Experimental Novel Investigation of Electrostatic Charged Multi Walled Carbon Nanotubes Reinforced Epoxy Based Polymer Composite

R. SARAVANAN* AND A. SURESHBABU

Department of Manufacturing Engineering, College of Engineering, Guindy, Chennai 600 025.

ABSTRACT

In this research work, multi walled carbon nanotubes (MWCNT) particulate filler of various (0.9, 1.2, 1.5, & 1.8 wt %) weight percentage was used along with epoxy resin. A novel method of distributing the MWCNT in epoxy had been employed to reduce the agglomeration problem by charging the MWCNT electrostatically. The electrostatic charged (MWCNT) and uncharged (MWCNT) were loaded on to matrix and then it was stirred by a mechanical mixer for 300 minutes continuously to achieve uniform distribution. The nano filler reinforced composite was fabricated by using hand layup method and mechanical testing (Tensile and Flexural) were performed as per ASTM standards. The electrostatic charged (MWCNT) results in better dispersion and shows an improved adhesion between the electrostatic charged (MWCNT) and matrix with the evident from the SEM analysis.

KEYWORDS: *Epoxy, Electrostatic charging MWCNT*

INTRODUCTION

The fundamental criteria for strong composites composed from matrices and fillers are: high aspect ratio fillers, alignment, high loading, large interfacial area, and efficient stress transfer between the filler and the matrix [1-2]. Epoxies are widely used as the polymer matrix for high performance laminated composites due to their good mechanical performance, processability, compatibility with most fibers, chemical resistance, wear resistance and cost [3]. Epoxy resins are inherently brittle and hence have reduced damage tolerance. Additionally, in reinforced plastics, damping is governed by the matrix properties and consequently the research effort has been oriented towards the modification of matrix resin systems. During the past decade, techniques

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Correspondence author e-mail: saravananr85@gmail.com
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that allow the tailoring of the resin properties are in the forefront of scientific research [4]. In recent years, micro and nano scaled particles have been considered as filler materials for epoxy to produce high performance composites with enhanced properties. Nanotube-reinforced epoxy systems hold the promise of delivering superior composite materials with high strength, light weight and multi functional features and have attracted great attention [5]. In particular, owing to their extraordinary thermal, mechanical and electrical properties, carbon nanotubes have finer applications as additives to various structural materials [6]. The theoretical predictions and the experimental results have shown that multi-walled carbon nanotubes (MWNTs) possess excellent combinations of electrical, thermal and mechanical properties which make them an ideal filler for polymers because most polymers either do not conduct electricity or have low electrical conductance. Themain applications of conductive polymer composites (CPCs) are in the electronics industry in which they are used as electromagnetic shields, strain gauges and diodes [7-11]. MWNTs can be produced in various ways such as arc discharge, laser ablation molten salt electrolysis and chemical vapor deposition. Depending on the synthesis method used and other parameters involved, the diameter range for MWNTs is from several nanometers to several hundred nanometers. The length of MWNTs is usually several micrometers. A few researchers have investigated the effect of MWNT dimensions on properties of nanocomposites and it has been shown that they can have a considerable effect on both mechanical and electrical properties [12]. The dispersion of electrically conductive nanoparticles, such as carbon nanotubes (CNTs), in a polymer matrix enables the production of an interesting class of materials in view of commercial applications: electrically conductive polymeric composites[13]. One major issue has been identified during the preparation of carbon nanotube polymer composite, that carbon nanotubes are usually tend to aggregate. Some studies have been carried out to solve this problem. It was found out that some methods can uniformly distribute CNTs into the matrix, like mixing method, ultra-sonic dispersion method, three-roll grinding dispersion method, and three-roll milling dispersion method. However, in the curing process of the CNTs-epoxy composites, CNTs still agglomerated and precipitated, resulting in poor binding force between the composite and the substrate [14].

