

Thermal Characterization of $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ Glasses Using Iso-Conversional Methods

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ABSTRACT

Alloys of $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ glassy system are obtained by quenching technique and crystallization kinetics has been studied using Differential Scanning Calorimetric [DSC] technique. Well defined endothermic and exothermic peaks are observed at glass transition temperature (T_g) and crystallization temperature (T_c). From DSC scans, T_c is obtained at different heating rates (5, 10, 15, 20, 25 K/min). It is observed that T_c increases with increasing heating rate for a particular glassy alloy. Activation energy of crystallization (E_c) has been calculated by different Non-isothermal Iso-conversional methods, *i.e.*, Kissinger-Akahira-Sunose [KAS], Friedman, Flynn-wall-Ozawa [FWO], Friedman-Ozawa [FO] and Starink methods. It is observed that E_c is dependent on extent of crystallization (α). Activation energy is also found to vary with atomic percentage of In in ternary $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ glassy system. The compositional dependence of E_c shows a reversal in the trend at $x = 15$ in $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$, which is explained in terms of mechanically stabilized structure at this composition.

Keywords: Crystallization Kinetics; DSC; Iso-Conversional Methods

1. Introduction

The technological applications of chalcogenide glasses are widespread and, therefore, the study of these materials is a matter of great interest. The most important application of these glasses is in switching action which is commonly known as Ovonic switch. In 1968, Ovshinsky and his team discovered memory and switching effects exhibited by some chalcogenide glasses [1]. The memory devices produced by amorphous semiconductors have great advantage of cheap preparation, unclean radiation resistant and moderate purity of raw material over same devices produced by crystalline semiconductors [2]. Phase change from amorphous to crystalline (a-c p) recording is promising technique for optical memory devices. This technique is more advantageous due to higher transfer rates, faster access time, improved endurance which ultimately provide higher standard of audio video respectively [3-14]. The process of optical recording is based on the simple phenomenon. The underlying concept is that some physical property of recording layer of the disc surface is changed. Microscopic spots of crystalline material on the disc surface are melted on exposure to short laser pulse for recording and recorded marks are erased

by changing the amorphous spots back to crystal-line form. For this purpose material is brought through annealing process with the help of long laser pulses. Long laser pulses are required to heat the material and to bring a-c phase change. That is why the study of a-c phase transformation is the matter of great interest. This study will help in developing the new Chalcogenide materials with enhanced qualities to make them suitable for optical recording materials.

In the present paper, different atomic percentages of In is added to $\text{Se}_{80}\text{Te}_{20}$ binary system to overcome certain problems in binary $\text{Se}_{80}\text{Te}_{20}$ glassy alloy like low glass transition temperature T_g and crystallization temperature T_c and limited reversibility [15]. Ternary alloys are better from the point of view of device preparation and its application [16-18]. It is expected that addition of In can modify the optical properties of binary $\text{Se}_{80}\text{Te}_{20}$ alloy [18]. This has been explained in terms of mean atomic mass of the alloy.

In addition to above mentioned applications, study of crystallization kinetics is important for the manufacturing of glass ceramics and in pervading devitrification. Differential scanning calorimetry (DSC) is valuable technique for the quantitative study of crystallization in different glassy systems and has been widely used [19-24].

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In crystallization kinetics, different models of independent treatments for non-isothermal crystallization have been suggested. Most of these treatments are based on the following relation:

$$\alpha = 1 - \exp[-(Kt)^n] \quad (1)$$

where, α is extent of crystallization and K is rate constant which is thermally activated [25-27].

In all non-isothermal methods one assumes a constant heating rate β that is $T = T_0 + \beta t$ where T_0 is the initial temperature. The best description of these methods is an iso-conversion method. The goal of all these methods is to identify two parameters which when plotted one against the other will result in a straight line. From the slope of such line, the overall effective activation energy E_c or the order of reaction n is calculated [28,29].

In the present paper, the study of the crystallization kinetics is made in glassy $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$) at different heating rates (5, 10, 15, 20, 25 K/min) and activation energy of crystallization (E_c) is calculated by non-isothermal and iso-conversional methods. Five different methods, *i.e.*, Kissinger-Akahira-Sunose [KAS], Friedman, Flynn-wall-Ozawa [FWO], Friedman-Ozawa [FO] and Starink are used to evaluate the value of E_c at different values of α . The dependence of E_c on atomic percentage of In in glassy $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$) is also discussed.

