Non-isothermal crystallization kinetics of UHMWPE composites filled by oligomer-modified CaCO$_3$

Zexiong Wu[1] · Zishou Zhang[1] · Kancheng Mai[1]

Abstract

The processability of ultrahigh molecular weight polyethylene (UHMWPE) improved by oligomer-modified calcium carbonate (CaCO$_3$) was observed in our previous work. In order to understand the effect of oligomer-modified CaCO$_3$ on the crystallization of UHMWPE, the non-isothermal crystallization behavior and crystallization kinetics of UHMWPE composites filled by oligomer-modified CaCO$_3$ was studied by differential scanning calorimetry in this work. Jeziorny and Mo methods were used to describe the non-isothermal crystallization kinetics of UHMWPE composites. The effect of modified filler content and cooling rate on the crystallization temperature and crystallization rate was discussed. The heterogeneous nucleation of modified CaCO$_3$ slightly increases the crystallization temperature of UHMWPE. The crystallization enthalpy of UHMWPE composites is significantly higher than that of UHMWPE. The crystallization rate of UHMWPE composites depends on the filler contents and cooling rate.

Keywords Non-isothermal crystallization kinetics · Ultrahigh molecular weight polyethylene · Oligomer-modified calcium carbonate · Crystallization

Introduction

Ultrahigh molecular weight polyethylene (UHMWPE) is an excellent thermoplastic crystalline plastic [1–4]. In comparison with other polyethylene, UHMWPE has lower coefficient of friction [5–7], higher mechanical strength [8, 9] and abrasion resistance [10–12]. Thus, UHMWPE has a wider foreground of application for pipeline industry [13, 14], biological medicine [15–17], textile industry [18, 19] and so on. Nevertheless, UHMWPE has extremely high molecular weight so that it is difficult to be processed by common processing method. Aim at this situation, a lot of researchers prepared a series of UHMWPE-based compounds to improve its performance [9, 20–24]. Meanwhile, UHMWPE as a crystalline polymer, addition of other materials can change the crystallization property of UHMWPE, which can influence the UHMWPE performance [25–29]. Therefore, it is necessary to study the crystallization behavior of modified UHMWPE materials.

Sattari et al. [30] reported the non-isothermal crystallization and melting behavior of UHMWPE hybrid composites reinforced with short carbon fiber (SCF) and nano-SiO$_2$ particles. It is observed that adding SCF to UHMWPE increased the melting peak temperatures and the crystallinity of UHMWPE. The amounts of nano-SiO$_2$ had no obvious effect on melting temperatures of UHMWPE and decreased the crystallinity of UHMWPE. Ozawa–Avrami method can be used to describe the non-isothermal crystallization kinetics of the composites.

Liu et al. [31] prepared a novel antibacterial UHMWPE/chlorhexidine acetate (CA)—montmorillonite (MMT) composites and investigated the crystallization kinetics of UHMWPE, UHMWPE/MMT, UHMWPE/CA and UHMWPE/CA-MMT. It is observed that the CA and MMT

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exhibited strong heterogeneous nucleation to increase the crystallization temperatures of UHMWPE. Addition of CA lowered the melting temperature of UHMWPE due to its plastization. The results of crystallization kinetics indicated that the addition of CA decreased the crystallization rate and broadened the range of crystal growth temperature, while MMT increased the crystallization rate due to its heterogeneous nucleation.

Zhang et al. [32, 33] studied the non-isothermal crystallization kinetics of UHMWPE in liquid paraffin (LP) systems and used the Avrami method modified by Jeziorny and Mo to describe the influence of UHMWPE content and cooling rate on crystallization mechanism and spherulitic structure of UHMWPE. Addition of LP increased the crystallization rate of UHMWPE. The Avrami plots of UHMWPE and UHMWPE/LP blends show a good linearity. At the primary crystallization stage, it is found that the Avrami exponent n is variable around 5 and decreases slightly as the cooling rate decreases. High values of n_1 for UHMWPE and UHMWPE/LP blends may be generated from their high viscosities yielding a more complicated crystallization mechanism. In

addition, the extent of secondary crystallization increases with increasing cooling rate. The Avrami exponent n_2, ranging from 0.80 to 1.96, indicated the simpler crystallization mode. Further, the value of $F(T)$ in the Mo method increased with an increase in relative crystallinity and UHMWPE content in the blends.

Shen et al. [34] researched the influence of compatibilizer and dispersant on the non-isothermal crystallization behavior of HDPE/UHMWPE/hydroxyapatite blends. Analyzed by Jeziorny method, the value of Avrami exponent n is between 2.7 and 3.5. At the same temperature, adding compatibilizer and dispersant increased the crystallization rate, while decreased the half crystallization time and the activation energy of crystallization. In addition, the compatibilizer and dispersant also improved the mechanical properties of blends.

