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Measurements of Glass Transition Temperature of Na-Borophosphate Glassesby Thermal Analysis

Umakant B. Chanshetti*, Amit L. Patil
Department of Chemistry, Arts, Science and Commerce, College, Naldurg, Osmanabad, Maharashtra, India

ABSTRACT

Glasses of the series of xNa2O - (100-x) B2O3 (x=25, 30, 35, 40, 45,..) and 30Na2O-(70-x) B2O3-xP2O5 (x= 15, 20, 25, 30, 35) have been prepared by standard melt-quench technique. The glass samples were characterized using X-ray diffraction (XRD), thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) techniques. The X-ray diffraction pattern shows that all the samples no sharp Bragg's peak, but only a broad diffuse hump around low angle region. The amorphous phase of the prepared glass samples was confirmed from their XRD, TGA and DTA profiles.

Keywords : Glass ; Thermal Analysis, Annealed Glass, X-Ray Diffraction, Melt Quenching.

I. INTRODUCTION

Research in glass gained much interest now-a-days due to the increasing applications in engineering and technological fields.Glasses have been most widely used to make metal-ceramic seals because they can be modified to have a very close match of thermal expansion with metal materials. Except that, the glass seals show good result along with thermal and environmental stability [1-5].The glass transition temperature(Tg)[6-11]of a sample is an important parameter that determines its stability during storage. While Tg can be measured by a variety of methods, it is a time consuming procedure, especially if the sample is to be kept at subzero temperatures, in anhydrous conditions, or if sampling a portion of the specimen for analysis is cumbersome.

The glass transition is described to be the reversible change in amorphous materials between a hard, brittle state into a rubbery molten state. Materials which are capable of going through a glass transition are named as glass. Glass transition temperature is a metastable transition. Below this temperature, the material is specified to be a glass it means is typically hard and brittle; above, it is first a super cooled liquid i.e. a rubber like viscous liquid, and finally liquid [12].Pure boron trioxide (B_2O_3) is a very good glass former, covalently bonded, with interesting physicochemical properties.The Mixed Glass Former Glasses is improved thermal, chemical and mechanical properties, these glasses are significant candidates for improved solid electrolytes in next generation batteries [13].

II. MATERIALS AND METHODS

Glass Preparation

The glass samples were prepared with thefollowing chemical compositions *xNa_2O - (100-x) B_2O_3 (x=25, 30, 35, 40, 45) and $30Na_2O$-(70-x) B_2O_3-xP_2O_5 (x= 15, 220, 25, 30, 35)*. The chemicals used $NaNO_3$, H_3BO_3, $(NH_4)_2HPO_4$, are of analar grade.The appropriate chemicals were weighed andmixed thoroughly. These chemicals were thoroughly mixed and ground for 30-40 min in a mortar pastel and then the batches of (30g) was melted in alumina crucible using muffle furnace for 4-5 hrs at temperature -1000°C. When the melt was thoroughly homogenized and attained desirable viscosity it was poured either onto metal plate or into graphite moulds. The prepared glass was the annealed at appropriate temperatures (between 300 and 400°C) for 2 hrs and stored in desiccators prior to evaluation.At the end of the annealing process the glasses were allowed to cool down naturally to room temperature. The obtained glasses were confirmed bubble-free, homogeneous and transparent, in a circular glass disc shape with dimension of 10 mm diameter and 6 mm thickness.

X-ray Diffraction

In general, the glassy nature of the obtained glass could be confirmed by crystallographic and thermal analysis. Prepared glass samples were characterized by X-ray diffraction technique to check for possible crystallinity of glasses using X-ray diffractometer (Model. RigakuMiniflex II Desktop) with Cu-Kα radiation the XRD patterns were recorded in the 2θ =(20-80^o)with scanning rate 1^0/mint.

