

Neeraj Mehta  
Ashok Kumar\*

Department of Physics,  
Harcourt Butler  
Technological Institute  
Kanpur – 208 002, India  
\*Email:

[dr\\_ashok\\_kumar@yahoo.com](mailto:dr_ashok_kumar@yahoo.com)

# Investigation of Amorphous to Crystalline Transition in Glassy $\text{Se}_{80}\text{Te}_{20}$ and $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$ ( $\text{M} = \text{Ag, Cd, Sb}$ ) Alloys

*Calorimetric measurements have been performed in binary  $\text{Se}_{80}\text{Te}_{20}$  and ternary  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  ( $\text{M}=\text{Ag, Cd, Sb}$ ) alloys to study the effect of metallic additives ( $\text{Ag, Cd, Sb}$ ) on the crystallization kinetics in  $\text{Se}_{80}\text{Te}_{20}$  alloy. Three different non-isothermal methods Kissinger's method, Matusita-Sakka theory and Augis-Bennett approximation method have been used in the present study. Different kinetic parameters such as the activation energy of crystallization ( $E_c$ ), the order parameter ( $n$ ), the growth morphology parameter ( $m$ ), the rate constant ( $K$ ) and its frequency factor ( $K_0$ ) have been determined.*

*The effect of third element on crystallization temperature of binary  $\text{Se}_{80}\text{Te}_{20}$  is explained in terms of Arrhenius dependence of rate constant  $K(T)$ . The increasing sequence of activation energy of crystallization in ternary  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  ( $\text{M}=\text{Ag, Cd, Sb}$ ) alloys is explained in terms of their average bond strength.*

**Keywords:** Chalcogenide glasses, Differential Scanning Calorimetry, Crystallization kinetics

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## 1. Introduction

Reversible optical information based on the amorphous to crystalline (a-c) transformation of chalcogenide glasses was first studied in the early 1970's [1,2]. A particular "phase change" technique, termed reverse mode recording, records data by quenching amorphous spots in the material with short laser pulses and erases data by crystallizing with long laser pulses. Phase change (PC) recording materials are thus designed so as to have two structural forms 'amorphous' and 'crystalline'.

Since the first observation of rapid rewritable laser-induced a-c transitions in chalcogenide glasses by Ovshinsky [1, 2], many chalcogenide glasses have been studied as potential recording materials [3-14]. These materials are Se- and Te-based alloys because Se-Te alloys have an easy amorphization process. Se-Te alloys have gained much importance as recording materials in PC technique because of their attractive advantages (higher photosensitivity, greater hardness and smaller aging effects) as compared to pure a-Se. However, these alloys are found to have some significant problems for this type of application. The two serious problems are the limited reversibility [15] and low crystallization temperature.

As it is widely accepted that addition of a third element to the binary chalcogenide glasses

produces stability in these glasses, the above problems can be removed by adding a third element as chemical modifier. In practice, the laser pulse duration used to write and erase is usually several hundred nano-seconds. It is difficult to erase a written spot in such a time scale, if a-c transformation rate of the recording material is not sufficiently high. Thus, the crystallization behaviour at various temperatures is one of the most important features in developing phase change materials. For this reason, the study of crystallization rate and the factors influencing it are very important for the development of new kinds of erasable PC recording materials. The crystallization kinetics of Se-Te alloys are, therefore, studied by various workers [16-23]

Recently, the effect of Ag, Cd and Sb additives on the glass transition kinetics of glassy  $\text{Se}_{80}\text{Te}_{20}$  alloy is reported by our group [24]. In the present work, three different methods of analysis (as described in next section) have been used to study the effect of these additives on the crystallization kinetics of glassy  $\text{Se}_{80}\text{Te}_{20}$  alloy.

## 2. Theoretical Basis

The a-c transformation in chalcogenide glasses can be investigated by isothermal and non-isothermal methods. In isothermal method, the sample is brought near to crystallization

temperature very quickly and then any physical quantity, which changes drastically, is measured as a function of time. In non-isothermal method, the sample is heated at a fixed rate and heat evolved is recorded as a function of temperature.

A disadvantage of the isothermal method is the impossibility of reaching a test temperature instantaneously and during the time which system needs to stabilize, no measurements are possible. However measurements can be achieved in a relatively rapid and precise manner by DSC technique, which also allows an immediate observation of the a-c transformation. Various theoretical methods have been suggested to determine activation energy of crystallization from non-isothermal DSC data.

