

# Crystallization behavior of partially melted poly(ether ether ketone)

## Influence of pre-crystallization conditions

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**Abstract** The crystallization behavior of partially melted poly(ether ether ketone) (PEEK) was investigated by differential scanning calorimetry. The influence of pre-crystallization conditions was studied in details. After partial melting, the maximum peak shifts to higher temperature by 8.4–11.8 °C during subsequent cooling, indicating that self-nucleation remarkably enhanced the overall crystallization rate of PEEK. An interesting finding is that the self-nucleation temperature domain of PEEK is way outside the melting curve, and its magnitude is greatly large (28–35 °C). Moreover, samples that were pre-crystallized nonisothermally or from the glassy state exhibit a larger self-nucleation effect, and a larger self-nucleation temperature domain. The isothermal crystallization data were analyzed by the Avrami model. The Avrami exponent is found to be remarkably different for melts with and without self-nucleation effects, indicating that self-nucleation changes the crystallization mechanism of PEEK.

**Keywords** Poly(ether ether ketone) · Partial melting · Crystallization behavior · Thermal analysis

## Introduction

It has been well established that primary nucleation is the crucial step to initiate the crystallization of polymers [1, 2]. There are two types of primary nucleation: homogeneous and heterogeneous nucleation. Homogeneous nucleation can be defined by spontaneous aggregation of polymer molecules to form a three-dimensional nucleus, in which large supercooling is required. While in heterogeneous nucleation, nucleation sites preexist in a sample, which can lower the surface free energy barrier toward nucleation, and thus initiate crystallization at higher temperature during cooling. This can usually be achieved through the addition of nucleating agents. Alternatively, self-nucleation has been demonstrated as an effective approach to overcome the problem of nucleation and to avoid large supercooling [1–13]. Self-nucleation typically refers to clusters of molecules that retain their crystallographic arrangement as a result of partial melting, and on subsequent cooling, these aggregates of clusters of molecules can act as pre-determined athermal nucleation sites which can greatly enhance the overall crystallization rate of polymers [4–6]. For example, after partial melting, the maximum crystallization peak shifts to higher temperature by 30 °C during subsequent cooling for poly(lactic acid) (PLA), which is a polyester with intrinsically slow crystallization rate [7].

Self-nucleation effect has been studied as a function of experimental variables such as heating rate, melting temperature and time [4–14]. Among the variables, melting temperature plays a critical role on the self-nucleation effect. According to Fillon et al. [5], self-nucleation temperature range (denoted  $T_s$ ) or domain II corresponds to the temperatures that are high enough to melt almost all crystals in the sample, but low enough to leave small crystal self-nuclei that can nucleate the polymer upon

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subsequent cooling from  $T_s$ . It is characterized by an exponential increase in nucleation density and a corresponding shift of the peak crystallization temperature to higher values. In contrast, domain III occurs at lower temperatures where partial melting and annealing take place simultaneously, while domain I is defined as the temperature range where complete melting is achieved [5]. It is noteworthy that the  $T_s$  range is strongly dependent on the chemical structures of polymers [4, 5, 13]. For example, for isotactic polypropylene (iPP) the  $T_s$  range is rather narrow (4 °C), and the domain is located between the maximum and its high-temperature foot of the melting peak [4, 5]. While for polyvinylidene fluoride (PVDF), the  $T_s$  range can be very large (15 °C) and its upper limit is far beyond the high-temperature foot of the melting curve [13].

Besides, the pre-crystallization conditions also play an important role on the self-nucleation effect of polymers [7, 12, 15, 16]. Maus et al. [12] investigated the self-nucleation behavior of syndiotactic polypropylene (sPP) pre-crystallized isothermally from the melt at various crystallization temperatures. It was found that the samples pre-crystallized at lower temperature exhibited a larger self-nucleation effect. For PLA, the self-nucleation behaviors of as received samples [15], single crystals [16] and the ones crystallized from the melt nonisothermally [7] were also reported in the literature.

Poly(ether ether ketone) (PEEK) is a high-performance engineering thermoplastic polymer, widely used in aerospace, automotive, electronics and chemical industries [17–20]. Nevertheless, a high degree of crystallinity is required to ensure optimal mechanical performances [21–23]. The understanding of the crystallization behavior allows a better control and optimization of the crystallinity and crystalline structure of polymers. In this context, the isothermal and nonisothermal behaviors of PEEK have been investigated extensively [24–32]. However, little work has been carried out to date on the influence of self-nucleation on the crystallization behavior of PEEK [33]. Tan et al. [33] investigated the crystallization kinetics of PEEK from its metastable melt, obtained from partial melting between the double endotherm. After partial melting, the type of lamellar crystal exhibiting the higher thermodynamic stability remained and the metastable melt recrystallized immediately just above the lower melting temperature and formed slightly thicker lamellae than the original ones [33].

