

Effect of loading rate on inter laminar shear strength (ILSS) of highly doped MWCNTs carbon/epoxy laminates

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Abstract. The present experiment investigates the sensitivity of strain rate on mechanical properties of MWCNTs (multi-walled carbon nanotubes) reinforced carbon woven composites. The SBS (short beam strength) tests were done to evaluate the dependence of strain rate on ILSS (inter laminar shear strength) of pristine neat and 1, 2, 3 and 4 wt. % MWCNTs doped CFRP (carbon fiber reinforced polymer). The symmetrical bi-directional carbon fiber laminate comprising of eight layers with 0.5 mm thickness each and ply orientation of [(0, 90) / (+45, -45) / (+45, -45) / (0, 90)]_s was designed. The laminates were prepared by hand lay-up method assisted by vacuum bagging technique. The prepared laminates were cut using the diamond cutter and the obtained samples were tested on H50KS, the UTM (universal testing machine) as per ASTM D2344. Three different loading rates of 1, 110 and 220 mm/min were used to see the mechanical performance of CFRP with no doping and with doping of higher percentage by weight of MWCNTs. The results revealed strain rate dependency of carbon/epoxy laminates on its mechanical performance. In the test, 3 wt. % MWCNTs doped CFRP showed maximum shear strength at the strain rate of 110 mm/min. Also, FEA (finite element analysis) using ANSYS were done and the results were found to be in good agreement with the experimental data.

1. Introduction

Fiber reinforced polymer (FRP) composites due to their higher strength to weight ratio, higher stiffness, better shock resistance fatigue resistance, low density, high strength and heat resistance are used in aerospace, automotive and defence services. FRP composites are subjected to different loading rates during their whole service life. For the authentic design of composite structures, it is very necessary to analyse the behaviour of FRP composites under different (low, moderate and high) strain rates. It is well known fact that as the rate of loading rate is amplified, there is a lesser amount of time for the crack to develop, consequently the sum of accumulated damage at a given strain-level decrease and the material can resist higher load and failure strain.

Yokoyama et al. [1] determined the interlaminar and in-plane shear strengths of unidirectional CFRP laminates and found that with increasing deformation rate both the shear strengths were enhanced to some extent. Kumar et al. [2] worked on the woven and chopped E-glass fiber at ambient and cryogenic conditions with varying loading rates from 2 mm/min and 500 mm/min and concluded that



higher values of ILSS at cryogenic conditions were obtained compared to atmospheric conditions. Harding et al. [3] investigated on the interlaminar shear strength of two plain-weave glass/epoxy plies, two plain-weave carbon/epoxy plies and a plain-weave glass/epoxy and a plain-weave carbon/epoxy ply and found that the change of loading rate from quasi-static to impact leads to the increment of interlaminar shear strength obtained at failure of the sample. Hallet et al. [12] designed the single lap shear specimen for considering the dependency of strain rate on the ILSS of cross-ply carbon epoxy (T300/914) and found that at impact strain rate there is small increment in both the ILSS and strain rate but decrement in the value of through thickness shear modulus was observed.

Li et al. [4] found that the tensile strength of carbon/epoxy composites enhanced with increasing strain rate for both types, whether woven fabric or unidirectional composites. For quasi-static loading, the strain rate was kept at 0.5 s^{-1} whereas for dynamic testing, the strain rate was varied from 200 s^{-1} to 2300 s^{-1} . Naresh et al. [6] studied the strain rate dependency of GFRP, CFRP and hybrid composites and varied the strain rate from 0.0016 s^{-1} to 542 s^{-1} . The results indicate that the tensile strength and tensile modulus of GFRP and hybrid composites increase and percentage of failure strain for GFRP, CFRP and Hybrid composites decreases with the increase in strain rate, whereas tensile strength and tensile modulus of CFRP remains approximately constant. Shokrieh et al. [8] found that the shear properties of bi-directional woven composites are more rate-dependant than their tensile behaviour. Higher maximum stress and initial undamaged tension and shear modulus were observed in dynamic tests compared to quasi-static ones. Naik et al. [7] found that the ILSS for the plain weave E-graphite/epoxy laminate and plain weave carbon/epoxy laminate increases by 59% and 67% respectively at the strain rate of 1000 s^{-1} .

