UNIVERSITI PUTRA MALAYSIA

OPTICAL PROPERTIES OF ZINC BOROTELLURITE GLASS SYSTEMS DOPED WITH DYSPROSIUM OXIDE AND DYSPROSIUM OXIDE NANOPARTICLES

AMI HAZLIN BINTI MOHD NOR

FS 2018 67

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By

AMI HAZLIN BINTI MOHD NOR

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

October 2017

DEDICATION

To mom and dad,
who were always be my supportive,
and encouraged me to go on every adventure,
without them none of my success would be possible,

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

OPTICAL PROPERTIES OF ZINC BOROTELLURITE GLASS SYSTEMS DOPED WITH DYSPROSIUM OXIDE AND DYSPROSIUM OXIDE NANOPARTICLES

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October 2017

Chair: Professor Halimah Mohamed Kamari, PhD
Faculty: Science

Two series of zinc borotellurite glass systems doped with dysprosium oxide and dysprosium oxide nanoparticles with chemical formula of $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{1-x}(Dy_2O_3)_x$ (where x = 0.01, 0.02, 0.03, 0.04, and 0.05 molar fraction) were fabricated by using melt-quenching method. The physical, structural and optical properties of the zinc borotellurite glass systems at various concentration of dopants have been studied. The amorphous nature of the two glass systems was confirmed by using XRD analysis. The infrared spectra obtained from the FTIR spectroscopy for both series of the glasses revealed the existence of five obvious bands which were assigned to the BO_3, BO_4, TeO_3 and TeO_4 vibrational groups. The presence of the Dy_2O_3 NP in the zinc borotellurite glass network was proven by TEM image. The values of the density of zinc borotellurite glass doped with Dy_2O_3 and Dy_2O_3 NP were found to increase from 4.4181 to 4.94941 g/cm^3 and from 4.4210 to 5.0081 g/cm^3 respectively with the addition of dopants. The decrement of the molar volumes of both glasses follow the relationship between the density and the molar volume which supposed to be inversely proportional to each other. For the zinc borotellurite glass systems doped with Dy_2O_3, the indirect and direct optical band gaps were found to increase from 3.0000 to 3.0430 eV and 3.0800 to 3.1150 eV, respectively as the concentration of Dy_2O_3 increased. On the other hand, the direct and indirect optical band gaps for the zinc borotellurite glass systems doped with Dy_2O_3 NP were found to decrease in the range of 3.2880 to 3.3110 eV and 3.0015 to 3.0309 eV as the concentration of Dy_2O_3 NP was increased. The values of Urbach energy for both glass series were inversely proportional to the optical band gaps of the glass samples. The Urbach energy of Dy_2O_3 doped glass systems was observed in the range of 0.3389 to 0.3411 eV while the Urbach energy for Dy_2O_3 NP doped glass systems was in between 0.4010 to 0.4219 eV. As the concentration of the Dy_2O_3 increased, the refractive index

of Dy_2O_3 doped zinc borotellurite glass was found to decrease from 2.0410 to 2.0310, which in turn reduced the electronic polarizability, oxide ion polarizability and optical basicity of the glass systems. As in case of Dy_2O_3 NP doped zinc borotellurite glass, the refractive index of the glass systems was found to increase from 2.0340 to 2.0410 as the concentration of Dy_2O_3 NP increased. The values of electronic polarizability, oxide ion polarizability and optical basicity were found to be inversely proportional to the refractive index of the zinc borotellurite glass systems doped with Dy_2O_3 NP. The increment in the values of the metallization criterion of the first series of the glass systems which were found in the range of 0.4866 to 0.4898 indicates that the materials were prone to act as an insulator. In contrast, the slight decrement of the metallization criterion of the second series of the glass systems from 0.4944 to 0.4934 suggests that the glass samples are metallizing. From the emission spectra of both series of glass system which were obtained from Luminescence spectrometer, two transition bands were observed which represent the transitions from $^4F_{9/2}$ to $^6H_{15/2}$ and $^6H_{13/2}$. In addition, the x and y CIE chromaticity coordinates which were determined from the emission spectra and were found to be located in the region of white light spectrum. In this research, it is also proven that the values of the correlated colour temperature obtained for both glass systems fall in the neutral white light region.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia Sebagai memenuhi keperluan untuk ijazah Master Sains

