Original Paper

Elastic properties of tellurite glasses

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Elastic properties of binary and ternary tellurite glass systems of the composition (in mol%) $(100-x)\ TeO_2-xNb_2O_5$, and $(100-x)\ TeO_2-0.5x\ (Nb_2O_5 + Li_2O)$ were calculated from the measured densities as well as from longitudinal and shear ultrasonic velocities at room temperature. Ultrasonic velocity measurements were taken at 4 MHz ultrasonic frequency using the pulse echo technique. Elastic moduli, and Debye temperature calculated from experimental data and calculated theoretically using the bond compression model were used to obtain quantitative details about the structure of these glasses. The effect of adding either Nb_2O_5 alone or Nb_2O_5 and Li_2O on the elastic moduli was investigated in terms of the number of network bonds of the glass systems. The average atomic ring size of the network was also calculated and it was found that it depends on the concentration of the modifiers. The obtained results show that these glasses become more stable and compact when modified with Nb_2O_5 or with Nb_2O_5 and Li_2O, which increases the elastic moduli in the two systems.

1. Introduction

The study of the structure of TeO_2-based glasses is of great interest [1 to 3]. The basic structural unit of glasses with high TeO_2 contents is an asymmetrical TeO_4 trigonal bipyramid with a lone pair electron in an equatorial position. As reported by infrared and Raman scattering [4 to 6], this unit forms an infinite three-dimensional network linked together by shared vertices. At glass formation some Te–O bonds are broken creating TeO_3 trigonal pyramids. In binary TeO_2 glasses containing alkaline oxides as network modifiers, the glass structure changes from TeO_4 trigonal bipyramids to TeO_{3+1} polyhedra and then to TeO_3 trigonal pyramids, as the alkaline oxide concentration increases. Further, tellurium oxide based glasses have been considered as promising materials for use in nonlinear optical devices or host materials [7 to 9]. The phase diagrams of the binary and ternary tellurium lithium niobate are known [10]. Ultrasonic studies of velocity in these glasses and, hence, elastic properties are particularly suitable for describing glasses as a function of composition because they are directly related to the interatomic forces and potentials.

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Recently, there have been a number of studies dealing with measurements of ultrasonic velocities and determination of the elastic moduli theoretically. The elastic moduli obtained were reported for pure, binary, ternary, and quaternary tellurite glasses [11 to 16]. The work under report aims to study the elastic moduli of these tellurite glasses by measuring the ultrasonic velocities and to examine the applicability of the theoretical models to this kind of glasses to throw more light on the previous work reported on tellurite glasses [10 to 17].

2. Experimental work

Glass samples were prepared in the form, $(100 - x)\ TeO_2 - x\ Nb_2O_5$ for the binary system where $x = 4$, 7.5, 10, 12.5, 15, 17.5, 20 and 22.5 mol% and $(100 - x)\ TeO_2-0.5x\ (Nb_2O_5 + Li_2O)$ for the ternary system where $x = 10$, 20, 25, and 30 mol%, by mixing together specific weights of tellurite oxide, niobium oxide, and lithium oxide (Aldrish Chemicals 99.99 %) in a ceramic crucible. In order to reduce tendency to volatilization, the mixture was kept at 400 °C for 30 min. The crucible was then transferred to a muffle furnace which was con-

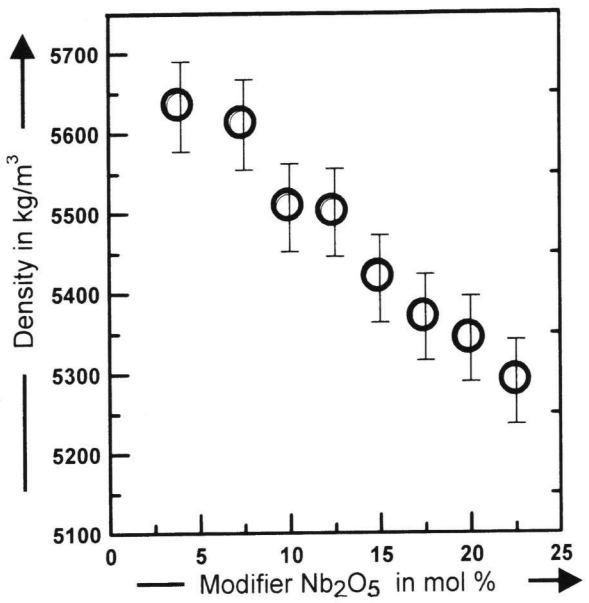

Figure 1. Density of the binary glass system versus the molar percentage of the modifier Nb_2O_5.

