

Nonisothermal Crystallization Behavior of LLDPE/Glass Fiber Composite

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Received 26 June 2007; accepted 5 November 2007

DOI 10.1002/app.27707

Published online 8 April 2008 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The nonisothermal crystallization behavior of linear low-density polyethylene (LLDPE)/glass fiber (GF) composite was investigated by differential scanning calorimetry (DSC). It was observed that the crystallization temperature peak (T_p) of LLDPE composite containing 5.0 wt % GF (LLDPE/GF5) was higher than that of the pure LLDPE at various cooling rates. The half-time of crystallization ($t_{1/2}$) of LLDPE/GF5 composite was shortened under the effect of GF. The nonisothermal crystallization kinetics of LLDPE and LLDPE/GF5 composite were analyzed through the Avrami, Ozawa, and Mo equations. The results indicated that the data of the nonisothermal crystallization for LLDPE and LLDPE/GF5 composite calculated based on the Ozawa equation did not have the good linear relationship, but the nonisothermal crystallization behav-

iors of LLDPE and LLDPE/GF5 composite could be successfully described by the modified Avrami and Mo methods. The crystallization rate Z_c of the modified Avrami parameter of LLDPE/GF5 composite was higher than that of pure LLDPE at the same cooling rate. The Mo parameter $F(T)$ of LLDPE/GF5 composite was lower than that of LLDPE at the same degree of crystallinity. Through the Kissinger equation, the activation energies E_d of LLDPE and LLDPE/GF5 composite were evaluated, and their values were 312.3 and 251.2 kJ/mol, respectively. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 782–788, 2008

Key words: LLDPE; GF; composite; DSC; nonisothermal crystallization

INTRODUCTION

LLDPE as one of the important polyolefins has been extensively studied from both the industry and academic points of view, for its good processability, great recyclability, and preferable compatibility with other polyolefins.^{1,2}

It is well known that the blending of polymers with inorganic fillers is an important method to optimize the properties and the processability of the final products. The mechanical properties of LLDPE, such as the impact strength, Young's modulus, and tensile strength, could be improved by adding inorganic fillers, and also the fillers could affect the optical,^{3,4} thermal,^{5,6} electrical,^{7,8} and penetration^{9,10} properties of LLDPE. These changes could be achieved mainly by controlling the contents, sorts, and sizes of fillers.^{11–18} In addition, the interaction between the matrix and the fillers would have a remarkable effect on the mechanical properties of the composites.^{19–23}

For semicrystallization polymers, the crystallization behavior plays a key role in determining the properties of the material, such as mechanical, optical, and penetration properties. According to the studies on the crystallization behavior of polymer-containing fillers,^{24–28} the inorganic fillers may influence the crystallinity, crystallization temperature, crystallization rate, crystal morphology, and the spherulite sizes of polymers, and many of researches have been published to explore the mechanism of the effect of fillers on the crystallization of polymers.^{27,28} Glass fibers (GF), as one of the very important inorganic fillers, have been effectively used to improve the properties of the polymers,^{29–31} and the effect of GF on the crystallization behavior of HDPE has been reported. Zhang^{32,33} found that the strain-induced crystallization was formed in the interface between the GF and the matrix, and the concentration of the GF, the interfacial bonding strength, and the cooling rate of samples in injection molding had the important effect on the crystallization behavior of HDPE. The crystallization of HDPE/GF was a process of heterogeneous nucleation, and the Avrami exponent did not change with the cooling rate.^{34,35} The structure of LLDPE contains some branch chains, which is different from that of HDPE, and the addition of GF into LLDPE materials would also influence both the mechanical properties and the

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Contract grant sponsor: National Basic Research Program of China; contract grant number: 2005BC623800.

