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Optical properties of graphene oxide-coated tellurite glass for potential fiber optics

Y Azlina[a,b], M.N. Azlan[a,b,*], A.B. Suriani[a,b], M.K. Halimah[c], S.A. Umar[d]

[a] Physics Department, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjung Malim, Perak, Malaysia
[b] Nanotechnology Research Centre, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjung Malim, Perak, Malaysia
[c] Physics Department, Universiti Putra Malaysia, 35900, Serdang, Selangor
[d] Department of Physics, Faculty of Science, Federal University Lafia, Nigeria

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ABSTRACT

In this work, we synthesize graphene oxide (GO)-coated tellurite glass for the improved optical performance of fiber optics. A series of tellurite glass system with composition of $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ where *y* (mol%) = 0.005, 0.01, 0.02, 0.03, 0.04and 0.05 mol% was successfully fabricated and coated with GO by using melt-quenching and spray coating methods. The structural properties were characterized by using XRD analysis and FESEM. The XRD patterns indicated that the tellurite glass were amorphous. The FTIR transmissions images revealed the surface morphology of GO on the glass surface. The optical band gap energy was found enhanced from 3.392 to 3.495 eV. The refractive index was found improved from 2.275 to 2.299 with the existence of GO. Hence, these results were useful for the application of fiber optics.

1. Introduction

Graphene is a 2D nanomaterial or the monolayer formed by carbon atoms possessing honeycomb structure and has attracted great interest among the researchers which exhibits outstanding photonic and optoelectronic properties [1–3]. In addition, graphene can be used as a promising material to enhance the optical performance of glass materials that can be applied in fiber optics technology. Graphene oxide is commonly produced by using electrochemical exfoliation method, which possesses the arrangement of oxygen-containing functional groups, including carboxyl, epoxy, hydroxyl, and carbonyl [4]. The wide-bandgap in graphene oxide has been stimulated from an oxidation process where the carbon atoms are transformed from sp^2 to sp^3 in the GO structure [5,6]. Therefore, the oxidation degree and arrangement of oxygen functionality groups may affect the bandgap energy variation in glass materials [4]. Hence, it would be a fascinating approach to combine the graphene oxide and tellurite glass as it may enhance the optical performance of the glass system.

The tellurite-based glass has high demand as a host of rare-earth trivalent ions compared to those phosphate, borate, germanate and silicate glasses due to its unique characteristics that include high rates of linear and nonlinear refraction [7], low melting point, high dielectric constant, low phonon energy, high transparency, high refractive index and excellent infrared transmission [8–11]. Thus, it should be noted tellurite glass has strong mechanical strength and good glass-forming network when combining with GO, which is ideal for fiber optics applications. The incorporation of rare-earth materials such as erbium oxide into the tellurite glasses has been focused on exploring the optical properties due to its emission at 1.53 µm which can be contributed in broadband amplifier and laser applications [12]. In addition, erbium-doped glasses has been successfully used in optical fibers as a dopant in order to amplify the signals in optical communication systems [13].

The aims of this study are to explore the optical properties of tellurite glasses coated with graphene oxide for potential application in fiber optics. The refractive index, optical band gap, and electronic polarizability of the glass system were performed as important parameters to determine the optical performance of graphene oxide-coated tellurite glass system. As far as we know, the investigation on the optical properties of tellurite glasses coated with graphene oxide has not been reported in previous research. Hence, the outcome of this research is the first step to develop novel materials for fiber optics.

* Corresponding author at. Nanotechnology Research Centre, Faculty of Science and Mathematics, Universiti Pendidikan Sultan Idris, 35900 Tanjung Malim, Perak, Malaysia

E-mail addresses: bazlanmn@fsmt.upsi.edu.my (M.N. Azlan), suriani@fsmt.upsi.edu.my (A.B. Suriani), halimahmk@upm.edu.my (M.K. Halimah).

