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EFFECT OF LANTHANUM OXIDE ON OPTICAL PROPERTIES OF ZINC BOROTELLURITE GLASS SYSTEM

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A series of zinc borotellurite glasses doped with lanthanum oxide with the chemical composition $\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.7}(ZnO)_{0.30}\}_{1-x}(La_2O_3)_x$ where x = 0.01, 0.02, 0.03, 0.04, and 0.05 molar fraction have been fabricated using conventional melt-quenching method. The structural properties of the fabricated glass samples were determined by X-ray diffraction (XRD) analysis and Fourier Transform Infrared (FTIR) analysis. XRD result confirmed that the fabricated glasses are amorphous. Density and molar volume of the prepared samples were measured and calculated. The optical properties of the prepared glasses were determined by UV-Vis analysis. The optical absorption spectra reported that the fundamental absorption edge shifts to lower wavelength as the amount of La_2O_3 increases. The refractive index, direct optical energy band gap, indirect optical energy band gap and Urbach energy had been calculated and analyzed.

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Keywords: Borotellurite glass; Optical band gap; Optical absorption coefficient

1. Introduction

Lanthanide and lanthanide doped glasses have received many attentions as lanthanide doped glasses offer a large, new and important application possibility especially in the area of optical communication. In the 1940s and 1950s, many lanthanum oxide-based new optical glasses were investigated where at that time, lanthanum borate glass and lanthanum silico-borate glasses have a higher refractive index and lower dispersions than the lead silicate glasses [1]. Lanthanide ions ($Ln^{3+)}$ have electron structure of [Xe] $4f^n5s^25p^6$ where n range from 0 to 14. The *f* electrons are known to have a dominant influence on the optical properties [2].A variety of lanthanide doped glass that shows linear and nonlinear optical properties that are applicable for the devices in the field of photonics have been investigated during the last decades.

Glass formation process bypasses the process of crystallization and almost all material can be prepared as amorphous solid by quick cooling. The reason for the different properties among the glasses found and created nowadays was because of the composition of these glasses. All glass properties reflect the intrinsic nature of the composition of elements that has been added into the glass mixture.

In a glass formation process, zinc oxide reduces the melting point and increases the glass forming ability [3]. The participation of zinc oxide in the glass formation would decrease the optical energy gap, increase the refractive index and it also act as glass stabilizer. Borate oxide is known to be a superior host matrix for rare earth metal oxide because it able to become glass over a wide range of composition without the addition of network modifiers. Borate oxide is also known as the best choice to stabilize the glass formation due to its good rare earth ion solubility and hardness. The borate matrix possesses BO_3 triangles and BO_4 tetrahedra that able to form stable borate groups such as diborate, triborate and tetraborate [4]. The ability of boron to exist in

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three and four oxygen coordinated environments and the strong covalent B-O bonds enable borates to form stable glasses.

Tellurite is well known as the best host matrix due to its unique optical and electrical properties. Tellurium oxide itself is a conditional glass former, as it needs a modifier like an alkali, alkaline earth and transition metal oxide or other glass former in order to form the glassy state easily. The introduction of TeO_2 into the matrix of a glass will help in enhancing the infrared transmission and improving the quality of glass where the glass formed will be less brittle and most likely free from bubbles [5].

Lanthanum is one of the major rare earth elements, which occupies the 14.1% of total rare earth resources [6]. Lanthanum is used in many fields such as piezoelectric material, electric material, thermoelectric material, luminescence material, optical glass, laser materials and a variety of alloy materials. Lanthanum oxide is assumed to be a network modifier in various type of glass [7]. Lanthanum is an exceptional case in the electronic configuration where its electronic configuration is [Xe] $5d^1$ $6s^2$ instead of [Xe] $6s^2$ $4f^1$ which show that lanthanum does not have any 4f electron to begin with. Doweidar and Saddeek (2010) investigated the influence of La_2O_3 on the structure of lead borate glasses and reported that La^{3+} ion compensates the negative charge on glass former ions and form non-bridging oxygen ions in the glass network. As a result, the density increase and the molar volume decrease with increasing La concentration.