2. Experimental Details

Various samples of $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ glassy system ($x = 5, 10, 15, 20$) are prepared in bulk form by conventional melt quenching technique. High purity (99.999%) Se, Te and In in appropriate atomic weight proportions are placed in a quartz ampoule and sealed under a vacuum of 10^{-5} Torr. The ampoules are then heated at 800°C for about 10 h with continuous rotation to facilitate homogenization of the sample. The molten samples are rapidly quenched in ice cooled water to obtain glassy state. X-ray diffraction pattern (XRD) of the samples confirmed the glassy nature of the material.

The samples so obtained were ground to powder form for carrying out DSC measurements. Five heating rates 5, 10, 15, 20 and 25 K/min. were selected for these measurements. About 10 - 15 mg powder was heated at constant heating rate and the changes in heat flow with reference to empty reference were measured. The presence of well defined endothermic peak at the glass transition temperature and an exothermic peak at the crystallization temperature is observed in each DSC scan. The studies were made under almost identical conditions so that the activation energy of crystallization (E_c) could be compared with other compositions. The T_c was taken as the temperature corresponding to the exothermic peak in

DSC scan. The extent of crystallization (α) at any temperature (T) is calculated as $\alpha = A_T/A$, where A is the total area of the exotherm and A_T is the area of the exotherm between onset of crystallization temperature and T [30].

3. Theoretical Basis

To evaluate the activation energy of crystallization E_c from Non Isothermal experiments, two types of analysis methods are adopted iso-conversional methods and peak methods. Iso-conversional methods are also of two types. One set of methods relies on approximating the temperature integral. This set mainly includes Kissinger method [31], Kissinger-Akahira-Sunose (KAS) method [32,33,34] and the Flynn-Wall-Ozawa (FWO) method [35,36]. While another set of methods does not use any mathematical approximation but uses a determination of the reaction rate at an equivalent stage of the reaction at different heating rates such as Friedman Ozawa (FO) method [37]. For various heating rates β , the Friedman method [38] can directly be used at a specific crystallization fraction α . To study the accuracy of known iso-conversional methods Starink [39] reported that the most accurate methods are KAS method and the methods developed by the author [39,40]. However the most reliable iso-conversional methods as reported by Ozawa [40] are FWO, KAS and Friedman Ozawa (FO) methods.

3.1. Kissinger-Akahira-Sunose (KAS) Method

All of the iso-conversion methods require the determination of the temperature T_{ai} at which a fixed fraction α of the total amount is transformed. In the KAS method, the relation between the temperature T_{ai} and the heating rate β is given by

$$\ln\left(\frac{\beta}{T_{ai}}\right) = \text{constant} - \frac{E_{c\alpha}}{RT_{ai}} \quad (1)$$

Plots of $\ln\left(\frac{\beta}{T_{ai}}\right)$ vs. $1000/T_{ai}$ are plotted and activa-

tion energy $E_{c\alpha}$ is calculated from the slopes of these plots.

3.2. Friedman-Ozawa (FO) Method

Friedman proposed this method for n^{th} order reaction in 1964. Later Ozawa found that it is applicable to other solid-solid transformation processes. According to Friedman-Ozawa method a linear relationship of $\ln(\beta \cdot d\alpha/dT)$ vs. $1000/T$ exists with slope proportional to E_c which can be calculated from different temperature data for fixed value of fraction transformed α by the following relation:

$$\ln\left(\beta \cdot \frac{d\alpha}{dT}\right) = \frac{-E_c}{RT} + \text{constant} \quad (2)$$

3.3. Friedman Method

For various heating rates β , the Friedman method can be used at specific crystallization fraction α as

$$\ln\left(\frac{d\alpha}{dt}\right) = \text{constant} - \frac{E_{c\alpha}}{RT_{ai}} \quad (3)$$

The parameter $E_{c\alpha}$ at this specific value of α , is then calculated from a plot of $\ln(d\alpha/dT)_{ai}$ vs. $1000/T_{ai}$ across different heating rates. The process is repeated for different values of α .

