



Ultrasonic and Thermal Properties of Bismuth Borotellurite Glasses Doped with NdCl₃



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BISMUTH borotellurite glass systems in the form of 65B₂O₃-12.5TeO₂-12.5Bi₂O₃ - (10-x)Na₂O - xNdCl₃, x=0, 1, 2, 5, and 10 mole % were melted by traditional method and their amorphous nature were examined by using XRD technique. Densities of the prepared glasses were measured and molar volumes were calculated. The ultrasonic pulse-echo technique at 4 MHz nominal frequency was used to calculate ultrasonic velocities (both of Longitudinal and Shear together). Many of known elastic moduli, such as, longitudinal (L), shear (G), Young (E) and bulk (K) moduli were obtained by using the measured density and ultrasonic velocities. Also Poisson's ratio (σ), the softening temperature (T_s), Debye temperature (θ_D), and microhardness (H) were calculated. The glass transition (T_g) temperature, the crystallization temperature (T_c) and the glass stability parameters (K_{sp}) could be calculated from the Differential thermal analysis (DTA) that has been carried out in the temperature range from 300 to about 1100 °K for all prepared samples. It is noticed that the mechanical properties (elastic moduli and hardness) of the prepared glass increase with increase of NdCl₃%. Also, the softening temperature, the glass transition temperature and stability increase with the increase of NdCl₃ % content.

Keywords: Borotellurite glasses; Ultrasonic velocities; Elastic moduli, Debye Temp.; Thermal properties.

Introduction

Recently, researchers give more attention to the structural changes and different properties of borate glasses containing heavy metals ions as these type of glasses can be used in a wide range of applications. Also, these types of special glasses can be used in optical and electronic engineering as sensors for temperature [1]. A lot of studies on borate glasses prove that these types of glasses have a lot of effectiveness for uses as soldering materials, welder for joints, coating and a lot of different uses [2-4]. It is well known that borate glasses containing alkaline earth oxides or alkali oxides are in a good agreement in the structure and that is completely proved by examining of their FTIR spectra. Also the alkaline and alkali borate glasses are mainly composed of two types of groups which known as tetrahedral (BO₄)

and triangular borate (BO₃) units which always vibrate in specific well known wave numbers [5]. Neodymium (Nd³⁺) ion is known as the most studied lanthanide ions in different glass types, such as silicate [6], borate [7, 8], phosphate [9], tellurite [10], germinate [11] and fluoride glasses. The properties of borate glasses mainly depend on two factors one of them is the types of borate groups present in the glass and the second is the kind and concentration of additives. The addition of rare earth ions to borate glasses (specially Nd₂O₃) leads to change in the structure of borate glasses due to the change in the boron coordination states. Thus, these remarkable changes lead to noticeable changes in both chemical and physical properties of glasses. For example, the addition of neodymium ions (Nd³⁺), which have special optical properties, to the borate glasses produce specific glasses which can be used for laser

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diode. The borate glasses containing Nd_2O_3 are characterized by their extraordinary hardness and very good chemical durability so they are considered as candidate glasses used to prepare glass refractories.

There has been increasing interest in measurement of elastic properties of solid materials by using ultrasonic techniques. They have a very desirable feature of being a form of nondestructive testing. The dynamic (ultrasonic) methods of measuring the elastic properties of solids have gained a wider interest over the other static methods due to its experimental simplicity and its versatility where few millimeters samples may be used in these measurements [12- 14]. Previously, physical properties of different tellurite glasses have been measured [15-18]. Also, the mechanical properties of different Glasses were studied by ultrasonic technique [19-21].

Recently, the studied glasses which contains NdCl_3 have been prepared and their optical parameters are estimated [22]. The studied glasses belong to bismuth borotellurite glasses which containing glass former (borate) with two conditional glass formers (tellurite and bismuthate).

The goal of the present work is to study how the replacement of alkali oxide, 10 mol% Na_2O , by rare earth ion (NdCl_3) on both the elastic moduli, hardness and thermal properties of glasses to estimate new glasses characterized by higher hardness. The composition of the base glass is mainly composed of 65mol% B_2O_3 with constant and equal concentrations of TeO_2 and Bi_2O_3 (12.5 mol%) with 10 mol% of Na_2O without NdCl_3 and other glasses produce by replacement of alkali oxide by rare earth chloride, where the concentration of the NdCl_3 is equal 0, 1, 2, 5, and 10 mol % respectively.

