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Study of glass transition kinetics of selenium matrix alloyed with up to 10% indium

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Abstract Glassy selenium matrix alloyed with up to 10% indium, namely $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$, was prepared by the melt quenching technique. Differential thermal analysis (DTA) has been used to study the glass transition kinetics of the two binary Se–In alloys in comparison with that of pure Se. DTA scans were recorded at different constant heating rates ($\beta = 1{:}20$ °/min). The glass transition temperature (T_g) is found to be shifting to a higher value with increasing of both β and In-content. Such a configurationally response is related to the observed increase of the stability parameters accompanied with the introduction of In into the Se matrix. The activation energy of glass transition (E_g) has been determined using two different non-isothermal models (Moynihan and Kissinger) where a good coincidence is obtained. The compositional dependence (0:10% In) of all considered parameters is discussed in terms of the introduced structural matrix.

Keywords DTA · Thermal analysis · Chalcogenide glasses · Se–In · Glass transition kinetics · Thermal stability

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Introduction

Since the seventies of the last century, considerable efforts have been exerted towards studying the structure and properties for semiconducting chalcogenide glasses for their potential applications in reversible phase change optical storage media or electrical switches. These applications utilized the differences in optical storage or electrical properties between the crystalline and amorphous phases of the same material (cf., [1–4] and references cited therein). Chalcogenide semiconductors have also been proposed for phase change nonvolatile random access memories (PCRAM), which is becoming the next generation for memory technology [5]. In this respect, the proper description of thermal behavior of semiconducting chalcogenide glasses is crucial to understand their properties and functions. In fact, the understanding of glass transition kinetics of chalcogenide glasses aims at establishing their thermal stability and glass-forming ability (GFA). This enables one to determine the useful range of operating conditions (e.g., temperature) for a specific technological application before an eventual crystallization takes place. Here, one of the important used techniques to study the glass transition kinetics is the differential thermal analysis (DTA) or differential scanning calorimetry (DSC).

Among the chalcogenide systems, selenium and selenium based glassy alloys have been intensively studied due to their wide technical applications, especially in the field of electronics and optoelectronics. Glassy selenium (g-Se) exists in two structural forms: long helical chains and eight-membered rings, with strong covalent bonds existing among atoms within the molecular units and weaker forces, perhaps of Van der Waals type, binding together neighboring units (cf., [6, 7] and references cited therein). Glassy-Se has some shortcoming from the point of view of some practical applications, e.g., low sensitivity and low thermal instability. Such properties can be improved by alloying g-Se with some elements. Various additives to g-Se that introduce binary chalcogenide systems, such as Se–In, Se–Tl, Se–Ge, Se–S, Se–Te, Se–Sb and others [8–15], are of great interest for their various properties accompanied with better hardness, better stability, higher sensitivity, and/or change (higher or lower) in crystallization ability.

The incorporation of indium atoms into selenium forms a layered structure; each layer consists of three atomic planes: one metal ion sheet sandwiched between two non-metal atom sheets. These three atomic sheets are strongly bounded by either a covalent or ionic bonds, but the layers are weakly bound with each other by Van der Waals interaction (cf., [8, 16] and references cited therein). In–Se compositions are characterized by their highly anisotropic properties resulted from their layered structure, where iono-covalent bonds are essentially confined within the layers; whereas the interactions between layers are very weak, mainly of Van der Waals type. Such a structure allows for the diffusion of guest atoms or molecules between two successive layers without altering the host structure. Therefore, it is possible to use this inserted reaction in new microdevices such as capacitors or microbatteries where the lamellar compounds are used as solid state ionic conductors. Semiconductors with such novel characteristics are suitable for sensors of small particles and ionizing radiation [17]. Moreover, the formation of heterostructures between an In–Se chalcogenide semiconducting layer and a tetrahedral one, such as silicon (Si), represents another challenge because of their potentialities for non-linear optics or as intermediate between highly mismatched semiconductors [18].

