

# A Survey on Isoconversional and Model Fitting Methods for Fe-based Metallic Glass

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**ABSTRACT:** Isoconversional methods like Kissinger-Akahira-Sunose(KAS), Ozawa-Flynn-Wall(OFW), Li-Tang(LT), Friedman(FR) and Vyazovkin as well as model fitting methods like Invariant Kinetic Parameter method(IKP), Coats and Redfern (CR) were utilized to understand the crystallization kinetics and to determine the kinetic triplet (activation energy for crystallization (E), pre-exponential factor (A) and the conversion function  $f(\alpha)$ ) for Fe-based metallic glass  $\text{Fe}_{77}\text{B}_{16}\text{Si}_5\text{Cr}_2$ (2605S3A). The non-isothermal differential scanning calorimetry (DSC) data obtained at different heating rates ranging from 2 to 10 K/min are used for the analysis. The E values for peak I and II obtained by the model fitting IKP method are in good agreement with the values obtained from the isoconversional KAS and OFW methods. The calculated kinetic triplets from IKP for peak I is:  $E=414.8\pm 47.1\text{kJ/mol}$ ,  $A= 4.3\times 10^{22}\text{ sec}^{-1}$  and  $f(\alpha) = [-\ln(1-\alpha)]^{1/4}$  and for peak II is:  $E= 340 \pm 6.4\text{ kJ/mol}$ ,  $A= 5.7\times 10^{16}\text{ sec}^{-1}$  and  $f(\alpha) = [-\ln(1-\alpha)]^{1/5}$ . The isoconversional and model fitting methods are discussed elaborately.

**KEYWORDS:** Activation energy for crystallization, Crystallization kinetics, Fe based metallic glass, Isoconversional and model fitting methods.

## I. INTRODUCTION AND RELATED WORK

The kinetic analysis of non-isothermal data (DTA, DSC etc.) is based on the rate equation [1]

$$\frac{d\alpha}{dt} = k(T) f(\alpha) = A \exp\left(-\frac{E}{RT}\right) f(\alpha) \quad (1)$$

where  $\alpha$  is the fractional conversion in the solid reactant, A the Arrhenius pre-exponential factor, T the reactant temperature,  $f(\alpha)$  is kinetic function that depends on the reaction mechanism (reaction model), E the activation energy for the reaction, and R the gas constant. In practice most of the non-isothermal experiments in thermal analysis are carried out at a constant heating rate ( $\beta$ ) =  $dT/dt$  and the kinetic parameters E and A are determined from several thermograms recorded at different  $\beta$ .

Isoconversional methods requires the information of temperature  $T_\alpha(\beta)$  at which fractional conversion  $\alpha$  is constant, for  $\beta$ [2]. This method is further divided into two categories: 1. linear isoconversional and 2. non-linear isoconversional methods. The linear isoconversional methods are again classified into two groups. One group of methods depends on approximating the temperature integral and requires data  $T_\alpha(\beta)$ . These are called linear integral isoconversional methods and proposed by Kissinger-Akahira-Sunose (KAS) [3,4] Ozawa-Flynn-Wall (OFW) [5,6] and Li and Tang [7]. In these methods E values are calculated at a specific  $\alpha$ . Kissinger, Augis & Bennett [8], Boswell [9] methods are the special cases of KAS and Ozawa is that of OFW, because they evaluate only a single value of E at peak crystallization temperature ( $T_p$ ). Second group of isoconversional methods depends on the rate of transformation ( $d\alpha/dt$ ) at  $T_\alpha(\beta)$  as well as on  $T_\alpha(\beta)$ [10]. These methods are referred to as linear differential isoconversional methods, which include Friedman [11] and Gao & Wang [12]. An advanced non-linear isoconversional method is described by Vyazovkin [13, 14] which assumes that the reaction model,  $f(\alpha)$  is independent of the  $\beta$ . In the linear isoconversional methods (integral and differential) the E is evaluated from the slope of a straight line while in the non-linear procedures, the E is deduced from a specific minimum condition. The isoconversional methods, being model-free, are considered to give accurate values of the kinetic parameters E and A. To utilize these isoconversional methods, DSC curves are recorded at various

$\beta$  and presumed that the kinetic parameters of the investigated process are independent of  $\beta$ . It was shown that if  $E$  is independent of  $\alpha$ , all these methods lead to the same value of  $E$ .

