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Crystallization kinetics of new copoly(ethylene terephthalate-imide)s

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Abstract

Crystallization kinetics of copoly(ethyleneterephthalate-imide)s (PETIs) of various imide contents were studied by the DSC technique. The results of isothermal and nonisothermal crystallization kinetics studies showed a consistent trend in that the crystallization of all copolyesterimides comprised a primary stage and secondary stage, and that the crystallization mechanism was a three-dimensional growth with athermal nucleation. The crystallization rate of PETIs first increased and then decreased as the content of imide units in the copolyesterimides increased. This indicates that the imide units in the copolymer decreased the chain-packing regularity of poly(ethylene terephthalate) (PET) and the same units also enhanced the copolymer chain rigidity. The copolyesterimide with 0.2 mol% imide unit content showed a dramatic increase of the crystallization rate, and exhibited the highest crystallization rate of all the studied compositions. Studies of isothermal and nonisothermal crystallization kinetics, showed that small amounts of imide units in the the backbone act as nucleating agents during the crystallization process of copolyesterimides. © 2002 Published by Elsevier Science Ltd.

Keywords: Copolyesterimides; Copoly(ethylene terephthalate-imide)s; Poly(ethylene terephthalate)

1. Introduction

The prediction of crystallization behavior at a reasonable semiquantitative level of accuracy is important in assessing the processability and performance [1–3] of different polymeric structures. For example, in both polypropylene and poly(ethylene terephthalate) (PET), the interplay of the effect of rheology and crystallization kinetics under various types of fabrication conditions is crucial in determining the crystalline morphology. In turn, the morphology affects the mechanical, optical, and gas barrier properties of the fabricated articles. Likewise the processing characteristics of semicrystalline polymers, whether done between the glass transition state and the crystalline melting point, or in the melting phase, are very dependent on the crystallization kinetics and the resultant morphology of the polymer [1].

PET, a typical semicrystalline polymer, is a major commercially important polymer and is widely used as fibers, films, and/or as engineering plastics [4,5]. However, the low crystallization rate of PET homopolymer makes it less attractive for injection molding applications than faster crystallizing polyesters like poly(butylene terephthalate). In the last several years, physical and chemical modifications of PET have been utilized and resulted in a series of new polyesters with superior performance over unmodified PET [5,6]. It is well known that copolymerization is always an effective method in chemical modification of PET or other polyesters. Furthermore, the increasing demand for materials with high performances has drawn considerable interest to the PET copolymers, especially in the field of liquid crystalline polymers (LCPs). Many papers [7–11] have reported on the incorporation of rigid units to PET segments to achieve superior performance. Among them, the PET/HBA copolyesters are successful examples and have become probably the first commercialized thermotropic liquid crystal polymers [10,11]. Up to now, there are only very few papers in the literature on incorporating rigid imide units into PET [8,12,13]. These reports focused on the synthesis and thermal properties of copolyesters and did not cover the crystalline behavior and processability. However, the crystallization behavior is an important property for PET and its copolymers, and plays an important role in their processing and application. In our previous studies [14,15], a series of new copolyesterimides, PETIs, were synthesized, which contain rod-like imide units in the

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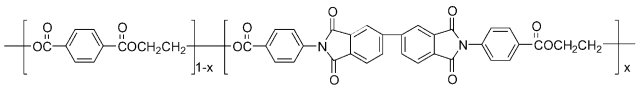
main chain of PET segments. The results of thermal properties and mechanical properties analyses showed that the glass transition temperature (T_g) increased significantly with higher content of the imide units. The T_g of copolymer rose by 12 or 19 °C as 5 or 10 mol% imide units were incorporated into PET. Some mechanical property tests showed that the tensile strength of the copolyesterimides increased significantly by up to 53.6% as 7 mol% of imide was incorporated into PET [14]. Since the crystallization behavior is very important for determining property profiles and in eventual applications, we have studied the crystallization characteristics of this new family of PETIs.

2. Experimental

2.1. Materials

The copolyesterimides (PETIs) utilized for the investigation were synthesized in one of our previous studies [14], by the melt-copolycondensation of dimethyl terephthalate (DMT), ethylene glycol and *N*,*N*′-bis(*p*-(methoxy carbonyl)phenyl)-biphenyl-3,3′,4,4′-tetracarboxydiimide (BMBI). PETIs were first dissolved in the mixture of 1,1,2,2-tetrachloroethane and phenol (1:1, v/v), and then precipitated by adding the solution into excess of methanol. The precipitate was then extracted by methanol for 24 h to remove the solvent. After vacuum dried at 80 °C for 12 h, the dried copolyesterimides were used for DSC analysis. A description of the materials investigated is shown in Table 1. The chemical structure for PETI series is shown as follows:



PETI

2.2. Characterization [16]

A Perkin–Elmer DSC-7 instrument with cold trap was used for thermal analysis and characterization. The instrument was calibrated by indium before the measurements. All measurements were conducted under a high-purity nitrogen atmosphere to minimize the possibility of moisture regain and consequent hydrolytic degradation. The weight of samples was about 5 mg for all copolyesterimides.