crystallization behavior of LLDPE. To make better use of GF reinforced LLDPE, it is necessary to investigate the effect of GF on the crystallization behavior of LLDPE. However, as far as we have known up to now, there was a lack of systematic studies on the effect of GF on the crystallization behavior of LLDPE.^{36–41} In this work, we focused on how the GF influenced the nonisothermal crystallization behavior of LLDPE and analyzed the nonisothermal crystallization kinetics of LLDPE/GF composite. In the study of crystallization kinetics of polymers, isothermal and nonisothermal kinetic analyses are generally used to study the crystallization behavior of crystalline polymers. The isothermal crystallization kinetics, however, is often limited to idealized conditions, in which the parameters of state (temperature, pressure, etc.) are constant. In real practical situations, the conditions of crystallization change continuously, which makes the kinetics of the crystallization rely on the instantaneous state conditions as well as on the rate of the condition change. Also, the study of nonisothermal crystallization in polymer composites is of greater practical significance because the polymer processing at industrial scale is normally under the nonisothermal conditions. In this article, the modified Avrami, Ozawa, and Mo equations^{42–45} were used to investigate the nonisothermal crystallization kinetics of LLDPE and LLDPE/GF composite, and the activation energy of LLDPE and LLDPE/GF composite were calculated through Kissinger equation⁴⁶ to explore the reason of the effect of GF on the crystallization behavior of LLDPE.

EXPERIMENTAL

Materials

LLDPE, a commercial grade of copolymer (DFDA-7042) with a MFI of 1.9 g/10 min was bought from Jilin Petrolic, China. It was a copolymer of ethylene and butylene with a comonomer concentration of 7.6%. Its weight-average molecular weight (M_w) and number-average molecular weight (M_n) were 2.38×10^5 and 6.10×10^4 , respectively. Its melting temperature (T_m) was 124°C tested through a differential scanning calorimetry (DSC204, NETZSCH, Germany).

GF (ESC-28-06) was provided by the Fiber Institute of Nanjing, Nanjing, China, with a length/diameter ratio of 3 mm/0.005 mm and without any of surface treatment by coatings in this work.

Samples preparation

LLDPE/GF composites were prepared in a parallel corotating twin-screw extruder with a length/diameter ratio of 32, and a screw diameter of 25 mm

(TSSJ-25/32, provided by the Research Institute of Plastic Machine of Chenguang, Chengdu, China). The composites were extruded at 165°C with 110 rpm. The sample of LLDPE containing 5.0 wt % GF was represented by LLDPE/GF5.

Differential scanning calorimetry

The crystallization behavior of LLDPE composites was investigated by DSC. Each of samples was first held at 423 K for 5 min to erase all previous thermal history. Then the melt was cooled to room temperature to nonisothermally crystallize at the cooling rate of 2.5, 5, 10, and 20 K/min, respectively. All measurements were carried out in nitrogen containing atmosphere.

RESULTS AND DISCUSSION

The effects of GF on the crystallization behavior of LLDPE were carried out through the DSC instrument and the nonisothermal crystallization curves of LLDPE and LLDPE/GF5 composite at various cooling rates were shown in Figure 1. The crystallization temperature T_{onset} and T_p (which were defined as the initial crystallization temperature and the peak maximum temperature and they could be obtained from the DSC data of crystallization curves, respectively) of pure LLDPE and LLDPE/GF5 composite were determined and listed in Table I.

It was observed that both T_{onset} and T_p of samples decreased greatly with the increase of cooling rate. The T_p of LLDPE and LLDPE/GF5 composite decreased about 9 and 10 K, respectively, when the cooling rate increased from 2.5 to 20 K/min. But at a given cooling rate, for example, at the cooling rate of 5 K/min, the T_p value of LLDPE/GF5 composite was 2–3 K higher than that of LLDPE. It has been reported that many inorganic fillers,^{24–28} such as montmorillonite, calcium carbonate, titania, and glass bead, could increase the crystallization temperatures of polymers due to the heterogeneous nucleation effect of inorganic fillers,⁴⁷ and the GF might play the same role as those fillers do. Furthermore, Kim et al.²⁵ found that the heterogeneous nucleation effect was related to the crystallization active energy of polymers. The addition of fillers could decrease the crystallization active energy of polymers, which would make the crystallization process occur at higher temperature. Therefore, to confirm this interpretation, the crystallization active energies of LLDPE and LLDPE/GF5 composite should be considered, and they would be calculated in the discussion later.