In fact, the thermal stability (TS) of a glass and its ability to devitrification gives an important conclusion for using such a glass in the proper application field. In other words, the memory and threshold behavior of chalcogenide glasses are determined essentially by their crystallization ability. Threshold switches are made in those glasses near the center of the glass-forming region where the glasses are stable and show little or hard tendency to crystallize when heated or cooled slowly. Memory switches come from the boundaries of the glass-forming region where the glass is more prone to crystallization; i.e., glasses which prone to easy devitrification show memory and stable glasses exhibition threshold behavior.

Saffarini [19], in his study on $Se_{100-x}In_x$ chalcogenides concluded that the TS of the quenched glasses decreases monotonically with increasing the In-content up to $x = 29$ at.% In. In contrast to this conclusion, Imran et al. [20] reported a maximum and minimum TS at $Se_{96}In_4$ and $Se_{90}In_{10}$ glasses, respectively. Singh et al. had also proved that the effective thermal conductivity as well as the effective thermal diffusivity are maximum and optical band gap is minimum at 10 at.% In ([21] and references cited therein]. In other words, these latter authors concluded from their studies on the thermal transport properties that $Se_{90}In_{10}$ chalcogenide glass is better thermally stable than other glasses of the binary Se–In series. Due to such contradictory results regarding the compositional range of In-content [19–21] in addition to the importance of TS in applications, lead us to study the effect of In-content, particularly at 5 and 10 at.%, on the TS, GFA and glass transition energy of g-Se.

Experimental

Two binary chalcogenide glasses of the composition $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ were prepared by the melt quenching technique. This has been done by mixing appropriate quantities of Se and In, of purity 99.999%, in accordance with their atomic percentage using a digital balance of type WA-31 with accuracy 10^{-4} g. The weighted materials of each composition were then sealed in an evacuated silica ampoule ($\sim 10^{-5}$ Torr) of length ~ 12 cm and thickness ~ 0.2 cm (internal diameter = 0.8 cm and external diameter 1 cm). The ampoules were heated gradually to 350 °C in an electric furnace and kept at this temperature for 2 h. Then, the temperature of the furnace was gradually raised by a rate ~ 200 °/h until reaching 800 °C and sustained at this temperature for 7 h before being raised to 900 °C for another 2 h. The ampoules were frequently agitated during the synthesis process to ensure optimum homogeneity of the melt. Afterwards, the ampoules were quenched in ice-water.

The glassy nature of the quenched ingots was checked by using X-ray diffractometer of the type Philip's XPERT-MPDUC PW 3040 with Cu K_α radiation source ($\lambda = 0.15406$ nm), at power of 1600 W (40 kV and 40 kA). Fine powder samples were prepared by grinding for DTA scans. The latter was carried out using hot-stage thermo-system of the type Mettler FP80. For each DTA run, 20 mg of the powder was heated starting from room temperature up to 280 °C. Five different constant heating rate scans were recorded for each investigated composition; these were at 1, 5, 10, 15, and 20 °/min.

Results and discussion

Figure 1 shows typical DTA curves for glassy $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ as recorded at the considered rates in the range of 1:20 °/min. Each curve demonstrates an endothermic step corresponding to glass transition at

