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INVESTIGATE THE APPROPRIATE MODEL FOR DESCRIBING THE CRYSTALLIZATION PROCESS OF $Te_{65}Sb_{25}Ga_{10}$ THIN FILM

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The sheet resistance, R_s, measurements versus temperature, T, for 1000 nm Te - Sb -Ga thin films were performed at different heating rates ($\beta =$ 5, 10, 20, 30 and 40 K/min) in the temperature range from 300 to 673 K. The kinetic transformation parameters of $Te_{65}Sb_{25}Ga_{10}$ thin film were examined via the theoretical method developed (TMD) and the Johnson–Mehl–Avrami model (JMA). The crystallization activation energy E_c with the transformed fraction is not constant but change with the conversion fraction. These results refer to the rate constant of the transformation is in fact determined through the nucleation and diffusion processes. The results of the impingement exponent γi dependence of the conversion fraction (χ) revealed that the mode of impingement is due to "anisotropic growth". In addition to that the average values of kinetic exponent parameters, n and m obtained by (TMD) and JMA models. are accounted for two and three-dimensional crystal growth with heterogeneous nucleation. Moreover, comparing the experimental curve of the transformed fraction indicated that the crystallization process of $Te_{65}Sb_{25}Ga_{10}$ thin film cannot be satisfactorily described by Johnson-Mehl-Avrami (JMA) model. On contrary, the theoretical curves of the developed model are more realistic for describing the crystallization process of the investigated chalcogenide bulk glass.

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1. Introduction

Chalcogenide glasses containing chalcogen elements (Se, Te and S) have attracted the great interest of the researchers all over the word. These glasses possess the physical property of transmitting wavelengths ranging from 1 to 16 μm and having energy gaps of 1-3 eV which make them highly efficient candidates for optical devices. The investigation of the pre-crystallization and crystallization of the amorphous-crystalline transformation kinetics in amorphous glass is essential to investigate the kinetics of chemical reactions of chalcogenide glass. In addition, the crystallization kinetics studies of the phase transitions are considered important phenomena from the point of view of physics and practical applications because they enable the optimizing of the crystallization parameters which are useful for several applications such as memory and storage devices. Furthermore, the doping of the chalcogenide alloys by the different interesting elements including, S [1], Sn, Ga, Sb [2, 3] and Ag [4, 5] has attracted the remarkable interest for investigating the crystallization and amorphous kinetics based on the introduction of impurity elements into the host alloys. The addition of the such elements into the host alloy could not only improve the physical properties but also participate in finding new materials for the practical applications. Therefore, chalcogenide glasses have a given place in the material industry, widely

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used for thermal cameras, infrared detectors (IR) and optical devices such as IR amplifiers, fiber lasers [1, 4] and high-performance multi-functional UV photodetectors[6]. Furthermore, the fabrication methods of chalcogenide glasses are simple and low cost [7].

Moreover, the kinetic investigations of the amorphous materials are among the most significant aspects of thermal analysis. The kinetics is closely associated with the thermal conversion reaction mechanism, which effectively assists in determining the thermal stability parameters from the information of the reaction mechanism [8]. In addition, it has been known that the kinetics knowledge of chalcogenide glasses is most effective in providing important information which is essential for obtaining the kinetic parameters which enable the development as well as the use of chalcogenide glasses in several applications [9, 10]. The investigations of the crystallization kinetics or amorphous crystallization transformation are carried out by widely used techniques such as differential scanning calorimetry (DSC) which is considered as, low-cost as well as rapid method [11-13].

In this regard, Te-Sb-Ga compound was chosen for study for several considerations, including the effect of adding the Gallium element to the binary Te-Sb compound and also applying several theoretical models to study the phenomenon of the transition from amorphous to crystalline phase in the suggested composition. Moreover, another objective of this study is to find out which theoretical model could be appropriate for analyzing the glass-crystallization kinetics of the proposed composition. Many experimental and theoretical approaches can be used to investigate the glass-crystallization transformation phenomenon of the amorphous glasses. Among those, the DSC technique is widely used for the study of the crystallization kinetics of the amorphous materials where the experiments are carried under iso-thermal conditions where such type of studies is important for the industrial applications [11-13]. On the other hand, several theoretical models have applied for analyzing the experimentally measured DSC data [9, 14]. Among the recent theoretical methods is the developed method (TDM) which makes use of the mutual interference of regions growing from separated nuclei. Furthermore, one of the advantages of such method is that it is applied under different theoretical and experimental conditions [15].

