a result, Varshneya *et al.,* (2010) proposed that glass be defined as a solid with a non-crystalline structure that will continuously convert to a liquid upon heating. Zarzycki (1996), on the other hand, came up with a more straightforward definition of glass which is a non-crystalline material that shows the glass transition phenomena. As a consequence, both criteria were successful in categorizing non-crystalline materials into two groups. Glass and amorphous materials were present.

1.3 Glass application

Black volcanic glass, commonly known as obsidian glass, is one of the oldest man-made glasses found in Anatolia, Turkey's south central region. It is mostly made up of granite that melted during volcanic activity but did not recrystallize when it cooled. It also has a convex surface and an impressive optical quality. Obsidian glass was one of the first materials exported from Anatolia (Enoch, 2006). It was used for spears, arrowheads, knives, axes, scrapers, and jewellery.

Shelby (2005), on the other hand, stated that the application of glass existed around 7000 Before Century through the combination of sea salt (NaCl) with either bone (CaO) or fire residues built on the sands (SiO_2). Several ways for producing shape glass, such as bottles, beads, tools, glass jars, and glass containers, have been discovered, including winding and cooling. The invention of the blowing method of glass production improves glass quality, such as clear sheet glasses without bubblesor other flaws, and it is also coloured.

Each of these glasses has unique properties that allow for a wide range of applications. Lead glass, for example, is a form of glass created by substituting lead oxide (PbO) for the lime and soda in regular glass to generate a glass with a lower melting point and greater refractive index. Lead glass is also extremely electrically resistant. As a result, it is widely used in electrical devices such as the cathode ray tube in televisions (Kolb *et al.,* 2000).

Laboratory glassware, domestic cooking utensils, and automotive headlights can all benefit from glasses having a low thermal expansion coefficient and a good chemical resistance (Gaafar and Marzouk, 2007). The type of glass with this mechanical qualities and structure is borosilicate. It can also withstand temperature shock and has greater chemical durability. Aluminosilicate glass, like borosilicate glass, is resistant to heat shock and chemical deterioration and can be used directly as a heating element.

The growing interest in art glass is another notable development in the field of glass production. According to Kolb *et al.,* (2000), glassmaking art peaked around 5,000 years ago under the Roman empire, when numerous great pieces of glass art were created. The spectacles have became such a valuable antique that the British Museum now owns them. There are now glass schools and private hot-glass workshops all over the world, offering courses in glassblowing, flameworking, and hot- glass sculpting.

1.4 Components in europium zinc borotellurite glass system doped with silver oxide

Several elements make up a europium zinc borotellurite glass that is doped with silver oxide which are zinc oxide (ZnO), boron oxide (B_2O_3), tellurium oxide (TeO_2), europium oxide (Eu_2O_3) and silver oxide (Ag_2O). Each of these elements have their own influence towards the properties of the zinc borotellurite glass.

1.4.1 Zinc oxide

Zinc oxide (ZnO) has been one of the most essential industrial materials for centuries, and it has piqued the interest of manufacturers. This is owing to a mixture of physical features including high temperature stability, high electrical and thermal conductivity, and ultra-violet optical absorption. Zinc oxide is also said to be capable of stabilising the components to which it is added (Usman *et al.,* 2018). Consequently, adding zinc oxide to a borotellurite glass network system is said to improve the glass's stability. It also borotellurite glass network system is said to improve the glass's stability. It also improved glass forming capabilities and ensured low crystallisation rates in the glass system. This is due to its ability to enter the glass network as either a glass former or a glass modifier.

Zinc oxide is an essential multifunctional material since it is also known as a broad band gap semiconductor with unique chemical surface and microstructural features (Usman *et al.*, 2018). Zinc oxide has the capacity to lower the optical band gap by increasing its refractive index, which will eventually improve the optical properties of a glass. Other common uses for zinc oxide include gas sensors, transparent electrodes, and catalysts. According to Sayyed (2016), zinc oxide is an intriguing glass forming since its presence in high quantities in glass will affects the features of the glass, such as low melting temperature.

1.4.2 Boron oxide

Boron oxide which is a chemical compound has molecular formula, B_2O_3. It can be used as a glass type former and can also form glass by itself. Boron oxide has a number of properties, according to Hasnimulyati *et al.*, (2016), including good rare-earth ion solubility, a low melting point, and a high phonon energy. Boron oxide has been widely used as a glass forming material since it can make a glass on its own. The ability of boron to form glass is influenced by its bond elongation, decreased size of cation, fusion heat is lower, and trivalence. Apart from these characteristics, boron oxide has a lot of potential in terms of technological applications, and it's used in a lot of large-scale industrial glasses (Khanisanij and Sidek, 2014).

According to Usman *et al.,* (2018), borate glasses are made up of triangle units known as BO triangles, which indicates they are randomly corner bonded. The BO triangles will be joined by a B-O-B connection to generate six (6) members (boroxol) rings. The boroxol rings will be changed into complicated borate groups such as diborate, triborate, and tetraborate when other metal oxides are added to the glass system. The gathering of BO_3 triangles and BO_4 tetrahedra that are held (possessed) by the borate matrix will also impact the complex borate groups, indicating that the addition of metal oxides to the glass system will produce modifications to the structural unit of the glass.

1.4.3 Tellurium oxide

Tellurium oxide (TeO_2) is a thermally stable element used in glass, optics, and ceramics. Since of their remarkable features, tellurium oxide-based glasses are now receiving scientific and technological interest. According to Faznny *et al.*, (2018), a tellurite-based glass has become one of the best components for synthesising photonics devices because of its unique features. Thermal imaging, fibre laser production, infrared laser power transmission, laser radar windows, aerial reconnaissance and surveillance, and many other uses are all possible with these glasses (Sayyed *et al.*, 2016).

Besides that, Halimah *et al.,* (2010) have mentioned on the TeO_4 trigonal bipyramid (tbp) basic structural unit, which consists of a lone pair of electrons and requires a Te-O-Te connection to produce a glass. Furthermore, it has unique qualities like thermal stability, chemical durability, high refractive index, and high solubility of rare earth ions, making it an excellent choice for optical device applications (Hasnimulyati *et al.*, 2016).

Tellurium oxide, on the other hand, is said to be a conditional glass forming by certain researchers (Nazrin *et al.*, 2018). This means that it cannot form a glass in normal circumstances unless other components or a modifier ion are added to it under specific settings (especially extreme ones), for as by altering the quenching rates during manufacturing. As a result, adding oxides to tellurite glasses is commonly recommended to improve the glass' forming capacity.

1.4.4 Europium oxide

The chemical compound europium (III) oxide (Eu_2O_3) is made up of europium and oxygen. It's frequently used in television sets and fluorescent lamps as a red or blue phosphor, as well as an activator for yttrium-based phosphors. It also acts as a catalyst in the production of fluorescent glass. The optical and spectroscopicproperties of Eu_2O_3 doped calcium boroaluminate glasses with Eu_2O_3 up to 10% wt% (Malo *et al.*, 2020). The electronic polarizability, refractive index, reflection loss, and optical basicity are all rise when Eu_2O_3 is added.

Besides that, after the addition of europium, small changes in the FTIR of the examined glass may be seen in the IR spectra. According to Pascuta *et al.*, (2020), rare earth glasses with the vitreous host glass matrix rigidity of bismuth borate is relatively high. As a result, the existence of europium ions in the substance tends to favor the creation of BiO_3 units by influencing the surrounds of the Bi^{3+} cations. When europium ions with greater charges and coordination numbers are introduced, a closely packed glass structure develops.

1.4.5 Silver oxide

The chemical composition Ag_2O stands for silver(I) oxide. It is a fine dark brown or black powder that is used to make various silver compounds. Although a revolutionary form of glass containing both rare earth (RE) and silver ions has just recently been produced, it has sparked a lot of curiosity. Surface plasmonic resonance (SPR) absorption bands in Ag nanoparticles (Ag NPs) are mainly found in glass as single Ag^+ ions (or Ag atoms), molecular-like Ag nanoclusters, or Ag nanoparticles (Ag NPs). According to Liu *et al.*, (2018), the distribution of the Na^+ ion and bridge oxygen in boron- rich and silicon-rich phases was shown to be affected by Ag^+ concentration, during the crystallization process, the BO_3 triangles transform into BO_4 tetrahedra. In addition, at a particular hour, the doped Ag^+ ion concentration attained its solubility in borosilicate glass.

Thus now, several research have been conducted to examine the mechanism of Ag NP formation in glass, as well as associated factors such as Ag doping concentration, glass host composition, and subsequent thermal treatment. Silver oxide additionally improves the characteristics of the glass network (Fudzi *et al.,* 2018). The presence of silver oxide in the glass sample results in a greater spectroscopic property when compared to the comparable glass without silver. The distribution of the Na^+ ion and bridge oxygen in boron-rich and silicon-rich phases was demonstrated to be changed by Ag^+ concentration, resulting in the crystallization of BO_3 triangles to BO_4 tetrahedra (Liu *et al.,* 2018).

1.5 Ultrasonic

The ultrasonic method was used to study the practical and theoretical approaches to elastic characteristics. Ultrasonic waves are used to test the ultrasonic measurement in the tested substance. To deliver sound into particular places, an ultrasonic transducer is utilized, which transforms electric signals from the machine into sound waves, which may then be transformed back into electrical impulses. A glass's microstructure, characterisation, mechanical properties, phase transitions, and elastic constants may all be determined using ultrasound (Eevon *et al.,* 2016).

The material’s ultrasonic nondestructive character is a useful instrument for detecting changes in materials properties such as microstructure, deformation process, and mechanical properties across a wide temperature range. Apart from that, this can be explained by the strong relationship between ultrasonic waves and the elastic and inelastic characteristics of materials. The information obtained from the ultrasonic propagation velocity mode assists in determining a set of elastic constants that can be used to calculate a variety of elastic properties of a material, including longitudinal, shear, bulk, and Young’s moduli (Hasnimuyalti *et al.,* 2016).

According to Franco *et al.*, (2011), the precision of mechanical testing in relation to the quality of the experiment setup is crucial in determining the variation of reported values. Aside from the mechanical test, variable materials qualities have an impact on the claimed value of different manufactures. For example, to explore some of the mechanical elements and standard materials used for the calibration of test equipment in quality control, an accurate mechanical test is required. As a result, ultrasound technique is one of the most suited method for meeting all requirements, as it uses the propagation velocity of waves through a solid medium, which is dependent on the solid elastic properties.