For isothermal crystallization kinetic studies, the samples sealed in aluminum pan were heated from 20 to 280 °C at 20 °C/min; held at the final temperature for 5 min to erase any previous thermal and/or mechanical history; then, quenched at 300 °C/min to the desired temperature. The exothermic curves as a function of time were then recorded. For nonisothermal crystallization kinetic studies, the samples were measured as the procedure of isothermal crystallization kinetic studies, from 20 to 280 °C at 20 °C/min; kept at 280 °C for 5 min to eliminate any effects of past thermal/mechanical history. Then, the samples were cooled at different rates to 100 °C. These cooling curves were recorded and used for nonisothermal crystallization kinetics analyses. The cooling rates used in this study were 40, 20, 10, 5, and 2.5 °C/min.

Table 1
Esterimide contents and intrinsic viscosity of synthesized PETIs copolyesterimides

Sample	BMBI/(DMT + BMBI) ratio		$[\eta]_{inh}$[a] (dl/g)
	Feed (mol/mol, %)	Found[b] (mol/mol, %)	
PET	0	0	0.83
PETI-A	0.2	0.21	0.90
PETI-B	0.5	0.47	0.80
PETI-C	1	0.96	0.75
PETI-D	2	2.00	0.94
PETI-E	5	4.53	0.98
PETI-F	7	7.10	0.84

[a] Measurement using TCE/phenol (50/50, v/v) as solvent at 25 °C with concentration of 0.5 g/dl.
[b] Chemical composition calculated from ^{1}H NMR.

3. Results and disscussion

3.1. Isothermal crystallization kinetics

The crystallization process is usually treated as a series of two stages: the primary and the secondary crystallization stage. The crystallization process was very marked by temperature dependence. Assuming that the relative degree of crystallinity increased with an increase in the crystallization time t, the Avrami equations (Eqs. (1a) and (1b)) [17,18] were used to analyze the isothermal crystallization process of copolyesterimides, as follows:

$$X(t) = 1 - \exp(-Kt^n) \tag{1a}$$

or

$$\ln\{-\ln[1 - X(t)]\} = n \ln t + \ln K \tag{1b}$$

where $X(t)$ is the relative degree of crystallinity, t the time, K the growth rate constant, and n is the exponent in Eqs. (1a) and (1b) represents a parameter revealing the nucleation mechanism and growth dimension. Generally, the value of n was found to vary between 1 and 4, corresponding to various growth forms from rod-like to sphere-like. As it is well known, the Avrami model lumps together the formation of nuclei and their subsequent growth.

The plots of $\ln\{-\ln[1 - X(t)]\}$ versus $\ln t$ are shown in Fig. 1. Each curve shows an initial linear portion, then subsequently tends to level off. This fact indicates the existence of a secondary crystallization, which is caused by spherulite impingement in the later stage of crystallization process of copolyesterimides [2,3,19].

