

Structure and Ultrasonic Properties of Vanadium Tellurite Glasses Containing Copper Oxide

Nadia S. ABD EL-AAL, Hesham A. AFIFI

National Institute for Standards

El-Haram, Giza, P.O. Box: 136 Giza, CN. 1221, Egypt

e-mail: n_nadia_99@yahoo.com, hmafifi@hotmail.com

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The elastic properties of vanadium tellurite glasses, $65\text{TeO}_2-(35-x)\text{V}_2\text{O}_5-x\text{CuO}$, with different compositions of Copper ($x = 7.5$ to 17.5 mol% in steps of 2.5 mol%) have been studied at room temperature (300 K). The ultrasonic velocity measurements have been made, using a transducer having resonating frequency of 4 MHz (both longitudinal and shear). The density, molar volume, and ultrasonic velocities show interesting features, which are used to explore the structural changes in the network. Elastic moduli, Poisson ratio, crosslink density, Microhardnes, and Debye temperature of the glasses have been determined using the experimental data. The composition dependence of the elastic properties explores useful information about the physical properties of the vanadium tellurite glasses doped with Copper. Quantitative analysis has been carried out in order to obtain more information about the structure of the glass under the study, based on bond compression model and the Makishima & Mackenzie model. The observed results through ultrasonic non-destructive evaluation, investigate the structural changes and mechanical properties of the glass.

Keywords: ultrasonic velocity, elastic constant, crosslink density, Debye temperature, and theoretical models.

1. Introduction

Tellurite glasses have extensively been investigated in the past several years due to their advantages such as high refractive index, high third-order nonlinear susceptibility, good infrared transmittance and low melting point [1, 2]. In addition, tellurite glasses could be used in the production of optical fibers and planar waveguides; these special optical properties encourage in identifying them as important materials for potential applications in high-performance optics and laser technology and optical communication network. It has been known that pure TeO_2 is only a conditional glass, former requiring fast quenching to form

glass. The conventional methods can not form a glass in a straightforward manner without adding other elements to the pure TeO_2 [3]. Several studies of tellurite glasses modified by the conditional glass formers WO_3 , V_2O_5 and GeO_2 have been reported [4–6]. Tellurite glasses containing a large amount of V_2O_5 -oxide are a relatively new class of vitreous materials. These glasses have relatively high electrical conductivity, as compared with vanadium phosphate glasses or other glass containing metal oxides with the same amount of charge carriers [7]. Ultrasonic non-destructive characterization of materials is a versatile tool for inspection of their microstructure and their mechanical properties [8]. This is possible because of close association of the ultrasonic waves with the elastic and inelastic properties of materials. Recent ultrasonic studies [9–11] on composition dependence of ultrasonic velocities, elastic moduli, Debye's temperature and Poisson's ratio showed that the mechanical structure of TeO_2 - V_2O_5 glasses depends on the percentage of V_2O_5 ; when the V_2O_5 concentration is below 20 mol%, the three-dimensional tellurite network is partially broken by the formation of TeO_3 trigonal pyramids which in turn reduce the glass rigidity; when V_2O_5 concentration is above 20 mol%, the glass structure changes from the continuous tellurite network to the continuous vanadate network.

Elastic properties are very informative about the structure of solids and they are directly related to the inter-atomic potentials. Very recently, the elastic properties of Gd^{3+} doped telluro-vanadate glasses [12] and new ternary tellurite glasses in the form $50(\text{TeO}_2)$ - $50-x(\text{V}_2\text{O}_5)$ - $x(\text{TiO}_2)$ have been studied using the pulse echo technique [13]. The addition of transition metal oxides to glasses, in general, permits the possibility for the glasses to exhibit semi-conducting behavior. This electronic behavior as well as the optical magnetic and structural properties for glasses, depends upon the relative ratio of the different valence states of the transition metal ions present [14–16]. Very little attention has been given in published literature to the tellurite glasses containing both transition metal and a significant content of rare-earth ions.

In this article, the effect of the third component CuO with different percentage in elastic moduli of tellurite-vandate glasses will be discussed. Although the conductivity and optical absorption data [17–20] on some binary and ternary copper tellurite glasses are available, no ultrasonic measurements on copper tellurite glass have so far been reported except PAUL *et al.* [20] who observed that the ultrasonic properties of $(\text{CuO})_x$ - $(\text{TeO}_2)_{1-x}$ glass system changed significantly with temperature if the CuO content is below 20 mol%. However, for the sample having more than 20 mol% CuO contents no such changes were observed. These changes in the behavior are attributed to the structural changes in coordination polyhedron, with variation in composition of the glasses.

