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Investigation of KH560 Coupling Agent with Carbon Fiber One Step Dipping Method Compared with Functionalized Multi-Walled Carbon Nanotubes

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ABSTRACT

Interlaminar and interfacial properties of composites are important parameters for any application, extensive research has been out bursting to enhance the above properties using nanotubes (NTs) as an alternate solution to minimize delamination of laminates. This research focused on significance of functionalized engineering materials for better efficiency in industries, especially stealth technology. Silane-coupling agent "3-glycidyletheroxypropyl-tri methoxy silane, KH-560" is one module among five modules designed in this work. Five modules are considered as (M1, M2, M3, M4 & M5) with (0.5wt % of AMINE, CARBOXYL, multi wall carbon nano tubes (MWCNTs), 5 % KH-560 treated carbon laminate and finally with base carbon fiber). PAN-based 12 k carbon fiber as base fiber with functionalized and non-functionalized nanocomposite are incorporated as fillers in the epoxy laminates fabricated using vacuum bagging technique. Findings indicated 5 % KH-560 silane treated carbon fiber (module M5) showed best results such as properties with 39 %, 55 % and 20 % in "tensile, flexural and interlaminar shear strength (ILSS)" respectively. FESEM analysis is conducted to understand morphology of the laminates.

Keywords: Carbon fiber; Surface treatment; Silane-coupling (KH-560); Delamination; Functionalized nanocomposite

1. INTRODUCTION

Polymer matrix reinforced materials with MWCNT-anchor carbon laminates is used as novel inclusion into CFRTPs. Process of esterification coupling-agent, functional Acid or flame scaling applied to form groups on carbon-fiber-coated MWCNTs. A suitable concentration with process technique for MWCNT reinforcement is suggested to enhance the ILSS and impact resistance[1]. Worked on non-treated besides treated MCNTs with nitric acid. CNTs cured with nitric acid to prevail carboxylic-functional cluster. Mixing times ranged from 24 hrs to 96 hrs with a continuous mixing speed of 2000 rpm for the epoxy resins and 0.3 %wt of multi-walled CNTs exhibits an improved in flexural strength of 17.4 % & 15.3 % during stir durations twenty-four (24) hours and ninety-six (96) hours respectively. The test results showed that treated multiwalled CNT-reinforced laminates had enhanced ILSS 14 % and 07 % greater at stir durations of twenty-four (24) hours and ninety-six (96) hours compared to control specimen[2]. CFRP has poor thermal & electrical conductivity, which restricts its application in the space industry. These are necessary characteristics for the design of space components. Tailored CNT-CFRP strengthened by 0.4 weight percent in epoxy, increasing the above said conductivities by 41 % and 500–625 %, respectively[3]. The study of CFRP changed by the inclusion of 0.1–0.5wt % single-wall carbon nanotubes and 0.5–5wt % milled carbon fibres. The addition of 0.3 weight percent of carbon nanotubes resulted in the greatest increases in tensile by "8.6 % (from 682 to 741 MPa) and the flexural by 14 % (from 649 to 740 MPa)". Shear strength of short beams enhanced by 18 %. Moreover, fatigue durability was much enhanced[4]. This study emphasizes properties of CNTs resin-matrix composites. Electrophoretic deposition, which was used to introduce CNTs, barely damaged the carbon fibres. Tensile tests were performed to see how mechanical characteristics changed after CNTs were deposited. The outcomes are evident after CNTs were deposited as interface, tensile strength by 9.86 %, failure strain by 44.01 %, and Young's modulus rose 12.4 % respectively. "Nitric-acid" treatment used to in reinforcing Possibilities of CNTs, and it was shown that this increased the material's tensile force, strain at failure, & young's modulus, respectively by 35.70 %, 21.70 % and 70. %[5].Novel applications of composite assemblies are examined replacing metals on robotics, mechanism and vehicles are the major parameter without effecting its stiffness design issues and made its mark to be inevitable in aero, defence, auto streams and other fields out of their best properties as highest specific strength to weight ratio, resistance to corrosion and modulus etc[6]. Graphene oxide/silica (GO/SiO2) laminas are fabricated with CFs. These are compact & uniformly spread on surface by monitoring assembling periods, experimental results showed increased surface roughness & wettability of CFs, significantly boost interfacial strength some of values are (IFSS, ILSS and flexural strength improved by 86.1 %, 89.3 % and 30.4 %) respectively[7].Discussed different "wet', 'dry' and