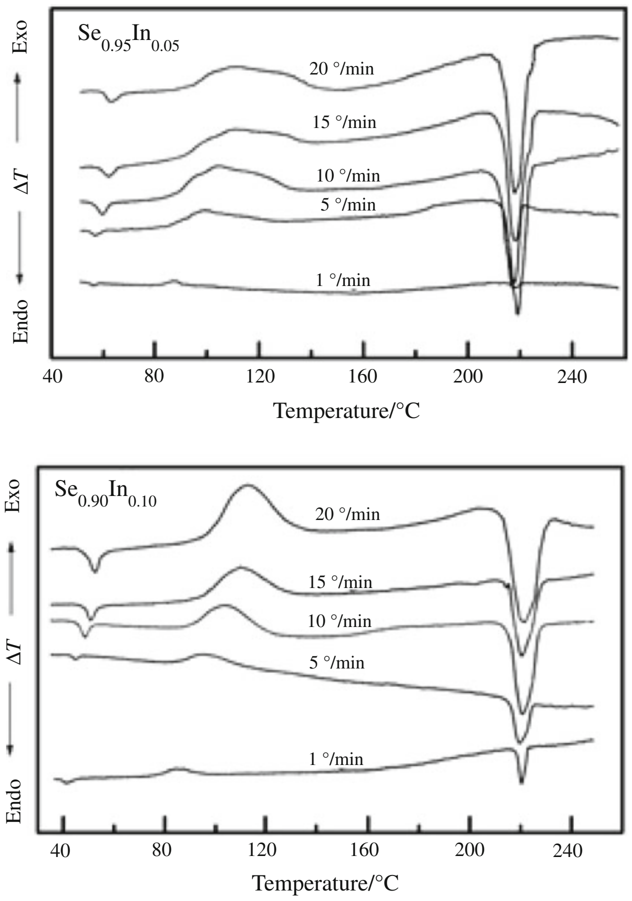

Fig. 1 DTA curves for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses as-scanned at different heating rates

temperature T_g, an exothermic peak due to crystallization with onset temperature T_0 and maximum rate of crystallization at T_c, and a well defined endothermic peak due to melting at T_m. Such recorded phase transition curves indicate the dependence of both T_g and T_c on the heating rate β; besides, the independency, as expected, of the value of T_m on β.

Heating rate dependence of T_g

An empirical relation between β and T_g has been suggested early by Lasocka [22]:

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

where A is a constant refers to the value of T_g at a heating rate equals 1 °/min. The proportionality constant B indicates the response of the configurationally changes within the glass transition region. The function $T_g = f(\beta)$ is plotted in Fig. 2 thus indicating a straight line relationship satisfying the previously mentioned relation (Eq. 1). Such linear dependence is observed for both glassy $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ compositions investigated. Each plotted line

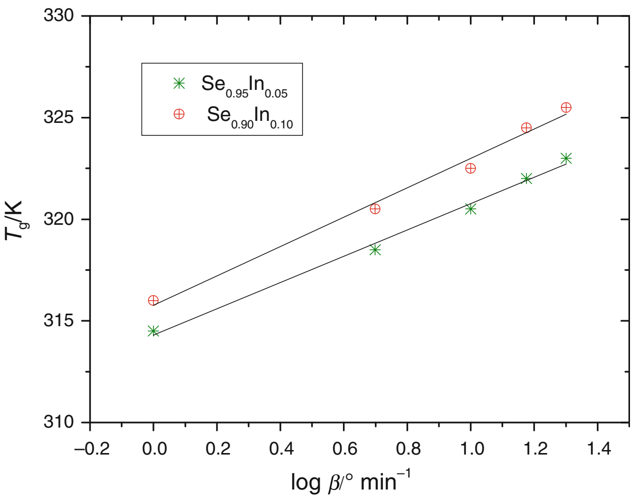

Fig. 2 Heating rate dependence of T_g for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses. The *solid lines* represent least-squares fitting

illustrates a least-squares fitting with $R^2 = 0.991$ for $Se_{0.95}In_{0.05}$ and $R^2 = 0.987$ for $Se_{0.9}In_{0.1}$. Here, it worth mentioning that the value of A that calculated from the intercept of Fig. 2 (314 K for $Se_{0.95}In_{0.05}$ and 316 K for $Se_{0.9}In_{0.1}$) is in a quite good agreement with the experimental value of T_g estimated from the DTA curves of Fig. 1 at 1 °/min.

Figure 2, also, reflects a more stable glass structure for $Se_{0.9}In_{0.1}$ than that of $Se_{0.95}In_{0.05}$, as the value of T_g is always higher for the former composition at the different heating rates considered. This observed compositional dependence on T_g is in accordance with the fact that there is a difference between the bond energies of Se–Se (1.9 eV [23]) and In–Se (2.09 eV [23]). That is the formation of Se–In bonds on the expense of Se–Se bonds mostly lead to the detected increase in T_g with In-content. In other words, the increase of indium content (up to 10%) in g-Se produces a more stable glassy network. This is in the same sequence as the value of B, where $B = 6.46$ min for 5% In and increases to 7.22 min for the introduction of 10% In in g-Se matrix. Moreover, the difference in the value of B indicates that the two binary glassy alloys undergo different structural configurations.

