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Structural and Thermal Investigations on Sodium Alumino Borate Glasses

P. Vasantharani* and S.Rajeswari

Department of Physics, Annamalai University, Annamalai Nagar, Chidambaram 608 002, Tamilnadu, India.

Abstract : Glasses of $70B_2O_3$-(30-x)Na_2O-xAl_2O_3 systems with different Al_2O_3 content (where x = 5, 10, 15, 20 and 25 mol%) have been prepared by melt quench technique. X-Ray diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform Infra Red (FT-IR) spectroscopy and Thermo Gravimetric Differential Thermal Analysis (TG-DTA) studies have been employed to study the role of Al_2O_3 on the structure of the investigated glass system. The amorphous nature of these samples was verified by XRD and SEM is used to study the morphology of these glass samples. FT-IR spectrum reveals the characteristic absorption bands due to various groups of triangular and tetrahedral borate network. Glass transition temperature, crystallization temperature and thermal stability were determined by TG-DTA investigations.
Keywords : sodium borate glass,FTIR,TG-DTA,XRD,SEM.

1. Introduction

In recent years, there has been a considerable interest in the study of alkali borate glasses doped with transition metal ions because of their potential applications as thermal ,optical ,lasers, photo-conductive devices, magnetic materials, especially for tuneable solid state lasers, efficient phosphors, etc[1] Borate is the best glass forming system and is present in almost all commercially important glasses. Borate glasses are very useful amorphous materials considering their specific structure and physical properties and also they act as inorganic hosts for transition metal ions. [2]. B_2O_3 can form glasses at low melting point and with high transparency, high thermal stability and good rare earth ion solubility. Borate glass system has been shown promising properties to several technological applications, such as amorphous films for battery and tissue engineering and nuclear waste disposals. Concerning to photonics applications, borate glass properties are compatible to optical fiber development due to their low melting temperature and high rare earth loading capacity. Transition metal ions are incorporated in to these glasses in order to study their optical, dielectric and conductivity behaviours. Glasses containing transition metal ions have become a subject of interest owing to their potential applications.[3]

Glasses doped with transition metal ions are more attractive because they exist different valence states with different coordination glass matrices.

Na_2O helps to reduce the melting point and facilitate the homogenization of the glass system, thereby decreasing the possible structural defects.[4] Sodium diborate is a type of glasses that consist of one-third of sodium oxide and two-third of boron oxide. This type of borate glasses draws great attention due to their improved electrical and optical properties when modified by phthalocyanine or by rare earth ions.

Boroalumino glasses are technologically important due to their high mechanical strength and chemical durability and are widely used in various applications, which include optical communication, glass to metal seals, ion exchange material is, nuclear waste immobilization, etc [5].

2. Experimental

Glass samples with a general formula of ($60B_2O_3$-(30-x)Na_2O- xAl_2O_3) were prepared by melt quench technique. Analytical grade chemicals of B_2O_3, Na_2O and Al_2O_3were used. The required amount of the chemicals for a composition were taken and then mixed together and ground to obtain a homogeneous mixture. Then it was melted using a muffle furnace around 1000K for 3hours. The mixture were kept into Porcelain crucible and rapidly quenched onto a copper mould plate maintained at room temperature. The prepared samples were transferred for annealing in an oven at 30 minutes to reduce thermal stresses. The photograph of the glass samples is shown in Figure 1. The composition of the glass samples were shown in Table 1.

Table 1: Composition of the glass samples

Sample code	**composition in mol(%)**		
B2O3	**Na2O**	**Al2O3**	
BNA1	70	25	0 5
BNA2	70	20	10
BNA3	70	15	15
BNA4	70	10	20
BNA5	70	05	25

3. Results and Discussion

3.1 XRD analysis

XRD spectrum of **BNAC3** glass sample is shown in Fig.1 The XRD spectrum show a broad halo, which reflects the characteristic of amorphous or glass structure, obtained at around 2θ=30°[6] The absence of sharp, strongly diffracted beams in the x-ray diffraction patterns from glass indicated that there were no well defined planes in the structure on or around which the constituent atoms were regularly arranged.

