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NON-ISOTHERMAL CRYSTALLIZATION KINETICS OF $Sb_{10}Ge_{10}Se_{80}$ CHALCOGENIDE GLASS

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A differential scanning calorimetry technique was used to study the crystallization kinetics of $Sb_{10}Ge_{10}Se_{80}$ chalcogenide glass under non-isothermal conditions. The crystallization parameters such as the order parameter (n), the frequency factor (k_0), the activation energy of crystallization (E_c), the activation energy of glass transition (E_g) and the activation energy of nucleation (E_n) were determined. The value of E_c was deduced by means of six methods and the average value was found to be equal to (76.10 ± 1.10) kJmol^{-1}. The most suitable method for crystallization kinetic studies was the Augis–Bennett approximated method at different heating rates, while the method of Coats–Redfern–Sestak was the most suitable one at constant heating rate. The results have been discussed on the basis of theoretical principles.

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Keywords: chalcogenide glass, crystallization, DSC, glass transitions

1. *Introduction*

The great interest in the condensed matter is mainly due to ever increasing applications in the recent technology. The thermal analysis has been widely used for studying the crystallization of glasses [1 − 3]. The crystallization kinetics of chalcogenide glasses has been applied to investigate their electrical and transport properties, as well as their thermal stability. The crystallization kinetic mechanisms of the chalcogenide glasses can be followed by means of isothermal and non-isothermal technique. In the former technique, a small piece of the sample is brought very quickly near to the crystallization temperature (T_c), then the specific physical property is recorded as a function of time. In the later one, the sample is thermally annealed at a fixed rate of heating and the physical property is recorded as a function of temperature. Several studies have been carried out on the physical properties of the system Sb-Ge-Se, such as glass forming region [4,5], structure [6], optical properties [7,8] and electrical properties [9−12]. The crystallization kinetics of several binary alloys of the system Sb-Ge-Se has been studied [13−18]. The objective of the present work is to study the crystallization kinetics of $Sb_{10}Ge_{10}Se_{80}$ chalcogenide glass using the non-isothermal differential scanning calorimetry (DSC) technique.

2. *Experimental details*

The bulk glass material $Sb_{10}Ge_{10}Se_{80}$ was prepared by the conventional melt-quenching technique. High purity elements (99.999%) of the constituents Ge, Sb and Se, according to their atomic percentage, were weighed in a silica tube that was sealed under vacuum (10^{-3} Pa). The tube was then heated at 1273 K for a period of 24 hours, during which it was frequently agitated to intermix the constituents in order to ensure the homogeneity of the melt. The molten sample was then rapidly quenched in ice-cooled water. The X-ray diffraction (XRD) pattern of the sample powder was recorded by Philips X-ray diffractometer system (X" Pert PRO) using CuK_α ($\lambda = 1.5406$ Å) as incident radiation at a 2θ scan rate of $0.03°/1.5$ s in $10° - 60°$ span. Differential scanning calorimetry thermograms of the bulk sample were carried out at different heating rates (5, 10, 15 and 20 $\mathrm{Kmin^{-1}}$) by means of Setaram thermal analyser model DSC131 (France). The thermograms were recorded in the temperature range 323−473 K. The temperature of both reference specimen and the sample under investigation was uniformly raised. However, the heat exchange between the test sample and its environment depends on absorption or release of heat that may occur leading to either endothermic or exothermic reaction, respectively.

3. *Results and discussion*

3.1. *The activation energy of crystallization*

3.1.1. **Matusita method**

The glassy nature of the prepared sample was confirmed by the XRD. The pattern of the as-prepared $Sb_{10}Ge_{10}Se_{80}$ is shown in Fig. 1. DSC thermograms of the $Sb_{10}Ge_{10}Se_{80}$ chalcogenide glass, obtained at the heating rate $\alpha = 10$ $\mathrm{Kmin^{-1}}$, are shown in Fig. 2. From the trace, three characteristic phenomena are seen. The first one is related to the glass transition temperature ($T_g = 392.3$ K), the second corresponds to the onset of temperature of crystallization ($T_c = 412.9$ K), while the

