UNIVERSITI PUTRA MALAYSIA

LINEAR AND NON-LINEAR OPTICAL PROPERTIES OF ZINC BOROTELLURITE GLASS DOPED WITH ERBIUM, ERBIUM NANOPARTICLES, NEODYMIUM AND NEODYMIUM NANOPARTICLES

MUHAMMAD NOORAZLAN BIN ABD AZIS

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By

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

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

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March 2016

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Faculty : Science

The multi-compositions of RE (Er_2O_3, Er_2O_3 nanoparticles, Nd_2O_3, Nd_2O_3 nanoparticles) doped zinc-borotellurite glass with chemical composition of $\{[(TeO_2)_{0.70} (B_2O_3)_{0.30}]_{0.7} (ZnO)_{0.3}\}_{1-y} (RE)_y$, y = 0.005, 0.01, 0.02, 0.03, 0.04 and 0.05 were synthesized from high purity raw materials by using conventional melt quenching method. The physical, structural, linear and nonlinear optical properties of the glass system were characterized by using densimeter, X-ray diffraction (XRD), Fourier transform infra-red (FTIR), transmission electron microscopy (TEM), Ellipsometer, UV-Visible spectrophotometer, photoluminescence, and Z-scan technique. The amorphous nature of all the glass samples is confirmed by using X-ray diffraction (XRD) analysis. The presence of nonbridging oxygens in the glass network is obtained from FTIR analysis that formed as TeO_3 and BO_3 structural units. The presence of erbium nanoparticles and neodymium nanoparticles in the range 15 – 30 nm in the glass network are proved from TEM images. The values of density of the glass samples are found increases with increasing content of erbium (3.650 – 3.690 kg/m^3), erbium nanoparticles (4.588 – 4.881 kg/m^3), neodymium (3.672 – 3.931 kg/m^3) and neodymium nanoparticles (3.719 – 3.936 kg/m^3). The increasing trend of density is mainly attributed to the high value of the atomic mass of the dopants than the tellurite oxide. It is observed that the values of molar volume of the glass samples are increased with increasing concentration of erbium (32.483 – 32.955 m^3/mol), erbium nanoparticles (25.868 – 26.737 m^3/mol), neodymium (32.258 – 32.612 m^3/mol) and neodymium nanoparticles (31.850 – 32.571 m^3/mol). The obtained trend is due to the greater value of ionic radii of the dopants than the tellurite oxide. The values of refractive index are found increased with increasing concentration of erbium (1.716 – 1.740), erbium nanoparticles (1.774 – 1.924), neodymium (1.760 – 1.863) and neodymium nanoparticles (1.947 – 2.046). This trend is due to the high value of density. There are several absorption band observed from the UV-Vis spectra of the glass samples which were caused by 4f-4f transitions in erbium, erbium nanoparticles, neodymium and neodymium nanoparticles. The values of optical band gap are decreased along with erbium (3.650 – 3.68 eV), erbium nanoparticles (4.440 – 4.360 eV), neodymium (3.184 – 3.151 eV) and neodymium nanoparticles (3.209 – 3.178 eV) concentration which are due to the high number of free electrons as the number of nonbridging oxygen increases. The non-linear trend of Urbach energy values is obtained in the glass samples (erbium: 0.18 – 0.153 eV, erbium nanoparticles: 0.265 – 2.76 eV, neodymium: 0.316 – 0.320 eV, neodymium nanoparticles: 0.316 – 0.323 eV). The electronic polarizability of the glass samples is increased with increasing content of erbium (5.071 – 5.274 Å), erbium nanoparticles (4.28 – 5.03 Å), neodymium (5.265 – 5.843 Å) and neodymium nanoparticles (6.091 – 6.655 Å). The values of oxide ion polarizability (erbium: 2.185 – 2.148 Å, erbium nanoparticles: 1.77 – 2.02 Å, neodymium: 2.279 – 2.361 Å, neodymium nanoparticles: 2.710 – 2.774 Å) and optical basicity (erbium: 1.195 – 1.181, erbium nanoparticles: 1.012 – 1.004, neodymium: 1.401 – 1.173, neodymium nanoparticles: 1.191 – 1.170) are found in nonlinear variations of all the glass samples. The glass samples are found to be more conductive as the values of metallization criterion (erbium: 0.607 – 5.967, erbium nanoparticles: 0.580 – 0.530, neodymium: 0.589 – 0.549, neodymium nanoparticles: 0.518 – 0.485) increases along with dopants concentrations. The Judd-Ofelt parameters of erbium and erbium nanoparticles doped zinc borotellurite glass are shown to follow the trend of $\Omega_2>\Omega_4>\Omega_6$. Meanwhile, the Judd-Ofelt parameters of neodymium and neodymium nanoparticles doped zinc borotellurite glass are shown to follow the trend of $\Omega_2 < \Omega_4 <\Omega_6$. Green emission is found in erbium and erbium nanoparticles doped zinc borotellurite glasses under 385 nm excitation wavelength. Orange and red emission peaks are found in neodymium and neodymium nanoparticles doped zinc borotellurite glasses respectively. The violet and green emission of upconversion are found in erbium doped zinc borotellurite glass, green emission of upconversion is found in erbium nanoparticles dopant, violet color of upconversion was found in neodymium doped zinc borotellurite glass and blue emission of upconversion is found in neodymium nanoparticles doped zinc borotellurite glass. Erbium and erbium nanoparticles doped zinc borotellurite glasses exhibit negative NLR, meanwhile neodymium and neodymium nanoparticles doped zinc borotellurite glasses exhibit positive NLR. The variations of the NLA of the glass samples are found to be nonlinear.

