Crystallization and mechanical properties of carbon nanotube/continuous carbon fiber/metalloocene polypropylene composites

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Abstract

In this work, a high fluidity polypropylene prepared with the metalloocene catalyst (mPP) was used as matrix, carbon nanotube (CNT) and continuous carbon fiber (CCF) were added to prepare composites, and their mechanical properties, melting and crystallization behavior were investigated. In the mechanical properties, the effects of tension force in the preparation process and compatibilizer maleic anhydride grafted polypropylene (MAPP) on the tensile strength of the composites were researched. The results show that the tensile strength of the composites increases first and then decreases with the increase of tractive force. In addition, the melting and crystallization behaviors and dynamic mechanical behaviors of mPP, CNT/mPP and CNT/CCF/mPP composites were characterized and studied by a differential scanning calorimetry (DSC) and dynamic mechanical analyzer (DMA). The results show that the melting point ($T_m$), crystallization temperature ($T_c$) and storage modulus ($E'$) of CNT/mPP are all increased by adding 1 wt% CNT, especially the $T_c$ is increased by 8.8 °C. It shows that after CNT was added to mPP as inorganic carbon material, it plays a prominent role in heterogeneous nucleation. After CCF was composited with CNT/mPP, the composites with CCF content of 30 and 42 wt% were prepared, and their $T_m$, $T_c$, crystallinity ($X_c$) and $E'$ were all improved, especially $E'$ was greatly improved, such as the initial $E'$ was increased by 5.64 and 11.74 times. Even at the end of the curve, the $E'$ of the composites with CF is still significantly higher than that of mPP and CNT/mPP. It indicates that adding CCF will greatly improved the deformation resistance and load deformation temperature of mPP.

1. Introduction

Polypropylene (PP) is one of the most widely used commercial thermoplastics due to its heat resistance, good mechanical properties and economy [1]. Since the birth of the first 6 kt/a industrial device in 1957, PP has become the second-largest amount of plastics in the world, and has been widely used. Almost all isotactic polypropylene (iPP) currently on the market is based on so-called Ziegler–Natta catalysts developed by Giulio Natta in 1954 [2]. However, the multiple active sites of Ziegler–Natta catalysts lead to broad molecular weight distributions (MWD). Conversely, metallocene catalysts having a single active site, can polymerize iPP with a narrower MWD [3]. The narrower MWD is very advantageous for the production of high-quality PP fiber [4]. In addition, due to the limitation of the Ziegler-Natta catalyst, the molecular weight of PP produced is relatively large. When high melt mass flow rate (MFR) is required, such as PP spinning, thin-wall injection molding and melt-blown fabric etc, the PP for high MFR were usually produced by a peroxide-degradation process of polypropylene (PP), and that was controlled-rheology polypropylene (CRPP) [5–9]. However, the CRPP, after adding peroxide was increased the Peculiar smell. The manufactured auto parts installed on the car increased the
VOC content in the car. The metallocene catalyst synthesized PP has overcome this disadvantage. The hydrogen regulation can control its molecular weight extensively to obtain the desired resin with low molecular weight and high fluidity [10].

Thermoplastic composites (TPC) are rapidly emerging as alternative materials for auto parts owing to recyclability and excellent stiffness- and strength-to-weight ratios [11–13]. Due to strengthening environmental regulations, thermoplastic composites (TPCs) are actively conducted to develop recyclable automobile composite components. Polyolefins such as polypropylene (PP) have been commonly used for TPCs [14]. As one kind of TPC, the continuous fibers reinforced polymeric composites (CFRPCs) have gained much attention and are widely used in numerous fields such as automotive industries, aerospace industries, sports equipment and military due to the high chemical stability, excellent solvent resistance, remarkable mechanical properties, outstanding thermal stability and high toughness/damage tolerance, infinite shelf-life and ease of processing [15, 16].

PP is the most used variety in automotive plastics parts. Carbon fiber composites based on PP will have significant potential applications in the process of automobile lightweight. As the matrix of composites, the viscosity of resin (i.e., low molecular weight and a high MFR) is essential for the excellent impregnation rate [17]. Low viscosity PP is usually obtained by adding peroxide in the PP by Ziegler-Natta catalyst (ZN PP), which is inevitable to increase odor.

