Optical and thermal properties of $\text{TeO}_2\text{--B}_2\text{O}_3\text{--Gd}_2\text{O}_3$ glass systems

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New glass samples with composition $(1 - x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ with $x = 0.2, 0.4, 0.6, 0.8$ and 1.0 in mol% have been synthesized by conventional melt-quenching techniques. X-ray diffraction (XRD) studies were performed in order to confirm the amorphous nature of the samples. The density of the samples has been found to vary with the Gd_2O_3 content, whereas an opposite trend has been observed in the molar volume. The analysis of Fourier Transform Infrared (FT-IR) spectroscopy of the samples showed that the glass network is mainly built of TeO_3 , TeO_4 , BO_3 and BO_4 units. The addition of Gd_2O_3 changed the refractive index, optical band gap and Urbach energy of the glass samples. The thermal properties of the studied glasses were investigated by measuring the thermal diffusivity of the samples by using photoflash method at room temperature.

Keywords: gadolinium oxide; borotellurite glass; optical band gap; Urbach energy; thermal diffusivity

1. Introduction

There is a considerable interest in the study of rare earth doped glasses for photonic, optoelectronic and sensor applications. Borotellurite glasses are known for having low phonon energy, high thermal stability, high chemical stability and ease of fabrication [1]. These features make borotellurite glasses promising candidates for photonic applications. In order to improve those qualities, rare earth oxides were used as glass modifiers to increase the rigidity and devitrification of the glass [2–4]. Gadolinium oxide has been used to explore the optical and thermal behavior of the borotellurite glasses due to the fact that Gd^{3+} ions possess high luminescence efficiency, play attractive role as active ions for optical materials and are useful for the development of photonic devices, such as broadband amplifiers, fiber laser and light-emitting diodes [1, 5]. The addition of gadolinium oxide to borotellurite glasses influences the homogeneity and stability of the glass system [6, 7].

Besides, these types of glass have unique properties such as mechanical resistance, enhanced magnetic properties, high chemical durability and photoinduced nonlinear optical properties resulting from substantial contribution of anharmonic electron-phonon interactions [8–11]. The magnetic properties result from the presence of gadolinium ions and antiferromagnetic interactions between gadolinium ions distributed in the glass system as reported in [6, 10]. Thermal stability of borotellurite glass system containing ZnO has reached 166°C [12]. Also, unique physical properties of tellurite glasses have been reported [13–16].

Gadolinium-doped glasses have been studied and reported by several researchers. It is commonly stated that all transitions in Gd^{3+} containing glasses occur mostly in UV region. Optical energy gap E_{opt} of lead germanate glasses increases with the addition of Gd_2O_3 which causes a change in the bonding of the glasses [17, 18]. Moreover, the spectroscopic studies show that ZBLAN: Gd^{3+} glasses have strongest absorption due to $^6\text{I}_7$ levels [19]. Gd^{3+} ions have strong emission around 310 nm to 312 nm which is very useful for producing narrow

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band UVB (ultraviolet broadband) light and this narrow band UVB has medical applications in the treatment of skin diseases. However, the optical [2], thermal [3] and elastic [4] properties of quaternary $\text{TeO}_2\text{--ZnO--Nb}_2\text{O}_5\text{--Gd}_2\text{O}_3$ glasses with different glass compositions have not been studied extensively.

The aim of the present work is to study the effect of gadolinium oxide on FT-IR spectra, refractive index, optical band gap, Urbach energy, and the thermal diffusivity properties of $(1 - x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ with $x = 0.2, 0.4, 0.6, 0.8$ and 1.0 in mol% glasses.

2. Experimental

2.1. Sample preparation

The synthesis of gadolinium borotellurite glasses with compositions $(1 - x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ ($x = 0.2$ mol%, 0.4 mol%, 0.6 mol%, 0.8 mol% and 1.0 mol%) was carried out by conventional melt-quenching technique. The chemical composition of the sample is recorded in Table 1. The starting materials, tellurium oxide, TeO_2 (Alfa Aesar, 99.99 %), boron oxide, B_2O_3 (Alfa Aesar, 97.5 %) and gadolinium oxide, Gd_2O_3 (Alfa Aesar, 99.999 %), were weighted using an electronic beam balance with an accuracy of ± 0.0001 g. The chemical mixtures were mixed thoroughly to ensure homogeneity and placed in a furnace at temperature of 400°C to remove any water vapor. Then the samples were melted at 900°C in the second furnace. The melt was poured into a stainless steel mould that was preheated at 350°C and annealed for an hour to remove thermal strain.

