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Flexural behaviour of CNT-filled glass/epoxy composites in an in-situ environment emphasizing temperature variation

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ABSTRACT

The paucity of structural defects in carbon nanotube (CNT) with unrivalled mechanical properties has always posed an interest to material scientists for its potential incorporation in soft polymer resins to achieve superior mechanical stability. Present investigation focuses on the assessment of flexural behaviour of glass/epoxy (GE) and multiwalled carbon nanotubes (MWCNT) embedded glass/epoxy (0.3 wt. % of epoxy) (CNT-GE) composites at different in-service environmental temperatures. In-situ 3-point bend tests were performed on GE and CNT-GE composites at −80 °C, −40 °C, room temperature (20 °C), 70 °C and 110 °C temperatures at 1 mm/min crosshead speed. The results revealed that at 110 °C temperature, the flexural strength of GE and CNT-GE composites was significantly decreased by 67% and 81% respectively in comparison to their strength at −80 °C temperature. Similarly, 38% and 77% decrement in modulus was noted for GE and CNT-GE composites respectively. Dynamic mechanical thermal analysis (DMTA) was carried out in the temperature range of −100 °C to 200 °C to correlate the mechanical and thermo-mechanical response of both the material systems. Addition of 0.3 wt. % MWCNT in GE composite resulted in lowering of glass transition temperature (T_g) by 12 °C. Furthermore, to understand various possible deformation and failure mechanisms, the post failure analysis of the fractured specimens, tested at different temperatures, was carried out using scanning electron microscope (SEM). The critical parameters needed during designing composite structures were calculated and modelled using Weibull constitutive model.

1. Introduction

In contrast to the constituent materials, FRP composites do exhibit quite reliable material properties. The tailorability of the interface/interphase in composite structures has successfully generated a new prospective era for material scientists to design materials with well desired properties for various high performance engineering applications. Restricting the premature failure of FRP composites in various isolated cases has been a major challenge for material engineers from the last decade. The durability and structural integrity of these materials are strongly affected by the in-service environments like high and low temperature [1], loading rate, humidity [2] and other harsh environmental conditions [3]. For laminated composites, the in-plane mechanical properties are governed by the fiber phase whereas the z-direction (perpendicular to the plane of the laminate) properties are mostly limited by the matrix phase [4,5]. Scientifically engineered nano-fillers have been reported to be more reliable choice to improve these properties, which have been acknowledged round the globe [6]. Along with this improvement, it is of great interest to investigate the effect of nanofiller incorporation on the environmental degradation of these potential materials. Undoubtedly, carbon nanotube (CNT) is one of the most promising nanofillers for effective enhancement of the mechanical, electrical and thermal properties of polymer matrix composites (PMCs) [7–13]. Addition of 1 wt.% of MWCNT into a carbon fiber/epoxy composite has shown significant enhancement in its tensile and inter laminar shear strength with a reduced dispersity [14]. The low and cryogenic temperature strength of epoxy has been reported to be significantly enhanced due to addition of CNTs [15]. As temperature induced damages further may degrade the properties of the composite material, its structural integrity must be ensured at the service temperature [16]. Several literature are available explaining the pros and cons of CNT modified polymer, but current time

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demands fabrication and effective evaluation of mechanical performance and integrity of CNT modified hierarchical composite materials at various temperature environments [17,18].

Present study aims to investigate the mechanical and thermo-mechanical performance of MWCNT modified glass fiber/epoxy (CNT-GE) composites at elevated and low temperature environments. This, in turn, proposes the applicability, reliability and predictability of the CNT-GE composite.

2. Experimental section

2.1. Materials and processing of composite laminates

The epoxy resin used in the present study is diglycidyl ether of Bisphenol A (DGEBA) and the hardener is Triethylene tetra amine (TETA), supplied by Atul Industries Ltd, Gujarat, India under the trade name of Lapox L-12 and K-6 respectively. The glass fiber used is a 3 K plain weave type with 15 μm filament diameter supplied by Saint-Gobain, India. The MWCNTs having an outer diameter of 6–9 nm with 5 μm length were purchased from Sigma–Aldrich. Some salient properties of the constituents of laminated composite are provided in Table 1.

