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STRUCTURAL AND MECHANICAL CHARACTERIZATION OF ZNF2–TEO2–B2O3–SM2O3 GLASSES

Akshatha Wagh[1], Raviprakash Y.[1], Sudha D. Kamath*[1]
1Department of Physics,
Manipal Institute of Technology
, Manipal University, Manipal-576104,
Karnataka, India.
*sudhakamath6@gmail.com

***Abstract*— Rare earth samarium doped zinc borate glass system based on 30 ZnF2 – 20 TeO2 – (50-x) B2O3 – x Sm2O3, (where x = 0, 0.5, 1.0, 1.5, 2.0, 2.5 mol%) were prepared. The density of each glass was measured and the molar volume was calculated. Makishima and Mackenzie (M-M) models gave reasonable estimation for different constants of elasticity (Young's modulus (E), Bulk modulus (K), Shear modulus (S), Poisson's ratio (ϑ)). Vickers (Hv) indentation tests were performed on the series of glass samples. These connections were used to relate the conventional Hardness (H). Fracture toughness (KIC), Dissociation energy (Gi) and Packing density (Vt) were also investigated. The relationship between Machinability and Brittleness of glass materials were studied. Exposition of the M-M model proved to be good for the constants of elasticity of ZnF2–TeO2–B2O3–Sm2O3 glasses..**

***Index Terms*— Vickers indentation, Young's modulus, Makishima and Mackenzie, Brittleness**

I. INTRODUCTION

Glasses containing rare earth ions have attracted considerable attention because of their wide applications in laser, up-conversion fluorescence, high density optical storage, solar concentrators, wave-guide lasers and in other photo-electronic fields [1-2]. The fluoride glasses possess high elasticity and its best attribute is that these glasses have a low optical attenuation. These rare earth doped fluoroborate glasses with various visible emissions will be useful in developing new light sources, display devices, UV sensors and tunable visible lasers [1]. Oxyfluoride glasses are of interest from the fundamental point of view: the replacement of fluorine by oxygen affects the glass formation and the structure of glass networks, namely, their connectivity. The oxyfluoride glass matrix can provide a unique environment for rare earth ions, which will differ from both the oxide and fluoride matrices. It is expected that oxyfluoride glasses with high content of rare earth elements can appear to be new functional materials [1]. Transparent oxyfluoroborate rare earth doped glasses are interesting class of materials, which combine the optical advantages of fluoride host with the mechanical advantageous of oxide glass plus highest glass formation tendency of borate atoms.

Glasses based on boron oxide (B_2O_3) are of great technological importance because of the wide range of their applicability. This range varied between glasses for lightening, cookware, medical and LCD screens to glass wool for thermal and acoustic insulation, textile fibers for the reinforcement of plastic, ceramics glazes (for example tiles and tableware) and optical glasses[3]. Increasing interest in new glass design has heightened the need for pre-estimation of properties suitable for their use. Mechanical properties like Elastic modulus, Fracture toughness, Hardness and Brittleness are among the interesting parameters when designing glass compositions [4]. Studies of the elastic constants of glasses gave valuable information about the structure of non-crystalline solids since they are directly related to the inter-atomic forces and potentials [1]. Makishima and Mackenzie have proposed a model to calculate the Elastic modulus of glasses based on their chemical composition, i.e., the constituent compounds from which the glass is assumed to be made.

Most previous works on borate glasses are concentrated to study the structure, electrical and thermal properties. The single bond strength of the B - O (809 kJ/mol) is considered to be very high, but boron ions are coordinated by three oxygen in B_2O_3 glass. Thus, pure B_2O_3 glass has low elastic modulus (~20 GPa) and low T_g (glass transition temperature),

indicating that plastic deformation may take place easily under high stress. As other cations are introduced into B_2O_3 glass, boron ions change their coordination number from three to four causing an increase in Young's modulus [5].
Samarium (Sm) containing glasses are known to have an unusual elastic behaviour due to valance instability. It is a rare-earth element that has a stronger affinity towards oxygen than most of the transition metals. It is thus reasonably expected that Sm may act as an oxygen scavenger and help to suppress heterogeneous nucleation, and consequently improve the glass forming ability of zinc fluoroborate based glasses. On the other hand, Sm is of relatively large atomic size, which is in favour to improve the glass forming ability [6].

