



Preparation and properties of graphene nanosheets/carbon foam composites



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ABSTRACT

Graphene nanosheets(GNs)/carbon foam composites were prepared by direct pyrolysis of GNs/cyanate ester (CE) resin mixtures at ambient pressure. Pyrolysis behaviors of the GNs/CE resin mixtures were studied using thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR). In addition, the effects of the additive amount of GNs on the microstructures, physical properties and compressive behaviors of the resulting GNs/carbon foam composites were investigated. Results show that the additive amount of GNs has great effect on the thermal stability of the CE resin as well as the structures and properties of the resulting composites. With the increasing of additive amount of GNs from 0 to 6 wt.%, the composite exhibits an increase in the bulk density and mechanical performance as well as structural uniformity. When the additive amount is 6 wt.%, its bulk density, compressive strength and modulus arrive at 0.38 g/cm³, 7.11 MPa and 115.9 MPa, respectively.

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1. Introduction

Carbon foams are lightweight porous carbon materials with three dimensional network structures, and therefore have been widely used in many fields [1,2], such as thermal control material, battery electrodes, catalyst carriers, and aerospace field [3–8]. However, the development and application of carbon foams is greatly restricted by their poor mechanical strengths and high costs [9]. In our previous work [10,11], we found that cyanate ester (CE) resins had self-foaming characteristics at high temperatures and consequently carbon foams could be prepared by direct pyrolysis of the CE resins at ambient pressure. The carbon foams prepared possessed well-developed pore structures without obvious cracks or voids, but their mechanical strengths were still low (about 3 MPa) and meanwhile their structural uniformity should be further improved [11]. Therefore, it is necessary to apply specific treatments to improve the mechanical strengths and structural uniformity of the resulting carbon foams from CE resins.

In recent years, several additives such as short carbon fiber [12,13], carbon nanofibers [14–17], clay-montmorillonite [18], and hollow ceramic microspheres [19] have been introduced into the matrix precursor to improve the performances of carbon foams, among which the addition of well-dispersed nanofillers has been

demonstrated to be quite effective in improving their mechanical strengths [15,20,21]. Graphene nanosheets (GNs), which possess extraordinary electronic, thermal and mechanical properties [22–26], are promising candidates as nanofillers in polymer composites [27–29]. Recently, Narasimman et al. [30] reported that graphene-reinforced carbon composite foams exhibited improved strength and EMI shielding from sucrose and graphene oxide. This suggests that the incorporation of GNs into carbon foam is effective for improving its mechanical strength. On the other hand, the structural uniformity of carbon foam from CE resin was closely related to the thermal distribution of the resin during pyrolysis [15]. The addition of GNs with excellent thermal properties to CE resin seems to be beneficial to the enhancement of structural uniformity of resulting carbon foam. In this work, we attempt the fabrication of GNs/carbon foam composites by ambient pressure carbonization of the GNs/CE resin mixtures, and then structures and properties of the composites were investigated.

2. Experimental

2.1. Raw materials

Bisphenol A cyanate ester (99% pure) was purchased from Jinliyan curatorial Co., Ltd., China. Bisphenol A epoxy resin (E-51) was purchased from Guangzhou Lihou trading Co., Ltd., China. Dichloromethane (CH₂Cl₂) was in analytical grade and supplied by Guoyao Chemical reagent Co., Ltd., China. Graphene nanosheets

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(GNs) were prepared according to our previous work [22]. The GNs obtained had an average thickness of 3.95 nm and a high C/O molar ratio of 60:1 with a crystalline structure.

2.2. Preparation of GNs/CE resin mixtures

For the preparation of GNs/CE resin mixtures, a fixed mass ratio of Bisphenol A epoxy resin to bisphenol A cyanate ester (1:10) was used and the additive amount of GNs was a certain weight percentage of bisphenol A cyanate ester containing EP resin (0 wt.%, 2 wt.%, 4 wt.%, 6 wt.%, or 8 wt.%). The preparation process was described as follows: Bisphenol A epoxy resin and GNs were milled for 3 h at a rotation speed of 900 rpm in a planetary ball mill with addition of CH_2Cl_2 as solvent. The mixture was then mixed with bisphenol A cyanate ester under vigorous stirring at 130 °C for 30 min to obtain a homogeneous melt. The CH_2Cl_2 was evaporated with vacuum distillation. Afterwards, the melt was degassed in a vacuum oven at 140 °C for 30 min, and then cast into a steel mold for curing via the procedures of 160 °C/2 h + 180 °C/2 h + 200 °C/2 h + 220 °C/4 h. The resulting GNs/CE resin mixtures were labeled mixture-0, mixture-2, mixture-4, mixture-6 and mixture-8 when the additive amount of GNs was 0 wt.%, 2 wt.%, 4 wt.%, 6 wt.% and 8 wt.%, respectively.

2.3. Preparation of GNs/carbon foam composites

The cured GNs/CE resin mixtures were heated up to 900 °C at a rate of 5 °C/min and kept for 1 h under a nitrogen atmosphere, and then cooled naturally to room temperature. The smooth surfaces of the pyrolysis products were ground off using sandpaper to obtain resultant GNs/carbon foam composites. The composites obtained were labeled GCFC-0, GCFC-2, GCFC-4, GCFC-6 and GCFC-8 when the mixture used was mixture-0, mixture-2, mixture-4, mixture-6 and mixture-8, respectively.

