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Morphology and Mechanical Properties of Nylon 6/PBT Blends Compatibilized with Styrene/Maleic Anhydride Copolymer*

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The mechanical properties and dynamic mechanical properties of blends composed of Nylon 6 and poly(butylenes terephthalate)(PBT), with styrene/maleic anhydride(SMA) as compatibilizer, were studied. The observation on the morphologies of the etched surfaces of the cryogenically fractured specimens *via* scanning electron microscopy(SEM) demonstrated that in the compatibilized Nylon 6/PBT blends, there exists a finer and more uniform dispersion induced by the *in-situ* interfacial chemical reactions during the preparation than that in the corresponding uncompatibilized blends. On the other hand, the overall mechanical properties of the compatibilized blends could be remarkably improved compared with those of the uncompatibilized ones. Moreover, increasing the amount of the compatibilizer SMA leads to a more efficient dispersion of the PBT phase in Nylon 6/PBT blends. Furthermore, there exists an optimum level of SMA added to achieve the maximum mechanical properties. As far as the mechanism of this reactive compatibilization is concerned, the enhanced interfacial adhesion is necessary to obtain improved dispersion, stable phase morphology, and better mechanical properties.

Keywords Nylon-6; PBT; Morphology; Dynamic mechanical property

Introduction

There have been some approaches employed to enhance the compatibility of the polymer blends, among which the formation of block or graft copolymers at the domain interface during processing by *in-situ* reaction of functional groups, *i. e.*, reactive compatibilization, has been considered to be an effective approach[1,2]. A polymer containing some functional groups like —OH, —COOH, and/or —NH_2 at the chain ends can react with another polymer bearing a reactive component such as maleic anhydride, acrylic acid, or epoxide. A compatibilized Nylon 6/PBT alloy offers certain advantages as the stiffness (modulus) and strength could be increased, depending on the composition ratio of the main constituents. On the other hand, the presence of PBT reduce not only the cost but also the moisture sensitivity of the final product. Kim *et al.*[3] studied the improvement of the toughness for PBT/Nylon 6 blends compatibilized by ethylene/vinyl acetate-*g*-maleic anhydride(EVA-*g*-MAH). Kamal *et al.*[4] reported that poly(ethylene terephthalate)(PET)/Nylon 66 blends are brittle even though the individual component is ductile failure, and owed the brittleness to the poor interfacial adhesion in those blends. Huang *et al.*[5] found that the epoxide end groups are able to react with the end groups of Nylon 66 and PBT *in-situ* to form a certain amount of the desired epoxy-co-PBT-co-Nylon 66 copolymer at the melt. Watanable *et al.*[6] reported that the blends of PBT and Nylon 6 are incompatible and pointed out that the compatibility between this pair of polymers could be enhanced *via* the addition of a copolymer, poly(styrene-maleic anhydride)(SMA), because SMA can react with both the amine end groups of Nylon 6 and hydroxyl end groups of PBT to form copolymers using the *in-situ* reactive extrusion. It is also assumed that *in-situ* copolymer can improve the interfacial adhesion between Nylon 6 and PBT and can control the dispersed-phase particle size, and consequently, may affect the strength and toughness of the blends. To our knowledge, few articles of the systematic investigation on the *in-situ* reactive compatibilization of the Nylon 6/PBT blends by SMA have been reported to date. This study is focused on the current industrial applications of compatibilized blends consisting of Nylon 6 with poly(butylenes terephthalate)(PBT). We attempted to probe the relationship between the morphology and mechanical properties of Nylon 6/PBT blends compatibilized by SMA copolymer.

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Experimental

1 Materials and Sample Preparation

Nylon 6 (1013B) was a commercial product of Ube Co. , Japan. A random SMA copolymer containing 24% (mass fraction) maleic anhydride from Sinopec Shanghai Petrochemical Corp. of China was used as the compatibilizer. PBT(S3130) was purchased from Engineering Plastics Plant of Yihua Group Corp. of China, contained 0. 02 mol/kg of carboxylic groups at its chain ends.