Based on the aforementioned aspects, the present paper aims to study the role of Sm_2O_3 (0, 0.5, 1.0, 1.5, 2.0, 2.5 mol %) on the constants of elasticity of zinc fluoroborate glasses through theoretical analysis. The density and molar volume of the glass samples were determined. The calculation of the constants of elasticity (Young's modulus, Bulk modulus, Shear modulus, Packing density, Poisson's ratio) is based on the Makishima-Mackenzie model. Fracture toughness, Brittleness, Hardness, Cutting energy and Machinability parameter of the samples were also studied.

1. Experimental

Glasses in the 30 ZnF_2 – 20 TeO_2 · (a) $_2O_3$ – x Sm_2O_3 system were fabricated by the conventional melt-quenching technique. The compounds were chemically pure reagents (99 %+) from Sigma Aldrich. The mixture was thoroughly mixed in an agate mortar - pestle and transferred into a porcelain crucible. The homogenous mixture in porcelain crucible is placed in an electric furnace set at 980 ^{0}C for 1 Hr 30 min. The melt was then poured into a stainless steel mould. The casted samples were annealed near the glass transition temperature (~300 ^{0}C) for 2 hours and then they were cooled to room temperature. The annealed samples were polished to have a good parallelism faces to be suitable for the mechanical properties to be studied.
The density was measured by Archimedes method using Xylene as an immersion liquid. The molar volume was calculated according to $V_m = M/\rho$, where M is the molecular weight of the glass sample and ρ its density.

2. Results and Discussion

3.1. Structural Analysis

The measurement of density (ρ) and refractive index (n) are the effective tools to explore the degree of structural compactness modification of the geometrical configurations of the glass network [2]. The density and refractive index of the glass samples determined in the present study are given in the Table 1 with probable errors ±0.001.The molar volume (V_m) of the glass samples, was calculated using the average molecular weight (M) and density (ρ) with the following relation:

$$V_m = M/\rho$$

The number density of rare – earth ions (N) of Sm^{3+} was determined using the formula given in equation [6]

$$N = [6.023 \times 10^{23} \times \text{mol\% of cation} \times \text{valency of cation}]/V_m$$

The dielectric constant (ε) was calculated using
refractive index (n) of the glass.

$$E = n^2$$

The Molar Refraction, (R_m) of the glass samples were calculated using the formula given [7] which is well – known as Volf and Lorentz-Lorenz formula,

$$R_m = \{[(n^2 - 1)/ (n^2 + 2)] * V_m$$

Where n is the refractive index of the glass sample. ρ is the density and M is the average molecular weight of the glass samples. V_m is molar volume (V_m), and $(n^2 - 1)/ (n^2 + 2)$ is the reflection loss.

A condition for predicting metallic or insulating behavior in the condensed state matter is metallization criterion, $M = 1 - [R_m/V_m]$. If $R_m/V_m > 1$, then the materials show metallic nature and if $R_m/V_m < 1$ they exhibit insulating behavior [8]. The so-called metallization parameter values of the present glasses are found to be less than one and are given in Table 1. Hence, the present glass systems with their metallization parameter values should exhibit insulating nature.
The molar refraction is related to the structure of the glass and it is proportion to the Molar Electronic Polarizability of the material (α_m) [7]. According to the Clausius-Mosotti relation [7], molar polarizability of the materials ($\alpha_m * 10^{-24}$ cm^3) is given by the relation

$$\alpha_m = [3 / 4* \pi * A_v] * R_m$$

Where A_v is the Avogadro's number.