The electrostatic forces between energetic nanoparticles (Al and Fe_2O_3) and multi-walled carbon nanotube (MWCNT) are exploited to prepare Al/Fe_2O_3/MWCNT nanostructured energetic materials in organic solvent. A negatively charged MWCNT can be used as a glue-like agent to direct the self-assembly of the well dispersed positively charged Al (fuel) and Fe_2O_3 (oxide) nanoparticles. This spontaneous assembly method decreases the aggregation of the same nanoparticles largely, moreover, the poor interfacial contact between the Al (fuel) and Fe_2O_3 (oxide) nanoparticles was improved significantly [15]. Electrostatic spraying method is another most common technique used for the powder coating, in which powder particles are charged electrostatically to a high voltage and then deposited onto the surface of the sample [16]. It has the advantages of simple operation and strong binding force between the coating and the substrate [17]. The electrostatic flocking is another method which is employed to attach multi-walled carbon nanotubes to face sheet prepregs. The voltage and the duration of process are adjusted to gain different attaching amount of MWCNTs. The face sheet prepreg stacks with different weight contents of MWCNTs are prepared and then sandwich samples are assembled and cured by autoclave process [18].

In this present work, a novel method of distributing the MWCNT in epoxy has been employed to reduce the agglomeration problem by charging the MWCNT electrostatically. Electrostatically charged MWCNT will repel each other due to similar polarities which in turn results in homogeneous dispersion. The uncharged and electrostatic charged MWCNT are loaded in epoxy with 0.9, 1.2, 1.5 & 1.8 wt % proportions and subsequently samples are fabricated using hand layup method [5]. A new Experimental Setup is also fabricated to charge the MWCNT electrostatically. The prepared samples are then subjected to microscopical evaluation and mechanical characterization. The results are then analyzed and interpreted.

MATERIALS AND METHODS

Materials

In this work, epoxy resin was used as matrix material and multi walled carbon nano tubes (MWCNT) powder was used as filler material with the specifications of outer diameter 10 nm to 30 nm and the length of 1 - 10 micrometer was purchased from Sri Sai Scientific Company, Coimbatore, Tamilnadu, India. The filler was extracted from the carbon. The epoxy resin Diglycidyl Ether of Bisphenol-A (DGEBA) and the cross linking agent (Triethylene Tetra Amine) was supplied by Araldite®, Petro Araldite Pvt. Ltd., Chennai.

Composite Preparation

Fabrication of Electrostatic Charging Experimental Setup

The Electrostatic Charging Experimental Setup is shown in Figure 1. The experimental setup has two main parts first one is the charging tube which has to be specially fabricated and the second one is power source which is available in the market.

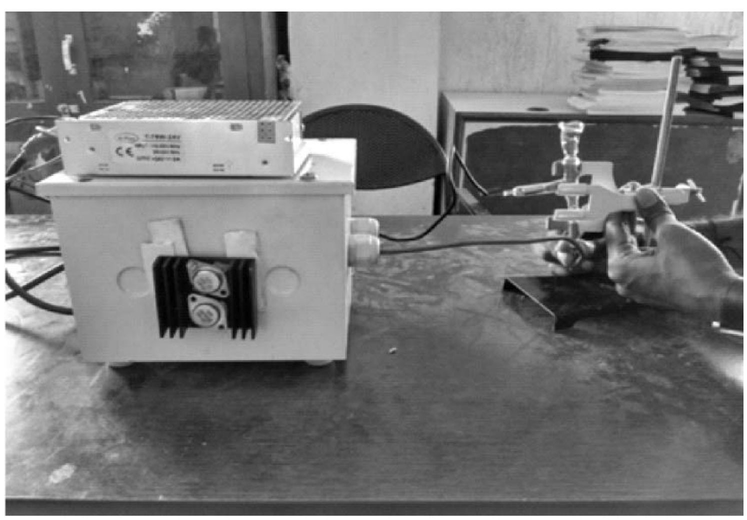

Fig. 1 Experimental Setup for Electrostatic Charging of MWCNT

The charging glass tube has metal electrode in it which act as anode and cathode, and a Glass cup which act as charging chamber for MWCNT. A 10 mm diameter lead glass tube is kept in a tube stand. At one end of glass tube cathode (glass tube with electrode) is joined using a flame torch, and the other end of glass tube is fully closed. Now anode (glass tube with electrode) is attached perpendicularly, one inch above the cathode tube. A small hole is made in the tube to vacuum out the air which is present in the tube, after vacuuming process is done the hole is sealed by applying heat. Another glass cup is attached on the closed end of the glass tube by applying heat, and this will be the place for the carbon nanotubes to get charged.