3.4. Flynn-Wall-Ozawa (FWO) Method

The Flynn-Wall-Ozawa (FWO) method has been developed for non-isothermal analysis of crystallization in which final reaction is as follows

$$\ln(\beta) = \text{constant} - 1.0518 \left(\frac{E_{c\alpha}}{RT_{ai}} \right) \quad (4)$$

By plotting $\ln\beta$ vs. $1000/T_{ai}$ for the chosen value of α , activation energy E_c is determined.

3.5. Starink Method

The KAS and FWO methods can be expressed in general form, *i.e.*,

$$\ln\left(\frac{\beta}{T^s}\right) = \frac{-AE_c}{RT_f} + \text{constant} \quad (5)$$

where T_f is the temperature at which a fixed fraction α of the total amount is transferred.

In the method proposed by Starink $A = 1.0008$ and $s = 1.92$, *i.e.*

$$\ln\left(\frac{\beta}{T^{1.92}}\right) = \frac{-1.0008E_c}{RT_f} + \text{constant} \quad (6)$$

4. Results and Discussion

Typical thermo grams obtained by DSC of glassy $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$) at heating rate of 5 K/min are given in **Figure 1**. Similar DSC thermo grams were obtained at other heating rates (10, 15, 20, 25 K/min.) also. It is clear from these thermo grams that well defined endothermic and exothermic peaks are obtained at glass-transition temperature (T_g) and crystallization temperature (T_c).

4.1. Heating Rate Dependence of Crystallization Temperature

Crystallization temperatures of glassy $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$) at different heating rates are calculated from typical thermograms obtained from DSC. These are given in **Table 1**.

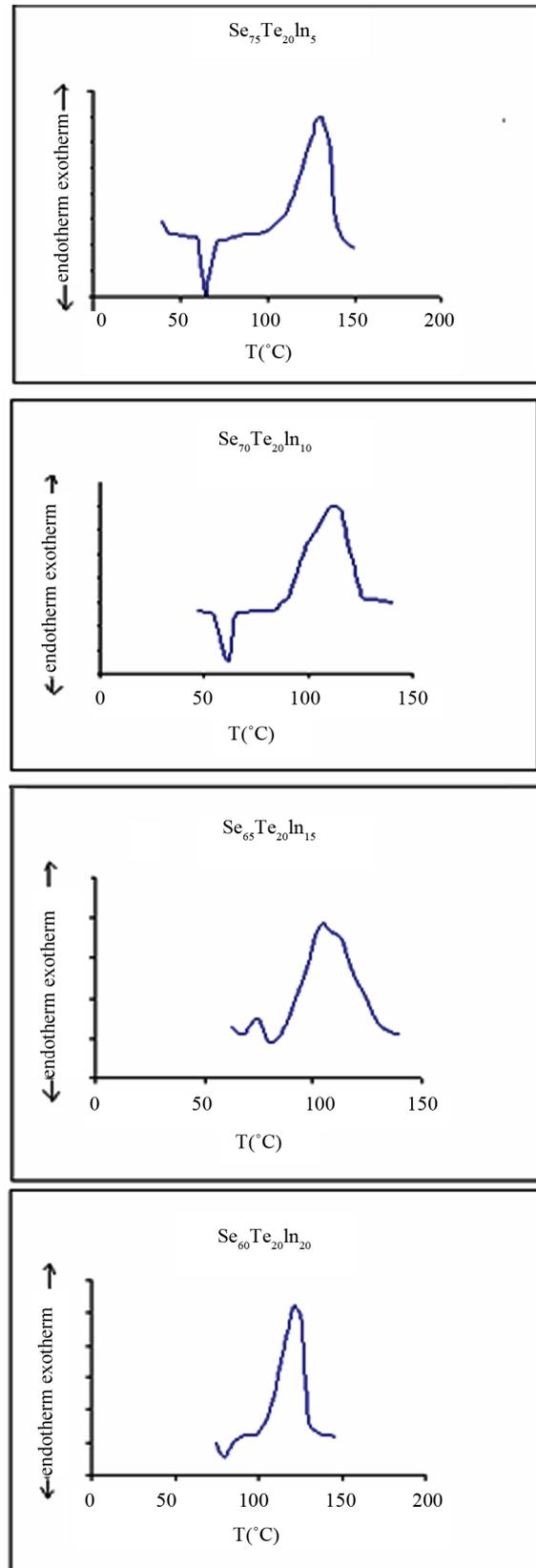


Figure 1. DSC thermograms for $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$) at 5 K/min.

