Research Article

High-Density Polyethylene and Heat-Treated Bamboo Fiber Composites: Nonisothermal Crystallization Properties

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The effect of heat-treated bamboo fibers (BFs) on nonisothermal crystallization of high-density polyethylene (HDPE) was investigated using differential scanning calorimetry under nitrogen. The Avrami-Jeziorny model was used to fit the measured crystallization data of the HDPE/BF composites and to obtain the model parameters for the crystallization process. The heat flow curves of neat HDPE and HDPE/heat-treated BF composites showed similar trends. Their crystallization mostly occurred within a temperature range between 379 K and 399 K, where HDPE turned from the liquid phase into the crystalline phase. Values of the Avrami exponent (n) were in the range of 2.8~3.38. Lamellae of neat HDPE and their composites grew in a three-dimensional manner, which increased with increased heat-treatment temperature and could be attributed to the improved ability of heterogeneous nucleation and crystallization completeness. The values of the modified kinetic rate constant (K_J) first increased and then decreased with increased cooling rate because the supercooling was improved by the increased number of nucleating sites. Heat-treated BF and/or a coupling agent could act as a nucleator for the crystallization of HDPE.

1. Introduction

Heat treatment of bamboo material under oil, steam, or oxygen-deficient conditions presents a physical modification technique for altering properties of bamboo fibers (BFs) such as strength and durability. Heat-treated BFs have been shown to have somewhat reduced mechanical properties, unchanged shrinkage, and a lowered equilibrium moisture content [1, 2]. As heat-treatment temperature increased, the relative contents of hemicellulose and cellulose decreased and lignin content increased in the heat-treated bamboo material [2, 3]. Heat treatment was also shown to lead to better color stability compared with untreated bamboo [4, 5]. With respect to composites, high-density polyethylene (HDPE)/heat-treated BF composites showed lower water absorption rates and similar mechanical properties compared with those from untreated BF/HDPE composites [3]. In a more recent study related to the thermal decomposition behaviors of HDPE/heat-treated BF composites, Li et al. [6]

showed that the values of activation energy were between 225 and 236 kJ/mol and decreased with increasing heat-treatment temperature.

The crystallization properties of polymers have an enormous influence on the performance and processing properties of polymer-based composites [7–9]. The crystallization process includes two stages: nucleation and crystal growth. The nucleation stage produces nucleating sites, in which crystal grain is generated. The crystallization process includes homogeneous nucleation and heterogeneous nucleation. Homogeneous nucleation refers to the spontaneous crystallization of neat resins with changing temperatures. With respect to heterogeneous nucleation, additives mixed in the neat HDPE can help form nucleating sites, leading to a fast crystallization rate of HDPE and higher crystallization temperature [10]. Crystallization tests can be done in both isothermal and nonisothermal conditions [11, 12].

Recently, more attention has been paid to the crystallization properties of polymer in wood/natural fiber composites.