Fabrication of Polymer Nano Composites

The steps for fabrication of polymer nano composites are shown in Figure 2. In Electrostatic Dispersion Method, first the measured quantity of carbon nanotubes are electrostatically charged by placing it in the vacuum flask of the experimental set up and by the use of the power supply, the nanotubes are charged and gain the similar polarities. The charging value of the charged MWCNT can be measured by the coulomb meter.

Fig. 2 Fabrication steps for Polymer Matrix Nano composite

The epoxy resin is measured and taken in the container and the charged MWCNT is then transferred in to the container. Then the mixture of MWCNT and epoxy is stirred with the help of a mechanical stirrer for 300 mins to achieve uniform dispersion. The composites were fabricated in a mild steel mold (300 mm X 300 mm X 4 mm) by Hand lay-up technique. Silicone spray was coated with a thin layer for releasing agent in the mould. The mould was then closed with another mild steel mould and the resin was left to cure for 24 h at room temperature followed by 24 h post curing at 70 °C. The same procedure is repeated by varying the weight percentage of MWCNT in to epoxy and the specimens could be fabricated as per ASTM standards. The fine powder of uncharged and electrostatic charged MWCNT was mixed in different weight proportions of 0.9, 1.2, 1.5 &1.8 wt % in epoxy resin and the samples were fabricated.

Mechanical properties

The mechanical characterization was carried out by longitudinal tension test (ASTM D-3039). The test involves straining a test piece by tensile force, generally to fracture, for the purpose of determining one or more of the mechanical properties defined in clause. The test is carried out at ambient temperature between 10 °C and 35 °C, unless so the wise specified. Tests carried out under controlled conditions shall be made at a temperature of 23 °C ± 5 °C. The three-point bending flexural test (ASTM D-790) provides guidelines for evaluating the flexural strength and flexural toughness of polymer nano composite materials through tests under displacement control, in the third point loading configuration, of (un-notched) moulded specimens.

Surface characterization

Surface morphological studies were carried out on the fracture surfaces of the electrostatic dispersed MWCNT epoxy matrix and mechanically dispersed MWCNT epoxy matrix using ZEISS EVO MA15 SEM and voltages in the range 10–20 kV.

RESULTS AND DISCUSSION

Sem Morphology

The SEM morphology of different weight proportions of both uncharged and charged MWCNT reinforced epoxy composites are shown in following figures. Figure 3a shows that the MWCNT are more agglomerated at the right hand top corner of the image and it gradually become root structure at the middle part of the image, and then at the lower left corner of the image it shows fewer MWCNT. Figure 3b shows that the distribution of MWCNT with varying aggregate size. It also shows the better distribution of MWCNT when compared with uncharged MWCNT sample. Figure 3c shows that the MWCNT with non-uniform aggregate size. The larger aggregates of MWCNT's are distributed at the middle portion of image and smaller aggregates of MWCNT are distributed more at the outer side of the image. Figure 3d shows the MWCNT with smaller / finer aggregate size which are uniformly distributed in all areas of the sample, but voids are present due to a defect in fabrication process and this could be avoided. Figure 3e shows the MWCNT with larger aggregate size and it is agglomerated with more concentration at some areas of the sample and boundaries of the voids. The image also shows the voids present in the sample. Figure 3f shows the MWCNT with moderate aggregate sizes and it is evenly distributed at all areas of the sample. The larger number of MWNTs can be responsible for increase in roughness of the surface.The density of agglomeration is less when it is compared with uncharged MWCNT sample. Even though the distribution of MWCNTs was homogeneous after electrostatic charging process, the higher content (1.5 wt %) of MWCNTs shows agglomerationin the matrix, which could be divided into MWCNTs-rich region and MWCNTs-poor region. The lower content (0.9 wt %) of MWCNTs seems to be more homogeneously dispersed in the epoxy while the agglomeration of MWCNTs may become more serious with increase of MWCNTs. The agglomeration of MWCNTs acts like defects in the composite and the crack seems to be easier to generate and extend to the whole composite. In other words, the agglomeration of MWCNTs produces a demoting effect on the mechanical properties and the effect is enhanced with increase of MWCNTs. Therefore, the reinforcing effect is weakened and the mechanical properties decrease as the increase of the content of MWCNTs.

