



Fracture behavior of epoxy nanocomposites modified with polyol diluent and amino-functionalized multi-walled carbon nanotubes: A loading rate study



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ABSTRACT

The synergistic effects of reactive polyol diluent and amino-functionalized multi-walled carbon nanotubes on fracture of two- and three-phase (hybrid) epoxy nanocomposites are investigated under quasi-static and dynamic loading conditions. Digital image correlation method with a drop-tower and high-speed camera are used for dynamic tests. The crack-tip deformation histories and fracture parameters for stationary and growing cracks are extracted. Tests show improved crack initiation toughness in modified-epoxies relative to the neat resin with the highest enhancement in hybrid nanocomposites. The dynamic crack initiation toughness values are found to be consistently lower than the static counterparts. Fractographic examinations reveal distinct rate-dependent morphologies.

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1. Introduction

Polymer matrix composites (PMCs) have been widely used in a variety of engineering applications. Among the various matrix systems, epoxy resins are commonly used due to their physical, thermo-mechanical and dielectric attributes. However, their brittleness often leads to structural damages due to poor crack growth resistance. A common approach to improve damage tolerance is to incorporate rubber particles [1–3], thermoplastics [4–6] or stiff fillers [7–9] that alter the overall mechanical and fracture performances favorably [10,11]. The rubbery or thermoplastic phases, however, generally improve ductility but diminish stiffness of thermoset resins whereas the rigid inorganic inclusions such as silica and alumina improve stiffness and strength at the expense of ductility. In addition to the type of fillers, other studies [12–17] on particulate PMCs suggest that fracture toughness can be affected by various factors such as filler size, shape, volume fraction, filler–matrix adhesion strength and the loading rate.

An alternative approach to counteract the reduction in thermo-mechanical properties of PMCs is simultaneous addition of compliant and stiff phases where each contributes its inherent

characteristics to produce optimum stiffness, strength and toughness. Previous works on such hybrid composites [18–20] have shown enhanced fracture toughness and energy absorption. In the recent years, a number of polyol based reactive diluents has been considered as a good choice for improving the fracture resistance of brittle epoxies [20,21]. In addition to the toughening effect, the lower viscosity and the extended pot life of polyols generally increase the level of filler loading as well as the resin wetting action without a substantial decrease in curing rate and thermal stability. These properties make polyols suitable for modification of epoxy resins to achieve improved peel and impact strengths, and facilitate processing of particle-filled and fiber-reinforced PMCs. For instance, the use of polyether polyol as a toughener for epoxy resins by Isik et al. [20] provided 160% enhancement in impact strength. In the past few decades, researchers have also successfully tailored the matrix properties by using nanofillers [22]. Since their discovery in 1991 by Iijima [23], carbon nanotubes (CNTs) have emerged as potential candidates for matrix modification because of their exceptional strength and stiffness [24], flexibility, diameter dependent specific surface area and high aspect ratio with low density [25]. These remarkable features of CNTs make them to act as bridges between crack faces and induce interlocking with the matrix material. However, to incorporate CNTs as effective reinforcements, good dispersion and interfacial adhesion between matrix and CNTs is desirable. Previously, it has been

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shown that surfactant treatment and amino-functionalization of CNTs enhance their dispersibility in the epoxy matrix [26–30]. Furthermore, multi-walled carbon nanotubes (MWCNTs) exhibit better dispersion than single walled carbon nanotubes (SWCNTs) because of lower specific surface area in the former than the latter. Considering these features of polyols and CNTs, the authors [31] have recently processed and characterized epoxy composites modified with reactive polyol diluents and randomly-oriented amino-functionalized multi-walled carbon nanotubes (NH₂-MWCNTs).

Nanocomposites modified with CNTs as reinforcements in polymer matrices have been extensively investigated over the years [25,32]. The dispersion of 0.1 wt.% of amino-functionalized double-wall carbon nanotubes (DWCNTs) in epoxy using a calendering technique by Gojny et al. [33] resulted in improved tensile strength, Young's modulus and quasi-static fracture toughness. A thermo-mechanical study by Fidelus et al. [34] showed 70% improvement in tensile impact strength at 0.5 wt.% of randomly oriented MWCNTs in epoxy resin. In another thermo-mechanical investigation, Zhou et al. [35] noted 90% improvement in storage modulus and an optimum flexural strength with 0.3 wt.% MWCNT/epoxy system. Seyhan et al. [36] used 3-roll milling to disperse CNTs into vinyl-ester-polyester hybrid resin. They found that nanocomposites containing MWCNTs and NH₂-MWCNTs exhibited higher tensile modulus, fracture toughness and fracture energy values relative to DWCNTs and NH₂-DWCNTs counterparts. Hsieh et al. [37] noted improved tensile, fracture and fatigue performances with increasing MWCNTs content in an anhydride-cured epoxy. Some efforts to enhance the fracture toughness of CNT/epoxy nanocomposites by simultaneous addition of a third phase filler such as rubber and/or nanosilica particles have also been made [38,39].