SIFAT OPTIK BAGI SISTEM KACA ZINK BOROTELURIT TERDOP DENGAN DISPROSIUM OKSIDA DAN DISPROSIUM OKSIDA NANOZARAH

Oleh

AMI HAZLIN BINTI MOHD NOR

Oktober 2017

Pengerusi: Profesor Halimah Mohamed Kamari, PhD
Fakulti: Sains

Dua siri sistem kaca zink borotelurit yang telah didopkan dengan disprosium oksida dan disprosium oksida nanozarah dengan menggunakan formula kimia $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(ZnO)_{0.3}\}_{1-x}(Dy_2O_3)_x$ (dimana x = 0.01, 0.02, 0.03, 0.04, dan 0.05 pecahan molar) telah difabrikasi dengan menggunakan kaedah sepuh lindap. Sifat fizikal, struktur dan optik bagi sistem kaca zink borotelurit pada kepekatan dopan yang pelbagai telah dikaji. Sifat semulajadi amorfus bagi kedua-dua sistem kaca telah disahkan menggunakan analisis XRD. Spektra inframerah yang diperolehi daripada spektroskopi FTIR untuk kedua-dua siri sistem kaca menunjukkan kewujudan lima jalur yang ketara yang merujuk kepada kumpulan getaran BO_3, BO_4, TeO_3 dan TeO_4. Kehadiran Dy_2O_3 NP di dalam jaringan kaca zink borotelurit telah dibuktikan melalui TEM. Nilai ketumpatan bagi kaca zink borotelurit yang didopkan dengan Dy_2O_3 dan Dy_2O_3 NP telah dijumpai bertambah masing – masing dari 4.4181 hingga 4.9491 g/cm^3 dan dari 4.4210.hingga 5.0081 g/cm^3 dengan bertambahnya pendopan. Penurunan isipadu molar untuk kedua-dua kaca adalah mengikut perhubungan di antara ketumpatan dan isipadu molar yang sepatutnya berkadar songsang di antara satu sama lain. Untuk sistem kaca zink borotelurit yang didopkan dengan Dy_2O_3, nilai jurang jalur optik terus dan tidak terus masing – masing telah dijumpai menaik dari 3.0000 hingga 3.0430 eV dan dari 3.0800 hingga 3.1150 eV apabila kepekatan Dy_2O_3 menaik. Sementara itu, nilai jurang jalur optik terus dan tidak terus untuk sistem kaca zink borotelurit yang didopkan dengan Dy_2O_3 NP telah dijumpai menurun dan berada di dalam julat 3.2880 hingga 3.3110 eV dan 3.0015 hingga 3.0309 eV apabila kepekatan disprosium oksida nanozarah menaik. Nilai tenaga Urbach untuk kedua-dua siri kaca adalah berkadar songsang dengan jurang jalur optik sampel kaca tersebut. Nilai tenaga Urbach bagi Dy_2O_3 dop sistem kaca telah diperhatikan berada di dalam julat 0.3389 hingga 0.3411 eV semetara nilai tenaga Urbach bagi Dy_2O_3 NP dop sistem kaca berada di antara 0.4010 hingga 0.4219 eV. Apabila kepekatan Dy_2O_3 bertambah, indeks pembiasan bagi Dy_2O_3 dop zink borotelurit didapati menurun dari 2.0410 hingga 2.0310, sekaligus telah menyebabkan pengurangan kebolehkutuban elektronik, kebolehkutuban ion oksida dan kebesan optik sistem kaca.

