

Study of Physical Properties of Lithium-borosilicate glasses

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Abstract:- Glass samples of the Lithium borosilicate were prepared by conventional melt-quench technique. The density and molar volume of some silicate glasses were determined in order to study their structure. Molar volume decreases linearly as mole % of SiO₂ increases. The physical parameters like Oxygen packing density, Ionic concentration, Inter ionic distance, Polaron radius, Dielectric constant, Reflection loss, Band energy gap, Refractive index, Molar refractivity, Molar polarizability, Metallization, Electronic polarizability and Conductivity are studied. Oxygen packing density is decreasing with increasing the concentration of SiO₂. Ionic concentration increases, where as inter ionic distance, polaron radius, decreases with increasing the concentration of SiO₂. Molar polarizability, Molar refractivity, Electronic polarizability varies alike with increasing mole % of SiO₂ respectively. Maximum values at 5 mol % of SiO₂ are 7.671×10^{24} ions/cm³, 19.348 cm³ and 0.33×10^{24} ions/cm³. Electronic polarizability, Reflection loss are varies similarly while Metallization is exactly opposite to them; at 5 mol % of SiO₂ Reflection loss is 0.359 and Metallization is 0.166. Conductivity increases according to glass transition temperature. Mole % of SiO₂ and ionic concentration varies linearly with slope 0.0222, intercept 10.9 and R² 0.999. Other linear variations are also studied.

Keywords:- Density, Molar volume, Oxygen packing density, Dielectric constant, Refractive index, Molar refractivity, Molar polarizability; Metallization, Eelectronic polarizability, Molar refractivity, Molar polarizability, Polarizability per unit volume, glass composition, mixed glass former effect.

I. INTRODUCTION

Oxide glasses are classically described as a network composed by building entities such as SiO₂, B₂O₃, P₂O₅, TeO₂ and modifiers such as alkaline oxides: Li₂O, Na₂O, K₂O, Ag₂O or alkaline earth oxides: CaO, MgO, SrO^[1, 2]. In such glasses, the oxygen from the metal oxide becomes part of the covalent glass network, creating new structural units. The cations of the modifier oxide are generally present in the neighborhood of the non-bridging oxygen (NBO) in the glass structure. The extent of the network modification obviously depends on the concentration of the modifier oxide present in the glass. A glass network affects various physical properties such as density, molar volume, glass transition temperature & polarization, etc.

The mixed glass former (MGF) and mixed glass former effect (MGFE) is defined as a nonlinear and non-additive change in the ionic conductivity with changing glass former composition at constant modifier composition. Lithium borosilicate glasses are characterized by an interesting structure on account of the presence of two glass-forming components.

Density of solids is mostly the simplest physical property that can be measured. However, it would be a highly informative property if the structure of material could be well defined. Density can be used for finding out the structure of different types of glasses. The density of the glass is additive and can thus be calculated on the basis of the glass composition

^[3,4,5,6]. Several formulas have been derived to correlate the glass density to the glass composition^[7, 8, 9, 10, 11]. The glass structure can be explained in terms of molar volume rather than density, as the former deals the spatial distribution of the ions forming that structure. The change in the molar volume with the molar composition of an oxide indicates the preceding structural changes through a formation or modification process in the glass network^[12, 13].

The density, molar volume and packing fraction^[14, 15, 16] could be directly related to the short range structure of alkali oxide modified borate glasses. The densities prove changes in both short range order and coordination as the modification, while the molar volume is sensible in terms of size and packing. The packing of the borate based glasses with ions having volume smaller than the oxygen is considered to be covalent, controlled by oxygen covalent network, and heavily dependent on the glass former.

The molar refractivity^[17, 18, 19, 20, 21, 22], is a constitutive-additive property which represents the real volume of the molecules. That is calculated by the Lorenz-Lorentz formula. Electric polarizability^[17-23], is the relative tendency of a charge distribution, like the electron cloud of an atom or molecule, to be distorted from its

normal shape by an external electric field; similarly ionic concentration^[17,22,23], inter ionic distance^[17,22]. When an electron in the conduction band of a crystalline insulator or semiconductor polarizes or otherwise deforms the lattice in its vicinity. The polaron comprises the electron plus its surrounding lattice deformation. (Polarons can also be formed from holes in the valence band.) If the deformation extends over many lattice sites, the polaron is “large,” and the lattice can be treated as a continuum. Charge carriers inducing strongly localized lattice distortions form “small” polarons^[22]. In present work, these parameters have studied to explain structural features.