The value of n and K were determined from the initial

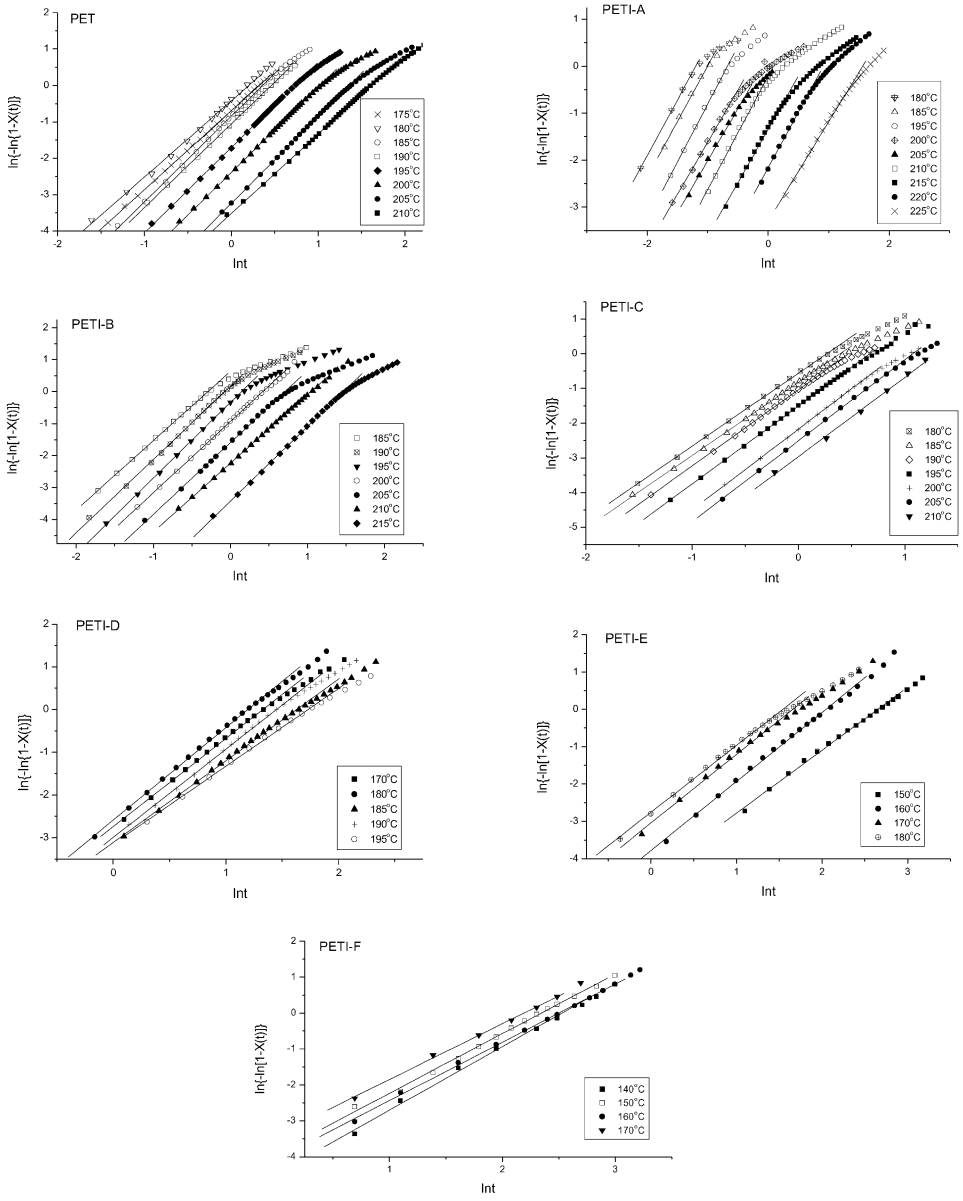


Fig. 1. Plots of $\ln\{-\ln[1 - X(t)]\}$ versus $\ln t$ for isothermal crystallization of PETIs copolymers and neat PET.

linear portion in Fig. 1 and are listed in Table 2. The Avrami exponent n decreases as the imide content in PETIs increases. The Avrami exponent n for PETIs with imide content below 2% is close to 2.5. This value means that the crystallization mode is of three-dimensional growth with an athermal nucleation, which has been detailed in a review paper by Bicerano on the crystallization kinetics of PET [1]. On the other hand, the value of n was found to decrease to below 2 for PETIs with the imide content above 2%. This indicates that the crystallization mode might shift to two-dimensional growth with an athermal (heterogeneous) nucleation.

The crystallization half-time $t_{1/2}$ is defined as the time at which the extent of crystallization is completed 50%. It can be determined from the measured kinetic parameters [1,2]. That is

$$t_{1/2} = (\ln 2/K)^{1/n} \tag{2}$$

Usually, the crystallization rate at 50% crystallinity, $\tau_{1/2}$ is described as the reciprocal of $t_{1/2}$. That is to say, $\tau_{1/2} = 1/t_{1/2}$. The values of $\tau_{1/2}$ and $t_{1/2}$ are also listed in Table 2.

For all copolyesterimides containing various contents of imide units, the crystallization rate constant K and crystallization rate $\tau_{1/2}$ were found to increase with decreasing crystallization temperature T_c. This result is easy to understand. A higher crystallization temperature means a lower supercooling degree, and the nuclei for the crystallization are more difficult to form.