Except as mentioned above, the effect of UHMWPE on the crystallization behavior and crystallization kinetics of HDPE has been reported [13, 35]. Nevertheless, fewer investigation focus on the crystallization behavior and the non-isothermal crystallization kinetics UHMWPE
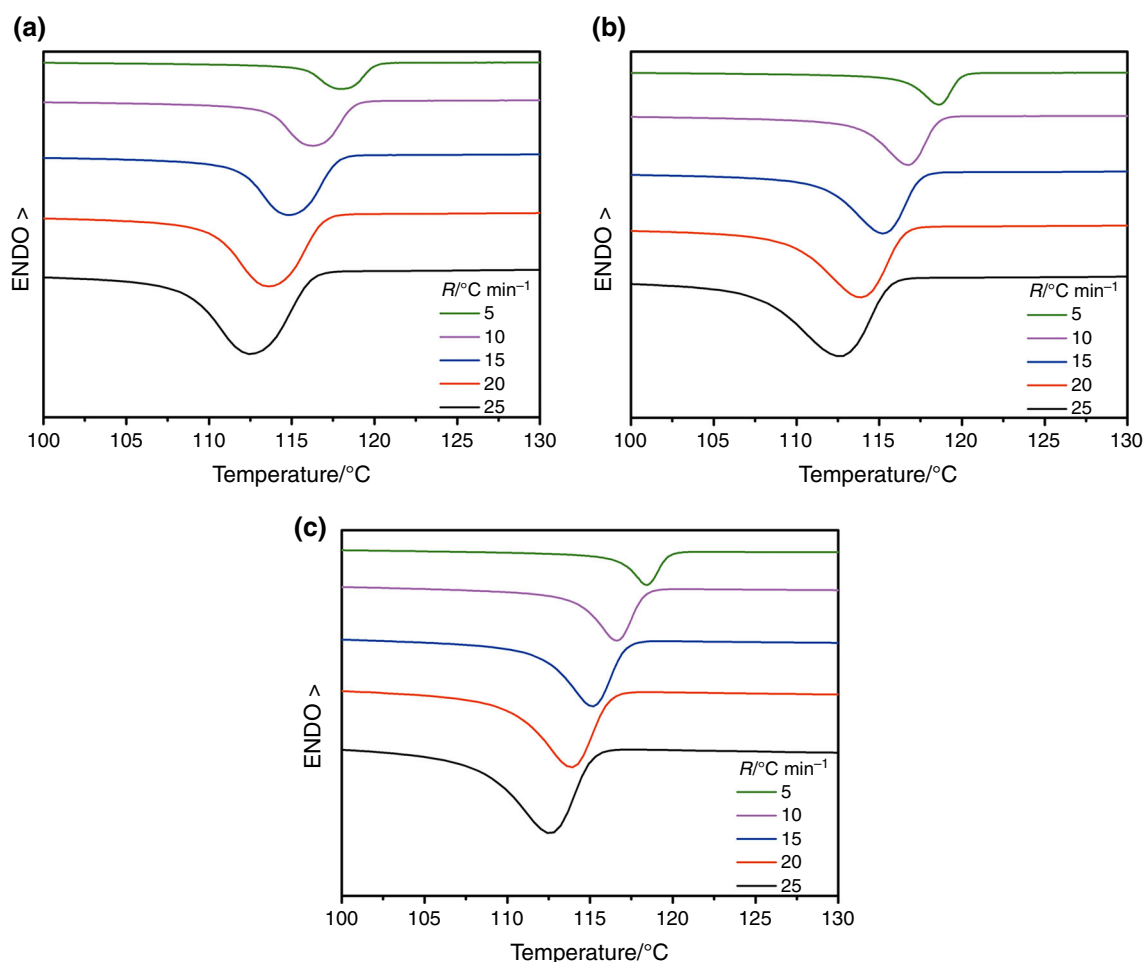

Fig. 1 DSC crystallization curves for PEW-g-CaCO₃/UHMWPE at different cooling rate PEW-g-CaCO₃/wt%: **a** 0, **b** 10 and **c** 20
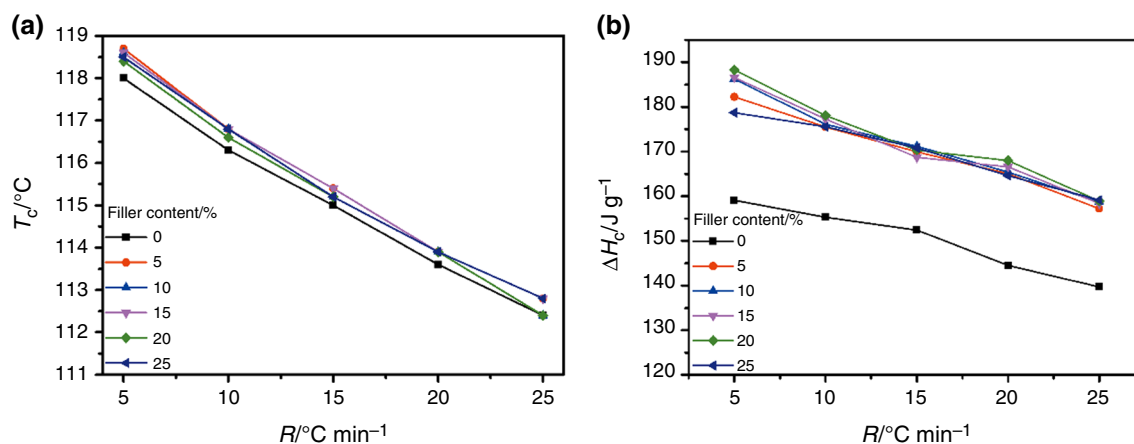
Fig. 2 Plots of crystallization peak temperature and crystallization enthalpy versus cooling rate
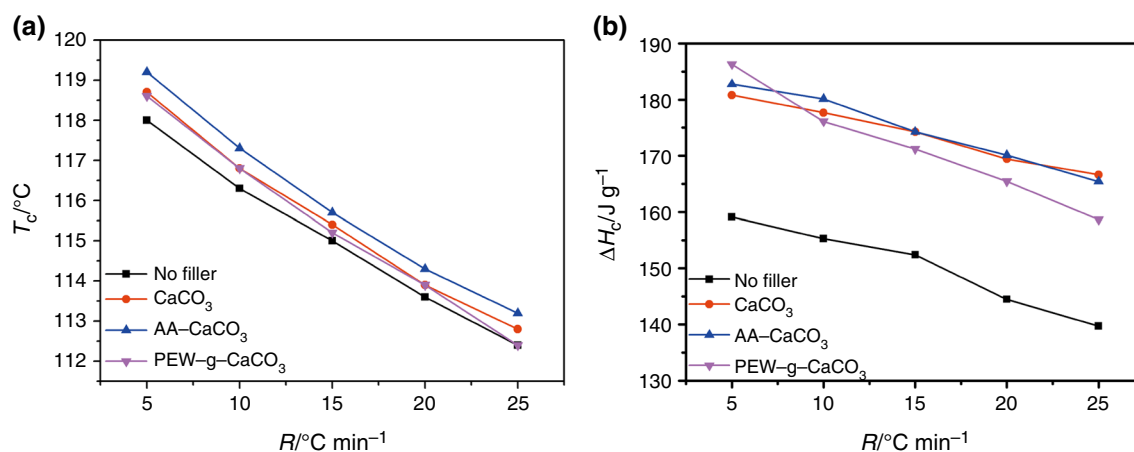

