ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com
International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 3, Special Issue 2, March 2016

SPECTROSCOPIC AND THERMAL STUDIES ON B_2O_3-K_2O-CaO GLASSES

R. EzhilPavai and M. Indhira
Department of Physics, Annamalai University, Annamalainagar, Tamil Nadu, India
Corresponding author: R.EzhilPavai. Email: ezhilpavaibalu@yahoo.com

ABSTRACT

Mixed alkali alkaline earth oxide borate glasses of compositionB_2O_3-K_2O-CaO glasses with different concentrations of CaO (0-20 mol. % in the steps of 5) were prepared by melt quench technique. Structural characterizations of these glasses were conducted through FT-IR, DTA measurements. The amorphous nature of the glasses was checked by X-ray diffractometry (XRD). The transformation of BO_3trigonals to BO_4 tetrahedral units has evidenced from the FT-IR spectra of the prepared glass samples and the BO_4 units increases with an increasing concentration of CaO content. The transition temperature (Tg), melting temperature(Tm) and crystallization temperature (Tc) have been identified using

DTA measurements. The transition temperature (Tg) increases with an increase of CaO content. The structural properties of these glasses were discussed in terms of the relative proportion of potassium and calcium oxides.

Key words: borate glasses, XRD, FTIR spectroscopy, DTA.

INTRODUCTION

Glasses are super cooled liquids, transparent, and amorphous in nature. They are inorganic product of fusion which has cooled to a rigid condition without any crystallization. Especially, borate glasses are a technologically important class and used in many devices required for high-temperature-bearing capacity[1-3].

The alkali-borate glasses are commonly used materials in the field of opto-acoustical electronics, in nonlinear devices for frequency conversion in the ultraviolet region and piezoelectric actuator.Meanwhile, these glasses and their crystalline counterparts are considered to be good candidates for the optically induced elastoopticity. The boron atom in borate crystals and glasses is usually coordinated with either three or four oxygen atoms forming [BO3] or [BO4] structural units. These two fundamental units can be arbitrarily combined to form either the so-called super-structure or different BxOy structural groups like boroxol ring, pentaborate, tetraborate, diborate groups etc. In the alkali borate glass systems, each alkali oxide is associated with a proportional quantity of B2O3; so that, the number of the structural units depends on both the nature and the total concentration of the added modifiers, and can often give rise to tightly organized structures resulting in intermediate order [4-5]

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com
International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 3, Special Issue 2, March 2016

The objective of the present paper is to probe the structure of 60B2O3- (40-x) K2O - xCaO (where x=0, 5, 10, 15and 20 mol %) glasses using spectroscopic techniques FTIR and DTA measurements. This study is significant because the new findings will provide information about the basic units forming this multi-component glass structure.

EXPERIMENTAL PROCEDURE

Glass preparation

The chemical composition of the investigated glass is chosen such that, contents of B_2O_3–K_2O–CaO were prepared by the melt-quench technique. A gradual increase in the concentration of CaO was done from 0 to 20 mol% while K2O concentration was decreased from 35 to 20 mol%. The raw materials of boron trioxide (B_2O_3), potassium oxide (K_2O) and calcium oxide (CaO) were mixed together by grinding to obtain a fine powder. The obtained mixture was melted in a silica crucible for 3 hours in a muffle furnace at 900 ºC until a bubble free liquid is formed. The melt was poured into a brass mould to form samples of dimensions 10mm diameter and 6mm thickness. Glass samples were annealed at 420 ºC for 2 hours to avoid the mechanical strain developed during the quench process. Then the furnace was switched off and glass was allowed to cool gradually to room temperature. Diamond disc and diamond powder were used to smoothen the prepared glass samples and to keep their surfaces perfectly plain.

Characterization

The amorphous nature of glasses was determined by X-ray diffraction technique. GE-Inspection technology 3003TT model made in Germany copper target operating voltage 40 Kv 300 mA. The prepared glass samples were grind with potassium bromide (KBr) pellet techniques.Using the pellet, the FT-IR spectra in the region 4000 cm^{-1} to 400cm^{-1} with a resolution of ± 4 cm^{-1} were recorded at room temperature using by SHIMADZU 8400 FT-IR SPECTROMETER. The Differential Thermal Analysis (DTA) was carried out on a SETARAM Labsys, TM TGDTA16 thermal analyzer between 100–1200K temperature range at heating rate of 10 °C min−1 .The nomenclature and the composition in mol% of different glasses are given in Table 1.