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'multi-scale" surface alteration techniques of fiber, i.e. "sizing, plasma, chemical treatment carbon nanotubes/nanoparticles coating", for improving the wettability and interfacial grip with polymeric matrices[8]. Introduced in-situ amalgamation of g-C3N4 with carbon-fibers proved to better roughness within the laminates. The face energy of lamina observed to increase by 67.81 %. ILSS and IFSS of composite laminates found increased from 51.84 to 72.09 MPa and 44.62–73.41 MPa, respectively[9]. Composites are mainly replaced over conventional materials due to its light weight, durable, custom design flexibility, and reinforcement. The output of the prepared laminate using different combination of both reinforcement and matrix ensures the load bearing characteristics, Surface sizing, and hybrid reinforcement drawing attention because of its excellent interfacial of composite[10]. Strength of the fibre and adhesion capability of the matrix solely responsible for any sort of interphase issues while dynamic or static failures[11]. Carbon-fiber epoxy composite (CF-EP) with 0.3wt % CNT laminate fabricated with vacuum-assisted resin infusion method (VRIM) found good response in both tensile along with ILSS by 17 % & 28 % respectively. Further, tested for delamination factor which proved to be 26.31 % more than the Pure Carbon-fibre[12]. MWCNTs reinforced with 0.4 weight percent epoxy improve heat and electrical conductivities by 41 % and 500-625 %, respectively. On MWCNTs hybrid, direct electroplating without surface activation is possible due to an increase in electrical conductivity, whereas this is not possible with neat CFRP[13]. Resin pre-coating (RPC) and three silane coupling agents' coating (CAC) procedures were also applied to porous Ti substrate surfaces. According to SEM pictures, nano-cavities have been produced to increase the contact area and vertical volume on the Ti substrate surface, which have then been completely coated with resin through RPC. The results show that specimens treated with "anodizing + RPC" have the best average shear strength of 20.73 MPa, which is at least 63.0 % greater than specimens coated with silane KH-550/560/792 and increased by 31.7 %[14].

2. METHODOLOY

The base material carbon fiber 12K PAN with no twist from Toray Advanced materials-korea Inc is mentioned in Table 1 and Table 2 epoxy resin Araldite LY-556 from Huntsman with Aradur HY-951. Silane agent "3-glycidyl ether-oxy-propyl-tri-methoxy-silane (KH560)" and three different types of functionalized nano fillers/tubes such as (Amine, Carboxyl and MWCNTs) from Adnano Technologies is used for study. The table below contains a list of the materials and their specifications.

Table 1. Properties of Carbon fiber

Fiber K12-T700SC-NoTwist	Properties
Tensile strength (Mpa)	515
Tensile modulus	234
Elongation %	2.2
Density103 kg/m^2	1.79

Table 2. Specifications of MWCNTs

Nano fillers	Properties
Purity	~99 %
Diameter	~5-15 nm
Length	~10 µm
Surface Area	260 m2/g

2.1 Fabrication

Five modules of carbon laminates are considered in this research each laminate designated as shown in table.3, Module-1 neat carbon laminate followed by (0.5wt % of Amine, carboxyl, MWCNTs, and 5 % KH-560-treated carbon fibre respectively till module-5. Multi-walled carbon nano tubes with agglomeration primarily have treated with sonication process to convert deagglomeration state by using systematic procedure shown in fig.1.

The process of deagglomeration starts with ultrasonic of 0.5wt % nano particles with 100 ml of acetone to make

