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Elastic properties of bismuth borate glasses

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Received 17 March 1999, in final form 3 August 1999

Abstract. The elastic properties of the vitreous system Bi₂O₃–B₂O₃ have been measured by ultrasonic pulse echo overlap technique at a frequency of 10 MHz and at room temperature. For the purpose of measuring the ultrasonic velocity with high accuracy, the McSkimin criterion for determining the correct cyclic overlap between echoes is used. The elastic properties of the present glass system as a function of composition are discussed on the basis of the elastic internal energy due to the deformation. Also presented is a full discussion of the Poisson’s ratio; which is found to be rather sensitive to the glass composition.

1. Introduction

Interest in glasses has rapidly increased in recent years because of diverse applications in electronics, nuclear and solar energy technologies and acousto-optic devices. Vitreous boron oxide, a well known oxide glass, has been studied since the early 1930s [1–4]. Pure boron oxide is a very good former, covalently bonded, with interesting physico-chemical properties. B₂O₃ and related binary borate systems (especially alkali borate glasses) are also of commercial and technological interest [1, 5].

The propagation of ultrasonic waves in solids provides important information regarding solid-state motion in the materials. Two types of acoustic waves are important [6]: bulk acoustic waves and acoustic surface waves. For basic studies in solids, bulk acoustic waves are mainly used, whereas for device applications, acoustic surface waves may be more appropriate. The propagation of acoustic waves in bulk glasses has therefore been of considerable interest in understanding mechanical behaviour.

The network structure and elastic properties of borophosphate glasses have been studied in terms of the fractions of four coordinated boron atoms, Osaka et al [7]. The elastic moduli of alkaline earth borate glasses was studied by Kodoma [8]. Paul et al [9] studied the ultrasonic velocity, absorption and elastic properties of barium borate glasses. They observed velocity dispersion with frequency and temperature. The elastic moduli of these glasses were calculated theoretically, assuming the Makishima and Mackenzie’s model [9, 10]. No particular trend was observed in the ultrasonic velocity at a given frequency for these glasses [9]. The acoustic velocity in several glasses in the temperature range 73–473 K was measured by Fuxi et al [11]. The relationship between the structure and properties or the glasses and changes in the coordination number of individual glass-forming cations in alkali oxide glasses have been analysed. The ultrasonic velocity and attenuation measurements in borate glasses at different temperatures were reported by Yawale et al [12, 13] and Pakade et al [14]. Nagate et al [15] measured the ultrasonic velocity and absorption in sodium borate and lead borate glasses at different temperatures. They suggested the the product of one-third power of the velocity of sound and the molar volume of the oxide melt was constant and independent of temperature.

In this work, the ultrasonic velocity, measured with high accuracy using the pulse echo overlap method [16], and the elastic properties as a function of composition, on the basis of the elastic internal energy due to the deformation of the bismuth borate glasses, are reported.

2. Theoretical considerations

The finite elastic strain theory [17, 18], in which the strain does not need to be infinitesimal, gives a complete description of the thermodynamic potentials, with stress and strain as the mechanical variables. In considering a deformed body at some temperature, we chose the undeformed state to be the state of the body at the same temperature in the absence of external forces. Let aᵢ, i = 1, 2, 3, denote the Cartesian coordinates of a material’s particle before the deformation and λ_k(a₁, a₂, a₃) denote the coordinates of the same particle after the deformation. Then the Lagrangian strain components η_ij which describe the finite elastic strains are given by

\[ η_{ij} = \frac{1}{2} \left( \frac{∂λ_k}{∂a_i} \frac{∂λ_k}{∂a_j} \right) - δ_{ij} \] (1)

where i, j = 1, 2, 3 and the symbol δ_ij is the Kronecker delta.

