



Reinforcing epoxy resin with activated carbon: A way of high rate of quality and price



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ABSTRACT

For the worldwide rapid growing pressure from environmental pollution and energy shortages, the lightweight epoxy based materials, which owe great potential to be qualified structure materials to replace steel alloys on transportations, are attracting increasing attentions. A way of high rate of quality and price to reinforce the epoxy is always desired. In this work, activated carbon (AC) was found to be an effective reinforcing nanofiller. A comprehensive improvement (tensile properties, fracture toughness, impact strength and dynamic mechanical properties) occurred after AC was further modified by nitrogen groups. The modification was simple, economic, efficient and eco-friendly. The reinforcing method for the epoxy matrix was of high rate of quality and price. The reinforcing mechanism was discussed. The present work aims to give out some ideas for the fabrication of low-price high-performance nanocomposites.

1. Introduction

Nowadays, the popularization of various kinds of transportations makes the consumption of petroleum and the emission of gases that are not eco-friendly by transportations cannot be ignored. With the increasing concerns on the energy shortages and environmental pollution from every corner of the world, designing lightweight materials to replace conventional steel alloys on transportations, which could effectively reduce the consumption of petroleum and the emission of exhausts, becomes more and more welcome. Characteristics like high specific strength, excellent weatherability and chemical resistance endow the epoxy based composite with great potential to be an outstanding structure material [1–3]. The lightweight nature of epoxy could largely reduce the body weight of transportations, and therefore reduce the energy consumption and the exhausts emission of cars, ships and airplanes. However, to be a qualified structure material for transportations, at least enough impact strength to protect transportations from damage, and enough fracture toughness to protect lives and properties inside the transportation should be necessary. For the high cross-linking degree, cured epoxy resin is always quite brittle, which largely limits the application [1]. To further extend the application scenarios, various nanofillers were used to reinforce the epoxy. The tensile strength, failure strain and fracture toughness at 77 K of the epoxy resins could be simultaneously enhanced by introducing modified nano-rubber in, however, the Young's modulus decrease a little

[4]. The incorporation of nanoclay could enhance the mechanical and fracture toughness of cured epoxy resins, nevertheless, it always occurred at a high nanoclay loading of around 5.0 wt% [1]. For modified silica nanoparticles, even a loading amount of as high as 20.2 wt% was applied to reinforce the Young's modulus of the neat epoxy [5]. Nanocarbons are no doubt one of the most flamboyant stars among various nanofillers [6]. The superior mechanical properties of nanocarbons (e.g. the Young's modulus of carbon nanotube (CNT) ranges from 600 to 1.4 TPa and tensile strength ranges from 10 to 200 GPa [7]; the Young's modulus of graphene ranges from 0.2 to 1.0 TPa and the tensile strength ranges from 90 to 130 GPa [8]) could help to improve the mechanical performance of the composite obviously at low loading amounts [2,9]. And at the same time, the advantage of low weight is kept. However, the relative high price of these nanocarbons largely limits the large-scale applications in industry.

In the family of carbon materials, AC should be one of the most common and low-price members. ACs are usually results of carbonization of biomass, therefore, they are always rich of oxygen groups. Thanks to the highly developed porous structure and large specific surface area, ACs are extensively used as adsorbents [10]. And, AC from biomass was reported with considerable mechanical strength [11,12]. Though AC has no mechanical properties as good as CNT and graphene, the highly developed porous structure and large specific surface area could induce widely existed physical interactions with epoxy, and the rich oxygen group could induce chemical interactions with epoxy.

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What's more, the active carbon is normally hundreds times cheaper than CNT or graphene. In the present work, AC/epoxy and modified AC/epoxy composites were reported to try to give out some ideas about composites of high rate of quality and price for transportations.

2. Experimental section

The AC chosen in this work was from Jiangsu Zuxi Activated Carbon Co., Ltd. The epoxy (zy5401A) and curing agent (zy5401B) used were from Nanjing Zhongying New Nanomaterials Co., Ltd. Concentrated aqueous ammonia was supplied by Sinopharm Chemical Reagent Co.

NAC was obtained after treating AC with aqueous ammonia solution by a simple, economic, efficient and eco-friendly method. Detailed information about the method could be found in our previous work [13].

The AC/epoxy nanocomposites were prepared with selected AC to zy5401A weight ratios. The mill technology was used to uniformly disperse AC into epoxy. 280 agate balls (6 mm) were equipped in two 250 mL agate vessels. The program was set at 40 Hz for 2 h in the same direction. zy5401B was then added in the obtained mixture with a zy5401A to zy5401B weight ratio of 3.62. After uniformly stirred, the final mixture was poured into stainless molds and cured at 80 °C for 2 h and then 100 °C for another 2 h.

Transmission electron microscopy (TEM) images were obtained by FEI T12. Scanning electronic microscopy (SEM) images were taken on FEI nano-450 after the fracture surface was coated by Au. The X-ray photoelectron spectroscopy (XPS) results were from an ESCALAB 250 XPS system with a monochromatized Al K α X-ray source (1486.6 eV). Tensile properties were tested on SHIMADZU AG-X (10 kN) mechanical testing machine following GB/T 2567-2008. Impact tests were carried out on Zwick/Roell HIT50P following GB/T 2567-2008. Dynamic mechanical analysis (DMA) results were obtained under the single-cantilever blending mode using NETZSCH DMA 242 E from 30 °C to 250 °C (3 °C/min) at 1 Hz.

Fracture toughness was tested following ASTM D5045-14 using SHIMADZU AG-X (10 kN). Single-edge notch-bend specimens were prepared for tests. The sample dimensions were 5 × 10 × 44 mm³, and the sharp notch (5 mm-deep) was prepared by machining.