Rawat et al. [5] studied the variation in mechanical performance by varying CNT (SWCNTs or MWCNTs) percentage in composite laminates and concluded variation of impact resistance with CNTs variation; also they observed that the optimized doping of MWCNTs provides better impact properties and the damage propagation is also affected by the size of CNTs used. Zhou et al. [16] studied the effect of CNTs distribution on inter-laminar shear strength and found that the fiber-matrix interface bonding was enhanced by adding CNTs into the fiber-sizing, the matrix was kept CNT free and the ILSS was found to be increased by 36-53 %. Also, they found that if CNTs was added in the matrix, it will increase the bonding capacity between fiber sizing and matrix, the material toughness and the ability to resist crack propagation due to delamination and the ILSS was increased by 77%. Singh et al. [11] worked on the effect of doping MWCNTs on low velocity impact response of quasi-isotropic asymmetric laminate of woven carbon/epoxy laminate and concluded that on doping of 2 wt. % and 5 % MWCNTs absorbed impact energy increases by 13.53% and decreases by 10.49% respectively. Boddu et al. [9] investigated the mechanical properties of an E-glass fabric composite doped with anchored MWCNTs. The interlaminar shear strength was decreased by 25.9% with composites doped with MWCNTs, it was also found that the specific energy absorption was enhanced by 106% at high strain rates and there was an increase in energy density dissipation by 64.3% after 5 cycles at quasistatic strain rates. Soliman et al. [10] investigated the role of MWCNTs on the tension (on axis tension test) and in-plane shear (off-axis tension test) strength of CFRP composites. The pristine and 0.1, 0.5, 1.0, and 1.5 wt. % MWCNTs doped CFRP were used for this test. From the work it was concluded that with 1.5 wt. % doped MWCNTs, toughness, ultimate strength and failure strain of the off-axis tension test was enhanced by 39%, 51%, and 121%, respectively. Rawat et al. [13] found that the ILSS and flexural properties of FRP laminates can be improved by adding MWCNTs because it reduces the brittle nature of epoxy. Tehrani et al. [14] concluded that the addition of MWCNTs to the matrix of carbon/epoxy laminate results in the improvement of inter as well as intra-laminar strength which leads in the improvement of impact resistance of FRP laminates. Godara et al. [15] noticed a substantial increase in mode-I fracture toughness by over 80% for the pristine MWCNTs, also other mechanical properties were enhanced.

Therefore, this paper aims to examine the sensitivity of loading rate with no doping and with doping of 0, 1, 2, 3 and 4 % by weight of MWCNTs on the mechanical properties like ILSS of carbon/epoxy composites.

2. Material fabrication

The CFRP laminate comprising of eight plies was symmetrically designed with the help of hand layup technique. The ply orientation of the laminate was $[(0, 90) / (+45, -45) / (+45, -45) / (0, 90)]_s$ and the thickness of each lamina was 0.5 mm.

