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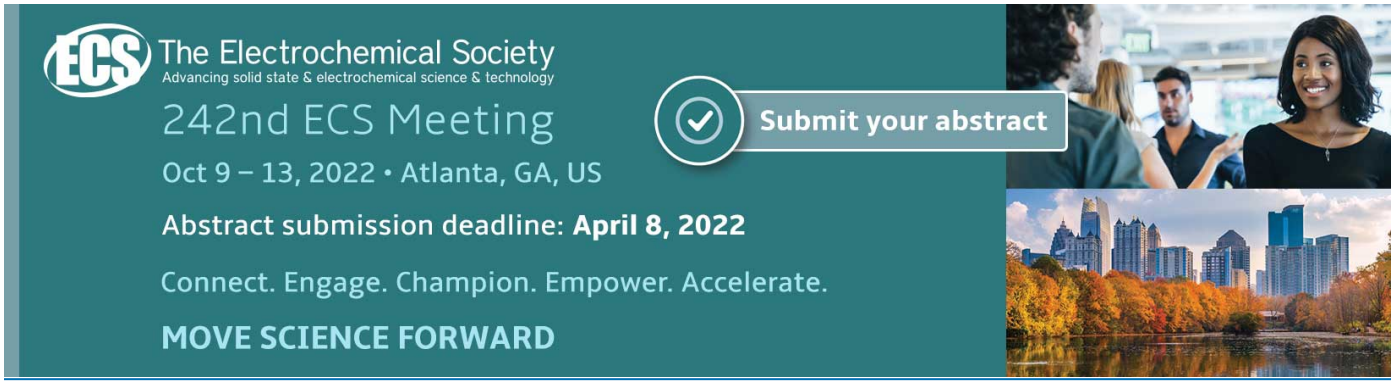

An Experimental Investigation of Fracture Toughness and Volume Resistivity of Symmetric Laminated Epoxy/ Glass Fiber/CNT multiscale composites

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Abstract. In this work an attempt is made to improve the fracture toughness and electrical conductivity of epoxy/glass fiber based laminates by the inclusion of carbon nanotube (CNT) fillers. The fiber orientation of the epoxy/ glass fiber (GF) fabric laminates was optimized based on estimation of mechanical properties. The carboxylic acid functionalized CNTs were incorporated into epoxy matrix by ultra-sonication method. The nano filled epoxy resin was used to prepare laminates with 30/45 GF fabric orientation. The CNT content was varied and its effect on the tensile properties was determined. The fracture toughness of multiphase composites was estimated using single edge notch bend (SENB) test. The presence of CNTs improved the fracture toughness by a crack bridging mechanism. The volume resistivity of multiphase composites was found to be superior to the conventional epoxy/CNT composite. The presence of glass fabric reduces the number of inter-tube contacts contributing to the reduction in volume resistivity.

Keywords: Symmetric Laminates, Fracture Toughness, Stress Intensity Factor, Electrical Conductivity

1. Introduction

Epoxy is the most widely used thermoset matrix material for composites developed for marine, civil and aerospace applications. The range of curing mechanisms, easy process ability and relatively lesser cost in comparison with other thermosetting polymers providing similar properties, have made epoxy the material of choice. A lot of research reports are available on the composites prepared from epoxy and synthetic fibers like glass, carbon, aramid etc. [1-6]Long glass fiber reinforced polymer composites are widely used in aerospace, automotive, sports and various structural applications because of their high strength to weight ratio and non-corrosive properties [3, 4].Compared to carbon, glass fiber is cheaper which is expanding its area of application [5, 6].Lot of research was carried out in epoxy matrix based on fiber reinforcement orientations. Wang et.al had investigated the effect of fiber orientation angles in the properties of composite materials [7].They inferred that load carrying capacity is superior in the fiber direction and higher the fiber content in composite, higher the Young's modulus. The curing of epoxy exhibits highly cross linked structure, which imparts good adhesion

properties and thereby improves the static mechanical [1]properties like tensile strength and modulus. Since epoxy develops a 3D network during curing, the high cross linking density leads to ductile-brittle transition which lowers the fracture toughness and limits its applications to structural engineering. Hence, impact modification has been a hot topic of research for the last ten to fifteen years [1, 2].