Lately, many researchers are trying to seek the best material to be used in photonic device by doping rare earth element in various glass compositions. Therefore, this research is concentrated on the effect of lanthanum oxide on the optical properties of zinc borotellurite glass system.

2. Experimental

Zinc borotellurite glasses doped with lanthanum oxide with the chemical composition $\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.7}(ZnO)_{0.3}\}_{1-x}(La_2O_3)_x$ where x = 0.01, 0.02, 0.03, 0.04, and 0.05 molar fraction have been fabricated using conventional melt-quenching method. The chemical powder of boron oxide, B_2O_3, (98.5%, Alfa Aesar), zinc oxide, ZnO, (99.99%, Alfa Aesar), tellurium (IV) oxide, TeO_2, (99.99%, Alfa Aesar) and lanthanum (III) oxide, La_2O_3, (99.99%, Alfa Aesar) were weighted at appropriate amount using a digital weighing machine with an accuracy of ±0.0001g.

The chemical powder mixture that been weight was transferred to the alumina crucible and mixed for 30 minutes to achieve homogeneity. Next, the mixtures in alumina crucible were put into an electric furnace for the preheating process at 400^oC for a period of 1 hour to remove water content in the chemical powder mixture.

The alumina crucible was next transferred to another electric furnace for 2 hours at 900^oC for melting process. At the same time, stainless steel split mould was polished and transferred to the first electric furnace for the pre-heating process at 400^oC.

After 2 hours, the molten chemical powders was poured into the pre-heated stainless steel mould and immediately transferred into the first furnace for annealing process at 400^oC for a period of 2 hours. The function of the annealing process is to remove air bubbles, reduce thermal strength and improve the mechanical strength of the glass samples. Last but not least, the electric furnace is switched off and the fabricated glass sample was left to be cooled to room temperature.

The prepared glass samples were cut to a thickness approximate to 2 mm using Isomet Buehler low speed saw machine and polished both side of the surface for optical absorption measurement. The polishing process will be done by using silicon carbide paper with different grid. The optical absorption was obtained by using UV-1650PC UV-Vis Spectrophotometer (Shimadzu) with the wavelength range from 220 to 2600 nm.

For the structural properties, the glass samples were crushed by using plunger, mortar and pestle to get a fine powder. The fine powder of glass sample was sent to X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) in order to analyze the structure of the glass sample.The density and molar volume of the fabricated glass samples were measured and calculated by using the Archimedes principle at room temperature using distilled water as the immersion liquid.

3. Result and Discussion

3.1 Density and molar volume

Density is an important tool that had been used to explore the degree of structural compactness of a substance [8] and the change in the geometrical configurations of the glass system. The density value of the glass samples will be used to calculate molar volume, molar refraction and optical parameters. The density of the glass samples can be obtained by using the following formula:

$$\rho_{\text{sample}} = \frac{W_{\text{air}}}{V} \tag{3.1}$$

where $\rho_{\text{water}} = 1$ g/cm^3 , W_{air} is the weight of glass sample in air (g) and V is the volume of glass sample (cm^3).

The calculated density of the prepared glass samples were listed in Table 1 and depicted in Figure 1. It can be observed that as the concentration of lanthanum oxide increases, the density increases. The increasing trend of the density might due to the replacement of lighter molecular mass with high molecular mass atom in the glass network leads to the increasing of density [7]. In this work, the replacement of lighter atomic mass of B_2O_3 with higher atomic mass of La_2O_3 leads to the increasing number of density of the glass samples.