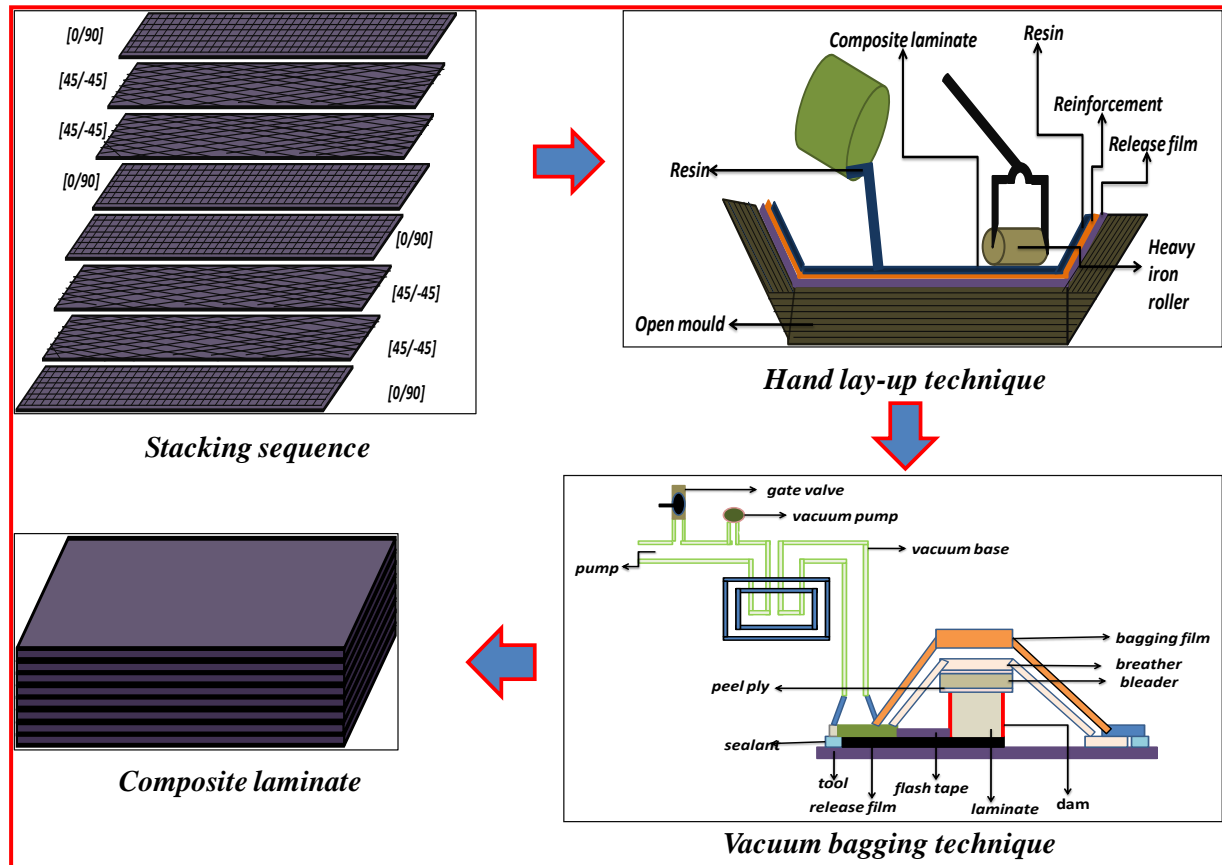


Fig. 1 Symmetric CFRP eight layered laminate preparation sequence

In epoxy (bisphenol-A), the MWCNTs were added and properly mixed using the ultrasonic bath for two hours. After that, hardener K6 was added and for 20 minutes, the solution was further sonicated. The ratio in which hardener and epoxy were mixed was 1:10.

Table 1. Description of resin and carbon/epoxy laminate

CFRP	Resin
Carbon fiber :600 gsm	Hardener : K6 Triethylenetetramine
Matrix : Epoxy resin	Epoxy :Diglycidyl ether of bisphenol-A

The first carbon fiber woven sheet was placed over a plane glass surface, then adhesive resins were applied on it with the help of a brush. Thereafter, the second carbon fiber layer was placed over first carbon layer and brushing was applied on it with adhesive resins. Between the two carbons layers, the extra resin which was present was squeezed by rolling a heavy iron roller after the addition of every

new carbon fiber woven sheets. In this way, the symmetrically designed laminate was prepared. The same laminate was kept inside the vacuum bag set up as shown in figure 2 at 720 mm of Hg for 1 hour and 30 minutes so that the maximum amount of resins can be squeezed out. Then, for 24 hours, the CFRP laminate was subjected to heavy normal load and after that curing was done in normal atmospheric conditions.

The SBS (short beam strength) samples of CFRP laminate of 0, 1, 2, 3 and 4 % doped MWCNTs were prepared for each loading rate 0.1, 110 and 220 mm/min.

3. Testing Method

Hounsfield machine *H50 KS* having a maximum load carrying capacity of 50KN was used to perform the SBS test. The specimens were cut, according to *ASTM 2344*, using the diamond cutter. Three different loading rates of 1, 110 and 220 mm/min were used to investigate the effect of loading rate on inter laminar shear strength of CFRP composites. The machine shown below in figure 2 is fully computerized *UTM* machine on which the tests were conducted.



Fig. 2 Computerised UTM, *H50KS* on which the SBS tests were performed

3.1. Short beam strength (SBS) test: The SBS tests were conducted according to ASTM D2344. The testing specimens, dimensions of specimen and the fixtures are shown in figures 3, 4 and 5 respectively..For calculating the ILSS, the following expression was used as given in Eq. 1.

$$ILSS = \frac{3P}{4bh} \quad (1)$$

Where,

P = maximum load (N)

b = specimen width (mm) and

h= specimen thickness (mm)

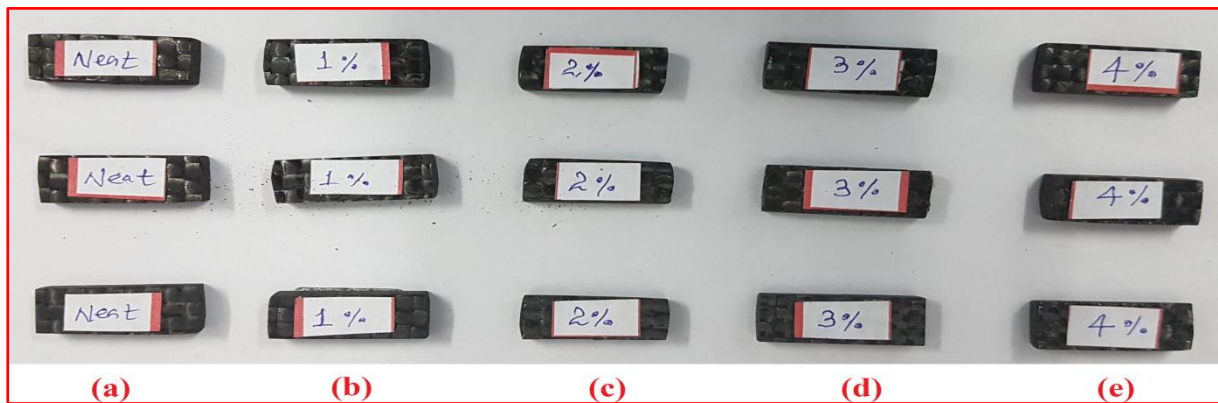


Figure 3. The samples of (a) neat (b) 1 % doped (c) 2 % doped (d) 3 % doped (e) 4 % doped carbon epoxy laminate.