Experimental

The glasses under study of nominal composition $65\text{B}_2\text{O}_3\text{-}12.5\text{TeO}_2\text{-}12.5\text{Bi}_2\text{O}_3\text{-(}10\text{-x)Na}_2\text{O - xNdCl}_3$ were prepared using laboratory analytical grade chemicals, Orthoboric acid (H_3BO_3) for B_2O_3 and Na_2CO_3 for Na_2O supplied from BDH Co., BaO was introduced in the form of anhydrous heavy barium carbonate (BaCO_3) supplied Prolab Co., while Bi_2O_3 was added as such from (Sigma Aldrich Co.). The conventional melting annealing technique was used. Well-mixed batches were melted using

Alumina crucibles in SiC furnace (Vecstar, UK) at temperature of 1000°C for 60 minutes. Crucible was swirled frequently at fixed time intervals to obtain adequate homogeneity. Viscous molten were then poured into preheated stainless steel molds and moved to annealing muffle adjusted at 400°C . Annealing muffle was then switched off after and left to cool with a rate nearly equal $30^\circ\text{C}/\text{hour}$ to room temperature.

The glasses densities are measured by using Automatic Gas Pycnometers for true density, Ultrapyc 1200e, and apparatus with helium gas. Molar volume which calculated by using the next equation (1):

$$V = \frac{M_w}{\rho} \quad (1)$$

where M_w is the glass molecular weight and ρ is glass density.

The ultrasonic velocities were measured using 4MHz frequency longitudinal wave probe for longitudinal velocity and 4MHz transverse wave probe for shear velocity measurements at room temperature ($21 \pm 2^\circ\text{C}$) by applying the pulse echo technique as explained before [19-21]. The uncertainty of measurements of the ultrasonic velocities is dependent on the samples preparation, to achieve accurate measurement; the samples were grounded and polished to obtain two parallel planes faces. The calculated expanded uncertainty for the longitudinal ultrasonic velocity measurement was better than ± 10 m/s and ± 12 m/s for the shear wave velocity measurements.

Fine powder samples were examined for differential thermal analysis using SEATRAM Instrumentation Regulation, Labsys TM TG-DSC16 (Setaram, Caluire, France) in inert gas. powder holder with Al_2O_3 as a reference material was heated along with powder holder with the glass under analysis.

Results and Discussion

Measured values of density (ρ) and the calculated values of molar volume (V_m) for all prepared glass samples of composition $65\text{B}_2\text{O}_3\text{-}12.5\text{TeO}_2\text{-}12.5\text{Bi}_2\text{O}_3\text{-(}10\text{-x)Na}_2\text{O-xNdCl}_3$ where x ranged from 0 to 10 mol% are presented in Table 1 and shown in Fig.1. From both table and figure we can notice that there is an increase in the values of the density from 3.95 to $4.17 \text{ gm}/\text{cm}^3$ with the increase of NdCl_3 from 0 to 10%. whereas the calculated values of the molar volume decrease gradually from 30.48 to $28.09 \text{ cm}^3 \cdot \text{mol}^{-1}$ with the

increase of NdCl_3 from 0 to 10%

It is well known that the values of both (ρ) and (V_m) for the glass network be influenced by many factors for example glass composition, structural units, number of coordination of glass former, cross link and interstitial spaces dimensions

[23]. As shown in Table 1 the density values are increasing with the addition of NdCl_3 to the glass system and this may be due to the fact that NdCl_3 have higher molecular weight (=250.6 g/mol) in compare to Na_2O (=61.98 g/mol). Accordingly the studied glasses become more compactness with

TABLE 1. Chemical composition, densities, and molar volumes for all glass sample Nd ion concentration (N), distance between two nuclie (r_i), Polaron radius (r_p) and field strength (F).