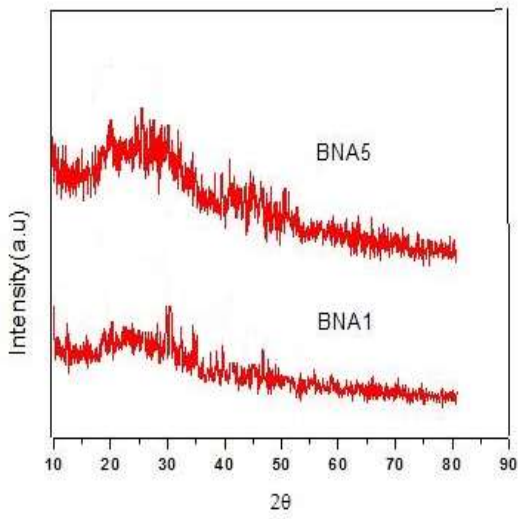

Figure 1: XRD spectrum of BNAC3 glass sample

3.2. SEM analysis

The glass homogeneity was characterized by scanning electron microscope. Figure 2 shows the SEM image of **BNA3** glass sample. It is observed that the sample exhibits surfaces without microstructure and different sized grain particles. The image clearly indicates that there is no crystalline phase existing in the overall surface of the samples. This further confirms the amorphous nature of the glass samples.

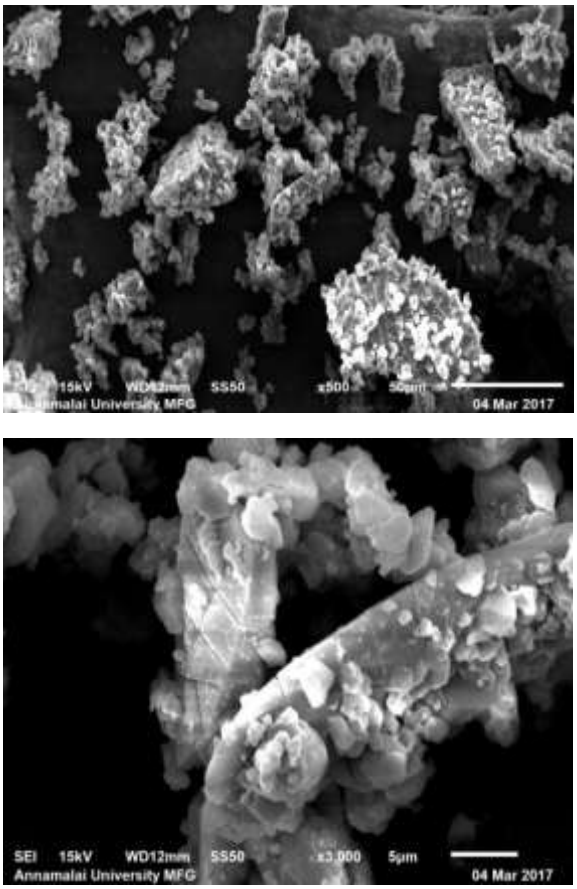

Figure 2 SEM analysis of BNAC3 glass sample

3.3 FT-IR Analysis

Infrared spectroscopic studies were used to get essential information about the arrangement of the structural units in the glass samples. It is assumed that the vibrations of a characteristic group of atoms in the glass network are independent of vibration of the other neighboring groups in the glass[7]. Fig. 3 shows the FTIR spectra of the glass samples, the spectra were obtained between 400-4000 cm^{-1}. The ftir spectra consist of four distinct regions. The first region 430-562 cm^{-1} is due to stretching vibrations of metal cations. The second region 568-612 cm^{-1} is due to B-O-B stretching vibrations involving oxygen atoms exterior borate rings.

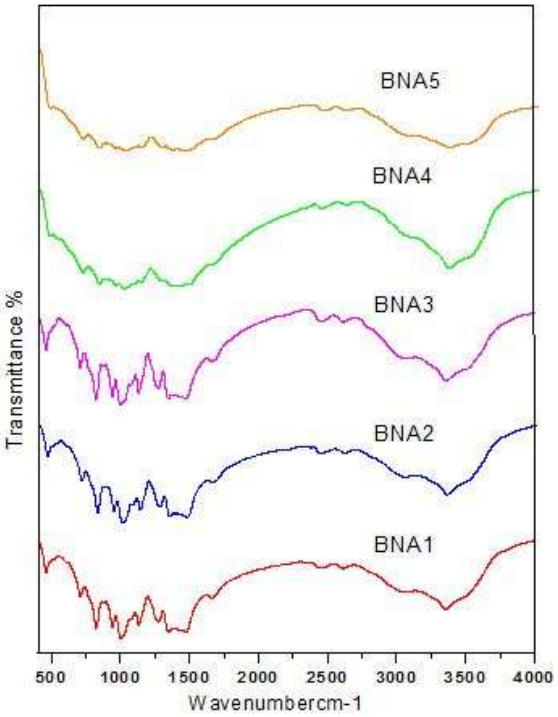

Figure 3.FT-IR Spectra of prepared glass samples

The third region from 700-1200 cm^{-1} is assigned to the stretching vibrations of tetrahedral BO_4 borate units. The fourth region from 1150-1600cm^{-1} is assigned to the stretching vibrations of tetrahedral BO_3 borate units. The small peaks are occurring above 1600 cm^{-1}, these peaks are attributed to O-H bending that give rise to absorption in this region and the possibility of some absorbed water.[8] The band at 710 cm^{-1} is due to bending vibrations of B-O-B triangles. The B-O stretching vibration in BO_4 units from di-borate groups was observed at 826 and 945 cm^{-1} .