The aim of the present study is to verify whether some of conclusions drawn in the previous studies on the behaviour of Cu ions in tellurite glasses also hold true in a simple binary system i.e. CuO - TeO_2 [18], and to see how the behaviour changes with the addition of another network formers V_2O_5 . Furthermore, an

attempt has been made to explore the structural changes, structural stability and physical properties of tellurite-vanadium glasses with different CuO contents, using ultrasonic measurements.

2. Experimental procedures

2.1. Materials

The tellurovanadate glasses containing different copper contents as 65TeO_2 - $(35-x)\text{V}_2\text{O}_5$ - $x\text{CuO}$ where $x = 0, 7.5, 10, 12.5, 15, 17.5$ mol% for copper content, have been prepared by conventional quenching method. The reagent oxides used in this study were analytical grade chemicals. The required amount in wt% of chemicals in powder form was weighted using a digital balance having an accuracy of ± 0.1 mg. The powders mixture were then put in a platinum crucible and heated in melting furnace. In order to reduce any tendency of volatilization, the mixture was kept at 400°C for 1h and melted for about 2 h at 1000°C . The melt was stirred several times during preparation to achieve high degree of homogeneity. The melt was then cast into copper block followed by annealing at 350°C for 2 h. The obtained glass samples were in the form of a cube (1 cm^3). The samples were polished using fine sand-paper and mirrored to obtain an optically flat and parallel faces which, suitable for ultrasonic measurements. The amorphous nature of the samples was checked by X-ray diffraction XRD analysis.

2.2. Density measurements and molar volume

The density of all glass samples was determined by a pycnometric technique with toluene as a buoyant liquid using the relation $\rho = [W_a/W_a - W_b]\rho_b$, where W_a is the glass sample weight in air, W_b – the glass weight in buoyant and ρ_b – the density of the buoyant. The overall accuracy in density measurement is $\pm 0.05\text{ kg m}^{-3}$.

The molar volume V_M could be calculated according to the following relation:

$$V_M = M/\rho_{\text{glass}}, \quad (1)$$

where M and ρ_{glass} are the molecular weight and density of the glass sample, respectively.

2.3. Ultrasonic detection

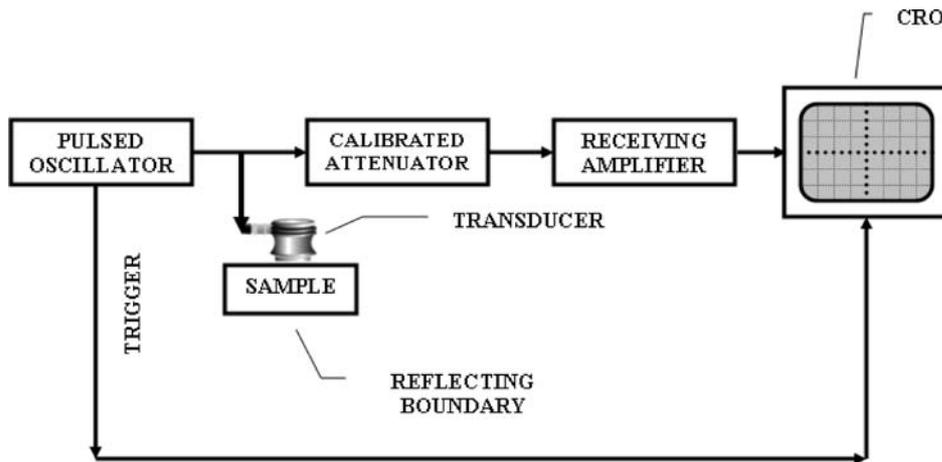
2.3.1. Ultrasonic principle, system and method of detection

Ultrasound is a kind of mechanical wave with frequency higher than 20 kHz. Ultrasonic waves can be generated from periodically vibrating piezoelectric transducer propagating in the viscoelastic polymer. There are four types of ultrasonic waves, namely longitudinal wave, shear wave, Rayleigh wave and Lamb wave;

since the velocity and attenuation of longitudinal wave in certain materials are two useful physical parameters to explore the materials properties. Longitudinal wave is often preferentially utilized to characterize such properties.

