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ULTRASONIC INVESTIGATION ON ELASTIC AND MECHANICAL PROPERTIES OF BORATE DOPED GLASS SPECIMEN

S.Thirumaran[1*], A.Priyadharsini[2], N.Karthikeyan[3]

[1]Department of Physics, Annamalai University, Annamalai nagar- 608 002

[2]Research Scholar, Department of Physics, Annamalai University, Annamalai Nagar- 608002

[3]Department of Physics, SBM College of Engineering & Technology - Dindugal, Tamil nadu

*e-mail: thirumaran64@gmail.com

Abstract. The present work divulges about ultrasonic characterization on the elastic and mechanical properties of borate doped ternary glass systems. The Ultrasonic wave velocity (longitudinal and shear) and density for the two ternary (B_2O_3-WO_3-Bi_2O_3) BTB and (B_2O_3-SiO_2- Bi_2O_3) BSB glass systems have been measured at room temperature by using Pulse–Echo technique at 5 MHz. The glass samples were prepared by melt-quenching method. The measured experimental values have been utilized to evaluate the Elastic modulus, Poisson's ratio, Acoustic Impedance, Debye Temperature, and Thermal Expansion Coefficient. The incorporation of heavy metal ion in BTB glass system is attributing for its decrease of most of its parameters. The elevation of Debye Temperature in this study advocates structural rigidity and compactness of BSB glass system over BTB glass system. The trends of other evaluated parameters supporting this. The UV-Vis (DRS) study on the band gap energies of these glass specimens confirming their amorphous nature and suggesting their nature of rigidity.

Keywords: Poisson's ratio, Acoustic impedance, Debye temperature, Thermal expansion Coefficient, Elastic Modulus

1. Introduction

Ultrasonic characterization of materials is a versatile tool for the inspection of their microstructure and their mechanical properties. The glassy materials have become increasingly important in the field of solid state ionics. In recent years, bismuth borate glasses attracted the attention of researchers due to their useful physical properties, their important applications in the field of glass ceramics, thermal and mechanical sensors, reflecting windows, and so forth. A survey of the literature shows that there are many reports available on ternary bismuth borate glasses. Borate glasses are widely used in many applications lower such as electronic products [1]. The studies on the borate glasses is not new since borate glasses offer good heat stability and melting temperature compared with other glasses [2]. Recently many researchers made modifications on the borate glass by changing modifiers or / and activators [3]. Recent work with, glasses containing bismuth oxide have received increasing interest due to their potential applications as nonlinear optical materials, glass-ceramics, super conductor materials and fast ion conducting glasses[4]. Tungsten oxide containing glasses is of great interest because they can exhibit unusual electro chromic or photosensitive properties related to the ability of tungsten atoms to exhibit various oxidation states (W^{4+}, W^{5+} OR W^{6+}). WO_3 has exhibited interesting optical property like electrochromism, which has important applications in gas sensor, optical smart window and

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display devices. It does not form glasses by itself but so readily in combination with other glass formers such as B_2O_3. The tungsten ion exists in different valent states W^{6+},W^{5+},W^{4+} etc. [5] hence its doping can affect the structure and optical properties of the host glasses.

The objective of the present work is to probe the structural characterization using ultrasonic study of ternary glass systems doped with borate as (i) B_2O_3 -WO_3- Bi_2O_3 (BTB glass system) and B_2O_3 -SiO_2- Bi_2O_3(BSB glass system) with a view to testifying their compactness and rigidity due to the doping with borate content. The UV-Visible study was also carried out in the light of confirming of its amorphous nature as well as identifying the soft or hard category of glass specimen.

2. Experimental techniques

The ultrasonic interferometer is a simple and direct device to determine the ultrasonic velocity in liquids. An ultrasonic interferometer (model: F81) supplied by M/S. Mittal Enterprises, New Delhi, having the frequency $2MH_Z$ (with an overall accuracy of ± 1 ms^{-1}) has been used for velocity measurements. For UV-Visible DRS studies, the glass samples were finely ground in an agate pestle and mortar. Diffuse reflectance spectra of the samples under investigation were taken using UV1600 series Shimadzu spectrophotometer at room temperature. The relative diffuse reflectance was measured with $BaSO_4$ powder taken as reference.

