Composites: Part B 69 (2015) 526–533

Contents lists available at ScienceDirect

Composites: Part B

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

Experimental investigation on the compression behaviors of epoxy with carbon nanotube under high strain rates

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ARTICLE INFO

Article history:
Received 16 August 2014
Received in revised form 26 September 2014
Accepted 29 September 2014
Available online 7 October 2014

Keywords:
A. Particle-reinforcement
A. Resins
B. Impact behaviour
B. Mechanical properties

ABSTRACT

The compressive properties of epoxy with different carbon nanotubes (CNTs) contents at quasi-static and high strain rates loading had been investigated via experiment to evaluate the compressive failure behaviors and modes at different CNTs contents and different strain rates. The results indicated that the stress train curves were strain rate sensitive, and the compressive stiffness, compressive failure stress of composites with various CNTs contents was increased with the strain rates and CNTs contents. The compressive failure stress and the compressive failure modes of the composites were apparently different as the change of CNTs contents.

1. Introduction

Fiber reinforced composites like carbon/epoxy composite have become more popular nowadays because of their low cost and high quality structures such as high strength to weight ratio and high resistance to corrosion and abrasion. The dynamic behaviors such as impact tension and impact compression properties are basic parameter for the fiber reinforced composites. Since the composites often employed in impulsive loading, the dynamic behaviors are important factors in designing because the mechanical properties of fiber reinforced composite are different under high strain rates, which also called strain rates effect. In the polymer field, epoxy presents good stiffness, specific strength, dimensional stability, chemical resistance and strong adhesion to the reinforcement [1–3]. Therefore, epoxy is a popular material to be the matrix of fiber reinforced composites. At the same time, the mechanical properties of epoxy can play a role part in the dynamic behaviors of fiber reinforced composites.

Since the synthesis of carbon nanotubes (CNTs), many researchers have been activated in the relative fields. During the past decades, carbon nanotubes have been widely used into polymers such as epoxy and polyurethane to improve the radiation resistance and mechanical properties. Gojny et al. [4] prepared the nanocomposites consisting of double-wall carbon nanotubes (DWCNTs) and epoxy matrix; they found that the mechanical properties had an obvious increasing. Thostenson and Chou [5] investigated the mechanical properties of nanotube/epoxy composites with different reinforcement fractions. The results showed that the fracture toughness increased obviously at low nanotube concentrations and the thermal conductivity increased linearly with nanotube concentration. Gojny et al. [6] observed the influence of various carbon nanotubes such as single-wall CNTs (SWCNT), double-wall CNTs (DWCNT) and multi-wall CNTs (MWCNT) on the mechanical properties of epoxy matrix composites. In their observation, the nanocomposites produced exhibited an enhanced strength and a significant increase in fracture toughness. Cooper et al. [7] employed the Raman spectroscopy to characterize the tensile deformation of a dilute dispersion of SWNTs and MWNTs in the epoxy composite and predicted the mechanical properties of high volume fraction composites reinforced with carbon nanotubes. Dassios et al. [8] investigated the compressive behavior of MWCNT/epoxy composite mats, they found that the strength, stiffness and toughness of nanocomposite were increased with the pure polymer. Loos et al. [9] prepared the epoxy resin with single walled carbon nanotubes (SWCNTs) and studied the mechanical, viscoelastic and thermal properties. The results showed that the addition of small amounts of SWCNTs to epoxy could lead slight structural change in the epoxy matrix and improve the mechanical and viscoelastic properties. Gou et al. [10] investigated the interfacial bonding of single-walled nanotube reinforced epoxy composites via computational and experimental methods. The results showed that the interface bonding of the nanotubes in the epoxy resin increased up to 250–300% and indicated that there

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http://dx.doi.org/10.1016/j.compositesb.2014.09.038
1359-8368/

Table 1
The properties of carbon nanotubes (CNTs) contents.

Property	Outer diameter (OD) (nm)	Purity (wt%)	Length (μm)	Specific surface area (m^2/g)	Ash powder (wt%)	Tap density (g/cm^3)
Index	<8	>95	10–30	>500	<1.5	0.27

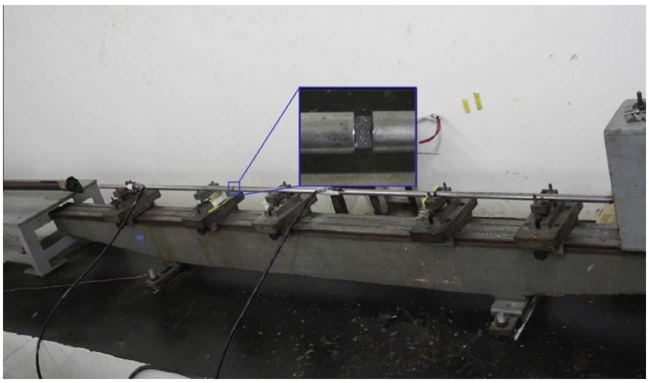


Fig. 1. Set up of split Hopkinson pressure bar.

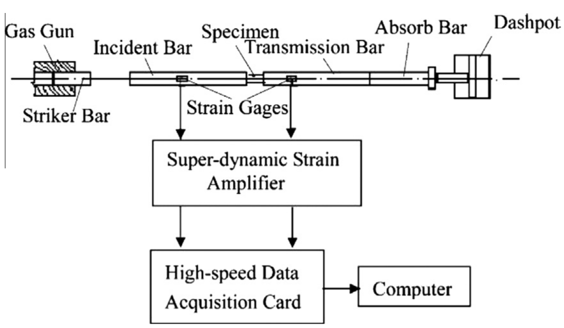


Fig. 2. Schematic of split Hopkinson pressure bar.