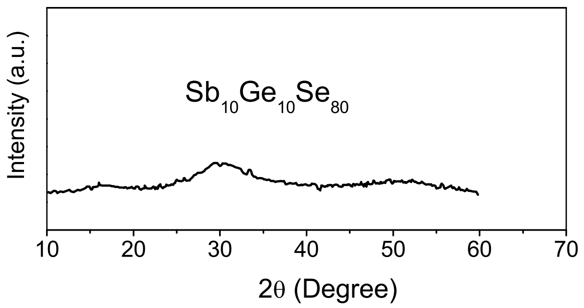

Fig. 1. XRD pattern of the $Sb_{10}Ge_{10}Se_{80}$ *glass.*

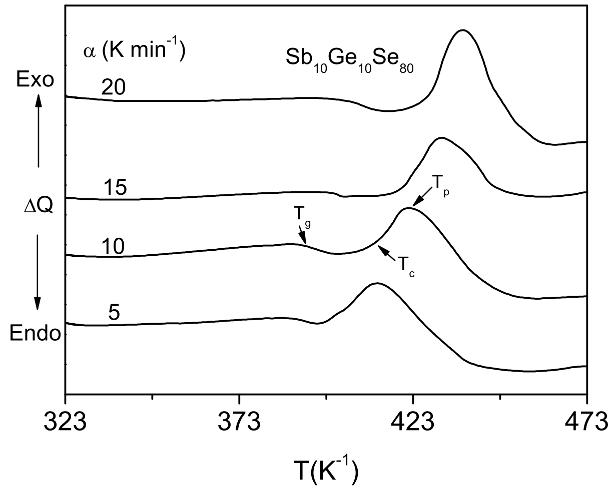

Fig. 2. DSC thermograms at different heating rates for the $Sb_{10}Ge_{10}Se_{80}$ *glass.*

third one is the peak temperature of crystallization ($T_p = 423.1$ K). Figure 2 shows the thermograms of $Sb_{10}Ge_{10}Se_{80}$ at different heating rates. It can be seen that for all heating rates, the exothermic peaks (T_p) are shifted towards higher temperature for increasing heating rates. Johnson [19] and Avrami [20−22] developed the kinetic transformation mechanism by which the volume crystallization fraction χ for an isothermal process is expressed as

$$\chi = 1 - \exp\{-\big(kt\big)^n\}\,, \tag{1}$$

where n is the Avrami exponent that depends on the mechanism and the dimensionality of the crystal growth and k is the reaction rate constant which is temperature dependent according to Arrhenius formula

$$\ln k = \ln k_0 - \frac{E_a}{RT}\,, \tag{2}$$

where k_0 is the frequency factor and E_a is the apparent activation energy of crystallization. The amorphous-crystalline transformation process is fully understood in terms of the crystallization parameters such as the activation energy of glass transition E_g, the order of crystallization mechanism n and the frequency factor k_0. The volume fraction χ, crystallized after a time t at the temperature T, is given by $\chi = A_T/A$, where A is the total area of the exothermic peak lying between the temperature at which the crystallization just begins and the temperature at which the crystallization is completed. A_T is the area between the initial temperature T_c and a generic temperature T. The crystallized fraction χ at the constant heating rate α is related to the activation energy of crystallization E_c through the relation [23,24]

$$\ln\left[-\ln(1-\chi)\right] = -n\ln\alpha - 1.052\, m\frac{E_c}{RT} + const\,, \tag{3}$$

where m and n are integers whose values depend on the mechanism and dimensionality of the crystal growth [25].