An important method of improving fracture toughness of epoxy is to provide reinforcement such as fibers and elastomeric based impact modifiers like polyburadiene. While fibres will not affect the static mechanical properties of the composite, the inclusion of impact modifiers enhances the performance of the composite but at the expense of tensile strength which is not desirable for structural applications

Nano particle reinforced polymers are considered to be one of the most important fields of materials research [8-9]. Multiple layers of graphite rolled on themselves to form MWCNTs, which are in tubular shape and exhibiting unique electrical as well as mechanical properties. The excellent mechanical strength allows the composite to be used as an important reinforcing material for structural applications [10].They also improve the stiffness and electrical conductivity. Modern field of research in nano composites combining CNTs with reinforcements like carbon, glass fiber were investigated by [11, 12], they found that addition of 0.1% of MWCNT with epoxy improved the mechanical properties by 20%. The toughening mechanism of epoxy reinforced with MWCNTs revealed that addition of small amount of MWCNT increases the strength and fracture energy of epoxy matrix; this is due to the effect of CNT bridging with epoxy matrix to resist fracture. However, as the concentration of MWCNTs increases there is a decline in fracture toughness and energy value as well [11]. According to Zhang et.al addition of small weight percentage of MWCNT can improve the fracture properties because MWCNTs can strengthen the interface between glass fibers and the matrix and it reduces the weak interfacial bonding between the fibers and matrix [12].Bakir et.al [13] investigated the effect of orientation angle of glass fiber and it was reported that the laminate had good strength when the fiber is perpendicular to the direction of tensile force and also at 45^{o} as well when the volume fraction of fiber was 30%. Midhun et.al [14] had investigated the fracture properties based on the fiber orientation, it was reported that fracture properties was were best for laminate with 15^{0} orientation.

Conductivity studies incorporating nanofillers allows the implication of damage detection and stress-strain monitoring, apart from the improvement in the mechanical properties of the composite [15, 16]. As investigated by Wang et.al [17] when CNT is dispersed into the epoxy matrix, CNTs can percolate through the matrix and introduce electrical conductivity to the composite. The conductivity property in the composite makes it suitable for electrostatic dissipation and electromagnetic shielding applications. Vivo et.al [18] had investigated the conductivity of epoxy based MWCNT with an optimized design and nonlinear effect with 0.6% wt of hydrotalcite clay and 1 wt% of MWCNTs multiphase which showed improved conductivity. Hendra et.al [19] studied the conducting composites by using nano particles such as graphite and CNTs which had helped in increasing the electrical conductivity and mechanical properties such as hardness and flexural strength.

In this work the emphasis is given on the preparation and properties of epoxy/ glass fiber/CNT laminates. The synergistic effect of multi-scale fillers on the fracture toughness of the composite prepared by vacuum bagging process is analyzed. The morphology and

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mechanical property studies are expected to throw light on the interface characterization. The volume resistivity of laminates are not widely reported, which forms a novel part of this work.

2. Materials and methods

2.1 Materials

Unidirectional E-Glass fabric sheet of 220 GSM and thickness of 0.23 mm and density 2.5 g/cc was used to fabricate the fiber reinforced laminate. Araldite LY556 resin which has density 1.4 g/cc and tensile strength of 50 Mpa and hardener HY991 were used as matrix for the laminate. The ratio between resin and hardener was 100:15 by volume. MWCNTs (United nanotech innovations Pvt. Ltd. Bangalore) had purity greater than 95 % and had an average length of 10 microns and an average outer diameter of 5-20 nm. It was available in black powder form.