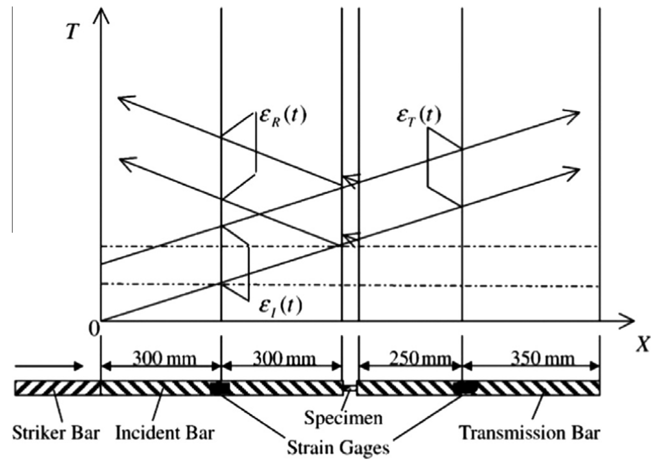


Fig. 3. Principle of split Hopkinson pressure bar.

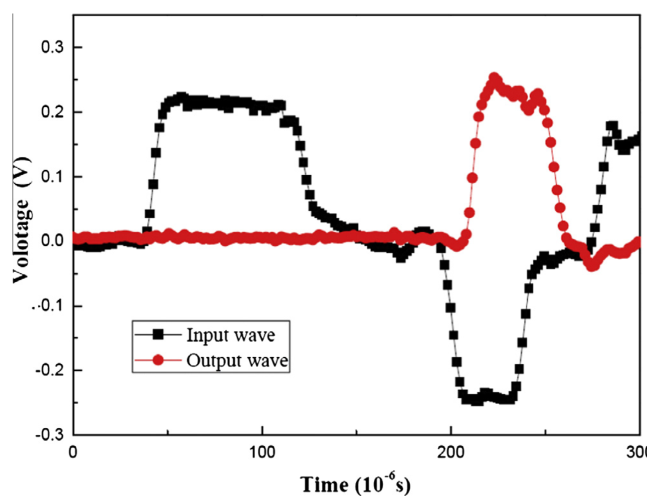


Fig. 4. Typical signals in input and output bar under compression with 1.0 wt% CNTs at strain rate of 1600/s.

could be an effective stress transfer from the epoxy to the nanotube. Shen et al. [11] discussed the reinforcement role of various amino-functionalized multi-walled carbon nanotubes in epoxy nanocomposites. They found that various kinds of amino-functionalized multi-walled carbon nanotubes would have different effects on the thermal and mechanical properties of the nanocomposites. Xu et al. [12] investigated the mechanical properties and interfacial characteristics of carbon-nanotube-reinforced epoxy tin films. The results showed that a 20% increase in elastic modulus when 0.1 wt% multiwalled carbon nanotubes were added compared to net resin thin films. Zhang et al. [13] Observed behavior of glass reinforced aluminum laminates with MWCNT modified epoxy resins via low-velocity experiments, they found that the impact resistance of composites with MWCNT was improved. Leininger et al. [14] investigated the influence of MWCNT content level on the quasi-static and dynamic tensile properties of epoxy. The results indicated that the mechanical properties were improved by the addition of MWCNTs. He and Tjong [15] analysis the correlation between state of nanotube dispersion and Zener tunneling parameters of carbon nanotube/epoxy resin composites. They found that the composites with homogeneous nanotube dispersion could exhibit larger static electrical conductivity and smaller percolation threshold han those with poorer nanotube dispersion.

Some researchers such as Zhang et al. [13] and Leininger et al. [14] investigated the dynamic mechanical behaviors of epoxy/CNT composites. However, their dynamic mechanical behaviors only focused on the low-velocity state. For the applications of impact resistance and impact protection, the mechanical parameters of the composite at high strain rates should be introduced because the composites often manifest the strain rate sensitivity. In this research, the epoxy with various content carbon nanotubes nanocomposites was prepared. The compression behaviors of the nanocomposites under quasi-static and high strain rates conditions will be presented. The compression strength, failure strain, energy absorption, compression stiffness and failure modes of various carbon nanotube contents nanocomposites under different compression strain rates also will be investigated.

2. Experimental procedure

2.1. Materials

The epoxy resin was Type 618 made by Shanghai Resin Factory of China, tensile modulus: 1.97 GPa, tensile strength: 68.10 MPa. The MWCNTs were MFG 110413 supplied by Chengdu Organic Chemicals Co. Ltd., Chinese Academy of Sciences. The properties of the CNTs have been shown in Table 1.

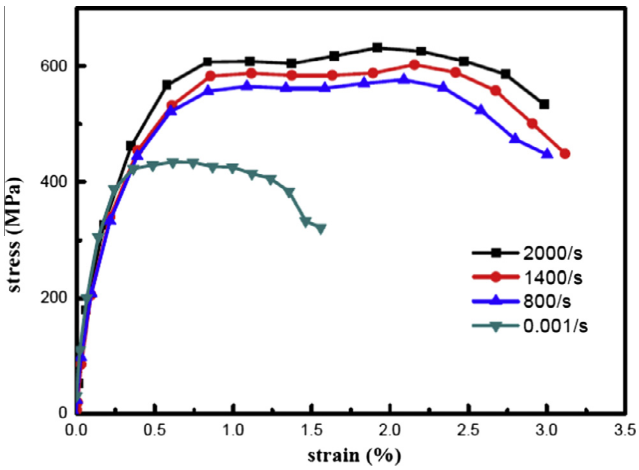


Fig. 5. Stress–strain curves of epoxy/CNTs (0 wt%) composites at various strain rates.