The ultrasonic measurements in this study were performed using an ultrasonic flow detector (USIP20), an oscilloscope (54615B), longitudinal transducer and shear transducer (both 4 MHz in frequency). As shown in Fig. 1a, the incident wave is generated from the transducer contacting directly the specimen. This is achieved by putting coupling agent of glycerin. Thus, the incident waves are transmitted in a sample of thickness X and reflected back and forth at the two surfaces. When the reflected wave reaches the upper surface and received by the transducer, an echo signal containing several oscillations will be gained. A series of

a)



b)

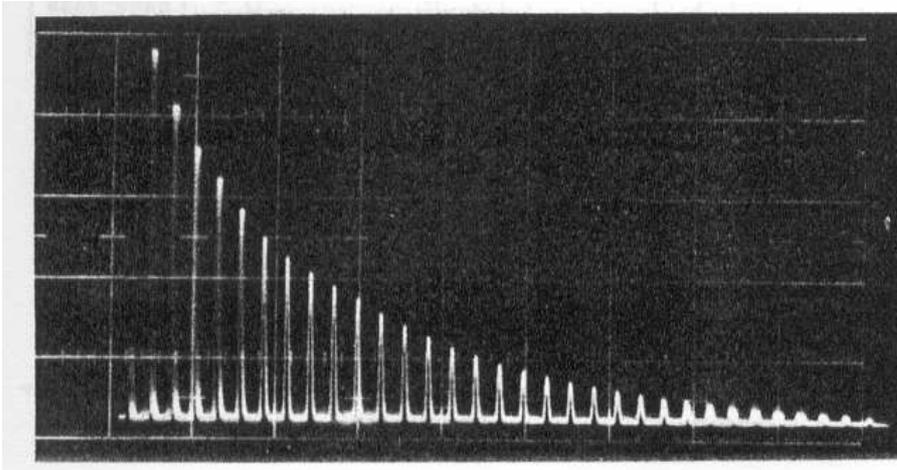


Fig. 1. Basic pulse-echo system (a), typical pulse-echo decay pattern (b).

echo signals can be obtained as ultrasound waves are reflected between the two surfaces. However, the amplitudes of the echo signals are gradually decreased with time due to ultrasound attenuation, as shown in Fig. 1b. The first two echo signals, of amplitudes (A_1 , A_2) and the corresponding times (t_1 , t_2), can be read and chosen to calculate the ultrasonic velocity v .

$$v = \frac{2X}{t_2 - t_1}. \quad (2)$$

The measurements of ultrasound velocities (longitudinal, shear) were repeated three times to check the reproducibility of the data. The estimated accuracies are about ± 9 m/s and 11 m/s, respectively.

3. Results and discussion

3.1. X-ray, density and molar volume results

X-ray diffraction patterns of the studied glass system show the absence of any discrete or continuous sharp crystalline peaks, but the existence of the characteristic halo of the amorphous solids. The experimental values of the density (ρ) and molar volume V_M of the ternary glass system: 65TeO₂-(35-x)V₂O₅-xCuO are given in Table 1.

Table 1. Density (ρ), molar volume (V_M), longitudinal ultrasonic velocity (V_l), shear ultrasonic velocity (V_s) and oxygen molar volume (V_o) of TeO₂-V₂O₅-CuO glasses.

Glass composition mol%			ρ [kg m ⁻³]	V_M [m ³ /mol]	V_l [m/s]	V_s [m/s]	V_o [cm ³]
TeO ₂	V ₅ O ₂	CuO					
65	35	0	3998	0.0387	3991	2361	12.68
65	27.5	7.5	4169	0.0383	3785	2212	13.93
65	25	10	4235	0.0371	3581	2075	14
65	22.5	12.5	4327	0.0353	3515	2036	14.01
65	20	15	4398	0.0346	3467	2000	14.11
65	17.5	17.5	4458	0.0335	3419	1956	14.27

The CuO dependence of density increases linearly from 3998 to 4458 Kg m⁻³. On the other hand, the molar volume decreases from 0.0387 to 0.0335 m⁻³/mol as the Cu content is varied from 7.5 to 17.5 mol%.