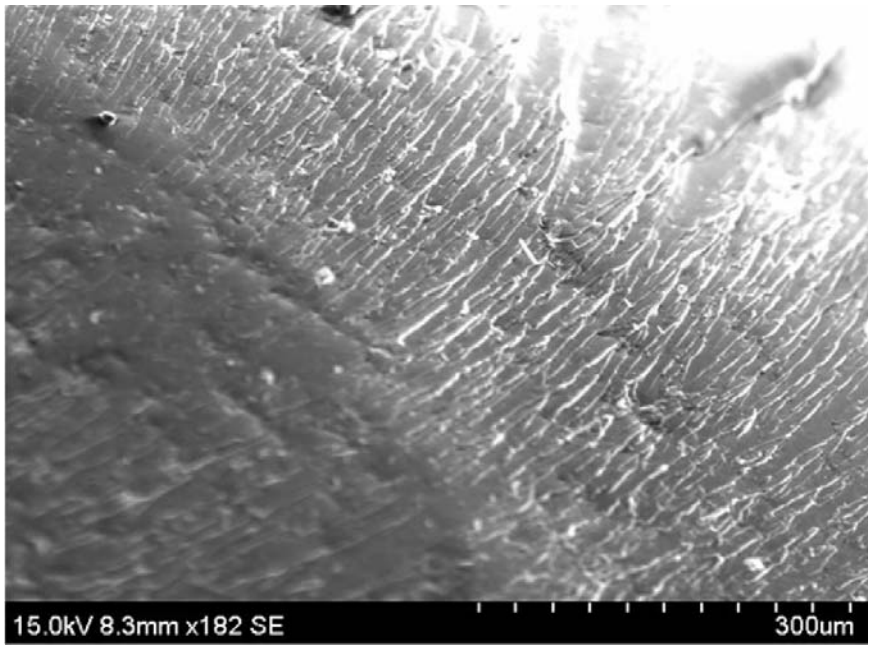

Fig. 3a SEM image of 0.9 wt % uncharged MWCNT

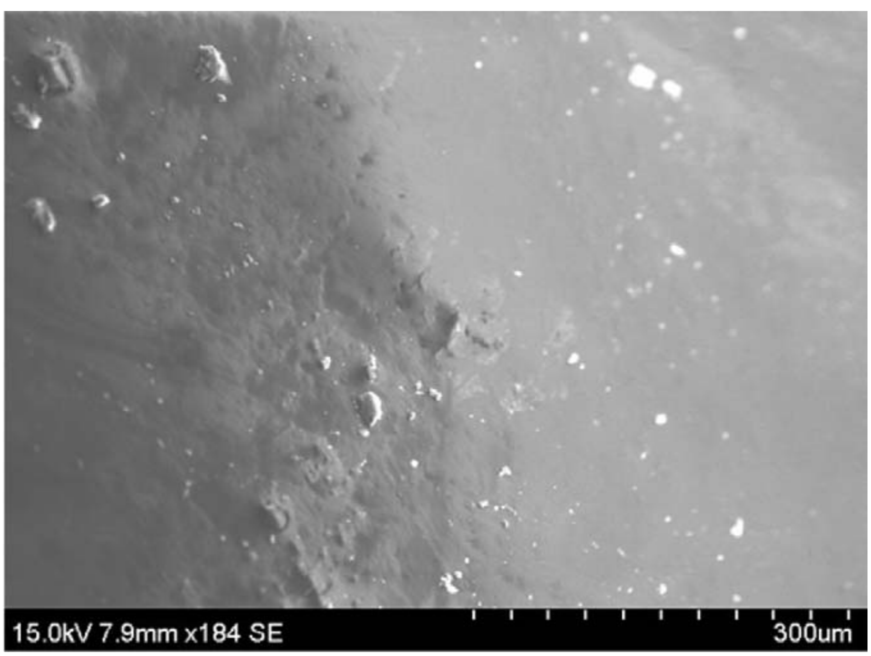

Fig. 3b SEM image of 0.9 wt % electrostatic charged MWCNT

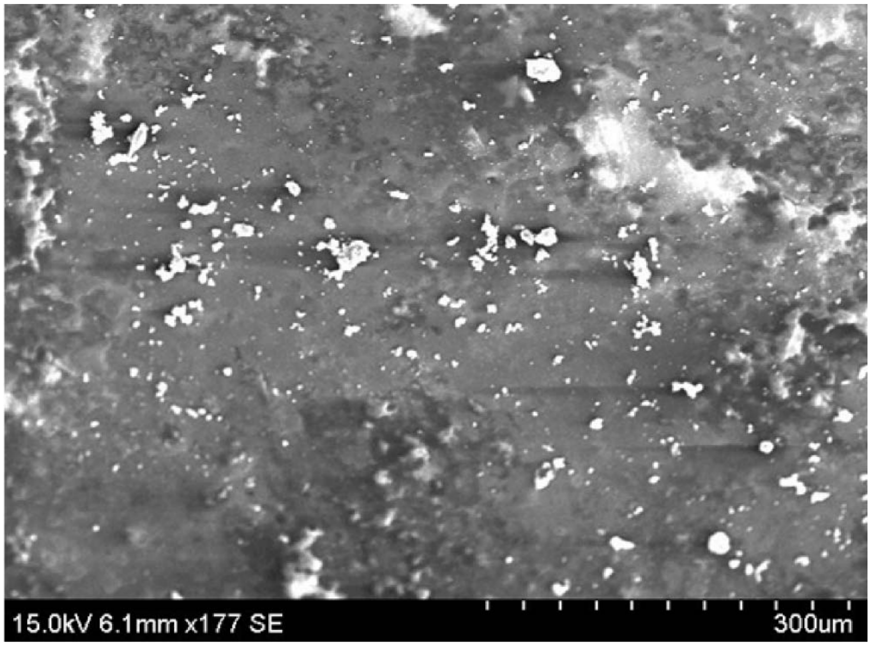

Fig. 3c SEM image of 1.2 wt % uncharged MWCNT

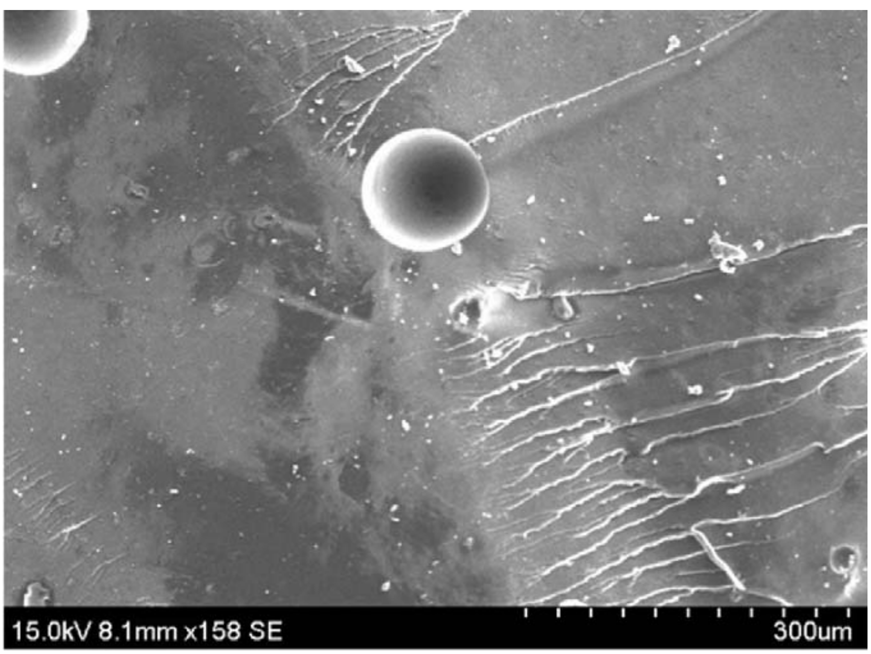

Fig. 3d SEM image of 1.2 wt % electrostatic charged MWCNT

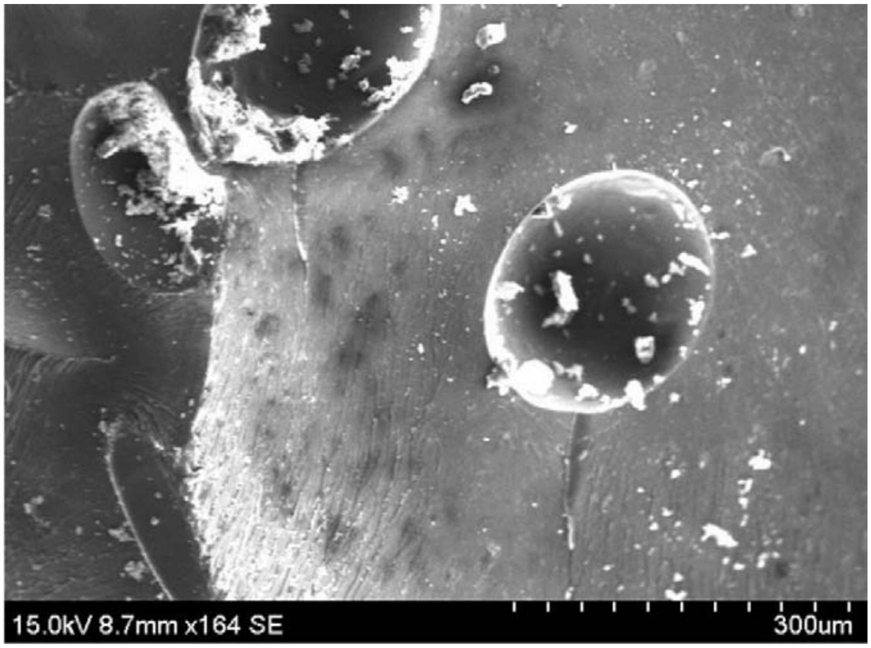

Fig. 3e SEM image of 1.5 wt % uncharged MWCNT

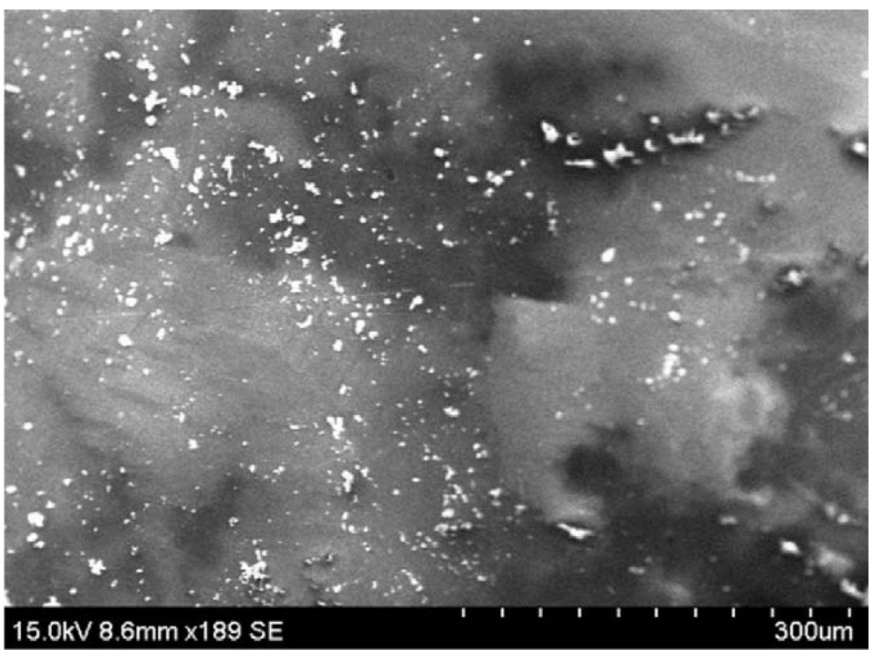

Fig. 3f SEM image of 1.5 wt % electrostatic charged MWCNT

Tensile Test

The tensile modulus of different weight percentages of MWCNT nano fillers (NF) reinforced epoxy composites are shown in Figure 4. The tensile modulus of MWCNT