1.6 Problem statement

The features of zinc borotellurite-based glass have recently attracted the curiosity of academics who want to learn more about them. This is owing to the intriguing qualities that this glass possesses. Some researchers have also attempted to inject dopants into this glass in order to gain better understanding its structural, physical, and elastic properties. Most of the dopants used in this glass came from one of two categories which is transition metals (TM) ions or rare-earth (RE) ions. However, no one has looked into the effect of silver oxide on elastic properties of zinc borotellurite glass. But there is information that silver oxide can result in a glass network's elastic characteristics being improved. Consequently, several glass samples with the chemical composition of $\{\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{0.96}(Eu_2O_3)_{0.04}\}_{(1-X)}(Ag_2O)_x$ and different molar fractions, x = 0.0011, 0.0012, 0.0013, 0.0014 and 0.0015 were fabricated in this work to investigate silver oxide's effect on the characteristics of zinc borotellurite glasses. The glass samples are characterised by performing physical and structural analyses on them.

1.7 Objectives of the study

The goal of this research is to investigate the effect of silver oxide on the elastic characteristics of a europium zinc borotellurite glass system. In order to achieve the aim, there are several objectives that have been identified:

I. To synthesize europium zinc borotellurite glass doped silver oxide with different molar fractions, x = 0.0011, 0.0012, 0.0013, 0.0014 & 0.0015.

II. To study the influence of silver oxide on the structural and physical properties of the europium zinc borotellurite network.

III. To identify the influence of silver oxide on the elastic properties of europium zinc borotellurite glasses.

1.8 Chapter organization

This thesis had been organized and divided into five chapters. The content for Chapter 1 is about the introduction and background of the project such as the definition, application and components used in this project. Meanwhile, Chapter 2 includes a literature review focused on the project's relevant topics, with references of the published publications. Chapter 3 describes the methodology used to create a zinc borotellurite glass system doped with europium oxide co-doped with silver oxide. Other than that, Chapter 4 will be covered on the results of the project and discussion as well. Finally, Chapter 5 contains the project's conclusion as well as some recommendations for further research in this area.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviewed some of the existing literature relevant to the study's components and methods, as well as some of the research linked to the glass system's elastic properties, which is the study's goal.

2.2 Components

The main component used in this study is europium zinc borotellurite but was further enhanced by doping it with silver oxide before analysing its elastic properties.

2.2.1 Zinc borotellurite

As mentioned in Chapter 1, there is very little literature on zinc borotellurite used as a component in a glass system because interest in this research is relatively new. According to Pavani *et al.*, (2011) and Usman *et al.* (2018), adding zinc oxide (ZnO) to the combination of components improves the quality and stability of the glass system (in particular its forming capability) by reducing crystallization rates. Apart from the aforementioned research, investigations on the properties of zinc borotullerite-based glass are still of interest, since this component has shown promising results when experimented with or doped with various elements or dopants.

The laminar network structure of tellurite glass is based on triangular TeO_3 pyramids or square TeO_4 pyramids, (Halimah *et al.,* 2007). The laminar network in the glassy form of boron oxide (B_2O_3) is made up of boron atoms that are 3-fold coordinated with oxygen. The presence of oxygen leads the network to transform from a 3-fold BO_3 to a 4-fold BO_4. Bridging oxygen is a network that is responsible for increasing the connectivity of the glass network. The glass-forming oxides are TeO_2 and B_2O_3. In borotellurite glasses, both TeO_4 and BO_4 units have asignificant tendency to correlate with one another, resulting in increased glass network connection. Aside from that, due to the transformation of TeO_3 and BO_3 units, the stiffness of these glasses increases with TeO_2.

Tellurite glasses have a low phonon energy compared to phosphate and silicate glasses, strong thermal and mechanical stability, large infrared transparency, and strong chemical durability, (Paz *et al.,* 2016). Tellurite glasses have a low melting temperature, a high dielectric constant, and a high refractive index, among other properties. Tellurite glass is an excellent for photonic applications such as optical communication, window materials, and up-conversion lasers. When tellurium oxide is added to another glass formation, such as borate glass, the phonon energy is reduced while the refractive index and IR transmission are increased, making borotellurite glasses suitable for optical applications.

2.2.2 Europium zinc borotellurite doped with silver oxide

Europium oxide is often used as a trace element in the glass industry for certain types of glass, typically involving borotellurite type glasses. The optical absorption spectrum of europium oxide is such that it blocks wavelengths ranging from 300 to 400 nm with high efficiency. For this reason, europium oxide is an ideal additive to glasses designed for its use in solar cells or other devices where undesirable spectral characteristics can interfere with the material's functionality.

According to Saudi *et al.*, (2020), europium oxide helps catalyze a chemical reaction that is responsible for zinc becoming a colourless gas and boron nitride becoming a black gas. They combine to create glass doped with zinc borotellurite. This glass then fluoresces an eerie green light. Europium oxide also has applications in controlled nuclear fusion reactions, high-temperature superconductivity, and as phosphors in organic light-emitting diodes. Based on study conducted by Binnemans *et al.*, (2018), europium oxide was already included in glass as a red dopant in the 17th century. In the early 1800's, boron was added to glass to create fluorescent light, but europium oxide remained highly sensitive and relied on ultraviolet light for illumination.

Glass structural analysis was used to investigate the effect of Ag^+ ions on the glass forming and crystallisation processes. Aggregated Ag NPs can operate as a nucleating agent for the crystallisation of stable oxide components in borosilicate glass when the doped Ag+ ion concentration is higher than its solubility in the glass. Apart from the above studies, studies focusing on the properties of zinc borotullerite-based glass continue to be of interest as this component has shown promising results when experiment or doped with different elements or dopants. For example, Fazzny *et al.*, (2018) found that when this component was doped with lathanum and silver oxide (La_3 $^+$and Ag^+), the optical characteristics of the lanthanum zinc borotellurite-based glass were improved by adding silver oxide to the glass matrix. In addition, the doped Ag^+ ion concentration attained its solubility in the borosilicate glass at a certain point.

2.3 Elastic properties

Elastic characteristics are inextricably linked to the solid's interatomic potential and structure. Longitudinal and shear velocities are two separate elastic constants that make up elastic characteristics. The ultrasonic measurement of longitudinal and shear velocities can yield these two values (Halimah *et al.*, 2005). At room temperature, longitudinal and shear velocities of borotellurite glass samples were measured using a 5 MHz frequency. The measured density and ultrasonic velocity can be used to derive elastic characteristics, Poisson's ratio, and hardness. Due to the stiffness of the glass samples, the elastic moduli and hardness of borotellurite glasses rise as the TeO_2 content increases, which can be attributed to the development of bridging oxygen from the production of TeO_3 and BO_4 in the glass network. Furthermore, an increase in TeO_2 content causes a decrease in the Poisson' ratio from 0.2576 to 0.1952, indicating an increase in the glass system's crosslink density.

According to Azianty *et al.,* (2012), ultrasonic velocity can be used to get non-destructively the longitudinal and shear modes for elastic characteristics of glass. In order to determine all the parameters under elastic characteristics, sound velocity serves as an intermediary component. Because it offers information on stiffness, structural alteration of the glass network, and crosslink density, the ultrasonic approach is highly beneficial for glasses. The creation of bridging oxygen and the glass network alteration on the stiffness and elastic characteristics of the glass can be determined using FTIR or Raman spectroscopy in combination with ultrasonic analysis.

2.4 Density and molar volume

According to Sidek *et al.,* (2012), the density and molar volume of bismuth lead germanate glasses vary monotonously and slowly as the Bi_2O_3 concentration increases. Due to changes in atomic mass and atomic of volume constituent components, the addition of Bi_2O_3 induced differences in density. The increase in density was matched by an increase in molar volume, indicating that the glass network had shifted structurally. The elastic moduli all follow the ultrasonic wave velocity's pattern. Elastic moduli increase as Bi_2O_3 concentration increases over the whole composition studied. Increases in elastic moduli are caused by an increase in cross-link density, and hence an increase in the stiffness of glass samples. A decrease in Poisson's ratio, according to Higazi and Bridge (1985), indicates an increase in glass cross-link density.

According to a study by Bootjomchai *et al.*, (2014), the average tensile force constant, average ring size diameter, and average crosslink density all contribute to the increasing and decreasing trends in elastic properties and glass density. The ratio between the lowest theoretical volume occupied by ions and the equivalent effective volume of glass is known as the glass packing density. An increase in the amount of non-bridging oxygen molecules is often related with a drop in ultrasonic velocity, resulting in a decrease in glass network connections. Modifiers like TiO_2, BaO, and Bi_2O_3 change the structure of the material by producing non-bridging oxygen.

In addition, according to Laoding *et al.,* (2016), the density increase with the increment of thulium(III) oxide. This is because Tm_2O_3 has a higher molecular weight than the weight of all other chemical compounds combined. Density rises because it is directly proportional to mass. Furthermore, the additional ions may fill the network's interstices, causing the network to become compact. It was discovered that the ultrasonic velocity increased, indicating that the glass network was strengthened and the sample's rigidity increased. Additionally, it contributed to an increase in packing density. However, when the concentration of Tm(III) ions is high, it causes more non- bridging oxygen to be produced than boron, lowering the ultrasonic velocity. According to the studies, the elastic moduli show an increasing trend as the concentration of Tm(III) ions increases. This can be attributed to the increased connectivity of the glass network.

2.5 X-ray diffraction

The glass sample was investigated using an X'Pert PRO MPD diffractometer, (PANalytical, Philips) (Matori *et al.,* 2017). The measurements are carried out by Ni- filtered Cu-Kα radiation (λ = 0.1542 nm) and an X-ray source with a power and current of 40 kV and 40 mA, respectively. Matori *et al.,* (2017) also highlighted that the glass sample is amorphous in nature. The X-ray diffraction pattern of the investigated glass, lead zinc phosphate glasses, similarly reveals no distinct or continuous sharp peaks (broad hump), indicating that the glass sample is amorphous solids (glassy phase).

Epp *et al.,* (2000) discovered that the ability of crystals to diffract X- rays in the XRD method frequently influenced the structure of the crystalline phases under investigation. As a result, the diffraction patterns obtained using this technique are made up of a variety of micro- and macro-structural characteristics of a sample. The peak position acquired from the XRD spectrum can also be used to investigate lattice characteristics, space group, chemical composition, macrostresses, and qualitative phase analysis. In addition, the peak intensity can be used to determine information about the crystal structure, such as atomic locations or occupancy.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discusses how to synthesize a glass sample such that a succession of europium zinc borotellurite glass systems doped with silver oxide may be created. The glasses are prepared by using high purity analytical chemicals of TeO_2, B_2O_3, ZnO, Eu_2O_3 and Ag_2O with composition of $\{\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{0.96}(Eu_2O_3)_{0.04}\}_{(1-x)}(Ag_2O)_x$. The molar fraction of x is equal to 0.0011, 0.0012, 0.0013, 0.0014 and 0.0015. Chemical oxides of tellurium (IV) oxide, TeO_2 (Alfa Aesar, 99.99%), Boron oxide, B_2O_3 (Alfa Aesar, 98.50%), Zinc oxide, ZnO (Alfa Aesar, 99.99%), Europium (III) oxide, Eu_2O_3 (Alfa Aesar, 99.99%) and Silver oxide, Ag_2O (Alfa Aesar, 99.99%). The glass sample will be characterised in terms of elastic properties, in order to understand the details information about the stress-strain relationship in the glass.