The density of a glass is an important property capable of evaluating the compactness and short-range structure of the glass. Addition of transition metal to the tellurite network causes some type of rearrangement of the atom as found by [21, 22]. The density of V₂O₅ and copper oxide are 3350 and 6310 Kg m⁻³ respectively. The replacement of vanadium atoms by Cu oxide with high density, explaining the observed gradual increase in the density, and hence the decrease in molar volume with the increase of Cu modifier content, is illustrated in Table 1.

SHELBY [23] reported that if the ionic radius of the modifier ions is smaller than the interstices of the network structure, their attraction to the oxygen ions can lead to decrease in the size of the interstices and consequently decreases the molar volume, since the ionic radius of Cu (0.074 Å) is smaller than that of V ions (0.880 Å). Moreover, the decrease in molar volume may be also due to the increase in the number of nonbridging Oxygens as reported earlier by KHALIFA *et al.* [24].

3.2. Ultrasonic velocities results

The ultrasonic velocities (longitudinal and shear) results for these glasses are displayed in Fig. 2 and listed in Table 1, as a function of CuO mol%. It shows a gradual decrease in ultrasonic longitudinal and shear velocities from 3991 and 2361 m/s to 3419 and 1956 m/s for longitudinal and shear velocities respectively, when CuO content increases from 0 to 17.5 mol %.

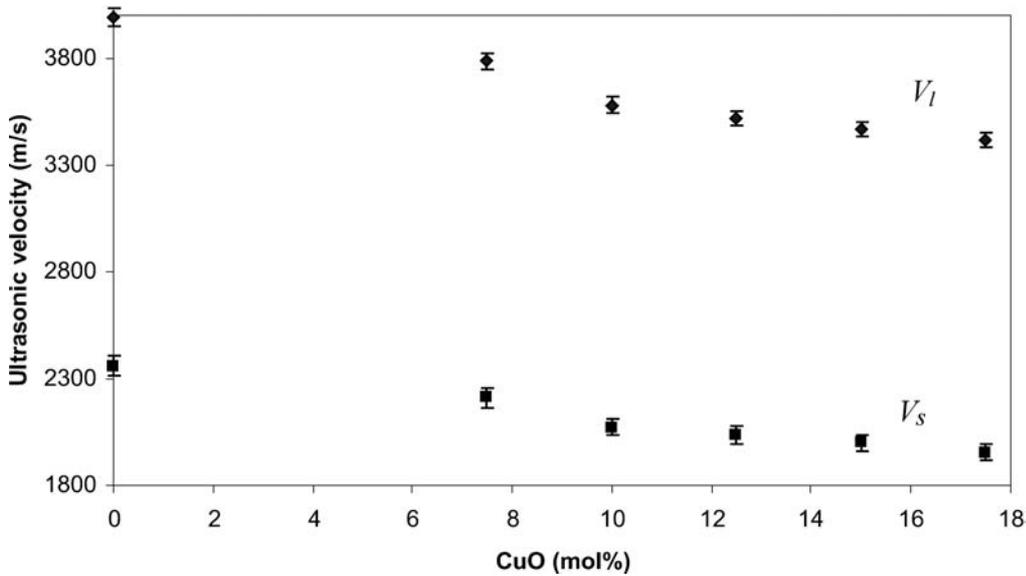


Fig. 2. Variation of longitudinal ultrasonic velocity (V_l) and shear ultrasonic velocity (V_s) with the mole percentage of TeO_2 - V_2O_5 -CuO glasses.

It has been reported [25] on the structural studies of CuO- TeO_2 that, for larger concentration of CuO, i.e. more than ten percent, the CuO ions cluster and a sort of demixing occurs. In these Cu-rich regions, the process of demixing implies emerging of the CuO ions from the strained Te-O network and forming almost pure amorphous CuO regions. We are undertaking this result to confirm our data indicating that the mixed tellurite-vanadium network is relatively more open, as reported before by studying DTA and IR absorption spectra of TeO_2 - V_2O_5 glasses in the range of 0–50 mol% V_2O_5 cf. [26]. However, more importantly the CuO-rich regions in vanadium-tellurite are resulting in more open structures

with higher oxygen molar volume, as shown in Table 1. This open structure decreases the resistance of the network to deformation which, in turn, results in loosening the structure, as presented by the observed decrease in the ultrasonic velocity values.