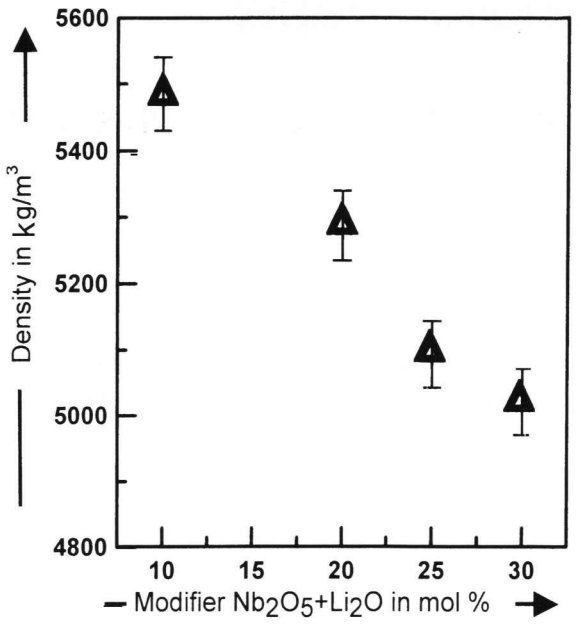

Figure 2. Density of the ternary glass system versus the molar percentage of the modifiers Nb_2O_5 and Li_2O.

trolled at a temperature in the range from 800 to 1000 °C depending upon the composition of each sample for one hour. The melt was then cast at room temperature in a cuboidal-shaped split-mold made of mild steel which had been preheated at 380 °C and then annealed at 350 °C for one hour. Pieces of about 1 cm^3 were lapped and polished on the sides to be suitable for use in ultrasonic velocity measurements. Nonparallelism of the side faces was less than 0.01 mm.

X-ray diffraction examination was carried out using Philips PW/1710 with Ni-filtered, CuK_α radiation (λ = 0.1542 nm) powered at 40 kV and 30 mA. The densities ϱ (in kg/m^3) of the glass samples were measured accurately to the third decimal (±50 kg/m^3) by the displacement method using toluene as an immersion liquid. The ultrasonic velocities v (in m/s) were obtained using the pulse echo technique, by measuring the time elapsed between the initiation and the receipt of the pulse appearing on the screen of the flaw detector (USM3-Krautkrämer) by standard electronic circuit (Philips PM 3055 Oscilloscope). The velocity was therefore obtained by dividing the round trip distance by the elapsed time. Random errors in the measurements were ± 60 m/s for longitudinal velocity v_L (in m/s) and ± 80 m/s for shear velocity v_s (in m/s).

3. Results and discussion

X-ray diffraction patterns for all samples of the binary and ternary glass systems show no discrete or continuous sharp peaks but the characteristic halo of the amorphous solids.

Figure 1 shows the variation of the density for the binary system with the molar percentage of the modifier. The figure shows a linear decrease in density with increasing the modifier (Nb_2O_5) concentration. The observed decrease in density values is expected taking into consideration the additive density rule as reported by Weast [18]. The density of Nb_2O_5 is 4470 kg/m^3, which is much lower than that of TeO_2 (5670 kg/m^3). As suggested by Komatsu et al. [19], the structural unit of TeO_2-based glasses changes gradually from asymmetrical TeO_4 trigonal bipyramid to TeO_3 trigonal pyramid accompanied with increasing amounts of other components such as alkali earth elements. The structural unit of the niobium oxide is NbO_6 octahedra with nonbridging oxygens. The three basic units, TeO_4 trigonal bipyramid, TeO_3 trigonal pyramid and NbO_6 octahedra, are linked randomly by sharing corners. In general, increasing of nonbridging oxygens decreases the density of the glass with increasing the modifier content.

