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Fatigue Life Assessment of Nano Composites

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ABSTRACT
Components failing due to fatigue loads, would cause changes in properties which can occur in any structural member, Fatigue is usually associated with these changes in properties and it can cause failure. The fatigue damage in a structural component can occur when defects initiate at the undamaged site and propagate leading to final failure. Fibre reinforced material losses its stiffness due to appearance of microscopic flaws, before any visible damage can occur under reversed loading. The polymeric resin is dispersed with Multi Walled Carbon Nano tubes in various percentages by weight and the effect of stacking sequence on fatigue life is also studied. The study revealed that the life of the Nano composites was better than virgin specimens. The samples were examined by Scanning Electron Microscope after testing.

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Introduction

Fatigue is one of the major reasons for failure of a component in a cyclic load, high temperature, corrosive environment, contact between parts, causing severe damage to the components. Such failures [1] generally occur when the applied loads are smaller than safe loads.
The assessment of the fatigue life involves in characterizing failure in terms of stress and strains till a small crack initiates and the remaining life of the component can be inferred from the number of cycles it can survive. The initiation of the crack is a predominant part in assessing the fatigue life of components subjected to high cycles; the stresses are large enough to cause plastic deformation before failure.
The heterogeneity of the composite makes it difficult to assess the total life of the component. The approach is similar to assessment of isotropic components [2] and due to the fact that damage mechanism is different from its metallic counterparts. The failure of the composite materials comprises of matrix cracking, debonding voids and micro flaws. These defects occur in the early life of the composite material.
The micro flaws are present before any visible damage occurs leading to loss in strength. Generally, the fatigue test would be conducted till the residual strength reduces to a pre-determined level.
The life estimation can be described for various composite materials by a straight line.

$$S=\sigma_u\,(m \log N + b) \qquad (1)$$

Where S = maximum ultimate stress
N = number of cycles to failure
σ_u= Average static strength and
m, b = constants.
The polymeric resins are reinforced with fibre but another novel way to strengthen the resin is by adding Carbon Nano Tubes (CNT), and other such particulate materials [3]. Its unique properties could improve the mechanical strength of the components [4]. The various fabrication methods [5] of CNTs are listed elsewhere. These nanocomposites have excellent properties at low particulate infusions due its aspect ratio and surface area of Nano-sized particles. However, the potential usage of Carbon Nano Tubes as a strengthening agent could not be fully achieved, because of the difficulties in casting the composite and load transfer limitations. CNT can agglomerate, helping to augment the interfacial bonding strength of the composite. The inherent attraction between the CNT particles bound together and is not soluble in many solvents.

Experimental

Materials and Methods

Sample Preparation

In this present work Bisphenol-A based epoxy resin is reinforced with stitched woven roving while the epoxy was toughened with Multi walled carbon Nano tubes [4, 6-7]. A cost-effective method to fabricate the laminate is by wet lay- up method which requires minimum tooling. Multi walled carbon Nano tubes (MWNT) were purchased from M/S Nanoshel Haryana, India. Stitched woven roving was obtained from St. Gobain, Chennai. The resin Ly 556 and the corresponding curing agent HY 951 was obtained from M/S Huntsman limited. A laminate of dimension300 mm x 300 mm was fabricated with three different orientations namely 0-degree (Unidirectional ply), cross ply (CP) and ±45 degrees (Angle Ply). All the samples were machined according to ASTM standards. The tension fatigue samples were fabricated according to ASTM D 3039-76 with the test coupons having dimensions of length of 229 mm, width 25 mm and with a thickness of 3 mm.

Fabrication of Nano Composite

The Nano tube must be investigated for purity, thermal stability and bonding characteristics before fabricating the Nano composite [8]. The multi walled carbon Nano tubes, which was purchased had amorphous carbon of about 2 to 3 per cent; the carbon content was about 90 per cent while the other metallic impurities were present up to 5 to 9 per cent. The impurities will cause adherence problems to the

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polymer thereby decreasing the properties that were intended to increase.

Purification Process

Purification must be attempted in order to remove the amorphous carbon content: (1) Heating the tubes in a muffle furnace at about 450^{o} C for three hours, (2) Stirred in nitric acid for four hours. Then this mixture was rinsed with ionized water. The suspension was degassed to remove the air bubbles. (3)The mixture is heated in an oven at a 80^{o} C so that moisture content can be removed.
The scanning electron micrographs were obtained on Carl Zeiss HR SEM-EDAX elemental analysis before (Figure1) and after the purification process (Figure 2).

