The Effect of rGO On Optical Properties of Neodymium Nanoparticles Doped Tellurite Glass System

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ABSTRACT

Nanomaterials based on graphene have outstanding properties, particularly in terms of optical performance. Using melt-quenching and low-cost spray coating procedures, we effectively generated a series of reduced graphene oxide-coated tellurite glasses doped with neodymium nanoparticles, denoted as rGO-ZBTNd (NPs) glasses. An X-ray diffraction study of glass samples validated the glass system's amorphous structure. FESEM morphological analysis was used to evaluate the appearance of rGO on the glass substrate and revealed the dispersion of rGO. UV-Vis spectroscopy was used to determine different absorption bands. The refractive index value calculated using the Dimitrov and Sakka equation was found to be more than n > 2.000 and in the range of 2.339 to 2.657 when compared to uncoated samples. On the basis of these findings, a new method involving rGO deposited on tellurite glass has been developed to improve the tellurite glass currently utilised in laser applications.

Keywords: *Tellurite Glass; Reduced Graphene Oxide; Optical Parameter; Refractive Index*

Introduction

Tellurium oxide is a potential glass forming that has been shown to be useful in current technological applications. Because of its unique qualities, tellurite

glass has been introduced. When compared to oxide material like phosphate and germanium, tellurite glasses have some of the most significant benefits, such as excellent malleability, low processing temperature, and superior mechanical strength [1]. Tellurite glass is useful in laser technology because of its many good qualities, including high transparency, large dielectric constant, large refractive index, a lower melting point, and remarkable nonlinear optical properties. Tellurite glass is also recognised for its strong solubility in rare earth oxide and low phonon energy. These qualities are critical in constructing optical fibre with high optical efficiency. Optical fibre is becoming increasingly significant in recent years, particularly in remote sensing, laser surgery, long-distance laser communications, and broadband amplifiers [2].

According to Novoselov 2004, graphene derived from carbon materials has significant features like strong electrical resistance, workability, and toughness [3]. Graphene is a single thick planar sheet of sp^2-bonded carbon atoms structured in a hexagonal arrangement in two dimensions. It possesses extraordinarily high carrier mobility, thermal expansion, and durability [4] Additionally, graphene exhibits extraordinary optical characteristics. However, graphene integration into lengthy lengths of fibre is limited due to the difficulties of handling graphene in the form of flakes, which results in transmission losses. A graphene derivative known as graphene oxide (GO) has been used in replacement of graphene flakes in the production of fibres for use in ultrafast laser and sensor applications, among other things [5]. Additionally, it has sparked interest in a variety of applications [6]-[7] including optoelectronics, super capacitors, or as power storage such as carbon-based rechargeable batteries, sensor (biosensor) solar cells, catalysts, photo catalysts, optics, photonics area and more applications. [8]–[10].

Based on the previous discovery, integrating graphene-based material and tellurite glass is still another method for improving the optical characteristic of the glass matrix [8]. Azlina et al [11] discovered that the existance of several rich-oxygen functional groups in GO layers increased optical properties, showing that GO has a significant impact on tellurite glass for optical fibre application. Furthermore, due to the high refractive index, the glasses offer promise in optical fibre laser technology [12] and producing the fibre core in optical fibre [8]. Reduced graphene oxide (rGO) has also attracted a lot of interest because of its optical features, which include saturable absorption and optical nonlinearity [13]-[14]. It has been intensively investigated for optical device and laser uses due to its high hydrophilicity, ease of functionalization, and water solubility qualities, as well as its flexibility and ease of processing in massive manufacturing [13]. Reduced graphene oxide (rGO) coating give the enhancement the sensing nanocomposite layer due to its high conductivity. However, reduced graphene oxide coated tellurite-based glass, on the other hand, has been reported infrequently. So, more investigation into the impact of reduced graphene oxide on the tellurite glass

system is thus required. As a result, the purpose of this research is to investigate the linear optical properties of rGO and give new information to the field of fibre laser applications.

Material and Method

Glass samples were prepared by melt-quenching and doped with neodymium NPs in a proportion of 0.01 mol% to 0.05 mol% in the chemical formula $(0.47(1-y)\ TeO_2 + (0.2(1-y))\ B_2O_3 + (0.29(1-y))\ ZnO + (y)\ Nd_2O_3$ (NPs). Table 1 shows the calculated weights of the compositions in 13 grams using the chemical powder that was used for the sample preparation from Aesar grade.

