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GLASS TRANSITION KINETICS OF SOME $Se_{70}Te_{30-x}Zn_x$ CHALCOGENIDE GLASSES

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The present paper reports the Differential scanning Calorimetric (DSC) study of some $Se_{70}Te_{30-x}Zn_x$ glasses. DSC runs are taken at different heating rates. Well defined endothermic and exothermic peaks are obtained at glass transition (T_g) and crystallization temperatures (T_c). From the dependence of the glass transition temperature on the heating rate β, the activation energy of glass transition has been calculated on the basis of Moynihan and Kissinger models. The value of (T_c - T_g) and stability parameters have also been calculated for each composition. Results indicate that thermal stability increases on addition of Zn to binary $Se_{70}Te_{30}$ glass.

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Keywords: Chalcogenide glasses, D S C technique,Thermal relaxation, Activation energy of glass transition

1. Introduction

Structural studies of chalcogenide glasses are very important for better understanding of transport mechanisms [1-5], application in optoelectronics as wave guide [6], infrared optical fiber [7], semiconducting properties [8, 9] and more recent in optical recording [10]. Among chalcogenide glasses, Se-Te glassy alloys have drawn great interest because of their higher photosensitivity, greater hardness, higher crystallization temperature and smaller ageing effects as compared to pure Se glass for better applications. Moreover, the properties of Se-Te alloys can be improved by adding third element in to it to make it more suitable for various applications. A lot of work has therefore been done on ternary alloys containing Se-Te. The present work deals with the calorimetric studies in some ternary Se-Te-Zn alloys to see the effect of Zn incorporation on various thermal parameters in binary Se-Te system.

Glassy solid state has large viscosity and hence the relaxation kinetics are very slow leaving a few opportunities for local arrangements of bonds and atomic displacements. This type of thermal relaxation depends upon the annealing temperature and may be quite fast near the glass transition temperature. One of the most important problems, in the area of glasses, is understanding of glass transition kinetics [11-13] which can be studied in terms of glass transition temperature (T_g) and activation energy of thermal relaxation (E_t). The glass transition is exhibited as an endothermic peak or a shift in the base line in the scan of Differential Scanning Calorimetry (DSC) due to the change in specific heat. However, in chalcogenide glasses, such an endothermic peak can also be observed due to fast change in enthalpy when the glassy system relaxes quickly due to decrease in viscosity at the glass transition temperature. DSC technique can, therefore, be quite useful in the study of thermal relaxation in these glasses.

The heating rate dependence of T_g is well known, which makes possible the study of glass transition kinetics. We have therefore used non-isothermal DSC technique for the study of glass transition kinetics in the present work. The present paper reports the glass transition kinetics in glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8) system. The activation energy of glass transition is calculated using Kissinger's relation and Moynihan's relation.

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2. Experimental Procedure

Glassy alloys of $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8, 10) were prepared by quenching technique. The exact proportions of high purity (99.999%) elements, in accordance with their atomic percentages, were weighed using an electronic balance (LIBROR. AEG -120), with the least count of 10^{-4} mg. The materials were then sealed in evacuated (~10^{-5}torr) quartz ampoules (length~5cm and internal diameter~8mm). Each ampoule was kept inside the furnace at 1000 ^{0}c temperature (where the temperature was raised at a rate of 3 - 4 ^{0}c ⁄ min.). During heating, all the ampoules were constantly rocked by rotating a ceramic rod to which the ampoules were tucked away in the furnace. This was done to obtain homogeneous glassy alloys.

After rocking for about 10 hours, the obtained melts were cooled rapidly by removing them from the furnace and dropping them to ice-cooled water rapidly. The ingots of the samples were then taken out by breaking the quartz ampoules. The glassy nature of alloys was checked by X-ray diffraction technique.

The glasses thus prepared, were ground to make fine powder for DSC studies.10 to 20 mg of the powder was heated at constant heating rate and the changes in heat flow with respect to an empty reference pan were measured. DSC plus instrument (Rheometric Scientific Company, U. K.) was used at four different heating rates of 5, 10, 15 and 20 ^{0}C/ min. Measurement were made under almost identical conditions.