2.2. Characterization

X-ray diffraction studies of the samples were performed using Phillips X-ray diffractometer X'Pert Pro PW 304. The density of prepared samples was measured using electronic densimeter MD-300S which is based on Archimedes principle. The measurement was carried out at room temperature using distilled water as the buoyant liquid. The Fourier transform infrared (FT-IR) transmittance

spectra of the samples were recorded at room temperature using a Mattson 5000 FT-IR spectrometer, in the wavenumber range of 2000 cm^{-1} to 200 cm^{-1} . The optical absorption measurements were carried using Shimadzu-1650PC UV-Vis spectrophotometer in the wavelength range of 200 nm to 1000 nm . The refractive index was measured using EL X-02 high precision ellipsometer at room temperature and wavelength of 632.80 nm . The thermal diffusivity of the glass samples was measured by photoflash technique by using a K-type thermocouple to measure the temperature profile at the rear sample surface.

3. Results and discussion

3.1. X-ray diffraction

Fig. 1. X-ray diffraction pattern of $(1 - x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

The X-ray diffraction (XRD) patterns of Gd^{3+} doped borotellurite glasses are shown in Fig. 1. The XRD patterns of the samples recorded in the range of 20° to 80° do not reveal any distinguishable peaks but exhibit broad diffusion hump at a low scattering angle. This confirms the amorphous structure of the studied samples. This result is in agreement with the previous work reported in [20, 21].

3.2. Fourier transform infrared spectroscopy (FT-IR)

The FT-IR spectra of the gadolinium borotellurite glasses recorded in the region of 200 cm^{-1} to 2000 cm^{-1} contain several absorption bands characteristic of its local structure. The FT-IR spectra

Table 1. Chemical composition of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}]-x(\text{Gd}_2\text{O}_3)$ glass sample.

x [mol%]	TeO_2	B_2O_3	Gd_2O_3	Total [%]
0.2	83.7904	15.6647	0.5448	100
0.4	83.3346	15.5795	1.0859	100
0.6	82.8818	15.4949	1.6233	100
0.8	82.4322	15.4108	2.1570	100
1.0	81.9856	15.3273	2.6871	100

Table 2. Assignment of IR transmission bands for $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}]-x(\text{Gd}_2\text{O}_3)$ glasses.

No.	0.2	0.4	0.6	0.8	1.0	Assignments
1	1369	1350	1370	1357	1370	Trigonal B–O bond stretching vibrations in isolated trigonal BO_3 units [22]
2	1222	1219	1225	1218	1224	Trigonal B–O bond stretching vibrations of BO_3 units from boroxyl groups [22]
3	630	625	645	643	622	TeO_4 groups presences in all tellurite containing glass [22]

Fig. 2. FT-IR transmittance spectra of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}]-x(\text{Gd}_2\text{O}_3)$ glasses.

are shown in Fig. 2. The peak positions and their assignments are presented in Table 2. Tellurium network includes two vibration modes [20, 22, 23]:

1. In 600 cm^{-1} to 640 cm^{-1} the vibration is due to the Te–O in trigonal by-pyramids, TeO_4 ,
2. In 680 cm^{-1} to 700 cm^{-1} the vibration corresponds to the Te–O in trigonal pyramid, TeO_3 .

The absorption spectra of borate glass can be divided into three regions: 600 cm^{-1} to 800 cm^{-1} ,

800 cm^{-1} to 1200 cm^{-1} and 1200 cm^{-1} to 1800 cm^{-1} :

- In 600 cm^{-1} to 800 cm^{-1} , bending vibration of various borate arrangements B–O–B occur [22, 23],
- In 800 cm^{-1} to 1200 cm^{-1} , it is assigned to B–O stretching of tetrahedral BO_4 units [20, 22, 23],
- In 1200 cm^{-1} to 1800 cm^{-1} , it is due to B–O stretching of trigonal BO_3 units [20, 22, 23].

The FT-IR spectra of the glasses exhibit several peaks at 621 cm^{-1} to 646 cm^{-1} , 1217 cm^{-1} to 1225 cm^{-1} and 1349 cm^{-1} to 1371 cm^{-1} . The absorption band around 621 cm^{-1} to 646 cm^{-1} is attributed to Te–O–Te bridges between tellurium atoms (TeO_4). The peaks at 1217 cm^{-1} to 1225 cm^{-1} are assigned to the trigonal B–O bond stretching vibrations of BO_3 units. The band at 1349 cm^{-1} to 1371 cm^{-1} indicates the B–O stretching vibration of BO_3 units.

