Glass transition and crystallization study of chalcogenide Se₇₀Te₁₅In₁₅ glass

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Abstract Differential scanning calorimetry data at different heating rates (5, 10, 15 and 20 °C min⁻¹) of Se₇₀Te₁₅In₁₅ chalcogenide glass is reported and discussed. The crystallization mechanism is explained in terms of recent analyses developed for use under non-isothermal conditions. The value of Avrami exponent (*n*) indicates that the glassy Se₇₀Te₁₅In₁₅ alloy has three-dimensional growth. The average values of the activation energy for glass transition, $E_{\rm g}$, and crystallization process, $E_{\rm c}$, are (154.16 ± 4.1) kJ mol⁻¹ and (98.81 ± 18.1) kJ mol⁻¹, respectively. The ease of glass formation has also been studied. The reduced glass transition temperature ($T_{\rm rg}$), Hruby' parameter ($K_{\rm gl}$) and fragility index ($F_{\rm i}$) indicate that the prepared glass is obtained from a strong glass forming liquid.

Keywords Chalcogenide glasses \cdot Differential scanning calorimetry \cdot Non-isothermal methods \cdot The ease of glass formation

Introduction

Chalcogenide glasses have recently emerged as multipurpose materials and have been drawn great attention because of their potential applications in various solid state devices [1–4]. The physical properties of chalcogenide glasses, being amorphous semiconductors, have attracted much

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B. S. Patial · N. Thakur Department of Physics, H. P. University, Summer Hill, Shimla, H.P 171 005, India interest due to their wide range of technological applications. Among these glasses, Se-Te alloys recently gained much importance as these alloys are found to be more photosensitive and harder than amorphous Se. They also have higher glass transition and crystallization temperatures and a smaller ageing effect than pure amorphous Se [5]. Moreover, addition of third element such as In to binary chalcogenide glass produces a higher stability [6–9]. The insertion of the third element expands the glass forming area and also creates compositional and configurational disorder in the system. It is observed that the addition of the third element helps in getting cross linked structure thus increasing the glass transition and crystallization temperature of the binary alloy. There are number of papers [6-9] found in the literature that deals the effect of addition of indium into Se-Te glasses on the crystallization kinetics, thermal stability, glass forming ability, fragility, etc. But no detailed study of crystallization kinetics for the higher content of indium (In > 10 at.%) of Se-Te-In system are reported in the literature. In this article, the effect of higher In impurity of 15 at.% on the crystallization kinetics of Se₈₅Te₁₅ at the cost of Se has been studied.

The crystallization of chalcogenide glasses plays an important role in determining transport mechanism, thermal stability and practical applications. The crystallization study in amorphous materials by differential scanning calorimetry (DSC) has been widely discussed in literature [10–12]. Thermally activated transformations in the solid state can be investigated by isothermal or non-isothermal conditions [13, 14]. In the isothermal method, the sample is brought rapidly to a temperature above the glass transition temperature and the heat evolved during the crystallization process at a constant temperature is recorded as a function of time. In the non-isothermal method, the sample is heated from room temperature generally at a fixed heating rate and

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heat evolved in this case is again recorded as a function of temperature or time. A disadvantage of the isothermal method is the impossibility of getting a test temperature instantaneously and during the time, which system needs to stabilize, no measurements are possible. However, the non-isothermal method does not have this drawback [15].

It is of interest to obtain the crystallization study using different methods for the same experimental data of a glass. The present work studies the crystallization kinetics and evaluates the activation energies for the $Se_{70}Te_{15}In_{15}$ glass under non-isothermal conditions. Using DSC scans of $Se_{70}Te_{15}In_{15}$ glassy alloy at four different heating rates, the values of activation energy for crystallization have been determined by Kissinger's method [16, 17], method of Augis and Bennett [18], Matusita's method [19] and Gao and Wang model [20] as described in the Theoretical basis section. The results obtained have been compared with each other to see the effect of using different methods in the determination of activation energy for crystallization.

