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Composition Dependent Structural and Thermal Properties of Sm_2O_3 Doped Zinc Fluoroborate Glasses

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ABSTRACT

Glasses based on Sm^{3+} doped zinc fluoroborate have been synthesized and characterized. Formation of glass has been investigated in the 30 ZnF_2-20 TeO_2-(50-x) B_2O_3-x Sm_2O_3 matrix. Fast quenching is required to prevent melt crystallization and adequate heat treatment to diminish thermal stress, which results in an efficient amorphous material. The Differential Scanning Calorimeter (DSC), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Analysis (EDAX), stability, density and refractive index have been recorded, calculated, measured and analyzed for the glass samples with different concentrations of Sm^{3+} ranging between 0 to 3 mol%. Density increases as dopant concentration increases and glass transition temperature T_g ranges between 395 and 420ºC. The increase of molar volume with Sm_2O_3 content indicates that the extension of glass network is due to the increase of the number of NBOs. The results found in this investigation showed that the refractive index of glass does not only depend on the density but also depends on the electronic polarizability of the glass. The increasing stability of the glass samples shows that they are thermally resistant. The presence of NBOs in the glass network is also approved by the decrease in glass transition temperature (T_g). The variation of the properties with different composition of dopant plays a dominant role in determining a good host material in the field of optics and photonics.

Keywords: Hruby's Parameter, DSC, Zinc Fluoroborate, Samarium Doped

1.INTRODUCTION

Glasses containing rare earth ions have attracted considerable attention because of their wide applications in laser, up-conversion fluorescence, high density optical storage, solar concentrators, wave-guide lasers and in other photo-electronic fields (Polishchuk *et al*., 2011; Lin *et al*., 2005). The fluoride glasses possess high elasticity and its best attribute is that these glasses have a low optical attenuation. These rare earth doped fluoroborate glasses with various visible emissions will be useful in developing new light sources, display devices, UV sensors and tunable visible lasers (Polishchuk *et al*., 2011). Oxyfluoride glasses are of interest from the fundamental point of view: The replacement of fluorine by oxygen affects the glass formation and the structure of glass networks, namely, their connectivity. The oxyfluoride glass matrix can provide a unique environment for rare earth ions, which will differ from both the oxide and fluoride matrices. It is expected that oxyfluoride glasses with high content of rare earth elements can appear to be new functional materials (Polishchuk *et al*., 2011). Transparent oxyfluoroborate rare earth doped glasses are interesting class of materials, which combine the optical advantages of fluoride host with the mechanical advantageous of oxide glass plus highest glass formation tendency of borate atoms.

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The investigations of absorption properties of the Sm^{3+} ions have indicated that the optical properties of these rare earth ions can be affected by varying the glass composition, thus opening up the possibility of engineering application-friendly compositions (Lin *et al*., 2005). With the increasing demand of various visible lasers and light sources, further investigations in other rare earth ions such as Sm^{3+} ions, are becoming more significant. Oxide glasses are attracting hosts for obtaining efficient luminescence in rare earth ions.

In them, borate glasses have been widely investigated due to their technological applications. Boroncan be considered as having the highest glass forming tendency because it in the form of B_2O_3 does not crystallize by itself even when cooled at the slowest rate (Lin *et al*., 2005). It is a suitable optical material with high transparency, low melting point, high thermal stability and good rare earth solubility. However, interest in borate glass is small due to its high phonon energy and it is difficult to obtain high efficient infrared and up-conversion visible emissions. On the other hand, the high phonon energy in borate glass is not detrimental to Sm^{3+} normal 4f transition emissions and sometimes it can accelerate the relaxation processes, which is necessary and beneficial for visible emissions. In this study, borate glass as a suitable host for Sm^{3+} is demonstrated (Lin *et al*., 2005).

Lanthanides themselves cannot form glasses, they can be incorporated into the structure by associating with the glass composition by introducing as dopants and act as active luminescence centers. It is noted that, when rare earth ions are involved in the matrices, their state determined by the 4f electronic configuration depends weakly on the surrounding ions. The glasses containing oxides and fluorides are appropriate objects for this purpose. Their properties are determined by the relatively low energy of phonons, which is explained by the presence of oxides and the chemical and mechanical stabilities associated with the fluorides (Polishchuk *et al*., 2011).