Dalam kes kaca Dy_2O_3 NP dop zink borotellurit pula, indeks pembiasan sistem kaca didapati menaik daripada 2.0340 kepada 2.0410 apabila kepekatan Dy_2O_3 NP bertambah. Nilai kebolehkutuban elektronik, kebolehkutuban ion oksida dan kebesan optik telah didapati berkadar songsang dengan indeks pembiasan sistem kaca zink borotellurit yang didopkan dengan Dy_2O_3 NP. Penambahan nilai kriteria pelogaman bagi sistem kaca siri pertama yang berada di antara julat 0.4866 hingga 0.4898 menunjukankan bahan tersebut lebih cenderung untuk bertindak sebagai penebat. Bertentangan dengan itu, sedikit penurunan kriteria pelogaman untuk sistem kaca siri kedua dari 0.4944 hingga 0.4934 telah mencadangkan bahawa sistem kaca tersebut bersifat logam. Berdasarkan spektra pancaran untuk kedua-dua sistem kaca yang dipeolehi daripada spektrometer kefotopendarchayaan, dua jalur peralihan yang mewakili peralihan daripada $^4F_{9/2}$ kepada $^6H_{15/2}$ dan $^6H_{13/2}$ telah diperhatikan. Tambahan pula, koordinat kroma bagi x dan y yang didapati daripada spektra pancaran telah ditemui berada di dalam rantau spectrum cahaya putih. Di dalam kajian ini, ia juga terbukti bahawa nilai suhu korelasi warna untuk kedua-dua sistem kaca adalah berada di dalam rantau cahaya putih berkecuali.

ACKNOWLEDGEMENT

First and foremost, I would like to extend my deepest praise to Allah S.W.T, the Almighty, who gave me strength, patience, courage and provide me with good health and intellectual ability to finish this research. My praise also goes to Prophet Muhammad S.A.W for his guideline which always useful throughout the research.

It is my proud privilege to express my heartfelt thanks and earnest gratitude to my respectable supervisor, Professor Dr Halimah Mohamed Kamari for her non-stop guidance, sustained encouragement, constructive criticisms, absolute ideas, and willingness to examine this thesis. I would like to express my deepest gratitude also to my co-supervisor, Dr Farah Diana Muhammad for her brilliant advices and valuable opinions and suggestions towards accomplishing this research.

It is a great pleasure for me to give thousands of thanks to my knowledgeable seniors and lovely friends, Faznny binti Mohd Fudzi, Hasnimulyati binti Laoding, Zaitizila binti Ismail, Chua Eevon and Nurhayati binti Mohd Nor for their big help and always lend their shoulders when in need.

A very heart warming and sincere love goes to my family who are always be my strength and spirit, always standing beside me in every steps of my life, gave me unconditional loves, and helped me to complete this research.

Thank you.

I certify that a Thesis Examination Committee has met on (19 October 2017) to conduct the final examination of (Ami Hazlin binti Mohd Nor) on her thesis entitled "Optical Properties of Zinc Borotellurite Glass Systems doped with Dysprosium Oxide and Dysprosium Oxide Nanoparticles" in accordance with the Universities and University Colleges 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 Master of Science.

Members of the Thesis Examination Committee were as follows:

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

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Signature: _____
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Member of
Supervisory
Committee: Dr. Farah Diana Muhammad

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
BO	Bridging oxygen
CCT	Correlated Colour Temperature
CIE	Commission Internationale de I'éclairage
E_{opt}	Optical band gap
FTIR	Fourier Transform Infrared
GaN	Gallium nitride
IUPAC	International Union of Pure and Applied Chemistry
IR	Infrared
LED	Light emitting diode
NBO	Non-bridging oxygen
NP	nanoparticles
PL	Photoluminescence
RE	Rare earth
SSL	Solid state lighting
TEM	Transmission electron microscopy
T_g	Glass transition temperature
T_m	Melting point
UV-VIS	UV-Visible
V-T	Specific volume vs. temperature
XRD	X-ray Diffraction
Y/B	Yellow to blue ratio
ΔE	Urbach energy
$\boldsymbol{\rho}$	Density
V_m	Molar volume
n	Refractive index
Λ	Optical basicity
ħ	Plank constant
v	Frequency of the absorbed light
A	Absorbance
d	Thickness of the materials
B	Band tailing parameter
$\hbar\omega$	Photon energy
$\alpha(\omega)$	Absorption coefficient
ω	Photon frequency
α_m	Electronic polarization
$\alpha_{O^{-2}}$	Oxide ion polarizability
R_m	Molar refraction

CHAPTER 1

INTRODUCTION

This chapter is a preamble to the zinc borotellurite glass systems doped with Dy_2O_3 and Dy_2O_3 NP and the basic explanation of the formation of the glass are elaborated. The problem statements and the objectives of the research are also presented in this chapter.