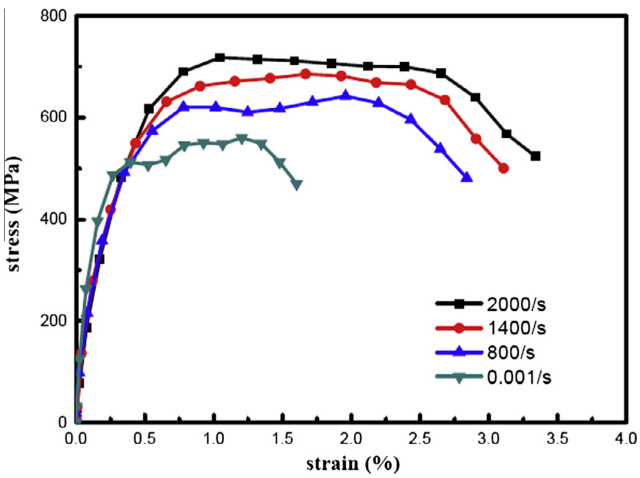


Fig. 6. Stress–strain curves of epoxy/CNTs (0.5 wt%) composites at various strain rates.

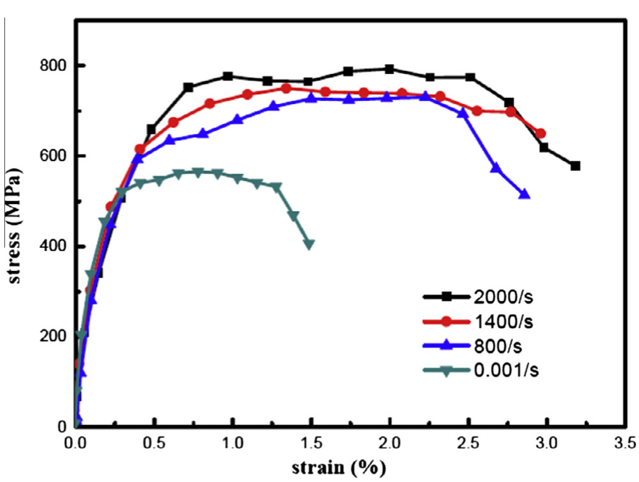


Fig. 7. Stress–strain curves of epoxy/CNTs (1.0 wt%) composites at various strain rates.

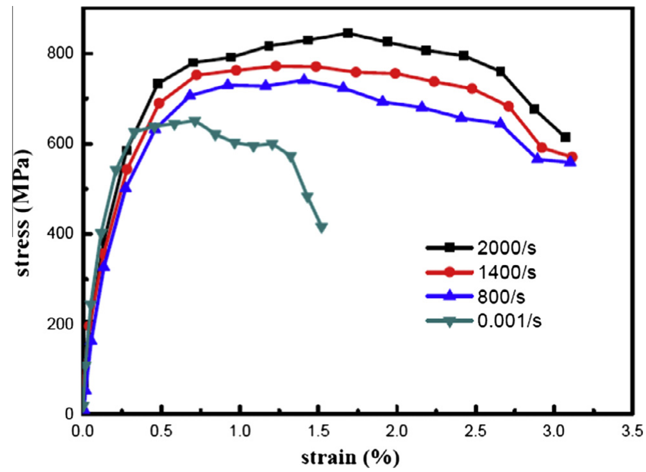


Fig. 8. Stress–strain curves of epoxy/CNTs (1.5 wt%) composites at various strain rates.

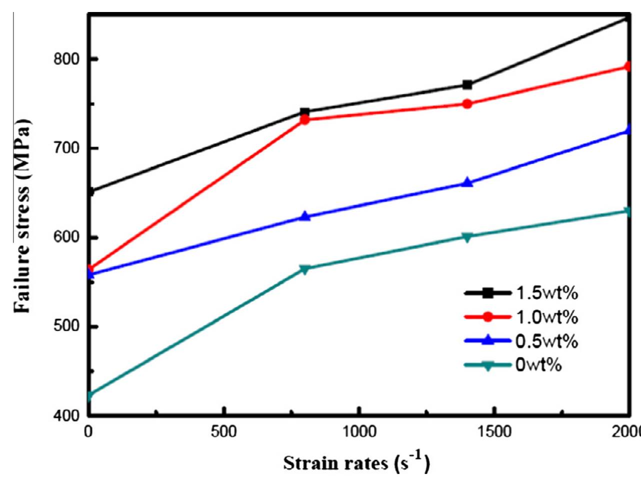


Fig. 9. Failure stress of epoxy/CNTs (with different contents) composites at various strain rates.