Figure 2 also depicts the variation of the density for the ternary glass system with increasing the modifier molar percentage (Nb_2O_5 + Li_2O). The addition of low-density modifier (Nb_2O_5 of density value 4470 kg/m^3, and Li_2O of density value of 2013 kg/m^3) at the expense of high-density oxide TeO_2 (5670 kg/m^3) decreases the density of the glass. Moreover, the addition of Li_2O to tellurite glass at the expense of Nb_2O_5 or TeO_2 will decrease the densities since the radius of the Li^+ ion (0.06 nm) is much smaller than that of the Te^{+4} (0.22 nm) or Nb^{+6} ion (0.07 nm).

The variation of longitudinal and shear velocities for the binary glass system with modifier molar percentage is shown in figure 3. Both velocities increase with increasing modifier concentration. This can be attributed mainly to the decrease in density and to the increase in elastic moduli of the glass.

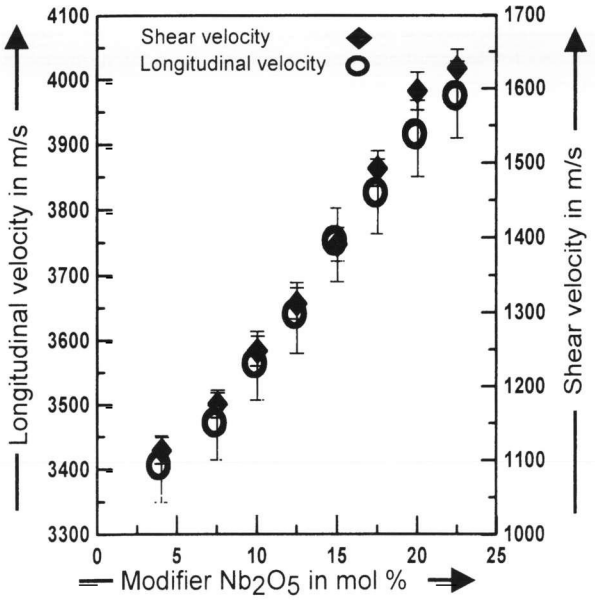

Figure 3. Velocity of the binary glass system versus the molar percentage of the modifier Nb_2O_5.

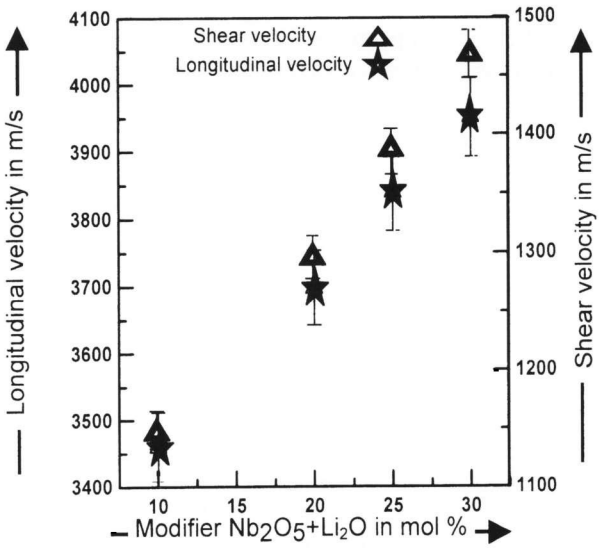

Figure 4. Velocity of the ternary glass system versus the molar percentage of the modifiers Nb_2O_5 and Li_2O.

For the ternary glass system, both longitudinal and shear velocities increase with increasing the molar percentage of the modifier concentration as shown in figure 4. The same explanation mentioned earlier is also true for the glass system.

