Ultrasonic Study and Physical Properties of Borotellurite Glasses

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Abstract: A series of glasses $(TeO_2)_x (B_2O_3)_{1-x}$ with x = 0.6, 0.63, 0.65, 0.70, 0.73, 0.75, 0.78 and 0.80 were synthesized by rapid quenching. Longitudinal and shear ultrasonic velocity were measured at room temperature and at 5 MHz frequency. Elastic properties, Poisson's ratio and micro hardness have been calculated from the measured density and ultrasonic velocity at room temperature. Estimated parameters based on Makishima-Mackenzie theory and bond compression model were calculated in order to analyze the experimental elastic moduli. Comparison between the experimental elastic moduli data obtained in the study and the calculated theoretically by other models has been discussed.

Keywords: Tellurite glass, borate glass, elastic moduli

INTRODUCTION

The study of tellurite glasses is of scientific and technical interest because they have low melting points, high refractive index, high dielectric constant and good infrared transmission[1]. Tellurium oxide (TeO_2) is a conditional glass former[2] and forms glass only with a modifier such as alkali, alkaline earth and transitional metal oxides or other glass formers. In a binary tellurite glasses, the basic structural unit of TeO_4 is a trigonal bipyramid (tbp) with lone pair of electrons and the structural units take the Te-O-Te bond for glass formation[3].

Ultrasonic nondestructive character of materials is a versatile tool for investigating the change in microstructure, deformation process and mechanical properties of materials over a wide range of temperatures[4]. This is possible due to the close association of the ultrasonic waves with elastic and inelastic properties of the materials. It is also due to the availability of different frequency range and many modes of vibration of the ultrasonic waves to probe into the macro, micro and submicroscopic levels.

Elastic properties are very informative about the structure of solids and they are directly related to the interatomic potentials. Glasses being isotropic and have only two independent elastic constant: longitudinal and shear elastic moduli. These two parameters are obtained from the longitudinal and shear velocities and density of the glass. The elastic constants could be deduced. Tellurite glass system with unique physical properties and applications were reported by El-Mallawany[5].

The main objectives of the present work are to study elastic moduli of the borotellurite glasses based on experimental measurements and theoretical models and also to investigate the structural modification of the borotellurite network induced by the introduction of TeO_2.

MATERIALS AND METHODS

The binary $(TeO_2)_x (B_2O_3)_{1-x}$ glasses were prepared by mixing together specific weights of tellurium dioxide (Aldrich 99.5%) and boron oxide B_2O_3 (Alfa Aesar, 97.5%), in a closed alumina crucible. The mixture were kept for 400^0C for a period of 30 minutes in the first furnace, the crucible was then transferred to a second furnace for 60 minutes at 800^0C. The crucible was constantly shaking in order to have a homogeneous melt. The melt was then poured in a stainless steel cylindrical shaped split mould which had been preheated and then the sample was annealed at $350^0 C$. The prepared samples were cut into required dimension for ultrasonic measurements. The samples were yellowish in colour and free from cracks and bubbles. The surfaces of samples were polished with sand paper to achieve a plane parallelism.

The density of the glasses was determined by Archimedes method as described elsewhere[6]. For measurement of ultrasonic velocity in the glass sample MATEC MBS 8000 was used. All measurements were taken at 5 MHz frequency and at room temperature. The prepared samples were ground into powder form for x-ray diffraction measurement, using X'pert Pro Panalytical.

Elastic moduli (longitudinal, shear, bulk and Young's), Debye temperature and Poisson's ratio of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses with different contents have been determined from the measured ultrasonic velocities and density using the standard relations[1].

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Longitudinal modulus:

$$L = V_L^2 \rho \tag{1}$$

Shear modulus:

$$G = V_s^2 \rho \tag{2}$$

Bulk modulus:

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

Young's Modulus:

$$E = (1+\sigma) 2G \tag{4}$$

Poisson's ratio:

$$\sigma = \left(\frac{(L-2G)}{2(L-G)}\right) \tag{5}$$

The microhardness H, softening temperature T_s and Debye temperature Θ_D were calculated using the following equation[1]:

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

$$T_s = \left(\frac{M_w}{c\rho}\right) v_s^2 \tag{7}$$

Where M_w is the molecular weight of the glass, c is constant equal to 0.5074×10^5 cm $k^{-1/2}s$ and ρ is the density.

