BEST: International Journal of Humanities, Arts, Medicine and Sciences (BEST: IJHAMS)
ISSN (P): 2348-0521, ISSN (E): 2454-4728
Vol. 4, Issue 5, May 2016, 69-74

COMPOSITION EFFECT OF Al_2O_3 ON DENSITY AND FTIR OF LITHIUM BORATE GLASSES

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ABSTRACT

Lithium aluminum borate glasses of composition $35Li_2O$: (65-X) B_2O_3: xAl_2O_3 (where X = 0, 5, 10, 15, 20) were prepared by melt quenching technique and investigated by XRD, DTA, FTIR and density measurement. X ray diffraction and scanning electron microscopy confirmed the nature of sample. The density and molar volume studies reported change of structure with increase in mole percent of aluminum. The FTIR analysis revealed that network structures of sample are mainly based on BO_3 and BO_4 unit.

KEYWORDS: XRD, FTIR and Density

INTRODUCTION

Chemical composition of glasses play important role in determining properties of the glasses. The glass is divided into main categories: network formers, network modifiers and intermediate species, which falls somewhere between network modifier and may be substituted for a network former in the glassy state. Borate is one of the important glass former and has been incorporated in much kind of glasses to attain desired chemical and physical properties. Glasses are receiving considerable attention due to their unique properties like hardness, good strength, transparency and excellent corrosion resistance. X-ray diffraction (XRD), infra-red spectroscopy (IR), differential scanning calorimetry (DSC) studies has been extensively employed over the years to investigate the structure of glasses [1-4]. Borate glasses, in particular, have been the subject of numerous infra-red studies due to their structural peculiarities [5-8]. In pure B_2O_3 glass structure most of the boron is involved in B_3O_6 (boroxol) ring. Addition of modifier breaks boroxol ring and thereby produced BO_3 and BO_4 units [6, 8]. In addition, modifier also changes the physical properties along with structural modifications. Recently, the study of oxide glasses doped with transition metal ions (TMI) has received considerable attention due to their attractive combination of physical and chemical properties. TMI doped borate glasses have application in microelectronics, optical glasses and solid state laser [9-11]. Continued effort for the development of new glassy materials either by doping or by adding TMI, and the study of their novel properties is highly relevant due to their potential applications in various technological fields [12, 13]. Keeping in mind the very fact of creating novel functionalities we have chosen an uncommon glass system. We report here the preparation, structural characterization, optical properties and physical property of aluminum oxide containing lithium borate glass, which may lead to a new composite material.

EXPERIMENTAL

The aluminum lithium borate glasses of composition $35Li_2O$: (65-X) B_2O_3: xAl_2O_3 (where X = 0, 5, 10, 15, 20) were prepared by melt quenching technique. The starting material lithium carbonate, boric acid and aluminum oxide of AR

grade purchased from Merc laboratory were used. A homogeneous mixture of different composition has melted in ceramic crucible by keeping it into Muffle furnace equipped with digital temperature controller. The materials were melted at1150^{0}C for two hours with heating rate 30^{0}C/min and molted material is quenched in aluminum mould at room temperature (27^{0}C).The samples were annealed at 200^{0}C for 2Hrs in hot air oven. The measurements of XRD were carried out by using XPERT PRO DIFFRACTOMETER. Scanning electron microscope of all samples was investigated from Tata Institute of Fundamental Research Mumbai by using ZEISS Ultra SEM instrument. FTIR of prepared samples were recorded by SHIMADZU FTIR spectrometer IR infinity1/8300 in the range 4000-400cm^{-1} with resolution 4 cm^{-1}.The density were measured by Archimedes principle.

Measurement

The density of glass samples have been measured by Archimedes's principle with pure benzene as the immersion fluid. All the measurement were made using K Roy balance. The experiment was repeated five times to get accurate value of density. The density was calculated according to known formula.

$$d_{exp} = \frac{Wa \times d_b}{(Wa - W_b)} \quad (1)$$

Where, Wa is the weight of sample in air, Wb is the weight of sample benzene and d_b is density of buoyant (benzene) at room temperature.

