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Glass transition kinetics and crystallization mechanism in $\text{Se}_{90}\text{Cd}_{8}\text{Bi}_{2}$ and $Seg_{90}Cd_{6}Bi_{4}$ chalcogenide glasses

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Differential scanning calorimeter (DSC), under non-isothermal condition, was used to study the glass transition kinetics and the crystallization mechanism of $\text{Se}_{90}\text{Cd}_{8}\text{Bi}_{2}$ and $\text{Se}_{90}\text{Cd}_{6}\text{Bi}_{4}$ glassy alloys. Two approaches, namely Moynihan and Kissinger, were used to calculate the relaxation activation energy (E_t) from the dependence of the glass transition temperature (T_g) on the heating rate (β). Results reveal that E_t decreases with an increase in Bi content which indicates that thermal stability is improved. This is attributed to the formation of Se–Bi bonds which are stronger than Se–Cd bonds and heavily cross-link the structure. In addition, the kinetic analysis of the crystallization peaks was performed using Ozawa, Kissinger, Takhor and Augis–Bennett relations. The values of the crystallization activation energy (E_c) and Avrami exponent (n) of the two alloys were evaluated. The obtained values of E_c , calculated from the above mentioned relations, were found to be in good agreement while the average calculated values of Avrami exponent (n) are (2.3 ± 0.1) for Se₉₀Cd₈Bi₂ and (1.7 ± 0.1) for Se₉₀Cd₆Bi₄ which indicating that the crystal growth in the two alloys occurs in one dimension.

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1. Introduction

After the publication of the pioneer work of Ovshinsky [\[1\]](#page-5-0) in which the reversible switching phenomenon in certain types of chalcogenide glasses was reported, a comprehensive survey was presented to characterize and to improve the properties of such type of glasses. A great deal of these researches has been focused on studying the glass transition kinetics [\[2–8\]](#page-5-0) and the crystallization mechanism [\[9–15\].](#page-5-0) The related parameters of these transformations are important for better understanding of the physics of these materials and for determination of the useful range of operating temperature at which these materials may be utilized in a specific technological application before crystallization takes place [\[9,14\].](#page-5-0)

Among the amorphous chalcogenide elements, selenium (a-Se) is widely used for fabrication of chalcogenide glasses because of its unique property of reversible transformation (amorphous to crystalline and back). To produce considerable changes in the properties of pure Se and to overcome some of its disadvantages such as short lifetime, low sensitivity and high resistivity, it is worth to add more than one component (like Bi, Te, Ge, Ga, Sb, As, Cd, Sn etc.)into selenium matrix. There were lots ofinvestigations showed that alloying of pure Se or binary selenium based glasses with Bi or Cd caused remarkable variation in the physical properties of the

original glass. Chalcogenide glasses are generally p-type semiconductors. It is observed that [\[16,17\],](#page-5-0) the addition of Bi at a certain atomic percentage into some binary chalcogenide glasses reverses the conduction from p to n-type. Besides, a survey of literature indicates that the addition of Bi with small content to pure Se [\[18\]](#page-5-0) or to binary Se–Te glass [\[19–21\]](#page-5-0) increases the glass transition temperature (T_{σ}) and decreases the relaxation activation energy while the crystallization activation energy takes several trends. Density of state (DOS) of Se–Te–Bi system is also found to increase with an increase in Bi content [\[22,23\].](#page-5-0) In the other side, the addition of metallic elements like Cd to pure Se or binary Se–Te glass is found to alter the electrical properties by inducing new impurity states in the band gap without any change in conduction type [\[24\].](#page-5-0) Singh et al. [\[25\]](#page-5-0) obtained that, the DOS decreased when 5 at% of Cd is added to pure Se–Te binary glass and further addition of Cd increased the DOS. $\text{Se}_{75}\text{S}_{25-x}\text{Cd}_x$ ternary glasses have been investigated in two separated studies. In the first one [\[26\],](#page-5-0) it is found that as Cd content increases, the glass transition temperature, the relaxation activation energy and the crystallization activation energy were increased. In the second work [\[27\],](#page-5-0) both of the optical band gap and the DC electrical conductivity are found to increase with an increase in Cd content.According to the author knowledge, no work has been done to investigate the thermal or any physical properties of Se-based ternary chalcogenide glasses containing both Bi and Cd as dopant materials.