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

SIFAT OPTIK LINEAR DAN TIDAK LINEAR BAGI KACA ZINK BOROTELLURIT TERDOP DENGAN ERBIUM, NANOZARAH ERBIUM, NEODIMIUM DAN NANOZARAH NEODIMIUM

Oleh

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Multikomposisi bagi RE (Er_2O_3, nanozarah Er_2O_3, Nd_2O_3, nanozarah Nd_2O_3) terdop kaca zink-borotellurit dengan komposisi kimia $\{[(TeO_2)_{0.70} (B_2O_3)_{0.30}]_{0.7} (ZnO)_{0.3}\}_{1-y} (RE)_y$, y = 0.005, 0.01, 0.02, 0.03, 0.04 dan 0.05 telah disintesis dari bahan mentah berketulenan tinggi dengan menggunakan kaedah lebur lindap konvensional. Sifat fizikal, struktur, optik linear dan tidak linear bagi sistem kaca telah dicirikan dengan menggunakan densimeter, pembelauan sinar-X (XRD), jelmaan Fourier inframerah (FTIR), mikroskopi penghantaran elektron (TEM), elipsometer, spektrofotometer UV-nampak, kefotopendarcahayaan, dan teknik imbasan-Z. Sifat semula jadi amorfus bagi kesemua sampel kaca telah disahkan dengan menggunakan analisis pembelauan sinar-X (XRD). Kehadiran oksigen bukan penitian di dalam jaringan kaca telah diperoleh daripada analisis FTIR yang membentuk sebagai TeO_3 dan BO_3 unit stuktur. Kehadiran nanozarah erbium dan nanozarah neodimium di dalam julat 15 – 30 nm di dalam kaca telah dibuktikan melalui imej TEM. Nilai ketumpatan sampel kaca telah dijumpai bertambah dengan pertambahan kandungan erbium (3.650 – 3.690 kg/m^3), nanozarah erbium (4.588 – 4.881 kg/m^3), neodimium (3.672 – 3.931 kg/m^3) dan nanozarah neodimium (3.719 – 3.936 kg/m^3). Trend pertambahan ketumpatan adalah kebanyakkannya ditentukan oleh ketinggian nilai jisim atom daripada dopan berbanding tellurit oksida. Telah dicerap bahawa nilai isipadu molar bagi sampel kaca adalah bertambah dengan pertambahan kepekatan erbium (32.483 – 32.955 m^3/mol), nanozarah erbium (25.868 – 26.737 m^3/mol), neodimium (32.258 – 32.612 m^3/mol) dan nanozarah neodimium (31.850 – 32.571 m^3/mol). Trend yang telah diperolehi adalah disebabkan daripada nilai jejari ion oleh dopan yang lebih besar berbanding tellurit oksida. Nilai indeks biasan telah dijumpai bertambah dengan pertambahan kepekatan erbium (1.716 – 1.740), nanozarah erbium (1.774 – 1.924), neodimium (1.760 – 1.863) dan nanozarah neodimium (1.947 – 2.046). Trend ini adalah disebabkan oleh ketinggian nilai ketumpatan. Beberapa jalur penyerapan telah dicerap daripada spektrum UV- nampak oleh sampel kaca yang disebabkan daripada transisi 4f-4f di dalam erbium (3.650 – 3.68 eV), nanozarah erbium (4.440 – 4.360 eV), neodimium (3.184 – 3.151 eV) dan nanozarah