Sample	B_2O_3 %	TeO_2 %	Bi_2O_3 %	Na_2O %	NdCl_3 %	Density (g/cm^3)	Molar volume (cm^3/mol)	N $\times 10^{20}$ ions/ cm^3	$r_i \times 10^{-8}$ cm	r_p Å	F $\times 10^{14}$ cm^{-2}
0 Nd	65	12.5	12.5	10	0	3.95	30.48	-	-	-	-
1Nd	65	12.5	12.5	9	1	3.97	30.21	0.00	17.68	3.31	13.18
2 Nd	65	12.5	12.5	8	2	4.01	30.06	0.36	3.02	0.57	451.01
5 Nd	65	12.5	12.5	5	5	4.12	29.60	0.89	2.24	0.42	822.39
10 Nd	65	12.5	12.5	0	10	4.17	28.09	1.69	1.81	0.34	1260.67

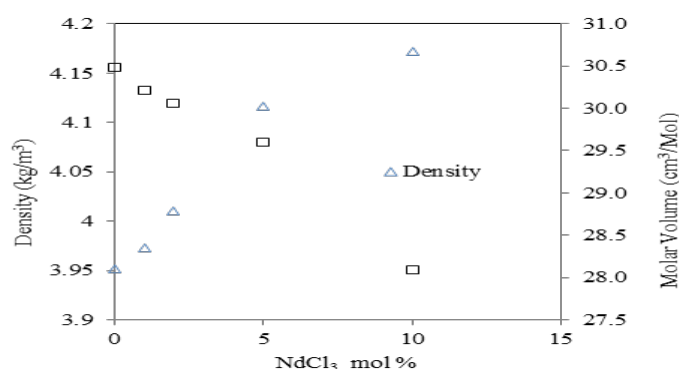


Fig. 1. Variation of density and Molar Volume with mole % of NdCl_3 .

the addition of rare earth ions.

The concentration of Nd^{3+} (N_{Nd}), distance between two nuclie (r_i), Polaron radius (r_p) and field strength (F) the bond between Nd and Cl in the investigated glasses were calculated [24] and given in Table 1.

$$\text{Nd ions concentration} = (\text{mol\% of } \text{NdCl}_3) \times \frac{(N_A)(\rho)}{\text{glass average molecular weight}} \quad (2)$$

$$\text{distance between two nuclie } (r_i) = \left(\frac{1}{N}\right)^{1/3} \quad (3)$$

$$\text{Polaron radius } (r_p) = \frac{1}{2} \left(\frac{\pi}{6N}\right)^{1/3} \quad (4)$$

$$\text{field strength } (F) = \frac{Z}{r_p^2} \quad (5)$$

where N_A is Avogadro's number, ρ is the density of glass and G is the mass of Nd ion.

As shown in Table 2, the addition of NdCl_3 increases both the Nd ion concentration (N_{Nd}) and field strength (F) whereas both (r_i) distance between two nuclie and polaron radius (r_p) decrease. These results may be due to the addition of NdCl_3 which leads the matrix to have a lot of Nd ions and there is a decrease in the distance between Nd and oxygen which creates stronger field strength (F) around Nd ions [8]. The decrease in distance between two nuclie (r_i) (r_i) and polaron radius (r_p) makes the glass network more compacted which explain the decrease in molar volume

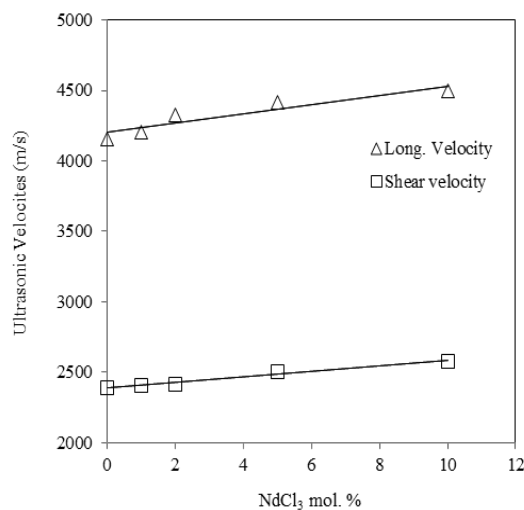


Fig. 2. Variation of longitudinal, V_L , and shear, V_s , ultrasonic velocities with mole percentage of NdCl_3 .

Figure 2 shows the variation of the values of the measured longitudinal (V_L) and transverse ultrasonic (V_s) velocities of all studied glass samples at room temperature with change in the concentration of NdCl_3 % from 0 to 10%. The ultrasonic longitudinal velocity increases from 4157 to 4496 m/sec and the ultrasonic shear velocity increases from 2387 to 2578 m/sec. The continuous and regular increase in both longitudinal and transverse ultrasonic velocities may be attributed to the fact that when the NdCl_3 % increases, Nd ions enter the bismuth borotellurite network glasses and consequently increase the density values, therefore the compactness and connectivity increases, hence, the longitudinal and shear ultrasonic waves transmit more easily and faster inside the glass network structure.