For every mechanical test, at least 5 specimens were tested to make sure the results.

3. Results and discussion

AC was incorporated into the epoxy matrix with different loadings. The impact strength, fracture toughness, DMA results and tensile properties, which are properties that always cared by transportation manufacturers, are reported and summarized in Table 1. Unlike nanodiamond or CNT, the mechanical performance of which always undergo a process that increase firstly and then decrease [14–16], varying the AC loading amount from 0.3% to 2%, the comprehensive mechanical performances of AC/epoxy composites just decrease. The best performance was obtained at 0.3% AC loading. After 0.3% AC was incorporated, the tensile strength increased by 19.4%, the tensile modulus increased by 30.7%, K_{1c} (the mode-I critical-stress intensity factor)

increased by 204.3%, G_{1c} (the mode-I critical strain energy release rate) increased by 607%, and G'30 (the storage modulus at 30 °C) increased by 24.6%. However, the elongation at break, the impact strength, and the glass transition temperature (T_g) showed some decrease. The incorporation of AC made a malformed reinforcement to the epoxy matrix. Just using AC to reinforce the epoxy matrix, when some properties were reinforced, some other properties that we cared were damaged. This is unexpected.

Researches showed that modifying nanocarbons with nitrogen groups could further improve the mechanical performance [14,17]. After AC was modified by nitrogen groups, the toughness of NAC/epoxy nanocomposite improved obviously. Compared with the 0.3% AC/epoxy nanocomposite, the elongation at break of the 0.3% NAC/epoxy nanocomposite increased by 92.7%, the impact strength increased by 32%, K_{1c} increased by 9.9%, G_{1c} increased by 60.5%, while the tensile strength, tensile modulus and G'30 showed some decrease. However, NAC made an obvious overall reinforcement to the epoxy matrix in almost all the properties that we concerned. Tensile strength, elongation at break, impact strength, K_{1c} and G_{1c} increases obviously, at the same time, without sacrificing tensile modulus, G'30 and T_g. The exciting reinforcement of NAC could also be easily sensed from the SEM images of the fracture surface taking from the specimens after the fracture toughness tests (Figure S1). After AC was replaced by NAC, the fracture surface was no longer typical brittle fracture surfaces. The fracture surface of the 0.3% NAC/epoxy nanocomposite was much rougher than others, which means the 0.3% NAC/epoxy nanocomposite has more ability to struggle against the external force.

To understand the reinforcing effects by AC and NAC, the structure and surface chemistry of AC and NAC were studied. The AC used was the carbonization product of coconut shells, with a specific surface area as large as 1500 m²/g. The porous structure and large specific surface area enabled a strong and widely existed physical interaction. From Fig. 1a, the amorphous structure of AC could be seen. In the high-magnification image of AC (Fig. 1b), graphene layers appear. Graphene is a material of excellent mechanical properties, and it could effectively reinforce the epoxy matrix [2,18]. Then, the improved mechanical performance should be mainly contributed by the graphene layers.

By XPS, it is known that the surface oxygen content of the AC chosen is as high as 10.6 at%. The XPS O 1s spectra of AC and NAC could be deconvoluted into four peaks at 530.7 eV, 531.5–531.8 eV, 532.5–532.8 eV, 533.5–533.9 eV, corresponding to highly conjugated forms of carbonyl oxygen such as quinone groups, C=O (ketonic carbonyl groups), O=C-O (a sum of carboxyl, anhydride, lactone, and ester groups), C-OH (phenol groups), respectively [19] (Fig. 2). From the deconvolution results (Table S1), it is known that most oxygen groups of AC are O=C-O species (2.97 at%) and C-OH (5.14 at%), which could react or induce some other kinds of interactions (such as hydrogen bond) with the epoxy group of epoxy resins. The high-content reactive oxygen groups of AC could result to widely existed interactions between AC and epoxy, which guaranteed the load transfer from epoxy matrix to AC. After AC was further modified with nitrogen groups, the surface oxygen content of NAC was 9.1 at%, which was a little lower than that of AC. From Table S1, it is found that the decrease of the

Table 1
The mechanical performances of the as-obtained samples.

Samples	Tensile strength (MPa)	Tensile Modulus (GPa)	Elongation at break (%)	Impact strength (kJ/m ²)	K _{1c} (Mpa m ^{1/2})	G _{1c} (kJ/m ²)	G'30 (MPa)	T _g (°C)
Neat epoxy	65.0 ± 2.4	2.77 ± 0.13	5.5 ± 0.6	26.3 ± 2.1	1.16 ± 0.10	0.43 ± 0.03	757.6	106.9
0.3% AC/epoxy	77.6 ± 0.63	3.62 ± 0.03	4.1 ± 0.2	20.7 ± 1.8	3.53 ± 0.20	3.04 ± 0.12	943.9	105.0
0.3% NAC/epoxy	69.7 ± 1.5	2.73 ± 0.06	7.9 ± 0.3	33.0 ± 3.1	3.88 ± 0.06	4.88 ± 0.08	755.1	106.9
0.5% AC/epoxy	61.8 ± 3.1	3.58 ± 0.05	2.4 ± 0.3	6.5 ± 0.8	2.75 ± 0.08	1.87 ± 0.04	953.8	104.7
1% AC/epoxy	54.0 ± 1.3	3.47 ± 0.03	1.9 ± 0.2	5.3 ± 0.7	2.56 ± 0.06	1.67 ± 0.03	887.9	104.7
2% AC/epoxy	45.5 ± 1.6	3.67 ± 0.14	1.6 ± 0.1	6.5 ± 0.2	2.14 ± 0.10	1.10 ± 0.03	798.3	105.0

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