Theoretical basis

The theoretical basis for interpreting differential thermal analysis (DTA) or DSC data is provided by the formal theory of transformation kinetics as the volume fraction (χ) crystallized in time (t), using the Johnson–Mehl–Avrami (JMA) equation [21–24]:

$$\chi = 1 - \exp[-(Kt)^n] \tag{1}$$

where n is an integer or half integer which depends on the mechanism of growth and the dimensionality of the crystal. K is defined as the effective overall reaction rate, which is usually assumed to have Arrhenian temperature dependence:

$$K = K_0 \exp\left(-\frac{E_c}{RT}\right) \tag{2}$$

where E_c is the effective activation energy describing the overall crystallization process and K_0 is the frequency factor.

In non-isothermal crystallization, it is assumed that there is a constant heating rate (α) in the DTA or DSC experiments. The relation between the sample temperature (*T*) and the heating rate (α) can be written in the form:

$$T = T_{\rm o} + \alpha t \tag{3}$$

where $T_{\rm o}$ is the initial temperature. The crystallization rate is obtained by taking the derivative of Eq. 1 with respect to time and reaction rate constant is a time function through its Arrhenian temperature dependence, results in the following equation:

$$\dot{\chi} = (1 - \chi) n K^n t^{(n-1)} \left[1 + \frac{t}{K} \dot{K} \right] \tag{4}$$

The derivative of K with respect to time is obtained from Eqs. 2 and 3 as follows:

$$\dot{K} = \frac{\mathrm{d}K}{\mathrm{d}T}\frac{\mathrm{d}T}{\mathrm{d}t} = \frac{\alpha E_{\mathrm{c}}}{RT^2}K\tag{5}$$

Thus, Eq. 4 becomes:

$$\dot{\chi} = (1 - \chi)nK^n t^{(n-1)}(1 + at)$$
(6)
where $a = \alpha E_c/RT^2$

Kissinger's formula

According to Kissinger [16, 17], the term *at* is neglected in comparison to unity when $(E_c/RT^2) < < 1$ and the heating rate α in terms of peak temperature of crystallization can be used to determine the activation energy for crystallization (E_c) and expressed as:

$$\ln\left(\frac{\alpha}{T_{\rm p}^2}\right) = -\frac{E_{\rm c}}{RT_{\rm p}} + \text{const.}$$
⁽⁷⁾

Method of Augis and Bennett

Augis and Bennett [18] developed an accurate method, which is based on substituting u for Kt in Eq. 6. The value of E_c can be derived from this method and the relation used by them is of the form:

$$\ln\left[\frac{\alpha}{(T_{\rm p} - T_{\rm o})}\right] \simeq -\frac{E_{\rm c}}{RT_{\rm p}} + \ln K_0 \tag{8}$$

Matusita's method

The most widely used model, the JMA model, is derived for isothermal crystallization. Attempt has been made to apply the JMA model for non-isothermal conditions. In the non-isothermal method, the crystallized fraction in a glass heated at a constant rate α is related to the activation energy of crystallization (E_c) is given by Matusita's [19] relation:

$$\ln[-\ln(1-\chi)] = -n\ln(\alpha) - 1.052mE_{\rm c}/RT + {\rm const.}$$
(9)

where χ is the crystallized fraction and *m* and *n* are integer or half integer numbers that depend upon the growth mechanism and the dimensionality of the glassy alloy. For as-quenched glass containing no nuclei *m* is equal to (n - 1) and for glass containing a sufficiently large number of nuclei, which might occur due to the annealing of the asquenched glass *m* is taken equal to *n*. Since the as-quenched samples are studied, the value of *m* is taken as m = (n - 1).

Gao and Wang model

A method suggested by Gao and Wang [17] uses the following expressions to determine the activation energy and frequency factor:

$$\ln\left(\frac{\mathrm{d}\chi}{\mathrm{d}t}\right)_{\mathrm{p}} = -\frac{E_{\mathrm{c}}}{RT_{\mathrm{p}}} + \mathrm{const.} \tag{10}$$

where $(d\chi/dt)_p$ is the rate of transformed volume fraction at the peak of crystallization. To obtain the information about morphology of growth and Avrami exponent, the following relations of Gao and Wang model [20] have been used:

$$K_{\rm p} = \frac{\alpha E_{\rm c}}{RT_{\rm p}^2} \tag{11}$$

$$\left(\frac{\mathrm{d}\chi}{\mathrm{d}t}\right)_{\mathrm{p}} = 0.37nK_{\mathrm{p}} \tag{12}$$

where *n* is Avrami exponent, K_p is the reaction rate constant and other symbols have their usual meanings.