Table 1. Heating rate dependence of crystallization temperature T_c (K).

Heating rates/samples	5 K/min.	10 K/min.	15 K/min.	20 K/min.	25 K/min.
$\text{Se}_{75}\text{Te}_{20}\text{In}_5$	404	404	418	419	425
$\text{Se}_7\text{Te}_{20}\text{In}_{10}$	386	392	394	399	405
$\text{Se}_{65}\text{Te}_{20}\text{In}_{15}$	378	383	396	397	397
$\text{Se}_{60}\text{Te}_{20}\text{In}_{20}$	385	389	393	398	402

For a particular sample, T_c increases with the increase in heating rate. It is also clear from this table that all crystallization temperatures are much higher than room temperature which prevents them from self transition from amorphous to crystalline phase. This characteristic of these materials make them useful as PC-optical recording materials.

4.2. Calculation of Activation Energy of Crystallization

Activation energy of crystallization plays a vital role in proving Chalcogenide as a basic material in various applications. Lot of research work has been done on activation energy of crystallization E_c . In the present work we have calculated E_c by using five non-isothermal and Iso-conversional methods *i.e.*, Kissinger-Akahira-Sunose (KAS), Friedman, Flynn-Wall-Ozawa (FWO), Friedman

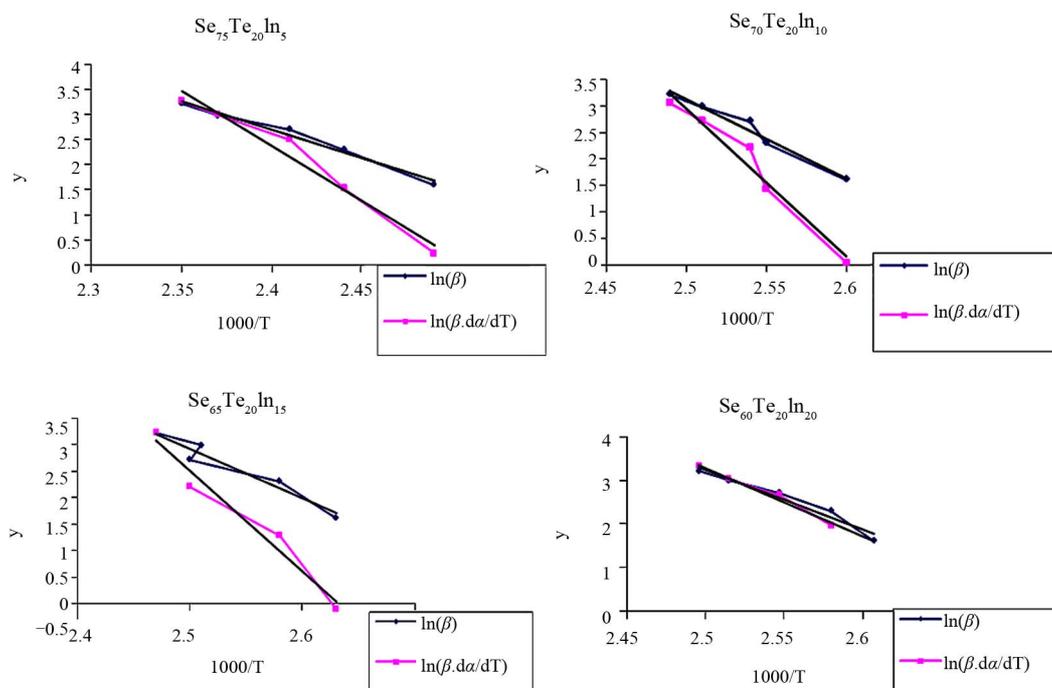
-Ozawa (FO) and Starink methods to study the transformation process (amorphous to crystalline). To do this best fits obtained by FWO and FO methods are given in **Figure 2**.