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2. Materials and methods

2.1. Glass preparation technique

A series of tellurite glass with composition $\{[(TeO_2)_{0.7}\ (B_2O_3)_{0.3}]_{0.7}\ (ZnO)_{0.3}\}_{1\text{-}y}\ (Er_2O_3)_y$ and varying concentrations of Er^{3+} ions from y = 0.005, 0.01, 0.02, 0.03, 0.04, and 0.05 mol% were fabricated via melt-quenching technique. The high purity raw materials used to synthesize the glass samples were TeO_2 (Alfa Aesar, 99.99%), B_2O_3 (Alfa Aesar, 98.5%), ZnO (Alfa Aesar, 99.99%) and Er_2O_3 (Alfa Aesar, 99.99%). All the chemicals at an appropriate amount were completely weighed, homogeneously mixed, and placed into a platinum crucible. The mixture was then transferred to an electric furnace and preheated at 400 °C for 1 h.

The mixture was then transferred to a high-temperature electrical furnace at 900 °C in a period of 2 h to undergo the melting process. The melt was then poured into a cylindrical mold that had been preheated at 400 °C and immediately annealed to release the thermal stress and mechanical strength. The furnace was turned off and allowed the glass samples to cool down at room temperature. All of the glasses were cut into the same thickness about 2 mm by using Isomet Buehler low speed saw machine and finally being polished with the sandpapers to obtain the highly smooth and transparent surface of glasses.

2.2. Synthesis and coating of graphene oxide onto glass surface through spray coating technique

Graphene oxide was synthesized using the chemical approach from electrochemical exfoliation method. Initially, the two graphite rods were connected to the power supply as anode and cathode electrodes. This was followed by immersing the graphite rods partially in the electrolyte solution. Then, the electrolyte used was a mixture of dissolved surfactant (SDS) in de-ionized water, and a direct current (DC) voltage was applied between the two of graphite electrodes. The exfoliation of bulk graphite into single sheet occurred in the presence of surfactant intercalated between interlayer of graphite. Finally, the glass samples were deposited with GO on the glass surface by using the spray coating method and ready for further characterizations.

3. Results and discussion

3.1. Morphological structure of GO on the glass surface

The microscopic morphologies and surface structures of graphene oxide deposited on the tellurite glass surfaces were examined using field scanning electron microscopy (FESEM) as illustrated in **Fig. 1**. The results confirm the existence of GO due to its large agglomerate structure and thick layers as observed at the edges of GO sheets. Moreover, the high degree of agglomeration of GO, as shown in Fig. 1(a) indicates the inhomogeneous of GO distribution on the glass surface. Meanwhile, the thick layers, as shown in Fig. 1(b) are due to the high amount of oxygen-containing functional groups that resulted from the oxidation process through electrochemical exfoliation method. The graphitic structure is found in high density with a large crumple structure which corresponds to the outcome of the oxidation process.

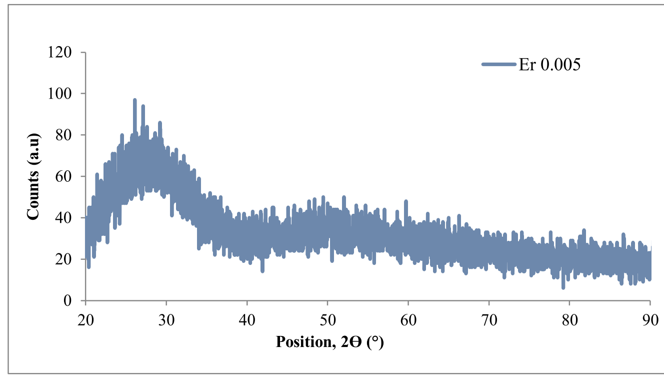

Fig. 2. XRD diffraction pattern of Er^{3+} doped tellurite glass.

3.2. X-ray diffraction (XRD)

The measured XRD pattern of Er^{3+} doped tellurite glass coated with GO is displayed in Fig. 2, respectively. It was found that the diffractogram of the synthesized glasses is similar in shape with a broad hump between 20° and 40° but the absence of any sharp discrete diffraction peaks, indicating the presence of long-range structural disorder and the amorphous nature [14].