Longitudinal elastic (L), shear elastic moduli (G), bulk elastic moduli (K), Young's elastic moduli (E) and Poisson's (σ) are calculated using the longitudinal ultrasonic velocity, the shear ultrasonic velocity and the density as shown in the next relations [20, 26].

$$L = \rho V_L^2 \quad (6)$$

$$G = \rho V_s^2 \quad (7)$$

$$K = L - \left(\frac{4G}{3}\right) \quad (8)$$

$$E = (1 + \sigma)2G \quad (9)$$

$$\sigma = \left(\frac{L-2G}{2(L-G)}\right) \quad (10)$$

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Value of these moduli listed in Table 2 and shown in Fig. 3. It is clear that as the NdCl_3 increases from 0 to 10%, the longitudinal elastic moduli L increases from 68.3 to 84.3 GPa, the shear elastic moduli G increases from 22.5 to 27.7 GPa, bulk elastic moduli B from 38.3 to 47.4 GPa, Young's elastic moduli E from 56.5 to 69.6 GPa. The increase in all elastic moduli with the increase of NdCl_3 % contents may be attributed to the increase in the rigidity of the studied samples [21].

Poisson's ratio is known as the ratio between the lateral to longitudinal strain produced when tensile forces are applied [25, 26]. It depends on dimensionality and cross link density. In glasses, the cross-links (cross link density) does not effect on the values of tensile strain founded in the network while the values of lateral strain is found to be affected by the cross link density in inversely way because these [27, 28] cross- links generate strong covalent force to resist lateral contraction and consequently decrease, Poisson's ratio if its value increases and vice versa.

The cross link density \bar{n}_c is calculated from the relation

$$\bar{n}_c = \frac{1}{\eta} \sum (n_c)_i (N_c)_i \quad (11)$$

$$\eta = \sum (N_c)_i \quad (12)$$

where n_c is the number of cross-links per cation (number of bridging bond per cation minus two)

TABLE 2. The longitudinal ultrasonic velocity V_L , the shear ultrasonic velocity V_s , the longitudinal moduli L , the shear moduli G , the bulk moduli B , Young's moduli E and Poisson's μ , the micro hardness H , the deby's Temperatur Θ_D , and the softening temperature T_s for all studied glasses.

Sample	V_L m/sec	V_s m/sec	L (GPa)	G (GPa)	B (GPa)	E (GPa)	μ	BO4 ratio	H (GPa)	Θ_D (K)	T_s (K)
0 Nd	4157	2387	68.3	22.5	38.3	56.5	0.25	0.47	3.69	344.74	630.0
1 Nd	4205	2402	70.2	22.9	39.7	57.7	0.26	0.38	3.70	346.27	645.8
2 Nd	4328	2417	75.1	23.4	43.9	59.7	0.27	0.35	3.54	348.77	661.8
5 Nd	4413	2504	80.2	25.8	45.8	65.2	0.26	0.33	4.09	359.81	735.6
10 Nd	4496	2578	84.3	27.7	47.4	69.6	0.26	0.27	4.53	365.02	823.6

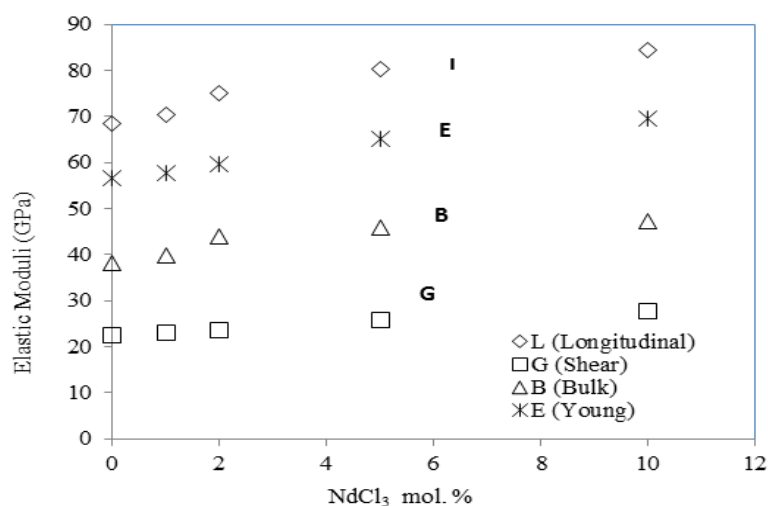


Fig. 3. Variation of Elastic moduli with mole percentage of $NdCl_3$.

