






Research Article

Cure Kinetics of a Carbon Fiber/Epoxy Prepreg by Dynamic Differential Scanning Calorimetry

Kaihua Chen ^{1,2,3} Xiaobao Zhu ^{1,2,3} Guozhen Zhao ^{1,2,3} Jing Chen ^{1,2,3}
and Shenghui Guo ^{1,2,3}

¹Faculty of Metallurgical and Energy Engineering, Kunming University of Science and Technology, Kunming, Yunnan 650093, China

²State Key Laboratory of Complex Nonferrous Metal Resources Clean Utilization, Kunming University of Science and Technology, Kunming, Yunnan 650093, China

³Key Laboratory of Unconventional Metallurgy, Ministry of Education, Kunming, Yunnan 650093, China

Correspondence should be addressed to Shenghui Guo; shguo78@hotmail.com

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Investigating the curing kinetics of a fiber prepreg system is beneficial to the controlling of prepreg laminate curing process. In the present work, a dicyandiamide (DICY)-cured carbon fiber/epoxy prepreg system was investigated by non-isothermal differential scanning calorimetry (DSC) at 2, 5, 10, and 20°C/min to attain the glass transition temperature for uncured prepreg and fully cured sample, which were estimated to be 7.6°C and 106.2°C, respectively. The activation energy (E_a) of the prepreg system was evaluated by Kissinger and Ozawa methods, and Friedman method was also employed to reveal the evolution of E_a as a function of curing degree. The kinetic parameters were determined by fitting the average E_a value obtained by Friedman method into Málek methodology, and the two parameters Šesták–Berggren model was found to best describe the curing kinetic of the prepreg system. The preexponential factor was calculated to be $6.0 \times 10^8 \text{ min}^{-1}$, with the overall reaction order at nearly 2.5. The prediction curves, based on Friedman method and autocatalytic model, were in good agreement with the experimental data.

1. Introduction

Epoxy resins have versatile utilization in modern society due to their attractive properties, such as excellent chemical and solvent resistance, high mechanical strength and modulus, good adhesion, and low shrinkage [1–3]. In the structural composite industry, epoxy resins are primarily applied as the matrix of fiber-reinforced polymer composites, and the majority of the composite parts are manufactured by using thermoset prepregs [4]. The prepreg is a kind of intermediate, wherein the fibers or fabrics are impregnated with a resin matrix, and the resins are in B-stage semi-cured state [5]. Curing the stacked prepreg laminate by using a predetermined cure cycle could yield composite parts, and the properties of the cured product are significantly influenced by the curing process [6]. Therefore, in order to sufficiently minimize the budget

throughout the curing process and develop a reasonable curing cycle, studying the curing kinetic would be a key factor for manufacturing high-performance epoxy composites.

The kinetic models employed to study cure reactions generally fall into two categories: mechanistic models and phenomenological models [7]. Although mechanistic models are based on detailed understanding of the chemical reaction throughout the curing process and generally have better predictions than phenomenological models, the mechanistic models are less popular than phenomenological ones since the complexity of the curing reactions. Furthermore, in most cases, the users are prohibited to examine the exact composition of commercial resin system [8]. On the contrary, the phenomenological models capture the main features of the reaction kinetics while ignoring the details of the reaction among each species. Additionally, the n th order model and