Experimental details

Glassy alloy of Se₇₀Te₁₅In₁₅ is prepared using melt quenching technique. 5 N pure materials are weighed according to their atomic percentages and sealed in a quartz ampoule (length ~ 5 cm, diameter ~ 12 mm) under a vacuum of $\sim 2 \times 10^{-5}$ mbar. The sealed ampoule is kept inside a furnace where the temperature is raised to 900 °C at a rate of 3-4 °C min⁻¹. The ampoule is rocked frequently for 10 h at maximum temperature to make the melt homogenous. The quenching is done in the ice cooled water. The bulk sample has been characterized by X-ray diffraction technique and is found to be amorphous in nature as no prominent peak is observed in the spectrum. The composition of the glassy alloy has been measured by an electron microprobe analyzer (JEOL 8600 MX). For the compositional analysis, the constitutional elements Se, Te and In has been taken as reference samples. The composition of the bulk Se₇₀Te₁₅In₁₅ sample is found to be uniform within the measurement accuracy of about $\pm 2\%$.

The thermal behaviour of the glasses is investigated using Mettler Toledo Star^e DSC system. Approximately, 5 mg of sample in powder form is sealed in standard aluminium pan and DSC runs are taken at four different heating rates from 5 to 20 °C min⁻¹ to obtain glass transition temperature (T_g), onset temperature of crystallization (T_o), peak crystallization temperature (T_p) and melting temperature (T_m). The crystallized fraction (χ) at a given temperature is given as $\chi = (A_T/A)$, where A is the total area of the exothermic peak between the initial temperature (T_i) where crystallization just begins and the temperature (T_f) where the crystallization is completed. A_T is the area between T_i and T. A best fit for the results is calculated by the least square fitting method for the activation energies and other kinetic parameters.

Results and discussion

Figure 1 shows the DSC curves of $\text{Se}_{70}\text{Te}_{15}\text{In}_{15}$ at four different heating rates (5, 10, 15 and 20 °C min⁻¹). The exothermic peaks are sharp and symmetric. From these DSC curves, glass transition temperatures and crystallization temperatures are observed at endothermic and exothermic peaks, respectively. It is observed from Fig. 1 that the glass transition temperature (T_g) and the peak temperature of crystallization (T_p) shift to a higher temperature with increasing heating rate. The values of glass transition temperature (T_g), peak temperature of crystallization (T_p) and melting temperature (T_m) at all heating rates are given in Table 1.

Glass transition activation energy

Glass transition kinetics can be described in terms of the glass transition temperature (T_g) and the activation energy of glass transition (E_g) . The glass transition temperature T_g represents the strength or rigidity of the glassy structure of the alloy. Two approaches have been used to study the dependence of T_g on the heating rate. The first one corresponds to the heating rate dependence of T_g expressed in terms of the following empirical relation [25]:

$$T_{\rm g} = A + B \ln(\alpha) \tag{13}$$

where A and B are constants depending upon the glass composition and α is the heating rate used in DSC scans.



Fig. 1 DSC curves of chalcogenide $\mathrm{Se}_{70}\mathrm{Te}_{15}\mathrm{In}_{15}$ glass at different heating rates

Heating rate	$T_{ m g}$	$T_{ m p}$	T _m	$T_{ m rg}$	$K_{ m gl}$	F_{i}	
5	344.79	392.38	537.55	0.64	0.33	33.41	
10	348.95	399.09	539.98	0.65	0.36	23.08	
15	351.65	405.10	541.29	0.65	0.39	19.47	
20	353.78	408.37	543.18	0.65	0.40	17.49	

Table 1 Parameters determined from the heating rate data for $Se_{70}Te_{15}In_{15}$ chalcogenide glass

Plot of $T_{\rm g}$ versus $\ln(\alpha)$ for Se₇₀Te₁₅In₁₅ glass is shown in Fig. 2, which confirms the validity of the relation. The calculated values of A and B are (334.31 ± 0.5) K and (6.44 ± 0.2) min for Se₇₀Te₁₅In₁₅ glass, respectively.