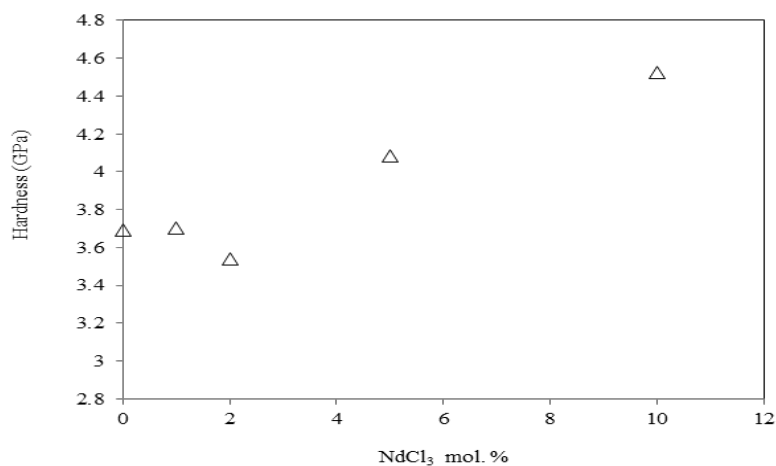


Fig. 4. Variation of microhardness with mole percentage of $NdCl_3$.

in the oxide I, N_c is the number of cation per glass formula and η is the total number of cations per glass formula unit. In the system under study the total number of cations per glass formula is decreasing consequently the cross link density is decreasing therefore the Poisson's ratio increases from 0.25 with 0% NdCl₃ to 0.27 with 2% NdCl₃ [25], the decrease of Poisson's ratio to 0.26 with higher concentrations of NdCl₃ (5 % and 10%) may be attributed to other predominant factors such as the increase of tensile strain or dimensionality [26].

Micro hardness (H) is calculated from Young's Modulus and Poisson's ratio Eq. (11) [29-31]. It is known as the stress required eliminating the free volume (deformation of the network) of the glass. The calculated values of micro hardness for investigated glass samples are listed in Table 2 and shown in Fig. 4.

$$H = \frac{(1 - 2\sigma)E}{6(1 + \sigma)} \quad (13)$$

As shown in Table 2 and Fig 4, there is an increase in the values of micro hardness which is expected from the increase in the elastic moduli and consequently an increase the softening temperature as indicated in Table 2. Debye temperature Θ_D , of B₂O₃-TeO₂-Bi₂O₃-Na₂O₃ glasses containing different concentrations of NdCl₃ contents were determined using the equations (14 and 15) [29-31].

$$\theta_D = \left(\frac{3}{4\pi}\right)^{1/3} \left(\frac{h}{k}\right) V_m \left(\frac{N_A}{V_a}\right)^{1/3} \quad (14)$$

where n equal the atom numbers present in the glass chemical formula, h is the constant of Planck, k_B the Boltzmann's constant, N_A the Avogadro's number, V_a is the molar volume calculated from the molecular weight and density (i.e. M/ρ), and V_m the mean ultrasonic velocity defined by the

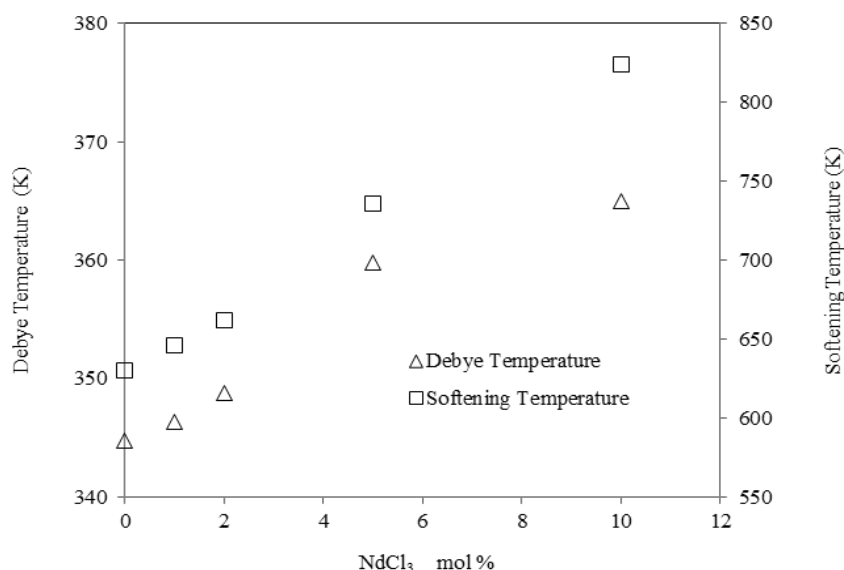


Fig. 5. Variation of Debye's and softening temperature.