II. EXPERIMENTAL TECHNIQUES

2.1. Preparation of Glasses

The lithium-borosilicate glass samples having the general chemical formula $42.5\text{Li}_2\text{O}\cdot(57.5-x)\text{B}_2\text{O}_3-x\text{SiO}_2$, ($x=0-57.5$ in the step of 5, 7.5, 10, 20) were prepared by conventional melt-quench technique from high-purity reagent grade Li_2CO_3 , H_3BO_3 and SiO_2 . The raw materials were mixed up in the desired proportions and thoroughly ground in an agate mortar. The mixtures were fired in air in a porcelain crucible in the following manner:

- Reaching 500°C and keeping steady at this temperature for 30 min to allow the evaporation of water from the powders to be as complete as possible;
- Reaching 1100°C depending on composition. 4-5 hrs and keeping steady at this temperature for 30 min to allow a complete homogenization of the melt and attained desirable viscosity it was poured onto metal plate.
- The prepared samples was then annealed at $300-400^\circ\text{C}$ temperature for 2 hrs and then kept in vacuum desiccators to avoid possible moisture absorption before testing.

The prepared glass samples are polished and the surfaces are made perfectly plane and smoothed by 120 No. emery paper. Thickness of the samples has been measured using digital vernier calipers with an accuracy of 0.0001mm .

III. RESULT AND DISCUSSION

3.1 Differential Scanning Calorimetry (DSC)

A typical DSC endothermic curve for a glass as shown in Figure 3.1, a very broad hump corresponding to the glass transition temperature T_g is 421.35°C . It is known that T_g is a measure of structural degradation.

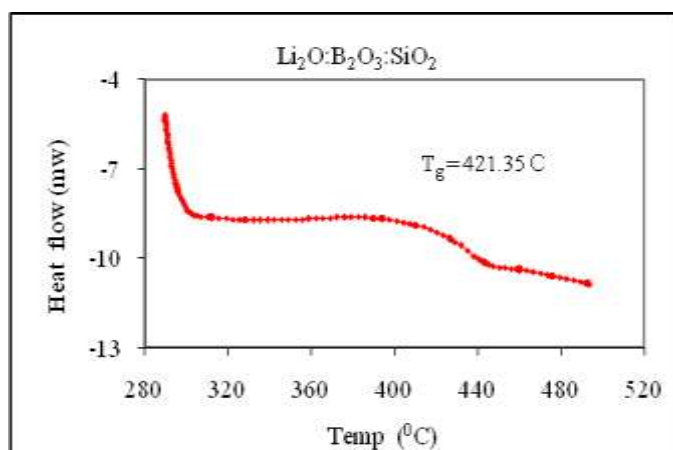


Figure 3.1. DSC thermo grams for LBS.2 glass sample.

The glass transition temperature varies with increasing of the SiO_2 content. Decrease in glass transition temperature is referred to increasing the loose packing and transformation structure and increase in number of non-bridging oxygen items which are assumed to interrupt the B-O bond. The oxygen packing density as a measure of tightness of packing of oxide network can also be explained decrease of glass transition temperature. The nominal composition of glass samples along with their conductivity, activation energy, and glass transition temperature is listed in Table 3.1.

Table 3.1: Composition of glass samples with their nomenclature, conductivity, activation energy, and glass transition temperature.

| Name of samples | Composition in mol % | | | T_g C | σ at 350 C | E_a (eV) |
|-----------------|----------------------|-------------------------------|------------------|---------|-------------------|------------|
| | Li ₂ O | B ₂ O ₃ | SiO ₂ | | | |
| LBS.1 | 42.5 | 57.5 | 0 | 440.35 | 2.6418 e-3 | 0.54 |
| LBS.2 | 42.5 | 52.5 | 5 | 421.35 | 1.8518 e-3 | 0.24 |
| LBS.3 | 42.5 | 47.5 | 10 | 410.5 | 4.1572 e-3 | 0.42 |
| LBS.4 | 42.5 | 37.5 | 20 | 429 | 1.5031 e-3 | 0.33 |
| LBS.5 | 42.5 | 30 | 27.5 | 439 | 2.134 e-3 | 0.5 |
| LBS.6 | 42.5 | 27.5 | 30 | 443.12 | 9.6272 e-4 | 0.31 |
| LBS.7 | 42.5 | 20 | 37.5 | 435.05 | 1.1081 e-3 | 0.58 |
| LBS.8 | 42.5 | 10 | 47.5 | 430.5 | 2.1712 e-3 | 0.51 |
| LBS.9 | 42.5 | 5 | 52.5 | 421.35 | 1.5000 e-3 | 0.52 |
| LBS.10 | 42.5 | 0 | 57.5 | 427 | 2.2656 e-3 | 0.53 |

3.2. X-ray Diffraction

X-ray diffraction pattern of the material gives the identification of the constituent phases present in the material. It gives the information about the state of the combination of the chemical elements present in the mixture and also about the formation of compound if any, as a result of the individual constituents.