Instead of the tensor notation, we use the abbreviated Voigt notation and the Lagrangian strain in the abbreviated
notation is defined by Brugger [18] as
\[
\eta_\alpha = 2\eta_i/(1 + \delta_{ij}).
\]
Because the sound wave propagates under adiabatic conditions, the internal energy is chosen in the present work as the most convenient thermodynamic potential. Let \( u \) and \( s \) be the internal energy and the entropy respectively, taken per unit mass; \( U \) and \( S \) taken per unit volume; and \( U_m \) and \( S_m \) taken per unit mole. Then, for the energy equation for conservative media [18], the internal energies can be expressed by
\[
du = T \ ds + (1/\rho) t_u \ d\eta_\alpha \quad (2a)
\]
\[
dU = T \ dS + t_u \ d\eta_\alpha \quad (2b)
\]
\[
dU_m = T \ dS_m + (M/\rho) t_u \ d\eta_\alpha \quad (2c)
\]
where \( T \) is the temperature, \( \rho \) is the density of the undeformed state, \( M \) is the molar mass, \( t_u = \rho(\partial u/\partial \eta_\alpha) \) is a quantity called the thermodynamic tension, and the summation is taken over the suffix \( \alpha \) from one to six. Equations (2a) and (2b) are the usual expressions when the composition change is not to be considered, while (2c) is the new expression suitable for discussing elastic properties as a function of composition. These are the fundamental thermodynamic relations for the internal energies of a deformed body.

In order to be able to apply the general formulae of the internal energy to any particular deformation, we must know the internal energy of the body as a function of the strain. This expression is easily obtained by using the fact that the deformation is small and by expanding the internal energy in powers of \( \eta_\alpha \). As the glass is elastically isotropic, we shall consider here only isotropic bodies. Since \( t_u = 0 \) when no external stresses are present, it follows that there are no first-order terms in the expansions of the internal energy in powers of \( \eta_\alpha \). The second derivatives of the internal energy with respect to the Lagrangian strain, keeping the entropy constant, are given by
\[
\left(\frac{\partial^2 u}{\partial \eta_\alpha^2}\right)_S = v^2 \quad (3a)
\]
\[
\left(\frac{\partial^2 U}{\partial \eta_\alpha^2}\right)_S = C^S_{uu} = \rho v^2 \quad (3b)
\]
\[
\left(\frac{\partial^2 U_m}{\partial \eta_\alpha^2}\right)_{S_m} = M v^2 \quad (3c)
\]
where \( v \) is the velocity of a sound wave having the strain component \( \eta_\alpha \) (where \( \alpha = 1, 2 \) and 3 refer to normal strains or stresses for the longitudinal wave and \( \alpha = 4–6 \) refer to the shear components for the transverse wave) and \( C^S_{uu} \) is the second-order isotropic stiffness coefficient.

If the strain occurs under adiabatic conditions, the internal energy of the body increases. Expanding each expression of the internal energies in powers of the Lagrangean strain \( \eta_\alpha \) at constant entropy about the state of zero strain, and neglecting all terms above the second order, we obtain the elastic internal energies:
\[
\Delta u = u(s, \eta_\alpha) - u(s, 0) = \frac{1}{2} v^2 \eta_\alpha^2 \quad (4a)
\]
\[
\Delta U = U(S, \eta_\alpha) - U(S, 0) = \frac{1}{2} C^S_{uu} \eta_\alpha^2 = \frac{1}{2} \rho v^2 \eta_\alpha^2 \quad (4b)
\]
\[
\Delta U_m = U_m(S_m, \eta_\alpha) - U_m(S_m, 0) = \frac{1}{2} M v^2 \eta_\alpha^2. \quad (4c)
\]
The elastic internal energies taken relative to unit mass, unit volume and unit mole are related to the strain component, \( \eta_\alpha \), by the material constants \( v^2, \rho v^2 \) and \( M v^2 \) respectively. Equations (4a)–(4c) hold for the longitudinal velocity, \( v_l \), and the transverse velocity \( v_t \). It should be noted that (4a)–(4c) have different meanings when discussing elastic properties as a function of composition. When the elastic strain is infinitesimal, (4b) is equivalent to the strain energy function [19, 20] in classical elasticity theory.