And:

$$\Theta_D = \frac{h}{k}\left(\frac{9N}{4\pi V}\right)^{1/3} v_m \tag{8}$$

Where:

$$v_m = \left[\frac{(1/v_l^3) + (1/v_s^3)}{3}\right]^{-1/3} \tag{9}$$

h is the Plank's constant, k is the Boltzman constant, v_m is the mean ultrasonic velocity and (N/V) is the number of vibrating atoms per unit volume and equal to (PN_A) where P is the number of atoms in the chemical formula and N_A is the Avogadro number.

THEORETICAL MODEL

Quantitatively analysis of experimentally determined elastic moduli is based on two theoretical models, bond compression model proposed by Bridge *et al.*[7] and Makishima-Mackenzie model[8,9].

According to the bond compression model[7], this model takes into account the atomic geometry of the oxides such as the bond length and the coordination number. Bridge noted that the average cross-link density per unit formula, $<n_c>$ increases with the first order stretching force constant. The average cross-link density per unit formula, n_c was given by:

$$<n_c> = \frac{1}{\eta}\sum_i x_i \left(n_c\right)_i \left(N_c\right)_i \tag{10}$$

Where n_c is the number of cross-links per unit cation which is equal to the number of bonds minus 2, N_c is the number of cations per unit glass formula unit and η is the total number of cations per unit glass formula unit given by:

$$\eta = \sum_i x_i \left(N_c\right)_i \tag{11}$$

The bond compression model is a useful guide for the structure containing only one type of bond. The bulk modulus on this case is given by:

$$K_{bc} = \frac{n_b r^2 f}{9} \tag{12}$$

Where n_b is the number of network bonds per unit volume, r the bond length and f is the first order stretching force constant and higher order force constant and bond-bond interaction are neglected. The relation between the ratio K_{bc} and that of experimental value K_e and the calculated atomic ring size l is:

$$K_e = 1.0106 F l^{-3.84} \tag{13}$$

Where l is in nm and F is in N/m

Makashima and Mackenzie[8,9] have presented a theoretical calculation model, in terms of oxides glasses, only taking into consideration the dissociation energy of the oxide constituents per unit volume (G_i) and the packing density (V_t). The elastic moduli and Poisson's ratio were given as follows:

$$E_m = 2V_t G_t$$
$$K_m = 100 V_t^2 \Sigma G_i x_i$$
$$G_m = \frac{3EK}{9K-E} \tag{14}$$
$$\sigma_m = \frac{E}{2G_m} - 1$$

Where G_m is shear modulus and x_i is the mole fraction of the component i of an oxide glass.

RESULTS AND DISCUSSION

X-ray diffraction patterns of the studied glass system reveal the absence of any discrete or continuous sharp crystalline peaks but show homogeneous glassy characters.

Table 1: Density (ρ), longitudinal ultrasonic velocity (V_L) and shear ultrasonic velocity (V_S), elastic moduli of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

Mol % X	ρ (g cm^{-3})	V_m(cm^{-3})	V_L (m s^{-1})	V_S (m s^{-1})	L(GPa)	G(GPa)	E(GPa)	K(GPa)
60	4.71	28.24	3467	1981	56.63	18.49	46.50	31.98
63	4.75	26.57	3471	2068	57.28	20.33	49.80	30.17
65	4.79	26.72	3581	2086	61.48	20.86	51.87	33.66
70	4.89	27.13	3608	2185	63.62	23.33	56.48	32.51
73	4.88	27.74	3692	2206	66.49	23.74	58.03	34.84
75	4.89	28.04	3700	2260	66.94	24.98	60.06	33.64
78	4.96	28.19	3800	2300	71.62	26.24	63.55	36.64
80	4.97	28.50	3900	2400	75.59	28.63	68.43	37.42

Table 2: Microhardness (H), Debye temperature Θ_D , softening temperature, T_s and Poisson's ratio σ of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