The electronic polarizability of oxide ions (α_O^{2-}) can be calculated on the basis of refractive indices using the following equation,

$$\alpha_O^{2-}(n) = [(R_m / 2.52) - \sum \alpha_i] * (N_O^{2-})^{-1}$$

Where $\sum \alpha_i$ in the above equation is molar cation polarizability and N_O^{2-} is the number of oxide ions in the chemical formula. For the studied glasses the values of N_O^{2-} is equal to 1.9. The value of $\alpha_{Zn} = 0.286$ Å^3 for Zn^{2+} ions, $\alpha_{Te} = 1.595$ Å^3 for Te ions, $\alpha_B = 0.003$ Å^3 for B^{3+} and $\alpha_{Sm} = 0.92$ Å^3 for Sm^{3+} ions [8].

The obtained values of N are used to calculate the polaron radius (r_p), ionic radius (r_i) [9,10] and field strength (F) using the relations:

$$r_p = \frac{1}{2}\left[\frac{\pi}{6N}\right]^{(1/3)} \qquad r_i = \left[\frac{1}{N}\right]^{(1/3)} \qquad \text{and} \qquad F = Z/(r_i)^2$$

It is evident from Table 1 that the density, molar volume, number density of the samarium ions, the electronic oxide ion polarizability of the present glass system increases with increase in the content of Sm_2O_3.The dielectric constant is directly correlated with the polarizability of the glass. The dielectric constant gradually increases with increase in the Sm_2O_3 content in the glasses.

The chemical compositions of the constituent elements for the thermally evaporated glass sample (where x = 2.5 mol %) have been investigated by means of an energy dispersive X-ray analysis [EDAX] unit interfaced with scanning electron microscope [SEM]. Our visual observation, the scanning electron microscope picture of the 2.5 % Sm_2O_3 doped glass shown if Figure 1(a), indicates the amorphous nature of the glass. Figure 2 (b) shows the EDAX spectrum for 2.5 mol% representative sample. The obtained data showed the glass chemicals like Sm, Te, Zn, O and B showing deficiency in Fluorine (F). The deficiency of F may be due to its low melting temperature and there by evaporating after 250°C which is clearly visible in the EDAX spectrum in Figure 1(b)

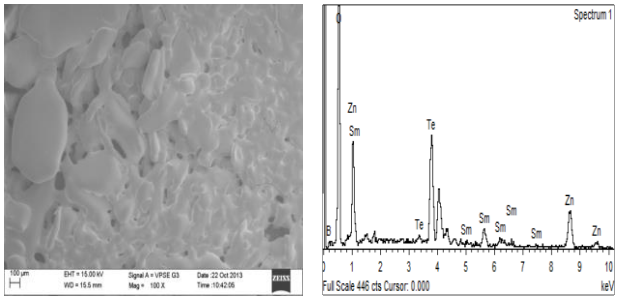

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Figure 1: (a) SEM image and (b) EDAX spectrum obtained from 2.5 mol% Sm_2O_3 content in the glass

Table 1: Physical parameters of the glass system 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3.

Physical Parameter	Glass code					
	Sm_0	Sm_1	Sm_2	Sm_3	Sm_4	Sm_5
Average Molecular Weight (g/mole)	97.7	99.1	100.5	101.9	103.3	104.7
Density (ρ) (g/cm^3) (±0.0001)	3.7240	3.7494	3.7860	3.8466	3.8903	3.9328
Refractive index (n) (±0.0001)	1.4510	1.4575	1.4610	1.4615	1.4620	1.4630
Optical dielectric constant (ε)	2.1054	2.1243	2.1345	2.1359	2.1374	2.1403
Molar volume (V_m) (cm^3/mol) (±0.01)	26.23	26.33	26.43	26.49	26.55	26.62
Molar refractivity (R_m) (cm^3/mol)	7.06	7.17	7.26	7.27	7.30	7.33
(1 - R_m/ V_m)	0.730	0.727	0.725	0.725	0.725	0.724
Number density (N) (*10^{20}/cm^3)	---	3.43	6.83	10.23	10.36	16.97
Inter ionic distance (r_i) (A^0)	---	14.29	11.34	9.92	9.88	8.38
Polaron radius (r_p) (A^0)	---	5.75	4.57	4.00	3.98	3.37
Field strength (F) (*10^{15} cm^{-2})	---	3.04	4.82	6.30	6.35	8.82
Electronic Polarizability (α_e^{2-} (n)) (Å^3)	1.474	1.497	1.516	1.518	1.525	1.531
Molar Electronic polarizability (α_m)	2.798	2.848	2.878	2.882	2.893	2.905
(*10^{-24} ions/cm^3)						