The relation between $\ln[-\ln(1-\chi)]$ and $(1/T)$ at different fixed heating rates is shown in Fig. 3. From the figure, it can be seen that all plots are nearly linear with the same slope over most of the temperature range. However, in the high-temperature range, deviation from linearity is observed. This temperature range corresponds to a large crystallized fraction; other chalcogenide glasses indicate a similar behaviour [26, 27]. This deviation has been generally attributed to the saturation of nucleation sites in the final stage of crystallization or to the restriction

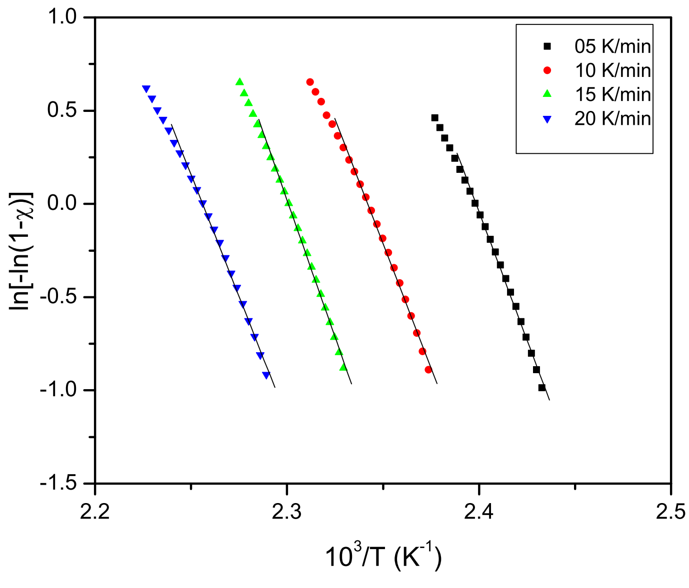

Fig. 3. $\ln[-\ln(1-\chi)]$ *as a function of* $10^3/T$ *at different heating rates for the* $Sb_{10}Ge_{10}Se_{80}$ *glass.*

of crystal growth by the small size of the particles. Figure 4 shows the relation between $\ln[-\ln(1-\chi)]$ and $\ln\alpha$ for two fixed temperatures ($T = 420$ and 430 K) according to the relation [28,29]

$$\frac{\mathrm{d}\ln\left[-\ln(1-\chi)\right]}{\mathrm{d}\ln(\alpha)} = -n\,. \tag{4}$$

From the figure, the order of crystallization n could be deduced using the least square fits. Its average value was found to be 2.71 ± 0.01. The non-integer value of n indicates that the crystallization occurs with fractal dimensionality [1]. There exist two crystallization mechanisms working simultaneously during the amorphous – crystalline transformation with equal shares [27]. They are considered as one- and three-dimensional crystal growth [30−32]. The value of n is taken to be equal to either $(m + 1)$ or m. The former case is taken when the nuclei formed during the heating run at a constant rate are dominant, while the later case is considered when the nuclei formed during previous heat treatment prior to thermal analysis are dominant [23]. According to Eq. (3), mE_c was calculated for all heating rates (see Table 1). It can be noticed that mE_c seems to be heating-rate-independent and, therefore, an average value of mE_c was calculated by considering all heating rates which is equal to (216.84 ± 5.55) kJmol^{-1}. From the value of $n = 2.71$ and the average value of mE_c, the value of activation energy of crystallization according to Matusita method was deduced and is listed in Table 1. In the present study, no specific heat treatment was given before the thermal analysis and accordingly n was taken to be $m + 1$.