In this paper, the influence of pre-crystallization conditions on the self-nucleation behavior of poly(ether ether ketone) (PEEK) was investigated by differential scanning calorimetry (DSC). Since PEEK can be crystallized either from the melt or the glassy state [28, 30, 32], it provides an adequate model to investigate the influence of pre-crystallization conditions on the self-nucleation behavior of

polymers. In general, the spherulites in melt-crystallized polymers are much larger than those in cold-crystallized counterparts [34, 35]. Moreover, a single polymer spherulite usually exhibits radial inhomogeneity on a micrometer scale [36]. In this context, the self-nucleation behavior of melt-crystallized and cold-crystallized PEEK samples may be different.

## Experimental

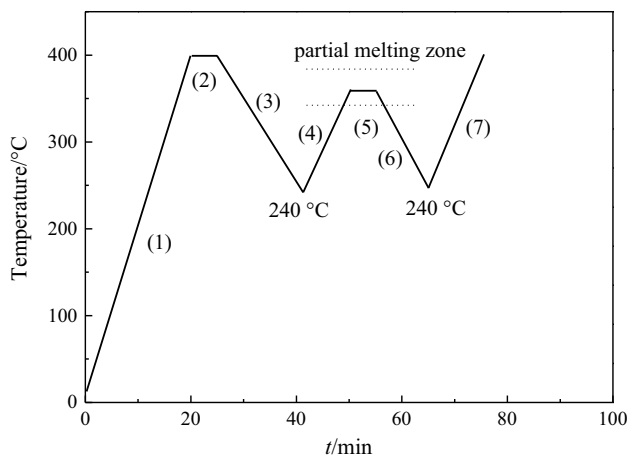
### Materials

The PEEK was purchased from Sigma–Aldrich. Its number- and weight-average molecular weights were  $1.03 \times 10^4$  and  $2.08 \times 10^4$ , respectively.

### DSC measurements

A DSC 2920 (TA Instruments) was used to study the crystallization behavior of PEEK. Calibration for the temperature and energy scales was carried out using a pure indium standard. Sample weight was about 5 mg. Each sample was used only once, and all the runs were carried out in a nitrogen atmosphere. Pre-crystallized PEEK samples were prepared by nonisothermal or isothermal crystallization from the melt or the glassy state, respectively. In details, the samples were first heated to 400 °C at  $20 \text{ K min}^{-1}$  and held for 5 min to erase previous thermal history; then, they were (a). cooled to 240 °C at  $10 \text{ K min}^{-1}$ ; or (b). cooled rapidly to 320 °C and held for 30 min; or (c). quenched into ice water to obtain amorphous state and then reheated at  $10 \text{ K min}^{-1}$ ; or (d). quenched into ice water and then cold-crystallized at 159 °C for 30 min, to obtain the pre-crystallized samples. To observe the melting behavior of the pre-crystallized samples, the DSC scans were recorded further during subsequent heating at  $10 \text{ K min}^{-1}$ .

As relevant for determining the partial melting temperature ( $T_s$ ) range of the pre-crystallized PEEK, Fig. 1 illustrates a representative thermal program employed for self-nucleation experiment of the sample pre-crystallized nonisothermally from the melt. The sample was first heated to 400 °C at  $20 \text{ K min}^{-1}$  (step 1) and held for 5 min (step 2); then, it was cooled to 240 °C at  $10 \text{ K min}^{-1}$  to obtain the pre-crystallized sample (step 3). The pre-treated sample was heated at  $10 \text{ K min}^{-1}$  to  $T_s$  (step 4) and held for 5 min to create self-nucleated sites (step 5). Then, it was cooled to 240 °C at  $10 \text{ K min}^{-1}$ , and the crystallization peak temperature of self-nucleated PEEK was determined (step 6). Finally, the sample was reheated to 400 °C at  $10 \text{ K min}^{-1}$  (step 7). This program is adapted to other pre-crystallized conditions (step 3 is modified, also the levels of steps 5 and 6).



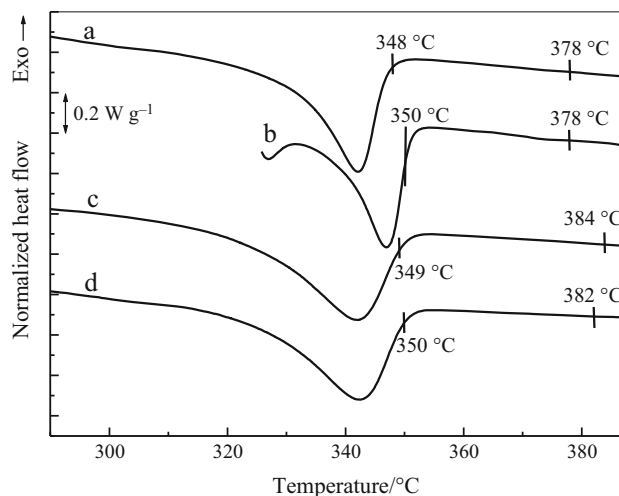
**Fig. 1** Representative thermal program employed for self-nucleation experiment of PEEK. This plotted temperature program corresponds to nonisothermally crystallized sample from the melt