Table.1 Properties of Glass Fiber and Epoxy

Properties	Density g/ cc	Modulus of Elasticity, E (Gpa)	Dielectric Constant
E-Glass fiber	2.5	72.3	6.13
Epoxy(LY556)	1.4	7.3	3.6

2.2 Methods

Fabrication of Glass Fiber Reinforced Symmetric Laminates

Laminates are mainly classified into symmetric and asymmetric laminates. A laminate is called symmetric when the material, angle of orientation and thickness of the layers are same

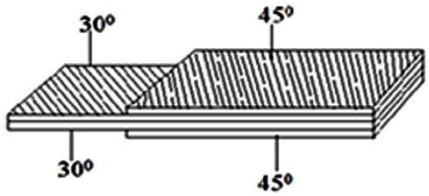

Figure 1. Representation of Symmetric Laminates

above and below the mid plane. The study is focused on symmetric laminates of various fiber orientations $[30/45]_{10}$, $[30/60]_{10}$, $[45/90]_{10}$, $[60/90]_{10}$, $[45/60]_{10}$, $[30/90]_{10}$.Unidirectional E-Glass fiber fabric of 300 mm × 300 mm were cut according to specified angles. Symmetric composite laminates of 10 layers of glass fiber fabric with different combinations of orientations were fabricated by wet layup process. Each fabric layer was coated with epoxy resin followed by stacking. After stacking the fabric as per the required sequence of orientation, release fabric, release material and bleeder material were laid. The consolidation and curing of the laminates

was carried out by vacuum bagging process [20]. The capacity of the vacuum pump used was 1 atmosphere and vacuum gauge was used to ensure required vacuum pressure inside the bag. The cure time was kept as 24 hrs at ambient conditions and at its end the peel plies were removed to take the consolidated sample and were cut to various ASTM test standard specimens for the evaluation of mechanical and fracture properties.

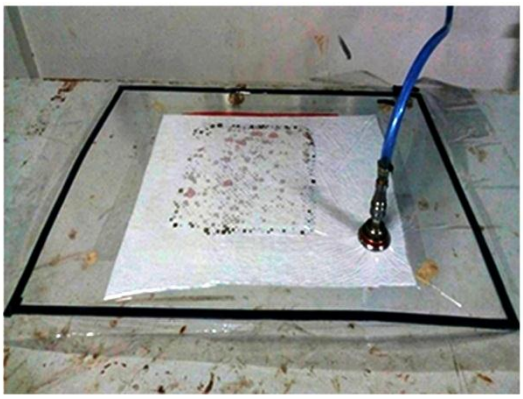

Figure 2. Vacuum Bagging for Fabrication of Laminates

2.3. Estimation of volume fraction

Fiber volume fraction in the composite was estimated using resin burn off test according to ASTM D 2584. The crucible containing the pre weighed composite sample is then kept in a muffle furnace and maintained at a temperature of 650^0 C for two hours. The resin burn off sample was then taken out and fiber weight was measured. The volume fraction of fiber for all the samples was maintained at 58%. The volume fraction was calculated using the following relation,

$$Fiber\ resin\ volume\ ratio,\ \frac{V_f}{V_m} = \left(\frac{W_f}{W_m}\right)\left(\frac{\rho_m}{\rho_f}\right) \quad (1)$$

$$Matrix\ Volume, V_m = \frac{1}{1+\frac{V_f}{V_m}} \quad (2)$$

$$Fiber\ Volume\ , V_f = 1 - V_m \quad (3)$$

2.4 Characterization techniques

Tensile tests were performed according to ASTM D 638, using a universal testing machine of 25kN capacity at a cross head speed of 1mm/min. Fracture properties were evaluated by single edge notch bend test carried out as per ASTM- D5045 in a universal testing machine. The morphology of the composites was investigated in a scanning electron microscope. The fracture surface of the composite was sputtered with gold to make it conductive prior to analysis. The analysis was carried out at an acceleration voltage of 20kV. Electrical conductivity test was also carried out using two probe methods. The sample size was maintained as 1cm length, 1cm width and 0.2cm thickness.