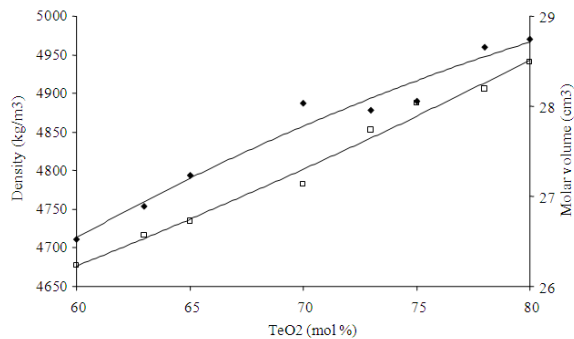| Mole %x	H(GPa)	Θ_D (K)	σ	T_s(K)
60	2.98	290	0.2576	762
63	3.73	299	0.2248	841
65	3.57	301	0.2432	860
70	4.50	310	0.2104	959
73	4.39	309	0.2224	998
75	4.96	314	0.2024	1059
78	5.06	317	0.2109	1103
80	5.82	328	0.1952	1214

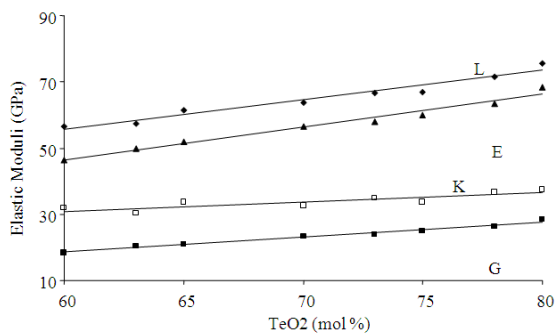Fig. 1: Density and molar volume of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

Fig. 2: Elastic moduli of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

Table 3: Bond length (r), first order stretching force constant (F), coordination number (n) of the oxides TeO_2 and B_2O_3[11,17]

Oxide	r(nm)	F(Nm^{-1})	n
TeO_2	1.99 x 10^{-10}	216	4
B_2O_3	1.38 x 10^{-10}	660	3

Figure 1 shows the variation of both density and molar volume for the glasses as function of TeO_2 content (mol %) while their values are listed in Table 1.

It can be seen that the density increases from 4.71 to 4.97 g cm^{-3} and the molar volume increases from 28.24 to 28.50 cm^3.

The dependence of density and molar volume on composition is in agreement with the weight and size of the constituent oxides. The increase in density is due to the atomic weight of Te atom and B atoms are 127.6 and 10.8 g respectively. The observed increase in the molar volume may be attributed to an increase in the bond length or inter-atomic spacing between the atoms or since the ionic radius of Te atom (2.21 Å) is much greater than that of B atom (0.20Å). This trend of density and molar volume has been found by Saddek[10].

The values of density molar volume lie in the same range as that of the tellurite glasses reported by El-Mallawany[11].

The measured values of the longitudinal and shear ultrasonic velocities with variation of TeO_2 content were listed in Table 1. The longitudinal ultrasonic velocity increases from 3467 to 2400 m s^{-1} and shear velocity increases from 1981 to 2400 m s^{-1}. The increase in velocities is attributed to the increase in rigidity of the glass network[10,12]. The structural unit TeO_4 trigonal bipyramids is converted into the structural unit TeO_3 trigonal pyramids and BO_3 trigonal planar to BO_4 tetrahedral which in turn is accompanied by creation of bridging oxygens (BO) that is the rigidity of the glass increases. TeO_3 trigonal pyramids formed by a part of tellurium atoms are connected with BO_4 tetrahedra by vertex sharing and that an excess and defiency of positive charges on TeO_3 and BO_4 units compensate each other and that formation of BO_4 unit is concurrent with that of TeO_3[13].

Figure 2 shows the variation of elastic moduli of borotellurite glass with TeO_2 mol percent and Table 1 compiles the longitudinal modulus L, shear modulus G, Young's modulus E, bulk modulus K. The results indicate that the elastic moduli increase with TeO_2 content.