The isokinetic analysis depends on the reaction model. It also assumes the Arrhenius temperature dependence of the rate constant  $K(T)$ . Model fitting methods do not achieve a clean separation between the temperature dependent  $K(T)$  and the reaction model  $f(\alpha)$ . Moreover, the temperature sensitivity of the reaction rate depends on the extent of conversion. As a result, these methods are considered to be approximate. Among the various methods of this type, the CR method [15] and the invariant kinetic parameters (IKP) method [16] are used.

Present investigation elaborately utilizes isoconversional and isokinetic (model fitting) methods to understand the crystallization kinetics of Fe-based  $Fe_{77}B_{16}Si_5Cr_2$  (2605S3A) metallic glass.

## II. EXPERIMENTAL METHODS

Specimens of amorphous 2605S3A ribbons were gifted from Metglas, USA. As-quenched samples were heated in DSC (Mettler Toledo) at five linear heating rates (2, 4, 6, 8 and 10 K/min) from room temperature to 840 K in air. The DSC scans were recorded by a thermal analyzer interfaced to a computer.

## III. RESULTS AND DISCUSSION

The DSC thermograms of 2605S3A bulk amorphous alloy at different heating rates are shown in Fig.1. All the DSC traces have double exothermic peaks in the temperature scanned. It is noticed that the  $T_p$  shifted to high temperatures with increasing heating rate. This indicates that the crystallization behaves in a marked kinetic feature. This shift in the peak forms the basis for determination of  $E$ . Due to increase in the sensitivity, the peak height also increases with increasing heating rate.

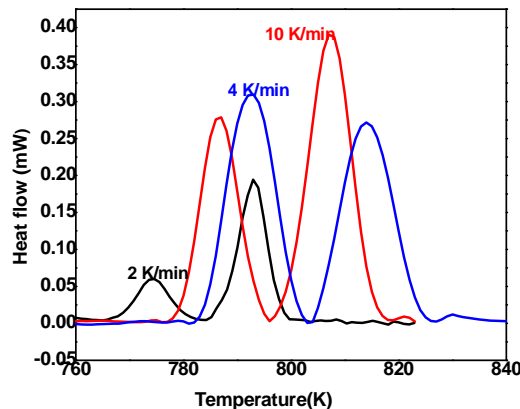


Fig.1. DSC curves at different heating rates

### A. Isoconversional Methods

#### 1. Linear Integral isoconversional method

Kissinger-Akahira-Sunose (KAS) Method

This method is based on the expression

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left(\frac{AR}{Eg(\alpha)}\right) - \frac{E}{RT} \quad (2)$$

The plot  $\ln(\beta/T_\alpha^2)$  vs  $1000/T_\alpha$  for constant conversion,  $\alpha$ , gets the local activation energy  $E(\alpha)$ , given for both the

peaks in Fig.2 and the values are in the Tables I & II.

**2. Ozawa -Flynn-Wall (OFW) method**

OFW method involves the measurement of the temperature  $T_\alpha$ , corresponding to a fixed value of  $\alpha$ , at different  $\beta$ . This method is based on the expression

$$\ln \beta = -1.0516 \frac{E(\alpha)}{RT_\alpha} + const \tag{3}$$

The plot of  $\ln \beta$  vs  $1000/T_\alpha$  (Fig. 3) gives the slope  $-1.0516 E(\alpha)/R$  from which the  $E$  at particular  $\alpha$  for both the peaks have been evaluated and given in Tables I & II.