3.3. Density and molar volume

The measured values of density ρ , molar volume V_m , experimental and theoretical refractive

Table 3. Density, molar volume and refractive index of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

mol%, $x(\text{Gd}_2\text{O}_3)$	Density [kg/m^3]	Molar volume [m^3/mol]	Experimental refractive index, $n(632.80 \text{ nm}) \pm 0.01$
0.2	3914	3.40×10^{-5}	2.029
0.4	4045	3.30×10^{-5}	1.940
0.6	3959	3.38×10^{-5}	1.951
0.8	4014	3.35×10^{-5}	1.964
1.0	4101	3.29×10^{-5}	2.058

Fig. 3. Density and molar volume of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

index are recorded in Table 3. The density of gadolinium borotellurite glass samples increases from 3914 kg/m^3 to 4101 kg/m^3 due to addition of 0.2 to 1.0 Gd_2O_3 mol%. The increase in density is due to the substitution of borotellurite, $\text{TeO}_2\text{--B}_2\text{O}_3$ by gadolinium atom, Gd_2O_3 (362.5 g/mol) which has a higher molecular weight than $\text{TeO}_2\text{--B}_2\text{O}_3$ (229.2 g/mol), resulting in a higher net weight of the glass structure [24, 25] as shown in Fig. 3. Increasing of the number of oxygen atoms and cation radius due to higher atomic weight of gadolinium atom, Gd_2O_3 contributes to higher packing density [26]. However, there is a decrease in density from 0.4 to 0.6 Gd_2O_3 mol%. The decrease in density at 0.6 mol% of gadolinium oxide may be attributed to the formation of Gd--O--Te or Gd--O--B linkages which might weaken the cross-linking within the glass network [6]. Molar volume of the present glass samples decreases from 3.40×10^{-5} to $3.29 \times 10^{-5} (\text{m}^3/\text{mol})$ as shown in Fig. 3. The change in V_m is due to bond length or the interatomic spacing between atoms and the rearrangement of the lattice. Molar volume

is inversely proportional to density which is a normal behavior between density and molar volume. Hence, the compactness of the glass will increase and more bridging oxygen ions will be created which will result in an increase in the rigidity of the glass [27]. The increase in the molar volume might also be due to the substitution of B_2O_3 by Gd_2O_3 in the glass matrix.

3.4. Refractive index

Fig. 4. Refractive index of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

The values of the experimental refractive index, $n(632.80 \text{ nm})$ have been collected in Table 3 and shown in Fig. 4 as a function of Gd_2O_3 mol%. The values of the experimental refractive index at first decreased from 2.029 to 1.940 and then increased to 2.058 mol% due to 0.2 to 1.0 Gd_2O_3 mol% addition. The decrease in refractive index might be due to the structural change from TeO_3 to TeO_4 and formation of bridging oxygen atoms after addition of Gd^{3+} ions into the glass. This is because bridging oxygen atoms have lower polarizability compared to non-bridging oxygen atoms.

However, the refractive index increased from 1.940 to 2.058 as the concentration of Gd_2O_3 increased from 0.4 mol% to 1.0 mol%. The changes in refractive index can be related to the effect of large ions as the concentration of Gd^{3+} ions increases. The addition of rare earth ions such as Gd^{3+} ions to the network leads to the formation of dense packing of rare earth modifiers in the host material, which increases the refractive index. This is because there is a directly proportional relationship between dense packing and the refractive index. Furthermore, the increase in the refractive index of gadolinium borotellurite glasses is also due to the size of the cation. As the concentration of Gd^{3+} ions increases, the effect of large ions Gd^{3+} ions becomes more pronounced. Hence, the substitution of Gd^{3+} ions (1.79 Å) which have higher atomic radius than the atomic radius of tellurite atom (1.6 Å) and boron atom (0.98 Å) into the network enhances the increase in refractive index with increasing the rare earth ion concentration. It is due to high polarization value of these ions resulting from increasing of cation size [28, 29]. The refractive index obtained in this work is higher than the refractive index reported for calcium borotellurite glass system, which is desirable for fiber optic design and waveguide applications [30].

