Combined effect of loading rate and percentage by weight of MWCNTs on inter laminar shear strength (ILSS) and flexural strength of CFRP

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Abstract. The mechanical performances of fiber reinforced polymer (FRP) composites depend upon the factors like types of reinforcement added and loading rate to which it is subjected. The present paper investigates the influence of multi-walled carbon nanotubes (MWCNTs) doping and strain rate on the properties like inter laminar shear strength (ILSS) and flexural strength of composites. Two samples i.e., neat carbon/epoxy composites and 0.25 wt. % of MWCNTs reinforced CFRP were prepared and tested. The specimens were tested on Hounsfild H50KS, universal testing machine (UTM). The SBS and three-point bend tests were done as per ASTM D 2344 and ASTM D 7264 standards. The hand lay-up technique assisted by vacuum bagging was used to fabricate the symmetrically designed CFRP laminates with eight layers of 0.5 mm thickness each. The prepared samples were tested at the three different crosshead speeds of 1, 100 and 250 mm/min. The results obtained in this paper indicate that the carbon/epoxy composites doped with MWCNTs showed higher strength as compared to neat CFRP at the corresponding loading rates. Also, it was detected that there was the existence of a critical loading rate at which the mechanical properties, ILSS and flexural strength was enhanced up to its maximum limit. Moreover, the FESEM images of fractured samples were taken to observe the inter laminar shear and bending failure.

1. Introduction

Low specific weight, high specific rigidity, and high specific strength are special properties of carbon/epoxy composites, due to which it got significant applications in aircraft structures, marine structures, automotive, etc. [1]. However, CFRP is rather susceptible to delamination (inter-laminar failure) which may lead to loss of stiffness and reliability of structures [2]. Failure due to delamination can be resisted by increasing the inter-laminar shear strength of carbon/epoxy laminates. Because of its simplicity and straightforward data analysis, short beam strength (SBS) test is used to measure the inter-laminar shear strength (ILSS) of FRP composites [3]. Three-point bending test is used to find the flexural strength of CFRP composites. It was found that the out of plane strength calculated by three-point bending test depends upon span length to thickness ratio of specimen [14].

The use of carbon nanotubes (CNTs) reduces the brittle nature of epoxy, which is commonly used as matrix material [4]. The addition of small quantity of CNTs into the matrix or fiber sizing increases the bending and inter-laminar shear strength of fiber-reinforced polymer [5]. Wang et al. [17]
concluded that, when the mass fraction of MWCNTs was varied from 0 to 2%, there was reinforcing effect of MWCNTs on the mechanical properties of carbon fiber non-crimp fabric. They also determined that, with 2% and 0.5% mass fraction of MWCNTs in carbon fiber non-crimp fiber, the flexural strength and ILSS was enhanced by 26% and 14% respectively. Zhou et al. [7] found that the inter laminar shear strength was improved by 22.3% with 2 wt.% of carbon nano-fibers in the matrix.

Some studies have been carried out to see the influence of loading rate on ILSS and bending strength of composites, but there is need to explore much more in this field. Bouette et al. [8] proposed a way to evaluate the ultimate ILSS and inter laminar shear modulus of unidirectional CFRP as functions of strain rate. Ray [10] found the change in ILSS, at a constant temperature, for GFRP and glass/polyester, by varying the number of humidity shock cycles for two different loading rates of 2 and 50 mm/min. Dong et al. [11] designed single-lap and double-lap shear specimens for comparing the ILSS of carbon/epoxy composites and observed that the value obtained by single-lap shear specimen was about 50% higher than the double-lap shear specimen. Sethi et al. [6] detected that the maximum increase in the ILSS of both carbon/epoxy and graphite/epoxy composites was about 85.72% at ambient temperature using 1mm/min loading rate. They also observed that the ILSS value of carbon/epoxy composites was decreased when loading rate was exceeded beyond 500 mm/min. Naik et al. [9] found that the ILSS for the plain weave E-GFRP and plain weave CFRP increases by 59% and 67% respectively at the strain rate of 1000/s. Miyano et al. [18] studied the variation in flexural strength at four different strain rates of 0.05, 0.5, 5 and 50 mm/min and at seven temperatures of 60, 80, 100, 120, 140, 160 and 180°C.

This paper aims to examine the sensitivity of loading rate with no doping and with doping of 0.25% by weight of MWCNTs on the mechanical properties like ILSS and bending strength of carbon/epoxy composites.

2. Material fabrication
Symmetric CFRP laminate of eight layers was prepared with the help of bi-directional woven carbon fiber by stacking it into different orientations of 0°/90° and 45°/-45°. The carbon/epoxy laminate stacking sequence and its thickness are shown below in figure 1.

![Figure 1. Symmetric CFRP laminate consisting of eight layers with given fiber orientation and thickness.](image-url)
The MWCNTs were added in epoxy (bisphenol-A) and were properly mixed using ultrasonic bath for one and a half hour. Thereafter, the hardener K6 was added and the solution was further sonicated for 10 minutes. The mixing ratio of hardener to epoxy was kept at 1:10.

