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Dramatic mechanical and thermal increments of thermoplastic composites by multi-scale synergetic reinforcement: Carbon fiber and graphene nanoplatelet

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ABSTRACT

We report an easy and efficient approach to the development of advanced thermoplastic composites based on multi-scale carbon fiber (CF) and graphene nanoplatelet (GN) reinforcement. Poly (arylene ether nitrile) (PEN)/CF/GN composites, prepared by the twin-screw extrusion, exhibited excellent mechanical properties. For example, the flexural modulus of PEN/CF/GN composites was 18.6 GPa, which is 1.7, 4.5 and 6.4 times larger than those of PEN/CF composites, PEN/GN composites and PEN host, respectively. Based on the SEM image observation, such mechanical enhancements can be attributed to the synergetic effect of micro-scale CF and nano-scale GN in the PEN matrix (decreased matrix-rich and free-volume regions and enhanced interfacial interactions). For 5 wt.% GN-filled PEN/CF/GN composites, the $T_{d30\%}$ of PEN/CF/GN composites was 145 °C and 62.8 °C compared with those of PEN host and PEN/CF composites, respectively. This study has demonstrated that multi-scale CF and GN have an obvious synergetic reinforcing effect on the mechanical properties and thermal stabilities of thermoplastic composites, which provides an easy and effective way to design and improve the properties of composite materials.

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1. Introduction

Carbon fiber (CF)-reinforced polymer composites have been extensively applied to the areas of aerospace, aircraft, rocket, sport and military industries due to the superior strength-to-weight, stiffness-to-weight ratio and high service temperature. Advanced composites have demonstrated weight savings for aircraft structure, outstanding corrosion and fatigue-damage resistance [1–5].

However, the limits of improving composite properties made of traditional micrometer-scale CF have been reached, since the composite properties, especially the mechanical properties, generally involve compromises [6,7]. One problem in these composites is that CF typically shows poor interfacial interactions with polymer matrix. Another critical drawback is the presence of matrix-rich and free-volume regions formed in the gaps between the interlaced fiber bundles [7]. These drawbacks lead to ineffective stress transfer and easy crack initiation and propagation.

To solve these problems, some researchers are dedicated to the CF surface treatment. At present, several techniques including plasma treatment have been used. These methods, on the one hand, have achieved strong interfacial interactions with polymer matrices and improved the mechanical properties of the composites. On the other hand, such methods always involve rigid chemicals and multiple fabrication steps. Consequently, defects are

preferentially observed at the CF surface while the existence of matrix-rich and free volume region still stands [8,9].

It is well-known that the mechanical properties of polymer/filler composites depend largely on the interface-to-volume ratio and filler size and a decrease in gain size significantly affects the yield strength and hardness [10-12]. In this sense, it is quite beneficial to incorporate nanoscale fillers such as fullerene [13,14], clay [6,15] and CNT [16-18] into polymer/CF systems to enhance mechanical properties of the composites. This approach benefits from the micro-scale reinforcement provided by traditional CF and from the complementary reinforcement on the nanoscale offered by clay or CNT. And 2-D and 3-D textile materials can be formed into the final net-shape of the desired product [7]. However, little work has been done on the preparation and property of nanofiller-reinforced polymer/CF nanocomposites. On the other hand, Xua and Hoa [6] have reported significant enhancement of flexural strength in the clay-reinforced epoxy/CF nanocomposites. Bekyarova et al. [18] have reported that CNT-filled epoxy/CF nanocomposites showed 30% enhancement of the shear strength as compared to that of epoxy/CF composites.

Recently, graphene nanoplatelet (GN) has attracted significant interest both in the fields of academia and engineering. Compared with clay, GN has similar layered structure but much higher mechanical properties. In comparison with quasi-1-D CNT, GN with 2-D lattice of carbon possesses similar mechanical performance but larger surface areas. Moreover, in the polymer/filler composites, stress and heat transfer can be taken much easily in