Table 1: Density and molar volume of lanthanum oxide doped zinc borotellurite glass

Molar Fraction, x	Density (g/cm³)	Molar Volume (cm³/mol)
0.01	3.5439	33.6701
0.02	3.6252	33.4903
0.03	4.7381	26.0642
0.04	5.9748	21.0184
0.05	6.6431	19.2179

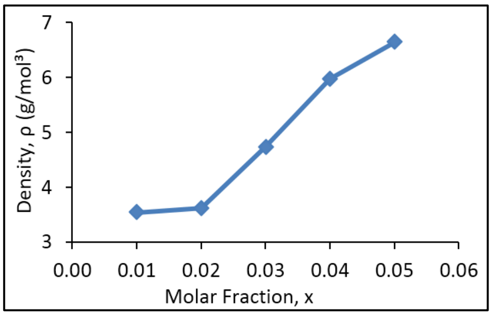

Fig. 1: Density for lanthanum oxide doped zinc borotellurite glass

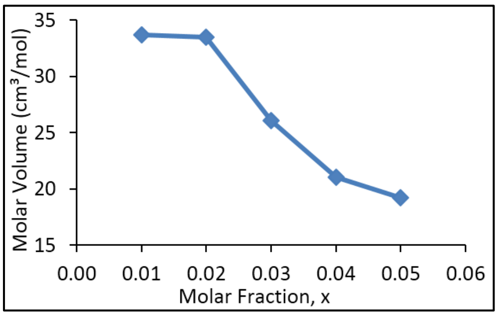

Fig. 2: Molar volume for lanthanum oxide doped zinc borotellurite glass

The molar volume of all glass samples can be calculated by using the following equation:

$$\text{Molar volume} = \frac{MW}{\rho} \quad (1)$$

where MW is the molecular weight of the glass sample (g/mol) and ρ is the density of the glass sample (g/cm^3).

The calculated molar volume of the prepared glass samples was recorded in Table 1. It can be observed that as concentration of lanthanum oxide increases, the molar volume decreases. It can be seen that the relationship between density and molar volume is inversed to each other which follow the theoretical relationship. The decrease in the molar volume is due to the decrease in the bond length on inter-atomic spacing between the atoms [11].

3.2 X-Ray Diffraction (XRD)

X-ray diffraction spectroscopy is the basic characterization technique to identify the crystalline phase in the glass sample. X-ray diffraction was based on the scattering and diffraction phenomena of an X-ray passed through the glass samples. The XRD spectra of the glass samples were recorded at 2θ angles in the range of $80° \geq \theta \geq 20°$.

Figure 3 show that the XRD spectra have no discrete or continuous sharp peaks. A broad hump is observed from Figure 3 that indicates the presences of long range structural disorder. The absence of sharp peaks in the XRD spectra indicate that there are no well-defined planes in the structure on or around which the constituent atoms are regularly arranged [11].Since there are no sharp line spectra were obtained in the XRD spectra, it shows that there is no evidence of crystalline phase in the glass samples. The broad hump pattern confirmed that the prepared glass samples are amorphous in nature.

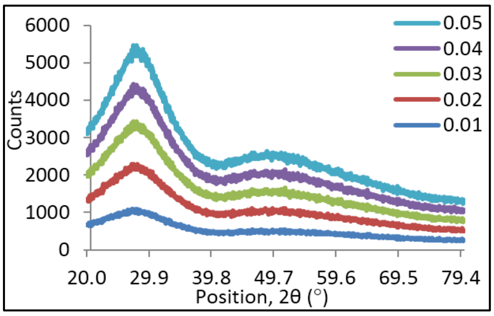

Fig. 3: XRD diffraction pattern of prepared glass sample with different concentration of lanthanum oxide

3.3Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier Transform Infrared (FTIR) spectroscopy is an analysis method which provides the structural studies to explore the fundamental and functional groups in crystalline and non-crystalline matrices [12]. FTIR exploits the fact that the molecules absorb specific frequencies which represent their characteristic of structural units. The frequency of the absorbed radiation matches the frequency of the bond or groups that vibrates. The result of FTIR spectroscopy is a spectral curve which is the characteristics of the elements and chemical bond that exist between them. Absorption peak in infrared spectrum represent the frequencies of vibrations between the bonds of the atom or molecule. There are no two compounds that produced the exact same infrared spectrum since each different material is a unique combination of atom. In addition, the size of the peak shows the amount of a particular material present.

The transmission spectra of the prepared glass samples are in between 280 and 2000 cm^{-1} region. The FTIR spectra of the glass samples are recorded in Figure 4. The observed broad bands are due to a combination of higher degeneracy of vibrational states, thermal broadening of the lattice dispersion and mechanical scattering of the powdered sample and the corresponding band assignments [13].

