

Elastic, Spectroscopic and Thermal Analysis of B₂O₃-CDO Binary Glasses

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Abstract

Borate based glass system of binary B₂O₃-CdO with different compositions are prepared by melt quenching technique. XRD, SEM, ultrasonic, FT-IR, and DTA studies have been employed to study the modification caused by CdO on the structure of the prepared glasses. The absence of Bragg peaks in the XRD pattern confirms the amorphous and homogeneous nature of the glass samples. The SEM image clearly depicts that there is no crystalline phase existing in overall surface of the samples and also confirms the amorphous nature of the samples. The ultrasonic (both longitudinal and shear) velocities are measured using the Pulse Echo Overlap method. Density of the glass samples is measured by Archimedes principle using water as immersion liquid. From the measured data, the elastic moduli and other parameters such as molar volume, Poisson's ratio, acoustic impedance, microhardness and Debye temperature have been evaluated and they are used to gain knowledge about the structural, mechanical properties and are correlated with the rigidity and compactness of the glass systems. IR spectroscopic studies have been carried out to explore the role of divalent cation in the structure of the studied glasses. The transition temperature (T_g), crystallization temperature (T_c) and melting temperature (T_m) is identified using Differential Thermal Analysis (DTA). The glass stability factor and Hruby's parameter are calculated from above mentioned temperatures.

Keywords—XRD, SEM, FTIR, DTA, Ultrasonic velocity

1. Introduction

In recent decades, various experimental methods were employed to study the Structure-property relations, among them elastic properties of the solid are of marked interest, since their measurements provide valuable information regarding the forces that are operative between the atoms and the ions forming the solids. Therefore, it is vital to interpret and understand the bonding nature of the solid and essentially gain knowledge regarding its mechanical properties in order to appropriate the material for a particular application. Several researchers, [1-4] have given manuscripts concerning elastic properties and discussed them to be more suitable for describing the glass structure as the function of composition.

Among various transition metal oxides, CdO is II-VI type semiconductor, acting as a divalent oxide in glass technology. Their appealing properties such as large band gap, low electrical resistivity and transmission in the visible region make them superior to be used in abundant applications. According to

Stanworth [5], they are classified as intermediate oxides with intermediate electronegativity under such a case, the cations form slightly more ionic bonds with oxygen and cannot form glasses by themselves, but can partially replace cations from the network formers.

Despite so many studies have been done on cadmium borate glasses [6,7] the role of cadmium oxides on mechanical properties of borate glasses is not appropriate. The purpose of this work is to elucidate the role of CdO on ultrasonic velocities as well as density of borate glasses. Based on the ultrasonic velocities and density data, some mechanical parameters such as elastic moduli, Poisson's ratio, microhardness and Debye's temperature are obtained for different cadmium borate glass compositions and analyses regarding the elastic property of the material under investigation are encountered. In addition, a detailed report on spectroscopic and thermal analysis of binary B₂O₃-CdO glass system is also presented.

2. Experimental Procedure

2.1 Synthesis

Glass samples belonging to some borate based binary glass system namely BCd were prepared by melt quench technique using the starting materials as B₂O₃ and CdO of reagent purity grade. The required amount (approximately 20g) in mol% of different chemicals in powder form was weighed using a single pan balance having an accuracy of ± 0.001 g. The homogenization of the appropriate mixture of the components of chemicals is attained by repeated grinding using a mortar. The homogeneous mixture is put in a silica crucible and placed in a furnace. Melting is carried out under controlled conditions at a temperature from 800-900 °C for all the samples. The molten mixture was cast into a copper mould having the dimension of 10mm diameter and 6mm thickness. Then the glass samples were annealed at 250 °C for two hours to avoid the mechanical strain developed during the quenching process. The prepared glass samples were polished and the surfaces were made perfectly plane and smoothed by diamond disc and diamond powder. The samples prepared were chemically stable and non-hygroscopic. The nominal composition of BCd glass samples is listed in Table 1.