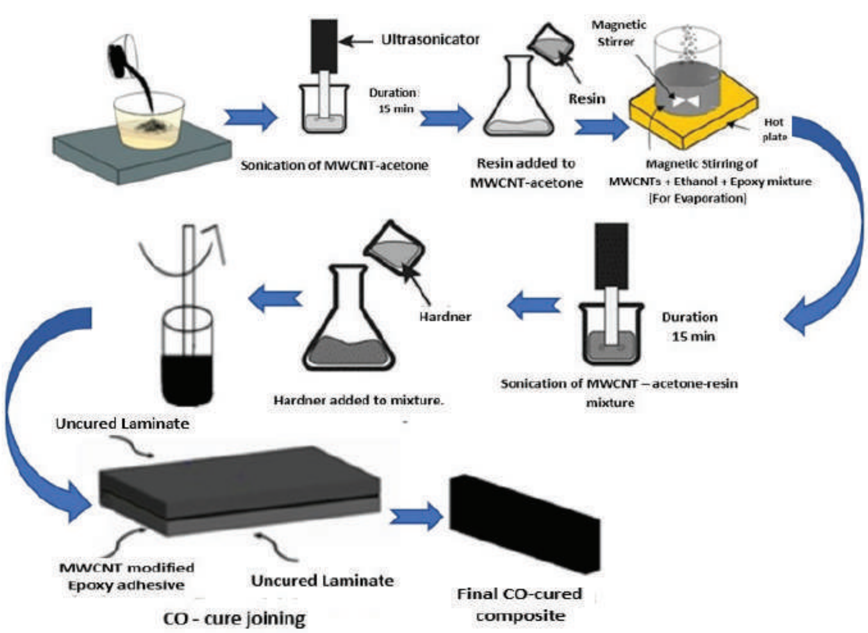

Figure 1. Sonication process (Deagglomeration MWCNTs).

homogenized dispersion of MWCNTs in resin for 15 minutes of sonication (40 Hz, 700 W).

Table 2. Samples and its Specifications

Module	Material	Nano material/ Chemical	Added (%)
M1	Neat carbon laminate	Not applicable	00
M2	Carbon fiber 12K	Amine	0.5 Wt
M3	Carbon fiber 12K	Carboxyl	0.5 Wt
M4	Carbon fiber 12K	MWCNTs	0.5 Wt
M5	Carbon fiber 12K	KH-560	05

2.1.1 Chemical Treatment Process (KH560)

Surface /Chemical treatment of lamina is a novel and highly recommended process for modifying the wettability of layers which technically leads to apply pressure of van der Waals tends to have better bonding of the laminate ultimately provides improved properties compared to neat fiber or even nano particles impregnated laminate and the process shown in fig 2 and fig 3. The next stage nanofillers with acetone suspension mixed into pre weighed amount of epoxy resin followed by magnetic stirring process carried at 70-degree Celsus with 4000 rpm for acetone to get evaporated. The epoxy/MWCNT suspension introduced 10:1 ratio of hardener and then stirred for 10 min followed by the final prepared suspension for preparing the composite laminate using a vacuum bagging for curing period about 24 hours at room temperature. carbon/epoxy/ MWCNT nanocomposite laminate obtained was 3.5 mm. The same process of vacuum bagging technique is used for other modules (M2 &M3) of modules. 5 % silane-chemical treated carbon fibre is prepared using vacuum bagging by considering respective procedure of fabrication and are explained in Fig. 4. Each lamina after trimming has soaked for 30 hours and then cleaned every lamina with utmost care for removing all the chemical agent over the surface of the lamina at least 3 times and then dried it for 3 hours in furnace maintaining 50 °C in order to make sure it completely free from wetness, followed by each lamina is placed one over the other

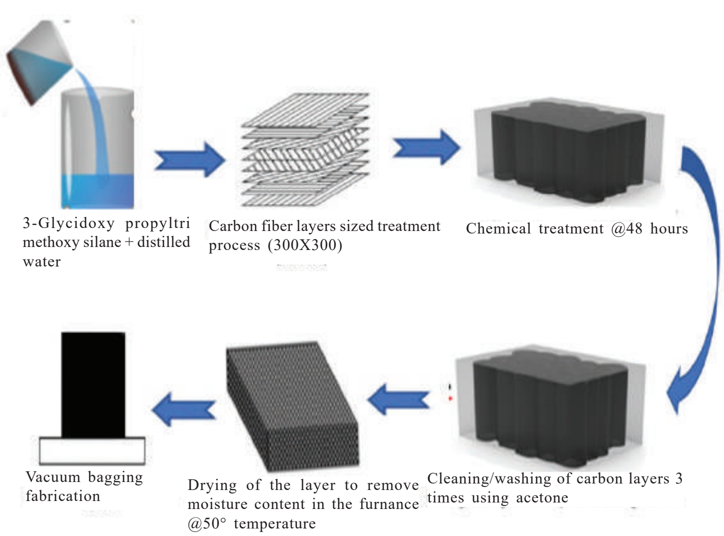

Figure 2. Surface treatment process.