Some studies assert that fillers such as wood and/or other natural fibers play a role as a nucleator and therefore enhance crystallization rate and relative crystallinity, especially for polypropylene (PP) composites. In addition, surface properties of the fibers (e.g., roughness) and modification methods (e.g., using coupling agents to improve compatibility) can affect crystallization properties [13–15]. Mi et al. [16] showed the β-form crystallinity differences in the melting and crystallization processes of neat PP matrix and PP/BF composites from the heat flow curves. Another study contended that this β-form crystallinity was mainly determined by various inorganic fillers, which in turn influenced the properties of those composites, especially the thermal properties [17, 18]. For PP/BF composites, the increasing fiber loading decreased the melting enthalpy, crystallization enthalpy, and crystallinity; and the crystallization temperature increased with the addition of BFs [19]. Under a certain cooling rate, peak temperature had a relationship with the loading level of fillers, and the fillers presented a multinucleating effect [20, 21]. Phuong and Gilbert [22] investigated the crystallization kinetics of recycled PP/bamboo composites, and the results showed that the peak and onset temperatures of crystallization moved to a lower temperature with increasing cooling rate. This was because of the slower cooling rate that provided sufficient time to stimulate the crystallization at a high temperature [22]. Nonisothermal crystallization of neat high-density polyethylene (HDPE), wood flour (WF)/HDPE composite (WPC), virgin Kevlar fiber- (KF-) reinforced WPC (KFWPC), and grafted Kevlar fiber- (GKF-) reinforced WPC (GKFWPC) was investigated by means of differential scanning calorimetry and wide-angle X-ray diffraction [23]. The values of crystallization peak temperature, half-time of crystallization ($t_{1/2}$), and other kinetic parameters showed that the crystallizability followed the following order: FKWPC > GKFWPC > HDPE > WPC. The X-ray diffraction data indicated that the crystalline thickness perpendicular to the reflection plane increased with the addition of KF. The results demonstrated that KF and GKF can act as nucleating agents and increase the crystallization rate of HDPE. Compared with GKF, KF is a more effective nucleating agent for HDPE. Though some of the aforementioned studies investigated the crystallization behavior of bamboo-based polymer composites, none of the published work has dealt with heat-treated bamboo material and the polymer crystallization process, according to our knowledge.

The objectives of this study were to investigate the effects of heat-treated BFs on the nonisothermal crystallization of HDPE and to acquire information about the characteristic parameters of HDPE's crystallization, which could help control the process of crystallization and analyze the properties of the resultant composites during processing.

2. Experimental

2.1. Raw Material and Preparation.
Moso bamboo (*Phyllostachys edulis*) was used for this study. Green bamboo sections with diameters varying from 70 to 100 mm were collected and cross cut and then sawn into strips. All strips were dried at $100 \pm 2°C$ for 48 h to reduce their moisture content to about 6%. The strips were then randomly divided into three equal weight groups. One group was used as the control and the two other groups were subjected to heat treatment at 150 and 180°C for 2 h. All three groups were then hammer-milled, and the crushed material was screened to pass a 40-mesh screen.

High-density polyethylene (HDPE AD60-007 with MFR = 0.7 g/10 min at 190°C/2.16 kg, density = 0.96 g/cm^3) was provided by ExxonMobile Chemical Co. (Houston, TX, USA). MAPE (Epolene G2608 with MFR = 6~10 g/10 min at 190°C/2.16 kg, MF = 65,000 g/mol, and acid number = 8 mgKOH/g) from Eastman Chemical Co. (Kingsport, TN, USA) was utilized to increase compatibility between the fillers and the plastic matrix.

2.2. Composite Sample Preparation.
Melt compounding of HDPE with other components (40 wt% BF, 58 wt% HDPE, and 2 wt% MAPE) was performed using an intermesh, counterrotating Brabender twin-screw extruder (Brabender Instruments Inc., Hackensack, NJ) with a screw speed of 40 rpm. The temperature profile of the barrels was 423-448-448-448-448 K. The extrudates were quenched in a cold water bath and then pelletized into granules, which were oven-dried at 373 K for 12 h and bagged for further testing.

2.3. DSC Test Procedure and Data Analysis.
The crystallization behavior of HDPE in the composites was measured using a differential scanning calorimeter (TA DSC Q2000) under nitrogen. Samples of 5~6 mg were taken and placed in aluminum capsules and were heated from 313 K to 433 K at a rate of 10 K/min and then kept for 5 min at 433 K for eliminating their thermal and mechanical history before crystallization. Samples were then cooled at 2.5, 5, 10, 20, and 35 K/min, respectively.