3. Theoretical Basis

The evaluation of activation energy of glass transition (E_g) from the heating rate dependence of glass transition temperature is widely used in the literature. The theory of glass transition kinetics and structural relaxation as developed by Moynihan and other workers [14-16] has been used for this purpose. Some attempts have also been made to evaluate E_g using Kissinger's relation [17-23]. Since E_g evaluated from this relation has less dependence on thermal history, this method seems to have some extra advantage. As this method is basically given for amorphous to crystalline transformation, the validity of its use for glass transition kinetics has always been questionable. The application of this relation for glass transition means that some kind of transformation is assumed in this case as well. Some authors have given the name of this transformation as the glass – to - amorphous transformation [21]. It is, therefore, interesting to see whether the Kissinger's relation can be applied in general for chalcogenide glasses for evaluating the activation energy of structural relaxation, which is normally obtained by Moynihan's relation .This motivates us to compare the values of activation energy of glass transition process by both the relation in glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4. 6, 8) alloys. The theoretical basis of the two non-isothermal methods is given below:

3.1 Moynihan's relation

Heating rate dependence of the glass transition temperature in chalcogenide glasses is interpreted by Moynihan et. al [16].in terms of thermal relaxation phenomena. In this kinetic interpretation, the enthalpy at a particular temperature and time, H (T, t) of the glassy system after an instantaneous isobaric change in temperature, relaxes isothermally towards a new equilibrium value H_c (T). The relaxation equation can be written as: [14]

$$(\partial H / \partial t)_T = -(H - H_g) / \tau \tag{1}$$

where τ is a temperature dependent structural relaxation time and is given by the following relation

$$\tau = \tau_0 \exp(-E_g / RT) \exp[-C(H - H_c)] \tag{2}$$

Where τ_0 and C are constants and E_g is the activation energy of the relaxation time.Using the above equations, it can be shown [5, 6] that:

$$d \ln \beta / d(1/T_g) = -E_g / R \tag{3}$$

From equation (3), the ln β versus $1/T_g$ plot should be a straight line and the activation energy involved in the molecular motions and rearrangements around T_g can be calculated from the slope of the plot.

3.2 Kissinger's relation

This method is most commonly used in analyzing crystallization data of DSC experiment. During the isothermal transformation, the extent of crystallization (α) of a certain material is represented by the Avrami's equation [24, 25]:

$$\alpha(t) = 1- \exp [-(kt)^{n}] \tag{4}$$

where n is Avrami exponent (order parameter) which depends on the mechanism of growth and on the dimensionality of the crystal growth, K is defined as the reaction rate constant, which is usually assumed to have an Arrhenius temperature dependency.

$$K= K_{o} \exp (-E_{c} / RT) \tag{5}$$

Where E_c is the activation on energy of crystallization and K_o is the frequency factor.
According to Kissinger Eq.(4) can be approximated as:

$$(d\alpha / dt) = n K^{n} t^{n-1}(1- \alpha). \tag{6}$$

Expressing t in terms of α from Eq. (4), the crystallization rate ($d\alpha/dt$) becomes

$$(d\alpha /dt) = An K (1-\alpha) \tag{7}$$

where $A= [-\ln (1-\alpha)]^{(n-1)/n}$

In non-isothermal crystallization, it is assumed that there is a constant heating rate in the experiment. The relation between the temperature T and the heating rate βcan be written in the form:

$$T = T_{i}+ \beta t \tag{8}$$

where T_i is the initial temperature
the derivative of k with respect to time can be obtained from Eq (5) and (8) as

$$\frac{dK}{dt} = \left(\frac{dK}{dT}\right).\left(\frac{dT}{dt}\right) = \left(\frac{\beta E_c}{RT^2}\right)K \tag{9}$$