Fig. 3 Plots of crystallization peak temperatures and crystallization enthalpy versus cooling rate for UHMWPE composites filled by 10% filler

composites filled by inorganic particles. In our previous works, a new UHMWPE composite filled by oligomer-modified $CaCO_3$ was prepared in our laboratory. It is found that our prepared oligomer-modified $CaCO_3$ can improve the processability of UHMWPE [36]. In order to understand the effect of oligomer-modified $CaCO_3$ on the crystallization of UHMWPE, the crystallization behavior of UHMWPE composites was investigated by DSC, while the non-isothermal crystallization kinetics was described by different kinetic models. The influence of the modified filler on the UHMWPE crystallization was discussed in this article.

Experimental

Materials

UHMWPE (M_w 2.2×10^6) was purchased from Mitsui Chemicals, Japan. PE wax (Product No.1020, Mw about 2000–5000 and melt point at 116 °C) was provided by SCG, Thailand. Micro-$CaCO_3$ was provided by Keynes

nanomaterial (Lianzhou, China). Benzoyl peroxide (BPO) and acrylic acid (AA) were purchased from Damao Chemical (Tianjin, China).

Sample preparation

In order to obtain acrylic acid (AA)-modified calcium carbonate (AA-$CaCO_3$), 500 g $CaCO_3$ was added in 2.5 L ethyl alcohol at room temperature, then 40 g AA was added and stirred 20 min by mechanical stirring so that $CaCO_3$ reacted with AA thoroughly. Then, the solvent was volatilized at room temperature, and the product of AA-modified calcium carbonate (AA-$CaCO_3$) was dried in at 80 °C for 1 h.

The AA-$CaCO_3$, PE wax (mass ratio 8:2) and 0.5 mass% BPO (relative to $CaCO_3$) as initiator were mixed in mixer chamber at 150 °C for 10 min. The long chain-modified $CaCO_3$ (PEW-g-$CaCO_3$) was prepared due to reaction between PEW and AA. Composites of UHMWPE filled by different contents of PEW-g-$CaCO_3$ were prepared in mixer chamber at 190 °C for 10 min.
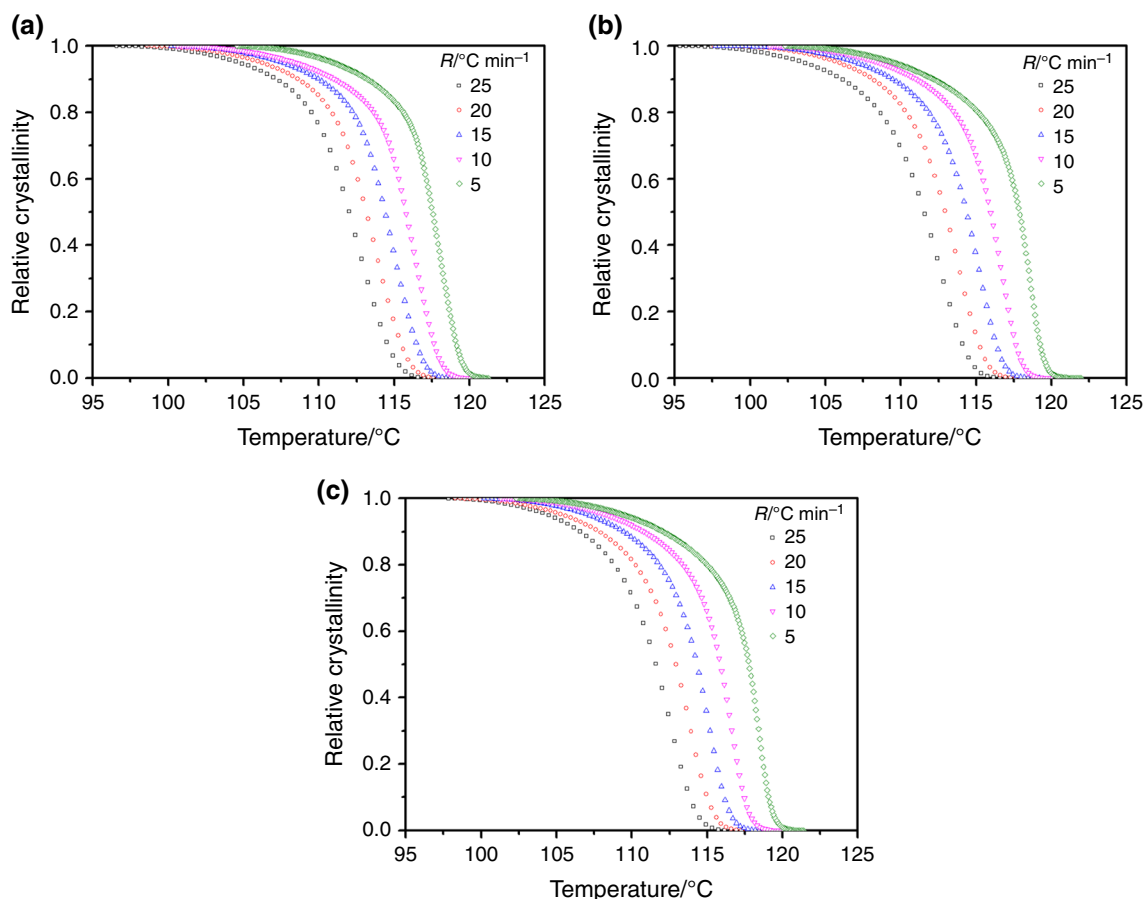
Fig. 4 Curves of relative crystallinity versus temperature of PEW-g-CaCO₃/UHMWPE PEW-g-CaCO₃/wt%: **a** 0, **b** 10 and **c** 20