Preparation of Glass Samples. The chemicals utilized for the preparation of glass samples were obtained from the reputed chemical companies. The glass composition was prepared in mole percentage (mol %). The Borate content is increased in steps of 2 mol % in both BTB and BSB glass systems. Heavy metal as WO_3 is kept as constant in BTB glass system and low density SiO_2 in BSB glass systems. Glass specimens have been prepared by using the conventional melt- quenching technique. The proper compositions were mixed together by grinding the mixture repeatedly to get powdered form. The mixture is melted in silica crucible and it was carried out under controlled muffle furnace with occasional stirring. The temperature in the controlled furnace was gradually raised to a higher temperature at the rate of 373 K per hour and a glassy structure was noticed at 1303 K for BTB glass systems and 1353 K for BSB glass systems respectively. Eventually the molten glass melt was immediately poured on a heavy copper molding block bearing the dimensions 12mm diameter and 4mm thickness kept at a room temperature. Then the glass samples were annealed at 673 K for two hours to avoid the mechanical strains developed during the quenching process. The muffle furnace was switched off and glass was allowed to cool gradually at room temperature. The prepared glass samples were polished and their surface is made perfectly plane and smoothened. The glass compositions in mol % are given in Table 1 and the photographs of BTB and BSB glass systems are displayed in Fig. 1.

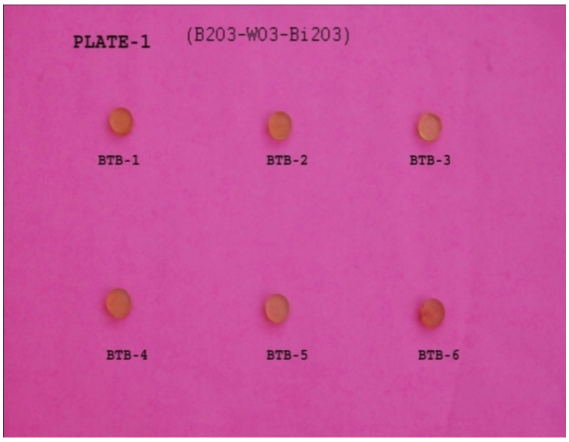
Measurement of density of glass samples. The density of the glass samples was measured using relative measurement method. Benzene was used as a buoyant liquid. The glass samples were weighed both in air and after immersing in benzene at 303K. The weight of the glass samples was measured in a single pan with an accuracy of 0.0001g. The density was calculated using

$$\rho = \rho_B \frac{W_1}{W_1 - W_2}, \qquad (1)$$

where W_1 and W_2 are the weights of the glass samples in air and in benzene and ρ_B is the density of the benzene at 303K.

Table 1. Composition of Glass Specimen

S. No.	Glass specimen	Composition in mol %	Remark
System I (B_2O_3 – WO_3- Bi_2O_3) BTB glass systems			
1	BTB-1	65-05-30	Mol% of WO_3 is kept constant
2	BTB-2	67-05-28	
3	BTB-3	69-05-26	
4	BTB-4	71-05-24	
5	BTB-5	73-05-22	
6	BTB-6	75-05-20	
System II (B_2O_3- SiO_2-Bi_2O_3) BSB glass systems			Mol% of SiO_2 is kept constant
1	BSB-1	65-07-28	
2	BSB-2	67-07-26	
3	BSB-3	69-07-24	
4	BSB-4	71-07-22	
5	BSB-5	73-07-20	
6	BSB-6	75-07-18	



