



Fracture toughness and failure mechanism of graphene based epoxy composites



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ABSTRACT

The present study investigates the effect of addition of three different types on carbon nano-fillers on fracture toughness (K_{IC}) and failure mechanism of epoxy based polymer nano-composites. The carbon nano-fillers were dispersed in the epoxy matrix using a three-roll mill and the three nano-fillers used for this experiment were (i) thermally reduced graphene oxide (TRGO); (ii) graphite nano-platelets (GNP); and (iii) multi-wall carbon nano-tubes (MWCNT). The fracture toughness was measured as a function of weight percentage of the filler using single edge notch three-point-bending tests. The toughening effect of TRGO was most significant resulting in 40% increase in K_{IC} for 0.5 wt% of filler. On the other hand, the enhancements in toughness were 25% for GNP/epoxy and 8% for MWCNT/epoxy. Investigations on fracture surface revealed that crack pinning or bi-furcation by TRGO and crack face separation initiated from TRGO contributed to enhance the fracture toughness. Based on the observations, a schematic explaining the crack propagation in graphene/epoxy composite and the interaction of crack front with graphene particles was proposed.

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1. Introduction

Epoxy polymers that fall under the class of thermoset polymers are used as matrices for fiber reinforced composites and also as adhesives. In view of their cross-linked network structure, these polymers are used in structural engineering applications owing to their low moisture absorption, high modulus and high temperature performance. However, they also come with an undesirable property of brittleness with low fracture resistance because of their structure. Thus, the enhancement in fracture toughness has been a critical issue when it comes to their application and hence engineers have been working on the toughening of epoxies for several decades [1,2].

Researchers have come out with a solution of incorporating secondary phase particles in the epoxy matrix in order to increase its toughness. The dispersed secondary phase particles can either be soft fillers (e.g., thermoplastic particles, rubber, etc.) or rigid fillers

(e.g., silica beads or titania or alumina) [3]. Here, the toughening effect of soft particles is significant on the epoxy resin; however, they also reduce the stiffness and the glass transition temperature (T_g). On the other hand, modification using rigid fillers enhances the stiffness and T_g of the epoxy composites, the toughening effect was generally not significant. To overcome these trade-off relationships, more effective rigid fillers had been desired [4–7].

With the emergence of nano-technology, the enhancement of nano-composites in mechanical properties (toughness) is considerable at lower loadings. These nano-composites are polymers (epoxy in our study) that are reinforced with nano-phase rigid fillers (nano-silica, nano-clay, carbon nanotubes, carbon black, graphene, and fullerene). These nano-fillers enhance the toughness to a larger extent than micro fillers at very low filler loading due to their high surface area [8–10].

Understanding the relationship between the observed fracture properties of epoxy composites with the microstructure of the dispersed secondary phase particles is important to identify the mechanism responsible for the enhanced toughness. For nano-composites, where the dispersed particles are in the nano-scale, several toughening mechanisms come to play depending on the filler type. For example, nano-sized silica particles increase the

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toughness of the epoxy matrix through de-bonding of particles which is followed by plastic void growth [11]. There were also reports on nano-void formation and presence of a dilatation zone when the interface is strong [12]. Whereas, for carbon nano-tubes reinforced epoxy system, pull-out of nano-tubes and de-bonding seems to contribute to the increase in fracture energy and the contribution from plastic void growth is minimal [13]. When it comes to fillers with layered structures such as nano-clay or silicates (layered), crack-deflection, micro-cracking and plastic void growth is the major toughening mechanisms [14,15]. With graphene a latest addition to the carbon nano-filler family, the major toughening mechanism in the graphene/epoxy composites still needs to be investigated.

It is seen from the reported literature that, the toughening effect of graphene is significant when compared to nano-tubes. Improvements in fatigue life and fracture toughness were observed in glass fiber reinforced polymers when infiltrated with graphene modified epoxies [16,17]. In addition to the enhanced fracture toughness, graphene based polymer composite also exhibit relatively high electrical conductivity (in thermoplastic matrix), thermal conductivity and Young's modulus [18,19]. Indeed, previous works have shown that the fracture toughness increases by 53% for graphene based epoxy nano-composites [20]. A comparative graph on the normalized fracture toughness of nano-composites based on nano-clay, multi walled carbon nano-tubes and graphene as a function of filler content is shown in Fig. 1.