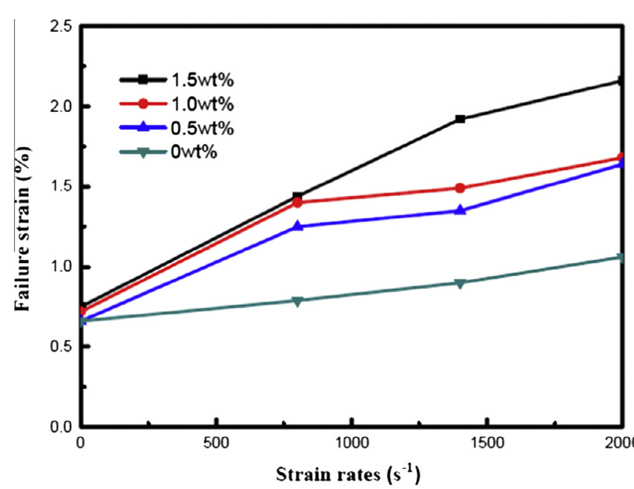


Fig. 10. Failure strain of epoxy/CNTs (with different contents) composites at various strain rates.

Table 2
The mechanical properties of epoxy with different carbon nanotubes (CNTs) contents under various strain rates.

CNT contents	Strain rates (s)	Compressive strength (MPa)	Failure strain (%)	Absorbed energy (J)	Modulus (GPa)
0 wt%	0.001	423	0.66	769.25	2.21
	800	565	0.79	1298.77	2.08
	1400	601	0.90	1525.85	1.93
	2000	630	1.06	1648.10	1.64
0.5 wt%	0.001	558	0.66	795.05	2.70
	800	623	1.25	1589.29	2.47
	1400	661	1.35	1819.07	2.28
	2000	720	1.64	1999.52	1.95
1.0 wt%	0.001	564	0.72	855.45	3.78
	800	732	1.40	1603.88	3.37
	1400	750	1.49	1879.81	3.16
	2000	792	1.68	2108.48	2.55
1.5 wt%	0.001	651	0.75	1008.63	3.79
	800	741	1.44	1986.45	3.12
	1400	771	1.92	2132.37	2.97
	2000	847	2.16	2270.98	2.58

2.2. Preparation of MWCNTs-filled epoxy resin

The MWCNTs were put in acetone solution. The concentration of MWCNTs was 1 wt%, and the mixture was ultra sonicated by an ultrasonic cell disruptor BILON92-IID (Shanghai Bilang) for about 6 h in order to get a homogenous solution. Then the mix of Bisphenol A epoxy (Type 618 made by Shanghai Resin Factory of China) was added to the solution and stirred at 40 °C for 12 h.

2.3. Composites fabricating

The composites were fabricated with the MWCNTs filled-epoxy resin. The mix of Bisphenol A epoxy with MWCNTs and agent Tri-methyl-hexamethylene-diamine (Type 593 made by Shanghai Resin Factory of China) in the volume proportion of 3:1.

The composite coupons for testing were prepared according to the length × width is 9 mm × 9 mm, and the thickness is 5 mm.

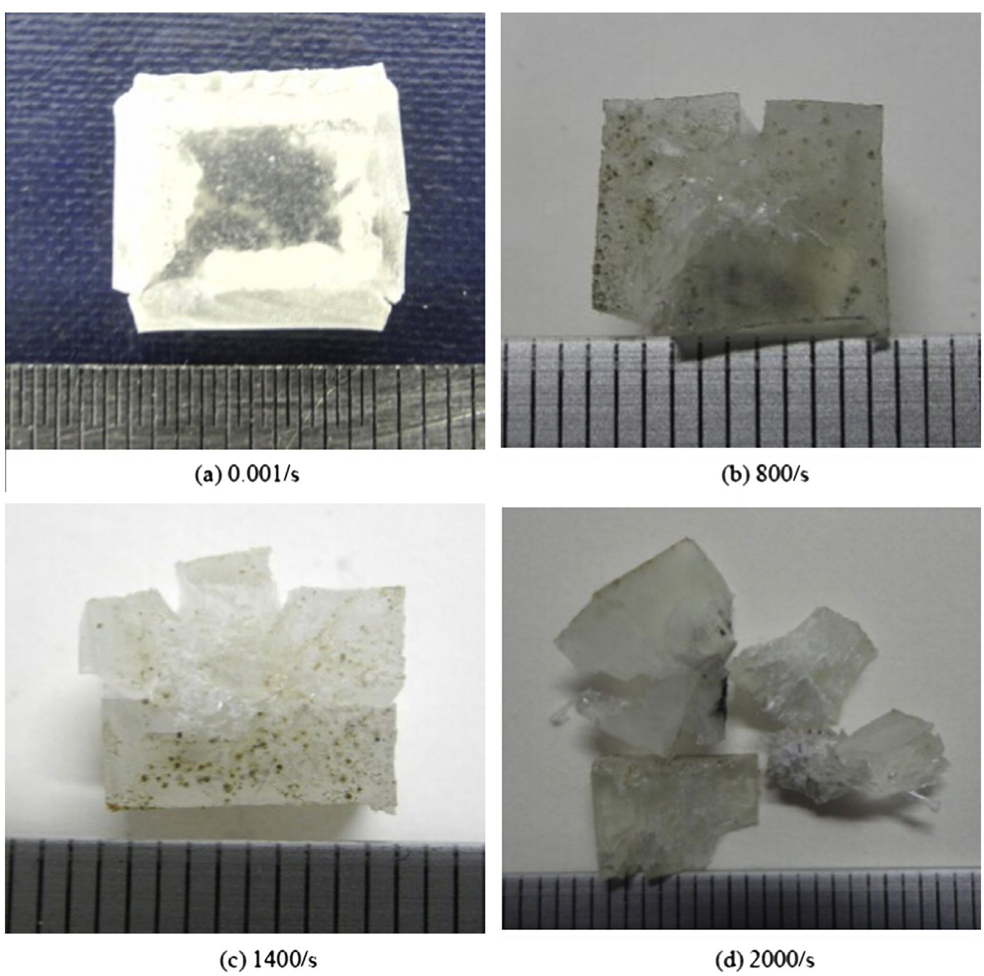


Fig. 11. Compression fractures of epoxy/CNTs (0 wt%) composites at various strain rates.