1.1. Zinc borotellurite glass systems doped with rare earth (RE) oxide

In recent times, extensive research has been done on the glasses to be applied as the materials for the optical device applications (Peng et al., 2016; Steudel et al., 2016; Karmakar et al., 2016). The tellurite based glass is one of the most promising glasses and is widely used as the main host to achieve good optical and dielectric properties. The tellurite based glass has a high quality of a glass forming ability (Noorazlan et al., 2015), good mechanical strength and chemical durability, low melting point, high refractive index and good infrared transmission (Maheshvaran et al., 2011; Sennaroglu et al., 2006). This characteristics of tellurite glass have made the tellurite glass as a good candidate for the further development of the optical systems. For instance, the addition of borate oxide into the tellurite glass systems is able to improve the properties of the glass and provide moderate stability and durability to the glass systems. Apart from the borate oxide, the addition of zinc oxide into the glass network helps to increase the glass forming ability and able to lower the crystallization rates within the glass systems (Mahraz et al., 2013). According to Kityk et al. (2002), doping rare earth element into the glass systems is important to enhance the quality of the glass. RE does not only act as an optically active ion but also can influence the structure and properties of the multicomponent glass systems. Besides, the addition of RE helps to improve the glass stability and enhance the resistance to crystallization (Pisarski et al., 2005).

1.2. Dysprosium oxide

Dysprosium is a rare earth element with the symbol Dy and the atomic number 66 (Fontani, 2015) had been discovered in 1886 by Paul-Émile Leloq de Boisbaudran (1838-1912) in Paris. This element was named as dysprosium because it has been *dysprositos* (difficult to get) (Emsley, 2011). Among the trivalent lanthanide ions, Dy^{3+} ion is the most suitable for investigating the dependence of its optical behavior against glass compositions (Karunakaran et al., 2010). Dy^{3+} ion is identified as *f*-localized trap-creating ion and forms some electron trapping level in the intra-band gap of the host materials (Laopaiboon and Bootjomchai, 2015). Other than that, the Dy^{3+} ion is identified as an active luminescence center. Furthermore, Dy^{3+} ion has been well incorporated into several glass systems in order to obtain two primary colours, yellow and blue luminescence materials (Rajesh et al., 2012) thus can be considered as promising materials in the solid state lighting (SSL) technology. Besides that, the Dy^{3+} ion can act as a well-known activated ion, which leads to light emission in the visible range and offers an excellent possibility for white light application due to its strong excitation band

around 454 nm which perfectly matches with the emission spectrum of GaN-based LED (Jayasimhadri et al. 2009; Nizamoglu et al., 2007). Moreover, Dy^{3+} ion doped glass systems are promising luminescent materials in the blue-yellow region, thus, by adjusting a suitable intensity of yellow to blue ratio (Y/B), the chromaticity coordinates of the glass that contains Dy^{3+} ion will be able to generate white light (Babu et al., 2015).

1.3. Formation of glass

Glass has been found for millions of years, has enchanted and captivated much interest both scientifically and technologically. Glass has been produced in many forms and shapes and is traditionally used as a drinking bottles, containers, jars and vases for centuries. Over time, glass has entering new applications that are appearing in the nanotechnology (fibres and displays). With the highly demands in the sophisticated optoelectronic devices, the urge to combine the optical and electronic devices for many applications has leads to the increased demand of the glass materials. It is well informed that glass is one of the most important materials in optics which has been used in optical fibers, lenses, mirror substrates, prisms and SSL materials (Hirao, 2001).

Glasses are formed from extremely viscous liquid and are found to possess ionic and covalent bonding interaction. Glasses can be defined as a non-crystalline amorphous solid which completely lacking in the long range, periodic atomic structure, and exhibiting a region of glass transformation behavior. According to Yamane and Asahara (2000), the American Society for Testing and Materials (ASTM) has defined glass as an inorganic product of fusion which has been cooled to a rigid condition without undergo crystallization process. All glasses have two common characteristics. These include having a short range periodic atomic arrangement and exhibiting time-dependent glass transformation behavior (Shelby, 2005). The atomic arrangement of the glass material is dissimilar from the crystalline material and is portrayed in Figure 1.1.