Tellurite glasses are very promising materials for linear and non-linear application in optics, due to some of their important characteristic features such as high refractive index, low phonon maxima and low melting temperature. $TeO_2$is known as a conditional glass former, as it needs a modifier in order to form the glassy state easily. Tellurite glasses continue to intrigue both academic and industry researchers not only because of their technical applications, but also owing to a fundamental interest in understanding their microscopic mechanisms. In general, application and utilities of glassy materials are enormous and are governed by the factors like composition, refractive index and dopants present in the glass. Moreover, rare earth in glassy matrix is strongly dependent on crystal field effects, local environment, phonon energies extended into the band gap. It has been found that glasses with a high density along with a low dispersion usually have high non-linear refractive index (Eraiah, 2010).

B_2O_3 is a glass forming oxide, TeO_2 is a conditional glass former, however, the structural role of zinc in such glasses is the subject of some controversy with evidence for behavior as a network former or a network modifier. Both Zachariasen and Warren's theory of glass structureand Dietzel's crystal field theory classify zinc as a network intermediate, i.e., able to adopt both network former and modifier roles depending upon glass matrix (Cassingham *et al*., 2010). Sm_2O_3 is a modifier in the network, with these chemicals in the glass matrix, a low rate of crystallization, moisture resistant, stable and transparent glasses could be achieved.

The aim of the present paper is to describe detailed thermal behavior of oxyfluoroborate glasses, containing different concentration of Sm^{3+} and to find correlations between rare-earth content, phase composition and thermal properties of these glasses. To the best of our knowledge, there is a lack of information in the literature about thermal properties of Sm^{3+}doped oxyfluoroborate glasses. The presented data from the research done indicates that the glass systems with rare earth element that serve as dopants leads to the design of a new material with wide scope in optics and photonics.

2. EXPERIMENTAL PROCEDURE

Within the glass forming region of ZnF_2-TeO_2-B_2O_3-Sm_2O_3 glass system, the following compositions with successive increase in the concentration of Sm_2O_3 are chosen for the present study:

Sm_0 = 30 ZnF_2-20 TeO_2-50 B_2O_3
Sm_1 = 30 ZnF_2-20 TeO_2-49.5 B_2O_3-0.5 Sm_2O_3
Sm_2 = 30 ZnF_2-20 TeO_2-49 B_2O_3-1.0 Sm_2O_3
Sm_3 = 30 ZnF_2-20 TeO_2-48.5 B_2O_3-1.5 Sm_2O_3
Sm_4 = 30 ZnF_2-20 TeO_2-48 B_2O_3- 2.0 Sm_2O_3
Sm_5 = 30 ZnF_2-20 TeO_2-47.5 B_2O_3-2.5 Sm_2O_3

Appropriate amounts (all in mol%) of reagent grades of ZnF_2, TeO_2, B_2O_3 and Sm_2O_3 powders were thoroughly mixed in an agate mortar-pestle and the homogenously mixed materials were heated in the temperature range 950-1000°C in a PID temperature controlled electric furnace for about 1 h 30 min until a bubble free transparent liquid was formed. The resultant melt was then squeezed on a preheated stainless steel mould and subsequently annealed at 250°C for 2 h. The samples were then ground and optically polished using polisher from Chennai Metco

Bainpol. The final dimensions of the samples used for structural and thermal analysis were about 0.8×0.8×0.2 cm. The density 'ρ' of these finely polishedglass samples wasfound through the standard principle of Archimedes in Contech Analytical Balance (with accuracy of 0.0001 g) using xylene (99.99% pure) as the buoyant liquid. The refractive index of these glasses was measured using Abbe refractometer using sodium-D line-589 nm as source of radiation in Medimeas Instrument. The characteristic temperatures of the glasses are measured by a Differential Scanning Calorimeter (DSC) Shimadzu DSC60 at a heating rate 10 K/min under controlled atmosphere (N_2). The samples (weight≈10-15 mg) are introduced into aluminum pans, which are sealed to prevent the possible contamination of the furnace cell. The estimated values of temperatures are given with an estimated accuracy of about±2°C. Chemical analysis was implemented in a Scanning Electron Microscope (SEM) using an Energy Dispersive X-ray Spectrometer (EDAX) from Zeiss EVO 18 Special.