Table 1. Composition of carbon/epoxy laminate and resin

| CFRP       | Resin                                                                 |
|------------|----------------------------------------------------------------------|
| Carbon fiber : 800 gsm | Hardener : K6 Triethylenetetramine (TETA)                            |
| Matrix : Epoxy resin          | Epoxy : Diglycidyl ether of bisphenol-A (DGEBA)                       |

By keeping the first carbon fiber woven sheet on a plane glass surface and applying adhesive resins on it with the help of a brush, the preparation of carbon/epoxy laminate was initiated. Thereafter, the second carbon fiber layer was kept over first carbon layer and brushing was applied in a similar manner on it with adhesive resins. The extra resins present between the two carbons layers was squeezed by rolling a heavy iron roller on the carbon fiber woven sheets. The same process was repeated for the preparation of eight layers of symmetric CFRP laminate as shown above in figure 1. The prepared carbon/epoxy laminate consisting of eight layers was put inside the vacuum bag set up as shown in figure 2 at 680 mm of Hg for 1 hour so that the maximum amount of resins can be squeezed out. Then, for next 24 hours, the CFRP laminate was subjected to heavy load and thereafter curing was done in normal atmospheric conditions.

For conducting both the SBS and three-point bend tests, samples of different dimensions were cut as shown in figure 3. For each case of neat carbon/epoxy and doped CFRP laminate with 0.25 wt. % of MWCNTs, twelve samples were cut, six for the SBS test and the other six for the flexural test.
3. Testing Methods

Tests were performed using Hounsfield machine which has maximum load carrying capacity of 50 KN. Three different loading rates of 1mm/min, 100mm/min, and 250 mm/min were used to see its effect on ILSS and flexural strength of FRP composites. According to ASTM D2344 and ASTM D7264 standards, the dimension of specimens for short beam shear strength and bending tests were cut using the diamond cutter. The machine shown below in figure 4 is fully computerized UTM machine on which the two tests were conducted.

Figure 4: Fully computerized Hounsfield Universal Testing Machine (UTM)

Short beam strength (SBS) test:
The SBS test was conducted according to ASTM D2344. The test specimen dimensions and the fixture setup are shown in figure 5. For calculating the ILSS, the following expression was used as given in equation 1:

\[ ILSS = \frac{0.75F}{bh} \]  

(1)

Where,  
F = maximum load (N) observed during the test,  
b = specimen width (mm) and  
h= specimen thickness (mm).  

Dimensions of specimen:
The thickness of specimen = 4mm

Figure 3: Dimensions of the (a) 3-point bend test specimen and (b) short beam strength (SBS) test specimen
Length of specimen = 6 x thickness = 24mm
Width of specimen = 2 x thickness = 8mm

Figure 5: Schematic representation of short beam strength (SBS) test according to ASTM D2344

Three point bending test:
3-point bending test was conducted using the ASTM D7264 standard. The test specimen and fixture used for the test is shown in figure 6. Maximum bending strength was calculated for each MWCNTs-embedded CFRP and neat laminate using the equation 2.

\[ \sigma = \frac{3PL}{2bh^2} \]  

(2)

Where,
b = beam width (mm)
h = beam thickness (mm)
L = support span length (mm)
P = applied force (N)
\( \sigma \) = stress in MPa at the outer surface of mid-span.

Figure 6. Schematic representation of the 3-point bend test according to ASTM D7264

Dimensions of specimen:
Length of specimen = 96 mm
Beam width = 13mm
Beam thickness = 4mm.
4. Result and discussion

Short beam strength (SBS) test is used to measure the ILSS of FRP with or without MWCNTs. In SBS test, it is not necessary that the specimen will fail by pure shear [12, 13 and 15]. The shear failure of CFRP laminate observed in this test was indeed the failure of the matrix by shear. Comparative calculations were done for each sample by altering the loading rates. For the SBS test, there was an increase in the value of ILSS of the neat carbon/epoxy samples by 39.976 % when loading rate was changed from 1 mm/min to 100 mm/min, but when the loading rate was altered from 100 mm/min to 250 mm/min, the ILSS decreased by 27.109 %. In the SBS test of 0.25 wt. % MWCNTs doped CFRP, it was seen that the ILSS enhanced by 28.564 % and dropped by 19.075 % due to change in loading rate from 1mm/min to 100mm/min and 100 mm/min to 250 mm/min respectively.

Three-point bend test is used to measure the bending strength of FRP with or without MWCNTs. The breakage of the fiber was responsible for the CFRP laminate failure [16]. Maximum bending strength was calculated for each sample by altering the loading rates. It was observed that in flexural test of the neat CFRP specimens, with change in loading rate from 1 mm/min to 100 mm/min, the bending strength was increased by 22.533 % but decreased by 30.332 % when the loading rate was increased from 100 mm/min to 250 mm/min. Similar pattern of the graphs were observed for the 0.25 wt. % MWCNTs embedded carbon/epoxy laminates in the bending test, there was increment and decrement in flexural strength by 29.984 % and 18.153 % when loading rate was changed from 1 mm/min to 100 mm/min and 100 mm/min to 250 mm/min respectively.