Table 1. Nomenclature and the composition of glass samples

Nomenclature	Composition (Mol %)		
	B_2O_3	K_2O	CaO
BKC 05	60	35	5
BKC 10	60	30	10
BKC 15	60	25	15
BKC 20	60	20	20

RESULTS AND DISCUSSION

X-ray diffraction

The XRD patterns of BKC 05 and BKC 20 glass samples are shown in Figure 1. Figure shows the X-ray diffractograms of the present glass samples. It gives absence of sharp peaks and conform the amorphous nature of present all the glasses[6].

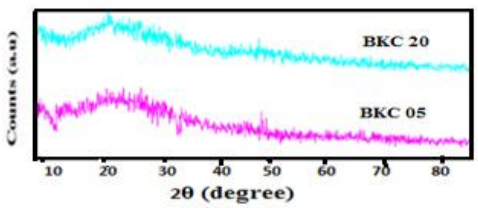


Fig.1. XRD of BKC05 and BKC20 glasses

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com
International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 3, Special Issue 2, March 2016

Infrared Spectroscopy

The infrared spectra of $60B_2O_3$- (40-x)K_2O - xRCaO(where x=5, 10, 15, and 20 mol%) glasses are recorded at 303K in the frequency range between 400 and 4000 cm^{-1} as shown in Figs.2. The observed bands along with their vibrational assignments of samples have been tabulated in Tables 2.

In the present investigation, the following bands are present in the IR spectra of BKC systems: 1321-1387cm^{-1}, 1006-1093 cm^{-1} and 2800-3600 cm^{-1}. The first band is due to B-O asymmetric stretching vibrations of BO_3 units. The second band is due to the stretching vibration of tetrahedral BO_4 units and is shifted to higher intensity with the addition of alkaline earth oxide to binary glass matrix. The introduction of alkaline earth oxide alters the structure of the glass to a large extent by the conversion of BO_3 units present in the glass into BO_4 units and it produces a more polymerized borate network. As the concentration of alkaline earth oxion increases, the intensity of the band due to [BO_3] decreases corresponding to increase in the intensity of [BO_4] units.

The bands at low frequencies (457 cm^{-1}) are observed in the spectra and can be attributed to vibration of metal cation such as Ca^{2+}Hence, network modifying behavior is observed in which these ions enter the interstices of the network [9]. The addition of CaO into BK glass matrix, makes an increase in the BO_4 units and decrease in the BO_3 structural units, indicating an increase in the compactness in the glass network.

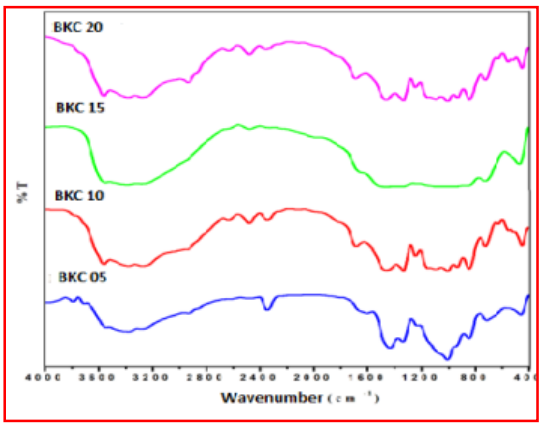
Therefore, FTIR spectra give information about the interactions between alkali, alkaline earth ions and borate glass network, which will be helpful in order to recognize the ionic transport phenomena in these glass samples.

Table. 2. Band positions and their corresponding assignments of infrared spectra of BKC glass system

Wavenumber cm-1	Assignment	Reference
~1319	B-O stretching vibration of the trigonal BO_3 unit	Karthikeyan and Mohan 2003
~1017	B-O stretching vibrations of tetrahedral BO_4.	Ramadevedu, et al. 2011
~717	Bending vibration of B-O-B	Nageswara Rao, et al. 2005
~457	Vibration of metal cations Ca^{2+}	Manisha Pal, et al. 2011

Thermal behavior of BKC glasses

In BKC system the DTA curve exhibits a small endothermic hump at lower temperature in the glass samples, which is characteristic of the transition temperature (T_g), single exothermic peak at high temperature regionis characteristic of crystallization temperature (T_c). The exothermic peak is followed by an endothermic peak, which is characteristic of the melting temperature (T_m). The DTA curves for BKC glasses show a small



ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com
International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 3, Special Issue 2, March 2016

endothermic hump corresponding to the glass transition temperature (T_g) in the range 127 - 162 ºC. This is followed by an exothermic peak corresponding to the crstallization temperature between 420 - 6y21 ºC and other endothermic events corresponding to the melting temperature (T_m) in the range 595 - 710ºC. The values of T_g, T_c and T_m increase with the introduction of CaO. The results indicate that the addition of CaO enhances the rigidity of the glass network

Fig. 2. Infrared spectra of BKC glasses with different concentration of CaO

.