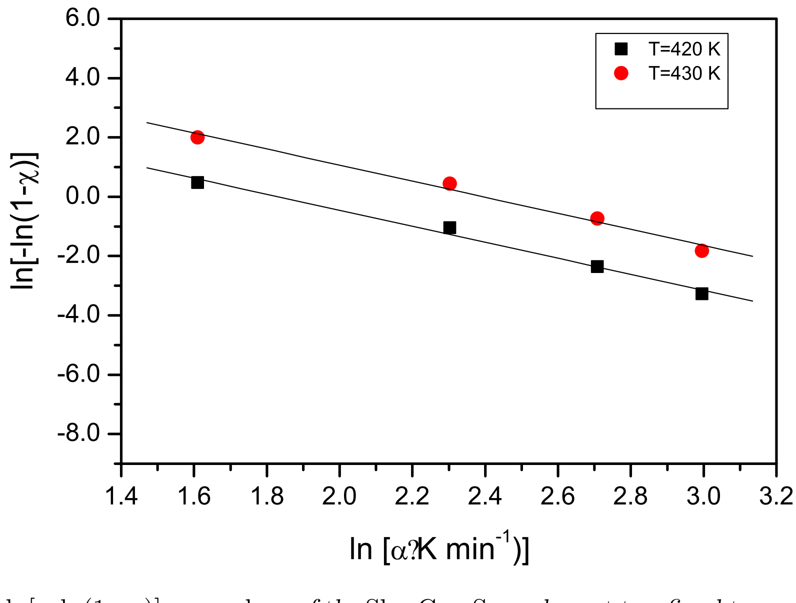

Fig. 4. $\ln[-\ln(1-\chi)]$ *versus* $\ln\alpha$ *of the* $Sb_{10}Ge_{10}Se_{80}$ *glass at two fixed temperatures* ($T = 420$ *and 430* K).

TABLE 1. The values of activation energy of crystallisation E_c, according to Matusita, Eq. (3).

α [Kmin^{-1}]	mE_c [kJmol^{-1}]	E_c [kJmol^{-1}]
5	217.37	127.49
10	211.25	123.90
15	232.14	136.15
20	206.61	121.18
Average value	216.84 ± 5.55	127.18 ± 3.26

3.1.2. Coats–Redfern–Sestak method

From the variation of $\ln[-\ln(1-\chi)/T^{2n}]$ with $(1/T)$ (Fig. 5), the activation energy of crystallization (E_c) was determined at different heating rates using CRS method [33] according to the relation

$$\ln\left[\frac{-\ln(1-\chi)}{T^{2n}}\right] = -n\frac{E_c}{RT} + n\ln\left(\frac{k_0 R}{n\alpha E_c}\right). \tag{5}$$

The average value of E_c was deduced and is listed in Table 2.

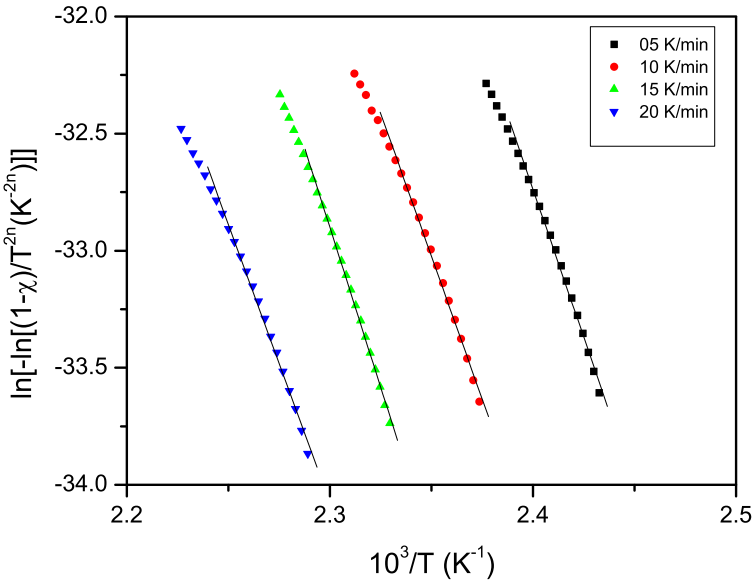

Fig. 5. $\ln[-\ln(1-\chi)/T^{2n}]$ *as a function of* $10^3/T$ *at different heating rates for the* $Sb_{10}Ge_{10}Se_{80}$ *glass.*

TABLE 2. The average values of the activation energy for crystallisation, E_c, glass transition, E_g, and the frequency factor, k_0, deduced by the different methods used.