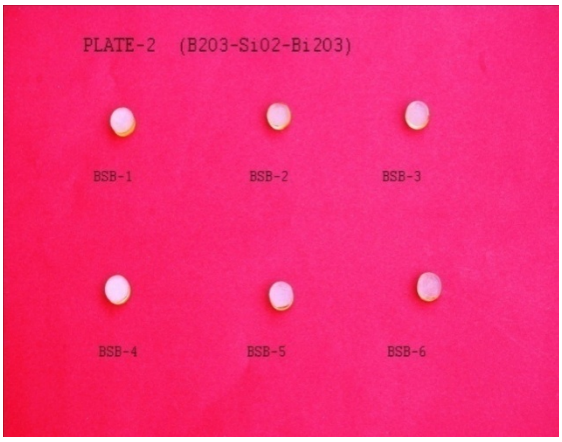


Fig. 1. Plate - 1. BTB glass system and Plate - 2. BSB glass system

Measurement of Ultrasonic Velocity. The ultrasonic longitudinal (U_L) and shear wave velocities (U_S) of the glass specimen were measured by using the pulse-echo method at room temperature at a frequency of 5 MHz with X-cut and Y-cut transducers. These transducers act as both transmitters and receivers of the ultrasonic pulse. The transducers were brought into contact with each of the twelve samples by means of a couplant, in order to ensure that there was no air void between the transducer and the specimen.

UV-visible DRS study. Diffuse Reflectance Spectroscopy (DRS) is a simple, but powerful spectroscopic tool to estimate the band gap energy (E_g) of powder samples unambiguously. Band gap is generally obtained from optical absorption edge energy, which is defined as the minimum photon energy required to excite an electron from the highest occupied molecular orbital (HOMO, at the top of the valence band in semiconductor domains) to the lowest unoccupied molecular orbital (LUMO, at the bottom of the conduction band) [6]. Electronic transitions are of two types – direct and indirect. Direct transitions require only that photons excite electrons, whereas indirect transitions require vibrations and energy from the crystal lattice (phonons). Kubelka–Munk (KM) theory provides the theoretical descriptions of diffuse reflectance spectroscopy [7-9]. When a semiconductor absorbs photon of energy larger than the gap of the semiconductor, an electron in transferred from the valence band to the conduction band, where there occurs an abrupt increase in the absorbency of the materials to the wavelength corresponding to the band gap energy whether the electronic transition is direct or indirect. UV-Vis DRS help to obtain the optical band gap energies, the experimental data, assuming Direct and Indirect band transitions [10].

3. Results and discussion

When the powdered sample is radiated with light, a portion is regularly reflected at a powder surface and the remaining enters the powder and diffuses. As light of particular wavelength is absorbed by the sample, the measurement of the diffuse reflected light at different wavelengths yields a spectrum called diffuse reflectance spectrum. Optical absorption is a useful method for investigating optically induced transition and to get information about the bond structure and energy gap of non-crystalline materials.

The calculated values of absorption edge or cut-off wavelengths and band gap energies for BTB and BSB glass systems are reported in Table (2).

Table 2. Cutoff Wavelength and Band Gap Energy for BTB and BSB Glass Systems

Sample	Cutoff Wavelength	Theoretical values of band gap (ev)	Band gape Eg (ev)	
			Direct	Indirect
		System I : B_2O_3- WO_3-Bi_2O_3		
BTB-1	330	3.75	3.35	2.90
BTB-2	327	3.79	3.40	3.04
BTB-3	325	3.81	3.45	3.06
BTB-4	322	3.85	3.46	3.08
BTB-5	321	3.86	3.49	3.10
BTB-6	320	3.87	3.50	3.18
		System II : B_2O_3- SiO_2-Bi_2O_3		
BSB-1	318	3.89	3.49	3.11
BSB-2	316	3.92	3.52	3.12
BSB-3	315	3.93	3.56	3.14
BSB-4	314	3.94	3.60	3.16
BSB-5	312	3.97	3.65	3.25
BSB-6	301	4.11	3.68	3.27

