



High-performance graphene oxide/carbon nanotubes aerogel-polystyrene composites: Preparation and mechanical properties

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ABSTRACT

High-performance graphene oxide/carbon nanotube aerogel-polystyrene (GOCA-PS) composites were successfully prepared. Three-dimensional (3D) GOCA was prepared by self-assembly and a freeze-drying method. The synthesized 3D aerogel has highly porous structure, low density, and good mechanical properties. Then PS was backfilled into the pores of the aerogel by in situ polymerization via vacuum assisted impregnation process to prepared GOCA-PS composites, which has extremely high microhardness (807.4 MPa) and compression modulus (1029.9 MPa) with only 0.41 wt% GO and 0.16 wt% carbon nanotubes.

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1. Introduction

Recently, graphene aerogel (GA) [1], a three dimensional (3D) monolithic graphene framework assembled by individual graphene sheets, has received great attention for its unique structure and properties due to their potential applications in oil absorption [2], catalysis [3], sensors [4] and supercapacitors [5], largely due to its outstanding performance with a low density, large specific surface area, and high electrical [6] and thermal conductivity [7]. The recent success of 3D GA fabrication and resulting properties has provided a new avenue for preparing new composites. Compared with conventional processing methods of graphene composites, GA resolves the problem of dispersion in polymers [8]. Recently, some studies have examined the backfilling of GA with polymers and successfully fabricated high performance GA/polymer composites [9]. For example, GA-Poly(methyl methacrylate) and GA-polydimethylsiloxane composites exhibited outstanding electrical and thermal properties [10,11]. Although the electrical conductivity of the GA/polymer composites improved, the mechanical properties were unchanged compared with pure polymers, mainly because of the weak interfacial interaction between GA and the polymer, resulting in the low infusion efficiency of the composites [10].

In the contrast, graphene oxide (GO) nanocomposites have wide application in many field such as, conductive switch, vivo bio-imaging [12,13]. GO has abundant oxygenated functional groups compared with original graphene [14]. These functional groups can reduce interplanar forces and improve the interfacial interaction between GO and polymers. Therefore, if the GO aerogel (GOA) is prepared and filled with polymers, the mechanical properties of the obtained composites were enhanced greatly.

Inspired by GAs, we prepared a 3D graphene oxide/carbon nanotubes aerogel (GOCA) by self-assembly sol-gel and freeze-drying. The GOCA was then backfilled with polystyrene (PS) which was in situ polymerized from the styrene monomer and infiltrated into the aerogel assisted by vacuum. The synthesized GOCA-PS composites had a high microhardness, compression modulus, and good mechanical properties. The composites will be used as engineering materials in the future.

2. Experiments

2.1. Preparation of GOCA

GO was prepared by a modified Hummers method [15]. Then, GOCA was fabricated by self-assembly and freeze-drying. Homogeneous carbon nanotube (CNT)/GO aqueous suspensions were prepared in a beaker loaded with 30 wt% CNTs and 70 wt% GO, and then placed in sample vials and frozen for 12 h at -25°C . GOCA was synthesized after freeze-drying for 36 h in a freeze dryer

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(SC21CL). The prepared sample named as GOC₃₀A (30 in GOC₃₀A means the mass content of carbon nanotubes in aerogel was 30%). For contrast, preparation of GOC₁₀A, GOC₂₀A, GOC₄₀A, and GOC₅₀A were similar to that of GOC₃₀A. Effects of more CNT content on the composites were added in Table 1.

2.2. Preparation of GOCA-PS composites

The GOCA-PS composites were prepared via in-situ polymerization. First of all, 20 g of styrene and 70 mg of azobisisobutyronitrile (AIBN) were mixed. Next, the aerogel was added into the above mixture to expel air bubbles in the mixture with a vacuum assisted method. Then, the mixture was heated at 70 °C for 8–12 h and 80 °C for 12 h, until it completely solidified. Finally, the GOCA-PS composites were obtained after continuous reaction for 12 h at 90 °C. To investigate the effect of CNTs on the composites, another sample of GOA was prepared under the same conditions. For contrast, GOC₁₀A-PS, GOC₂₀A-PS, GOC₃₀A-PS, GOC₄₀A-PS, and GOC₅₀A-PS were prepared.

2.3. Characterization

Thermal gravimetric analysis (TGA, Shimadzu DTG60H) was carried out on a thermal analyzer under a nitrogen atmosphere at a heating rate of 10 °C/min. The morphologies of the aerogels and composites were observed with a JSM-7800F field emission-scanning electron microscope (Hitachi, Japan) at an accelerating voltage of 10 kV. Microhardness was measured by HXD-1000 digital microhardness tester using indentation force of 245 mN for a duration of 15 s. Compressive strength, compression modulus, and stress-strain curves of the composites were investigated by A1-7000 tensile testing machine (Taiwan, China) at a compression speed of 1 mm/min over a stress range of 0–30,000 N, with cylindrical samples (diameter of 20 mm, height of 20 mm).

