



Enhanced Mechanical Properties of Multi-layer Graphene Filled Poly(vinyl chloride) Composite Films

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ARTICLE INFO

Article history:

Received 11 June 2014

Received in revised form

6 September 2014

Accepted 23 September 2014

Available online 10 December 2014

Key words:

Multi-layer graphene

Poly(vinyl chloride)

Composites

Mechanical properties

Film

In order to improve mechanical properties of soft poly(vinyl chloride) (PVC) films, we used commercial multi-layer graphene (MLG) with large size and high structural integrity as reinforcing fillers, and prepared MLG/PVC composite films by using conventional melt-mixing methods. Microstructures, static and dynamic mechanical properties of the MLG/PVC composite films were investigated. The results showed that a small amount of MLG loading could greatly increase the mechanical properties of the MLG/PVC composites. The tensile modulus of the 0.96 wt% MLG/PVC composites was up to 40 MPa, increasing by 31.3% in comparison to the neat PVC. Such a significant mechanical reinforcement was mainly attributed to uniform dispersion of the large-size MLG, good compatibility and strong interactions among MLG and plasticizers and PVC.

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1. Introduction

Poly(vinyl chloride) (PVC), one of the most consumed thermoplastics with low cost, excellent corrosion resistance, good flame retardance, and easy operation, has been widely used in industrial fields^[1–4]. Generally, adding plasticizers into PVC resin can obtain soft PVC products, and they can be applied as electrical cables and films in some fields of building construction, automobile, agriculture, and communication^[2]. In those circumstances, mechanical strength of soft PVC products is an important evaluating index^[5]. How to improve mechanical properties of soft PVC has been a critical issue, which has aroused great interest in industrial and academic fields. Some inorganic fillers, such as calcium carbonate, clay, and nanosilica, have been added to improve mechanical properties of PVC^[4,6–8], while there are still some drawbacks of low mechanical reinforcement, poor dispersion, and poor compatibility^[6–9]. Recently, nanocarbon materials like carbon nanotubes and graphene have been used to improve mechanical properties of PVC by virtue of their high strength, low density, and good compatibility with polymer^[4,10–14]. Especially, graphene shows some advantages of lower cost and better dispersion over

the CNTs, revealing its great potential for commercial large-scale application of nanocomposites.

In the light of its excellent mechanical strength, extraordinary physical properties, good dispersion and compatibility with polymer matrix^[15–17], graphene has been added as a novel nanofiller into PVC matrix to improve mechanical properties of nanocomposites. Vadukumpully et al.^[18] used a solution blending method to obtain graphene/PVC composite films with high mechanical strength, high thermal stability, and high electrical conductivity of composites. Salavagione et al.^[19] found that the presence of chemically modified graphene oxide (GO) could remarkably improve storage modulus and glass transition temperature of graphene/PVC composites. Wang et al. and Deshmukh et al.^[20,21] reported that the addition of GO could also improve thermal and mechanical properties of GO/PVC composite films. However, all these graphene/PVC composites mentioned above were obtained by using the solution blending method. Such a method has its intrinsic disadvantages of complicated operation, solvent impurity, and high cost, and it is difficult to be utilized for large-scale production of nanocomposites. Furthermore, the graphene oxide used in all these above studies has some drawbacks of small size and many structural defects, which is not beneficial to obtaining remarkable mechanical reinforcement of nanocomposites^[22,23].

Mechanical properties of graphene/polymer composites are strongly dependent on many influencing factors, such as intrinsic mechanical properties of graphene, dispersion of graphene, compatibility and interaction between graphene and polymer. It

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has been reported that large size and high structural integrity of graphene can be beneficial to improving mechanical properties of nanocomposites^[15,24–26]. Fortunately, an interlayer catalytic exfoliation (ICE) method has been developed recently for obtaining high-quality multi-layer graphene (MLG) in a large quantity^[23]. The commercial MLG exhibits larger size, higher structural integrity, higher electrical and thermal conductivity than those graphene or GO which were prepared by traditional Hummer's method^[27,28]. We believe that addition of the MLG may significantly improve mechanical properties of the MLG/PVC composites. As far as we know, enhanced mechanical properties of the commercial MLG filled PVC composites have not been reported so far.

The aim of this work is to obtain MLG/PVC composites with high mechanical properties by taking full advantages of the large size and high integrity of the MLG. Conventional melt-mixing method was used for preparation of MLG/PVC composites. Microstructure, tensile strength and modulus, and dynamic mechanical behavior of the MLG/PVC composites were investigated in detail.

2. Experimental

2.1. Materials

Multi-layer graphene (MLG) powders, produced by using interlayer catalytic exfoliation (ICE) method, were supplied by Sichuan Jinlu Group Co., Ltd., China. Layer number of the used MLG was less than 10 graphitic layers, and its lateral size was as large as 10–15 μm and thickness is about 1–3 nm. The MLG powders possess high structural integrity and less defects (its C/O ratio was as high as 20), and its electrical conductivity was over 700 S/cm. Poly(vinyl chloride) (PVC) resin powders (general type SG-5) and rear-earth stabilizer were also supplied from Sichuan Jinlu Group Co., Ltd., China. Plasticizers of dioctyl phthalate (DOP) were supplied from Sinopharm Chemical Reagent Co., Ltd.