The isothermal crystallization behavior of self-nucleated PEEK was also investigated. Following the steps 1–3 (see Fig. 1), the nonisothermally pre-crystallized sample from the melt was obtained. Then, it was heated at  $10 \text{ K min}^{-1}$  to  $350 \text{ °C}$  (step 4) and held for 5 min to create self-nucleated sites (step 5). Finally, the sample was cooled rapidly to the selected crystallization temperature ( $T_c$ ), and the DSC traces were recorded during the isothermal process. The  $T_c$  was selected based on a rule that it could give a reliable crystallization exotherm while the completion of crystallization did not take too long. As a reference, the isothermal crystallization behavior of PEEK without pre-existing nuclei was also investigated. The samples were heated to  $400 \text{ °C}$  at  $20 \text{ K min}^{-1}$  and held for 5 min to erase previous thermal history. Then, they were cooled rapidly to the selected  $T_c$ , and the DSC traces were recorded during the isothermal process.

## Results and discussion

### Melting temperature as a function of pre-crystallization conditions

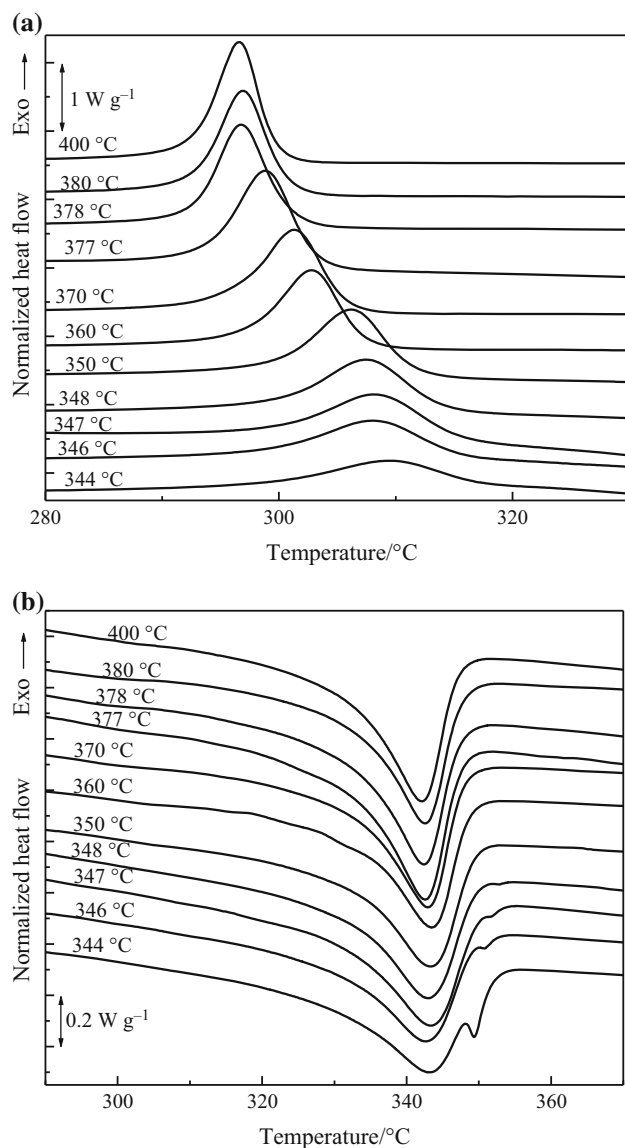
As a start, we examine first the melting behavior of PEEK samples prepared under various conditions. As shown in Fig. 2, PEEK pre-crystallized nonisothermally or isothermally from the melt presents a melting peak at  $342.0$  or  $346.8 \text{ °C}$ , respectively. For PEEK pre-crystallized nonisothermally or isothermally from the glass, its melting peak appears at  $341.9$  or  $342.3 \text{ °C}$ , respectively. Obviously, the melting curve is broader for the samples pre-crystallized from the glassy state.



**Fig. 2** DSC heating scans (at  $10 \text{ K min}^{-1}$ ) of PEEK samples pre-crystallized (a) nonisothermally and (b) isothermally from the melt, and (c) nonisothermally and (d) isothermally from the glass, respectively. The self-nucleation range of each sample is also indicated. Note that the curves are only shifted for clarity reason

