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Material Behaviour

Non-isothermal crystallization kinetics of nylon 6/attapulgite nanocomposites

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ABSTRACT

The non-isothermal crystallization behaviour of nylon 6 (PA6)/attapulgite (AT) composites was studied by differential scanning calorimetry (DSC), which indicated that AT nanoparticles acted as nucleating agents during the crystallization of PA6/AT blends. The Avrami analysis modified by Mandelkern and the method combining Avrami and Ozawa equations were employed to analyze the non-isothermal crystallization behaviour. It was found that the Avrami analysis modified by Mandelkern was only valid for describing the middle stage of PA6 and PA6/AT nanocomposites crystallization. The method combining Avrami and Ozawa equations was able to satisfactorily describe the crystallization behaviour of PA6 and PA6/AT nanocomposites. In addition, X-ray diffraction (XRD) analysis showed that the addition of AT into nylon 6 did not change the crystalline form of nylon 6.

1. Introduction

Polymer/inorganic nanocomposites, wherein the inorganic components are dispersed on a nanometer scale and can, therefore, maximize their functions, have been explored extensively in recent years [1–3]. Attapulgite (AT) is a hydrated magnesium aluminum silicate present in nature as a fibrillar mineral [4,5]. The particles are approximate 20 nm in diameter and the length is from several hundred nanometers to several micrometers. The high aspect ratio of the AT fiber makes it extensively used in polymer composites [6–8]. Wang et al. studied the preparation and properties of polypropylene (PP) filled with modified attapulgite using silane [6]. Pan et al. prepared waterborne polyurethane/attapulgite nanocomposites [9]. Nylon 6/attapulgite nanocomposites were also reported by Subramani et al. [10], Pan et al. [7] and Shen et al. [11], etc. The results showed that the mechanical behaviour and thermal stability of nylon 6/attapulgite nanocomposites were improved to some extent. However, few studies reported the crystallization behaviour of nylon 6/attapulgite nanocomposites.

Filler particles can influence the flow and, hence, the crystallization behaviour and final morphology during processing [12]. The crystalline phase has a significant effect on physical properties of polymer/inorganic nanocomposites, such as mechanical and thermal properties [13]. Industrial nylon 6 composite processes are operated under dynamic, non-isothermal conditions. As a result, it is important to investigate the behaviour of nylon 6/attapulgite composites during non-isothermal crystallization. In this paper, the study focuses on the effect of attapulgite on the crystallization behaviour and kinetics of nylon 6/attapulgite composites under non-isothermal conditions from the melt by differential scanning calorimetry. Further, the structure and morphology of nylon 6 and nylon 6/attapulgite composites also are studied by X-ray diffraction and scanning electron microscopy.

2. Experimental

2.1. Materials

Nylon 6 (PA6) pellets used in this study were kindly supplied from Nanjing Julong Ltd. China. Attapulgite clay was provided by Xuyi Hongfei Company, China.

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2.2. Sample preparation

All materials were dried in an oven at 90 °C for 12 h before melt extrusion. PA6/AT nanocomposites were prepared by melt-mixing using a twin-screw extruder at a rotation speed of 60 rpm. The blending temperature profiles were 235/240/245/245/245/245/240 °C from hopper to die. Nanocomposites containing 0, 5% and 10% AT were prepared.

2.3. Differential scanning calorimetry

Non-isothermal crystallization behaviour was studied using a Perkin–Elmer Pyris-1 differential scanning calorimeter, with the temperature being calibrated with indium. All DSC measurements were performed under a nitrogen atmosphere. Sample weights were about 5 mg. The samples were heated at a rate of 20 °C/min to 250 °C, and allowed to stay for 5 min to remove all memory of previous thermal history, and then cooled to 50 °C to crystallize at different cooling rates of 5, 10, 15 and 20 °C/min. The exothermal curves of heat flow as a function of temperature were recorded to analyze the non-isothermal crystallization kinetics.

2.4. X-ray diffraction

The crystal structures of the pure nylon 6 and nylon 6/AT nanocomposites were examined by a Bruker D8 Advance X-ray diffractometer using Cu Kα radiation at ambient temperature. The scanning rate was set as 2°/min in the range from 5° to 70°.