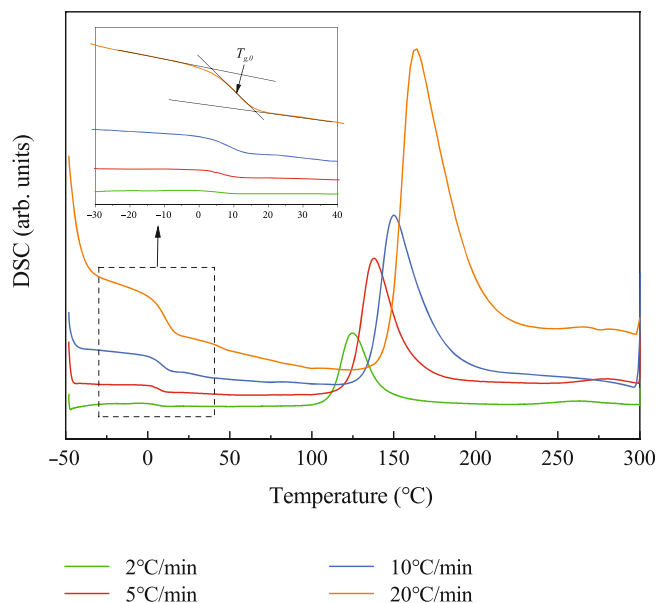


FIGURE 1: Dynamic DSC measurements at heating rates of 2, 5, 10, and 20 °C/min.

autocatalytic model are two kinds of iconic phenomenological models [9]. The n th order model assumes the maximum curing rate is obtained at the beginning of the curing process, while the autocatalytic model considers an initial da/dt of zero. When the resin system undergoes vitrification, the rate-controlling mechanism would transfer from chemical reaction to species diffusion, and the diffusion factor should be added into the kinetic model.

Numerous technologies have been proposed to characterize the curing kinetics of epoxy resin, such as dynamic mechanical analysis (DMA) [10], rheology [11], differential scanning calorimetry (DSC) [12], and dielectric analysis (DEA) [13, 14]. Among these technologies, the DSC analysis might be the most widely accepted means. In DSC measurements, two approaches, namely dynamic and isothermal scan, are commonly utilized to derive the cure kinetics [15], and the heat released during the exothermal reaction of a thermoset is assumed to be directly proportional to the rate of cure [16]. The dynamic DSC scan is measuring the variation of sample heat flow during a heating process in a predetermined temperature range, and the heating rate is normally fixed at a constant value. On the other hand, in the isothermal scanning, the sample is maintained at a constant temperature for various time intervals to obtain the variation of heat flow during each period. For both DSC methods, the heat of cure is achieved by integrating the total area of the heat flow curve with the construction of a baseline [17].

Currently, various papers have studied the curing kinetics of epoxy resins and thermoset prepregs by using DSC. For example, Hwang et al. [18] reported the cure kinetics of a vacuum-bag-only prepreg by using DSC and DMA, and both phenomenological and chemo-rheological models were utilized to predict the curing process. Jouyandeh et al. [19] used model-free integral Kissinger and differential Friedman isoconversional methods to derive the activation energy of epoxy/Fe₃O₄ nanocomposites from DSC data, and the kinetic model was

determined by Málek method. Kudisonga et al. [20] reported the curing kinetic of a commercially available preceramic polymer by deriving the kinetic parameters from DSC data, and they also built up a time-temperature transformation diagram for this system. Vafayan et al. [21] studied the curing reaction of epoxy prepreg by using advanced model-free, semi-model fitting, and model-fitting methods to fit the DSC data, and gave an optimal fitting by the advanced model-free method. Liang et al. [22] studied the curing kinetics of neat phenolic (PF) resin and its prepreg by dynamic DSC experiments, and they reported the lower E_a value of prepreg compared with neat resin since the fibers could improve the heat conduction.

In this study, the cure behaviour of a carbon fiber/epoxy prepreg was studied by using dynamic DSC scans. This prepreg was based on a dicyandiamide (DICY)-cured epoxy resin system. The activation energies of resin prepreg in dynamic cure condition were derived by fitting the data upon the Kissinger, Ozawa, and Friedman approaches. Moreover, the Málek methodologies were employed to determine the most appropriate kinetic model for the curing process, and the two parameters Šesták-Berggren model was found to best fit the DSC curves. All these fundamental understanding of the curing kinetic were supposed to be beneficial to the use of epoxy prepreg in large scale.