Table 1: Weight of each chemical composition in the fabricated glasses

Neodymium (NPs) molar fraction	Chemical component (g)				Total (g)
	TeO_2 (99.99% Alfa Aesar)	B_2O_3 (99.99% Alfa Aesar)	ZnO (98.5% Alfa Aesar)	Nd_2O_3 99.99% Nanostructured & Amorphous Materials, Inc	
0.01	8.427	1.576	2.631	0.366	13
0.02	8.192	1.531	2.557	0.719	13
0.03	7.965	1.489	2.486	1.059	13
0.04	7.745	1.448	2.418	1.388	13
0.05	7.534	1.408	2.352	1.706	13

An appropriate amount, with composition as stated in Table 1. A total of 13 g of chemicals mixed powder were measured using electronic balance equipment with a precision of ±0.0001 g. The alumina crucible mixtures were then preheated for 1 hour at 400 °C in an induction furnace to eliminate any moisture that may have been present in the chemical combination during the first weighing operation.The alumina crucible was then moved to the second furnace for a 2-hour melting operation at 900 °C. During the heating phase, the cylindrical stainless steel split mould was heated in the first furnace at 400 degrees Celsius to avoid the glass sample fracturing due to mechanical strain when the glass was cast in the glass system. After 2 hours, the molten material was immediately quenched in the mould before being annealed at 400 °C for an hour. The annealing technique was used to minimize heat stress in the glass sample and to stabilize the glass structure. After the glass sample had been annealed for an hour, the furnace was turned off, and it was left there overnight to cool to ambient temperature. The glass sample was polished with several sandpaper grades, including 1500 grid, 1200 grid, and 1000 grid. The glass

sample was crushed into a fine powder using a mortar and pestle for structural characterization. To examine the glass sample's structure, the fine powder was conducted to X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). Meanwhile, the high smooth bulk transparent glass samples were carried out to UV-Vis Spectrometer (PerkinElmer Model) in the wavelength range of 200-1000 nm.

Reduced graphene oxide preparation and coating method

Electrochemical exfoliation was used to synthesize graphene oxide (GO) [15]. Extra oxygen functional groups in GO were chemically reduced by using hydrazine hydrate (Merck, 80% soluble in water) at a ratio of 100:1 as a reducing agent to remove them (GO: hydrazine hydrate). This process was done for 24 hours at 95 °C to make an rGO solution depict at Figure 1 [16]. The rGO was then sprayed on the polished glass samples using the spraying technique.
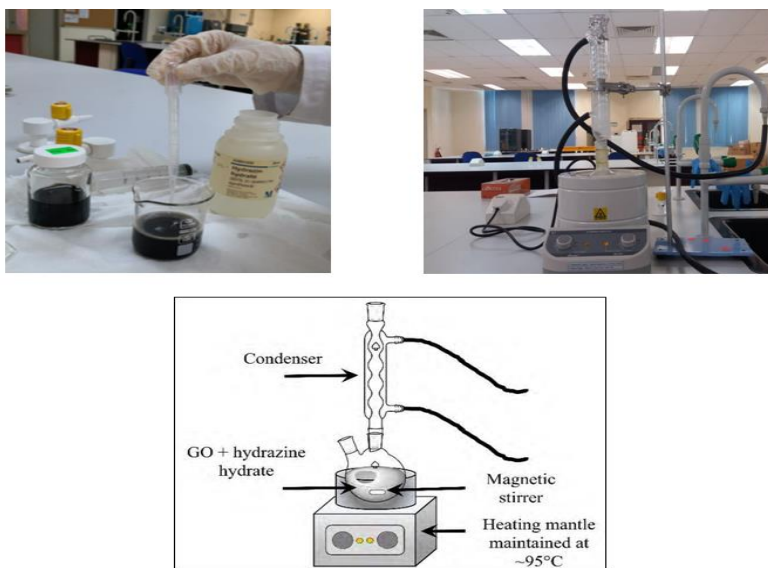

Figure 1: Reduction process of rGO used heating mantel instrument [17]

Result and Discussion

X-Ray diffraction (XRD)

The XRD analysis was utilised to confirm whether the materials inside the glass network were amorphous or crystalline following glass formation [12].