The crystallinity levels (χ_c) of HDPE in the blends were evaluated as

$$\chi_c = \frac{\Delta H_{\exp}}{\Delta H} \times \frac{1}{W_f} \times 100\%, \tag{1}$$

where $\Delta H_{\exp}$ is the experimental enthalpy of crystallization, ΔH is the assumed enthalpy of crystallization of fully crystalline HDPE, and W_f is the weight fraction of HDPE in the blends. For fully crystalline HDPE, the heat of crystallization is 276 J/g [23]. The half-time of crystallinity ($t_{1/2}$) was determined using the χ_c value. The crystallization time (t) and crystallization temperature (T) are interrelated through the cooling rate φ as

$$t = \frac{T_o - T}{\varphi}. \tag{2}$$

The usual method for analyzing isothermal crystallization kinetics is the Avrami equation, which assumes that the relative crystallinity and crystallization time have the following mathematical relationship:

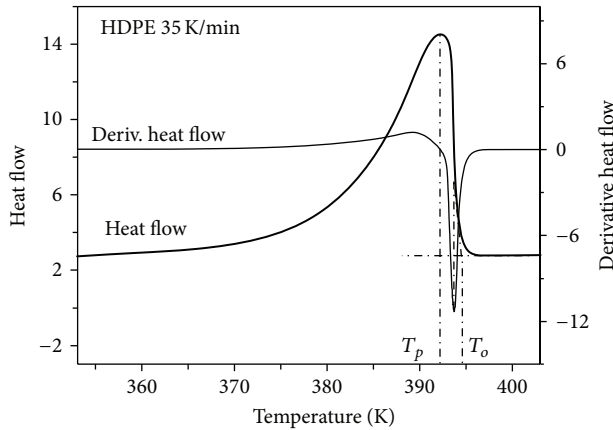$$1 - X_t = \exp\left(-Kt^n\right), \tag{3}$$

FIGURE 1: Determination of crystallization characteristic parameters of HDPE and its composites by using neat HDPE as an example (cooling rate = 35 K/min).

where n is the Avrami exponent, t is crystallization time, X_t is relative crystallinity, and K is the parameter for the crystallization rate. The parameter n shows the geometry of the growing crystals and nucleation process. Equation (3) can also be written as follows:

$$\log\left[-\ln\left(1 - X_t\right)\right] = \log K + n \log t. \tag{4}$$

The Jeziorny method is the modified Avrami equation for nonisothermal crystallization. The values of n and t do not have the same physical significance since the temperature is constantly reduced in isothermal crystallization. Therefore, it is assumed that the crystallization temperature is constant. Jeziorny [12] asserted that K should be modified because the cooling rate should be taken into consideration. The final equation is

$$\log K_J = \frac{\log K}{\varphi}, \tag{5}$$

where K_J is the corrected kinetic rate constant.

3. Results and Discussion

3.1. Nonisothermal Crystallization Behavior of Heat-Treated BFs and HDPE Blends. Figure 1 shows a typical plot of heat flow for neat HDPE. As temperature decreased (from the right side to the left side), the heat flow increased and reached a peak value and then gradually decreased. The obvious absorption peak indicated that HDPE turned from a liquid phase into a crystalline phase. Crystallization characteristic parameters were calculated from heat flow and derivative heat flow curves are shown in Figure 1. The extrapolated onset temperature of crystallization, T_o, was obtained by extrapolating the tangent line of the minimal inflection point on the heat flow curve and down to the baseline level of the heat flow axis. The peak temperature, T_p, was the maximum temperature on the heat flow curve.

Figure 2 shows a comparison of heat flow values of HDPE/heat-treated BF composites at different cooling rates,
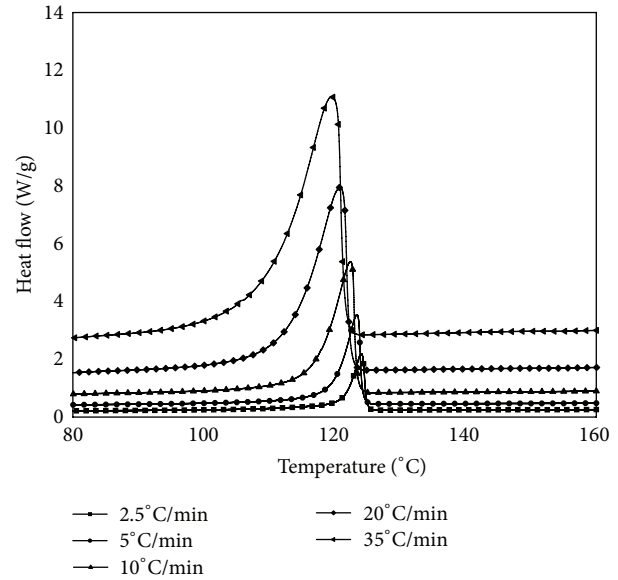