Method	Eq.	E_c [kJmol^{-1}]	E_g [kJ mol^{-1}]	k_0 [s^{-1}]
Kissinger	(6)	71.99 ± 0.04	61.56	–
Mahadevan et al.	(7)	79.07 ± 0.04	68.15	–
Augis–Bennett	(8)	74.36 ± 0.06	–	2.15×10^{11}
Augis–Bennett approx.	(9)	75.53 ± 0.04	–	6.93×10^{9}
Ozawa–Chan	(10)	78.38 ± 0.10	–	–
Coats–Redfern–Sestak	(5)	77.25 ± 2.35	–	1.31×10^{8}
Average value		76.10 ± 1.10	64.86 ± 3.29	7.39×10^{10}

3.1.3. Kissinger method

The activation energy of crystallization was determined using the Kissinger method of crystallization analysis [35, 36] according to the relation

$$\ln\left(\frac{\alpha}{T_p^2}\right) = -\frac{E_c}{RT_p} + const\,. \tag{6}$$

For this purpose $\ln(\alpha/T_p^2)$ versus $(1/T_p)$ was plotted. The results are shown in Fig. 6, from which E_c was determined. The result is given in Table 2.

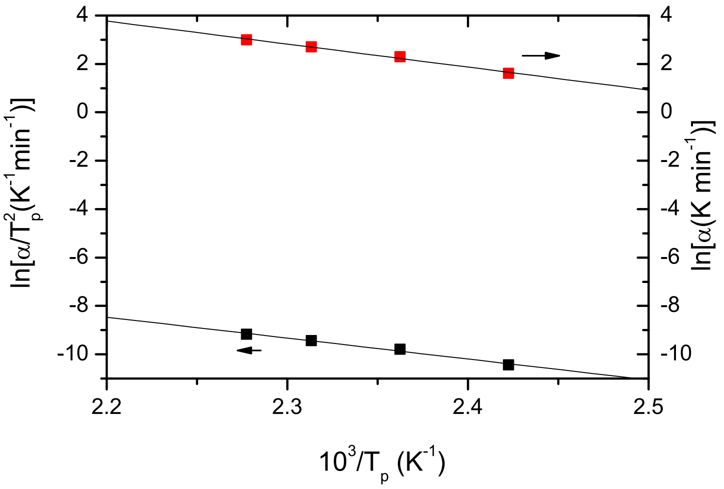

Fig. 6. $\ln(\alpha/T_p^2)$ *and* $\ln\alpha$ *versus* $10^3/T_p$ *for the* $Sb_{10}Ge_{10}Se_{80}$ *glass.*

3.1.4. **Mahadevan method**

The activation energy of crystallization according to Mahadevan method [26] was deduced using the relation

$$\ln(\alpha) = -\frac{E_\mathrm{c}}{RT_\mathrm{p}} + const\,. \tag{7}$$

The relation between $\ln(\alpha)$ and $(1/T_\mathrm{p})$ is shown in Fig. 6 and the resultant value of the activation energy (E_c) is given in Table 2.

3.1.5. **Augis and Benett method**

On the basis of Piloyan [39] and Brochardt [40], Augis and Benett [38] deduced the activation energy of crystallization (E_c) from the variation of $\ln[\alpha/(T_\mathrm{p} - T_\mathrm{c})]$ with $(1/T_\mathrm{p})$ (Fig. 7), according to the relation

$$\ln\left(\frac{\alpha}{T_\mathrm{p} - T_\mathrm{c}}\right) = -\frac{E_\mathrm{c}}{RT_\mathrm{p}} + \ln k_0\,. \tag{8}$$

The values of E_c are listed in Table 2. Augis and Benett considered that $T_\mathrm{p} \gg T_\mathrm{c}$ in Eq. (8), therefore this equation was approximated to the form

$$\ln\left(\frac{\alpha}{T_\mathrm{p}}\right) = -\frac{E_\mathrm{c}}{RT_\mathrm{p}} + \ln k_0\,. \tag{9}$$

The value of E_c was deduced from the variation of $\ln(\alpha/T_\mathrm{p})$ with $(1/T_\mathrm{p})$ (see Fig. 7) and is listed in Table 2.

