

Effects of process parameters and surface treatments of graphene nanoplatelets on the crystallinity and thermomechanical properties of polyamide 6 composite fibers



Sang-Ha Hwang^{a, d}, Byeong-Joo Kim^{a, d}, Jong-Beom Baek^{b, d}, Hyeon Suk Shin^{c, d}, Il-Joon Bae^e, Seong-Young Lee^e, Young-Bin Park^{a, d, *}

^a Department of Mechanical Engineering, Ulsan National Institute of Science and Technology, UNIST-gil 50, Ulsju-gun, Ulsan, 44919, Republic of Korea

^b School of Energy and Chemical Engineering, Ulsan National Institute of Science and Technology, UNIST-gil 50, Ulsju-gun, Ulsan, 44919, Republic of Korea

^c School of Natural Science, Ulsan National Institute of Science and Technology, UNIST-gil 50, Ulsju-gun, Ulsan, 44919, Republic of Korea

^d Low Dimensional Carbon Materials Center, Ulsan National Institute of Science and Technology, UNIST-gil 50, Ulsju-gun, Ulsan, 44919, Republic of Korea

^e Carbon Materials Research Group, Research Institute of Industrial Science and Technology, 67 Cheongam-ro, Nam-gu, Pohang, Gyeongbuk, 37673, Republic of Korea

ARTICLE INFO

Article history:

Received 21 November 2015

Received in revised form

2 May 2016

Accepted 14 June 2016

Available online 16 June 2016

Keywords:

Fibres

Nano structures

Mechanical properties

Thermomechanical

Melt-spinning

ABSTRACT

Polyamide 6 composite fibers containing various kinds of pristine graphene nanoplatelets [P-graphene nanoplatelets (GnPs): P-M5 and P-C750] as well as oxidized GnPs (acid-M5, acid-C750) were prepared via melt spinning. Both P-GnPs and acid-GnPs were used as reinforcements to enhance the thermo-mechanical properties of the composite fibers. GnPs were dispersed in the polymer matrix via solution mixing followed by coagulation. Composite fibers were melt-spun using a plunger-type extrusion system, followed by stretching to the maximum draw ratio using a Godet setup. The tensile and dynamic mechanical properties of the composite fibers were tested, as well as the thermal properties. The results showed considerable enhancements in the mechanical and thermal properties of the polyamide 6 fibers due to loading with 0.5–5 wt% GnP, and the composite fibers containing 1 wt% of acid-C750 exhibited an increase of up to 76% in the tensile strength compared to that of the neat polyimide 6 fibers. Dynamic mechanical analysis, differential scanning calorimetry, X-ray diffraction, and scanning electron microscopy were carried out to investigate the effects of the crystallinity, crystallization kinetics, and interfacial bonding on the tensile properties of the polyamide 6 composite fibers.

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1. Introduction

Since the discovery of carbon nanotubes (CNTs) in 1991 and the first experimental investigation of graphene in 2004, the field of nanocomposites has gained significant momentum [1,2]. Owing to the unique honeycomb structure of the sp^2 -hybridized carbon atoms, these structures exhibit large mechanical moduli; they are tough and exhibit excellent electrical, thermal, and optical properties [3,4]. Thermoplastic polymers are widely used organic materials, with a wide variety of industrial applications, and have the benefit that they can usually be recycled [5]. Thermoplastic

polymers also exhibit process flexibility from the macro to nano-scale, and enhancements of the physical properties may further add to their potential applications [6]. Thermoplastic polymer composites reinforced with CNTs [7–11] or with graphene [12–17] have received the most interest, with remarkable improvements reported in their mechanical, electrical, and rheological properties. Among the shapes and processing methods of polymer composites, fabrication of composites with fibers that align with the nanomaterials and polymer chains has been demonstrated [18]. Accordingly, a great deal of effort has gone toward improving the mechanical properties of these fibers [19–21].

To fully exploit the nanoparticles as reinforcements in the composite, they should not aggregate but be well dispersed. However, this is challenging because of the large specific surface area and the presence of van der Waals forces. Numerous methods have been used to achieve well-dispersed polymer composites, including optimal physical blending methods (e.g., ultrasound and

* Corresponding author. Department of Mechanical Engineering, Ulsan National Institute of Science and Technology, UNIST-gil 50, Ulsju-gun, Ulsan, 44919, Republic of Korea.

E-mail address: ypark@unist.ac.kr (Y.-B. Park).

shear mixing) [22–24], surfactants [25–27], surface treatments (e.g., oxidation, functionalization, and polymer chain grafting) [28–31], and in situ polymerization [32–36]. Attaching different covalently bonded functional groups to the carbon nanotubes leads to changes not only in the dispersion characteristics, but also in the adhesion and interfacial bonding between functionalized nanotubes and the polymer matrix [37]. A similar effect on the mechanical properties was reported by chemically functionalizing graphene to form composite materials [38]. More recently, the advantages of graphene in mechanical reinforcement over existing carbon fillers, such as carbon black, expanded graphite, and CNTs, have been discussed by Ramanathan et al. [38] and Rafiee et al. [39]. They attributed the improvements in the mechanical properties to the strong hydrogen bonding of oxygen-functionalized graphene and the mechanical interlocking at the wrinkled surface, which resulted in inhibition of polymeric segmental motion near the interface. Shin et al. [18] reported synergistic toughness enhancement using reduced graphene oxide flakes and CNTs as a fiber form. However, few studies have reported on graphene-containing commercial-grade polymer nanocomposite fibers and the effects of the nanomaterials on their thermomechanical properties.