2. Materials and Methods

2.1. Materials. The materials employed in the present study were consistent with those used in [23], i.e., unidirectional carbon fiber/epoxy prepreg supplied by SINOPEC, Shanghai Research Institute of Petrochemical Technology Co., Ltd. The prepreg system was based on DICY-cured epoxy reinforced by T300 grade carbon fibers. The mass fraction of resin matrix was 40 wt%, and the thickness of uncured ply was 0.2 mm. The uncured prepreg was stored at -18°C before use. Unfortunately, the exact chemical composition of the resin system was not disclosed by the manufacturer.

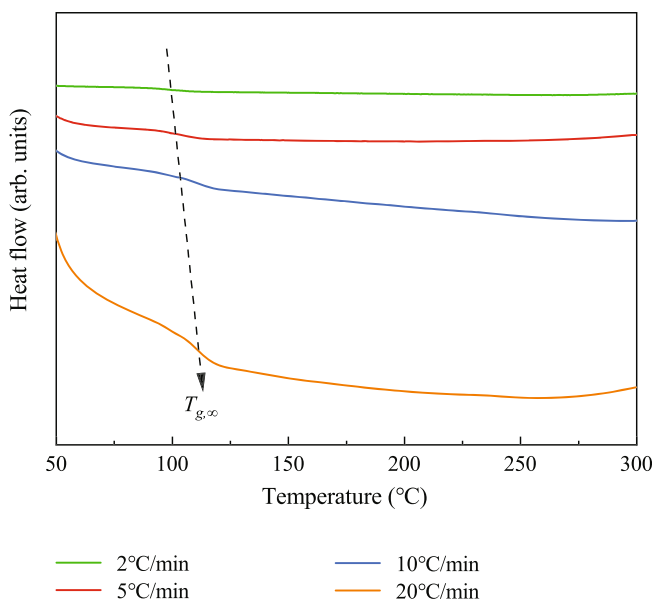
2.2. DSC Measurements. The DSC measurements were performed on a DSC 200F3 Netzsch, Germany. Samples were prepared by placing a small amount of uncured prepreg (9 ~ 10 mg) in an aluminium crucible equipped with a pierced lid, and all the experiments were carried out under a constant flow of nitrogen of 50 ml/min. A series of two-round dynamic DSC measurements were carried out to evaluate the curing behaviour of the prepreg system: the DSC cell was initially pre-cooled to -50°C , and, respectively, heated at a constant rate of 2, 5, 10, and 20 °C/min over a temperature range of -50 to 300°C . The temperature changed linearly with time, and the heating rate (β) could be expressed as follows:

$$\beta = \frac{dT}{dt} = \text{const}, \quad (1)$$

where T denotes the absolute temperature, and t represents the time (min). After the first round of DSC scan, the specimen was cooled down to 30°C at a cooling rate of $50^{\circ}\text{C}/\text{min}$. Finally, the second run was reheating the sample from 30°C to 300°C at the same heating rate as in the first run.

TABLE 1: The values of ΔH_T , $T_{g,0}$, and $T_{g,\infty}$ derived from dynamic ramps.

Heating rate β ($^{\circ}\text{C}/\text{min}$)	T_i ($^{\circ}\text{C}$)	T_p ($^{\circ}\text{C}$)	$T_{g,0}$ ($^{\circ}\text{C}$)	$T_{g,\infty}$ ($^{\circ}\text{C}$)	Total enthalpy ΔH_T (J/g)
2	75.4	124.6	5.4	99.6	107.1
5	82.5	138.0	6.5	104.5	88.33
10	105.0	150.0	7.8	109.3	66.66
20	126.0	164.0	10.1	111.5	66.90
Mean values	N/A	N/A	7.5	106.2	N/A

FIGURE 2: Second run indicating $T_{g,\infty}$ values for different heating rates (2, 5, 10, and 20 $^{\circ}\text{C}/\text{min}$).

