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# STRUCTURAL CHARACTERISTICS OF DIFFERENTIAL SCANNING CALORIMETRIC (DSC) LEAD BORATE GLASSES

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## ABSTRACT

Lead Silver borate glasses were prepared in different Composition by using rapid quenching method. The appropriate property of  $PbO_2 - Ag_2O - B_2O_3$  having ratio  $Ag_2O / B_2O_3 = 0.5$ . This technique provides the understanding of glass transition and crystallization behavior of amorphous solids. This can be used as a quality control for monitoring glass synthesis because the overall behavior of the glass depends on glass transition temperature ( $T_g$ ) and the different phase formation in the samples.

## 1. INTRODUCTION:

The structure of borate glasses is different than other glasses such as phosphates and silicates. Soppe and Marcel (1988) suggested that the structure of borate glasses heavily depend upon the cooling rate of the melt through the range of glass transition temperature. The structure of glass was proposed by Zachariasen (1932). In borate glasses  $B_2O_3$  is a basic glass former because of its higher bond strength, lower cation size, smaller heat of fusion and valence (=3) of B. In borate glasses  $B^{3+}$  ions triangularly co-ordinate by oxygen to form glasses easily. In  $B_2O_3$  the units are triangles, which are covalently bonded in a random configuration (Yawale *et al* 2000, Barde V S, Pakade S V *et al* 2007, Motke S G, Yawale S P *et al* 2002).

The evolution of the ionic conduction have been reported in  $(AgI)_x-(Ag_2O)_y-(B_2O_3)_{1-(x+y)}$  glasses containing nanocrystallites of  $\alpha$ -AgI. The predominant role of Au/AgI nonoparticles is discussed (Kiyoshi Nozaki *et al* 2006; Zacheo *et al* 2011).

The nature of charge carriers and their transport numbers in glasses of the  $Ag_2O-B_2O_3$  system has been studied by Sokolov, *et al* (2006).

It has been observed from electrons spin resonance experiment that  $Ag^+$  ions are capable of trapping both types of charge carriers electrons and holes becoming the neutral species  $Ag^+$  and  $Ag^{++}$ . In borate glasses  $Pb^{2+}$  cations play the role of network modifier as well as glass former. The dual role of lead ions disrupted the glass network from  $BO_4^-$  tetrahedral.

## 2. EXPERIMENTAL PROCEDURE:

The glasses of different compositions were prepared from  $Ag_2O$ , (Sd Fine) AR grade,  $B_2O_3$  and GR grade  $PbO$ . Appropriate amounts in mole % of  $PbO$ ,  $Ag_2O$  and  $B_2O_3$  in powder form were weighted on K Roy monopan balance, having accuracy  $\pm 0.00001$  g and mixed thoroughly in acetone in an agate mortar.

The dried mixture was taken in porcelain crucible and heated at moderate rate in an automatically temperature controlled muffle furnace, (Gallenkamp Co. Ltd., England) operating on

CURRENT GLOBAL REVIEWER- Special Issue (UGC APPROVED) NO. 1100/2017. The general formula of the present system is  $(100-3x) \text{PbO}_2 \cdot x \text{Ag}_2\text{O} \cdot 2x \text{B}_2\text{O}_3$ . The ratio of  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3$  was kept constant = 0.5.

Repeated grinding of mixture was done to insure the homogenization. The melting temperature, which depend on the composition of different glasses ranges between 923 to 1023 K. The molten mass soaked at a temperature 323K above the melting point for two hours to achieve homogeneity and to minimize the dispersion of resistivity of the material. The molten mass was then finally, quenched between two flat surfaces of metallic block at room temperature in air.

The conducting silver paint applied on two surfaces of the glass sample, provide two electrodes, without any air gap between the surfaces of the sample. The dc electrical conductivity is measured by simple voltage drop method in the temperature range 353 to 513K.

This technique provides the understanding of glass transition and crystallization behaviour of amorphous solids. This can be used as a quality control for monitoring glass synthesis because the overall behavior of the glass depends on glass transition temperature ( $T_g$ ) and the different phase formation in the samples.

Glasses are generally prepared by sudden quenching technique. During cooling such glasses may undergo a second order phase transition called glass transition and temperature at which this occurs is called glass transition temperature.

## RESULT AND DISCUSSION:

In the present work the calorimetric investigation of  $\text{PbO}_2\text{-Ag}_2\text{O-B}_2\text{O}_3$  glasses is made in the temperature range of 303 to 1100 K at heating rate of  $10^\circ \text{K/min}$ . The differential scanning calorimetric (DSC) curves of present glasses are given in figures (a to f) for series. The DSC studies helps in understanding the glass transition and crystallization behaviour of solids.