reinforced epoxy composites were found to be increased by increasing the weight percentages of NF and it has been restricted to 1.2 wt % NF when compared with neat epoxy (NE). The neat epoxy shows the tensile modulus of 1850 MPa and it has been increased to the maximum value of 2650 MPa when the same was reinforced with 1.2 wt % of elecrostatic charged MWCNT. This is due to the phenomenon that the electrostatically charged MWCNT could be deposited onto the epoxy substrate and mechanical bonding was formed under the double effects of electrostatic attraction and driving force of Particles. Gojny FH et al. in his theory, the reinforcement potential of the MWCNT can only be activated if there is an effective load transfer from the surrounding epoxy matrix into the MWCNT. Therefore, a good dispersion into the matrix, together with astrong interfacial adhesion has to be ensured. However, the larger the provided interface, the more difficult it is to efficiently disperse the reinforcing phase. Mean while it was found that the tensile modulus of electrostatic charged samples showed an continuous increasing trend when compared with the neat epoxy. This is achieved by good dispersion of MWCNT in to the epoxy and reduction in formation of agglomeration of MWCNT particles.

The tensile modulus of electrostatic charged MWCNT with different weight proportions 0.9, 1.2, 1.5 & 1.8 wt % was found to be increased by 9.9 %, 12.15 %, 29.80 % & 42.62 % when compared with uncharged MWCNT. Montazeri et al. demonstrated the uncharged MWCNT,