3. Results and Discussion

3.1. Dynamic DSC Measurements. The dynamic DSC curves for uncured carbon fiber/epoxy prepreg in the temperature range of $-50 \sim 300^{\circ}\text{C}$ at heating rates of 2, 5, 10, and 20 $^{\circ}\text{C}/\text{min}$ are presented in Figure 1. The glass transition temperature of uncured prepreg ($T_{g,0}$) was taken as inflection point between extrapolation of the baseline to lower temperatures [17], and the results are shown in Table 1. For each dynamic curve, a single exothermic peak could be observed, and the peak temperature in the exotherm shifted to a higher value with the increasing heating rate. After the construction of baselines, the total heat of the reaction was relatively calculated via the integration of the heat flow curves for specified time limits, and it could be observed that the total heat exotherm was decreased with the increasing heating rate. This decreasing trend might be attributed to the reduced reaction time at higher heating rate, leading to a reduced heat exotherm [17]. Moreover, it has been reported that volatile by-products would be generated during the curing process, and some of the intermediates were capable of promoting the dissolution and reaction of DICY. The higher heating rate would result in the increased loss of volatile in open configuration [24],

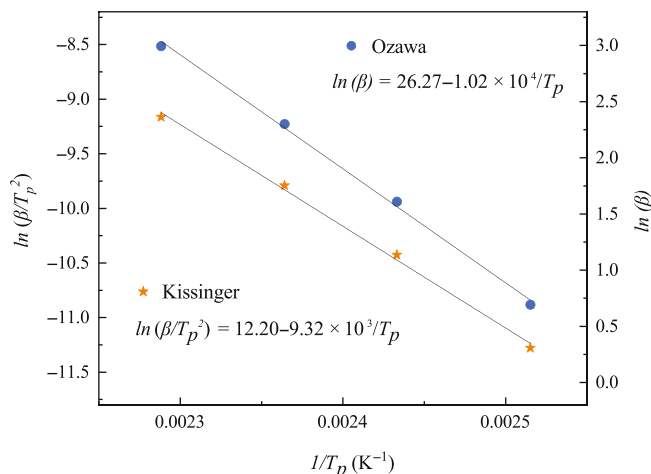


FIGURE 3: Determination of kinetic parameters by Kissinger and Ozawa methods.

leading to the decrease of the curing exotherm of DICY/epoxy system. In the present study, the sample was placed in aluminium pan with a pieced lid, which should be the same case as the published paper. Figure 2 demonstrates the second heating run of dynamic DSC scans of the prepreg sample. According to these results presented in Figure 2, complete polymerization of the resin matrix could be confirmed due to the absence of exothermal curing residue. In this case, the inflection points were taken as the glass transition temperature for the totally crosslinked polymer structure ($T_{g,\infty}$) obtained at various heating rates. Table 1 summarizes the characteristic parameters of the dynamic DSC scans, and it could be observed that the value of initial temperature and peak temperature increases from 75.4 $^{\circ}\text{C}$ to 126.0 $^{\circ}\text{C}$ and from 124.6 $^{\circ}\text{C}$ to 164.0 $^{\circ}\text{C}$, respectively.

3.2. Kissinger and Ozawa Methods. In the DSC measurements, the recorded heat evolution is assumed to be proportional to the extent of consumption of the reactive groups [25]. On the basis of this assumption, the degree of cure (α) and curing rate ($d\alpha/dt$) during the process could be described as follows:

$$\alpha = \frac{\Delta H(t)}{\Delta H_T}, \quad (2)$$

$$\frac{d\alpha}{dt} = \frac{(dH/dt)}{\Delta H_T}, \quad (3)$$

where $\Delta H(t)$ is the enthalpy of the reaction up to time t , and ΔH_T refers to the total enthalpy of the reaction at a certain heating rate.

The kinetic analysis of thermosetting resin is commonly described in the following equation:

$$\frac{d\alpha}{dt} = K(T)f(\alpha), \quad (4)$$

where $K(T)$ is the temperature-dependent reaction rate constant, and $f(\alpha)$ is the kinetic model determined by the cure

TABLE 2: Kinetic parameters obtained by Kissinger and Ozawa methods for carbon fiber/epoxy prepreg.

