



Fracture resistance, thermal and electrical properties of epoxy composites containing aligned carbon nanotubes by low magnetic field



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ABSTRACT

The alignment of carbon nanotubes (CNTs) with residual catalyst Ni particles in epoxy resin (EP) was achieved under a low magnetic field of 0.4 T. EP composites containing different loading of CNTs were prepared at 60 °C based on the rheology results of CNT/EP suspensions. The effects of CNT loading and magnetic field on the fracture toughness (K_{IC}), glass transition temperature (T_g) and electrical properties of the composites were studied. The obtained results showed significant enhancements in K_{IC} , independent of the orientation of the CNTs, compared to neat epoxy. The maximum increase (~51%) was achieved by composites with 3 wt.% aligned CNTs transverse to crack growth. Additionally, composites with magnetically aligned CNTs displayed anisotropy in K_{IC} . Compared to CNT/EP composites with no applied magnetic field, the K_{IC} increase was less effective at high CNT loading. Finally, toughening mechanisms, T_g and electrical conductivity results were discussed.

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1. Introduction

Epoxy resins are an important class of thermosetting resins widely used in many applications owing to their high elastic stiffness, excellent adhesion, high thermal stability, good chemical resistance, low costs and flexible processability [1]. However, their relative brittleness limits their applications as structural materials where high fracture resistance is often required. Hence, many previous studies were mainly focused on the toughening methods of epoxy resins [1–3]. These include early work using liquid rubber and thermoplastic microparticles as well as recent work using nanoparticles such as silica, rubber and carbon nanotubes (CNTs). Several studies [4,5] also examined the toughness (and stiffness) improvements of epoxy resins by incorporation of two or more types of nanoparticles to achieve a synergistic effect.

Over the past decade, CNTs have attracted much interest for increasing the mechanical properties, especially fracture toughness (K_{IC}) (typically 10–50%), of epoxy resins [6–12] with quite low CNT loading (typically 0.1–1.0 wt.%) as given in Table 1. The major reported toughening mechanisms are crack-bridging, crack

deflection at CNTs agglomerates, localized matrix plasticity and void nucleation/growth [9,10]. Of these mechanisms, crack-bridging is found in almost all CNT/EP composites, which in turn dissipates energies due to subsequent interface debonding and pull-out of CNTs.

Experimental evidence indicates that interfacial bonding strength (which depends on surface treatment) and dispersion of CNTs are two important factors for effective toughening. For the first factor, surface treated CNTs are always better than untreated CNTs. Thus, Park et al. [11] showed that adding 0.5 wt.% untreated and oxyfluorinated CNTs in neat epoxy increased K_{IC} of CNTs/EP by 7.34% and 24.3%, respectively; similarly, Tang et al. [13] reported 30% and 53% K_{IC} improvements compared to neat epoxy with 1.0 wt.% pristine and ozone treated MWCNTs, respectively. Also, Ma et al. [12] showed that untreated CNTs decreased K_{IC} and silane-treated CNTs only increased K_{IC} moderately. These results suggest that toughness increase is controlled by the specific surface treatments applied to the CNTs. It should be noted that stronger interfacial bonding between CNTs and EP does not provide higher fracture toughness to the composites [14–16].

For the second factor, the state of dispersion of CNTs in the epoxy matrix is important for enhancement of toughness. A combination of well-dispersed nanotubes and micro-scale agglomerates was effective for toughening since the agglomerates could

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Table 1

Summary of the different K_{IC} increase on CNT/EP composites due to different loading of CNTs, CNT surface treatment and orientation due to magnetic and electric fields in the reviewed publications.

Matrix	Curing agent	CNT ^a type	CNT loading (wt.%)	CNT surface treatment	Orientation method	$K_{IC}/K_{IC,m}$	Ref.
Epoxy (EPON 862)	Aromatic diamine (Epi-Kure W)	MWCNT (15 nm)	1.0	N ^b	N ^b	1.21	[7]
Epoxy (EPON 828)	Aromatic diamine (Epikure 3402)	MWCNT (14.38 nm)	0.5	N	N	1.65	[8]
Epoxy (L135i)	Amine hardener (H137i)	SWCNT (<2 nm)	0.3	N	N	1.12	[9]
		DWCNT (2.8 nm)	0.3	N	N	1.31	
		DWCNT (2.8 nm)	0.3	-NH ₂ modified	N	1.41	
		MWCNT (15 nm)	0.3	N	N	1.23	
		MWCNT (15 nm)	0.3	-NH ₂ modified	N	1.31	
Epoxy (EPON 862)	Anhydride (MTHPA)	SWCNT (1.1 nm)	0.24	Fluorinated	N	1.43	[10]
Epoxy (YD-128)	Aromatic diamine (DDM)	MWCNT	0.5	N	N	1.07	[11]
		MWCNT (10–20 nm)	0.5	Oxyfluorinated	N	1.24	
Epoxy (EPON 828)	Aromatic diamine (m-PDA)	MWCNT (10–20 nm)	0.5	N	N	-1.26	[12]
				Silane coupling agent	N	1.08	
Epoxy (EPON 828)	Anhydride (MHHPA)	MWCNT ($D = 15$ nm)	1.0	N	N	1.30	[13]
				Ozone functionalized	N	1.53	
Epoxy (LY1564)	Triethylenetetramine	MWCNT (10–20 nm)	0.5	Ozone functionalized	Electric field	1.13	[23]
Epoxy (Araldite-F)	HY951	MWCNT (60 nm)	1.0	N	N	1.33	This work
					Magnetic field	1.49	

^a Number in parenthesis represent diameter of CNTs.

