



Research paper

Preparation and properties of montmorillonite/carbon foam nanocomposites

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ARTICLE INFO

Article history:

Received 8 December 2016

Received in revised form 24 January 2017

Accepted 30 January 2017

Available online xxxx

Keywords:

Carbon foam

Montmorillonite

Nanocomposite

Microstructure

Property

ABSTRACT

Montmorillonite (Mt)/carbon foam nanocomposites were prepared by direct carbonization of organic montmorillonite (OMt)/bismaleimide (BMI) resin mixtures at ambient pressure. Carbonization behaviors of the OMt/BMI resin mixtures were studied using X-ray diffraction (XRD) and thermogravimetric analysis (TGA). In addition, the influences of the additive amount of OMt on the microstructures, physical properties and compressive behaviors and oxidation resistance of the resulting nanocomposites were investigated. Results show that with the increasing of the additive amount of OMt, the pore walls of the nanocomposites become thick and their average pore sizes decrease. When the additive amount of OMt varies from 0 to 1.5 wt%, the mechanical properties, bulk density and oxidation resistance of the nanocomposites are improved and their thermal conductivity is markedly decreased. For the nanocomposite prepared with 1.5 wt% of OMt, its bulk density, thermal conductivity, compressive strength and modulus arrive at 0.35 g/cm³, 0.108 W/m·K, 6.90 MPa and 207 MPa, respectively.

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1. Introduction

Carbon foam, a novel structural and functional material, has attracted more and more considerable attention from both scientific research and applications (Chen et al., 2015). Due to its low density, high porosity and excellent thermal properties (Wang et al., 2009; Fawcett and Shetty, 2010; Lin et al., 2013; Liu et al., 2014), carbon foam has been widely used in many fields ranging from aeronautics, astronautics, chemicals, electronics, energy, environmental protection to catalysis (Friedrich et al., 2004; Luo et al., 2011; Narasimman and Prabhakaran, 2012; Narasimman et al., 2015). However, poor mechanical strengths and high production costs of carbon foams hinder their development and application (Li et al., 2007). As is well known, lots of thermosetting polymers like phenolic resin were adopted as precursors to obtain carbon foams with low thermal conductivity through the conventional process of preparation and stabilization of polymeric foams followed by carbonization (Chen et al., 2006; Liu et al., 2007; Lorjai et al., 2009; Lin et al., 2014). During the process, the polymeric foams underwent thermal decomposition causing large shrinkage stress, which led to the formation of structural defects like cracks and voids thus deteriorating the mechanical strengths of resultant carbon foams. Meanwhile, complex-shaped carbon foams could not be directly fabricated owing

to the large shrinkage stress, but generally had to first be prepared in block shape, which necessitated a series of subsequent machining or grinding steps (Tsytarski et al., 2010; Lin et al., 2013). This resulted in further increase in their production costs. Therefore, it is hoped to develop a simple method to fabricate carbon foams with higher mechanical strength and more flexible shapes.

As a solution, allyl condensed polynuclear aromatic (COPNA)-modified bismaleimide (BMI) resin was introduced to fabricate carbon foams (Liu et al., 2016; Wang et al., 2016). The results showed that near net-shape carbon foam with well-developed pore structures could be fabricated directly by carbonization of casting part from the allyl COPNA-modified BMI (ACMB) resin. The ACMB resin could easily keep its complex shape upon carbonization, which was attributed to its self-foaming characteristics leading to effective removal of shrinkage stress during thermal decomposition (Liu et al., 2016). The resulting carbon foam had a compressive strength of 2.37 MPa with a bulk density of 0.26 g/cm³ and a thermal conductivity of 0.44 W/m·K when prepared at a carbonization temperature of 900°C, suggesting it could be a promising thermal insulation material. Nevertheless, it is necessary to apply specific approaches to further improve the mechanical strength of the carbon foam and reduce its thermal conductivity as well when intended for use as thermal insulation.

In recent years, various approaches have been developed to improve the mechanical strengths of carbon foams (Lin et al., 2014), among which the addition of well-dispersed nanofillers into carbon foams has been demonstrated to be quite effective. Organic montmorillonite

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(OMt) is a kind of layered nano-silicates treated covering the chain length and acidity (Shi et al., 1996) as well as the compatibility (Meng and Hu, 2004) with the monomers to be intercalated, which has small size effect, surface effect and interface effect (Jo et al., 2008; Gărea et al., 2010; Liu et al., 2013; Yu et al., 2015), and often used as a nanofiller to prepare polymer nanocomposites for improving not only the mechanical properties of the polymer but also some of the physical properties like barrier and thermal properties (Alexandre and Dubois, 2000; Suprakas and Masami, 2003; Cao et al., 2005; Wang et al., 2006; Mirabedini et al., 2012; Mazeyar and Eslami, 2012). More importantly, only the exfoliated state, in which silicate nano-layers are delaminated and individually dispersed in the polymer matrix, can give the resulting nanocomposites with excellent properties (Meng and Hu, 2004). In this work, montmorillonite (Mt)/carbon foam nanocomposites were fabricated by ambient pressure carbonization of the OMt/ACMB resin mixtures prepared via in situ polymerization, and structures and properties of the resulting Mt/carbon foam nanocomposites were investigated.