From the present UV-Vis DRS measurements, it is seen that the band gap values increases around 3.35ev to 3.50ev for BTB glasses for direct method and 2.90ev to 3.18ev for indirect and similarly for BSB glasses found around 3.49ev to 3.68ev for direct method and 3.11ev to 3.27ev indirect method. From the evaluated both direct and indirect transitions, it is clear that the increase of band gap energies observed for both glass systems. The present investigation through UV–Vis DRS study finds that (i) increasing of band gap energy for both glass systems and (ii) decreasing of cut-off wavelength. Increasing band gap suggests a poor rigidity of the glass network after irradiation. This result further supported by the observed blue-shift of the cut-off wavelength [11]. (Towards lower wavelength side). In addition, the increasing band gap can be attributed to the decreasing of non-bridging oxygen atoms after breaking the bonds between boron and oxygen atom. For the amorphous materials, the values of the band gap energy are above 3ev independent of the transition types [12]. Our measured direct and indirect transition values exhibit more than 3ev, confirming the amorphous nature of the glass specimen and this increasing of band gap energy is resulting in decreasing of non-bridging oxygen atoms in the glassy matrix [13,14]. The increasing band gap can be attributed that there is an increasing number of energetic electrons in the glass network with increasing of borate content. This electronic ionization will increase the electronic transition between localized states, so that the band gap increases. It may also further be interpreted as, for the band gap increase it may due to high doped concentrations which causes the broadening of the impurity band and the formation of bond tails on the edges of conduction and valence bands [15]. Hence, probing through UV-Visible study identifies increasing band gap energies of glass specimen confirming their amorphous nature and suggesting their nature of rigidity.

The measured values of density and ultrasonic velocity [both longitudinal (U_L) and shear (Us) of BTB and BSB glass systems and the calculated values of elastic moduli such as Longitudinal modulus (L), Shear modulus (G), Bulk modulus (K), and Young's modulus (E) are reported in Table 3. The perusal of Table 4 shows the values of Poisson's ratio (σ), Acoustic impedance(Z), Debye temperature (θ_D), and Thermal expansion coefficient (αp) and molar volume (Vm) for the two glass systems (BTB and BSB).

Table 3. Values of Density (ρ), Longitudinal Wave Velocity (U_L), Shear Wave Velocity (U_S), and Elastic Moduli of BTB and BSB glass systems

Name of the glass samples	Ultrasonic Wave Velocity U (m/s)			Elastic Modulus			
	Density (ρ) Kg/m^3	Longitudinal Wave Velocity (U_L) m/s	Shear Wave Velocity (Us) m/s	Longitudinal Modulus (L) 10^9 Nm^{-2}	Shear Modulus(G) 10^9 Nm^{-2}	Bulk Modulus (K) $10^9 Nm^{-2}$	Young's Modulus (E) 10^9 Nm^{-2}
System I (B_2O_3- WO_3- Bi_2O_3) BTB-glass systems							
BTB-1	1192.39	3681.81	1021.42	16.1637	1.2440	14.5050	3.6283
BTB-2	1177.58	3666.66	1000.00	15.8318	1.1775	14.2617	3.4381
BTB-3	1160.21	3600.00	983.33	15.0363	1.1218	13.5405	3.2751
BTB-4	1151.51	3482.00	975.90	13.9612	1.0966	12.4990	3.1965
BTB-5	1143.79	3262.00	954.21	12.1706	1.0414	10.7820	3.0268
BTB-6	1135.80	2950.00	939.02	9.8842	1.0015	8.5489	2.8915
System II (B_2O_3- SiO_2-Bi_2O_3) BSB-glass systems							
BSB-1	1210.19	2827.58	968.67	9.6757	1.1355	8.1616	3.2556
BSB-2	1213.79	3272.00	976.19	12.9948	1.1566	11.4525	3.3570
BSB-3	1235.42	3448.78	980.48	14.6941	1.1876	13.1106	3.4585
BSB-4	1242.71	3663.63	982.92	16.6798	1.2006	15.0790	3.5087
BSB-5	1253.84	3754.54	985.54	17.6748	1.2178	16.0510	3.5634
BSB-6	1279.66	3828.57	995.18	18.7571	1.2673	17.0673	3.7102