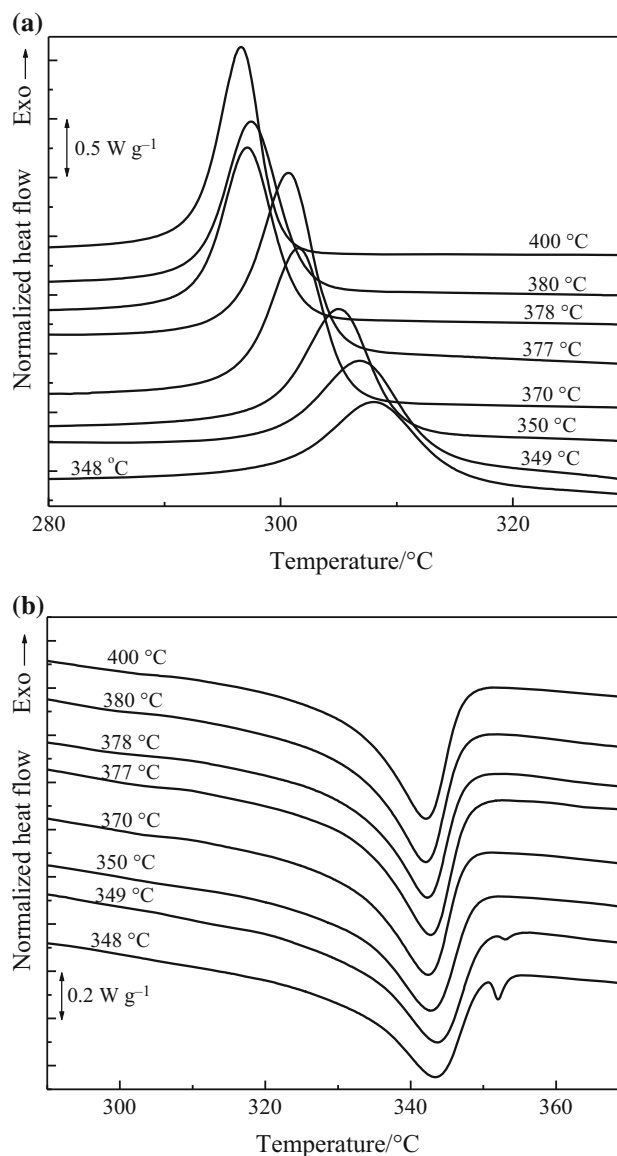
### Self-nucleation domains as a function of pre-crystallization conditions

The above comparison of melting peak temperatures deals with only one aspect of the melting behavior of the polymers. Comparison of the self-nucleation domains makes it possible to cover the broader spectrum of thermal stabilities of crystals present in any pre-crystallized PEEK sample. Two criteria have been used to define this nucleation range: the magnitude of the range and its position relative to the melting peak [13]. For the sample pre-crystallized from the melt nonisothermally, the DSC curves of self-nucleated PEEK are presented in Fig. 3. Figure 3a shows the cooling runs at  $10 \text{ K min}^{-1}$  after thermal conditioning at the indicated melting temperatures for 5 min, and Fig. 3b shows the subsequent heating runs at  $10 \text{ K min}^{-1}$ . After being melted at temperatures between  $400$  and  $378 \text{ °C}$ , all the DSC cooling profiles present one exothermic peak at around  $296.5 \text{ °C}$ , while the subsequent heating profiles reveal one melting peak also. This indicates that  $378 \text{ °C}$  is the lowest temperature at which complete melting occurs. Thermal conditioning at  $377 \text{ °C}$  was able to self-nucleate PEEK and, therefore, its crystallization temperature shifts to  $298.9 \text{ °C}$ . With further decrease in the melting temperature, its crystallization temperature shifts to higher levels and reaches  $307.5 \text{ °C}$  for a melting temperature of  $348 \text{ °C}$ . At a melting temperature of  $347 \text{ °C}$ , a weak crystallization peak and double melting peaks are present on cooling and subsequent heating, respectively. With further decrease in the melting temperature, its crystallization peak gets weaker on cooling. During the



**Fig. 3** DSC curves of PEEK pre-crystallized nonisothermally from the melt: **a** during cooling runs at  $10 \text{ K min}^{-1}$  after thermal conditioning at the indicated melting temperatures for 5 min, and **b** the subsequent heating runs at  $10 \text{ K min}^{-1}$ . Note that the nonisothermal pre-crystallization from the melt corresponds to a cooling at  $10 \text{ K min}^{-1}$  down to  $240 \text{ }^\circ\text{C}$ , the cooling run at  $10 \text{ K min}^{-1}$  after partial melting corresponds to step 6 (see Fig. 1), the partial melting is step 5 and the subsequent heating at  $10 \text{ K min}^{-1}$  is step 7. The curves are only shifted for clarity reason

subsequent heating process, the lower endotherm gets weaker while the higher one gets stronger. This can be explained as follows: the sample was not sufficiently melted at or below  $348 \text{ }^\circ\text{C}$  so that self-nucleation and annealing of un-melted crystals took place simultaneously. Similar multiple endothermic peaks after self-nucleation of PP [5, 9] and PLA [7] have been reported. These results clearly show that partial melting temperature  $348 \text{ }^\circ\text{C}$  corresponds to the highest achievable crystallization peak

