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Preparation and characterization of high performance of graphene/nylon nanocomposites



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ABSTRACT

The potential of using functionalized graphene (FG) as reinforcement for producing high performance of nylons 11 and 12 nanocomposites was explored. A series of FG/nylons 11 and 12 nanocomposites fabricated by a direct melt blending with assistance of pre-mixing was assessed for their mechanical and barrier properties as a function of FG loading. The results revealed that the ultimate tensile strength, elongation at break, impact strength, toughness and permeation resistance characteristics were improved by the incorporation of a very small amount of the FG into the nylon matrices. In the nylon12, the ultimate tensile strength increased by ~35%, elongation at break by ~200%, fracture toughness by ~75% and the impact failure energy by ~85%, respectively, when only 0.6 wt% FG was incorporated. In contrast, the tensile mechanical properties and fracture toughness of the nylon11 was only slightly improved by addition of FG, but a dramatic enhancement of ~250% in impact strength was achieved by adding 1 wt% FG. In addition, the graphene sheets were dramatically effective for improvements of vapor and gas barrier properties for both nylons at a very significant low loading. Especially, the nylon11 films with FG loading as low as 0.3 wt% showed a superior reduction of water vapor and oxygen permeability by ~49% and ~47%, respectively. In this communication, the effects of FG on these property enhancements of the nylons have been well discussed. It can be concluded that to achieve the maximally improved properties, aside from good dispersion of the filler, strong interface between the polymer and graphene sheets, flexibility of graphene in the polymer matrix could be considered to be also an important factor due to the characteristics of graphene wrinkled structure in the polymer matrix.

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1. Introduction

Polymer nanocomposites based on layered silicates, carbon nanotubes and graphene have attracted tremendous attention in recent years from both the scientific and engineering communities as a result of the substantial property enhancements obtained from relatively low nanofiller loadings [1–5]. Among these nanofillers, graphene has been recognized as a leading candidate to offer the best possible material properties in almost all

applications since it reunites several outstanding properties never observed before in a single material [5,6]. Its extraordinary performance has led many to call it the ‘superlative’ or ‘wonder’ material [6].

Graphene is the basal plane of graphite, a one-atom-thick two dimensional honeycomb layer of sp² bonded carbon [7]. It has been reported to be the strongest material ever tested in the world with the superior ultimate strength of 130 GPa and Young’s modulus of 1TPa [8]. Graphene sheets exhibits high transparency with absorption of ~2.3% towards visible light [9]. It has a thermal conductivity of 5000 W/(m³ K) at room temperature and very high electrical conductivity of up to 6000 S/cm. Defect-free graphene sheets are impermeable to all gas molecules [10].

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The theoretical specific surface area of graphene sheet is 2630–2965 m² g^{−1} with a large aspect ratio even higher than 2000 [11], which makes graphene an outstanding reinforcement for altering all matrix properties, such as mechanical, rheological and permeability properties, and thermal stability.