In fact, such pronounced dependence of T_g on β detected for the binary Se–In glassy investigated compositions, has not been verified from DTA studies for pure g-Se scanned at different heating rates in the range of 5:50 °/min, see Fig. 3 [9]. This is also the case recognized with the isoelectronic additions (S or/and Te) to g-Se. In fact, such additives do not impact the two structural fragments of Se; i.e., chains and rings. This means that there is no detectable change in the recorded value of T_g with β for g-Se as well as for the Se-like structure materials (Se–S, Se–Te, and Se–S–Te) [11, 13, 24]. Here, it is worth keeping in mind that

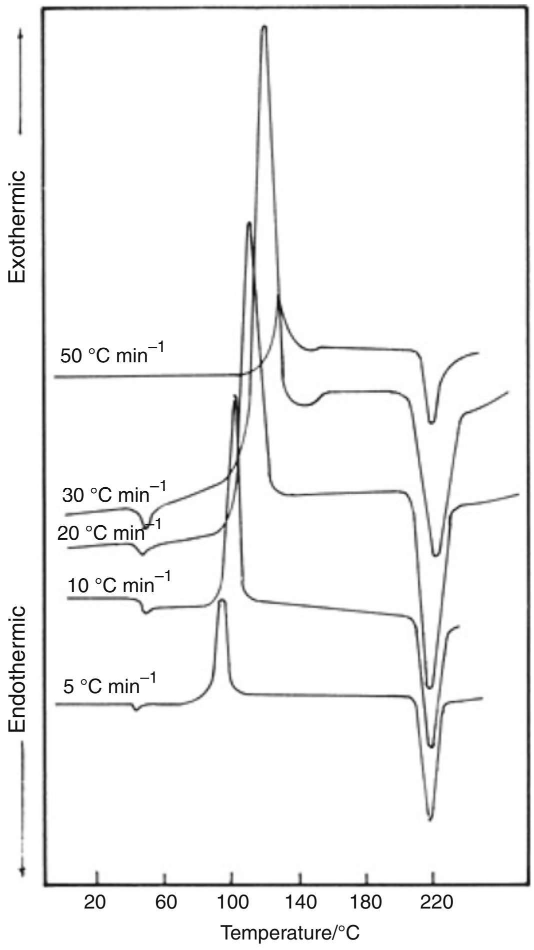

Fig. 3 DTA curves for g-Se at different heating rates [9]

the technique of preparation as well as the preparation condition greatly affects the configurational structure characterization of the considered glass [6, 7]. Apart from the Se-like structural materials, addition of elements such as As, Sb or Bi to Se, that forming layered structure, tends to change the value of T_g with the heating rate [15, 25, 26].

TS and glass-formation tendency

The TS of a glass reflects its resistance towards the crystallization transform through nucleation and growth stages. Also, the GFA is related to the ease of glass formation while avoiding the crystal growth. Both of TS and GFA play a significant role in determining the utility of a chalcogenide material for introducing a device [27, 28]. In fact, the value of T_g represents the strength or rigidity of the glass structure in the chalcogenide system. Hence, T_g offers valuable information on TS of the glassy state. However, the value of T_g alone does not give any information about the GFA. So, it has been found that the difference between the onset temperature of crystallization (T_0) and T_g is a strong indicator for both TS and GFA.