3. Results and discussion

Compared to all the samples, the properties of GOC₃₀A-PS are better than that of others. The TG curves of pure PS, GOC₃₀A-PS, and GOA-PS composites are shown in Fig. 1. The thermal stability of the GOA-PS and GOC₃₀A-PS composites appeared remarkably improved, which can not only be attributed to the incorporation of homogeneously dispersed GO and modified CNTs, but also to the strong interaction between the aerogel and PS, which acts as a barrier to restrain the mobility of PS chains during pyrolysis [16].

Fig. 2 shows the morphology of the aerogels and composites. Seen from 2a and b, the microstructure of GOC₃₀A (Fig. 2a and b) is like honeycomb (pore size of 50–80 μm) consisting of unfolded oxidized graphene sheets. Compared with GOA (Fig. 2c and d), the pore structure of GOC₃₀A is uniform and regular, with a thick and stable pore wall. This is likely because the modified CNTs can interact strongly with GO. Fig. 2e and f show that GOC₃₀A and GOA 3D networks are almost completely infiltrated with PS. As

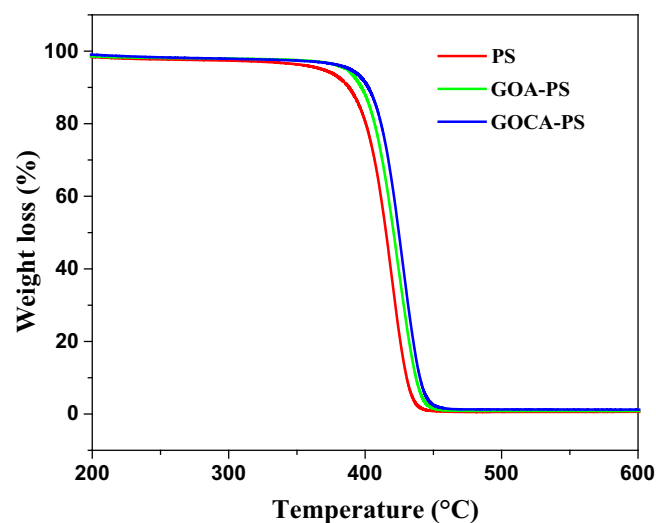


Fig. 1. TG curve of PS, GOA-PS, GOC₃₀A-PS composites.

GOC₃₀A has stable structure due to the introduction of modified CNTs, the morphology of the GOC₃₀A-PS composites is uniform. Therefore, the structure of GOC₃₀A is not easily destroyed during backfilling with PS, which resulted in the high infusion efficiency and density of the GOC₃₀A-PS composites. Specifically, if the densities of GO, modified CNTs, and PS are assumed to be 2.10, 2.20, and 1.05 g/cm³, respectively, the theoretical densities of the GOC₃₀A-PS and GOA-PS composites can be calculated to be 1.0364 and 1.0355 g/cm³, respectively. The actual density value is slightly lesser than the theoretical density because the composite aerogel cannot be filled completely (Table 1).

To studies the mechanical behavior, the compressive properties of the prepared composites were investigated with a tensile testing machine. The stress-strain curves of PS, GOA-PS, and GOC₃₀A-PS are shown in Fig. 3a. Although the stress-strain curve of PS shows that it bears the largest stress (100 MPa), the steep slope of the curve is the smallest among the three curves. Compared with PS, the GOC₃₀A-PS and GOA-PS composites bear greater stress when the strain ranges from 0 to 8%. Especially for GOC₃₀A-PS, it not only bears 83 MPa compressive stress at the strain of 8%, but also has the biggest compression modulus according to the relation $E = \sigma / \varepsilon$ (where σ and ε are the stress value and corresponding strain value of the linear range of stress-strain curves, respectively). As shown in Table 1, GOA-PS and GOC₃₀A-PS exhibit outstanding compression modulus with only 0.41 wt% of GO and 0.16 wt% of modified CNTs, and are superior to that of pure PS.

The microhardness of PS, GOA-PS, and GOC₃₀A-PS was measured with a digital microhardness tester. Fig. 3b shows a photo of the indentation of the GOC₃₀A-PS composites after the microhardness test. The specific microhardness values are listed in Table 1. At the same content of GO (0.41 wt%), though the GOC₃₀A-PS composites contains 0.16 wt% of modified CNT compared

Table 1
Properties and composition of the composites.

Sample	GO (wt%)	CNT (wt%)	Aerogels Porosity (%)	Backfill rate (%)	Density (g/cm ³)	Compression Modulus (MPa)	Microhardness (MPa)
PS	–	–	–	–	1.032	789.8	190.4
GOA-PS	0.41	0	99.80	>96	0.994	943.5	698.3
GOC ₁₀ A-PS	0.41	0.043	99.78	>96	0.997	987.2	756.4
GOC ₂₀ A-PS	0.41	0.097	99.75	>96	1.008	1008.3	784.5
GOC ₃₀ A-PS	0.41	0.16	99.72	>96	1.016	1029.9	807.4
GOC ₄₀ A-PS	0.41	0.258	99.69	>96	1.017	1035.2	821.7
GOC ₅₀ A-PS	0.41	0.385	99.61	>96	1.014	1024.6	829.4

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