The density is a vital parameter capable of casting the light on the structure of a glass and it is a very important tool to detect the structural changes in the glass network and are reported in the Table 3.The present study of density notices a decreasing trend in BTB glass system with addition of B_2O_3 and an increasing trend in BSB glass system with the addition of the same content. The increasing density value in BSB glass system may be attributed due to the possession of long chains of BiO_6 distorted polyhedral and octahedral units in Bismuth–Borate glasses, which may increase the randomization of the glassy structure resulting in elevation of density values. The decreasing density values from Table 3 in BTB glass system can be interpreted that the formation of BO_4 units [16,17] due to inclusion of metal ion (WO_3) contents. The tetrahedral BO_4 groups are strongly bonded due to their bond strength, resulting in decrease of optical band gap. However, the inclusion of tungsten ions (WO_3) which are positioned in the cavities of empty band gap space of the network obstructing the formation of BO_4 units further. This makes the density to decrease. The decrease in density assuring the less compact structure of the glass. It is quite expected that the density and the molar volume should exhibit opposite behavior to each other. In the present investigation, the BSB glass systems obey the usual trend, however in BTB glass system, it seems to vary, ie both density and molar volume exhibiting the decreasing behavior in BTB glass system. Such anomalous behavior between density and molar volume were reported earlier [18,19].

Table 3 reporting the evaluated values of longitudinal (U_L) and shear velocities (Us) of BTB and BSB glass systems. These velocities decrease in BTB glass systems linearly with mol % of WO_3 with B_2O_3 content and however increase with mol % SiO_2 with same content for BSB glass systems. The decreasing velocities in BTB glass systems can be interpreted as due to the involvement of tungsten (WO_3) ions as modifiers breaking up tetrahedral bonds of B_2O_3 units and oxygen of WO_2 ions. Consequently, resulting in rupturing of its symmetry of B2O3 and its cations (negative charged atom) occupy the interstitial position. The rupturing of the symmetry of tetrahedral B_2O_3 network and a creation of negative charged atoms (cation) lead to decrease in ultrasonic velocity. In short, the inclusion of heavy metal ion leading the way for decrease of ultrasonic velocities in the glassy matrix. Bhatti and Singh et al. [20] suggested that the addition of SiO_2 will enhance the ultrasonic velocity due to the change in the coordination number of boron from 3 to 4. The elevation of ultrasonic velocity in BSB glass systems points out that SiO_2 plays a dominant role in increasing the ultrasonic velocities. It is obvious that increasing of borate content with SiO_2 would cause a swift movement of the ultrasonic wave inside the network of the glass structure. This will not only to enhance the elevation of ultrasonic velocity but too for making the glassy structure harder and resist deformation [21-23].

The elastic properties yield information regarding the forces that operate between atoms or ions in solid materials. This information provides important clues in interpreting and understanding the nature of bonding in solid materials. These properties are suitable in describing the glass structure as a function of its composition, dimensionality and the connectivity of glass structure. The perusal Table 3 reporting the values of elastic moduli such as longitudinal modulus (L), shear modulus (G) bulk modulus (K) and Young's modulus (E). These values are decreasing in BTB glass systems on increasing the mole% of B_2O_3 with WO_3 and it finds a reverse trend on increasing the mole% of B_2O_3 with SiO_2 in BSB glass systems. The increasing trend of elastic modulus in BSB glass systems indicating the presence of number of layers of covalent bonds which resist the deformation of glassy structure and the addition of glass modifier (SiO_2) in the glass system has contributed to increase in coulomb contribution to the lattice energy too. Such increase of elastic modulus indicating the formation of a complete structure [24] due to creation of bridging oxygen atoms and bond strength of Si-O of the silicate network.

Poisson's ratio is an effective tool in exploring the degree of cross-link density of the glass network and its magnitude increases the cross-link density. In the present study, the value of Poisson's ratio showing an increasing trend in BSB glass system and a reverse trend is seen in BTB glass system. Rajendran et al. [25] reported that Poisson's ratio is affected by the changes in the cross-link density of the glass network, and suggested that structure with high cross-link density have Poisson's ratio in the order of 0.1 – 0.2, while structures with low cross-link density have Poisson's ratio in the order of 0.3-0.5. In BTB glass system, the values of Poisson's ratio are decreases and it is varied from 0.45 to 0.44 and in BSB glass system, it is increased from 0.43 to 0.46 which are presented in Table 3. On noticing the trends of present two glass systems [BSB & BTB] it is obvious that these glass structure posses low cross-link density [26]. The increasing trends of the parameter in BSB glass system is attributed to the strengthening and hardening of the glassy network structure.