2. Experimental

2.1. Materials

OMt, DK4, modified with dimethyl dehydrogenate tallow ammonium chloride, was supplied by Zhejiang Fenghong New Material Co., Ltd. (Zhejiang, China). 4,4-bismaleimidodiphenyl methane (BDM), mp 156–158°C, was obtained from the Fenguang Chemical Co., Ltd. (Honghu, China). Dichloromethane (CP) was purchased from the Shanghai Chemical Reagent Corporation (Shanghai, China). Allyl condensed polynuclear aromatic (AC) resin was prepared according to the procedures described in the literature (Lin et al., 2010). The AC resin obtained had a softening point of 52.6°C and an allylation degree of 93.5%.

2.2. Preparation of OMt/ACMB resin mixtures

For the preparation of OMt/ACMB resin mixtures, a fixed mass ratio of AC resin and BDM (1:1.1) was used to prepare ACMB resin, and the ACMB resin containing weight fractions (0 wt%, 0.5 wt%, 1.0 wt%, and 1.5 wt%) of OMt were prepared. The preparation process was described as follows: OMt and AC resin were mixed thoroughly in dichloromethane at 60°C and sonicated for 30 min using a high-power (240 W) ultrasonic machine. Afterward the dichloromethane was evaporated with vacuum distillation. BDM was added into the mixture of OMt and AC resin and vigorously stirred at 150°C for 30 min until a homogeneous melt was achieved. The melt was then degassed to remove entrapped air at 140°C for 1 h in a vacuum oven. After that, the melt was cast into molds, followed by curing via the procedures of 150°C/2 h + 180°C/2 h + 220°C/2 h + 250°C/2 h. The resulting OMt/ACMB resin mixtures were labeled mixture-0, mixture-0.5, mixture-1, mixture-1.5, and mixture-2 when the additive amount of OMt was 0 wt%, 0.5 wt%, 1.0 wt%, 1.5 wt%, and 2 wt%, respectively.

2.3. Preparation of Mt/carbon foam nanocomposites

The OMt/ACMB resin mixtures were put into a furnace and heated up to 900°C at a rate of 2°C/min in argon atmosphere and maintained for 1 h, and then cooled naturally to room temperature to obtain resultant carbonization products. The smooth surfaces of the carbonization products obtained were ground off using sandpaper to achieve Mt/carbon foam nanocomposites. The resulting Mt/carbon foam nanocomposites were labeled MCF-0, MCF-0.5, MCF-1, MCF-1.5, and MCF-2 when the OMt/ACMB resin mixture used was mixture-0, mixture-0.5, mixture-1, mixture-1.5, and mixture-2, respectively.

2.4. Characterization

2.4.1. Thermogravimetric analysis (TGA)

For TGA of OMt/ACMB resin mixtures, ca 10 mg of sample, placed in a platinum crucible, was heated to 1000°C at 10°C min⁻¹ under a nitrogen flow of 100 mL/min using a TA SDT-Q600 thermogravimetric analyzer.

2.4.2. X-ray diffraction (XRD)

XRD analysis of OMt/ACMB resin mixtures was carried out in PHILIPS X'Pert diffractometer with Cu K α radiation. Scans were taken from 2 θ at 1° to 10°, step size 0.10°, and scan time per step of 20 s using the high resolution mode. XRD analysis of Mt/carbon foam nanocomposites was carried out in Rigaku Ultima III diffractometer with Cu K α radiation. XRD was performed on powder samples with a 40 kV generator voltage and a 35 mA current, and the scan range 2 θ = 10–70°.

2.4.3. Scanning electron microscopy (SEM)

Morphological measurements of samples were performed using a field emission electron microscope (FESEM, Carl Zeiss ULTRA 55 FESEM).

2.4.4. Bulk density test

Bulk density of the samples was determined by weighing the geometrically shaped foam and dividing the weigh by the density of the samples was determined by weighing the geometrically shaped foam and dividing the weigh by the measured volume. The true density of the samples was measured by a helium pycnometer (Micromeritics AccuPyc 1330). The porosity and percentage of open cell were calculated by the following formula:

$$\text{Porosity (\%)} = (1 - D^*/D_p) \times 100, \quad (1)$$

$$\text{Open cell (\%)} = D_s/D_p \times 100, \quad (2)$$

where D^* is the bulk density, D_s is the skeleton density of the sample, D_p is the true density of pulverized sample.

2.4.5. Electrical conductivity test

A four probe technique was used to measure the electrical conductivity of the samples. The electrical conductivity was measured on both sides of each specimen and the two measured values were then averaged. All the results were calculated based on the average of ten tests.

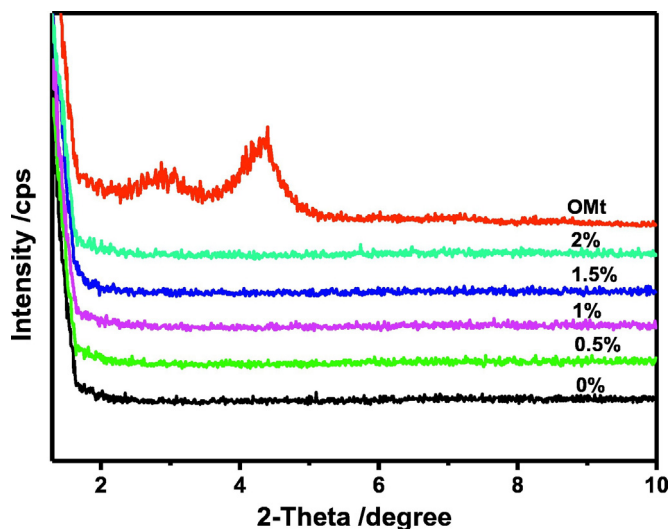


Fig. 1. XRD patterns of the OMt/ACMB resin mixtures prepared with different additive amount of OMt.

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