Using Eq.(7)&(9), Kissinger showed that:

$$\ln (\beta/T_c^{2}) = \text{constant}- E_c /RT_c \tag{10}$$

Where T_c is peak crystallization temperature. Although originally derived for the crystallization process, it is suggested that this relation is valid for glass transition process [26-27] and hence the above equation takes the following form for its use in glass transition kinetics:

$$\ln (\beta/T_g^{2})= -E_g / R T_g + \text{constant} \tag{11}$$

4. Results and discussion

Figs. 1 & 2 show typical DSC thermograms for glassy $Se_{70}Te_{30-x}Zn_x$ (x = 2, 4, 6, 8) at heating rate 20^{0}C/min.Similar thermograms were obtained for other heating rates also (not shown here). It is clear from these figures that well defined and single endothermic and exothermic peaks are observed at glass transition temperature T_g and crystallization temperature T_c respectively, which indicates the homogeneity of these glasses.

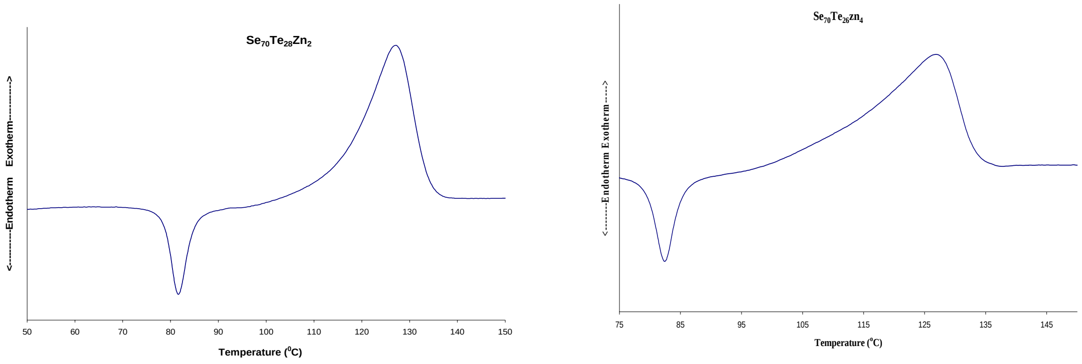

Fig 1.DSC thermograms for glassy $Se_{70}Te_{28}Zn_2$and $Se_{70}Te_{26}Zn_4$ alloy for heating rate20 (^{0}C/min).

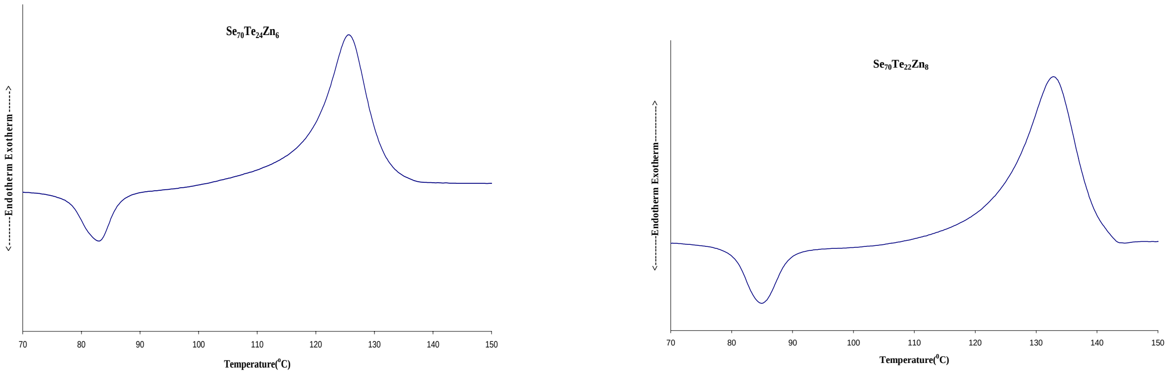

Fig 2. DSC thermograms for glassy $Se_{70}Te_{24}Zn_6$ and $Se_{70}Te_{22}Zn_8$ alloy for heating rate20 (^{0}C/min)