The Debye temperature is the temperature at which nearly all modes of vibration in a solid are excited and its increasing trend implies an enhancement in rigidity of the glass and vice-versa. It plays an important role in solid materials in the determination of elastic moduli and atomic vibrations. From the perusal Table 4, one can notice the values of Debye temperature is increasing in BSB glass system and decreasing in BTB glass system. The elevation of Debye temperature indicating the strengthening of the glass structure and higher values of this parameter are reported in BSB glass system comparatively to BTB glass system [27]. This suggests that BSB glass system possess more compactness and rigidity over BTB glass system.

Table 4. Values of Poisson's ratio (σ), Acoustic Impedance (Z), Debye Temperature (θ_D) Thermal Expansion Coefficient (α_p) and Molar volume (V_m) of BTB and BSB glass systems

Name of the glass samples	Poisson's ratio (σ)	Acoustic impedance (Z) ($X10^7$ $kg.m^2s^{-1}$)	Debye temperature (θ_D)K	Thermal expansion co-efficient (α_p).K^{-1}	Molar volume (V_m) (cm^3/mol)
System I (B_2O_3- WO_3- Bi_2O_3) BTB-glass systems					
BTB-1	0.4583	0.4390	597.5803	85404.6619	164.9067
BTB-2	0.4598	0.4317	590.8341	85053.1819	160.2492
BTB-3	0.4596	0.4176	586.478	83506.6699	155.8161
BTB-4	0.4573	0.4009	589.144	80769.0699	150.1095
BTB-5	0.4532	0.3731	583.5908	75665.0699	144.1924
BTB-6	0.4436	0.3350	581.772	68426.6699	138.2277
System II (B_2O_3- SiO_2-Bi_2O_3) BSB-glass systems					
BSB-1	0.4335	0.3421	568.0559	65586.5259	148.6770
BSB-2	0.4511	0.3971	582.1045	75897.0699	141.7054
BSB-3	0.4560	0.4260	596.7896	79998.3659	132.8081
BSB-4	0.4612	0.4552	608.7223	84982.8859	125.6504
BSB-5	0.4629	0.4707	621.8081	87091.9979	118.2130
BSB-6	0.4637	0.4899	642.4795	88809.4939	109.6334

Table 4 reporting the values of acoustic impedance (Z) which are found to be increased in BSB glass system and a reverse trend is observed in BTB glass system. The increasing trend of acoustic impedance can be presumed as the propagation of ultrasonic waves and the corresponding decreasing trend advocate the slowing down of ultrasonic waves in the glass specimen. The other parameter Thermal Expansion Coefficient from Table 4 provides the values for two glass systems. One can observe that thermal expansion coefficient increases with increasing concentration of borate with SiO_2 in BSB glass system and the same decreases with increasing concentration of borate with WO_3 in BTB glass system. Srinivatsava and Srinivasan [28] observed that the thermal expansion coefficient of materials depends on the strength of the bonds. The decrease in thermal expansion values in BTB glass system advocate the decrease in number of bonds per unit volume and hence account for the poor rigidity of the glassy structure. Hence it is obvious that BSB glass system possessing more structural rigidity and compactness over BTB glass system.

4. Conclusion

The presence of heavy metal ion in BTB glass system is attributing for its decrease of most of its parameters such as elastic modulus, Poisson's ratio (σ), Acoustic impedance (Z) etc., the elevation of Debye temperature in this study advocates structural rigidity and compactness of BSB glass system over BTB glass system. The trends of other evaluated parameters confirming this. The UV-Visible DRS investigation confirms their amorphous nature and both glass systems fall under soft glassy structure category.

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