Table 1: Physical parameters of the glass system 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3.

The composition (mol%), density (ρ) and molar volume (V_m) of the present glasses which were designed as Sm_0, Sm_1,……,Sm_5 are given in Table 1. The variation of density and molar volume with the different concentration (mol%) of Sm_2O_3 is shown in Figure 2.

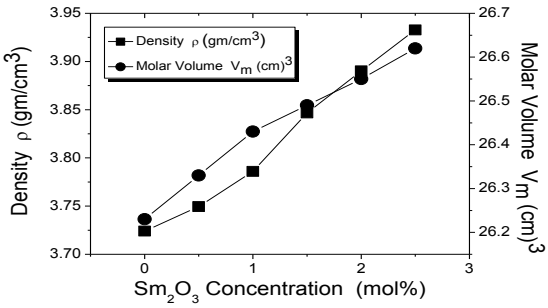

Figure 2: The variation of density and molar volume

Various types of continuous indentation tests have come into general use for the determination of mechanical properties of materials. The indentation method is preferred because relatively small amounts of testing material are needed and there are no strict requirements for the shape of the samples, moreover the measurements can be performed without the destruction of the samples. For these investigations a wide variety of testing devices were developed with indenters of various forms working in a scale from the nanoindentation to macrohardness region. The common feature of these tests is that the applied load is registered as a function of indentation depth during both the loading and unloading period [11].

Micro indentation experiments were carried out on series of glass samples for evaluation of Hardness (H) and Fracture toughness (K_{IC}). SEM micrograph of the indenter impression which produced cracks is depicted in Figure 3. Radial cracks were propagated along the indenter corners in half penny configuration. The crack length (C) gives the resistance of the sample to fracture. Various models were developed to estimate K_{IC}. The following equation which has been standardized for ceramics, glasses and glass-ceramics is used to estimate the K_{IC} of the glasses [12],

$$K_{IC} = 0.018 \ (E/H)^{1/2} (P/C^{3/2})$$

Where P is the applied load, E and H are Young's modulus and Hardness of the material respectively. H is obtained from Vickers micro-hardness tester through H_v values obtained from the same (Load: 25 gf, Dwell time: 10s, Objective: 400x, Magnification: 40x and Eyepiece: 10x).

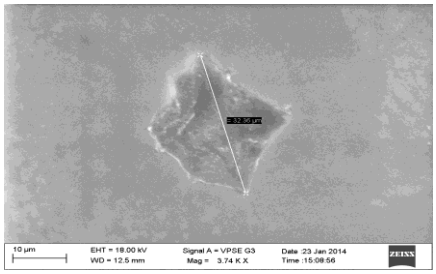

Figure 3: SEM image of indentation cracks produced along the corners of Vickers indenter

In the present study the elastic moduli was calculated by applying the Makishima and Mackenzie's theory. The Young's modulus of a crystalline oxide is given by the following

$$E = 2\alpha U / r_o^3$$

Where α is the Madelung constant, U is the attraction electrostatic energy and r_o is the interatomic distance.

