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Elastic Constants and Thermal Properties of Lead-bismuth Borate Glasses

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Abstract: Systematic series of lead-bismuth borate glasses, where PbO, Bi_2O_3 and B_2O_3 content change for every series based on their weight percentage have been prepared. The ultrasonic and glass transition temperature (T_g) of this glass system have been studied using the Matec MBS-8000 Digital Signal Processing and conventional Differential Thermal Analysis (DTA) method. Elastic properties of the glass have been calculated together with Poisson's ratio from the measured densities as well as longitudinal (V_L) and shear (V_S) ultrasonic velocities. The T_g determined from the change of the base line in the DTA chart. The result showed that both properties are much depends on the changes of their atomic arrangement behaviour with an addition of the modifiers.

Key words: Lead-bismuth borate glasses, glass transition temperature, Differential Thermal Analysis (DTA), ultrasonic velocities, elastic moduli

INTRODUCTION

Glass can be prepared either by the conventional method using a mixture of oxides as starting materials and quenching the melts or by the sol-gel method involving hydrolysis and poly condensation of organometallic compounds to form a gel that is converted by heating into a glass (Pernice *et al*., 1997).

Lead oxide based glasses is an interesting system to study because the glass phase can be formed over a wide concentration. Moreover, PbO can enter the glass network both as a network modifier and also as a network former (Meera *et al*., 1989). It was suggested that the addition of one or, more of SnO, PbO, ZnO, Al_2O_3 and Fe_2O_3, results in the formation of Sn-O-P, Pb-O-P, Zn-O-P, P-O-Al and P-O-Fe bonds and leads to dramatically improvement in the chemical durability of the phosphate glasses (Shih *et al*., 1998).

In recent years, several new uses for bismuth have been developed as non-toxic substitutes for lead in various applications. Bismuth is known as semimetals of rhombohedral crystal structure (Nagao *et al*., 1999). Among the heavy metals, bismuth is the heaviest and the only non-toxic element. Because of this, borate and phosphate glasses containing bismuth have obtained great attention, since the melt of the glass vitrify over a wide range of composition (Saddeek, 2004). Bismuth is a leading candidate for replacing lead in applications that have an environmental impact. Today bismuth in its elemental form has many uses, including recent developments as a nanowire. Bismuth compounds are well known in pharmaceutical applications both as medicines and more recently as radio-opaque agents (Lavallee, 2001).

In many anhydrous, borate systems melts of composition that rich in B_2O_3 show a rather high viscosity and a marked tendency to glass formation. This, on one hand, hinders the growth of large single crystals, but, on the other hand, it also allows the preparation of glasses that may posses interesting physical properties (Becker, 2003). It is known that, the boron atom usually coordinates with either three or four oxygen atoms forming $[BO_3]^{3-}$ or $[BO_4]^{5-}$ structural units (Opera *et al*., 2004). On borate glasses, a lot of work has been going on during the last decade, especially concerning an optimization of glass preparation, investigation of properties and efforts to acquire information about the structure of glasses.

Combining bismuth oxide (Bi_2O_3) and lead oxide (PbO) with boron oxide (B_2O_3) allows one to tune the physical properties in a wide range depending on the glass composition. The present research intends to study the ultrasonic and thermal properties of this glass system ($PbO\text{-}Bi_2O_3\text{-}B_2O_3$) over a wide range of composition.

MATERIALS AND METHODS

Analar PbO, Bi_2O_3 and B_2O_3 were used to prepare 20 glass samples with different composition using the conventional method. These reagents were weighed and

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mixed together with appropriate amounts where B_2O_3 content ranging from 47.6% to 38.2%. The PbO and Bi_2O_3 content increase ranging from 31.5% to 55.7% and 3.5% to 16.6%, respectively and their amorphous nature was confirmed by X-ray Diffraction (XRD) technique and made at room temperature using a Philips X-ray Diffractometer.