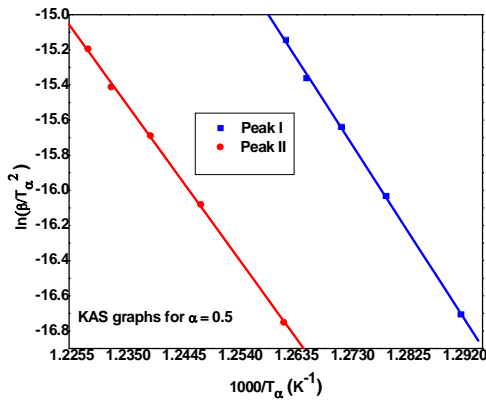


Fig.2 plot of  $\ln(\beta / T_\alpha^2)$  vs  $1000/T_\alpha$

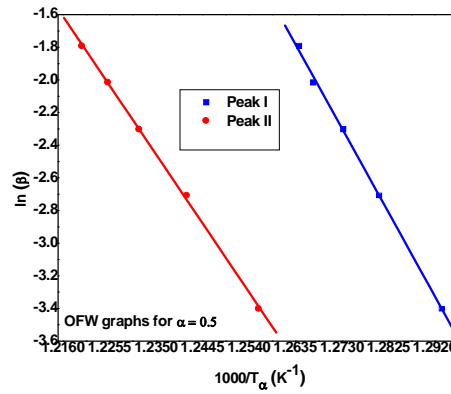


Fig. 3 plot of  $\ln \beta$  vs  $1000/T_\alpha$

**3. Li and Tang method**

According to this method the expression is

$$(4) \int_0^\alpha \ln \left( \frac{d\alpha}{dt} \right) d\alpha = G(\alpha) - \frac{E}{R} \int_0^\alpha \left( \frac{1}{T} \right) d\alpha$$

A plot of  $\int_0^\alpha \ln \left( \frac{d\alpha}{dt} \right) d\alpha$  vs  $\int_0^\alpha \left( \frac{1}{T} \right) d\alpha$

for a set of heating rates at constant conversion  $\alpha$  will have the slope  $-E/R$  (Fig.4, Tables I & II). Fig.4 shows the Li and Tang plot for peak I and II.

**B. Linear differential isoconversional method**

**Friedman method:** This method by Friedman is based on the expression

$$\ln \beta \frac{d\alpha}{dT} = \ln A + \ln f(\alpha) - \frac{E}{RT} \tag{5}$$

For a constant  $\alpha$ , the plot of  $\ln(d\alpha/dt)_\alpha$  vs  $1000/T_\alpha$  (Fig.5), obtained from thermograms. Fig 5 is a Friedman plot for peak I and II.

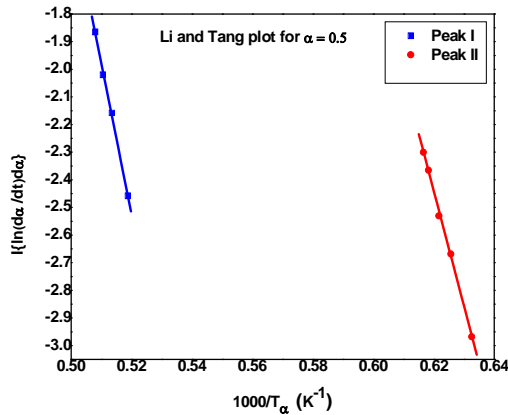


Fig.4 Plot of  $I\{\ln(da/dt)d\alpha\}$  vs  $I\{da/T\}$

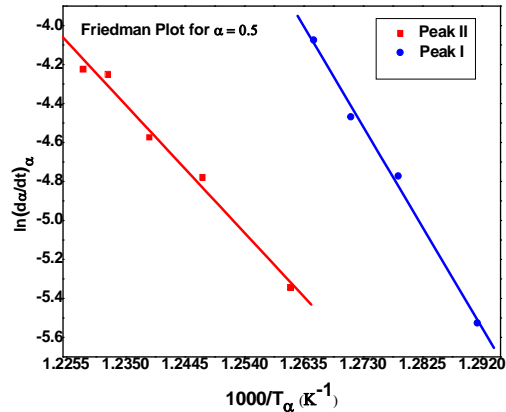


Fig.5 Plot of  $\ln(da/dt)_\alpha$  vs  $1000/T_\alpha$

**C. Non-linear integral isoconversional method**

An advanced isoconversional method has been described by Vyazovkin which is based on the assumption that the reaction model,  $f(\alpha)$  is independent of the  $\beta$ . So, for any two experiments conducted at different  $\beta$  the ratio of the temperature integral  $I(E, T_\alpha)$  to the  $\beta$  is a constant. For a given conversion and a set of  $n$  experiments performed under different heating rates, the activation energy can be determined at any particular value of  $\alpha$  by finding the value of  $E_\alpha$  for which the function