TABLE 3. Glass temperature T_g , crystallization temperature T_c , and K_{sp} of (65B₂O₃-12.5 TeO₂-12.5Bi₂O₃-(10-x) Na₂O-x NdCl₃), where (0 ≤ x ≤ 10 mol %) glasses.

Sample	NdCl ₃ %	T _g	T _c	ΔT	K _{sp}
0 Nd	0	420	598	152	9.409
1 Nd	1	425	613	159	10.8
2 Nd	2	432	610	184	10.7
5 Nd	5	453	596	121	9.94
10 Nd	10	460	600	120	7.826

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equation (15)

$$V_m = \left[1/3 \left(\frac{1}{v_l^3} + \frac{2}{v_s^3}\right)\right]^{-1/3} \quad (15)$$

where M is the effective molecular weight.

The calculated Debye temperatures are listed in Table 2 and also shown in Fig 6. It is clear in Fig. 6 that NdCl₃% increases the debye temperature increases from 344.74 to 365.02 K. The increase of Debye's temperature as the NdCl₃ increase is interpreted on the base of change of both the number of atoms in the glass chemical

formula and the increase of mean ultrasonic velocity. Softening temperature (T_s) of glass refers to the temperature at which the substance attains a particular degree of softening under specified conditions of test. It is related to the ultrasonic velocity of shear waves (V_s) by the equation (16).

$$T_s = \frac{v_s^2 M}{c^2 n} \quad (16)$$

where M is the effective molecular weight and C a constant of proportionality and has the value $507.4 \text{ m.s}^{-1}.\text{K}^{1/2}$, n equal the atom numbers present in the glass chemical formula. The calculated softening temperatures are listed in Table 3 and shown in Fig. 5. It is clear in this figure that, as the $\text{NdCl}_3\%$ increases from 0 to 10% the softening temperature increases from 630 to 823.6 K. The increase of softening temperatures as the $\text{NdCl}_3\%$ increase is attributed to the increase of the thermal

energy needed to overcome the increase in the field strength (F) as shown in Table 1.

The DTA curves of all studied systems are shown in Fig. 6, the interpreted glass transition temperature T_g and crystallization temperature T_c from these curves are listed in Table 3. As shown in Fig. 6 and Table 3 the values of glass transition temperature, T_g , of studied glasses increase with the addition of Nd ions. The T_g is ranged from 420 for base glass to 460 °C for glass containing 10 mole % of NdCl_3 which indicated that glass becomes more stable with the addition of the NdCl_3 . It is known that the glass transition, T_g , is a kinetic process, and it mainly influenced by heating rate of the DTA measurement. Generally, T_g is occurring at higher temperature when a higher heating rate is used [31-32]. Glasses with high T_g are closely packed compared to glasses with low T_g , that are loosely packed structure [32],