FIGURE 2: A comparison of heat flow curves of HDPE/heat-treated BF composites with heat-treated BFs at different cooling rates.

and Table 1 summarizes the calculated data values. The values of T_o and T_p of both HDPE and the composites reduced gradually, the shoulder of the curves was broadened, and the areas of the curves were enlarged. This is because the slower cooling rate provided better fluidity and diffusivity for the molecules to crystalize at high temperatures [23]. In contrast, faster cooling rates shortened the time for the arrangement of HDPE's molecules and decreased their ability to form the regular structure. Therefore, the faster cooling rate impeded crystallization of HDPE, and more supercooling was needed to reach the same crystallinity at higher cooling rate. The temperature gradient of the samples was presented from the inside outward. Therefore, the crystallization temperature at a lower cooling rate had to be lower in order to attain the same crystallization level. The absorption peak moved to a lower temperature with increasing crystallization time. Furthermore, crystallization properties weakened as the activation energy of HDPE decreased within the low surrounding temperature, which made the peak broaden.

Compared with the nonisothermal crystallization parameters of HDPE, the crystallization temperatures of HDPE/heat-treated BF composites (T_o and T_p) were slightly higher than those of HDPE. This was because of the enhancement effect of the crystallization and relaxation process of HDPE, which was caused by the fact that the addition of BFs and/or a coupling agent could weaken the interfacial interaction. This implies that the BFs could act as a nucleating agent in the HDPE matrix, which provided heterogeneous nucleation sites.

To exclude the influence of the cooling rate, T_o and T_p were obtained using a linear extrapolation to $\varphi = 0$ in the plots of cooling rate against temperature. The crystallinity level corresponding to $\varphi = 0$ was designated as the final crystallinity level. The χ_c values of HDPE and

TABLE 1: Nonisothermal crystallization parameters of HDPE and its composites.

Samples	Φ (K/min)	T_o (K)	T_p (K)	$t_{1/2}$ (min)	ΔH_{exp} (J·g^{-1})	X_c (%)	X_c $\varphi = 0$ (%)
HDPE	2.5	397.9	396.7	1.141	122.0	44.2	
	5	397.3	396.7	0.652	126.8	45.9	
	10	396.3	395.0	0.411	121.1	43.9	45.2
	20	395.5	393.7	0.263	122.5	44.4	
	35	394.2	392.2	0.194	116.8	42.3	
HDPE/heat-treated BF (373 K)	2.5	398.1	397.1	1.033	84.4	51.0	
	5	397.4	396.4	0.585	80.6	48.6	
	10	396.5	395.4	0.378	78.1	47.1	49.9
	20	395.5	393.8	0.253	75.2	45.4	
	35	394.6	392.6	0.192	73.6	44.5	
HDPE/heat-treated BF (423 K)	2.5	398.1	397.1	1.007	81.9	49.5	
	5	397.4	396.4	0.596	79.4	48.0	
	10	396.5	395.4	0.373	76.7	46.3	49.4
	20	395.5	393.9	0.253	73.6	44.4	
	35	394.3	391.9	0.207	67.9	41.0	
HDPE/heat-treated BF (453 K)	2.5	398.1	397.2	1.210	83.2	50.0	
	5	397.3	396.4	0.657	78.7	47.5	
	10	396.6	395.4	0.368	74.0	44.7	51.5
	20	395.5	395.3	0.262	70.8	42.7	
	35	394.7	392.7	0.183	70.1	42.3	

