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Carbon nanotubes induced brittle-ductile transition behavior of the polypropylene/ethylene-propylene-diene terpolymer blends

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ABSTRACT

Brittle-ductile transition is a well-known phenomenon in the elastomer-toughened polymer blends, and it is usually used to value the toughening efficiency of the elastomers. In this work, a small quantity of carbon nanotubes (CNTs) were introduced into the polypropylene/ethylene-propylene-diene terpolymer (PP/EPDM) blends to investigate the effect of CNTs on the brittle-ductile transition of the blends. The mechanical properties, morphologies and the crystallization behaviors of samples were comparatively investigated. The results showed that CNTs not only enhanced the impact strength of the materials but also promoted the occurrence of brittle-ductile transition at relatively low EPDM content. The crystallization and melting behaviors of PP matrix were not apparently influenced by CNTs, which could be attributed to the selective localization of CNTs in the EPDM component. Further results demonstrated that the toughening efficiency of CNTs in the blend composites was dependent upon the matrix ligament thickness (τ), and there was an appropriate range of τ (0.85–1.15 μm), at which CNTs exhibited high toughening efficiency. This work provides new insight on the toughening effect of CNTs in the immiscible polymer blends.

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1. Introduction

Introducing elastomers into brittle plastics has been demonstrated an efficient way to improve the impact strength of the materials. The toughening efficiency of elastomers is determined by many factors, such as the interfacial interaction between elastomers and matrix, the elastomer particle size and its distribution, the content of elastomers, the matrix ligament thickness between elastomer particles, and the inherent toughness of the matrix, etc. [1]. Generally, the impact strength of the elastomer-toughened blends increases with increasing elastomer content, and a brittle-ductile transition occurs at a certain amount of elastomer. So far, several toughening mechanisms have been proposed [2,3], such as the multiple crazing, shear yielding, rubber cavitation, matrix ligament thickness (τ), etc. Among these toughening mechanisms, the matrix ligament thickness which was proposed by Wu [4] has been widely accepted by researchers. It is suggested that there is a critical matrix ligament thickness (τ_c). If τ is smaller than τ_c, the material is tough, otherwise the material is brittle.

Studying the brittle-ductile transition of the elastomer-toughened blends is very significant. For example, if the brittle-ductile transition can be induced at relatively low elastomer content, the processing flowability of the materials cannot be impaired and the material cost can be maintained at relatively low level. Therefore, much work has been carried out to seek an appropriate way to induce the occurrence of the brittle-ductile transition at relatively low elastomer content. Through changing the crystalline structures of matrix, which can be achieved by adding nucleating agent, is demonstrated a simple strategy to influence the brittle-ductile transition of the elastomer-toughened blends [5]. The other strategy is related to adding nanoparticles into the elastomer-toughened blends [6]. If nanoparticles selectively locate in the elastomer component, elastomer and nanoparticles can form the core-shell structure [7]. In other scenario, nanoparticles selectively locate in the matrix but disperse around elastomer particles, once the particle-network structure is formed, the impact strength of the blend composites can be greatly enhanced [8]. Furthermore, it has also been demonstrated that the brittle-ductile transition of the elastomer-toughened blends can be influenced by other factors, such as the properties of the matrix [9], the volume of cavitation [10], and the notch radius [11], etc.

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Carbon nanotubes (CNTs) have been also demonstrated a novel impact modifier for brittle polymers, and the toughening mechanisms are suggested to be related to the bridging effect of CNTs in the crack surface and the enhanced stress transferring ability [12]. Different from the slight toughening effect in brittle polymers, the toughening effect of CNTs in the immiscible polymer blends are much more apparent [13]. Many researches have demonstrated that the impact strength of the blend composites increases with increasing CNT content [14–16], and the excellent toughening effect is mainly related to the percolated network structure of CNT which facilitates the homogeneous distribution of stress field in the sample under the impact load. Although much work has been carried out to investigate the toughening effect of CNTs on the immiscible polymer blends, the effect of CNTs on the brittle-ductile transition of the elastomer-toughened blends is still unclear.

In this work, a certain amount of CNTs (1 and 2 wt%) are introduced into the polypropylene (PP)/ethylene-propylene-diene terpolymer (EPDM) blends with variant EPDM contents. The effect of CNTs on the brittle-ductile transition of the blends is systematically investigated. The results demonstrate that the brittle-ductile transition of the PP/EDPM blends can be induced at relatively low EPDM content by adding CNTs.

2. Experiment

2.1. Materials

All the materials used in this work are commercially available. PP (trade name of 2401) was purchased from Beijing Yanshan Petrochemical Co. China. The melt flow rate of PP was 2.5 g/10min (230 °C/2.16 kg). EPDM (trade name of JSR EP35) was obtained from Japan Synthetic Rubber Co. The content of propylene in EPDM was 43%. CNTs (trade name of TNIMC4, with carboxyl groups of about 1.55 wt%) were obtained from Chengdu Institute of Organic Chemistry, Chinese Academy of Science (Chengdu, China). The length of a single CNT and the outer diameter of the CNT were 10–20 μm and 10–30 nm, respectively.

