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Impact toughness of polypropylene/glass fiber composites: Interplay between intrinsic toughening and extrinsic toughening

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ABSTRACT

There are principally two mechanisms to improve the impact resistance of polymer-based composites, intrinsic toughening and extrinsic toughening. But the interplay between them is far from being well understood. Here, glass fiber was incorporated into polypropylene to promote extrinsic toughening mechanism, while addition of elastomer and annealing were adopted for intrinsic toughening. In this way, the interaction among glass fiber, elastomer and annealing could be discussed based on various characterizations, and their combined effect on mechanical properties of the composites could be determined. The results show that the intrinsic toughening mechanism of elastomer will be suppressed by glass fiber irrespective of the support of annealing, though annealing could work synergistically with glass fiber to toughen polypropylene. The possible structure-property relations are discussed. This work will provide deeper insight into the toughening behavior of polymer composites and practical guidance for the design of composites with excellent stiffness-toughness balance.

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1. Introduction

Toughness of materials, which is measured as the energy needed to cause fracture, is one of the most important properties that determine their potential applications in industry. However, most polymer-based materials with excellent strength and stiffness show a brittle fracture behavior at high strain rates, particularly at the presence of flaws and notches [1–6]. So the impact behavior of polymers and their composites is one of the key factors to evaluate their usage, and the subject of toughening polymers/composites has received not only academic but also industrial attention throughout the years.

Overall, there are two mechanisms to improve the impact properties of polymers and their composites, intrinsic toughening and extrinsic toughening [7,8]. Intrinsic toughening, mainly including massive crazing and shear yielding, is the plastic deformation mechanism that acts ahead of the tip of a crack against its initiation and/or propagation [7,8]. It is strongly related to plasticity and enlarges the plastic zone of the matrix polymer [9–13]. The well-documented toughening approach of blending with elastomers is a typical intrinsic process. The strong toughening effect of annealing on some semi-crystallized polymers, which promotes crystalline network perfection and severe plastic deformation, works also intrinsically. Extrinsic toughening, on the other hand, is the crack-tip-shielding mechanism that operates principally behind the crack tip to inhibit damage, mainly including bridging, fiber pull-out and interfacial debonding [7]. By operating mostly in the crack wake, extrinsic mechanisms are valid in resisting crack growth [7,14–18]. The effectiveness of strong fibers in improving impact resistance of polymers is the result of extrinsic toughening. When the stress is transferred from the polymer matrix to the fibers at the crack tip, a large amount of strain energy, which would otherwise have been used to extend the crack, can be stored on the fibers, and then dissipated after fracture [14]. This is the fiber bridging mechanism. The fiber pull-out and interfacial debonding mechanism could also dissipate much energy [15–18].

In the abundant previous works, both the intrinsic and extrinsic toughening mechanisms are well established and investigated, but the combined effect of them is less reported. Paul and coworkers [19–22] conducted a series of work to investigate the impact toughness of polyamide 6/short glass fiber (SF)/elastomer composites. The results show that the elastomer type, droplet size and

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fiber surface chemistry would affect the toughness of the composites to different degrees, but the intrinsic toughening effects of the elastomers are all suppressed in the presence of glass fiber, irrespective of these influencing factors. Tjong and coworkers [23,24] studied the toughening behavior of polyamide 66/SF/elastomer composites using the essential work of fracture concept, it was found that the addition of SF is beneficial to enhancing the essential work of fracture, indicating a extrinsic toughening effect, while non-essential plastic work was absent in the composites, suggesting a restriction of shear yielding by the SF and consistent with Paul's work. In our previous work [25], long glass fiber (LF) was used to promote the extrinsic toughening mechanism of polypropylene (PP) owing to its better stress transfer effect than SF, while β-modification and annealing were adopted to provide intrinsic toughening. A synergetic effect between them was verified and composites with stiffness-toughness balance were obtained. Nonetheless, these results seem controversial to the previous works demonstrating the conflict between intrinsic and extrinsic toughening. What makes this conflict? Since no such work has ever been conducted to deliberate the toughening behavior of polymer/LF/elastomer composites as well as the effect of annealing on their toughness, more systematic work is needed to further investigate the interplay between intrinsic and extrinsic toughening mechanisms.

So in this work, an extrusion-impregnation device was used to produce PP/LF composites with and without elastomers (ethylene-octene copolymer, POE) to determine the combined effect of LF extrinsic toughening and POE intrinsic toughening. In addition, annealing was also adopted to exert further intrinsic toughening. Mechanical properties of the composites were measured, and various characterizations were employed. In light of structure-property relations, the interplay between intrinsic and extrinsic toughening mechanisms was discussed.