Molar volume is calculated by the formula.

$$Vm = \frac{M}{d_{exp}} \quad (2)$$

Where, M is the molecular weight of sample.

The Number of ions per c.c. (N), ploaron radius (r_p) and Hopping distance (R) are calculated.

RESULTS AND DISCUSSIONS

- **X- Ray Diffraction (XRD)**

The XRD spectra of investigated samples have been found as shown in Figure 1. X – Ray diffraction patterns recorded for all samples show a diffuse scattering over range of angles (2θ from 10˚ to 100˚), which confirms amorphous nature of the samples.

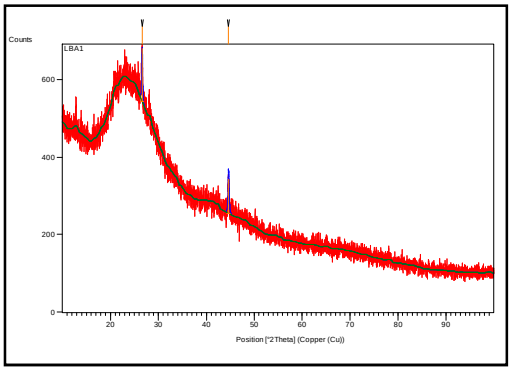

Figure 1: XRD of LB1A Sample

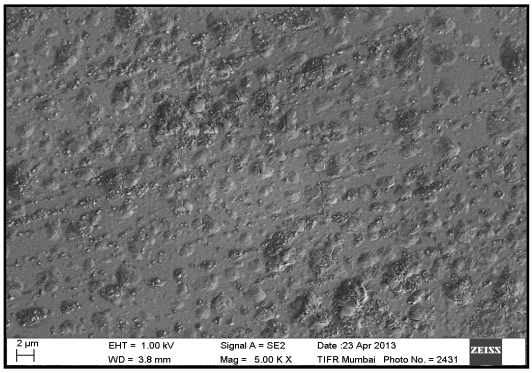

Figure 2: SEM of LB1A Sample

- **Scanning Electron Microscopy (SEM)**

To study the surface morphology, scanning electron microscope results are obtained and are shown in Figure 2. It is clearly visible from the scan that the prepared glasses appear a homogeneous structure of glass flake suggesting highly amorphous phase. The result of XRD and SEM shows that the prepared glasses are amorphous in nature.

In the photograph of SEM, It has been observed that the structure of glass sample is changed and compactness increases due to addition of glass former Al_2O_3, glass converted in to glass ceramics. It reflects the mixed former effect.

- **Density and Molar Volume**

All the values for these samples which have been calculated are listed in the Table 1. The variation of density and molar volume with mole percent of Al_2O_3 is depicted in Figure 3. The density of samples is gradually increased from 2.3 to 2.6 g cm^{-3} and molar volume decreases from 24.17 to 23.886 cm^3 with increase of mole percent of Al_2O_3 content. This increase in the density can be due to replacement of low density B_2O_3 by high density aluminum oxide. Also non bridging oxygen decreases in the glass network on increasing Al_2O_3 content, which tends to consolidate their structure, maintaining homogeneity of the glasses and thus increasing density. Molar volume indicates the spatial distribution of the oxygen in the glass network. The gradual decrease in molar volume can be attributed to closing up of glass structure. As we have more content of Li_2O than Al_2O in our system therefore net result is to shift coordination (BO_3) to (BO_4) and hence increase in density. The observed results agree well with the literature [14-15].