4.1 Heating rate dependence of T_g

The glass transition temperature T_g represents the strength or rigidity of the glassy structure of the alloys. It is well known that T_g of glassy alloys varies with the heating rateβ [31-35]. The empirical relation used to analyze the dependence of T_g on β is of the form:

$$T_g = A + B \log \beta \qquad (12)$$

Where A and B are constants .The values of A indicates the glass transition temperature for the heating rate of 1 K/min. It has been found by various workers that the slope B in eq. (12) is related to the cooling rate of melt. Lower the cooling rate of melt, lower the value of B. The physical significance of B seems to be related with the response of the changes in configuration within the glass transformation region. A plot of T_g vs. log β for glassy $Se_{70}Te_{30-x}Znx$ (x = 0, 2, 4, 6, 8) alloys is shown in Fig.3. Similar curves are obtained for the other glassy alloys. The values of A and B for different alloys are given in Table1. The values of B for glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8) alloys have been found to be different, indicating that these glassy alloys undergo different structural changes. The results shown in Table 1 indicate the validity of this relationship for glassy $Se_{70}Te_{30-x}Znx$ (x = 0, 2, 4, 6, 8) alloys

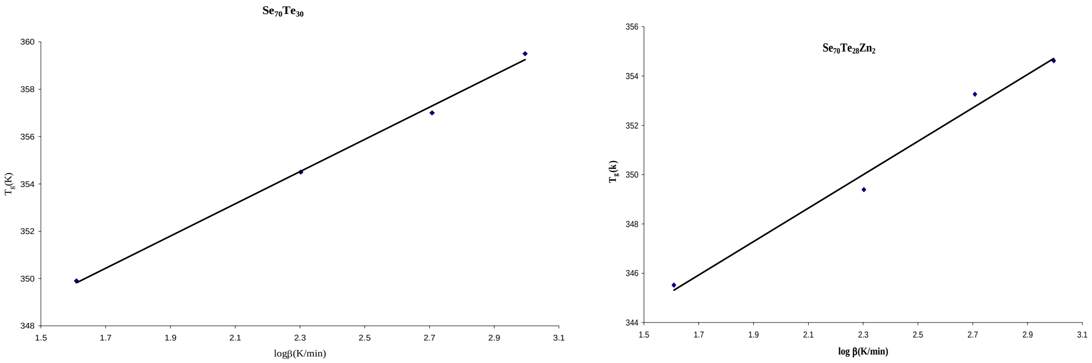

Fig.3 Variation of T_g *with* $\ln\beta$ *for* $Se_{70}Te_{30-x}Zn_x$ *(x =0 & 2)alloys.*

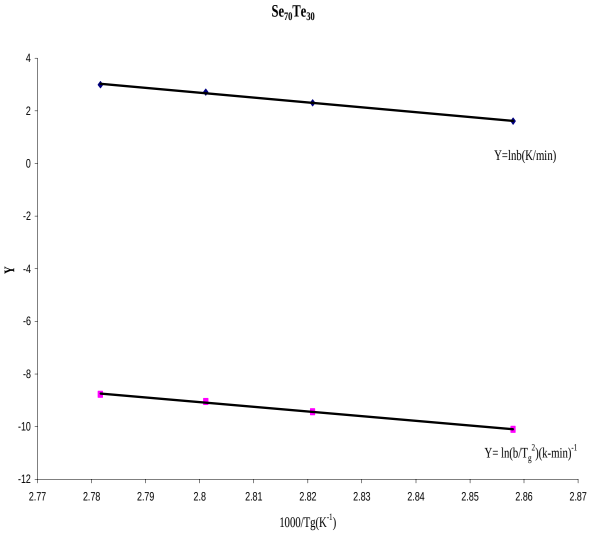

Fig.4. Plots of $\ln(\beta/T_g^2)$*,* $\ln(\beta)$ *against* $10^3/T_g$ *for* $Se_{70}Te_{30}$