their composites (BFs with a heat-treatment temperature of 373 K, 423 K, and 453 K) were 45.2%, 49.9%, 49.4%, and 51.5%, respectively, which displayed an increasing trend as pretreatment temperatures for BFs increased. One possible reason might be that the addition of heat-treated BFs and/or the coupling agent promoted the nucleation of HDPE. A shorter time was needed to acquire higher crystallinity of the blends, leading to better properties. Another cause could be the interaction between the heat-treated BFs and neat HDPE. It should be pointed out that BF itself is a complex polymer made of cellulose, hemicellulose, and lignin. After heat treatment, the orientation of cellulose molecule chains in the amorphous region could be somewhat improved; the noncrystalline region rearranged to form the crystalline region, and then the bridging reaction might take place in the noncrystalline regions.

Relative crystallinity-crystallization time data were obtained based on the crystallinity-temperature data (Figure 3(a)) and using cooling rate data (Figure 3(b)). The crystallization time of 373 K BF-reinforced HDPE composites decreased significantly as the cooling rate increased. The curve between crystallization time and relative crystallinity had an approximate "S" shape, and the size and shape of the "S" were largely affected by the cooling rate. The parameter $t_{1/2}$ is the half-time of relative crystallinity (from 0 to 50%), which is determined by the driving force for the crystal growth. Figure 3(b) shows that $t_{1/2}$ was less than the medium value of the crystallization time. This was because the HDPE crystallized much faster at the beginning of the crystallization process. The $t_{1/2}$ value was reduced as the cooling rate increased. At the same cooling rate, values of $t_{1/2}$ of heat-treated HDPE/heat-treated BF composites were slightly lower than those of HDPE. This indicates that BF (and/or the coupling agent) accelerated the crystallization rate of HDPE. Thus, the BF could act as a nucleating agent in the HDPE matrix. However, there was no distinguished trend among the $t_{1/2}$ values of various HDPE/heat-treated BF composites.

3.2. Crystallization Kinetics of Heat-Treated BFs and HDPE Blends. Typical plots of $\log[-\ln(1 - X_t)]$ against $\log(t)$ are presented in Figure 4 (composites that contain 373 K heat-treated BF as an example). The left side of the x-axis indicates the early period of crystallization and the right side showed the late period of crystallization. As shown in Figure 4, in the early period of crystallization, the relationship between $\log[-\ln(1-X_t)]$ and $\log(t)$ displayed positive linear trends. At the beginning of crystallization, HDPE molecules crystallized around nucleating sites with freedom in a sufficient space. Toward the end of crystallization, lamellae began to grow from the nucleating sites and their size increased. The lamellae squeezed and collided, which restrained crystal growth in all directions and thus affected the nucleating pathway. The initial data with a higher linearity were used to calculate the parameters n and K_J. Nonisothermal kinetic parameters of the heat-treated BF-reinforced HDPE composites are listed in Table 2.