As a new carbon nanomaterial with excellent conductivity and mechanical properties [18], carbon nanotubes can significantly improve the conductivity, thermal conductivity and mechanical properties of thermoplastic resins [19, 20]. The properties of composites can also be improved by modifying the matrix of composites. In recent years, the conductivity [21], mechanical properties [22, 23], crystallization behavior and morphology [24, 25], rheological characteristics [25, 26] of carbon nanotube modified polypropylene were researched.

Over the past three decades, the preparation [27], interface [28, 29], welding [30], mechanical properties [31, 32], flame retardant properties [33] and stamping forming [34] of continuous glass fiber reinforced polypropylene composites were comprehensively and systematically researched. However, there were few studies on continuous carbon fiber reinforced polypropylene composites [12].

The previous work had studied the tensile properties, conductivity, thermal conductivity, storage modulus, melting point and crystallization temperature of CNT/CCF/PEEK composites prepared by the powder impregnation method with special engineering plastics peek as matrix, and it is found that CNT and CCF play a very obvious role [35, 36].

In this work, a high fluidity PP (MFR is 113 g/10 min) prepared by the metallocene catalyst (mPP) was used as the matrix to replace the traditional high MFR ZN PP prepared by adding peroxide, which is compounded with carbon nanotube (CNT) and continuous carbon fiber (CCF) to prepare CNT/CCF/mPP composites. The tensile properties, dynamic mechanical behavior, melting and crystallization behavior of the composites were researched, and the effect of CNT and CCF on properties of mPP matrix composites was investigated.

2. Experimental

2.1. Materials

Carbon fiber (CF) was produced by Toray Co., Ltd, Japan (commercial cord: T700SC-12k). The iPP (mPP powder) using metallocene catalysts was prepared by the Advanced Synthetic Materials Research Institute of Shandong Qinghe Chemical Technology Co., Ltd., China, as shown in table 1. High flow impact polypropylene copolymer (IPC) (Type K7780) was produced using Ziegler–Natta catalyst by , China, MFR was 80 g/10 min (2.16 kg). Multi walled carbon nanotube (MWNT) was produced by Shandong Exhibition Nanomaterials Co. Ltd, China, the grade was GT-210. Maleic anhydride grafted polypropylene (MA-g-PP) (grade: PP-G-1) was produced by Nanjing Deba Polymer Materials Co., Ltd, China. The antioxidants were produced in Suzhou

| Table 1. Preparation of the composites matrix. |
|---------------------------------------------|
| Number | Matrix (mPP powder) (wt%) | Antioxygen (wt%) | Carbon nanotube (wt%) | Process | Shap |
| CMP 0  | 100                      | —            | —                  | —       | Powder |
| CMP 1  | 99.6                     | 0.2          | 0.2                | —       | Dry mixing and Pelleting Pellets |
| CMP 2  | 98.6                     | 0.2          | 0.2                | 1       | Dry mixing and Pelleting Pellets |
Liangcai Chemical Co. Ltd, China, and the types were 1098 and 168, respectively. The isotactic polypropylene (powder) T30S was produced by SINOPEC Qilu Petrochemical Co. Ltd, China. Basic parameters of matrix resin were shown in table 1.

2.2. Preparation of prepreg tapes

2.2.1. mPP pellets

According to the ratio in table 1, mPP powder (CMP 0) with antioxidants 1010 and 168 were mixed dryly by a plastics high-speed blender for 3 min, the blender was produced by Dongguan Huanxin Machinery Co., Ltd,
China. And then these dry blends powder were put into a twin-screw extruder unit produced by Harbin Hapu Electric Technology Co., Ltd, China to extrude, cool in water and granulate to obtain mPP pellets (CMP 1), as shown in table 1. The basic physical properties and structural parameters are shown in table 2.

2.2.2. CNT/mPP pellets
According to the ratio in table 1, the CNT and the antioxidants 1010 and 168 were added in the mPP, the composites matrix CMP 2 with mPP, CNT and antioxidants was obtained by the same process as CMP 1, as illustrated in figure 1 and table 1.

