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Enhancement of mechanical properties of buckypapers/polyethylene composites by microwave irradiation



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ABSTRACT

Buckypapers (BPs) were generally laid up interleaved with polyethylene (PE) films to form BPs/PE composites after hot press. However, the mechanical properties of BPs/PE composites as-prepared were not good enough owing to poor impregnation and weak interfacial interactions between the matrix and carbon nanotube (CNT). In this paper, controlled microwave radiation treatment was applied on the BPs/PE composites. It is found that the microwave irradiation can effectively improve the tensile strength and stiffness of BPs/PE composites. Specifically, the tensile strength of BPs/PE composites increases from 20.9 MPa upto 34.1 MPa, 2.8 times that of pure PE, while the modulus increases from 880 MPa to 1778 MPa, 3.0 times that of pure PE. In addition, the structures (including fractured morphology, the interfacial composition between CNT and PE, structure of CNT and PE matrix, and crystallization) of BPs/PE nanocomposites with and without microwave irradiation were observed by SEM, FTIR, Raman, X-ray, and DSC, and then a mechanism of microwave irradiation to enhance the mechanical properties of BPs/PE composites was also proposed.

1. Introduction

Buckypapers (BPs) are non-woven carbon nanotube (CNT) membranes, in which nanotubes are well-dispersed and entangled together. Recently, BPs have attracted considerable interests as reinforcements for polymeric materials, because the use of BPs is almost the only way to obtain well-dispersed CNT composites at high loadings [1–3], which may lead to ultra-high mechanical performance polymer composites.

Similar to the carbon fiber reinforced polymer composites, BPs composites are usually fabricated with thermosetting resins (e.g., epoxy and bismaleimide) as the matrices. However, most of thermosetting resins have low elongations and thus their BPs composites are even more brittle. Heretofore, many efforts have been dedicated to employ BPs as reinforcements in the thermoplastic resins, such as polycarbonate (PC) [4,5], poly-ether-ether-ketone (PEEK) [6], polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), and polystyrene (PS) [7–9], and the resultant composites usually exhibit higher elongations than those of BPs/thermosetting resin composites.

However, there are two intractable problems which reduce the

reinforcing effects of BPs in the thermoplastic resins [4-6]: (i) poor impregnation and (ii) weak interfacial interactions between matrix resin and CNT. First, due to the nanoscale pore structure of the CNT membranes, the melting thermoplastics are difficult to efficiently infiltrate into BPs, and thus the reinforcing effect is minimized. Thus, solvent are often used to reduce the viscosity of thermoplastics. However, it is hard to find a good solvent for some insoluble thermoplastics, such as PE, etc., and the residual solvent could damage the mechanical properties of resultant composites. Second, it is well known that the interfacial nature usually plays a decisive role in mechanical properties of polymer composites. For this purpose, the CNTs are usually functionalized to endow better interfacial interaction with polymer. However, the existing surface treatment methods tend to sacrifice the mechanical properties and aspect ratio of CNT. Moreover, for non-polar thermoplastic resins, such as polyethylene (PE), the traditional functionalizing methods have little effects on the interfacial bonding between CNT and polymer. Apparently, it is still a challenge work to fabricate a BPs/insoluble and non-polar thermoplastic composites with excellent impregnation and strong interfacial interactions.

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Microwave irradiation (heating) is long used in polymer processing to directly couple electromagnetic energy with a material [10,11]. It is known that the CNTs are excellent microwave absorbents [12]. When CNTs are exposed to microwaves, energy can be transferred from microwave field to CNTs via dipole polarizations induced by alternating electric field as well as mechanical vibration caused by phonon-phonon interaction [13], thus producing intense heating, outgassing, and light emission [14]. It was reported that microwave irradiation could significantly improve the interfacial bonding for nanotube composites [15–17]. For example, Wang and his coworkers employed an ultrafast microwave welding technology to create a new paradigm for the formation of very strong interfacial strength between CNT and polyethylene terephthalate (PET) without any surface treatment [15]. Shim et al. demonstrated an enhancement of adhesive strength between CNT and PET, and the tensile strength of resultant composite showed 16% greater than that of pure PET [16]. However these works almost focused on low CNT content composites, rare work applies microwave irradiation onto high CNT content composites, such as BPs/thermoplastic nanotube composites.