3. RESULTS

3.1. Structural Analysis

The measurement of density (ρ) and refractive index (n) are the effective tools to explore the degree of structural compactness modification of the geometrical configurations of the glass network (Eraiah, 2006). The density and refractive index of the glass samples determined in the present study are given in the **Table 1** with probable errors ±0.001. The molar volume (V_m) of the glass samples, was calculated using the average Molecular weight (M) and density (ρ) with the following relation:

$$Vm = M / \rho$$

The number density of rare-earth ions (N) of Sm^{3+} was determined using the formula given in equation (Saritha *et al*., 2008):

$$N = [6.023\times10^{23}\times\text{mol\% of cation}\times\text{valency of cation}]/V_m$$

The dielectric constant (ε) was calculated using refractive index (n) of the glass:

$$E = n^2$$

The Molar Refraction, (R_m) of the glass samples were calculated using the formula given (Prajnashree*et al*., 2013, Chimalawong *et al*., 2010, Sidek*et al*.,2009) which is well-known as Volf and Lorentz-Lorenz formula:

$$R_m = \{[(n^2-1)/(n^2+2)]*V_m$$

where, n is the refractive index of the glass sample. ρ is the density and M is the average molecular weight of the glass samples. V_m is molar Volume (V_m) and (n^2-1)/(n^2+2) is the reflection loss.

A condition for predicting metallic or insulating behavior in the condensed state matter is metallization criterion, M = 1-[R_m/V_m]. If R_m/V_m>1, then the materials show metallic nature and if R_m/V_m<1 they exhibit insulating behavior (Dimitrov*et al*., 2010). The so-called metallization parameter values of the present glasses are found to be less than one and are given in **Table 1**. Hence, the present glass systems with their metallization parameter values should exhibit insulating nature.

The molar refraction is related to the structure of the glass and it is proportion to the molar electronic polarizability of the material (α_m). According to the Clausius-Mosotti relation, molar polarizability of the materials (α_m*10^{-24} cm^3) is given by the relation(Prajnashree*et al*., 2013, Chimalawong*et al*., 2010, Sidek*et al*., 2009):

$$\alpha_m = [3/4*\pi*A_v]*R_m$$

where, A_v is the Avogadro's number.

The electronic polarizability of oxide ions (α_O^{2-}) can be calculated on the basis of refractive indices using the following equation:

$$\alpha_O^{2-}(n) = [(R_m/2.52) - \sum \alpha i]*(N_o^{2-})^{-1}$$

where, $\sum \alpha_i$ in the above equation is molar cationpolarizability and N_O^{2-} is the number of oxide ions in the chemical formula. For the studied glasses the values of N_O^{2-} is equal to 1.9. The value of α_{Zn} = 0.286 Å^3 for Zn^{2+} ions, α_{Te}= 1.595 Å^3 for Te ions, α_B = 0.003 Å^3 for B^{3+} and α_{Sm} = 0.92Å^3 for Sm^{3+} ions (Dimitrov*et al*., 2010).

The obtained values of N are used to calculate the polaron radius (r_p), ionic radius (r_i) (Moustafa *et al*., 2008; Rao *et al*., 2011) and Field strength (F) using the relations:

$$r_p = \frac{1}{2}\left(\frac{\pi}{6N}\right)^{(1/3)} \quad r_i = \left(\frac{1}{N}\right)^{(1/3)} \quad and\ F = \frac{z}{r_{p2}}$$

The variation of density, molar volume, electronic polarizability, number density with different composition of Sm_2O_3 is shown in **Fig. 1a and b**.