3.3. Optical absorption, optical band gap energy, Urbach energy

Measurement of optical absorption, particularly the absorption edge is important to investigate the optically induced transitions, the band structure, and energy band gap of non-crystalline materials [15]. The optical absorption spectra of erbium ions doped tellurite glass were observed through UV–Visible Spectrometer (Agilent Cary 60). The UV–VISIBLE spectroscopy was carried out in the range of 200–800 nm at room temperature.

The optical absorption spectra of $\{[(TeO_2)_{0.7}\ (B_2O_3)_{0.3}]_{0.7}\ (ZnO)_{0.3}\}_{1\text{-}y}\ (Er_2O_3)_y$ coated with GO are shown in Fig. 3. As can be seen from the figure, it can be observed clearly that there are few absorption bands located in various wavelengths. These absorption bands indicate the characteristics of erbium ions, Er^{3+} arise from the ground state to the excited states. In addition, the 4f shell electrons are shielded by outer 5 s and 5 p bonding electrons, which lead to the sharp absorption

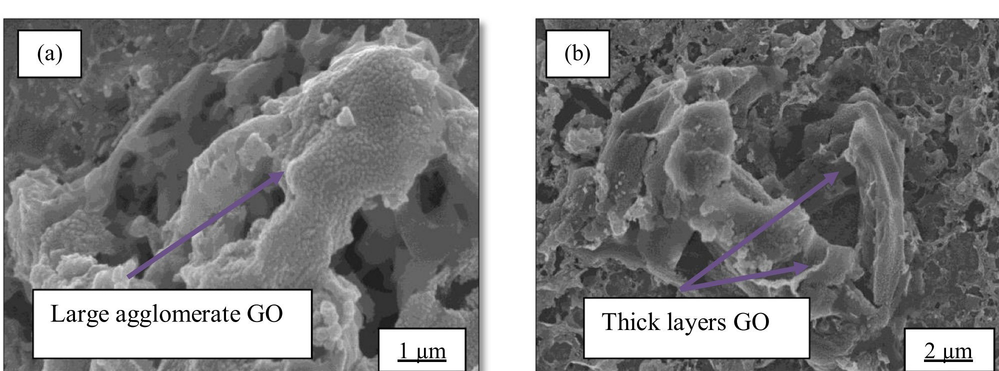

Fig. 1. FESEM micrographs of GO deposited onto tellurite glass surface at magnification 50 KX (b) 20 KX.

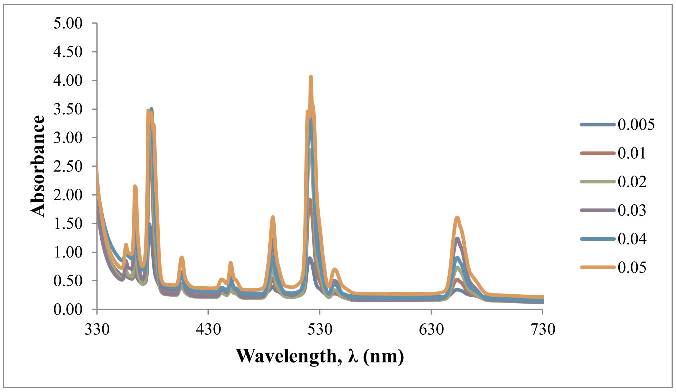

Fig. 3. Optical absorption spectra of $\{[(TeO_2)_{0.7}\ (B_2O_3)_{0.3}]_{0.7}\ (ZnO)\ _{0.3}\}_{1\text{-}y}\ (Er_2O_3)\ _y$ coated with GO.

