Impact of hybrid reinforcement (nano- and macro-) over quasi-isotropic symmetrically designed GFRP composites on short beam strength properties

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Abstract. Carbon fillers like carbon nanotubes, short carbon fibers, carbon black and graphene are considered to be highly potential fillers (from macro to nanoscale) to improve mechanical characteristics of polymer matrix composites. In this work, three different samples of glass fiber reinforced composite laminates (GFRP) were prepared (1) neat GFRP, (2) MWCNT reinforced GFRP composite and (3) MWCNT with short-carbon fiber fillers (SCFF) or hybrid reinforced GFRP and tested for short beam strength (SBS). The SBS tests were performed on Hounsfield H50KS universal testing machine (UTM) at a strain rate of 0.05 mm/min. The fabrication of eight layered symmetrical glass/epoxy laminate was done by using press molding machine at 200 KN load. The results justified the significance of MWCNTs reinforcement and hybrid reinforced GFRP over neat GFRP specimen.

1. Introduction

Although polymer matrix composites have been successfully replaced conventional materials in many industries such as an automobile, aerospace, military, marine, construction, etc., their interlaminar shear strength is still considered one of the major constraints for mainstream acceptance in conventional engineering applications [1]. Thus, it becomes necessary in the composite field to enhance the interlaminar shear strength of FRP and to attain this, various approaches have been made.

The most common through-thickness reinforcement techniques are 3D weaving [2-4], braiding [3-4], stitching [5] and z- pinning. Traulis et al. [6] found that using z- pins, ILSS increased fairly. However, Abate et al. [7] have reported that these techniques are labor intensive and require additional manufacturing processes that can significantly increase the cost of resulting component. Many researchers have explored other ways to improve ILSS by using nano, micro and macro fillers such as carbon nanotubes, graphene, micro Al₂O₃, SiO₂, Ti₂O₃, short carbon fibers etc. Ramesh et al. [8] showed that addition of microparticles like Al₂O₃, SiO₂, Ti₂O₃ improve the ILSS and flexural strength. Carbon nanotubes as a filler have a high potential to improve the mechanical properties of FRP. The properties offered by CNTs are high stiffness (500GPa – 1000GPa) and strength (50GPa – 100GPa), diameter dependent specific surface area (SSA) of up to 1300m²/gm as well as aspect ratio in the range of several thousand, high thermal conductivity and electrical conductivity [9]. Zhou et al. [10] found that by the addition of 2wt% CNT into GFRP, ILSS increased by 22.3%. Gojny et al. [11] in 2005 improved the ILSS by addition of 0.3wt% DWCNTs. Z Fan et al. [12] concluded that by adding up to
2wt% of OWCNT in GFRP laminate, 33% improvement in ILSS is possible. Macro modifiers like short carbon fiber fillers (SCFF) has also been considered as economical fillers to enhance ILSS of GFRP. K.K. Singh et al. [13] showed that ILSS of GFRP doped with short carbon fiber fillers had increased 5% to 10% significantly.

However, comparative study on mechanical properties with nano and macro fillers together have not been studied much for GFRP composite. In this paper, MWCNTs and short carbon fiber fillers (SCFF) are used as secondary reinforcement in order to enhance short beam strength. The hand layup technique is used with the assistance of hydraulic press molding for curing the composite laminates.

2. Experiments

2.1. Materials

Plain woven glass fiber fabrics with a nominal weight of 600GSM, manufactured by Vetrotex India Limited and supplied by M.S. Industries, Kolkata were used as primary reinforcement. The MWCNTs used were purchased from United Nanotech Innovations Pvt. Limited, Bangalore, India and having a length of 1-10 microns, thickness of 5-20 nm with 98% purity. The carbon fibers were chopped in the range of 1-5mm in length. Bisphenol-A based thermosetting epoxy, brand name Lapox L-12 (ARL-12) was used as matrix along with N,N’- Bis (2-amino ethyl) ethane -1,2- diamine hardener, brand name Lapox K-6 (AN312).

2.2. Specimen preparation

The glass woven was cut in two orientations, i.e. (0/90) and (+45/-45) as shown in figure 1a. The hand layup technique assisted by the hydraulic press, has been used to prepare symmetrically designed quasi-isotropic GFRP laminates (figure 1b) with stacking sequence [(0,90)/(+45,-45)/(+45,-45)/(0,90) // (0,90)/(+45,-45)/(+45,-45)/(0,90)].