For isotropic systems, such as glass, the following relations hold:

$$L = \varrho\, v_L^2 , \tag{1}$$

$$G = \varrho\, v_s^2 , \tag{2}$$

$$\mu = 1/2 - G/(2(L - G)) , \tag{3}$$

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

$$K = L - \frac{4}{3} G , \tag{5}$$

$$H = (1 - 2\,\mu) E/(6(1 + \mu)) \tag{6}$$

where L is the longitudinal elastic modulus, G is the shear modulus, K is the bulk modulus, E is Young's modulus, μ is Poisson's ratio, and H is the micro-hardness of the glass. Values of the elastic moduli, Poisson's ratio and micro-hardness for the glasses studied are tabulated in table 1. It can be seen from this table that increasing the modifier content in the binary and ternary glass systems causes an increase in the longitudinal, shear Young's and bulk modulus, as well as in the micro-hardness while Poisson's ratio decreases.

In the binary glass system, the replacement of tellurium oxide by niobium oxide means that a tetrahedral TeO_4 structural unit is replaced by an octahedral NbO_6 structural unit. The coordination number of niobium oxide (six) is larger than that of tellurium oxide (four), i.e. niobium oxide has a higher number of network bonds. This leads to a regular formation of a strengthened structure, which in turn minimizes the spaces in the network. As a direct result, the rigidity of the glass and consequently the elastic moduli increase.

In the ternary glass system, the observed increase in elastic moduli is mainly due to the replacement of tellurium oxide by both niobium and lithium oxide. This means that a tetrahedral structural unit (TeO_4) is replaced by an octahedral structural unit (NbO_6) and tetrahedral (LiO_4). The higher coordination number of niobium and lithium oxide creates more network bonds. Thus, the spaces in the network are minimized, which leads to an increase in the rigidity of the glass.

The micro-hardness expresses the stress required to eliminate the free volume (deformation of the network) of the glass. The increase in micro-hardness with increasing modifier content indicates the increase in glass rigidity.

The decrease in Poisson's ratio with increasing the modifier content in the two glass systems is attributed to the increase in cross-link density of the glass, which affects the lateral strain as proposed by Higazy and Bridge [20].

Debye temperature θ_D represents the temperature at which nearly all modes of vibrations in a solid are excited and it gives an indication of the rigidity of the glass. The Debye temperature is given by Mukherjee et al. [21] as:

$$\theta_D = (h/k)\, \bar{v} \left[\frac{9Z\, N_A}{4\pi\, V}\right]^{1/3} \tag{7}$$

$$\bar{v} = \left[\frac{1}{v_L^3} + \frac{2}{v_s^3}\right]^{-1/3} \tag{8}$$

Table 1. Glass composition (in mol%) of binary $(100 - x)TeO_2-Nb_2O_5$, and ternary glass formula $(100 - x)TeO_2 - 0.5x\ (Nb_2O_5 + Li_2O)$; and respective calculated values (with standard deviation in %) for longitudinal elastic modulus L, shear modulus G, Young's modulus Y, bulk modulus K, micro-hardness H, and Poisson's ratio μ

glass composition			physical properties					
TeO_2	Nb_2O_5	Li_2O	L in GPa (±5 %)	G in GPa (±5 %)	Y in GPa (±4.5 %)	K in GPa (±3 %)	H in GPa (±4 %)	μ (±3 %)
addition of Nb_2O_5								
100.0	0	0	59.12	20.58	50.75	31.68	3.663	0.233
96.0	4.0	0	65.11	6.98	20.10	55.81	0.279	0.440
92.5	7.5	0	67.40	7.76	22.27	57.05	0.337	0.435
90.0	10.0	0	69.78	8.58	24.53	58.35	0.401	0.430
87.5	12.5	0	72.64	9.47	26.98	60.01	0.473	0.425
85.0	15.0	0	76.02	10.48	29.77	62.04	0.559	0.420
82.5	17.5	0	78.37	11.95	33.71	62.43	0.717	0.410
80.0	20.0	0	81.51	13.59	38.04	63.39	0.906	0.400
77.5	22.5	0	83.33	14.00	39.18	64.66	0.943	0.399
addition of Nb_2O_5 and Li_2O								
90.0	5.0	5.0	65.67	7.24	20.83	56.02	0.299	0.438
80.0	10.0	10.0	72.29	8.88	25.39	60.45	0.414	0.430
75.0	12.5	12.5	75.11	9.77	27.85	62.09	0.487	0.425
70.0	15.0	15.0	78.39	10.21	30.73	63.96	0.570	0.420