Method	E_a (kJ/mol)	A (min^{-1})
Kissinger method	77.49	1.85×10^9
Ozawa method	80.24	N/A

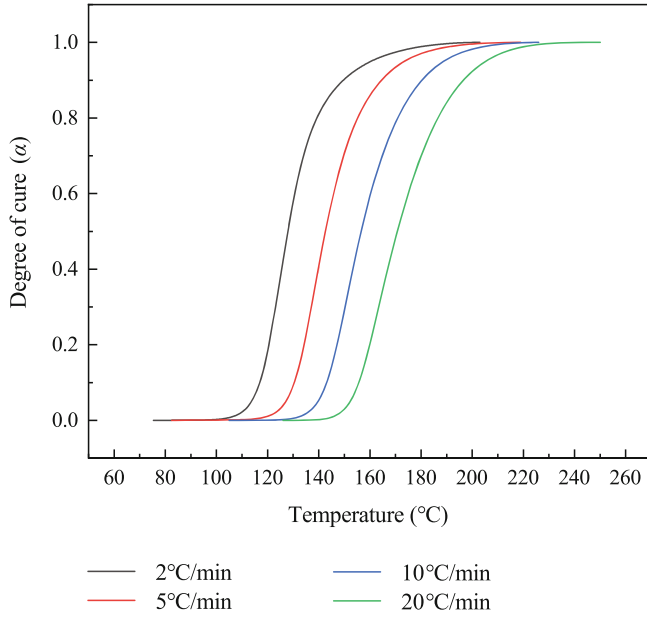


FIGURE 4: Conversion as a function of cure temperature for carbon fiber/epoxy prepreg at different heating rates.

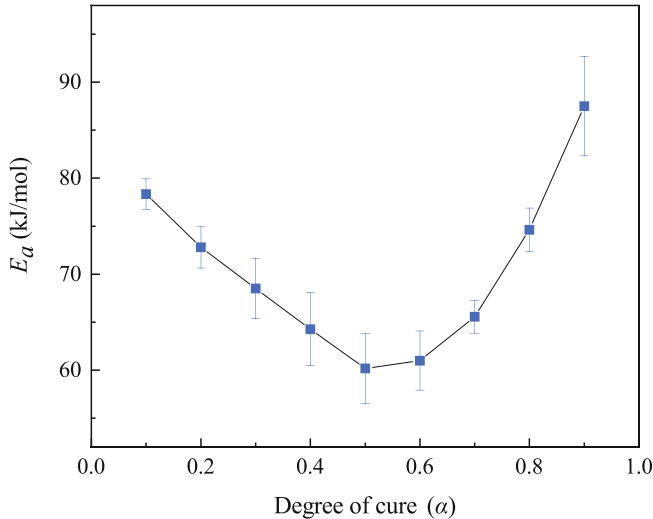


FIGURE 5: Variation of E_a versus conversion α for carbon fiber/epoxy prepreg.

mechanism. Taking the Arrhenius equation into account, the curing rate could be expressed as follows:

$$\frac{d\alpha}{dt} = A \exp\left(\frac{-E_a}{RT}\right) f(\alpha), \quad (5)$$

TABLE 3: The values of α_p^∞ , α_M , and α_p obtained from Malek model in terms of β .

Heating rate ($^{\circ}\text{C}/\text{min}$)	α_p^∞	α_M	α_p
2	0.26	0.26	0.36
5	0.35	0.24	0.33
10	0.32	0.23	0.29
20	0.32	0.20	0.32

where A and E_a are the preexponential factor and activation energy, respectively, and R is the universal gas constant ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$). Since the α , T , and t could be obtained from the experiments, the left two parameters A and E_a , as well as the kinetic model $f(\alpha)$ should be derived from the experimental data.

In order to estimate the activation energy and preexponential factor, the Kissinger and Ozawa methods were applied in this study. These two methods are typical model-free kinetics, which do not require prior knowledge about the reaction mechanism [20]. Moreover, both of these two methods assume the curing degree of the DSC exothermic peak is independent to the heating rate [26]. The Kissinger equation could be expressed as follows:

$$\ln\left(\frac{\beta}{T_p^2}\right) = \ln\left(\frac{AR}{E_a}\right) - \frac{E_a}{RT_p}, \quad (6)$$

where T_p is the peak exothermic temperature at a certain heating rate. The plots of $\ln(\beta/T_p^2)$ versus $1/T_p$ are shown in Figure 3, and the values of E_a and A could be obtained from the slope and intercept of the linear fitting curve, respectively.