The other approach is the use of Eq. 7 for the evaluation the activation energy for glass transition, E_g . In spite of the fact that the Kissinger's equation is basically meant for the determination of activation energy for crystallization process, the similar equation can be used for the evaluation of glass transition activation energy. The justification of applying this method for the evaluation of the glass transition activation energy comes from the shifting of glass transition peaks at different heating rates similar to crystallization peaks and may be written as:

$$\ln\left(\frac{\alpha}{T_g^2}\right) = -\frac{E_g}{RT_g} + \text{const.}$$
(14)

where R is the universal gas constant.

In addition, the heating rate dependence of the glass transition temperature in chalcogenide glasses is interpreted by Moynihan et al. [26] in terms of the thermal relaxation phenomenon. 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 $H_e(T)$. The relaxation equation can be written in the following form [27]:



Fig. 2 T_g versus $\ln(\alpha)$ plot for the Se₇₀Te₁₅In₁₅ chalcogenide glass

$$\left(\frac{\delta H}{\delta t}\right)_{T} = -\frac{(H - H_{\rm e})}{\tau} \tag{15}$$

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

$$\tau = \tau_0 \exp(-E_g/RT) \exp[-c(H - H_e)]$$
(16)

where τ_o and *c* are constants and E_g is the activation energy of relaxation time. Using the above equations it can be shown [26–28] that:

$$\ln(\alpha) = -E_{\rm g}/RT_{\rm g} + \text{const.}$$
(17)

Equation 17 states that $\ln(\alpha)$ 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.

Figure 3 shows the plots of $\ln(\alpha/T_g^2)$ and $\ln(\alpha)$ versus 1000/ T_g for the Se₇₀Te₁₅In₁₅ glass, displaying the linearity of the equations used. The values of the activation energy calculated using Eqs. 14 and 17 are (151.26 ± 0.5) kJ mol⁻¹ and (157.06 ± 0.5) kJ mol⁻¹, respectively. From the comparison of these two results, it is evident that the two deduced values, so obtained by using Eqs. 14 and 17, are in good agreement with each other. This means that one can use either of the two relations to calculate glass transition activation energy, E_g . The average value of E_g obtained for the glass transition is (154.16 ± 4.1) kJ mol⁻¹.



Fig. 3 $\ln(\alpha/T_g^2)$ and $\ln(\alpha)$ versus $1000/T_g$ for Se₇₀Te₁₅In₁₅ chalcogenide glass

Crystallization activation energy and crystallization reaction order

The activation energy of crystallization is normally obtained using different theoretical models. The most widely used model is the JMA model for non-isothermal kinetics. This model implies that the Avrami exponent, n, and the activation energy (E_c) should be constant during the transformation process. The analysis of the experimental crystallization data is given on the basis of four different approaches [16–20] pertaining to the kinetics of phase transformation involving nucleation and growth process under non-isothermal conditions. In general, the crystallization process is well characterized when the three kinetic parameters, activation energy for crystallization (E_c), Avrami exponent (n) and frequency factor (K_0) are determined.

Using Kissinger's method, Eq. 7, the activation energy for crystallization (E_c) of the chalcogenide glass can be obtained from the heating rate dependence of the peak temperature of crystallization. Figure 4 shows the plot of $\ln(\alpha/T_p^2)$ versus 1000/ T_p for the glassy Se₇₀Te₁₅In₁₅ bulk sample. The deduced value of E_c is (106.60 ± 0.8) kJ mol⁻¹.

The activation energy for crystallization can also be calculated using Eq. 8 suggested by Augis and Bennett [18]. Augis and Bennett (Eq. 8) considered the effect of $\ln(T_p - T_o)$ in the calculation of E_c . When $\ln[\alpha/(T_p - T_o)]$ is plotted against $1000/T_p$, a straight line is obtained whose slope is E_c/R as shown in Fig. 4. The value of E_c determined by this method is (86.18 ± 1.6) kJ mol⁻¹. The frequency factor K_0 can be calculated from the vertical axis intersection as given by using Eq. 8 given by Augis and Bennett method [18] and the obtained value of K_0 is (2.55 × 10⁹) s⁻¹.

