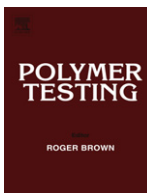




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Material properties

Preparation and properties of graphene oxide nanosheets/cyanate ester resin composites

Qilang Lin^{a,*}, Lijuan Qu^a, Qiufeng Lü^a, Changqing Fang^{b,*}^a College of Materials Science and Engineering, Fuzhou University, 2 Xue Yuan Road, University Town, Fuzhou, Fujian Province 350116, PR China^b College of Printing and Packing Engineering, Xi'an University of Technology, Xi'an 710048, PR China

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ABSTRACT

Graphene oxide nanosheets (GONSs)/cyanate ester (CE) resin composites were prepared via a solution intercalation method. The structures of the GONSs and the composites were studied using Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The mechanical and tribological properties of the composites were investigated. In addition, the thermal behavior of the composites was characterized by thermogravimetric analysis (TGA). Results show that the GONSs/CE resin composites were successfully prepared. The addition of GONSs is beneficial to improve the mechanical and tribological properties of the composites. Moreover, the composites exhibit better thermal stability in comparison with the CE resin matrix.

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1. Introduction

Polymer composites have been increasingly applied as structural materials in aerospace, automotive and chemical industries [1]. A number of these applications are tribological components, where the self-lubrication properties of polymer composites are of special advantage [2–4]. The addition of well-dispersed nanofillers into a polymer matrix has been demonstrated to be quite effective in improving the properties of the polymer matrix, including friction and wear properties [5]. It is known that graphene nanosheets possess extraordinary electronic, thermal and mechanical properties and, therefore, are promising candidates as nanofillers in polymer composites [6–8]. However, the pure graphene nanosheets cannot be dissolved or dispersed even after ultrasonic treatment for long

times, which severely limits their application in the manufacture of polymer composites [9,10].

To solve this problem, various approaches to modify graphene nanosheets by chemical means or non-covalent functionalizations have been reported [10–12], among which graphene oxide nanosheets (GONSs) can be easily synthesized [13]. Graphite oxide (GO), an oxygen-rich carbonaceous layered material, readily exfoliates in water under mild ultrasonic treatment to form stable dispersions of GONSs. However, since GO is hydrophilic, its direct exfoliation into non-aqueous solvents is not favored. Thus, it is necessary to make GO become less hydrophilic via chemical functionalization [14–16]. Stankovich et al. [17] have demonstrated that isocyanate-treated GO with reduced hydrophilic properties can be exfoliated into functionalized GONSs; the GONSs can form a stable dispersion in polar aprotic solvents such as *N,N*-dimethylformamide (DMF) and *N*-methyl-2-pyrrolidone (NMP).

Cyanate ester (CE) resins are a family of high-performance thermosetting resins, which are often used in production of friction materials. Compared with other kinds of thermosetting resins, CE resins possess

* Corresponding authors. Tel./fax: +86 591 22866531.

E-mail addresses: linqilang@hotmail.com (Q. Lin), fcqxaut@163.com (C. Fang).

outstanding thermal properties and superior dielectric behavior [18–20]. Many modified CE resin systems have been developed to meet the requirements of various applications. Among them, a CE resin system based on bisphenol A dicyanate ester (BADCy) and bismaleimide (BMI) has been proved to have good processability, superior mechanical and thermal properties [21]. In this paper, isocyanate-treated GO and the CE resin were used to prepare graphene oxide nanosheets (GONSs)/CE resin composites via a solution intercalation method, and then structures and properties of the composites were studied.

2. Experimental

2.1. Material

Natural graphite was supplied by Tianhe graphite Co., Ltd. (Qingdao, China). Bisphenol A dicyanate ester (BADCy) was obtained from Jinliyan curatorial Co., Ltd. (Zhejiang, China). 4,4'-bismaleimidodiphenyl methane (BDM), mp 156–158 °C, was purchased from the Fenguang Chemical Co., Ltd. (Honghu, China). 0,0'-diallylbisphenol A (DBA) was purchased from Fenguang Chemicals Co., Ltd. (Hubei, China). *N,N*-dimethylformamide (DMF) and methylene chloride were obtained from Guoyao Chemical reagent Co., Ltd. (Fujian, China). Phenyl isocyanate was supplied by Ouhe technology Co., Ltd. (Beijing, China).

