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Fracture toughness and fatigue life of MWCNT/epoxy composites

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ABSTRACT

The present work experimentally characterizes the mode-I fracture toughness and stress–life curve of multi-walled carbon nanotube-(MWCNT-)reinforced epoxy-matrix composites. The effects of carbon nanotube weight fraction and voids on the composite fracture toughness are studied. The average fracture toughness of 1 wt%- and 3 wt%-MWCNT/epoxy composites is 1.29 and 1.62 times of that of pure epoxy, respectively. The 0.5 wt%-MWCNT/epoxy composites' fatigue lives are 10.5 and 9.3 times of the average fatigue life of neat epoxy, when they are subjected to cyclic loadings with stress amplitudes of 8.67 MPa and 11.56 MPa, respectively. The micrographs indicate that the separation and uniform distribution of MWCNTs in the matrix and the formation of voids significantly affect the fracture and fatigue behavior of MWCNT-reinforced composites.

1. Introduction

Due to their excellent stiffness and strength, carbon nanotubes (CNTs) are considered as an ideal candidate for the reinforcement in composites. While most of the existing works are focused on the stiffness and strength of CNT-reinforced composites (see [1] for an extensive review), relatively few works are devoted to the fracture and fatigue behavior of CNT-reinforced nanocomposites. In the work of Park et al. [2], the mode-I fracture toughness (K_{IC}) of multi-walled carbon nanotube-(MWCNT-)reinforced epoxy-matrix composites is determined by single edge notched three-point bending tests, and is in the range of 2.45–3.05 MPa m$^{1/2}$. Gojny et al. [3] conduct compact tension tests and report that the K_{IC} is 0.77 MPa m$^{1/2}$, 0.76 MPa m$^{1/2}$, 0.77 MPa m$^{1/2}$ and 0.82 MPa m$^{1/2}$, for composites containing 0.1% carbon black, 0.1% double-wall carbon nanotubes (DWCNTs), 0.1% DWCNT–NH_2, and 1% DWCNT–NH_2, respectively. The fracture toughness of composites containing 0.3 wt% DWCNT–NH_2 is 42% (K_{IC} = 0.92 MPa m$^{1/2}$) higher than that of the neat epoxy, K_{IC} = 0.65 MPa m$^{1/2}$ [4]. Adding 0.3 wt% unfunctionalised DWCNTs as well as carbon black into an epoxy resin shows a minor increase in K_{IC}, and increasing the CNT fraction up to 0.5 wt% does not lead to a further improvement in fracture toughness [5]. The addition of 1 wt%-MWCNT in epoxy increases its strength from 70 MPa to 170 MPa and the fracture toughness from 1.3 MPa m$^{1/2}$ to 4.0 MPa m$^{1/2}$ [6]. Satapathy et al. [7] report that a tough-to-brittle transition is observed in 4 wt%-MWCNT/PC. The fracture toughness determined by single edge notch tests is 0.66 MPa m$^{1/2}$ for neat epoxy, 0.73 MPa m$^{1/2}$ for 0.1 wt%-MWCNT/epoxy and 0.84 MPa m$^{1/2}$ for 2 wt%-MWCNT/epoxy, respectively [8]. It is noted that most of the existing works consider composites with a specific CNT weight fraction only, and data among various works are scattered. In the present work, the effects of CNT weight fraction on the fracture toughness of CNT-reinforced composites are studied in a systematic manner. The fracture toughness of CNT-reinforced ceramic composites is reported by Ning et al. [9] and Sun et al. [10].

The fatigue of CNT-reinforced composites has not been well studied yet. The stress–life (S–N) curve of unidirectional single-walled carbon nanotube-(SWCNT-)reinforced epoxy-matrix composite is similar to that of carbon/epoxy [11], and a fatigue failure model is established [12].

2. Experiments

The diameters of the as-received MWCNTs are 40–60 nm and their lengths range 5–15 μm. The epoxy resin is DER-331 (Dow Chemical) and the associated curing agents are HN-050 (Polystar Ltd.) for fracture specimens, and DER 26 (Dow Chemical) for fatigue specimens, respectively.

2.1. Materials processing

MWCNTs are added into a DER-331 epoxy resin of 30-ml together with 0.5-ml degassing agent (Model No. 5123, Polystar Enterprise Co. Ltd.) and separated from each other by sonication.

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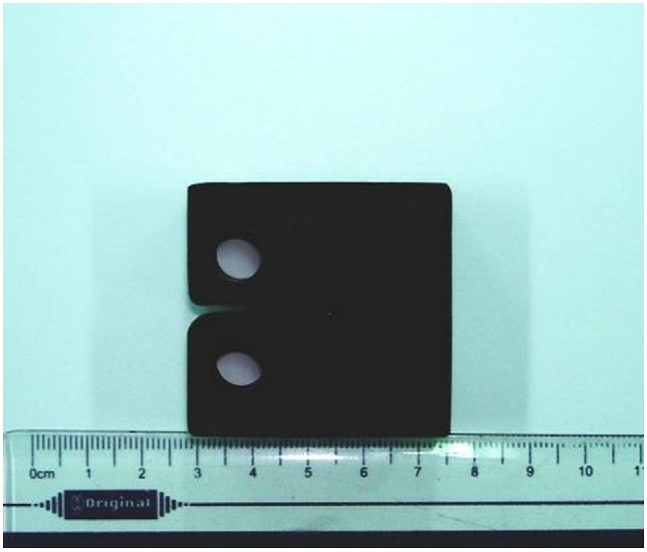

Fig. 1. Compact tension fracture specimen.