The densities of the samples were measured at room temperature using Archimedes principle with acetone as buoyant liquid. A cylinder shape glass sample was weighed in air (W_{air}) using electronic balancer (± 0.0001 g) manufactured by Metler Toledo, W_{ac} the glass sample weight in buoyant and ρ_{ac} the acetone density of the buoyant. The acetone density using here is 0.7899 g cm^{-3}. The relative density is given by following the relation (Halimah, 2005):

$$\rho_s = \rho_{ac} \frac{W_{air}}{W_{air} - W_{ac}} \quad (1)$$

Ultrasonic pulse-echo technique is widely used nowadays in studying the physical properties of material. In this research, all the glass samples were cut using Isomet Low Speed Saw machine to obtain 0.5 to 1.0 cm height samples and the ultrasonic wave propagation can be obtained by using the Matec Instruments MBS-8000 computer controlled ultrasonic test system at frequency of 5 MHz. The Matec DSP system utilizes a high precision, high-resolution measurement technique, which calculates time of flight, velocity, attenuation and dispersion of ultrasonic waveforms automatically, at single or over a multiple frequencies (Halimah, 2001; Khamirul, 2000).

Supercooled liquids are often called rubbers and the solids are glasses, transformation between the supercooled liquid and solid states occur over a temperature range and the transition is known as the glass transition. One of the methods used to determine the glass transition is Differential Thermal Analysis (DTA).

DTA is a technique by which the thermally induced physical or chemical changes in a substance can be studied (Mondal, 1997).

In this study, all measured glass samples were crushed to fine grains and weighted about 150 to 200 mg before placed in special shaped Platinum (Pt) cup with the capacity of 100 μL. The glass transition temperature is determined from the DTA curve as obtained from Setaram DTA/DSC equipment heated in temperature range from 30 to 500°C at a constant rate of 20 K min^{-1}. In order to reach constant combustion during measurement process, an inert nitrogen (N_2) atmosphere was used.

RESULTS AND DISCUSSION

The physical properties of lead-bismuth borate glasses used in this study, shows clearly in Table 1.

The XRD patterns of the glasses obtained are shown in Fig. 1. All selected quenched materials were found to be fully in the glass form and a broad halo, which is characteristic of amorphous structure, was obtained at around $2\theta \cong 30°$.

This indicates the absence of long range atomic arrangement and also the periodicity of the three dimensional network in the quenched material (Aksan *et al*., 2000).

The addition of Bi_2O_3 in the B_2O_3 network, increased the number of Non-bridging Oxygen (NBO) atoms which in turn increased the density of glass and this may be attributed to replacement of a low-density oxide (B_2O_3,

Table 1: Physical properties of lead-bismuth borate glasses

	Weight of substance (%)						
Glass sample	PbO	Bi_2O_3	B_2O_3	Density (kg m^{-3})	V_L (m s^{-1})	V_S (m s^{-1})	T_g (°C)
A1	42.5	9.9	47.6	3920	4013	2301	470
A2	57.8	13.4	28.8	5026	3896	2229	457
A3	65.7	15.2	19.1	5187	3440	2114	422
A4	70.5	16.4	13.1	5774	3428	1924	418
A5	73.7	17.1	9.2	6325	3261	1787	377
B1	38.3	14.1	47.6	4033	4082	2310	469
B2	52.0	19.2	28.8	4878	3930	2256	462
B3	59.1	21.8	19.1	5164	3523	2172	436
B4	63.5	23.4	13.1	5660	3439	1979	419
B5	66.3	24.5	9.2	5802	3289	1858	414
C1	34.4	18.0	47.6	3819	4156	2343	465
C2	46.8	24.4	28.8	4774	4045	2285	464
C3	53.1	27.8	19.1	5630	3650	2184	439
C4	57.1	29.8	13.1	5961	3451	2006	417
C5	59.7	31.1	9.2	6063	3301	1923	402
D1	30.9	21.5	47.6	3904	4271	2402	468
D2	42.0	29.2	28.8	4813	4230	2323	462
D3	47.7	33.2	19.1	5789	3714	2246	431
D4	51.2	35.7	13.1	5880	3524	2089	410
D5	53.6	37.2	9.2	6199	3389	2052	405