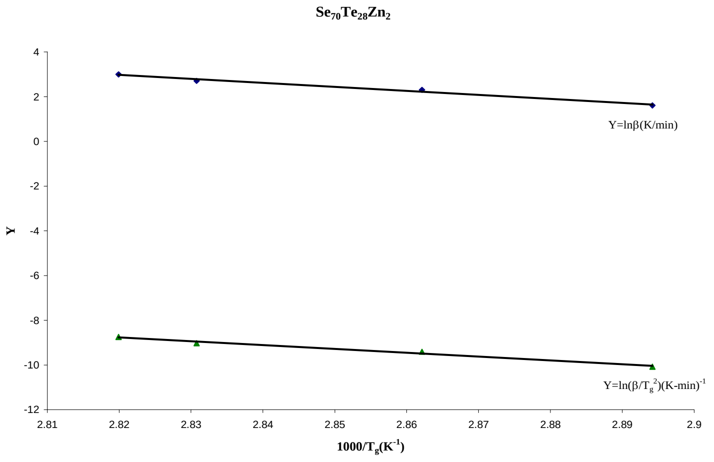

Fig.5. Plots of $\ln(\beta/T_g^2)$*,* $\ln(\beta)$ *against* $10^3/T_g$ *for* $Se_{70}Te_{28}Zn_2$

Table 1. Kinetic parameters of glass transition process in glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8) alloy.

Sample	A(K)	B(min)
$Se_{70}Te_{30}$	338.88	6.80
$Se_{70}Te_{28}Zn_2$	334.41	6.78
$Se_{70}Te_{26}Zn_4$	337.10	6.33
$Se_{70}Te_{24}Zn_6$	338.77	6.13
$Se_{70}Te_{22}Zn_8$	330.43	8.54

Table 2. Glass transition temperature T_g at different heating rates.

Heating rate	$Se_{70}Te_{30}$	$Se_{70}Te_{28}Zn_2$	$Se_{70}Te_{26}Zn_4$	$Se_{70}Te_{24}Zn_6$	$Se_{70}Te_{22}Zn_8$
5^0c/min	349.90	345.52	347.08	348.33	344.88
10^0c/min	354.50	349.39	351.73	352.81	349.56
15^0c/min	357.00	353.26	355.21	357.01	351.46
20^0c/min	359.50	354.62	355.30	355.87	357.95

4.2 Composition Dependence of T_g

From Table 2, it is clear that in glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8), the glass transition temperature T_g decreases with increasing Zn content. The slight decrease of T_g with increasing Zn in the present system may be explained by considering the structural changes occurring due to the further addition of Zn content. The generally accepted structural model of amorphous Se includes [28] two molecular species, meandering chains, which contain helical chains of trigonal Se and Se_8 ring molecules of monoclinic Se. Therefore, as Zn is incorporated in Se-Te, they are probably dissolved in the Se chains increasing relatively the number of Se_8 rings while the numbers of chain Se-Se are decreased [29]. It is known [30] that the glass transition temperature T_g should increase with increasing chain length and decrease with increasing ring concentration. In the present case, the decrease in T_g may, therefore, be related to the increase in ring concentration on increasing Zn concentration.