In the present study, the transformation kinetic parameters such as the activation energy E_c, and Avrami exponent n ware deduced utilizing both the theoretical method developed (TMD) and the Johnson-Mehl-Avrami (JMA) model. The transformation kinetic parameters and the experimental curve of the transformed fraction (χ) are used to test the capability of theoretical models such as Johnson-Mehl-Avrami model and developed methods in analyzing the $Te_{65}Sb_{25}Ga_{10}$ thin film.

2. Theoretical background approach

The theoretical approach for interpreting DSC and DTA data was provided via the theory of kinetic transformation of solid-state[16-18]. This theory postulate that the crystal- growth is anisotropic, in accordance with this theory the volume $V\,(t,\,\tau)$ could be expressed as follows[16, 17, 19].

$$V(t,\tau) = g\prod_i \int_{(1-\sigma)\tau}^{t} \nu_i(t'dt') \tag{1}$$

where τ is the nucleation period, " *σ is a parameter in the case of site saturation equals to unit or in the case of the continuous nucleation equals to zero*" [18] and g is a geometric factor based on the shape of the crystal- growth and the dimensionality[14, 19]. Otherwise, the nucleation frequency per unit volume, $I_v(\tau)$ and crystal- growth rate v(t) which depends on the time as a power law are generally described by [14, 18, 19].

$$I_v(\tau) = I_{vo}\tau^{p}\exp(-E_N/RT) \tag{2}$$

$$v(t) = v_o t^q \exp(-E_G/RT) \tag{3}$$

where E_N , E_G are the effective activation energies for nucleation and growth, respectively, p and q are the exponents for each of the quoted power laws. In accordance with *Cardenas-Leal et al* [18], the transformation fraction χ_e, under non-isothermal condition is written as:

$$\chi_e = D(T^2/\beta)^n \exp(-E_c/RT) \tag{4}$$

where D, in $(\mathrm{Ks})^{-n}$ is a constant which is a constant which concerning with the probability of the effective collisions and E_c (kJ mol^{-1}) apparent crystallization activation energy , Further, considering the hypothesis of random nucleation the relation between the change in the extended volume, V_b, and the actual volume, V_a, can be written in the following form:

$$dV_b = \left(1 - \frac{V_b}{V}\right)^{\gamma i} dV_e = (1-\chi)^{\gamma i}\, dV_e \tag{5}$$

where $\chi = V_b/V$ is the actual transformation volume fraction, V is the sample volume and γ_i is impingement exponent and assuming $dV_e = V\, d\chi_e$, Eq.(5) can be described in the following equation*:*

$$(1-\chi)^{-\gamma i}\, d\chi = d\chi_e \tag{6}$$

according to define of an impingement factor $\delta_i = (\gamma_i - 1)^{-1}$, the general solution of the previous differential equation is given as

$$\chi = 1 - (1 + \chi_e \delta_i^{-1})^{-\delta_i} \tag{7}$$

Through substituting Eq. (4) into Eq. (7) the general expression of the actual transformed fraction for non-isothermal process corresponding to the theoretical model developed (TMD) can be given as[18, 20].

$$\chi = 1 - \left[1 + \frac{1}{\delta_i} D(T^2/\beta)^n \exp(-nE_c/RT)\right]^{-\delta i} \tag{8}$$

On the other hand, according to the Johnson-Mehl- Avrami (JMA) model the impingement exponent $\gamma_i = 1$, $\delta_i \rightarrow \infty$ so, the Eq.4 from Eq.7 becomes.

$$\chi = 1 - \lim_{\delta_i \to \infty} \left[1 + \left(\frac{\delta_i}{\chi_e}\right)^{-1}\right]^{-\delta_i} = 1 - \exp(-\chi_e) = 1 - \exp\left[-D\left(\frac{T^2}{\beta}\right)^n \exp\left(\frac{-nE_c}{RT}\right)\right] \tag{9}$$

In the subsequent results section, will be deducing that the values of the constant D and the kinetic exponent are different for each model, while the value of the activation energy is the same in both models.