2.2.3. Prepreg tape prepreg
The CNT/CCF/mPP and CCF/IPC prepreg tapes were prepared by a self-designed and assembled continuous fiber-reinforced thermoplastic composite melting laboratory impregnation device, flow chart and photos as shown in figure 2, figures 3(a) and (b). Yarn frame (Type ADC-2) was produced by Guangzhou Aosai Carbon Fiber Technology Co., Ltd, China. The single screw extruder with a screw diameter of 20 mm and three-roll calendar (CL-T110) were produced by Harbin Hapu Electric Technology Co., Ltd, China. The melt impregnation die was designed by ourselves.

After CNT/mPP pellets and compatibilizer maleic anhydride grafted polypropylene (MAPP) (the content was 8% of matrix CNT/mPP) were blended dryly, they were added into a single screw extruder for melting and plasticizing extrusion. The melt entered the impregnation mold chamber, and the fiber yarns were pulled over multiple pins, which were positioned in a chamber filled with the melt [37]. The fiber yarns were impregnated well with the melt, and the prepreg tape blank was pulled out and formed in the exit of the slit die of the mold, and press polished by a three-roll calendar. CNT/CCF/mPP prepreg tapes with smooth surfaces were obtained and finally coiled. The formula and preparation process conditions of the prepreg belt are shown in table 3. Among them, the thickness of the slit of the die and the content of carbon fiber in the prepreg corresponding to 0.3, 0.4 and 0.6 mm are 49, 42 and 30 wt% respectively.

Figures 3(a) and (b) are photos of CNT/CCF/mPP and CCF/IPC unidirectional prepreg tapes. As shown in the figure, CNT/CCF/mPP and CCF/IPC unidirectional prepreg tapes with flat surface were prepared using the self-designed and assembled laboratory assembly line for melt impregnation unidirectional prepreg tape preparation. It can also be seen that the surface of CNT/CCF/mPP prepreg tape is obviously smoother than that of CCF/IPC prepreg tape.

2.3. Measurements
2.3.1. Tensile strength
The tensile property was measured using universal material testing machine according to ASTM D3039/D3039M-14, the machine model WDW-20 was produced by Jilin Guanteng Automation Technology Co., Ltd. China, and the tensile speed was 10 mm min\(^{-1}\). The tensile strength of every sample was obtained by the average value of 5 effective data.

| Number | Matrix | Tension force (N) | \(V_d (r/min)\) | 0.3 | 0.4 | 0.6 |
|--------|--------|------------------|-----------------|-----|-----|-----|
| CCMP 1 | CMP 2  | 1                | 5               |     |     |     |
| CCMP 2 | CMP 2  | 5                | 5               |     |     |     |
| CCMP 3 | CMP 2  | 10               | 5               |     |     |     |
| CCMP 4 | CMP 2  | 15               | 5               |     |     |     |
| CCMP 5 | CMP 2  | 20               | 5               |     |     |     |
| CCMP 6 | CMP 2  | 25               | 5               |     |     |     |
| CCMP 7 | CMP 2  | 15               | 10              |     |     |     |
| CCMP 8 | CMP 2  | 15               | 15              |     |     |     |
| CCMP 9 | CMP 2  | 15               | 20              |     |     |     |
| CCMP 10| CMP 2  | 15               | 25              |     |     |     |
| CCMP 11| CMP 2  | 15               | 30              |     |     |     |
| CCMP 12| CMP 2  | 15               | 5               | √   |     |     |
| CCMP 13| CMP 2  | 15               | 15              |     |     |     |
| CCMP 14| CMP 2  | 15               | 5               | √   |     |     |
| CCMP 15| CMP 2  | 15               | 5               |     | √   |     |
| CCMP 16| CMP 2  | 15               | 5               |     |     | √   |

Table 3. Recipes and process conditions of composites.
2.3.2. Differential scanning calorimetry
The melting and crystallization behaviors of mPP, CNT/mPP and CNT/CCF/mPP composites were measured with a Netzsch differential scanning calorimeter model DSC 3500 Sirius. It took the weigh 5–8 mg of each sample, and did at a rate of 10 °C min⁻¹ in the temperature range of 50 °C to 180 °C and kept the temperature for 3 min in nitrogen atmosphere. Then, the crystallization curves of the material were obtained by cooling to 50 °C at a cooling rate of 10 °C min⁻¹; keep the temperature constant for 3 min, raised the temperature again to 220 °C at the heating rate of 10 °C min⁻¹, and recorded the melting curves of the materials.