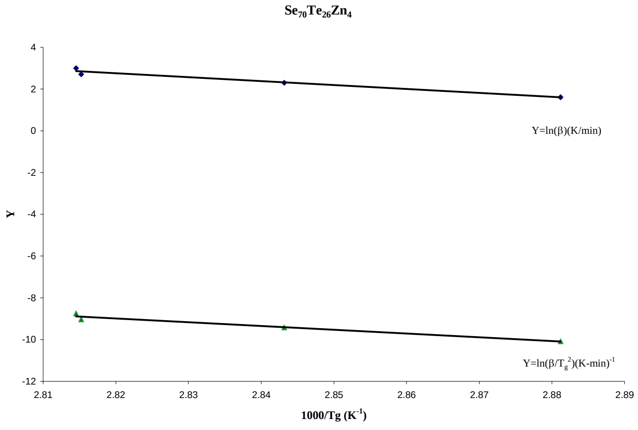

Fig.6-Plots of ln (β/T_g^2), ln (β) against $10^3/T_g$ for $Se_{70}Te_{26}Zn_4$

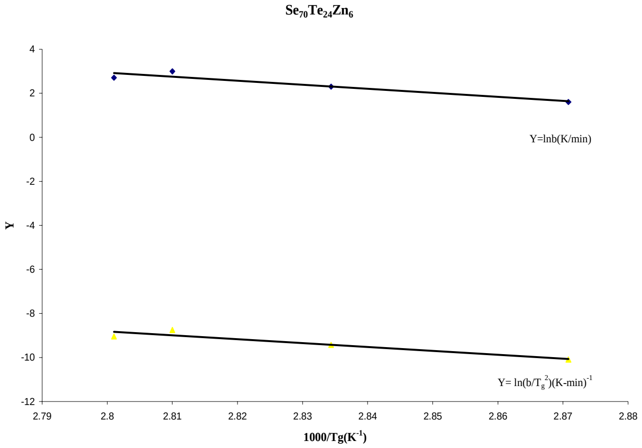

Fig. 7. Plots of ln (β/T_g^2), ln (β) against $10^3/T_g$ for $Se_{70}Te_{24}Zn_6$

4.3 Evaluation of activation energy of glass transition E_g

Using Moynihan's relation, eq.3, the plots of lnβ against $10^3/T_g$ are plotted for various glassy alloys. These plots are shown in Figs. 4 -8. for various glassy alloys of $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8). The slopes of these plots are used to calculate the activation energy of glass transition process. Table 3 shows the E_g values obtained from eq.(3). The values of E_g are also evaluated from the slopes of plots of ln(β/T_g^2) against $10^3/T_g$ for various glassy systems using Kissinger's relation eq.(11). The plots of ln (β/T_g^2) vs. $1000/T_g$ are also shown in Figs. 4 - 8 for glassy alloys of $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8). These values of E_g are given in Table 3.

Table 3: Activation Energy of glass transition E_g (eV).

Sample	Moynihan's relation	Kissinger's relation
$Se_{70}Te_{30}$	1.59	1.52
$Se_{70}Te_{28}Zn_2$	1.54	1.48
$Se_{70}Te_{26}Zn_4$	1.62	1.56
$Se_{70}Te_{24}Zn_6$	1.58	1.52
$Se_{70}Te_{22}Zn_8$	1.13	1.07

It is clear from Table 3 that E_g values obtained from Kissinger's relation are in good agreement with the E_g values obtained using Moynihan's relation. This means that one can use either of equation (3) & (11) to calculate the activation energy of glass transition.

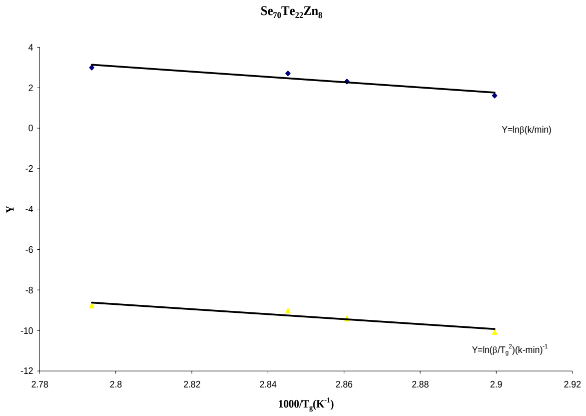

Fig. 8. Plots of $\ln(\beta/T_g^2)$, $\ln(\beta)$ *against* $10^3/T_g$ *for* $Se_{70}Te_{22}Zn_8$

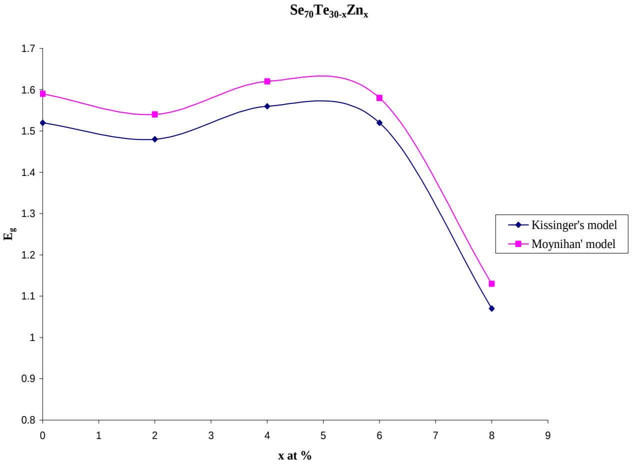

Fig. 9. E_g *vs atomic percentage of Zn in glassy* $Se_{70}Te_{30-x}Zn_x$

It is evident from the Table 3 that E_g does not change significantly at low concentration of Zn. However, at higher concentration (8 at.%), E_g decreases drastically. Fig. 9 shows composition dependence of E_g in glassy $Se_{70}Te_{30-x}Zn_x$.