At a given crystallization temperature T_c, the trends of

Table 2
Isothermal crystallization parameters for PETIs and PET

Sample	T_c (°C)	n	K (min^{-n})	$t_{1/2}$ (min)	$\tau_{1/2}$ (min^{-1})
PPET	175	2.17	0.50	1.16	0.86
	180	2.18	0.63	1.05	0.96
	185	2.35	0.43	1.23	0.82
	190	2.32	0.33	1.37	0.73
	195	2.34	0.18	1.77	0.56
	200	2.47	0.08	2.42	0.41
	205	2.39	0.04	3.33	0.30
	210	2.24	0.03	4.27	0.23
PETI-A	180	2.30	9.28	0.32	3.08
	185	2.48	12.72	0.31	3.23
	195	2.58	6.12	0.43	2.33
	200	2.34	1.30	0.76	1.31
	205	2.34	1.39	0.74	1.35
	210	2.46	0.84	0.93	1.08
	215	2.31	0.27	1.51	0.66
	220	2.37	0.11	2.13	0.47
	225	2.16	0.04	3.94	0.25
PETI-B	185	2.20	1.95	0.62	1.60
	190	2.34	1.20	0.79	1.27
	195	2.35	0.70	0.99	1.01
	200	2.22	0.40	1.29	0.78
	205	2.26	0.20	1.72	0.58
	210	2.13	0.11	2.40	0.42
	215	2.26	0.04	3.58	0.28
PETI-C	180	2.12	0.58	1.09	0.92
	185	2.10	0.44	1.25	0.80
	190	2.19	0.35	1.36	0.74
	195	2.26	0.22	1.65	0.61
	200	2.28	0.11	2.21	0.45
	205	2.33	0.08	2.51	0.40
	210	2.35	0.05	3.04	0.33
PETI-D	170	2.05	0.06	3.18	0.31
	180	2.10	0.05	3.53	0.28
	185	1.92	0.04	4.19	0.24
	190	1.96	0.06	3.55	0.28
	195	1.86	0.04	4.55	0.22
PETI-E	150	1.61	0.01	11.47	0.09
	160	1.80	0.02	6.39	0.16
	170	1.76	0.05	4.66	0.21
	180	1.75	0.07	3.86	0.26
PETI-F	140	1.74	0.01	10.36	0.10
	150	1.71	0.02	8.49	0.12
	160	1.60	0.02	9.62	0.10
	170	1.58	0.03	6.97	0.14

variation of the crystallization rate constant K and crystallization rate $\tau_{1/2}$ were quite complicated. It was found that the values of K and n increased first and then decreased as the content of imide units in PETIs increased. For a direct comparison, K and n of different copolyesterimides at crystallization temperatures of 185 and 200 °C, respectively, were extracted from Table 2 and re-listed in Table 3, and also exhibited as plots in Fig. 2.

Fig. 2 shows clearly the above tendency. Apparently, the copolyesterimide with 0.2% imide unites has the highest

Table 3
Isothermal crystallization parameters for PETIs

T_c (°C)		PET	PETI-A	PETI-B	PETI-C	PETI-D
185	$\tau_{1/2}$ (min^{-1})	0.82	3.23	1.60	0.80	0.24
	k (min^{-n})	0.43	12.72	1.95	0.44	0.04
200	$\tau_{1/2}$ (min^{-1})	0.41	1.31	0.78	0.45	–
	k (min^{-n})	0.08	1.30	0.40	0.11	–

crystallization rate, which is also much higher than that of PET and other copolyesterimides synthesized in this study. The crystallization rate of other copolymers decreased as the content of imide units increased. This result is consistent with the results in the literature on the crystallization kinetics of other copolymers [1]. In general, it is well-known that the incorporation of different size and shape of comonomers into a polyester backbone hinders crystallization of the backbone because of the disruptions in the regular chain structure which inhibits the regular chain packing necessary for crystallization. In our studies, the comonomer was a rod-like imide unit, which is very rigid and is commonly used as mesogenic units for the LCPs [20]. As more imide units were incorporated into PET backbone, the polymer chain becomes more rigid, and the movement of the polymer chain is inhibited, thus the crystallization rate decreased.

However, incorporation of a small amount of comonomer unit in the polymer chain can play a different role for the crystallization rate of copolymers. The incorporation of small amounts of the imide group only slightly changes the chemical structure of the main PET chain, but the unit can act as a nucleating agent. So small amounts of comonomer units can be expected to increase the crystallization rate. In this study, it was found that the crystallization rate of copolyesterimides with imide contents below 1% increased when compared to that of pure PET, and PETI-A with

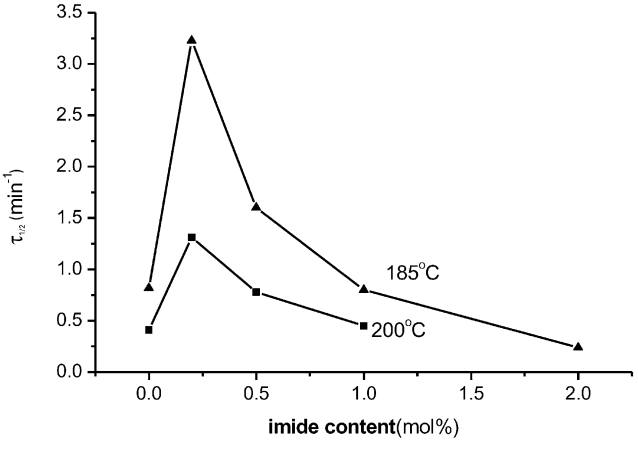


Fig. 2. Plots of $\tau_{1/2}$ as a function of imide content for PETIs and PET at temperature of 185 and 200 °C.