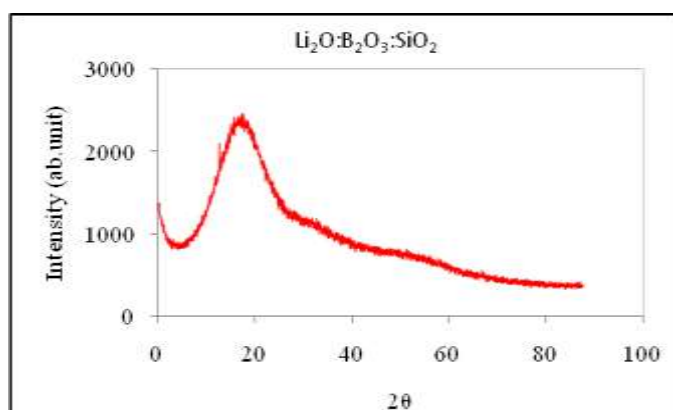


Figure 3.2. X-ray diffraction pattern for, LBS.2 glass sample.

The X-ray diffraction patterns of all the samples under study were recorded by using-Phillips, Holland, X-ray counter diffractometer model PW 1710 employing filtered CuK α radiation ($\lambda=1.5418^\circ\text{A}$). The X-ray diffraction patterns of the entire sample under study were recorded from IUC Indore. A typical XRD curve for a glass as shown in Figure 3.2, from the X-ray diffraction patterns of all the samples under study were recorded as amorphous in nature.

3.3 Optical band gap (E_g) and refractive index (n) studies

Figure 3.3 shows the typical absorption intensity in arbitrary units as a function of wavelength (1/cm) for the glass sample. It is clear from the figure there is no sharp absorption edge and this edge this is the characteristics of glassy nature. In the present work absorption edge in the samples determine by oxygen bond strength in the glass forming network. The quantity $(\alpha hf)^{1/2}$ is plotted as a function of the photon energy (hf) for glass sample is as shown in the Figure 3.4. The straight portion of the graph is then extrapolated to intersect with x axis. The value of hf at the point where $(\alpha hf)^{1/2}$ becomes zero yields a direct measure of the optical band gap energy Altaf^[23]. The optical band gap energy was found to be 2.95eV.

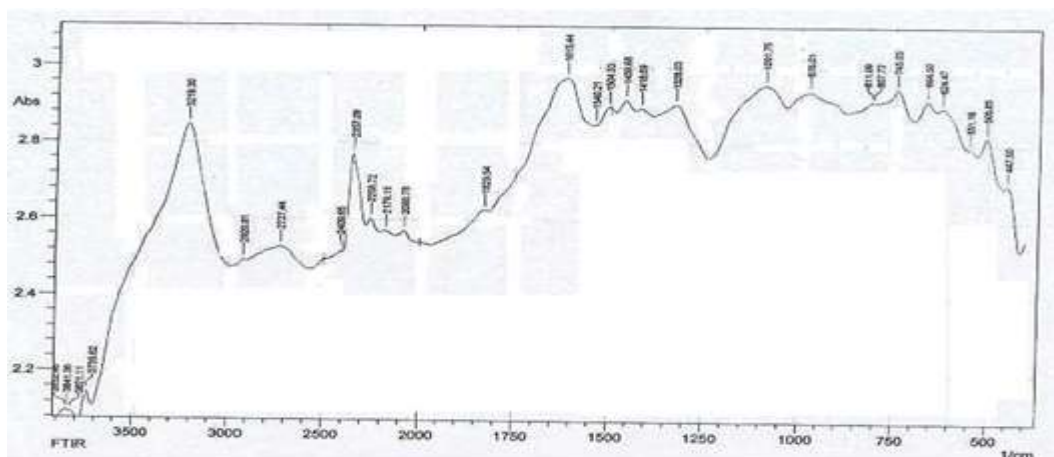
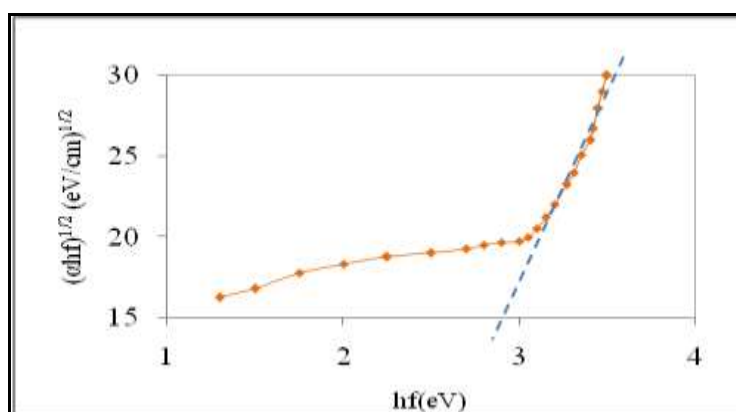