3. Results and discussion

3.1. Evaluation of tensile properties

The Stress- strain curves obtained for the various symmetric laminates are delineated in Figure 3. It is observed that there is a considerable role for the fiber orientation in the tensile strength of the composite. In a symmetric laminate, the main load bearing constituent is the fiber. In the present study symmetric laminate of $[30/60]_{10}$ orientation shows a maximum tensile strength of 51.6 MPa but the elongation at break of the laminate is less. The energy absorbing capacity of this laminate can be considered to be less when compared with $[30/45]_{10}$, as reported earlier [7]. In their study, they concluded the load along the 45^{o} fiber orientation was higher and the elongation was more for $[30/45]_{10,}$ laminate aiding higher energy absorption.

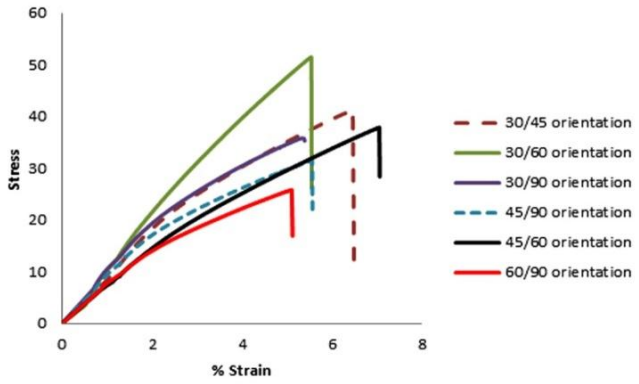

Figure 3. Stress-Strain graph of various symmetric laminates

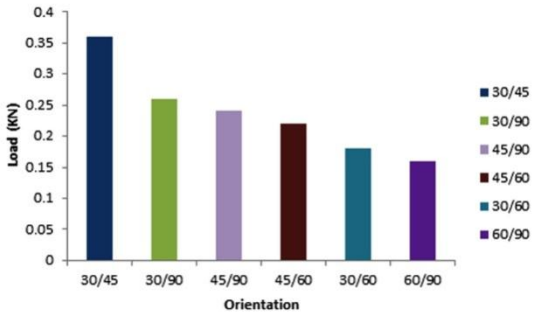

Figure 4. Variation Maximum load of different symmetric laminates

3.2. Evaluation of fracture properties

For the evaluation of fracture behavior various methods are being practised of which Single Edge Notch Bend (SENB) test was employed to evaluate the stress intensity factor value of various symmetric laminates [23].The scheme for the test is given in the Figure 5.

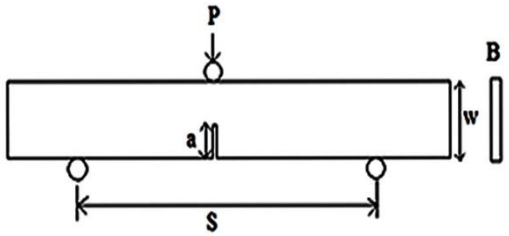

Figure 5 Schematic representation of Single Edge Notch Bend Test

Where a = Crack length, P = Load applied, W = Width of the specimen, B = Thickness of specimen. The crack was provided exactly at mid span of the specimen. When the load was applied in the compression side of the flexured sample, the crack propagated along the fibre interface. From the load data the stress intensity factor values were evaluated using the following empirical .relations the term $f(\frac{a}{w})$ represents the geometric factor for the SENB.

