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Effect of severely thermal shocked MWCNT enhanced glass fiber reinforced polymer composite: An emphasis on tensile and thermal responses

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Abstract. Fiber reinforced polymeric (FRP) composite materials are exposed to diverse changing environmental temperatures during their in-service period. Current investigation is aimed to investigate the influence of thermal-shock exposure on the mechanical behavior of multiwalled carbon nanotube (MWCNT) enhanced glass fiber reinforced polymeric (GFRP) composites. The samples were exposed to $+70^{\circ}$ C for 36 hrs followed by further exposure to -60° C for the similar interval of time. Tensile tests were conducted in order to evaluate the results of thermal-shock on the mechanical behavior of the neat and conditioned samples at 1 mm/min loading rate. The polymer phase i.e. epoxy was modified with various MWCNT content. The ultimate tensile strength (UTS) was raised by 15.11 % with increase in the 0.1 % MWCNT content GFRP as related to the thermal-shocked neat GFRP conditioned samples. The possible reason may be attributed to the variation in the coefficients of thermal expansion at the time of conditioning. Also, upto some extent the pre-existing residual stresses allows uniform distribution of stress and hence the reason in enhanced mechanical properties of GFRP and MWCNT filled composites. In order to access the modifications in the glass transition temperature (T_g) due to the addition of MWCNT in GFRP composite and also due to the thermal shock temperature modulated differential scanning calorimeter (TMDSC) measurements are carried out. Scanning electron microscopy(SEM) was carried out to identify different modes of failures and strengthening morphology in the composites.

1. Introduction

Fiber reinforced polymeric (FRP) composites finds a widespread usage in the structural as well as in different filed of applications. The very extensive fields where these are used are aerospace, automobile, structural, sporting goods, oil and gas pipelines, cryogenic containers. In aircrafts and space vehicles, the structural components are come in direct contact with the environment that varies with different temperatures. Hence, investigation of mechanical properties and various failure micro mechanisms of these composites are highly essential with respect to temperature variations. Nowadays different nanofillers are used in polymeric matrix to enhance the mechanical, thermal and electrical properties. Carbon nanotubes are used as nanofiller material as its possesses high modulus (around 1 TPa [1]) and strength (approximately 22 GPa [2]). Various material investigators have performed exploration with the mechanical behavior of GFRP composites with inclusion of different types of nano-fillers [3–5] at numerous environmental parameters and different nature of loadings [6-10]. The present experimental investigation describes the inclusion of various contents of MWCNTs in polymer matrix that resulted in enhancement in tensile behavior over neat GFRP composite. The polymer matrix was reformed with different contents of

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| Properties | Epoxy | Glass Fibers (Woven Fabric) |
|--|-------|-----------------------------|
| Density (g/cm) | 1.162 | 2.58 |
| Tensile Strength (GPa) | 0.11 | 3.4 |
| Tensile modulus (GPa) | 4.1 | 72.3 |
| Strain at failure (%) | 4.6 | 4.8 |
| Areal weight of fabric (g/m ²) | - | 360 |

MWCNTs i.e. 0.1 wt. %, 0.3 wt. % and 0.5 wt. % and in accordance to that different laminated composites were prepared.

Table 1. Various mechanical properties of epoxy and glass fibers.

2. Materials and Methods

2.1 Testing Materials

The present study involves preparation of neat GFRP and MWCNT filled GFRP composites. The resin used as matrix material established with the trade name as Diglycidyl Ether of Bisphenol A (DGEBA) and hardener having the trade name as triethylene tetra amine (TETA) was used. The orientation of E-glass fiber (woven fabric) utilized in the present work was 0/90° and it was procured from Owens Corning Industries. Hardener and resin (epoxy) were acquired from Atul Industries Ltd, Gujarat having the trade name of K-6 and Lapox L-12 respectively. At the time of preparation of laminates, the hardener to resin weight ratio was maintained by 1:10.

2.2 Fabrication of neat GFRP and MWCNT filled GFRP composites

The laminated composites were prepared with 9 stacks of glass fibers having 50:50 weight fractions of epoxy and glass fibers respectively. The different mechanical properties of epoxy resin and glass fibers are illustrated in table 1. Hand layup process was carried out to prepare the GFRP and MWCNT filled composite laminate. To achieve reasonable mechanical properties of the composites, it is vital to ensure proper distribution of MWCNTs in the polymer matrix. Therefore, the different weight fractions of MWCNTs were processed thoroughly in magnetic stirrer and ultra-sonicator. Hot compression press at 60°C was used for the curing of laminated composites at a pressure of 490 kPa for 20 minutes. According to ASTM D3039 the composites were cut with the help of diamond tipped cutter. Further, post curing of composites specimens was carried out in an hot chamber for 6 hours at 140°C [11].

2.3 Testing Process of laminated GFRP and MWCNT filled composite

Universal testing machine having the model number Instron 8862 was accomplished for conducting the tensile tests. The tensile tests are carried out in the room temperature for neat GFRP and MWCNTs filled GFRP composites at 1 mm/min loading rates. Glass transition behavior of the composites are examined by means of TMDSC. SEM was used for determining the several failure and strengthening mechanisms occurred in laminated composite surfaces of the tested fractured samples.

Results and discussion

3.1 *Effect of MWCNT filled GFRP composites on the tensile properties at thermal shock environmental conditions*

It is evident from figure 1 that the stress-strain behavior for neat GFRP, 0.1 wt.% MWCNT, 0.3 wt.% MWCNT and 0.5 wt.% MWCNT at 1 mm/min loading speeds. It can be seen that upto 0.1 wt.% MWCNT content tensile strength was increased (figure1) in comparison to the neat GFRP composite. Further, 0.3 and 0.5 wt. % MWCNT filled composites shows decrease in the tensile strength and the possible reason of decrement in strength may be agglomeration of MWCNTs in the matrix phase. The agglomeration of MWCNTs contributes to improper transmission of stress at the interfacial region.