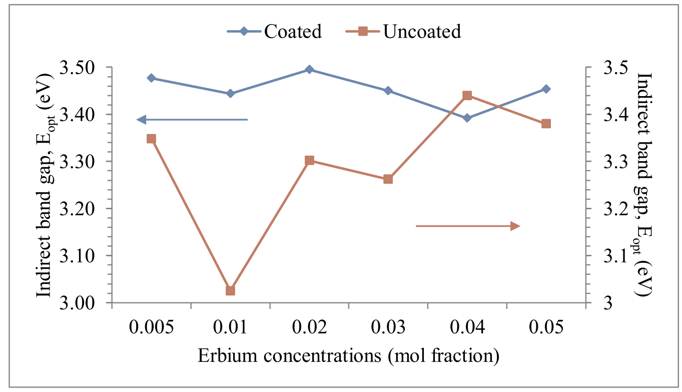

Fig. 5. Variation of optical band gap values for uncoated and coated glass with GO.

and emission bands [16]. The change in oxygen bond strength may affect the absorption characteristics. It can be seen from Fig 3 that there are changes in absorption edge which is due to the different number of oxygen bond strength in each of the glass samples.

By referring to Mott and Davis [17], the relationship between the absorption coefficient, $\alpha(\omega)$ and photon energy, $\hbar\omega$ has been proposed to determine the indirect transition occurred in the band gap energy by following equation:

$$\alpha(\omega) = \frac{B(\hbar\omega - Eopt)^n}{\hbar\omega} \tag{1}$$

where B is an energy-independent constant known as band tailing parameter, E_{opt} is the optical band gap energy, and n is a constant index that shows different values, 1/2, 2, 3/2, and 3 depending on the type of optical transitions. These values correspond to the direct allowed, indirect allowed, direct forbidden and indirect forbidden transitions, respectively [18].

The value of absorption coefficient, $\alpha(\omega)$ can be identified from the absorbance of glass sample at the varying wavelengths by using following relation:

$$\alpha(\omega) = 2.303\left(\frac{A}{d}\right) \tag{2}$$

where A refers to absorbance and d is the thickness of glass samples.

Fig. 4 indicates a plot of $(\alpha\hbar\omega)^{1/2}$ against $\hbar\omega$ for indirect allowed transition, which is known as Tauc's plot and is used to determine the optical band gap energy of a given transition. The optical band gap can be understood in terms of the energy difference between the valence band and the conduction band [19]. It was revealed that the values of optical band gap energy, E_{opt} have been estimated by extrapolating the curves and the values show a non-linear trend with the increase of erbium oxide, varying from 3.392 to 3.495 eV as shown in Fig. 5 and Table 1. This trend is related to the increase of non-bridging oxygen (NBOs), which leads to the change in absorption characteristics. The comparison of the optical band gap values of the present glass system with uncoated tellurite glass studied by Azlan et al. had been shown in Fig. 5 and Table 1 [7]. Fig. 5 shows that the values of optical band gap energy of coated glass are higher than the uncoated glass system [7]. This trend was due to the high presence of oxygen functional groups with localized Π electrons occurred in the oxidation process. Moreover, the optical band gap of GO is mainly affected by the Π and Π^* states [20, 21]. The presence of localized finite-sized molecular sp^2 clusters within on sp^3 matrix may lead to the confinement of Π-electrons in GO [22]. Consequently, the high degree of oxygens in GO enhance the optical band gap energy of glass materials as compared to the uncoated glass materials. On the other hand, the optical absorption is high in GO, which affects the optical band gap of GO-coated tellurite glass [4]. This trend is due to the number of active layers existed in GO surfaces, as reported by Lai et al. [21]. In addition, the absorption coefficient increases with the presence of GO on the tellurite glass surface [23] and hence, increase the optical bandgap energy.

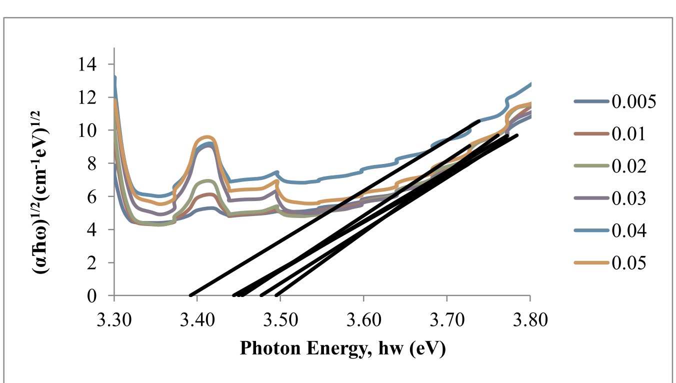

Fig. 4. Plot of $(\alpha\hbar\omega)^{1/2}$ against photon energy, $\hbar\omega$ of $\{[(TeO_2)_{0.7}\ (B_2O_3)_{0.3}]_{0.7}\ (ZnO)\ _{0.3}\}_{1\text{-}y}\ (Er_2O_3)\ _y$ coated with GO.