The values of n reflected the dimensions of the crystal growth, which were in a range of 0~4. A value of n closer to 4 indicates more complete crystallization. Table 2 shows that the n values of neat HDPE and its composites decreased with increased cooling rates, and thus the degree of crystallization of HDPE lamellae reduced. The values of n were not integers
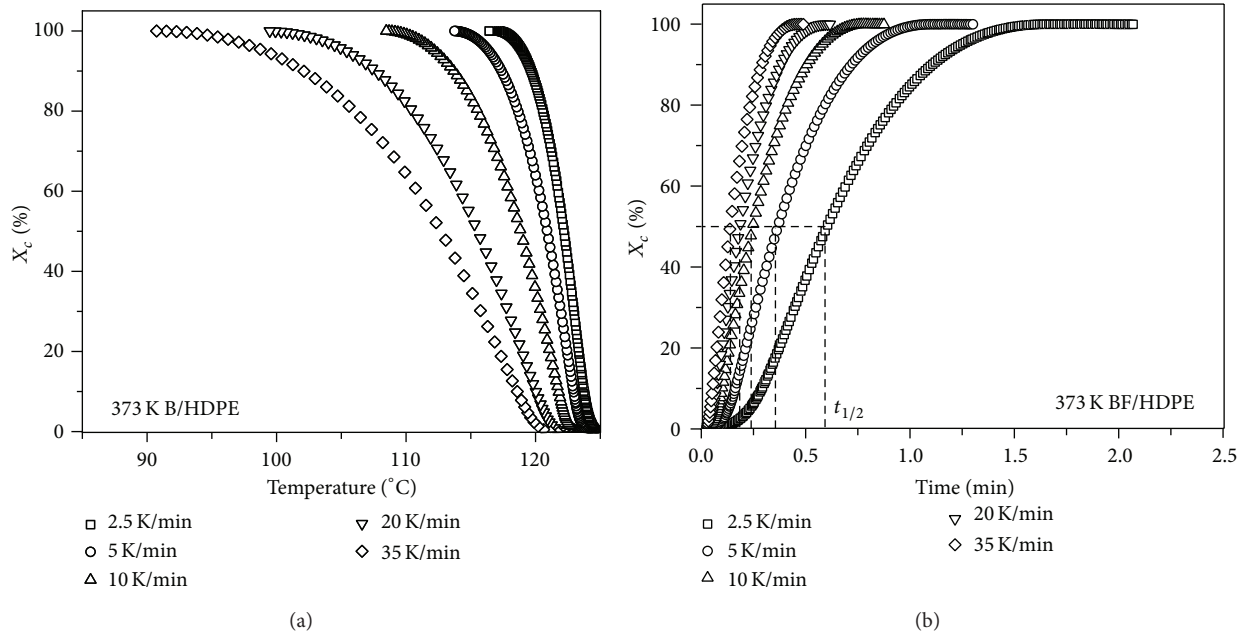
(a)

(b)

FIGURE 3: (a) Curves of relative crystallinity for HDPE/heat-treated BF composites (BF with a heat-treatment temperature of 373 K) versus temperature. (b) Curves of relative crystallinity for HDPE/heat-treated BF composites (BF with a heat-treatment temperature of 373 K) versus time.
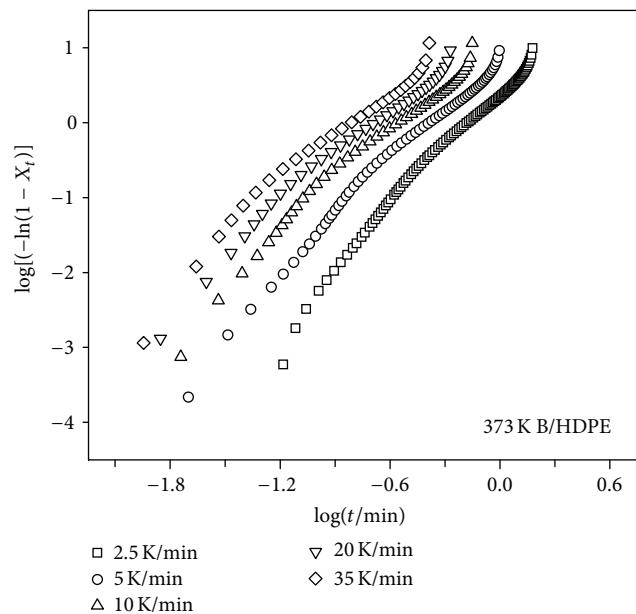

FIGURE 4: Plots of $\log[-\ln(1 - X_t)]$ versus $\log(t)$ for HDPE/heat-treated BF composites (BF with a heat-treatment temperature of 373 K).

