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Microstructure and impact strength of polyamide 6 composites

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ABSTRACT

Impact strength and tensile strength of materials are among the most important properties in sport equipment. The objective of the paper is to greatly increase impact strength with no compromise of tensile strength of both polyamide 6 (PA 6) and short glass fibre reinforced PA 6 (GFPA 6), the two commonly used materials in sport equipment. For PA 6, ternary nanocomposites have been designed with the addition of nanoclay as strengthening agent and ethylene-octene elastomer grafted maleic anhydride (POE-g-MAH) elastomer as toughening agent. The microstructure of the nanocomposites is found to be affected by the blending sequence of the various constituents. With the control of the microstructure and formulation, polyamide 6 nanocomposites with impact strength increased by more than 140% without compromising their tensile properties have been successfully developed. For the GFPA 6 composite, the formulation and process have been optimized to avoid reduction in the glass fibre length. Additionally, the POE-g-MAH is found to also act as compatibilizer to increase interfacial adhesion between the glass fibre and the PA 6. The impact and tensile strength of the 30% glass fibre reinforced PA 6 have been increased by 145.4% and 8.9% with the addition of 5% POE-g-MAH.

1. Introduction

Polymer materials have been widely used in sport equipment such as ski bindings, in-line skates and hockey sticks because of their high strength to weight ratio, excellent fatigue properties, ease of processing and long service life [1–3]. Polyamide 6 (PA 6) and glass fibre reinforced polyamide 6 (GFPA 6), among the most commonly used engineering thermoplastics, can be a better choice for sport industries owing to their high heat resistance, high abrasion resistance and high chemical resistance. However, PA 6 based materials are usually brittle, which limits their applications as toughness is one of the most important material properties in sport equipment. The impact properties of the polymers can be effectively improved by addition of elastomeric toughening agent, but this usually achieves at the expense of tensile strength [4–6].

The objective of this work is to develop polyamide 6 nanocomposites and short glass fibre reinforced polyamide 6 composites with much improved impact strength without sacrificing tensile strength. Different strategy has been used to design the microstructure of PA 6 nanocomposites and GFPA 6 composites, the effect of reinforcement and process on their impact and tensile properties have been studied.

2. Experimental

2.1. Materials

Polyamide 6, Ultramid B3K, was purchased from Bayer, chopped glass fibre of HP3610 was purchased from Behn Meyer Speciality Chemicals. Ethylene-octene elastomer grafted maleic anhydride (POE-g-MAH) was purchased from Chinese HKH national engineering research center. Nanoclay of Cloisite 93A was purchased from Southern Clay Products Inc. Cloisite 93A is a natural montmorillonite modified with a methyl dehydrogenated tallow ammonium (M2HT), the fine powder possesses a cation exchange capacity of 90 meg/100 g clay and a density of 1.88 g/cm^3.

2.2. PA 6 nanocomposites and GFPA 6 composites fabrication

All the materials were dried at 80 °C overnight prior to use. PA 6 nanocomposites and GFPA 6 composites were prepared by melt compounding using Leistritz twin-screw extruder (MICRO 27). For GFPA 6, the short glass fibres were fed into extruder via side feeder. The temperature range of the extruder for both PA 6 and GFPA 6 composites was 220–240 °C with screw speed of 150 rpm. Both tensile and notched impact test pieces were injection molded at 245 °C using Netstal injection molding machine (HP 1000). All the dimension of the impact and tensile test pieces were consistent to the ASTM D6110 and D638.

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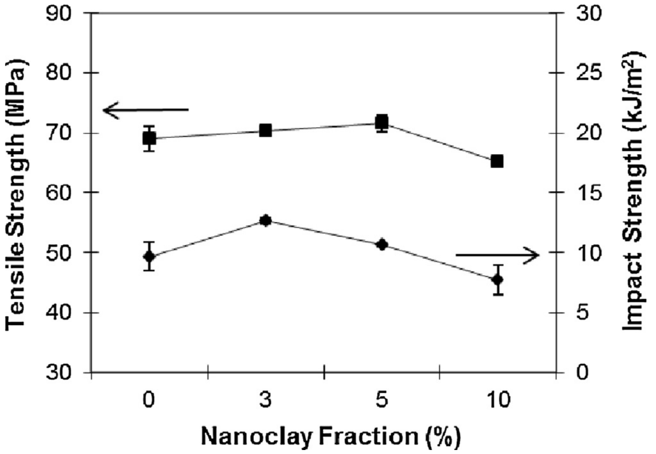

Fig. 1. Effect of the nanoclay fraction on tensile and impact strength of the PA 6.

2.3. Characterization

The microstructure of the nanocomposites was observed and analyzed by transmission electron microscopy (TEM) (Philips CM300 FEGTEM) and scanning electron microscopes (SEM, Carl Zeiss EVO 50). X-ray diffraction (XRD) of the specimens was performed using a Bruker diffractometer with Cu Kα radiation ($\lambda = 1.54$A°) at 40 kV and 30 mA, the scan speed is 0.2 s/step.