Polyethylene is one of typical insoluble thermoplastics with excellent ductility. Introducing CNT into PE can improve its stiffness and strength [18–20], and the microwave irradiation may further improve these properties, leading to a new promising material which is strong and ductile. Although microwave radiation can improve the interfacial bonding between nanotubes and the polymer matrix, excess exposure of microwave radiation may inversely cause serious degradation of polymer matrix, especially for these BPs composites with high content of CNT which have a very short distance between the adjacent CNT. Also, it is found that directly applying microwave to BPs/PE composites often gets no effects or worse effects on the ultimate mechanical properties. In addition, the processing and mechanism should be well studied to fully exploit the advantage of microwave irradiation on BPs composites.

In this paper, a series of BPs/PE composites were prepared. In order to avoid all heat generated from microwave irradiation immediately transferred from CNT to the surrounding polymer and then seriously damage the chemical bonding of PE matrix, water loading is carried out to serve as a cooling reservoir for the junctions between the adjacent CNT, and then a microwave radiation was employed with controlled process to improve the properties of BPs/PE composites. In addition, the effect of structures on the mechanical properties of BPs/PE composites with and without microwave irradiation was investigated from the view of process-structure-property relation. To the best of our knowledge, BPs/PE composites are prepared utilizing microwave irradiation for the first time in this work. The use of microwave irradiation shows significant improvement of mechanical properties of BPs/PE composites.

2. Experimental

2.1. Materials

The multi-walled carbon nanotubes (Flo Tube TM 9000, MWCNT) with average diameter of 11 nm and average length of $10 \,\mu$ m were purchased from CNano Technology Limited and used without further purification. PE film was supplied by Mitsui Chemicals Inc. Triton X-100 surfactant was obtained from the Aladdin Chemistry Co. Ltd.

2.2. Preparation of BPs

MWCNT and Triton X-100 were dispersed in water using a high intensity probe sonicator to make a stable suspension of MWCNT. Subsequently, a BP was prepared by filtering the MWCNT suspension through a nylon filtering membrane with the aid of vacuum. Following filtration, the BP was thoroughly washed with deionized water and acetone to remove the surfactant. Finally, the BP was carefully peeled



Fig. 1. Photograph of BPs.

off from the filter and dried in a vacuum oven. The obtained BP has a thickness around $70\,\mu$ m, appearing as uniform, smooth, and self-supporting matrix, as seen in Fig. 1.

2.3. Preparation of BPs/PE composites

The process used for the fabrication of BPs/PE composites is a traditional hot-compression technique. In a typical experiment, the PE films and BPs were firstly placed in an oven at 100 °C for 24 h to remove the absorbed moisture. Subsequently, the BPs and PE films were carefully laid-up as sandwich-like structures as shown in Fig. 2, and then the laminates were put between the two platens of hot-press machine and held at 220 °C for 2 h consequently. Then the press heater was turned off and the samples were allowed to cool under pressure. Finally, BPs/ PE composite plates were obtained.

2.4. Microwave irradiation of BPs/PE composites

Microwave radiation was applied on the prepared BPs/PE composite plates using deionized water as a carrier at 700 W for 30 s under different pulse time. The modified composite samples were marked as "BPs-n/PE", where n represents the pulse time of microwave radiation, n is 1, 2, 3, 5, 10, 15, and 30 s, respectively.

2.5. Characterization

A Scanning Electron Microscope (SEM) (JEOL JEM-2010, Japan) was employed to observe the morphologies of the samples. All samples were dried at 50 $^{\circ}$ C for 24 h before test.



Fig. 2. Schematic illustration of fabricating BPs/PE composites through hot-compress method.

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