



Temperature dependent reinforcement efficiency of carbon nanotube in polymer composite



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ABSTRACT

The transcendent mechanical properties of carbon nanotube (CNT) hold the promise of delivering contributory reinforcing effect in soft polymeric materials. But, the built-in-risk of the environmental susceptibility of the CNT/polymer interface should be well explored before certifying it for a particular application. Present investigation reports the reinforcement efficiency of CNT in epoxy as a function of environmental temperature. Nanocomposite with 0.2% CNT, which shows maximum strength and modulus at room temperature, exhibits the poorest strength and modulus at 90 °C. Dynamic mechanical thermal analysis (DMTA) has also been carried out to study the variation of thermomechanical properties of nanocomposites with temperature. A decrement in the glass transition temperature (T_g) of the polymer was obtained due to CNT reinforcement upto 0.2%. Post-failure fracture surface analysis was done to underneath the dominating strengthening, toughening and weakening mechanisms.

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1. Introduction

Replication of the unique properties of the carbon nanotube in the polymer is the key target to achieve superior mechanical, thermal and/or electrical properties of the materials. Many research articles have reported the beneficial mechanical performance of CNT reinforced polymers at room temperature [1]. Various environmental parameters have been reported to influence the mechanical behavior of polymeric composites [2]. Addition of CNT into polymeric materials has shown remarkably improved cryogenic mechanical performance [3]. But to the best of our knowledge there is a dearth of open literature on the mechanical performance of such CNT embedded polymer composite when the service temperature is relatively high. These nanocomposites are potential materials for applications like EMI shielding, where there is a significant chance of internal heat generation, leading to substantial temperature increment in the material [4]. So, the performance of the material must be well ensured to avoid any unprecedented failure. "Whether the strength enhancement mechanism still remains valid at elevated temperatures and what is the role of CNT content in the nanocomposite on its elevated temperature durability", these two questions have been tried to answer in the current paper.

2. Experimental procedure

Epoxy resin used in this study was diglycidyl ether of Bisphenol A (DGEBA) and hardener was Triethylene tetra amine (TETA), both were supplied by Atul Industries, India under the trade name Lapox, L-12 and K-6 respectively. The MWCNT used has an outer diameter 6–9 nm, length 5 μ m, purchased from Sigma Aldrich, USA.

Preweighed amount of CNT (0.1, 0.2, 0.3 wt% of epoxy) was added to acetone and then stirred at 1000 rpm for 30 min followed by 30 min sonication. This mixture was then added to epoxy and stirred at 70 °C and 1000 rpm until entire acetone was evaporated, followed by 1 h sonication at 70 °C. The suspension was kept under vacuum for 18 h. Then, to the suspension, required amount of hardener (10% of epoxy) was mixed properly and poured into moulds. Neat epoxy (with hardener) was also poured in similar moulds. These moulds were kept in vacuum for 10 min followed by room temperature curing for 24 h. The samples were removed from the moulds carefully and well-polished to have better surface finish and uniform geometry. The final samples were then post cured at 120 °C for 6 h.

3-point flexural tests were carried out (ASTM D790) using UTM (Instron 5967) attached with environmental chamber at various temperatures with loading rate 1 mm/min with 10 min holding at each temperature. In DMTA (Netzsch DMA242E) samples were loaded on a 3-point bending fixture and heated from 40 °C to

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200 °C at a heating rate of 5 °C/min and 1 Hz (ASTM D7028). The fractographic images of the failed samples were captured using SEM (FEI NOVA NanoSEM-450).

3. Result and discussion

The stress-strain curves of all nanocomposites tested at room temperature (RT), i.e. 30 °C, and 90 °C, and the variation in strength and modulus with temperature and CNT content are reported in Fig. 1. Results revealed that addition of 0.1% and 0.2% CNT into epoxy resulted in 7.1% and 17.4% improvement in strength respectively, at RT. But, after that, modulus becomes saturated whereas strength drops. The high specific surface area of CNTs brings out high CNT/epoxy interfacial area, giving rise to better mechanical properties in nanocomposites, only when the CNTs are well dispersed in the polymer. The increment in the flexural properties upto 0.2% might be attributed to the uniform dispersion of CNTs in the polymer resulting availability of larger interfacial area through which effective stress transfer takes place from the soft matrix to the ultra-strong CNT. In addition, well dispersed CNTs may reduce the free volume of the polymer matrix by their mating accommodation into the polymer network [5], thus increasing its rigidity. The interfacial region (interphase) extended from the CNT surface to bulk polymer exhibit non-uniform characteristics and may be considered to be composed of two types of polymer layer. First layer which is close to CNT surface can be designated as part of the polymer which is immobilized or tightly bound as proposed by Tsagaropoulos and Eisenberg [6]. The second layer may be composed of polymer of reduced mobility or loosely bound. The tightly bound layer can contribute towards the

stiffness enhancement and the loosely bound layer offers effective stress transfer from the polymer to CNTs. When the CNT content was increased from 0.2% to 0.3%, the strength enhancement was dropped from 17.4% to 11.2% (with respect to neat epoxy). In addition, it was also found that addition of 0.2% CNT helps in improving the strain to failure by 28.7%. The reason may be attributed to various toughening mechanisms like CNT pull-out and polymer crack bridging by CNT, which are also discussed later from their microscopic images. Hence, CNT incorporation into epoxy not only strengthens the material, but also makes it tougher, which widens its scope of engineering applications. At higher CNT content (here 0.3%), all the CNTs may not be dispersed in the epoxy and remains as undispersed CNT bundles [7] or agglomerates. In addition, relatively higher amount of CNTs may reduce the net volume of the loosely bound polymer in the interphase due to overlapping of the adjacent restructured polymer zones. As the volume of the loosely bound polymer is lower in the interphase zone, the flexural strength of the nanocomposite with 0.3% CNT is lower than 0.2% CNT.