Figure 3.3 Typical UV spectra of the sample.


 Figure 3.4.A representative plot of the quantity $(\alpha hf)^{1/2}$ as a function of the photon energy (hf) .

IV. PHYSICAL PROPERTIES

4.1. Density Measurements

Density of all glass samples are measured at room temperature using xylene as the immersion liquid. Density is generally measured by the fluid displacement method depending on Archimedes principle. According the Archimedes principle, the buoyancy equals the weight of the displaced fluid. Archimedes Principle using xylene as the buoyant medium evaluated the density of the glass samples. The density was obtained by employing the relation:

$$\rho = \frac{W_a \rho_b}{(W_a - W_b)}$$

Where W_a is the weight of glass sample in air, W_b is the weight of glass sample in buoyant liquid, $(W_a - W_b)$ is the buoyancy, ρ_b is density of buoyant. All the measurements were made using a digital balance.

4.2. Molar weight calculation

Step I – Calculation of wt/mole

Weight/mole = molar weight of the constituents * mol% / 100

Step II – Calculation of molecular weight of sample (M)

The molecular weight of the sample (M) is nothing but the summations of Wt/mole of its constituents.

Step III – Calculation of Molar volume (V_m)

Using molecular weight and density calculated as from above, the molar volume of the glass samples can be calculated from following expression:

$$V_m = \frac{M}{\rho}$$

Here, V_m is molar volume, ρ is the density of the sample and M is the molecular weight of the sample.

4.3. Oxygen packing density (O)

The oxygen packing density of the glass samples were calculated using the following relation ^[24]

$$O = n \left(\frac{\rho}{M} \right)$$

where ρ , the density of desired glass samples, M, molecular weight of the sample and n, the number of oxygen atoms in the composition.

4.4. The ionic concentrations (N)

The ionic concentrations of the glass samples are determined using the following relation,

$$N = \left(\frac{6.023 \times 10^{23} \text{ mol}^{-1} * \text{mol\% of cation} * \text{valency of cation}}{V_m} \right)$$

4.5. Inter-ionic distance (R)

Inter ionic distance (R) of the glass samples is given as,

$$R = \left(\frac{1}{N} \right)^{\frac{1}{3}}$$

Where N = ionic concentrations.

Average molecular weight, Density, Molar volume, Oxygen packing density Ionic concentrations and Inter-ionic distance, Parameters values of lithium borophosphate glasses are presented in Table 4.4.

From Table 4.5 and Figures 4.5.1, 4.5.2, shows increasing the mol% of SiO₂ with respect to the B₂O₃ by keeping modifier Li₂O constant, the molar volume, inter ionic distance, polaron radius decreasing, suggests the increased free space within the glass structure, ^[25, 26]; it means that the glass structure becomes loosely packed ^[27]. Oxygen packing density, ionic concentration decreases, because the polaron comprises the electron plus its surrounding lattice deformation. Hence, the deformation extends over many lattice sites and the lattice can be treated as a continuum. Transformation of BO₃ triangle units to BO₄ tetrahedral units can be expected to increase the network linkage of the glass which is reflected in the monotonically increasing density.

