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Fabrication, Characterisation, and Multiscale Modelling of an Epoxy Composite of Amine-functionalized Carbon Nanotubes Included Three Stages of Carbon Fibre

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ABSTRACT

The goal of this research is to analyse the effect that functionalization has on the material characteristics of multiscale carbon epoxy composites, in addition to determining how efficiently carbon nanotubes are distributed throughout the material (CNTs). As part of this experiment, carbon nanotubes, sometimes referred to as CNTs, were incorporated into an epoxy matrix. After that, carbon fibres were woven into the matrix in order to give it more strength. The resulting combination was used to construct unidirectional carbon fibre laminates. This procedure included infusing an epoxy matrix with a certain number of carbon nanotubes that had been amine-functionalized in the ideal manner. The inclusion of CNTs in the resin at a weight percentage of 1.1 had the impact of creating a significant rise in the Young's modulus. This was the result of the presence of CNTs. The flexural modulus showed observable signs of improvement as a direct result of this action. This event took place as a direct consequence of the presence of CNTs inside the system. However, the overall characteristics of the three-phase composites deteriorated when the epoxy resin was loaded with CNTs at a concentration of 1.6 weight percent. In order to do an examination of the mechanical characteristics of multiscale composites. The two different strategies were combined in order to get this result. The results of this investigation shed light on the disparities that exist between the values that were predicted and the values that were actually found in the data. It is hoped that the multiscale composites in question will be used in the aerospace and missile industries in order to investigate the possibility of structural applications.

Keywords: *Carbon Nanotubes; Polymer; Fabrication; Carbon Fibre; Amine-functionalized Multiwalled Carbon Nanotubes; Tensile.*

1.0 Introduction

Carbon nanotubes (CNTs), which have excellent mechanical properties and a low density, have a unique use as a reinforcing nanofiller in composite materials [1, 2]. This application was made possible by the combination of these two factors. The discovery of CNTs opened the door for the implementation of this technology. The tensile, shear, and flexural properties of multiscale composites are increased as a result of the high strength and stiffness of CNTs [3]. Even negligible amounts of loading of CNTs (less than five weight percent) result in large gains in both the mechanical and physical features of the material that is being reinforced with CNTs, which is proof that the CNT reinforcing approach is effective. There have been a number of studies [4, 5] conducted

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with the objective of producing high-performance CNT/polymer composites. These composites contain a proportion of CNTs ranging from 0.1 to 5% by weight. However, it has been shown that the dispersion of CNTs in a variety of polymers in a way that is both uniform and consistent is a very difficult task, which often leads to a deterioration of the features of composites. Another significant issue is the insufficient interfacial adhesion, which is primarily responsible for the efficient distribution of load transfer that takes place between the CNTs and the matrix. This is a significant problem because it is primarily responsible for the efficient distribution of load transfer. Because of this, the CNTs and the matrix are unable to work together to efficiently disperse the load, which is an issue. It is still difficult to experimentally evaluate the interfacial strength of composites made of carbon nanotubes (CNT) and polymers (polymers). According to Zhang et al. [6], the interfacial shear strength of an epoxy composite that was reinforced using a CNT/carbon fibre (T300) hybrid has the potential to reach as high as 106.55 MPa. This finding was published in the journal Composites Science and Technology. This is about 150 percent greater than the interfacial shear strength of composites that do not include CNTs as reinforcement. On the other hand, the theoretical side has achieved a significant amount of success, whilst the experimental side has only gained a limited amount of success. It is feasible to significantly quicken the transfer of shear stress across the contact by chemically functionalizing the surfaces of the CNTs [7]. This will cause the transfer to occur across the interface. This transpires as a consequence of the matrix's shear stress being transferred to the reinforcement by the reinforcement.

Within the field of novel, high-performance materials, multiscale composites that comprise carbon nanotubes, fibres, and matrix have received a significant degree of attention. This is due to the fact that these composites combine many scales of material. There have only been a select few research that have been documented that have used three-phase composites [9– 13]. According to the findings of Gojny et al. [8], it was discovered that the amine functionalization of CNT surfaces contributed to an increase in the interlaminar shear strength (ILSS) by a factor of twenty percent. According to Zhihang et al. [9], increasing the amount of CNTs in a direction that is perpendicular to the surface of the fibre resulted to an increase in ILSS that was thirty percent higher than before. [10] [12] Godara et al. [13] evaluated the tensile strength of unidirectional laminates that were reinforced with carbon fibre in a number of different orientations. The laminates were put through a series of tests.