The observed decrease of the ultrasonic velocity values support the fact that addition of CuO into TeO₂-V₂O₅ glass system creates non-bridging oxygen atoms due to the transformation of TeO₄ trigonal bi-pyramids to TeO₃ trigonal pyramids [27, 28]. This causes loose packing density in the glass structure, as we will see in the theoretical interpretation of the data, and also a reduction in the vibration of the lattice, as observed earlier by BRIDGE *et al.* [29].

3.3. Elastic constants, Poisson's ratio, cross-link density and micro-hardness results

In an amorphous solid (such as glass), the elastic strain produced by a small stress can be described by the longitudinal modulus (L) and shear modulus (G) given as

$$L = \rho v_l^2, \quad (3)$$

$$G = \rho v_s^2,$$

where v_l and v_s are the longitudinal and transverse sound velocities and ρ is the density of glass samples.

The sound velocities also allow the determination of Young's modulus (E), the bulk modulus (K), the micro-hardness (H) and Poisson's ratio (σ), using the following equations [30]:

$$\begin{aligned} K &= L - (4/3)G, \\ E &= 2G(1 + \sigma), \\ \sigma &= (L - 2G)/2(L - G), \\ H &= (1 - 2\sigma)E/6(1 + \sigma). \end{aligned} \quad (4)$$

Table 2 gives the experimental values of the elastic moduli, Poisson's ratio σ , (E/G) ratio and microhardness of the glasses. It is clear from Table 2 and Fig. 3 that all elastic moduli values are gradually decreased with different percentage of CuO mol%. It is worth mentioning that all values of elastic moduli are smaller than those calculated for binary TeO₂-V₂O₅ glasses [9] and nearly similar to our previous study on the TeO₂-V₂O₅-TiO₂ glasses [13]. All the elastic moduli variations with composition are similar to the variation of ultrasonic velocities with composition. The addition of CuO to the TeO₂-V₂O₅ network decreases both ultrasonic velocities and this in turn leads to decrease the rigidity of glass and hence, to a decrease in elastic moduli.

Table 2. Longitudinal modulus (L), shear modulus (G), bulk modulus (K), Young's modulus (E), Poisson's ratio (σ), cross-link density (Nc), (E/G) ratio, microhardness (H), Debye temperature (θ_D) and softening temperature (T_s) of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses.

CuO mol%	L [GPa]	G [GPa]	K [GPa]	E [GPa]	σ	Nc	(E/G)	H [GPa]	θ_D [K]	T_s [K]
0	63.68	22.29	33.97	54.86	0.231	2.16	2.46	4	310	467
7.5	59.73	20.40	32.53	50.62	0.241	1.82	2.48	3.52	275	375
10	54.3	18.24	30	45.49	0.247	1.65	2.49	3.08	257	320
12.5	53.46	17.94	29.55	44.76	0.248	1.62	2.49	3.01	252	297
15	52.87	17.59	29.41	44.00	0.251	1.55	2.50	2.92	247	277
17.5	52.11	17.06	29.37	42.87	0.257	1.41	2.51	2.76	242	257

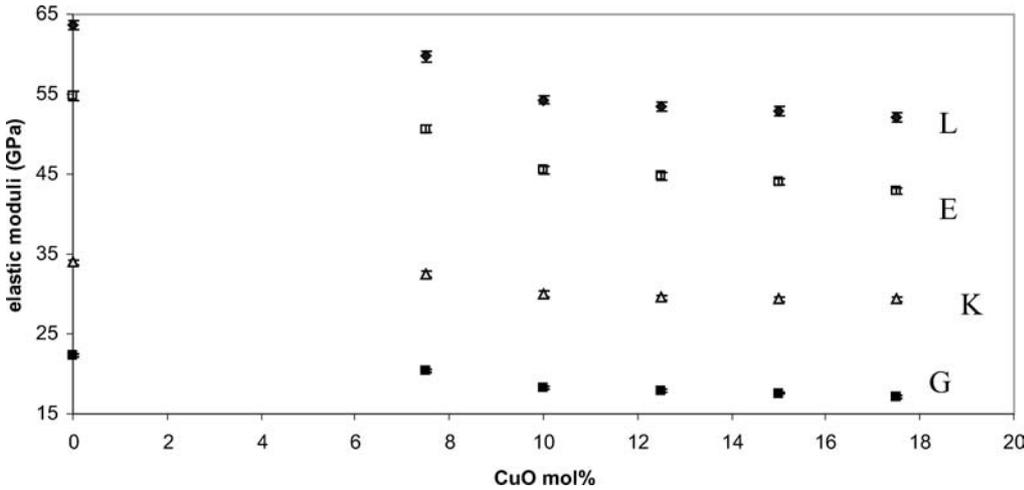


Fig. 3. Variation of elastic moduli: longitudinal (L), shear (G), and bulk modulus (K), and Young's modulus with the mole percentage of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses.