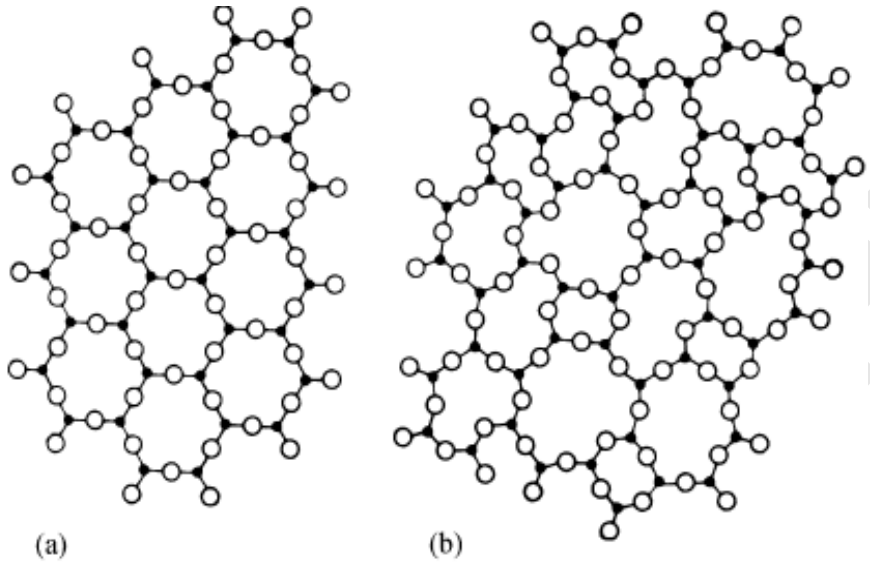

Figure 1.1: Schematic illustration of the atomic arrangement of the (a) crystalline material and (b) glass material (Yamane and Asahara, 2000)

The formation of glass involves the bypassing the crystallization process (Paul, 2012). Usually glass can be fabricated by using melt-quenching method through the quick cooling process as illustrated in Figure 1.2. The rate of cooling process varies depending on the type of materials. During the glass fabrication process, the materials will undergo the cooling process below a critical temperature. This critical temperature is also known as glass transition temperature (T_g). After passing the T_g, the molecular movement of the atoms within the material has slowed and the materials form a glass. The glass transition process can be demonstrated via specific volume vs. temperature (V-T) plot as shown in Figure 1.3.

