

## In-situ Elevated Temperature Mechanical Performance of MWCNT/epoxy Nanocomposite

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**Abstract:** The present investigation has been focused on the effects of multi-walled carbon nanotube (MWCNT) addition on the mechanical performance of epoxy under different in-service elevated temperature environments. Room temperature flexural test results revealed that addition of 0.1 wt. % MWCNT into epoxy resin resulted in modulus and strength enhancement of 21 % and 9 % respectively. With increase in service temperature, significant decrement in both modulus and strength was noticed for both materials (neat epoxy and MWCNT/epoxy nanocomposite), but the rate of degradation was found to be quite drastic for the nanocomposite. At 90 °C temperature, the CNT/epoxy nanocomposite exhibited inferior modulus and strength, which are 41 % and 59 % lower than neat epoxy respectively. The variation trend in elastic modulus with temperature obtained from both flexural testing and DMA for both these materials was also analyzed. It was found that addition of 0.1 % CNT in the epoxy reduced the glass transition temperature by about 16°C.

**Keywords:** Multi-walled carbon nanotube (MWCNT), Nanocomposite

### 1. Introduction

Replication of the unique properties of nano-fillers in the polymer is the key target to manipulate the mechanical, thermal and/or electrical properties of polymer based materials. In the last couple of decades, many nano-sized materials have been successfully synthesized, but none of them have been globally acknowledged as much as carbon nano-tube (CNT). The motivation behind reinforcing a polymer by CNT is due to its extra-ordinary strength (as high as 22 GPa), modulus (order of 1 TPa), low density and extremely high aspect ratio (500 - 100000). The potential exploitation of CNT for achieving the desirable mechanical properties lies in:

1) Good interfacial bonding between the polymer and CNT;

2) Uniform dispersion to avail high interfacial area to facilitate effective stress transfer.

Many research articles have reported the beneficial mechanical performance of CNT reinforced polymers at room temperature [1-2]. Addition of CNT into polymeric materials has shown remarkably improved cryogenic mechanical performance [3-4]. But, to the best of authors' knowledge there is a dearth of open literature reporting the elevated temperature performance of CNT/polymer nanocomposites. "Whether the strength enhancement mechanism still remains valid at elevated temperature environment", this question has been tried to answer in the current article.

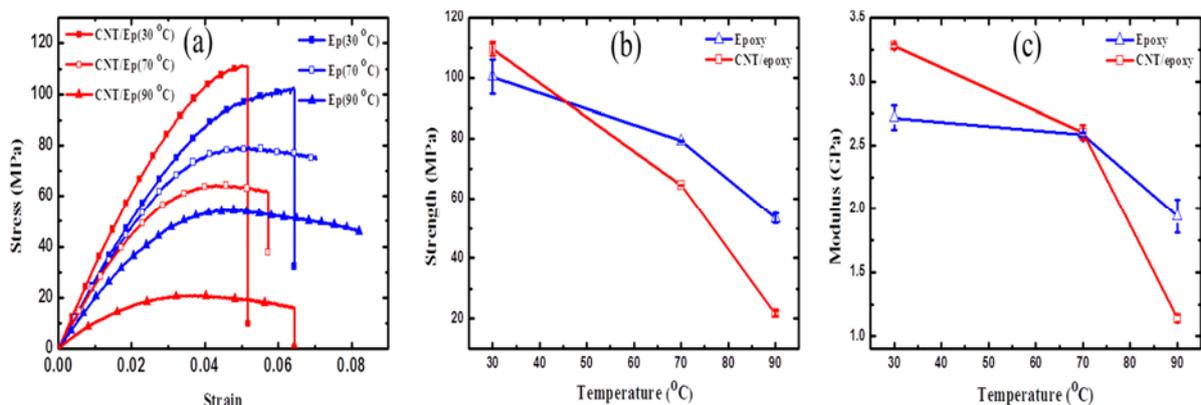
## 2. Experimental Details

The polymer used in the current study was a Diglycidyl ether of Bisphenol A type epoxy resin with Triethylene tetra amine as hardener, which were manufactured by Atul Industries, India. The MWCNT (6-9 nm outer diameter with 5  $\mu\text{m}$  length) used for reinforcing the epoxy resin was purchased from Sigma-Aldrich. 0.1 wt. % MWCNT (w.r.t. epoxy) was initially weighed and then mixed in 150 ml acetone. This acetone/MWCNT mixture was then stirred at 1000 rpm for 30 minutes followed by 30 minutes of ultra-sonication. The mixture was then transferred to a flask containing epoxy resin which was preheated at 70°C for 30 minutes. This epoxy/acetone/MWCNT mixture was then stirred at 70°C temperature and 1000 rpm till complete evaporation of acetone is ensured. The remaining epoxy/MWCNT suspension is then ultra-sonicated for 1 hour. After sonication this suspension was kept in vacuum for 18 hours. Required amount of hardener (10 wt. % of resin as specified by the manufacturer) was then added to the suspension and mixed gently using a glass rod for 2 minutes and then slowly poured into the moulds of appropriate dimensions. Neat epoxy was also poured into moulds only after mixing required amount of hardener. All the moulds were then again placed in vacuum for 10 minutes followed by room temperature curing for 24 hours. The solidified samples were then taken out from the moulds and fine polished to make all the surfaces smooth. Post-curing of all the samples were then carried out in an oven at 120 °C for 6 hours. Flexural test was then performed on neat epoxy and CNT/epoxy nanocomposites in an Instron 5967 attached with an environmental chamber at various temperatures (room temperature (30 °C), 70 °C and 90°C) at a loading speed of 1 mm/min as per ASTM D790. Thermo-mechanical properties of both epoxy and CNT/epoxy nanocomposite was evaluated by Netzsch DMA 242E in a temperature range of 40-170 °C at a heating rate of 5°C/min and 1 Hz frequency. Fractured surfaces after flexural

testing were then observed using scanning electron microscope (SEM).