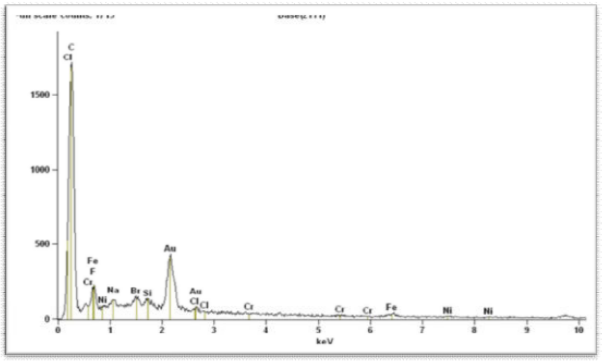

Figure 1: SEM Elemental Analysis before purification

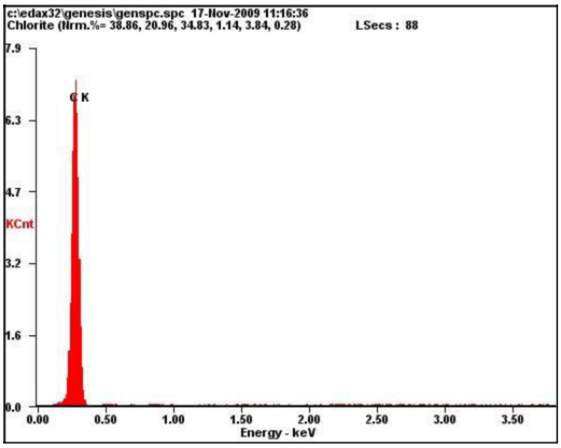

Figure 2: SEM-EDAX after purification

Dispersion of Multi Walled Carbon Nano Tubes

A better dispersion makes the Nano particle to be infused into the resin without forming lumps of CNTs. And thus, shearing of Nano tubes during composite loading will decrease the performance of the composites [3, 9-10].

The Nano tubes have a large surface area so that few polymer molecules can penetrate between them. For even distribution shear forces must be introduced in the suspension to prevent agglomeration. Dispersion can be achieved by various methods like sonication, stirring and calendaring and for this study sonication was used to disperse the MWNT into the epoxy resin [11].
The MWNT is mixed with an appropriate solvent such as toluene so that the property of the polymer is not changed and then sonicated. The viscosity of the polymer is reduced and thereby better adhesion is achieved by this process. The MWNT is mixed with toluene is taken in a beaker of 350 gm, then0.035gm of MWNT was infused into the resin and the suspension was stirred for one hour. The solvent could evaporate and the resin with filler material is ready for casting the Nano composite. The sonicator bath oscillated at 47 kHz, (figure3) causing an even dispersion to be achieved.

Figure 3: Sonication in progress

Testing Method

The samples were tested for tension fatigue at a frequency of 3 Hz on a closed loop 250 kN servo hydraulic Instron testing machine available at Structural Engineering Research Centre, Chennai, the maximum and minimum loads were calculated on the ultimate tensile strength. The test was conducted on a calibrated universal test machine at room temperature. All the samples were tested till failure. The samples had different orientations namely: 0 degree, ±45 degrees and cross ply. The epoxy was infused with multi walled carbon Nano tubes in different percentages namely 0.2, 0.5, 1, 3 and 5%.

Results and Discussion

Three samples were tested at each normalized value of applied stress. The effect of changing the orientation (Figure 4) on the fatigue life was studied and it can be observed that the angle ply had least fatigue life and unidirectional ply had maximum strength.

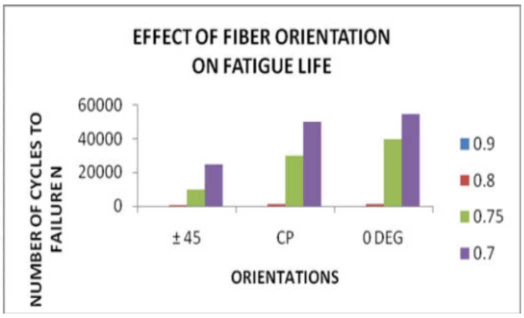

Figure 4: Orientation effect on life of Composites

The polymer was dispersed with multi walled carbon Nano tubes in various percentages and comparison is presented in the Figures (3,10,12) while the data are also considered for different fibre orientations. Individually the fatigue life improved for different orientations at various values of stress ratios.
Three specimens were tested at each normalized stress level varying the stress level between 0.9 to 0.7 per cent of ultimate load. The ultimate tensile strength was 240 MPa for unidirectional ply, 230 MPa for cross ply and for the angle ply it was 225 MPa. The unidirectional ply having 0 degree exhibits exceptional tension behavior. While the

cross ply is better at resisting fatigue. And the least was that of ±45-degree coupons.