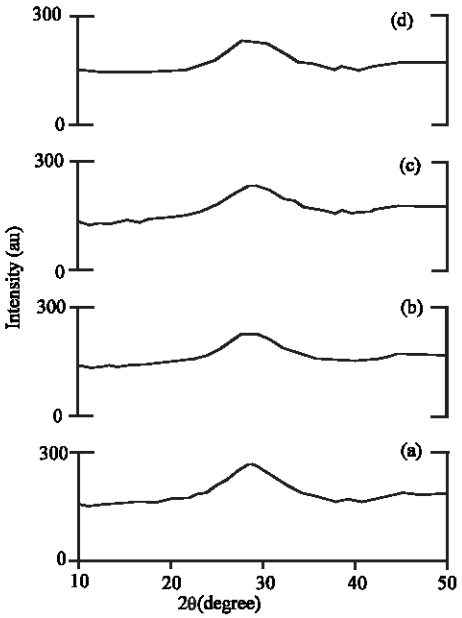

Fig. 1: XRD patterns of the glasses, (a) Glass sample A3, (b) Glass sample B3, (c) Glass sample C3 and (d) Glass sample D3

2550 kg m^{-3}) by a high-density oxide (Bi_2O_3, 8990 kg m^{-3}) (Saddeek, 2004). The increases in the density also consistent with the previous work on the bismuth borate glass system by Becker (2003) and Chowdari (1996).

Also, the addition of PbO into the glass system caused the densities to increase and the increases indicated that the Pb^{2+} as a network modifier altered the structure of the glass by creating the NBOs in the network, so that the structure turns out to be more randomly oriented (Azman, 2000).

The measured longitudinal and shear velocities (V_l and V_s, respectively) for lead-bismuth borate glass are also depicted in Table 1. An interesting feature occurs that there is a decrease in values for both wave velocities and this happen for every series of glass. For example the longitudinal velocity for A1 to A5 glass samples decreases from 4013 to 3261 m s^{-1} and shear velocity from 2301 to 1787 m s^{-1}, as PbO and Bi_2O_3 content increases from 42.5 to 73.7% and 9.9 to 17.1%, respectively.

The decrease of ultrasonic velocity linearly with the increasing of Bi_2O_3, indicating that Bi_2O_3 plays a dominant role in the velocity (Yawale *et al*., 1995). An addition of more Bi_2O_3 in glass interstices causes more ions being open up in the network. Thus, weakening of the glass structure or reduction in the rigidity of the network takes places. As consequences, both velocities V_l and V_s decrease with the addition of Heavy Metal Oxide (HMO).

The observed higher values in velocity at low Bi_2O_3 content and low values in velocity at high Bi_2O_3 content confirm a substantial change in glass structure. The Bi^{3+} cations are incorporated in the glass network as [BiO_6] octahedral units, on the expense of the Bi^{3+} cation which is incorporated as BO_3 and then converted in to BO_4, namely Bi_2O_3 has higher number of network bonds. The stretching force constant for Bi_2O_3 (216 N m^{-1}) is less than that for B_2O_3 (450.7 N m^{-1} for BO_3 and 401.2 N m^{-1} for BO_4) and the molar volume of Bi_2O_3 is 5.24×10^{-6} m^3 mol^{-1} which is larger than that of B_2O_3, which is 27.3×10^{-6} m^3 mol^{-1}. Further addition of Bi_2O_3 causes more and more discontinuity and, hence, a decrease in rigidity and velocity results (Saddeek, 2004).

The addition of PbO to the borate glass system will also decrease both of velocities and cause the glass properties to be changed (Azman *et al*., 1997). To interpret the compositional dependence of the wave propagation in the lead borate glasses, one should consider the effect of bond strength, packing density, coordination number and crosslinking in the glass, where those parameters play important roles in determination of the structural changes.

By considering the character of such oxide in this glass system which plays as modifier, it will modify the glass structure, thus making the glass getting softer (Sidek *et al*., 1996). In fact, when the wave propagated through the sample, a harder material will produce higher velocity whereas a softer material will produce lower velocity (Carini *et al*., 1984). Although the glass is softer, it does not mean that the glass is less dense (Azman, 2000).