2.5. Scanning electron microscopy (SEM)

The fracture surfaces of impact samples were plated with a thin layer of gold before scanning electron microscopy (SEM) observations. The SEM observations were then done using a LEO1530 VP SEM.

3. Results and discussion

3.1. Non-isothermal crystallization analysis

The non-isothermal crystallization exothermal curves of PA6 and PA6/AT nanocomposites at different cooling rates are shown in Fig. 1. It is found that the crystallization peak temperature (T_p) decreases with increasing cooling rate.

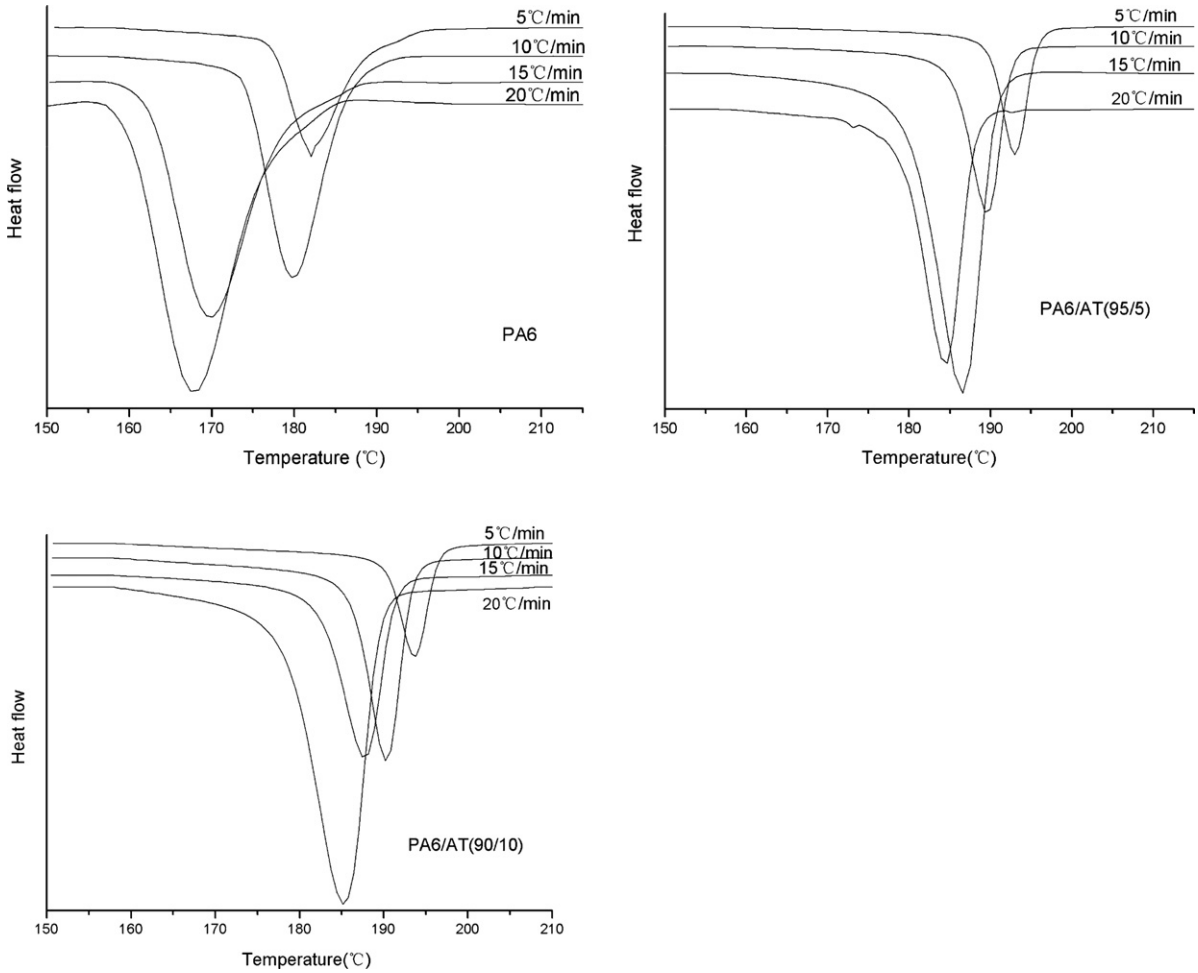

Fig. 1. DSC crystallization curves of PA6 and PA6/AT nanocomposites at various cooling rates.