In this study, X-ray Diffraction using a PANanalytical EMPYREAN diffractometer was used to determine the structural characteristics of the glass samples. The diffraction patterns were determined at room temperature in the theta range of $10° < 2\theta < 80°$ using a wavelength of $\lambda = 1.5418$ Å Cu-Kα. Figure 2 illustrates the disordered nature of glass for rGO-coated tellurite glass doped with Nd^{3+} nanoparticles. The XRD pattern of the present glass exhibits a wide hump between $20°$ and $35°$ for 2 theta values, which is typical of amorphous materials rather than any sharp peaks [18], [19], [20].
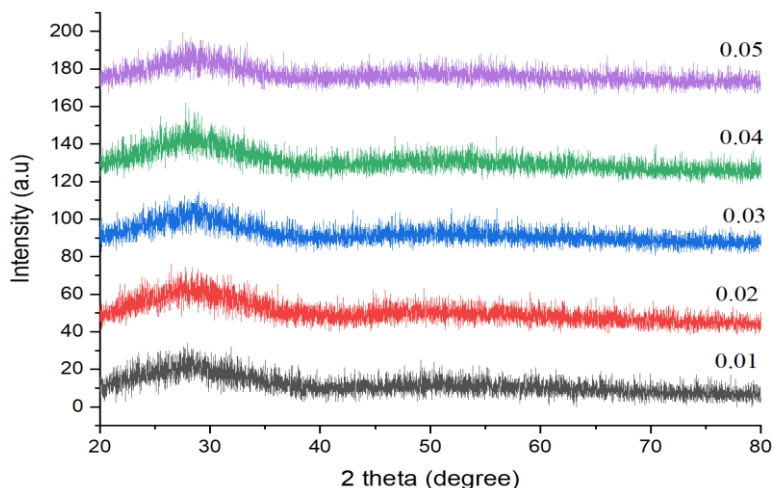

Figure 2: XRD pattern for rGO - ZBTNd (NPs) glasses

Fourier transform infrared (FTIR) analysis

The FTIR NEXUS Thermo 69000 Nicolet instrument was used to study the structural unit of the glass system in the frequency range 300 - 4000 cm^{-1} in order to produce FTIR spectra. Table 2 lists the peak positions and their assignments. Figure 3 displays the rGO-ZBTNd (NPs) FTIR spectra that were collected between 300 and 1700 cm^{-1}. The stretching vibration of the Te-O bond places the infrared properties of the two kinds of tellurium oxide structural units in the 600-700 cm^{-1} region. TeO4's trigonal bipyramidal structural unit band has a wavelength of 600–650 cm^{-1}[12], [21]-[23].

According to Azlan et al. [2], the trigonal pyramidal TeO_3 structural unit band is found around 650 and 700 cm^{-1}. The transmission spectra of the glass series have bands at 639-649 cm^{-1}, 1220-1357 cm^{-1}, 897-1017 cm^{-1}, and 1625 cm^{-1}. The band between 600 and 690 cm^{-1} shows that the glass series is composed of trigonal bipyramid, TeO_4, and trigonal pyramidal, TeO_3 groups, which correspond to bridging and non-bridging oxygen, respectively. Bridging

oxygen occupies "two equatorial and two apical oxygen sites in the bipyramidal structure, whereas lone pairs of electrons (LPEs) in tellurium's valence band occupy the third equatorial site" stated by [25].

Three spectral areas are primarily involved in the production of pure borate oxide B_2O_3 structural units. The first group of the band's tetrahedral structural units measure between 600 and 800 cm^{-1} (bending vibrations of various borate arrangements B-O-B). The second group is around between 800 and 1200 cm^{-1} for B-O tetrahedral BO_4 stretching, whereas the third group is located between 1200-1800 cm^{-1} for B-O stretching of trigonal BO_3 units [26]. Figure 3 displayed two wide absorption bands, one at 1623 cm^{-1}, which were corresponds to the B-O trigonal, BO_3 structural unit [12], [27]. The other at 1221-1359 cm^{-1}, which is assigned to the trigonal B-O bond stretching vibrations of BO_3 units from boroxyl groups that was identified in this experiment from the produced glass samples [27].