Figure 4 shows the relations of bulk and Young's moduli with molar volume. It is noted that a decrease in volume should lead to a decrease in bulk and Young's moduli. Thus it is reasonable to speculate that the type of bonding in the network plays a dominant role in deciding on the rigidity of these glass structures. Also, the observed decrease in the bulk and Young moduli are associated with the change in cross-linkage/co-ordination, Table 2, of the network structure [31].

The variation of Poisson's ratio, σ , with composition for the investigated glass system is listed in Table 2. It can be seen that the behavior of the variation is nearly opposite to that of the elastic moduli variation, i.e. σ increases with the increase of CuO from 0 to 17 mol% content. The increase in σ values is related to the decrease in cross-link density (number of bridging bonds per cation minus two) [32], which is due to the replacement of CuO with 4 co-ordinations on the

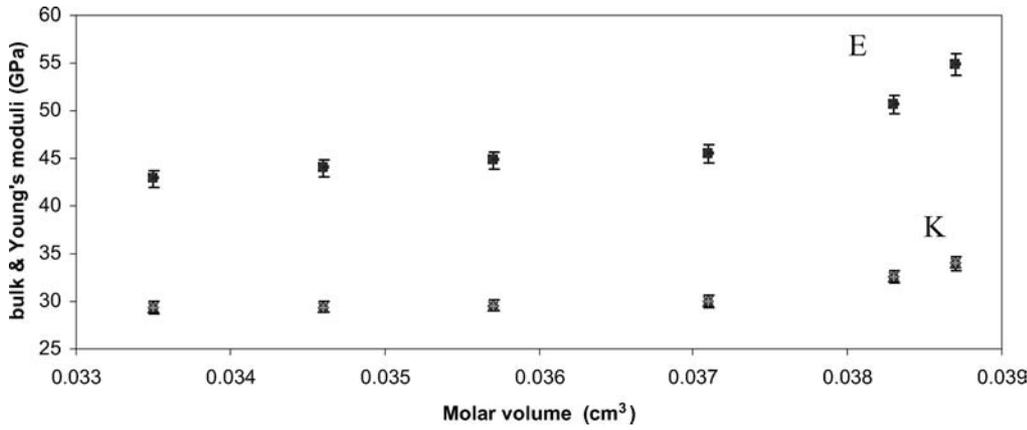


Fig. 4. Variation of bulk modulus (K), and Young's modulus with molar volume of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses.

expense of V_2O_5 with 5 and 6 co-ordination sites. Therefore, this means a stronger effect of CuO than V_2O_5 in transforming TeO_4 units to TeO_3 units. One more factor could affect the Poisson's ratio of glass, which is the relation of Poisson's ratio to (E/G) ratio, applied to the three chains network [33]. From Table 2, one can note that the behavior of σ is similar to that of E/G ratio with variation of the content CuO mol%.

Microhardness (H) expresses the stress required to eliminate the free volume of the glass. The free volume in the glass is the openness of the glasses over that of the corresponding crystals [34]. Therefore, application of high hydrostatic pressure will reduce this free volume. The variation of H with the contents of CuO mole% is tabulated in Table 2. It can be seen that the microhardness has the same attitude as the elastic moduli with increase of CuO mol% concentration, and hence the observed decrease in H is related to the decrease of the rigidity of glass.