Table 4.5 Average molecular weight, Density, Molar volume, Oxygen packing density Ionic concentrations and Inter-ionic distance for Li₂O·B₂O₃-SiO₂ glass system.

| Name of samples | Average molecular weight M (gm/mole) | Density ρ (gm/cm ³) | Molar volume V _m (cm ³ /mole) | Oxygen packing density O (10 ⁻⁶ m ³ /mole) | Ionic Concentration N (10 ²¹ /cm ³) | Inter ionic distance r _i (Å ^o) | Polaron radius r _p (Å ^o) | E _g (eV) |
|-----------------|--------------------------------------|--------------------------------------|---|--|--|---|---|---------------------|
| LBS.1 | 52.731 | 2.249 | 23.447 | 91.698 | 10.918 | 9.712 | 0.182 | 1.152 |
| LBS.2 | 52.254 | 2.25 | 23.224 | 90.423 | 11.022 | 9.681 | 0.181 | 0.556 |
| LBS.3 | 51.778 | 2.25 | 23.012 | 89.083 | 11.124 | 9.651 | 0.181 | 0.954 |
| LBS.4 | 50.824 | 2.249 | 22.599 | 86.289 | 11.327 | 9.593 | 0.179 | 0.934 |
| LBS.5 | 49.87 | 2.25 | 22.165 | 83.466 | 11.549 | 9.531 | 0.178 | 1.112 |
| LBS.6 | 47.248 | 2.251 | 20.99 | 75.036 | 12.195 | 9.36 | 0.175 | 1.01 |
| LBS.7 | 47.725 | 2.25 | 21.211 | 76.611 | 12.068 | 9.393 | 0.176 | 1.088 |
| LBS.8 | 48.202 | 2.25 | 21.423 | 78.187 | 11.949 | 9.424 | 0.176 | 1.116 |
| LBS.9 | 49.155 | 2.251 | 21.837 | 81.284 | 11.722 | 9.484 | 0.177 | 1.044 |
| LBS.10 | 50.109 | 2.25 | 22.271 | 84.192 | 11.494 | 9.546 | 0.179 | 1.186 |

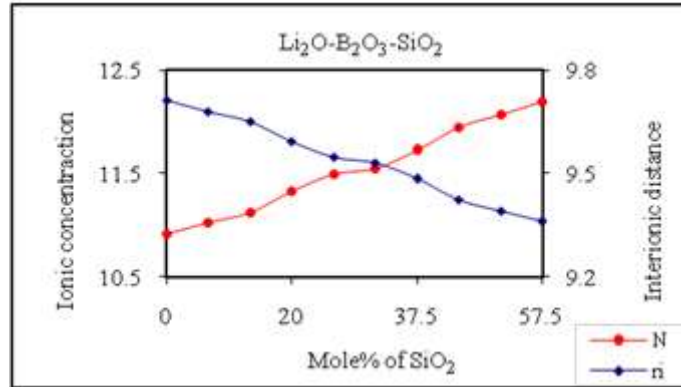


Figure 4.5.1. Variation of Ionic concentration, Inter-ionic distance with respect to mol% of SiO₂.

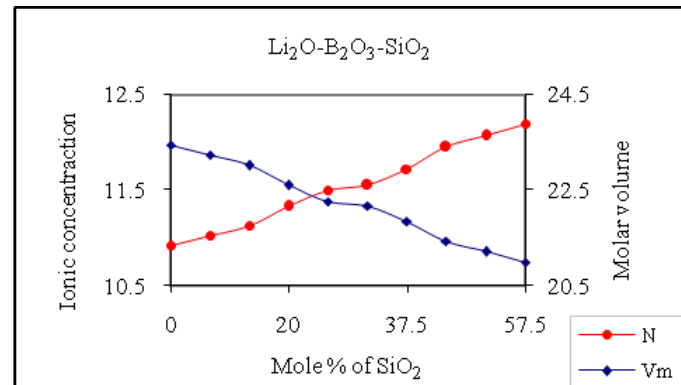


Figure 4.5.2. Variation of Ionic concentration, Molar volume with respect to mol% of SiO₂.

4.6. Refractive index (n), Dielectric constant (ε)

The mean atomic volume (V_M) of each glass was obtained from the values of densities (ρ) and mean atomic weights. The calculated values are given in Table 4.5. Other physical parameters such as, Refractive index (n) of samples was calculated by using the following relation^[28].

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{E_g / 20} \quad \text{Where, } E_g \text{ is the energy gap.}$$

The dielectric constant (ϵ) was calculated from the refractive index of the glass using^[29].

$$\epsilon = n^2$$

4.7. Reflection loss (R), Molar refraction (R_m)

The reflection loss (R) from the glass surface was computed from the refractive index by using the Fresnel's formula as^[30].

$$R = \left(\frac{n-1}{n+1} \right)^2$$

The Lorentz-Lorenz equation^[31, 32, 33, 34] relates molar refraction R_m to refractive index n and molar volume V_m of the substance by,

$$R_m = \left[\frac{(n^2 - 1)}{(n^2 + 2)} \right] V_m$$

where V_m is equal to the molar volume.