2.1. Evaluation of the kinetic parameters

The kinetic analysis of the amorphous–crystalline conversion of materials under non-isothermal conditions can be performed utilizing several different ways[13, 21, 22]. In this present work, will be used the theoretical method developed (TMD) model to evaluate the values of kinetic parameters as the impingement factor δ_i, and the kinetic exponent n, in terms of the conversion characteristics [T_p, χ_p, and $(d\chi/dt)_p$].

i. the impingement factor δ_i: will be evaluated at different heating rates β from the equation

$$(1-\chi_p) = \left(\frac{\delta_i}{\delta_i - 1}\right)^{\delta_i} \tag{10}$$

ii. The crystallization activation energy E_c of $Te_{65}Sb_{25}Ga_{10}$ thin film can be deduced via two methods:

1. Linear relationship according to equation given as

$$\ln \frac{T_p^2}{\beta} = \frac{E_c}{RT_p} - \ln h \tag{11}$$

The slope and intercept give respectively E_c and the quantity $h = D^{1/n}$, which is related to the probability of effective collisions.

2. Obtain the activation energy E_c according to TMD model[18, 23] of the amorphous–crystalline transformation of the glass as a Function of the transformation volume fraction χ as well temperature T. From the above mention Eqs. 4 and 7.

$$D^{1/n} \frac{T^2}{\beta} \exp(\frac{-E_c}{RT}) = \left\{\delta_i \left[(1-\chi)^{1/\delta_i} - 1\right]\right\}^{1/n} = F(\chi) \tag{12}$$

The $F(\chi)$ function depends on only of the transformation volume fraction, by taking the logarithmic expression for Eq. (12).

$$\ln \frac{\beta F(\chi)}{T^2} = \frac{1}{\langle n \rangle} \ln D - \frac{E_c}{RT} \tag{13}$$

Plotting ln [$\beta F(\chi)/T^2$] vs. $1000/T$ the slope and intercept give respectively E_c and the constant D .

iii. The kinetic exponent, n_p and m_p can be Determined according to TMD and JMA models from the following equations[20, 24, 25].

iv.

$$n_p = RT_p^2 (\frac{d\chi}{dt})_p [(1-\chi)^{\gamma i} \beta E_c]^{-1} \tag{14}$$

$$m_p = \frac{RT_p^2 (d\chi/dt)_p}{(1-\chi)\beta E_c} \tag{15}$$

$$n_p = R(dx/dt)_p (T_p^2)/(0.37\beta E_c) \quad \text{For JMA model} \tag{16}$$

3. Experimental procedures

The synthesis steps of the chalcogenide Te-Sb-Ga glass by the melt quenching technique are summarized as follow. Te, Sb and Ga of 6N purity were carefully weighted according to the elements concentration in the proposed composition. In the next step the weighted materials were inserted in clean quartz glass ampoules which are sealed under a vacuum of 10^{-5} Pa to prevent the oxidation or the impurity contamination during the heating process. The melt of mixed materials were obtained by heating the sealed ampoules in a furnace at 950 K for 24 h. In the final step the melt was rapidly ice water at 273K-quenched. The prepared bulk glass was cleaned and grinded to get powder sample for the DSC experiments. Thin films were deposited onto well-cleaned glass substrate at room temperatures by thermal evaporation technique using a high vacuum coating unit (Edward E 306A, UK). The thickness of the investigated films was kept ~ 1000 nm. In order to obtain resistance–temperature measurements, the electric current at a constant voltage supply was measured against temperature and the phase transition temperature, T_c of the investigated film was computed. Post deposited Al electrodes in a square geometry (5.0 mm) were used for resistance-temperature measurements, carried out in the running vacuum. The current was measured by a digital picoammeter (DPM-111 Scientific Instruments, Roorkee) and two-point probe method was used for measuring electrical properties.