The volume fraction of fibres is approximately 50%. For fabricating CNT-GE composite, the epoxy resin was modified with MWCNT prior to hand lay-up method. The MWCNT content in the epoxy resin was kept at 0.3 wt. % of the epoxy taken. Required amount of CNTs was taken and dispersed in 250 mL acetone. The suspension was then stirred at 1000 rpm for 30 min followed by sonication for 30 min at room temperature. At this stage, stirring and sonication were done to facilitate deagglomeration of the as supplied CNTs in acetone medium. The CNT/acetone mixture was then transferred to a flask containing the pre-weighed epoxy, which was already heated at 70 °C for 30 min to impart sufficient fluidity. Then the epoxy/CNT/acetone mixture was stirred at 1000 rpm for 2 h at 70 °C. Sonication was again carried out at 70 °C for 1 h. At this stage, removal of all acetone was ensured. Then the epoxy/CNT suspension was kept in vacuum for 12 h. This was done for the removal of air bubbles entrapped in the suspension during stirring and sonication. Then to the suspension, required amount of hardener (10 wt. % of epoxy as specified by the supplier) was added and mixed well. The laminated composites were prepared by hand lay-up method with 14 layers of woven fabric cloth of reinforcement and then placed in a hot press. Then the curing of the laminate was carried out at 60 °C temperature and 10 kg/cm^2 pressure for 20 min. Similarly, the controlled glass fiber/epoxy (GE) composite laminate was fabricated using 14 layers of glass fiber and neat epoxy by hand lay-up method followed by hot compression using the same parameters as were in CNT-GE composite. The laminates were then kept at room temperature for 24 h. The flexural (as per ASTM D7264) and DMTA (as per ASTM D7028) samples were cut from the laminates using a diamond tipped cutter. After cutting, all the samples were post-cured at 140 °C for 6 h.

Table 1
Salient properties of epoxy and glass fiber.
Specifications of glass woven fabric-
Warp and weft density: 16 and 14 yarns/inch respectively;
Fabric weight: 360 gsm;

Property	Epoxy	Glass fibres
Density (g/cm^3)	1.162	2.58
Tensile modulus (GPa)	4.1	72.3
Tensile strength (GPa)	0.11	3.4
Strain at failure (%)	4.6	4.8
Poisson's ratio	0.3	0.2

2.2. Thermo-mechanical and mechanical characterization

The thermo-mechanical analysis was carried out as per ASTM D7028 in the dynamic mechanical thermal analyser (DMTA) (Netzsch DMA 242E) from −100 °C to 200 °C temperature range at a heating rate of 10 °C/min. In DMTA, the specimens were loaded in a 3-point bending mode using a frequency of 10 Hz.

In-situ flexural testing of the samples was carried out at −80 °C, −40 °C, room temperature (20 °C), 70 °C and 110 °C (with a holding time of 10 min) with a loading rate of 1 mm/min using the 3-point fixture in the environmental chamber of universal testing machine (Instron 5967) following ASTM D7264 standard. The instrumental set-up with the loading fixture for DMTA and in-situ flexural testing is shown in Fig. 1.

3. Results and discussion

3.1. Dynamic mechanical thermal analysis (DMTA)

DMTA is a tool to evaluate the viscoelastic response of the material for a wide range of temperature. The instrument applies a dynamic load to the sample and the response of the material is recorded in the form of dynamic displacement. For a perfectly elastic solid, the applied stress and resulted strain remain in phase, whereas there is a phase difference in case of polymeric (viscoelastic) material. The storage modulus (E′) obtained from DMTA is a representation of the elastic modulus of the material, whereas the loss modulus (E″) reflects the viscous modulus. The damping tendency of the material is determined from the parameter tanδ (ratio of E″ to E′). The E′, E″ and tanδ are determined using the following equations.

$$E' = \frac{\sigma_o}{\varepsilon_o}\cos\delta \tag{1}$$

$$E'' = \frac{\sigma_o}{\varepsilon_o}\sin\delta \tag{2}$$

$$\tan\delta = \frac{E''}{E'} \tag{3}$$

where, σ_o and ε_o represent the peak stress and peak strain respectively and δ is the phase difference between the stress and strain.

Fig. 2 refers the variation in E′, E″ and tanδ with temperature for GE and CNT-GE composites. It is evident from Fig. 2(a) that at temperature below glass transition temperature (T_g), the E′ for CNT-GE composite is higher than that of GE composite. The rate of decrement of E′ with temperature (upto T_g) is higher in CNT-GE composite than that of GE composite. The onset of sharp change in slope of the E′ verses temperature curve is taken as the T_g of the material. From Fig. 2(a) it can be observed that addition of 0.3 wt. % CNT results in lowering of the T_g from 139 °C to 127 °C. This decrement in T_g may be attributed to the hindrance of crosslink formations due to the entrapment of CNT between the polymeric chains. Fig. 2(b) shows the change in E″ due to CNT incorporation in the composite. The tanδ value reduces due to CNT addition showing more induced brittleness in the composite as observed from Fig. 2(c).

3.2. Flexural behaviour at various testing temperatures

The stress–strain curves of the GE and CNT-GE composites at low, room and elevated temperature environments are shown in Fig. 3. As the T_g of both the composites are below 140 °C, in-situ