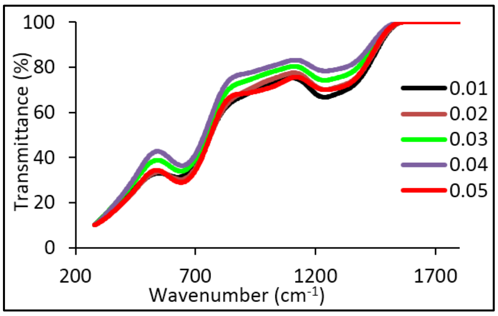

Fig. 4: FTIR spectra of prepared glass sample with different concentration of lanthanum oxide

From Figure 4, it can be seen that the spectra consist of several peaks specifying its local structure. The peak position and the assignments are recorded in Table 2. The transmission spectra of the prepared glass samples consist of two wide absorption bands at 631 – 645 cm^{-1} and 1239 – 1243 cm^{-1} while there is small absorption band at 1050 – 950 cm^{-1}from 0.05 molar fraction of lanthanum oxide.

Tellurite oxide consists of two types of structural configuration units that are trigonal bipyramid, TeO_4 and trigonal pyramid, TeO_3. Pure TeO_2 is characterized at around 640 cm^{-1}.The absorption band at 600 – 700 cm^{-1} is assigned by stretching vibration of Te-O bonds: trigonal bipyramid, TeO_3 and trigonal pyramid, TeO_3. The stretching vibration of TeO_3 group is between 650 – 700 cm^{-1} while stretching vibration of TeO_4 which in between 600 – 650 cm^{-1}. Thus, the broad peak in the range of 631 – 645 cm^{-1} detected form the prepared glasses indicates that TeO_4 group exists in all tellurite containing glass system.

The absorption band of pure borate glass, B_2O_3 is centered at 806 cm^{-1} which indicates the characteristic of boroxyl ring [13]. The absorption band of boroxyl ring disappears after the glass formation with the substitution of BO_3 and BO_4 structural units. The absorption spectra of the borate glass can be divided into 3 regions: 600 – 800 cm^{-1} which corresponds to the bending vibrations of various borate arrangements B-O-B, 800 – 1200 cm^{-1} that indicate B-O stretching of tetrahedral BO_4 units and 1200 – 1800 cm^{-1}which attributes to the B-O stretching of trigonal BO_3 units. In this experiment, the absorption band positioned at 1239 – 1243 cm^{-1} detected from the prepared glass samples is assigned to the trigonal B-O bond stretching vibrations of BO_3 units from boroxyl groups.

Table 2: Assignment of infrared transmission bands of prepared glass sample with different concentration of lanthanum oxide

No	0.01	0.02	0.03	0.04	0.05	Assignments
1	1239	1241	1239	1241	1243	Trigonal B-O bond stretching vibrations of BO_3 units from boroxyl groups. [12]
2	-	-	-	-	975	B-O bond stretching vibrations in BO_4 tetrahedral from tri-, tetra-, and penta – borate groups. [13]
3	631	637	639	645	640	TeO_4 group exist in all tellurite containing glass. [12]

3.4 Optical Absorption

The measurement of optical absorption and particularly the absorption edge is important for investigating optically induced transitions and for getting information about the band structure and energy gap of non-crystalline materials [9]. Absorption spectrum of the glass sample in ultraviolet and visible region was observed by using UV- Visible Spectrophotometer (UV-1650PC, SHIMADZU). The UV-Visible spectroscopy was carried out in the range 200 – 800 nm under room temperature.

The optical absorption spectra of $\{[(TeO_2)_{0.70}(B_2O_3)_{0.30}]_{0.7}(ZnO)_{0.3}\}_{1-x}(La_2O_3)_x$ are shown in the Figure 5. From Figure 5, it can be seen clearly that there are no sharp absorption peaks that indicates the characteristics of the glassy state. The change of oxygen bonding in the glass network, for instance, the formation of nonbridging oxygen, changes the characteristics absorption edge [11].