The only independent elastic constant for isotropic solids and glasses are longitudinal modulus (C_{11}) and shear modulus (C_{44}), where calculation for other elastic constants and Poisson ratio (σ) depending on the values of density and both velocities.

In an amorphous solid (such as glass), the elastic strain produced by a small stress can be described by C_{11} and C_{44} given as:

$$C_{11} = \rho V_L^2 \quad (2)$$

$$C_{44} = \rho V_S^2 \quad (3)$$

where, ρ is sample density.

The sound velocities also allow the determination of Young's modulus (Y) which defined as a ratio of linear stress over the linear strain and is related to the bond strength. Also, the bulk modulus (B) defined as the changing in volume when a force is acted upon it at all direction, where both of this given as:

$$B = \frac{\rho\left(3V_L^2 - 4V_S^2\right)}{3} \quad (4)$$

$$Y = \frac{\rho V_S^2\left(3V_L^2 - 4V_S^2\right)}{V_L^2 - V_S^2} \quad (5)$$

The values of elastic moduli for the studied glasses are shown in Fig. 2 and 3. It can be seen for every glass series, there is similar pattern in elastic moduli with the increasing of PbO and Bi_2O_3 weight content in the ternary glass system. The values increased at the earlier stage and then decreased subsequently.

For the longitudinal modulus, the values increased from 63.1 to 76.3 GPa before decreasing to 67.3 GPa for A1 to A5 glass series. This situation also occurred to the rest of sample series. Meanwhile for the shear modulus, for similar series, the values increased from 20.8 Gpa to 25.0 GPa then decreased to 20.2 GPa.

Based on this result, it can be seen that longitudinal modulus values for every glass series were higher than shear modulus. This shows that, the materials formed are easier to bend than to be elongated (Azman, 2000).

This interesting behaviour continues in the Young's and bulk modulus patterns. As an example, for A1 to A5 series, the Young's modulus increases from 52.1 to 62.8 GPa before decreases to 51.9 GPa and bulk modulus increases from 35.5 to 42.3 Gpa then decreases to 40.3 GPa. This pattern is applied to the rest of glass series.

The existence of PbO and the nature of Bi_2O_3 will cause a sudden increase (at earlier stage) in the elastic moduli (Saddeek, 2004). In general, addition of Bi_2O_3 to B_2O_3 network glass will decrease the rigidity, the velocity and hence the elasticity (Yawale *et al*., 1995). The decrease in the rigidity of the glasses contributes a decrease in velocity and, hence, elastic moduli in the other hand. In another study of binary system PbO-B_2O_3 by Azman (2000) also found similar changes in some of the elastic moduli and observed that the elastic properties have a potential to have a strong relationship with the structural changing.

Poisson's ratio is defined as the ratio between lateral and longitudinal strain produced when tensile force is applied or function of the ratio of the longitudinal and shear velocities. Micro-hardness 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. Therefore, application of high hydrostatic pressure will reduce this free volume, for an example the glasses will be compacted.

In this glass study, the Poisson's ratio calculated using the following relation:

$$\sigma = \frac{\left(V_L^2 - 2V_S^2\right)}{2\left(V_L^2 - V_S^2\right)} \quad (6)$$

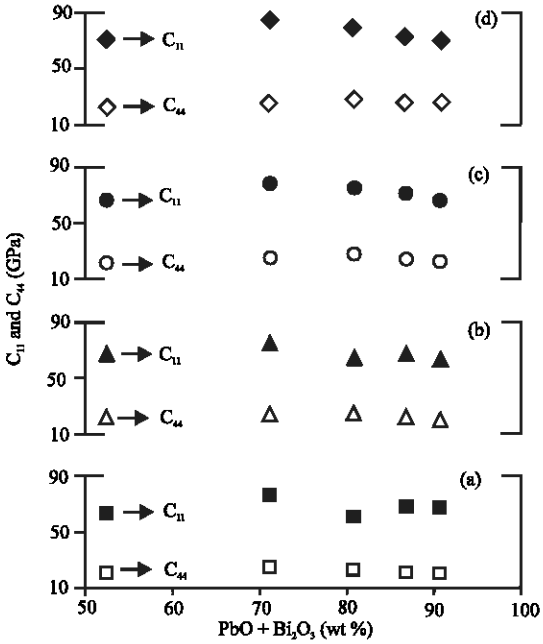

Fig. 2: Longitudinal and shear modulus of glasses, where (a) A1-A5, (b) B1-B5, (c) C1-C5 and (d) D1-D5