This equation gives the average molar refraction of isotropic substances, i.e., for liquids, glasses and cubic crystals. The Lorentz-Lorenz equation presents the polarizability, i.e., the magnitude of the response of the electrons to an electromagnetic field.

On the other hand, Duffy^[35] has obtained an empirical formula that relates energy gap E_g to molar refraction R_m .

$$R_m = V_m \left(1 - \sqrt{E_g / 20} \right)$$

The ratio of $\frac{R_m}{V_m}$ is called polarizability per unit volume.

According to the Herzfeld theory of metallization ^[36], If $\frac{R_m}{V_m} > 1$ and $\frac{R_m}{V_m} < 1$ samples predicting metallic or insulating. From Table 2.3 it is clear that present glass samples behave as non-metal. The difference $M = 1 - R_m/V_m$ is so-called metallization criterion ^[37]. Materials with large M close to 1 are typical insulators. The small value of M close to zero means that the width of both valence and conduction bands become large, resulting in a narrow band gap and increased the metallicity of the solid.

4.8. Molar polarizability (α_m), Electronic polarizability (α_e)

The molar refraction R_m , can be expressed as a function of molar polarizability α_m as

$$R_m = 4\pi\alpha_m A_v / 3$$

Where A_v is Avogadro's number introduced, with α_m in (\AA^3) this equation can be transformed to,

$$R_m = 2.52\alpha_m$$

Hence molar polarizability α_m can be calculated.

The electronic polarizability (α_e) was calculated using the formula ^[38].

$$\alpha_e = \frac{3(n^2 - 1)}{4\pi A_v (n^2 + 2)}$$

Where, A_v is the Avogadro number. The measured and calculated values of densities, molar volumes and polarizability of oxide ions of P_2O_5 doped lithium-borate glasses are listed in the Table 4.8.

Table 4.8 Refractive index, Dielectric constant, Reflection loss, Molar refractivity, Electronic polarizability, Metallization, Molar polarizability and Polarizability per unit volume.

| Refractive index (n) | Dielectric constant (ε) | Reflection loss R | Molar Refractivity $R_m(\text{cm}^3)$ | Electronic polarizability $\alpha_e *(10^{24}\text{ions}/\text{cm}^3)$ | Metallization M | Molar polarizability $\alpha_m*(10^{24}\text{ions}/\text{cm}^3)$ | Polarizability /unit volume R_m/V_m |
|----------------------|-------------------------|-------------------|---------------------------------------|--|-----------------|--|---------------------------------------|
| 3.24 | 10.5 | 0.279 | 17.814 | 0.301 | 0.24 | 7.063 | 0.76 |
| 3.999 | 15.993 | 0.36 | 19.348 | 0.33 | 0.167 | 7.672 | 0.833 |
| 3.426 | 11.736 | 0.3 | 17.985 | 0.31 | 0.218 | 7.131 | 0.782 |
| 3.447 | 11.882 | 0.303 | 17.708 | 0.311 | 0.216 | 7.021 | 0.784 |
| 3.275 | 10.723 | 0.283 | 17.019 | 0.303 | 0.236 | 6.748 | 0.764 |
| 3.369 | 11.35 | 0.294 | 17.18 | 0.307 | 0.225 | 6.812 | 0.775 |
| 3.296 | 10.862 | 0.286 | 16.746 | 0.304 | 0.233 | 6.64 | 0.767 |
| 3.271 | 10.7 | 0.283 | 16.36 | 0.303 | 0.236 | 6.487 | 0.764 |
| 3.336 | 11.131 | 0.29 | 16.364 | 0.306 | 0.228 | 6.488 | 0.772 |
| 3.212 | 10.32 | 0.276 | 15.879 | 0.3 | 0.244 | 6.296 | 0.756 |

Variation of band energy gap and refractive index with increasing mol% of SiO_2 , are exactly apposite to each other Figure (4.8.1). This indicates that is due to break down of borate bonds to create non-bridging oxygen atoms; which also supports from the decreasing of oxygen packing density as well as decreasing the values of ionic concentrations of the atoms (Table 4.5). This is due to the glass structure becomes loosely packed.