Table 2. Glass composition (in mol%) of binary $(100 - x)TeO_2-Nb_2O_5$, and ternary glass formula $(100 - x)TeO_2-0.5x\ (Nb_2O_5 + Li_2O)$; and respective calculated values (with standard deviation in %) for number of atoms per unit volume Z (x 10^{28}), Debye temperature θ_D, and softening temperature T_s

glass composition			physical properties		
TeO_2	Nb_2O_5	Li_2O	Z in m^{-3}	θ_D in K (±3.5 %)	T_s in K (±3 %)
addition of Nb_2O_5					
96.0	4.0	–	4.69	152.1	249.48
92.5	7.5	–	4.77	161.5	272.74
90.0	10.0	–	4.74	171.0	302.88
87.5	12.5	–	4.80	180.4	330.22
85.0	15.0	–	4.79	191.0	366.42
82.5	17.5	–	4.81	205.0	416.37
80.0	20.0	–	4.83	219.2	470.85
77.5	22.5	–	4.85	223.6	483.80
addition of Nb_2O_5 and Li_2O					
90.0	5.0	5.0	4.78	158.0	253.86
80.0	10.0	10.0	4.93	179.8	301.70
75.0	12.5	12.5	4.91	191.8	333.52
70.0	15.0	15.0	5.00	204.3	362.91

where h is Planck's constant, k is Boltzmann's constant, $\bar{v}$ is the mean ultrasonic velocity, N_A is Avogadro's number, V is the molar volume, and Z is the number of atoms. Table 2 gives the Debye temperature values for the glass samples studied. The observed increase in Debye temperature with increasing modifier content is attributed to a change in the number of atoms per unit volume and therefore, the increase in the number of bonds between atoms and rareness of nonbridging oxygens.

Softening temperature for the glasses studied are given in table 2 as calculated from an equation given by Anderson [22] as;

$$T_s = (v_s/C)^2 \frac{M}{Z} \tag{9}$$

where the constant C equals 507.4 m/(s $K^{1/2}$), and M is the molecular weight of the oxide. It can be seen from table 2 that the softening temperature increases as the modifier content increases in both binary and ternary glass systems studied.

A trial to interpret the experimental results obtained for elastic behavior based on the bond compression model [23] is attempted. According to this model, the elastic moduli depend on the number of network bonds per unit volume. In a quantitative way, the theoretically calculated bulk modulus K_{bc} is given by Bridge et al. [23] as:

$$K_{bc} = \left(\sum_i x_i n_i r_i^2 f_i\right) \frac{N_A \varrho}{9M} \tag{10}$$

where x is the mole fraction of component oxide, n is the coordination number of cation, r is the bond length, f is the first order stretching force constant given by $f = 1.7/r^3$. The average ring size $\bar{l}$, defined as the ring perimeter (number of bonds times bond length divided by π), is given by the equation:

$$\bar{l} = \left[0.0106 \frac{F_b}{K_e}\right]^{0.26} \tag{11}$$

where K_e is the experimental bulk modulus and F_b is the bond bending force constant of the glass which is

proportional to the average bond stretching force constant $\bar{F}$ and given by

$$\bar{F} = \frac{\sum_i x\, n f}{\sum_i x\, n} . \tag{12}$$

The theoretical Poisson's ratio can be calculated from the equation given by [20] as:

$$\mu_{th} = 0.28\ (\bar{n}_c)^{-1/4} \tag{13}$$

where $\bar{n}_c$ is the average crosslink density per unit formula and is given as:

$$\bar{n}_c = (1/\eta) \sum_i x_i\, (n_c)_i\, (N_c)_i \tag{14}$$

where n_c is the number of crosslinks per unit cation which equals the number of bonds less 2, N_c is the number of cations per glass formula unit and η is the total number of cations per glass formula unit given as:

$$\eta = \sum_i x_i (N_c)_i . \tag{15}$$

The theoretically calculated values of the bulk modulus K_{bc}, number of bonds per unit volume N_B, atomic ring size l, average stretching force constant $\bar{F}$, average crosslink density $\bar{n}_c$, theoretically calculated Poisson's ratio μ_{th}, and the ratio of the calculated to experimental bulk modulus K_{bc}/K_e are given in table 3.