3.2 Glass preparation

3.2.1 Weighing of chemical

Melt quenching was used to make a europium zinc borotellurite glass system doped with silver oxide sample. The glass samples were weighed and proportioned according to their molar fraction values. **Table 3.1** shows the required amount of TeO_2, B_2O_3, ZnO, Eu_2O_3 and Ag_2O powder, creating a 13 g of glass sample.

Table 3.1: Weight of each chemical element in the prepared glass.

Molar fraction of silver oxide, x	Weight of TeO_2, (g)	Weight of B_2O_3, (g)	Weight of ZnO, (g)	Weight of Eu_2O_3 (g)	Weight of Ag_2O (g)
0.0011	7.6920	1.4380	2.4016	1.4423	0.0261
0.0012	7.6906	1.4377	2.4012	1.4420	0.0285
0.0013	7.6892	1.4374	2.4007	1.4417	0.0309
0.0014	7.6878	1.4372	2.4003	1.4415	0.0333
0.0015	7.6864	1.4369	2.4000	1.4412	0.0356

To begin, tellurium (IV) oxide, boron oxide, zinc oxide, europium (III) oxide, and silver oxide were precisely weighted with a digital weighing machine with an accuracy of 0.0001 g to get the required proportion for making the glass sample. After that, the chemical mixtures were transferred to an alumina crucible, where they were properly mixed. The liquid was then stirred with a glass rod for 20–30 minutes to ensure that the chemicals were uniformly distributed throughout the liquid.

3.2.2 Pre-heat process and melting process

For the pre-heating procedure, the mixed chemical mixture was placed in the furnace at 400℃ for one hour. To ensure that the combination is devoid of water vapour, it was pre-heated. After one hour, the crucible was transported to the second furnace, where it underwent a two hour of melting procedure at 900℃. The melting process was used to verify that the chemical granules completely reacted.

3.2.3 Glass casting

As the powders was still in the melting process in the second furnace, a stainless steel split mould was cleansed and polished before being utilised in the glass casting process. Before pre-heating the mould, it must be polished using sand paper to eliminate impurities and dust. The purpose of the pre- heating phase for the stainless steel mould was to prevent thermal shock to the glass sample during the casting process.

Glass casting is the technique of pouring molten metal into a mould. To avoid solidification before all of the molten has been poured into the stainless steel mould, the liquid must be transported quickly into the mould. If the melt cooled too quickly, stress built up in the glass, resulting in the breaking of the glass. On the other hand, crystal will begin to form within the glass, resulting in a murky glass with poor optical qualities.

3.2.3 Annealing

The annealing procedure takes around two hours after the glass casting procedure to eliminate any residual stress in the glass sample. This procedure was also used to eliminate the production of air bubbles and improve the sample's mechanical strength. In the annealing process, molten metal in a stainless steel mould was heated at 400˚C in a first furnace. Finally, the first furnace's electricity was switched off, and the sample was allowed to cool to ambient temperature for 24 hours. The glass sample was removed from the furnace the next day and placed in a silica gel containing scintillation vial to absorb any moisture.

3.2.4 Polishing and grinding

The glass samples' surfaces were polished using Silicon Carbide (SiC) as an abrasive paper, with each grade level of smoothness being polished in ascending order. Starting with 800, 1000, 1200, 2400 and 4000, the polishing procedure progressed until the glass sample's surface was parallel, flat, and mooth. Following the preparation of the glass samples, each sample was polished and ground. The purpose of this grinding operation was to eliminate any damage or deformed surface from the glass samples. The goal of polishing on the other hand, is to provide a clean and parallel surface finish on both sides of the sample. The thickness of the glass sample was measured with a vernier calliper with an accuracy of 0.0001 mm until it reached a thickness of 5 mm, which was suitable for ultrasonic testing.

3.1 Structural analysis

In structural analysis, the purpose of conducting structural analysis is to study the behaviour, properties, and characterization of a material. Therefore, in order to determine the structure of the glass system, a powder form sample was prepared before analysing it with the X-ray Diffraction (XRD) method. To prepare samples in the powder form, the glass samples that were fabricated were crushed by using a plunger before grinding them with an agate pestle and mortar. The grinding process is necessary in order to obtain samples that are in the form of fine powder.

3.1.1 X-ray diffraction (XRD)

The basic characterization method is X-ray diffraction spectroscopy, which detects or identifies any crystalline species in the glass samples and validates the crystallinity of the manufactured samples have short range order of crystalline structure or not. All the glass samples were recorded at 2Θ angles in the range of $80^o \geq \Theta \geq 20^o$. The XRD investigation was performed using a Philips X'PERT PRO PW304 with Cu K1 radiation. According to Epp *et al.* (2016), X-ray Diffraction or also known as XRD is a method that allows a precise study of the structure of crystalline phases of a material. It is a non-destructive analysis method that relies on the targeted sample's ability to diffract X-rays in a specific way. Furthermore, X-ray diffraction crystallography for powder samples has become one of the most popular and widely used methods in materials research, as it is widely used in the field of material characterization to obtain information on the atomic scale structure of various materials in various states. This XRD method is expected to confirm the amorphous nature of the glass sample by producing a broad hump rather than discrete or sharp peaks, indicating that the glass samples are indeed amorphous.

3.2 Physical analysis

Physical analysis of the glass sample includes the density, molar volume and elastic properties.

3.2.1 Density and molar volume

Density is a measure of compactness of matter within a substance. It is defined as:

$$\text{Density} = \text{mass} / \text{volume} \quad \textbf{(3.1)}$$

With distilled water as the immersion liquid at room temperature, the Archimedes Principle was used to determine the density of the glass sample. Because of its high precision and low surface tension, distilled water was chosen to prevent the air bubble from clinging to the sample. Since distilled water does not evaporate at normal temperature, it is also safer than other liquids like acetone. The Electronic Densimeter MD-3005 was used to measure the weight, and the instrument's precision was $\pm$ 0.001 g/cm^3.

According to this principle, the buoyant force on an immersed item is equal to the weight of the fluid displaced by the object. Initially, the glass samples were weighed in the air. After that, the glass samples are weighed and soaked in distilled water. The density of the glass sample may be calculated using the following formula:

$$\rho_{sample} = \frac{W_{air}}{W_{air} - W_{water}} \times \rho_{water} \quad \textbf{(3.2)}$$

ρ_{water} : 1 g/cm^3

w_{air} : Weight of the glass sample in air

w_{water} : Weight of the glass sample in distilled water

From the density of the glass sample, molar volume can be calculated by using this formula:

$$\frac{m}{\rho} = molar\ volume \tag{3.3}$$

m: molecular weight of the glass sample (g/mol)

ρ: density of the glass sample (g/cm^3)

3.2.2 Elastic properties

An ultrasonic method was used to investigate the elastic properties of the glass samples. According to Matori *et al.*, (2013), the ultrasonic method has been used in the field of glass research for many years. The information obtained from the ultrasonic propagation velocity mode assists in determining a series of elastic constants that can be used to express a material's elastic behaviour, such as the longitudinal, shear, bulk, and Young's modulus (Hasnimuyalti *et.al.*, 2016). Debye temperature, Poisson's ratio, microhardness, and softening temperature, may be calculated using established relations from recorded ultrasonic velocities and density.

The following formula was used in this investigation to compute the ultrasonic wave velocity:

$$v = \frac{2d}{\Delta t} \tag{3.4}$$

where Δt is the time interval between two (2) successive echoes and d is the sample thickness. Meanwhile, longitudinal and shear ultrasonic measurements were taken at room temperature utilising the RITEC SNAP RAM-5000 high power ultrasonic system at a frequency of 5 MHz.

It determines the velocity of ultrasonic stress pulses in solids by giving the longitudinal modulus zero lateral strain. The total stiffness of a composite is also influenced by the longitudinal modulus of elasticity. The shear modulus characterizes a solid's elastic characteristics when transverse internal forces are applied. It is the ratio of the shear stress required to bend a material at a particular angle. If a material is too stiff to shear, the shear energy will be transferred relatively quickly. To put it another way, this feature describes how resistant a material is to shearing deformation.The longitudinal and shear velocities were computed using the following formulas:

$$U = \frac{2x}{\Delta t} \tag{3.5}$$

Where U denotes the longitudinal and shear velocity, x is the thickness of the glass samples, and Δt denotes the period between two successive echoes.

Bulk modulus distinguishes a solid’s elastic capabilities when it is subjected to pressure from all sides. When the pressure is removed, the material’s volume returns to its original state. This is because the material’s volume is reduced as a result of the pressure. Bulk modulus is a measurement of a substance’s capacity to withstand volume changes when compressed on all sides. A substances with a high bulk modulus but low compressibility has a high bulk modulus. A substance with a high compressibility but a low bulk modulus is easy to compress. Hooke’s law of elasticity is formed when the bulk modulus is constant.

Young's modulus is a special form of Hooke's Law of Elasticity, and the metric unit for it is newton per square metre (N/m^2). The stress in Young's modulus is proportional to the strain. When the external force is removed, the material returns to its original dimensions. Young's modulus may no longer remain constant as stresses increase, but instead decrease, or the material may flow, suffering persistent deformation, or finally break.

Poisson's ratio (σ) is a relationship between deformation in materials in right-angle directions. This means that when a material sample is stretched in one direction, it tends to thin down in the other two. To put it another way, it is the ratio of transverse to longitudinal strain. Most typical materials have a ratio between 0 and 0.5. For examples, aluminium, bronze, copper and magnesium.

The hardness of a substance is defined as its resistance to localised deformation. Identification, scraping, cutting, and bending are all examples of deformation. The deformation discussed in metals, ceramics, and most polymers is surface plastic deformation. The resistance to elastic deformation of the surface is defined as hardness for elastomers and certain polymers. Because there is no universal definition for hardness, it is assumed that it is a composite attribute that includes contributions from yield strength, work hardening, real tensile strength, modulus, and other characteristics.

CHAPTE R 4

RESULT AND DISCUSSION

4.1 Introduction

In this chapter, the results obtained for $\{\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{0.96}(Eu_2O_3)_{0.04}\}_{(1-X)}(Ag_2O)_X$ glass system have been recorded in order to analyze the properties of the glass sample. Three separate analysis were performed during the process: structural, physical, and elastic analysis. To identify the nature of the glass samples and to examine the arrangement of various structural units that could exist in the glass samples, structural analysis such as X-ray diffraction (XRD) spectroscopy was used. In addition, density and molar volume, as well as ultrasonic velocity in both shear and longitudinal velocity, are obtained in this discovery to assess the glass sample's physical qualities.