It can be seen that nano-clay toughened epoxies show an increase in K_{IC} but at higher filler contents (1.0–10.0 wt%) [34]. At the same time, MWCNT reinforced epoxies, show slightly higher K_{IC} but at lower filler content (0.1–1.0 wt%). Graphene based epoxy composites show better improvement in K_{IC} at lower filler content varying from 0.01 to 1.0 wt%. Though there are literature available on the fracture toughness of graphene based epoxy composite, the potential of toughening effect and toughening mechanism of graphene based epoxy composite have not been well discussed.

The main aim of this article is to understand the toughening effect for epoxy nano-composites reinforced with three different types of carbon nano-fillers (MWCNT, TRGO and GNP). In particular, toughening mechanisms of graphene fillers (TRGO and GNP) were discussed in detail by fracture surface observation and filler dispersion state observation of the epoxy nano-composites through scanning electron microscopy.

2. Materials and methods

2.1. Materials

The matrix chosen for this work is an epoxy resin. It has a modulus of 2.7–3.0 GPa and glass transition temperature (T_g) \sim 140 °C and can be used later in preparing structural components. The epoxy resin used is diglycidyl ether of bisphenol A (Araldite LY556) with an anhydride hardener (Aradur CH917) and imidazole accelerator (DY070) in ratios of 100:90:1 by weight. All components for matrix resin were supplied by Huntsman Corporation. For the nano-fillers, the MWCNTs used were NC7000 series supplied by Nanocyl S.A, Belgium. The tubes have an average diameter of 9.5 nm, 1.5 μ m long with a purity of 90% (from the datasheet). The graphite nano-platelets GNPs were supplied by Punto Quantico, Italy. These have a purity of 96% and the lateral size of the sheets varies from 20 to 50 μ m (from the datasheet). The TRGO sheets were produced by co-authors in Freiburg University, Germany. A detailed procedure on the synthesis of TRGO was described in previous papers [35,36]. The SEM images of as-received MWCNT, GNP and TRGO are shown in Fig. 2.

2.2. Preparation of the nano-composite

Composite suspensions were produced using three-roll-mill (EXAKT® 120E). The three-roll mill works on the principle of applying very high shear rates (approx. $250,000 \text{ s}^{-1}$) on the mixture which enables to disperse the nano-fillers in a highly viscous medium like epoxy. Appropriate amounts of the nano-filler and the epoxy resin were mixed inside a glove box before milling. The mixture is initially fed through the feed roll and was collected at the apron roll. The distance between the rollers was varied from 120 μ m down to 5 μ m and the speed of the apron roll was maintained at 300 rpm [37]. To this suspension, the hardener and accelerator are added and mixed manually for 10 min. The mixture was then degassed under vacuum at 50 °C to remove the air bubbles and was poured into the curing mold and cured in the oven at 80 °C for 4 h and further tempered at 140 °C for 8 h.

The nano-composite for each filler type was prepared at varying filler content from 0.05 wt% till 2.0 wt%. In some cases, higher filler content could not be reached owing to the difficulty in processing due to high viscous nature of the nano-composite suspension. The

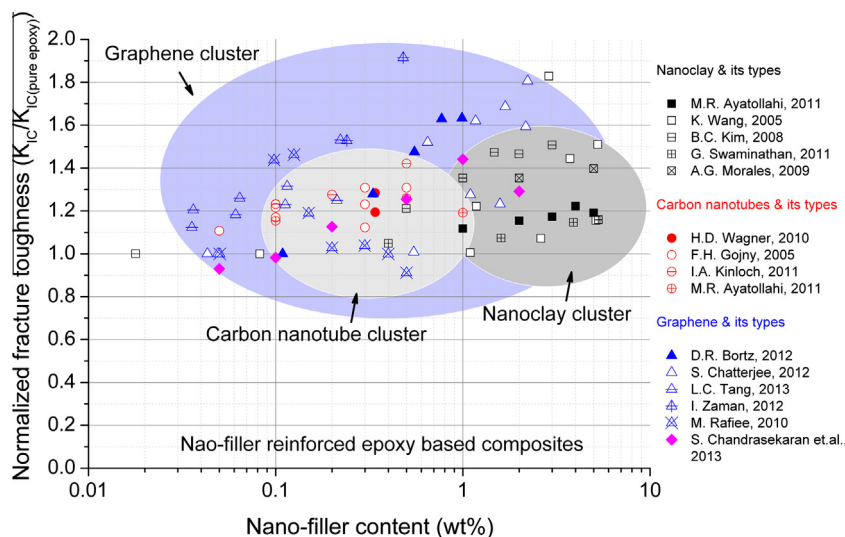


Fig. 1. Normalized fracture toughness as a function of filler content for different nano-filler reinforced epoxies. References used for the plot [13,21–33].

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