The Urbach energy, ΔE of the glass system can be identified by using the relation:

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

where β is a constant and ΔE is the band tailing parameter or known as the Urbach energy. This exponential behavior can be ascribed to band tails associated with the valence and conduction bands which extend into the band gap [12]. The magnitude of the Urbach energy corresponds to the width of localized states which can be used to characterize the degree of disorderliness in the amorphous or crystalline materials [9]. The materials with the larger value of Urbach energy have the tendency to convert weak bond into defects [15]. The values of ΔE were calculated by taking the reciprocals of slopes of the linear portion plotted on 1n α versus $\hbar\omega$ curves. The optical indirect band gap

Table. 1
Comparison of optical band gap (E_{opt}) between coated and uncoated for $\{[(TeO_2)_{0.7}\ (B_2O_3)_{0.3}]_{0.7}\ (ZnO)\ _{0.3}\}_{1\text{-}y}\ (Er_2O_3)_y$ glasses.

Optical band gap (E_{opt})		
Er concentration	**Uncoated**	**Coated**
0.005	3.348 [7] ± 0.1 nm	3.477 ± 0.1 nm
0.01	3.025 [7] ± 0.1 nm	3.444 ± 0.1 nm
0.02	3.302 [7] ± 0.1 nm	3.495 ± 0.1 nm
0.03	3.262 [7] ± 0.1 nm	3.450 ± 0.1 nm
0.04	3.440 [7] ± 0.1 nm	3.392 ± 0.1 nm
0.05	3.380 [7] ± 0.1 nm	3.454 ± 0.1 nm

Table. 2
Urbach energy (ΔE) for uncoated and coated glass with GO for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glass system.

Er molar fraction, x	Uncoated	Coated
0.005	0.180 ± 0.1 nm [24]	0.286 ± 0.1 nm
0.01	0.200 ± 0.1 nm [24]	0.339 ± 0.1 nm
0.02	0.175 ± 0.1 nm [24]	0.302 ± 0.1 nm
0.03	0.180 ± 0.1 nm [24]	0.255 ± 0.1 nm
0.04	0.161 ± 0.1 nm [24]	0.314 ± 0.1 nm
0.05	0.153 ± 0.1 nm [24]	0.123 ± 0.1 nm

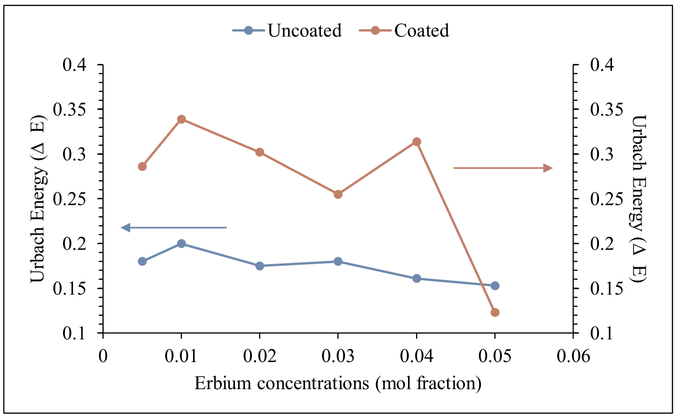

Fig. 6. Variation of Urbach energy for uncoated and coated glass with GO for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glass system.