As a result, after glass formation, the BO_3 and BO_4 structural units dominate the tellurite glass matrix. Thus, borate oxide in the glass systems increases the quantity of non-bridging oxygen in the tellurite glass network. Meanwhile, the zinc oxide and Nd_2O_3 atoms are not exist because the zinc lattice was entirely broken down after glass production [28] and was undetected by the instrument due to the low concentration of rare-earth [29], similar with others rare-earth confirm by [2], [24], and [28] etc.
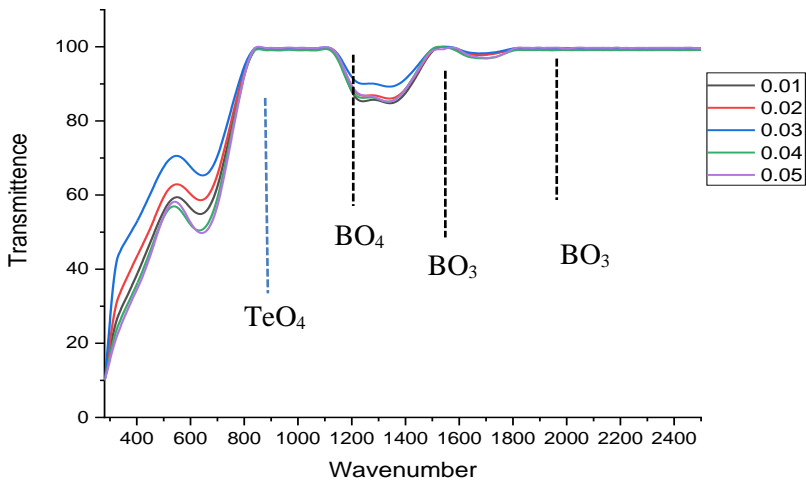

Figure 3: FTIR spectra of rGO - ZBTNd (NPs) glasses

Table 2: Assignment of infrared transmission band of rGO- ZBTNd (NPs) glasses

No	0.01	0.02	0.03	0.04	0.05	*Assignment
1	639	649	644	643	640	TeO_4 group [30]
2	899	1018	895	913	909	B-O stretching of BO_4 units [31]
3	1359	1350	1338	1370	1367	Trigonal B-O stretching vibration in isolated trigonal BO_3[24]
4	1239	1227	1221	1226	1229	Trigonal B-O bond stretching vibration of BO_3 unit from boroxyl groups [27];.
5	1623	1622	1623	1622	1623	Trigonal B-O bond stretching vibration of BO_3 unit from boroxyl groups [27]

FESEM

FESEM morphology was used to validate the presence of rGO on the glass surface [11]-[12]. Figure 4 shows a FESEM image of the rGO that was successfully deposited utilising a spraying technique on the ZBTNd NPs glass surface. The glass samples displayed a range of uneven, irregularly ordered distribution structures due to van der Waal's interactions and reduction effects [32]. As a result of what may have happened during the electrochemical exfoliation of the GO synthesise process, the FESEM image analysis of rGO displays a stacked structure as represented by the white arrow and an exfoliated structure as shown by the red arrow.

Absorption spectra

The optical absorption was carried out by a UV-Vis spectrometer in the region of 350-950 nm. From this method, it can measure the absorption edge, which is necessary for exploring optically induced rare-earth transitions and obtaining information on the band structure and energy gap of non-crystalline materials in tellurite glass systems [8], [31], [33]. The absorption edges of the rGO-ZBTNd (NPs) glass samples were not sharply defined, which indicates the noncrystalline/glassy nature as confirmed in XRD [34]-[35].

Figure 5 shown the absorption spectra consisting of several eight bands which rise from the ground state ($^4I_{9/2}$) to the excited state. These absorption bands represent the properties of the neodymium ion in the current glass matrix. To identify, a few absorption peak bands at 430, 478, 529, 586, 683, 750, 799, and 877 nm are ascribed from ground state to the excitation state [34], [36]-[37]. Because of the π-π^* electronic transition of sp^2 hybridised C = C bonds, the intensity of the optical absorption spectra was also altered by the presence of rGO [38]. The higher peaks at 589 nm, 750 nm, and 799 nm at 0.05