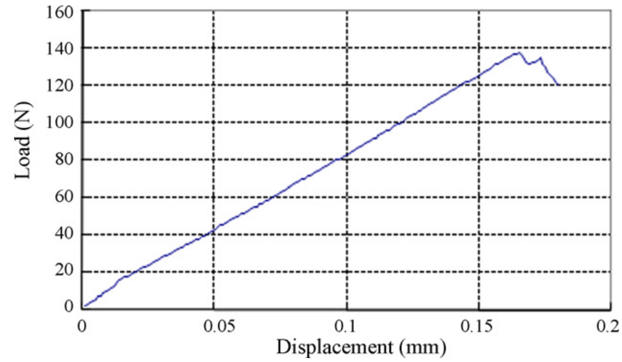

Fig. 3. Load–displacement curve of composite specimen no. CA031.

The degassing agent is used to prevent the formation of voids in the epoxy resin. The mixture is stirred for 180 min to ensure the separation of entangled MWCNTs and the uniform distribution of MWCNTs in epoxy, and is vacuumed in an oven for 15 min and then released to the atmospheric pressure for 5 min. This step is repeated three times for removing air bubbles produced from stirring. The temperature is kept at 40 °C to reduce the viscosity and enhance the fluidity of the mixture.

The MWCNT/epoxy mixture is cooled down at room temperature for 30 min. One then adds 15 ml curing agent into the mixture and stirs for 10 min to enhance the fluidity of the mixture using a magnetic stirrer. The stirrer's rotational speed is keep at 300 rpm. The mixture is vacuumed for 10 min and then released to the atmospheric pressure for 5 min. This step is repeated three times to reduce bubbles produced from stirring. It is noted that a large amount of air is indeed extracted from the mixture at the first two vacuum-release cycles. At the third vacuum-release cycle, extracted air bubbles are significantly reduced.

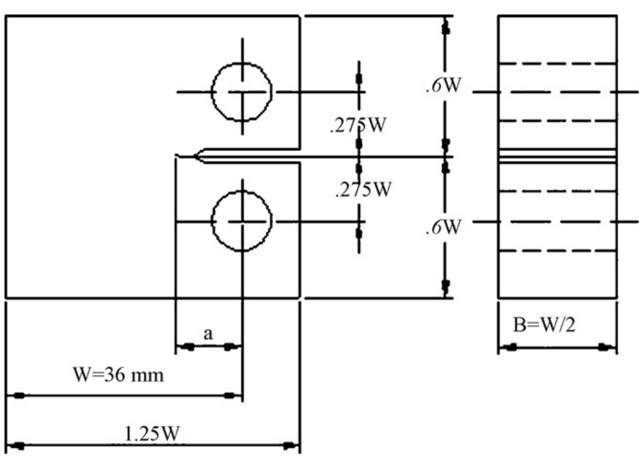

Fig. 2. Dimensions of ASTM D5045 compact tension specimen.

2.2. Fracture specimen preparation and testing

The MWCNT/epoxy mixture is poured into an aluminum mold for fracture specimen. The mixture is vacuumed for 10 min and then released to the atmospheric pressure for 5 min. This step is repeated three times to reduce the air bubbles resulting from MWCNT/epoxy being poured into the mold. The mixture is cured at room temperature for 3 h. A notch is made at this stage by using a razor. The composite is post-cured at room temperature in atmosphere for 24 h.

After simple machining to achieve the required thickness of specimen, one drills two holes in the specimen by using a computer numerical control (CNC) drilling machine. The specimen is then ready for standard compact tension fracture toughness test; see Fig. 1. The dimensions of the specimen are shown in Fig. 2. The cardinal width of specimen, W, is 36 mm, and the specimen thickness, B, is 18 mm. The ratio W/B is equal to two. The tests are conducted in accordance with ASTM D5045-99 *Standard Test Method for Fracture Toughness and Strain Energy Release Rate of Plastic Materials* [13].

Table 1
Fracture toughness of MWCNT/epoxy composites

Sample no.	CNT (wt%)	Crack length (mm)	Compliance ($\times 10^{-6}$ m/N)	P_{max} (N)	P_Q (N)	P_{max}/P_Q	K_{IC} (MPa m$^{1/2}$)	Increase in K_{IC}	t (10^{-4} m$^{1/2}$)
EN001	0	18.7	1.5	146.3	142.8[a]	1.02	0.42	0	6.7
EN002	0	18.46	1.2	138.1[a]	134.1	1.03	0.41	0	6.4
EA001	0	18.76	1.5	150.3	143.8[a]	1.05	0.44	0	7.3
EA002	0	18.96	1.8	143.3[a]	141.7	1.01	0.45	0	7.7
CN011	1%	19.55	1.7	158.3[a]	154.9	1.02	0.53	26%	5.2
CN012	1%	19.43	1.3	148.2[a]	142.9	1.04	0.55	31%	5.6
CA011	1%	17.92	2	213	208.7[a]	1.02	0.59	34%	6.5
CA012	1%	17.28	1.5	203.2[a]	193.2	1.05	0.56	29%	5.8
CN031	3%	17.59	1.9	210.3	210.1[a]	1.0	0.53	26%	3.9
CN032	3%	17.97	2.0	203.3[a]	188.5	1.08	0.57	40%	4.5
CA031	3%	18.34	1.2	232.7[a]	231.1	1.01	0.73	66%	7.4
CA032	3%	18.66	2.3	219.3[a]	208.3	1.05	0.71	62%	7.0

Specimen code: E, epoxy; C, composite; N, no degassing agent; A, with degassing agent.
[a] The P value used in Eq. (1).