At 50 °C, neat epoxy and all the nanocomposites exhibit almost similar strength, suggesting a higher degree of strength degradation for the nanocomposites with increase in temperature. As a consequence of temperature increment, both CNT and epoxy try to expand as per their respective co-efficient of thermal expansion (CTE). The differential CTE between epoxy ($6.2 \times 10^{-5} \text{ K}^{-1}$ [8]) and CNT ($0.73 - 1.49 \times 10^{-5} \text{ K}^{-1}$ [9]) eventually results in development of a residual stress at the interface which favors interfacial debonding. Thus not only the stress transfer efficiency reduces, but also interfacial debonding occurs at a lower external stress. The maximum strength deterioration was noticed for 0.2% CNT/epoxy nanocomposites. The reason may be attributed to the availability

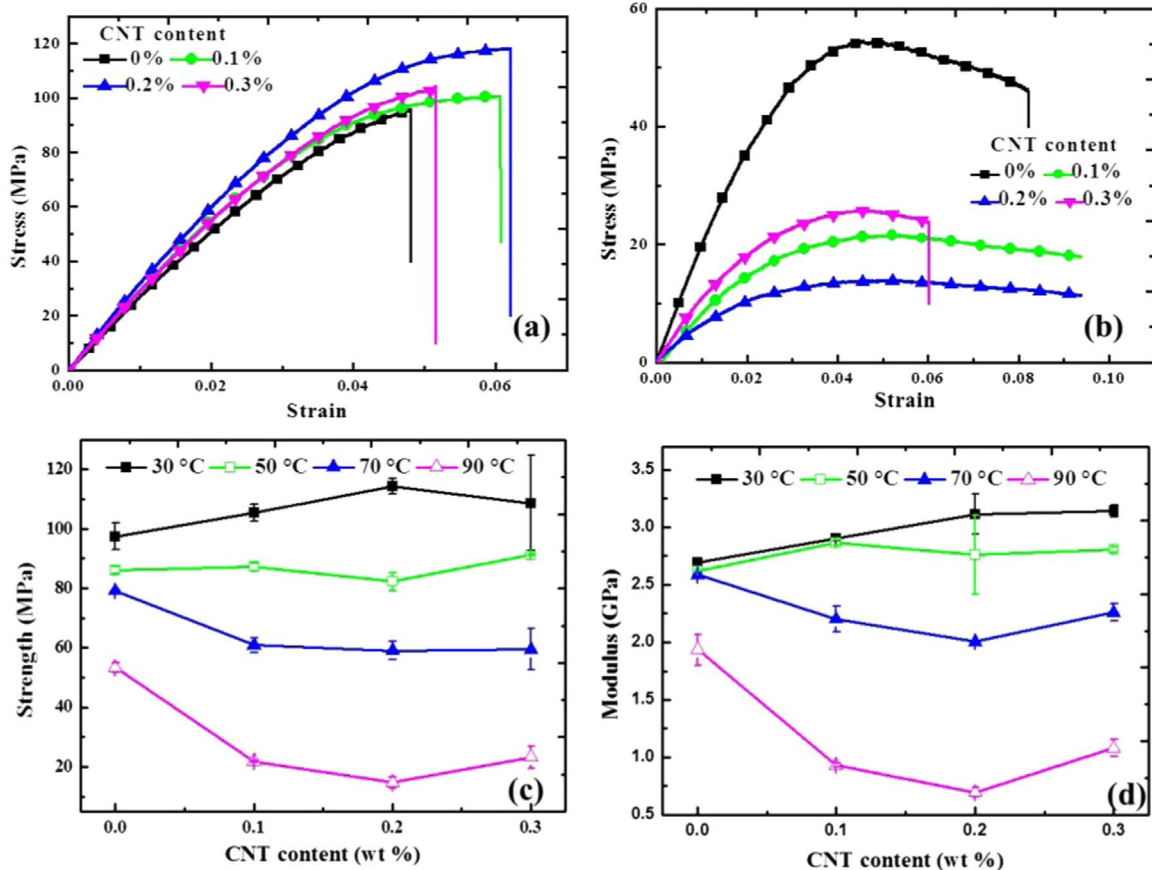


Fig. 1. Flexural stress–strain curves for nanocomposites at various MWCNT contents at (a) room temperature (30 °C), (b) 90 °C. Variation in (c) flexural strength, and (d) flexural modulus with CNT content at various in-situ temperatures.

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