In the glass, because of the disorder structure, it is difficult to get a meaningful Madelung constant as for a crystalline oxide. Makishima and Mackenzie proposed to take the dissociation energy of oxides per unit volume, G_t, instead of the Madelung energy multiplied by dimensionless term, relative to the packing density factor, V_t. Therefore, Young's modulus may then be written as follows [2,5],

$$E = 2 G_t V_t = 2 \frac{\rho}{M} \sum V_i x_i \sum G_i x_i$$

The factor G_t can be obtained from the dissociation energy per unit volume,

$$G_i = (\rho_i / M_i) [X \Delta H_f (M, gas) + Y \Delta H_f (O, gas) - \Delta H_f (M_xO_y \text{ crystal}) - (X + Y) RT]$$

ρ_i and M_i are the density and molecular weight of the oxide, i, respectively, and $\Delta_f H$; the molar heat of formation from their standard states of the oxide and gaseous atoms [2]. R is the gas constant and T is the room temperature (273 +25^{0}C=298 K). For the present glass system the dissociation energy of the ZnF_2, TeO_2, B_2O_3 and Sm_2O_3 is obtained from the Hess's cycle, by knowing the enthalpies of each component [(Zn: 130.40 kJ/mol, F: 79.38 kJ/mol, ZnF_2: 40 kJ/mol) (Te:196.6 kJ/mol, O: 249.229 kJ/mol, TeO_2: 28.9 kJ/mol) (B:565 kJ/mol, O:249.229 kJ/mol, B_2O_3:24.56 kJ/mol) (Sm:206.7 kJ/mol, O:249.229 kJ/mol, Sm_2O_3:119 kJ/mol)] [11,12]; Calculated G_i values of each glass system are given in the Table 2. In the multi component glass, if x_i is the molar fraction of the component i, then:

$$G_t = \sum G_i x_i$$

In order to calculate the V_t factor of an oxide M_xO_y glass, it is necessary to calculate firstly the individual density factor, V_i factor of each oxide linked to the packing compactness is :

$$V_i = (4\pi N/3) (x r_M^3 + y r_O^3)$$

Where N is the Avogadro number, r_M and r_O the ionic radii of the cation and anion respectively. Hence, the total packing density factor is given by:

$$V_t = (\rho / M) \sum V_i x_i$$

Table 2: Volume unit (V_i) and dissociation energy per unit volume (G_i) of each component.

Compound	V_i [(*10^{-6}) (m^3)]	$\Delta_f H$ (kJ/mol)	G_i (kJ/mol/m^3)
ZnF_2	14.89	40	12.28
TeO_2	15.49	28.9	23.93
B_2O_3	20.80	24.56	65.93
Sm_2O_3	16.98	119	25.25

The variations of total packing density factor (V_t) with the mol% of Sm_2O_3 is shown in Figure 4. With the spread in the atomic arrangements or the spread in particle size the packing density decreases with increase in Sm^{3+} concentration. For the present glass system volume unit (V_i) and the dissociation energy (G_i) of ZnF_2, TeO_2, B_2O_3 and Sm_2O_3 are obtained from the Hess's cycle [11], by knowing the enthalpies of each component which are given in Table 2.

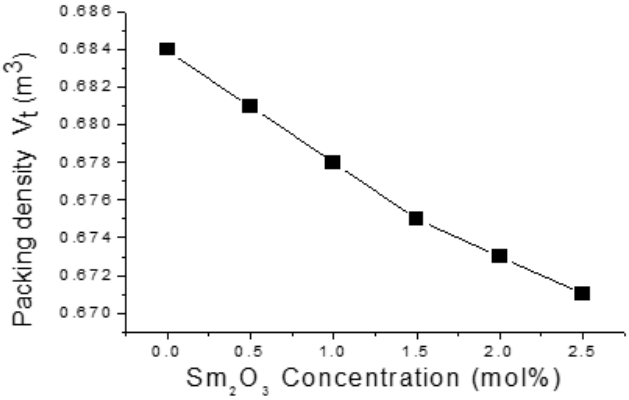

Figure 4: The variations of total packing density factor (Vt) with the different concentration (mol%) of Sm2O3