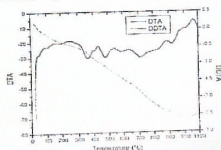


Fig. (a) : DSC curves of lead borate silver glasses series  $10\text{PbO}_2\text{-}30\text{Ag}_2\text{O-}60\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

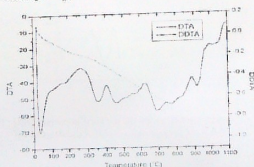


Fig. (b) : DSC curves of lead borate silver glasses series  $25\text{PbO}_2\text{-}25\text{Ag}_2\text{O-}50\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

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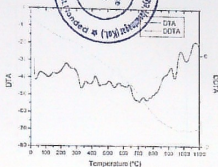


Fig.(c) : DSC curves of lead borate silver glasses series  
 $40\text{PbO}_2-20\text{Ag}_2\text{O}-40\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

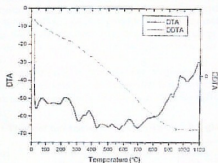


Fig. (d) : DSC curves of lead borate silver glasses series  
 $55\text{PbO}_2-15\text{Ag}_2\text{O}-30\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

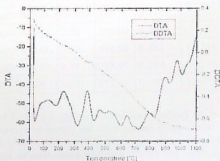


Fig. (e) : DSC curves of lead borate silver glasses series  
 $70\text{PbO}_2-10\text{Ag}_2\text{O}-20\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$



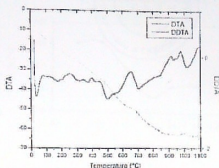


Fig. (f) : DSC curves of lead borate silver glasses series  
 $85\text{PbO}_2\text{-}5\text{Ag}_2\text{O-}10\text{B}_2\text{O}_3$  for  $\text{Ag}_2\text{O/B}_2\text{O}_3 = 0.5$

Materials that are obtained by cooling from the molten state are called glasses and generally have a smaller tendency to crystallize compared with those that can be prepared only by deposition. In certain, cases this reluctance to crystallize makes it possible to heat the material through the softening range of temperature in to the liquid state without any discontinuous change in properties. Such stable glasses may, however undergo a second phase transformation, so called glass transition (transformation) temperature ( $T_g$ ).

In the present work, DSC technique has been employed to determine glass transition temperature ( $T_g$ ), change in heat capacity ( $\Delta C_p$ ), crystallization temperature ( $T_c$ ), crystallization enthalpy ( $\Delta H_c$ ) obtained from the thermograms are reported in table for different compositions of lead silver borate glasses.

Table : Thermal parameters for  $\text{Ag}_2\text{O/B}_2\text{O}_3 = 0.5$  glasses

Sr. No.	Glass Composition	Total Weight loss (%)	$T_g$ (°C)	$\Delta C_p$ (J/g)	$\Delta H$ (J/g)	$E_a$ (J/g-mole K)
1	$10\text{PbO}_2\text{-}20\text{Ag}_2\text{O-}60\text{B}_2\text{O}_3$	07.38	363.98	0.599	13.4079	-
2	$25\text{PbO}_2\text{-}25\text{Ag}_2\text{O-}50\text{B}_2\text{O}_3$	28.992	359.31	0.717	-	-
3	$40\text{PbO}_2\text{-}20\text{Ag}_2\text{O-}40\text{B}_2\text{O}_3$	7.60	336.09	0.538	-	-
4	$55\text{PbO}_2\text{-}15\text{Ag}_2\text{O-}30\text{B}_2\text{O}_3$	7.87	401.80	0.481	1.4726	10157.37
5	$70\text{PbO}_2\text{-}10\text{Ag}_2\text{O-}20\text{B}_2\text{O}_3$	6.78	558.97	0.344	0.6273	1071.193
6	$85\text{PbO}_2\text{-}5\text{Ag}_2\text{O-}10\text{B}_2\text{O}_3$	12.61	455.34	0.197	1.6927	10173.05

The values of  $T_g$  for the glasses studied here (series.) are in well agreement with the values reported by many workers for borate and other glasses (Soppe *et al* 1988, Selvaraj *et al* 1989, Ghosh 1995 and Chaudhary 1995) from the DSC curves the glass transition temperature ( $T_g$ ), change in heat capacity ( $\Delta C_p$ ) and crystallization enthalpy ( $\Delta H_c$ ) are evaluated. The glass transition temperature variation with  $\text{Ag}_2\text{O}$  mol % for different ratio of  $\text{Ag}_2\text{O/B}_2\text{O}_3 = 0.5$  are shown in Figure.