It is also one of the most intriguing areas of research. Both of these subfields of inquiry are still in their formative stages at the moment. The Halpin-Tsai equations [14–16] and the Mori-Tanaka methodology [18–21] are the two methodologies that are presently being used for the bulk of the time when it comes to CNT/polymer composites. [14–16] and [17–20] in their own separate sequences. In the research carried out by Qian et al. [16], the composite films made of multiwalled carbon nanotubes (MWCNTs) and polystyrene were broken down into randomly oriented discontinuous fibre lamina. After that, they used the Halpin-Tai equations to analyse the data, which allowed them to calculate the tensile modulus of the composite material. Yeh et al. These are two of the several methods that may be used to alter the equations. This action was carried out in order to make adjustments to the Halpin-Tsai equations. Kim et al. [21] determined the tensile modulus of three-phase woven laminates by a combination of theoretical analysis and research that was conducted in the laboratory. The results of their investigation are detailed in the following phrase. They discovered that the theoretical tensile modulus is 11.8 percent higher than the value that was observed; the discrepancy between the two may be traced to the assumptions that were made in the theoretical models.

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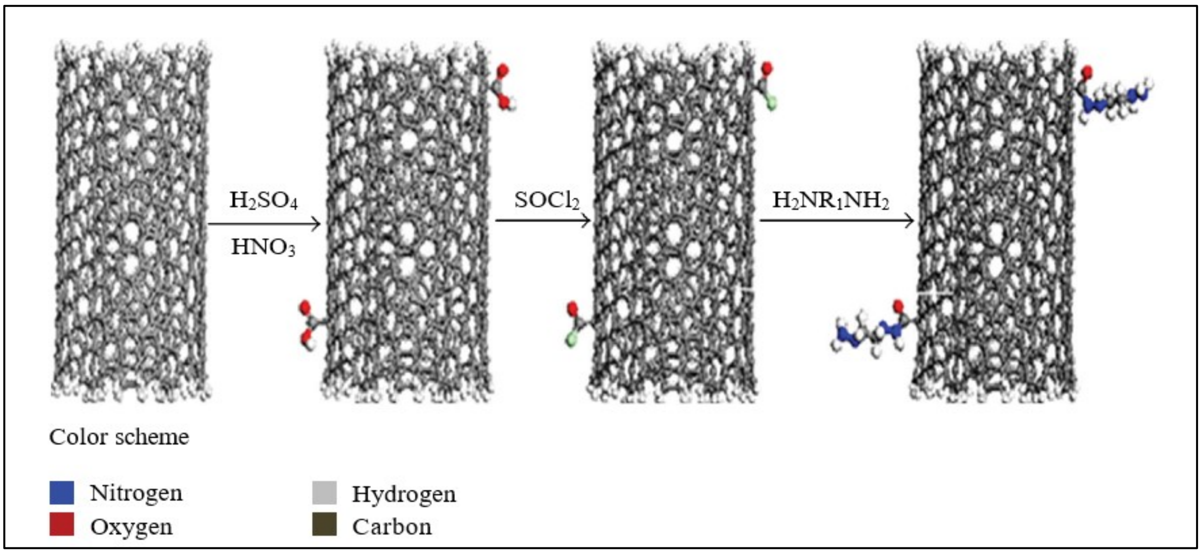
This study aimed to get a greater understanding of the theoretical and practical concerns involved in producing three-phase multiscale CNT composites and the influence of CNTs on the material's characteristics. This study aims to build a durable interface between amine-functionalized CNTs and epoxy. Achieving a homogenous dispersion of AFMWCNTs in epoxy is also a primary objective of this study. If we are successful in reaching our objectives, then we will have successfully completed both of these aims. In order to achieve a more even dispersion, many different methods have been studied and analysed. This research also examines how CNT functionalization affects the mechanical characteristics of multiscale composites. The next phrase explains their investigation's results. The gap between the theoretical and observed tensile moduli is due to assumptions made in the theoretical models.