3.5. UV-Vis spectroscopy

UV-Vis absorption spectra of the prepared glasses are shown in Fig. 5. The absorption coefficient, $\alpha(\omega)$ near the absorption edge can be calculated by the following formula:

$$\alpha(\omega) = 2.303 \frac{A}{d} \quad (1)$$

where A denotes the absorbance and d corresponds to the thickness of the glass samples [22]. The optical absorption edges do not exhibit any sharp absorption edges which is in agreement with X-ray diffraction spectra shown in Fig. 1 and confirms that the present glass samples are an amorphous material. The relationship between optical band gap energy, E_{opt} and the absorption coefficient, $\alpha(\omega)$ is given by the following formula:

$$\alpha(\omega) = \frac{A(\hbar\omega - E_{\text{opt}})^n}{\hbar\omega} \quad (2)$$

Fig. 5. Optical absorbance spectra for $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

Fig. 6. Direct energy band gap of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

Fig. 7. Indirect energy band gap of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

where A is a constant, E_{opt} is the energy of optical band gap, $\hbar\omega$ is the photon energy and $\alpha(\omega)$ is the absorption coefficient as reported in [21]. The values of n are $\frac{1}{2}$ and 2 for the allowed direct and allowed indirect transitions, respectively. Both of these band gaps obtained from the above dependences are interband transitions but the latter involves phonon interactions [31]. In order to determine whether the optical data of the glass samples

Fig. 8. Variation of direct and indirect optical band gap with glass composition.

fit better to direct or indirect band gap formula, the relationships between $(\alpha\hbar\omega)^2$ versus photon energy ($\hbar\omega$) and $(\alpha\hbar\omega)^{1/2}$ versus photon energy ($\hbar\omega$) have been plotted as shown in Fig. 6 and Fig. 7, respectively. The value of optical band gap E_{opt} for either direct or indirect band gap can be determined by extrapolating the linear parts of the curves [31]. The values of obtained optical band gap energy are tabulated in Table 4. The direct and indirect energy band gap decreased from 3.253 eV to 3.199 eV and from 2.535 eV to 2.525 eV due to the increase of Gd₂O₃ from 0.2 to 1.0 mol%. The disorder in the glass structure causes larger extension of the localized states within the gap according to Mott and Davies relation [32]. The increase in the direct and indirect energy band gap for the glass system can be related to the variation of density as well as the creation of bridging oxygen [32]. The increase in the direct and indirect energy band gap might due to electronic defects in the glass network which allow the glass structure to relax and fill the relatively large interstices that exist in the interconnected network of boron, tellurium and oxygen atoms causing expansion followed by volume compaction [33]. The fundamental absorption edge usually follows the Urbach rule as follows:

$$\alpha(\omega) = \alpha_0 \exp\left(\frac{\hbar\omega}{\Delta E}\right) \quad (3)$$

where α_0 is a constant, ΔE is a measure of the band tailing and is known as Urbach energy as reported in [21]. The values of the data obtained are tabulated in Table 4 and shown in Fig. 8. The Urbach energy values of the present glass systems decrease

from 0.397 eV to 0.354 eV. The high values of Urbach energy ΔE suggest that there are more defects in the glass system. The values of Urbach energy ΔE decrease with the increase of Gd₂O₃ content which causes reduced fragility of the vitreous network. The slight increase might be due to the increase of TeO₄ pyramids as the content of Gd₂O₃ increases and the presence of TeO₄ results in less stable structure and lower connectivity of the glass network.

3.6. Thermal properties

Fig. 9. Thermal diffusivity of $(1 - x)[(\text{TeO}_2)_{70} (\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

Thermal diffusivity is used to measure the speed of heat propagation through a material [34]. Thermal diffusivity, β has been calculated according to the formula 4:

$$\beta = \frac{0.1388L^2}{t_{1/2}} \quad (4)$$

where L is the thickness of the sample and $t_{1/2}$ is the time required for the specimen to reach half of the maximum temperature rise as reported in [34]. Values of thermal diffusivity are recorded in Table 5 and shown in Fig. 9. The thermal diffusivity of gadolinium borotellurite glasses varies from $2.32172 \times 10^{-7} \text{ m}^2/\text{s}$ to $2.46902 \times 10^{-7} \frac{\text{m}^2}{\text{s}}$ as the concentration of Gd₂O₃ increases from 0.2 mol% to 1.0 mol% as shown in Fig. 9. Thermal diffusivity of gadolinium borotellurite glass achieves the maximum at $x = 1.0$ mol% with the value of $2.46902 \times 10^{-7} \frac{\text{m}^2}{\text{s}}$, which is lower than that of window glass 3.4. The values of thermal diffusivity obtained in the present work are lower

Table 4. Direct energy band gap, E_{opt}^1 indirect energy band gap, E_{opt}^2 and Urbach energy ΔE of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

mol%, $x(\text{Gd}_2\text{O}_3)$	Direct energy band gap, E_{opt}^1 [eV]	Indirect energy band gap, E_{opt}^2 [eV]	Urbach energy, ΔE [eV]
0.2	3.253	2.535	0.397
0.4	3.252	2.555	0.359
0.6	3.168	2.511	0.353
0.8	3.167	2.526	0.351
1.0	3.199	2.525	0.354