It is quite clear from table 3 that the calculated values of K_{bc} decrease with increasing modifier content in both binary and ternary glass systems. The theoretically calculated bulk modulus K_{bc} depends on the mole fraction of component oxides, the density of the glass, and the molecular weight of the glass. As the concentration of the modifier increases, the density decreases and also the number of network bonds per unit volume, and this leads to the decrease in K_{bc} values with decreasing the ring diameter in both glass systems as shown in figures 5 and 6.

The dependence of the ratio (K_{bc}/K_e) on the modifier content in the binary and ternary glass systems are shown in figures 7 and 8. Generally, this ratio is a measure of the extent to which bond bending is governed by the configuration of the network bonds, i.e. this ratio is assumed to be directly proportional to the ring diameter. For the binary glass system, the insertion of niobium oxide with coordination number six at the expense of tellurium oxide with coordination number four increases the number of bonds per unit volume and the average force constant. This will increase the connectivity of the structure and both the atomic ring diameter and the ratio K_{bc}/K_e decrease. The same explanation is applicable also to the ternary glass system.

The ratio K_{bc}/K_e was found to be in the form:

- $K_{bc}/K_e \equiv 0.047 \cdot V - 0.19$ with a correlation factor 99 %; for the binary glass system.

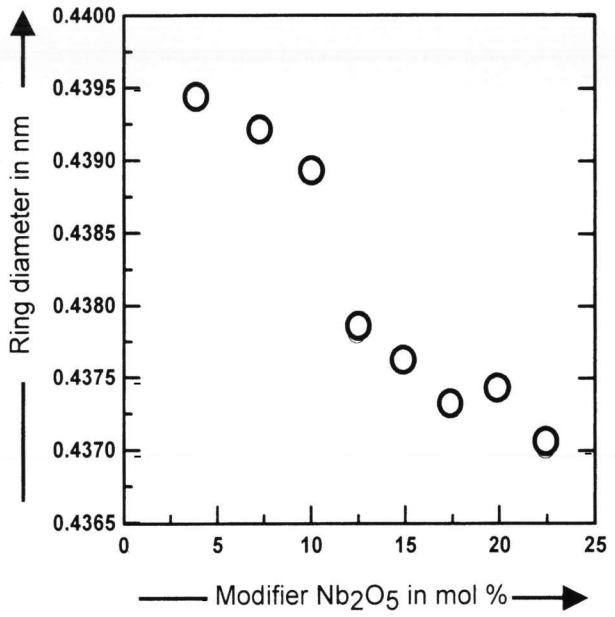

Figure 5. Ring diameter of the binary glass system versus the molar percentage of the modifier Nb_2O_5.

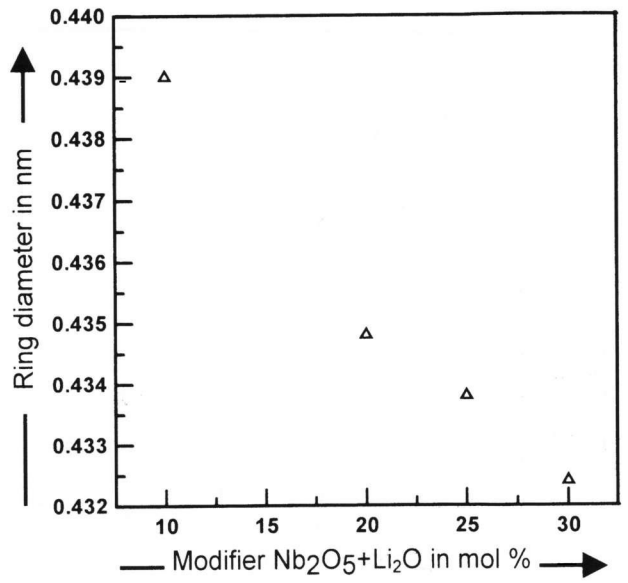

Figure 6. Ring diameter of the ternary glass system versus the molar percentage of the modifiers Nb_2O_5 and Li_2O.