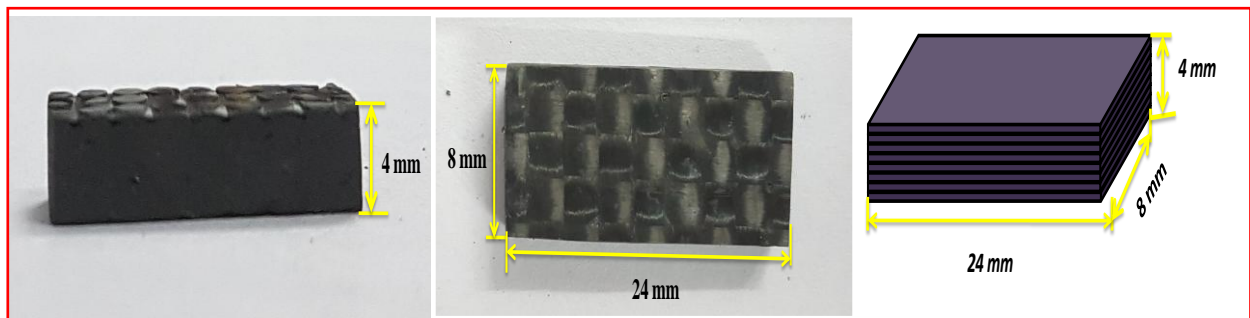


Figure 4. Dimensions of the short beam strength (SBS) test specimen

Dimensions of specimen:

Thickness of specimen = 4mm

Length of specimen = 6 x thickness = 24mm

Width of specimen = 2 x thickness = 8mm

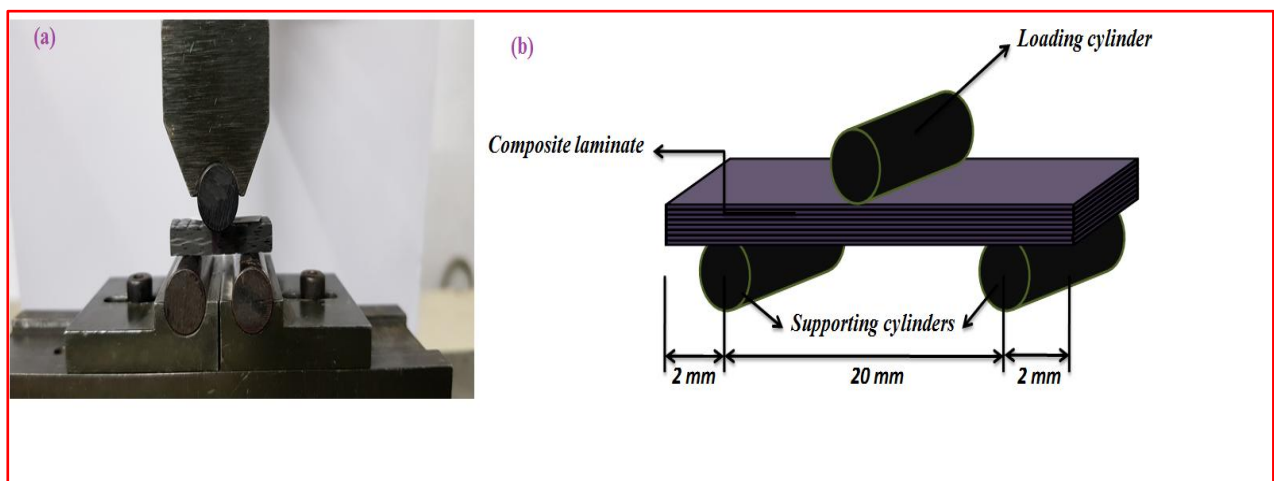


Figure 5. Schematic representation of the short beam strength (SBS) test used to determine the ILSS of fiber reinforced composites.