neodimium (3.209 – 3.178 eV). Nilai jurang jalur optik telah berkurangan selari dengan kepekatan erbium, nanozarah erbium, neodimium dan nanozarah neodimium yang disebabkan oleh ketinggian nilai elektron bebas apabila nilai oksigen bukan penitian bertambah. Trend tidak linear oleh tenaga Urbach telah diperolehi di dalam sampel kaca (erbium: 0.18 – 0.153 eV, nanozarah erbium: 0.265 – 2.76 eV, neodimium: 0.316 – 0.320 eV, nanozarah neodimium: 0.316 – 0.323 eV). Kebolehkutuban elektronik oleh sampel kaca telah berkurangan dengan pertambahan kandungan erbium (5.071 – 5.274 Å), nanozarah erbium (4.28 – 5.03 Å), neodimium (5.265 – 5.843 Å) dan nanozarah neodimium (6.091 – 6.655 Å). Nilai kebolehkutuban ion oksigen (erbium: 2.185 – 2.148 Å, nanozarah erbium: 1.77 – 2.02 Å, neodimium: 2.279 – 2.361 Å, nanozarah neodymium: 2.710 – 2.774 Å) dan kebesan optik (erbium: 1.195 – 1.181, nanozarah erbium: 1.012 – 1.004, neodimium: 1.401 – 1.173, nanozarah neodymium: 1.191 – 1.170) telah dijumpai dalam variasi tidak linear dari semua sampel kaca. Sampel kaca telah dijumpai cederung untuk menjadi lebih konduksi apabila nilai kriteria pelogaman (erbium: 0.607 – 5.967, nanozarah erbium: 0.580 – 0.530, neodimium: 0.589 – 0.549, nanozarah neodimium: 0.518 – 0.485) bertambah selari dengan kepekatan dopan. Parameter Judd-Ofelt oleh neodimium dan nanozarah neodimium terdop kaca zink borotellurit telah menunjukkan mengikuti trend $\Omega_2 > \Omega_4 > \Omega_6$. Manakala, parameter Judd-Ofelt oleh neodimium dan nanozarah neodimium telah menunjukkan mengikuti trend $\Omega_2 < \Omega_4 < \Omega_6$. Pancaran hijau telah dijumpai di dalam erbium dan nanozarah erbium terdop kaca zink borotellurite di bawah pengujaan panjang gelombang 385 nm. Puncak pancaran jingga dan merah telah dijumpai di dalam neodimium dan nanozarah neodimium terdop kaca zink borotellurit masing-masing. Pancaran unggu dan hijau oleh penukaran naik telah dijumpai di dalam erbium terdop kaca zink borotellurit, pancaran hijau oleh penukaran naik telah dijumpai di dalam nanozarah erbium, pancaran unggu oleh penukaran naik telah dijumpai di dalam neodimium terdop kaca zink borotellurit dan pancaran biru oleh penukaran naik telah dijumpai di dalam nanozarah neodimium terdop kaca zink borotellurit. Erbium dan nanozarah erbium terdop kaca zink borotellurit menunjukkan indeks biasan tidak linear negatif, manakala neodimium dan nanozarah neodimium terdop kaca zink borotellurit menunjukkan indeks biasan tidak linear positif. Variasi pekali penyerapan oleh sampel kaca adalah didapati tidak linear.