Makishima and Mackenzie found a good linearity between the bulk modulus, packing density and Young's modulus of the 30 different glasses. The slope found through the interpretation was 1.2 and the bulk modulus of glass was expressed semi-empirically as follows:

$$K = 1.2\ V_t\ E = 2.4\ V_t^2\ G$$

Shear modulus (S) and Poisson's ratio (ϑ) are shown by the following well known equations [10]:

$$S = 3EK / (9K - E)$$

$$\vartheta = E / (2S - 1)$$

Calculated Young's modulus (E), Bulk modulus (K), Shear modulus (S) and Poisson's ratio (ϑ) of the 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3 glasses are given in Table 3. Variation of Young's modulus (E), Shear modulus (S) and Bulk modulus (K) versus Sm_2O_3 concentration and variation of Fracture toughness (K_{IC}), Brittleness (B) and Hardness (H) versus Sm_2O_3 concentration is shown in Figure 5 (a) and 5 (b) respectively.

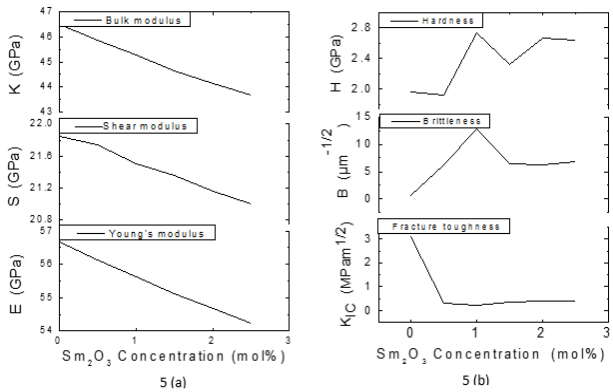

Figure 5: (a) Variation of Young's modulus (E), Shear modulus (S) and Bulk modulus (K) versus Sm_2O_3 concentration. (b) Variation of Fracture toughness (K_{IC}), Brittleness (B) and

Table 3: Calculated Young's modulus (E), Bulk modulus (K), Shear modulus (S) and Poisson's ratio (ϑ) of the 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3 glasses.

Sample	G_t (*10^9)	V_t (m^3)	E (GPa)	K (GPa)	S (GPa)	ϑ
Sm_0	41.435	0.684	56.68	46.52	21.85	0.297
Sm_1	41.231	0.681	56.15	45.88	21.74	0.291
Sm_2	40.020	0.678	55.63	45.26	21.50	0.293
Sm_3	40.824	0.675	55.11	44.63	21.36	0.290
Sm_4	40.621	0.673	54.67	44.15	21.16	0.291
Sm_5	40.418	0.671	54.24	43.67	21.00	0.291

Although machinability can be simply assessed qualitatively as the ease with which a given material is cut; its accurate quantitative measurement is difficult. Various parameters have been suggested as the 'measurement' of the machinability, depending on the testing conditions employed, including tool wear, surface roughness, cutting force, cutting energy, drilling rates etc. These parameters will depend strongly on the microstructure and properties of the sample. In particular, fracture strength, hardness and fracture toughness have been considered for the prediction of the machinability of glass.

In this contribution the use of a brittleness index is suggested for estimating the machinability of the glass sample. Brittleness is a measure of the relative susceptibility of a material to deformation and fracture, relating Hardness (H), which quantifies the resistance to deformation and toughness (K_{IC}), which quantifies to resistance to fracture. On the basis of an indentation-mechanics analysis, the ratio of hardness to toughness has been proposed as a simple index of brittleness, B = (H / K_{IC}) and the general applicability of B for determining the question of ductility (deformation) versus fracture has been suggested. The value of B varies between ~ 0.1 $\mu m^{-1/2}$ for steels and ~17 $\mu m^{-1/2}$ for Si monocrystal, with values for glasses and ceramics varying in general within the range 3 – 9 $\mu m^{-1/2}$.