Similar linear plots are obtained by KAS, Friedman and STARINK methods which are given in **Figure 3**.

By calculating the slopes of above linear fits, activation energy E_c is calculated at different extent of crystallization. The activation energy so obtained is given in **Tables 2-5** at different values of α . Activation energy is plotted against α for each glassy alloy and plots are shown in **Figure 4**.

It is clear from the above figure that activation energy of crystallization E_c varies with the extent of crystallization. It is also evident from the above figure that activation energy obtained by FO method is relatively larger as compared to other methods. This disagreement is due to approximation involved in the method developed by Friedman-Ozawa. Variation of activation energy is also examined with atomic percentage of In incorporation at a particular value of α . The values of activation energy at different atomic percentage of In incorporation at $\alpha = 0.5$ is given in **Table 6** and values are plotted in **Figure 5**.

It is clear from the above figure that E_c increases with increase in atomic percentage of In in ternary $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ alloys up to 10 at %. Such an increase in E_c with increasing percentage of In concentration may be analyzed by considering the atomic weights of Se and In as Te content in each alloy is kept constant. It is known that

**Figure 2. Linear plots in case of FWO, FO methods at $\alpha = 0.7$.**

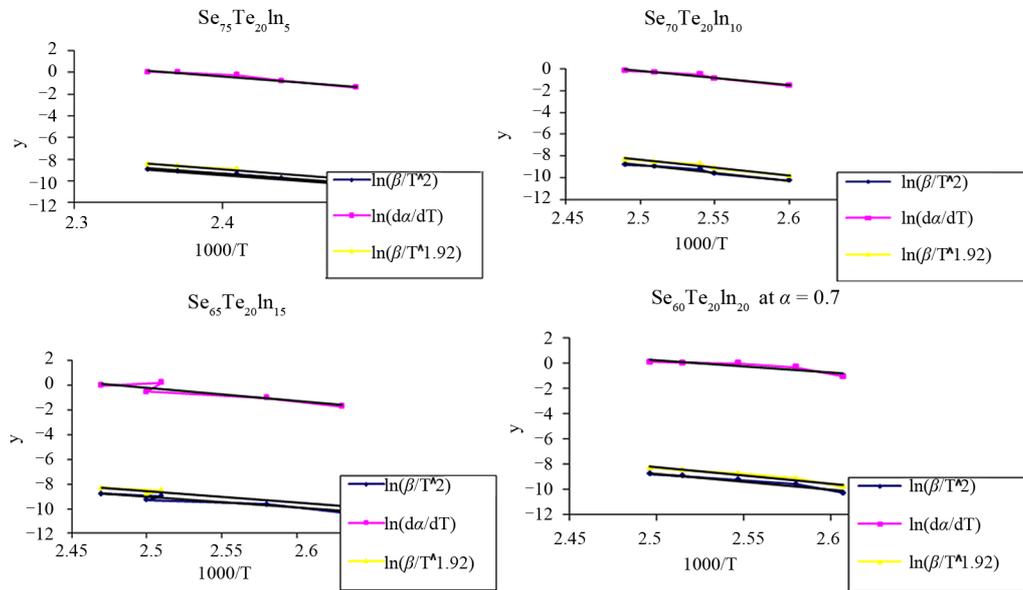


Figure 3. Linear plots in case of KAS, Friedman & STARINK methods at $\alpha = 0.7$.

Table 2. Activation energy E_c (eV) for $\text{Se}_{75}\text{Te}_{20}\text{In}_5$ at different α .

α	KAS	Friedman	FWO	FO	Starink
0.3	0.66	0.69	0.70	1.43	0.66
0.4	0.65	0.55	0.69	1.28	0.65
0.5	1.00	0.93	1.01	2.00	1.01
0.6	0.93	0.88	0.96	1.88	0.94
0.7	0.90	0.92	0.93	1.89	0.92
0.8	0.99	0.89	1.02	1.99	0.99
0.9	0.94	1.55	0.97	2.66	0.95

Table 3. Activation energy E_c (eV) for $\text{Se}_{60}\text{Te}_{20}\text{In}_{10}$ at different α .