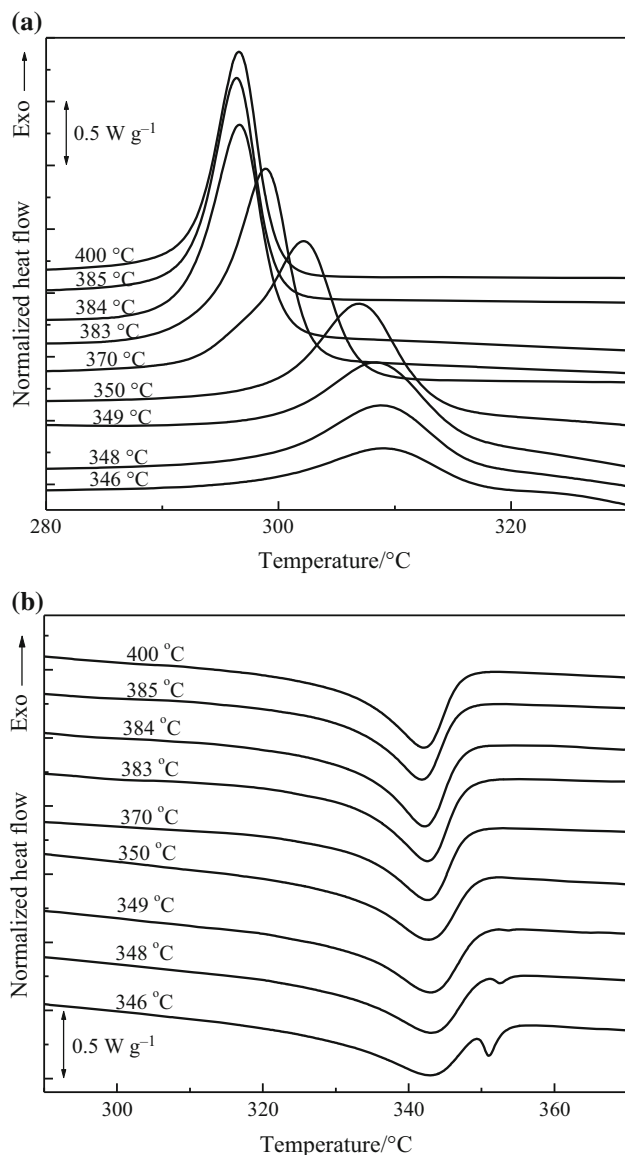


**Fig. 4** DSC curves of PEEK pre-crystallized isothermally from the melt: **a** during cooling runs at  $10 \text{ K min}^{-1}$  after thermal conditioning at the indicated melting temperatures for 5 min, and **b** the subsequent heating runs at  $10 \text{ K min}^{-1}$ . Note that the isothermal pre-crystallization from the melt corresponds to a rapid cooling down to  $320 \text{ }^\circ\text{C}$  and holding at the temperature for 30 min, the cooling run at  $10 \text{ K min}^{-1}$  after partial melting corresponds to step 6 (see Fig. 1), the partial melting is step 5 and the subsequent heating at  $10 \text{ K min}^{-1}$  is step 7. The curves are only shifted for clarity reason

temperature of PEEK during cooling. From above results, it can be concluded that, for PEEK pre-crystallized nonisothermally from the melt, the corresponding magnitude of the  $T_s$  range ( $348\text{--}378 \text{ }^\circ\text{C}$ ) can be obtained to be  $30 \text{ }^\circ\text{C}$  and the maximum peak shifts to higher temperature by  $11.0 \text{ }^\circ\text{C}$ .

For the samples pre-crystallized from other thermal conditions, the DSC curves of self-nucleated PEEK are presented in Figs. 4–6. For PEEK pre-crystallized

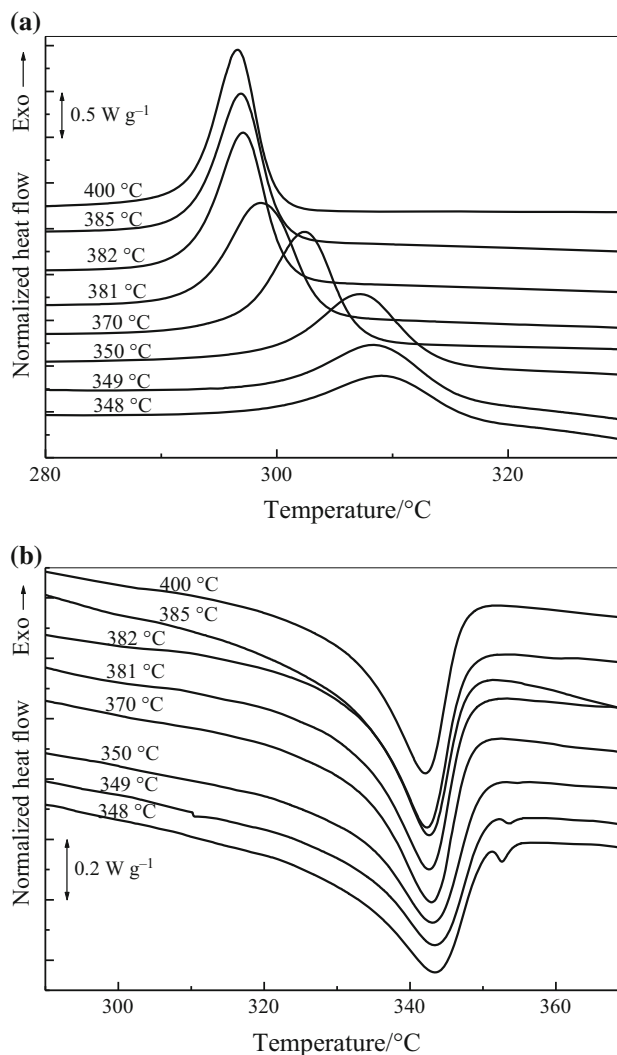
isothermally from the melt, the corresponding magnitude of the  $T_s$  range (350–378 °C) can be obtained to be 28 °C and the maximum peak shifts to higher temperature by 8.4 °C during subsequent cooling (Fig. 4). While for PEEK pre-crystallized nonisothermally from the glass, the magnitude of the  $T_s$  range (349–384 °C) is 35 °C and the maximum peak shifts to higher temperature by 11.8 °C during subsequent cooling (Fig. 5). And while for PEEK