Table 1 Heating rate dependence of ΔT and H' for the glasses investigated

β/°/min	$Se_{0.95}In_{0.5}$		$Se_{0.9}In_{0.1}$	
	$\Delta T \pm 0.5/°C = T_0 - T_g$	H'	$\Delta T \pm 0.5/°C = T_0 - T_g$	H'
1	33	0.11	37	0.12
5	29	0.09	39	0.13
10	28	0.09	38	0.12
15	32	0.1	37	0.13
20	31	0.96	43	0.14

In this respect, Saad and Poulain [29] proposed two interesting parameters: weighted TS (H') and S-parameter, to determine the stability of the glass. The S-parameter is defined as:

$$S = \frac{(T_0 - T_g)(T_c - T_0)}{T_g} \tag{2}$$

As the S-parameter reflects the resistance to devitrification after the formation of the glass, so higher value of $(T_0 - T_g)$ delays nucleation, and $(T_c - T_0)$ is related to the rate of devitrification transformation of the glass phases. The other parameter, i.e., the weighted TS (H'), has the following form:

$$H' = \frac{(T_0 - T_g)}{T_g} \tag{3}$$

The values of H' and $\Delta T = (T_0 - T_g)$ have been calculated for the two Se–In investigated glasses and are listed in Table 1 as a function of heating rate. The table illustrates that the variation of each parameter has a non-monotonic character for both $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ compositions. But, the values of H' and ΔT are always higher for the g-Se alloyed with 10% In than those of 5% In. This represents another evidence for the higher relative stability of $Se_{0.9}In_{0.1}$ than that of $Se_{0.95}In_{0.05}$. Figure 4 shows a plot of the calculated values of S-parameter as a function of β for both $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses. This figure summarizes the increase of S-parameter with both of β and In-content.

The numerical values of the coefficient of the glass-forming tendency, K_{gl}, have been calculated by Hruby [30] according to the formula:

$$K_{gl} = \left(\frac{T_0 - T_g}{T_m - T_0}\right) \tag{4}$$

where the assumptions considered in its derivation are reviewed in [30, 31]. Higher values of $(T_0 - T_g)$ refer to delaying the nucleation process and the small values of $(T_m - T_0)$ retard the growth process. Values of K_{gl} can be

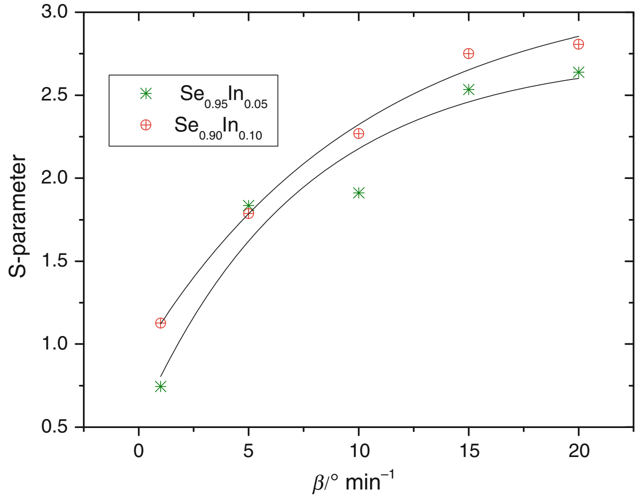

Fig. 4 Variation of *S*-parameter with heating rate for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses. The *solid lines* represent regression fitting

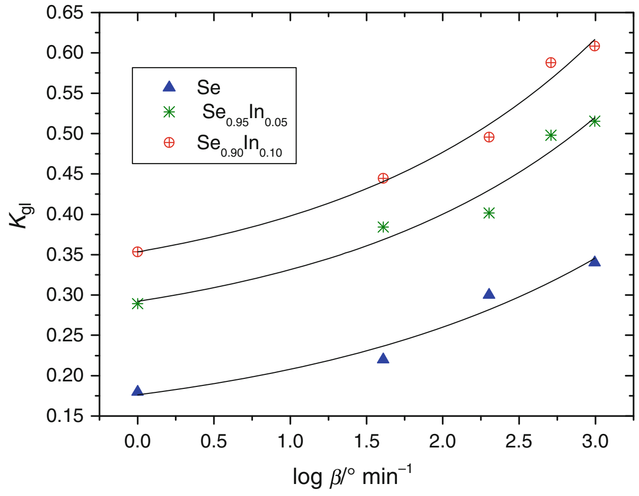

Fig. 5 Variation of K_{gl} with heating rate for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses together with that for g-Se [6, 7]. The *solid lines* represent regression fitting