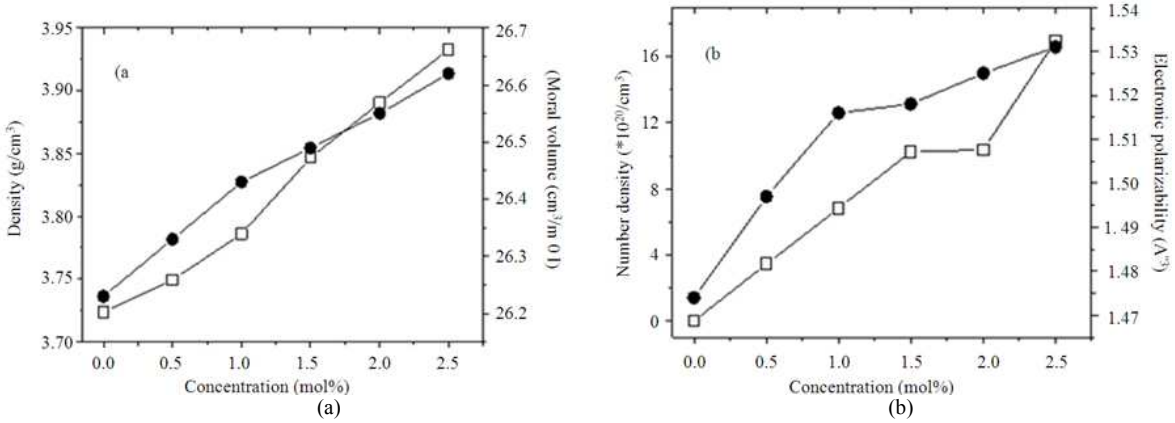


Fig. 1. Variation of (a) density and molar volume (b) number density and electronic polarizability with different composition of Sm_2O_3

Table 1. Physical parameters of the glass system 30 ZnF_2-20 TeO_2-(50-x) B_2O_3-x Sm_2O_3

	Glass code					
Physical parameter	Sm_0	Sm_1	Sm_2	Sm_3	Sm_4	Sm_5
Average Molecular Weight (g/mole)	97.7000	99.1000	100.5000	101.9000	103.3000	104.7000
Density (ρ) (g/cm^3) (±0.0001)	3.7240	3.7494	3.7860	3.8466	3.8903	3.9328
Refractive index (n) (±0.0001)	1.4510	1.4575	1.4610	1.4615	1.4620	1.4630
Optical dielectric constant (ε)	2.1054	2.1243	2.1345	2.1359	2.1374	2.1403
Molar volume (V_m) (cm^3/mol) (±0.01)	26.2300	26.3300	26.4300	26.4900	26.5500	26.6200
Molar refractivity (R_m) (cm^3/mol)	7.0600	7.1700	7.2600	7.2700	7.3000	7.3300
Metallization criterion (M) (1-R_m/V_m)	0.730	0.7270	0.7250	0.7250	0.7250	0.7240
Number density (N) (*10^{20}/cm^3)	---	3.4300	6.8300	10.2300	10.3600	16.9700
Inter ionic distance (r_i) (A^0)	---	14.2900	11.3400	9.9200	9.8800	8.3800
Polaron radius (r_p) (A^0)	---	5.7500	4.5700	4.0000	3.9800	3.3700
Field strength (F) (*10^{15} cm^{-2})	---	3.0400	4.8200	6.3000	6.3500	8.8200
Electronic Polarizability (α_o^{2-} (n)) (Å^3)	1.474	1.4970	1.5160	1.5180	1.5250	1.5310
Molar Electronic polarizability (α_m) (*10^{-24} ions/cm^3)	2.798	2.848	2.8780	2.8820	2.8930	2.9050