Sample characterization

The melting and crystallization behavior of UHMWPE composites were measured by differential scanning calorimetry (DSC) at nitrogen atmosphere with the DSC-8500 (PE, America), calibrated with indium. About 2–3 mg sample was firstly heated up to 180 °C at the rate of 100 °C min^{-1} and kept this temperature for 3 min to eliminate the thermal history of sample. Then, the sample was cooled at the rate of 5, 10, 15, 20, 25 °C min^{-1} from 180 to 60 °C to obtain the non-isothermal crystallization curves. At last, the sample was reheated up to 180 °C at the rate of 10 °C min^{-1} to obtain the melting curves. The crystallinity of sample (X_c) is calculated by Eq. 1.

$$X_c\% = \frac{\Delta H_m}{\Delta H_m^0 \cdot w} \times 100\% \tag{1}$$

where ΔH_m is the melting enthalpy of sample measured by DSC, ΔH_m^0 is the melting enthalpy of 100% crystalline UHMWPE (290 J g^{-1}) [30] and w is the mass content of UHMWPE in the composites.

Results and discussion

Effect of modified-CaCO₃ on non-isothermal crystallization behavior of UHMWPE

Figure 1 shows the crystallization curves of UHMWPE composites cooled at different rates. The plots of crystallization peak temperatures and crystallization enthalpy versus cooling rate are shown in Fig. 2. It is observed that the crystallization peak of UHMWPE is shifted to the lower temperature and crystallization peak becomes wider with an increase in cooling rates. The crystallization peak temperature of UHMWPE composites is higher than that of UHMWPE, and the contents of PEW-g-CaCO₃ have little influence on the crystallization peak temperature of UHMWPE composites. The crystallization enthalpy of composites also decreases with an increase in cooling rate. At the same cooling rate, the crystallization enthalpy of composites is significantly higher than that of UHMWPE and increases with an increase in the PEW-g-CaCO₃ contents at cooling rate of 5 °C min^{-1}. However, the contents of PEW-g-CaCO₃ have little influence on the
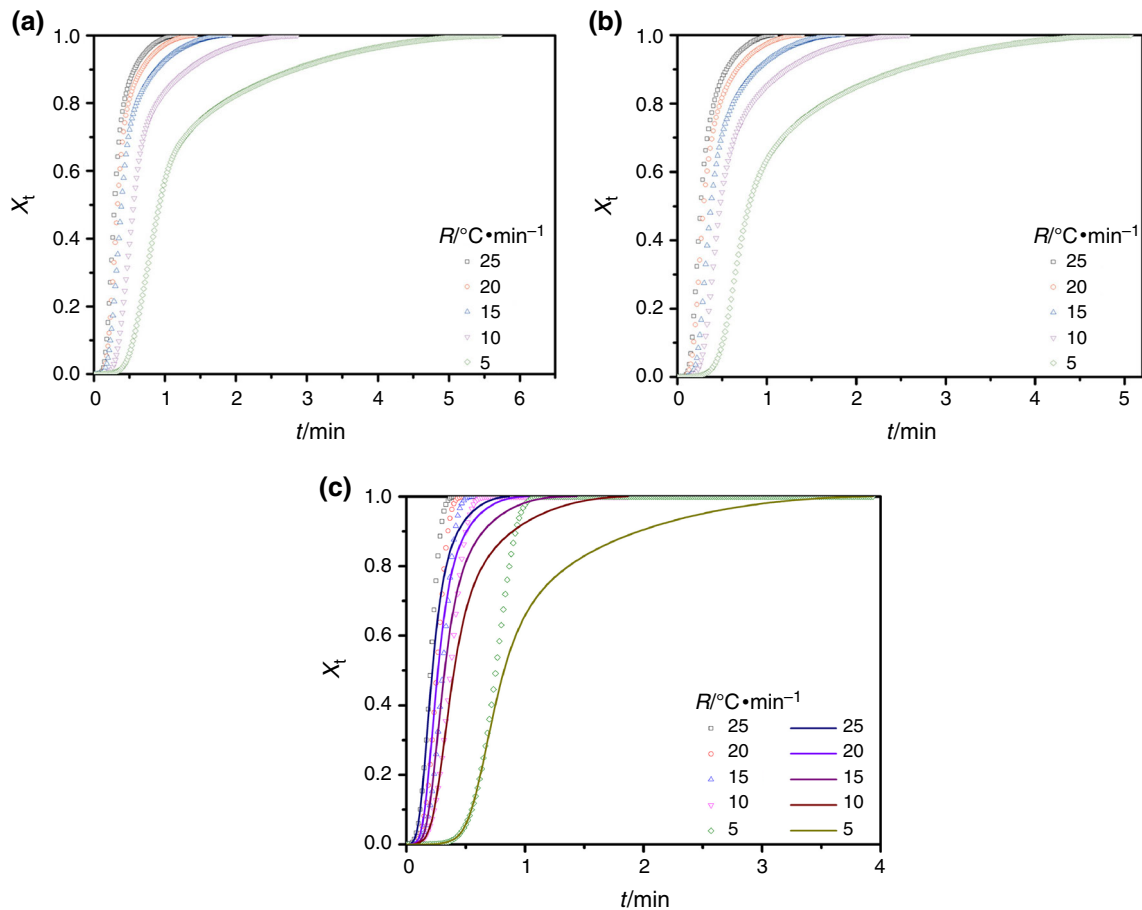
Fig. 5 Curves of relative crystallinity versus time of PEW-g-CaCO₃/UHMWPE PEW-g-CaCO₃/wt%: **a** 0, **b** 20 and **c** 10. Line: the experiment results, point: the kinetic calculation results