4. Result and discussions

The mechanical behaviour of fiber reinforced composites is dependent on the loading rate applied to it. The properties of FRP composites depend upon the stacking sequence, the thickness of the laminate, the crack propagation rate, the time taken by the crack to propagate and the number of plies which are actually taking the load applied. There is less time for the crack to propagate at higher loading rates, therefore the value of shear strength was decreased. At lower loading rates, the crack propagation rate is not enough that all the fiber can take the load resulting in lower shear strength values. The crack propagation rate and the time for crack propagation are sufficient at average loading rates so that maximum number of plies takes the applied load, resulting in a higher value of inter laminar shear strength. The value of ILSS was maximum for 3% MWCNTs doped carbon/epoxy laminate at 110 mm/min and minimum for neat CFRP at 220 mm/min loading rate.

Table 2. Results obtained in the SBS test

Serial no.	Loading rate (mm/min)	Percentage of MWCNTs in CFRP (wt.%)	Maximum load (N)	ILSS (MPa)	Extension at max. load
1.	1	0	1171.196	27.449	0.736
		1	1422.632	33.343	0.907
		2	1029.891	24.138	0.652
		3	1556.522	36.481	0.942
		4	1613.056	37.806	0.539
2.	110	0	1348.017	31.594	0.477
		1	1482.734	34.752	0.423
		2	1500.000	35.156	0.478
		3	2228.261	52.225	2.095
		4	1747.252	40.951	0.832
3.	220	0	937.500	21.973	0.447
		1	1029.891	24.138	0.652
		2	1073.792	25.167	0.689
		3	2000.000	46.875	0.434
		4	1291.304	30.265	0.600

Table 3. Influence of variation of loading rate on ILSS of CFRP in SBS test

Loading rate variation (mm/min)	Weight fraction of MWCNTs	% change in ILSS
1 to 110	0	15.102 % Inc.
	1	1.49 % Inc.
	2	3.127 % Inc.
	3	43.157 % Inc.
	4	8.319 % Inc.
110 to 220	0	30.454 % Dec.
	1	30.541 % Dec.
	2	30.584 % Dec.
	3	10.243 % Dec.
	4	26.096 % Dec.

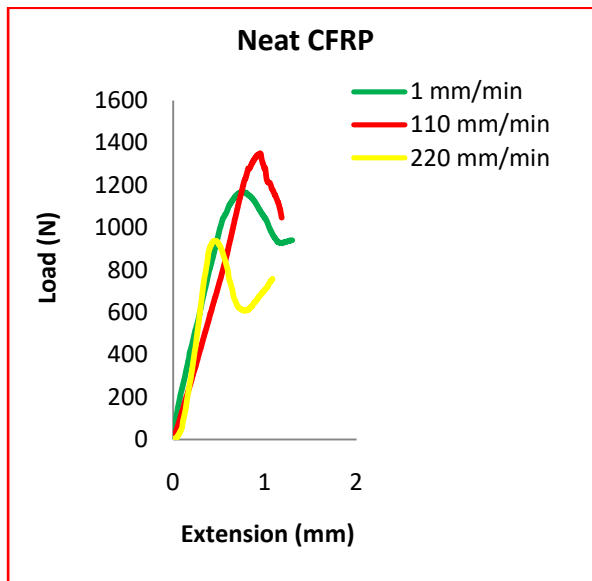


Figure 6. Load vs. extension graph of neat carbon/epoxy laminate at loading rates of 1,110 and 220mm/min.

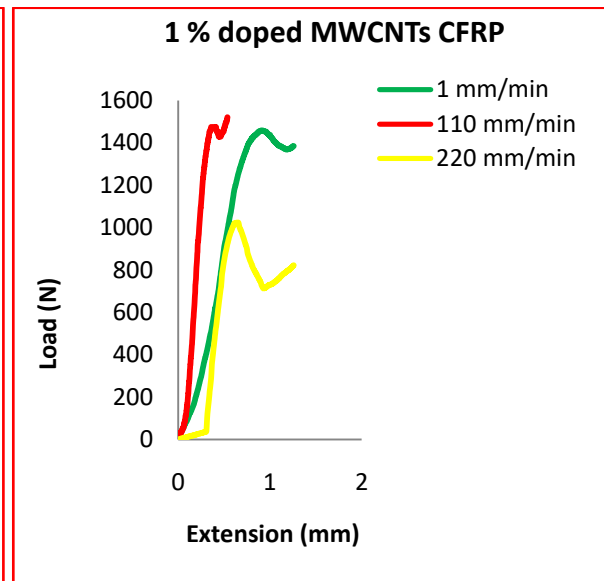


Figure 7. Load vs. extension graph of 1% doped MWCNTs carbon/epoxy laminate at loading rates of 1,110 and 220mm/min.

