



# Fracture properties of nanographene reinforced EPON 862 thermoset polymer system



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## ARTICLE INFO

### Article history:

Received 12 November 2014

Received in revised form 25 February 2015

Accepted 9 April 2015

Available online 16 April 2015

### Keywords:

Graphene

A. Nano composite

B. Fracture toughness

D. Scanning Electron Microscopy (SEM)

D. Atomic Force Microscopy (AFM)

## ABSTRACT

This paper studies the differences in fracture properties (stress intensity factors and energy release rates) of nanocomposites of thermosetting polymer EPON 862 and its nano-graphene reinforced counterparts. Extremely low (0.1 and 0.5) weight percent nano-graphene platelets were dispersed in EPON 862 matrix and compact tension (CT) fracture experiments were conducted under quasi static loading conditions using displacement control. Significant enhancements in fracture toughness (~200%), and energy release rate (~570%) respectively were observed for nano-graphene reinforced matrix for only 0.5 wt% of graphene platelets. Fractography analysis of the fractured CT specimens was used to qualitatively visualize and understand the mechanism(s) responsible for the enhancement in these properties using Scanning Electron Microscopy (SEM). Evidence of crack deflection due to increased surface roughness, graphene platelet pullout and plastic deformation of the matrix causing filler-matrix debonding, was observed from SEM micrographs, caused by the addition of nano-graphene platelets (NGP). Atomic Force Microscopy (AFM) was also used to quantify the magnitude of surface roughness changes between the NGP reinforced and reinforced nano-composite samples, and correlate surface roughness changes due to crack deflection to increased fracture toughness.

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## 1. Introduction

Graphene's wide range of applications from energy storage applications to immunosensors has made it an ideal test candidate as a filler for polymer systems due to its superior mechanical, electrical and thermal properties [1,2]. Polymer/nano graphene composites have attracted widespread interest in industry and research due to superior mechanical enhancements at very low filler volume fractions [3–6]. Carbon nanotubes (CNT) have also been widely researched for similar purposes due to their compatibility with various polymer systems and well as possibility of surface modification of CNTs, but the higher cost of manufacturing high-grade CNTs has made researchers lean toward graphene as a better option for large scale applications and superior epoxy-based composite materials [7–12].

The macro scale properties of polymer based nanocomposites depend on thermodynamic factors which include interfacial compatibility of the polymer with the nano-filler phase, and nano-scale dispersion and distribution of the filler, which in turn depends on

the aspect ratio of the filler, dispersion techniques, time of mixing and applied shear, bonding between the filler and the matrix and the volume fraction of the filler, etc. [13–16]. Full advantage of the NGP fillers can only be taken by considering all of the processing factors mentioned above, which would lead to better load transfer between the polymer matrix and the filler surface, leading to superior mechanical properties.

Various strategies have been proposed to disperse the platelets in the polymer matrix with solution mixing and shear mixing being the most widely used. Solution mixing involves dispersing the NGPs into suitable organic solvents using sonication and adding the polymer followed by evaporation of the solvent [17–20]. Shear mixing mechanically disrupts the low strength shear bonds between individual NGPs to disperse the platelets. King et al. [21] reported excellent dispersion of NGPs using purely high shear mixing in the epoxy matrix. The major drawback of the solvent mixing method is the time required to evaporate the solvent from the polymer/NGP blend. It has also been shown recently that even traces of solvents left in the blend can cause reduction in crosslinking of the polymer chains [22,23].

Chemical functionalization of the NGPs and the epoxy matrix has been used as another alternative for better dispersion of the

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filler, which is mainly used to obtain exfoliated NGPs in the matrix [24–26]. Kim et al. [27] achieved good dispersions of NGPs by functionalization of low density polyethylene matrix. Rafiee [16,28] reported similar dispersion of the GNPs using chemical functionalization of the GNPs through oxidation of bulk graphite in acid, followed by rapid heating in a tube furnace. Although this process reduces the agglomeration of the graphene sheets and leads to better phase mixing, it also adds impurities to the system, which are difficult to remove [29]. Graphite oxide sheets have been observed to form stable dispersions in water after ultra-sonication which, after de-oxygenation, can be reduced back to graphene. However, most graphene sheets exhibit crumpled and wrinkled morphology, which several authors have debated to be an added advantage toward better load transfers and mechanical interlocking between the matrix and the filler [16,29,30].

