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Effect of γ-ray irradiation on the microstructure and self-heating property of carbon fiber/polyethylene composite films

Ping Tang [a], Rong Zhang [b], Zhiqiang Chen [a], Bo Yang [a], Yuezhen Bin [a,*]

[a] Department of Polymer Materials and Engineering, Faculty of Chemical, Environmental and Biological Science and Technology, Dalian University of Technology, Dalian 116024, PR China

[b] School of Material Science and Engineering, Hubei University of Technology, Wuhan 430068, PR China

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ABSTRACT

High-density polyethylene composite films filled with various contents of carbon fiber (CF) were manufactured by melt mixing. The electrical and self-heating properties of the composite films were investigated. The composite films containing 10 wt% CF were exposed to γ-ray irradiation. The structural, morphological, and self-heating properties of the irradiated composite films were examined. The results indicated that the surface temperature (T_s) of the composite films was strongly dependent on the applied voltage and filler content. The T_s of the irradiated composite films was higher than that of the non-irradiated films, which contributed to the lower thermal expansion and the higher degree of crystallization of the irradiated composite films. In addition, the mechanical properties of the irradiated composite films were significantly improved. Using a rechargeable battery as the applied voltage source to evaluate the self-heating property of the irradiated composite films, a heating temperature of 54.2 °C was achieved, which lasted for 6 h.

1. Introduction

Conductive polymer composites (CPCs) are one of the most important and interesting areas in polymer composite research due to their ease of processing, tunable properties and wide range of applications [1–7]. Self-heating or electrical heating materials are also one of many applications for CPCs [8–15], which are a type of electrical resistor that easily and quantitatively converts electrical energy to thermal energy via Ohmic joule heating [16]. The advantages of electrical heating materials, include quietness, absence of byproducts, precise control of a readily achieved temperature, and uniform distribution of heat energy compared to that of other forms [17].

Self-heating or electrical heating materials require high electrical conductivity. Carbon fiber (CF) [8,15,18], graphite (G) [11] and carbon nanotubes (CNTs) [16,17] are commonly used as conductive fillers for electrical heating materials. Chung [8] reported that the interlaminar interface between continuous carbon fiber laminae provides a two-dimensional array of heating elements, and a DC electrical power input of 1.1 W results in a maximum temperature of 85 °C. An and Jeong [11] investigated the electrical heating behavior of graphene/epoxy composite films using solution casting, and a maximum temperature of ~126 °C was maintained over a cyclic voltage variation of 30 V for the composite film containing 10 wt% graphene. In addition, the polymer matrix of composites used for electrical heating should exhibit good thermal and mechanical properties that satisfy practical requirements. Therefore, a thermosetting matrix, such as an epoxy resin [11,19] and aramids [12,17], have been used to prepare electrical heating materials. However, the processing method of these composites most likely limits their application as electrical heaters. Therefore, the irradiation of general plastics should be adopted to prepare electrical heating materials using mixing methods. The exposure of polymers to γ-rays induces changes in structure and morphology as well as chemical and physical properties [20]. Due to its high efficiency and ease of operation, γ-ray irradiation is a popular approach for modifying polymers and improving the performance of polymer-based composites. Several studies have investigated the effect of γ-irradiation on the structural [21–24], mechanical [25] and electrical properties [20,26] of various composites. However, no studies on the effect of irradiation on self-heating or electrical heating properties have been reported.

In this study, high-density polyethylene (HDPE) composite films filled with carbon fibers (CFs) were prepared by melt mixing. The electrical and self-heating properties of the composite films

* Corresponding author. Tel.: +86 411 84986093.
E-mail address: binyz@dlut.edu.cn (Y. Bin).

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were investigated as a function of the CF content. Then, the composite film containing 10 wt% CF was exposed to a ^{60}Co γ-ray irradiation source at various irradiation dose ranging from 0 to 400 KGy at room temperature. The effect of γ-irradiation on the microstructures, thermal properties, self-heating properties, and mechanical properties were studied in detail.

2. Experimental section

2.1. Materials

High-density polyethylene (HDPE) with a viscosity average molecular weight of 5×10^5 g/mol was obtained from Exxon Mobil Chemical Company, China. CFs with an average length of 6 mm (12 K) were purchased from DaLian XingKe Carbon Fiber Co., Ltd., China. Antioxidant 1010 was purchased from Tokyo Chemical Industry Co., Ltd. (TCI, Shanghai).

2.2. Preparation of the sample

The HDPE was dried at 80 °C, and the CFs were dried at 120 °C under vacuum to remove moisture prior to use. A predetermined quantity of the HDPE, CFs, and antioxidant 1010 were then mixed in a HAAKE Polylab system (HAAKE Rheomix) at a rotation speed of 20 r/min at 160 °C for 15 min. The mass of the antioxidant 1010 was 0.2 wt% of the raw materials. The samples were hot molded by a plate vulcanizing machine (GT-7014-H30) at 160 °C and 5 MPa. Next, composite films containing 10 wt% CF were irradiated using a ^{60}Co γ source at a dose rate of 10 KGy/h in air at room temperature, and irradiation doses of 0, 50, 100, 150, 200, 250 and 400 KGy were applied according to the different irradiation time. Finally, all of the irradiated composite films were annealed at 110 °C for 1 h.