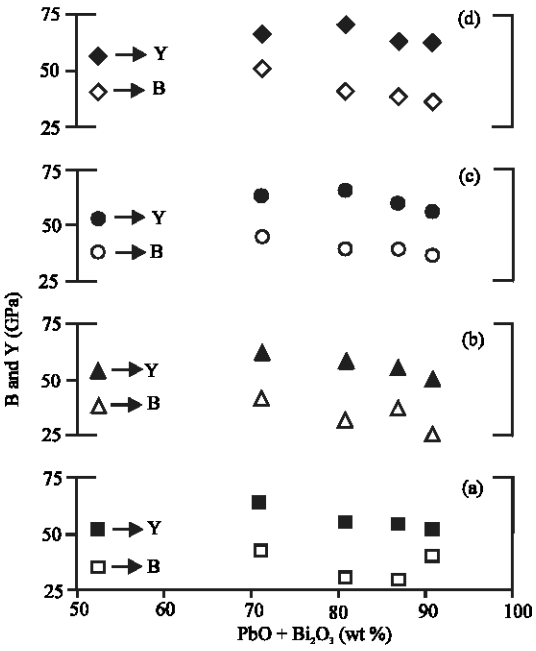

Fig. 3: Bulk and Young's modulus of glasses, where (a) A1-A5, (b) B1-B5, (c) C1-C5 and (d) D1-D5

Here, the value shows reverse changes and trend as compared with those of other elastic moduli. The values of Poisson's ratio for the studied glasses are shown in Fig. 4. It can be seen, for A1 to A5 series as an example, the Poisson's ratio decreases from 0.26 to 0.20 before it increases to 0.29.

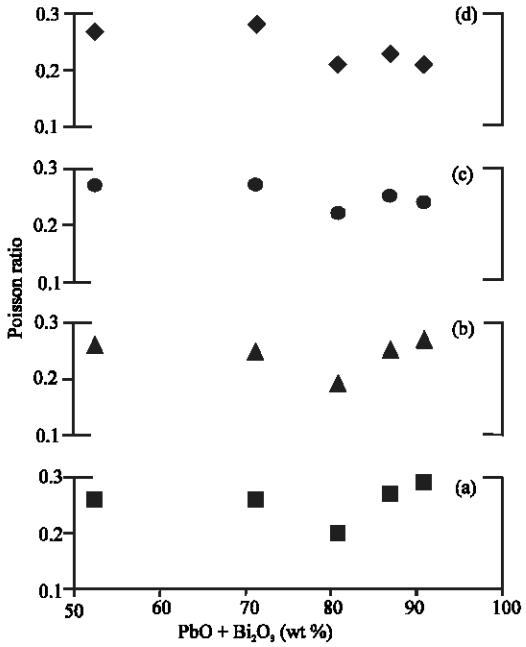

Fig. 4: Poisson's ratio of glasses, where (a) A1-A5, (b) B1-B5, (c) C1-C5 and (d) D1-D5

As mentioned earlier, existence of PbO and the nature of Bi_2O_3 not only cause a sudden increase in the elastic moduli, but also a decrease in Poisson's ratio at one stage. From the data obtained, the turning point of this changes occurred at B_2O_3 values of 19.1% and this happen for every glass series. The variation of Poisson's ratio with composition should be exactly the reverse of elastic moduli variation (El-Adawy and Moustafa, 1999). In this work, the Poisson's ratio would fall steeply (at B_2O_3 values of 47.6 to 19.1%) where the rate of increase of cross-link density with Bi content is high, as most of the Bi^{3+} (octahedral) form with its high cross-link density (El-Adawy and Moustafa, 1999).