Recent studies using graphite oxide sheets as the precursors to generate nano-graphene platelets have reported tremendous potential of graphene nanocomposites in improving the elastic modulus (~100%), fracture toughness (~65%), fracture energy (~115%) and reduction in fatigue crack propagation rates (~25-fold), for very low 0.1–1 wt% NGP for different thermoset systems [16,28–35]. The major improvement in these reports has been attributed to the increased surface roughness, crack pinning and crack deflection processes due to the presence of NGP. It has also been reported that particle matrix debonding and plastic deformation of the matrix could be potential mechanisms for the drastic increase in mechanical properties [35]. Thus, it is clear that graphene opens new avenues in the future of lightweight nano-composite structures, with its exceptional mechanical properties. However to the best of the authors' knowledge, such high increases in fracture energy for the thermoset polymer/nano-graphene system reported herein have not been previously observed and studied thoroughly.

EPON 862 is an emerging epoxy system used for research applications due to its low processing viscosity and better mechanical properties for composite applications. EPON 862 is an aerospace grade di-functional epoxy resin with very low molecular weight. It is categorized as a thermoset polymer system with high crosslink density when mixed with curing agent 'W'. Although the polymer itself is strong, it is relatively brittle with low stiffness.

In this study, the thermoset epoxy EPON 862 was selected to investigate the changes in fracture properties (toughness and

energy) of its nano-graphene reinforced counterpart. Compact Tension (CT) specimens with nanometer sized starter cracks were used in this study, with four replicate specimens per NGP loading. NGP loadings of 0 wt% (baseline), 0.1 wt% and 0.5 wt% were employed. ASTM standard 5045 was used to perform the fracture testing under room temperature and quasi-static loading conditions. Nano-graphene reinforced epoxy specimens were observed to increase the fracture toughness by 141% and 200% for 0.1 and 0.5 wt% respectively, and fracture energy by 347% and 567% for 0.1 and 0.5 wt% NGP respectively, compared with baseline epoxy. The major toughening mechanisms were studied using SEM and AFM, and both qualitative and quantitative rationales behind such dramatic increase in toughness are discussed.

## 2. Experimental section

### 2.1. Materials

Graphene nanoplatelets with an average diameter of 15  $\mu\text{m}$  were procured from XG-Sciences Inc. The platelets were supplied in small stacks (15–20 graphene platelets) as depicted in Fig. 1(A) with 99.9% purity. The stack has an average thickness of 6 nm and a typical surface area of 120–150  $\text{m}^2/\text{g}$  [36]. The epoxy used in the study was EPON 862, which is di-glycidyl ether of bisphenol-F epoxy (DGEBF) from Momentive Inc. and the curing agent used was Curing agent 'W' (DETDA (diethyl toluene diamine)). Fig. 1(B) and (C) illustrates the molecular structures of the thermoset polymer and curing system used in the study. DETDA amine groups act as the crosslink centers.

### 2.2. Dispersion of NGP

The desired wt% of NGP was weighed and dispersed in the epoxy using a high shear mixer (EuroStar power-b) at 2000 rpm for 45 min. The high shear mixing was performed based on the dispersion notes from the NGP manufacturer [36]. The curing agent was then added to the mixture and the blend was stirred for 3 min at 2000 rpm. The epoxy to curing agent weight ratio was 100:26.4 as provided by the manufacturer.

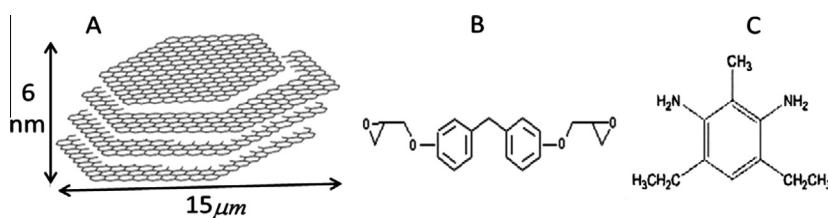


Fig. 1. (A) Schematic of the supplied nano graphene platelet (NGP) stack [36], molecular structure, (B) EPON 862 and (C) DETDA [39].



Fig. 2. CT samples after curing process (a) baseline, (b) 0.1 wt% and (c) 0.5 wt% NGP.

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