Composite Structures 92 (2010) 2984–2991

Contents lists available at ScienceDirect

Composite Structures

journal homepage: www.elsevier.com/locate/compstruct

Dynamic compressive response and failure behavior of fiber polymer composites embedded with tetra-needle-like ZnO nanowhiskers

Mao-Sheng Cao [a,*], Wei-Li Song [a], Wei Zhou [a,b], Da-Wei Wang [a], Ji-Li Rong [a], Jie Yuan [c,**], Simeon Agathopoulos [d]

[a] School of Materials Science and Engineering, Beijing Institute of Technology, Beijing 100081, PR China
[b] College of Quality and Technology Supervision, Hebei University, Baoding 071002, PR China
[c] School of Information Engineering, Central University for Nationalities, Beijing 100081, PR China
[d] Materials Science and Engineering Department, University of Ioannina, GR-451 10 Ioannina, Greece

ARTICLE INFO

Article history:
Available online 16 May 2010

Keywords:
Dynamic compressive response
Failure behavior
ZnO nanowhiskers
Composites

ABSTRACT

Glass fiber polymer composites embedded with tetra-needle-like zinc oxide (ZnO) nanowhiskers were prepared. The prepared composites exhibited excellent mechanical properties after the effective dispersion of ZnO nanowhiskers in the resin. The static and dynamic compressive properties of the composites were tested in the thickness and in-plane directions. The macro- and microfracture morphologies of the damaged specimens were obtained by using a scanning electron microscope. The results show that the compressive properties of the composites could be significantly affected by strain rates. As the strain rate increases, the composites have a higher strength. The compressive properties of the composites are affected by the content of ZnO nanowhiskers in the resin. The high strength of the composites can be attributed to the three-dimensional structures of ZnO nanowhiskers and the corresponding stress transfer.

1. Introduction

Zinc oxide (ZnO) nanostructures [1–6] have been fabricated by using various approaches. Further investigations demonstrate that ZnO nanostructures have exceptional piezoelectric [7], optical [8–11], electrical [12–14], mechanical [15–29], dielectric [30] and microwave absorption [31–36] properties. In recent years, fiber (e.g., carbon fibers and glass fibers)-reinforced composites have been widely applied in a remarkable variety of fields because of their excellent complex properties [37,38]. Therefore, the good functional properties [39–41] of ZnO nanostructures and the possibility of combining them with fiber reinforcements have speeded the preparation of ZnO composites with both structural and functional properties.

Besides functional properties, studying the mechanical properties is also important for structural and functional composites [42–48]. Thus far, the mechanical properties of composites have been characterized under quasi-static loading conditions [49–51]. Since many applications of composites are prone to serve under dynamic loading conditions such as high velocity impact, the mechanical response of composites under dynamic loading has attracted increasing attention [52–65]. The split Hopkinson pressure bar (SHPB) might be the most extensive experimental method for characterizing materials at high strain rates [54]. Tay et al. [55] studied the dynamic compressive properties of glass fiber-reinforced epoxy and pure epoxy resin by using SHPB. In their work, the mechanical behaviors of glass fiber-reinforced polymer and pure epoxy are shown to be strain rate sensitive, and an empirical strain rate-dependent constitutive relationship is proposed according to the experimental results. The dynamic compressive strength and strain rate-dependent behavior of polymeric composites have been investigated by Sun et al. [56–59]. They reported that the stress–strain relationship is linear only when the sample is loaded in the longitudinal direction. However, the response is nonlinear when the sample is loaded in the off-axis direction. The compressive behaviors of carbon/epoxy composites have been investigated at high strain rates by Hosur et al. [60,61]. The results indicated that the dynamic strength and stiffness exhibit an evident increase compared with the static values in the testing range of strain rates [60]. Failure features are characterized via optical and scanning electron microscopy [61]. Pintado et al. [62] studied the dynamic stress–strain response of graphite–epoxy composite laminates and found a scale effect: larger specimens are stiffer than smaller ones. The quasi-static and dynamic failure strengths and modes of the woven S2 glass-reinforced polyester thick laminates were

---

* Corresponding author. Tel.: +86 10 68914062.
** Corresponding author.
E-mail addresses: caomaosheng@bit.edu.cn (M.-S. Cao), yuanjie4000@sina.com (J. Yuan).

0263-8223/$ - see front matter © 2010 Elsevier Ltd. All rights reserved.
doi:10.1016/j.compstruct.2010.05.010

studied by Khan et al. [63]. The material was found to be significantly strain rate sensitive. Similarly, the strain to failure for both CFRP and GFRP was found to decrease with an increasing strain rate in the work of Ochola et al. [64]. The quasi-static and dynamic mechanical properties of TKW/PA66 composites were investigated by Gai and coworkers [65]. The results showed a 20–100% increase in the reference modulus and 20% increase in the compression strength. Most research focuses on the mechanical responses of composites, whereas fewer studies concern failure behaviors, such as the fracture processes and fracture morphologies of composites, particularly the failure behaviors of nanocomposites at high strain rates.

In this work, glass fiber-reinforced polymer composites were prepared by dispersing ZnO nanowhiskers in a resin matrix. The original ZnO nanowhiskers and dispersive ZnO nanowhiskers in the composites were examined by an X-ray diffractometer (XRD) and a scanning electron microscope (SEM). Quasi-static compressive experiments were performed by using an Instron 1186, and the dynamic compressive properties were tested in both normal and in-plane directions by SHPB. Furthermore, the microstructures and fracture morphologies were characterized using a SEM. More importantly, the effect of ZnO nanowhiskers on the mechanical properties of the composites was intensively discussed.