1.1 Materials

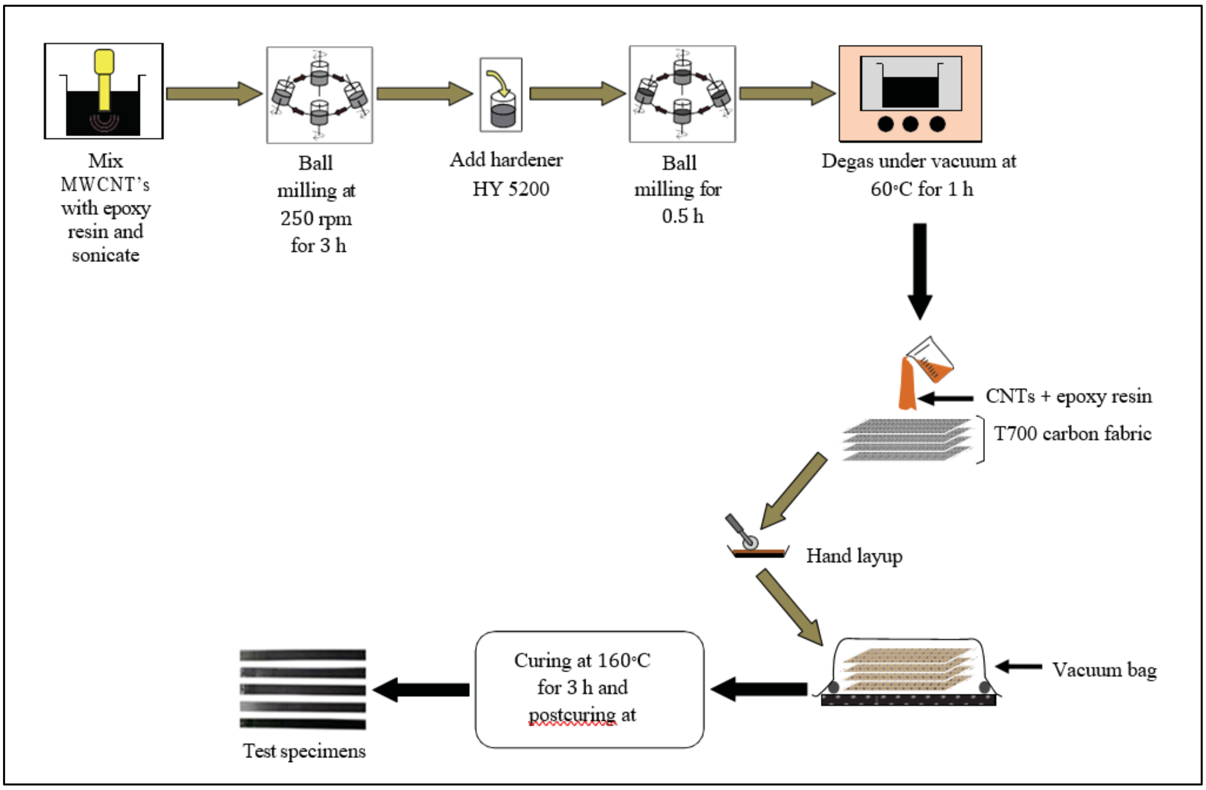
The AFMWCNTs came from Chemapol Industries, and they had a diameter of 21–29 nm, a length of 21–29 m, a density of 1.81 g/cc, and a Young's modulus of 410 GPa. In addition, their length ranged from 21–29 m. (Mumbai, India). Diethylamine is mixed together with resin at a ratio of 7.2 parts by weight to 120 parts by weight in order to functionalize the resin. The diethyl group was used for the amine functionalization procedure since it was recommended by the research conducted by Gojny et al. and Shen et al. [3, 22]. [CNT] stands for carbon nanotube (CNT), and [epoxy] stands for epoxy. The procedure that was carried out in order to functionalize MWCNTs is shown in Figure 1, along with the modifications that were applied to their chemical structure as a result of the procedure. These quantities accounted for 0.25, 0.51, 1.1, and 1.51 percent of the total weight, respectively. The sonicator that was used for this study endeavour will have more of its background explained to the reader in the next paragraph. In the end, the three-phase multiscale composites were created by using the hand layup method, with a carbon fibre content of sixty percent and for a variety of CNT loadings, as shown in Table 1. For this study, the authors made use of carbon fibre of the T700 grade, which was produced by the Toray Company in the United States and consisted of 605 individual filaments in each tow. The usual modulus of T700 fibre is 232 GPa, and its tensile strength is around 5500 MPa; both of these values indicate that it is a very durable material.

Figure 1: The Procedure that is Used in the Functionalization of MWCNTs



2.2 In order to produce a weight fraction of 0.3 percent for the CNTs in relation to the resin and curing agent, an ultrasonic probe sonicator equipped with a Horn Sonicator 3000 was employed for a duration of one hour and eighteen minutes. The Horn Sonicator 3000 has a tip diameter of 3.2 millimetres, an operating frequency of 22.5 kHz, and a maximum power output of 100 watts. Additionally, its maximum power output is 100 watts. Following this procedure, the combination of AFMWCNTs and the resulting product was ball milled for an additional thirty minutes at a rate of two hundred fifty revolutions per minute. After that, the mixture was heated before being evenly dispersed over the carbon fibre using the hand layup technique. This was done after the mixture had been degassed in a vacuum oven for an hour at a temperature of sixty degrees Celsius. After the carbon fibre was infiltrated with the mixture of LY556 and epicure W/MWCNT, it was placed in a vacuum oven and heated to 110 degrees Celsius for two hours. After that, it was subjected to a post-curing process that lasted three hours and was conducted at a temperature of 180 degrees Celsius. The conduct of this study resulted in the production of a total of five distinct carbon fibre reinforced laminates. One of these laminates consisted entirely of resin.