Table 4: Number of bonds per unit volume (n_b), bond compression bulk modulus (K_{bc}), ratio of (K_{bc}/K_e), atomic ring size (*l*), average force constant (F) and cross link density (n_c) of $(TeO_2)_x$ $(B_2O_3)_{1-x}$ glasses

Mole % x	n_b x 10^{28} (m^3)	K_{bc}(GPa)	K_{bc}/K_e	*l* (nm)	F(N/m)	n_c
60	82.63	90.82	2.84	0.5765	364	1.429
63	82.28	89.43	2.96	0.5801	352	1.460
65	82.26	88.74	2.64	0.5604	344	1.481
70	82.12	86.96	2.68	0.5569	324	1.538
73	80.99	84.82	2.43	0.5418	312	1.575
75	80.55	83.74	2.49	0.5432	305	1.600
78	80.77	83.05	2.27	0.5261	294	1.639
80	80.32	82.00	2.19	0.5198	286	1.667

Table 5: Elastic moduli calculated according to Makishima-Mackenzie model, packing density (V_t) and dissociation energy (G) of $(TeO_2)_x$ $(B_2O_3)_{1-x}$ glasses

Mole % x	E_m (GPa)	K_m (GPa)	G_m (GPa)	V_t	G x 10^7 (kJ m^{-3})
60	69.16	22.27	27.86	0.644	5.370
63	67.54	21.23	27.35	0.629	5.372
65	66.65	20.67	27.07	0.620	5.374
70	64.39	19.28	26.36	0.599	5.378
73	62.26	18.01	25.69	0.579	5.380
75	61.11	17.35	25.33	0.568	5.382
78	60.07	16.75	25.00	0.558	5.384
80	58.95	16.13	24.65	0.547	5.386

Table 6: Experimental elastic moduli (E_e, G_e and K_e), bond compression model (E_{bc}, G_{bc}, K_{bc}) and Makishima and Mackenzie (E_m, G_m and K_m) for Young's, shear and bulk modulus, respectively and Poisson's ratio (σ_e, σ_{bc}, σ_m) of $(TeO_2)_x$ $(B_2O_3)_{1-x}$ glasses

X (mol%)	E(GPa)			G(GPa)			K(GPa)			σ		
	E_e	E_{bc}	E_m	G_e	G_{bc}	G_m	K_e	K_{bc}	K_m	σ_e	σ_{bc}	σ_m
60	46.50	136.80	69.16	18.49	54.77	27.86	31.98	90.82	22.27	0.2576	0.2489	0.2843
63	49.80	135.32	67.54	20.33	54.22	27.35	30.17	89.43	21.23	0.2248	0.2478	0.2791
65	51.87	134.68	66.65	20.86	54.00	27.07	33.66	88.74	20.67	0.2432	0.2471	0.2760
70	56.48	132.94	64.39	23.33	53.38	26.36	32.51	86.96	19.28	0.2104	0.2452	0.2680
73	58.03	130.21	62.26	23.74	52.33	25.69	34.84	84.82	18.01	0.2224	0.2441	0.2599
75	60.06	128.91	61.11	24.98	51.84	25.33	33.64	83.74	17.35	0.2024	0.2434	0.2554
78	63.55	128.36	60.07	26.24	51.66	25.00	36.64	83.05	16.75	0.2109	0.2424	0.2510
80	68.43	127.07	58.95	28.63	51.16	24.65	37.42	82.00	16.13	0.1952	0.2417	0.2462

Longitudinal modulus ranged from 56.63 GPa to 75.59 GPa, shear modulus from 18.49 GPa to 28.63 GPa, Young's modulus from 46.50 GPa to 68.43 GPa and bulk modulus from 31.98 GPa to 37.42 GPa. The increase in elastic moduli is due to an increase in the rigidity of glass samples. The rigidity may be attributed by creation of bridging oxygens from the formation of BO_4 and TeO_3 in the glass network.

Poisson's ratio decreases from 0.2576 to 0.1952 when TeO_2 content increases as shown in Fig. 3. The decrease in poison's ratio is attributed to the increase in the crosslink density of the glass as proposed by Higazy and Bridge[7]. Debye temperature represents the temperature at which nearly all modes of vibration in a solid are excited and its increase implies an increase in the rigidity of the glass[14]. Figure 3 describes the variation of Debye temperature with tellurium oxide content. The gradual increase of Debye temperature from 290K to 328K indicates the increase in the rigidity of these glasses. The increase in Debye temperature is attributed to the increase in the number of atoms in the chemical formula of the glass and the increase in the mean ultrasonic velocity[14] and indicates the strengthening in the glass structure which due to the creation of bridging oxygens[12].