2.3.3. Dynamic mechanical analysis
The dynamic mechanical behaviors of mPP, CNT/mPP and CNT/CCF/mPP composites were tested by a Netzsch dynamic mechanical analyzer model DMA 242E. Sample size was 40 × 4 mm, the frequency 1 Hz, the temperature range −60 °C to +100 °C, and the heating rate 3 °C min⁻¹.

3. Results and discussion

3.1. Melting and crystallization behaviors
3.1.1. Melting
Figure 4 shows the melting behavior curves of mPP, CNT/mPP and CNT/CCF/mPP composites. The results show that the melting point ($T_m$) increased by 152.6 °C from 151.0 °C after adding CNT to mPP. While the $T_m$ of CF composites (CF content of 30 wt% and 42 wt%) prepared with CNT/mPP as matrix were 153.8 °C and 154.0 °C respectively, increased by 1.2 °C and 1.4 °C. The results show that the melting point of the materials increases with the addition of CNT and CCF. This is because the added CNT played the role of heterogeneous nucleation in mPP, which could be filled in the vacancy of molecule, the free volume will be decreased [38].
When the composites were heated up, the CNT hindered the movement of mPP molecular chain, so the $T_m$ of CNT/mPP composites increases. When CCF was added to CNT/mPP, CCF also played the role of heterogeneous nucleation, and limited further the movement of molecular chain, and the $T_m$ of CNT/CCF/mPP composites increased ulteriorly [39].

Figure 5 shows the melting curves of CNT/CCF/mPP composites with three fiber contents and with or without compatibilizer. It can be seen that when no compatibilizer was added, the $T_m$ of the composites with three fiber contents were 153.6 °C, 153.5 °C and 153.3 °C respectively, and the difference was only 0.3 °C. After adding compatibilizer MAPP, the $T_m$ of the three composites were 154.0 °C, 153.8 °C and 154.2 °C respectively, and the difference was only 0.4 °C. It shows that within the above fiber content range, the fiber contents had a little effect on $T_m$. In addition, the difference between the average values of $T_m$ of the three composites with and without compatibilizer was only 0.53 °C. The results show that indicated the effect of compatibilizer on $T_m$ of the composites was also very small.

3.1.2. Crystallization
The calculation method of crystallinity ($X_c$) is shown in formula (1):

$$X_c(\%) = \frac{\Delta H_c}{(1 - W_C) \Delta H_f} \times 100\%$$  \hspace{1cm} (1)

Where: $\Delta H_c$: melting enthalpy of the materials; $\Delta H_f$: theoretical melting enthalpy of mPP, which was 317 J/g; $W_C$: mass fraction of CCF.

Figure 6 shows the crystallization behavior curves of mPP, CNT/mPP and CNT/CCF/mPP composites. It can be seen that after adding 1 wt% CNT to mPP, the crystallization temperature $T_c$ increased sharply from 110.1 °C to 118.9 °C, and the $X_c$ increased by 0.76 wt%. Using the above CNT/mPP as the matrix, the composites with CF content of 30 and 42 wt% were prepared, and their $T_c$ was increased by 0.8 and 1 °C.
respectively. It can be seen from table 1 that when 30, 42 and 49 wt% CCF were added, the \( X_c \) was increased by 2.23, 4.38 and 4.59 wt% respectively. The results show that after CNT and CCF as inorganic carbon materials were added to mPP, they play an obvious role of ‘heterogeneous nucleation’, the molecular chain of mPP was induced to crystallize earlier. Therefore, \( T_c \) and \( X_c \) were raised, as shown as table 4.

Figure 7 shows the crystallization curves of CNT/CCF/mPP composites with three fiber contents, and with or without compatibilizer. It can be seen that when no compatibilizer was added, the \( T_c \) of the composites with three fiber contents were 122.2 °C, 121.9 °C and 122.1 °C respectively, and the maximum difference was only 0.3 °C. After adding compatibilizer MAPP, the \( T_c \) of the three composites were 119.7 °C, 119.9 °C and 119.5 °C respectively, and the difference was only 0.4 °C. It shows that there was a little effect of the carbon fiber content on \( T_c \) within the above fiber content range. In addition, the difference of \( T_c \) corresponding to the presence or
absence of compatibilizer in the three CCF content composites was 2.5, 2.0 and 2.6 °C respectively, that is, the \( T_c \) of the three CCF content composites with compatibilizer was lower than that without compatibilizer.