On the other hand, Ozawa method is related to the logarithm of the heating rate and the inverse of the exothermic peak temperature:

$$\ln(\beta) = \ln\left(\frac{AE_a}{Rf(\alpha)}\right) - 5.331 - 1.052 \frac{E_a}{RT_p}. \quad (7)$$

The linear fit results of Ozawa method are also displayed in Figure 3. The calculated parameters (E_a and A) are listed in Table 2. The activation energies obtained from the Kissinger and Ozawa methods were 77.49 kJ/mol and 80.24 kJ/mol, respectively. However, it has been reported that Ozawa method is relatively inaccurate [27] and should not be used without performing an iterative correction procedure for the value of E_a [28].

3.3. Friedman Method. The isoconversional method of Friedman was also applied since this method could provide the activation energy as a function of conversion. For different heating rates, the Friedman method directly evaluates Equation (8) at a specific degree of cure α :

$$\ln \frac{d\alpha}{dt} = \ln [A_\alpha f(\alpha)] - \frac{E_a}{RT_\alpha}. \quad (8)$$

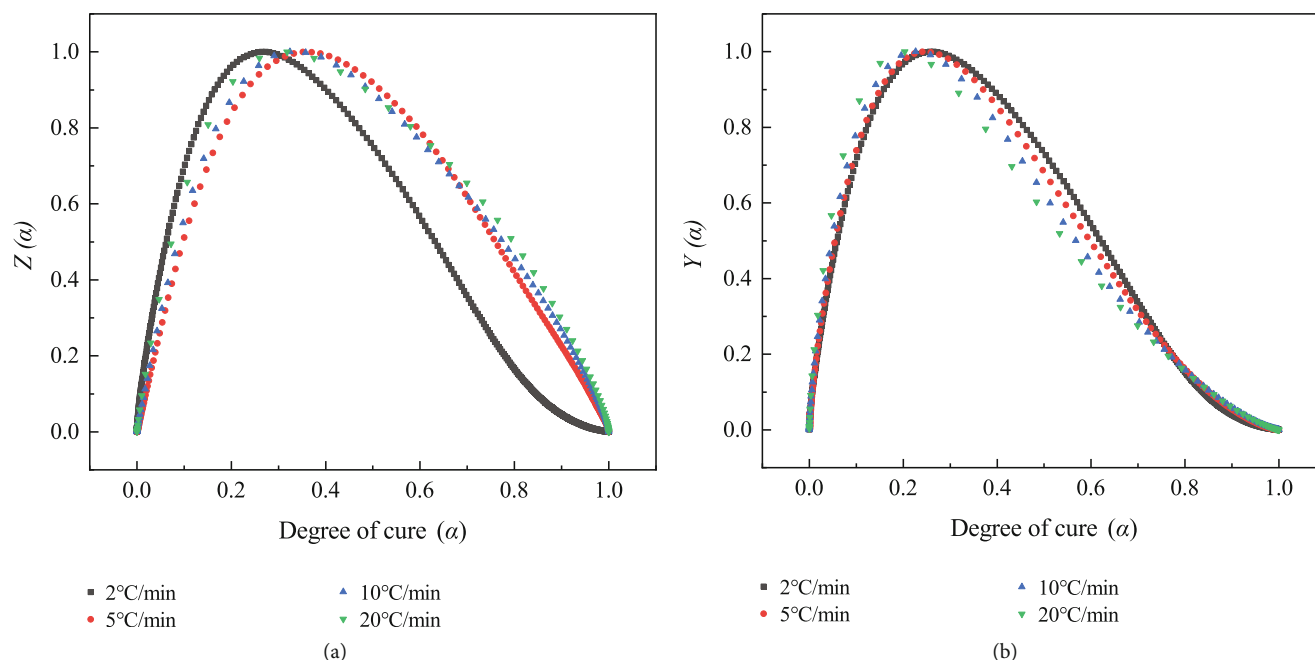


FIGURE 6: Variation of (a) $z(\alpha)$ and (b) $y(\alpha)$ function versus conversion.