4. Results and discussion

4.1. Thermal analysis

The differential curves versus temperature could be re-drawn after determining the temperature region for $Te_{65}Sb_{25}Ga_{10}$ thin film at different heating rates ($5 \leq \beta \leq 40$ K/min) (see in Fig. 1). It is clear that, there are one onset crystallization temperature T_c, and one exothermic peak T_p corresponding to the crystallization. In addition, the values of the characteristic temperatures T_c, T_p, and the full width at half maximum of the crystallization peak ΔT with various heating rates were summarized in Table 1.

Table 1. values of characteristic temperatures and maximum crystallization rate, $(d\chi/dt)|_P$, corresponding temperature, T_P, and crystallized fraction, χ_P of $Te_{65}Sb_{25}Ga_{10}$ thin film.

Experimental data of $Te_{65}Sb_{25}Ga_{10}$					
β C^o/min	T_c(C^o)	T_p(C^o)	ΔT (C^o)	($d\chi/dt$) × 10^{-3} s^{-1}	χ_p
5	198	226	21.1	3.2	0.484
10	205	238	21.5	6.5	0.524
20	214	249	22	12.7	0.538
30	219	256	22.2	19.2	0.550
40	577	258	22.6	25.2	0.571

It is obviously seen that T_c, T_p, and ΔT increase with increasing the heating rates. Otherwise, the actual volume fraction χ crystallized at a given temperature T is given as a ratio between A and A_T, where A_T is the total area of the exothermic between the onset temperature T_c as crystallization just begins and the temperature T_f where the crystallization is completed and A is the area between T_c and T_i i.e. $\chi = (A/A_T)$ (see in Fig. 2a)). The derivative of this ratio with respect to the time gives the transformation rate ($d\chi/dt$). As shown in Fig. 2b and Table 2, the value of the maximum transformation rate, $(d\chi/dt)_P$ increases with increasing the heating rate. This behavior is widely observed in literatures [21, 26-28].

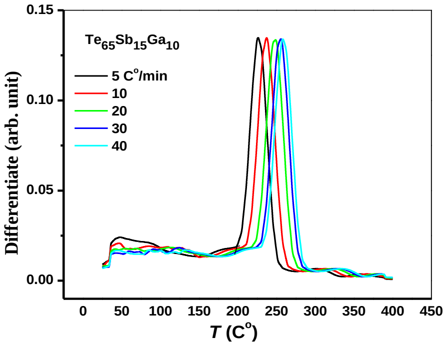

Fig. 1. The differential curves versus temperature for $Te_{65}Sb_{25}Ga_{10}$ thin film at different heating rate.

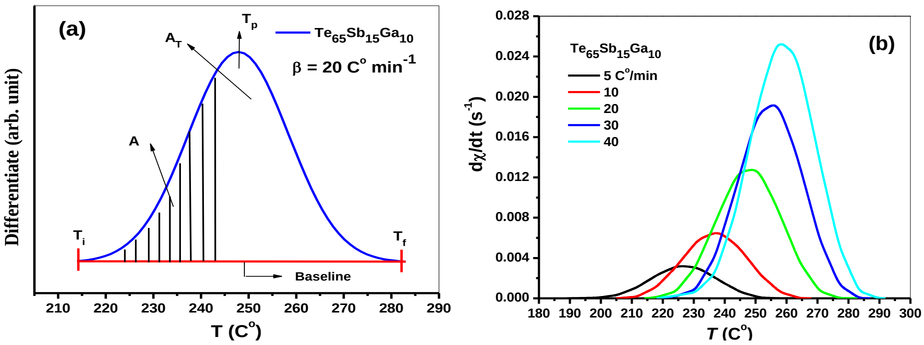

Fig. 2. (a) DSC traces of $Te_{65}Sb_{25}Ga_{10}$ thin film at β = 20 C° min^{-1}. A_T is the area between the temperature T_i and T_f. (b) The crystallization rate as a function of temperature of $Te_{65}Sb_{25}Ga_{10}$ thin film at different heating rates.