2. Experimental

2.1. Materials and sample preparation

Isotactic polypropylene (PP) T30S, with the density of 0.91 g/cm^3 and melt flow index (MFI) of 2.5 g/10 min (210 °C, 2.16 kg), was nicely supplied by CNPC Dushanzi Chemical Company, China. POE Engage 8150, with the octene content of 25 wt%, density of 0.868 g g/cm^3 and MFI of 3.5 g/10 min (210 °C, 2.16 kg), was a commercial product of DuPont Dow Elastomers. Glass fiber (GF) roving was purchased from Jushi Group (Chengdu, China). 0.2 wt% antioxidant (Irganox 1010) was blended into the materials during processing.

An extrusion-impregnation device, in which a twin screw extruder (SHJ-35, Nanjing Haote Machinery Company) was connected to an impregnation die by a joint, was designed and used to prepare the LF reinforced PP continuous strands. The details of this equipment were described elsewhere [26]. The temperatures of the extruder were set at 170 °C, 195 °C and 220 °C from hopper to die and the screw speed was 90 rpm. The temperature of the impregnation die was maintained at 225 °C. The obtained continuous strands were chopped into 8 mm-length pellets, which were then mixed with PP (or melt-blended PP/POE blends) and injection-molded using a PS40E5ASE injection machine at 210 °C. The glass fiber contents were 0 wt%, 5 wt%, 10 wt%, 15 wt%, and 23 wt%, respectively, while the POE contents were 0 wt%, 5 wt%, 10 wt%, 20 wt%, 30 wt% and 40 wt%, respectively. The PP/SF control samples were prepared by an extrusion mixing-injection molding process. In detail, PP and GF were firstly co-extruded with a twin screw extruder (SHJ-35, Nanjing Haote Machinery Company), in this way, GF will experience the high shear stress exerted by the screws, and SF reinforced composites could be prepared, which were then injection-molded using a PS40E5ASE injection machine. The processing parameters and GF contents used to prepare the PP/SF samples are identical to those employed to prepare the PP/LF specimens. For annealing experiment, the samples were placed in a vacuum oven set at 135 °C for 2 h, after which they were gradually cooled down in ambient air. Prior to the tests, the samples were conditioned at ambient conditions for 48 h.

The samples are named as *m*OE*n*LF or *m*OE*n*SF, in which *m* is the weight percentage of POE, *n* is the weight percentage of glass fiber, LF means long glass fiber, SF means short glass fiber. When $m = 0$ or $n = 0$, the samples are named as nLF/nSF or mOE, respectively. When the sample is annealed, we add "-A" after its original name. For instance, 23SF means the untreated PP/SF composites with 23 wt% SF, and 20OE10LF-A stands for the annealed composite with 20 wt% POE and 10 wt% LF.

2.2. Characterization

Notched Izod impact strength was measured using a VJ-40 Izod machine at room temperature (23 °C) according to ASTM D256-04 standard. Tensile test was conducted at room temperature using an SANS universal tensile testing machine. A two-step program was used, which is, the crosshead moving speed was set as 5.00 mm/min and 50.00 mm/min for modulus and tensile strength tests, respectively. The average values of the tested properties were obtained from at least five specimens.

Optical microscope (OM) observation was carried out using a Leica DMIP microscope equipped with a digital camera. The injection molded specimen was burned in a muffle furnace for 4 h at 600 °C to isolate the glass fibers from the composites. The extracted fibers were dispersed on a glass slide for observation. The length of more than 500 fibers was measured with Image J software system. Number average fiber length was calculated as described in our previous work [27].

Dynamic mechanical analysis (DMA) was performed on a DMA Q800 analyzer (TA instruments, USA). The three-point-bend mode was used, and the measurement was carried out from −90 to 150 °C at a heating rate of 3 °C/min, an oscillatory strain of 0.1% and a frequency of 1 Hz.

Wide-angle X-ray diffraction (WAXD) spectra was acquired using A Philips X'Pert pro MPD apparatus with Ni-filtered Cu Kα radiation ($\lambda = 0.154$ nm) at 40 kV and 40 mA. The diffraction angle (2θ) range was 10°—25°. The analysis method of WAXD spectra to obtain the overall crystallinity was described elsewhere [12,13].

Scanning electron microscope (SEM) was applied to observe the rubber domain morphology observation using an FEI Inspect F SEM instrument with an acceleration voltage of 20 kV. The specimen was cryo-fractured parallel to the flow direction in liquid nitrogen and then etched in xylene at 60 °C for an hour. The samples were coated with gold before each test.

3. Results and discussion

3.1. Mechanical properties

Tensile properties of various composites are shown in Fig. 1. As expected, Young's modulus and yield strength show linear dependence on GF content. With 23 wt% GF content, Young's modulus of PP increases remarkably from 1320 MPa to 2690 MPa, almost irrespective of the fiber length (Fig. 1(A)). But fiber length has an obvious influence on the yield strength, which increases gradually from 33.5 MPa for PP to 56.5 Mpa for 23SF, and rises distinctly to 83.6 MPa for 23LF (Fig. 1(B)). The addition of POE, on the other hand, will reduce the Young's modulus and yield strength of PP and its

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