- $K_{bc}/K_e = 0.073 \cdot V - 0.87$ with a correlation factor 77 %; for the ternary glass system.

The theoretically calculated bulk modulus is found to be considerably larger than that measured experimentally [23]. This may be due to an increase in the average force constant. Compression proceeds via a mechanism requiring much less energy than is needed for pure compression of network bonds.

As observed from table 2, the theoretically calculated Poisson's ratio decreases with increasing the modifier mole content. This decrease is mainly due to the increase in cross-link density. The addition of the modifier at the

Table 3. Glass composition (in mol%) of binary (100 − x)TeO_2−Nb_2O_5, and ternary glass formula (100 − x)TeO_2−0.5x (Nb_2O_5 + Li_2O); and respective calculated values (with standard deviation in %) for bond compression bulk modulus K_{bc}, number of bonds per unit volume N_B (x 10^{31}), ring diameter l, ratio of calculated to experimental bulk modulus K_{bc}/K_e, average force constant $\bar{F}$, and calculated Poisson's ratio μ_{th}

glass composition			physical properties					
TeO_2	Nb_2O_5	Li_2O	K_{bc} in GPa (±2 %)	N_B in m^3 (±2 %)	l in nm (±3 %)	K_{bc}/K_e (±4 %)	$\bar{F}$ in N/m	μ_{th}
addition of Nb_2O_5								
100.0	–	–	73.10	8.65	0.5036	2.31	215.7	0.236
96.0	4.0	–	81.00	8.45	0.4386	1.45	223.37	0.231
92.5	7.5	–	80.91	8.37	0.4393	1.42	229.83	0.228
90.0	10.0	–	79.56	8.18	0.4389	1.36	234.31	0.226
87.5	12.5	–	79.63	8.15	0.4378	1.33	238.69	0.224
85.0	15.0	–	78.58	7.99	0.4361	1.27	242.97	0.222
82.5	17.5	–	78.03	7.90	0.4373	1.25	247.15	0.221
80.0	20.0	–	77.64	7.82	0.4374	1.23	251.24	0.219
77.5	22.5	–	77.12	7.73	0.4370	1.19	255.23	0.218
addition of Nb_2O_5 and Li_2O								
90.0	5.0	5.0	82.14	8.55	0.4390	1.47	225.08	0.230
80.0	10.0	10.0	82.65	8.51	0.4348	1.37	234.01	0.227
75.0	12.5	12.5	81.31	8.32	0.4338	1.31	238.32	0.225
70.0	15.0	15.0	81.85	8.37	0.4324	1.28	242.53	0.224

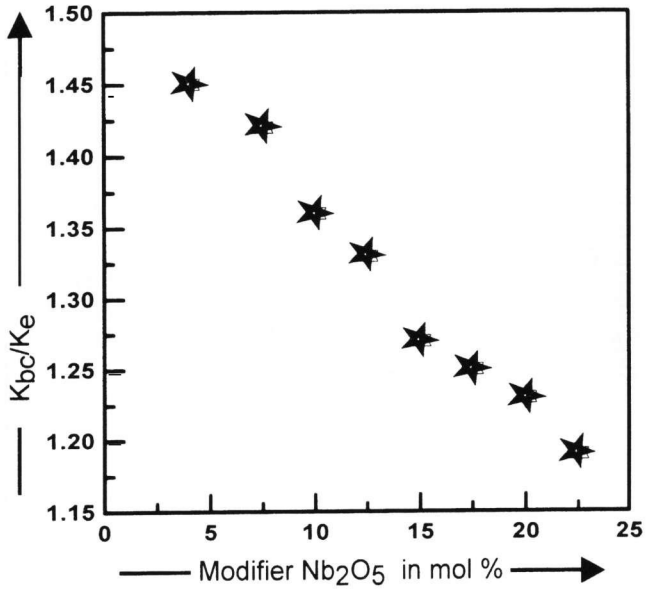

Figure 7. Ratio K_{bc}/K_e of the binary glass system versus the molar percentage of the modifier Nb_2O_5.