Stress Intensity Factor $K_I = \left(\frac{P}{B\sqrt{W}}\right) f\left(\frac{a}{w}\right)$ (4)

$$f\left(\frac{a}{w}\right) = \frac{3\frac{s}{w}\sqrt{\frac{a}{w}}}{2\left(1+2\frac{a}{w}\right)\left(1-\frac{a}{w}\right)^{\frac{3}{2}}}\left[1.99 - \frac{a}{w}\left(1-\frac{a}{w}\right)\left\{2.15 - 3.93\left(\frac{a}{w}\right) + 2.7\left(\frac{a}{w}\right)^2\right\}\right] \quad (5)$$

Where P is the applied load, a = 12.5 mm, w = 25 mm, B = 2.3 mm. The maximum load obtained during SENB test for the different laminates is presented in Figure 6. The maximum load capacity of 0.36 KN was shown by the $[30/45]_{10}$ laminate. This laminate had obtained an optimum load carrying capacity and showed a higher stress intensity value of 9.45 $Mpa\sqrt{m}$. All other samples recorded stress intensity values less than 7 $Mpa\sqrt{m}$.

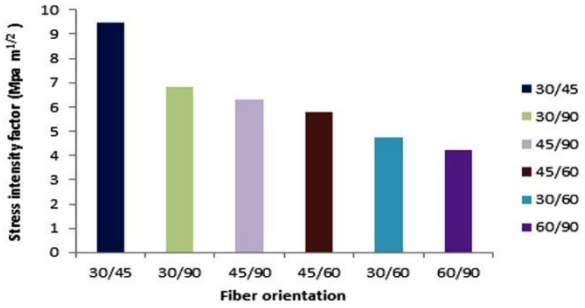

Figure 6.Variation of Stress Intensity Factor and Fiber Orientation angle

Since the stress intensity values were found to be the maximum for $[30/45]_{10}$laminates, further studies using MWCNTs were carried out in this particular symmetric laminate anticipating improvement in fracture toughness of MWCNTs. The nano tubes were added to the epoxy matrix by ultra-sonication technique [29]. The scheme for the preparation of epoxy nano composites is shown in Figure 7 .CNTs were first added to the epoxy resin and the sonicator probe was then inserted into the mixture. The sonication process was carried out for 1 hour to ensure proper mixing of CNTs into the matirx. In an earlier study better results were obtained for sonication that was conducted for a duration of 1 to 2 hours [30].

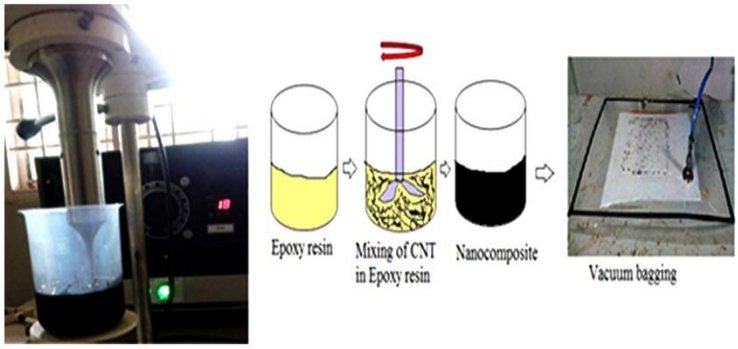

Figure 7.Schematic representation of fabrication of Epoxy Nanocomposites

The ultra-sonicated epoxy resin containing CNTs was used for laminate preparation. The CNT content was varied from 0.25 wt% to 1.25 wt%. After curing of the CNT containing [30/45]10laminates, the specimens were subjected to tensile test. The stress-strain plot for various weight percentages of CNTs in [30/45] laminates is shown in Figure 9.
From the plot it is inferred that tensile strength is increased with weight percentage of CNT up to 1 %, further addition of CNT showed a decrease in tensile strength. CNT/epoxy multiscale

composite showed an improvement tensile strength because of CNT's ability to act as secondary reinforcements on the surface of the fiber and they help in carrying tensile loads by improving the interfacial adhesion between the epoxy matrix and glass fiber, glass fibre being an inorganic material will not have any chemical interaction with the epoxy resin. MWCNTs which are acid functionalized can react with the epoxy groups to form β – hydroxy propyl ester. The improvement in the composite strength with modified MWCNT with epoxy system [resin and curing agent] can be understood from the reaction mechanism in the figure below.