The decrease in E_g on increasing Zn concentration in ternary alloys $Se_{70}Te_{30-x}Zn_x$ can be explained in terms of atomic weights of Te and Zn. The atomic weight of Zn (65.39 gm/mol) is much less than that of Te (127.60 gm/mol). In the present case, Zn is added in binary $Se_{70}Te_{30}$ at the cost of Te. Thus, the mean atomic weight of ternary alloys is decreased .This may probably be the reason why glass transition occurs in present ternary alloys at comparatively lower activation energies in the present glassy systems.

It is reported that [7], [36-39] E_g is responsible for the molecular motion and rearrangement of the atom around T_g and the glass with lower E_g is the most stable. In the next section, we have shown that thermal stability is more in the present glasses at the composition where E_g is found to be lesser i.e, for x = 8.

4.4 Thermal stability of glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8)

The thermal stability and glass forming tendency (GFT) play an important role in determining the utility of chalcogenide alloys as recording materials due to the fact that phase change optical recording and erasing techniques are based on the laser induced thermal amorphyzation and crystallization of chalcogenide glasses. The origin of the thermal stability and GFT is, therefore, a subject of great interest [40-51]. Recently, much work has been done in this area [41, 52].

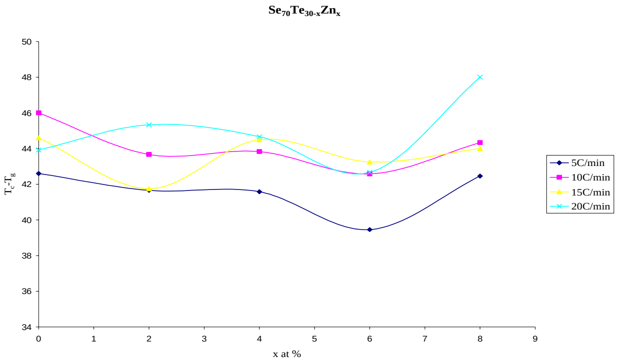

Fig.10 T_c-T_g vs atomic percentage of Zn in glassy $Se_{70}Te_{30-x}Zn_x$

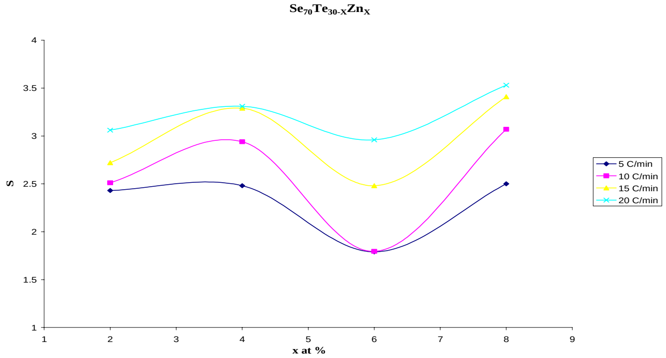

Fig.11. 'S' vs atomic percentage of Zn in glassy $Se_{70}Te_{30-x}Zn_x$

The glass transition temperature T_g represents the strength or rigidity of the glass structure in chalcogenide glasses. Hence, T_g offers valuable information on the thermal stability of glassy state but T_g alone does not give any information about the GFT. However, it has been found that the difference of T_c and T_g is a strong indicator of both the thermal stability and GFT. The higher the values of (T_c-T_g), the greater is the GFT, because the higher the value of this difference, the more the delay in the nucleation process [52]

Table 4 shows the crystallization temperature T_c at various heating rates for all the glasses studied. Values of difference of glass transition temperature (T_g) and crystallization temperature (T_c) for glassy alloys of $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8) are given in Table 5. Fig.10 shows (T_c- T_g) for different compositions at five heating rates. It is interesting to note that the values of (T_c - T_g) is highest at x = 8 in $Se_{70}Te_{30-x}Zn_x$ glassy system. This indicates that GFT is highest in this case among all the glassy alloys studied.