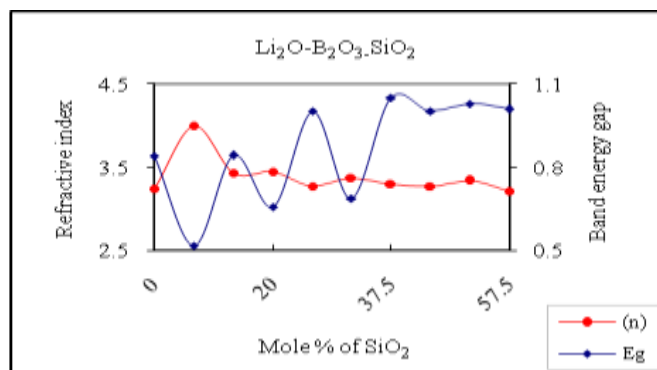


Figure 4.8.1: Variation of band energy gap and refractive index with mol% of SiO_2

Comparative study of electronic polarizability and density (Figure 4.8.2) shows, the alternately minimum and maximum values with increasing mol% of SiO₂, and electronic polarizability is exactly apposite to that of density. This is due to break down of borate & silicate bonds to create non-bridging oxygen atoms. Due to higher molecular weight of B₂O₃ compared to that of SiO₂ and the formation of BO₃ & BO₄ which modify the glass structure by creating NBOs in the network.

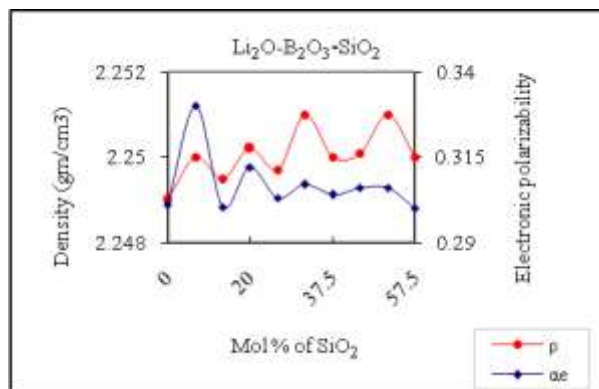


Figure 4.8.2: Comparative study of Electronic polarizability and Density

The behavior of dielectric constant and metallization are exactly apposite to each other (Figure 4.8.3); which is also due to break down of borate & silicate bonds to create non-bridging oxygen atoms, with constant 42.5 mol% of Li₂O.

Molar polarizability, Molar refractivity, Electronic polarizability varies alike with increasing mole % of SiO₂ respectively. Maximum values at 5 mol % of SiO₂ are 7.671×10^{24} ions/cm³, 19.348 cm³ and 0.33×10^{24} ions/cm³ (Figure 4.8.4)

Electronic polarizability, reflection loss is varies similarly while Metallization is exactly opposite to them; at 5 mol % of SiO₂ reflection loss is 0.359 and metallization is 0.166 (Figure 4.8.5), higher values are due modification the glass structure by BO₃ units with non-bridging oxygen atoms in the network.

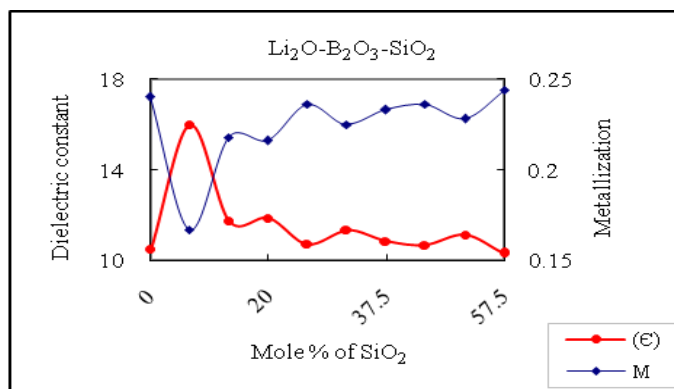


Figure 4.8.3: Dielectric constant and Metallization verses mol %. Of SiO₂

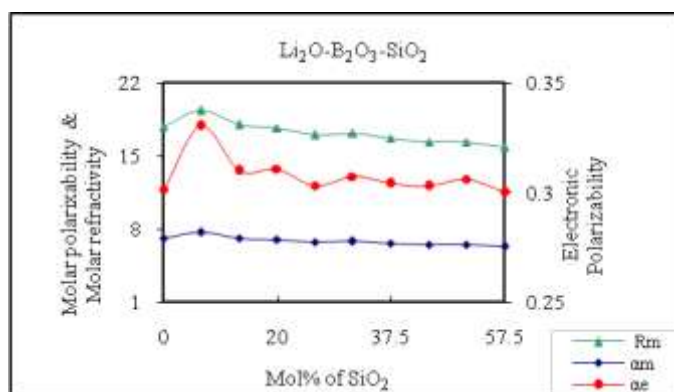


Figure 4.8.4: Comparative study of molar polarizability, molar refractivity and electronic Polarizability against mol % of SiO₂.