2.4. Compression tests under various strain rates

The compression tests were conducted under both quasi-static and high strain rates. Strain rate is the rate of change in strain (deformation) of composites with respect to time. For the strain rate at high rates of straining,

$$\dot{\varepsilon}(t) = \frac{d\varepsilon}{dt} = \frac{d}{dt}\left(\frac{L(t) - L_0}{L_0}\right) = \frac{1}{L_0}\frac{dL}{dt}(t) = \frac{\upsilon(t)}{L_0} \tag{1}$$

where $\dot{\varepsilon}(t)$ is strain rate, ε is the strain, L_0 is the original length, $L(t)$ is the length at each time, $\upsilon(t)$ is the speed at which the ends are moving away from each other.

For the strain rate in quasi static, the approximate strain can be calculated from follow equation,

$$\dot{\varepsilon}(t) = \frac{\upsilon}{L} \tag{2}$$

where $\dot{\varepsilon}(t)$ is strain rate, υ is the speed of compression, L is the length of sample. The compression test under the quasi-static condition was performed on an MTS810.23 system at a speed of 1 mm/min.

The high strain rate compression tests were carried out on a split Hopkinson pressure bar (SHPB) apparatus. The schematic illustration and principle of the SHPB employed in this study are shown in Figs. 1 and 2. A small sandwich specimen is between two elastic bars of the same cross sectional area and modulus, which are named as the incident bar and transmission bar separately. The material of the striker and pressure bars is steel subjected to maraging with extremely high yield strength in order to withstand a very high impact velocity. An elastic stress pulse is imparted to the incident bar by impacting it with a striker bar of the same cross sectional area and modulus. The impact of the striker bar generates as elastic stress wave with twice the length of the striker bar and propagates through the incident bar with the velocity of sound in the bar media and passes through the specimen while deforming it. The particle velocity imparted on the incident bar is half the impact velocity of the striker bar. The stress in the bar is given by

$$\sigma = \rho C_0 \upsilon_P \tag{3}$$

where ρ is the density of the bar material, C_0 is the bar sonic velocity and υ_P is the particle velocity.

When the elastic wave reaches the specimen-incident bar interface, part of it will be reflected back, and part of it will be transmitted through the specimen and pass through the transmission bar. To acquire the direct incident pulse, the reflected pulse and the transmitted pulse, strain gages are mounted on both the incident and transmission bars. One-dimensional wave propagation is assumed to analyze the strain signals from the strain gauges (Type BF(BH)350-8KA made by Shanghai Shendi Informational Technology Co., Ltd.).

If E_b, A_b and ρ_b represent the modulus, cross section area and density of the bar and E_s, A_s and ρ_s those of specimen then the equations for the strain rates ($\dot{\varepsilon}$), strain (ε) and stress (σ) of the specimen are as follows:

$$\dot{\varepsilon}(t) = -\frac{2C_0}{L_s}\varepsilon_R(t) \tag{4}$$

$$\varepsilon(t) = -\frac{2C_0}{L_s}\int_0^t \varepsilon_R(t)dt \tag{5}$$

$$\sigma(t) = \frac{E_b A_b}{A_s}\varepsilon_T(t) \tag{6}$$

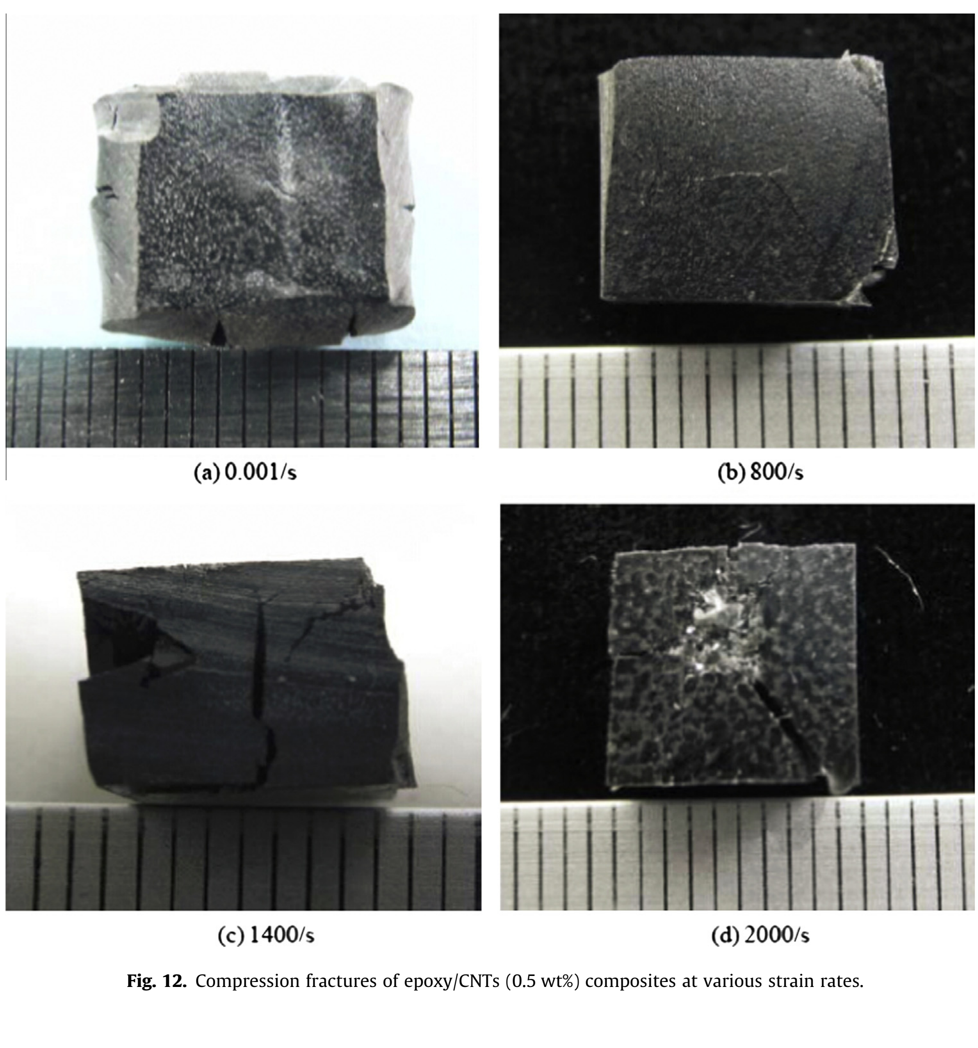


Fig. 12. Compression fractures of epoxy/CNTs (0.5 wt%) composites at various strain rates.

where $C_0 = \sqrt{E_b/\rho_b}$ represents the longitudinal wave velocity in the bar L_s represents the specimen length $\varepsilon_R(t)$ and $\varepsilon_T(t)$ are the strain gauge signals of the reflected and the transmitted pulses, respectively.

The average stress and strain in the specimen can be known as a function of time and the mechanical properties of the composite, such as strain rates and stress–strain behavior, along the loading direction can be determined from Eqs. (4)-(6).

The SHPB employed in this study was equipped with a momentum trapping device, a gas gun of 14.5 mm inner diameter. The diameter of the incident and transmission bars was the same (diameter 14.5 mm and length 600 mm). Compressed nitrogen gas was employed to operate the gas gun and propel the striker bar to move a distance of 200 mm. The impact velocity of the gas gun (strike bar) could be changed by changing gas pressure to get required strain rates. The schematic and principle of split Hopkinson pressure bar are shown in Figs. 2 and 3 respectively.

3. Results and discussion

3.1. Stress–strain relations

Compression tests of epoxy composites with various CNT contents were conducted at different strain rates. As abovementioned, at least five samples were tested at each strain rate (including quasi-static state). Fig. 4 shows the typical signals in input and output bar under compression with 1.0 wt% CNTs at strain rate of 1600/s. According to the one dimensional stress wave propagation theory of the SHPB apparatus, the stress strain curves under high strain rates can be calculated. The results are shown in Figs. 5–8. It can be observed that the stress–strain curves are sensitive to strain rate for different CNT contents.

Fig. 5 shows the stress–strain curves of epoxy without CNTs at various strain rates. Fig. 5 indicates that the compressive failure stress increases, the compressive stiffness decreases and the failure strain increases with increasing strain rate at high strain rate compression.

Fig. 6 shows the stress–strain curves of epoxy composites with 0.5 wt% CNTs at various strain rates. Similar to Fig. 5 which is for the composites without CNTs, the compressive failure stress and compressive failure strain are increasing with increasing strain rate. However, the stiffness of composites is inclined with the CNTs content increasing, because the CNT can increase the interface strain in the resin.

Figs. 7 and 8 are the stress–strain curves of epoxy composites with 1.0 wt% CNTs and 1.5 wt% CNTs at various strain rates, respectively. Similar with Fig. 6, the compressive failure stress increases as the strain rate grows and the CNTs content increases, the compressive failure strain also increases when strain rate increases at high strain rates compression. Meanwhile, the compressive stiffness of composites with CNTs is smaller than that of the composites without CNTs.