3.4. Debye temperature and softening temperature results

Debye temperature [35] of the glass samples is obtained from the relation.

$$\theta_D = (h/K_B)(3PN_A/4\pi V_M)^{1/3}U_m, \quad (5)$$

where h is the Planck constant, K_B is the Boltzman constant, N_A is the Avogadro number, P the number of atoms in the molecular formula and U_m is the mean sound velocity defined by the relation:

$$U_m = (1/3)(2/v_s^3 + 1/v_l^3)^{-1/3}. \quad (6)$$

The compositional dependence of Debye temperature of the glass system showed the same trend as the ultrasonic wave velocities and elastic moduli. Another

parameter could be calculated from the shear velocity, which is the softening temperature T_s given by BHATTI [36] as,

$$T_s = v_s^2 M / nc^2, \quad (7)$$

where c is the constant of the proportionality and equals $0.5074 \cdot 10^5 \text{ cm s}^{-1} \text{ k}^{-1/2}$ and n is the number of atoms in the chemical formula. Softening temperature plays a crucial role in determining the temperature stability of glass. The higher is the value of softening temperature of glass, the greater will be the stability of its elastic properties. Both the Debye and softening temperatures show a decreasing trend with CuO concentration, as shown in Fig. 5.

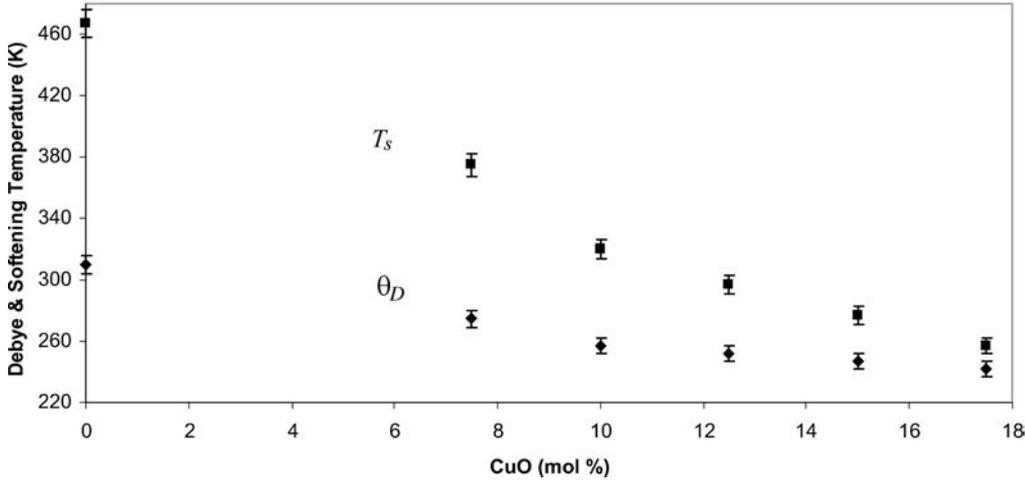


Fig. 5. Variation of Debye temperature (θ_D) and softening temperature (T_s) with the mole percentage of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses.

3.5. Theoretical models data

It is interesting to interpret the variation in the experimental elastic behavior observed in this study with bond compression model put forward by BRIDGE *et al.* [29], and MAKISHIMA and MACKENZIE model [37] for predicting the compositional dependence of elastic moduli of polycomponent oxide glasses.

Firstly, the bond compression model depends on coordination number of cation, compression bulk modulus [K_{bc}], [K_{bc}/K_{exp}] ratio, atomic ring diameter (l), the total number of cations per glass formula (η), the bond stretching force constant (F), in addition to the cross-link density (N_c) and Poisson's ratio (σ_{bc}). All these parameters of the glasses under study are summarized in Table 3.

The calculated values of (K_{bc}) were found to increase from 73.16 to 76.64 GPa, since the values of (N_c) and (F) are decreased from 2.16 and 254 (N/M) to 1.41 and 240 (N/M) respectively. Thus the increase in K_{bc} values is due to the increase in density with addition of CuO on the expense of V_2O_5 . Also, the reduced values

Table 3. Bond compression bulk modulus (K_{bc}), Ratio of (K_{bc}/K_e), average force constant (F), atomic ring size (l), total number of cations per glass formula (η) and Poisson's ratio (σ_{bc}) of TeO₂-V₂O₅-CuO glasses.

CuO mol%	K_{bc} [GPa]	K_{bc}/K_e	F [N/m]	l [nm]	η	σ_{bc}
0	73.16	2.15	254	5.17	1.35	0.212
7.5	70.93	2.18	248	5.19	1.28	0.215
10	72.23	2.41	246	5.29	1.25	0.216
12.5	73.99	2.50	244	5.30	1.23	0.218
15	75.40	2.56	242	5.30	1.20	0.219
17.5	76.64	2.61	240	5.29	1.18	0.221

of (N_c) and (F) confirm the decrease behaviors of ultrasonic velocities and elastic moduli.