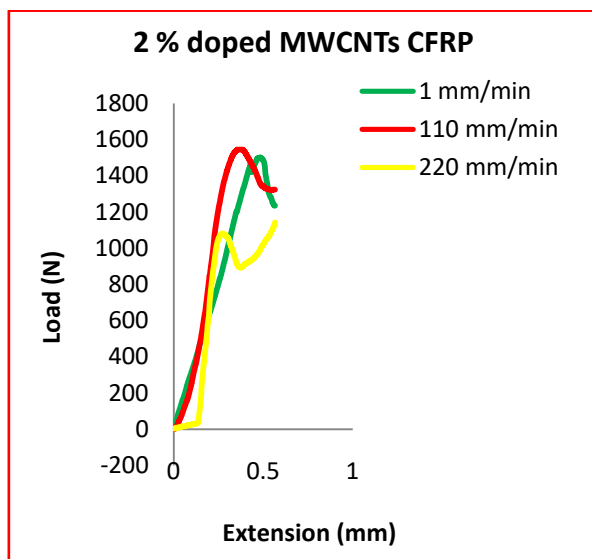


Figure 8. Load vs. extension graph of 2% doped MWCNTs carbon/epoxy laminate at loading rates of 1,110 and 220mm/min.

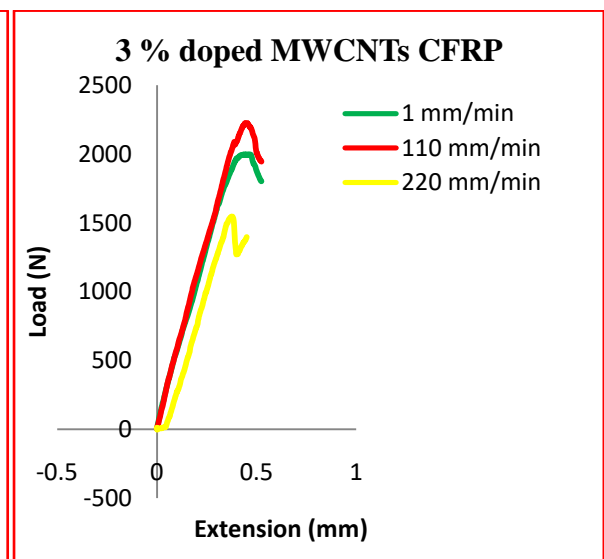


Figure 9. Load vs. extension graph of 3% doped MWCNTs carbon/epoxy laminate at loading rates of 1,110 and 220mm/min.

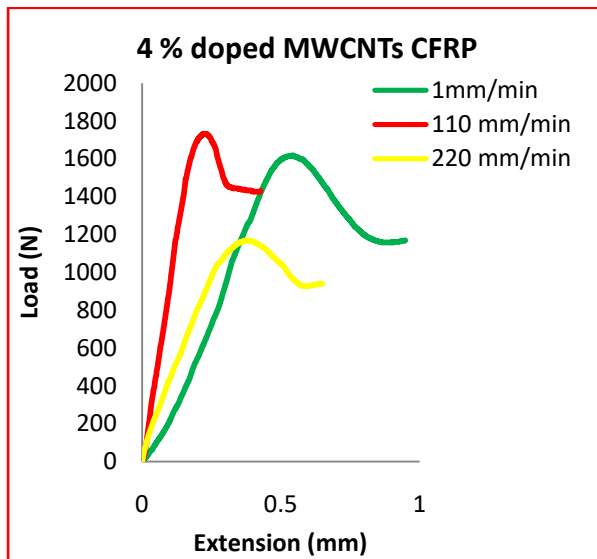


Figure 10. Load vs. extension graph of 4% doped MWCNTs carbon/epoxy laminate at loading rates of 1,110 and 220mm/min.