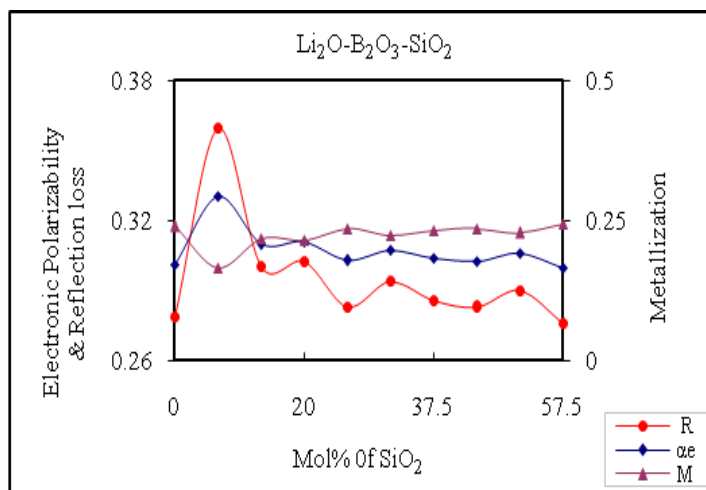


Figure 4.8.5: Comparative study of electronic polarizability, Reflection loss and Metallization against mol % of SiO₂.

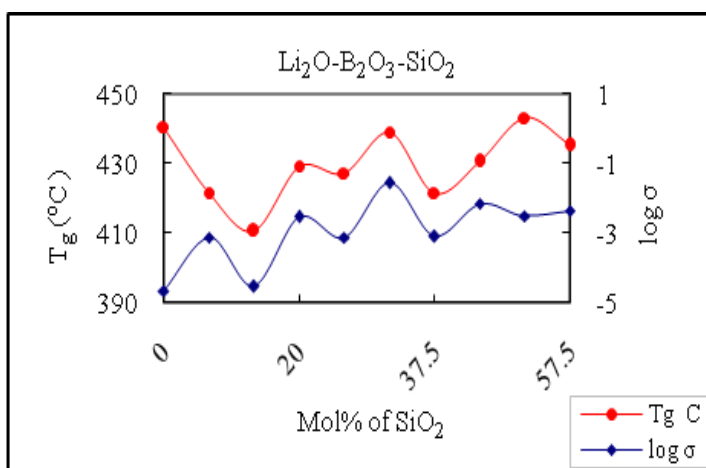


Figure 4.5.6: Comparative study of glass transition temperature, log σ against mol % of SiO₂.

Conductivity increases according to glass transition temperature with mol % of SiO₂. The maximum value of conductivity $4.1572 \times 10^{-3} \text{ S cm}^{-1}$ is at 10 mol% of SiO₂, at T_g 410.5 °C at 42.5 constant mol% of Li₂O ((Figure 4.5.6)); due to the formation of BO₃ and BO₄ which will modify the glass structure by creating NBOs in the network. Figure 4.5.7 shows, as usual behavior in log σ and activation energy.

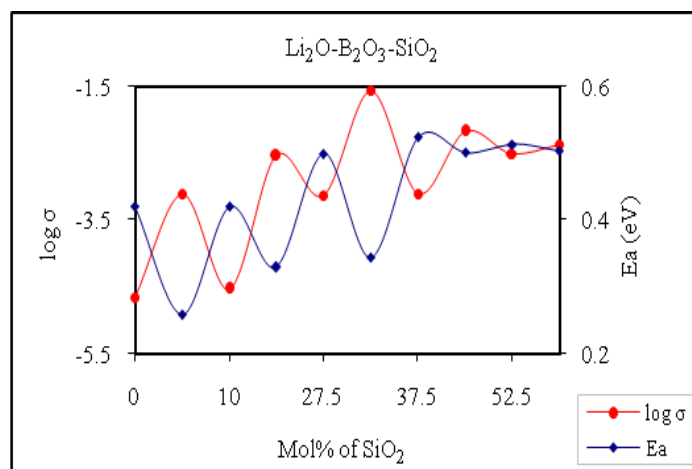


Figure 4.5.7: Comparative study of activation energy, log σ against mol % of SiO₂.

Hence mixed glass former effect (MGFE) gives a nonlinear and non-additive change in the ionic conductivity with changing glass former composition at constant modifier

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