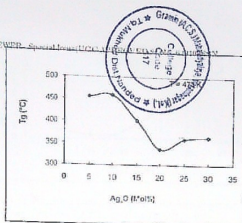


Fig. : Variation of glass transition temperature  $T_g$  with  $\text{Ag}_2\text{O}$  (mol%) for ratio  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

The glass transition temperature ( $T_g$ ) decreases up to 15 mol % of  $\text{Ag}_2\text{O}$  and then increases further for ratio  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$  glasses, the decreasing  $T_g$  with  $\text{Ag}_2\text{O}$  concentration suggested the network structure of glass and consequently at higher concentration of  $\text{Ag}_2\text{O}$  more than 15 mol % the ( $T_g$ ) increases sharply which is an indication of strong network structure of glass. It is noted that the glass transition temperature ( $T_g$ ) varies from 336 to 558.97°C for entire series of glasses.

The decrease in the  $T_g$  decreases in case like vibrational frequency of the most mobile species in the tissue portion of the glass such as  $\text{Ag}^+$  ion. According to the cluster model of glass transition the clusters are the more order groups and the tissue is disorder and distorted regions of the glass structures with the addition of  $\text{Ag}_2\text{O}$ . Addition of metal oxide  $\text{Ag}_2\text{O}$  increases the stability of the glass as compare to the undoped boron oxide glass. This fact is very much related to the ( $T_g$ ) where it is lower for undoped glasses.

The increasing ( $T_g$ ) value after specific mol %  $\text{Ag}_2\text{O}$  in all the glasses of the series, suggests that with increasing composition of  $\text{PbO}_2$  at the cost of  $\text{Ag}_2\text{O}$ , strengthen the network structure of the glass which ultimately decreases the formation of the non bridging oxygen. Variation of change in heat capacity ( $\Delta C_p$ ) with  $\text{Ag}_2\text{O}$  mol% in glass transition region for the ratio  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$  is shown in figure.

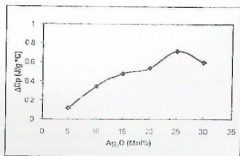


Fig. : Variation of change in heat capacity ( $\Delta C_p$ ) with  $\text{Ag}_2\text{O}$  (mol%) in glass transition region for ratio  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$

It is observed that  $\text{Ag}_2\text{O}/\text{B}_2\text{O}_3 = 0.5$  the change in specific heat increases and is maximum at 25 mol% of  $\text{Ag}_2\text{O}$ . The magnitude of  $\Delta C_p$  is generally indicative of the nature of bonding and is higher for ionic glasses and lower for covalent ones. The higher magnitude of  $\Delta C_p$  in these glasses indicative of ionic bonding.

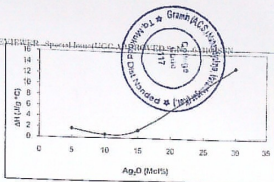


Fig. : Variation of change in enthalpy ( $\Delta H_c$ ) with  $Ag_2O$  (mol%) in glass transition region for ratio  $Ag_2O/B_2O_3 = 0.5$

The area under exothermic peak gives crystallization enthalpy ( $\Delta H_c$ ). The variation of ( $\Delta H_c$ ) with  $Ag_2O$  mol % is shown in figures., for the ratio  $Ag_2O/B_2O_3 = 0.5$ . glass series. The release energy  $\Delta H_c$  are associated with the metastability of the glasses. Larger value of  $\Delta H_c$  are associated with least stable glasses. In the present glass systems  $\Delta H_c$  values are comparatively higher therefore these glasses are least stable.

In DSC curves no prominent exothermic peaks are seen but DDTA curve shows exothermic peaks around  $550^\circ C$  and  $630^\circ C$  which could be corresponds to the formation of the  $Ag_2O \cdot 4B_2O_3$  and  $Ag_2O \cdot 2B_2O_3$  (Boulos *et al* 1971). This fact may be confirm from the X-ray spectra where the peaks are observed at  $2\theta$  angles.

## CONCLUSION:

The magnitude of  $\Delta C_p$  is generally indicative of the nature of bonding and is higher for ionic glasses and lower for covalent ones. The higher magnitude of  $\Delta C_p$  in these glasses indicative of ionic bonding. The exothermic peaks gives crystallization enthalpy ( $\Delta H_c$ ). The variation of  $\Delta H_c$  with  $Ag_2O$  mol% is presented. In DSC curves no prominent exothermic peaks are seen but DDTA curves shows exothermic peaks around  $550^\circ C$  and  $630^\circ C$  which could correspond to the formation of  $Ag_2O \cdot 4B_2O_3$  and  $Ag_2O \cdot 2B_2O_3$ . From the study of physical properties it is observed that the density and molar volume of all glasses studied are composition dependent. The density and molar volume changes with composition which suggests that all the glasses are probably in a single phase with random network structure. It is concluded that the glasses are not soft because they have higher values of  $T_g$  glass transition temperature.

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