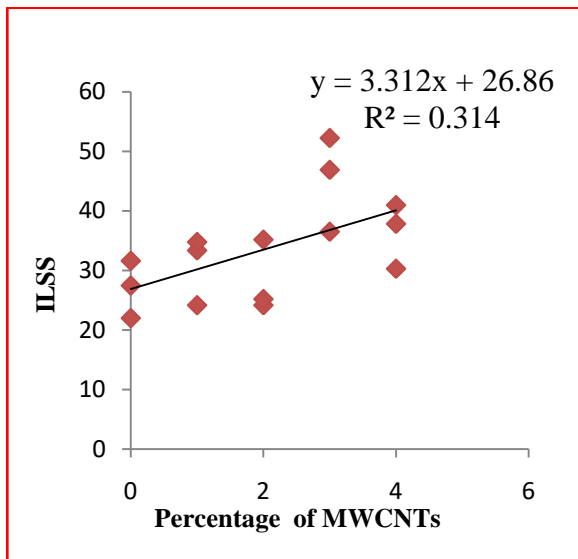


Figure 11. ILSS vs. weight fraction of MWCNTs

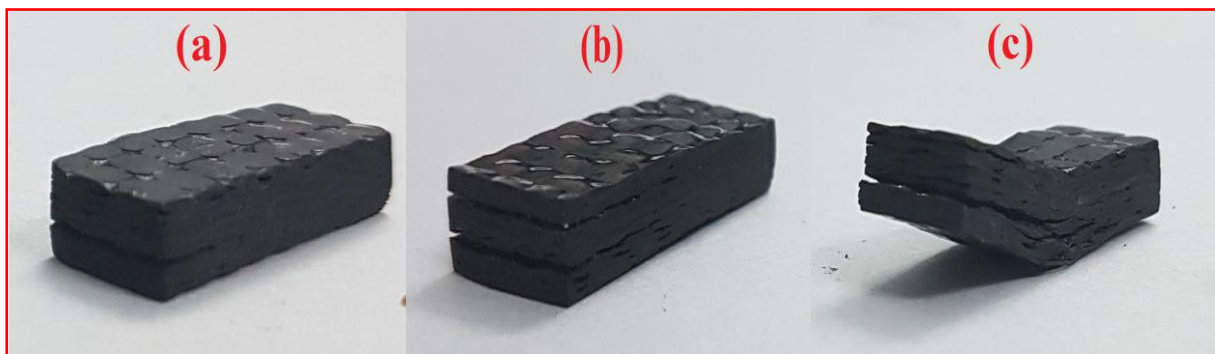


Figure 12. Specimen failure pattern after testing of neat carbon/epoxy laminate at (a) 1 mm/min (b) 110 mm/min (c) 220 mm/min.

4.1. Numerical investigation.

The numerical analysis of the SBS test was also done in ANSYS to validate the experimental results obtained. The data used for the analysis is given in table 4.

Figure 6,7,8,9 and 10 depicts the graph of 0, 1, 2, 3 and 4 % doped MWCNTs CFRP at 1,110 and 220 mm/min loading rate. In all the tests, the load were transferred to matrix which transfers the load to fibers, after which delamination took place. Since the dimensions of specimens were small, the failure was dominated by shear and shear strength was determined. On increasing the percentage of MWCNTs, we noticed that the load carrying capacity of carbon/epoxy laminates were improved. But it was also found that MWCNTs were able to increase the strength of CFRP only upto a certain doped percentage or when the dispersion of MWCNTs in epoxy was good enough to restrict the agglomeration of MWCNTs. This was observed for 1, 2 and 3 % doped MWCNTs in CFRP composites. For 4 % doped MWCNTs, it was found that the value of shear strength decreased due to agglomeration of MWCNTs.

Figure 12 depicts the specimen which have failed of neat at loading rate of 1, 110 and 220 mm/min. We can see that the failure took place due to matrix delamination and stress was higher at 110 mm/min and the strain was maximum at 220 mm/min.

Table 4. Carbon/epoxy laminate material properties

Material properties		Values
Young's Modulus (GPa)	E_{11}	52.32
	E_{22}	52.32
	E_{33}	30.86
Poison's ratio (GPa)	μ_{12}	.36
	μ_{23}	.28
	μ_{31}	.28
Shear modulus (GPa)	G_{12}	.155
	G_{23}	.622
	G_{31}	.622

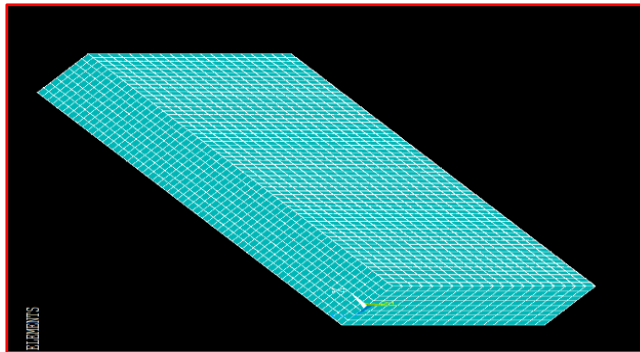


Figure 13. The symmetrical bi-directional woven CFRP laminate comprising of eight plies with 0.5 mm thickness each and ply orientation of $[(0, 90) / (+45, -45) / (+45, -45) / (0, 90)]_S$ was designed in ANSYS for finite element analysis at different strain rate during the SBS test, used to determine the ILSS of composites.