2.2. Sample preparation

The composites were prepared through a two-step processing procedures. First, EPDM and CNTs were melt-compounded using a twin screw extruder SHJ-30 (Nanjing Ruiya, China) to prepare a master batch with 10 wt% CNTs. The melt compounding was carried out at a screw speed of 140 rpm and melt temperatures of 150–160–170–180–190–190–185 °C from hopper to extruder head. After that, a certain amount of the master batch was further compounded with PP and EPDM to prepare the blend composites. Here, the content of EPDM in the blend composites was varied from 20 to 40 wt% while the contents of CNTs were 1 and 2 wt%, respectively. After being granulated, the pellets were injection-molded to obtain the specimens with a thickness of 4 mm and a width of 10 mm using an injection-molding machine EM80-V (Chen Hsong Machinery, China). The melt temperatures were set to 195–200–195 °C from hopper to nozzle while the mould temperature was 25 °C. The sample notation was defined as P*x*E*y*C*z*, where *x*, *y* and *z* represent the contents of PP, EPDM and CNTs, respectively. For comparison, the binary PP/EDPM blends were also prepared, and the sample notation was defined as P*x*E*y*.

2.3. Mechanical properties measurements and microstructure characterizations

Notched Izod impact strength was measured using an impact tester XC-22Z (Chengde Jinjian, China) according to ASTM D 256-04. Before measurements, a notch with a depth of 2.0 mm was prefabricated on the specimen and the ligament width was about 8.0 mm. Tensile measurements were conducted on a universal tensile machine AGS-J (SHIMADZU, China) according to ASTM D638. During the measurements, the gauge distance was set to 50 mm and a cross-head speed of 50 mm/min was used. Impact measurements were carried out at two different environmental temperatures, i.e. room temperature (23 °C) and low temperature (0 °C), while tensile measurements were carried out at 23 °C.

The impact-fractured surface morphologies, the morphologies of EPDM, and the dispersion of CNTs were characterized using a scanning electron microscope (SEM) FEI Inspect (FEI, the Netherlands). To characterize the morphology of EPDM, the sample was first cryogenically fractured in liquid nitrogen, and then the cryo-fractured surface was etched in *n*-heptane at 23 °C for 3 h. After being carefully washed using fresh *n*-heptane and ethanol, the treated surface was then characterized. The crystallization behaviors of samples were investigated using a differential scanning calorimeter (DSC) STA449C Jupiter (Netzsch, Germany) in the nitrogen atmosphere. During the measurements, a sample of 8 mg was heated from 30 to 200 °C at a heating rate of 10 °C/min and maintained at 200 °C for 3 min to erase any thermal history, then the sample was cooled down to 30 °C at a cooling rate of 5 °C/min. The degree of crystallinity (X_c) was calculated according to the following equation:

$$X_c = \frac{\Delta H_m}{\Delta H_m^0 \times \phi} \times 100\% \quad (1)$$

where, ΔH_m was the DSC measured value of fusion enthalpy, ΔH_m^0 was the fusion enthalpy of the completely crystalline PP, and ϕ was the relative weight fraction of PP in the samples. Here, ΔH_m^0 was taken as 207 J/g [17].

3. Results and discussion

3.1. CNTs induced brittle-ductile transition

Fig. 1a shows the variations of notched Izod impact strength of samples measured at room temperature (23 °C) *versus* EPDM content. As expected, the binary PP/EPDM blends exhibit increased impact strength with increasing EPDM content, and a brittle-ductile transition occurs at EPDM content of about 35 wt%. Interestingly, with incorporation of 1 wt% CNTs, higher impact strength is achieved for all the blend composites. Specifically, the P70E30C1 sample exhibits the impact strength of 36.6 kJ/m^2, which is much higher than the 13.4 kJ/m^2 of the P70E30 sample. This indicates that the brittle-ductile transition occurs at EPDM content of 30 wt%. When the content of CNTs is increased up to 2 wt%, although the P80E20C2 sample exhibits similar impact strength to those of the P80E20 and P80E20C1 samples and also the P60E40C2 sample exhibits similar impact strength to that of the P60E40C1 sample, largely enhanced impact strength is achieved for the P75W25C2, P70E30C2 and P65E35C2 samples. Specifically, the P75E25C2 sample exhibits the impact strength of 39.4 kJ/m^2. This indicates that the brittle-ductile transition of the blend composites with 2 wt % CNTs is induced at EPDM content of 25 wt%.

To further demonstrate the variation of brittle-ductile transition of the PP/EPDM blends induced by incorporating CNTs, the impact strength of the binary PP/EPDM and PP/EPDM blend composites with 2 wt% CNTs were also measured at environmental temperature of 0 °C. As shown in Fig. 1b, at EPDM content lower than 40 wt %, the impact strength of the binary blends is relatively small and the samples exhibit the brittle fracture mode. Specifically, the P65E35 sample exhibits the impact strength of 13.3 kJ/m^2, which is

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