Since machinability involves deformation and micro-fracture, the brittleness index, combing the response of the material to both of these phenomena, should be better parameter for tis quantification than either hardness or fracture toughness taken separately. Under this premise, samples with good machinability should behave in a less brittle manner, i.e., the index B should assume a low value.

Two parameters were proposed for evaluating the machinability; (i) n, the slope of the log-log plot of the specific cutting energy versus cutting rate, and (ii) u_1, the specific cutting energy at low cutting rate, neglecting elastic impact effects. It was shown that good machinability is related to positive values of n, whilst negative values of n represent the predominance of brittle fracture along the glass phase over the continuous delamination through mica crystals, resulting in poor machinability. In the paper alluded to the machinability

parameters n and u_1 were fitted to the hardness of the material H to give the following relationships:

$$n = 0.643 – 0.122H$$

$$u_1 = k\ H^{2.25}$$

where k is a proportionality constant.

Calculated Hardness (H), Brittleness (B), parameter of machinability (n) and cutting energy (u_1) of the 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3 glasses are tabulated in Table 4. The correlation between n and H, and between u_1 and H were, however poor. Indeed, a better result is obtained by the fitted equations in this case are [13]:

$$n = 0.457 – 0.106\ B$$

$$u_1 = 0.23\ B^{1.55}$$

Table 4: Calculated Hardness (H), Brittleness (B), parameter of machinability (n) and cutting energy (u_1) of the 30 ZnF_2 – 20 TeO_2 – (50-x) B_2O_3 – x Sm_2O_3 glasses

Sample	H (GPa)	K_{IC} (MPam$^{1/2}$)	B ($\mu m^{-1/2}$)	n	u_1
Sm_0	1.964	3.112	0.631	0.390	0.112
Sm_1	1.922	0.311	6.180	-0.198	3.780
Sm_2	2.739	0.214	12.799	-0.899	11.963
Sm_3	2.320	0.362	6.408	-0.222	4.093
Sm_4	2.669	0.427	6.250	-0.205	3.938
Sm_5	2.640	0.390	6.769	-0.260	4.456

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3. Conclusion

V_m is found to be increasing with gradual increase in density with increased Sm^{3+} concentration in the glass matrix. This is due to the presence of $Sm3^+$ ions that results in the expansion of the network to adjust within small interstitial sites (of B_2O_3 network) thereby increasing the molar volume. In the present glass system, Sm^{3+} ions may be acting as a network modifier that creates two non-bridging oxygen (NBO) by breaking the B-O-B network and results in converting some of B_2O_3 into BO_4 units. In borate glasses, fractions of tetrahedral [BO_4] as well as trigonal [BO_3] structural units influence the properties. This model depends on the dissociation energy of each type of bonding inside the network structure and the packing factor of each constituent compound. Any modification in the network during glass formation may lead to a deviation in the estimated Young's modulus. Increasing the cation size in the glass network caused a decrease in Young's modulus, which can be associated with weaker bond linkages and lower packing density in the respective glass networks. The increasing ionic radius and decreasing field strength of a cation lead to a decrease in the packing density of the glass compositions. Therefore, the decrease in the packing density of the Sm^{3+} doped glass system leads to a lower Young's modulus. This result reflected in the Bulk modulus, Shear modulus and the Poisson's ratio as well.

The brittleness index (B) is preferred over Hardness (H) or fracture toughness (K_{IC}) in order to predict the Machinability parameter (n) of the glass sample. The good machinability occurs when n > 0 and brittleness index of the material is lower than B ~ 4.3 $\mu m^{-1/2}$. The results prove that the Sm^{3+} doped glasses are having larger fracture toughness indirectly increasing the brittleness of the samples.

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