4.2 Structural analysis

4.2.1 X-ray diffraction (XRD) spectroscopy

The XRD analysis was used to determine if the materials were crystalline or amorphous. By evaluating the powder form sample using XRD, the X-ray diffraction pattern of a manufactured glass sample of a borotellurite glass system doped with silver oxide was obtained. Silver oxide concentrations range between x = 0.0011, 0.0012, 0.0013, 0.0014, and 0.0015 molar fractions. **Figure 4.1** shows that no discrete sharp peaks can be found in any XRD diffraction pattern. The absence of sharp peaks in the range of $20^o \leq \Theta \leq 80^o$ indicates that the glass sample has an amorphous structure.

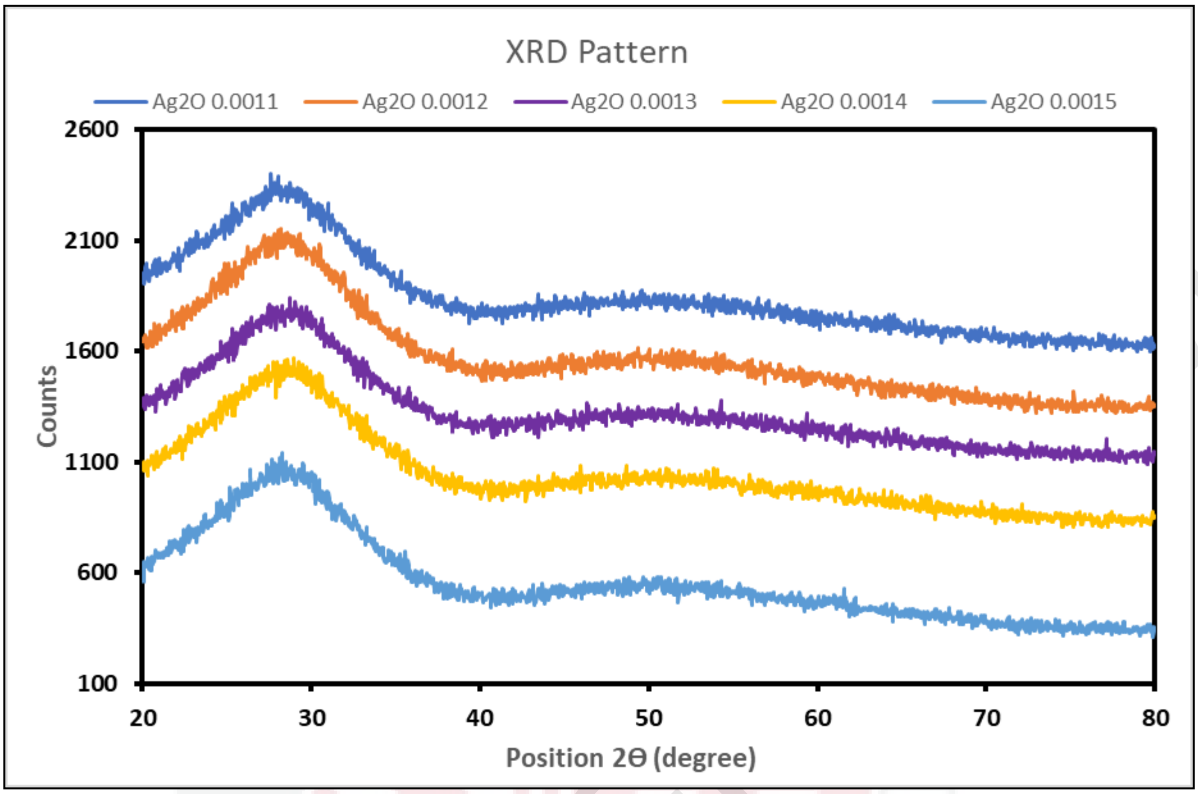

Figure 4.1: XRD diffraction patterns of silver-doped europium zinc borotellurite glasses.

The XRD scattering of produced glass samples having different amounts of silver oxide is depicted in **Figure 4.1**. The XRD patterns exhibited by the glass samples form broad diffusions or humps at reduced scattering angles, indicating the absence of long ranged atomic arrangement in the glass samples, as shown in the figure above. Kaky *et al.*,(2019) had stated that glass is well known to be a non-crystalline or amorphous solid, which lacks translational periodicity. Glasses, unlike crystalline solids, lack long-range order and instead consist of the repetition of a well-defined unit cell in a three-dimensional network. The amorphous and crystalline nature of a material can be determined using XRD analysis. It also provides data on particle size and structure. According to Hasnimulyati (2017), the broad hump confirmed the non-crystallinity (amorphous nature) of the glasses.

4.3 Physical analysis

4.3.1 Density and molar volume

Measuring the density of glass samples is one of the simplest physical analyses that can be performed. Density is an important property that can represent a material's structural compactness (Azuraida *et al.*,2015). The density of all glass samples is estimated at room temperature using the Achimedes Principle and distilled water as the immersion liquid. The results of density and molar volume are recorded in **Table 4.1**, **Figure 4.2** and **Figure 4.3**.

Table 4.1: Density and molar volume of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction, x	Density (g/cm^{-3})	Molar volume (cm^{3}mol^{-1})
0.0011	4.806	26.372
0.0012	4.846	26.156
0.0013	4.852	26.128
0.0014	4.820	26.300
0.0015	4.834	26.230

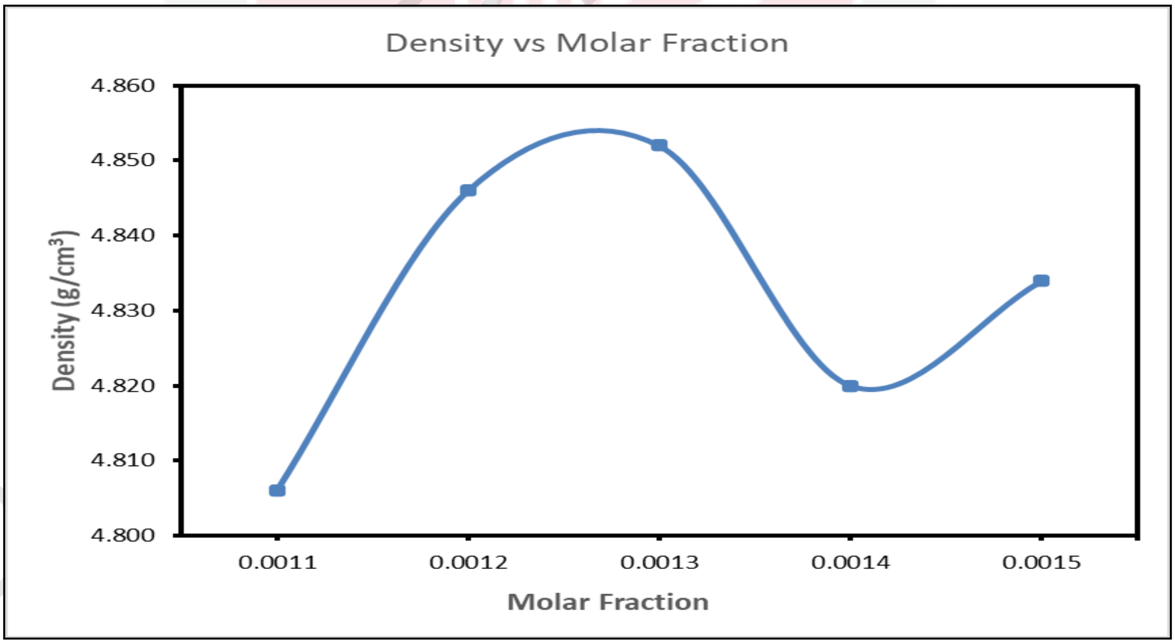

Figure 4.2: Density of europium zinc borotellurite glass system doped with various concentration of silver oxide.

The connection between the density and molar volume of a zinc borotellurite glass system doped with various concentrations of silver oxide is depicted in **Figure 4.2**. The density of the glass samples increases from 4.806 gcm^{-3} to 4.852 gcm^{-3} when the molar percentage of silver oxide increases from 0.0011 to 0.0013, according to the recorded data. The density then exhibits a declining tendency from 0.0013 to 0.0014, followed by an upward trend from 0.0014 to 0.0015. According to Hamza *et al.,* (2019), a rise in the number of BO in the glass network, which increases the glass's connectivity, may impact the density of the glass samples increasing. The structural arrangement would be in a tightly packed condition until there is no increase of free volume in the glass system, resulting in an enhancement of the glass network's stiffness. A glass with better connectivity shows that the glass network has become more compact. The net molecular weight drops when a lower molecular weight ingredient is replaced by a higher molecular weight substance, resulting in poor connection in the glass network. Boron glass modifier has a molecular weight of 69.63 g/mol, which is lower than that of europium, which is 351.93 g/mol. As a result, the density drops as the molar fraction of silver oxide increases.

The molar volume of the glass sample grows and declines as the silver oxide level increases, as seen in **Figure 4.3** from this study. The inverse relationship exists between density and molar volume (Eevon *et al.,* 2010). Because molar volume is inversely related to density, when the density lowers, the molar volume increases.