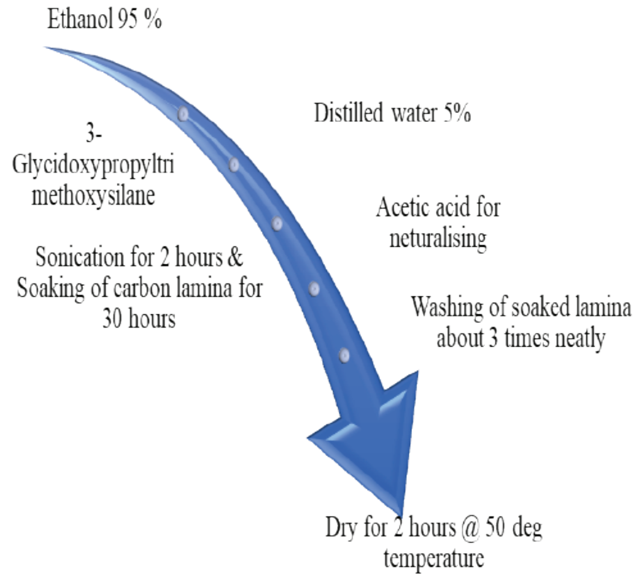

Figure 3. Process for chemical surface treatment.

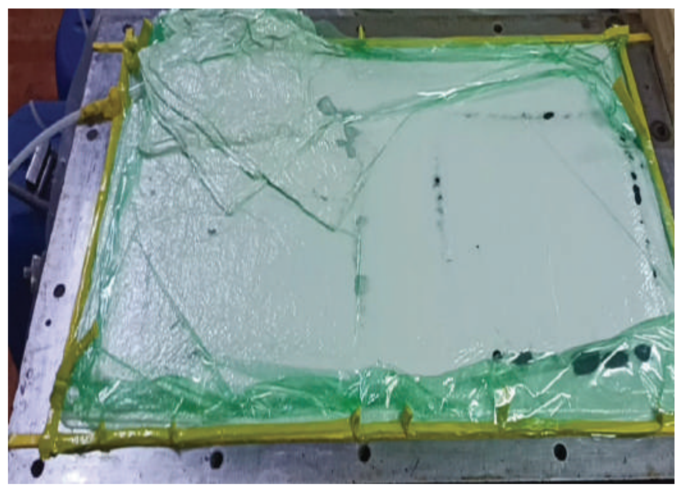

Figure 4. Laminate preparation by vacuum bagging.

with matrix reinforcement using vacuum bagging technique for preparation of final composite laminate.

From Fig. 4(a), it shows the fabrication process of laminate using vacuum bagging technique which strictly follows a sequence of procedure like initial laminas are trimmed, measured for the dimension followed by calculation of number of laminas required as per the requirement of ASTM testing and lamina placed one over the other with binders in between, later the process of vacuum starts till the laminate is prepared/ solidified.

3. RESULTS

3.1 Tensile Test ASTM D 3039

From Fig.5 samples (M1, M2, M3, M4 &M5) are shown with (25 mm *250 mm) are placed in the ultimate tensile testing machine and secured with fixtures firmly. Each specimen progressively applied with the load until it breaks, and the values are automatically recorded with respective values of each module. From Fig. 6 results are very clear and concludes the best laminate with respective to the test performed as per the ASTM standards. Compared to all modules, module M5

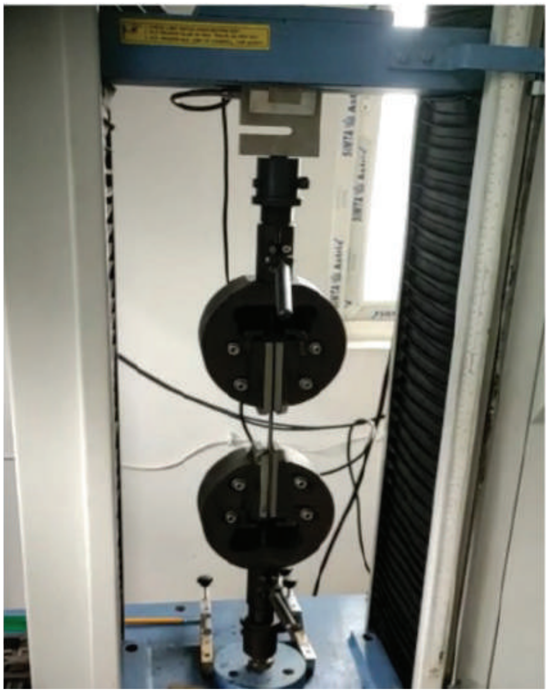

Figure 5. UTM-tensile test.