Table 2. Values of the activation energy, E_c, kinetic exponent n_p, m_p, impingement factor δ_i, quantity h and D constant for different heating rates of the $Te_{65}Sb_{25}Ga_{10}$ thin film.

	TMD							JMA	
	β C° /min	δ_i	n_p	m_p	E_c K.Cal.mol^{-1}	h (C° s)$^{-1}$	D(C° s)$^{-n}$	n_p	D(C° s)$^{-n}$
$Te_{65}Sb_{25}Ga_{10}$	5	0.853	2.18	1.036	28	125	5.48*10^4	1.71	1.48*10^4
	10	1.305	2.19	1.242				1.86	
	20	1.555	2.22	1.389				1.99	
	30	1.838	2.33	1.512				2.1	
	40	2.604	2.37	1.62				2.21	

4.2. Glass - crystal transformation

4.2.1. Evaluation of the impingement factor δ_i

In this study, the kinetic transformation parameters of $Te_{65}Sb_{25}Ga_{10}$ thin film will be examined through the TMD model. According to the experimental χ_p values given in Table 2, it is possible to procure the parameter of impingement factor δ_i identical with different heating rates β for crystalline peak of $Te_{65}Sb_{25}Ga_{10}$ thin film. The results indicate that the average values of parameter δ_i are 1. 63 ± 0.292, bearing in mind when $\gamma_i > 1$ $\delta_i = (\gamma_i - 1)^{-1}$ the mode of impingement is appropriate to "anisotropic growth"[16, 17, 29]. In addition, the values of the transformation fraction χ_P decreases with increases γ_i as well as showed in Fig. 3. The increase of the impingement effect may be because addition the Ga-atom to Sb-Te, which increases the impingement effect of the complicated structural units in the composition of the glass this conclusion is in good agree with the result anisotropic growth[16, 21, 30].

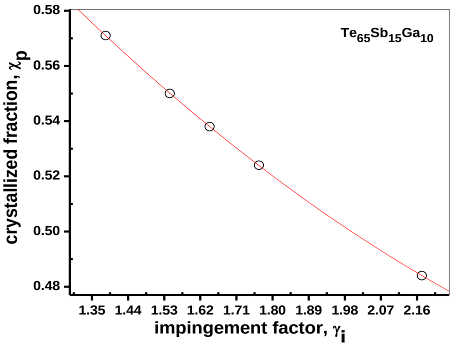

Fig. 3. The dependence of the conversion fraction χ_p, corresponding to the maximum transformation rate, on the impingement exponent γi, of $Te_{65}Sb_{25}Ga_{10}$ thin film.

4.2.2. Evaluation of the activation energy of crystallization E_c

The kinetic parameters E_c and D of the two crystallization peaks of $Te_{65}Sb_{25}Ga_{10}$ thin film could be obtained according to the linear Eq.(11). Plotting $\ln \beta / T_P^2$ against $1000/T_P$ with different heating rates β as shown in Fig. 5 from the slope of the straight lines the values of E_c and constant h $(=D^{1/n})$ could be obtained from the intercept. The obtained values showed that the activation energy E_c, can be interpreted in terms of the rule of Ga in enhancing the crystallization process which made to changes the short-range order in the structure in this glassy system[31, 32]. The deduced values of the activation energy E_c of the both crystallization stage and the values of h of $Te_{65}Sb_{25}Ga_{10}$ thin film are given in Table 2.