Figure 2: Fabrication Process of Carbon Fibre/CNT/Epoxy Three-phase Composites



2.0 Experimental Results and Discussion

FTIR spectroscopy was used in the initial stage of this research effort in order to provide conclusive evidence that AFMWCNTs had been functionalized. During the second stage of this experiment, five examples of each kind of composite were examined and analysed. The findings of the tensile, flexural, and shear strength tests that were performed on multiscale composites are described in this part. The tests were carried out on the multiscale composites.

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2.1 Effect of functionalisation

Due to the nanotubes' lack of infrared transparency, FTIR spectroscopy has only shown a limited capacity to investigate the structure of AFMWCNTs. AFMWCNTs have a significant absorption because to their black colour; as a result, it is often hard to discern them from the surrounding noise. This is because their colour is dark, which contributes to this trait. Because of this problem, it is necessary to use a very low concentration of the nanotubes in the potassium bromide (KBr) powder in order to make up for it. This is done in order to compensate for the problem. Figure 5 demonstrates that the peak at 3425 cm1 corresponds to the NH2 stretch, whereas the peaks at 1635 and 1187 cm1 correspond, respectively, to the stretching of the NH in-plane and the C–N bond in AFMWCNTs. This is demonstrated by the fact that the NH2 stretch corresponds to the peak at 3425 cm1. This is shown by the fact that the NH2 stretch coincides with the peak at 3425 cm1, which demonstrates the point. One technique to demonstrate that MWCNTs include an amine group is to look for a peak at 3425 cm-1. This peak is caused by the flexible variation of primary amine and may be seen when this variation occurs. If one is looking for it, it is possible to find this summit.

Tensile Test*:* The specimen was broken into many pieces as a result of the tensile testing that was performed on it, as shown in Figure 6. Each kind of composite material that was listed in Table 1 was put through a total of five different rounds of testing. Figure 7(a) displays the usual stress-strain graphs for each composition, whereas Figures 7(b) and 7(c) exhibit the mean values of tensile modulus and tensile strength, respectively, for each multiphase composite. Figure 7(a) shows stress-strain charts for the varied compositions. The longitudinal tensile modulus and tensile strength of unidirectional multiphase composites are fiber-dominated. When one examines the plot of the longitudinal tensile modulus in Figure 7, one can see this quite well (b). whereas the tensile strength decreases for 4CNTCFEC. The creation of a covalent link between the AFMWCNTs and the epoxy matrix, which results in a more effective transmission of stress, is one potential explanation for this phenomenon.

Table 2: Tensile Properties of Neat Epoxy and CNT/Epoxy Composites

% CNT in CNT/epoxy composite	Tensile modulus □ (GPa)	Poisson's ratio]	Shear modulus □ (GPa)
0.0% CNT (neat epoxy)	3.11	0.38	1.16
0.20%	3.45	0.38	1.23
0.45%	3.87	0.38	1.46
1.1%	4.22	0.38	1.53
1.6%	4.47	0.38	1.68

Table 3: Elastic Properties of Carbon Fibre/Epoxy and Carbon Fibre/CNT/Epoxy Multiscale Composites

Composite description	Longitudinal tensile modulus E_{11} (GPa)	Transverse tensile modulus □22 (GPa)	In-plane Poisson's ratio]12	In-plane shear modulus □12 (GPa)
CFEC	98.95	7.54	0.286	2.78
1CNTCFEC	124.45	8.42	0.286	3.08
2CNTCFEC	128.90	9.31	0.286	3.37
3CNTCFEC	138.31	10.33	0.286	3.79
4CNTCFEC	142.53	10.78	0.286	3.87

Figure 6: Fractured Specimen of Multiscale Composites

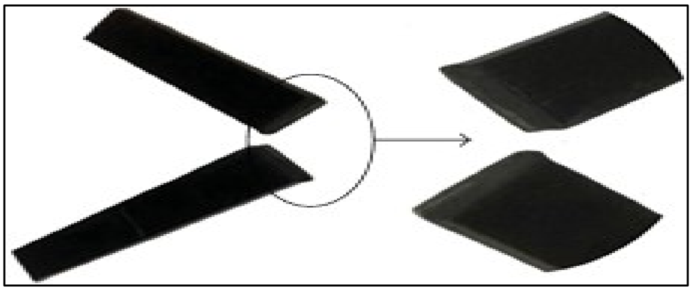

Figure 7 reveals that the value of Young's modulus for 1CNTCFEC has increased by 40.5% in comparison to CFEC. This can be shown to be the case (c). The reason for this is owing to the fact that unidirectional composite laminates are typically used. Because these characteristics are assessed in the longitudinal direction, this is the situation that exists. It turns out that AFMWCNTs are substantially more effective than was previously believed to be the case, and this is due to the fact that the polymer chains are aligned along the CNTs in the axial orientation. This might be due to the higher tension that has built up around the fibres.