Nd^{3+} NPs concentrations were referred to by a hypersensitive transition (HST) following the selection rule $|\Delta L| \leq 2$ and $|\Delta J| \leq 2$ (HST), which was "sensitive to the environment, local structure, and symmetry of the glass network" according to [39]-[40].
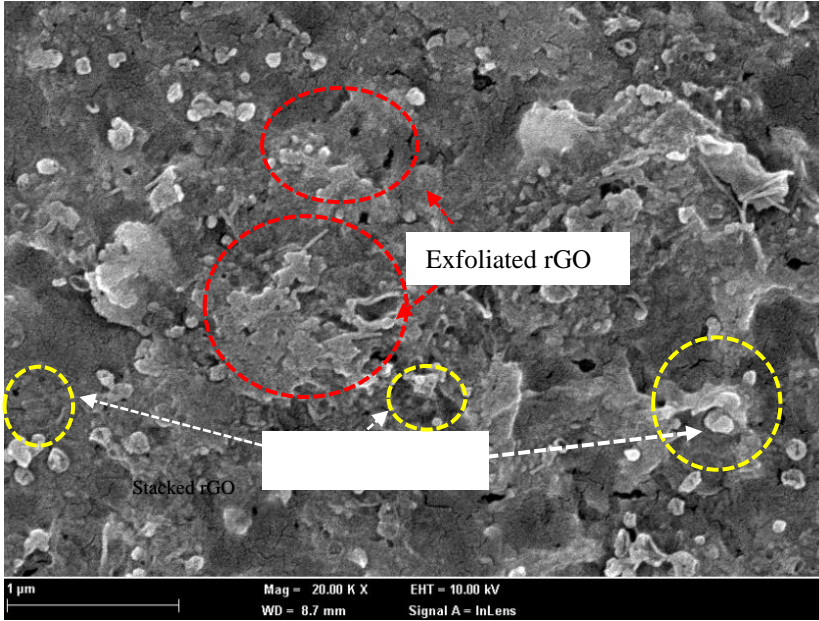

Figure 4: FESEM image rGO- ZBTNd (NPs) glasses

Optical band gap

Mott and Davis stated [41], the optical band gap can be computed using the correlation between absorption coefficients and photon energy. The absorption coefficient $\alpha(\omega)$ could be determined using Equations (1) and (2) using the absorbance data collected from UV-Vis spectroscopy.

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

A denote an absorbance, and d is the thickness of the glass samples

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

where B is an energy-independent constant called band tailing parameter, $\hbar\omega$ is photon energy and n (n = ½ for indirect band gap or n = 2 for direct band gap) is a constant that determine the type of optical transition.
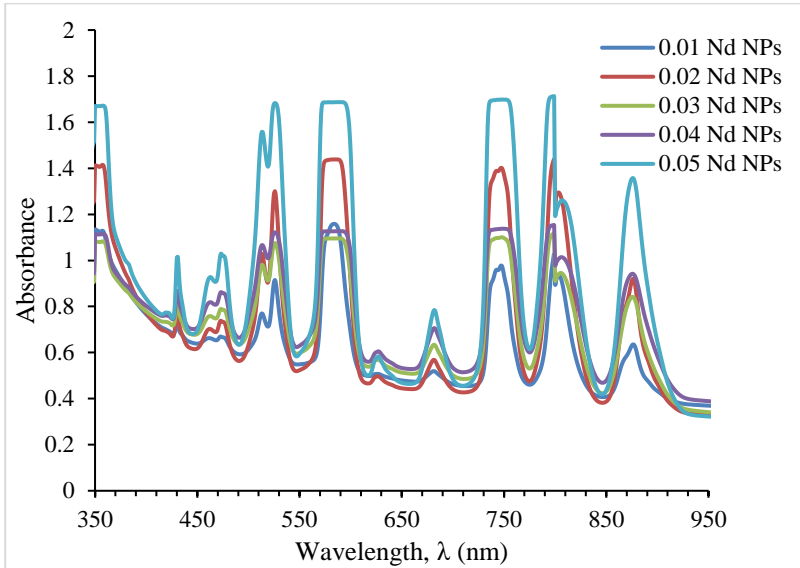
Figure 5: Absorption spectra of rGO - ZBTNd (NPs) glasses

Optical band gap energies (E_g) has been defined from the extrapolating of the linear region meet ħω axis. Tauc's plot $(\alpha\hbar\omega)^{1/2}$ shown as Figure 6 [42]. The data of the indirect bandgap is tabulated in Table 3. The increase value of the indirect of bandgap of the rGO- ZBTNd NPs coated is due to presence of rGO layer on glass surface. Compare to uncoated ZBTNd nano glass the value of the indirect bandgap is show decreasing trend with increase of Nd^{3+} ions due to rGO deposited on glass samples. Decreasing into rGO coated samples are might from reduction process when Addition hydrazine hydrate remove the functional group for sp3 to sp2 transitions from GO to RGO and tune the bandgap of rGO [4], [43].
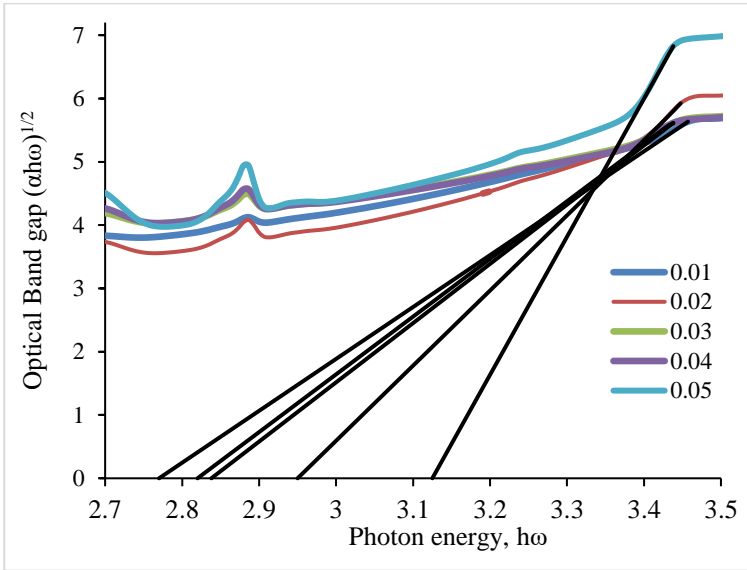
Figure 6: Plot of $(\alpha h\omega)^{1/2}$ versus photon energy hω, rGO- ZBTNd (NPs) glasses for indirect band gap