**Fig. 5** DSC curves of PEEK pre-crystallized nonisothermally from the glass: **a** during cooling runs at  $10 \text{ K min}^{-1}$  after thermal conditioning at the indicated melting temperatures for 5 min, and **b** the subsequent heating runs at  $10 \text{ K min}^{-1}$ . Note that the nonisothermal pre-crystallization from the glass corresponds to quenching into ice water from the melt to obtain amorphous state and then reheating at  $10 \text{ K min}^{-1}$  to finish cold crystallization, the cooling run at  $10 \text{ K min}^{-1}$  after partial melting corresponds to step 6 (see Fig. 1), the partial melting is step 5 and the subsequent heating at  $10 \text{ K min}^{-1}$  is step 7. The curves are only shifted for clarity reason

pre-crystallized isothermally from the glass, the magnitude of the  $T_s$  range (350–382 °C) is 32 °C and the maximum peak shifts to higher temperature by 10.7 °C during subsequent cooling (Fig. 6). The  $T_s$  ranges of PEEK pre-crystallized at various conditions are also indicated in Fig. 2.

From above results, it can be concluded that, after partial melting, the maximum crystallization peak shifts to higher temperatures by 8.4–11.8 °C, indicating that self-nucleation significantly enhances the overall crystallization rate of PEEK. Moreover, the self-nucleation effect is more



**Fig. 6** DSC curves of PEEK pre-crystallized isothermally from the glass: **a** during cooling runs at  $10 \text{ K min}^{-1}$  after thermal conditioning at the indicated melting temperatures for 5 min, and **b** the subsequent heating runs at  $10 \text{ K min}^{-1}$ . Note that the isothermal pre-crystallization from the glass corresponds to quenching into ice water from the melt to obtain amorphous state and then holding at  $159 \text{ °C}$  for 30 min to finish cold crystallization, the cooling run at  $10 \text{ K min}^{-1}$  after partial melting corresponds to step 6 (see Fig. 1), the partial melting is step 5 and the subsequent heating at  $10 \text{ K min}^{-1}$  is step 7. Note that the curves are only shifted for clarity reason

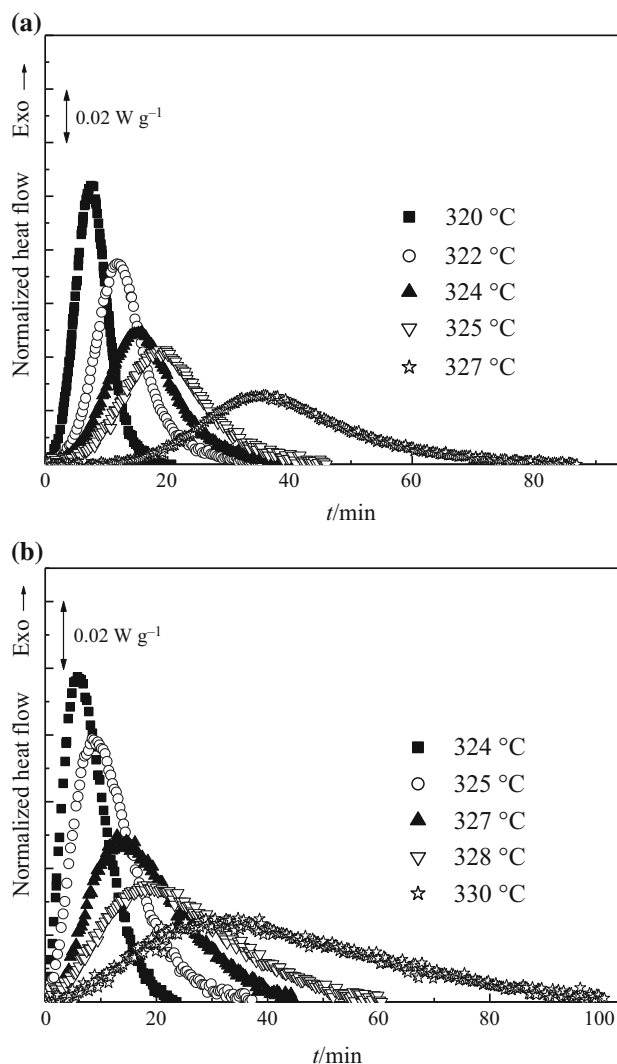
significant for the samples pre-crystallized nonisothermally or from the glassy state. On the other hand, the magnitude of the  $T_s$  range varies from 28 to 35 °C, depending on the pre-crystallization conditions. The range is larger for the samples pre-crystallized nonisothermally or from the glassy state. It is noteworthy that the magnitude of the  $T_s$  range is rather narrow for iPP (4 °C) and even more so for polyethylene (1.5 °C), and the domain is usually located between the high-temperature foot of the melting peak and its maximum [4, 5]. For PVDF, the  $T_s$  range can be larger (15 °C) and its upper limit is beyond the high-temperature foot of the melting curve [13]. Interestingly, the self-nucleation temperature domain of PEEK is way outside the melting curve, and its magnitude is greatly large (28–35 °C).