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

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ε_0	Permittivity in vacuum (8.85 x 1012)	Fm^{-1}
ω	Radian Frequency	Fm^{-1}
χ	Susceptibility	-
A	Area	m^2
d	Thickness	m
T_g	glass transition temperature	^0C
NBO	Non Bridging Oxygen	-
BO	Bridging Oxygen	-
ΔE	Energy	Joule (J)
ɳ	Refractive index	-
λ	Wavelength	m
XRD	X-ray Diffraction	-
E_{opt}	Optical energy gap	Joule (J)/eV
$\hbar\nu$	Photon energy	Joule (J)/eV
E_c	Urbach energy	Joule (J)/eV
MR	Molar Refraction	-
JO	Judd-Ofelt	-
NLR	Nonlinear refractive index	-
NLA	Nonlinear absorption	-
Nd	Neodymium	-
Er	Erbium	-
TEM	Transmission Electron Microscopy	-

CHAPTER 1

INTRODUCTION

1.1 Preamble

The extensive research on glass science and technology that explore the new findings on photonic and optical applications is undeniable. The novel findings on glass applications are reported continuously. The excitement in the investigation of the novel glass applications motivates the researchers around the world. The high demand and interest on communication system increase the development of potential glass materials to be applied in optical communications.

There is various kind of glass materials are being developed for optical applications. The silicate-based glass is currently used as the main core in fiber optics. Besides that, silicate based glass has high melting point, weak absorbance, and weak nonlinearity. The high quality of optical glass is essential to improve the current optical applications. Whilst tellurite-based glass is the best candidate as a high quality of glass materials.

The tellurite-based glass appears to be one of the unique and high demand glass materials as it has high refractive index, high dielectric constant, a wide band infrared transmittance and large third order nonlinear optical susceptibility. The tellurite-based glass had been widely developed in many optical applications such as fiber optics, optoelectronics, light emitting diode (LED), and glass sensors. Moreover, the improvement of tellurite-based glass in optical applications is still in progress. The tellurite-based glass is compatible with many oxides materials. Various oxides had been added in tellurite glass network to improve their optical properties.

The glass formation strongly depends on the cooling rate and the size of the melt, especially in the TeO_2-rich region (Sidek *et al.*, 2009). The zinc containing tellurite glass system is used as a basis for multi-component optical glass synthesis and a good candidate for super heavy optical flint glass (Sidek *et al.*, 2009). Lanthanide (rare-earth) oxide group is the best candidate to enhance the optical properties of tellurite-based glass. The recent research on rare-earth oxide doped tellurite glass has been extensively discovered due to their potential applications in many areas, especially in optical communication.

Rare earth doped fiber amplifier is used to enhance the optical communication, and one of the applications is Er^{3+}-doped fiber amplifier (EDFA) devices. The photonic-based system is an excellent application for ultra-high speed transfer and processing. Glass is one of the examples of the system application because of its superior properties of tunable nonlinear optical (NLO) properties and low phonon cut energy (Duffy and Ingram, 1971). The demand for such properties increases the development of photonic materials. Designing the photonic-based systems require the application of nonlinear optical (NLO) properties. One of the most significant properties in the field of optical and electronics is electronic polarizability of ions (Duffy and Ingram, 1971).

Nonlinear optical (NLO) properties are monitored by the electronic polarization of material in the exposure of intense light beams which can be related to several properties of the material such as electro-optical effect, conductivity and refractivity (Rafaella *et al*., 2001). The new kind of materials in which possesses excellent nonlinear optical (NLO) properties has to be found to design the photonic based system, which are accessible and understandable.