Based on network forming groups, all the glass samples have a connectivity of 3 (crosslink density of 1) and Poisson's ratio ~ 3 which is typical for the of As_2O_3, B_2O_3 and P_2O_5 glasses (Higazy and Bridge, 1985). Crosslink densities here defined as the number of bridging bonds per cation less 2.

Table 1 shows that the transition temperature of every series decreased as PbO and Bi_2O_3 content increased. This is mainly due to the addition Bi_2O_3 which weaken the bond between each atom in sample (increases the number of NBOs atoms). The bond getting easier to break and hence the T_g of the sample decreased. For an example, the glass transition for A1 to A5 samples decreased from 470.18 to 376.87°C and for B1 to B5 samples the glass transition dropped from 469.25 to 414.42°C. It was found that approximately 1.06 to 9.74% and 1.67 to 5.65% decreased for each samples in both series.

The current finding is comparable to the thermal properties of the Bi_2O_3-B_2O_3 glass measured by Becker (2003); the variation in glass transition temperature with glass composition is caused by the subsequent formation of dipentaborate units with increasing amounts of Bi_2O_3.

The decrease of Bi_2O_3 concentration in borate glasses lead to a decrease in density and an increase in glass transition temperature (Khanna *et al*., 2003). It was suggested that the following structural transformations (Hung *et al*., 1993):

$$B\emptyset_4^- \Rightarrow B\emptyset_2O^- \quad (7)$$

where, Ø represents a bridging oxygen and O^- a non-bridging oxygen, take place slowly in the borate melt and are responsible for the change in various glass properties. $B\emptyset_2O^-$ units are estimated to have volume about 1.70 times more than that of $B\emptyset_4^-$ unit by the NMR studies carried out by Karki (1987). Meanwhile, if the breaking up of boron tetrahedral units, $[B\emptyset_4]^-$, into a triangular boron units, containing two bridging and one NBO, $B\emptyset_2O^-$, had been the only transformation occurring in the melt, the glass transition temperature should have decreased drastically (Martin and Angell, 1984).

It was also found that, Li^+ or Na^+ were stabilized around tetrahedral boron units, $[B\emptyset_4]^-$, while Pb^{2+}, Bi^{3+} and Sn^{2+} were stabilized around $B\emptyset_2O^-$ (Khanna *et al*., 2003). This fact has been supported by NMR and X-ray photoelectron spectroscopic studies on SnO-B_2O_3 glasses by Hayashi (2002). Thus, tetrahedral boron unit are inherently unstable in the presence of ions like Pb^{2+} and Bi^{3+} and break into much larger metaborate triangular unit, $B\emptyset_2O^-$ (Khanna *et al*., 2003), which in result creating more NBOs.

Furthermore, the study on the thermal properties of glass system revealed that T_g is dependent on the strength of chemical bond in the glass structure (Nitta *et al*., 2001). In general, alkaline metal oxides play a role of network modifier in silicate and borate glasses and NBOs increase with an increasing of alkaline metal oxides in these glasses. It is widely known that the bond between alkaline atom and oxygen is an ionic bond which is in general weaker than covalent bond. Hence, T_g (Table 1) decrease with increasing alkaline oxide content.

CONCLUSIONS

New ternary bismuth based glass samples were successfully prepared and their glassy natures were

determined using the XRD method. Based on the result obtained, it demonstrated that the density and molar volume increase with glass modifier content, which more attributed to the replacement of Bi_2O_3 and PbO; both had larger density and molar volume than B_2O_3 in glass networks.

The observed higher values in velocity at low modifier content and low values at high modifier content for this glass types, confirmed a substantial change in glass structure. Additional increment of Bi_2O_3 and PbO, causing more discontinuity and, hence, decrease in rigidity and velocity results. Meanwhile, there was a similar pattern in elastic moduli with the increasing of PbO and Bi_2O_3 weight content, where the values increased at the earlier stage and then decreased subsequently.

For the thermal properties of glass, we found in lead-bismuth borate glasses, the T_g of every glass series was decreased with increasing content of PbO and Bi_2O_3, which probably due to slightly change in the glass structure.

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