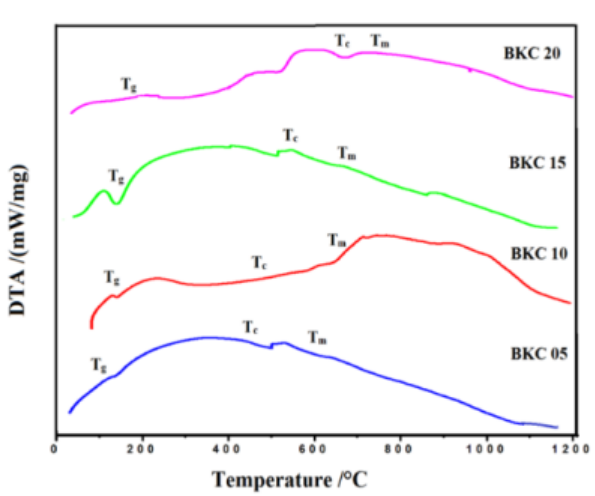


Fig. 3. DTA curves for BKC glasses

Table 3. Values of glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m), thermal stability (S) andHruby's parameter (K_{gl}) of BKC glasses

Name of the sample	T_g/°C	T_c/°C	T_m/°C	S	K_{g1}
BKC 05	127	420	595	293	1.67
BKC 10	147	481	629	334	2.25
BKC 15	158	535	637	377	3.69
BKC 20	162	621	710	459	5.15

Tables 3 show the values of T_g,T_c, T_m, glass stability factor(S) and Hruby's parameter (K_{gl}). Hruby's parameter gives the information on the stability of the glass against devitrification.The values of S and K_{g1}increase with increasing the alkaline earth ion in BKC systems.

Conclusion

In summary, it is concluded that the glass samples of composition $60B_2O_3$ - (40-x) K_2O - xMgO (where x=0, 5, 10, 15 and 20 mol. %) has been successfully developed which is transparent and stable. From the XRD profiles, the amorphous nature of the glasses sample is confirmed. The FT-IR spectral studies have indicated the transformation of BO_3 triangles to BO_4 tetrahedral for the glass samples with an increase in CaO content. Thermal stability of the investigated glasses increases with increasing CaO content at the expense of K_2O.

REFFERENCES

1 Avadheshkumaryadavand C. R. Gautam. Spectroscopy Letters, 48: 514–520

2 Yamane, M.,Asahara, Y. Glasses for Photonics; Cambridge University Press: Cambridge, 2000.

3 Srinivas,G., RameshBoda , Siva Kumar,J,. Shareefuddin , M.N. Chary,Sayanna, R. Int. J. Innovative Resec in Science, Eng& Tech, 4(8): 2015.

4 Edukondalu,A., Ch. Srinivasu, Syed Rahman, K. Siva kumar. Int. J. Scientific &Eng.Resc, 5(3): 2014.

5 Wollenhaupt, Ahrens, Frobel,

ISSN 2394-3777 (Print)
ISSN 2394-3785 (Online)
Available online at www.ijartet.com
International Journal of Advanced Research Trends in Engineering and Technology (IJARTET)
Vol. 3, Special Issue 2, March 2016

Barner, Giessinger, Braunstein, J. Non- Cryst. Solids, 194, 191:1996.

6 ChandkiramGautam, Avadhesh Kumar Yadav , Vijay Kumar Mishra , KunwarVikram. Open Journal of Inorganic Non-metallic Materials. 2(47-54):2012

7 Nageswara Rao, P., C. LaxmiKanth, D. Krishna Rao and N. Veeraiah,.*J. Quant. Spectro. Radia. Transf*., 95(3)2005: 373–386.

8 Pal Singh, G. and D.P. Singh, *J. Mal. Struct.,* 1012(2012):137-140.

9 Manisha Pal, Baishakhi Roy and Mrinal Pal. *J. Modern Physics*, 2,(2011): 1062-1066.