As shown in Figure 8, it was found that the theoretical tensile modulus was about 16 percent higher than the actual value. This was observed in the case of CFEC. [Further citation is required] [Further citation is required] This may be explained by the assumptions that are made in the Halpin-Tsai equations. These assumptions include that the fiber-matrix bonding is perfect, that the dispersion is excellent, and that the l/d ratio is very high. The morphology of the surface that had been broken was examined using a scanning electron microscope (SEM) in order to investigate the interaction between carbon fibre, carbon nanotubes, and epoxy. A coating of gold was put to the specimen before it was analysed by the SEM. This was done so that the specimen could be seen more clearly when viewed via a microscope. The images in Figures 9(a) and 9(b) were captured by a scanning electron microscope, and they demonstrate the weak bonding that exists at the interface between the fibres and the matrix.

Shear Test: Testing with shear was carried out, and the results of the examination of the specimen are presented in Figure 10. The shear force test was carried out on five test specimens of each type of composite, and the outcomes of the test were noted down. The results of the tests are shown in Figure 11 as the mean values of the five test specimens, together with bars that indicate the standard deviation. These figures may be found beside one another in the same figure. The findings of the tests suggest that there is a progressive rise in ILSS up to 3CNTCFEC, and then there is a subsequent reduction of 38.72 percent with the addition of additional CNTs. This pattern continues when more CNTs are added. When compared to CFEC, it was found that 3CNTCFEC had a value that was greater by 28 percentage points than CFEC. The presence of AFMWCNTs causes a change in the surface, which in turn causes a strong interfacial contact. This increased interfacial contact is the cause of the rise in ILSS. Although the findings that were published by Gojny et al. [8] and Zhihang et al. [9] were somewhat identical to those that we got, the matrix that was used by these other researchers was different from the matrix that we employed (name it here). In the research that they carried out [13], Godara and his colleagues discovered that modified MWCNTs had an ILSS value of 67 MPa. This value for modified MWCNTs was reported to be 13.38 percent higher than the ILSS value that was measured for unmodified MWCNTs.

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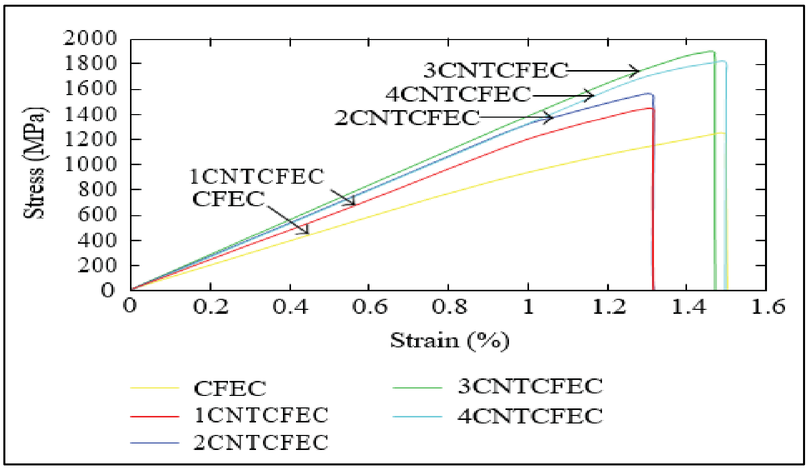
Figure: Stress-strain Plots



Other possible causes include a CNT surface that has been damaged less severely, the maintenance of the aspect ratio, and the introduction of an ideal quantity (up to 10 percent) of –NH2 functional groups during the CNT functionalization process. Other variables, such as step duration and temperature, may also have an impact on the grafting of a variety of functionalized groups onto the CNT surface. The researchers Park et al. [23] discovered that when the temperature rose, there was an effect on the CNT/epoxy composite in the form of a decreased matrix toughness. They also emphasised the significance of optimising the functionalization of the CNT surface, pointing out that this has a significant impact on the polarity of the CNT surface as well as the amount of contact it has with the matrix. In addition, they emphasised the importance of minimising the amount of space between the CNTs and the matrix. In addition, Ci and Bai [23] offered an explanation of the lowest influence that CNT reinforcement may have on the features of a composite if the matrix is highly resilient. This explanation was presented in the context of the characteristics of a composite. Because of this, it is very difficult to detect any sort of impact that the CNT may have on the strength of the epoxy matrix. This is true regardless of the kind of CNT. They also found that soft and ductile matrices provide better interface coupling than rigid matrices, which have poor interfacial contact due to the full cross-linking of polymer molecules around the CNTs. This was in contrast to rigid matrices, which have excellent interfacial contact due to the fact that they are rigid. In the present experiment, laminates were manufactured using a matrix that had a modulus of around 3.10 GPa. This value is much more than the limit that Ci and Bai [23] indicate.