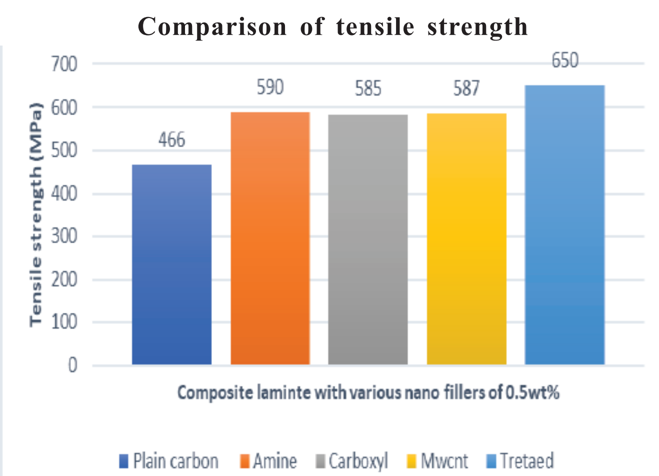

Figure 6. Tensile testing.

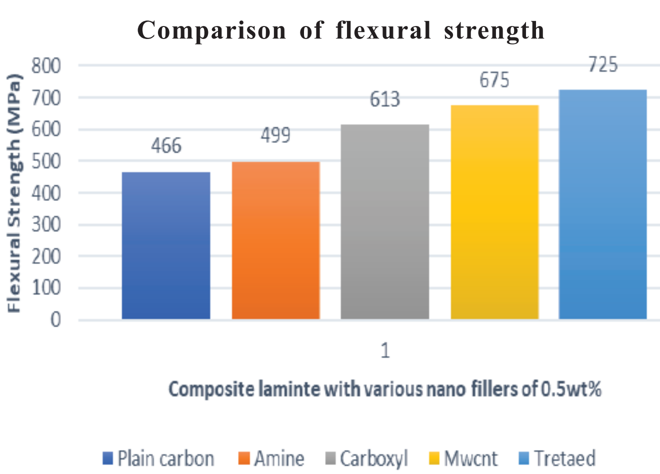

Figure 7. Flexural testing.

chemical treated composite laminate showed the best tensile strength with almost 39 % more.

3.2 Flexural Tests ASTM D790

The load-deflection plots of the all five modules varying different functionalised MWCNTs along with chemical treated laminate are tested using three-point bending experiments values shown in Fig. 7 and the results are evident surface treated laminate has made remarkable by showing the highest among all other modules (M1, M2, M3, M4 &M5).

3.3 Interlaminar shear strength (ILSS) ASTM D2344

Module(M5) surface treated laminate along with all other modules are tested with ASTM standards using UTM (universal testing machine) and the response are collected and determined shear response plotted in fig 8. The maximum shear strength 35MPa has observed followed by 33MPa from functionalised carboxyl-Mwcnts and remaining responses are plotted.

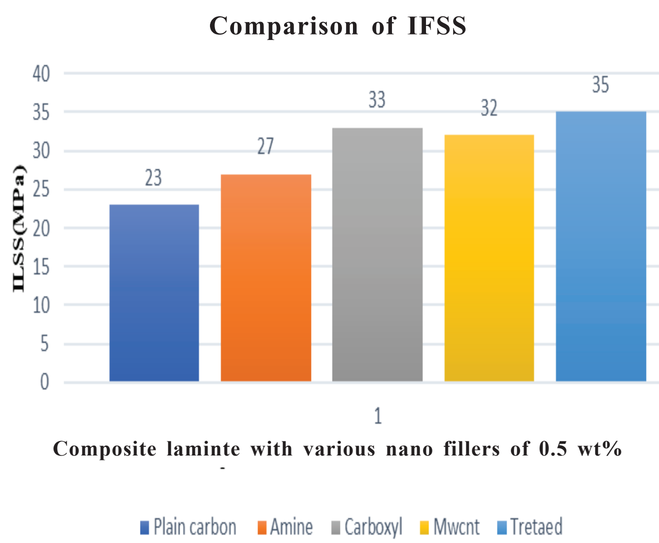

Figure 8. ILSS comparison on all modules.

3.4 Fractography

Post mechanical testing failure samples used for surface morphology. FE-SEM technique revealed reinforcement is outstanding between fiber/epoxy, but more amount of epoxy resin has accumulated at a place made the area smoother and it led to carbon fiber pull-out. Studied all the fractured surfaces specimens in fig. 9 represents fractography of all the module. Functionalised nano particles and epoxy resin matrix at fracture point of test samples are concentrated with (a,a1) 100µm and 50 µm magnification to identify level of its bonding and particles in all modules (M1, M2, M3, M4 &M5).