α	KAS	Friedman	FWO	FO	Starink
0.3	1.37	1.22	1.26	2.54	1.33
0.4	1.25	1.25	1.26	2.57	1.22
0.5	1.28	1.24	1.25	2.56	1.28
0.6	1.26	1.16	1.24	2.46+	1.23
0.7	1.21	1.13	1.22	2.42	1.22
0.8	1.20	1.05	1.21	2.32	1.22
0.9	1.10	0.88	1.11	2.02	1.11

Table 4. Activation energy E_c (eV) for $\text{Se}_{65}\text{Te}_{20}\text{In}_{15}$ at different α .

α	KAS	Friedman	FWO	FO	Starink
0.3	0.88	0.72	0.90	1.67	0.89
0.4	0.80	0.53	0.82	1.40	0.81
0.5	0.74	0.67	0.77	1.03	0.81
0.6	0.71	0.75	0.75	1.53	0.79
0.7	0.74	0.94	0.77	1.7	0.79
0.8	0.81	0.0	0.77	1.80	0.74
0.9	0.83	1.16	0.86	2.07	0.76

Table 5. Activation energy E_c (eV) for $\text{Se}_{60}\text{Te}_{20}\text{In}_{20}$ at different α .

α	KAS	Friedman	FWO	FO	Starink
0.3	1.18	0.84	1.19	2.10	1.20
0.4	1.14	0.94	1.15	2.15	1.14
0.5	1.17	0.99	1.13	2.18	1.13
0.6	1.11	0.83	1.10	1.37	1.13
0.7	1.09	0.83	1.11	1.35	1.11
0.8	1.00	0.93	1.13	1.33	0.99
0.9	0.96	1.31	0.98	3.07	0.93

Table 6. Values of activation energy of crystallization at different at % of In at $\alpha = 0.5$.

at % of In	KAS E_c (eV)	Friedman E_c (eV)	FWO E_c (eV)	FO E_c (eV)	Starink E_c (eV)
5	1.00	0.93	1.01	2.00	1.01
10	1.13	1.24	1.25	2.56	1.28
15	0.74	0.67	0.77	1.03	0.81
20	1.17	0.99	1.13	2.18	1.13

activation energy of crystallization is guided by nucleation and growth process, which prevents the Chalcogenide glasses from devitrification [41,42]. The atomic weight of In is 114.8 gm/mol and that of Se is 79.0 gm/mol. Since the atomic weight of In is more than Se, the mean atomic weight of $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ alloy increases on increasing the In at the cost of Se. This increase in atomic wt. lowers the nucleation & growth rate and hence increases the activation energy

In the present glassy alloys, composition dependence of E_c also shows a trend reversal at $x = 15$ where it shows