Figure: Tensile Modulus

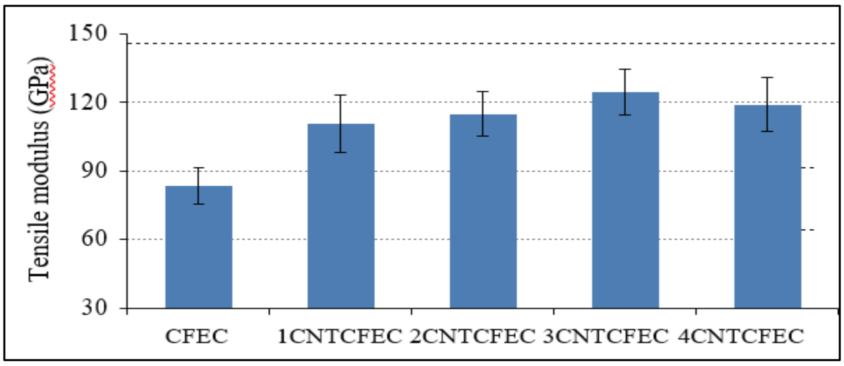


Figure: Tensile Strength of Different Multiphase Composites

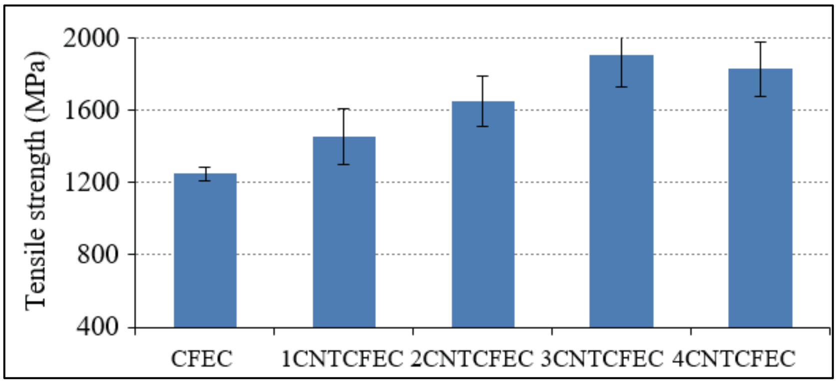
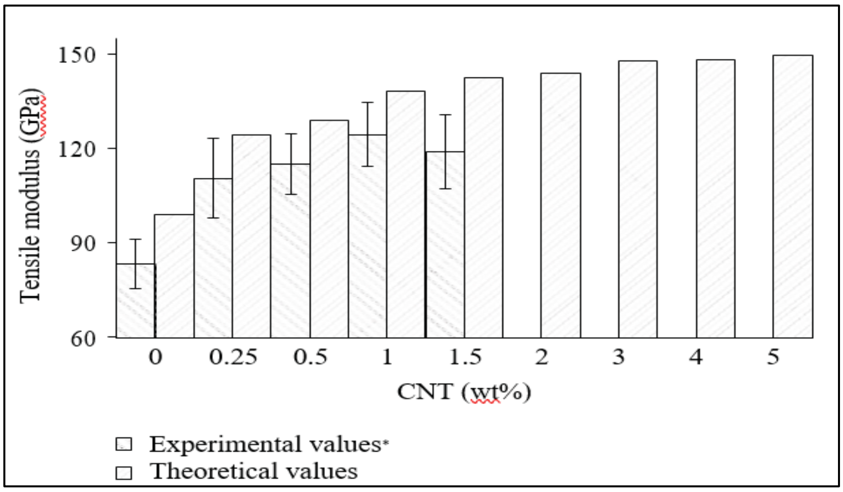


Figure 8: The following table provides a visual representation of a comparison between the theoretical and actual values for the longitudinal tensile modulus. Only up to 1.6 weight percent of CNT reinforcement was used in the experiments, and only then were values obtained.