The slope of $\ln(d\alpha/dt)$ versus $1/T_\alpha$ for the same value of α obtained at four heating rates could give the value of activation energy. Figure 4 presents the degree of cure as a function of temperature at various heating rates. After treating the isoconversional plots of $\ln(d\alpha/dt)_\alpha$ versus $1/T_\alpha$, the variation of E_a values with increasing conversion is presented in Figure 5. It could be observed that the E_a value initially decreased from 78.37 kJ/mol to 60.18 kJ/mol in the region of $\alpha = 0.1 \sim 0.5$, and then continuously increased after the α exceeded 0.5. Similar E_a variation was also reported in other thermosetting polymer curing kinetics [29, 30]. In this study, the initial descending trend of E_a could be related to the lowered viscosity induced by increasing temperature, which would facilitate the mobility of polymer chains. However, after the conversion exceeded 0.5, the molecular weight would significantly increase, and the movement of chains was restrained by the cross-linked structure. On the other hand, the sharp increase of E_a at the final stage of cure might be related to the transfer of reaction mechanism from chemical reaction control to diffusion control. The average value of E_a obtained by Friedman method was 70.3 kJ/mol, which was lower than that of the obtained by Kissinger method. However, the Kissinger method is based on the assumption that the curing degree of the DSC exothermic peak is independent to the heating rate [26], while the results summarized in Table 3 indicated that the curing degree at the exothermic peak obtained at four dynamic scans were different from each other. In this case, the assumption of Kissinger method was not satisfied in the present study. Therefore, the average E_a value obtained by using Friedman method (70.3 kJ/mol) was used in the following calculations.

3.4. Model-Fitting Method. Treating the experimental data by using isoconversional Friedman method could give the activation energy, and then the evaluated E_a could be utilized

to find the kinetic model, which best describes the curing process. In this study, the Málek method was performed to determine the kinetic model and preexponential factor by using these two special equations [31, 32]:

$$y(\alpha) = \left(\frac{d\alpha}{dt}\right) e^x, \quad (9)$$

$$z(\alpha) = \pi(x) \left(\frac{d\alpha}{dt}\right) \frac{T}{\beta}, \quad (10)$$

where x is the reduced activation energy (E_a/RT), β is the heating rate (K/min), T is absolute temperature (K), and $\pi(x)$ is the expression of the temperature integral. As was pointed out [33], $\pi(x)$ function could be well approximated by using the following expression proposed by Senum and Yang [34]:

$$\pi(x) = \frac{x^3 + 18x^2 + 88x + 96}{x^4 + 20x^3 + 120x^2 + 240x + 120}. \quad (11)$$

Figures 6(a) and 6(b) show the dependence of $y(\alpha)$ and $z(\alpha)$ values on the curing degree, respectively. For practical reasons, the values of both $y(\alpha)$ and $z(\alpha)$ were normalized between the (0, 1) intervals for various heating rates. Based on these results, the combination of the maximum values of $y(\alpha)$ and $z(\alpha)$, which were denoted as α_M and α_p^∞ , respectively, allowed the determination of the most suitable kinetic model [31]. The calculated values are summarized in Table 3, and it could be observed that the α_M values of various heating rates were within the range from 0 to α_p , where α_p denotes the conversion corresponding to the maximum on DSC curve. Moreover, all α_p^∞ values were lower than 0.632.

TABLE 4: The kinetic parameters evaluated for the curing of the tested prepreg.