The tensile properties of the nanocomposites were measured with Instron 5569 tensile machine at room temperature. The load cell used was 10 kN and crosshead speed used was 50 mm/min. The Charpy impact strength of the notched specimens was measured using Ceast Resil Impactor at room temperature, the hammer used is 2.7 J. For both tensile and impact tests, at least five test pieces of each type of the formulation were tested. The final property data was the average of the measured results.

3. Results and discussion

3.1. Effect of the nanoclay fraction on impact and tensile strength of the PA 6

Nanoclay, due to its rigid inorganic nature, nano-scale dimension and huge adequate interfacial contact area between clay and polymer matrix, can effectively increase mechcnial strength of polymers at low filler fraction [7,8]. However, there are numbers of studies showed that the addition of nanoclay can only increases very modest or even drops toughness of polymer matrix [9–14], this is because the presence of the stiff nanoparticle hindered the polymer chain mobility. Fig. 1 shows the effect of nanoclay fraction on tensile and impact strength of the PA 6. It can be seen that the tensile strength of the PA 6 increases with nanoclay fraciton until 5%, then the tensile strength decreases by 5.5% at relative high filler fraction of 10% due to the nanoclay agglomeration. The impact strength of the PA 6 is increased by 31.08% at nanoclay fraction of 3%, but further increase in nanoclay fraction causing a decrease in impact strength. The increase in impact strength at nanoclay of 3%, perhaps, is due to the formation of micro-cracks resulted from the delamination of the nanoclay layers under a sudden load [15]. The decrease in impact strength at higher nanoclay fraction is due to the presence of the rigid nanoclay particles, they reduce the polymer plastic deformation under a sudden load. Plastic deformation is usually responsible for high toughness values.

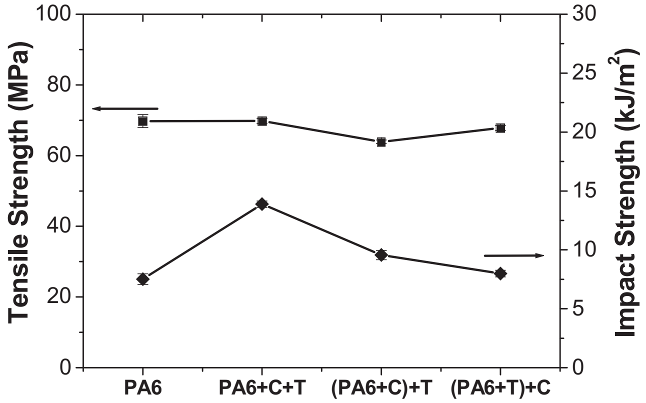

Fig. 2. Effect of the blending sequence on mechanical properties of the PA 6 ternary nanocomposites.

3.2. Developing the PA 6 ternary nanocomposites with high impact and tensile strength

In order to further markedly increase the tougness of the polymer without compromising tensile strength, PA 6 ternary nanocomposite is designed with POE-g- MAH elastomer as a toughening agent, and the nanoclay 93A as a strengthening agent. For composites with multi-phases, the microstructure can be affected by blending sequence of the constituents [16]. Three blending sequences have been employed in the work: (a) PA 6 + nanoclay 93A (C) + toughening agent POE-g-MAH (T) (one-step blending): PA 6, nanoclay and toughening agent are blended simultaneously; (b) (PA 6 + C) + T (two-step blending): PA 6 blends with nanoclay first and then blends with toughening agent; (c) (PA 6 + T) + C (two-step blending): PA 6 is blended with toughening agent first and then mixed with nanoclay. Fig. 2 shows the mechanical properties of the ternary nanocomposites containing 10% nanoclay and 15% toughening agent with different blending sequences.

Unlike PA 66/nanoclay 30B/(maleated styrene–ethylene/butylene–styrene triblock copolymer (SEBS-g-MAH)) ternary nanocomposites in which highest impact strength is achieved at two-step blending process of (PA 66 + nanoclay 30B) + SEBS-g-MAH [17], it can be seen in Fig. 2, for the PA 6/nanoclay/POE-g-MAH nanocomposites, the highest impact and tensile strength have only been resulted from the one-step blending (a) of (PA 6 + C + T) – the impact strength remarkably increases by 84.5%, while the two-step blending of (b) and (c) can only increase the impact strength by 27.1% and 6.1%, respectively. Morphological study of the nanocomposite from one-step blending (Fig. 3) also indicates that the nanoclay particles is exfoliated and uniformly dispersed in the PA 6 phase.

The phenomenon that PA 6 nanocomposite with higher impact strength is obtained from one-step blending is most probably a consequence of interaction among the nanoclay, matrix material and toughening agent. The nanoclay 93A is modified with methyl dehydrogenated tallow ammonium, it has increased polarity as compared to the pristine nanoclay. On the other hand, the PA 6 is very polar due to the presence of amide (—CO—NH—) groups. Thus, the nanoclay 93A has higher affinity to the PA 6 than to the POE-g-MAH, that is, the nanoclay layers are richer in the PA 6 phase than in the POE-g-MAH phase during the one-step blending process, both the strengthening effect of the nanoclay and toughening effect of the POE-g-MAH are not compromised. For the PA 6 nanocomposites obtained from both two-step compounding of (b) and (c), the impact strength is lower than those from the one-step compounding of (a). Degradation caused by shear stress and heat during the second process is one of the reasons. Addition-