2. Experimental details

2.1. Materials and specimens

Tetra-needle-like ZnO nanowhiskers were synthesized by the combustion oxidization [6,66–68] of metal zinc powders (purity: 99.99 wt.%) without a catalyst. Then, as-prepared ZnO nanowhiskers were dispersed into the resin matrix (the weight ratio of the E-44 epoxy to 616 phenolic resin was about 1:1) with different proportions (0, 5 and 20 wt.% ZnO nanowhiskers in the resin). The glass fiber prepreg (the volume fraction of woven glass fibers was about 65%) was carried out, and the composites were formed by the hot-press at 448 K for about 3 h. The thicknesses of the composites were about 12 mm.

The laminates were machined into two different specimens by a turning process on an engine lathe. As shown in Fig. 1, these two cylinders with a diameter $D = 10$ mm and height 8 mm are in the normal direction (left in Fig. 1) and in-plane direction (right in Fig. 1), respectively. The specimens were polished before the test to reduce the friction between the specimens and pressure bar.

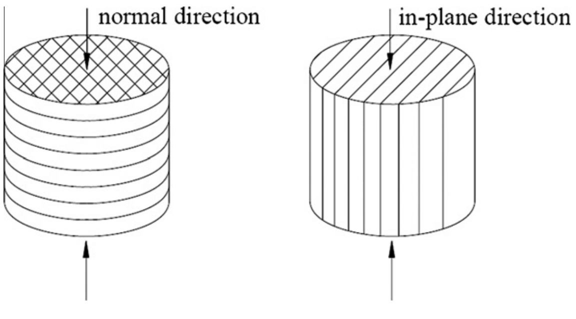

Fig. 1. Geometry and loading direction of two types of specimens.

2.2. Quasi-static and high strain rate testing

The quasi-static compressive properties of the composites were evaluated by an Instron machine (Instron 1186). SHPB, one of the most extensive experimental methods for characterizing the mechanical response of materials, was used to study the response of specimens at high strain rates [69,70]. Stress and strains with varying strain rates of materials could be obtained based on the following equations:

$$\sigma(t) = \frac{AE\varepsilon_t(t)}{A_s}, \tag{1}$$

$$\dot{\varepsilon} = -\frac{2c\varepsilon_r(t)}{L_s}, \tag{2}$$

$$\varepsilon(t) = \int_0^t \dot{\varepsilon}(\tau)d\tau, \tag{3}$$

where $\varepsilon_r(t)$ and $\varepsilon_t(t)$ are the axial strains of the reflecting pulse and transmitting pulse, respectively, which could be measured in the incident and transmitter bars as functions of time t. A, E and c are the cross-sectional area, Young's modulus and wave speed in the bar, respectively. Herein, $c = (E/\rho)^{1/2}$, and ρ is the mass density of the bar. L_s and A_s are the length and cross-sectional area of the specimens.

2.3. XRD and SEM analysis

Tetra-needle-like ZnO nanowhiskers were characterized by XRD (Rigaku DMAX2500 Cu K). The morphology of tetra-needle-like ZnO nanowhiskers, together with the macro- and microfracture fractography of the damaged specimens, were obtained by the SEM (Hitachi S-3500).

3. Results and discussion

3.1. Tetra-needle-like ZnO nanowhiskers and their composites

SEM, energy dispersive spectrometry (EDS) and XRD images of tetra-needle-like ZnO nanowhiskers are shown in Fig. 2. The products consist of abundant tetra-needle-like ZnO nanowhiskers and the morphology is uniform. The length of the whisker needles is 10–50 μm, revealing that a single tetra-needle-like ZnO nanowhisker consists of a core and four legs. The XRD spectrum of ZnO nanowhiskers shows that the diffraction peaks such as (1 0 0), (0 0 2) and (1 0 1) are sharp, whereas the peaks of other impurity phases were not found. The results indicate that the products have high quality single crystal structures, and the peaks indexed to JCPDS Card No. 75-1526 reveal that the products are hexagonal wurtzite-structured ZnO. The EDS of ZnO nanowhiskers shows that the products consist of two elements Zn and O, and the atomic composition ratio of Zn to O is about 1:1, which is in agreement with the ratio of ZnO compositions. The peak of silicon appears because the specimen was tested on the silica. According to the experimental results, tetra-needle-like ZnO nanowhiskers with high quality single crystal were obtained.

Fig. 3 shows the XRD spectrum of the composites. The diffraction peaks such as (1 0 0), (0 0 2) and (1 0 1) are consistent with the original ZnO nanowhiskers, indicating that ZnO nanowhiskers are dispersed in the composites.

The section image of the composites is shown in Fig. 4a. Fig. 4b–d exhibit the element maps of Si, Zn and C in the selected area of Fig. 4a, respectively. The results indicate that ZnO nanowhiskers were dispersed uniformly in the resin matrix.

3.2. Compressive properties of the composites

Tables 1 and 2 exhibit the static compressive properties of the composites with various proportions of ZnO nanowhiskers in the normal direction and in-plane direction, respectively. Both the quasi-static compressive strength and Young's modulus of the composites embedded with ZnO nanowhiskers were higher than the neat ones. From Table 1, the quasi-static compressive