Furthermore, the total number of cations per glass formula unit (η) decreased from 1.35 to 1.18 with increasing the content of CuO mol%.

In general, the ratio K_{bc}/K_{exp} is measured of the extent to which bond bending is governed by the configuration of network bonds, i.e. this ratio is assumed to be directly proportional to the ring diameter (l) and inversely proportional to the experimentally determined elastic moduli. The values of K_{bc}/K_{exp} ratio and (l) were found to increase from 2.15 and 5.17 nm to 2.61 and 5.29 nm, respectively (Table 3).

Poisson's ratio is defined for any structure as the ratio between the lateral and longitudinal strain produced when tensile force is applied. For tensile stresses applied parallel to the chains. The produced longitudinal strain will be the same and is unaffected by the cross-link density. However, the lateral strain is greatly affected by the cross-link density (N_c). Therefore, the increase in theoretically estimated Poisson's ratio (σ_{bc}) from 0.212 to 0.221 is due to the decrease in (N_c) (Table 2).

MAKISHIMA and MAKENZIE [37] presented a theoretical model to calculate the elastic moduli of oxide glasses in terms of chemical composition, which depends only on packing density (V_t) and dissociation energy (G_t) of the oxide constituents.

The calculated values of packing density, dissociation energy, and Poisson's ratio (σ_{M-M}) are listed in Table 4. By introducing CuO with packing factor equal to 7.5 cm³ on the expense of V₂O₅ with packing factor equal to 35.6 cm³, the packing density of the glass decreases from 0.526 to 0.510 g/cm³ with increasing CuO content from 0 to 17.5 mol%. This consequently causes the decrease in theoretically estimated Poisson's ratio (σ_{M-M}) from 0.236 to 0.228, which is inversely proportional to the packing density values. Furthermore, the calculated values of the dissociation energy (G_t) of the present glass compositions were found to decrease from 56.7 to 48.3 KJ/cm³ describing the decrease in rigidity of

Table 4. Packing density (V_t), dissociation energy (G_t), shear modulus (G_{m-m}), bulk modulus (K_{m-m}), Young's modulus (E_{m-m}), and Poisson's ratio (σ_{m-m}) due to Makishima-Mackenzie model of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses.

CuO mol%	V_t [g/cm ³]	G_t [k Joule/cm ³]	G_{m-m} [GPa]	K_{m-m} [GPa]	E_{m-m} [GPa]	σ_{m-m}
0	0.526	56.7	24.55	34.79	59.62	0.236
7.5	0.520	53.1	22.78	31.82	55.18	0.233
10	0.518	51.9	22.20	30.84	53.76	0.232
12.5	0.518	50.7	21.70	30.17	52.51	0.232
15	0.515	49.5	21.10	29.12	50.97	0.230
17.5	0.510	48.3	20.43	27.87	49.25	0.228

the network structure and hence the decreasing in elastic moduli with increasing CuO from 0 to 17.5%. The estimated values of both bulk modulus and Young's modulus due to applying Makishima and Makenzie model show comparatively good agreement with those obtained from experimental results, as seen in Table 4 and Table 2.

4. Conclusion

The conclusion drawn from the study of ultrasonic properties of $\text{TeO}_2\text{-V}_2\text{O}_5\text{-CuO}$ glasses with a varied CuO content is summarized here. The studies of the ultrasonic velocities, elastic moduli, Poisson's ratio, cross-link density, microhardnes, Debye temperature and softening temperature of the glasses reveal a decrease in the rigidity of the glass network when CuO concentration increases from 7.5 to 20 mol%. The variation of Poisson's ratio with composition should be the reverse of the elastic moduli variation.

The elastic moduli computed theoretically (Bond's compression model and Makishima&Makenzie model) are found to be strongly affected by stretching force constant, ring diameter, crosslink density, packing density, and dissociation energy of the glass system. The agreement between the elastic moduli calculated from the bond compression model and the experimental values are satisfied.

Finally, it should be concluded that the ultrasonic pulse echo method is a good tool to study and characterize the mechanical properties of glass materials.

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