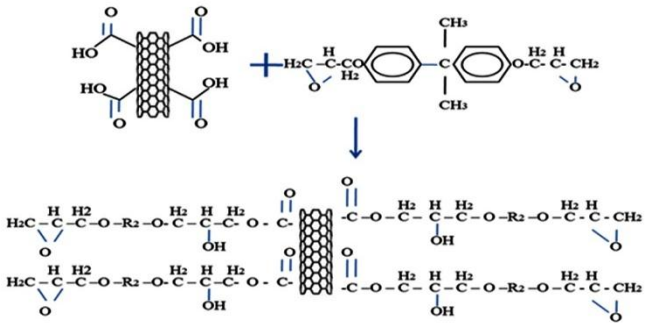

Figure 8. Reaction Mechanisms for MWCNT with Epoxy resin

The carboxyl group participates in opening of the epoxide rings, resulting in an ester bond and formation of –OH group [33]. Strong covalent bonding would lead to a more effective stress transfer at the matrix-nanotube interface for these composites. Carbonyl groups on nanotubes can bind to the polymer matrix through hydrogen bonds. Crosslinking takes place because both the surface and ends of the nanotube are expected to carry more than one functionalized group. Carboxylic acid groups can undergo esterification reactions with the epoxy.

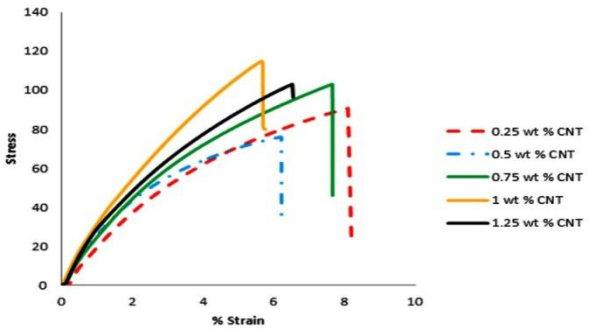

Figure 9. Stress Vs Strain for [30/45]10 laminates with different percentage CNT

A main potential of CNT as reinforcement in polymer matrix is its ability to improve fracture toughness. Figure 9 shows the variation of load with wt % of CNT. Up to 0.75 wt % there was a linear relationship between stress intensity value and quantity of CNT. Further addition of CNT decreases the stress intensity value. It is shown that 0.75 wt % CNT can withstand a maximum bending load of 1.02 KN and a maximum Ki value of 26.78 Mpa $m^{1/2}$(Fig 10).

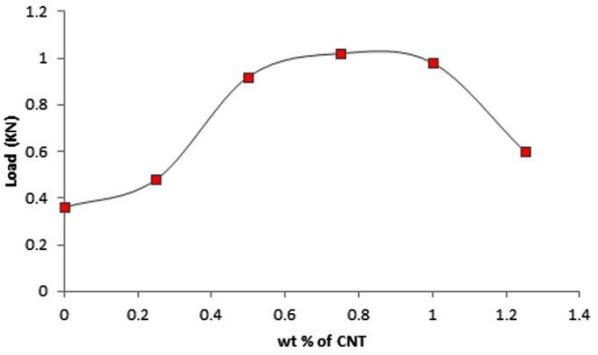

Figure 10. Variation of Maximum Load and Percentage amount of CNT

The fall in strength of these composites for higher concentration MWCNT can be attributed to the tendency of agglomeration of the bundles of MWCNT as a result of increase in the viscosity of the suspension which ultimately becomes a source of crack initiators in the composites under stress. Another contributing reason could be the inadequate wetting of MWCNT with epoxy resin because of large increase in the surface-to-volume ratio of the reinforcement. This would lead to increased porosity and defect sites in the resulting composites which become the ultimate reason for the fall in strength.