### Isothermal crystallization kinetics of self-nucleated PEEK

The crystallization kinetics of self-nucleated PEEK was investigated further. The samples melt-crystallized nonisothermally were melted partially at 350 °C for 5 min to create the self-nucleation site. Figure 7a, b presents the isothermal crystallization curves of PEEK at various crystallization temperatures ( $T_c$ ) after completely melted at 400 °C for 5 min and partially melted at 350 °C for 5 min, respectively. For both completely and partially melted PEEK samples, the subsequent isothermal crystallization exotherm gets broader with increasing  $T_c$ , although the investigated  $T_c$  range was much lower for the completely melted PEEK than for the partially melted one.

The relative degree of crystallinity  $X(t)$ , as a function of crystallization time  $t$ , can be calculated from the integrated area of the DSC curve from  $t = 0$  to  $t$  divided by the integrated area of the whole heat flow curve. Figure 8 shows the plots of  $X(t)$  as a function of  $t$  for completely and partially melted PEEK samples crystallized at indicated crystallization temperatures. An important parameter which can be taken directly from Fig. 8 is the halftime of crystallization  $t_{0.5}$ , which is the change in time from the onset of crystallization to the time at which  $X(t)$  is 50%. The values are summarized in Table 1. The  $t_{0.5}$  values increase with increasing  $T_c$  for both PEEK samples, indicating that the overall isothermal crystallization rate decreases with increasing  $T_c$ . Such results are reasonable since it is difficult for the samples to nucleate at high  $T_c$ , thereby resulting in the reduction in the overall crystallization rate. At the same  $T_c$ , the  $t_{0.5}$  value of PEEK with self-nucleation is much lower than that of the one without, indicating that self-nucleation accelerates the crystallization of PEEK.

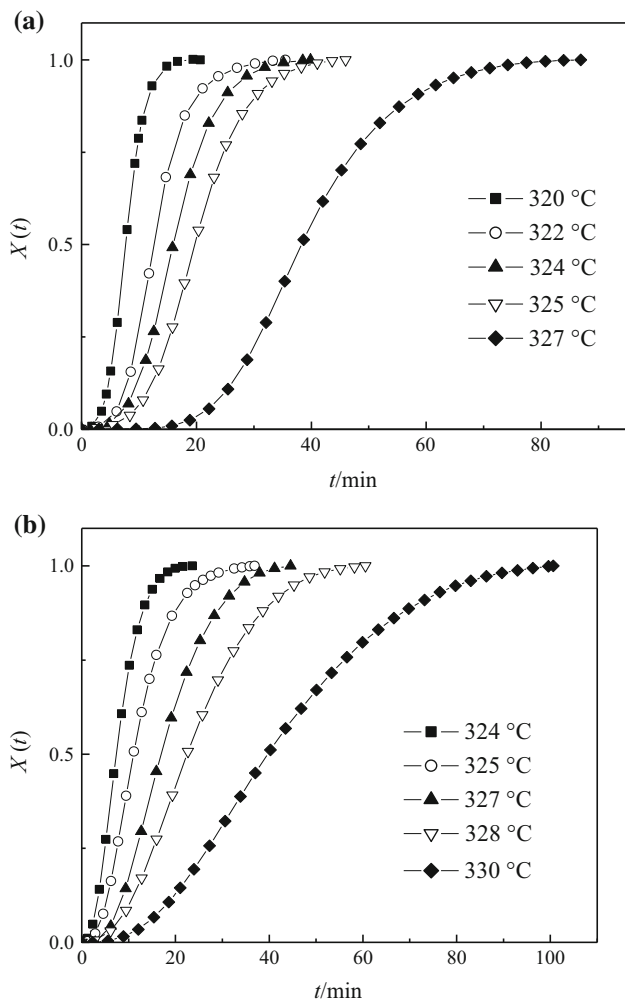
The well-known Avrami equation [37, 38] was used to describe the overall isothermal crystallization kinetics, which is shown as follows:



**Fig. 7** DSC exotherms of isothermal crystallization of PEEK at indicated  $T_c$ : **a** after melting at 400 °C for 5 min, and **b** after melting at 350 °C for 5 min

$$1 - X(t) = \exp(-kt^n) \quad (1)$$

where  $n$  is the Avrami index, and  $k$  is the overall rate constant associated with both nucleation and growth rate parameters. Figure 9 shows the double logarithmic plots of  $\log[-\ln(1 - X(t))]$  versus  $\log t$  for completely and partially melted PEEK samples crystallized at indicated crystallization temperatures. Each curve has a linear portion, most of which is followed by a gentle deviation at longer time. Usually, this deviation is considered to be due to the secondary crystallization, which is caused by the spherulite impingement in the later stage. By fitting the linear portion, the values of  $n$  and  $k$  can be obtained, which are included in Table 1. For PEEK without self-nucleation, the value of  $n$  ranges from 3.2 to 3.6 at  $T_c = 320$ – $325$  °C (with the exception that  $n = 5.1$  at  $T_c = 327$  °C), whereas it is from 2.4 to 2.8 at  $T_c = 324$ – $330$  °C for the self-