Amorphous chalcogenide glasses obtained by quenching from the liquid phase tend to change their structure in two subsequent

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processes: retrification (glass transition) and devitrification (crystallization). Both ofthem are thermally activated processes causing distinct changes in the physical properties of the glassy material. The glassy state may be one of many possible quasi-stable states, of different atomic configuration, which may differ only slightly from one another and results in very similar physical properties [\[14\].](#page-5-0) The glass transition temperature and the energy of thermal relaxation are the most important parameters for characterization of the glassy state. In addition, the amorphous glassy state in chalcogenide glasses is in general unstable one. Therefore, it inherently possesses the possibility of transforming into a more stable crystalline state which causes a drastic change in the properties of the glass [\[11\].](#page-5-0) Crystallization mechanism is always associated with the study of some thermal parameters like onset crystallization temperature (T_c) , peak crystallization temperature (T_n) , crystallization activation energy (E_c) , and the dimensionality of crystal growth (m) that is related with Avrami exponent (n) .

Differential scanning calorimeter (DSC) is an extremely popular thermo-analytical tool for studying thermal processes. In DSC measurements, two basic independent methods can be used: isothermal and non-isothermal. In the isothermal method, the sample is brought quickly to a temperature above the glass transition temperature (T_g) 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 at a fixed rate (β) , and the heat evolved is mainly 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 [\[11\].](#page-5-0) However, the non-isothermal method does not have this disadvantage and it also posses several advantages such as the quick performance and the rapid information it provides about the transformation temperatures over a wide temperature range. In addition, non-isothermal analysis using DSC helps us in not only understanding the thermal behavior but also provides information about the stability of the glass against devitrification, glass forming ability and monitoring of both the nucleation and growth processes, which are responsible for the devitrification of most glassy materials.

The aim of the present work is to study, using differential scanning calorimeter under non-isothermal conditions, the glass transition kinetics and the crystallization mechanism of $\text{Se}_{90}\text{Cd}_{8}\text{Bi}_{2}$ and $Seg_0Cd_6Bi_4$ glassy alloys. From the heating rate dependence of the glass transition and the crystallization temperatures, the glass transition and crystallization activation energies have been calculated using different theoretical relations reported in literature [\[28–32\].](#page-5-0) The dimensionality of crystal growth of the two studied glassy alloys has also been investigated.

2. Samples preparation and experimental technique

The two studied $Seg_0Cd_8Bi_2$ and $Seg_0Cd_6Bi_4$ glasses were prepared by conventional melt quenching technique. The appropriate amount of Bi, Cd and Se of 5 N purity were weighed and sealed into quartz ampoules under a vacuum of 10−⁵ Torr. The ampoules were then heated at 900 ℃ for about 15 h with continuous rotation to ensure the homogenization of the sample. The melt was then rapidly quenched in ice cooled water to produce the glassy material. The ingot of the so-produced glassy sample was taken out of the ampoule by breaking the ampoule and then grinded gently in a mortar and pestle to obtain a powder form. Calorimetric measurements were carried out using DSC (Model Perkin Elmer DSC7) under non-isothermal condition to deduce the temperatures T_{g} , T_c and T_p . DSC scans were done from room temperature to about 170 \degree C at five heating rates (5, 7, 10, 15 and 20 K/min) by heating/cooling at the same rate, in the DSC furnace, about (10 \pm 1) mg of

the powder samples to obtain the above mentioned characteristic temperatures. The powder samples were capsulated in aluminum pans with an empty reference pan. These pans are often covered by lids that act as a radiation shield. The DSC apparatus was calibrated prior to measurements, using high purity standards Pb, Sn and In with well-known melting points. The temperature precision of this equipment is \pm 0.1 K with an average standard error of about 1K in the measured values of glass transition and crystallization temperatures. A best fit for the results is calculated by the least square fitting for the activation energies and other kinetic parameters.