crystallization enthalpy of UHMWPE composites at high cooling rate.

The crystallization enthalpy of composites decreases with an increase in cooling rate, while crystallization peak becomes wider. It is suggested that the molecular chain of UHMWPE at higher cooling rate has poor mobility which is difficult to crystallize, resulting in a wider crystallization peak. At the same cooling rate, addition of modified filler increases the crystallization temperature of UHMWPE which indicates the presence of the heterogeneous nucleation of PEW-g-CaCO₃. The higher crystallization enthalpy of UHMWPE is attributed to improved mobility of molecular chain of UHMWPE by oligomer-modified CaCO₃.

In order to understand the effect of different modified-CaCO₃ on crystallization of UHMWPE, the non-isothermal crystallization behavior of UHMWPE composites filled by CaCO₃, AA-CaCO₃ and PEW-g-CaCO₃ are investigated by DSC. The plots of crystallization peak temperature and crystallization enthalpy versus cooling rate are shown in Fig. 3. It can be observed from Fig. 3 that the crystallization peak temperatures from high to low are AA-CaCO₃/

UHMWPE > PEW-g-CaCO₃/UHMWPE ≈ CaCO₃/UHMWPE > UHMWPE at the same cooling rate. It is indicted that the heterogeneous nucleation of AA-CaCO₃, PEW-g-CaCO₃ and CaCO₃ can increase the crystallization peak temperature of UHMWPE and the heterogeneous nucleation of AA-CaCO₃ is higher than that of PEW-g-CaCO₃ and CaCO₃. The crystallization enthalpy of UHMWPE composites is also higher than that of UHMWPE due to the heterogeneous nucleation of fillers. It can also be seen the crystallization enthalpy of PEW-g-CaCO₃/UHMWPE is higher than that of AA-CaCO₃/UHMWPE, CaCO₃/UHMWPE and UHMWPE at cooling rate of 5 °C min⁻¹. In our previous work [36], it is found that the oligomer-modified CaCO₃ can improve the processability of UHMWPE. It is suggested that the presence of oligomer in the surface of PEW-g-CaCO₃ is benefit to the mobility of molecular chain of UHMWPE to increase the degree of crystallization. However, the crystallization enthalpy of PEW-g-CaCO₃/UHMWPE is lower than that of AA-CaCO₃/UHMWPE and CaCO₃/UHMWPE at high cooling rate above 5 °C min⁻¹. It is considered that the mobility of molecular chain of UHMWPE is restricted in PEW-g-CaCO₃/UHMWPE at

Table 1 Kinetic parameters of UHMWPE and its composites from Jeziorny method

Filler/wt%	R/°C min^{-1}	$t_{1/2}$/min	n	Z_c
0	5	0.92	4.7	1.10
	10	0.56	5.3	1.37
	15	0.40	4.8	1.36
	20	0.33	5.3	1.35
	25	0.29	5.4	1.32
5	5	0.89	4.9	1.16
	10	0.53	4.9	1.37
	15	0.37	5.2	1.37
	20	0.32	5.0	1.32
	25	0.29	4.7	1.27
10	5	0.83	5.4	1.28
	10	0.47	4.9	1.47
	15	0.39	5.6	1.45
	20	0.31	5.2	1.37
	25	0.28	5.2	1.32
15	5	0.8	5.2	1.28
	10	0.48	4.8	1.46
	15	0.37	5.4	1.44
	20	0.30	4.9	1.36
	25	0.26	5.0	1.32
20	5	0.80	5.0	1.26
	10	0.48	5.0	1.45
	15	0.37	5.3	1.41
	20	0.30	4.7	1.33
	25	0.26	5.0	1.31
25	5	0.91	4.6	1.11
	10	0.50	4.1	1.33
	15	0.39	4.5	1.32
	20	0.33	4.6	1.29
	25	0.29	4.8	1.27

high cooling rate and the heterogeneous nucleation of PEW-g-CaCO$_3$ is lower than that of AA-CaCO$_3$, resulting in the decrease in the crystallization enthalpy of PEW-g-CaCO$_3$/UHMWPE with an increase in cooling rate.