For laminate without any secondary reinforcements (neat GFRP), the epoxy was mixed with hardener in the ratio of 10:1 (as suggested by the supplier) but for laminates with MWCNTs, weighed amount of MWCNTs was mixed in epoxy using ultra sonication bath for 30 minutes followed by mixing of hardener. Further, the solution was stirred for 15 minutes using high-speed stirrer at 1200 rpm. To prepare composite laminates, the woven glass sheet was kept on flat surface and resin was applied to the sheet using a brush. In the next step, second fiber layer was kept on the first one and brushing action was repeated. In order to remove excess resin, a heavy iron roller was rolled over wet woven sheets. This process is repeated up to eight layers of proposed design laminate. Finally, to remove the maximum amount of resin and to cure the laminate, the wet laminate was placed in hydraulic press molding machine (figure 1(c)) at 200 KN load. Moreover, to prepare hybrid GFRP reinforced with MWCNTS and SCFF, SCFF is divided into seven equal weights and added manually and randomly between each layer.

Thus, three composite laminates were prepared as (1) pristine GFRP, (2) GFRP doped with 1wt% MWCNTs and (3) hybrid nano- and macro- modified GFRP doped with 0.5% MWCNTs and 0.5% SCFF. Four specimens were cut from each composite laminate using a diamond cutter with dimensions as per ASTM D2344.
2.3. Testing procedure

The ILSS or short beam strength test was conducted on fully computerized Hounsfield machine, with a maximum load carrying capacity of 50KN and loading rate varied from 0.01mm/min to 50mm/min. The proposed tests were conducted on loading rate of 0.05mm/min. Also, the interlaminar shear strength of the symmetrical laminate is calculated using the formula in equation (1) as per ASTM D2344 [14] as shown in figure 2. The mean, standard deviation, and coefficient of variance of measured values are given by formula (2), (3) and (4) respectively.

\[
\frac{ILSS/F_{sbs}}{F_{sbs}} = 0.75 \frac{P_m}{b \times h} 
\]

\[
\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n} 
\]

\[
S_{n-1} = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - n(\bar{x})^2}{n-1}} 
\]

\[
CV = 100 \times \frac{S_{n-1}}{\bar{x}} 
\]

Where,

- \(ILSS/F_{sbs}\) = Inter laminar Shear strength or short-beam strength, MPa
- \(P_m\) = Maximum load observing during test, N
- \(b\) = Measured specimen breadth, mm
- \(CV\) = Coefficient of variation, %
- \(d\) = Specimen thickness, mm
- \(n\) = Number of specimens
- \(\bar{x}\) = Sample mean (average)
- \(S_{n-1}\) = Sample standard deviation
- \(x_i\) = Measured property
3. Result and discussion
The values of maximum force and ILSS or short beam strength along with standard deviation and coefficient of variation of various samples have been represented in table 1. The plot showing load versus deflection of tested specimens are shown in figure 3.

Figure 2. Fixture setup for ILSS as per ASTM D2344

Figure 3. Load displacement curves for (a) Neat GFRP, (b) 1 wt% MWCNT doped GFRP and (c) 0.5 wt% MWCNT + 0.5 wt% SCFF doped GFRP
Table 1. Values for ILSS (MPa) and maximum force

| Specimen | \( P_m \) (N) | \( \text{ILSS/} F_{\text{sbs}} \) (MPa) | Specimen | \( P_m \) (N) | \( \text{ILSS/} F_{\text{sbs}} \) (MPa) | Specimen | \( P_m \) (N) | \( \text{ILSS/} F_{\text{sbs}} \) (MPa) |
|----------|----------------|----------------|----------|----------------|----------------|----------|----------------|----------------|
| 1        | 1061.55        | 24.88          | 1        | 1179.30        | 27.64          | 1        | 1293.65        | 30.32          |
| 2        | 862.293        | 20.21          | 2        | 1249.71        | 29.29          | 2        | 1226.24        | 28.74          |
| 3        | 938.24         | 21.99          | 3        | 1329.49        | 31.16          | 3        | 1089.28        | 25.53          |
| 4        | 982.893        | 23.06          | 4        | 1366.61        | 32.03          | 4        | 1259.52        | 29.52          |
| Mean (\( \bar{x} \)) | 961.494 | 22.54 | Mean (\( \bar{x} \)) | 1281.28 | 30.03 | Mean (\( \bar{x} \)) | 1217.17 | 28.53 |
| \( SD \) \( (S_{n-1}) \) | 83.46 | 1.954 | \( SD \) \( (S_{n-1}) \) | 83.46 | 1.96 | \( SD \) \( (S_{n-1}) \) | 89.64 | 2.098 |
| CV (%)    | 8.68 | 8.67    | CV (%)    | 6.53    | 6.53    | CV (%)    | 7.36    | 7.36    |