It is observed from Figure 5 that as the concentration of lanthanum oxide increases, the fundamental absorption edge tends to shifts to the lower wavelength and higher energy. This trend may be due to the higher rigidity of the glass system as a result of increasing concentration of lanthanum oxide [14]. Besides that, the shifting of absorption band to lower energy can be related to the decreasing formation of nonbridging oxygen that binds electrons of nonbridging oxygen less tightly than bridging oxygen [15]. As the atomic number of the lanthanide ion increases, all these absorption bands shift to low wavenumbers also because of increasing field strength of the La^{3+} ion [2].

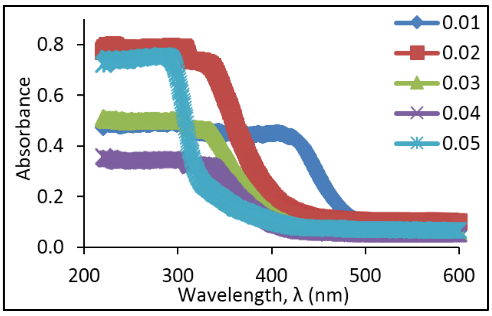

Figure 5: Optical absorbance spectra for lanthanum oxide doped zinc borotellurite glass

The optical absorption coefficient, α(*w*) can be calculated using absorbance through the equation:

$$\alpha(w) = 2.303(\frac{A}{d}) \tag{3.3}$$

where d is the thickness of the glass sample in cm and A is the absorbance.

Optical absorption result that was obtained from UV-Vis is important in the process of determining the optical band gap energy and Urbach energy. Mott and Davis proposed the relation between absorption coefficient with photon energy to calculate direct and indirect transition occurred in the band gap [12]. The equation can be generally written as follows:

$$\alpha(w) = \frac{B(\hbar\omega - Eopt)^n}{\hbar\omega} \tag{3.4}$$

where $\hbar = 4.14 \times 10^{-15}$ eV, n = 2 for indirect transition and n = ½ for direct transition.

In amorphous material, the value of indirect band gap can be obtained by using the Equation (3.5) while the value of direct band gap can be obtained using the Equation (3.6).

$$(\alpha\hbar\omega)^{1/2} = B\,(\hbar\omega - E_{opt}) \tag{3.5}$$

$$(\alpha\hbar\omega)^2 = B\,(\hbar\omega - E_{opt}) \tag{3.6}$$

Equation (3.5) is used to plot$(\alpha\hbar\omega)^{1/2}$ versus $\hbar\omega$ to determine the indirect optical band gap (Figure 6). Meanwhile, Equation (3.6) is used to plot$(\alpha\hbar\omega)^2$ versus $\hbar\omega$ to get the values of the direct optical band gap (Figure 7).

The values of indirect optical band gap E_{opt}^{1} and direct optical band gap E_{opt}^{2} can be obtained by the intercepts of the extrapolating of the linear portion of the curve with the linear axis which represents the photon energy [13]. Figure 8 and Figure 9 show the direct and indirect band gap of the glass samples respectively. The values of the direct and indirect band gap are tabulated in Table 3. Both direct and indirect band gap have same increasing trends along with lanthanum oxide.

Optical band gap for both direct and indirect depend on the changes in the structure of glass after the addition of modifier [5]. The formation of TeO_4 in the glass samples leads to the tightly bound of oxygen anions to the host materials. Hence, the optical band gap increases with the increasing number of bridging oxygen in the glass system. Moreover, the increasing value of optical band gap might be due to decreasing number of non-bridging oxygen [13].