Effect of modified-CaCO$_3$ on non-isothermal crystallization kinetics of UHMWPE

Some models were used to describe the non-isothermal crystallization kinetics of polymer and its blends and composites, including Ozawa, Jeziorny and Mo models, etc. In our work, effect of modified-CaCO$_3$ on non-isothermal crystallization kinetics of UHMWPE was investigated by the Avrami theory modified by Jeziorny and Mo models.
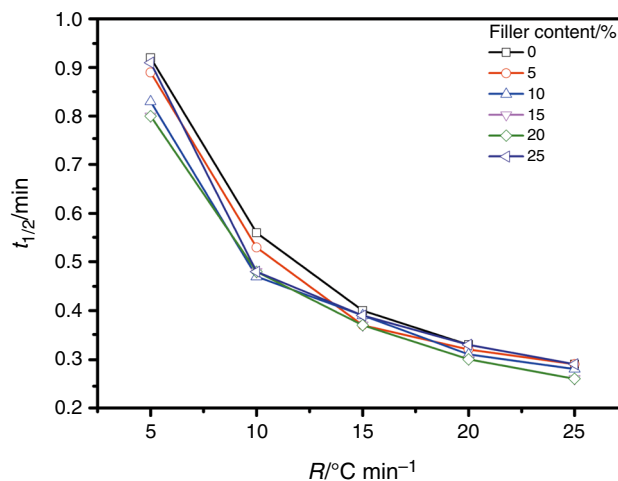
Fig. 6 Plots of half crystallization time versus cooling rate

Based on the Avrami theory modified by Jeziorny [33, 37, 38], the non-isothermal crystallization kinetics can be described as follows:

$$X_t = 1 - e^{-z_t t^n} \tag{2}$$

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

$$\lg Z_c = \frac{\lg Z_t}{R} \tag{4}$$

where n is the Avrami exponent, Z_t and Z_c are the Avrami and Jeziorny crystallization rate constants, respectively, R is the cooling rate, the time-dependent relative crystallinity, X_t, can be calculated by Eqs. 5 and 6.

$$X_T = \frac{\int_{T_0}^{T} \frac{d_H}{dT} dT}{\int_{T_0}^{T_\infty} \frac{d_H}{dT} dT} \tag{5}$$

$$X_t = \frac{\int_{t_0}^{t} \frac{d_H}{dt} dt}{\int_{t_0}^{t_\infty} \frac{d_H}{dt} dt} \tag{6}$$

where T_0 and t_0 are the crystallization onset temperature and time, T and t are the arbitrary crystallization temperature and time, T_∞ and t_∞ are the ultimate crystallization temperature and time, respectively, X_T is the temperature-dependent relative crystallinity. If $\lg[\ln(1 - X_t)]$ is linear on $\lg t$, the Avrami exponent n and the crystallization rate constant Z_t can be obtained by the slope and intercept. The crystallization rate constant Z_c can be calculated by Eq. (4).

Figures 4 and 5 are the representative curves of relative crystallinity of UHMWPE composites versus temperature or time, respectively. The plot of half crystallization time ($t_{1/2}$) (Table 1) obtained from Fig. 5 versus cooling rate is shown in Fig. 6. It can be seen that the half crystallization time for all samples decreases with an increase in cooling rate, indicating that accelerating the cooling rate can increase the crystallization rate so that the crystallization
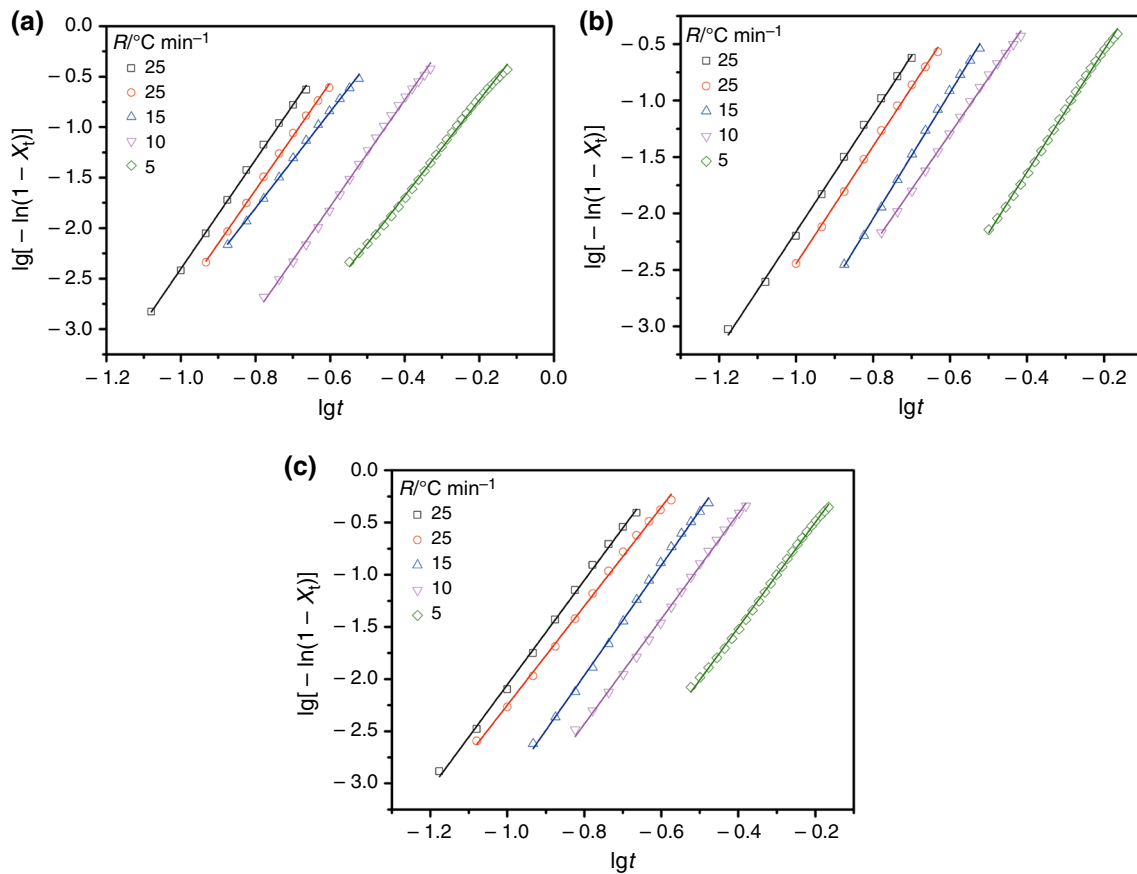
Fig. 7 Plots of $\lg[\ln(1 - X_t)] - \lg t$ for non-isothermal crystallization of PEW-g-CaCO₃/UHMWPE PEW-g-CaCO₃/wt%: **a** 0, **b** 10 and **c** 20
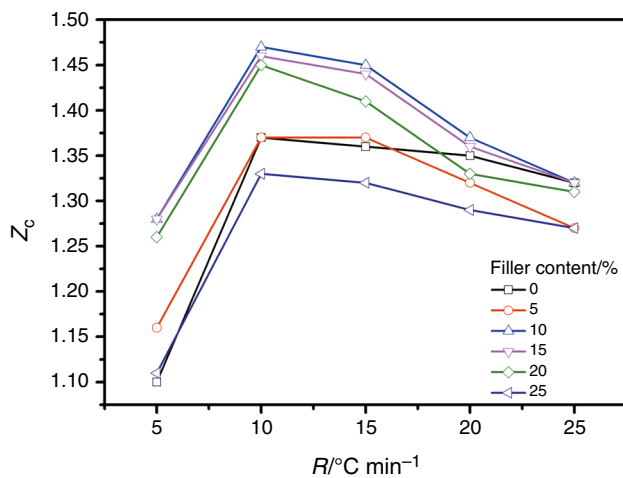