The crystallization kinetics of amorphous alloys have been intensively studied using the classical Johnson-Mehl-Avrami (JMA) theoretical model [25-27] in which the crystallization fraction ( $\alpha$ ) can be described as a function of time (t) according to the formula:

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

where  $n$  is the Avrami index and  $K$  is the rate constant which is given by:

$$K = K_o \exp(-E_c / RT) \quad (2)$$

here  $E_c$  is the activation energy of crystallization,  $R$  is the universal gas constant and  $K_o$  is also a constant.

Based on JMA model, different authors have developed very diverse methods to study a-c transformation of various alloys. Given below is the details of three important and useful methods, which have been used in the present study:

### 2.1 Kissinger's relation

According to Kissinger [28], the heating rate  $\beta$ , in terms of peak temperature of crystallization  $T_c$ , can be expressed as:

$$\ln(\beta / T_c^2) = -E_c / R T_c + \text{constant} \quad (3)$$

This equation is used to calculate the activation energy of crystallization by plotting  $\ln \beta / T_c^2$  vs.  $10^3 / T_c$  curve.

### 2.2 Matusita and Sakka Theory

The extent of crystallization ( $\alpha$ ) at a temperature  $T$  is well expressed by the expression:

$$\ln(1-\alpha)^{-1} = (C/\beta^n) [(-nE_c) / RT] \quad (4)$$

derived by Matusita and Sakka [29,30] from the classical JMA equation. For constant temperature, this equation can be written as:

$$\ln[\ln(1-\alpha)^{-1}] = -n \ln \beta + \text{constant} \quad (5)$$

From this equation, the value of  $n$  can be calculated by plotting  $\ln[\ln(1-\alpha)^{-1}]$  vs  $\ln \beta$  curves at different temperatures.

Further, since the values of  $\alpha$  are independent of  $\beta$  at  $T=T_c$  [31], so at  $T=T_c$ , the equation (4) takes the form:

$$\ln \beta = -E_c / RT_c + \text{constant} \quad (6)$$

This equation is used to calculate the activation energy of crystallization by plotting  $\ln \beta$  vs.  $10^3 / T_c$  curve.

### 2.3 Method of Augis and Bennett

The activation energy of crystallization  $E_c$  can also be determined by an approximation method developed by Augis and Bennett [32]. The relation used by them is of the form:

$$\ln \beta / T_c = -E_c / RT_c + \ln K_o \quad (7)$$

The activation energy of crystallization can be evaluated by this equation using the plots of  $\ln \beta / T_c$  against  $10^3 / T_c$ .

## 3. Experiment

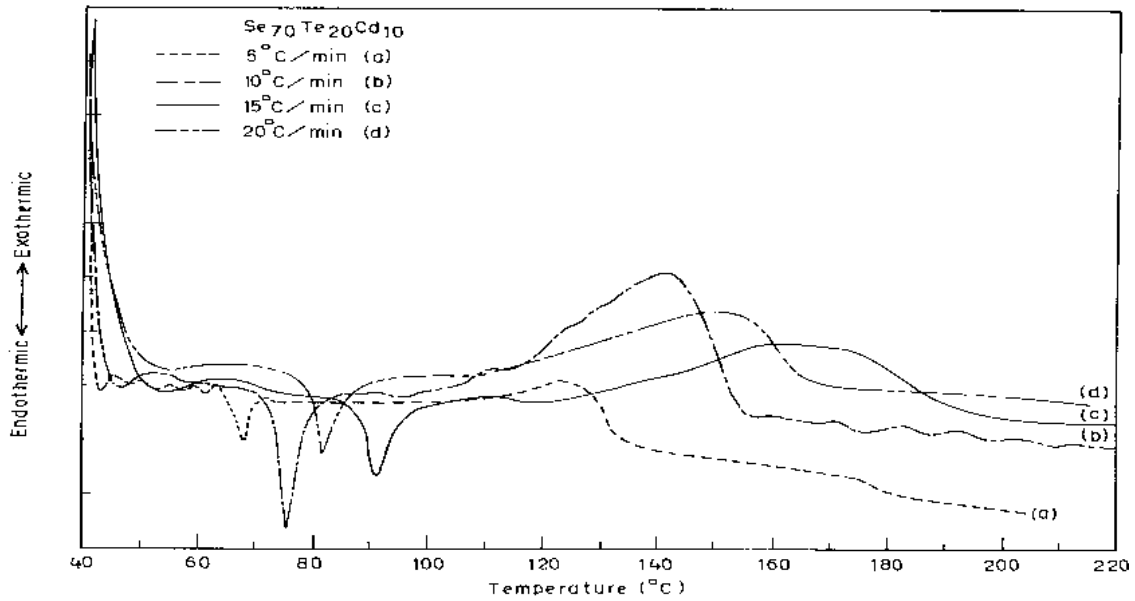
Glassy alloys of  $\text{Se}_{80}\text{Te}_{20}$  and  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) were prepared by quenching technique. High purity materials (5N pure) were weighed according to their atomic percentages and were sealed in quartz ampoules under the vacuum of  $10^{-5}$  Torr. Each ampoule was kept inside the furnace at an appropriate temperature (where the temperature was raised at a rate of 3-4°C/min.). The ampoules were rocked frequently for 10 hrs at the maximum temperature to make the melt homogeneous. Quenching was done in ice water and 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 each sample was heated at a constant heating rate and the changes in heat flow with respect to an empty pan were measured. Four heating rates (5, 10, 15 and 20°C/min) were chosen in the present study. For the sake of accuracy, four measurements were conducted for each heating rate and each specimen under identical conditions. Throughout this paper, the experimental data points are taken as the average value of the four supposedly identical measurements. Best-fit methods are used for plotting the experimental data.