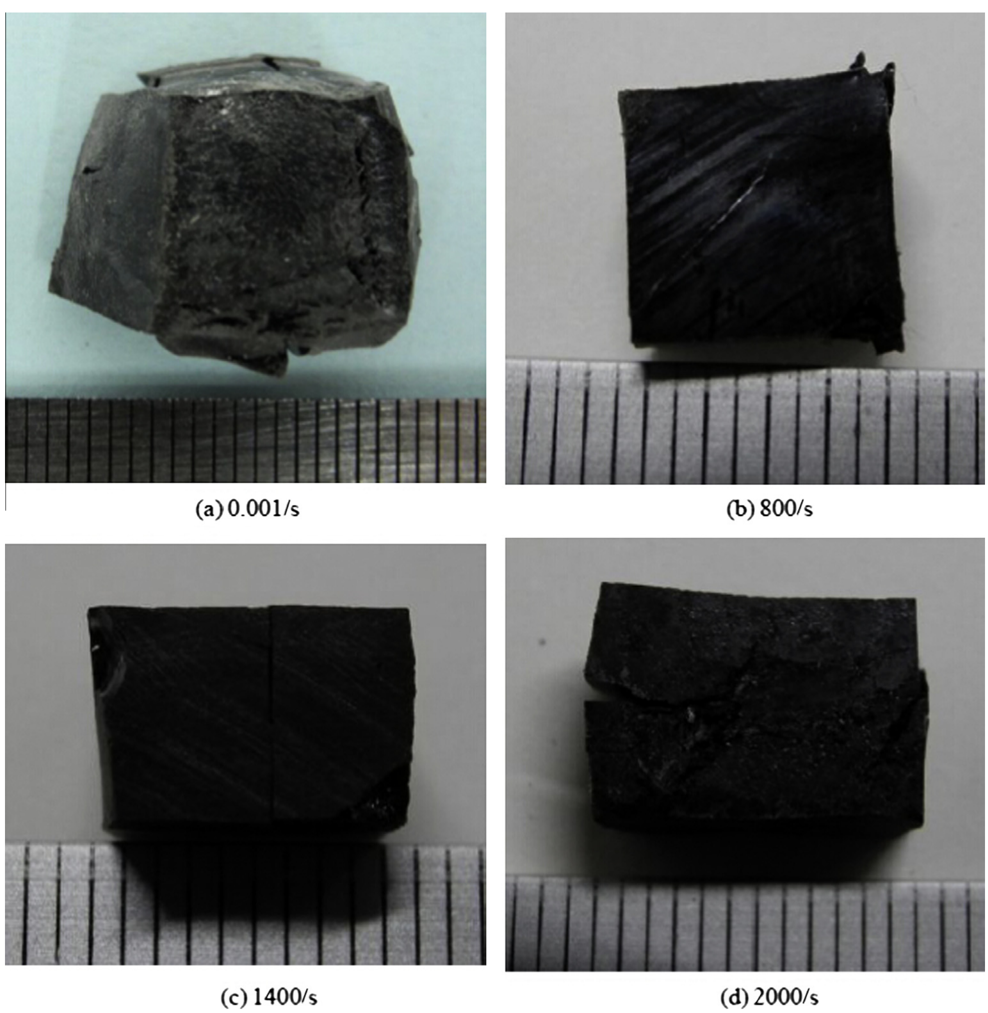


Fig. 13. Compression fractures of epoxy/CNTs (1.0 wt%) composites at various strain rates.

3.2. Mechanical properties

Fig. 9 shows the compressive failure stress of epoxy composites with various CNTs contents under different strain rates. It can be seen that the compressive failure stress increases with the strain rate and CNTs contents. For high strain rate loading, the failure stress of epoxy composites with various CNTs contents is much larger than that in quasi-static loading.

Fig. 10 shows the compressive failure strain of epoxy composites with various CNTs contents under different strain rates. Similar to Fig. 12, it can be seen that the compressive failure strain increases with the strain rate and CNTs contents.

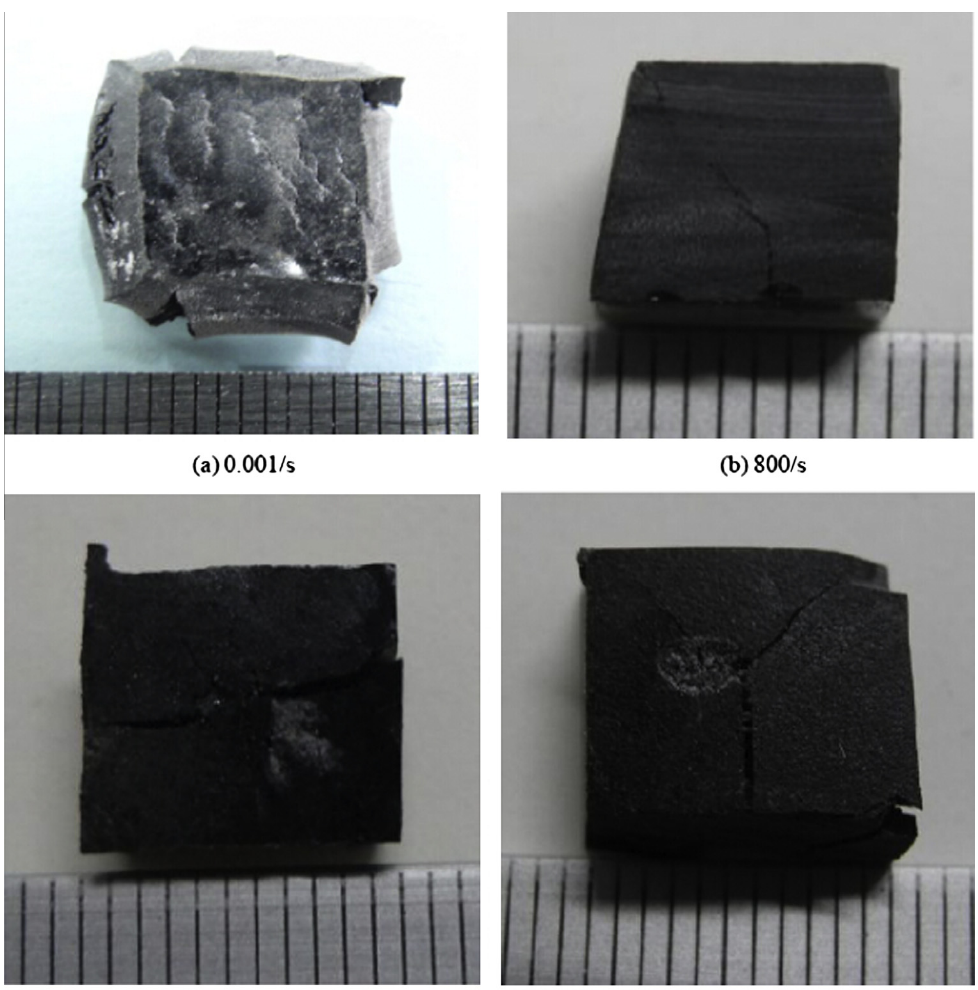
The mechanical properties of epoxy with various contents of CNT and different strain rates tests are summarized in Table 2. The absorbed energy is calculated by integrating the stress–strain curves. It can be shown that the compressive strength, failure strain and absorbed energy of epoxy composite increase with the increase of strain rate and CNT contents, and the modulus of epoxy composite is decrease with the strain rates and it is increase with the CNT contents.