The preparation was conducted as follow. (1) Samples were prepared by extrusion in a twin-screw co-rotating extruder($L/D = 40$, $D = 40$ mm) at 250 ℃ and a screw speed of 100 r/min unless otherwise specified. All the materials were dried in a vacuum oven at 80 ℃ for at least 24 h before melt-mixing. (2) The specimens for mechanical properties were prepared by injection molding using a injection molding machine (CJ-80, Chende Plastics Machinery Co. , Ltd). The temperatures of cylinders 1, 2, 3 and nozzle were set at 220, 230, 240, and 250 ℃, respectively.

2 Measurements of Properties

The dynamic mechanical analysis(DMA) was conducted using a TA Q800 DMA(TA Instruments, USA) at a frequency of 1 Hz and a heating rate of 10 ℃/min in a temperature range of 0—175 ℃. The low-temperature measurement was performed in a stream of dry air cooled with liquid N_2, and the high-temperature measurement was carried out in a stream of dry N_2.

Izod impact tests were performed using a ZBC-4 Impact Pendulum(Shenzhen SANS, China) under ambient conditions according to ISO 179/96 standard. Tensile tests (ISO 527-1/93) were carried out at a crosshead speed of 50 mm/min with an Instron 4302 universal tensile machine(Instron, USA). Experimental errors were calculated from five specimens for impact and tensile tests. The average deviations based on five specimens were of 5%.

On the basis of the results of solubility tests, it has been found that SMA is soluble in boiling toluene, whereas Nylon 6 and PBT are insoluble. Nylon 6/PBT/SMA(mass ratio, 70 : 30 : 9) and Nylon 6/PBT(mass ratio, 70 : 30) blends were extracted with toluene. A total of approximately 30 g of each of the two blends was added respectively to 250 mL of toluene and stirred at 140 ℃ for five days. The suspensions were filtered off and the clear solutions obtained were precipitated with acetone, and consequently dried in a vacuum oven until constant weight of the samples reached. FTIR spectra were recorded on a Nicolet 710 FTIR spectrometer(Nicolet Instrument Corporation, United States) to confirm the grafting reaction.

The observation of the blends with the aid of scanning electron microscopy (SEM) was conducted using a KYKY-2800 microscope (KYKY Technology Development Ltd. , China) at an accelerating voltage of 25 keV. Samples for observing surface morphology were obtained from cryogenically fractured molded plaques as well as from the fractured Izod specimens. As the PBT phase of the blends was preferentially hydrolyzed by potassium hydroxide (KOH), the morphology of the blends was obtained by etching the surface PBT with a 10% solution of KOH in ethanol at room temperature, followed by careful washing of the surface with water. After being dried in a vacuum oven at room temperature, the surface of the specimens was coated with Au and used for the test.

A semi-automatic digital image analysis technique (Image Pro-plus software) was employed to determine the effective average diameters of the particles ($\bar{d}_n$) based on SEM photomicrographs. For the accurate statistical samplings, more than 200 particles from the multiple SEM photomicrographs were analyzed.

Thermal treatment was carried out at 100, 120, 140 and 160 ℃ for 6 h in an oven.

Results and Discussion

1 Characterization of *in-situ* Compatibilization

In general, a reactive compatibilizer is a copolymer containing reactive functional groups reactable with one or both of the blend components to form a block or a graft copolymer, and the *in-situ* copolymer tends to reside at the interface to compatibilize the components in the blend. The major chemical reaction responsible for this reactive compatibilized system involves the anhydride groups of SMA, the terminal amine groups of Nylon 6, and the hydroxyl groups of PBT. The reaction between amine and carboxyl end groups would take place at the interface of compatibilized Nylon 66/PBT blends[3], whereas the grafted maleic anhydride reacts with both the hydroxyl end groups of PBT[7] and the amine end groups of Nylon 6[8—11]. On the basis of the order of the reaction reported by Zhubanov[12] and Flory[13], it is assumed that in the presence of MAH, the order of the reaction rate of Nylon 6 and SMA is similar to that of PBT and SMA. Hence, the *in situ* copolymer is preferentially located at the interface to act as an effective compatibilizer.

Fig. 1 gives the FTIR spectra of the SMA separated from Nylon 6/PBT/SMA(mass ratio, 70 : 30 : 9) ternary blend by extraction in boiling toluene. Some characte-

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