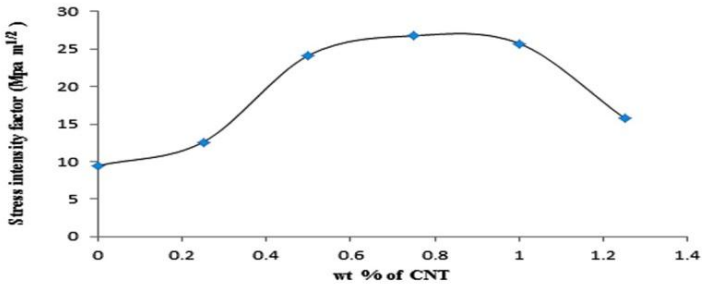

Figure11. Variation Stress Intensity Factor and Percentage amount of CNT

4. Morphology Studies on the Fracture Surface.

During SENB test it was noticed that the failure of the specimens was due to debonding, fiber breakage, delamination and matrix cracking. As the loading progresses, the interfacial crack propagation initiated from the crack tip, which advances through the matrix along the fiber direction. Addition of CNT hinders the crack propagation in a considerable way and hence improves the fracture toughness. Figure (12) represents the schematic representation of crack bridging effect produced by CNTs, thereby increasing both maximum load as well as the stress intensity factor. The surface of fibres act as a bridge between matrix and fiber. The crack bridging is one of the important mechanisms that contributed an improvement in fracture toughness [30-32].

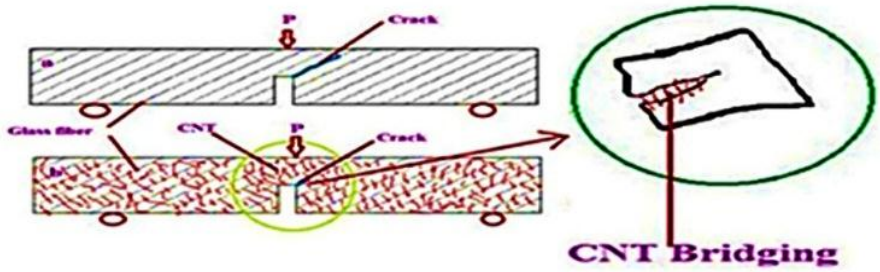

Figure 12. Schematic diagram representing the CNT bridging effect

The morphological examination using SEM as shown in figure 13 revealed the presence of MWCNT in the periphery of glass fibres, which hindered the crack propagation and provided stiffness at the interface, and finally the carbon nanotubes bridging crack at the interface which was observed by the presence of roughness on the fiber surface. Hence presence of CNT in matrix can enhance the fracture toughness up to an optimum level of 8, beyond which it declined.

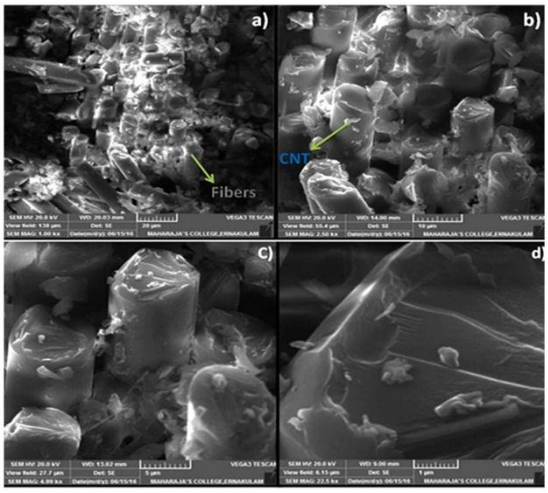

Figure 13. Scanning Electron Microscopic images of the fractured surface showing the CNT bridging with Glass Fiber

5. Volume resistivity and electrical conductivity

Four probe method was used, the sample size was 1cm in length, 1cm in width and 0.2cm in thickness. The electrical conductivity of epoxy/ CNT composites increased with the nanofiller content. The percolation threshold was obtained between 0.8 and 0.9 wt% as evident from Figure 14.