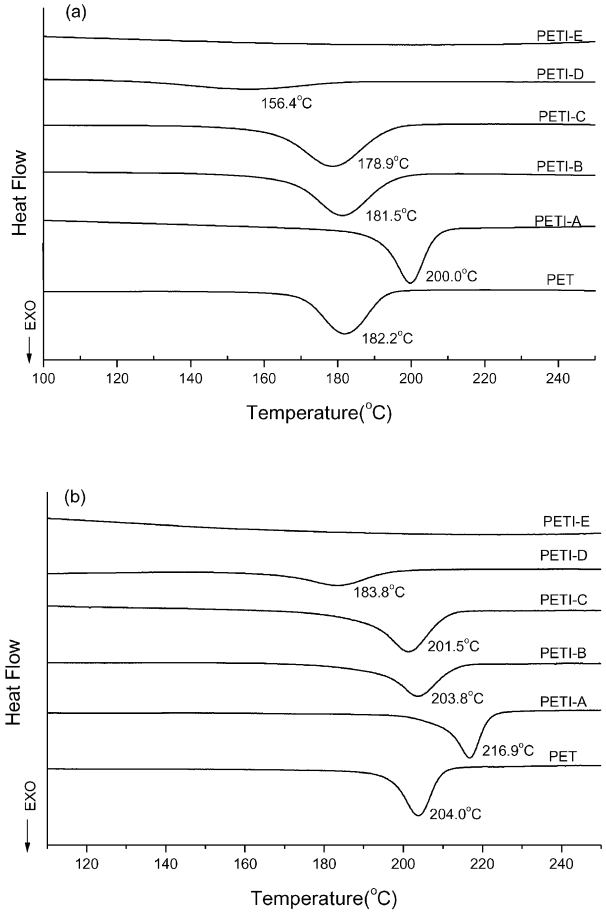


Fig. 3. DSC curves of PETIs and PET at a cooling rate of: (a) 20 °C/min, and (b) 5 °C/min.

0.2 mol% imide content showed a dramatic increase of crystallization rate. For PETI-0.2, the K and n at 185 °C were, respectively, 29.6 and 3.9 times that of neat PET. At 200 °C, these values were 16.2 and 3.2 times that for the neat PET.

3.2. Nonisothermal crystallization kinetic analysis

The isothermal crystallization behavior of copolyesterimides has been discussed in Section 3.1. However, nonisothermal crystallization behavior is of more practical interest than isothermal behavior. This is because polymers are usually processed under nonisothermal cycles rather than in an isothermal condition. Thus, nonisothermal crystallization kinetics of copolyesterimides will be discussed in the following sections.

Fig. 3 describes two series of DSC curves of PETIs at a cooling rate of 20 and 5 °C/min, respectively. Just from crystallization temperature (T_c) during the cooling scan, a general tendency about the crystallization rate of copolyesterimides can be seen. Crystallization temperature T_c of PETIs from the melt decreased with increasing imide content, this phenomenon indicated that the crystallization rate decreased with increasing imide content. But the T_c of PETI-A with 0.2 mol% imide content was higher than that of neat PET or any other PETIs. This fact indicates that PETI-A has the highest crystallization rate.

Table 4
Nonisothermal crystallization parameters for PETIs and PET

Sample	R (°C/min)	n	Z_t ($\min^{-n}$)	$\tau_{1/2}$ ($\min^{-1}$)
PET	2.5	3.18	0.01	0.22
	5	3.86	0.02	0.39
	10	3.55	0.18	0.68
	20	3.13	1.21	1.20
	40	2.84	4.11	1.87
PETI-A	2.5	2.92	0.01	0.22
	5	3.32	0.02	0.36
	10	3.28	0.24	0.73
	20	3.52	1.46	1.24
	40	2.85	4.07	1.86
PETI-B	2.5	2.73	0.01	0.21
	5	2.80	0.03	0.33
	10	2.80	0.22	0.66
	20	2.80	0.65	0.98
	40	2.71	2.02	1.48
PETI-C	2.5	2.89	0.00	0.18
	5	2.73	0.03	0.33
	10	2.84	0.15	0.58
	20	2.97	0.50	0.90
	40	2.77	1.72	1.39
PETI-D	2.5	3.20	0.00	0.20
	5	2.95	0.02	0.30
	10	2.71	0.07	0.43
	20	2.45	0.42	0.82