4. DISCUSSION

It is evident from **Fig. 1a and 1b** and also **Table 1** that the density, molar volume, number density of the samarium ions, the electronic oxide ion polarizability of the present glass system increases with increase in the content of Sm_2O_3. The dielectric constant is directly correlated with the polarizability of the glass. The dielectric constant gradually increases with increase in the Sm_2O_3 content in the glasses. The chemical compositions of the constituent elements for the thermally evaporated glass sample (where x = 2.5 mol%) have been investigated by means of an Energy Dispersive X-ray Analysis (EDAX) unit interfaced with Scanning Electron Microscope(SEM). Our visual observation, the scanning electron microscope picture of the 2.5% Sm_2O_3 doped glass shown if **Fig. 2a**, indicates the amorphous nature of the glass. **Figure 2b** shows the EDAX spectrum for 2.5 mol% representative sample. The obtained data showed the glass chemicals like Sm, Te, Zn, O and B showing deficiency in Fluorine (F). The deficiency of F may be due to its low melting temperature and there by evaporating after 250°C which is clearly visible in the EDAX spectrum in **Fig. 2b** and weight loss region in the TGA plot (**Fig. 3a**).

3.2. Thermal Analysis

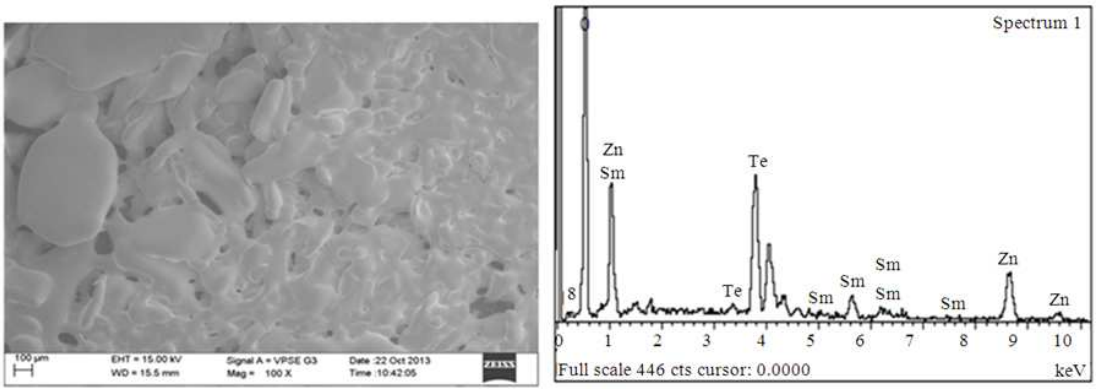
Differential Scanning Calorimeter (DSC) and Thermo Gravimetric Analysis (TGA) no simple way presently exists to formulate the correlation between the ideal composition and the stability of these types of glasses. Different simple quantitative methods have been suggested in order to evaluate the level of stability of the glass alloys.

Most of them as Dietzel and Hruby, are based on the characteristic temperatures such as the glass transition Temperature (T_g), the temperature at which crystallization begins (T_c), the temperature corresponding to the maximum crystallization rate (T_p), or the melting temperature (T_m). Some of the suggested methods are based on the crystallization activation energy. The characteristic Temperatures (T_g, $T_{c,Tp}$ and T_m) are easily and accurately obtained by the Differential Scanning Calorimetery (DSC) during the heating process of the glass sample. Dietzel introduced the first glass criterion, $\Delta T= T_c-T_g$, which is often an important parameter to evaluate the glass forming ability of the glasses (Sailaja *et al*., 2001; Aly *et al*., 2009). In the present work, the results are focused over 2.5 mol% doped glass sample which showed good results compared to other samples. The above mentioned criteria have been applied to the 2.5 mol% Sm^{3+} doped glass with temperature range of 100-600°C. Weight loss was observed in the temperature range between 350-580°C which is clearly indicating the DSC plot under almost the same temperature range. The DSC curve includes temperature of glass Transition (T_g), the onset of crystallization (T_c) and the exothermal maximum (T_p). In most cases the melting Temperature (T_m) does not appear in DSC traces, as it is higher than 600°C. An appropriate value may be found by applying the classical two-third rule expressed as $T_g/T_m = 2/3$ (Bouchaour *et al*., 2005).

The curve exhibits an endothermic effect due to glass transition temperature T_g; the value of T_g is evaluated from the point of inflection of this change. At still higher temperature, an exothermic peak T_c duetocrystal growthfollowedby an endothermic effect due to the melting effect denoted by T_m is also observed (Sailaja *et al*., 2011).