As occurred with other nanofillers, the main challenge in designing graphene-based polymer nanocomposites with maximally enhanced properties is, therefore, to effectively disperse the graphene sheets inside the host polymer matrix. Often, the fabrication of polymer nanocomposites is hindered by the tendency of nanoparticles to form agglomerates. The presence of agglomerates in polymer nanocomposites can negate the advantages of the nanofillers. The nature of graphene makes it hard to disperse within the majority of polymers since it can only interact efficiently with a limited group of polymers typically containing aromatic rings [6]. In addition, the low solubility of pristine graphene also limits its applications. In order to make graphene dispersible in – or compatible with – a variety of polymer matrices, as well as to maximize the interfacial interactions, chemical modification is generally required, introducing functional moieties that confer other properties on the pristine material. Recently, functionalization of graphene has been widely devoted to the production of highly exfoliated graphene sheets in organic solvents which has opened new horizons of using the nanosheets for developing new kind of polymer nanocomposites. Very active edge functional groups are the advantage for the functionalized graphene (FG) to form a good dispersion and strong interactions with polymeric matrices. To date, various graphene and its derivatives/polymer nanocomposites have been widely produced including polystyrene [4,12,13], polycarbonate [14], polyimides [15] and poly (methyl methacrylate) [16] and so on by solution mixing and achieved high reinforcement efficiency [4,5]. Many reports demonstrate that graphene and graphene based materials with the two-dimensional platelet geometry offer certain remarkable property improvements of polymer matrices combining the laminar properties of layered silicates with the unique characteristics of carbon nanotubes [4,5]. At very low filler contents most of these properties were better than those observed for other carbon-based reinforced nanocomposites, especially improved toughness and gas permeation resistance of the composite, due to the higher aspect ratio of graphene [17–20]. Rafiee et al. [17] firstly reported considerably enhanced fracture toughness of graphene filled epoxy composites. At a significant low loading of 0.125 wt% the toughness increased ~65%. For carbon nanotube epoxy composites, the best enhancement in K_{Ic} reported is ~43%, which occurs at about fourfold higher nanofiller weight fraction [18]. In the case of nanoclay/epoxy composites it required about 3.5% nanoclay weight fraction to achieve the similar level of K_{Ic} enhancement (~61%) [19]. Improvements in permeability reduction to gases and moisture resulting from the addition of low concentration graphene-based nanofillers are as effective as clay-based nanofillers at about 25 times higher loadings to thermoplastics [20]. Graphene allow for much lower loading levels than other nanofillers to achieve optimum

performance, which significantly impact weight reduction of nanocomposite materials. The versatility of graphene polymer nanocomposites suggests their potential applications in automotive, electronics, aerospace and packaging. Relative to solution mixing, melt blending is often considered as the most economically attractive, scalable and environmentally friendly method for applicable applications. However, use of this method has so far been limited to a few studies because of dispersion difficulty occurs as FG fillers in dried state, which could affect graphene future applications [21,22].

Nylons 11 and 12, which are members of the polyamide family with longer aliphatic chains and lower melting point, are one of the commonly used engineering plastic materials due to their key features such as their lightweight, easy fabrication, exceptional processability, durability and relatively low cost [23,24]. These engineering thermoplastics offer a distinctive combination of good mechanical properties, low wear and abrasion with high chemical resistance [25,26]. Thus, the nylons are widely used in a wide range of industrial fields from automotive to offshore applications, such as tubing and pipes for petroleum production lines and submarine flexible pipes for offshore oil production. For such particular applications, the materials are generally required to have high strength, modulus and toughness as well good barrier performance [24–26]. Accordingly, recent studies suggest that graphene sheets are excellent nanofiller, which can provide a combination of extraordinary physical properties [5,6]. In order to optimize their properties to meet desirable applications, our research group has produced series of nylon11/FG and nylon12/FG nanocomposites by a direct melt-compounding with assistance of pre-mixing. In this communication, their tensile mechanical properties, fracture toughness, impact strength and permeation resistance of these nanocomposites are reported.

2. Experimental

2.1. Materials

Nylons 11 and 12 (polyamides PA11 and PA12) with density of 1 g/cm³ and moisture absorption (24 h) of 0.07% with a low molecular weight about Mn: 3000 g/mol used in this study was kindly provided by 3T RPD Ltd., UK. Expandable graphite flakes were supplied by Qing Dao Graphite Company, China. All materials for chemical functionalization of graphite, such as concentrated sulfuric acid (H₂SO₄, 98%), potassium permanganate (KMnO₄), barium chloride (BaCl₂), hydrochloric acid (HCl, 36–38%) and hydrogen peroxide solution (H₂O₂) were purchased from Fisher Co. Ltd., UK.

2.2. Synthesis of chemical functionalized graphene (FG)

Chemical functionalized graphene (FG) was synthesized from expandable graphite according to Hummer's method [27] and the experimental details can be found in Ref. [28].

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