Mandelkern [21] considered that the primary stage of nonisothermal crystallization could be described by the Avrami equation, which was based on the assumption that the crystallization temperature was constant. Mandelkern obtained the following equations [21,22]:

$$X(t) = 1 - \exp(-Z_t t^n) \tag{3a}$$

$$\ln\{-\ln[1 - X(t)]\} = n \ln t + \ln Z_t \tag{3b}$$

where $X(t)$ is the relative degree of crystallinity at time t, Z_t the rate constant, and n is the Avrami exponent in the nonisothermal crystallization process.

Fig. 4 shows that by drawing straight lines of $\ln\{-\ln[1 - X(t)]\}$ versus $\ln t$ by using Eq. (3b), the values of Avrami exponent n and the rate constant Z_t can be obtained from the slope and intercept of the lines. The values of n, Z_t, and $\tau_{1/2}$ are summarized and shown in Table 4.

Similar to the previous treatments for isothermal crystallization kinetics (Fig. 1), all curves were divided into the primary crystallization stage and the secondary crystallization stage. The result also indicated the existence of a secondary crystallization in the process of nonisothermal crystallization for copolyesterimides. At the primary stage, the Avrami exponent was found to be $n \approx 3$, which suggested

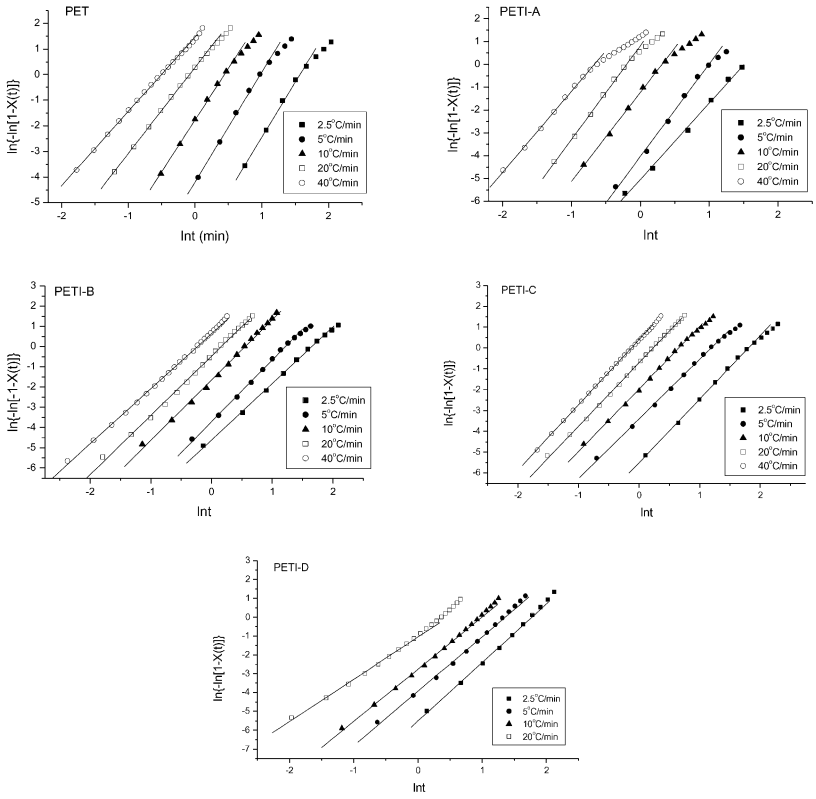


Fig. 4. Plots of $\ln\{-\ln[1-X(t)]\}$ versus $\ln t$ for nonisocrystallization of PETIs and PET.

that the mode at primary stage of the nonisothermal crystallization of copolyesterimides was of three-dimensional growth with an athermal nucleation mechanism [1]. This is about the same as that found in the isothermal crystallization process.

In addition, all the values of crystallization rate constant Z_t and crystallization rate $\tau_{1/2}$ of PETIs showed the same tendency as those found in the isothermal crystallization studies. The crystallization rates increased first, and then decreased as the imide content increased. However, the degree of variation was less than that of the isothermal crystallization process, which may be attributed to that Avrami equation was limited to describe the nonisothermal crystallization process of polymers.