To quantitatively discuss the relationship between crystallinity and temperature, an important param-

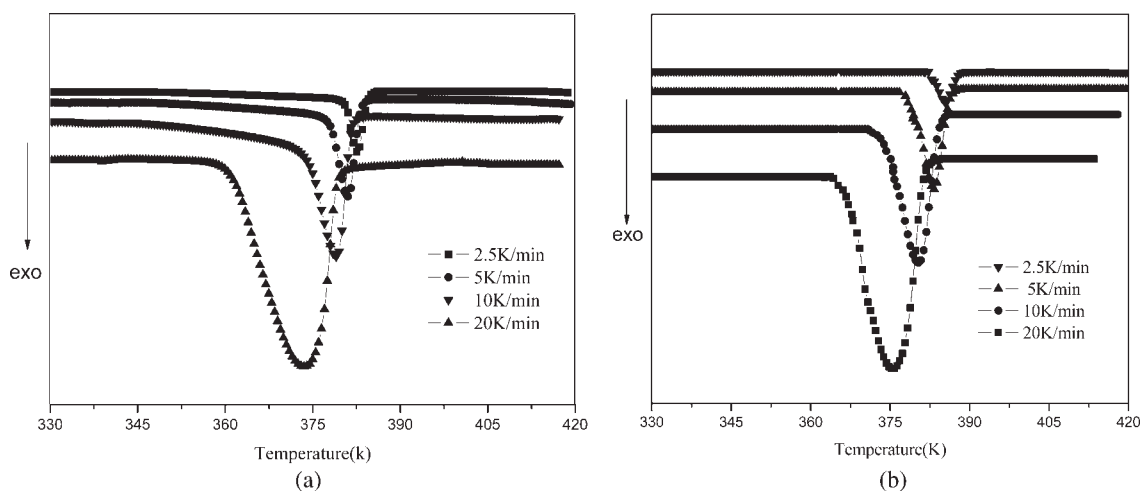


Figure 1 DSC thermograms of nonisothermal crystallization at various cooling rates for (a) LLDPE and (b) LLDPE/GF5 composite.

ter of the relative degree of crystallization X_t ⁴² should be introduced:

$$X_t = \int_{T_0}^T (dH/dT)dT / \int_{T_0}^{T_\infty} (dH/dT)dT \quad (1)$$

where T_0 is the onset of crystallization temperature, T_∞ is the end of crystallization temperature, and dH is the enthalpy of crystallization released during an infinitesimal temperature range dT .

The relationship between X_t and crystallization temperatures (Fig. 2) could be obtained from the data of Figure 1. When compared with Figure 1, the crystallization curves of Figure 2 indicated that the crystallization temperature decreased with the increase in the cooling rates. The crystallization temperatures of LLDPE/GF5 were higher than those of LLDPE, and these conclusions were consistent with the results of Figure 1.

Through the equation $t = (T_{\text{onset}} - T)/\Phi$,⁴⁸ the relationship between crystallinity and temperature could be transformed into the relationship between crystallinity and time, and the results were presented in Figure 3. From Figure 3, the half-time of crystallization $t_{1/2}$ was obtained, which was defined as the time from the onset of the crystallization to the time at which X_t was 50%, and the data were listed in Table I. The $t_{1/2}$ could be used as the characterization of crystallization rate, that the smaller the value of $t_{1/2}$ is, the more rapid crystallization rate the polymer has. As could be seen from Table I, the values of $t_{1/2}$ decreased with the increase of cooling rates of the LLDPE and the LLDPE/GF5 composite. At the cooling rates of 2.5 and 20 K/min, the values of $t_{1/2}$ of LLDPE were 1.11 and 0.41 min, respectively, indicating that the crystallization rate of the former was slower than that of the latter. Fur-

thermore, the value of $t_{1/2}$ of LLDPE/GF5 composite was smaller than that of pure LLDPE at a given cooling rate, which presented once again that the addition of GF increased the crystallization rate of LLDPE. The research on the effect of reinforced fibers on the crystallization of polypropylene by Manchado et al.⁴⁹ also indicated the similar results. The fibers acted as heterogeneous nucleating agents to facilitate crystallization, and the $t_{1/2}$ of composite was shortened under the effect of fibers. In addition, Misra and coworkers⁵⁰ found that the $t_{1/2}$ was not only related to the addition of inorganic fillers, but also the contents and particle sizes of fillers.