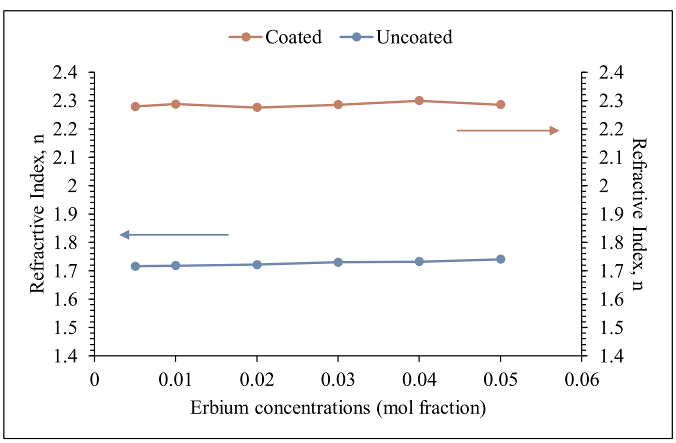

Fig. 7. Refractive index for uncoated and coated glass with GO for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glass system.

(E_{opt}) and Urbach energy for tellurite glass system was shown in Table 2. As can be referred in Fig. 6, the observed values of ΔE lie between 0.123 and 0.339 eV. The high amount of Urbach energy indicates the high degree of defects in the glass network [9]. The comparison values of Urbach energy with the uncoated tellurite glass studied by Azlan et al., are shown in Fig. 6 and Table 2 [24]. It can be seen that the Urbach energy of coated glass is higher than the uncoated glass. This trend is due to the high degree of defects in GO resulted from the high agglomerations, high oxygen numbers and large crumpled structure as shown in Fig. 1(a) and Fig. 1(b).

3.4. Refractive index and electronic polarizability

Refractive index, n is a fundamental optical parameter which is useful for photonic devices particularly in fiber optics and amplifiers. The refractive index is highly affected by the electron density and electronic polarizability of glass network [24]. The refractive index value of the glass samples can be determined by using optical band gap energy from the relation proposed by Dimitrov and Sakka [12]:

$$\frac{n^2-1}{n^2+1} = 1 - \sqrt{\frac{Eopt}{20}} \quad (4)$$

where n represents the refractive index and E_{opt} denotes as the indirect band gap values.

It can be observed from Fig. 7 that the refractive index increases with the increasing of Er_2O_3 concentration but decrease slightly at 0.02 mol% and 0.05 mol%. The comparative values of refractive index between coated and uncoated tellurite glass with GO are shown in Fig. 7 and listed in Table 3 [24]. The high value of the refractive index in GO-coated tellurite glass than the uncoated tellurite glass may be due to the high number of defects in GO and the presence of a high degree of oxygen atoms. The comparative value of Urbach energy in the previous result confirms that the GO-coated tellurite glass has a high degree of defects. The enhancement of refractive index in GO coated-tellurite glass than the uncoated tellurite glass confirms that the proposed glass materials have a high optical capability. The transmission of light ability is one of the important parameters for fiber optics application. Furthermore, the high number of refractive indices in tellurite glass is crucial to be used in fiber core in which the refractive index of fiber core must be higher than the index of the cladding. Hence, the coating of tellurite glass with graphene oxide is suitable to be used as a fiber core. The nonlinear trend of refractive index for GO-coated tellurite glass is attributed to the atomic number and also oxidation number of GO [25–26].

Table 3 Refractive index and electronic polarizability values for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glasses coated with GO and uncoated values from [24]

Electronic polarizability, α_e is the magnitude of electrons responds to an electric field which can be calculated by following well-known Clausius–Mosotti relation [27]:

$$\alpha_e = \left(\frac{3}{4\pi N_A}\right)\left(\frac{n^2-1}{n^2+2}\right) \quad (5)$$

where N_A is the Avogadro number, n is the refractive index and V_m refers to the molar volume of glasses. The electronic polarizability is an important parameter to determine the properties such as refraction, ferroelectricity and optical nonlinearity [19].

From Fig. 8 and Table 3, it can be observed that the electronic polarizability of coated tellurite glass increase from 8.815 to 8.894 Å^3

Table. 3
Refractive index and electronic polarizability values for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glasses coated with GO and uncoated values from [24].