1.2 Introduction of glass

Alemi *et al*. (2009) defined glass as a homogeneous material with a random, liquid-like (noncrystalline) molecular structure. Meanwhile, Rawson (1980) stated that glass is an X-ray amorphous material that exhibits glass transition. Glass had been used in many applications since ancient time. The formation of natural glass is from the cooling of molten in various compounds including alkali, alkali earth and transition metal oxides. The rules of glass formation had been proposed by Zachariasen (1932). Zachariasen summarized that the formation of glass may occur if:

1. Each oxygen atom is linked to no more than two cations
2. The cation coordination number is small: 3 or 4
3. Oxygen polyhedral share corners, not edges or faces
4. For 3D networks, at least three corners must be shared

Moreover, he stated that the melt must be cooled under suitable conditions to allow the formation of glass. The glass structure consists of random network and amorphous structural arrangement as shown in Figure 1.1.

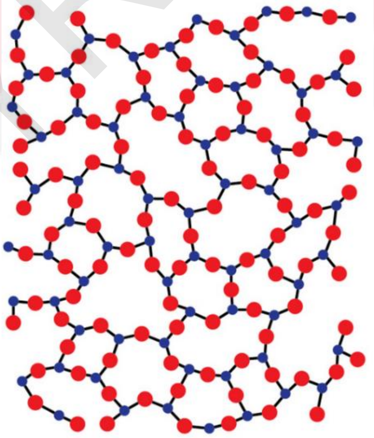

Figure 1.1: Basic glass structure (Stachurski, 2011)

Glass system does not consist of any crystalline phase, but it has high rigidity while retaining its liquid structure. The atoms in a liquid form are connected to one another, and they form in a random structure. There are two mechanisms occur when a liquid is heated which are;

1. The vibration of atoms
2. The movement of atoms or molecules in a random motion

When the temperature is lowered, the heat energy is slowly lost until the freezing point takes place. As a consequence, the liquid is changed to solid in which the structure becomes regular order. Besides that, if the liquid is cooled down from high temperature to room temperature, the structure will become rigid and maintain the internal structure of a liquid. This process is known as a “supercooled” liquid.

The state of the supercooled liquid is in the state of metastable. At the freezing point, the liquid is turned to a solid state. In this process, the internal energy is lowered until it reaches a minimum point and achieve a stable state. A supercooled liquid is not in a stable state, but it can achieve the stable state by passing through an intermediate state at higher energy. Thus, the supercooled liquid can form glass if the internal energy is supplied in which the crystalline state is attained. The supercooled liquid in which has the possibility to form a glass (glass-forming liquid) may show a rapid increment in stiffness or viscosity as the temperature is lowered down at below than the melting point.

At below the melting point, the glass-forming liquid needs to be cooled down rapidly to make sure the glass-forming liquid does not reach crystalline phase. The slow cooling process may result in the formation of the crystalline phase, and the glass system is not purely amorphous.

Figure 1.2 shows the mechanisms of the glass-forming process. Starting with the liquid at A, freezing would be expected to occur at a temperature corresponding to point B. From A to B the liquid contracts by two means. The amplitude of the vibratory motion of the atoms decreases as the temperature falls, and this has the effect of reducing the interatomic spacing and so causing the material to contract slightly. As well as this normal thermal contraction there is configurational shrinkage. Rearrangement of the interatomic bonds occurs as the temperature is reduced to reach a stable configuration at any particular temperature. This effect causes the material to assume a less open structure that occupies less space, (if the material crystallizes at B there will be a sudden decrease in volume, followed by thermal contraction only from C to D (room temperature). The crystal undergoes no configurational change.

