

Material Behaviour

Non-isothermal crystallization properties of polypropylene composites filled with multi-walled carbon nanotubes

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ABSTRACT

The influence of the filler size and content on the crystallization behavior of polypropylene (PP) composites filled with three multi-walled carbon nanotubes (MWCNTs) with different sizes was investigated by means of a differential scanning calorimetry (DSC). It was found that the values of the onset crystallization temperature, crystallization peak temperature and end crystallization temperature increased with increasing MWCNT weight fraction and with decreasing filler average diameter; the value of the crystalline degree of the composites increased slightly with increasing filler weight fraction. The values of the crystalline degree of the composites filled with MWCNTs with wide length-diameter ratio and small specific surface area were the lowest when the MWCNT weight fraction was lower than 4%, which can be attributed to weak interaction between the matrix and inclusions due to small specific surface area. Moreover, the total crystallization rate of the composites filled with a small amount of the MWCNTs could be significantly increased.

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1. Introduction

It is generally believed that crystallization properties including crystal type, crystal grain size and crystalline degree are related to the mechanical properties of crystalline polymeric materials, such as strength and impact fracture toughness. Polypropylene (PP) is a general thermoplastic resin, which is used in high volume because of advantages including easy of processing and recycling, low cost and good comprehensive properties. However, PP has some disadvantages such as high flammability, low strength and high notch sensitivity. For improving the mechanical properties including stiffness, strength and toughness, PP is usually filled with inorganic particles, such as calcium carbonate [1], glass beads [2,3], diatomite [4], Al(OH)₃ and Mg(OH)₂ [5], as well as clay [6]. Most of these research results showed that there were certain toughened and reinforced effects of these inorganic particles in PP resin.

Carbon nanometer tubes (CNTs) are considered to be tubes formed with a rolled-up graphene layer. CNTs possess some properties of general nano-materials, including excellent mechanical, electrical and thermal conductive properties due to its specific surface area and length to diameter ratio, in addition to the special

seamless nanometer tube structure. In general, CNTs can be divided into two types: single wall carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). Usually, the cost of SWCNTs is much higher than that of MWCNTs. The physical and mechanical properties of polymeric materials can be improved or some new properties can be obtained when they are filled with CNTs [7–9], such as electromagnetic field shielding and energy materials [10] and biocomposites [11]. Many results presented show that introduction of only a tiny amount of CNTs could change the flow properties [12] and crystallization [13,14], as well as enhance the thermal stability [15–17], electrical conductivity [18,19] and mechanical properties [20–23] of filled PP composites.

The introduction of inorganic particles could change the crystallization properties of filled semi-crystalline polymers such as polyethylene [24] and PP [25]. Bhattacharyya et al. [13] prepared PP/SWCNTs composites using the melt blending method and investigated the crystallization properties of the composites, and noted that the addition of the SWCNTs did not change the crystal type of the PP, but could become as a nucleating agent in the matrix to increase the crystalline rate of the composites, in which the size of spherules became smaller and had narrower distribution. In general, the crystallization properties of polymer composites are closely related to the content, shape and size of the inclusions, in addition to the dispersion of the filler in the matrix under given conditions. More recently, Liang et al. [26] studied the thermal

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properties and thermal stability of PP/MWCNT composites and found that the MWCNT could improve the thermal stability. The question then as to how does the size of CNTs affect the crystallization properties of polymer composites? However, there have been relatively a few comprehensive studies in this field, especially for polymer composites reinforced with MWCNTs. The objective of the present study is to investigate the effects of the content and size (e.g. diameter, length-diameter ratio, etc.) of MWCNTs on the non-isothermal crystallization behavior of the PP/MWCNTs composites, to provide useful data for development of MWCNTs reinforced PP composites.

2. Experimental

2.1. Materials

The polypropylene with trademark CJS-700 supplied by the Guangzhou Petrochemical Works in Guangdong province (Guangzhou, China) was used as the matrix material, and the density in the solid state and the melt flow rate were respectively 910 kg/m³ and 10 g/10min (230 °C, 2.16 kg).

Three types of multi-walled nanotubes (MWCNTs), designated respectively A, B, and C, were used as the fillers to investigate the effects of the size on the crystallization properties of the composite systems. These MWCNTs were supplied by the Chengdu Organic Chemical Co., Ltd. of the Chinese Academy of Sciences (Chengdu, China), and were prepared by means of the chemical vapor deposition method with a purity of greater than 90 wt%. Table 1 lists the main characteristics of the three MWCNTs. It can be seen that the average diameter of the three MWCNTs are arranged in the order of A < B < C, while the average length-diameter ratio of the three MWCNTs follows the order of B > A > C. Moreover, the specific surface area of the B type MWCNTs is the smallest.