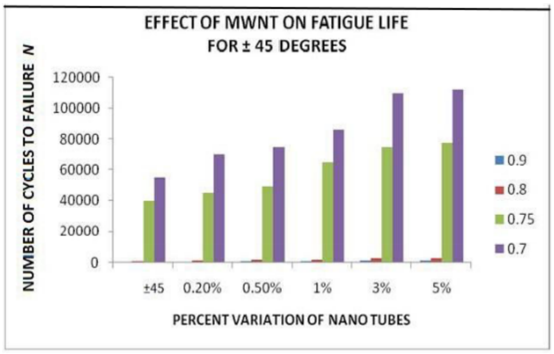

Figure 5: Effect of MWCNT on angle ply

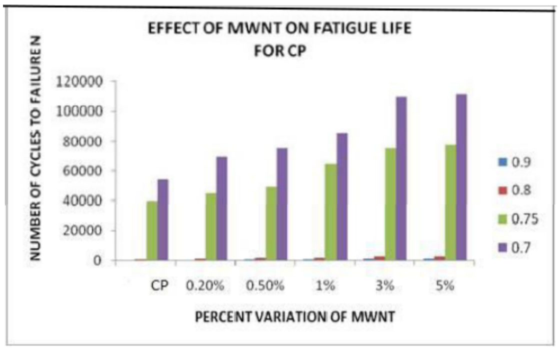

Figure 6: CP with MWNT infusion

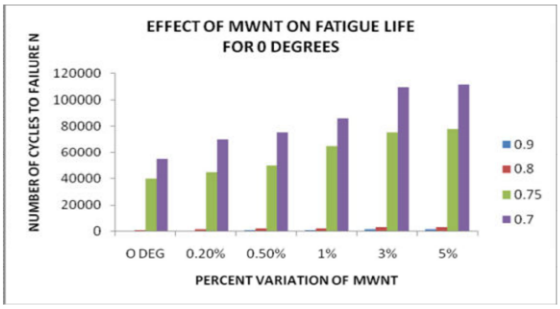

Figure 7: CP with MWNT infusion

The MWNT are particulate materials [8] with geometric features and chemical make-up would lend itself to strength of the base material especially the polymeric composite, these particulate materials are stiffer than the polymer and hence decreasing the strain in the neighbourhood of the resin- particulate boundaries.

But on the other side at higher percent of dispersion of MWNT led to the fatigue strength decreased since the CNT may have agglomerated. The fatigue samples exhibited the typical fibre pull out shown in Fig. 8-9.

Figure 8: Failed Unidirectional ply

Figure 9: Failed MWNT angle ply

Table 1: Percent improvement in fatigue life

Angle (Degrees)	MWNT infusion, % 0.2	0.5	1	3	5
0	71	62.5	56.3	33	27
CP	66	53	47	44	33
±45	60	40	33	31	27

SEM Characterization

Several samples were examined under Carl Zeiss scanning electron microscope at SAIF in IIT (M). Most of the samples were sputtered and vacuumed to remove the dust flecks. The broken surfaces from the failed samples were scanned. The initiation of the cracks can be due to the defects in manufacturing, surface impacts. The major contribution to deformation and fracture is by the polymer matrix and fibres of the individual laminae, whereas only a minor effect can be attributed to fibre fracture events. The broken samples under fatigue loading were examined for a unidirectional ply, cross-ply and angle ply. Regardless of the per cent of dispersion of MWNT failure mechanism remained the same but a slight improvement in the fatigue life was observed.