While numerous studies have been reported on plasticized epoxies and/or CNT reinforced nanocomposites, they mostly deal with material processing aspects, thermo-mechanical characterization, and are limited to fracture behavior under quasi-static loading conditions. A few works, however, have addressed dynamic fracture behavior of nano-size spherical particle-filled nanocomposites. For example, Shukla et al. [40] and Evora et al. [41,42] reported improved fracture toughness and higher crack velocities in TiO₂ (35 nm) and Al₂O₃ (14 nm) nanoparticle filled composites relative to the neat resin. A recent study by Jajam and Tippur [16] on fracture behavior of particulate composites showed that PMCs are indeed loading rate dependent. They observed higher crack initiation toughness for nano-silica (20 nm) filled epoxies under quasi-static loading when compared to low velocity impact loading while both showed improvement relative to unfilled epoxy. Note that much of the published research to date on fracture behavior of nanocomposites has been performed quasi-statically and very limited data exists from the perspective of dynamic crack growth caused by rapid loading. Further, the reported ones primarily deal with low aspect ratio fillers. Higher aspect ratio of stiff fillers in conjunction with plasticizers, however, could vary the mechanical response in general and fracture behavior in particular. To the best of authors' knowledge, no study on dynamic fracture related to the combined effect of CNTs and plasticizers on epoxy system, has yet been documented in the literature. These

gaps need to be bridged if such materials are to find engineering applications where stress-wave loading conditions dominate. Thus, the objective of the present research is to study fracture behavior of epoxy composites modified with reactive polyol diluent and randomly-oriented amino-functionalized MWCNTs under dynamic loading conditions. The loading rate effects and synergistic characteristics of NH₂-MWCNTs and polyether polyol on epoxy resin system are of particular interest.

2. Materials processing and characterization

2.1. Materials

A low viscosity epoxy system (Applied Poleramics Inc., USA) consisting of unmodified diglycidylether of bisphenol-A (DGEBA) resin cured by cycloaliphatic amine hardener was used as the matrix. An epoxy terminated polyether polyol (triglycidyl ether of propoxylated glycerin) (Applied Poleramics Inc., USA) was used as a reactive diluent and toughener. The amino-functionalized multi-walled carbon nanotubes (NH₂-MWCNTs) synthesized by catalytic chemical vapor deposition (purity > 95%, average diameter ~10 nm, average length ~1 μm) received from Nanocyl, Belgium, were used as stiff fillers.

2.2. Composites manufacturing process

Four categories of samples were prepared in this study: neat epoxy, epoxy/CNT(0.3 wt.%), epoxy/polyol(10 phr) and hybrid epoxy/CNT(0.3 wt.%)/polyol(10 phr). Table 1 presents the sample codes and formulations of all the epoxy composites used in this work. The choice of 0.3 wt.% NH₂-MWCNTs and 10 phr polyol was based on previous studies [31,43] that offered optimum gain in mechanical properties.

For EP–CNT system, the 0.3 wt.% NH₂-MWCNTs were dispersed in unmodified DGEBA resin at room temperature using a sonicator probe at 35% amplitude and a 30 s 'on'/30 s 'off' cycle in pulse mode for 1 h. To overcome the increase in pressure and temperature, the mixture was kept in a cooling bath during sonication. For effective dispersion of CNTs, the sonicated mixture was subsequently subjected to a three-roll shear mixing process, as shown schematically in Fig. 1. The rollers 1 and 3 rotate in the same direction and opposite to the middle roller 2 thereby inducing shear to the mixture. A gap setting between the rollers of 20 μm (1st pass), 10 μm (2nd pass) and 5 μm (3rd pass) was used to induce a high degree of shear to the mixture. The speed of the rollers was maintained at a ratio of 1:3:9 with a maximum speed of 200 rpm in all the three passes.

A conventional mechanical mixing technique was used to prepare EP–POL system by blending 10 phr polyol into the unmodified epoxy resin. For hybrid EP–CNT–POL system, 0.3 wt.% NH₂-MWCNTs were dispersed in 10 phr polyol modified epoxy resin using sonication and three-roll shear mixing process described above. The schematic shown in Fig. 1 depicts the manufacturing process for polyol modified epoxy (EP–POL) and epoxy/NH₂-MWCNTs/polyol (EP–CNT–POL) hybrid composites.

Table 1
Formulation of neat and modified epoxy samples.

Sample nomenclature	DGEBA ^a (phr)	Polyol content (phr)	NH ₂ -MWCNTs content (wt.%)
EP (neat epoxy)	100	0	0
EP–CNT (Epoxy + NH ₂ -MWCNT)	100	0	0.3
EP–POL (Epoxy + Polyol)	90	10	0
EP–CNT–POL (Epoxy + NH ₂ -MWCNT + Polyol)	90	10	0.3

^a The hardener content was 30 phr for all formulations.

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