^b N denotes no surface treatment or orientation.

interact with the crack front [7]. Clearly, the length and diameter of CNTs have significant influences on the composite toughness. Long CNTs are superior to short CNTs [17]; small-diameter CNTs limit the pull-out energy dissipation and reduce the dispersibility in the epoxy matrix [18]. In fact, CNTs having a larger aspect (length to diameter) ratio and aligned transverse to crack growth impart higher toughness than randomly oriented CNTs [19,20] due to their more efficient bridging effect discussed earlier.

Various methods for the alignment of CNTs were reviewed in [21–23]. They include electro-spinning and inductions by mechanical force, magnetic field (MF), electrical field and liquid crystalline phase. Due to the anisotropic magnetic susceptibility of CNTs, they are easily aligned contact-free in the applied magnetic field direction, improving the thermal, electrical and mechanical properties with CNT alignment as demonstrated by Kimura et al. [24] and Choi et al. [25]. Although this contact-free MF alignment of CNTs is a distinct advantage over electric field alignment [22], ultra-strong magnetic fields of 7–25 T are often required which are unavailable in most research laboratories. To overcome this difficulty, CNTs were modified by metal fillers [26] or with attached magnetic particles [27] so as to obtain good alignment under low MFs. Furthermore, due to the residual catalysts, e.g., Fe, Ni or Co, used in fabricating CNTs, incompletely purified CNTs can respond well to low MFs (typically 0.1–0.6 T) and are hence aligned in the polymer matrix with improved physical properties [28,29]. However, there are, hitherto, no published reports on the effect of aligned CNTs by (low or strong) MFs on the anisotropic toughness of CNT/EP composites. This issue is addressed in the present work.

Herein, MWCNTs containing residual catalyst Ni particles are used to fabricate magnetically aligned CNT/EP composites under a relatively low applied magnetic field of 0.4 T. Modified single edge notched bend (SENB) specimens are utilized for measurements of fracture toughness along and transverse to MF aligned CNTs. Also, the effects of CNT alignment on the glass transition temperature T_g and electrical conductivity of the composites are studied.

2. Experimental work

2.1. Materials

Diglycidyl ether of bisphenol A (DGEBA) epoxy with an epoxide equivalent weight of 185 g/mol (Araldite-F) and an aliphatic amine hardener (Aradur-HY951) were purchased from Huntsman Advanced Materials Co. Ltd, Australia. Pristine CVD-MWCNTs with an average diameter of 60 nm and average length of 3 μ m, >90% purity, were supplied by Chengdu Organic Chemicals Co. Ltd, China. The CNTs also contained residual catalyst particles and amorphous carbon. The applied magnetic field to the CNTs/EP composites was provided by NdFeB permanent magnets (N38, AMF Magnetics, Australia) with dimensions 50 \times 50 \times 12.5 mm³.

2.2. Preparation of nanocomposites

A master batch epoxy resin containing 5 wt.% CNTs was first prepared: 5 g CNTs were dispersed in 100 g acetone by mechanical stirring for 0.25 h, followed by ultrasonication for 1 h using a 100 W ultrasonic homogenizer (JY 96-IIN, Ningbo Scientz Bio-Tech Co. Ltd, China). Then 95 g epoxy (Araldite-F) was added to the suspension and mixed with a magnetic stirrer for 0.25 h. After that, the mixture was ultrasonicated for 2 h at 60 $^{\circ}$ C being assisted by magnetic stirring to obtain better dispersion of CNTs in epoxy. Acetone was completely removed in a vacuum oven at 100 $^{\circ}$ C for 24 h to yield the master batch which could be diluted by mechanical stirring at 80 $^{\circ}$ C for 0.5 h to obtain homogeneous suspensions with 0.5, 1.0 and 3.0 wt.% CNTs. These suspensions were subsequently degassed in a vacuum oven of -100 kPa for 0.5 h at 80 $^{\circ}$ C. After cooling down to room temperature, curing agent HY951 was added into the suspensions in the resin/hardener (Araldite-F/HY951) weight ratio [30] of 100/10, and gently stirred for 5 min to obtain homogenous mixtures. Then, the mixtures were poured into a silicon rubber mold and placed on a hot steel plate preheated for 3 min at 60 $^{\circ}$ C following the viscosity results obtained in the rheology study (see Section 3.2). Finally, a magnetic field 0.4 T was immediately applied to the preheated mixtures

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