3. Results and discussion

3.1. DSC curves

Amorphous and crystalline materials are often made starting from the liquid phase, either by solidification of a melt to form single crystals when the cooling rate is very low (crystallization), or when the liquid could be supercooled sufficiently quickly (quenched) so that crystallization could be bypassed, then a glass without any long-range lattice order (amorphous) would result [\[33\].](#page-5-0) Crystallization and glass transition phenomena can easily be observed by differential scanning calorimeter (DSC) when the material sample is reheated in the DSC furnace at a given heating rate. DSC is designed such that the sample holder temperature increases linearly as a function of time. By observing the difference in heat flow between the sample and reference, DSC is able to measure the amount of energy absorbed or released during any transitions. When there are no thermodynamic or chemical processes occurring, the heat flow difference between the sample and reference varies only slightly with temperature, and shows up as a flat base line of the DSC curve. However, glass transition occurs as the temperature of an amorphous solid is increased and characterized by a decrease in viscosity. This transition is an endothermic process and appears, at the glass transition temperature, as a step or a small dip of the DSC curve. At some point the molecules will obtain enough freedom of motion to spontaneously arrange themselves into a crystalline form. This transition from amorphous solid to crystalline solid is an exothermic process, and results, at the crystallization temperature, as a peak in the DSC curve. As the temperature increases, the sample eventually reaches its melting temperature. The melting process results as an endothermic dip-like peak in the DSC curve [\[34\].](#page-5-0)

The DSC curves, for the two studied glasses, were recorded at five heating rates of 5, 7, 10, 15 and 20K/min and presented in [Figs.](#page-2-0) 1 and 2. In the temperature range of investigation, the DSC curves are characterized by the evident of two peaks: Endothermic peak corresponds to the glass transition phenomenon which is represented by the glass transition temperature (T_g) , followed by exothermic peak corresponds to the crystallization process and is represented by the onset (T_c) and peak (T_p) temperatures of crystallization. Both T_g and T_c have been defined [\[9,10\]](#page-5-0) as the temperatures which correspond to the intersection of two linear portions adjoining the transition elbow of the DSC traces in the endothermic and exothermic directions, respectively, as shown in [Fig.](#page-2-0) 1. The values of T_g , T_c and T_p were obtained from [Figs.](#page-2-0) 1 and 2 and listed in [Table](#page-2-0) 1. As evident from this table, it is observed that all of these characteristic temperatures shift towards higher values as the heating rate increases from 5 to 20 K/min. In addition, T_g increases with an increase in Bi content at the same heating rate while both T_c and T_n decrease. From the dependence of these temperatures on heating rate, all the parameters that are required for analysis of the glass transition kinetics and crystallization mechanism of the studied samples have been deduced.

Fig. 1. Typical DSC curves for Se₉₀Cd₈Bi₂ glass at different heating rates.

3.2. Glass transition kinetics

The glass transition temperature (T_g) is one of the most important parameters for characterization of the glassy state as it is related to the rigidity of the glassy network [\[2\].](#page-5-0) Studying the glass transition kinetics, from the dependence of T_g on heating rate, was carried out by several researchers [\[2–8\].](#page-5-0) In all these studies, the recorded T_g is found to increase with increasing heating rate. In the present work, three approaches were used to analyze

Fig. 2. Typical DSC curves for Se₉₀Cd₆Bi₄ glass at different heating rates.

Table 1

Glass transition temperature (T_g) , onset (T_c) and peak (T_p) crystallization temperatures of the two studied glasses.