Measurements were made under almost identical conditions so that a comparison of kinetic parameters ( $n$ ,  $m$ ,  $K$  and  $E_c$ ) could be made in order to understand the effect of changing the additive element (Ag, Cd, Sb) in ternary alloys  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$ .

## 4. Results and Discussion

Fig.1 shows the typical DSC thermogram for ternary alloy  $\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$  at different heating rates. Similar thermograms were obtained for other glassy alloys. It is clear from Fig.1 that well defined endothermic and exothermic peaks are observed at glass transition temperature ( $T_g$ ) and crystallization temperatures ( $T_c$ ), respectively.



**Fig.(1):** DSC Thermograms for  $Se_{70}Te_{20}Cd_{10}$  alloy at different heating rates

*Evaluation of Rate constant and frequency factor*

The values of  $T_c$  at different heating rates for various glassy alloys are given in Table 1. It is clear from this table that  $T_c$  increases on addition of Cd to binary  $Se_{80}Te_{20}$  alloy. However, a decrease in  $T_c$  is observed in case of Sb and Ag. This can be explained in terms of Arrhenius dependence of  $K(T)$ . Knowing the values of  $E_c$  and  $lnK_o$  from Eq. (7), the values of rate constant  $K(T)$  have been determined by equation (2). The values of  $lnK$  at different temperatures in the crystallization region are given in Table 2.

**Table (1)** Peak crystallization temperatures of  $Se_{80}Te_{20}$  and  $Se_{70}Te_{20}M_{10}$  (M=Ag, Cd, Sb) alloys at different heating rates.

Sample	$T_c$ (°C)			
	5 °C/min	10 °C/min	15 °C/min	20 °C/min
$Se_{80}Te_{20}$	118.50	127.40	131.32	135.20
$Se_{70}Te_{20}Ag_{10}$	103.30	109.70	115.30	118.40
$Se_{70}Te_{20}Cd_{10}$	125.60	130.00	136.47	139.23
$Se_{70}Te_{20}Sb_{10}$	87.62	92.00	95.00	100.00

The values of  $K(T)$  are increased in the sequence  $Se_{70}Te_{20}Cd_{10} < Se_{80}Te_{20} < Se_{70}Te_{20}Ag_{10} < Se_{70}Te_{20}Sb_{10}$ . However, the values of  $T_c$  are decreased in the sequence  $Se_{70}Te_{20}Cd_{10} > Se_{80}Te_{20} > Se_{70}Te_{20}Ag_{10} > Se_{70}Te_{20}Sb_{10}$ . This shows that higher the value of  $K(T)$ , greater the speed of crystallization and hence lower the crystallization temperature.