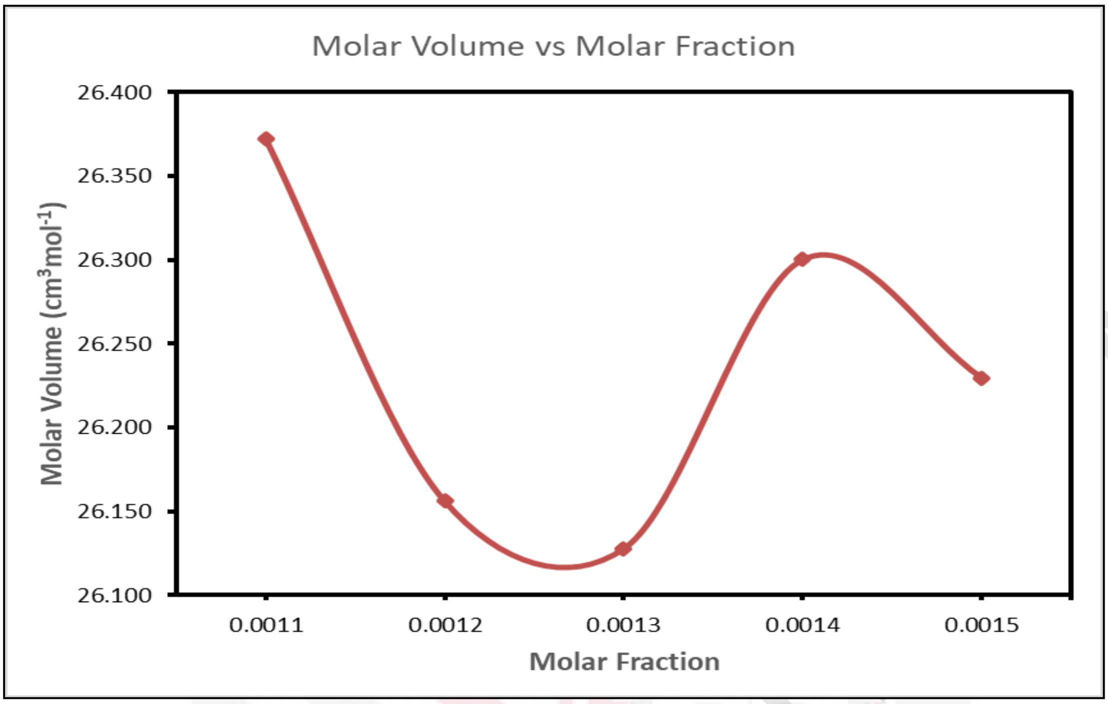

Figure 4.3: Molar volume of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar volume is another parameter that can be measured to study the physical properties of the glass samples. As stated by Mahraz *et al.,* (2013), the molar volume is highly dependent on the density of a glass as it will always follow the opposite trend of the density. Based on this relationship, the volume can be computed using a formula shown in **Equation (3.3)**. Referring to **Table 4.1**, the molar volume shows a decrease trend from 26.372 cm^3/mol to 26.128 cm^3/mol with molar fraction from 0.0011 to 0.0013. Then, the molar volume shows increasing trend from 0.0013 to 0.0014 and decreasing trend from 0.0014 to 0.0015. This indicates that the trend for the molar volume corresponds with the aforementioned theoretical relationship where the molar volume will always exhibit an opposite trend from the density. Based on Azianty *et al.*, (2012), the increasing number of non-bridging oxygen (NBO) in the glass network is one of the contributions to the increment of molar volume. Other than that, the differences in metal-oxygen bond have been proposed to cause the increase in molar volume for the glass system. This phenomenon might also be influenced by the atomic radius of europium (240 pm) that is larger than the atomic radius of boron (85 pm). The larger atomic radius of the dopant oxide causes an increase in the atomic packing density of the glass network, which in turn causes the glass network to become more tightly packed and compact, hence producing a denser glass (Hajer *et al.*,2014).

4.4 Elastic analysis

According to Halimah *et al.*, (2010), the importance of studying a glass's elastic properties is to determine a material's ability to return to its original shape or size after a load is applied. As a result, according to Bansal and Doremus (1986), a material with ideal elastic properties tends to deform immediately when subjected to a stress, but it also recovers quickly from the deformation process once the subjected stress is removed. The elastic parameters studied in this study are ultrasonic velocities, elastic modulus, Poisson's ratio, microhardness, softening temperature, Debye temperature, thermal expansion coefficient and acoustic impedance, which are obtained using the ultrasonic pulse technique. The analysis also derived a relationship between some of the elastic parameters in order to further construct the structure of the studied glass.

4.4.1 Ultrasonic velocities

The two main parameters of ultrasonic velocities are the longitudinal velocity and the shear velocity. The longitudinal velocity is the velocity of an ultrasonic pulse traveling perpendicularly, whereas the shear velocity is the velocity of glass samples when ultrasonic pulses travel parallel to the glass's surface (Halimah *et al.*, 2010). According to Sekhar *et al.*, (2021), the longitudinal (V_L) and shear (V_S) ultrasonic wave velocities are fundamental parameters used to determine the elastic constants, Poisson's ratio, micro-hardness, and other elastic related parameters of a glass material. The ultrasonic velocities can be determined by using the equation shown in **Chapter 3** as follow:

$$v = \frac{2d}{\Delta t} \qquad \textbf{(3.6)}$$

where $\boldsymbol{v}$ represents the longitudinal or shear ultrasonic wave velocity (depending on the type of probe that was used during the ultrasonic pulse echo measurement), $\boldsymbol{d}$ represents the thickness of the glass and $\boldsymbol{\Delta t}$ represents the time interval between the two echoes. The calculated ultrasonic velocities are illustrated in both **Table 4.2** and **Figure 4.4**.

Table 4.2: Longitudinal and shear velocity of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction, x	V_L (m/s)	V_S (m/s)
0.0011	3607	2112
0.0012	3764	2231
0.0013	3917	2317
0.0014	4066	2437
0.0015	4225	2511

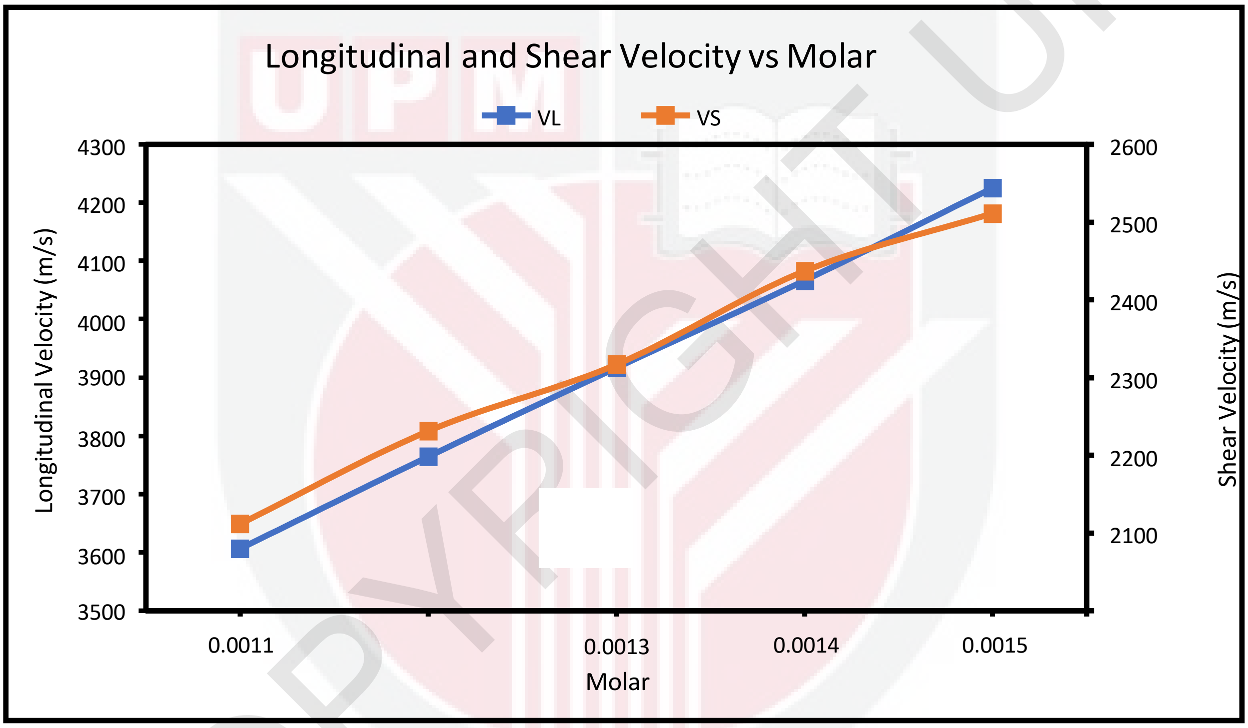

Figure 4.4: Longitudinal and shear velocity of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Figure 4.4 depicts the longitudinal and shear velocity of silver oxide-doped zinc borotellurite glass samples. The figure shows that the longitudinal velocity and shear velocity increase as the molar fraction increases from 0.0011 to 0.0015. The shear and longitudinal velocity variations are affected by the glass's elastic properties and changes in rigidity. Elokr and Aboudeif (2016) also noted that as the concentration molar fraction rises, the glass network stiffness rises and the NBO concentration falls, resulting in an increase in both ultrasonic velocities. As a result of the coordination transition, both velocities in the examined glass system increased, affecting the creation of additional bridging oxygen (Zaitizila *et al.*, 2018). It is possible to deduce that the longitudinal velocity is greater than the shear velocity by comparing the two ultrasonic velocities.

4.4.2 Elastic moduli

Longitudinal modulus (L), shear modulus (G), bulk modulus (K), and Young's modulus (E) are the elastic moduli that are calculated using the ultrasonic velocities and density values of the produced glass samples. The following diagram depicts the relationship between each modulus and ultrasonic velocities:

$$L = v_L{}^2\rho \qquad \textbf{(3.7)}$$

$$G = v_s{}^2\rho \qquad \textbf{(3.8)}$$

$$K = L - (^4/_3)G \qquad \textbf{(3.9)}$$

$$E = (1+\sigma)2G \qquad \textbf{(4.0)}$$

where $\boldsymbol{\rho}$ represents the density, $\boldsymbol{V_L}$ represents the longitudinal velocity, $\boldsymbol{V_S}$ represents the shear velocity and σ represents the Poisson's ratio of the glass. The calculated elastic moduli of the prepared glass samples at different molar fraction are illustrated in **Table 4.3** and **Figure 4.5.**

Table 4.3: Elastic moduli of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction,x	L (GPa)	G (GPa)	K (GPa)	E (GPa)
0.0011	62.51	21.43	33.93	53.12
0.0012	68.67	24.13	36.50	59.31
0.0013	74.44	26.05	39.71	64.12
0.0014	79.70	28.63	41.53	69.84
0.0015	86.30	30.49	45.65	74.81

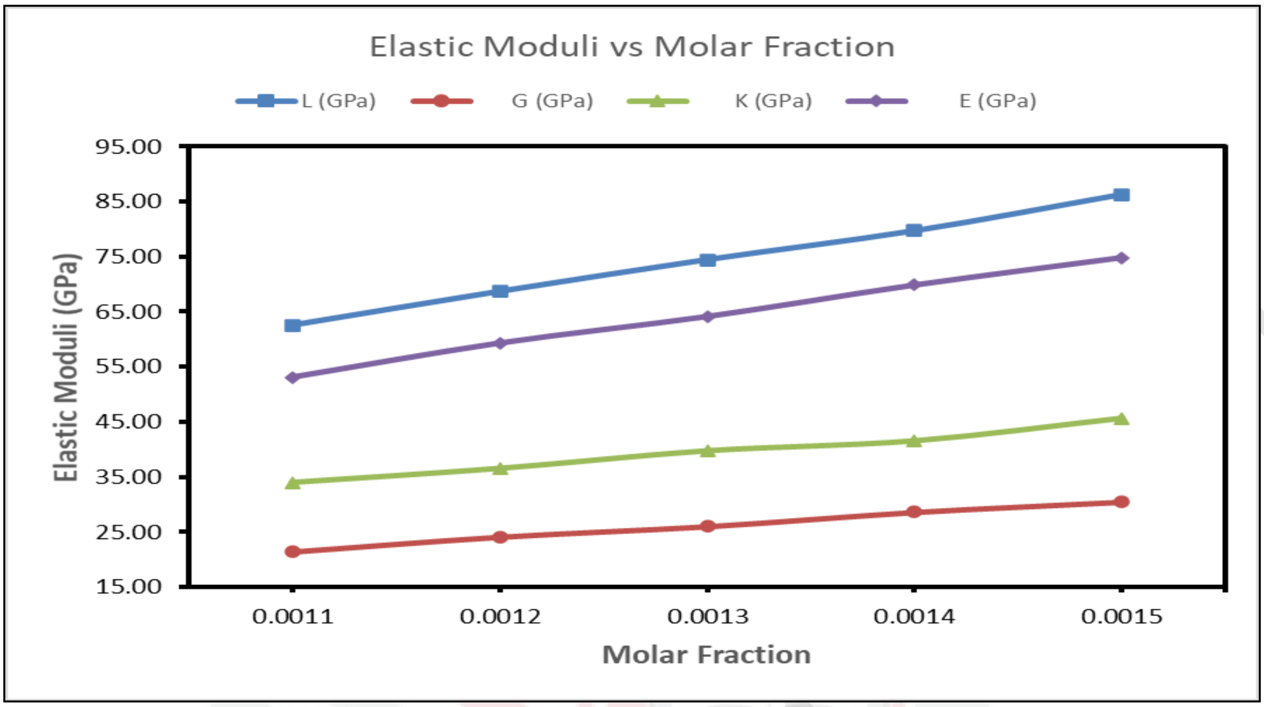

Figure 4.5: Elastic moduli of europium zinc borotellurite glass system doped with various concentration of silver oxide.