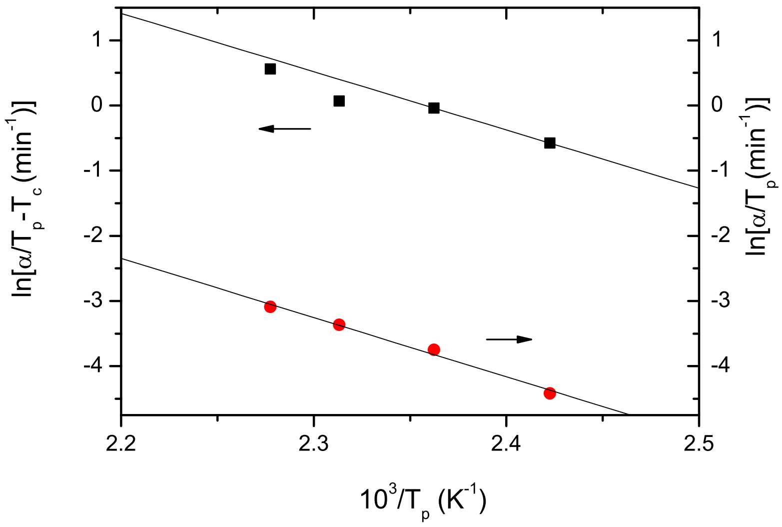

Fig. 7. $\ln[\alpha/(T_\mathrm{p} - T_\mathrm{c})]$ *and* $\ln(\alpha/T_\mathrm{p})$ *versus* $10^3/T_\mathrm{p}$ *for the* $\mathrm{Sb_{10}Ge_{10}Se_{80}}$ *glass.*

3.1.6. **Ozawa–Chen method [37, 41]**

Figure 8 shows the variation of $\ln(\alpha/T^2)$ versus $(1/T)$ for different crystallization fractions (χ), for all heating rates (5, 10, 15 and 20 Kmin^{-1}) according to the relation

$$\ln\left(\frac{\alpha}{T^2}\right) = -\frac{E_c}{RT} + const\,. \tag{10}$$

The average value of E_c is listed in Table 2. The frequency factor k_0 which was deduced according to the Coats–Redfern–Sestak and the Augis–Benett methods are listed in Table 2. The average value of k_0 is equal to 7.39×10^{10} s^{-1}.

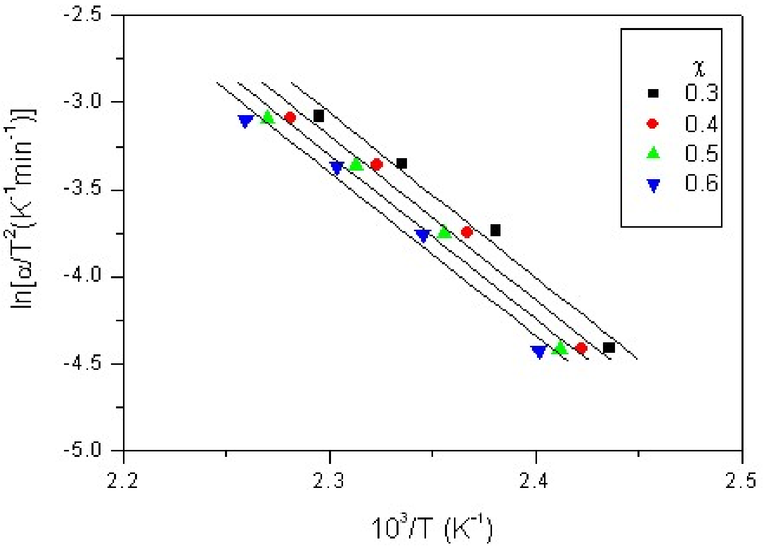

Fig. 8. $\ln(\alpha/T^2)$ *versus* $10^3/T$ *for the* $Sb_{10}Ge_{10}Se_{80}$ *glass.*

3.2. The activation energy of glass transition

Two approaches are used to determine the dependence of T_g on the heating rate α.

The first is the empirical relationship of the form [45]

$$T_g = A + B\ln\alpha\,, \tag{11}$$

where A and B are constants. Plots of T_g versus $\ln\alpha$ are shown in Fig. 9, which confirm the validity of the relation. The calculated values of A and B for $Sb_{10}Ge_{10}Se_{80}$ chalcogenide glass are 352.58 and 18.03 K, respectively.