Table 1: Density, Molar Volume, and Ions per c.c., Hopping Distance and Polaron Radius

Sample	Mole % of Al_2O_3	Molecular Weight(M)	Density (Dexp)	Molar Volume (Vm)	Ions per c.c.	Hopping distance(A^0)	Polaron Radius (A^0)
LB1A	0	55.724	2.305	24.170	2.578E+22	3.381	1.363
LB2A	5	57.34	2.378	24.107	2.584E+22	3.378	1.362
LB3A	10	58.956	2.453	24.029	2.593E+22	3.376	1.360
LB4A	15	60.572	2.528	23.955	2.601E+22	3.371	1.359
LB5A	20	62.188	2.603	23.886	2.608E+22	3.368	1.358

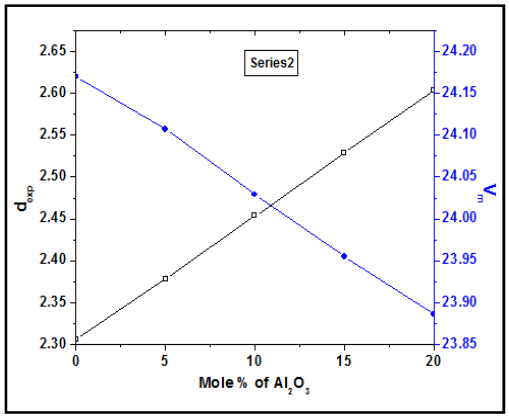

Figure 3: Variation of Density and Molar Volume

- **Fourier Transform Infrared Radiation (FTIR)**

The results have been discussed on the basis of method given by Condrate and Tarte [16-17] by comparing experimental data of the glasses with those of related crystalline compounds. The characteristic curve for B_2O_3, Li_2O and

Al_2O_3 were used as reference point in the discussion. The structural analysis of oxide glasses is carried out by using following information [18-19].

- Type of the bridged bonds of oxygen which link the co-ordination polyhedral of the framework and composition of chemical in homogeneities in the structure of the glass.
- The co-ordination number of the compound with respect to oxygen, especially of the network former.
- The change in oxygen bonds of framework induced by cation modifiers which combine with the oxygen bonds.