![Figure 7. Force vs. extension graph obtained during the SBS test of neat CFRP sample with loading rates of 1mm/min, 100mm/min and 250 mm/min.](image-url)
Figure 8. Force vs. extension graph obtained in the SBS test of 0.25 wt. % MWCNTs carbon/epoxy composite samples with loading rates of 1mm/min, 100mm/min and 250 mm/min.

Figure 9. Load vs extension graph of neat CFRP samples in the 3-point bend test with loading rates of 1mm/min, 100mm/min and 250mm/min.
Figure 10. Load vs. extension graph of 0.25 wt. % MWCNTs doped carbon/epoxy composites in the three-point bend test with loading rates of 1mm/min, 100mm/min and 250 mm/min.

Table 2. Influence of loading rate on ILSS of neat and 0.25 wt.% MWCNTs doped CFRP in SBS test.

| Loading rate (mm/min) | % increase or decrease in ILSS of neat CFRP | % increase or decrease of 0.25 wt. % MWCNTs doped CFRP |
|-----------------------|--------------------------------------------|---------------------------------------------------------|
| 1 to 100              | 39.976 % increase                          | 28.564 % increase                                       |
| 100 to 250            | 27.109 % decrease                          | 19.075 % decrease                                       |

Table 3. Effect of loading rate on the flexural strength of neat and 0.25 wt. % MWCNTs doped carbon/epoxy composites in 3-point bend test.

| Loading rate (mm/min) | % increase or decrease in bending strength of neat carbon/epoxy composites | % increase or decrease in flexural strength of 0.25 wt.% MWCNTs embedded carbon/epoxy composites |
|-----------------------|---------------------------------------------------------------------------|---------------------------------------------------------------------------------------------|
| 1 to 100              | 22.533 % increase                                                         | 29.984 % increase                                                                         |
| 100 to 250            | 30.332 % decrease                                                         | 18.153 % decrease                                                                         |

Figure 11. Carbon/epoxy laminates after failure in (a) SBS and (b) 3-point bending test
Table 4. Maximum load (N) at which failure of specimens took place in SBS test.

| Loading rate (mm/min) | % by weight of MWCNTs in CFRP | Maximum load (N) at which failure is observed |
|-----------------------|------------------------------|---------------------------------------------|
| 1                     | 0                            | 288.729                                     |
| 100                   | 0                            | 481.026                                     |
| 250                   | 0                            | 350.622                                     |
| 1                     | 0.25                         | 663.961                                     |
| 100                   | 0.25                         | 929.456                                     |
| 250                   | 0.25                         | 752.164                                     |

Figure 12. Graph representing the ability to resist maximum load of MWCNTs doped CFRP compared to neat carbon/epoxy composites at loading rates of 1, 100 and 250 mm/min in SBS test.

Table 5. Maximum load (N) detected in flexural test of each CFRP specimen.

| Loading rate (mm/min) | % by weight of MWCNTs in carbon/epoxy composites | Maximum load (N) at which failure takes place |
|-----------------------|-------------------------------------------------|---------------------------------------------|
| 1                     | 0                                               | 378.073                                     |
| 100                   | 0                                               | 488.045                                     |
| 250                   | 0                                               | 340.012                                     |
| 1                     | 0.25                                            | 403.279                                     |
| 100                   | 0.25                                            | 575.98                                      |
| 250                   | 0.25                                            | 471.423                                     |
Figure 13. Graph representing the ability to resist maximum load of MWCNTs embedded CFRP compared to neat carbon/epoxy composites at loading rates of 1, 100 and 250 mm/min in flexural test.

![Graph](image1)

Figure 14. FESEM images showing (a) inter laminar shear and (b) bending failure of CFRP composites.

![FESEM Images](image2)

5. Conclusions:
From the experimental study, following results can be concluded:
1. The mechanical properties, ILSS and bending strength of FRP laminates were improved by doping MWCNTs in the carbon/epoxy composites.
2. There exist a critical loading rate at which maximum mechanical performance of FRP composites can be achieved for both the SBS and flexural tests.
3. The ILSS and bending strength of FRP laminates are more sensitive at low and moderate loading rates and this sensitiveness decreases at higher loading rates.
For the SBS test, with or without the embedment of MWCNTs, the ILSS increased when loading rate was altered from 1mm/min to 100mm/min, but the same dropped when the loading rate was changed from 100mm/min to 250 mm/min.

In the 3-point bending test, the flexural strength was improved with change in loading rate from 1mm/min to 100mm/min, but there was decrease in its value when loading rate was increased from 100mm/min to 250mm/min.

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