Thermal Analysis

In thermal study, Thermo gravimetric analysis (TGA) and differential thermal analysis (DTA) were performed on a Shimadzu DTG 60 simultaneous instrument from 0.00 to 800^{o}C. The thermal analysis of the very finely polished (powder) glass samples was done using Dilatometer (Model Shimadzu DTG 60 TGA-DTA). The heating rate was kept to 40^{o}C/min for all measurements. The properties like glass transition temperature, crystallization temperature were determined by using this technique. The glass transition temperatures (T_g) were taken as the inflection point of the endothermic changeof the calorimetric signal. Crystallization onset temperatures (T_c) were specified as the beginning of the reaction where the crystallization first starts and peak temperatures represent the maximum value of the exothermic.

III. RESULTS AND DISCUSSION:

XRD Analysis

The X-ray diffraction patterns of all the Sodium borateand Sodium borophosphateglass samples show no sharp peaks, indicating the absence of crystalline nature. This is clear indication of amorphous nature of glasses.

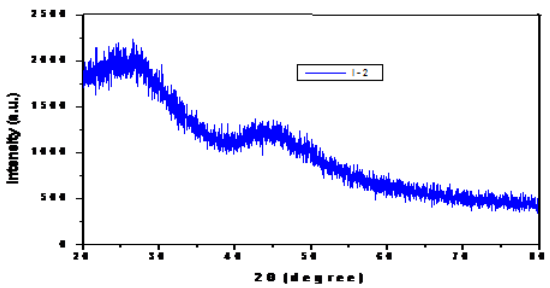

Figure 1. XRD profile for typical sample I-2 (X=20%).

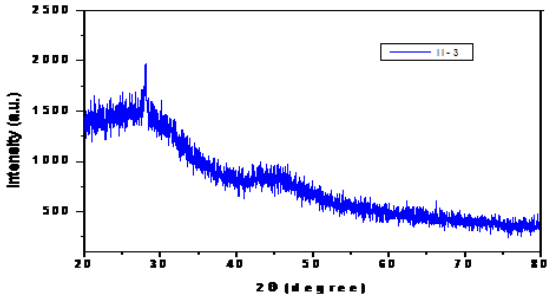

Figure 2. XRD profile for typical sample II-3 (X=25%).

TGA-DTA Analysis

The characteristic temperatures of the obtained glass were determined by TGA-DTA curves that were obtained for the as quenched glasses corresponding to the compositions x=30%, 35% of Na_2O and x=15%, 25% of $P_2O_5$are shown in figures respectively.

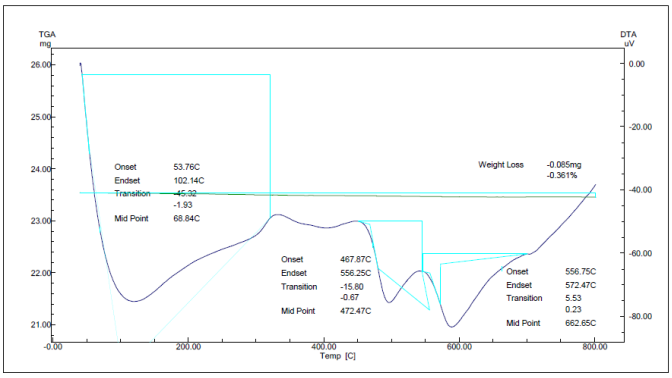

Figure 3. TGA-DTA curve and characteristic temperature determined for the compositions x=30% of Na_2O glass at heating rate of 40^{o}C/min.

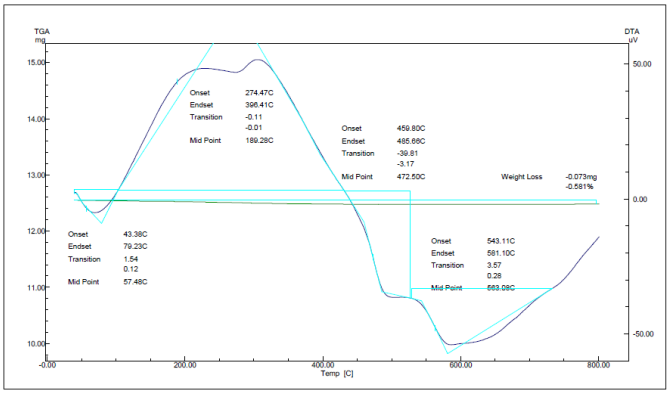

Figure 4. TGA-DTA curve and characteristic temperature determined for the Compositions x=35% of Na_2O glass at heating rate of 40^{o}C/min.