Regarding compression tests, the compressive strength of the pure epoxy is 423 MPa, and the compressive strength of epoxy with various CNT contents (0.5 wt%, 1.0 wt% and 1.5 wt%) increases about 31.9%, 33.3% and 53.9%. The modulus of epoxy with various CNT contents grows comparison with pure epoxy (2.21 GPa) about 22.2%, 71.0% and 71.5%. Loos et al. [9] found that the modulus and compressive strength of SWCNT/epoxy composite increased from 1.2 GPa to 1.3 GPa (8%) and from 42.1 MPa to 45.1 MPa (7.1%). Schadler et al. [16] obtained an increase from 3.6 GPa to 4.5 GPa (24%) in the compression modulus of MWCNTs/epoxy composite.

3.3. Compression fracture features

The photographs in Fig. 11a–d display the fracture features of epoxy without CNTs at various strain rates under compressive loading. It is found that breakage of epoxy specimen occur at various strain rates. As the strain rate increasing, the specimen is compressed into several pieces of fragment which is shown in Fig. 12d. The obvious beginning crack happens at the strain rate of 800/s, and drastic at the strain rate of 1400/s. The failure state of the sample without CNTs at various strain rates under in-plane loading is shown in the figures. It is can be observed that the specimen is cracked because of the radicalized stress transmission though the little epoxy block.

Fig. 12a–d shows compression fractures of epoxy/CNTs (0.5 wt%) composites at various strain rates. The crazes appear at the strain rate of 800/s, and the apparent crack arise at the strain rate of 1400/s. However, the specimen keeps as an entirety, instead of being the fragments, which is quite different from the state of the sample without CNTs at the same strain rate. And then, at



(a) 0.001/s (b) 800/s (c) 1400/s (d) 2000/s

Fig. 14. Compression fractures of epoxy/CNTs (1.5 wt%) composites at various strain rates.

the strain rate of 2000/s, the composites fractured. The crack happens under high strain rate compression.

The photographs in Fig. 13 illustrate compression fractures of epoxy/CNTs (1.0 wt%) composites at various strain rates. It is found that the failure modes under quasi-static state and high strain rates are rather different. The performance of crack of is evidence under the strain rate of 0.001/s. On the contrary, the morphological feature under the high strain rates is rather stable, which the shape of the specimen hardly change under 800/s and 1400/s expect a single craze, furthermore, the integrity of the sample is well kept ever under 2000/s.

Compression fractures of epoxy/CNTs (1.5 wt%) composites at various strain rates is shown in Fig. 14a–d. Similar with the situation of epoxy/CNTs (0.5 wt%) composites, the serious crack happens under low strain rate, which shows several crevices and is loose texture in appearance. On the opposite, under the high strain rates, the regular crazes occur, and there are hardly tiny cracks that can be noticed. The samples after test still keep hard quality. There is no obvious deformation, because the high content CNT can increase the interface strength of composites, which indicates that the composites can absorb more energy with smaller deformation.

4. Conclusions

The impact compression, including quasi-static and high strain rate compression properties of epoxy with various CNTs contents (0 wt%, 0.5 wt%, 1.0 wt% and 1.5 wt%) was investigated via split Hopkinson pressure bar (SHPB). The compression stress vs. strain curves of the composites with various CNTs contents have been obtained and compared with those for quasi-static compression. The stress strain curves are strain rate sensitive. For high strain rate loading, the failure stress of epoxy composites with various CNTs contents is much larger than that in quasi-static loading, while compressive stiffness of composites with CNTs contents under various strain rates is much smaller than that of the composites without CNTs. And the compressive stiffness of composites under different strain rates is lower when the CNTs contents increase. The fracture pictures of the damaged composites with different CNTs contents at various strain rates show the different crack situations and morphology of the specimens. The cracks happened drastically with the increase of the CNTs contents under 0.001/s, and on the contrary, there were less crazes and the morphology became more stable with the increase of the CNTs contents under high strain rates. The composites with higher contents of CNTs improved the energy absorption of composites because they could elevate the roughness of composites. The mechanical parameters of composites with CNT contents at high strain rates can help the design of composites structures.

Acknowledgments

The authors acknowledge the financial support from the National Science Foundation of China (No. 11302085), the National Science and Technology Support Program of China (No. 2012BAF13B03), the Fundamental Research Funds for the Central Universities (Nos. JUSRP1043 and JUSRP51404A), and the Innovation fund project of Cooperation among Industries, Universities & Research Institutes of Jiangsu Province (Nos. BY2014023-34 and BY2014023-20).

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