$$\sum_{i \neq j}^n \sum_{j=1}^n \frac{I(E_\alpha, T_{\alpha_i}) \beta_j}{I(E_\alpha, T_{\alpha_j}) \beta_i} \tag{6}$$

is a minimum. The minimization procedure is repeated for each value of  $\alpha$  to find the dependence of activation energy on the extent of conversion. Using this procedure we have processed our non-isothermal experimental data and the results are shown in Figs.6 and 7 recorded at several heating rates. The values of  $E$  at different  $\alpha$ , obtained using different methods are listed in Tables I & II for peaks I and II respectively. The dependence of activation energy  $E$  with fractional crystallization for the peaks I and II are shown in Figs.6 & 7.

Table I:  $E$  (kJ/mol) for peak I

$\alpha$	KAS	OFW	Li & Tang	Friedman	Vazovkin
0.1	407.4±1.3	399.9±1.3	415.7±1.7	397.4±1.2	405.2
0.2	404.2±1.1	396.7±1.1	394.1±1.2	350.9±0.8	398.4
0.3	398.2±1.0	391.2±1.0	374.1±0.9	320.9±1.3	387.4
0.4	392.4±0.8	385.7±0.8	358.3±0.8	301.0±1.8	377.4
0.5	385.8±0.7	379.3±0.7	345.0±1.0	286.8±2.3	367
0.6	379.1±0.5	373.0±0.5	333.4±1.1	270.2±2.4	357.3
0.7	372.1±0.5	366.7±0.5	324.2±1.3	269.4±2.5	347.7
0.8	364.3±0.5	358.8±0.5	315.9±1.5	266.0±2.4	337.6
0.9	355.8±0.6	350.9±0.6	310.9±1.6	274.4±2.3	328.6

$\alpha$	KAS	OFW	Li & Tang	Friedman	Vazovkin
0.1	434.0±2.6	424.4±2.6	439.0±3.1	492.2±2.2	369.0
0.2	438.1±2.0	429.1±2.0	446.5±0.6	474.7±2.5	398.0
0.3	438.1±1.5	429.1±1.5	453.1±0.8	458.1±2.8	405.6
0.4	436.5±1.1	427.6±1.1	453.9±1.3	449.8±3.0	407.2
0.5	434.0±0.8	425.2±0.8	453.1±1.6	451.5±3.0	407.0
0.6	431.5±0.6	422.8±0.6	452.3±1.8	451.5±3.0	407.1
0.7	430.7±0.6	421.2±0.6	453.1±2.0	459.8±3.0	408.2
0.8	429.8±0.9	421.2±0.9	454.8±2.1	482.2±3.5	411.2
0.9	434.0±1.5	424.4±1.5	--	523.8±6.7	420.3

Table II: E (kJ/mol) for peak II

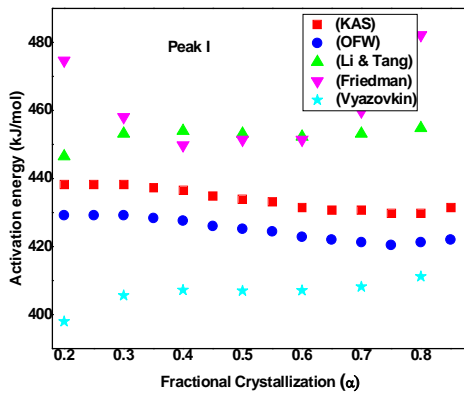


Fig.6. Dependence of E on  $\alpha$  for peak I

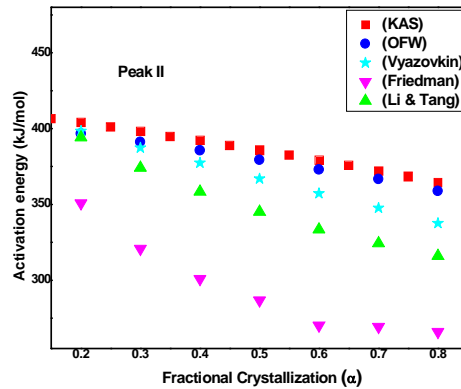


Fig.7. Dependence of E on  $\alpha$  for peak II

#### D. Model fitting methods

It is difficult to get an idea about the reaction model with the help of the model-free isoconversional methods which provide accurate estimates of E. Among the various model fitting methods, the CR and the IKP methods are used to examine the kinetics of non-isothermal crystallization in this metallic glass 2605S3A.