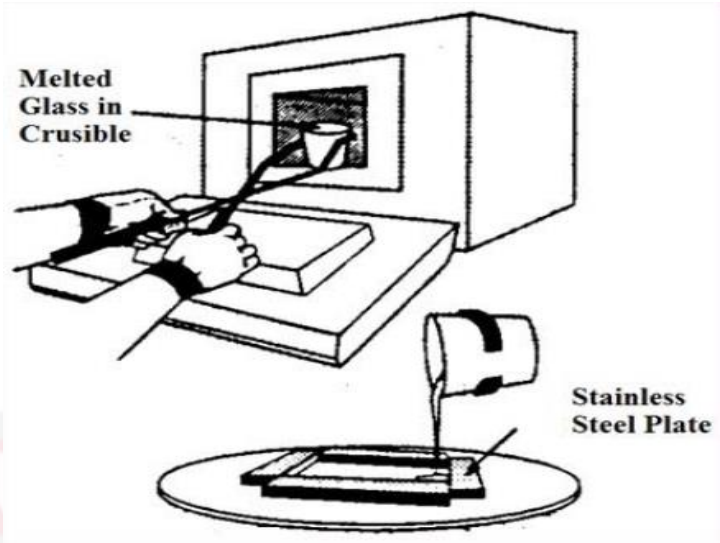

Figure 1.2: Glass fabrication process through melt-quenching method

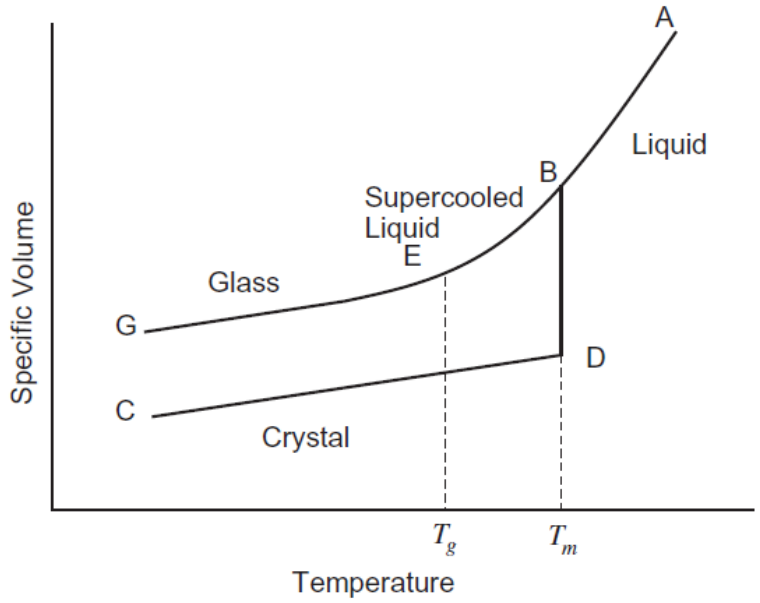

Figure 1.3: The V-T plot of the formation of glass (Kitamura, 2013)

In the glass forming process, the material will be transformed into liquid as it is heated above the melting point from A-B. As the liquid is cooled below the melting point (T_m), a sudden change in the specific volume is observed which follows path B-D. Cooling the liquid in the slow rate below T_m along the path D-C will usually produce a crystallization and exhibit a long range, periodic atomic arrangement. If the cooling process below the

T_m happens without the crystallization and the specific volume continues to decrease, a super-cooled liquid will be obtained along B-E. The decrement of the volume on cooling process is due to the decrease in the amplitude of the atomic vibrations and also due to the change in the structure of the melt which becomes more compact as the temperature decreases. As the cooling process takes place, the structure of the liquid will rearrange and the viscosity of the liquid will increase rapidly due to the less mobility of the molecules causes the atoms can no longer to rearrange completely to the equilibrium liquid structure. The path from E-G is called as the glassy state where the glass material starts to form (Shelby, 2005; Paul, 2012; Kitamura, 2013; Zanotto, 1998).

1.4. Problem statements

Recently, research on glass as an optical material has gained more attentions due to their versatile applications in science and technology. The sophisticated technological applications have led to an outstanding research on various type of glasses. The silicate based glasses have been applied in the optical devices and have been considered to be more compatible with the fabrication process in the development of the optical devices (Chimalawong et al., 2012). However, the usage of the silicate based glasses might be limited since silicate based glasses possess a high energy of phonons which might limits the contribution of rare earth ions which are optically active for optical devices (Jose et al., 2006) hence will lessen and restrict the ability of optical devices.
Some modifications are needed in order to enhance the ability of optical devices. The usage of other glass systems may lead to an interesting application such as lasers, luminescence materials and also in solid state lighting applications. Apart from the silicate based glasses, the tellurite based glasses are very promising materials and have been a subject of highly interest for optical devices since tellurite based glass possess high refractive index and low phonon energy (Rajeswari et al., 2010). Addition of other glass former and rare earth oxide into the tellurite based glasses are essential in order to improve the properties of the tellurite based glass systems.

It is known that Dy_2O_3 possesses a high potential to enhance the luminescence properties of tellurite based glass systems (Tuyen et al., 2016). The nanoparticles are also known as a promising materials to improve the optical properties of tellurite based glass. The zinc borotellurite glass doped with dysprosium oxide and dysprosium oxide nanoparticles are expected suitable for the white light LED application since the dysprosium itself can act as a promising active luminescence center. Using a glass material with low phonon energy is more suitable to be used in the optical devices since a low phonon energy glasses enable higher quantum efficiency of photoluminescence and higher lifetime of the exited states.