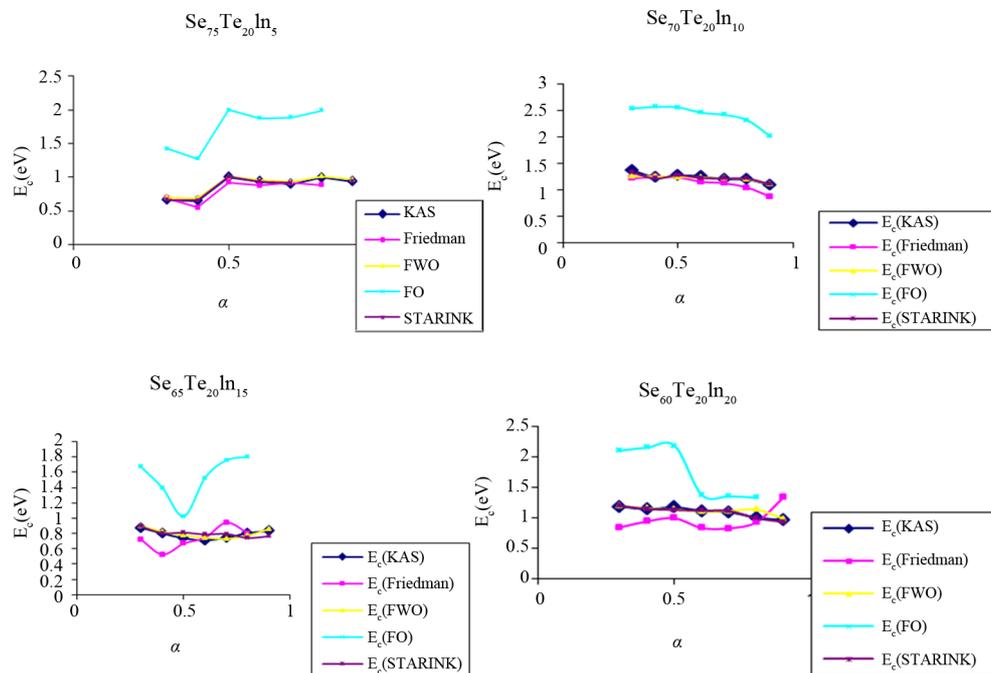


Figure 4. Variation of activation energy E_c with extent of crystallization.

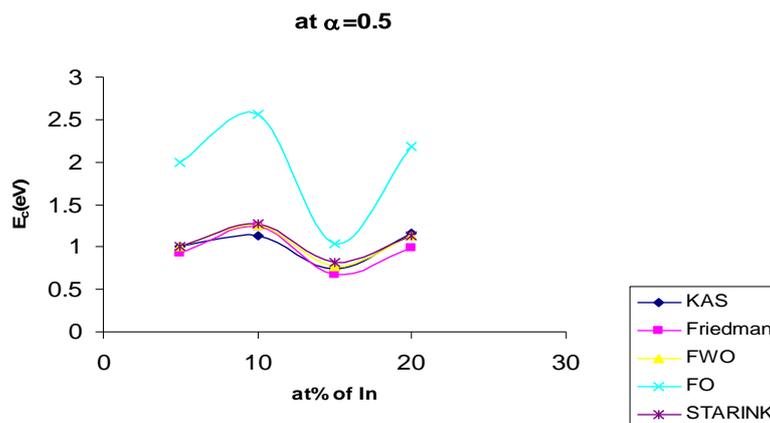


Figure 5. Composition dependence of activation energy of crystallization at $\alpha = 0.5$.

a minima. Similar kind of discontinuity is reported in various physical parameters [42-44] in Chalcogenide glasses when average co-ordination number $\langle r \rangle$ reaches 2.4. This is explained by Phillips and Thorpe in terms of mechanically optimized structure at critical glass composition [45]. The co-ordination number of Se and Te is 2 and that of In is 3 hence in glassy $\text{Se}_{65}\text{Te}_{20}\text{In}_{15}$, average co-ordination number comes out to be $\langle r \rangle = 2.15$. This value of $\langle r \rangle$ is lesser than 2.4. This may be due to an important limitation of Phillips & Thorpe model [45].

Phillips considered that the interaction between atoms is purely covalent while reaching at the balanced condition. In present glasses, addition of In is expected to change the character of some of the Se-Se homo-polar bonds. The In-Se bonds are created as a result of break-

ing up of Se_8 rings which are polar in nature and in turn disturb the covalent Se-Se bonds to some extent. Thus this alteration in the bond character is responsible for the reversal trend found in ternary $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ alloy at 15 at percentage of In.

5. Conclusion

The study of crystallization kinetics is done in glassy $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ ($x = 5, 10, 15, 20$). DSC scans are recorded at different heating rates (5, 10, 15, 20 K/min). Crystallization temperature is found to increase with increase in heating rates. Activation energy E_c of a-c phase change is recorded by employing five non-isothermal iso-conversional methods, *i.e.*, KAS, Friedman, FWO (Flynn-Wall-

Ozawa) FO (Friedmann-Ozawa) & Starink. Activation energy E_c is found to vary with the extent of crystallization α and also with atomic percentage of In. E_c varies on varying the extent of crystallization. E_c increases on increasing the atomic percentage of In except at $x = 15$ in $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$, where it shows minima. This reversal trend at $x = 15$ in $\text{Se}_{80-x}\text{Te}_{20}\text{In}_x$ is explained in terms of lattice dissociation due to breaking up of Se_8 rings to polymeric chains and due to change of nature of bonds which brings the reversal at much lower value of average co-ordination number.

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