The addition of Al_2O_3 into BN glass matrix makes an increase in BO_4 units and decrease in the BO_3 structural units, indicating an increase in the compactness of the glass network

AlO_4 tetrahedron is obtained and Al_{3+} is supposed to absorb its centre and acts with B_2O_3 producing BO_4 units. The band appears at 893 cm^{-1} which corresponds to Al-O units of the group AlO_4.[9]

The absorption band at 1006 cm^{-1} is probably due to the vibration of BO_4 tetrahedral which is present at di borate and tetra borate groups [13]. The absorption band at 1270 cm^{-1} was assigned to B-O asymmetric stretching vibration in BO_3 units. The band at 1346 cm^{-1} is assigned to the stretching vibrations the B-O trigonal BO_3 units [10].

3.4 TG-DTA Analysis:

The crystallization process of the glass during the thermal treatment is known to be connected to the nature and proportions of the glass oxide constituents. The ability of some cations to build glass forming units or to be housed as modifiers in interstitial positions in the glass structure must also be considered.[11] The DTAdata of the glasses are shown in Fig 4. The endothermic peaks in the 612-667°C temperature range. These endothermic peaks are to be attributed to the glass transition (Tg), at which the atoms begin to arrange themselves in preliminary structural elements subsequent to crystallization. One exothermic peak, which indicating crystallization reaction. These endothermic peaks are to be attributed to the glass transition (Tg), at which the atoms begin to arrange themselves in preliminary structural elements subsequent to crystallization. One exothermic peak, which indicating crystallization reaction in the glasses, is also recorded.

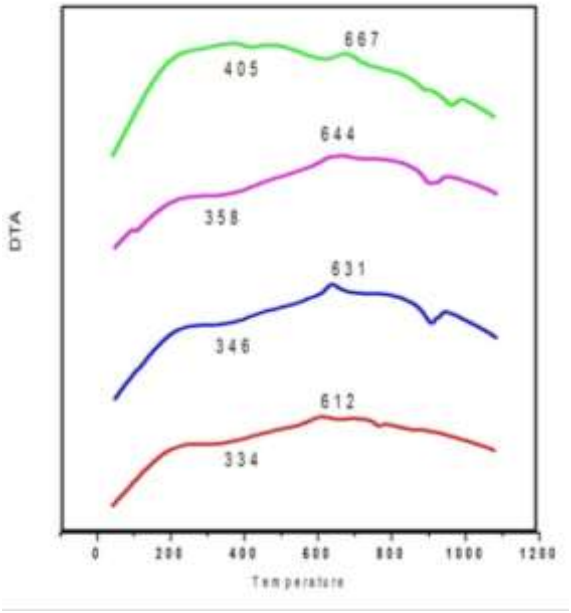

Figure 4. DTA analyses of prepared glass samples

According to Dietezel, the thermal stability of glasses (ΔT) can be expressed by the temperature difference between T_c and T_g.

$\Delta T = T_c - T_g$

The values of T_c, T_g, Δ T are mentioned in table. Increasing Δ T delays the nucleation process, indicating a better stability of the glass[12] The glasses have ΔT exceeding 100 c indicating that these samples are stable against devitrification[13]. Table 2 shows the calculated values of the samples.

Table 2: Calculated parameters from TG-DTA.

Samples	TgTc	ΔT	
BNAC2	405	667	262
BNAC3	358	650	292
BNAC4	346	638	292
BNAC5	334	612	298

4. Conclusion:

Aluminium doped sodium borate glass in the form of ($30B_2O_3$-$20Na_2O$-(30-X)Al_2O_3) were prepared and structural and thermal properties have been studied. The following conclusions were made. XRD patterns have confirmed their amorphous nature of these prepared glass samples. Morphological analysis of SEM shows the amorphous nature of the prepared glass samples. The FT-IR spectral analysis confirms the presence of absorption bands due to characteristic groups of BO_3 and BO_4 units. The TG-DTA results confirm good thermal stability of these glass samples.

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