The elastic internal energy per mole is chosen, if the elastic internal energy is taken relative to unit mass or unit volume; it relates to different amounts of the substances when the composition is changed. Let \( U_1 \) and \( U_2 \) be the partial molar internal energies of component one (in the present work, B2O3) and component two (Bi2O3), respectively. Then the mean molar internal energy \( U_m \), can be expressed by
\[
U_m = U_1 x_1 + U_2 x_2 = (U_2 - U_1) x_2 + U_1. \quad (5)
\]
The second-order derivative of (5) with respect to \( \eta_\alpha \) at constant entropy gives
\[
M v^2 = \left(\frac{\partial^2 (U_2 - U_1)}{\partial \eta_\alpha^2}\right)_S x_2 \left(\frac{\partial^2 U_1}{\partial \eta_\alpha^2}\right)_S \quad (6)
\]
where \( x_1 \) and \( x_2 \) are the mol% of the B2O3 and Bi2O3 respectively. When we plot \( M v^2 \) against \( x_2 \), each of
\[
\left(\frac{\partial^2 U_1}{\partial \eta_\alpha^2}\right)_S
\]
and
\[
\left(\frac{\partial^2 U_2}{\partial \eta_\alpha^2}\right)_S
\]
can be determined in principle from the tangential line of the curve with respect to \( x_2 \). If we define the elastic internal energies of component one and two respectively as
\[
\Delta U_1 = \frac{1}{2} \left(\frac{\partial^2 U_1}{\partial \eta_\alpha^2}\right)_S \eta_\alpha^2 \quad (7)
\]
and
\[
\Delta U_2 = \frac{1}{2} \left(\frac{\partial^2 U_2}{\partial \eta_\alpha^2}\right)_S \eta_\alpha^2 \quad (8)
\]
then
\[
\left(\frac{\partial^2 U_1}{\partial \eta_\alpha^2}\right)_S
\]
and
\[
\left(\frac{\partial^2 U_2}{\partial \eta_\alpha^2}\right)_S
\]
represent, respectively, the elastic internal energies per unit mole of B2O3 and Bi2O3 generated for a given strain. Hence, they express the elastic resistance of the respective components (B2O3 and Bi2O3) to the deformation.
3. Experimental procedure

3.1. Glass preparation

A series of bismuth–borate glasses were prepared from laboratory reagent grades of Analar boron oxide (B$_2$O$_3$, molecular weight 69.622) and Analar bismuth oxide (Bi$_2$O$_3$, molecular weight 465.9608), using alumina crucibles (of 100 cm$^3$ capacity) heated in an electric furnace that was open to the atmosphere. The reagents were mixed and placed in a furnace held at 850 $^\circ$C for complete fusion. Then, the furnace temperature was raised to 1100 $^\circ$C and kept for 2 h at this temperature. The glass melts were stirred occasionally with an alumina rod to ensure homogeneous melts. Each melt was case into two mild-steel molds to form glass rods 1 cm long by 1.6 cm diameter. Then, the mold halves were released to prevent cracking, the thin walls of the mold (2 mm) also served to minimize the risk of cracking. After casting, each glass was immediately transferred to an annealing furnace held at 300 $^\circ$C for 1 h. The furnace was switched off and the glasses were allowed to cool to room temperature at an initial cooling rate of 3 $^\circ$C min$^{-1}$. This procedure was employed to prepare glasses with a glass formation range of 5–45 mol% Bi$_2$O$_3$ (starting compositions). The compositions of the specimens studied are listed in table 1. The densities of the glasses were measured by the Archimedes method using toluene as the immersion liquid and they are accurate to ±0.001 g cm$^{-3}$.

3.2. Ultrasonic measurements

Specimens used for ultrasonic measurements were in the form of cylindrical rods, 1.6 cm diameter of 0.5 cm thick, with end faces optically polished to a parallelism of 1–2 arcsec, using a polishing machine (Metal Research, Multipol 2), with a special jig (MR, MK2) holding the specimen.

The ultrasonic travel time was measured by the pulse echo overlap method [16] at a frequency of 10 MHz and at room temperature.

X-cut and Y-cut quartz transducers were used for the generation and detection of the longitudinal and transverse waves, respectively. The transducer was bonded to the sample with Nonaq stopcock grease $\sim 2.2 \times 10^{-4}$ cm thick and with an acoustic impedance $Z_b = 2.7 \times 10^3$ mech.ohm cm$^{-2}$.