Following the method suggested specifically for nonisothermal data suggested by Matusita et al. [19], Eq. 9, the order of crystallization reaction (Avrami exponent n) and the activation energy for crystallization can be calculated for the amorphous-crystalline transformation. A plot of $\ln[-\ln(1-\chi)]$ versus 1/T at a uniform heating rate should give a straight line. Figure 5 shows linear $\ln[-\ln(1-\gamma)]$ versus 1000/T plots measured at different heating rates for $Se_{70}Te_{15}In_{15}$ glassy sample. According to Eq. 9, the average value of the mE_c is found equal to 45.78 kJ mol⁻¹. Figure 6 shows linear plots of $\ln[-\ln(1 - \chi)]$ versus $\ln(\alpha)$ at a fixed temperature T = 397.53 K for Se₇₀Te₁₅In₁₅ glass. The average value of n has been taken. From the mean value of n it is possible to postulate a crystallization mechanism. It has been that n may be 4, 3, 2 or 1 [29], depending upon the type of nucleation and growth of the system.

The average value of *n* for Se₇₀Te₁₅In₁₅ chalcogenide glass is found to be 4.45. Normally, *n* should not exceed 4 (i.e. the value of three-dimensional bulk nucleation). While the high values of Avrami exponent (n = 4.45) for the composition Se₇₀Te₁₅In₁₅ indicates very high nucleation rate with three-dimensional growth. But high values of *n* have been reported for other glasses [29–31] also. From the value of *n* and the average mE_c , the activation energy for crystallization of the Se₇₀Te₁₅In₁₅ glassy alloy can be calculated and is found equal to (120.61 ± 19.1) kJ mol⁻¹.

Gao and Wang [20] proposed a slightly different method to analyse DSC curves in terms of the activation energy E_c , the dimensionality, the rate constant in atomic diffusion, the microstructure of amorphous alloy, the frequency factor K_0 , etc. during the crystallization process. This equation is also derived from JMA transformation equation. This equation shows the relationship between the maximum crystallization rate and the crystallization peak temperature



Fig. 4 $\ln(\alpha/T_p^2)$ and $\ln[\alpha/(T_p - T_o)]$ versus $1000/T_p$ for $Se_{70}Te_{15}In_{15}$ chalcogenide glass



Fig. 5 $\ln[-\ln(1 - \chi)]$ against 1000/*T* for Se₇₀Te₁₅In₁₅ chalcogenide glass at different heating rates



Fig. 6 $\ln[-\ln(1-\chi)]$ against $\ln(\alpha)$ for the Se₇₀Te₁₅In₁₅ chalcogenide glass at different temperatures

as given in Eq. 10. Moreover, this equation has successfully predicted that the maximum rate of crystallization in DSC increases by the same factor as the heating rate does. Figure 7 shows the plot of $(d\gamma/dt)$ against temperature (T) at different heating rates for $Se_{70}Te_{15}In_{15}$ glass. It is clear from Fig. 7 that the peak height increases and shifts towards higher temperature values with the increase in heating rate. This is due to the fact that as the heating rate is increased from 5 to 20 °C min⁻¹, the rate of crystallization increases and crystallization shifts towards higher temperatures hence, the peak shifts towards higher temperature values. On the other hand we can say that with this increased rate of crystallization, a greater volume fraction is crystallized in a smaller time as compare to the low heating rate, which further signifies the increased peak height with increase in heating rate in these curves. The observed increase in $(d\chi/dt)_p$ values as the heating rate does has been widely discussed in the literature [20].