Er molar fraction, x	Refractive index, n (uncoated)	Refractive index, n (coated)	Electronic Polarizability, α_e (Å^3) (uncoated)	Electronic Polarizability, α_e (Å^3) (coated)
0.005	1.716 ± 10 µm [24]	2.279 ± 10 µm	5.071 [24]	8.723
0.01	1.718 ± 10 µm [24]	2.287 ± 10 µm	5.123 [24]	8.826
0.02	1.721 ± 10 µm [24]	2.275 ± 10 µm	5.156 [24]	8.815
0.03	1.730 ± 10 µm [24]	2.285 ± 10 µm	5.232 [24]	8.894
0.04	1.732 ± 10 µm [24]	2.299 ± 10 µm	5.216 [24]	8.892
0.05	1.740 ± 10 µm [24]	2.285 ± 10 µm	5.274 [24]	8.873

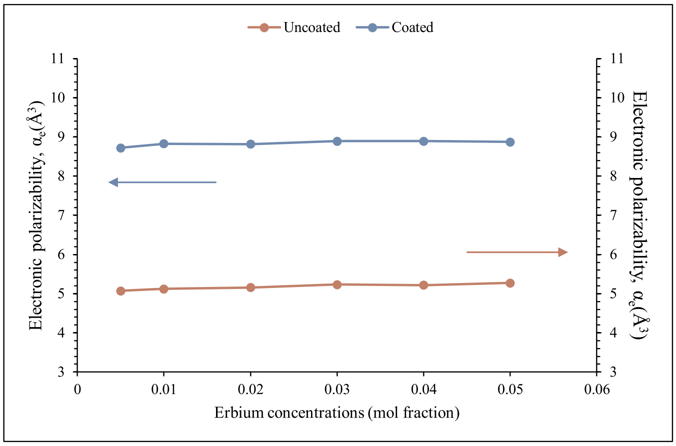

Fig. 8. Electronic polarizability for uncoated and coated glass with GO for $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.7} (ZnO)_{0.3}\}_{1-y} (Er_2O_3)_y$ glass system.

compared to uncoated glass within the value of 5.0706 to 5.2740 Å^3 [24] with the increase of Er^{3+} ions. This trend can be related to the graphene oxide coating possesses a high surface area and relatively low particle density [28]. On the other hand, the inclusion of graphene onto the tellurite glass system may exhibit the strong polarization-dependent of optical absorption under the total internal reflection (TIR) [29,30]. Additionally, the perfect absorption structures can greatly enhance the efficiency of graphene-based due to its high optical absorption and high field intensity in graphene [30]. However, at molar fraction 0.02 of Er_2O_3, the electronic polarizability value seems to decrease. This has been observed as the same trend for the refractive index of glasses and may be related to the formation of non-bridging oxygen (NBOs) from the incorporation of ZnO which acts the modifier into TeO_2 in the glass system network. As a result, the non-bridging oxygen bonds present the higher polarizability and cation refraction compared to bridging oxygen [24]. Another important factor is maybe due to the mobility of electrons, which causes higher polarizability in this work. Thus, we can conclude that the increase in electron mobility will lead to an increase in polarizability. This may be correlated to the atomic size increases and also the larger electrons must distort more easily.

4. Conclusion

The optical band gap energy of the GO coated glass showed a high value than the uncoated glass. It is noted that the graphene oxide coating enhances the optical properties of glass samples. The XRD analysis confirmed that the glass samples are in amorphous nature. The FESEM results proved that the existence of the GO structure onto the tellurite glass surfaces. The increasing value of optical band gap energy for tellurite glass, which has been deposited with GO is due to the influence of oxygen-containing functional groups on the surface behavior of GO. The refractive index and electronic polarizability after deposited also demonstrated the increase values compared to uncoated glass because due to the graphene-based effect, which consists of the high surface area and low particle density on GO. Therefore, this research is applicable to fiber optics technology applications.

Author statement

Azlina Y.: Fabrication of glass, coating technique, manuscript preparation. Azlan M.N.: Supervision. Suriani A.B: Graphene oxide preparation, morphology analysis. Halimah M.K: UV–Visible analysis, FTIR analysis. Umar S.A: Data analysis, validation

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

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