### 3.2. Tensile properties

#### 3.2.1. Effect of tension force

Figure 8 shows the tensile strength curve of CNT/CCF/mPP prepreg tapes with the different tension force. It can be seen that the tensile strength of the composites increased gradually with the increase of tension force. When the tension force reached 15 N, the tensile strength was the maximum value (653.84 MPa). This is because the greater the tension force, the straighter the carbon fibers in the prepreg tapes were pulled, the easier it was to expand laterally on the impregnation axis, the better the impregnation, and the higher the tensile strength of the composites. When the tension force continued to increase, the tensile strength of the composites decreased significantly. When the tension force was 25 N, it decreased to the lowest value (593.27 MPa). This is because the tension force was too large and the carbon fibers were broken partly, which reduced the overall tensile strength of the composites. The greater the tension force, the more the carbon fiber broke.

#### 3.2.2. Effect of the compatibilizer

Figure 9 is a histogram of the effects of fiber content and compatibilizer on the tensile strength of the composites. It can be seen that with the increase of carbon fiber content, the tensile strength of the composites increased greatly with or without compatibilizer. After adding compatibilizer MA-g-PP, the tensile strength of the composites with three carbon fiber contents was higher than that without compatibilizer. The addition of MA-g-PP played a role in transferring load [40].

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**Figure 10.** Relationship curves between \( E' \) and temperature of mPP and CNT/mPP composites.

**Figure 11.** Relationship curves between \( E' \) and temperature of mPP, CNT/mPP and CNT/CCF/mPP composites.
3.3. Dynamic mechanical analysis

Figure 10 shows the curves of storage modulus ($E'$) with temperature for mPP and CNT/mPP composites. It can be seen that the change trend of the two curves was the same within the initial stage of $-65$ to $+11.5$ °C, and the curves coincided basically. The downward trend of mPP curve was obvious after $11.5$ °C, the two curves began to separate, and the CNT/mPP curve was on the top of the mPP until the end of the experiment at $99$ °C. This shows that after the addition of CNT, CNT did not work until the glass transition temperature $T_g$ was reached. After reaching $T_g$, the amorphous part in the mPP was completely thawed. With the increase of temperature, the intermolecular force decreased gradually, the fluidity of melt increased and the modulus decreased, so $E'$ descended significantly. After the addition of CNT, the movement of molecules for mPP was hindered, the viscosity increased, and the decrease of modulus was slowed down. Therefore, the decrease of $E'$ was slowed down.

Figure 11 shows the relationship between $E'$ and temperature of mPP, CNT/mPP and CNT/CCF/mPP composites. At the starting point of the curves, the $E'$ of the composites were greatly increased after adding CF, which was 5.64 and 11.74 times higher than that of pure mPP, respectively. Even at the end of the curves, the $E'$ of the composites with CF was still significantly higher than that of mPP and CNT/mPP. This is because the $E'$ of mPP as the carrier to transfer load decreased significantly with the increase of temperature, which resulted in a significant decrease in the $E'$ of the composites. However, due to the support of continuous CF, the $E'$ of the composites was significantly higher than that of mPP and CNT/mPP. The higher the CF content, the greater the $E'$ of the composites.

4. Conclusion

CNT/CCF/mPP prepreg tape with smooth surface was prepared using a high fluidity mPP as matrix. During the preparation of CNT/CCF/mPP composites, the tensile strength of the composites increased first and then decreased with the increase of tension force. The $T_m$, $T_c$ and $E'$ of CNT/mPP were increased with the addition of CNT, especially the $T_c$ was increased by 8.8 °C, and the effect of CNT on heterogeneous nucleation of mPP was very obvious. CCF reinforced CNT/mPP, the composites with CCF content of 30 and 42 wt% were prepared, and their $T_m$, $T_c$, crystallinity and $E'$ were increased, especially $E'$ was greatly improved, such as the initial $E'$ were 5.64 and 11.74 times higher than CNT/mPP. Even at the end of the curves, the $E'$ of the composites with CF were still significantly higher than that of mPP and CNT/mPP. It shows that the deformation resistance and the heat resistance of the materials will be greatly improved after adding CCF.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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