<table>
<thead>
<tr>
<th>Variables</th>
<th>I</th>
<th>$\alpha$</th>
<th>$\beta$</th>
<th>$\gamma$</th>
</tr>
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<tbody>
<tr>
<td>$\rho$</td>
<td>4.01</td>
<td>0.0459</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>SE</td>
<td>0.027</td>
<td>0.0014</td>
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<td>—</td>
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<tr>
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<tr>
<td>SE</td>
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<td>0.012</td>
<td>0.000</td>
<td>34</td>
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<tr>
<td>$V_l$</td>
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<td>0.0603</td>
<td>$-0.001$</td>
<td>378</td>
</tr>
<tr>
<td>SE</td>
<td>0.126</td>
<td>0.016</td>
<td>0.000</td>
<td>44</td>
</tr>
<tr>
<td>$G$</td>
<td>109.96</td>
<td>6.3451</td>
<td>$-0.1103$</td>
<td></td>
</tr>
<tr>
<td>SE</td>
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<td>0.71</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
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<td>$-0.04386$</td>
<td></td>
</tr>
<tr>
<td>SE</td>
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<td>0.54</td>
<td>0.015</td>
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</tr>
<tr>
<td>$\beta$</td>
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<td>$-0.004$</td>
<td>14</td>
<td>0.0000952</td>
</tr>
<tr>
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<td>0.0000485</td>
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<td>$-0.001$</td>
<td>436</td>
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<tr>
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<td>0.00498</td>
<td>0.0000843</td>
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</tr>
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</table>

Table 2. Regression analysis of the variables (density, $\rho$; longitudinal and shear ultrasonic velocities, $v_l$, $v_t$; elastic moduli, $L$, $G$; Poisson’s ratio $\sigma$) of the bismuth borate glasses $\alpha$, $\beta$, and $\gamma$ are constants for a quadratic equation, as explained in the text.

When the ultrasonic transit time between echoes is measured, it is essential to determine which carrier cycles should properly be compared in observing two echoes. The criterion (known as $n = 0$) proposed by McSkimin [21] and in addition McSkimin and Andreatch [22] has been used to solve this problem [16]. Once a pair of echoes is properly overlapped, the pulse echo overlap technique can measure the absolute transit time within an accuracy of one part in 5000 for round trips greater than 5 $\mu$s [16].

3.3. Statistical data analysis

The data was analysed using the statistical package for social sciences (SPSS), by fitting regression curves, and the results are given in table 2. The regression coefficients and their standard errors (SE) are given for the curves shown in figures 1–9. In table 2 $\hat{Y}$ stands for the variables shown in the first column and $\hat{x}$ is the Bi$_2$O$_3$ concentration given in mol%. As can be seen in figures 1–9, for most of the variables a curvilinear ($\hat{Y} = \alpha + \beta \hat{x} + \gamma \hat{x}^2$) gives the best fit. However, for the density, the best fit is a straight line ($\hat{Y} = \alpha + \beta \hat{x}$), while the Poisson’s ratio is constant ($\hat{Y} = \hat{Y}$).
Figure 1. Variation of density with mol% Bi₂O₃ in Bi₂O₃–B₂O₃ glass systems.

Figure 2. Dependence of molar volume on the composition in Bi₂O₃–B₂O₃ glass systems.

Figure 3. Compositional dependence of the velocity of longitudinal acoustic waves in Bi₂O₃–B₂O₃ glass systems.

Figure 4. Compositional dependence of the velocity of shear acoustic waves in Bi₂O₃–B₂O₃ glass systems.

Figure 5. Dependence of longitudinal modulus on the composition of Bi₂O₃–B₂O₃ glass systems.

Figure 6. Dependence of the shear modulus on the composition of Bi₂O₃–B₂O₃ glass systems.

Figure 7. Variation of the longitudinal elastic internal energy with composition of Bi₂O₃–B₂O₃ glass systems.

Figure 8. Variation of the shear elastic internal energy with composition of Bi₂O₃–B₂O₃ glass systems.