The elastic moduli of the prepared silver-doped europium zinc borotellurite glass samples are shown in **Figure 4.5**. As illustrated in the figure, as the concentration of the dopant increases from one point to another, the elastic moduli exhibit varying trends. As the molar fraction of the dopant increases from 0.0011 to 0.0015, the longitudinal modulus (**L**) increases from 62.51 GPa to 86.30 GPa. The shear modulus (**G**) then exhibits an increasing trend, steadily increasing from 21.43 GPa to 30.49 GPa. In the figure above, the bulk modulus (**K**) shows an increasing trend from 33.93 GPa to 45.65 GPa as the molar fraction increases from 0.0011 to 0.0015. The Young's modulus (**E**) exhibits a similar trend to the longitudinal modulus (**L**), shear modulus (**G**), and bulk modulus (**K**), where it can be significantly observed increasing from 53.12 GPa to 74.81 GPa, in accordance with the molar fraction increment.

According to Halimah *et al.,* (2019), the increasing trend is linked to increases in glass density, which causes the glass structure to become compacted and rigid. A stiffer glass typically has a higher modulus value and will deform less when tested with the same amount of load. However, each of these moduli may play a different role in describing the elastic properties of a glass from different perspectives. The longitudinal modulus (**L**) is the ratio of a longitudinally applied stress to the longitudinal strain when all other strains are equal to zero, whereas shear modulus (**G**) is the constant between the shear stress and shear strain, which can also represent a material's tendency to deformin a direction parallel to the applied force (Wang *et al.*, 2019). Furthermore, bulk modulus (K) describes the change in volume of a material when a small or sufficient amount of force is applied to multiple surfaces, whereas Young's modulus (**E**) is defined as the measure of a glass' resistance to elastic deformation caused by a load applied from the normal direction.

According to Nazrin *et al.,* (2018), an increase in the longitudinal modulus (K) and shear modulus (**G**) indicates an improvement in the rigidity of the glass network, which is primarily influenced by the formation of BO ions. The declination of these two moduli, on the other hand, is caused by the formation of NBO ions, which reduces the rigidity and connectivity of the glass because the network of the glass has a more open structure. The study also mentioned that the dopant can affect the modulus increment because the addition of silver oxide as the dopant causes a structural modification in the glass network where the intensity of TeO_4 increases as the concentration of dopant increases. This occurrence contributes to the tendency for BO formation in the glass system. Because the Young's modulus (**E**) of the studied glass samples increased steadily, it is possible to conclude that there is a higher concentration of BO compared to NBO in the glass network, which influenced the inclination of crosslinking in the glass samples' network, improving both the connectivity and stiffness of the studied glass samples.

In terms of overall observation, the longitudinal modulus (**L**) is found to have the highest value when compared to the other elastic moduli. According to Lee *et al.,* (2017), materials with a higher bulk modulus (**K**) than its Young's modulus (**E**) indicate that the glass is more resistant to stress applied from multiple directions rather than just one. However, the results of this study show that the prepared glass samples' Young's modulus (**E**) is greater than their bulk modulus (**K**), implying that the samples are more resistant to stress applied from one direction.

In other words, if stress is applied from different directions, the prepared glass samples from this study will easily fracture or deform. In terms of the other two moduli, Sidek *et al.*, (2004) discovered that if the longitudinal modulus (**L**) is greater than the shear modulus (**G**), the glass can withstand longitudinal stress better than shear stress. This finding is also supported by Wang *et al.*, (2018), who claimed that a glass with high connectivity has a longitudinal modulus (**L**) value that is greater than the shear modulus (**G**).

4.4.3 Poisson's ratio

The connection between lateral (transverse) and axial (longitudinal) strain is described by the Poisson's ratio. According to Makishima and Mackenzie (2003), loosely packed glasses have a lower Poisson's ratio and lateral strain than compact glass structures. As a result, a high cross-link density material will have a Poisson's ratio of 0.1 to 0.2, whereas a low cross-link density material will have a ratio of 0.3 to 0.5. The following equation was used to compute the Poisson's ratio of the prepared glass samples:

$$\sigma = \left(\frac{(L-2G)}{2(L-G)}\right) \qquad \textbf{(4.1)}$$

where $\boldsymbol{L}$ represents the longitudinal modulus and $\boldsymbol{G}$ represents the shear modulus. The calculated values of the Poisson's ratio for the prepared strontium-doped zinc borotellurite glass samples are illustrated in **Table 4.4**.

Table 4.4: Poisson's ratio of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction, x	σ
0.0011	0.239
0.0012	0.229
0.0013	0.231
0.0014	0.220
0.0015	0.227

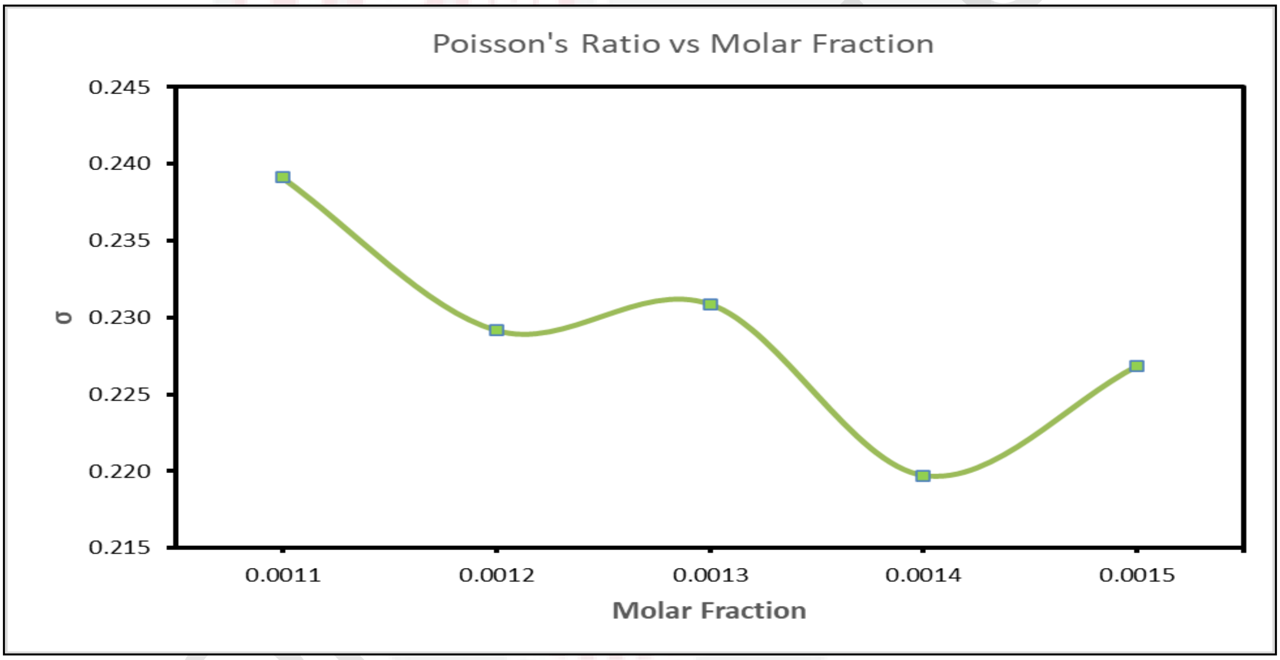

Figure 4.6: Poisson's ratio of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Figure 4.6 depicts the relationship between the molar fraction and Poisson's ratio of silver-doped zinc borotellurite glass samples. The figure shows that the Poisson's ratio of the prepared glass samples fluctuates, with increasing and decreasing trends clearly visible at different dopant concentrations. As the molar fraction rises from 0.0011 to 0.0012, the Poisson's ratio falls from 0.239 to 0.229. However, as the molar fraction reaches 0.0013, the ratio increases to 0.231 and continues to decrease as the molar fraction reaches 0.0014. Then, from 0.220 to 0.227, it increases once more until it reaches the highest molar fraction. As previously stated, the Poisson ratio is impacted by the glass network's size and cross-link density, with large cross-link densities resulting in Poisson ratios in the 0.1 to 0.2 range. A material with a low cross-link density, on the other hand, indicates a higher value of the ratio, ranging between 0.3 and 0.5. According to Yousef *et al.,* (2016), the increase in cross-link density is linked to the increase in dimensionality, whereas the formation of NBO occurs only when the dimensionality is reduced. However, because dimensionality increases with cross-link density in this study, it can be inferred that NBO formation is less in the prepared glass network.

The decreasing trend in Poisson's ratio is caused by an increase in the quantity of BO in the glass structure. As a result, there will be more room and less network connectivity. According to Umar *et al.,* (2020), bismuth ions gradually integrate into the glass structure, increasing the quantity of polar bonding and glass ionicity. Variations in the number of atoms coordinated in the glass network, according to El-Mallawany *et al.*, (2016), might be caused by an increase in dopant concentration content, with the number of atoms coordinated normally increasing with concentration. As a result, the presence of silver oxide as a dopant in the glass network for this investigation has an effect on the number of atoms coordination and the Poisson's ratio of the manufactured glass samples to some extent.

4.4.4 Microhardness

The stress required to suppress excess free volume (network deformation) in a material is referred to as microhardness **(H)** (Tafida *et al.*, 2020). In other words, microhardness is a measure of the surface characteristic of a material. The usual conclusion is that the rigidity of a glass increases with its microhardness (Awshah *et al.,* 2017). The microhardness of the prepared glass samples was determined using the following formula:

$$H = \frac{(1-2\sigma)E}{6(1+\sigma)} \quad \textbf{(4.2)}$$

where ***E*** represents the Young's modulus while ***σ*** represents the calculated Poisson's ratio calculated for the prepared glass samples. The microhardness of the prepared glass system is illustrated in **Table 4.5** and **Figure 4.7** shows the relationship between microhardness with the dopant content.