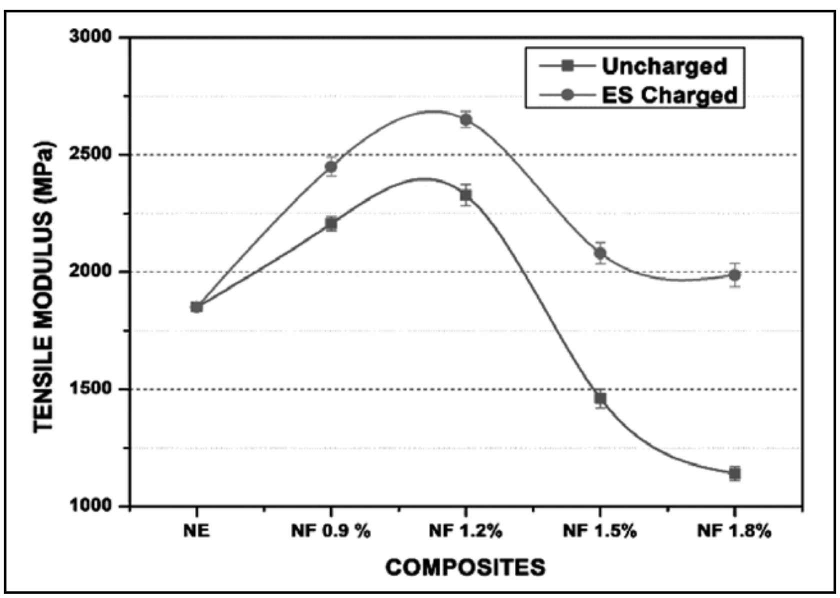

Fig. 4. Tensile Modulus of (uncharged & ES charged) MWCNT reinforced polymer composites

this was explained based on the fact that when MWCNT arepoorly dispersed the agglomerates trap polymer resin in the voids between the tubes and effectively reduce the volume fraction. The incorporation of MWCNT may lead to the improvement of other properties which are more related to the damage tolerance of then an composite or its resistance to crack initiation and propagation. It should be mentioned that the electrostatic charged mehod of dipersing the MWCNT resulted very effectively when compared with the uncharged method of dispersion.