4. Results and discussion

Table 1 gives the compositions and the properties of the Bi₂O₃–B₂O₃ glasses. From inspection of this table, it will be observed that there is a change in the behaviour of the compositional dependence of all the properties mentioned so far at a Bi₂O₃ content of approximately 25 mol% (see also figures 1–9).

The variation of the molar volume with the molar per cent of Bi₂O₃ oxide is seen to increase up to 45 mol% Bi₂O₃. The rate of increase is slightly less beyond 25 mol% Bi₂O₃ (see figure 2). The plot of density against mol% (see figure 1)
showed an increase with increase in mol%, with a point of inflection at about 25 mol%. The increase of the density of the glasses accompanying the addition of Bi₂O₃ is probably attributable to a change in cross-link density and coordination numbers of Bi³⁺ ions.

Figures 3 and 4 show the plots of longitudinal and shear wave velocities \( v_t \) and \( v_s \) against mol% Bi₂O₃, respectively. Both increase at first with increasing Bi₂O₃ mol% up to a maximum at 25 mol% Bi₂O₃ and then decrease as the Bi₂O₃ mol% increases further. At a given strain, the elastic internal energies of the longitudinal and shear strains per unit mass of the glasses, as functions of composition, follow the tendencies of \( v_t \) and \( v_s \), respectively.

Figures 5 and 6 show the variations of longitudinal \( C_{l11} \) and shear \( C_{s44} \) moduli against mol% Bi₂O₃. At a given strain, the elastic internal energies of the longitudinal and shear strains per unit volume of the glasses, as functions of composition, follow the tendencies of longitudinal and shear moduli, respectively. Both longitudinal and shear moduli exhibit maxima at different compositions (longitudinal modulus at 25 mol% Bi₂O₃ and shear modulus at 20 mol% Bi₂O₃). This difference arises numerically from the fact that the different magnitudes of \( v_t \) and \( v_s \) as a function of mol% Bi₂O₃ curves are multiplied by the density, which increases monotonically with increasing Bi₂O₃ composition. One reason for this difference may come from the volume effect, in that \( C_{s44} \) expresses the resistance of the body to deformation where no change in volume is involved, while \( C_{l11} \) expresses the resistance where compressions and expansions are involved.

Figures 7 and 8 show the variations of the elastic internal energies of the longitudinal and shear strains per unit mole of the glasses (\( Mv_t^2 \) and \( Mv_s^2 \), respectively, as functions of the Bi₂O₃ composition. The magnitude of \( Mv_t^2 \) and \( Mv_s^2 \) show maxima at different Bi₂O₃ compositions (\( Mv_t^2 \) at 35 mol% and \( Mv_s^2 \) at 30 mol%). This difference arises numerically from the fact that the different magnitudes of \( v_t^2 \) and \( v_s^2 \) as functions of Bi₂O₃ compositions curves are multiplied by the molar mass, which increases linearly with increasing Bi₂O₃ composition, this will be explained later.

By applying (6) to figures 7 and 8, the following relations may be obtained for \( Mv_t^2 \) and \( Mv_s^2 \) as functions of composition:

\[
\left( \frac{\partial^2 U_1}{\partial n_o^2} \right)_{S_n} > \left( \frac{\partial^2 U_2}{\partial n_o^2} \right)_{S_n} \quad (9)
\]

\[
\left( \frac{\partial^2 U_1}{\partial n_o^2} \right)_{S_n} = \left( \frac{\partial^2 U_2}{\partial n_o^2} \right)_{S_n} \quad (10)
\]

where (9) is at compositions of less than 35 and 30 mol% Bi₂O₃; (10) is at compositions of 35 and 30 mol% Bi₂O₃; and (11) is at compositions of more than 35 and 30 mol% Bi₂O₃, respectively (see figures 7 and 8).

Equation (9) shows that the network former, Bi₂O₃, is soft and easily deformed by stress while the modifier, Bi₂O₃, resists deformation more strongly. Except in the vicinity of the maxima (the composition region where boron atoms change from the threefold to the fourfold coordination by addition of modifier Bi₂O₃) the inequality of (9) may therefore arise from the fact that the modifier is enclosed within the network, so that it resists the deformation of the network.