It is observed that the errors at  $\alpha$  range lower than 0.2 or higher than 0.80 tend to be rather high because the deflection of the DSC curve from the base line is very small at these ranges. Therefore, the kinetic study was restricted to the range  $0.20 \leq \alpha \leq 0.80$ .

#### 1.Coats & Redfern (CR) method

One of the most popular model fitting methods is the CR method which uses the expression

$$\ln \frac{g(\alpha)}{T^2} = \ln \left[ \frac{AR}{\beta E} \left( 1 - \frac{2RT}{E} \right) \right] - \frac{E}{RT} \cong \ln \frac{AR}{\beta E} - \frac{E}{RT} \quad (7)$$

where E and A are the Arrhenius kinetic parameters corresponding to a particular reaction model  $g(\alpha)$ . According to CR

the graph of  $\ln \frac{g(\alpha)}{T^2}$  vs  $\frac{1}{T}$  gives a straight line and from its slope and intercept, E and A are calculated for a particular reaction model  $g(\alpha)$ . The single heating rate non-isothermal data of amorphous 2605S3A alloy are fitted to various reaction models and repeated for four different heating rates, 2, 4, 6 and 8 K/min for peaks I and II and are given for 2 K/min in the Tables III& IV

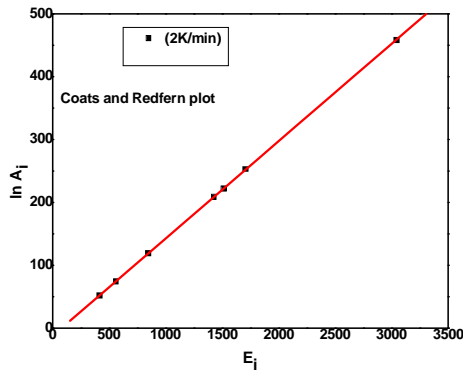


Fig.8. Plot of  $\ln A_i$  vs  $E_i$

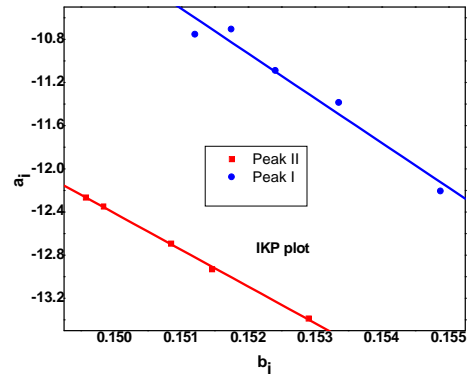


Fig.9. Plot of  $a_i$  vs  $b_i$

Table III: E and A, Coats–Redfern ( $0.20 < \alpha < 0.80$ ) **Peak I**  
**Bold font indicates the most suitable model**

Reaction models $g(\alpha)$	$\beta = 2$ K/min		
	E (kJ/mol)	$\ln A$	$ r $
$(1-(1-\alpha)^{1/3})$	1515.1±3	222.2±4	0.993
$(1-(1-\alpha)^{1/2})$	1426.3±3	208.7±4	0.991
$(1-(1-\alpha)^{1/3})^2$	3042.9±6	458.6±7	0.993
$(-\ln(1-\alpha))$	1707.1±2	253.2±3	0.996
<b><math>(-\ln(1-\alpha))^{1/4}</math></b>	<b>416.9±1</b>	<b>51.8±1</b>	<b>0.996</b>
$(-\ln(1-\alpha))^{1/3}$	560.4±1	74.4±1	0.996
$(-\ln(1-\alpha))^{1/2}$	846.4±1	119.2±2	0.996