Fig. 8 Plots of crystallization rate constant versus cooling rate

time can be shortened. On the other hand, the half crystallization time of UHMWPE composites is lower than that of pure UHMWPE, indicating high crystallization rate in UHMWPE composites filled by PEW-g-CaCO₃.

According to Eq. (3), the plots of $\lg[\ln(1 - X_t)]$ versus $\lg t$ are showed in Fig. 7. A good linear relation means that Jeziorny method is suitable to describe the non-isothermal

crystallization kinetics of UHMWPE and its composites. The Avrami exponent n and crystallization rate constant Z_t obtained by the slope and intercept are shown in Table 1. It can be observed from Table 1 that the crystallization rate constant of all samples increases with accelerating the cooling rate from 5 to 10 °C min⁻¹ and then decreases with an increase in the cooling rate. The Avrami exponent n is about 5, which may be generated from their high viscosities, yielding a more complicated crystallization mechanism [32].

The relationship of crystallization rate constant of UHMWPE composites versus cooling rate is shown in Fig. 8. It can be seen that the crystallization rate constant of UHMWPE composites depends on the filler contents and cooling rate. At cooling rate of 10 °C min⁻¹, UHMWPE and its composites exhibit maximum crystallization rate. At cooling rate above 10 °C min⁻¹, the crystallization rate of UHMWPE decreases with an increase in cooling rate. At the filler content below 10 wt%, the crystallization rate of UHMWPE composites increases with an increase in filler contents. At the filler content above 10 wt%, the crystallization rate of UHMWPE composites decreases with an increase in filler contents. It is suggested that higher contents of filler and high cooling rate should hinder the
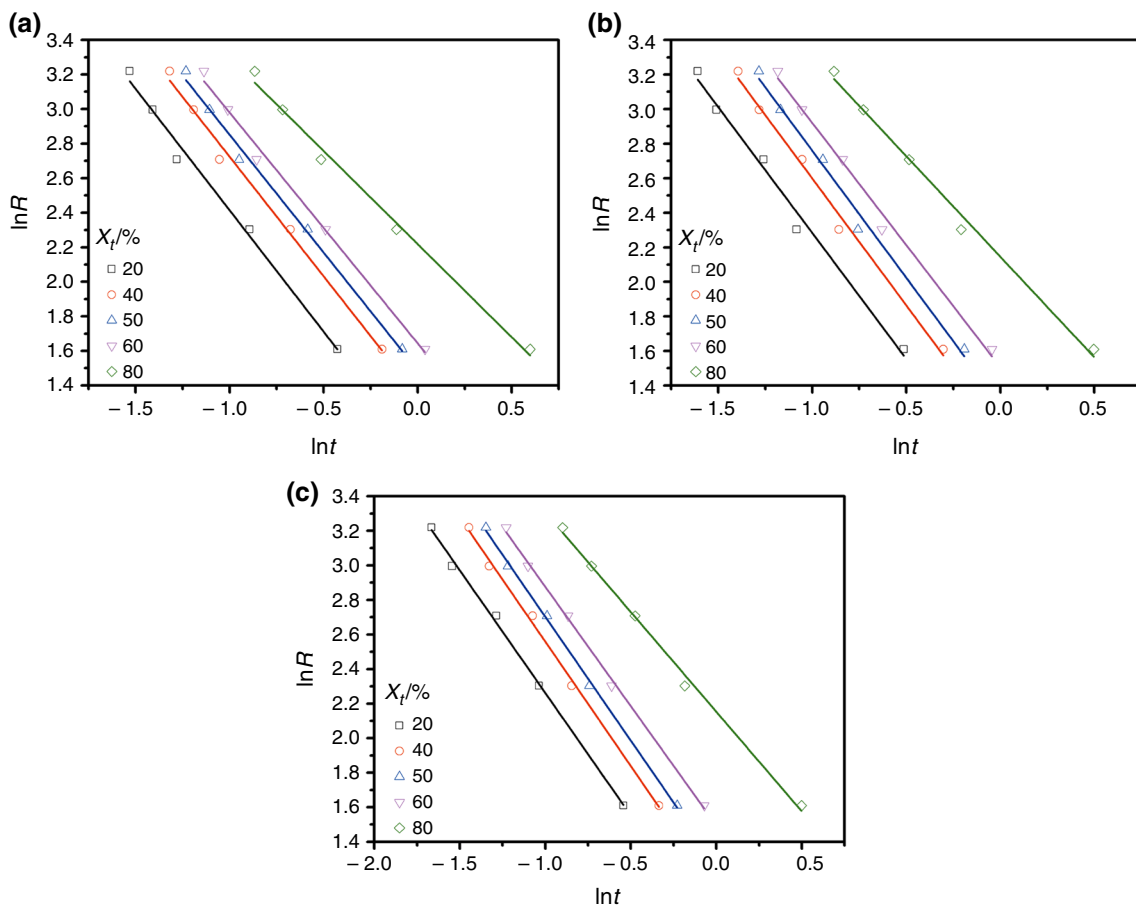
Fig. 9 Plots of $\lg R - \lg t$ for non-isothermal crystallization of PEW-g-CaCO$_3$/UHMWPE PEW-g-CaCO$_3$/wt%: **a** 0, **b** 10 and **c** 20
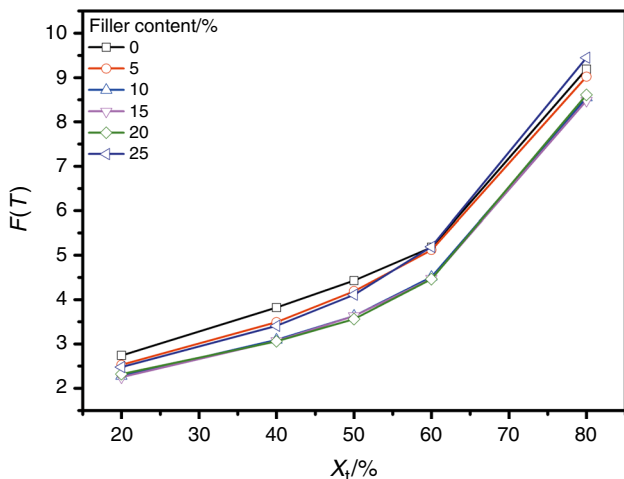