Here, we investigated the effects of graphene nanoplatelets (GnPs) on the mechanical properties of polyamide 6 (PA6) fibers with and without chemical surface treatments. PA6 fibers were spun using a laboratory-scale plunger-type fiber extruder with different GnP types, contents and functionalizations. The mechanical enhancement of the fiber with the inclusion of GnP was examined using crystallinity and crystallization kinetic analyses; i.e., wide-angle X-ray diffraction (WAXD) and differential scanning calorimetry (DSC). Further investigations were carried out using dynamic mechanical analysis (DMA) to monitor the glass transition temperature (T_g) and the effects of restricting the segmental motion of the polymer chains.

2. Experimental

2.1. Materials

Two kinds of commercially available GnPs (i.e., M5 and C750) were purchased from XG Sciences Inc. (Lansing, MI, USA). As shown in the transmission electron microscopy (TEM) and scanning electron microscopy (SEM) images in Fig. 1a and b, the M5 and C750 GnPs were composed of a few sheets of graphene with a thickness in the range of 10–20 nm (M5) and 3–7 nm (C750). The average

diameter of the M5 GnPs was 5 μm , and that of the C750 particles was 750 nm. The PA6 was sourced from DuPont (Zytel 7335F; Wilmington, DE, USA) and was supplied in pellet form. Chemical reagents including nitric, sulfuric, and formic acids were purchased from Samchun Chemical Inc. (Pyeongtaek, Korea).

2.2. Surface treatment of the GnPs

To achieve oxidized GnPs (i.e., acid-M5 and acid-C750), 3 g of GnPs was added to 300 mL of a 1:3 mixture of nitric acid and sulfuric acid. The resulting mixture was stirred and reacted for 12 h at 100 $^{\circ}\text{C}$, and then filtrated and washed with methanol until the pH reached ~ 6 . The remaining reagents were removed by freeze-drying for 48 h.

2.3. Preparation of GnP/PA6 composites

PA6 pellets were dissolved in formic acid at a concentration of 0.25 g/mL, while GnPs were added and stirred at a concentration in the range of 0.5–5 wt% polyamide, then sonicated for 2 h using a horn-type sonicator. Subsequently, coagulation was achieved by pouring the PA6 solutions containing GnPs into deionized water. Following filtration, the composite powder was dried in a vacuum oven for 24 h at 130 $^{\circ}\text{C}$ to remove any residual solvent.

2.4. GnP/PA6 composite fiber spinning

Prior to compounding the PA6 composite powders and pellets, they were dried at 80 $^{\circ}\text{C}$ for 48 h in a vacuum oven. The composite materials and neat PA6 were spun into fibers using a plunger-type extruder with a fiber spinneret (Fig. 2). The speed of the supply piston, take-up roll speed, heating elements, and winder all affect the fiber draw ratio (DR), and the Godet temperature for drawing was electronically controlled. The compound was melt-spun at 275 $^{\circ}\text{C}$, and the filament was heated to 150 $^{\circ}\text{C}$ to increase the crystallinity and facilitate drawing. The maximum DR of the M5 fiber was 3, and that of C750 was 4.

2.5. Characterization

The morphology of the nanofillers and the fracture surface of composite fibers were observed using SEM (Nanonova 230; FEI, Hillsboro, OR, USA). Single filament tensile tests and dynamic mechanical tests were carried out using a DMA tester (Q800; TA

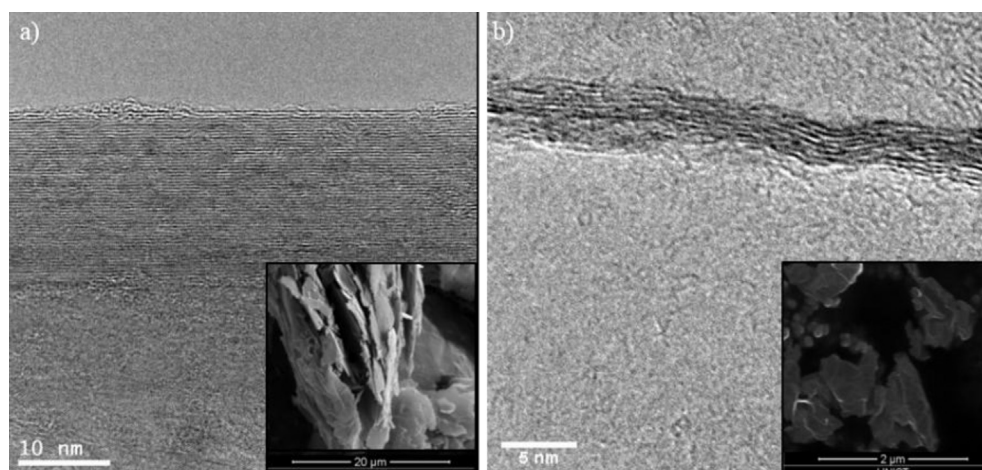


Fig. 1. TEM and SEM (inset) images of the GnPs. (a) M5 and (b) C750.

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