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Abstract

Fiber reinforced polymer (FRP) Composites are being used globally in many structural applications due to their superior specific properties. The in-plane mechanical performance of such FRP composite to a large extent is governed by the fibers. However, the out-of-plane response gets substantial contribution from the matrix and/or interfaces. In this regard, to improve the out-of-plane performance of such laminated composites, CNT incorporation has been found to be beneficial. Further, the performance of such CNT filled FRP composites in various environments is an active field of research. However, it has always been a curious concern to predict the long term behavior of such materials in real inservice applications. And, doing it under the exact conditions for such a long time period (like years) in laboratory scale is quite cumbersome. The long-term creep performance of these composites gives us an idea about the life-time of such materials in the real field of application. In this article, an attempt has been taken to predict the long-term creep performance of glass fiber reinforced polymer (GFRP) composite with and without CNT reinforcement using time temperature super-position (TTSP) principle. Step-wise (with 5°C step size) isothermal creep and recovery tests were performed using DMA (Netzsch DMA 242E) in the temperature range of 30°C - 120°C with a constant creep load (corresponding stress was 40 MPa). At each temperature both creep and recovery were performed for 1 hr each. Results indicated that Creep temperature has a decisive role to play on the interfacial stress transfer of the CNT/polymer composites and hence on the long-term durability of CNT embedded GFRP Composites.

Keywords: Carbon nanotube, FRP Composite, temperature, creep

1. Introduction

Fiber reinforced polymer (FRP) Composites are being used globally in many structural applications due to their superior specific properties. The in-plane mechanical performance of such FRP composite to a large extent is governed by the fibers. However, the out-of-plane response gets substantial contribution from the matrix and/or interfaces. In this regard, to improve the out-of-plane performance of such laminated composites, CNT incorporation has been found to be beneficial. Further, the performance of such CNT filled FRP composites in various environments is an active field of research [1-3].

However, it has always been a curious concern to predict the long term behavior of such materials in real in-service applications. And, doing it under the exact conditions for such a long time period (like years) in laboratory scale is quite cumbersome. The long-term creep performance of these composites gives us an idea about the life-time of such materials in the real field of application. In this article, an attempt has been taken to predict the long-term creep performance of glass fiber reinforced polymer (GFRP) composite with and without CNT reinforcement using time temperature super-position (TTSP) principle [4].

2. Materials and Method

Woven fabric glass fiber was used in this study, manufactured by Owens Corning, India. The polymer used in this study was a Diglycidyl ether of Bisphenol A based epoxy and the hardener was Triethylene tetra amine, both procured from Atul Industries, India. Multi-walled carbon nanotubes were procured from Sigma-Aldrich with 6-9 nm outer diameter and ~5 µm length. 0.1 wt.% CNT was first dispersed in epoxy resin using a combination of stirring and sonication as discussed in reference [5]. Both control GFRP and CNT-GFRP laminates were fabricated using 5 glass fiber layers by hand lay-up method followed by hot compression. Samples of required dimensions were then cut from the laminates and post-cured at 140 °C for 6 hrs. Step-wise (with 5°C step size) isothermal creep and recovery tests were performed using DMA (Netzsch DMA 242E) in the temperature range of 30°C - 120°C with a constant creep load (corresponding stress was 40 MPa). At each temperature both creep and recovery were performed for 1 hr each.

3. Results and Discussion

Creep compliance (C) vs. time (t) plots for both GFRP and CNT-GFRP composites has been shown in Figure 1. To obtain the master creep data at a particular reference temperature (T_{ref}) , a suitable shift factor (a_T) shall be chosen to convert the actual experimental time period (t) to a reduced time (t_r) , which is basically the expanded time at the reference temperature, using the following expression.

 $\log a_T = \log t - \log t_r \dots (1)$



Figure 1: Experimental creep compliance (C) vs. actual time (t) in the temperature range of 30° C to 120° C with a step size of 5° C.

The shift factor (a_T) for temperatures well below the glass transition temperature (T_g) may be determined by the Arrhenius relationship as given by [6],

$$\log a_T = \frac{\Delta H}{R} \left(\frac{1}{T} - \frac{1}{T_{ref}} \right) \log e \quad \dots (2)$$

 ΔH represents the activation energy for glass transition.



Figure 2: Linear fitting between $\ln f$ and $1000/T_g$. ΔH may be obtained by performing a standard DMA at different frequencies. The T_g for each frequency (*f*) may then be obtained by determining the peak position of the tan δ vs. temperature (*T*) peak. ΔH may then be determined from the slope of the linear fitting of the ln *f* as follows

$$\Delta H = -R \frac{d(\ln f)}{d(1/T_g)} \dots (3)$$

R represents the universal gas constant (8.314 \times 10⁻³ kJ K⁻¹ mol⁻¹).

The shift factor determination using Arrhenius relationship is valid well below T_g of the material, and doesn't hold good close to T_g . Hence, in the close vicinity of T_g , manual shifting has been done to obtain a continuous master creep plot. The master creep data at 2 different reference temperatures (30 °C and 70 °C) obtained using this technique is plotted

and shown Figure 3 for both GFRP and CNT-GFRP composites.



Figure 3: Master creep plots for GFRP and CNT-GFRP at (a) 30°C, and (b) 110°C reference temperatures.

It can be seen that CNT-GFRP composite has a better creep resistance (lower compliance) than that of control GFRP composite in the earlier stage for both the reference temperatures. This is due to stress transfer from the soft epoxy polymer to the hard CNT. However, it can be observed that the difference in the compliance gradually decreases with time. Interestingly, it can also be observed that there exists a transition time for the reference temperature 110°C beyond which the CNT-GFRP composite exhibits a higher compliance then control GFRP composite. The reason for this may be attributed to the development of thermal stresses at the CNT/polymer interface as temperature increased from 30°C to 110°C, which adversely affects the stress transfer efficiency of the CNT/polymer interface. The thermal tensile stress generated at elevated temperature reduces the interfacial shear strength and thus increases the possibility of interfacial slippage/pull out [7].

4. Concluding remarks

Creep temperature has a decisive role to play on the interfacial stress transfer of the CNT/polymer

composites and hence on the long-term durability of CNT embedded GFRP Composites.

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