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2-D (in plane) than in 1-D (in line) and the way in which GN mainly contacts each other by plane-to-plane may further offer convenience for stress and heat transfer. Thus, GN is expected to be a potential alternative to both clay and CNT since it combines the low cost and layered structure of clay and the superior mechanical properties of CNT [19]. In the present work, poly (arylene ether nitrile) (PEN) is the main polymer of interest since it is a high performance thermoplastic for special uses such as aerospace, military and electronic areas. PEN exhibits excellent mechanical strength, high radiation resistance and strong chemical inertia. The polar and potential cross-linking nitrile groups on the aromatic ring in PEN promote adhesion of the polymer to many substrates and PEN can easily be readily processed into shaped forms [20,21]. Thus it is the good polymer matrix which can be used to prepare advanced materials. Herein, PEN composites reinforced with CF or/and GN were prepared by an economically and environmentally viable method of melt-mixing and molding. The objectives of this work are to: (i) determine the flexural properties of CF-filled PEN composites to find out the optimal CF content for the preparation of GN-filled PEN/CF/GN composites; (ii) systematically investigate the effect of different GN loadings on the mechanical properties, i.e., impact strength, flexural strength and modulus of PEN/CF/GN nanocomposites and (iii) assess the morphology of the nanocomposites to study the reinforcement mechanism of CF and GN in the PEN matrix; (iv) study the thermal stabilities of PEN/CF/GN composites.

2. Experimental details

2.1. Materials

Poly (arylene ether nitrile) (PEN) was kindly provided by Union Laboratory of Special Polymers of UESTC-FEIYA, Chengdu, China. It is a copolymer derived from 2, 6-difluorobenzonitrile with hydroquinone (HQ) and resorcin (RS) (HQ: RS = 95:5 by mol) with inherent viscosity of 1.38 dL/g (By Ubbelohde viscosity method, at 25 °C and with a concentration of 0.005 g/mL in *N*-methyl-2-pyrrolidinone). PAN-based Carbon fiber (CF) (T300–3 k, tensile strength: 4.0 GPa; tensile modulus: 240 GPa; density: 1.7 g/cm³; diameter: 7 µm) was purchased from Toray Industries, Inc. (Japan) and used without any further treatment. Graphene nanoplatelet (GN, in Fig. 1b) was prepared by acid-intercalated expanded natural graphite (EG, in Fig. 1a) followed by applying a cost- and timeeffective exfoliation process in a microwave environment as we reported before [20]. The thickness and diameter of GN are in the range of 10–30 nm and 5–20 um, respectively.

2.2. Processing

The processing method in this study is melt-mixing due to its simplicity and compatibility with existing industrial polymer processing techniques extrusion and injection molding. The PEN/CF composites were first prepared and analyzed to find out the optimal CF content. Consequently, 20 wt.% CF content was chosen to prepare GN-reinforced PEN/CF composites.

The preparation processes for PEN/CF/GN composites were carried out by four steps. (1) Drying; 20 wt.% CF filled-PEN pellets and GN were dried at 100 °C in a vacuum oven for 2 h to get rid of moisture. (2) Blending; weight-measured 20 wt.% CF filled-PEN pellets and GN (GN content: 1 wt.%, 3 wt.%, 5 wt.%, 8 wt.%, 10 wt.%) were mixed in a blending machine for 10 min. (3) Melt-mixing and granulation; the blending obtained above was melting-mixed in a TSSJ-2S twin-screw extruder (China Blue star Chengrand Chemical Co., Ltd., Chengdu, China). The temperature was maintained at 310, 320, 330, 330, 330, and 325 °C from the hopper to the die, and



Fig. 1. SEM images of (a) expanded graphite (EG) and (b) graphene nanoplatelet (GN).

the screw speed was about 120 rpm. (4) Hot-press molding; the granulation was transferred to a Daca Micro-Injector operating at T_{barrel} = 340 °C and T_{mold} = 300 °C. The injection pressure used was 1.1 MPa and was held for 10 s. The standard molds designed for different experimental measurements were taken out after the hot-press was finished. The PEN/GN composites with 5 wt.% GN content were prepared with similar procedure for comparison.

2.3. Characterization

Mechanical measurements: Mechanical tests were performed with a SANS CMT6104 series desktop electromechanical universal testing machine (Shenzhen, China). All tests were undergone at room temperatures and the reported values were calculated as averages of five specimens for each PEN/CF and PEN/CF/GN composite. Flexural tests (three-point bending mode) were held according to the GB/T9341-2008 standard test method and the moving speed of the crosshead was 5 mm/min [22]. The dimensions of the samples for flexural tests were 80 mm * 10 mm * 4 mm. Impact resistance tests (Izod type) were carried out following the GB/ T1843-1996 standard test method [23]. The dimensions of the samples for impact resistance tests were 80 mm * 10 mm * 4 mm with a gap diameter of 0.25 mm in the middle.

Morphological characterization: The morphology of the EG, GN and fracture surfaces of the PEN-based composites were observed with scanning electron microscope (JEOL JSM-5900LV). The SEM samples were coated with a thin layer of gold prior to examinations. Download English Version:

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