Table IV: E and A, Coats–Redfern ( $0.20 < \alpha < 0.80$ ) **Peak II**  
**Bold font indicates the most suitable model**

Reaction models $g(\alpha)$	$\beta = 2$ K/min		
	E (kJ/mol)	$\ln A$	$ r $
$(1-(1-\alpha)^{1/3})$	1935.5±2.4	280.7±3.0	0.99687
$(-\ln(1-\alpha))$	2177.4±1.7	318.8±2.1	0.99880
$(-\ln(1-\alpha))^{1/4}$	534.6±0.4	68.4±0.5	0.99876
$(-\ln(1-\alpha))^{1/3}$	716.7±0.6	96.4±0.7	0.99877
$(-\ln(1-\alpha))^{1/2}$	1082.5±0.8	152.1±1.1	0.99879
$(-\ln(1-\alpha))^{1/5.5}$	384.9±0.3	45.4±0.4	0.99873
<b><math>(-\ln(1-\alpha))^{1/5}</math></b>	<b>424.8±0.3</b>	<b>51.5±0.4</b>	<b>0.99874</b>

It can be seen from these tables that the values of E's and A's strongly depend on the choice of the reaction model which is usually based on a statistical characteristic such as coefficient of linear correlation,  $r$ . The general practice in this method to determine E is to look for the model corresponding to maximum  $r$ . In some cases, the so-obtained value of E is significantly different from those obtained from other methods. For example, in the present case,  $r$  is maximum for the model  $g(\alpha) = -\ln(1-\alpha)$ . But the corresponding E is high compared to those obtained using different isoconversional methods. Thus, using statistical criterion it is difficult to say which model is the real one. For such cases an  $r$  value lower than  $r_{max}$  could correspond to the true kinetic model. The true analytical form of the conversion function can be determined from the E for a single  $\alpha = \alpha(T)$  curve that equals the E obtained by means of isoconversional methods. By applying this criterion for peak I of the analyzed data,  $g(\alpha) = (-\ln(1-\alpha))^{1/4}$  is found to be more suitable for the description of the solid state reaction and similarly for peak II, it is  $(-\ln(1-\alpha))^{1/5}$ .

## 2. The invariant kinetic parameter (IKP) method

The IKP method depends on the confirmation that the experimental curve  $\alpha = \alpha(T)$  in a thermally stimulated reaction process for a solid can be described correctly by several functions of fractional conversion  $f(\alpha)$ , and correlated through the compensation effect [16]. To apply this method, DSC at several heating rates  $\beta_i$ , are recorded and also different set of reaction models,  $g_i(\alpha)$ , are considered. For each  $\beta$  and particular  $g(\alpha)$ , using Coats and Redfern,  $\ln A$  and E are determined. Repeating this process for different  $\beta_i$  and  $g_i(\alpha)$ , pairs  $(A_{ij}, E_{ij})$ , are determined. These parameters are correlated through an apparent compensation effect equation

$$\ln A_i = a_i + b_i E_i \quad (8)$$

where  $a_i$  and  $b_i$  are constant parameters (compensation effect parameters). By plotting  $\ln A_i$  vs  $E_i$  for different  $\beta_i$ , the compensation parameters ( $a_i$  and  $b_i$ ) are determined. The straight lines  $\ln A_i$  vs  $E_i$  for several  $\beta$  must intersect in a point, which corresponds to the true values of A and E. These are called invariant activation parameters ( $A_{inv}$ ,  $E_{inv}$ ). Certain variation in the experimental conditions actually determine a region of intersection in the space  $\ln A$ , E. Because of this

reason the determination of the invariant activation parameters are determined by using the super correlation expression [17]

$$a_i = \ln A_i - b_i E_i \quad (9)$$

Thus, the plot of  $a_i$  vs  $b_i$  is a straight line and gives the invariant activation parameters. For different heating rates  $\beta$  and  $g(\alpha)$ ,  $\ln A_i$  and  $E_i$  are given in the Tables III & IV. These are plotted in Fig 8. The compensation effect parameters  $a_i$  and  $b_i$  are determined (Table V) from the slopes of these graphs and plotted in Fig.9 for both the peaks. These give invariant activation parameters for peak I,  $E = 414.8 \pm 47.1$  kJ/mol,  $A = 4.3 \times 10^{22}$  sec<sup>-1</sup> and for peak II,  $E = 340 \pm 6.4$  kJ/mol,  $A = 5.7 \times 10^{16}$  sec<sup>-1</sup>.