The technological applications of the tellurite based glasses as optical devices are still ongoing, so a better understanding of the optical properties of the tellurite based glasses could lead to further applications of tellurite glass systems. The research on the effect of the dysprosium oxide and dysprosium oxide nanoparticles on the zinc borotellurite glass systems are not yet vigorously discovered by the other researchers. In other words, there are still limited to numbers in the study of dysprosium oxide and dysprosium oxide nanoparticles doped zinc borotellurite glass systems. Therefore, by taking into

consideration of the advantages of tellurite glasses as well as the industrial importance of Dy^{3+} ion, the investigations on Dy_2O_3 and Dy_2O_3 NP doped zinc borotellurite glass system are necessary since there are still limited information to support their future optical applications.

1.5. Research objectives

Based on the problem statements stated above, the objectives of this research are:

i. To determine the physical and structural properties of the zinc borotellurite glass systems doped with Dy_2O_3 and Dy_2O_3 NP at different concentration of dopants
ii. To investigate the optical properties of zinc borotellurite glass doped with Dy_2O_3 and Dy_2O_3 NP at various concentration of dopants
iii. To compare the optical properties between the zinc borotellurite glass doped with Dy_2O_3 and zinc borotellurite glass doped with Dy_2O_3 NP at various concentration of dopants

1.6. Hypotheses

i. The addition of Dy_2O_3 and Dy_2O_3 NP into the zinc borotellurite glass systems are expected to increase the density of the glass systems due to the high molecular weight of Dy_2O_3 and Dy_2O_3 NP. The molar volume for both series of the glass systems are expected to decrease due to its relationship which is inversely proportional to the density of the glass systems. The XRD patterns for both of the glass systems are expected to exhibit a broad hump without sharps peaks which indicates the amorphous nature of the glass systems. Besides that, the inclusion of the Dy_2O_3 and Dy_2O_3 NP into the zinc borotellurite glass network are expected to reveal the existence for BO_3, BO_4, TeO_3 and TeO_4 vibrational groups.
ii. The addition of Dy_2O_3 and Dy_2O_3 NP into the zinc borotellurite glass systems are expected to improve the optical properties of the glass systems by decreasing the indirect and direct optical band gaps while increasing the Urbach energy, refractive index, electronic polarizability and oxide ion polarizability. These expected results are the evident that the addition of Dy_2O_3 and Dy_2O_3 NP into the zinc borotellurite matrices might have contributes to more number of non-bridging oxygens since they are strongly polarizable. Other than that, the inclusion of the modifier into the glass matrices might be able to enhance the optical properties of the glass systems because it will breaks the Te-O-Te and B-O-B linkages and creates non-bridging oxygens. On the other hand, the emission spectra of Dy_2O_3 and Dy_2O_3 NP are expected to be observed at transition from $^4F_{9/2}$ to $^6H_{15/2}$ and $^6H_{13/2.}$
iii. The optical band gaps of the zinc borotellurite glass systems doped with Dy_2O_3 NP are expected to be larger compared to the zinc borotellurite doped with Dy_2O_3 glass systems. The larger optical band gaps of the Dy_2O_3 NP doped zinc borotellurite glass systems are expected to reduce the other optical parameters. This might be due to the characteristics of nanoparticles which are made up of the only finite number of particles thus results in the decrement of the overlapping orbitals and causes the width of the bands starts to narrow.

1.7. Thesis overview

This thesis is organized into six chapters. Chapter one has the introduction of the zinc borotellurite glass systems and the dysprosium oxide as well as the brief explanation regarding the formation of the glass. The problem statements of the research have been stated throughout this chapter. From the problem statements, three objectives are listed in this chapter. In chapter two, the general findings regarding the glass structure from the previous research are reported. The reviews on the physical, structural and optical properties of the glass systems are also reported. Next in chapter three, the general theoretical approach on the parameters used in this study are elaborated. Then, chapter four discusses the method of the glass fabrication and also the overview on the instruments used for the glass characterization. The full analysis and discussion on the findings and the results obtained are discussed in detailed in chapter five. Finally, the conclusions are made based on the results obtained and some suggestions and recommendations for the future works are provided in chapter six.

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