Note: the data was provided by the Chengdu Organic Chemical Co., Ltd. (the Chinese Academy of Sciences).

2.2. Preparation

Firstly, the PP was separately mixed with the three MWCNTs in a high speed compounding machine, model GH-10, supplied by the Beijing Plastics Machinery (Beijing, China). Then, the PP/MWCNTs blends were melt-blended in a twin-screw extruder, (Model: SHJ-26) supplied by the Nanjing Chengmeng Machinery Ltd. Co. (Nanjing, China), at a screw speed of 100 rev/min and in a temperature range from 190 to 210 °C, to prepare the three PP/MWCNTs composites with designations of PP/A, PP/B and PP/C composite systems. Systems with MWCNTs weight fractions were 1, 2, 3, 4 and 5 wt% were prepared. The diameter and the length-diameter ratio of the extruder screw were respectively 26 mm and 40. The extrudate of the composites was granulated, and the granules were dried at 80 °C for five hours before testing.

2.3. Methodology

The crystallization properties of the PP/MWCNTs composites was measured using a differential scanning calorimeter (DSC),

Table 1
Characteristics of multi-walled carbon nanotubes.

MWCNTs type	d (nm)	L (μm)	L/d	ρ(g/cm ³)	Specific surface area (m ² /g)
A	8–15	30–50	2000–6250	2.1	250–300
B	10–20	20–100	1000–10000	2.1	100–200
C	≥50	10–20	≤400	2.1	>40

model DSC204C supplied by NETZCH Company (Selb, Bavaria, Germany). The test temperature range was from 30 to 200 °C, the heat preheat time was 5 min and the heating rate was 10 °C/min.

3. Results and discussion

3.1. DSC curves

DSC curves demonstrate a relationship between heat flow and temperature during heating or cooling of specimens. Figs. 1–3, respectively, present the DSC curves during cooling of the unfilled PP resin and the three PP/MWCNTs composite systems. It can be observed that the height and width, as well as position of the crystallization peak, vary with increasing MWCNTs weight fraction, and the area of the crystallization peak for the three PP/MWCNTs composite systems is more than that of the unfilled PP resin. Furthermore, the location of the crystallization peaks of the three composites moves to the right with reference peaks of the unfilled PP resin.

The area and location of the crystallization peak of a DSC curve presents the degree of crystallization of polymeric materials. The values of the area of the crystallization peak for the three composite systems are greater than that of the unfilled PP resin; this means

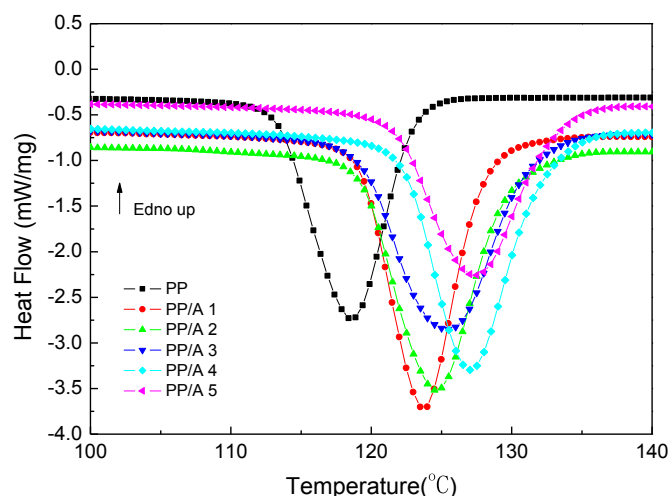


Fig. 1. DSC curves of PP and PP/A system.

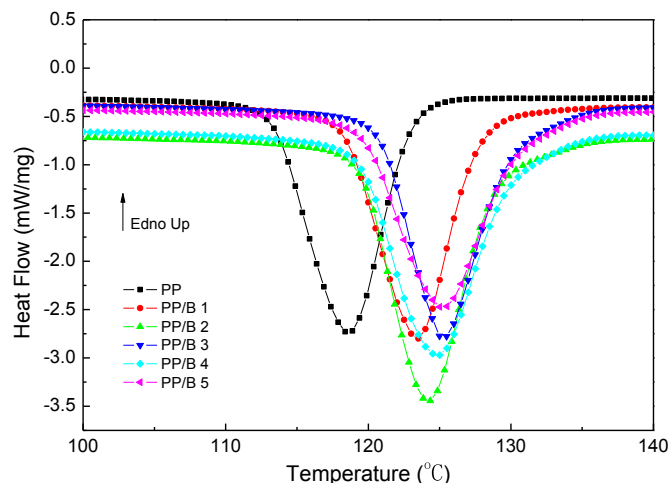


Fig. 2. DSC curves of PP and PP/B system.

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