Heating rate (K/min)	$Seq_0Cd_8Bi_2$			$Seq_0Cd_6Bi_4$		
	T_{σ} ($^{\circ}$ C)	T_c (\circ C)	T_n (°C)	T_{σ} (°C)	T_c (\circ C)	T_n (°C)
5	57	97.4	107.2	58.7	96.1	106.3
7	58.5	101	110.5	60.8	99	109.9
10	60	104.2	113.8	62.3	102.6	113.1
15	61.6	108.4	118.2	64.5	106.3	117
20	63.1	111.6	121.7	66.1	110.8	120.9

Fig. 3. Plot of T_g vs. ln β for Se₉₀Cd_{10−x}Bi_x (x = 2 and 4) glasses (solid lines are leastsquare fits to Lasocka relation).

the dependence of T_g on the heating rate β . The first one is the following empirical relationship that suggested by Lasocka [\[35\]:](#page-5-0)

$$
T_g = A + B \ln \beta \tag{1}
$$

where A and B are constants for a given glass composition. The value of A indicates the glass transition temperature for the heating rate of 1 K/min. However, B is related to the cooling rate of the melt; the lower the cooling rate of the melt, the lower the value of B. This signifies that B is related with the response of the configurational changes within the glass transition region [\[36\].](#page-5-0) It is found that this relation holds good for the studied glassy samples as shown in Fig. 3. The values of A and B were deduced from the slopes and intersections of the resulting straight lines, respectively, and are listed in Table 2.

The second approach, which was derived by Moynihan et al. [\[32\],](#page-5-0) suggested that the glass transition temperature varies with the heating rate according to the following relation:

$$
\frac{d\ln\beta}{d(1/T_g)} = -\frac{E_t}{R}
$$
 (2)

Where R is the universal gas constant (=8.314 JK⁻¹ mol⁻¹). The values of E_t for the two samples were obtained from the slopes of the plots of ln β as a function of $(10^3/T_g)$ as shown in [Fig.](#page-3-0) 4. The values of E_t obtained by this method are listed in Table 2.

Finally, Kissinger relation [\[29\]](#page-5-0) has been widely [\[2–8\]](#page-5-0) used to describe the variation of the glass transition temperature with the heating rate β . This relation was originally developed for crystallization processes to evaluate the crystallization activation energy. Besides, this relation has also been derived to describe the glass

Table 2 Lasocka parameters A and B of Eq. (1) and the glass transition activation energy (E_t) of the two studied glasses.

Composition	Lasocka parameters (K)		Glass transition activation energy (E_t) (kJ/mol)	
	А		Moynihan	Kissinger
$Seq_0Cd_8Bi_2$ $Seq_0Cd_6Bi_4$	323.1 ± 0.2 4.3 \pm 0.1 $323.4 + 0.3$ $5.2 + 0.1$		$213 + 5$ $178 + 5$	$208 + 4$ $173 + 5$

Fig. 4. Plot of ln β vs. (10³/T_g) for Se₉₀Cd_{10−x}Bi_x (x = 2 and 4) glasses (solid lines are least-square fits to Moynihan relation).

transition kinetics as suggested by Ruitenberg [\[37\]](#page-5-0) and takes the following form:

$$
\frac{\partial \ln[\beta/T_g^2]}{\partial[1/T_g]} = -\frac{E_t}{R}
$$
\n(3)

Following this equation, Fig. 5 shows the plots of $ln(\beta/T_g^2)$ vs. $(10^3/T_g)$ for the two glasses. The values of E_t , which were calculated from the slopes of the resulting straight lines, are listed in [Table](#page-2-0) 2. From this table, it is observed that the values of the activation energy (E_t) calculated using the two models, are in good agreement with each other and the difference is within the experimental error. Besides, since the glass transition activation energy is correlated with the relaxation energy, it means that it represents the amount of energy that a structure needs to jump from its quasi-stable state to another one of more stability within the glassy region. Hence, the glass with lower amount of E_t was originally formed in a local region of more stability and needs this amount of energy to relax to a stable structure and hence the least is the

Fig. 5. Plot of $\ln(\beta/T_g^2)$ vs. (10³/T_g)for Se₉₀Cd_{10-x}Bi_x (x=2 and 4) glasses (solid lines are least-square fits to Kissinger relation).