Glass stability may be evaluated using semi-empirical relations based on characteristic temperatures. The thermal stability range (T_c-T_g) provides a good estimate of the tendency of the glass to crystallization state. It should usually be larger than 100°C to obtain thick samples (Sailaja *et al*., 2011; Aly *et al*., 2009; Bouchaour *et al*., 2005). A popular stability scale is based on the Hruby's criterion (Bouchaour *et al*., 2005; Pablick, 2009) defined as $H_r= [(T_c-T_g)/(T_m-T_c)]$. H_r gives information about the nucleation rate of the sample in (T_c-T_g) and growth rate between T_m and T_c embedded in (T_m-T_c). Another stability scale introduced by (Bouchaour *et al*., 2005) takes into account the difference between T_p and T_c, which depends on the width of the crystallization peak. When the ($T_p–T_c$) factor is high the growth rate decreases. This criterion is defined as follows: $S= [(T_p-T_c).(T_c-T_g)]/T_g$(Bouchaour *et al*., 2005).

The values of T_g, T_c, T_p and T_m of all glass samples obtained are furnished in **Table 2**. For 2.5 mol% doped Sm_2O_3 glass the quantity (T_c-T_g), whichis proportional to glass forming ability, is found to be the largest whereas the quantity(T_m-T_c)which is inversely proportional to glass forming ability is the smallest (**Table 2**). The variation of the parameter $(T_c-T_g)/(T_m–T_c)$ with the concentration of Sm_2O_3 shows the maximum value for glass Sm_5 (**Table 2 and Fig. 4a**) indicating its highest glass forming ability among all the glasses under investigation. All the quantities of the glass samples (ΔT, H_g, H_r and S) follow the same trend which indicated the good homogeneity of the glasses prepared. The content Sm_2O_3 increases into the glass systems which results in splitting of glass network former bonds and hence the Bridging Oxygens (BOs) are converted into Non Bridging Oxygen's (NBOs). This results in weakening of the glass structure. Hence, the glass transition temperature decreases with the increase in Sm_2O_3 content.



(a) (b)

Fig. 2. (a) SEM image and (b) EDAX spectrum obtained from 2.5 mol% Sm_2O_3 content in the glass matrix

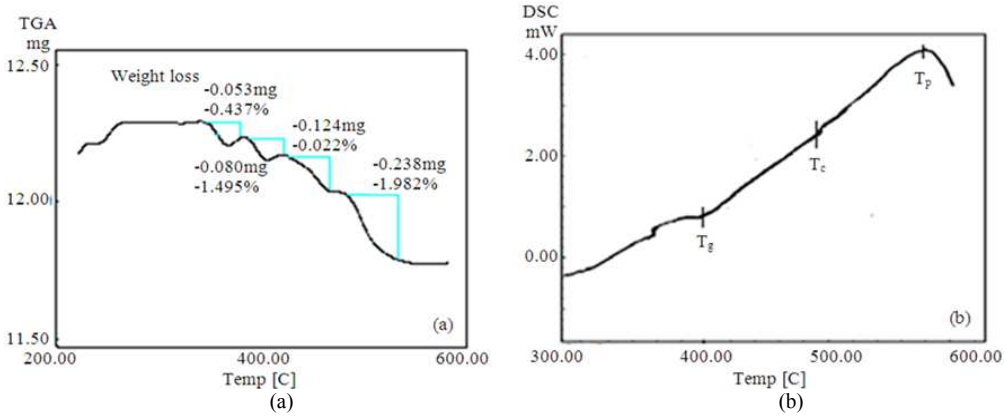


Fig. 3. (a) Thermal Gravimetric Analysis (TGA) (b) Differential Scanning Calorimeter (DSC) thermogram of 2.5 mol% Sm_2O_3 doped sample