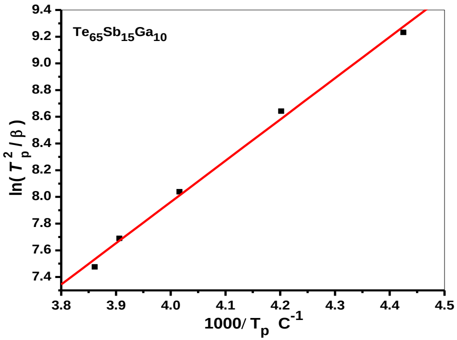

Fig. 4. Plot of $\ln\left(\beta/T_p^2\right)$ versus1000/T_p of $Te_{65}Sb_{25}Ga_{10}$ thin film.

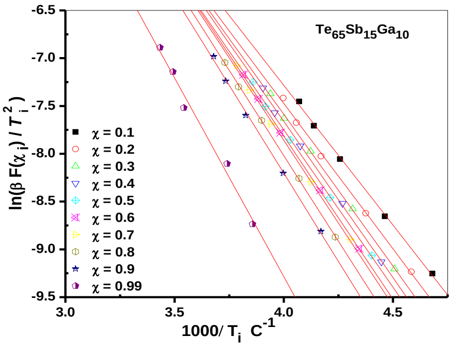

Fig. 5. Linearization plots for using a theoretical method developed TMD of $Te_{65}Sb_{25}Ga_{10}$ thin film.

Moreover, it is possible that evaluated the activation energy E_c as a function of the conversion (χ) using a theoretical method developed TMD. According to the Eq.(13). Plotting the $\ln\left[\beta F(\chi)/T^2\right]$ various $1/T$ for each conversion level $0.1 \leq \chi \leq 0.9$ can give the activation energies of crystallization of the two crystallization peaks of $Te_{65}Sb_{25}Ga_{10}$ thin film. Fig. 6 show the linear plots obtained (given by Eq. 13). From the slope of the straight lines the values of E_c are obtained and plotted against the conversion (χ) as shown in Fig. 7. The result show that the activation energy E_c increase from about 24.3 to 32.5 Kcal mol^{-1} with increasing χ from 0.1 to 0.9, respectively. A similar behavior has been observed in other chalcogenide compositions [33, 34]. The percentage change of E_c with χ is about 10 % which could be accounted for one step mechanism controlling the crystallization process of $Te_{65}Sb_{25}Ga_{10}$ thin film [35-37]. Moreover, the variation of the activation energy may be attributed to there are two crystallization mechanisms are accountable for the transformation process [38, 39]. Also, the values of the activation energy of crystallization E_c of $Te_{65}Sb_{25}Ga_{10}$ thin film, obtained as a function of the conversion (χ) (Fig.6) are in a good agreement with that obtained with the linear Eq. 11 (Table 2).

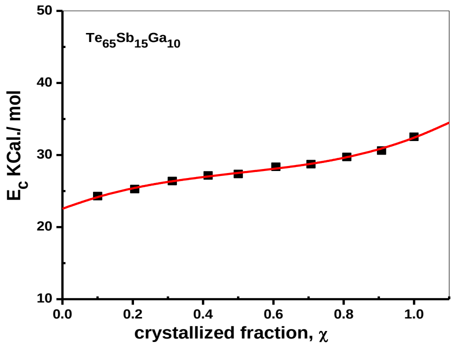

Fig. 6. The activation energy E_c as a function of conversion χ a theoretical method developed TMD of $Te_{65}Sb_{25}Ga_{10}$ thin film.

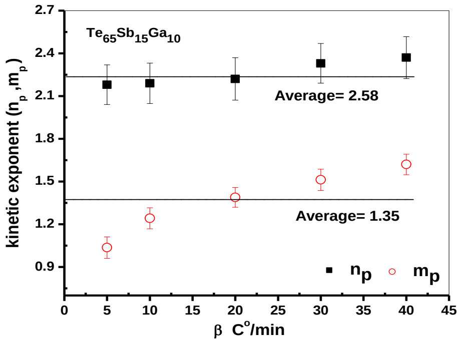

Fig. 7. The dependence of the kinetic exponents (n_p and m_p) as a function of different heating rates β of $Te_{65}Sb_{25}Ga_{10}$ thin film.