Flexural Text

The flexural modulus of different weight percentages of nano fillers (MWCNT) reinforced epoxy composites are shown in Figure 5. The flexural modulus of different weight proportions of NF reinforced epoxy were found to be increased for both the uncharged MWCNT and charged MWCNT when compared with the neat epoxy. Manoharan et al. showed that the load transfer properties are the main issue that ensures the composite system functions properly. Since the reinforcement role of MWCNT depends on the load transfer from matrix to MWCNT, the interfacial strength between these two is of crucial importance. The cylindrical particles with smaller diameter build a stronger interface with the matrix. The highest value of flexural modulus of 2693 MPa was found with 1.2 wt % MWCNT electostatic charged sample. This is due to the fact that, when flexural load is applied to the sample, the MWCNT takes up the load and acts as a barrier in between the load and the mtrix.

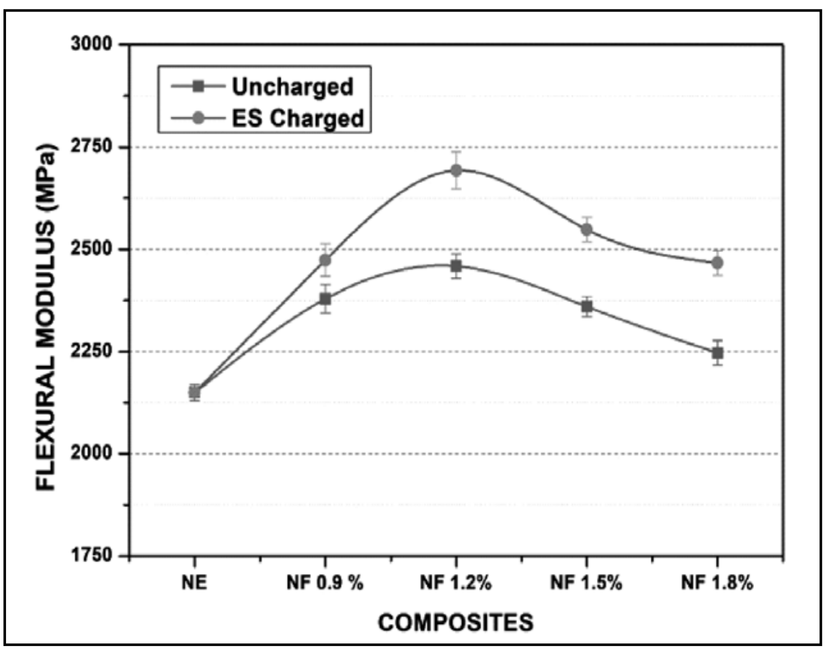

Fig. 5. Flexural strength of (uncharged & ES charged) MWCNT reinforced polymer composites.

The flexural modulus of electrostatic charged MWCNT with different weight proportions 0.9, 1.2, 1.5 & 1.8 wt % was found to be increased by 3.8 %, 8.6 %, 7.3 % & 8.9 % when compared with uncharged MWCNT process. This is because, the electostatic charged samples have their MWCNT thoroughly dispersed in all the areas of the sample and when loaded flexurally at three points, the MWCNT could be found which takes up the maximum load and then transfers to the matrix. But the above said phenomenon was found lagging in the uncharged MWCNT samples which would resulted in the lower flexural modulus.

CONCLUSION

A new novel design of experimental setup was fabricated for elestrostatic charging of MWCNT. The major problem in polymer nano composites is the formation of agglomeration which had been reduced by this electrostatic charging process. The specimens are fabricated by loading different weight percentage of 0.9, 1.2, 1.5 & 1.8 wt % MWCNT (uncharged MWCNT and electrostatically charged MWCNT) in to epoxy matrixand tested for its mechanical properties of tensile, flexural and also characterized by SEM images. The SEM images interprets that the dispersion of MWCNT by electrostatic charged process is more uniform and homogeneous when compared with uncharged MWCNT process. The tensile modulus of electrostatic charged MWCNT with different weight proportions 0.9, 1.2, 1.5 & 1.8 wt % was found to be increased by 9.9 %, 12.15 %, 29.80 % & 42.62 % when compared with uncharged MWCNT process. The flexural modulus of electrostatic charged MWCNT with different weight proportions 0.9, 1.2, 1.5 & 1.8 wt % was found to be increased by 3.8 %, 8.6 %, 7.3 % & 8.9 % when compared with uncharged MWCNT. It has been evident that the electrostatic charging mehod of dipersing the MWCNT had resulted very effectively when compared with the uncharged method of dispersion.

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