determined from DTA scans. This coefficient is demonstrated to be a valid indicator of GFA for wide range of materials, with the good glass formers having $K_{gl} \geq 0.1$ (cf., [9] and references cited therein). Figure 5 shows the heating rate dependence of K_{gl} for the Se–In glasses investigated with those of g-Se [6, 7]. This figure declares that the relative dependence of the coefficient K_{gl} is more pronounced than those of *S*-parameter, H', and ΔT. This emerges the monotonic increase of K_{gl} with the introduction of up to 10% In in g-Se that accompanied the increase of both TS and GFA. Such a trend of the two latter parameters agrees with other reported parameters as the macroscopic density and atomic density [19].

Kinetics of glass transition

Understanding the glass transition kinetics is one of the most important issues in the area of glasses applications. This can be realized in terms of the glass transition temperature (T_g) and activation energy of glass transition (E_g). Such activation energy is involved in the molecular motion and rearrangements of the atoms around the glass transition temperature. Throughout this work, evaluation of E_g for the two quenched $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ chalcogenide alloys, has been considered using the two most widely used models, namely Moynihan's [32] and Kissinger's [33].

Moynihan's model

Evaluating E_g for chalcogenide glasses, using the theory of structural relaxation that developed by Moynihan et al. [32], is widely used in literature (cf., [34–36]). This theory depends on the heating rate dependence of T_g. In this kinetic concept, the enthalpy at particular temperature and time, $H(T, t)$, of glassy system after an instantaneous isobaric change in temperature relaxes isothermally towards a new equilibrium value $H_c(T)$. Here, the relaxation equation can be written as:

$$\left(\frac{\partial H}{\partial t}\right)_T = -\frac{(H - H_c)}{\tau} \tag{5}$$

where τ is a temperature dependent structural relaxation time and is given by:

$$\tau = \tau_0 \exp\left(-E_g/RT\right) \exp[-C(H - H_c)] \tag{6}$$

where τ_0 and C are constants, R is the universal gas constant, and E_g is the activation energy of structural relaxation. Using Eqs. 5 and 6, it can be shown [37] that:

$$\frac{d(\ln \beta)}{d(1/T_g)} = -\frac{E_g}{R} \tag{7}$$

So, a plot of ln β versus $(1/T_g)$ should give a straight line where the activation energy involved in the molecular motions and atoms rearrangements around T_g can be calculated from the slope of such a plot. Figure 6 shows a plot of the function: $\ln \beta = f(1/T_g)$ for the two binary Se–In glasses investigated. The respective calculated values of E_g are found to be 299 and 270 kJ/mol for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$. That is the value of E_g shows a decrease with In-content in g-Se matrix. Such a decrease of E_g is accompanied with the increase in the internal energy of the structural matrix.

Kissinger's model

A model given by Kissinger [33] was originally derived for the determination of activation energy of crystallization

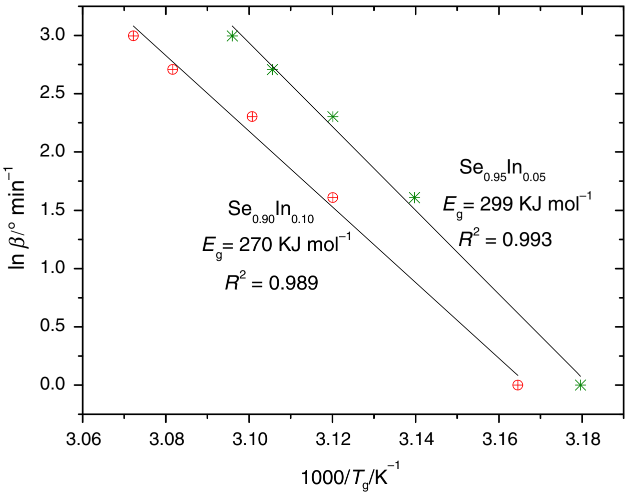

Fig. 6 Plots of ln β versus $1000/T_g$ for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses scanned at different heating rates. The *solid lines* represent least-squares fitting