Table 4. Crystallization temperature T_c at different heating rates.

Heating rate	$Se_{70}Te_{30}$	$Se_{70}Te_{28}Zn_2$	$Se_{70}Te_{26}Zn_4$	$Se_{70}Te_{24}Zn_6$	$Se_{70}Te_{22}Zn_8$
5^0c/min	392.5	387.18	388.66	387.79	387.34
10^0c/min	400.5	393.06	395.56	395.39	393.89
15^0c/min	401.6	395.01	399.71	400.26	395.46
20^0c/min	403.4	399.95	399.97	398.54	405.95

Table 5. $T_c - T_g$ *for various glassy alloys of* $Se_{70}Te_{30-x}Zn_x$.

Heating Rate	$Se_{70}Te_{30}$	$Se_{70}Te_{28}Zn_2$	$Se_{70}Te_{26}Zn_4$	$Se_{70}Te_{24}Zn_6$	$Se_{70}Te_{22}Zn_8$
5^0c/min	42.60	41.66	41.58	39.46	42.46
10^0c/min	46	43.67	43.83	42.58	44.33
15^0c/min	44.60	41.75	44.50	43.25	44.00
20^0c/min	43.92	45.33	44.67	42.67	48.00

The thermal stability parameter is also defined in the literature [48] by the following expression:

$$S = (T_c - T_o)(T_c - T_g) / T_g \qquad (13)$$

Here T_o represents the on set crystallization temperature, i.e., the temperature where crystallization peak starts.

Thermal stability parameter reflects the resistance to divitrification after formation of the glass. In eq. (13), the term (T_c- T_o) is related to the rate of divitrification transformation of the glassy phases. On the other hand, the higher values of the term (T_c-T_g) delay the nucleation process. The values of 'S' are given in Table 6 for various glassy alloys at all the heating rates. Fig.11 shows the concentration dependence of S in glassy $Se_{70}Te_{30-x}Zn_x$ at five heating rates. The highest values of 'S' occur at 8 at % of Zn, which shows that this composition is most stable among the various compositions studied here.

Table 6: 'S' values for various glassy alloys of $Se_{70}Te_{30-x}Zn_x$

Heating Rate	$Se_{70}Te_{28}Zn_2$	$Se_{70}Te_{26}Zn_4$	$Se_{70}Te_{24}Zn_6$	$Se_{70}Te_{22}Zn_8$
5^0c/min	2.43	2.48	1.79	2.50
10^0c/min	2.51	2.94	1.795	3.03
15^0c/min	2.72	3.29	2.48	3.41
20^0c/min	3.06	3.59	2.96	3.32

5. Conclusions

Calorimetric measurements have been performed in glassy $Se_{70}Te_{30-x}Zn_x$ (x = 0, 2, 4, 6, 8**)** alloys. DSC scan of these alloys show the well defined single endothermic and exothermic peaks at glass transition temperature T_g and crystallization temperature T_c respectively, which shows homogeneity of these glasses. The activation energy of glass transition E_g has been determined by using Moynihan's relation and Kissinger's relation. On comparison of the values of E_g obtained from these two relations, a good agreement has been found . The composition dependence of E_g values are similar for both the relations (Moynihan's relation and Kissinger's relation). Thus one can use any of the two relations for evaluation of E_g. E_g is found to decrease at higher concentration of Zn (8 at%).

The values of (T_c- T_g) is highest for the sample with 8 at % of Zn, the stability factor S is also highest for the same sample which confirms maximum stability of $Se_{70}Te_{22}Zn_8$ glasses. Hence, one can conclude that the activation energy of glass transition process is related to thermal stability and GFT. Other workers have also found that the stable glass requires less activation energy for glass transition process [36, 53].

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