To investigate the effect of GF on the nonisothermal crystallization of LLDPE, the crystallization kinetic equation of the Avrami equation⁴² could be rewritten through the logarithmic transformation as follows:

$$\ln[-\ln(1 - X_t)] = \ln Z_t + n \ln t \quad (2)$$

TABLE I
Values of the ΔH_c , T_{onset} , T_p , and $t_{1/2}$ for Pure LLDPE and LLDPE/GF5 Composite at Various Cooling Rates

	Φ (K/min)	T_{onset} (K)	T_p (K)	ΔH_c (J/g)	$t_{1/2}$ (min)
LLDPE	2.5	384.51	382.62	93.81	1.11
	5	383.09	381.02	91.93	0.73
	10	381.49	379.01	90.64	0.54
	20	379.11	373.59	90.37	0.41
LLDPE/GF5	2.5	387.84	385.43	89.73	0.84
	5	386.15	383.23	89.22	0.69
	10	384.05	380.51	88.21	0.49
	20	381.24	375.44	87.93	0.36

Φ , cooling rates (K/min); T_{onset} , the initial crystallization temperature (K); T_p , the peak maximum temperature (K); ΔH_c , the crystallization heat enthalpy (J/g); and $t_{1/2}$, half-time of crystallization (min).

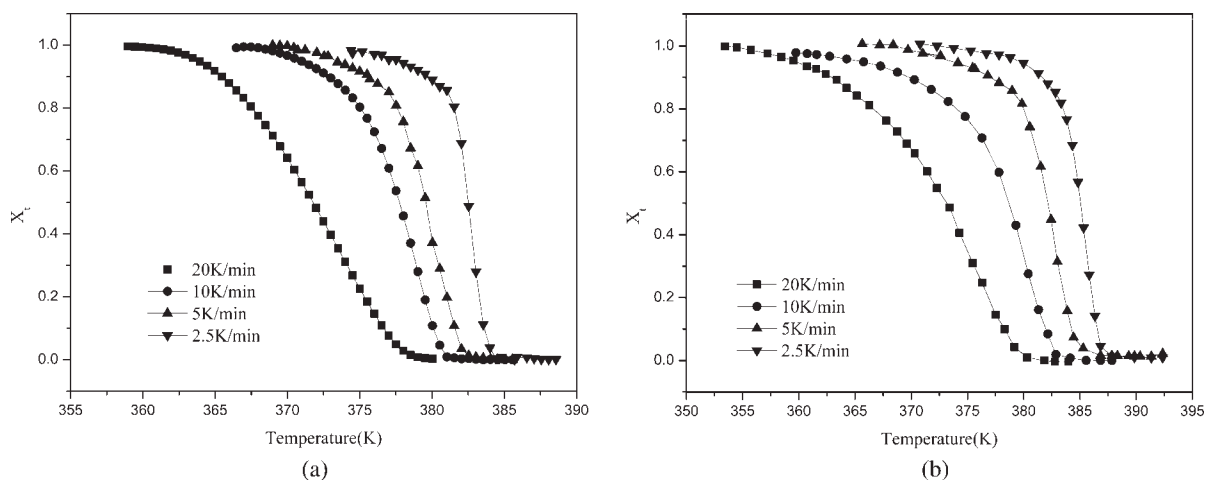


Figure 2 Plots of X_t versus temperature of crystallization for (a) LLDPE and (b) LLDPE/GF5 composite at various cooling rates.

where X_t is the crystallization fraction in the crystallization material at time t , Z_t is the crystallization rate constant and is temperature dependent, and n is the Avrami index and is related to the information of nucleation and growth geometry of polymers.

By plotting $\ln[-\ln(1 - X_t)]$ versus $\ln t$ at different cooling rates, a series of curves were obtained in Figure 4. From the interception and the slope of the curve linear part, the Avrami parameters Z_t and n were obtained and listed in Table II.

It was found that the data for both of LLDPE and LLDPE/GF5 composite did not have the good linear relationship between $\ln[-\ln(1 - X_t)]$ and $\ln t$ with the increase of crystallization time. Obviously, the extent of deviated linearity of the LLDPE/GF5 composite was larger than that of the LLDPE and this might be attributed to the addition of GF, which greatly influenced the growth rate of the spherulites at the end of crystallization.