3.2.1. Ozawa analysis in nonisothermal crystallization kinetics

It is well known that the Avrami equation can describe the isothermal crystallization process, but it is not applicable to describe the nonisothermal crystallization kinetics. The Avrami equation for a nonisothermal crystallization process may be considered as just the same mode for an isothermal crystallization process, but it does not take into account of the factors that are special for nonisothermal processes, i.e. cooling rate and the temperature variation at different time. Considering the effect of cooling rate R, Ozawa [23] shifted the Avrami equation (Eqs. (3a) and (3b)) into the process of nonisothermal crystallization, as follows:

$$1 - C(T) = \exp[-K(T)/R^m] \tag{4a}$$

$$\ln\{-\ln(1 - C(T))\} = -m \ln R + \ln K(T) \tag{4b}$$

where $C(T)$ is the relative degree of crystallinity, m the Ozawa exponent, and $K(T)$ is the crystallization rate constant. Drawing the plot of $\ln\{-\ln[1 - C(T)]\}$ versus $\ln R$ according to Eqs. (4a) and (4b), we should obtain a series of straight lines. However, we did not obtain straight lines according to the procedure of Ozawa analysis. This experimental fact indicates that Eqs. (4a) and (4b) is not quite applicable to describe the kinetics in the nonisothermal crystallization process [2], which have an appreciable extent of secondary crystallization.

3.2.2. Combined Avrami equation and Ozawa equation

In order to find a method to describe exactly the nonisothermal crystallization process, Liu et al. [24] have developed a new method for nonisothermal crystallization process and proved to be successful. They obtained the following equations by relating Eqs. (3a) and (3b) to Eqs. (4a) and (4b) [24,25]:

$$\ln Z_t + n \ln t = \ln K(T) - m \ln R \tag{5a}$$

$$\ln R = 1/m \ln[K(T)/Z_t] - n/m \ln t \tag{5b}$$

Define $F(T) = [K(T)/Z_t]^{1/m}$, and $a = n/m$. The parameter

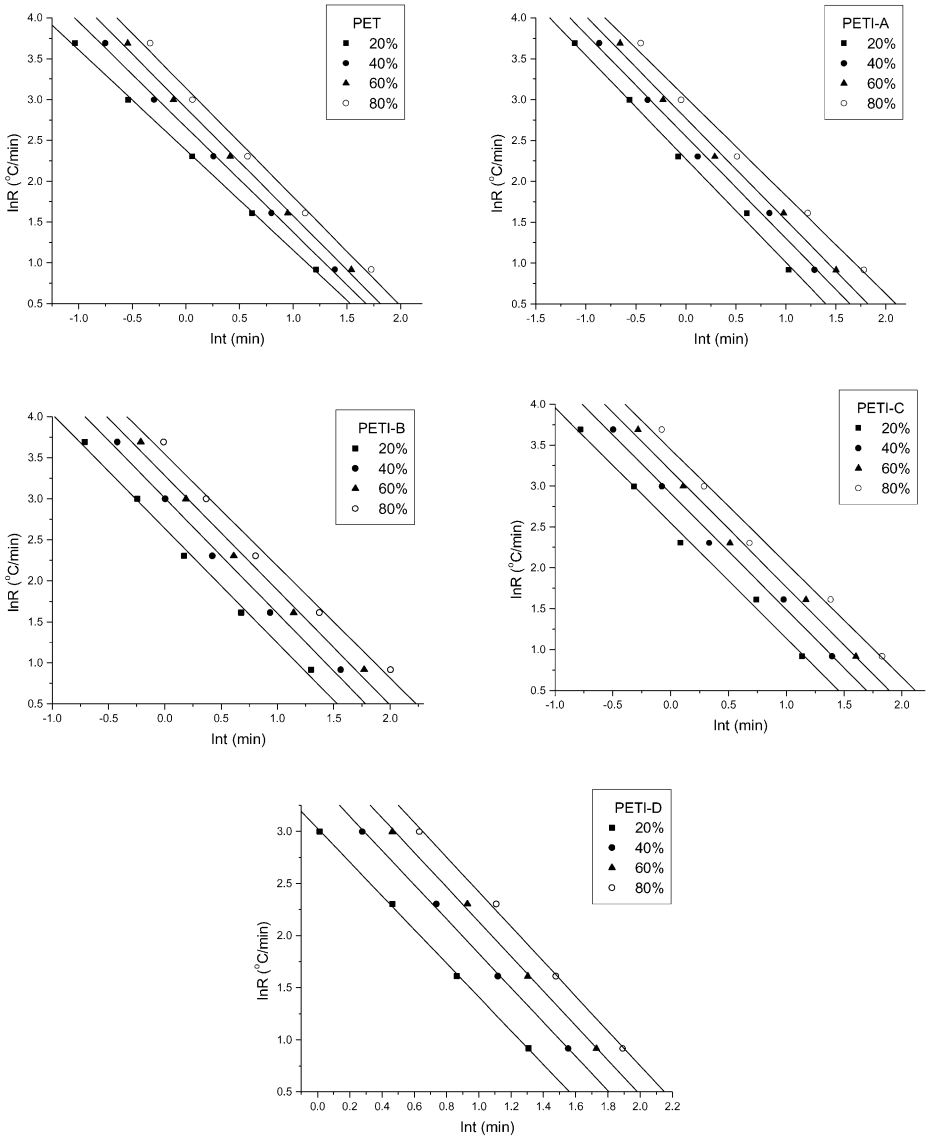


Fig. 5. Plots of ln R versus ln t for PETIs and PET at various $X(t)$.

$F(T)$ is the value of cooling rate, which has to be chosen at unit crystallization time when the measured system amounted to a certain degree of crystallinity. The smaller is the value of $F(T)$, the higher the crystallization rate becomes. Therefore, $F(T)$ has a definite physical meaning. By the above assumptions, Liu et al. have obtained the following expression [24]

$$\ln R = \ln F(T) - a \ln t \tag{6}$$

At above a certain degree of crystallinity of copolyesterimides, the plots of ln R versus ln t according to Eq. (6) are shown in Fig. 5. Using linearity fitting with these data, one can obtain a series of lines with slope $= -a$ and intercept $= \ln F(T)$. The values of a and $F(T)$ are also listed in Table 5. In general, the value of $F(T)$ for all copolyesterimides increased with an increase of the imide content, which indicates the crystallization rate decreases as the imide content increases. But the value of $F(T)$ for PETI-A (0.2 mol% imide content) is the lowest when compared to that of neat PET and other copolyesterimides, which means that PETI-A had the highest crystallization rate among all PETIs and neat PET.

A conclusion similar to that from the isothermal crystallization studies can be drawn. During the nonisothermal process, the crystallization rate of PETI-A was found to be the highest, and crystallization of other copolyesterimides decreased as the content of imide units increased. The interpretation is that high amounts of imide units in the copolymers may disturb the chain-packing regularity and make the backbone more rigid. Therefore, the crystallization rate decreases as the content of imide units increases. On the other hand, small amounts of incorporated imide units in the PET

Table 5
The values of parameters a and $F(T)$ at a certain degree of crystallinity $X(t)$ estimated from Eq. (6) for PETIs and PET

Sample	$X(t)$ (%)	a	$F(t)$
PET	20	1.23	10.81
	40	1.28	14.32
	60	1.32	18.07
	80	1.33	23.12
PETI-A	20	1.27	9.72
	40	1.25	12.82
	60	1.24	15.99
	80	1.20	20.65
PETI-B	20	1.41	12.76
	40	1.42	18.36
	60	1.42	24.21
	80	1.39	30.90
PETI-C	20	1.39	13.95
	40	1.40	20.18
	60	1.39	26.54
	80	1.36	34.43
PETI-D	20	1.62	20.60
	40	1.64	32.21
	60	1.66	44.36
	80	1.67	59.29

backbone, appear to have little effect on the chain regularity and these units appear to function as nucleating agents during the crystallization process. Therefore the crystallization rate of PETI-A was higher than that of neat PET or those of any other copolyesterimides.

4. Conclusions

The Avrami analysis of a series of new copolyesterimides indicated that the isothermal crystallization process consisted of a primary stage and secondary stage. During the primary stage of the isothermal crystallization process, an Avrami exponent $n = 1.6–2.5$ was found for copolyesterimides, and the process of crystal nucleation and growth were mainly three-dimensional growth with athermal nucleation. During the primary stage of the nonisothermal crystallization process, the Avrami exponent was found to be $n \approx 3$, indicating a mechanism of spherulitic growth and athermal nucleation, which was about the same mechanism as that in the isothermal crystallization process for copolyesterimides.

The Ozawa equation was found to fail to describe the nonisothermal crystallization process. But with a combined approach based on both the Avrami and Ozawa equation, one can describe the nonisothermal crystallization process, and obtained the same result as that in the isothermal crystallization process. The results of isothermal crystallization process and nonisothermal crystallization process of PET and copolyesterimides showed that the crystallization rate increased first and then decreased as the content of imide units in the copolymer increased. Finally, PETI-A, which is the copolyesterimide synthesized with 0.2 mol% imide content, had the highest crystallization rate. Its values of $\tau_{1/2}$ at 185 and 200 °C were, respectively, 3.9 and 3.2 times that of neat PET, which is a dramatic increase of crystallization rate over that for the neat PET. We report in a future publication our study of PETIs with very low levels of imide unit incorporation.

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