**Table (2)** The values of  $lnK$  for  $Se_{80}Te_{20}$  and  $Se_{70}Te_{20}M_{10}$  (M=Ag, Cd, Sb) alloys at different temperatures

Sample	ln K		
	70 °C	80 °C	90 °C
$Se_{80}Te_{20}$	-8.94	-7.87	-6.87
$Se_{70}Te_{20}Ag_{10}$	-7.57	-6.51	-5.51
$Se_{70}Te_{20}Cd_{10}$	-10.49	-9.23	-8.04
$Se_{70}Te_{20}Sb_{10}$	-6.23	-5.02	-3.88

Sample	ln K		
	100 °C	110 °C	120 °C
$Se_{80}Te_{20}$	-5.92	-5.00	-4.16
$Se_{70}Te_{20}Ag_{10}$	-4.56	-3.66	-2.81
$Se_{70}Te_{20}Cd_{10}$	-6.91	-5.84	-4.82
$Se_{70}Te_{20}Sb_{10}$	-2.80	-1.77	0.80

*Evaluation of Avrami index n and growth morphology parameter m*

The fraction ' $\alpha$ ' crystallized at any temperature  $T$  is given as  $\alpha = A_T/A$ , where  $A$  is the total area of the exothermic peak between the temperature  $T_b$  where the peak begins (i.e. the crystallization starts) and the temperature  $T_e$  where the peak ends (i.e. the crystallization is complete).  $A_T$  is the partial area of the exothermic peak between the temperatures  $T_b$  and  $T$ .

Fig. 2 shows the variation of  $ln[ln(1-\alpha)^{-1}]$  with  $ln\beta$  at three constant temperatures for ternary  $Se_{70}Te_{20}Cd_{10}$  alloy. Similar curves were obtained for other glassy alloys. Using Eq. (5), the values of  $n$  have been determined from the slopes of these curves at three different temperatures for all the glassy alloys. The values of Avrami index ' $n$ ' at three different temperatures are given in Table 3.

**Table (3)** Values of order parameter  $n$  for different alloys

$\text{Se}_{80}\text{Te}_{20}$		$\text{Se}_{70}\text{Te}_{20}\text{Ag}_{10}$		$\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$		$\text{Se}_{70}\text{Te}_{20}\text{Sb}_{10}$	
T (°C)	n	T (°C)	n	T (°C)	n	T (°C)	n
80	2.4	105	3.3	115	1.1	85	1.1
90	2.3	110	3.2	120	0.9	90	1.2
100	2.4	115	3.3	130	1.0	95	1.3

**Table (4)** Dimensionality factor (growth morphology parameter)  $m$  of different alloys

Sample	$m$
$\text{Se}_{80}\text{Te}_{20}$	2
$\text{Se}_{70}\text{Te}_{20}\text{Ag}_{10}$	3
$\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$	1
$\text{Se}_{70}\text{Te}_{20}\text{Sb}_{10}$	1

The numerical factor  $m$  (growth morphology parameter) of crystallization mechanism representing its dimensionality is given in Table 4. From this table, it is clear that the crystal growth in  $\text{Se}_{80}\text{Te}_{20}$  is two-dimensional. The crystal growth in  $\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$  and  $\text{Se}_{70}\text{Te}_{20}\text{Sb}_{10}$  occurs in one dimension whereas it occurs in three dimensions in  $\text{Se}_{70}\text{Te}_{20}\text{Ag}_{10}$ .

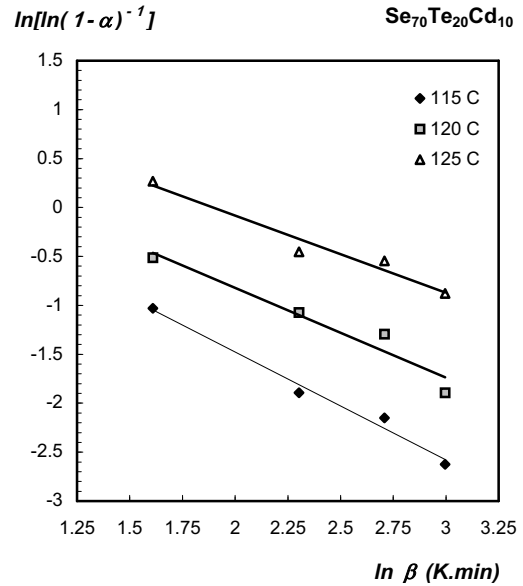
#### Evaluation of activation energy of crystallization

The activation energy of crystallization of each alloy has been calculated by Kissinger's relation, Matusita and Sakka theory and method of Augis and Bennett. The plots of  $\ln(\beta/T_c^2)$  vs.  $10^3/T_c$ ,  $\ln\beta$  vs.  $10^3/T_c$  and  $\ln(\beta/T_c)$  vs.  $10^3/T_c$  for glassy  $\text{Se}_{80}\text{Te}_{20}$  and  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) alloys are shown in Figs. 2 to 4, respectively. The values of  $E_c$  obtained for all the samples using the above three methods are given in Table 5. Comparison of  $E_c$  values of different alloys obtained from equations (3), (6) and (7) shows that the values are in good agreement. This means that one can use any of the three equations to calculate activation energy of crystallization.