4.2.3. Analysis of the kinetic exponent, n_p and m_p

With the aim to obtain information concerning of the kinetic exponent, n_p and m_p as a function of the heating rate by using Eqs. (14), (15) and (16) and according to the values of the impingement factor δ_i obtained above-mentioned section 4.2.1. The dependence of kinetic exponent, n_p and m_p $Te_{65}Sb_{25}Ga_{10}$ thin film on the heating rate β shown in Fig 7 and listed in table 2. The observed values of n_p and m_p of an average value of 2.85 and 1.35 also, from the average values of n_p and m_p indicated that the n_p value could be taken as ($n_p = m_p + 1$). Additionally, these results indicated that the nuclei formed through the heating at constant rate β are dominant and there are two crystallization mechanisms (volume nucleation with two- and three-dimensional growth) are accountable for the transformation process, which take place simultaneously through the amorphous-crystalline transformation process of the $Te_{65}Sb_{25}Ga_{10}$ thin film [38, 39].

4.2.4. Attributive behavior of the crystallization kinetics

In this section, using all obtained transformation kinetic parameters according to TMD analysis of $Te_{65}Sb_{25}Ga_{10}$ thin film. to give the expressions of the theoretical transformed fraction χ as functions of the temperature T. Considering Eqs. (8) and (9) corresponding to the two quoted models. Fig. 8 (a and b) for heating rate 20 C°/min of every peak as an example represent the experimental and theoretical curves χ various T. These results refer to a satisfactory agreement between the experimental curve and the theoretical curves of the TMD whereas the theoretical curve of the JMA model shows an enough large disagreement with the experimental curve. This fact is the second reason which advises to recommend the TMD in order to analyze the non-isothermal glass-crystal transformation kinetics of as-quenched materials with “site saturation”[15, 18].

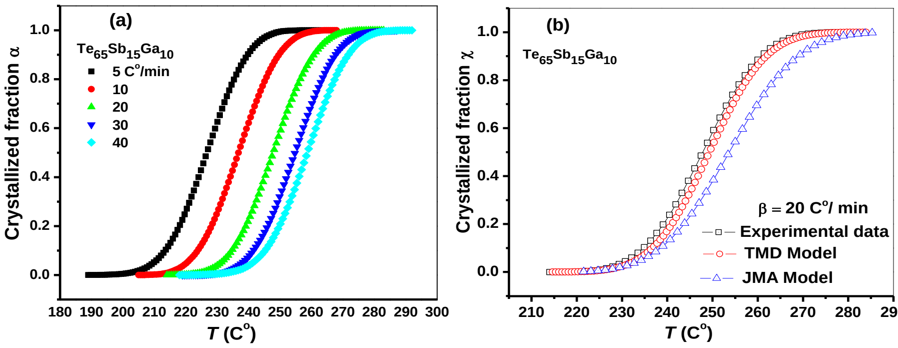

Fig. 8. (a, b) Comparison of the experimental curve of the transformed fraction with calculated one using the JMA and theoretical method developed (TMD) models, at different heating rates of $Te_{65}Sb_{25}Ga_{10}$ thin film.

Finally, this so-called site saturation assumption is an important to this crystallization process where the crystallization rate is only known by the temperature and exhibits a little dependence on the thermal history [13, 40, 41].

5. Conclusions

The kinetic of crystallization parameters were deduced via the theoretical method developed (TMD). The crystallization activation energy E_c with the transformed fraction is not constant but change with the conversion fraction. These results refer to the rate constant of the transformation is in fact determined through the nucleation and diffusion processes. The results of the impingement exponent γi dependence of the conversion fraction (χ) revealed that the mode of impingement is due to “anisotropic growth”. In addition to that the average values of kinetic exponent parameters, n and m obtained by (TMD) and JMA models. are accounted for two and three-dimensional crystal growth with heterogeneous nucleation. Finally, analyzing of the experimental curve of the transformed fraction using the JMA and theoretical method developed (TMD) models revealed that the (TMD) model is suitable for describing the crystallization process of $Te_{65}Sb_{25}Ga_{10}$ thin film

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