2.4. Characterization and measurements

Thermogravimetric analysis (TGA) was performed on a thermogravimetric analyzer (TASDT-Q600) at a heating rate of 10 °C min⁻¹ under a nitrogen flow of 100 ml min⁻¹. Fourier transform infrared (FTIR) spectra were recorded on a FTIR spectrophotometer (Nicolet 5700) in KBr pellets. X-ray diffraction (XRD) patterns were recorded on a X-ray diffractometer (Ultima III) using Cu K α radiation at $\lambda = 0.15406$ nm. Morphological measurements were performed using a field emission scanning electron microscope (FESEM, Carl Zeiss Supra 55). The electrical conductivity, bulk density, porosity

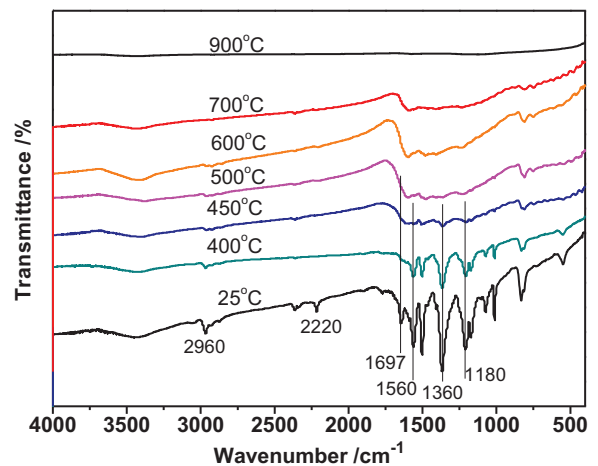


Fig. 1. FT-IR spectra of the GNs/CE resin mixture (mixture-6) at different temperatures.

and percentage of open cell were determined according to Ref. [14]. For the compressive tests, the samples were machined to cylinders of 12 mm in diameter and 10 mm in height. The tests were carried out at room temperature on an electron universal testing machine (model CMT-5505) with a cross-head speed of 2.5 mm/min, and all the results were calculated based on the average of five tests.

3. Results and discussion

3.1. Pyrolysis behaviors of the GNs/CE resin mixtures

Fig. 1 shows the FT-IR spectra of the GNs/CE resin mixture (mixture-6) at different temperatures. When the temperature is 25 °C, there exist the obvious characteristic absorption peaks at 1570 cm⁻¹, 1180 cm⁻¹ and 1360 cm⁻¹, corresponding to the stretching vibrations of triazine ring [10]. In addition, it exhibits the weak characteristic absorption peak of -OCN at 2220 cm⁻¹ and the obvious characteristic absorption peak of isocyanate urea at 1679 cm⁻¹ [31], but the characteristic absorption peak of cyclic ether cannot be observed, indicating that epoxy resin had completely reacted with cyanate ester and meanwhile the CE resin was not fully cured when preparing the mixture. When the temperature is 400 °C, the characteristic absorption peak of -OCN disappears whereas other characteristic absorption peaks still exist. This suggests that the CE resin was fully cured at this temperature and had

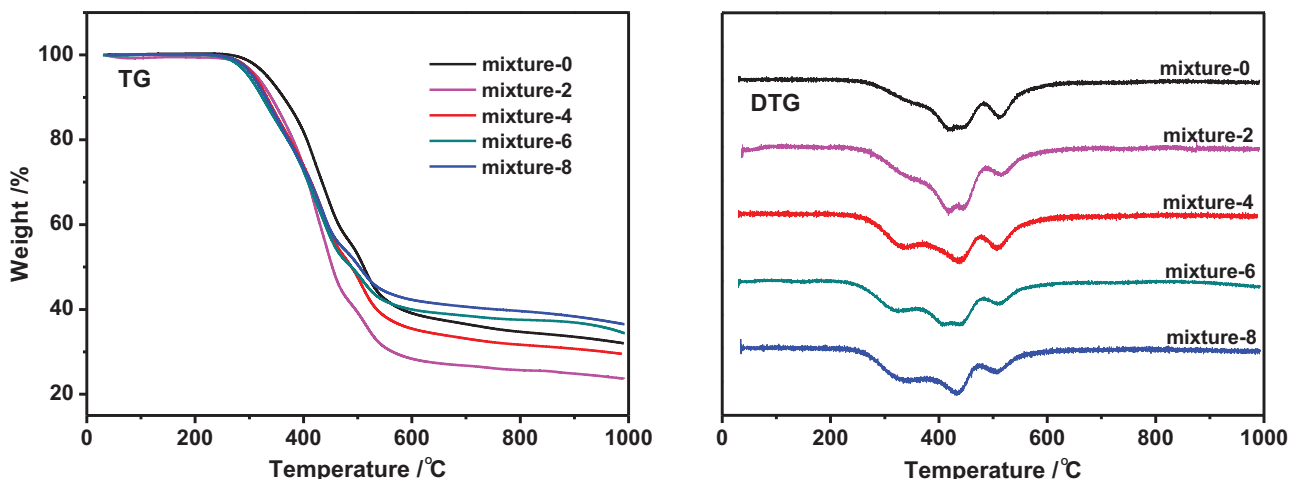


Fig. 2. TG-DTG curves of the GNs/CE resin mixtures prepared with different additive amount of GNs.

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