Table.1. Nomenclature and composition of B₂O₃-CdO binary glass systems

Nomenclature	Composition in mol %	Remarks
BCd30	70 – 30	
BCd35	65 – 35	Mol% of B ₂ O ₃ is decreasing with increasing CdO content
BCd40	60 – 40	
BCd45	55 – 45	

2.2 Characterization

2.2.1 Spectroscopic study

The amorphous nature of the samples is confirmed by X-ray diffraction technique using diffractometers De Rayons X- Inel Equinox 1000 at the range of $2\theta = (10-100)$ degrees utilizing copper radiation with operating voltage 40 Kv 30 mA anode current. The samples were compressed into thin

pellets using KBr for the infrared measurement. The infrared spectra were recorded using Nexus FT-IR at a range of 400 cm⁻¹ to 4000 cm⁻¹ at room temperature.

2.2.3 Ultrasonic study

The longitudinal and shear velocities of the glass specimen were measured using the Pulse –Echo method at 303K by making use of 5MHz X-cut and Y-cut transducers. These transducers were brought into contact with each of the samples by means of a couplant, in order to ensure that there was no air void between the transducers and the specimen. By applying constant pressure on the probe, the echo waveforms were obtained on the display unit and stored in the memory.

Ultrasonic velocity is calculated using the relation.

$$U = \frac{2d}{t}$$

where d and t are the thickness of the specimen (mm) and transit time in microsecond respectively.

2.2.3 Density

The density (ρ) of the prepared glass samples is determined by Archimedes method using double distilled water as buoyant at room temperature. The following relation is used:

$$\rho = \left[\frac{a}{a-b} \right] \rho_w$$

where, a is the weight of the glass samples in air, b is the weight of the glass sample in water and ρ_w is the density of water.

Using the measured values of density and ultrasonic velocities, the elastic moduli and other parameters of the glasses were calculated using the following standard relations.

- (i) Molar Volume (V_m) = $\frac{M}{\rho}$
- (ii) Longitudinal modulus (L) = ρU_ℓ^2
- (iii) Shear modulus (G) = ρU_s^2
- (iv) Bulk modulus (K) = $L - \left(\frac{4}{3}\right)G$
- (v) Poisson's ratio (σ) = $\left(\frac{L - 2G}{2(L - G)}\right)$
- (vi) Young's modulus (E) = $(1 + \sigma) 2G$
- (vii) Acoustic impedance (Z) = $U_t \rho$
- (viii) Microhardness (H) = $(1 - 2\sigma) \frac{E}{6(1 + \sigma)}$
- (ix) Debye temperature (θ_D) = $\frac{h}{K} \left(\frac{9N}{4\pi V_m} \right)^{1/3} U_m$

Where h is Planck's constant, K is Boltzmann's constant, N is Avogadro's number and U_ℓ , U_s and U_m are longitudinal, shear and mean ultrasonic velocities respectively.

U_m is measured using the relation

$$\text{Mean ultrasonic velocity}(U_m) = \left[\frac{1}{3} \left(\frac{2}{U_s^3} + \frac{1}{U_l^3} \right) \right]^{-1/3}$$

2.2.4 Thermal study

Differential thermal analysis (DTA) has been carried out using ThermalAnalyser Netzsch-STA449F3 Jupiterinstrument at a heating rate of 20°C/min in nitrogen gas atmosphere. Following parameters are calculated using DTA spectrogram.

- (i) Glass stability factor (S) = $T_c - T_g$
- (ii) Hruby's Parameter (K_{gl}) = $(T_c - T_g) / (T_m - T_c)$
 where T_g , represents glass transition temperature, T_c , crystallization and T_m , melting temperature.

3. Results and discussion

Spectroscopic study

XRD

The lack of long-range structural order is usually defined by the technique of XRD patterns as shown in fig1. The absence of Bragg peak confirmed that the prepared samples are amorphous and homogeneous in nature. Moreover, the presence of broad humps indicates that there is an existence of short-range order in the glass network[8].