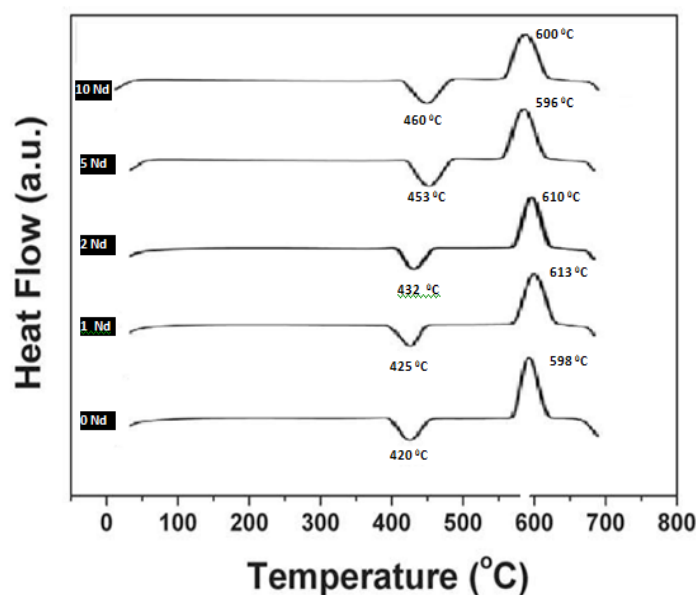


Fig. 6. DTA curves of the $(65\text{B}_2\text{O}_3-12.5\text{TeO}_2-12.5\text{Bi}_2\text{O}_3 - (10-x) \text{Na}_2\text{O} - x \text{NdCl}_3)$, where $(0 \leq x \leq 10 \text{ mol } \%)$.

with a modified number of NBO's in the network. The increase of T_g indicated that the glass samples become more compact and NBO decrease. It is well known that the glasses with higher T_g are more desirable due to overcoming the change in the temperature (resist thermal damage). T_g for studied glasses is higher than many studied previously glasses [32-36]. Stability of glasses against crystallization can be measured as $\Delta T = T_x - T_g$. The ΔT values are listed in Table 3. Values

of ΔT were varied in the temperature range 120 to 184 °C, which mean that the melted glass samples are highly stable. when the value of ΔT is > 100 °C that means the studied samples have good thermal stability for optical fibers fabrication. The variation of ΔT is may be due to the effect of Nd ions in the structure of the studied glasses. Glass stability parameters (K_{sp}) [36], was calculated to evaluate the thermal stability of the studied

glasses more appropriately by using the common expression:

$$K_{sp} = (T_c - T_x)(T_c - T_g) / T_g \quad (17)$$

Where T_x is the crystallization onset temperature.

Values of glass stability parameters changed from 9.409 for base glass to 10.747 for glasses containing 2 mole % NdCl_3 , then decrease to 7.826 for 10 mole % of glass containing 10 mole %. All obtained values indicate that, all studied glasses have high stability against crystallization and also depends on amount of doped NdCl_3 according to Table 3.

Conclusion

Bismuth borotellurite glass system mainly composed of 65mol% B_2O_3 , 12.5mole % of TeO_2 , 12.5 mol% Bi_2O_3 , $(10-x) \text{Na}_2\text{O}$ and $x\text{NdCl}_3$ where x ranged from 0 to 10 mol% are prepared by melting technique. With the addition of NdCl_3 , density of the prepared glasses were found to be increasing from 3.95-4.17 g/cm^3 meanwhile the volume of one mole (V_m) was found to be decreased from 28.1 to 30.5 cm^3/Mol .

Ultrasonic and thermal study show increase in longitudinal velocity, shear velocity and elastic moduli (longitudinal, shear, Bulk and Young). Glass temperature T_g , were increased from 420 to 460 °C and KSP were decreased from 9.4 to 7.8. all studied glasses have high stability against crystallization and also depends on amount of doped NdCl_3 according to Table 3.

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الخصائص فوق الصوتية والحرارية لزجاج البوروتيلوريت المطعم بكلوريد النديميوم

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تم صهر نظام من الزجاج ذو التركيب الكيميائي $-12.5\text{TeO}_2-12.5\text{Bi}_2\text{O}_3 - (10-x) \text{Na}_2\text{O} - x\text{NdCl}_3$, $x=0, 1, 2, 5, \text{ and } 10$ mole % بطريقه تقليديه واختبار طبيعة الزجاج الناتج باستخدام XRD. وتم قياس قيم الكثافه وحساب الحجم الجزئي وقياس سرعة الموجات فوق الصوتية الطولية والعرضية عند تردد 4 ميغاهرتز بطريقة النبض فوق صوتي واستخدمت تلك القياسات في حساب معاملات المرونه الطولية والعرضيه والحجميه وكذا معامل يونج ونسبة بواسون ومعامل الصلاده. أيضا تم حساب درجة حرارة التليين ودرجة حرارة انتقال الزجاج. وباستخدام تقنية التحليل الحراري التفاضلي تم تعيين درجة حرارة التبلور ومعمل ثبات الزجاج في المدى من 300 م⁵ الى 1100 م⁵. ووضحت النتائج زيادة الخصائص الميكانيكيه (معاملات المرونه والصلاده) بزيادة نسبة كلوريد النديميوم المضافه وكذا زيادة درجة حرارة التليين وانتقال الزجاج وثباته.