The second approach that is commonly used in analysing crystallization data in DSC analysis for evaluation of the activation energy for glass transition, E_g, was developed by Kissinger [35 – 37,42], which is expressed as

$$\ln\left(\frac{\alpha}{T_{\rm g}^2}\right) = -\frac{E_{\rm g}}{RT_{\rm g}} + const\,. \tag{12}$$

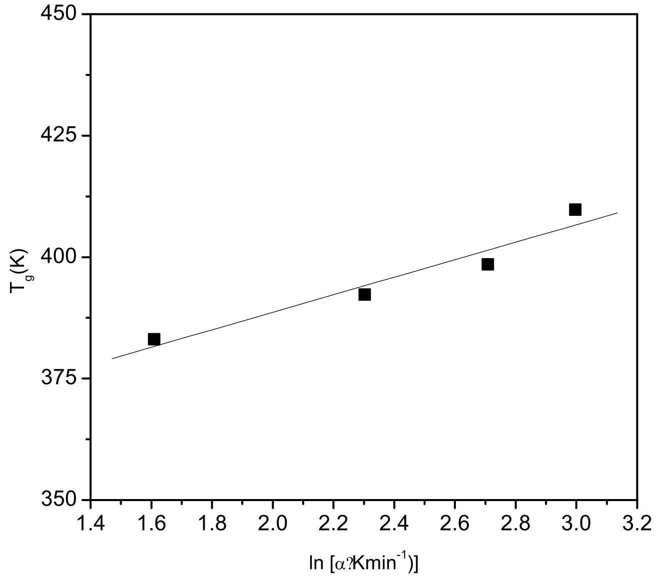

Fig. 9. $T_{\rm g}$ *versus* $\ln\alpha$ *for the* $\mathrm{Sb_{10}Ge_{10}Se_{80}}$ *glass.*

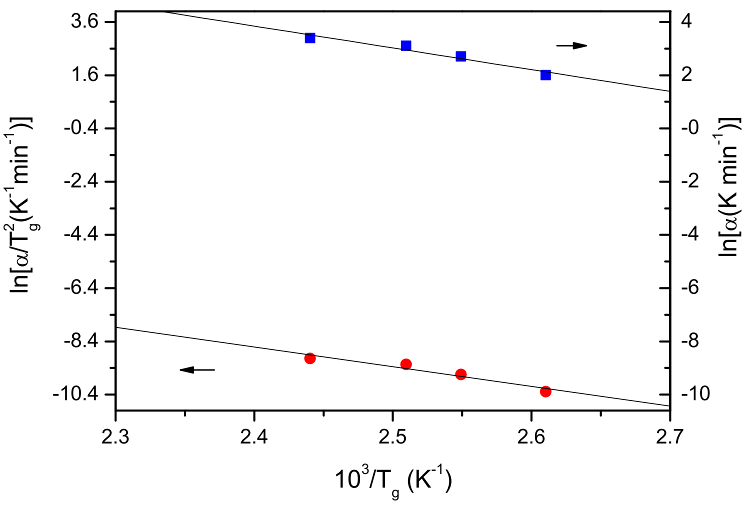

Fig. 10. $\ln(\alpha/T_{\rm g}^2)$ *and* $\ln\alpha$ *versus* $10^3/T_g$ *for the* $\mathrm{Sb_{10}Ge_{10}Se_{80}}$ *glass.*

Figure 10 shows the plots of $\ln(\alpha/T_g^2)$ versus $(10^3/T_g)$ from which the activation energy of the glass transition (E_g) was deduced and is listed in Table 2. The Kissinger equation is approximated by Mahadevan [26] to the form