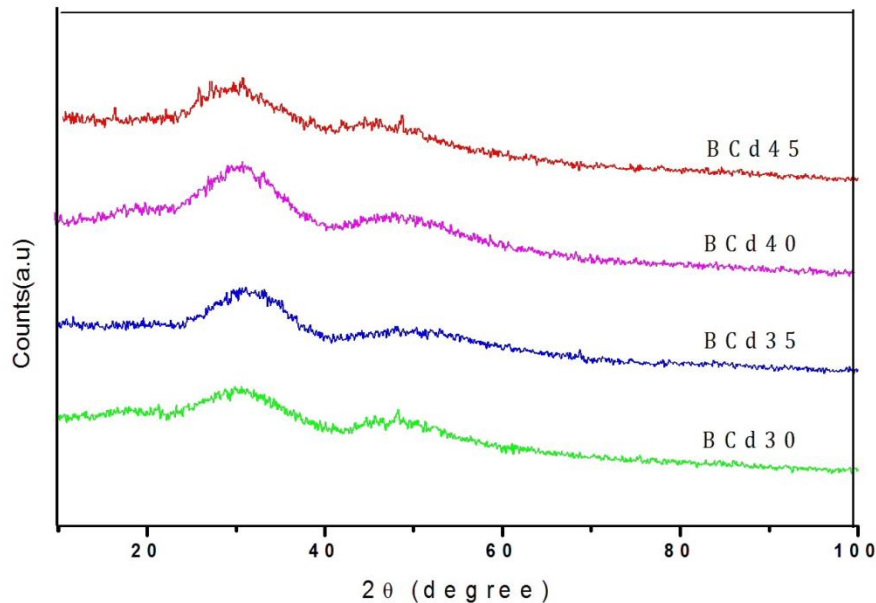


Fig.1.X-Ray diffractogram of $(100-x) B_2O_3 - xCdO$ glass specimens

SEM

The morphological investigation of the prepared glass samples of different stoichiometric ratio are taken using Scanning Electron Micrograph are shown in Fig 2. From the picture, it is observed that different sized grain particles are distributed and the size of the particles varies in each micrograph. The particles are extremely angular and spherical in nature.

Some sphere like agglomerates were found spreading in the glass surface, due to the deposition of amorphous apatite. These results are in agreement with the information provided by the XRD patterns.

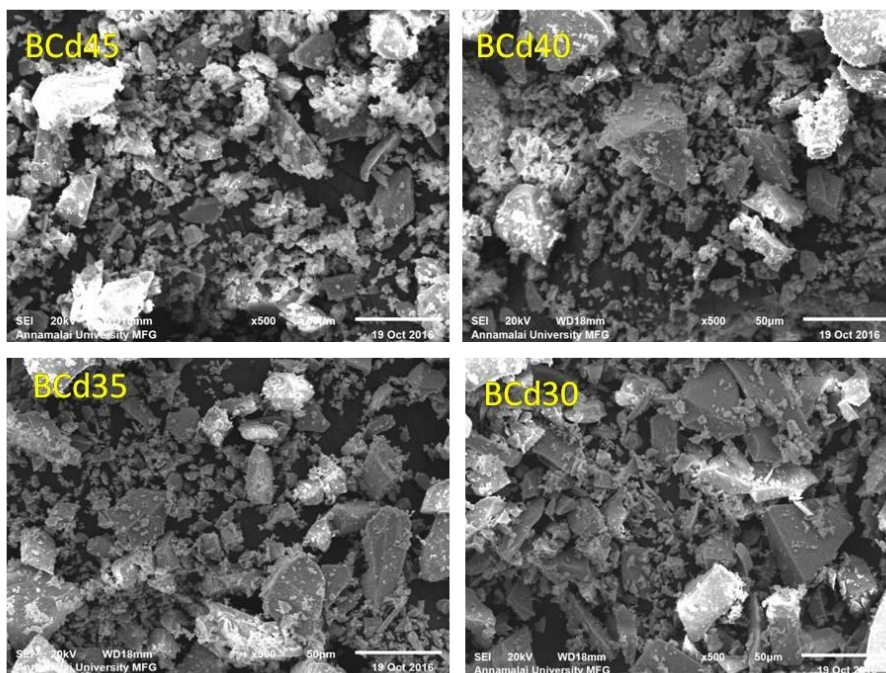


Fig.2. SEM photograph of BCd glass specimen

FT-IR

The infrared transmittance spectra of borate glasses, is a subject of interest for many researchers [9-11] due to their structural peculiarities. Generally, the structure of both crystalline and amorphous B₂O₃ is made up of planar triangles. In case of amorphous B₂O₃, most of these triangles are structured as boroxol rings, where three oxygen atoms become the part of the rings and remaining three out of the ring [12]. These rings are randomly interconnected with loose BO₃ units, on addition of cadmium oxide some of the BO₃ units are converted into BO₄ units with non-bridging oxygen. From fig.3 the broadening of IR bands indicates that there is no detectable variation in peak positions to crystallization. These spectra exhibit absorption bands as a consequence of the general disorder in the network, mainly due to a wide distribution of structural units occurring in these glasses. The observed band positions and their corresponding assignments of samples have been tabulated in Table 2.