Flexural Test: This improvement is a result of the comparison. ∗ In the trials, only up to 1.4 weight percent of CNT reinforcement was employed; as a result, only those figures are provided here. When opposed to rigid matrices, ductile matrices provide greater interface coupling. On the other hand, the interfacial contact in stiff matrices is poor due to the complete cross-linking of polymer molecules surrounding the CNTs. In this particular experiment, laminates were created utilising a matrix that had a modulus of around 3.15 gigapascals (GPa). The limit that Ci and Bai [23] mention is far lower than this number, which is significantly greater. The substantial decrease in ILSS that was seen for 4CNTCFEC is most likely attributable to the fact that the matrix stiffness was enhanced in addition to the interfacial contact being increased. This behaviour suggests that it was created by the reinforcing action of the CNTs since it is determined that the matrix, and not the fibres, is responsible for determining the flexural features of the material. Because 4CNTCFEC has a lower flexural strength in comparison to 3CNTCFEC, it is possible to deduce that there is a factor that governs the matrix-dominated mechanical properties that is more important than the degree of CNT dispersion. This can be inferred from the fact that 4CNTCFEC has a lower flexural strength. This may be shown

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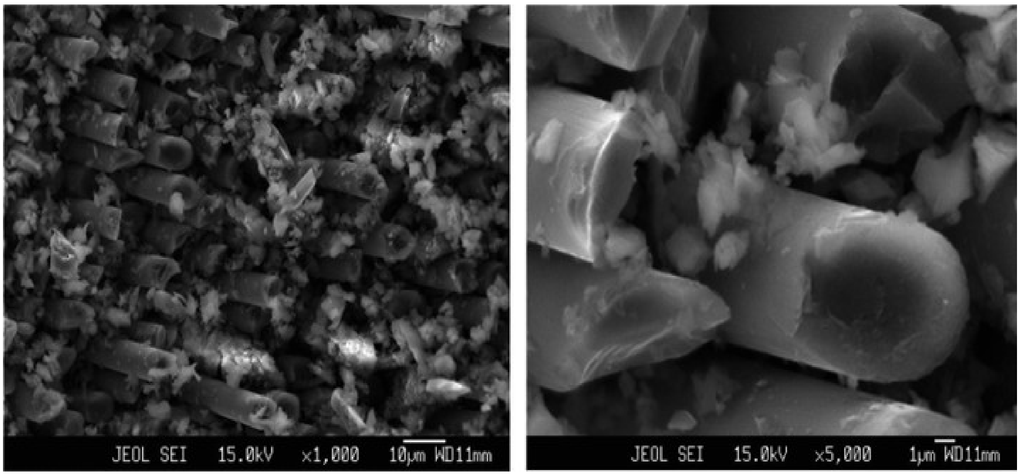
by contrasting the flexural strengths of the two different types of material. Another aspect that may be analysed and contrasted is the degree of curing that has already taken place in the epoxy resin by the time that it is injected.

Figure 9: SEM Images of Carbon Fibre Epoxy Interface Showing Debonding and Void in (a) 2CNTCFEC and (b) 3CNTCFEC Sample

(a)



(b)

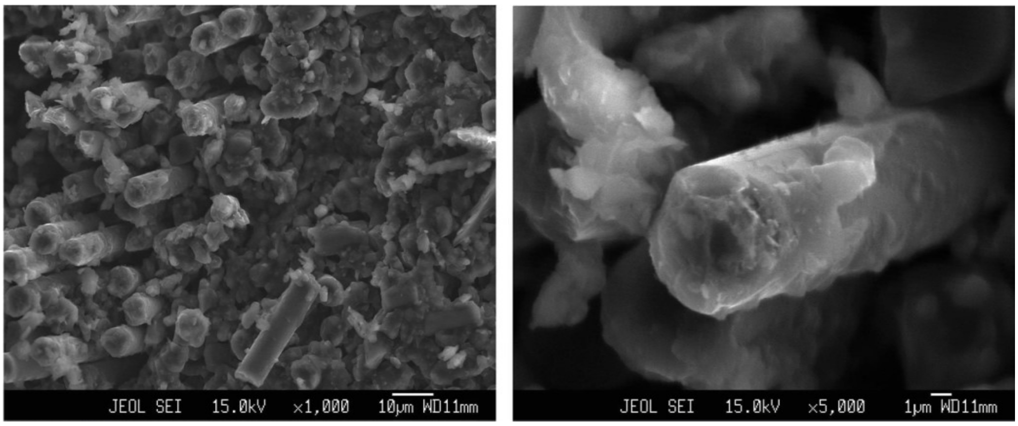


Figure 10: Shear Tested Specimen of Multiscale composite.

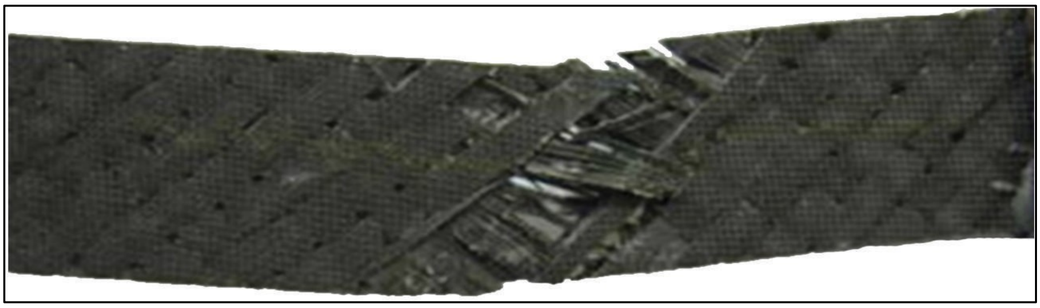


Figure 11: ILSS Variation with CNT Concentration (wt%)