(E_c). In fact, this model is the most commonly used one that was developed for the use of thermal analysis (DTA or DSC) to study of non-isothermal phase transformations (cf., [11, 15, 38–40]). In this model, efforts have been made to generalize the phenomenological formal Avrami equation [41, 42] thus describing the transformation kinetics under isothermal conditions to be used for non-isothermal (or continuous) heating crystallization conditions. This has been implemented by applying simplified assumptions to determine the fractional volume transformed, α (i.e., the ratio of the volume of new phase to the total volume) from the experimental DTA (or DSC) curves and consequently the kinetics of the transformation process.

According to Kissinger, the value of E_c can be determined from the formula [33]:

$$\ln\left(\beta/T_c^2\right) = -\frac{E_c}{RT_c} + \text{constant} \tag{8}$$

Few years ago, Soliman [43] used the theoretical principles of Kissinger to calculate the value of E_g; i.e., replacing T_c by T_g and E_c by E_g, where the above equation takes the following form for the glass transition kinetics:

$$\ln\left(\beta\Big/T_g^2\right) = -\frac{E_g}{RT_g} + \text{constant} \tag{9}$$

So, the value of E_g can be determined from the slope of $\ln\left(\beta\Big/T_g^2\right)$ versus $1/T_g$ plots that shown in Fig. 7 for the glasses investigated. The calculated values of E_g are found to be 293 and 264 kJ/mol for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$, respectively. These latter values of E_g are in good agreement with those obtained using Moynihan's equation for the considered glassy alloys. Some other parameters, regarding the crystallization kinetics (n, K, E_c) in correlation with the structural nature of these two Se–In chalcogenide glasses are now under consideration and will be published soon.

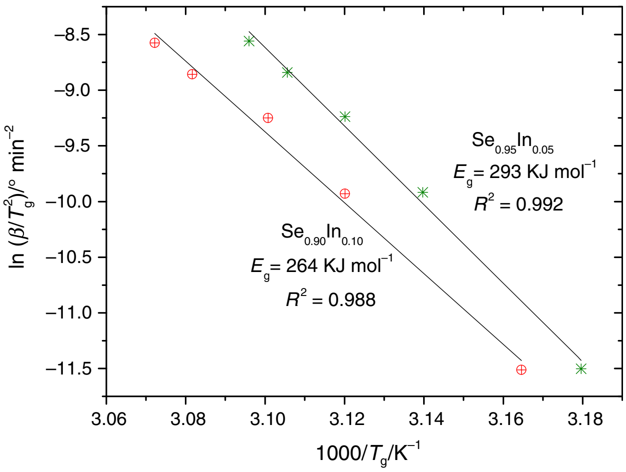

Fig. 7 Plots of $\ln\left(\beta/T_g^2\right)$ versus $1000/T_g$ for $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$ glasses scanned at different heating rates. The *solid lines* represent least-squares fitting

Conclusions

A non-isothermal calorimetric study on the effect of adding 5 and 10% In to g-Se, forming glassy $Se_{0.95}In_{0.05}$ and $Se_{0.9}In_{0.1}$, leads to various interesting concluding remarks. Among these: The value of T_g is found to be dependent on the heating rate. In such case, it is in contrast with that found for either pure g-Se or Se-like structural glasses. The difference in the relative increase of T_g with β refers to the addition effect of In on the structural configuration of the quenched glassy matrix. The increase of the value of T_g with increasing the introduction of In-content (up to 10%) has a positive effect on the calculated stability parameters of g-Se. This emerges the increase of K_{gl} with the introduction of In in g-Se that accompanied the increase of both TS and GFA. The value of E_g showed a decrease with In-content irrespective of the monotonic TS parameters. The coincidence of the calculated values of E_g using Moynihan's and Kissinger's models (296 ± 3 and 267 ± 3 kJ/mol) confirms the validity of both models.

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