Table 3: Indirect bandgap Eopt(dir) , refractive index of rGO-ZBTNd NPs and uncoated ZBTNd (NPs) [46]

Neodymium (NPs) concentration	Eopt, Uncoated, eV (±0.1 nm) [46]	Eopt, rGO-coated, eV (±0.1 nm) Present study	Refractive Index,n uncoated (±0.1µm) [46]	Refractive Index, rGO coated (±0.1µm) Present study
0.01	3.201	2.770	1.951	2.657
0.02	3.198	2.980	1.959	2.602
0.03	3.193	2.820	1.983	2.644
0.04	3.179	2.838	1.996	2.339
0.05	3.178	3.125	2.046	2.567

Refractive index

The refractive index (n) is a important variable to consider when analysing the optical properties of a glass solid.The refractive index value of a material can be used to identify its appropriateness for use as an optical or photonic device. A few parameters, such as electron density or electronic polarizability of the glass structure, have a significant impact on the refractive index. The optical

band gap energy derived from Dimitrov and Sakka's [44] relationship may be utilized to determine the refractive index value of prepared samples using the Equation (3).

$$\frac{n^2-1}{n^2+1} = 1 - \sqrt{\frac{E_{opt}}{20}} \qquad (3)$$

The refractive index is represented by n, while the band gap values are represented by E_{opt}. Table 3 shows the refractive index of rGO-ZBTNd (NPs) as well as the neodymium oxide concentrations. It is clear that the number of refractive indexes for prepared samples is increasing, ranging from 2.339 to 2.657. The rise in the refractive index might be attributed to the conversion of the BO_4 structural unit to BO_3, which results in non-bridging oxygen in the glass network, which is more polarizable than bridging oxygen. According to the refractive index value in Table 4, the current glass sample had a greater refractive index than the previous research.It could be related to rGO's high defect rate [12]. Polarizability is also linked to the morphological properties of the rGO structures when the larger surface area [32] of the rGO thin film makes it easy for electrons to move around. The elimination of additional oxygen-rich functional groups from graphene sheets most likely increased the charge transfer barrier between rGO sheets and tellurite glass [17].

Table 4: Refractive index of Nd^{3+} nanoparticles doped tellurite glass and previous study

Composition	Refractive index	Literature
$TeO_2.B_2O_3.ZnO.Nd_2O_3$	2.046	Azlan et al. [46]
$(TeO_2)(ZnO)(Nd_2O_3 NPs)x$	2.609	Halimah et al. [45]
GO coated $TeO_2.B_2O_3.ZnO.Nd_2O_3$	2.197	Shaari et al.[12]
rGO coated $TeO_2.B_2O_3.ZnO.Nd_2O_3$	2.657	Present study

Conclusion

Using the melt quenching process and spraying the coating with rGO, the five series of rGO-ZBTNd (NPs) glasses were successfully prepared. According to the XRD data, the glass materials are amorphous. FESEM results shows the existence of a rGO thin layer coated onto tellurite glass, revealing that the irregular distribution changes the optical characteristics due to the reduction of oxygen functional. Based on the aforementioned significant discoveries, the rGO coated on the tellurite substrate indicates that the glasses have promise in fiber laser materials due to their high refractive index more than 2.0 value.

Because of the effect of the rGO coating on the optical properties of tellurite glass, these glass materials are potential near-infrared laser active media.

Acknowledgment

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