To obtain a more accurate crystallization rate in the nonisothermal crystallization, the effect of the cooling rate should be taken into account. This method was proposed by Jeziorny⁴³ and also called as a modified Avrami equation:

$$\ln Z_c = \ln Z_t / \Phi \quad (3)$$

where Z_t is the crystallization rate constant in the Avrami equation and Z_c is the modified crystallization rate constant in the Jeziorny equation.

The Jeziorny parameters of LLDPE and LLDPE/GF5 composite were listed in Table II, and the Z_c could be applied to characterize the crystallization rate under the condition of nonisothermal crystallization. From Table II, values of Z_c varied from 0.49 to 1.07 for LLDPE and from 0.78 to 1.08 for LLDPE/GF5 composite, respectively, at the cooling rate region from 2.5 to 20 K/min. These results displayed

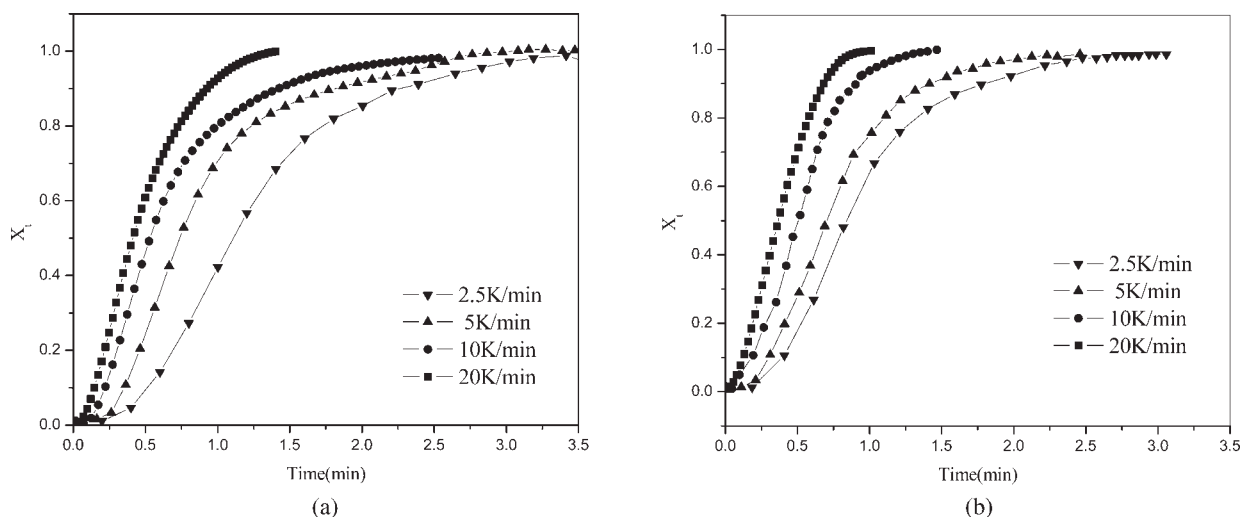


Figure 3 Plots of X_t versus t of crystallization for (a) LLDPE and (b) LLDPE/GF5 composite at various cooling rates.