A comparative study has been performed, and it was analyzed that ILSS of GFRP doping with 1% MWCNTs was 30.03 ± 1.96 MPa and was maximum with the increment of 33.26% when compared to neat GFRP which has ILSS value of 22.54 ± 1.954 MPa. It can be attributed to the fact that addition of MWCNTs causes an increase in ILSS due to toughening mechanism imparted by the nanoparticles to the matrix. Also, crack bridging effect of these particles causes resistance to crack propagation [15]. Nanomechanical interface between MWCNTs and matrix causes better interface between MWCNTs and matrix and better load transfer from matrix to MWCNTs [16]. The ILSS of hybrid MWCNTs/SCFF modified laminate was 28.53 ± 2.098 MPa and 26.54% higher than neat GFRP. Both MWCNTs and SCFF increase the strength of epoxy matrix as they both are very stiffer as compared to epoxy. The load transfer from matrix to macro and nanofillers increases SBS as it is a matrix dominant property.

Figure 4. Comparative values for ILSS (in MPa) with error bars
Comparing the ILSS of hybrid MWCNTs/SCFF modified composite with MWCNTs modified laminate, it was found that ILSS of MWCNTS modified laminate was only 5.27% higher than that of hybrid MWCNTs/SCFF modified laminate. It is because the effective load transfer from matrix to nanofillers happened only when there is a good dispersion of MWCNTs in a matrix [17] which may not be so in case of 1% MWCNTs/epoxy GFRP (agglomeration occurred). Whereas laminate with 0.5% MWCNTs and 0.5% SCFF, the proper dispersion may be there as compared to the sample with 1% MWCNTs.

4. Conclusions
This paper investigated the significance of hybrid reinforcement over MWCNTs doped GFRP laminates. The findings of experimental results can be concluded as
1. Multi walled carbon nanotubes and hybrid reinforcement have positive influence on interlaminar shear properties.
2. MWCNTs enhanced short beam strength by 33.261% and hybrid reinforcement improved SBS properties by 26.54% as compared to neat GFRP composite.
3. Hybrid doping ensured the reduction in the use of MWCNTs doping up to 50% by weight. Therefore, hybrid mixing method of strengthening composites material is economical over GFRP doped with MWCNTs only as secondary reinforcements.

References
[1] Rosselli F and Santare MH 1997 Compos Pt A: Appl Sci Manuf 28(6) 586-94.
[2] Chou T-W, Ko F 1989 Three-dimensional fabrics for composites (Elsevier Science publishers).
[3] Mouritz AP, Bannister MK, Falzon PJ and Leong KH 1999 Review composites A 30 1445-61.
[4] Toug L, Mouritz AP and Bannister MK 2002 3D fiber reinforced polymer composites Elsevier London.
[5] Dransfield K, Baillie C and Mai Y-W 1994 Compos Sci techno 50 305-17.
[6] Traulis M, Cartie DDR, Bartattoni L and Partridge IK 2001 In: Proceeding of the 6th international conference on deformation & fracture of composites Manchester, UK.
[7] Abali, F, Pora, A and Shivakumar K 2003 Journal Composite Materials 37 453–64.
[8] Nayak R K, Dash A and Ray B C 2014 Procedia Material Science 6 1359-64.
[9] Thostenson E T, Ren Z F and Chou T W 2001 Comp Sci Technol 61(13) 1899-12.
[10] Zhou Y, Pervin F, Rangari VK, Jeelani S 2006 Mater Sci Eng A 426 221-8.
[11] Wichmann M H G, Fiedler B, Bauhofer W and Schulte K 2005 Composite Part A: Applied Science and Manufacturing 36(11) 1525-35
[12] Fan Z, Santare M H and Advani S G 2008 Composites: Part A 39 540–54.
[13] Singh K K, Rawat P and Rai A K 2016 ARPN journal of Engineering and Applied Sciences 11.
[14] D2344/D2344M–13. Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates.
[15] Iwahori Y, Ishiwata S, Sumizawa T and Ishikawa T 2005 Comp. Part: A 36 1430.
[16] X Xu, M M Thwe 2002 Appl. Phy. Lett. 81 2833.
[17] Gojny F H, Malte H G Wichmann, Fiedler B and Schulte K 2005 Composites Science and Technology 65 2300-13.