2.3. Measurements

The electrical resistivity of the samples was measured using a two-terminal method, in which two ends of the specimen were clamped between copper metal jaws. The size of the specimen was 30 mm in length, 10 mm in width, and approximately 0.2 mm in thickness.

The self-heating properties were measured by a homemade computer-controlled instrument [9]. The specimen was cut into strips that were 140 mm in length and 70 mm in width, which are similar to the dimensions required for practical application. Copper foils (yellow parts in Fig. 1(a)) were hot-pressed to both ends of the sample under 1 MPa at 160 °C for 2 min as electrodes to eliminate the contact resistance. A schematic diagram of the sample is shown in Fig. 1(a). A fine thermocouple (AM-8000K, Anritsu METER, Japan) was attached to the center of the sample surface. The surface temperature (T_s) of the specimens was detected, and the corresponding circuit is shown in Fig. 1(b) [9,14]. The voltage was supplied by a DC power supply.

The positive temperature coefficient (PTC) was measured at a heating rate of 5 °C/min from 30 °C to 150 °C. Due to the large difference in the electrical resistivities of the samples at room temperature and high temperature, two machines with different measurement ranges were combined (i.e., R6441A digital multimeter and HP 4339B high resistivity meter for resistances lower and higher than 10^7 Ω, respectively).

The crosslinking percentage of the irradiated composites was determined using the extraction method to measure the insoluble content of the irradiated composites. The sample placed in the Soxhlet extractor was exposed to a refluxing boiling mixture of *m*-, *o*-, and *p*-xylene for 36 h at 160 °C [27,28]. The insoluble portion was then washed with ethanol and dried in a vacuum oven to a constant weight at 80 °C.

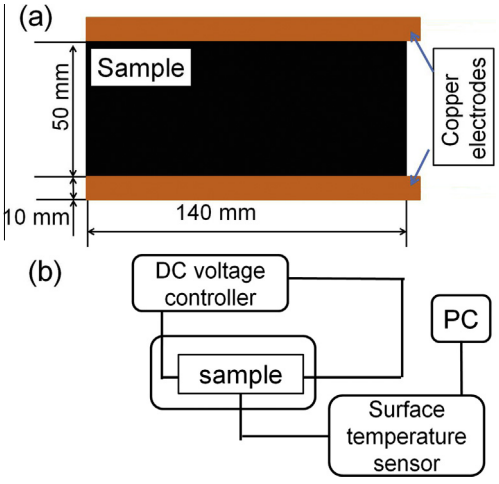

Fig. 1. (a) Samples and (b) measurement system used to examine self-heating. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The thermal properties of the composites were estimated using differential scanning calorimetry (DSC) on a DSC-Q20 (TA Instruments), and the experiments were performed at a heating rate of 10 °C/min from 10 to 160 °C.

The temperature dependence of the dynamic tensile modulus was measured for the composites using a viscoelastic spectrometer from Iwamoto Machine Co., Ltd. (Kyoto, Japan) at 10 Hz from 15 °C to 140 °C at a heating rate of 2 °C/min. The composite films were cut into strips with a length of 40.0 mm, a width of 2.0 mm, and a thickness approximately 0.2 mm. The applied external strain was 0.01 mm.

The coefficient of thermal expansion for the composites was estimated using a dynamic mechanical thermal analyzer (Q400, TA Instruments) at a heating rate of 5 °C/min from 20 °C to 110 °C under a preload of 0.02 N; the samples measured 5 mm in length, 5 mm in thickness and 5 mm in width.

Scanning electron microscopy (SEM) was performed using a Nava NanoSEM 450 (FEI Company, USA). The morphology of a cross-section of a composite film was observed.

3. Results and discussion

3.1. Electrical properties

Fig. 2 displayed the electrical conductivity of composite films with different CF contents at room temperature. As shown in Fig. 2, the electrical conductivity of the composite films was strongly dependent on the CF content. The electrical conductivity of the composite film containing 5 wt% CF was measured to be $\sim 10^{-11}$ S/cm. However, for the composite film containing 10 wt% CF, the electrical conductivity was measured to be ~0.1 S/cm, which indicated an increase of 10 orders of magnitude. This result showed a typical percolation behavior. The electrical conductivity of the composites with an increasing amount of conductive filler can be described by a scaling law according to classical percolation theory [1,29]:

$$\sigma \propto \sigma_0 (p - p_c)^t \quad (1)$$

where σ is the electrical conductivity of the composites, σ_0 is a scaling factor, p is the filler content in the composites (when $p > p_c$), p_c is the percolation threshold, and t is an exponent that is related to