as a result of various nucleating pathways brought about by heterogeneous nucleation. The average value of n for neat HDPE was 2.87, while the average values of BF-HDPE composites that contain 373 K, 423 K, and 453 K heat-treated BF were 2.96, 2.87, and 2.88, respectively. This demonstrated that crystals in both neat HDPE and the composites primarily grew in a three-dimensional manner, and the BFs and/or coupling agent enhanced heterogeneous nucleation and caused more complete crystallization, which could be attributed to the nucleating effect of BFs and the compatibilizing effect exerted by the coupling agent. However, heat treatment slightly reduced the values of n, which were close to those of the neat HDPE. Perhaps the heat treatment reduced the surface polarity of bamboo, though further investigation is needed.

K_J reflected the nonisothermal parameters of the crystallization rate with a modified cooling rate. With increasing cooling rate, the values of K_J first increased and then decreased. This is because supercooling was improved by the increased cooling rate to enhance the nucleating sites. The shortened distances between different molecules facilitated the completion of crystallization and boosted crystallization rate. The K_J values of HDPE/heat-treated BF composites were slightly higher than those of neat HDPE.

4. Conclusions

(1) Differential scanning calorimetry under nitrogen was used to investigate the crystallization processes of neat HDPE and the resultant BF-HDPE composites. The heat flow curves of neat HDPE and heat-treated BF-HDPE composites had similar trends. Crystallization of the materials mostly occurred within a temperature range between 379 K and 399 K. With increased cooling rate, the peak temperatures of these composites moved to lower temperature values and the peaks became broadened due to the

TABLE 2: Nonisothermal kinetic parameters at different relative crystallization of HDPE and its composites based on the Jeziorny model.

Samples	φ/K/min	N	$\log K$	R^2	$\log K_J$	K_J	R^2
HDPE	2.5	2.96	0.47	2.95	0.19	1.54	0.9999
	5	3.1	1.42	26.23	0.28	1.92	0.9995
	10	2.88	1.82	65.68	0.18	1.52	0.9999
	20	2.74	2.31	202.19	0.12	1.30	0.9988
	35	2.65	2.65	446.34	0.08	1.19	0.9982
373 K BF/HDPE	2.5	3.05	0.67	4.65	0.27	1.85	0.9990
	5	2.96	1.35	22.5	0.27	1.86	0.9992
	10	2.89	1.94	87.54	0.19	1.56	0.9996
	20	2.93	2.44	275.99	0.12	1.32	0.9993
	35	2.98	2.9	795.39	0.08	1.21	0.9945
423 K BF/HDPE	2.5	3.3	0.86	7.33	0.35	2.22	0.9996
	5	2.8	1.16	14.53	0.23	1.71	0.9987
	10	2.68	1.75	56.02	0.17	1.50	0.9973
	20	2.68	2.11	128.54	0.11	1.27	0.9994
	35	2.89	2.63	422.02	0.08	1.19	0.9995
453 K BF/HDPE	2.5	2.56	0.4	2.49	0.16	1.44	0.9991
	5	2.33	0.74	5.49	0.15	1.41	0.9964
	10	3.38	2.44	273.11	0.24	1.75	0.9993
	20	3.16	2.66	455.38	0.13	1.36	0.9990
	35	2.96	2.96	911.61	0.08	1.21	0.9997

increased cooling rate that improved supercooling and decreased crystallization temperature.

(2) Values of the Jeziorny model parameter n were in the range of 2.8~3.38. Crystal lamellae of neat HDPE and the resulting composites grew in a three-dimensional manner, which increased with increasing BF heat-treatment temperature. This indicates that the addition of BFs and/or a coupling agent was helpful for improving the ability of heterogeneous nucleation and crystallization completeness. The values of the Jeziorny model parameter K_J increased and then decreased when the cooling rate increased because supercooling was improved by the increased cooling rate.

(3) Heat-treated BFs and/or coupling agent could act as a nucleator for the nucleation of HDPE polymer in the composites.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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