$$\ln(\alpha) = -\frac{E_g}{RT_g} + const\,. \tag{13}$$

Figure 10 shows the plots of $\ln(\alpha)$ versus $(10^3/T_g)$ from which the activation energy of glass transition E_g was deduced and is listed in Table 2. From this table, it can be seen that E_g value obtained both by the Kissinger method and the Mahadevan method are roughly the same, which indicates that one can use either of the two methods to evaluate the activation energy of glass transition E_g for $Sb_{10}Ge_{10}Se_{80}$ chalcogenide glass. The average effective value of activation energy of crystallization E_c calculated for all above mentioned values is equal to (76.10 ± 1.10) kJmol^{-1}. By comparing the different values of E_c, which are deduced by different methods of calculation, and the average value of E_c, it can be concluded that all methods, except for Matusita method, can be used to determine the value of activation energy of crystallization E_c for the sample $Sb_{10}Ge_{10}Se_{80}$ but with different precisions. The value of E_c could be determined by two methods, either at constant heating rate (i.e., using one thermogram) or at different heating rates (i.e., form different thermograms). In the present work, the most precise method for the determinations of E_c at constant heating rate was found to be that of Coats–Redfern–Sestak, $E_c = (77.25 \pm 2.35)$ kJ mol^{-1}, since it yields the best precision. On the other hand, the evaluation of E_c from different heating rates, by the Augis–Bennett approximated method, $E_c = (75.53 \pm 0.04)$ kJ mol^{-1}, gives a minimum deviation from the average value (0.75 %), and, therefore, it can be considered the most suitable one. Generally the Coats–Redfern–Sestak method can be considered optimal, not only for the precision of E_c, but as it gives the chance to calculate the average value of E_c from different theromgrams (one value of E_c for each heating rate).

3.3. The activation energy of nucleation

According to Vogel [43] and Lu [44], the activation energy of nucleation could be deduced by two methods using the equation

$$E_c = \frac{aE_n - rbE_g}{a + rb}\,, \tag{14}$$

where E_c is the activation energy of crystallization, E_g is the activation energy of glass transition, r is considered to be either 1 or 1/2 for linear and parabolic growth, respectively and $b = 1$, 2, 3 for one-, two- and three-dimensional growth, respectively. $a = 0$ for no nucleation and $a = 1$ for constant nucleation. For the present study, the following values were considered: $a = 1$, $r = 1/2$ and $b = 2.71$ (fractal dimensionality). The values of E_n due to Kissinger [35, 36] and Mahadevan [26] method are 252.96 kJmol^{-1} and 278.55 kJmol^{-1}, respectively.

4. *Conclusions*

From the non-isothermal crystallisation study of $Sb_{10}Ge_{10}Se_{80}$ cahlcogenide glass, we obtained a non-integer value of the Avrami exponent $n = 2.71 \pm 0.01$. This indicates that two crystallisation mechanisms, one and two-dimensional crystal growth, are working simultaneously with equal shares through the amorphous – crystalline transformations. The optimal method to evaluate E_c is the Coats–Redfern–Sestak method. The values of activation energies for glass transition, E_g, using two different methods, are in good agreement with each other. So it can be safely calculated using one of them. The average values of E_c, E_g, k_0 and E_n for $Sb_{10}Ge_{10}Se_{80}$ cahlcogenide glass are (76.10 ± 1.10) kJmol^{-1}, (64.86 ± 3.29) kJmol^{-1}, 7.39×10^{10} s^{-1} and 265.78 kJmol^{-1}, respectively.

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KINETIKA NEIZOTERMNE KRISTALIZACIJE KALKOGENIDNOG STAKLA $Sb_{10}Ge_{10}Se_{80}$

Primijenili smo diferencijalnu pretražnu kalorimetriju za proučavanje kinetike kristalizacije kalkogenidnog stakla $Sb_{10}Ge_{10}Se_{80}$ u neizotermnim uvjetima. Odredili smo sljedeće parametre kristalizacije: parametar reda (n), faktor frekvencije (k_0), aktivacijsku energiju kristalizacije (E_c), aktivacijsku energiju staklenog prijelaza (E_g) i aktivacijsku energiju nukleacije. E_c smo izveli na osnovi šest metoda i našli srednju vrijednost (76.10 ± 1.10) kJmol^{-1}. Najpogodnija metoda za proučavanje kinetike kristalizacije za različite brzine grijanja je Augis–Bennettova približna metoda, dok se metoda Coats–Redfern–Sestak-a pokazala najbolja za stalnu brzinu grijanja. Postignuti ishodi raspravljaju se na osnovi teorijskih postavki.