Heating rate (°C/min)	E_a (kJ/mol)	A (min ⁻¹)	m	n
2			0.71	2.02
5			0.61	1.93
10	70.3	6.02×10^8	0.58	1.92
20			0.45	1.80
Mean			0.59	1.92

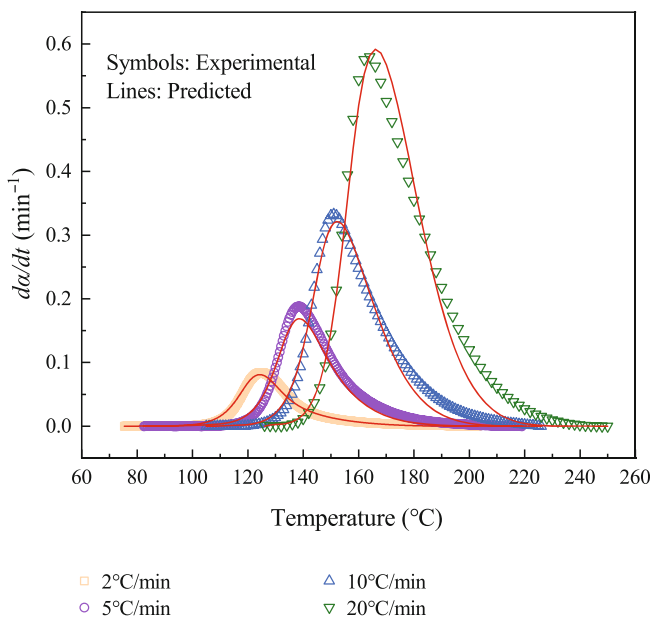


FIGURE 7: Comparison of experimental (symbols) and calculated (full lines) DSC curves.

These results suggested that the Šesták–Berggren (m , n) model would give a reasonable description of the cure kinetics of the carbon fiber/epoxy prepreg [19]:

$$f(\alpha) = \alpha^m(1 - \alpha)^n, \quad (12)$$

where m and n are the kinetic exponents. In this case, the reaction order n could be obtained by the slope of the linear dependence $\ln[(d\alpha/dt)e^x]$ versus $\ln[\alpha^p(1 - \alpha)]$, while $m = pn$, and $p = \alpha_M/(1 - \alpha_M)$ [35].

Additionally, the preexponential factor A could be calculated according to the following equation [36]:

$$A = - \left\{ \frac{\beta x_p}{T f'(\alpha_p)} \right\} \exp(x_p), \quad (13)$$

where $f'(\alpha_p)$ is the differential form of the kinetic model $[df(\alpha)/d\alpha]$ and p denotes the maximum of DSC curve.

Table 4 lists the kinetic parameters evaluated for the Šesták–Berggren kinetic model. The activation energy was the mean value of Friedman method (70.3 kJ/mol), and the preexponential factor was 6.02×10^8 . The average values of m

and n were 0.59 and 1.912, respectively. The kinetic model determined in this study was verified by comparing the curing rate obtained in the experiments (symbols) with the predicted values (lines) in Figure 7. It could be concluded that the two parameters Šesták–Berggren model reasonably well described the studied curing process.

4. Conclusions

The present work investigated the curing kinetics of a carbon fiber/epoxy prepreg system by using non-isothermal DSC scans. The two-round dynamic DSC scans at four heating rates indicated that the glass transition temperatures of uncured prepreg ($T_{g,0}$) and fully cured prepreg ($T_{g,\infty}$) were 7.6°C and 106.3°C, respectively. The activation energy (E_a) determined by Kissinger and Ozawa methods were 77.49 and 80.24 kJ/mol, respectively. The dependence of activation energy on the curing degree was revealed by the isoconversional Friedman method, which indicated that the E_a initially decreases until $\alpha = 0.5$ and then continuously increased in the following curing process. The two parameters Šesták–Berggren model was found to best describe the curing kinetics of the studied epoxy prepreg by using Málek method. The mean value of E_a obtained by Friedman method was utilized to calculate the kinetic parameters and build up the theoretically DSC curves. Finally, the predicted DSC curves showed good agreement with the experimental curves.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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