Table 3: Indirect optical band gap (E^1_{opt}), Direct optical band gap (E^2_{opt}) and Urbach energy of lanthanum oxide doped zinc borotellurite glass

Molar Fraction, x	Indirect Band Gap, E^1_{opt}, (eV)	Direct band gap, E^2_{opt} (eV)	Urbach energy, ∆E (eV)
0.01	2.20	2.20	0.47
0.02	2.49	3.10	0.45
0.03	2.60	3.20	0.42
0.04	2.61	3.30	0.41
0.05	3.43	3.90	0.33

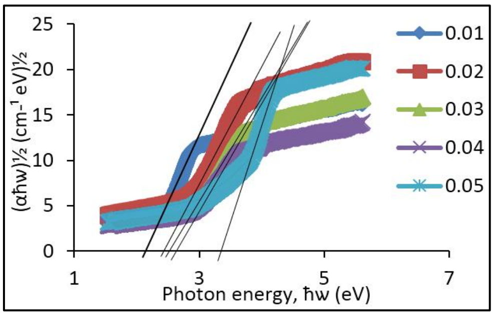

Fig 6: Plot of $(\alpha\hbar\omega)^{1/2}$ versus photon energy, $\hbar\omega$ of lanthanum oxide doped zinc borotellurite glass for indirect band gap measurement.

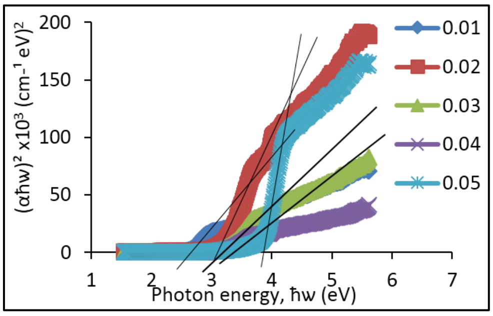

Fig 7: Plot of $(\alpha\hbar\omega)^2$ versus photon energy, $\hbar\omega$ of lanthanum oxide doped zinc borotellurite glass for direct band gap measurement

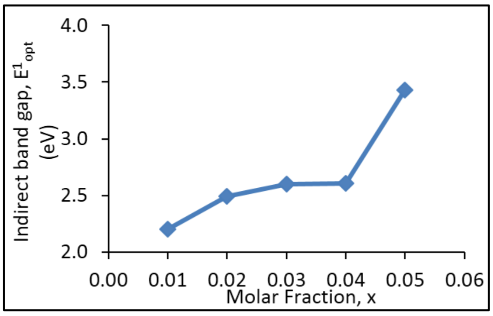

Fig. 8: Variation of indirect optical band gap with various glass compositions for lanthanum oxide doped zinc borotellurite glass

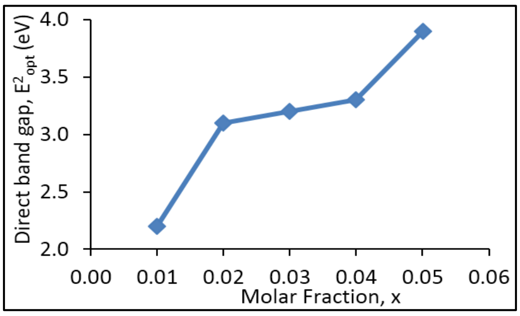

Fig. 9: Variation of direct optical band gap with various glass compositions for lanthanum oxide doped zinc borotellurite glass

Urbach energy (ΔE) gives information on the degree of disorder in the amorphous and crystalline materials. Materials will have greater tendency to convert weak bonds into defects when it has larger Urbach energy [11]. The observed linear correlation of optical band gap energy enable the calculation of Urbach energy, ΔE [16]:

$$\alpha(w) = ß \exp \frac{\hbar v}{\Delta E} \tag{3.7}$$

where ß is a constant, ħ is the plank constant, v is the photon frequency and ΔE is the Urbach energy.

The values of ΔE were calculated by taking reciprocal of the slopes of linear portion of ln α against ħw as shown in Figure 10. The obtained Urbach energy is shown in Figure 11. It is found that the Urbach energy decreases with increase of La_2O_3 content. As a consequence, the fragility of the glass samples decreases. The low Urbach energy is related to the decrease of degree of disorderness in glass structure [5]. As the concentration of the lanthanum ion increases, the number of nonbridging oxygen and BO_3 units in the glass structure decrease, and as a result, the Urbach energy of the glass system is also decreased.