It is clear from Table 5 that the activation energy of crystallization indicates the speed of the crystallization. The activation energy of crystallization in ternary alloys have been found to be increase in the sequence  $(E_c)_{\text{Ag}} < (E_c)_{\text{Sb}} < (E_c)_{\text{Cd}}$ . This sequence can be explained in terms of average heat of atomization for these alloys. The average heat of atomization  $H_S$  based on chemical bonding aspects, is defined for an alloy  $\text{X}_a\text{Y}_b\text{Z}_c$  as a direct measure of cohesive energy i.e., of the average bond strength [33].  $H_S$  can be given as:

$$H_S = [a(H_S)_X + b(H_S)_Y + c(H_S)_Z] / (a+b+c) \quad (8)$$

where  $(H_S)_X$ ,  $(H_S)_Y$ ,  $(H_S)_Z$  are the heat of atomization of atoms X, Y and Z, respectively. The value of  $H_S$  for ternary alloys  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) are given in Table 6.


**Fig. (2):** Plot of  $\ln[\ln(1-\alpha)^{-1}]$  vs  $\ln\beta$  for  $\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$  alloy

**Table (5)** Activation energy of crystallization for different alloys

Sample	$E_c$ (eV)		
	Eq. (3)	Eq. (6)	Eq. (7)
$\text{Se}_{80}\text{Te}_{20}$	1.081	1.149	1.115
$\text{Se}_{70}\text{Te}_{20}\text{Ag}_{10}$	1.074	1.140	1.106
$\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$	1.284	1.354	1.319
$\text{Se}_{70}\text{Te}_{20}\text{Sb}_{10}$	1.282	1.294	1.263

**Table (6)** The average bond strength of ternary  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) alloys

Sample	$H_S$ (kJ/mole atoms)
$\text{Se}_{70}\text{Te}_{20}\text{Ag}_{10}$	226.7
$\text{Se}_{70}\text{Te}_{20}\text{Cd}_{10}$	209.5
$\text{Se}_{70}\text{Te}_{20}\text{Sb}_{10}$	224.5

The average bond strength of ternary alloys  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) increases in the sequence  $(H_S)_{\text{Cd}} < (H_S)_{\text{Sb}} < (H_S)_{\text{Ag}}$ . This shows that higher the average bond strength of the ternary alloy, lower is the activation energy and hence the speed of crystallization. The variation of  $E_c$  with  $H_S$  is shown in Fig. 6.