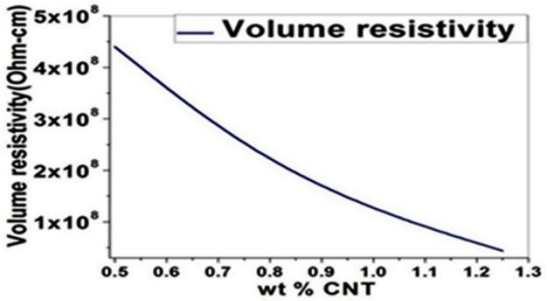

Figure 14. Variation Volume resistivity and Weight % of CNT

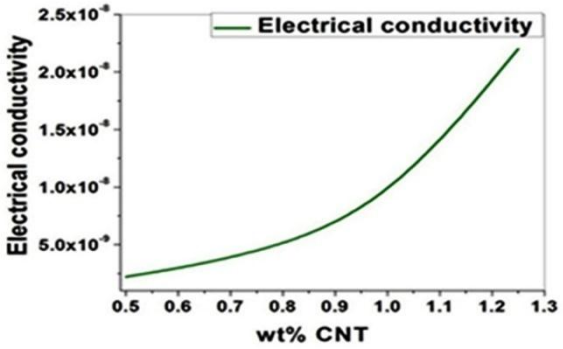

Figure 15. Variation electrical conductivity and weight % of CNT

This is owing to the formation of conductivity network which influenced the electron tunneling and hopping mechanism in the epoxy matrix. .Figure 16 illustrated the effect of fiber on the conductivity in the epoxy matrix , the results are indicating that the fiber reinforced composites showed lower resistivity value than absence fiber in the epoxy matrix .This is due to the formation of continuous network of CNT on the surface of fiber .From SEM image ,it could be confirmed that the CNT are more localized on the fiber surface which is inferred that the fiber

is more stable in thermodynamic phase for CNT than epoxy .Hence this stable state would help to form an excellent electrically conducting network in the epoxy matrix than without fiber nanocomposites.

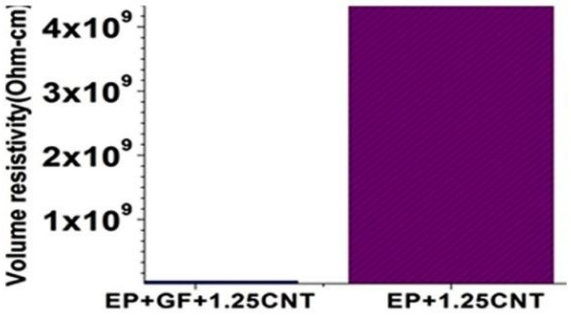

Figure 16.Volume resistivity comparison of Epoxy/Fiber/CNT with Epoxy/CNT.

Conclusion

From the experiments it was concluded that fracture properties of glass fiber reinforced symmetric laminates can be affected by the fiber orientation. The laminate $[30/45]_{10}$ shows the best stress intensity factor during SENB. Nanocomposite laminate was prepared by adding different weight percentages of MWCNTs to the epoxy matrix with a fixed fiber orientation of 30/45. It is noticed that there was a considerable increase in stress intensity factor values by the addition of CNT into the epoxy matrix. Stress intensity factor increases with increase in the percentage of CNT up to 0.75 wt % and further addition of CNT shows a decrease in fracture toughness. It was observed that as the weight percentage of CNT was increased there was an improvement in stress intensity factor along with the conductivity of the composite. Thus for $[30/45]_{10}$ symmetric laminates there was an increase in the stress intensity values which is an advantage for structural composites. They also showed highly conducting behavior in order to ward off the EMI signals, because of this there was an increase in the conductivity values as percentage amount of CNT was added to the laminates.

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