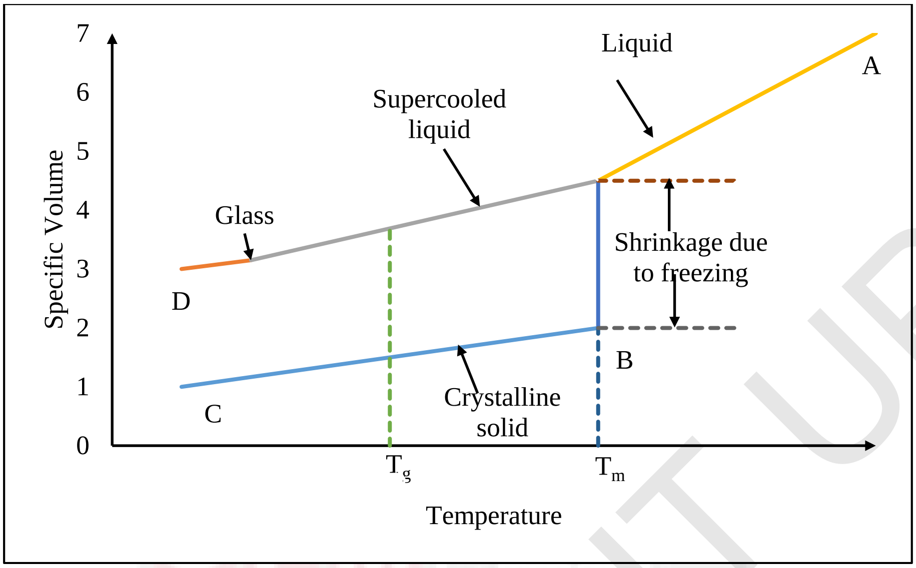

Figure 1.2: Glass transition diagram (Stachurski, 2011)

Figure 1.3 shows the transformation temperature varies with cooling rate. A rapidly cooled glass follows line 2. If it is cooled more slowly than glass 2, the configurational shrinkage can keep pace with the cooling to a lower temperature (line 1) and the final volume occupied by the glass at room temperature is smaller than glass 2. Thus, the rate of cooling affects its final internal structure. The faster the glass is cooled, the higher the temperature at which configurational rearrangement effectively ceases.

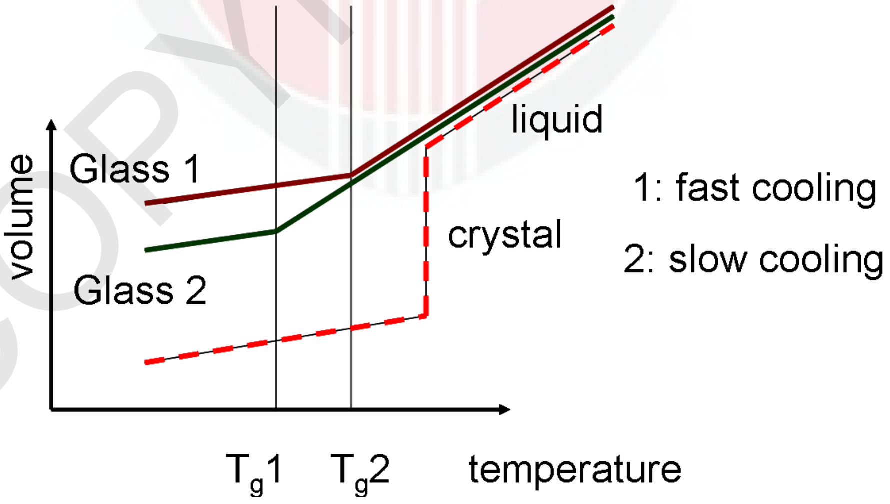

Figure 1.3: Variation of final glass volume with cooling rate (Stachurski, 2011)

1.3 Problem statements

The investigations on the new fiber optics materials with superior properties than silica based fiber optics have been made since many years ago. Only a small relative numbers of research have been investigated. The applications of silica fibers are still limited to non-telecommunications and short-haul applications which are due to their low optical and mechanical properties. Moreover, the current silica fibers are only applicable to tens of meters of fiber to be suited for telecommunications applications rather than kilometers of length.