The assignment of band positions for different structural units according to various authors [13-15] as follows.

1. 1200-1500 cm⁻¹ is due to the asymmetric stretching vibration of the B-O bond of trigonal BO₃ units containing non-bridging oxygen ions.
2. 800 and 1200 cm⁻¹ is due to the B-O bond stretching of the tetrahedral BO₄ units and
3. Band around 700 cm⁻¹ and is due to bending of B-O-B linkages in the borate network.

The characteristic band (at 806 cm⁻¹) of vitreous B₂O₃ assigned to the symmetric stretching vibration of the boroxolring is found missing in the FT-IR spectra of BCd glasses, which indicates that the boroxol ring in the glass structure is substituted by BO₃ and BO₄ structural units. The band around 2400-3600 is attributed to the molecular water or OH vibrations.

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

Wavenumber cm ⁻¹	Assignment	References
~1384	B-O stretching vibration of the trigonal BO ₃ unit	Abdel Aziz, et al. 2014[16]
~1019	B-O stretching vibration of the tetrahedral BO ₄	Gaafar, et al. 2014[17]
~683	Bending vibration of B-O-B linkage	Rani, et al. 2008[18]
~418	Vibration of metal cation Cd ²⁺	Marzouk, et al. 2014[19]

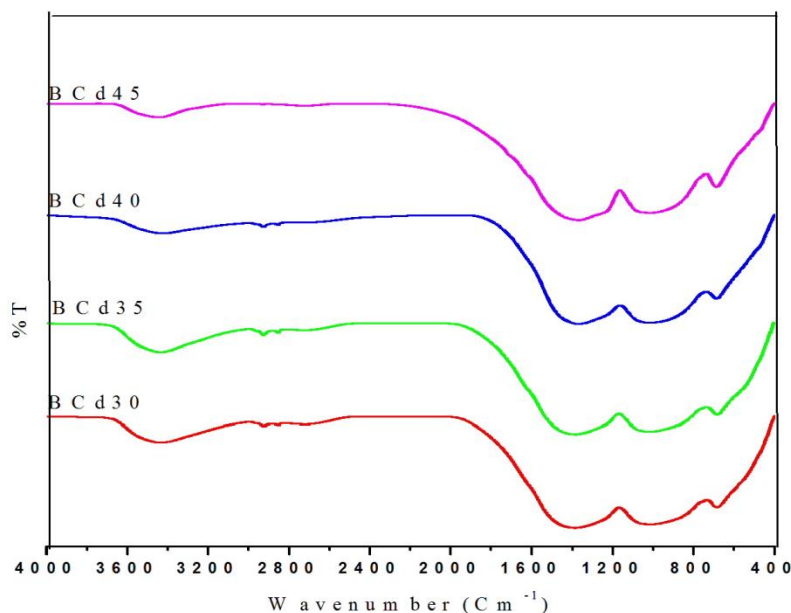


Fig. 3 FTIR spectra for BCd glasses

The studied cadmium borate of glasses of above-mentioned composition consists of BO₃ and BO₄ units in their defined wavenumbers. The presence of tetrahedral structural units confirms the similarity of the role played by CdO like alkali oxides, alkaline earthoxides or multivalent oxides about the ability to

transform some BO₃ units to BO₄ units up to a certain limit and the excess CdO promotes the formation of non-bridging oxygens. The interpretation of fig 3. explains the obvious increase of the intensity of the bands in the range(1200-1600 cm⁻¹) than the intensity of the bands in the range(800-1200 cm⁻¹). The above phenomena explains that the cadmium borate glasses are very similar to great extent to the IR spectra of alkaline earth borate glasses [20]and lithium borate glasses [21].