value of E_t , the most is the glass stable [\[4,14\].](#page-5-0) The calculated values of E_t indicate that Se₉₀Cd₆Bi₄ is more stable than Se₉₀Cd₈Bi₂ and hence it has the larger probability to jump to a state of lower configurational energy. The increase of both T_g and thermal stability with bismuth content can be explained as follows. The replacement of Se–Cd bonds (bond energy = 37.1 kcal/mol [\[24\]\)](#page-5-0) by Se–Bi bonds, which have higher bond energy (40.7 kcal/mol [\[20\]\),](#page-5-0) increases the cohesive energy of the system which heavily cross-link the structure. This is the reason why both T_g and thermal stability increase with Bi content. This result is in good agreement with the results reported by other workers [\[18–21\].](#page-5-0)

3.3. Crystallization mechanism

The theoretical basis for interpreting the crystallization mechanism for many amorphous materials is provided by the general Johnson–Mehl–Avrami (JMA) equation [\[38,39\].](#page-5-0) According to this equation, the fraction χ of the material that crystallized after a time t is given by:

$$
\chi = 1 - \exp[-(Kt)^n] \tag{4}
$$

where n is the Avrami exponent that gives the details of the dimensionality of crystal growth $[40]$ and K is actually a measure of the rate of crystallization, and usually assigned Arrhenian temperature dependence:

$$
K = K_0 \exp\left(-\frac{E_c}{RT}\right) \tag{5}
$$

Where K_0 is a frequency factor indicates the number of attempts to overcome the energy barrier and E_c is the activation energy for crystallization. Based on JMA equation and from the heating rate dependence of the onset (T_c) and peak (T_p) crystallization temperatures, several theoretical relations [\[28–31\]](#page-5-0) were developed under certain approximations to calculate the crystallization parameters such as E_c and n. These parameters were calculated, in this work, according to the following relations. The first one is the relation which was derived by Ozawa [\[28\]](#page-5-0) and is given as:

$$
\ln \beta = -\frac{E_c}{RT_c} + C \tag{6}
$$

According to this equation, the data of $\ln\beta$ against (10³/T_c) for the two glasses are plotted in Fig. 6. From the slopes of the resulting

Fig. 6. Plot of ln β vs. (10³/T_c) for Se₉₀Cd_{10−x}Bi_x (x = 2 and 4) glasses (solid lines are least-square fits to Ozawa relation).

Table 3

Crystallization activation energy (E_c) and the average value of Avrami exponent (n) of the two studied glasses.

Composition	Crystallization activation energy (E_c) (kJ/mol)				n
	Ozawa	Kissinger	Takhor	Augis-Bennett	
$Seg_0Cd_8Bi_2$ SegnCd ₆ Bi ₄		$117 + 2$ $114 + 3$ $119 + 3$ $115 + 4$		$120 + 4$ $117 + 3$ $121 + 4$ $118 + 4$	$2.3 + 0.1$ $17 + 01$

straight lines, the values of the activation energy (E_c) for the two studied glasses are obtained and listed in Table 3.

Kissinger relation is one of the most commonly used equations to evaluate E_c . This relation is given as [\[29\]:](#page-5-0)

$$
\ln\left(\frac{\beta}{T_p^2}\right) = -\frac{E_c}{RT_p} + C\tag{7}
$$

Depending on this equation, the data of ln (β/T_p^2) and $(10^3/T_p)$ for the studied glasses are plotted in Fig. 7. The values of the activation energy (E_c) are deduced from the slopes of the obtained straight lines and are listed in Table 3.

However, Takhor [\[30\]](#page-5-0) used a procedure similar to that of Ozawa, but here he used the variation of (T_p) , instead of (T_c) , with heating rate and hence his relation is given as:

$$
\ln \beta = -\frac{E_c}{RT_p} + C \tag{8}
$$

Where:

$$
C = \frac{1}{n} \left\{ \ln \left[\frac{n}{n-1} \right] + \ln K_0 \right\} + \ln(T_p - T_0)
$$
\n(9)

On this basis, the activation energies for the two samples were calculated from the slopes of the resulting straight lines in the plots of ln β as a function of (10³/T_p) as depicted in Fig. 8. The values of E_c deduced by this method are also listed in the same table.