The fiber cable consists of high light packed in which the silica fiber is not able to withstand the high intensity of light. The high vibrations of atoms in the silica fiber at high power lead to the conversion of light energy into sound energy. This effect will reduce the capabilities of fiber optics to transmit more power. This limitation will lessen the ability of fiber optics to transmit light and restrict the number of information that can be transmitted in a telecommunications system.

Heavy metal tellurite glasses are the excellent materials to replace the current silica fibers and to be used as fiber amplifiers and lasers. The future applications of tellurite-based glass may be more promising in fiber optics when doped with rare earth materials that enhance their ability in a telecommunications system. Moreover, rare-earth doped tellurite glass is very useful in evanescent wave spectroscopy (EWS). One of the advantages of an IR fiber EWS sensor is that the infrared region of the spectrum is adamant.

Broadband optical amplifiers to improve the bandwidth is highly required in a future communications system. The research is more efficient and excellent optical glass materials, however, is still ongoing. The promising materials of rare-earth doped tellurite glass have driven the improvement in the communication system. The growth of bandwidth will require the investigations of new materials.

The tellurite-based glass is among the higher potential materials than silicate based glass which are due to the broadband emission cross section. Such properties can allow the tellurite-based glass materials to achieve excellent optical efficiency than in other oxide glasses. There are still limited to numbers in the study of rare-earth and rare-earth nanoparticles doped tellurite-based glass system. It is known that erbium oxide and neodymium oxide possess the high potential to enhance the optical properties of tellurite-based glass.

The nanoparticles are also known as the promising materials to improve the optical properties (optical absorption, refractive index and optical nonlinearity) of tellurite-based glass. Therefore, the investigations on erbium, erbium nanoparticles, neodymium and neodymium nanoparticles doped tellurite-based glass system are still needed since there are limited data to support their future optical applications.

1.4 Research Objectives

The research objectives are;

1. To synthesize the glass samples with compositions $\{[(TeO_2)_{0.3}(B_2O_3)_{0.7}]_{0.7}(ZnO)_{0.3}\}_{1-y}(RE)_y$; RE: Nd_2O_3 microparticles, Nd_2O_3 nanoparticles, Er_2O_3 microparticles, Er_2O_3 nanoparticles; y = 0.005, 0.01, 0.02, 0.03, 0.04, 0.05; by using melt-quenching method

2. To determine the refractive index, optical absorption, optical band gap and Urbach energy of erbium, erbium nanoparticles, neodymium and neodymium nanoparticles doped zinc borotellurite glass system

3. To analyze the electronic polarizability, oxide ion polarizability, optical basicity and metallization criterion of erbium, erbium nanoparticles, neodymium and neodymium nanoparticles doped zinc borotellurite glass system

4. To study the Judd-Ofelt parameters, visible and upconversion luminescence of erbium, erbium nanoparticles, neodymium and neodymium nanoparticles doped zinc borotellurite glass system

5. To characterize the nonlinear refractive index and nonlinear optical absorption of erbium, erbium nanoparticles, neodymium and neodymium nanoparticles doped zinc borotellurite glass system

1.5 Outline of thesis

Chapter are divided into sections as well as sub-sections. Chapter 1 is the overview of the research work and a brief introduction of glass materials. It is also emphasized the focus of the research and underline the objectives of the study. The crucial issues are stated in the problem statements. Chapter 2 review the previous reports related to the research work. It is also described the different perspectives and approaches related to this study. Chapter 3 described the theory that being used in this study. The derivation of equations is also presented in this chapter. Chapter 4 discussed in details the method of fabrication and collection of data. The overview of the instrument is also presented in this chapter. Chapter 5 highlighted the critical discussion of this study and the comparative result between the glass series and previous studies. The discussions are cover several type of measurements which are: X-ray diffraction (XRD), Fourier transform infrared (FTIR), Density, Transmission electron microscopy (TEM), UV-Visible spectroscopy, Photoluminescence, and Z-Scan technique. Chapter 6 summarized the important outcome of the research and suggestions of the future works.

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