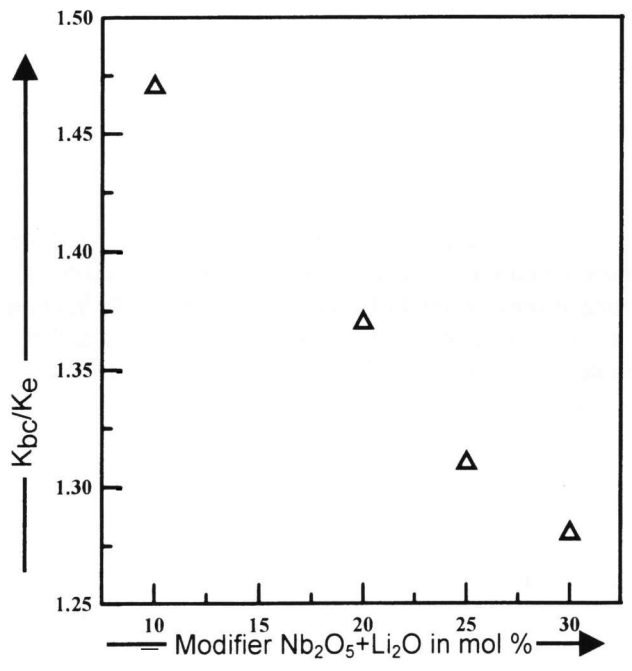

Figure 8. Ratio K_{bc}/K_e of the ternary glass system versus the molar percentage of the modifiers Nb_2O_5 and Li_2O.

expense of TeO_2 will increase the cross-link density, which means an increase in the structural linkage.

4. Conclusions

a) In the binary system, the density decreases with the increase in molar volume and this was attributed to the substitution of TeO_2 (having a density 5670 kg/m^3) by Nb_2O_5 (having a density 4470 kg/m^3). While for the ternary system, the density decreases with the increase in the molar volume.

b) The longitudinal and transverse ultrasonic velocities increase for binary and ternary glass systems. This was attributed to the change in the rigidity of the glass and the variation of coordination number of TeO_2, Nb_2O_5 and Li_2O.

c) Debye and softening temperatures behave in a similar way to the ultrasonic velocities.

d) The elastic moduli increase in the binary and ternary glass systems with increasing modifier content. This was attributed to the increase in cross-link density and the

number of network bonds per glass formula. This in turn decreases the ring diameter and also the ratio K_{bc}/K_e.

e) Poisson's ratio, as calculated experimentally and theoretically, decreases as a result of strengthening in the structure of the binary and ternary glass systems.

5. List of symbols

C	constant = 507.4 m/(s $K^{1/2}$)
E	experimental Young's modulus in GPa
f	first order stretching force constant in N/m
$\bar{F}$	average bond stretching force constant
F_b	bond bending force constant
G	experimental shear modulus in GPa
h	Planck's constant = $6.6 \cdot 10^{-34}$ J s
H	micro-hardness in GPa
k	Boltzmann's constant = $1.38 \cdot 10^{-23}$ J K^{-1}
K	bulk modulus in GPa
K_e	experimental bulk modulus in GPa
K_{bc}	calculated bulk modulus in GPa
L	experimental longitudinal modulus in GPa
l	ring diameter in nm
$\bar{l}$	average ring size
M	molecular weight of the glass
n	coordination number
N_A	Avogadro's number = $6.022 \cdot 10^{23}$ mol^{-1}
N_B	number of bonds per unit volume in m^{-3}
n_c	number of crosslinks per unit cation
$\bar{n}_c$	average crosslink density per unit formula
N_c	number of cations per glass formula unit
r	cation-anion bond length in nm
T_s	softening temperature in K
V	molar volume in m^3
v	ultrasonic velocity in m/s
$\bar{v}$	mean ultrasonic velocity in m/s
v_L	longitudinal ultrasonic velocity in m/s
v_s	shear ultrasonic velocity in m/s
x	mol fraction of component oxide
Z	number of atoms per unit volume in m^{-3}
η	total number of cations per glass formula unit
θ_D	Debye temperature in K
μ	Poisson's ratio
μ_{th}	calculated Poisson's ratio
ϱ	density of glass in kg/m^3

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