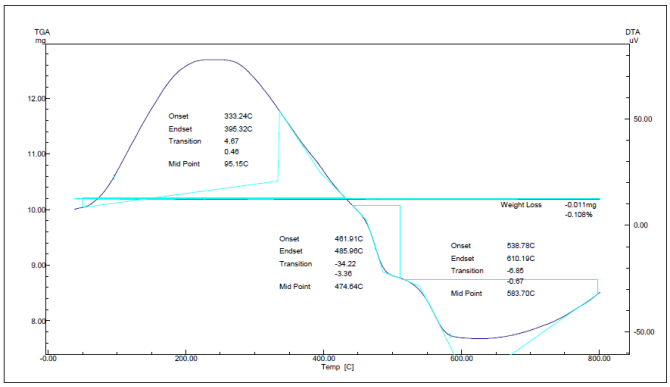

Figure 5. TGA-DTA curve and characteristic temperature determined for the Compositions x=15% of P_2O_5 glass at heating rate of 40^{o}C /min.

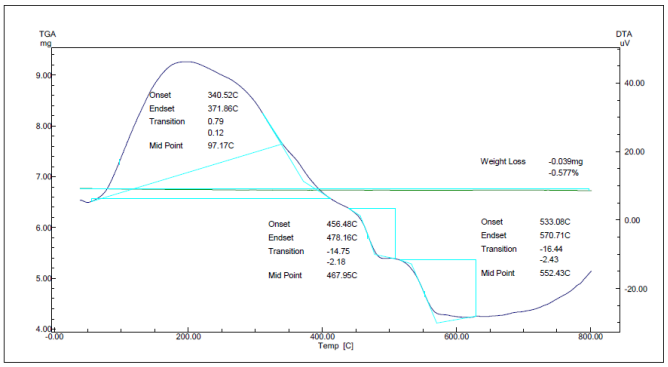

Figure 6. TGA-DTA curve and characteristic temperature determined for the Compositions x=25% of P_2O_5 glass at heating rate of 40^{o}C/min.

The glass transition temperatures (T_g) and crystallization temperature (T_c) of the Sodium borateand Sodium borophosphateglass samples are listed in table 1 and 2 respectively.

Table.1. Transition temperatures of sodium borate glass System indicated by TGA - DTA curves.

Sr. No.	Glass Code	Mole % Na_2O	T_g (^{o}C)	T_c (^{o}C)
1	I-2	30	472.47	556.75
2	I-3	35	472.50	543.11

Table 2. Transition temperatures of sodium borophosphate glass System indicated by TGA - DTA curves.

Sr. No.	Glass Code	Mole %P_2O_5	T_g (^{o}C)	T_c (^{o}C)
1	II-1	15	447.64	538.78
2	II-3	25	467.95	533.08

From table 1 it is observed that slight change with increasing the Na_2O content. It is due to the change in network structure; the material becomes soft. From table 2 it indicates that the T_g increase with increasing the P_2O_5 content. It may be due to the change in network structure (the material become soft) of the sodium borophosphate glass samples. It confirms that larger changes in glass structure occur.

IV. CONCLUSIONS

In the present investigation sodium borateand sodium borophosphateglass sampleswere prepared by conventional melt quench method.The amorphous phase of the prepared glass samples was confirmed from their XRD.Although glass is a very common material, its increasing technicality for special applications and issues linked with its worldwide scale manufacturing requires state of the art analytic tools. Apart from the glass transition temperature, which corresponds to the softening of the glass, TGA- DTA can become a key technique to study melting, crystallization and other phase transitions. Further, the increasing behavior of $T_g$indicates increasing the strength and thermal stability of the investigated glass systems with the increasing of Na_2O and P_2O_5 content in sodium borate and sodium borophosphate glasses.

V. ACKNOWLEDGEMENTS

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