The slope of plot of $\ln(d\chi/dt)_p$ versus $1000/T_p$ (Fig. 8) gives the activation energy of crystallization (E_c) and is found equal to (81.86 ± 1.3) kJ mol⁻¹. The Gao and Wang method is also used to evaluate the Avrami exponent and the value of Avrami exponent using Eq. 12 is found equal to 4.46 which is in good agreement with that obtained from Matusita's method. From the value of *n* using Eqs. 9 and 12, the crystallization mechanism is concluded to be three-dimensional growth.

Table 2 lists the values activation energy of crystallization (E_c) of Se₇₀Te₁₅In₁₅ glassy system. A considerable difference has been observed in the values of E_c evaluated by different formulations. This may be due to different approximations that have been adopted while arriving at the final equation of the various formalisms. On the other hand, the simplified Eq. 8 is the conventional equation for the evaluation of the activation energy for crystallization



Fig. 7 Crystallization rate versus temperature of the exothermic peaks at different heating rates



Fig. 8 $\ln(d\chi/dt)_p$ versus $1000/T_p$ plot for Se₇₀Te₁₅In₁₅ chalcogenide glass

because of the convenience and accuracy of the measurements of heating rate.

The ease of glass formation

The ease in glass formation is determined by calculating the reduced glass transition temperature [32]:

$$T_{\rm rg} = \frac{T_{\rm g}}{T_{\rm m}} \tag{18}$$

Two-third rule holds good for this composition at all heating rates (Table 1). The average value of reduced glass transition temperature $T_{\rm rg}$ of this composition is 0.65 showing that the ease of glass formation of Se₇₀Te₁₅In₁₅ chalcogenide glass is high.

Hruby [33] has introduced a parameter, K_{gl} , as an indicator of the glass forming ability (GFA) and is given by:

Table 2 The average values of activation energy for crystallization E_c deduced from different methods used

Method	Equation	$E_{\rm c}/{\rm kJ}~{\rm mol}^{-1}$	
Kissinger's method	(7)	106.60 ± 0.8	
Augis and Bennett method	(8)	86.18 ± 1.6	
Matusita's method	(9)	120.61 ± 19.1	
Gao and Wang model	(10)	81.86 ± 1.3	
Average value		98.81 ± 18.1	

$$K_{\rm gl} = \frac{(T_{\rm p} - T_{\rm g})}{(T_{\rm m} - T_{\rm p})}$$
(19)

where $T_{\rm m}$ is the melting temperature. The values for different heating rates vary from 0.33 to 0.40 and are listed in Table 1.

Since there is no absolute criterion to parameterize the glass formation, the empirical parameters extensively used for its quantitative characterization have been evaluated. The fragility index F_i is an important parameter of GFA and it is a measure of the rate at which the relaxation time decreases with increasing temperature around T_g and is given [34, 35] by:

$$F_{\rm i} = \frac{E_{\rm g}}{RT_{\rm g}\ln(\alpha)} \tag{20}$$

where E_g is the apparent activation energy for the glass transition. According to Viglis [36] glass forming liquids that exhibit an approximate Arrhenius temperature dependence are defined as strong and specified with a low value of F_i ($F_i \approx 16$), while the limit of fragile glass forming liquids characterized by a high value of F_i ($F_i \approx 200$). The values of F_i for all heating rates have been calculated (Table 1) and indicates that fragility index F_i for the present glass is 17.49–33.41. Since these values of F_i are within the above mentioned limit, it is reasonable to state that Se₇₀Te₁₅In₁₅ glass has been obtained from the good glass forming liquid.

Conclusions

Crystallization kinetics of Se₇₀Te₁₅In₁₅ glass has been studied successfully using DSC under non-isothermal conditions using methods deduced specifically for isothermal conditions. From the heating rate dependences of the glass transition temperature (T_g) and the peak temperature of crystallization (T_p), the kinetic parameters; activation energy for glass transition, activation energy for crystallization, kinetic exponent, *n*, reduced glass transition temperature, Hruby's parameter and fragility index have been deduced. The average value of activation energy for the crystallization process of the investigated glass using different methods is (98.81 ± 18.1) kJ mol⁻¹. According to the Avrami exponent, the crystallization mechanism may be interpreted as three-dimensional growth. The average value of the activation energy of the glass transition (E_g) is found equal to (154.16 ± 4.1) kJ mol⁻¹.

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