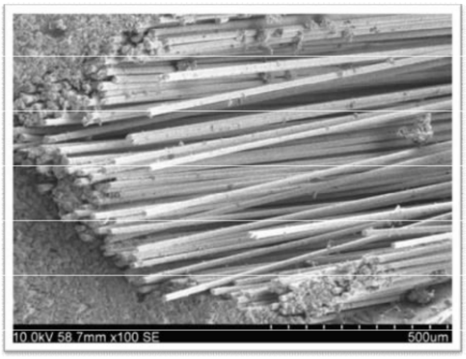

Figure 10: Broken bundles of uni-directional ply under fatigue loading

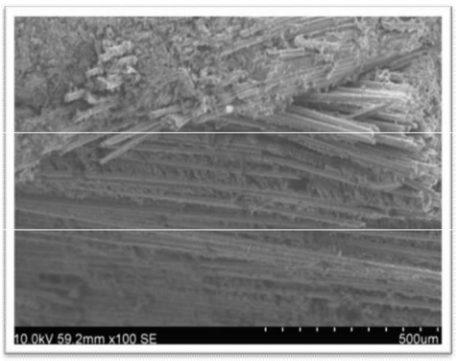

Figure 11: Fatigue failure of cross ply

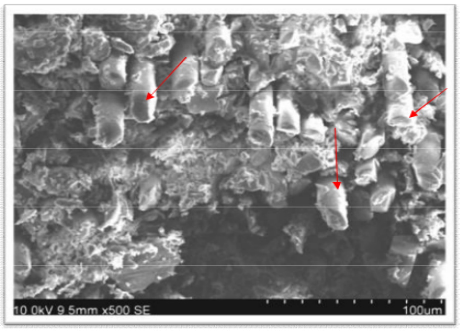

Figure 12: Dense network of broken fibre stubs indicated by arrows

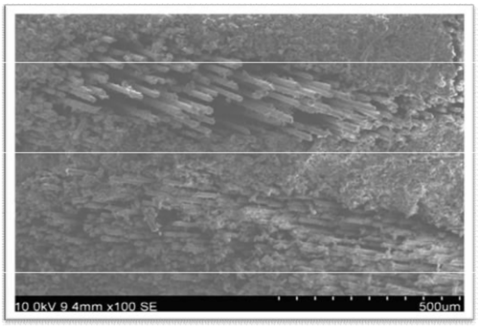

Figure 13: Fatigue failure of ±45 degrees sample

The failure mechanism was mainly dominated by fibre pull out, debonding and void formation. The defects like ribbons, cusps were not pronounced in the samples undergoing fatigue loading. The SEM micrographs are shown in figure 10-13, which is typical fatigue failure.

Figure 12 shows the dense network broken fibre stubs indicated by arrows and attaching itself to matrix. Broken matrix pieces could also be seen.

Figure 13 shows the fatigue failure of the angle ply. It can be observed that large chunks of matrix pieces are scattered on the sample.

Conclusions

Fatigue samples were fabricated by using wet layup technique, cured at room temperature. These samples were tested for fatigue in tension with the stress ratio of 0.1 using a fatigue rated 250kN load cell Universal Testing Machine at a frequency of 3 Hz. All the fatigue samples were infused with 0.2%, 0.5%, 1%, 3% and 5% of multi walled carbon tubes by weight. The fatigue life of the samples with MWNT was compared with that of samples without the infusion of MWNT and another parameter was stacking sequence while the samples were fabricated according toASTM D 3039-76 standards. It was found that the samples with 0/0 as the stacking sequence had better fatigue life, followed by cross ply, the least fatigue life was exhibited by angle ply.

Dispersing MWNT into the resin had similar effect except that the number of cycles to failure increased prominently indicating that there was little loss of stiffness. Increasing the MWNT per cent into the epoxy resin from 0.2% to 5% improved the fatigue life up to 3%, beyond that the addition of 5% of MWNT decreased the per cent improvement of the number of cycles to failure. The lower per cent infusion had better improvement in fatigue life. This indicates that MWNT infusion worked quite well with lower values of infusion. One reason could be that the Nano tubes had agglomerated at higher MWNT percentages causing a decrease in the per cent improvement in fatigue life. At 90% ofthe ultimate stress, 0.5% infusion of MWNT gave better fatigue life, while at higher per cent diffusion the fatigue life improvement decreased, (table 1). Extensive subsurface examination of the broken samples was done using a Scanning Electron Microscope at SAIF in IIT (M). Several failed samples were cut at the fracture surface, cleaned and then scanned. The fatigue samples exhibited the characteristic fatigue failure modes: fibre pull out, matrix cracking and debonding.

Acknowledgements

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