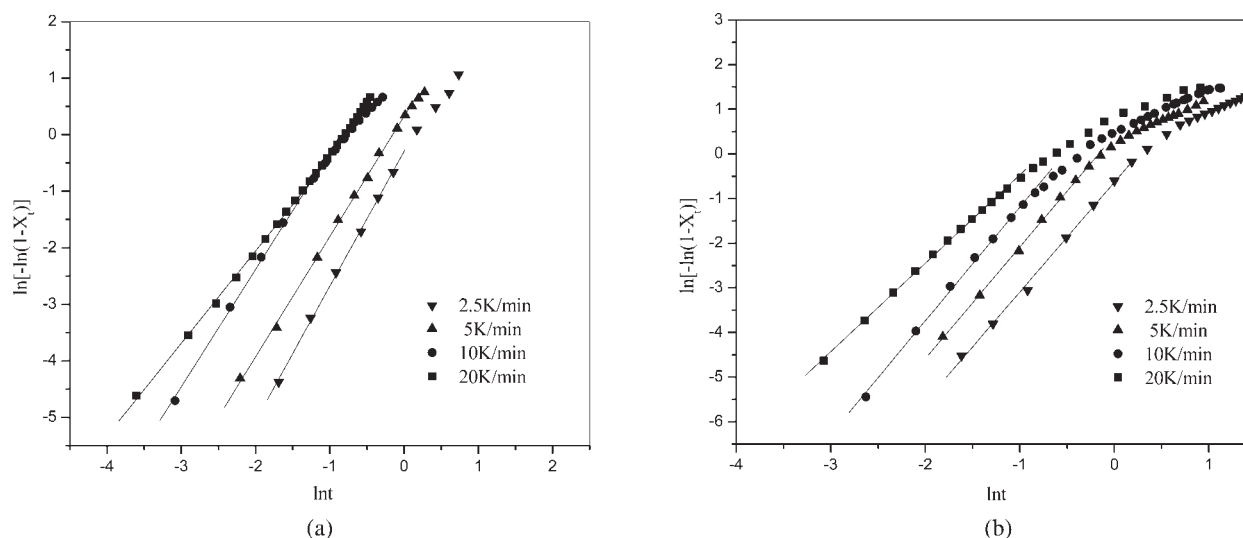


Figure 4 Plots of $\ln[-\ln(1 - X_t)]$ versus $\ln t$ by Avrami equation for (a) LLDPE and (b) LLDPE/GF5 composite at various cooling rates.

that the crystallization rate increased with the increase in the cooling rate. On the other hand, at a given cooling rate, the Z_c of LLDPE/GF5 composite was larger than that of LLDPE, indicating that the crystallization rate of LLDPE/GF5 was more rapid than that of LLDPE.

Ozawa⁴⁴ had also put forward an equation to analyze the nonisothermal crystallization.

$$1 - X(T) = \exp[-P(T)/\Phi^m] \quad (4)$$

where $P(T)$ is the function of cooling rate, Φ is the cooling rate, and m is the Ozawa exponent depending on the dimension of crystal growth.

The logarithmic form of the eq. (4) could be written as follows:

$$\ln[-\ln(1 - X(T))] = \ln P(T) - m \ln \Phi \quad (5)$$

The results from eq. (5) were presented in Figure 5. The results indicated that the data of the nonisothermal crystallization for LLDPE and LLDPE/GF5 composite based on the Ozawa equation did not have the good linear relationship. Although Ozawa method had been successfully applied to describe the nonisothermal crystallization of some polymers,⁴⁸ such as poly (trimethylene terephthalate), nylon 6, and poly (*p*-phenylene sulfide), it failed to describe the nonisothermal crystallization of LLDPE and LLDPE/GF5 composite. More information about the nonisothermal crystallization could not be obtained from Ozawa plots due to its poor linear relationship between $\ln[-\ln(1 - X_t)]$ and $\ln \Phi$.

For better describing the nonisothermal crystallization kinetics of LLDPE and LLDPE/GF5 composite, Mo method⁴⁵ was applied, which was proposed by

Mo and his coworkers, through combining the Avrami and Ozawa equations:

$$\ln \Phi = \ln F(T) - \alpha \ln t \quad (6)$$

where $\alpha = n/m$ is the ratio of the Avrami exponent n to the Ozawa exponent m and $F(T) = [K(T)/k]^{1/m}$, which refers to the cooling rate selected at a certain crystallization and mainly reflects the crystallization rate of polymers: the larger the $F(T)$ value, the slower the crystallization rate of polymer.

According to eq. (6), at a given degree of crystallinity, a series of plots of $\ln \Phi$ versus $\ln t$ were obtained in Figure 6, and there was a good linear relationship between $\ln \Phi$ and $\ln t$. In other words, the Mo method successfully described the nonisothermal crystallization process of LLDPE and LLDPE/GF5 composite. Thus, from the intercept and slope of the lines, the kinetic parameter $F(T)$ and α were obtained and listed in Table III.