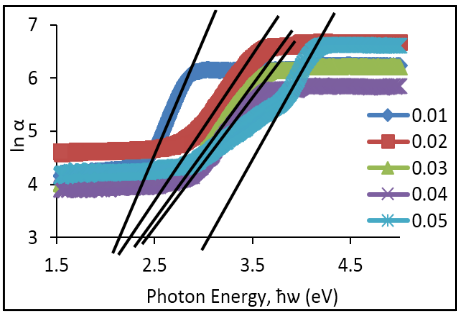

Fig. 10: Plot of ln α versus photon energy, ħω *of lanthanum oxide doped zinc borotellurite glass for Urbach energy measurement*

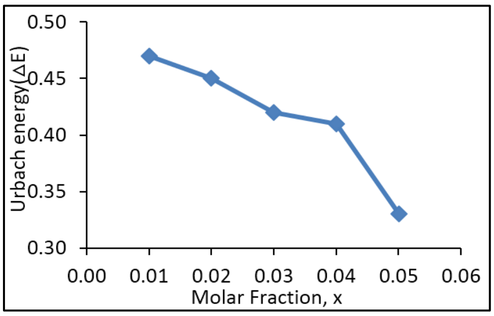

Fig. 11: Variation of Urbach energy with various glass compositions for lanthanum oxide doped zinc borotellurite glass

3.5 Refractive Index

Refractive index is an important property to be considered with respect to the optical features of a glass material [17].Refractive index value of a material can be used to determine the suitability of a glass material to be optical devices [18]. Thus, many researchers have started to investigate the relationship between refractive index and glass composition [19].The refractive index value of each prepared glass sample is calculated from the optical band gap value by using the equation proposed by Dimitrov and Sakka (1996).

$$\frac{n^2-1}{n^2+2} = 1 - \sqrt{\frac{E_{opt}}{20}} \qquad (3.8)$$

where n is the refractive index and E_{opt} is the value of indirect band gap.

The refractive index value is listed in Table 4 and plotted in Figure 12. Refractive index of a glass is related to density and polarizability of the constituent ions of the glass system [20]. From Table 4 and Figure 12, it can be clearly seen that the refractive index decreases with the increasing concentration of lanthanum oxide. This is explained by the decreasing number of nonbridging oxygen as the content of lanthanum oxide increases.

Table 4: Refractive index of lanthanum oxide doped zinc borotellurite glass

Molar Fraction, x	Refractive index, n
0.01	2.50
0.02	2.31
0.03	2.30
0.04	2.29
0.05	2.19

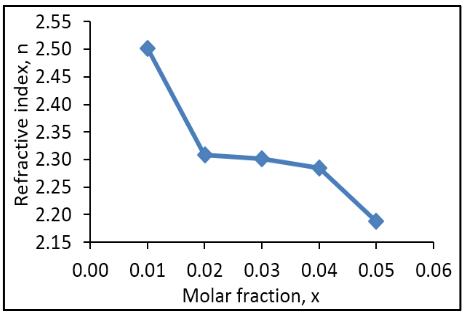

Fig. 12: Refractive index of lanthanum oxide doped zinc borotellurite glass

4. Conclusion

Zinc borotellurite glass doped lanthanum oxide had been successfully fabricated by using the conventional melt-quenching method. The XRD spectra show that the prepared glasses are amorphous in nature. The FTIR result proved the presence of TeO_4 group and the stretching vibrations of BO_3 group. As concentration of lanthanum oxide increases, the density of the prepared glass samples increases and the molar volume decreases. The increasing trend of the density is due to the replacement of lighter atomic mass of B_2O_3 with higher atomic mass of La_2O_3. Meanwhile, the decrease in the molar volume is due to the decrease in the bond length on inter-atomic spacing between the atoms. The fundamental absorption edge tends to shifts to lower wavelength as the concentration of lanthanum oxide increases. Both direct and indirect band gap increases as the concentration of lanthanum oxide increases. This trend might be related to the decreasing number of nonbridging oxygen in the glass system. Meanwhile, Urbach energy is increased with increase of La_2O_3 content that indicates the increasing fragility nature of the glass network. Refractive index of the prepared glass samples decreases with the increasing concentration of lanthanum oxide which is due to decreasing number of high polarizability nonbridging oxygen.

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