Table 4.5: Microhardness of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction, x	H (GPa)
0.0011	3.73
0.0012	4.36
0.0013	4.67
0.0014	5.35
0.0015	5.55

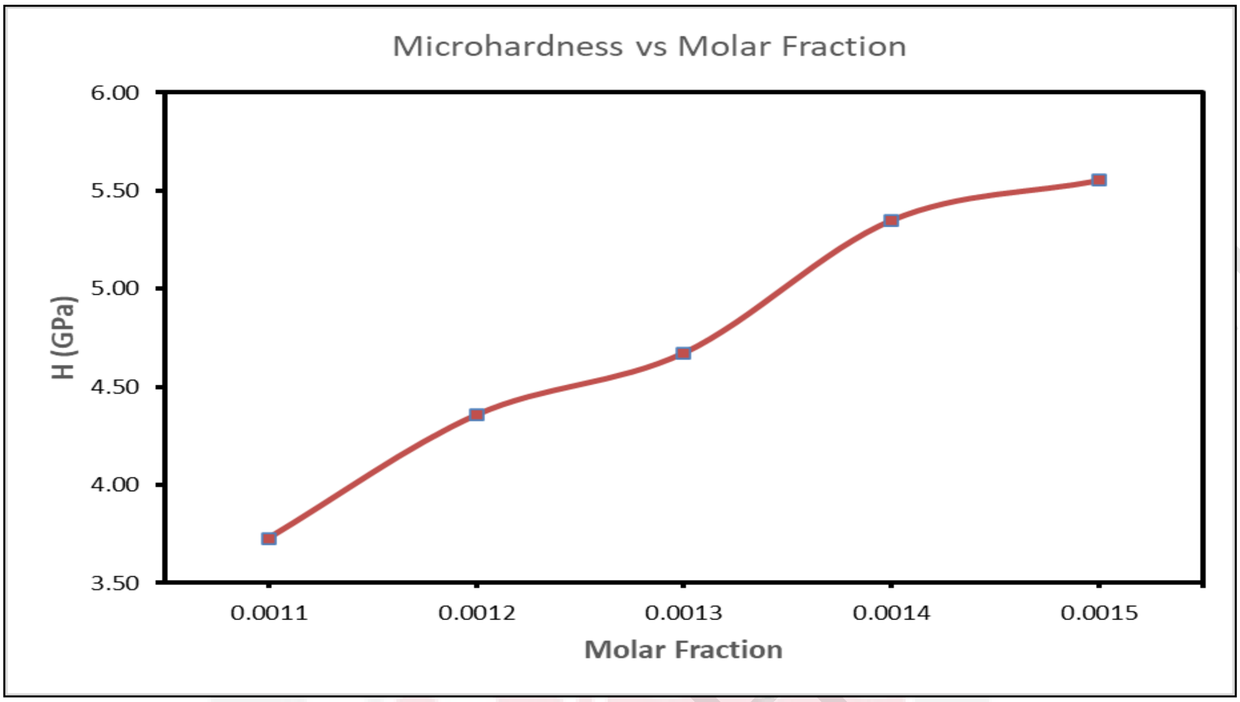

Figure 4.7: Microhardness of europium zinc borotellurite glass system doped with various concentration of silver oxide.

The relationship between the molar fraction of silver-doped zinc borotellurite glass samples and their microhardness is illustrated in **Figure 4.7**. The graph shows that the values of microhardness of the glass samples increase as dopant concentrations increase. The microhardness continuously increases from 3.73 GPa to 5.55 GPa when the silver oxide level increases from 0.0011 to 0.0015 molar fraction. The rise in microhardness reflects an improvement in the stiffness of the glass influenced by BO formation, leading in a more compact and tighter glass network. Furthermore, because the microhardness of the glass increases as the Ag_2O concentration content increases, the presence of the dopant has a significant impact on the improvement in the rigidity of the glass. Umar *et al.*, (2020) also stated that the increase in microhardness with progressive dopant content insertion is related with an increase in the glass sample's stiffness and connectivity.

Halimah *et al.*, (2019) also explained that the presence of dopant as a glass modifier may contribute to the distortion of structural unit arrangement in a glass network where TeO_4 to TeO_3 conversion or vice versa may occur. As a result, the strength of the bond in the glass network will be affected. In other words, as the strength of the network bonds increases, it indicates that more stress is required to deform the structure, demonstrating an improvement in the glass's microhardness.

4.4.5 Fractal bond connectivity

A useful parameter is fractal bond connectivity of a glass sample (d), which offers details about the glass arrangement's dimensions (Bahari *et al.*, 2013). The fractal bond connectivity of the following equation was used to compute prepared glass samples:

$$d = \frac{4G}{K} \qquad \textbf{(4.3)}$$

where ***K*** represents the bulk modulus, ***G*** is the shear modulus. The data of fractal bondconnectivity are listed in **Table 4.6** and presented in **Figure 4.8**.

Table 4.6: Fractal bond connectivity of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Molar fraction, x	d
0.0011	2.5264
0.0012	2.6441
0.0013	2.6237
0.0014	2.7576
0.0015	2.6717

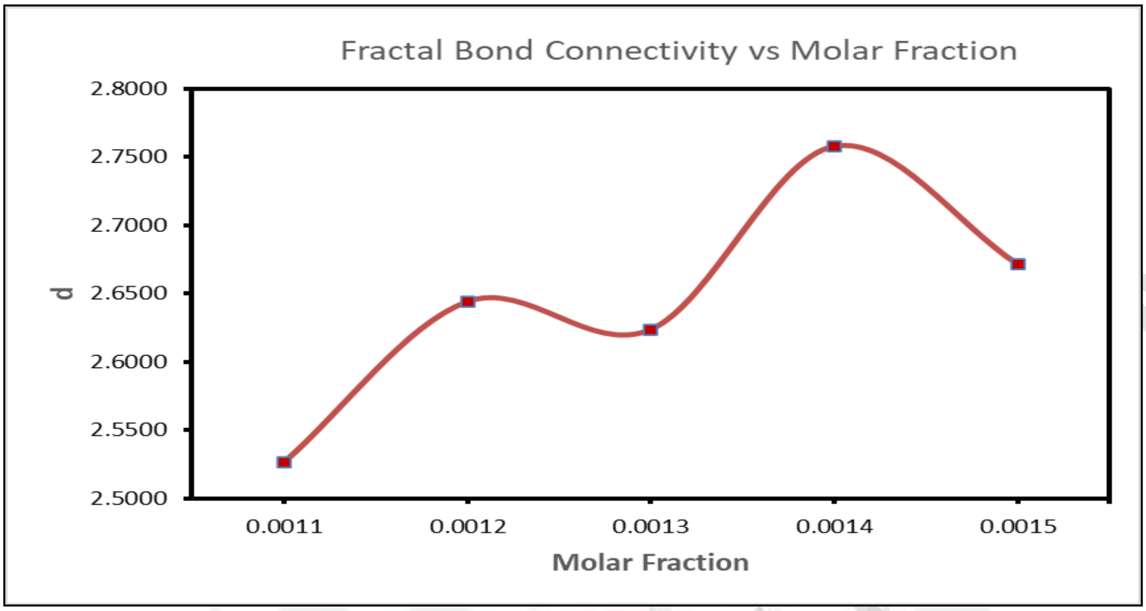

Figure 4.8: Fractal bond connectivity of europium zinc borotellurite glass system doped with various concentration of silver oxide.

The values of fractal bond connectivity of the glass samples show a changing (that is, growing, reducing, increasing, and decreasing again) tendency at different dopant concentrations, as shown in **Figure 4.8**. The fractal bond connectivity, according to Sidek *et al.*, (2013), connects the elastic characteristics of the glass to its structure. **Table 4.6** and **Figure 4.8** demonstrate that the molar fraction falls from 0.0012 to 0.0013 and from 0.0014 to 0.0015. It displays the effective dimensionality of the glass network. The observed alterations in the glass as a result of adding additional dopant indicate that the glass is migrating from a 3D network structure to a 2D network structure (Gouraud *et al.*, 2015). Furthermore, decreased fractal bond connectivity values have been linked to an increase in BO and a reduction in glass stiffness. An increase in the number of NBO and stiffness, on the other hand, is expected to result in an increase in the fugacity and bond connectivity of the manufactured glasses (Alazoumi *et al.*, 2017).

4.4.6 Softening temperature & debye temperature

Another important parameter that is heavily used in many studies relating to the elastic properties of a glass material is softening temperature. According to Laopaiboon *et al.,* (2016), softening temperature is the temperature at which a material transitions from viscous flow to plastic flow. Temperature stability of a glass can be established by determining its softening temperature. The softening temperature of the prepared glass samples was calculated in this study using the following equation:

$$Ts = \frac{MVs^2}{ZC^2} \qquad \textbf{(4.4)}$$

where **M** represents the effective molecular weight, **Z** represents the number of atoms in the chemical formula and **C** represents the constant of proportionality assumed for the prepared glass samples. The preceding equation demonstrates that the softening temperature is proportional to the glass's shear velocity, suggesting that the softening temperature will grow as the value of shear velocity increases.

The Debye temperature is the temperature at which the high pulse mode is excited (Laopaiboon *et al.,* 2016). It is proportional to the material's ultrasonic velocity (glass sample). As a nutshell, every change in ultrasonic velocity has an impact on Debye temperature. The temperature of a host glass network changes to the Debye temperature when a modifier is applied. The temperature at which practically all modes of vibration in a solid material are stimulated is also assumed to be represented by this characteristic (Sidek *et al.,* 2013). The following equation was used to compute the debye temperature of the produced glass samples in this study:

$$\theta_D = \left(\frac{h}{K_B}\right)\left(\frac{3PN_A}{4\pi V_m}\right)^{\frac{1}{3}} U_m \qquad \textbf{(4.5)}$$

where h is the Plank's constant, K_B is the Boltzmann's constant, U_m is the mean ultrasonic velocity, N_A is the Avogadro's constant, V_m is the calculated molar volume calculated from the effective molecular mass, P is the number of atoms in the chemical formula and C is the constant of proportionality assumed for the prepared glass samples, demonstrating that the Debye temperature is directly dependent on the mean ultrasonic wave velocity of the glass. **Table 4.7** and **Figure 4.9** show the calculated softening temperature and debye temperature for the prepared silver-doped zinc borotellurite glass samples.