From Table III, the values of α for pure LLDPE varied from 1.73 to 2.42, and for LLDPE/GF5 composite varied from 1.99 to 2.51. The value of $F(T)$

TABLE II
Parameters of Samples During Nonisothermal Crystallization Process

	Φ (K/min)	n	Z_t	Z_c
LLDPE	2.5	2.59	0.17	0.49
	5	2.08	1.35	1.06
	10	1.92	3.32	1.13
	20	1.74	4.22	1.07
LLDPE/GF5	2.5	2.46	0.53	0.78
	5	2.50	1.51	1.09
	10	2.55	3.87	1.15
	20	1.98	4.55	1.08

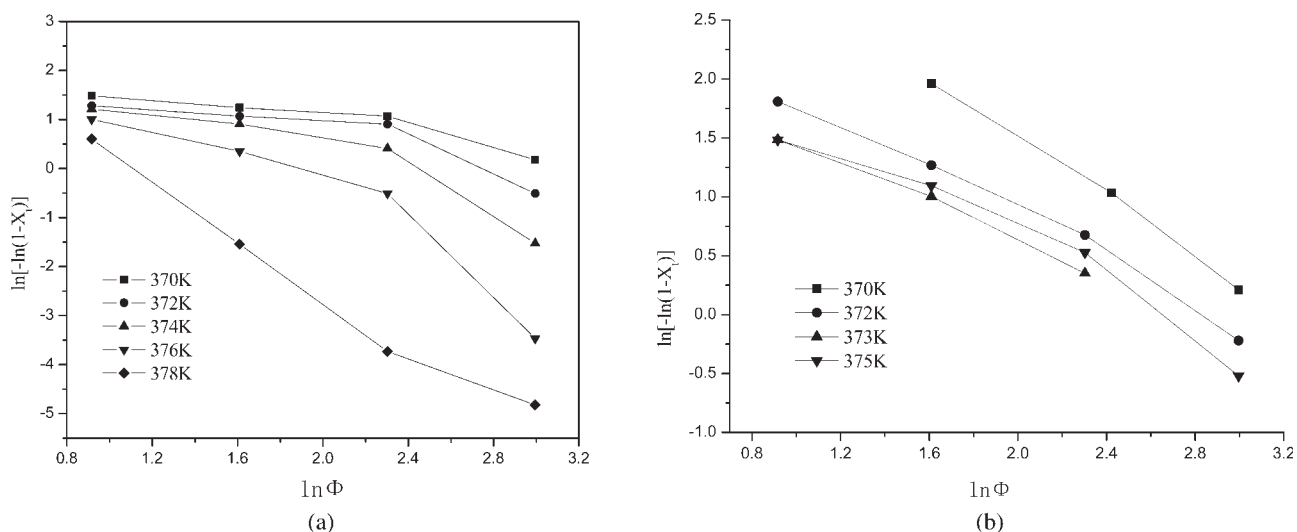


Figure 5 Relationship between $\ln \Phi$ and $\ln[-\ln(1 - X_i)]$ calculated by Ozawa equation for (a) LLDPE and (b) LLDPE/GF5 composite.

increased with the increase of the relative crystallinity for both pure LLDPE and LLDPE/GF5 composite. Furthermore, the $F(T)$ values of LLDPE/GF5 composite were smaller than those of pure LLDPE. These implied that the crystallization kinetics of LLDPE/GF5 composite was faster than that of pure LLDPE, which agreed with our previous observation.

To account for the reason of the essential effect of the GF on the crystallization process of LLDPE, the crystallization activation energy E_d should be calculated. According to the theory of crystallization,⁴⁷ the crystallization process of polymers is controlled by the dynamic and static factors. The dynamic factor is related to the E_d for the transport of crystalline units across the phase, and the static factor is related to the free energy barrier for nucleation.

And through the Kissinger equation,⁴² the E_d of LLDPE and LLDPE/GF5 composite could be easily calculated:

$$\frac{d\left(\ln\frac{\Phi}{T_p}\right)}{d\left(\frac{1}{T_p}\right)} = -\frac{E_d}{R} \quad (7)$$

where T_p , R , and Φ are the peak temperature, universal gas constant, and cooling rate, respectively. The activation energy E_d can be determined from the slope of $\ln\frac{\Phi}{T_p}$ versus $\frac{1}{T_p}$, and they were listed in Table III. The results indicated that the activation energy of nonisothermal melt crystallization of pure LLDPE and LLDPE/GF5 composite were 312.3 and 251.2 KJ/mol, respectively, which meant that the addition of GF had a significant influence on E_d of LLDPE. In