Table 5. Thermal diffusivity of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

mol%, $x(\text{Gd}_2\text{O}_3)$	Thickness L [m]	Time t [s]	Thermal diffusivity $\beta [\times 10^{-7} \text{ m}^2/\text{s}]$	Error $[\times 10^{-9} \text{ m}^2/\text{s}]$
0.2	0.00249	4.062	2.32172	4.64344
0.4	0.00249	4.464	2.09430	4.18860
0.6	0.00247	4.308	2.15262	4.30525
0.8	0.00250	4.453	2.11854	4.23708
1.0	0.00250	3.872	2.46902	4.93803

as compared with the results obtained for other tellurite glasses, $2.7 \times 10^{-7} \text{ m}^2/\text{s}$ to $3.0 \times 10^{-7} \text{ m}^2/\text{s}$ and from $3.1 \times 10^{-7} \text{ m}^2/\text{s}$ to $3.4 \times 10^{-7} \text{ m}^2/\text{s}$ as reported in the literature [35, 36]. Previously in [37] it has been reported that the maximum in thermal diffusivity corresponds to the minimum molar volume. Sensor with high thermal diffusivity will give faster initialization time [38]. In the last report, the optical and dielectric constant remained nearly the same on adding B_2O_3 into tellurite network but the stability against devitrification increased considerably [39]. The changes in the thermal diffusivity of the present glass can be attributed to the size of the rare earth ion, Gd^{3+} . Thermal conductivity increases with the increase of mean free path of phonon. When $\text{TeO}_2\text{--B}_2\text{O}_3$ is substituted by the bigger atom Gd_2O_3 , there is an increase in the size of the atom in the glass network which causes the mean free path to increase and hence, an increase in thermal diffusivity [34]. Therefore, at mole fraction of 0.01, the content of the gadolinium atoms is the highest and it gives the highest value of the thermal diffusivity.

Fig. 10. Thermal diffusivity and density of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses. Fig. 11. Thermal diffusivity and molar volume of $(1-x)[(\text{TeO}_2)_{70}(\text{B}_2\text{O}_3)_{30}] - x(\text{Gd}_2\text{O}_3)$ glasses.

In Fig. 10, it is noted that when density increases, the thermal diffusivity decreases from 0.2 – 0.8 mol concentration. Meanwhile, the thermal diffusivity achieves high value at the highest number of density and concentration. This behavior may be explained by the substitution of gadolinium atom, Gd_2O_3 in the glass network. As explained in the previous part, gadolinium atom has higher atomic weight (362.5 g/mol) than the atomic weight of TeO_2 – B_2O_3 (229.2 g/mol). Therefore, the change of thermal diffusivity is due to the change in density. Fig. 11 shows that at the point with the highest thermal diffusivity value, the density has the smallest molar volume. The minimum molar volume of the glass system may be due to the closely packing of the structure network. Decreasing in molar volume may cause a decrease in interatomic distance and increase in compactness of the glass network. Therefore, the distance for the vibration of molecules in the lattice or phonons is shortened and the heat energy can be transported faster.

4. Conclusions

New gadolinium borotellurite glasses $(1 - x)[(TeO_2)_{70}(B_2O_3)_{30}] - x(Gd_2O_3)$ with $x = 0.2$ mol%, 0.4 mol%, 0.6 mol%, 0.8 mol% and 1.0 mol% have been fabricated. The change in density, molar volume and FT-IR spectra of the obtained gadolinium borotellurite glasses arise from the changes in bonding configuration, mainly Gd–O–Te or Gd–O–B linkages and stretching vibrations of TeO_4 and BO_3 units in the glass structure. The refractive index of the glass samples at 632.80 nm is due to the presence of bridging oxygen (BO) and the effect of large concentration of rare earth ions. Urbach energy in the glasses varied from 0.351 eV to 0.397 eV which suggests that more defects are present in the glass network after the addition of Gd_2O_3 . Thermal diffusivity of the glass samples was investigated and it was found that thermal diffusivity of gadolinium borotellurite glass achieved the highest value of $2.46902 \times 10^{-7} \text{ m}^2/\text{s}$ at 1.0 mol% Gd_2O_3 concentration which was related to the substitution by bigger atoms, larger density and molar

volume. The presently studied glasses may be good candidates for optoelectronic applications.

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