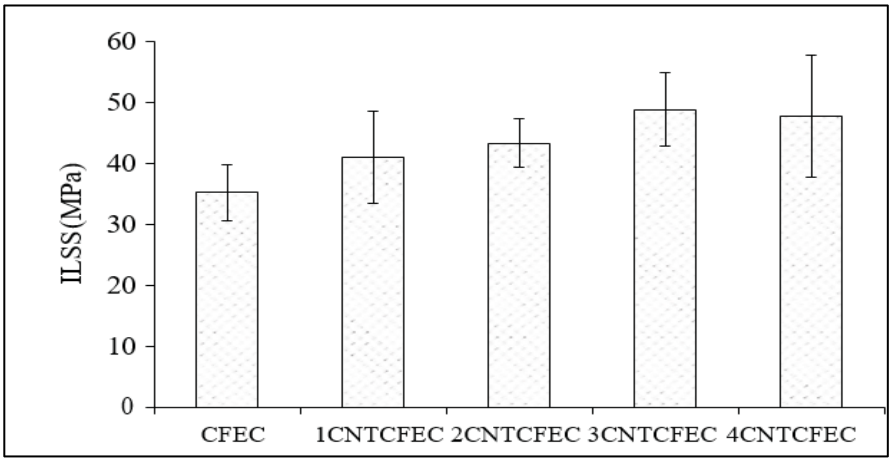
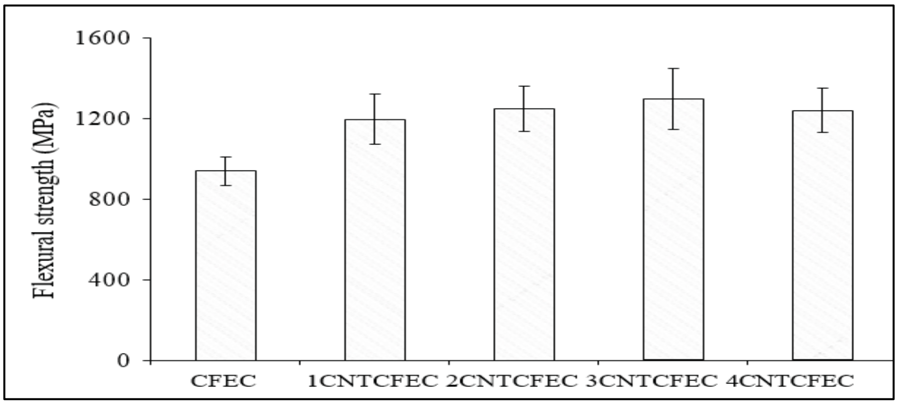


Figure 12: Flexural strength variation with CNT concentration (wt%)



To maximise the CNTs' reinforcing effect, they must establish firm contact with the matrix. This maximises CNT value. CNTs enhance the carbon fiber-matrix interaction. This transfers the load from the fibre to the matrix while bridging the mismatched properties [8]. Optimising the CNTs' surfaces for load transmission may be the most effective way. This improves the mechanical characteristics of multiscale composites.

3.0 Conclusion

In this study, we provide a complete analysis and discussion of the results of the tests that were carried out and how those results were obtained. Carbon nanotubes, and epoxy, the two most critical considerations were ensuring excellent dispersion and perfect bonding between the component elements. Ultrasonic treatment, either on its own or in conjunction with high-speed mechanical steering or stirring, was used to achieve the goal of uniform distribution of AFMWCNTs throughout the carbon fiber-epoxy matrix. This was the consequence of the method. When AFMWCNTs were dispersed in the matrix at a concentration of just 1.1 percent. The increase in Young's modulus was 48 percentage points, and the increase in tensile strength was 51 percentage points. Similarly, the flexural strength and the ILSS both observed improvements in their levels of improvement of 38 percent and

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39 percent, respectively. The presence of AFMWCNTs is responsible for the development of an interface between the carbon fibres and the epoxy matrix in multiscale composites, which is responsible for the better mechanical properties of multiscale composites. A parallel three-phase composite modelling technique was carried out in order to produce an accurate forecast of the tensile properties of the material. This was accomplished by using the equations. When it came to carbon fiber/epoxy composites. It's possible that the assumptions that are built into the Halpin-Tsai equations are to blame for this issue. These presumptions include a high l/d ratio, good dispersion, and strong fiber-matrix bonding.

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