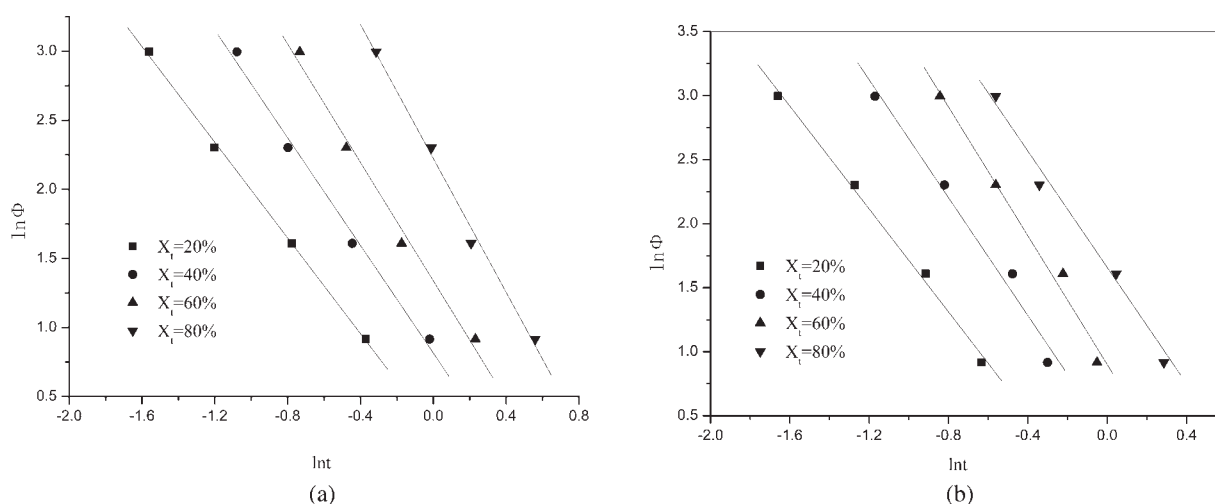


Figure 6 Plots of $\ln \Phi$ versus $\ln t$ by Mo equation for (a) LLDPE and (b) LLDPE/GF5 composite.

TABLE III
Nonisothermal Crystallization Kinetic Parameters by
Mo Equation for LLDPE and LLDPE/GF5 at Various
Relative Crystallinity

X_t (%)	20	40	60	80
LLDPE				
α	1.73	1.95	2.10	2.42
$F(T)$	1.30	2.25	3.79	9.18
ΔE (KJ/mol)		312.3		
LLDPE/GF5				
α	1.99	2.30	2.51	2.27
$F(T)$	0.74	1.43	2.45	5.22
ΔE (KJ/mol)		251.2		

other words, the occurrence of crystallization for LLDPE/GF5 composite was more easily than that of pure LLDPE due to the lower E_d caused by the addition of GF fillers.

CONCLUSIONS

The crystallization temperature peak (T_p) of LLDPE composite containing 5.0 wt % GF (LLDPE/GF5) was higher than that of the pure LLDPE at various cooling rates. The half-time of crystallization ($t_{1/2}$) of LLDPE/GF5 composite was shortened under the effect of GF. The nonisothermal crystallization kinetics of LLDPE and LLDPE/GF5 composite was analyzed through the Avrami, Ozawa, and Mo equations. The results indicated that the data of the nonisothermal crystallization for LLDPE and LLDPE/GF5 composite calculated based on the Ozawa equation did not have the good linear relationship, but the nonisothermal crystallization behaviors of LLDPE and LLDPE/GF5 composite could be successfully described by the modified Avrami and Mo methods. The crystallization rate Z_c of the modified Avrami parameter of LLDPE/GF5 composite was higher than that of pure LLDPE at the same cooling rate. The Mo parameter $F(T)$ of LLDPE/GF5 composite was lower than that of LLDPE at the same degree of crystallinity. Through the Kissinger equation, the activation energies E_d of LLDPE and LLDPE/GF5 composite were evaluated, and their values were 312.3 kJ/mol and 251.2 kJ/mol, respectively.

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