This is the expected result as it is known that the transformation of the boron from three to four ceases after certain percentage of the modifier oxide (CdO) and is assumed to be (16- 20 mol%). On further addition, excess modifier oxide promotes the formation of non -bridging oxygens. This explains the increases of triangular units than tetrahedral units. The detailed observations of the IR vibrations are tabulated in the table 2. Thus, the progressive addition of CdO supports the formation of BO₃ units with non-bridging oxygen atoms. Therefore, new weaker B-O-Cd linkages are expected to replace the strong B-O-B linkages thereby making them loosely packed.

Ultrasonic study

Ultrasonic examinations are considered in understanding the structural characteristics of glass network. The properties of glass such as elastic properties are dependent on the interatomic forces and potential in lattice structure. The ultrasonic wave velocities as well as density of glass can be taken into account to discover the elastic properties of glass network which are strictly related to the glass structure. Therefore, changes in the glass structure due to modifier doping can be directly noted. Young's, bulk, shear, and longitudinal modulus can be obtained from the density of the solid and longitudinal wave transmission velocity through the solid.

The density is an intrinsic property capable of casting the light on the short-range order structure of a solid like glass. It is known that, B₂O₃ in its glassy form is a laminar network consisting of boron atoms three-fold coordinated with oxygen. Upon modification with divalent oxide cause, the additional oxygen, obtained by the oxide dissociation, favoring conversion from the trigonal boron units BO₃ into four-fold BO₄ coordinated boron atoms. Each BO₄ structural group is negatively charged and the four oxygen are included in the network as bridging oxygen. These units are responsible for the connectivity in the glass network. As a result, the degree of the structural compactness, modification of the geometrical configuration, etc in the glass network can be varied with a change in the composition. Thus, the atomic density clearly reflects the underlying atomic arrangements in a quantitative manner and lends to the support and ideas of Krogh-Moe[22].

The addition of modifier oxide in the glass system changes the density of the structural units by changing the density of the glass system. Because density is an additive property it can be proposed that each oxide in glass would have its contribution towards density. From table 3. density varies from 3.5556 - 3.0968 g/cm³ and the molar volume varies from 24.55-31.23cm³/mol%. These results show that the glass structure relaxes as a result of progressive substitution of CdO. The observed decrease in the density may be explained by the factor that, the cadmium oxide act like an alkali metal oxide promoting the formation of four co-ordinated boron upto certain mol% later the excess of Cd²⁺ ion remains as holes and excess oxygen is unavailable for further conversion. Unlike alkali metal oxide the Cadmium oxide is less probable to occupy the intrinsic position between the linkages. Thus, in the studied glass the molar volume increases which in turn increases the free volume of the glass matrix. This trend supports the so-called open structure, which may be attributed to decrease packing density of the studied glass samples. i.e loose packing increases as the molar volume increases. Fig 4. shows the variation of density and molar volume with respect to mol% and interprets that they show opposite behavior to each other.

According to table.3 and table.4, it was found that the both the ultrasonic velocity, elastic moduli, microhardness and Debye temperature decreases and Poisson ratio increase with the increase of CdO content. The observed decrease in ultrasonic velocity can be explained by two phenomena, first assumption may be due to Cd²⁺ ions enter interstitially as a result of addition of CdO into borate glass and causes some type of modification of B-O-B linkages, that already exist in the glass. The conversion in these linkages results in an increase in the molar volume and decrease in packing density which expands the glass network.

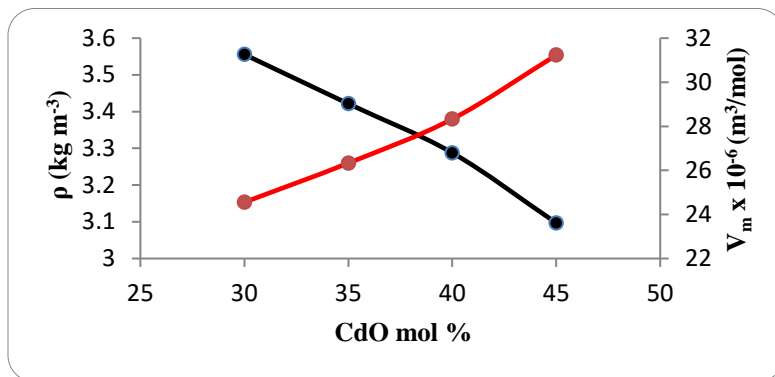


Fig. 4 Variation of density and molar volume with CdO mol%

Therefore, reduction in the rigidity is encountered due to the formation of the NBOs, as observed from the IR spectra. Secondly as proposed by Higazy and Bridge [23], the strain being dependent on the bond stretching force constant, since addition of CdO affects the longitudinal strain in the main chains (B-O-B linkages) causing overall increase in bond length of these linkages, thus decreasing longitudinal velocity.