Finally, Augis and Bennett [\[31\]](#page-5-0) developed an approximation method to determine E_c according to the following relation:

$$
\ln\left(\frac{\beta}{T_p}\right) = -\frac{E_c}{RT_p} + \ln K_0 \tag{10}
$$

This relation was also used in the determination of the activation energy of crystallization (E_c) for the two samples. Fig. 9 shows the plots of $\ln(\beta/T_p)$ against $(10^3/T_p)$ and the slopes of the obtained

Fig. 7. Plot of $\ln(\beta/T_p^2)$ vs. ($10^3/T_p$) for Se₉₀Cd_{10-x}Bi_x (x=2 and 4) glasses (solid lines are least-square fits to Kissinger relation).

Fig. 8. Plot of ln β vs. (10³/T_p) for Se₉₀Cd_{10−x}Bi_x (x = 2 and 4) glasses (solid lines are least-square fits to Takhor relation).

straight lines are used to find the two values of E_c . These values are also listed in Table 3.

In the other side, the order of crystallization reaction (Avrami exponent n) for the two studied samples was obtained using the relation [\[31\]:](#page-5-0)

$$
n = \frac{2.5RT_p^2}{(\Delta T)_{FWHM}E_c}
$$
\n(11)

where (ΔT) FWHM is the width of the crystallization peaks at half maximum and the other symbols carry their usual meanings that are mentioned earlier. For as-quenched glass containing no preexisting nuclei, the dimensionality of crystal growth (m) is equal to $(n-1)$ and m will be 1 if n is less than or equal 2, whereas for a glass containing a sufficiently large number of nuclei, which might occur due to any heat treatment of the as-quenched glass $m = n$ [\[38\].](#page-5-0) The values of m in this work are taken as $m = n - 1$ since as-quenched samples are studied. The average values of n , for the two glasses, are calculated from the last equation and listed in Table 3. From

Fig. 9. Plot of $\ln(\beta/T_p)$ vs. ($10^3/T_p$) for Se₉₀Cd_{10−x}Bi_x (x = 2 and 4) glasses (solid lines are least-square fits to Augis and Bennett approximation relation).

this table, it is clear that the values of n are not integers. A noninteger value of n indicates that two crystallization mechanisms were working during amorphous–crystalline transformation [41]. The obtained values of Avrami exponent (n) indicate that the crystal growth, in the two studied samples, occurs in one dimension (or growth from surface nuclei). It is also observed, from [Table](#page-4-0) 3, that the values of E_c , obtained from the above four methods, are in good agreement with each other. Besides, it is clear that E_c of the two samples is approximately the same. Since the crystallization activation energy is an indication of the speed of crystallization, this may give an indication that, to complete amorphous–crystalline transformation, higher percentage of Bi must be added at the cost of either Se or Cd.

4. Conclusions

Studies of glass transition kinetics and crystallization mechanism of $Seq_0Cd_8Bi_2$ and $Seq_0Cd_6Bi_4$ glasses, under non-isothermal condition, were performed using differential scanning calorimeter (DSC). The following conclusions were drawn:

- The DSC curves obtained for the two studied glasses show a single glass transition and crystallization region, confirming the homogeneity of the glassy alloys which are under investigation.
- The glass transition activation energy (E_t) has been calculated using two relations derived by Moynihan and Kissinger. The value of E_t of Se₉₀Cd₆Bi₄ is smaller than that of Se₉₀Cd₈Bi₂ which indicates that thermal stability is improved with an increase in Bi concentration.
- The values of the crystallization activation energy (E_c) obtained using Ozawa, Kissinger, Takhor and Augis–Bennett methods are in good agreement with each other. Thus, one can use any of these methods to deduce the activation energy of crystallization.
- The obtained values of the Avrami exponent (n) indicating that the crystal growth in the two glasses occurs in one dimension.

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