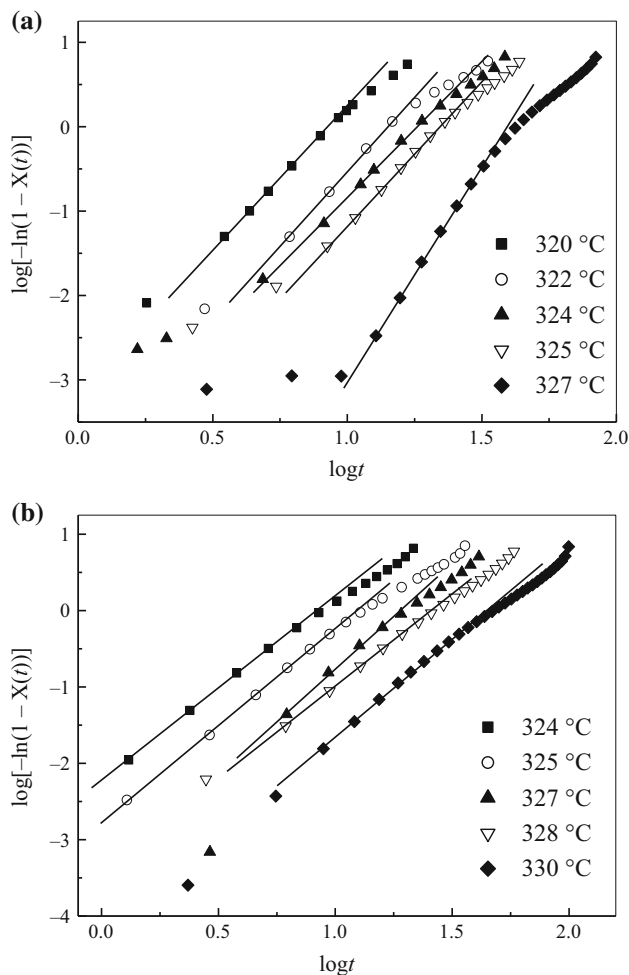


**Fig. 8** Variation of  $X(t)$  with crystallization time  $t$  of PEEK at indicated  $T_c$ 's: **a** after melting at 400 °C for 5 min, and **b** after melting at 350 °C for 5 min

**Table 1** Isothermal crystallization kinetic parameters based on the Avrami analysis

Sample	$T_c/^\circ\text{C}$	$n$	$k/\text{min}^{-n}$	$t_{0.5}/\text{min}$
PEEK after melted at 400 °C	320	3.3	$7.5 \times 10^{-4}$	7.7
	322	3.6	$7.4 \times 10^{-5}$	12.6
	324	3.2	$9.3 \times 10^{-5}$	15.8
	325	3.4	$2.5 \times 10^{-5}$	19.6
	327	5.1	$8.4 \times 10^{-9}$	38.2
PEEK after melted at 350 °C	324	2.4	$5.9 \times 10^{-3}$	7.5
	325	2.5	$1.7 \times 10^{-3}$	11.2
	327	2.8	$2.9 \times 10^{-4}$	17.0
	328	2.4	$3.8 \times 10^{-4}$	22.8
	330	2.6	$4.8 \times 10^{-5}$	39.6

nucleated one. Obviously, the value of  $n$  for partially melted PEEK is lower than that of the completely melted one, indicating that self-nucleation does change the



**Fig. 9** Plots of  $\log[-\ln(1 - X(t))]$  versus  $\log t$  for isothermal crystallization of PEEK at indicated  $T_c$ 's: **a** after melting at 400 °C for 5 min, and **b** after melting at 350 °C for 5 min. The solid lines are guides for the eyes

crystallization mechanism of PEEK. Similar conclusions have to be drawn for iPP [8, 39]. Moreover, it is reasonable to conclude that the crystallization of PEEK without self-nucleation is a homogeneous nucleation mechanism, while the one with self-nucleation is a heterogeneous nucleation mechanism.

### Conclusions

The influence of pre-crystallization conditions on the self-nucleation behavior of PEEK has been investigated. Samples that were pre-crystallized nonisothermally or from the glassy state exhibit a larger self-nucleation effect than the ones pre-crystallized isothermally or from the melt. Moreover, an interesting finding is that the self-nucleation temperature domain of PEEK is way outside the melting curve, and its magnitude is greatly large (28–35 °C). The

Avrami exponent for partially melted PEEK is lower than that of the completely melted one, indicating that self-nucleation changes the crystallization mechanism of PEEK from homogeneous nucleation to heterogeneous one.

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