Equation (11) shows that the network former now becomes hard and resists deformation more strongly than the modifier. The composition region corresponding to (11) is in the destruction region, so that the three-dimensional connection of the network has been partly broken up by the formation of non-bridging oxygen ions. Bi₂O₃ forms into smaller rigid anion groups so that it resists deformation more strongly.

Equation (10) indicates the intermediate state between the two states above, showing that the network former and the modifier equally resist deformation.

The reason why \( Mv_t^2 \) and \( Mv_s^2 \) exhibit maxima at different compositions can be explained as follows. The partial molar internal energies are regarded as functions of the independent variables \( S_n \), \( \eta_n \) and \( x \) (mol% Bi₂O₃), in which \( \eta_n \) are up to quadratic terms. We consider \( \frac{\partial^2 U_1(S_n, \eta_n, x)}{\partial \eta_n^2} \) and \( \frac{\partial^2 U_2(S_n, \eta_n, x)}{\partial \eta_n^2} \) as functions of \( x \), these depend on each of the strain components. Then, compositions satisfying (10) are generally different between the longitudinal and the shear strain respectively.

By increasing the modifier’s composition, Bi₂O₃, becomes more rigid and the degree to which the modifier prevents the deformation of the surrounding network decreases until, finally, the modifier itself is deformed by the application of stress. These properties depend to an extent on the nature of the strain component.

To interpret our data on the compositional dependence of Poisson’s ratio (\( \sigma \)) we first give a general qualitative model of the variation of \( \sigma \) with vitreous composition, developed from an idea expressed by Bridge et al [23], which is summarized as follows. (i) \( \sigma \) decreases with cross-link density (for constant ratio of bending- to stretching-force constant) and (ii) \( \sigma \) decreases with the increasing ratio of bond-bending to stretching force constant \( F_b/F \) (at constant cross-link density). If these mechanisms are then applied in our case, we find that the variation of Poisson’s ratio with composition should be exactly the reverse of the elastic moduli variation.

The experimental data indicate that the Bi³⁺ in octahedral coordination is usually weakly directional—producing a low ratio of \( F_b/F \) and correspondingly a high Poisson’s ratio in the compositional range of 25–45 mol% Bi₂O₃. In the compositional range of 5–20 mol% Bi₂O₃, \( \sigma \) would fall steeply (see figure 9) where the rate of increase of cross-link density with Bi content is high, as most of the bismuth is in the Bi³⁺ (octahedral) form with its high cross-link density.
5. Conclusion

The elastic properties of the vitreous Bi$_2$O$_3$ was measured by ultrasonic pulse echo overlap technique at a frequency of 10 MHz and at room temperature. The obtained conclusions are as follows.

1. From table 1, one can observe that there is a change in behaviour of the compositional dependence of all the properties measured at around 25 mol% Bi$_2$O$_3$.
2. The density increases with an increase in mol% Bi$_2$O$_3$. This is probably attributable to a change in cross-link density, the greater mol wt. of the bismuth atom and the coordination numbers of Bi$^{3+}$ ions.
3. The longitudinal and shear wave velocities and the elastic moduli of the present glass systems increase with increasing Bi$_2$O$_3$ composition, up to 25 mol%, and then decreases as Bi$_2$O$_3$ composition increases further.
4. The variations of the elastic internal energies of longitudinal and shear strains per unit mole of the glasses show that: at compositions less than 35 and 30 mol% Bi$_2$O$_3$, the network former Bi$_2$O$_3$ is soft and easily deformed by stress while the modifier Bi$_2$O$_3$ resists deformation more strongly. At 35 and 30 mol% Bi$_2$O$_3$, the network former and the modifier equally resist deformation. At compositions of more than 35 and 30 mol% Bi$_2$O$_3$, the network former becomes hard and resists deformation more strongly than the modifier.
5. The variation of Poisson’s ratio with composition should be exactly the reverse of the elastic moduli variation. It decreases with increasing Bi$_2$O$_3$ composition up to 25 mol%, and then increases with increasing Bi$_2$O$_3$ composition over 25 mol%.

Acknowledgments

The authors thank Dr A Fuith in Institute for experimental physics, Vienna University, Austria, for measuring the ultrasonic velocities of the present glass system.

References