Micro-hardness expresses the stress required to eliminate the free volume (deformation of the network) of the glass. The increase in the micro-hardness indicates the increase in the rigidity of glass. The softening point is the temperature at which viscous flow changes to plastic flow. It determines the temperature stability of the glass. The higher the higher the value of softening temperature, the greater is the stability of its elastic properties[14]. As seen in Fig. 4 micro-hardness and softening temperature increase with increasing TeO_2 content. These trends of increase imply an increase in the rigidity of the glass system.

Quantitatively analysis of experimentally determined elastic moduli based on bond compression model[7] and Makishima-Mackenzie model[8, 9]. The values of the constants used in this calculation are listed in Table 3[12,17].The calculated value of number of bonds per unit volume n_b, bond compression bulk modulus K_{bc}, the ratio K_{bc}/K_e, the atomic ring size *l*, the average stretching force constant F and average cross link density n_c are given in Table 4. From the Table 4 it can be seen that the value of K_{bc} decrease from 9.82 GPa to 82 GPa with increasing TeO_2 content. The number of bonds per unit volume n_b also decrease from 82.63 x 10^{28} m^3 to 80.32 x10^{28} m^3.
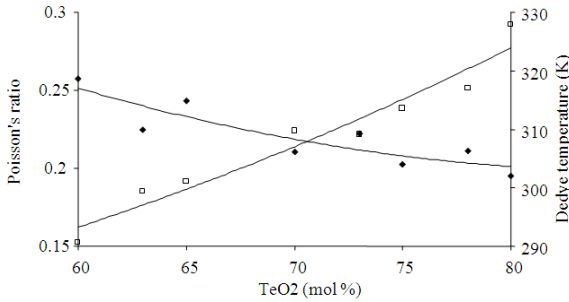
Fig. 3: Poisson's ratio and Debye temperature of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

Fig. 4: Softening temperature and micro hardness of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

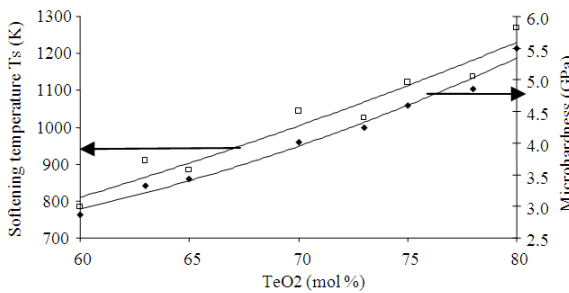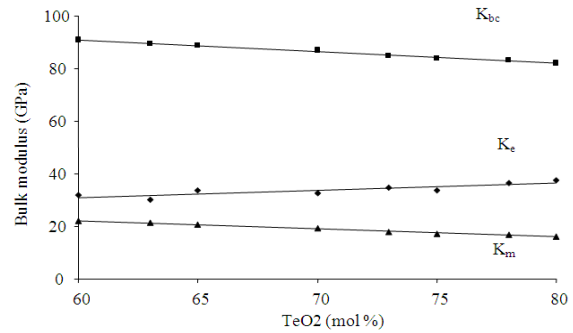Fig. 5: Atomic ring size and packing density of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

Fig. 6: The experimental bulk modulus (K_e), the bond compression bulk modulus (K_{bc}) and Makishima-Mackenzie bulk modulus (K_m) of $(TeO_2)_x (B_2O_3)_{1-x}$ glasses

The decrease number in the number of bonds per unit volume n_b leads to a decrease in the calculated bond compression bulk modulus K_{bc}[15]. The increase in the average cross link density n_c from 1.429 to 1.667 means that the addition of TeO_2 modify B-O-B bridges by increasing the boron atom coordination number from 3 to 4. This leads to the creation of extra bridging oxygen atoms and increase the connectivity of the network[14] Since K_e is less than K_{bc} compression proceeds via a mechanism requiring much less energy than that required for pure compression of network bonds series[14]. This in turns results in a more cross-linked and compact structure and this explains the observed increase in the K_e values. The decrease in the average atomic ring size l from 0.5765 to 0.5198nm with increasing TeO_2 is due to the gradual increase in the average cross link density n_c. Figure 5 shows the dependence of tellurium oxide with the atomic ring size l.