Table 4.7: Softening temperature and Debye temperature of europium zinc borotellurite glass system doped with various concentration of silver oxide

Molar fraction, x	Θ_d (K)	T_S (K)
0.0011	291.30	687.16
0.0012	308.28	767.20
0.0013	320.28	827.27
0.0014	335.75	915.46
0.0015	346.57	972.20

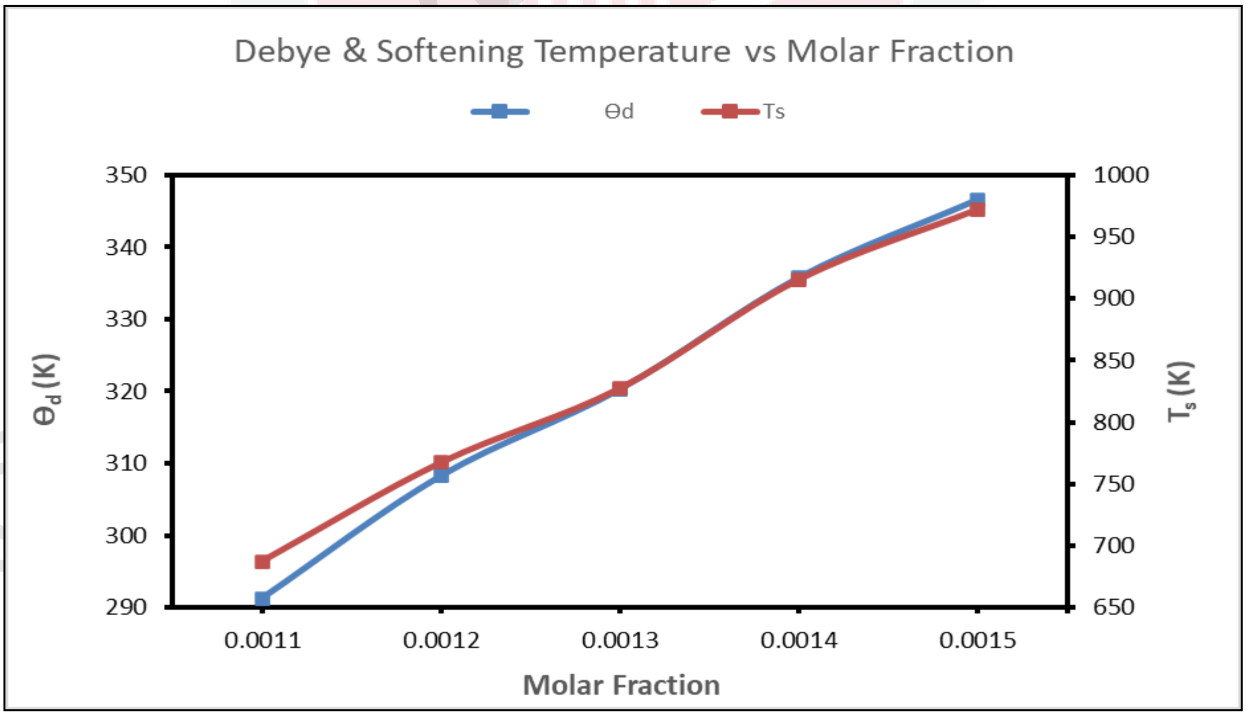

Figure 4.9: Softening temperature and Debye temperature of europium zinc borotellurite glass system doped with various concentration of silver oxide.

Figure 4.9 illustrates the softening temperature and Debye temperature exhibited by the silver-doped europium zinc borotellurite glasses based on their molar fraction. From the figure, it can be seen that the value of the softening temperature steadily shows increasing trend from 687.16 K to 972.20 K when approaches the higher molar fraction. According to The observed increase, according to Abdulbaset *et al.,* (2021), is due to the continual insertion of bismuth oxide, which reinforces the glass interconnectivity and enhances stability. And the softening temperature can be attributed to an significant decrease in molar weight as an result of the substitution of Eu_2O_3 (heavy) with B_2O_3, which is much ilighter. This resulted in a significant reduction in the molecular weight of the glasses, outweighing the rise in mean ultrasonic velocity (Chinnamat *et al.,* 2017). While for the Debye temperature, the figure shows the same trend as softening temperature which is when the molar fraction increases the debye temperature also increases from 291.30 K to 346.57 K. As a result, the increase in the Debye temperature observed in this study is thought tobe influenced by the fact that the glass has a good bond strength due to the increase in the number of bonds per unit volume, which triggers the atomic vibrations in the glass network. When a glass has a relatively high bond strength, it means that more energy is required to vibrate the atoms because the atoms are closer together.

According to Zaitizila *et al.,* (2018), when the temperature rises, the elastic characteristics of the material become more stable. This is due to the increasing number of atoms and ultrasonic velocities in the glass sample. Furthermore, an increase in ultrasonic velocities and the number of atoms in the chemical formula causes an increase in softening temperature, showing that the glass structure is strengthened by the creation of bridging oxygen (Eevon *et al.,* 2016). Because the Debye temperature of the prepared glass system rises with molar fraction, the concentration of dopants may have aided in the creation of BO in the glass network, resulting in an increase in mean ultrasonic velocity of the examined glass samples.

Simultaneously, this finding demonstrates that the prepared glass network has a superior connectivity, resulting in a more resilient and compact glass system. According to Pavani *et al.*, (2011), the observed rise in the two parameters indicates an increase in glass stiffness, which may be ascribed to an increase in the number of atoms in the chemical formula and an increase in the glass system's mean ultrasonic velocity. Furthermore, greater Debye temperatures and softening temperatures may be predicted as a result of the glass structure strengthening caused by the production of BO in the generated samples (Saddeek *et al.*, 2005).

CHAPTER 5

CONCLUSIONS

5.1 Conclusion

In this study, five (5) europium zinc borotellurite glass samples doped with silver oxide were prepared using traditional melt-quenching techniques. The general formula for the chemical composition of the prepared glass samples is $\{\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{0.96} (Eu_2O_3)_{0.04}\}_{(1-x)} (Ag_2O)_X$, with each sample composed of different molar fractions x = 0.0011, 0.0012, 0.0013, 0.0014 and 0.0015. Several analyses were carried out to determine the effect of silver oxide (Ag_2O) on the elastic properties of the zinc borotellurite glass system. The initial examination employed X-ray Diffraction (XRD) Spectroscopy to look at the structural characteristics of the produced glass samples. The lack of strong peaks in the XRD spectra of the examined glass samples verifies their amorphous nature. The density and molar volume of the produced glass samples are the two key parameters examined in this research for physical analysis. The density of all the manufactured glass samples increase and decrease as the quantity of Ag_2O increases, according to the observations. The molar volume, on the other hand, is inversely proportional to density, illustrating that the link between density and molar volume should always be indirect. The atomic radius of the dopant inserted into the glass network, which influences the bonding strength and connectivity of the glass to some extent, was also shown to effect variations in the density and molar volume of manufactured glass samples in this study.

At 5 MHz, the elastic properties were investigated further using the pulse echo overlap technique to quantify longitudinal and shear velocities. This approach was used to calculate the four main elastic moduli: longitudinal modulus (L), shear modulus (G), bulk modulus (K), and Young's modulus (E). The studied glass samples' elastic moduli, softening temperature, and Debye temperature all showed a continuous rising trend, which may be linked to the presence of BO. Because there is less concentration of NBO, the elastic moduli rise at particular concentration points, indicating that the examined glass system is more stiff and hence increases glass network connectivity. At different phases of dopant concentrations, the Poisson's ratio, microhardness, and fractal bond connectivity all displayed a changing tendency. As a result, our research has shown that adding Ag_2O as a dopant to the manufactured europium zinc borotellurite glass system impacted the elastic characteristics of the glass network to some amount. The function of Ag_2O content on glass elastic characteristics is believed to be helpful for a range of glass industry applications, including polishing glass, window glass, optical lenses, and others.

5.2 Future research

Further investigation of the elastic properties of a zinc borotellurite glass system doped with europium oxide co-doped with silver oxide should be carried out utilizing an echo of a pulse overlap procedure on a regular basis other than 5 MHz to measure shear and longitudinal velocities. Aside from that, the elastic properties of glass samples can be looked into using multiple glass thicknesses samples to see how thickness affects elastic qualities. Optical, thermal, electrical, dielectric, magnetic, and microstructural properties of the zinc borotellurite glass system doped with europium oxide co-doped with silver oxide can be studied instead of elastic properties.

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APPENDICES

The calculation of the molar fraction of chemical powders that was used to fabricate zinc borotellurite glasses system doped with europium oxide co-doped with silver oxide, with the chemical composition $\{\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{0.96}(Eu_2O_3)_{0.04}\}_{(1-X)}(Ag_2O)_X$.

Atomic weight of each element:

Boron, B 10.81 g/mol	Tellurium, Te 127.6 g/mol	Europium, Eu 151.96 g/mol	Oxygen, O 15.999 g/mol	Zinc, Zn 65.38 g/mol	Silver, Ag 107.9 g/mol

Molecular weight of element:

B_2O_3 69.63 g/mol	TeO_2 159.598 g/mol	Ag_2O 231.74 g/mol	ZnO 81.38 g/mol	Eu_2O_3 351.93 g/mol

Molecular weight of compound:

Molar fraction, x	Weight of TeO_2 (g)	Weight of B_2O_3 (g)	Weight of ZnO (g)	Weight of Eu_2O_3 (g)	Weight of Ag_2O (g)	Total (g/mol)
0.0011	74.9926927	14.0193489	23.4143630	14.0613155	0.2549094	126.7426296
0.0012	74.9851852	14.0179455	23.4120190	14.0599078	0.2780830	126.7531405
0.0013	74.9776777	14.0165420	23.4096750	14.0585002	0.3012565	126.7636513
0.0014	74.9701701	14.0151385	23.4073310	14.0570925	0.3244301	126.7741622
0.0015	74.9626626	14.0137350	23.4049870	14.0556848	0.3476037	126.7846731

Molar percent of compound:

Molar fraction, x	TeO_2 (%)	B_2O_3 (%)	ZnO (%)	Eu_2O_3 (%)	Ag_2O (%)	Total (%)
0.0011	59.169273	11.061274	18.473944	11.094385	0.201124	100
0.0012	59.158444	11.059249	18.470563	11.092355	0.219389	100
0.0013	59.147616	11.057225	18.467183	11.090324	0.237652	100
0.0014	59.136790	11.055201	18.463803	11.088295	0.255912	100
0.0015	59.125966	11.053178	18.460423	11.086265	0.274169	100

Weight of each element needed to form 10 g of glass sample:

Molar fraction, x	TeO_2 (g)	B_2O_3 (g)	ZnO (g)	Eu_2O_3 (g)	Ag_2O (g)	Total (g)
0.0011	7.692006	1.437966	2.401613	1.442270	0.026146	13
0.0012	7.690598	1.437702	2.401173	1.442006	0.028521	13
0.0013	7.689190	1.437439	2.400734	1.441742	0.030895	13
0.0014	7.687783	1.437176	2.400294	1.441478	0.033269	13
0.0015	7.686376	1.436913	2.399855	1.441214	0.035642	13