Table V: Compensation effect parameters  $a_i$  and  $b_i$  calculated from Table III for Peak I & Table IV for Peak II

$\beta$	Peak I			Peak II		
	$b_i \times 10^{-4}$	$a_i$	r	$b_i \times 10^{-4}$	$a_i$	r
2	0.155±2.7	-12.207±0.4	1	0.15 3±2	-13.387±0.1	1
4	0.1535±2.5	-11.386±0.4	1	0.1513±2	-12.932±0.1	1
6	0.1525±2.8	-11.090±0.5	1	0.151±3	-12.696±0.2	1
8	0.152±2.5	-10.707±0.5	1	0.150±1	-12.346±0.1	1
10	0.141±3.6	-10.754±0.5	1	0.150±2	-12.267±0.1	1

#### IV. CONCLUSION

The model free isoconversional methods proposed by KAS and OFW gives E which are dependent on  $\alpha$ . The values of E determined from KAS and OFW are very close to each other for both the peaks I and II and also fall near to the non-linear isoconversional method proposed by Vyazovkin and Wight. This trend is more pronounced for the peak II. The OFW method is less accurate because of the Doyle's approximation [18] than the KAS analysis. The E values remains almost constant with  $\alpha$  in the case of peak I, whereas continuously decreases for the peak II. Crystallization kinetics is a combined mechanism of nucleation and growth. The higher value of E in the initial stage for both the peaks indicates the dominant nature of nucleation and the decreasing trend of E with  $\alpha$  reveals the process of saturation of nucleation. That means the nucleation process takes place during the initial stage of transformation and negligible at the end. This is the reason why the isoconversional methods provide  $\alpha$  dependent E and clearly reveals the crystallization mechanism. The results of Li-Tang are deviating from consistency for both the peaks I and II in the entire range of  $\alpha$ . This is probably due to the absence of approximations involved in the derivation and shows clearly the dominant nature of nucleation. However, the values of E are not reliable at smaller values ( $\alpha < 0.2$ ) and at larger values ( $\alpha > 0.7$ ) due to the uncertainties in locating  $\alpha$ . The values of E from FR method clearly showed no systematic trend for peak I and II. This discrepancy may be due to the reason that the FR method uses the point values of the overall reaction rate while the integral isoconversional methods use the integrals, which describe the history of the system in the range 0.1 to 0.9.

The model fitting isokinetic methods, on the other hand gives single value of E. The CR method has been utilized to evaluate E and  $g(\alpha)$  and gave  $E = 441$  kJ/mol and  $g(\alpha) = (-\ln(1-\alpha))^{1/4}$  for peak I and  $E = 362.3$  kJ/mol and  $g(\alpha) = (-\ln(1-\alpha))^{1/5}$  for peak II. The IKP method is also used in conjunction with the CR method to determine E and A values for peak I and II and the E and A values to be  $\approx 415$  kJ/mol,  $4.3 \times 10^{22}$  sec<sup>-1</sup> and 340 kJ/mol,  $5.7 \times 10^{16}$  sec<sup>-1</sup>. The evaluated E values from CR and IKP methods are consistent with the values determined from isoconversional KAS and OFW methods and are 434 kJ/mol and 373 kJ/mol for peak I and II. Based on the studies of isoconversional and isokinetic methods it is evident that the isoconversional methods gave  $\alpha$  dependent E values which bears the signature of the crystallization mechanism. On the other side isokinetic methods gave single valued E and showed only one mechanism (reaction model) throughout the crystallization process for Fe-based metallic glass 2605S3A. The present investigation showed that, even though diverse approaches (isoconversional and isokinetic) are used, the results were consistent and complimentary to each other and the same trend is seen in other Co and Fe based metallic glasses [19, 20]

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