2.2. Preparation of the MLG/PVC composite films

The weighed PVC powders and DOP (weight ratio of 2:1) were mixed together at room temperature by using a high-speed mixer,

and then aged for 1 h in a drying oven at 80 $^{\circ}\text{C}$. Thereafter, the obtained mixtures were mixed with stabilizers and MLG powders, further heated and squeezed into thin MLG/PVC composite films by using a two-roll mill. The MLG/PVC composites with various MLG loadings of 0, 0.32, 0.64, 0.96, 1.28 wt% were obtained by following same procedures.

2.3. Characterization

Microstructure and morphology of the used MLG was characterized by scanning electron microscopy (SEM, NavoSEM430, FEI) and transmission electron microscopy (TEM). Fracture surfaces of the MLG/PVC composites were coated with gold particles and then observed by SEM. Tensile properties of the MLG/PVC composite films were performed on a universal testing machine (type WSM-50KG, Changchun Intelligent Equipment Co., Ltd.) at a speed of 5 mm/min referring to ASTM D882-10. A dynamic mechanical analyzer (DMA Q800, TA Instrument) was used for measuring storage modulus, loss modulus, and loss factor of the composite films in a tensile mode at a heating rate of 3 $^{\circ}\text{C}/\text{min}$ from -50°C to 100 $^{\circ}\text{C}$ in air, to investigate the effect of MLG on dynamic mechanical behavior and glass transition temperature of the MLG/PVC composites.

3. Results and Discussion

3.1. Microstructure of the MLG and MLG/PVC composite films

Fig. 1 shows the microstructure of the MLG powders and MLG/PVC composites. It can be seen clearly from the SEM images of Fig. 1(a, b) that the MLG exhibits typical crumpled morphology, totally different from the so-called two-dimensional planar morphology of graphene^[29–31], which is mainly attributed to high flexibility of the used MLG due to its large size of about 10–15 μm , thin thickness of 1–3 nm, and large aspect ratio of over 1000^[32]. Such large size and wrinkled morphology (high flexibility) of the MLG can also be clearly observed from TEM image in Fig. 1(c). On the other hand, we can see from SEM images of fractured MLG/PVC composite films (Fig. 1(d–f)) that MLG are uniformly dispersed

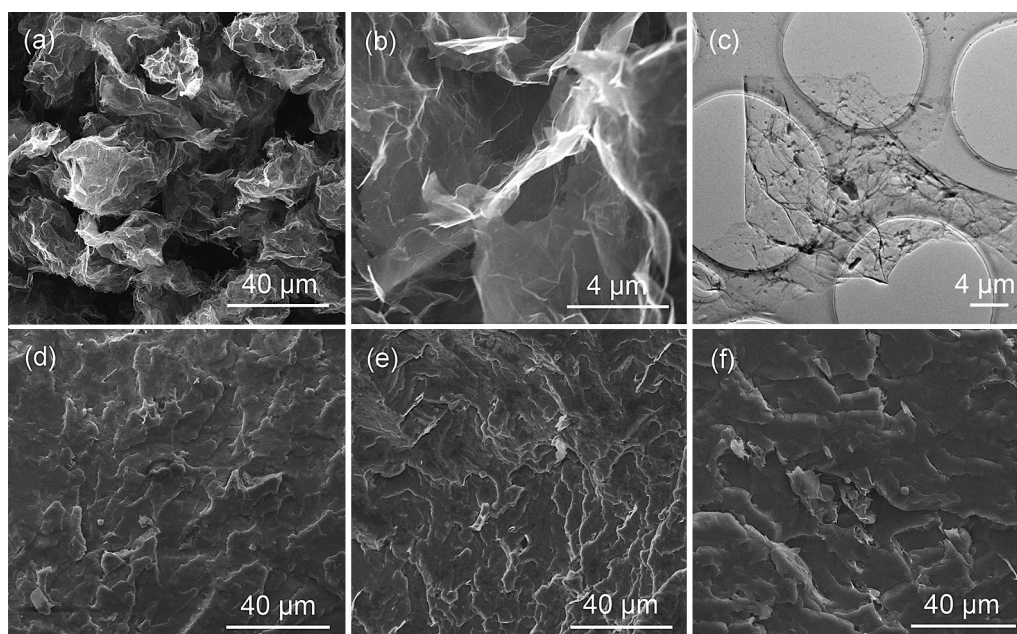


Fig. 1. Microstructure and morphology of the MLG and MLG/PVC composites. SEM images of the MLG (a, b), TEM image of MLG (c), SEM images of neat PVC (d), 0.96 wt% (e), and 1.28 wt% (f) MLG/PVC composites.

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