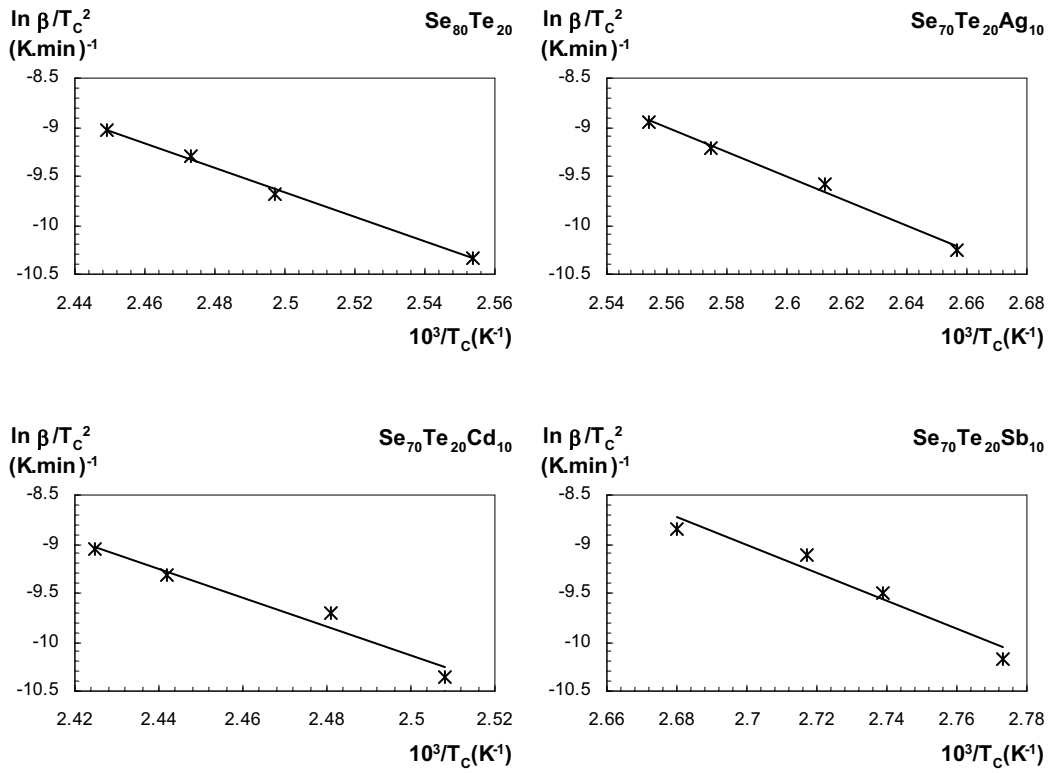


Fig. (3): Plots of  $\ln \beta / T_c^2$  vs  $10^3 / T_c$  for different alloys

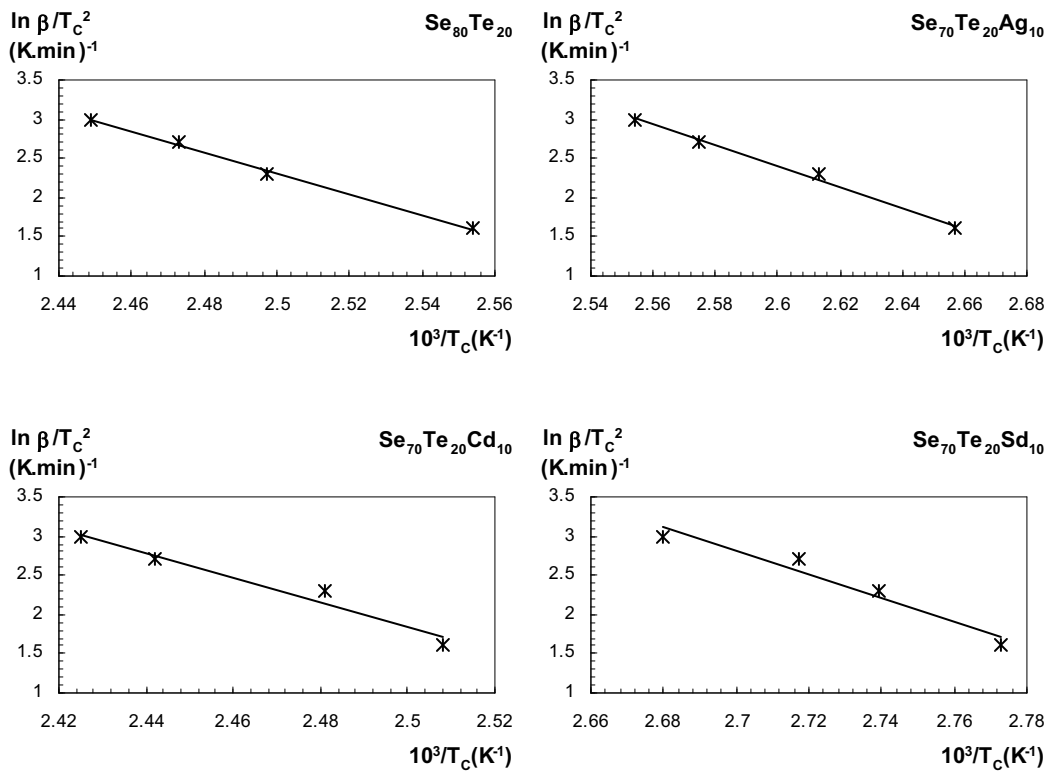


Fig. (4): Plots of  $\ln \beta$  vs  $10^3 / T_c$  for different alloys

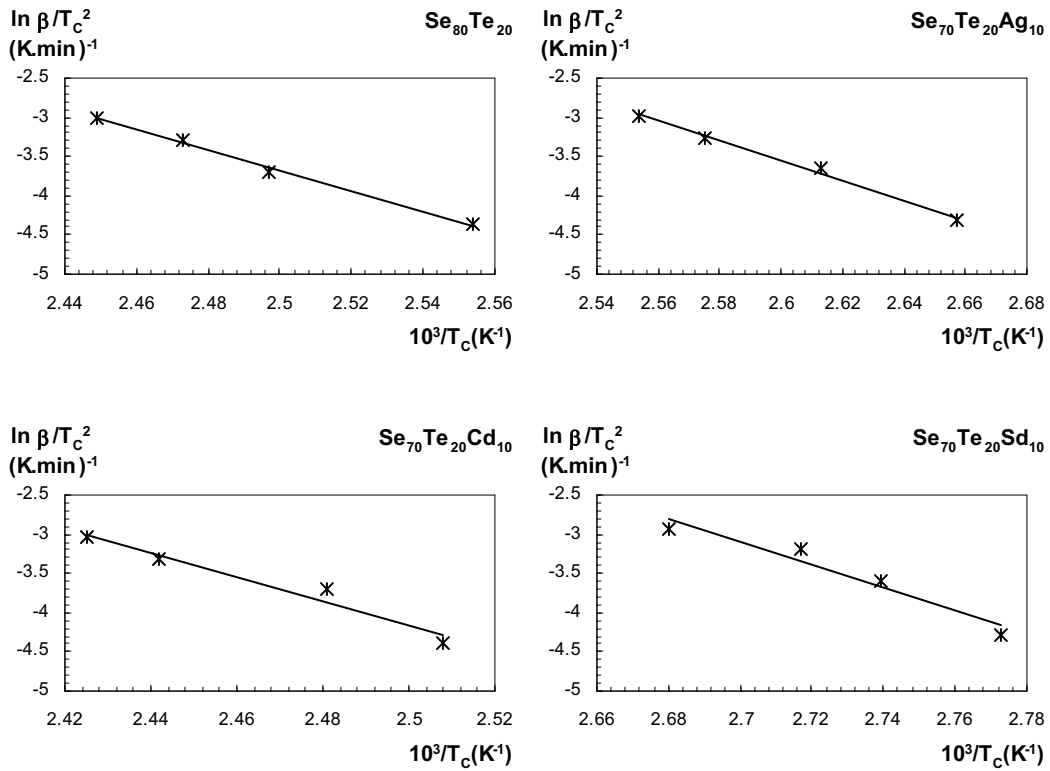


Fig. (5): Plots of  $\ln\beta/T_c$  vs  $10^3/T_c$  for different alloys

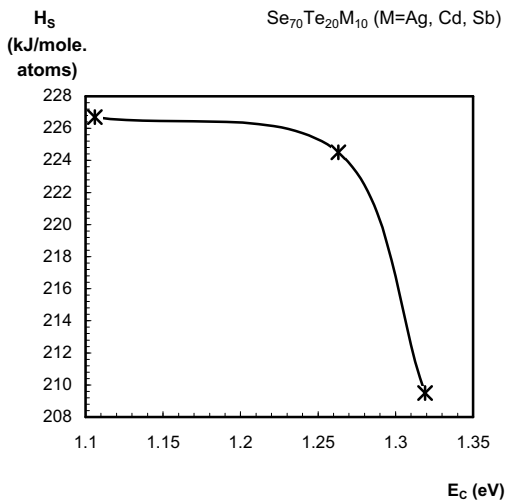


Fig. (6): Plot of  $E_c$  vs  $H_s$  for ternary  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) alloys

### 5. Conclusions

The effect of some metallic additives (Ag, Cd, Sb) on the crystallization kinetics in glassy  $\text{Se}_{80}\text{Te}_{20}$  alloy has been studied by three different methods under non-isothermal condition. DSC technique has been used in the present study to calculate the activation energy of crystallization. It has been found that  $E_c$  values obtained by three different methods are in good agreement with each other.

Different kinetic parameters such as the activation energy of crystallization ( $E_c$ ), the order

parameter ( $n$ ), the growth morphology parameter ( $m$ ), the rate constant ( $K$ ) and its frequency factor ( $K_0$ ) have been determined.

The effect of third element on crystallization temperature of binary  $\text{Se}_{80}\text{Te}_{20}$  is explained in terms of Arrhenius dependence of rate constant  $K(T)$ . The present results shows that the speed of crystallization in ternary  $\text{Se}_{70}\text{Te}_{20}\text{M}_{10}$  (M=Ag, Cd, Sb) alloys depends on their average bond strength.

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## MEETINGS

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