The calculated Poison's ratio of this glass system decreases gradually from 0.2489 to 0.2417. The decrease in Poisson's ratio is attributed to the gradual increase in the average cross-link density with increasing TeO_2 content in the glass. On the other hand, the average stretching force constant F decrease from 3.64 Nm^{-1} to 2.80 Nm^{-1}. This result proved that the increasing of TeO_2 in TeO_2-B_2O_3 glasses results in an increase in the cross-link density due to the transformation of three-fold-coordinated boron into four- fold-coordinated ones[15] and the transformation of TeO_4 trigonal bipyramid to TeO_3 trigonal pyramid which in turns is accompanied by the creation of the bridging oxygens that is the rigidity increases[12].

This in turns raises the resistance of the network to deformation and explains the increase in the observed density and elastic moduli of this glass system.

The ratio K_{bc}/K_e and atomic ring size l are listed in Table 4, it shows that the ratio K_{bc}/K_e decreases from 2.84 to 2.19 with decreasing atomic ring size from 0.5765 to 0.5198. The ratio (K_{bc}/K_e)>1 , indicates a relatively open three-dimensional network structure with ring size l directly proportional to (K_{bc}/K_e)[15]. The values of atomic ring size and ratio K_{bc}/K_e are nearly in the same range as observed by El-Mallawany[5].

The values of computed theoretical elastic moduli according to the Makishima-Mackenzie model[8,9] for all glass samples are given in Table 5. The values of the elastic moduli are lower than those measured experimentally. These lower values may be due to the decrease in the packing density and the increasing molar volume of the glass. Addition of TeO_2 with a packing factor 4.7 x 10^6 m^3 mol^{-1} will reduce the packing density as the packing factor of borax is 17.6 x 10^6 m^3 mol^{-1}. The decreasing trend in the packing density with TeO_2 content can seen in Fig. 5. On the contrary, the dissociation energy of the borate glass modified by TeO_2 increases with the increase in the TeO_2 content, as the dissociation energy

per unit volume of TeO_2 (54×10^6 kJm^{-3}) is higher than the dissociation energy per unit volume of the borax or of B_2O_3 (16.4×10^6 kJm^{-3} and 77.9×10^6 kJm^{-3})[17]. Table 5 shows the increase of dissociation energy with TeO_2 content.

Table 6 summarizes the results of the experimental values of elastic moduli and those of calculated from the bond compression model and Makishima-Mackenzie model for $(TeO_2)_x$ $(B_2O_3)_{1-x}$ glasses. The calculated value of K_{bc} is higher than experimental value K_e as shown in Fig. 6. K_{bc} is always greater than the experimental value, typically by a factor of 3-10[1] and the value of K_m is lower than the experimental value K_e is due to the decreasing packing density and the increasing molar volume[17]. The results show a fairly good agreement between the experimental and the calculated values of Poisson's ratio. The calculated shear, Young's and bulk modulus from the bond compression model and Makishima-Mackenzie model, are different from those experimental by an average correction factor 1.62.

CONCLUSION

The density and the molar volume of this glass system increases with increase in mole percentage of TeO_2 is due to the atomic weight and size of the constituent oxides. The increase in ultrasonic velocity reveals that adding of TeO_2 in the glass system causes an easy movement for the ultrasonic waves inside the network of the glass structure and hence the ultrasonic velocity increases as TeO_2 content increases.

The results of the elastic moduli and those of Poisson's ratio, micro hardness, softening temperature and Debye temperature shows a tightening in the bonding of the glass structure and increase in rigidity of the glass structure.

As the content of TeO_2 increases, the average ring diameter and the ratio K_{bc}/K_e decreases, while number of bonds per unit volume increases. This ascribed to the increase of the molar volume and the increase of the rigidity of the glass structure.

Continuous addition of the TeO_2 according to Makishima-Mackenzie model will slight decrease in the elastic moduli due to the decrease in packing density irrespective of the increase in dissociation energy of the glass.

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