On the other hand, the decrease in the shear wave velocity indicates that the addition of modifiers has no bending effect on the behavior of bond bending force constant, i.e., the increase in the modifier cations did not contribute to fill the network interstices. A similar interpretation was made by Yasser.B. Saddeek [24] on lead sodium diborate glasses, where the proportion of PbO is varied between 0 to 80 mol%.

Among the various experimental techniques available for studying structure property relations, elastic property of solids is of considerable significance. Because, their measurement yields information concerning the forces that are operative between the atoms or ions comprising solid.

Table 3. Values of density, molar volume, longitudinal velocity and shear velocity of BCd glass systems

Name of the sample	Density (ρ) $\times 10^3 \text{ kgm}^{-3}$	Molar volume (V_m) cm^3/mol	Ultrasonic velocity ms^{-1}	
			Longitudinal (U_l)	Shear (U_s)
BCd30	3.5556	24.55	4697.2	2698.6
BCd35	3.4214	26.33	4512.2	2556.5
BCd40	3.2877	28.32	4338.7	2419.3
BCd45	3.0968	31.23	4229.3	2314.7

This is fundamentally important in interpreting and understanding the nature of the bonding in the solid-state. Therefore, the choice of the most appropriate materials for particular application requires knowledge of its mechanical properties. Hence elastic properties are suitable for describing the glass structure as a function of composition.

As stated by Rajendran et al [25], Young's modulus is sensitive for any changes in the nature of the chemical bond and its strength, which characterize the glass structure, while the bulk modulus is more sensitive in exploring the changes in the cross-link density. From table.4, all the moduli value falls with the increasing concentration causing decrease in the strength and cross-link density.

Debye temperature decreases as the modifier content increases. The decrease in θ_D can be explained by two factors. The first is clarified from the shift of the band towards higher frequency (increase of BO₃ units). The second may be due change in the relative bond strength in the glass network as discussed earlier. Poisson's ratio increases by increasing CdO content due to the presence of large BO₃ with non-bridging oxygen, thereby decreasing dimensionality of the glass network.

Table.4. Values of longitudinal, shear, bulk and young's moduli and Poisson's ratio of BCd binary glass systems

Name of the sample	Longitudinal modulus $L \times 10^9 \text{ Nm}^{-2}$	Shear modulus $G \times 10^9 \text{ Nm}^{-2}$	Bulk modulus $K \times 10^9 \text{ Nm}^{-2}$	Young's modulus $E \times 10^9 \text{ Nm}^{-2}$	Poisson's ratio σ
BCd30	78.45	25.89	43.93	61.16	0.2537
BCd35	69.66	22.36	39.85	53.50	0.2636
BCd40	61.89	19.24	36.23	46.99	0.2744
BCd45	55.39	16.59	33.27	41.24	0.2862

Thermal Properties

It is known that the glass transition temperature (T_g) is affected by the alteration of the glass structure, and the structure of the thermally stable glasses are closely packed one. DTA curves for the studied glass samples with different CdO contents have been obtained. Fig. 5. shows a typical DTA curve for B₂O₃- CdO glass, in which the single endothermic peak is related to the glass transition temperature (T_g), followed by the crystallization temperature (T_c) and endothermic peak (T_m). The single peak of T_g reveals good homogeneity of the prepared glasses.