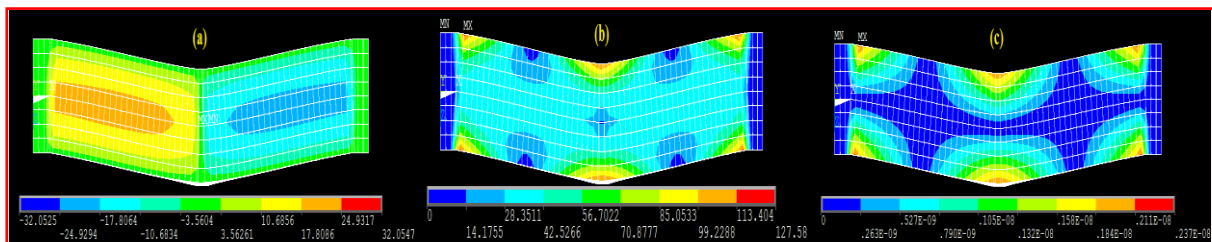


Figure 14. (a) Shear stress, (b) von-mises stress and (c) von-mises strain obtained during the finite element analysis of neat CFRP at 1 mm/min loading rate

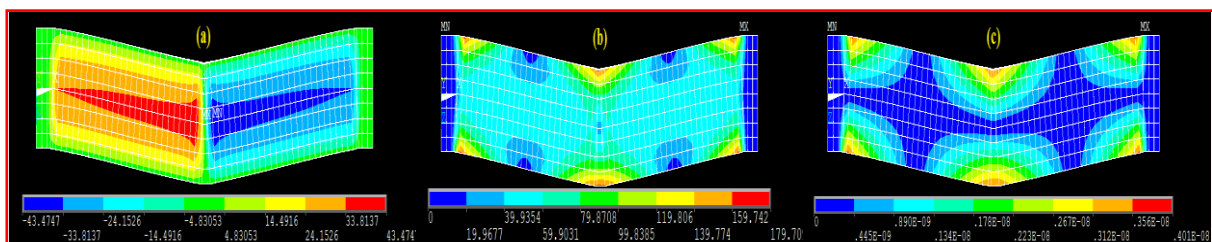


Figure 15. (a) Shear stress, (b) von-mises stress and (c) von-mises strain obtained during the finite element analysis of neat CFRP at 110 mm/min loading rate

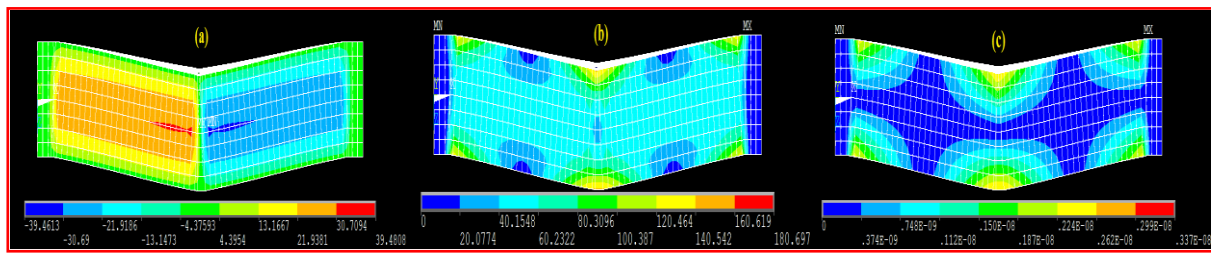


Figure 16. (a) Shear stress, (b) von-mises stress and (c) von-mises strain obtained during the finite element analysis of neat CFRP at 220 mm/min loading rate.

The values of shear stress was greater at plies closer to midplane of the laminate but the intensity of shear stress was greater at 110 mm/min. The values of von-mises stress and strain were greater at points where the loading cylinder strikes the specimen and the bottom portion of the laminate present exactly present below the loading cylinder as shown in figure 14, 15 and 16.

5. Conclusions

Following can be concluded from the above results

1. With increasing strain rate, the load carrying capacities of CFRP increases and hence inter-laminar shear strength (ILSS) increases but there is the existence of a loading rate beyond that the strength decreases.
2. At higher loading rate, the time for crack propagation is very less, the load transfer rate from matrix to fiber decreases and minimum number of plies take the load, and therefore decrement in the value of shear strength is observed.
3. At lower loading rate, the crack propagation time is sufficient but due to less loading rate the crack propagation rate is not enough so the load transfer from matrix to fiber decreases such that all the plies cannot take the load applied resulting in decrease in the value of ILSS.
4. At medium loading rate, both the time for crack propagation and the load transfer rate from matrix to fiber are sufficient, so that maximum number of plies can take the load resulting in increase in the value of ILSS.
5. The doping of MWCNTs increases most of the mechanical properties of FRP laminates.
6. Due to agglomeration of MWCNTs in fiber reinforced composites, even the one doped with a higher percentage of MWCNTs showed a lower value of inter laminar shear strength properties.
7. Finite element analysis is a sound technique to carry out the simulation based on the mechanical properties of composite materials.

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