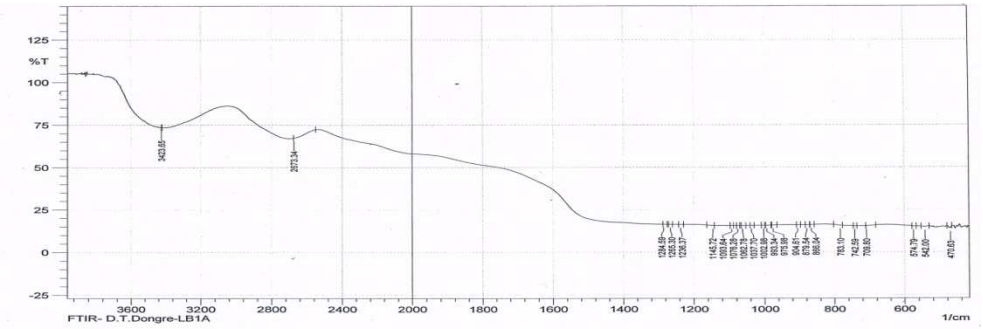

Figure 4.(a): FTIR of LB1A

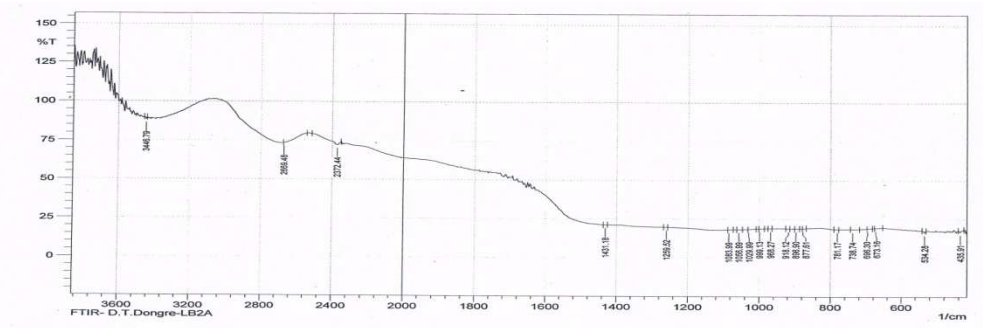

Figure 4.(b): FTIR of LB2A

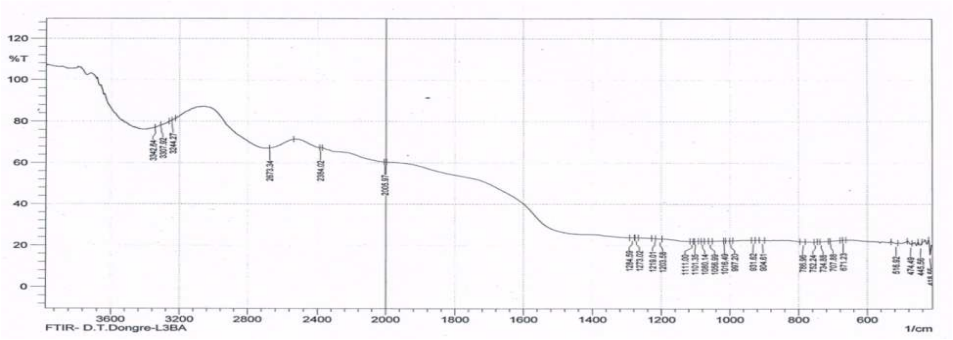

Figure 4.(c): FTIR of LB3A

The FTIR absorption spectra of investigated samples for the region 400-4000 cm^{-1} are shown in Figure 3. The IR spectra of these investigated samples show the number of sharp, medium and broad absorption peaks. Due to close and overlapping bands, obtained spectra appear more complex. The vibration modes of borate network are mainly active in three infrared spectra region, which are similar to those reported by several workers [16-22]. First band occurrence at

1200-1600cm^{-1} is due to asymmetric stretching relaxation of B-O band of trigonal BO_3 unit. Second band between 800cm^{-1} and 1200cm^{-1} is due to B-O bond stretching of the tetrahedral BO_4 unit and third group observed around 700cm^{-1} is due to bending of B-O-B linkage in the borate network.

The FTIR absorptions bands obtained for this series (LB1A-LB5A) are at 422 cm^{-1}-486cm^{-1}due to presence of lithium oxide in the samples. The bands situated in the range 501cm^{-1}-574cm^{-1} is assigned by B-O-B bands bending vibrations of various borate segment and next one is at 700cm^{-1}-794 cm^{-1} is attributed to the deformation and bending vibrations B-O-B linkage in borate network. It has been observed that LB3A and LB4A samples show maximum absorption and shift toward lower wave number. The maximum bands appear in these samples (LB3A and LB4A) in the range 800cm^{-1}-1200cm^{-1} are due to stretching of tetrahedral BO_4 unit. In the region 1200-1600cm^{-1}, bands appear in the range 1236cm^{-1}-1477cm^{-1} due to B-O bond of trigonal BO_3 unit. The band that appears above 1500 cm^{-1} can be assigned to presence water group and O-H vibration.

CONCLUSIONS

According to the results obtained, it can be concluded that investigated glass samples are composed of glassy phase. The increase in mole percent of Al_2O_3 changes the structure of sample from partial crystalline phase to fully glassy phase. The results of SEM are same as that of XRD results. From FTIR results, it is concluded that Al_2O_3 enters in the glass network as modifier and former. The BO_3 and BO_4 act as network structural group while lithium and aluminum appear in interstitial positions. Formation of BO_3 and BO_4 units with increase in mole percent of Li_2O and Al_2O_3 is also confirmed by FTIR spectroscopic studies. The density of glass increases and molar volume decreases with increase in mole percent of aluminum oxide.

AKNOWLEDGEMENT

The Authors are grateful to Principal Vidyabharti Mahavidyalaya Amravati, Principal S.S.S.K.R.Innani Mahavidyalaya Karanja (Lad) and TIFR Mumbai for providing facility to complete this work.

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