It is found from the figure.5 that the thermal transition parameters such as T_g , T_c and T_m data shifted to lower temperature with increasing CdO concentration. This is directly reflected on the thermal stability of the investigated glasses. Table 5 shows that the thermal stability and Hruby's parameter decreases with the increasing concentration of Cd²⁺ ions. The bond strength plays a competitive role in decreasing T_g . Since a much higher bond strength of B-O (808.8 kJ/mol) linkage being replaced with weaker bond of Cd-O (235.6 kJ/mol) linkage. A similar result was given by El-Falaky et.al in [26] the study of ultrasonic relaxation in zinc- borate glasses, where the decrease in the T_g was attributed to the replacement of B-O

linkage by the weaker Zn-O linkage. Therefore, for the observed decrease in T_g of the studied glass may also be due to increase of non-bridging oxygen.

Table 5. Values of glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m), Thermal stability (S) and Hruby's parameter (K_{gl}) of BCd glasses

Name of the sample	Glass transition temperature $T_g/^\circ\text{C}$	Crystallization temperature $T_c/^\circ\text{C}$	Melting temperature $T_m/^\circ\text{C}$	Thermal stability S	Hruby's parameter K_{gl}
BCd30	380	632	741	252	2.3119
BCd35	355	597	721	242	1.9516
BCd40	325	566	701	241	1.7852
BCd45	307	480	584	173	1.6635

In general the thermal study of glasses were performed because any change in the coordination number of the network forming atoms, or the formation of non-bridging oxygen, is known to be reflected in T_g . Since it is believed that T_g depends directly on the strength of chemical bonds in the structure the added Cd^{2+} plays the role of a network modifier and the non-bridging oxygen increases with the increase of CdO, thus indicates the breaking of chemical bond and decreases the strength and connectivity in the glass structure.

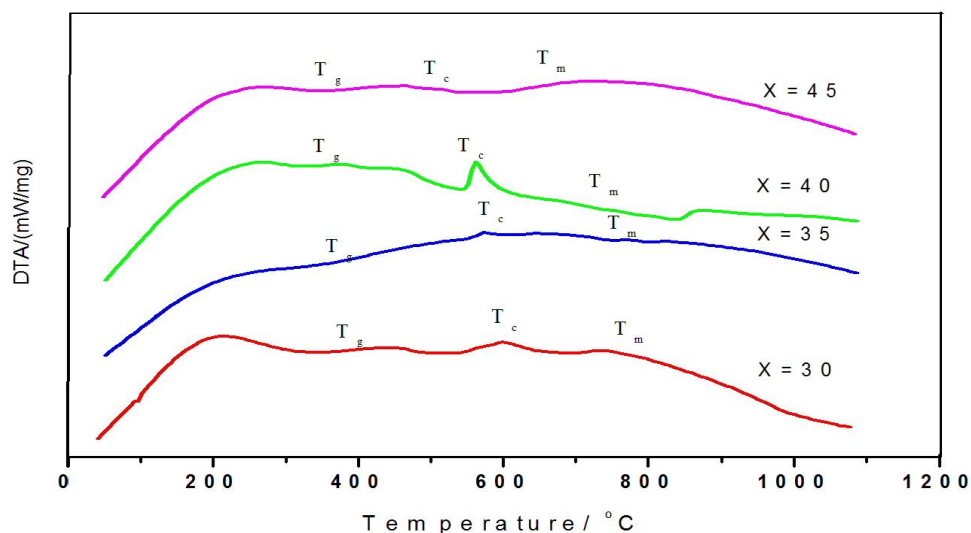


Fig.5. DTA Curve for BCd glasses

CONCLUSION

The XRD and SEM analysis supports the amorphous nature of the semiconducting glasses. From FT-IR analyses, the absence of boroxol ring and its substitution as BO₃ and BO₄ units are reported. Further, the presence of Cd²⁺ ions and the replacement of strong B-O-B linkage by weak B-O- Cd linkage is also seen. The increase in intensity of the BO₃ confirms the presence of a greater number of non-bridging oxygen and account for more loosely packed structure. Ultrasonic and elastic study reveals the decrease in compactness, rigidity, cross-link density and dimensionality of the glass subjected to decreased density, ultrasonic velocities, elastic moduli, microhardness and Debye temperature. On progressive substitution of CdO increase in the molar volume and Poisson's ratio shows the increase in the free volume and proves the presence of more non-bridging oxygen and loosely packed network structure. The decrease in T_g with the corresponding fall in the thermal stability and Hruby's parameter shows overall bond strength decreases with increase in CdO content.

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