2.2. Preparation of stable dispersions of graphene oxide nanosheets (GONSs)

Graphite oxide (GO) was prepared as reported by Hummers and Offeman [22], and phenyl isocyanate-treated graphite oxide (iGO) was obtained according to the procedure reported by Stankovich et al. [17]. Stable dispersions of GONSs were prepared by ultrasonic exfoliation (150 W) of the iGO for 1 h in DMF (1 mg/mL). Here, the amount of iGO added was considered as the amount of as-formed GONSs in the dispersion.

2.3. Preparation of CE prepolymer

BADCy was modified with a bismaleimide (BMI) prepolymer to improve its performance. The BMI prepolymer was prepared by mixing 20 g BDM and 20 g DBA at 120 °C until a clear homogeneous melt was achieved; and then 120 g BADCy was added and stirred at 130 °C for 30 min. The resultant liquid was defined as cyanate ester (CE) prepolymer.

2.4. Preparation of GONSs/CE resin composites

A certain amount of the CE prepolymer was added into the stable dispersion of GONSs in DMF and dissolved under stirring. The DMF was then evaporated with vacuum distillation. The mixture was stirred at 110 °C until a low viscosity was obtained, and then degassed to remove entrapped air at 140 °C for 1 h in a vacuum. After that, the mixture was cast into a mold for curing and postcuring with the sequence 150 °C/2 h + 180 °C/2 h + 200 °C/2 h and 240 °C/5 h. The GONSs/CE resin composites obtained were used for mechanical and tribological testing.

2.5. Characterization of the composites

FT-IR spectra were recorded on a Nicolet-5700 FT-IR spectrometer in the transmittance mode. The frequency of scanning for each spectrum was 15 times s^{-1} . KBr discs were prepared in the usual way from very well dried mixtures of about 1 mg sample and 100 mg KBr.

The carbon, hydrogen and nitrogen weight percentages of sample were determined using a Vario EL III elemental analyzer.

XRD analysis was carried out in a Philips X'Pert diffractometer with $CuK\alpha$ radiation. Scans were taken with 2θ from 1.2–30° and step size of 0.10° using a high resolution mode.

Transmission electron microscopy (TEM) was performed with a JEM-2000EX electron microscope. Stable dispersion of the GONSs was prepared by ultrasonic treatment. A drop of stable dispersion was placed on a copper grid and then dried before it was transferred to the sample chamber. The composites sample was trimmed using an ultramicrotome, and sliced into about 80 nm thickness with a diamond cutter.

For thermogravimetric analysis (TGA), ca 10 mg of sample was placed in a platinum crucible, and heated to 1000 °C at 10 °C min^{-1} under a nitrogen flow of 100 mL min^{-1} using a TA SDT-Q600 thermogravimetric analyzer.

Scanning electron microscopy (SEM) was carried out using a Philips XL30 environmental scanning electron microscope. The accelerated voltage was 20 kV, and the samples were coated with gold in vacuum.

The average value of ten replicated measurements of Vickers hardness was adopted as the microhardness of the specimens. Flexural strength and impact strength of the specimens were tested according to Chinese national standards GB3356-3382 and GB1843-1880, respectively.

Friction and wear tests at ambient temperature were performed on a MMW-1 friction and wear tester. The friction face of the test specimen was held in continuous sliding contact with the counter friction member in a ring-on-plate system, and a schematic diagram of the ring-on-plate system is given in Fig. 1. The counter friction member was carbon steel S45C (contact area of 2 cm^2) with the surface prepared using 1000 grade emery paper. The friction and wear tests were conducted at a rotating speed of 500 r/min and a load of 200 N with test duration of 30 min.

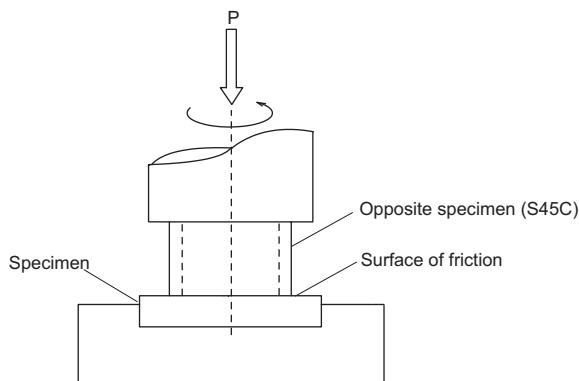


Fig. 1. A schematic diagram of the ring-on-plate system.

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