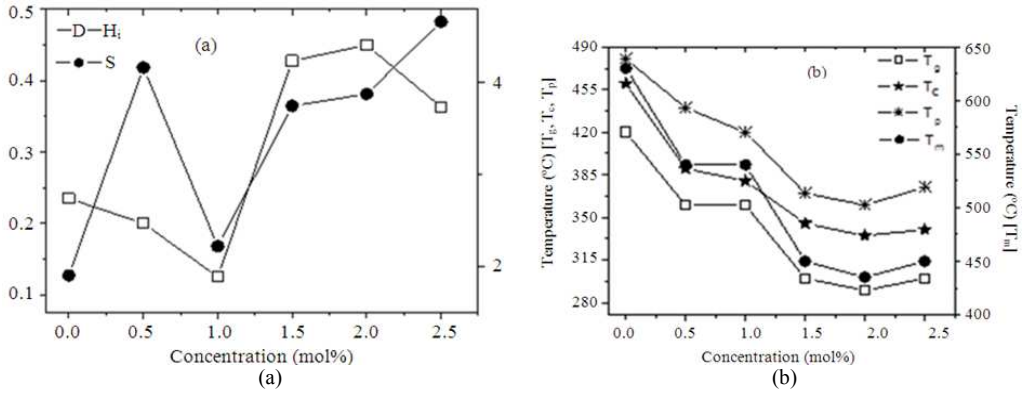


Fig. 4. (a) Stability criteria (b) Evolution of characteristic temperatures versus Sm_2O_3 content in 30 ZnF_2-20 TeO_2-(50-x) B_2O_3-x Sm_2O_3 (x = 0, 0.5, 1.0, 1.5, 2.0, 2.5)

Table 2. Thermal stability parameters of the fluoroborate glasses for the samarium series at a heating rate of 10 K/min. Note that Tg, T_c, T_p, T_m are annotated in degree Celsius [°C] for the calculation of the thermal stability parameters

Sm sample	Sm (mol%)	T_g(mol%)	T_c[°C]	T_p[°C]	T_m[°C]	$\Delta T=T_c-T_g$	$\Delta T = T_m-T_c$	H_g	H_r	S
Sm_0	0.0	420	480	568	630	60	150	0.142	0.400	12.6
Sm_1	0.5	410	495	560	615	85	120	0.207	0.708	13.5
Sm_2	1.0	408	480	555	612	72	132	0.176	0.545	13.2
Sm_3	1.5	405	480	548	608	75	128	0.185	0.585	12.6
Sm_4	2.0	395	464	545	593	69	129	0.174	0.534	14.1
Sm_5	2.5	401	486	589	602	85	116	0.211	0.732	21.8

5. CONCLUSION

The structural and thermal properties for the ZnF_2-TeO_2-B_2O_3-Sm_2O_3 glasses has been evaluated by using various criteria. The H_r criterion has been considered in the present work for the evaluation of glass stability by using DSC data. The obtained results of ΔT, H_g, H_r and S criteria agree satisfactorily with the DSC and TGA data. Sm_5 glass sample showed the good stability factor. This nonmetallic glass with good density exhibits fine structural properties where density reflecting the refractive indices increases with increase in concentration on Sm^{3+} ion in the matrix. This again reproduced in the molar volume, number density, electronic polarizability and different other physical parameters also. SEM reveals the amorphous nature of the glass sample [2.5 mol% glass

sample] and the weight loss in the sample during the DSC is demonstrated in TGA as well in EDAX plot. These findings indicate the structure of Sm_5(30 ZnF_2-20 TeO_2-47.5 B_2O_3-2.5 Sm_2O_3) is more stable when the concentration of Sm_2O_3 present in the glass network is 2.5 mol%.The rise of electronic polarizability in the present glasses indicates the higher ability of oxide ions to transfer electrons to the surrounding cations.The stability of the glasses also increased with increase in Sm_2O_3 concentration. Since our glass samples show good electronic polarizability and stability factor (S), theyare suitable material for the optoelectronic devices. Our study was limited upto 2.5 mol% of Sm_2O_3, further improvement/ degradation in the properties can be investigated beyond this Sm_2O_3 concentration.

6. ACKNOWLEDGMENT

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