Fig. 10 Plots of $F(T)$ versus relative crystallinity

movement of molecular chain to decrease the crystallization rate of UHMWPE.

For the verification of the kinetic calculation results, we compare the experiment results with the calculation results by kinetic constants, shown in Fig. 5c. It can be found that the experiment results are agreed with the kinetic calculation results at relative crystallinity range of 0–50%. Above 50% relative crystallinity, the experiment results deviated with the kinetic calculation results may be due to the effect of second crystallization.

Mo method is generally used to investigate the non-isothermal crystallization kinetics of polymer and its composites [33, 37, 38].

$$\ln R = \ln F(T) - b \ln t \qquad (7)$$

where the R is the cooling rate, $F(T) = [k/Z_t]^{1/m}$ can be used to characterize the crystallization rate. The value of $F(T)$ is bigger, the crystallization rate is slower. If $\lg R$ is linear on $\lg t$, the values of $F(T)$ can be calculated by the intercept.

Figure 9 is the plots of $\lg R$ versus $\lg t$ for UHMWPE and its composites. The plots of $\lg R$ have a good linearity with $\lg t$, which means that Mo method is suitable to describe the non-isothermal crystallization kinetics of UHMWPE and its composites. The plots of value of $F(T)$ calculated by the intercept versus relative crystallinity is shown in Fig. 10. The value of $F(T)$ increases with an increase in relative crystallinity, indicating that the crystallization rate becomes slower in high crystallinity. The $F(T)$ valve of

pure UHMWPE is higher than those of all composites, also indicating that the PEW-g-CaCO$_3$ can accelerate the crystallization of UHMWPE. Besides, the lower $F(T)$ of UHMWPE composites filled by 10–20% PEW-g-CaCO$_3$ also indicated the higher crystallization rate, conforming to the conclusion obtained by Jeziorny method.

Conclusions

In order to improve the properties of UHMWPE composites filled by CaCO$_3$, oligomer-modified CaCO$_3$ was prepared and the effect of oligomer-modified CaCO$_3$ on the non-isothermal crystallization behavior and crystallization kinetics of UHMWPE was investigated by differential scanning calorimetry. The results indicated that the addition of oligomer-modified CaCO$_3$ increases the crystallization temperature and crystallization enthalpy of UHMWPE. It is attributed to the heterogeneous nucleation of oligomer-modified CaCO$_3$ and the improved mobility of molecular chain of UHMWPE by oligomer-modified CaCO$_3$. Jeziorny and Mo methods can be used to describe the non-isothermal crystallization kinetics of UHMWPE and its composites. The crystallization rate of UHMWPE composites depends on the filler contents and cooling rate. UHMWPE and its composites exhibit maximum crystallization rate at cooling rate of 10 °C min^{-1}. The crystallization rate of UHMWPE decreases with an increase in cooling rate at cooling rate above 10 °C min^{-1}. The crystallization rate of UHMWPE composites increases with an increase in filler contents at the filler content below 10 wt% and decreases with an increase in filler contents at the filler content above 10 wt%. The higher contents of filler and high cooling rate should hinder the movement of molecular chain to decrease the crystallization rate of UHMWPE.

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