Figure 1. Tensile Stress-strain plot of neat GFRP and MWCNT filled composites at thermal shocked conditioning temperature at 1 mm/min loading speed

The different tensile behavior i.e. strength and modulus acquired from figure 1 are presented along with various MWCNT content for neat GFRP and MWCNTs filled GFRP composite in figure 2. The tensile properties are strongly depending upon the MWCNT content for GFRP composites as shown in figure 2. Incorporation of 0.1 wt % MWCNT/GFRP thermal shocked composite resulted in improvement in tensile strength in comparison to neat GFRP composite as shown in figure 2(a).



Figure 2. Variation in tensile properties (a) Strength and (b) Modulus with different content of MWCNT in the composites.

An improvement of 15.11 % in tensile strength was observed in case of 0.1 wt. % MWCNT filled GFRP composite in comparison to neat GFRP. The improvement in strength may be owed to high specific surface zone of MWCNTs in the polymer matrix. This imparts higher interfacial space in the ultimate composites. Therefore, transfer of stress is easily facilitated across the interfacial region. This contributes to

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enhancement in strength of MWCNT filled GFRP composites in comparison to neat GFRP composites even after thermal shocked environment. Further addition of MWCNTs content leads to decrease in strength and modulus values. Agglomeration of the MWCNT nanoparticles in the polymer matrix was the prime cause decrease in strength value. At the fiber/matrix interface due to agglomeration of MWCNTs causes poor wettability. This leads to interfacial debonding between the MWCNT filled fiber/matrix composites. Figure 2 (a) show that the tensile strength MWCNT/GFRP composite is increasing upto 0.1 wt % MWCNT content. The modulus vs MWCNT content value was illustrated in figure 2 (b). This shows decrease in the modulus value for more than 0.1 MWCNT-GFRP composites. Improper adhesion of matrix/MWCNT/fiber in the composite and inadequate transfer of stress from polymer matrix to the fiber. the bulk images of the composites with various failure patterns tested at thermal shock environment at 1mm/min loading rate are illustrated in figure 3. The opening of cracks mainly develops at the middle portion of the samples.



Figure 3. Bulk images of failed tensile specimens i.e. GFRP and MWCNT filled GFRP composites (a) GFRP (b) 0.1 wt. % MWCNT/GFRP (c) 0.3 wt. % MWCNT/GFRP and (d) 0.5 wt. % MWCNT/GFRP

3.2 Fractography analysis

After the tensile tests, different modes of failures and strengthening morphology in the composites were analyzed with the help of nano nova FESEM.



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Figure 4. SEM micrographs of (a) Neat GFRP (b) Neat GFRP(c) 0.1 wt. % MWCNT/GFRP (d) 0.5 wt. % MWCNT/GFRP (e) 0.1 wt. % MWCNT/GFRP and (f) 0.1 wt. % MWCNT/GFRP

Figure 4(a) and 4(b) reveals SEM micrographs of neat GFRP composites showing matrix cracking and fiber pullout. From figure 4(c) 0.1 wt. % MWCNT/GFRP composite reveals better dispersion as compared to 0.5 wt.% MWCNT/GFRP composites [figure 4(d)] in polymer matrix. Better distribution of MWCNTs in the polymer matrix delivers proper transfer of stress from the polymer matrix to fiber. Agglomeration of MWCNTs at a specific area leads to improper transfer of stress across the matrix resulting in decrease in strength and modulus value. Bridging of MWCNTs in the polymer matrix certainly enhances the tensile strength value in case of 0.1 wt % MWCNT enhanced GFRP composites [figure 4(e)-4(f)].

3.3 Thermal analysis

TMDSC measurements were carried out in order to know the T_g of the polymer part of the composites material.



Figure 5. TMDSC graph of neat and MWCNT filled GFRP composites

Figure 5 illustrates the TMDSC curves for neat GFRP and MWCNTs enhanced GFRP composite at 0.1wt. % MWCNT, 0.3 wt. % MWCNT and 0.5 wt. % MWCNT. The neat GFRP composites shows T_g around 119.42 °C, while the MWCNT/GFRP with 0.1 wt. %, 0.3 wt. % and 0.5 wt. % of MWCNT reveals 100.9 °C, 94.59 °C and 88.22 °C respectively. The T_g of 0.5 wt. % MWCNT enhanced GFRP composites is found to be decreasing from 119.32 °C to 88.22 °C as compared neat GFRP composites. This decrease in T_g value may be attributed to increase of MWCNT content in the GFRP composites. The hindrance of crosslink formations due to the entrapment of MWCNTs particles between the polymeric chains of the polymeric network resulting lowering in the T_g value.

4. Conclusions

The current study involves on the tensile behavior of different contents of MWCNTs in the glass reinforced composites at thermal shocked environment and comprises the following conclusions

• MWCNT content upto 0.1% enhanced GFRP composite exhibits improvement in the tensile strength by 15.11 % as compared to the neat GFRP composite.

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- Tensile modulus was found to be maximum at 0.1 wt.% MWCNT and with increased weight percentage of MWCNT in the polymer matrix resulted in decrease in modulus value attributed to accumulation of the MWCNT at a particular region in the composites.
- The T_g of 0.5 wt. % MWCNT enhanced GFRP composites was found to be decreasing from 119.42 °C to 88.22 °C in comparison to neat GFRP composites.

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