## 3. Results and Discussion

The results of flexural tests are shown in Fig. 1. Results revealed that addition of 0.1 wt. % CNT in epoxy resulted 9 % and 21 % enhancement in strength and modulus, when tested at room temperature. But, it can also be seen from Fig. 1(a) that the failure strain is reduced by 20 % due to this addition of CNT into epoxy. With increase in temperature both modulus and strength of both the materials continue to decrease, but interestingly the rate of decrement is relatively higher for the nanocomposite than the neat epoxy. At 70 °C temperature, the relative decrement in strength for epoxy and CNT/epoxy nanocomposite was 21 % and 41 % respectively compared to their respective room temperature strength. Similarly, the modulus was found to be reduced by 5 % and 20 % for epoxy and CNT/epoxy nanocomposite respectively. When the test was performed at 90 °C temperature, the nanocomposite was found to exhibit strength and modulus which were 59 % and 41 % lower than neat epoxy. The reduction in strength and modulus for neat epoxy can be explained in terms of polymer softening, increased polymer chain mobility which results in lower resistance to applied load. Further, the rate of this degradation is more as the polymer approaches its glass transition temperature ( $T_g$ ). In comparison to neat epoxy, the nanocomposite exhibits fairly high rate of degradation due to the differential co-efficient of thermal expansion of CNT ( $0.73\text{--}1.49 \times 10^{-5} \text{ K}^{-1}$  [3]) and epoxy ( $6.2 \times 10^{-5} \text{ K}^{-1}$  [5]). Hence, the radial expansion of epoxy is faster than CNT which induces a residual stress at the CNT/epoxy interface. This residual stress increases with temperature and/or external stress and ultimately gets relieved by interfacial debonding which reduces the strength of the material drastically.



**Fig. 1.** Variation in flexural (a) stress-strain plots, (b) strength and (c) modulus of epoxy (Ep) and CNT/epoxy nanocomposite (CNT/Ep) at various in-situ elevated temperature.

From Fig. 2, it can be seen that the storage modulus ( $E'$ ) also shows the similar trend as that of flexural modulus with temperature. At a lower temperature the nanocomposite exhibits higher  $E'$  than the neat epoxy, but after a temperature of 90 °C, epoxy exhibits a higher  $E'$  than nanocomposite. The difference in magnitude of the elastic modulus and transition temperature obtained from flexural testing and DMA might be attributed towards their differential thermal history. The  $T_g$  (obtained from the onset of  $E'$ -temperature plot) for the nanocomposite is 16 °C lower than the neat polymer. This might be due to penetration of the finely dispersed CNTs into the inter-chain region of the resin which hinders formation of crosslinks during curing.

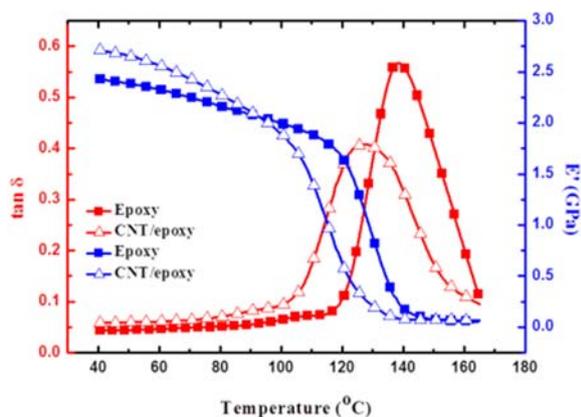


Fig. 2. Variation in thermomechanical behavior of epoxy and CNT/epoxy nanocomposites.

Mirror, mist and hackle zones [6] were observed as shown in Fig. 3 in both materials at all testing temperatures. Fig. 3(a) and Fig. 3(b) represent the hackle zones of neat epoxy and nanocomposite respectively for room temperature tested samples showing distinct appearance. The mist zone of epoxy and nanocomposite at 70 °C is shown in Fig. 3(c) and Fig. 3(d) respectively.

#### 4. Conclusions

From the present investigation it can be concluded that CNT reinforced epoxy remains a suitable candidate over neat epoxy at room temperature, but beyond a critical temperature a negative reinforcement

effect was noticed. This confirms that the positivity of the CNT/epoxy nanocomposite over neat epoxy is in-service temperature dependent.

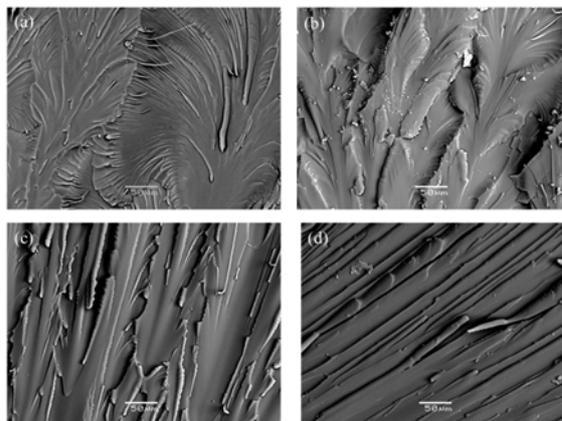


Fig. 3. SEM micro-graph of fractured surfaces of (a) epoxy and (b) CNT/epoxy nanocomposite at room temperature, (c) epoxy and (d) CNT/epoxy nanocomposite at 70 °C.

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