4. CONCLUSIONS

The current research describes novel approach of surface treatment of lamina individually before fabrication all modules (M1, M2, M3, M4 &M5) using vacuum bagging technique with various functionalised MWCNTs with 0.5wt % and matrix PAN based 12k Carbon fiber reinforcement to investigate the effect of mechanical properties comparing with surface treatment (silane coupling agent KH-560) of plain carbon

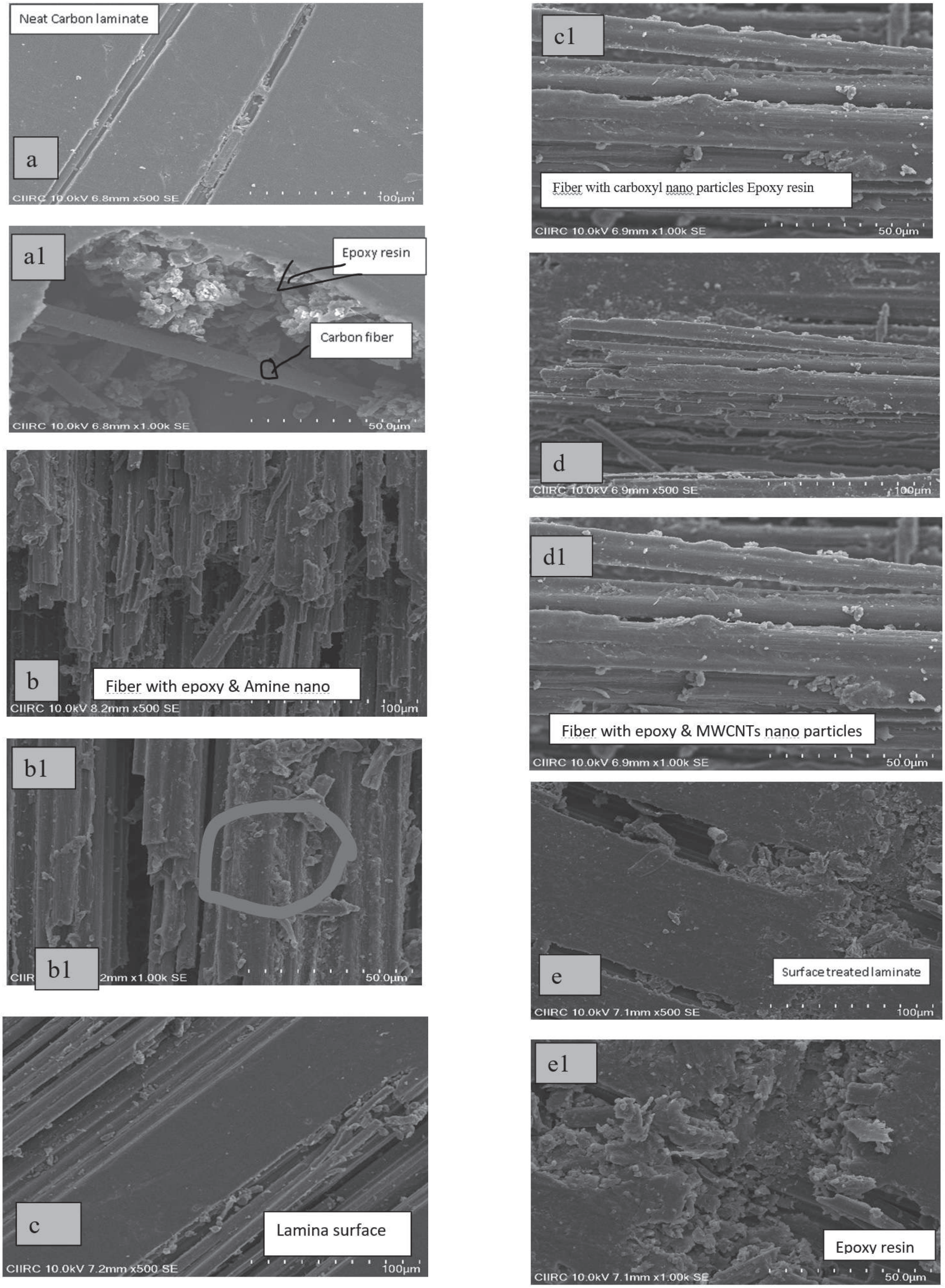

Figure 9. FE-SEM images of: (a) Neat carbon fiber; (b) 0.5 Wt % Amine; (c) 0.5 Wt % carboxyl; (d) 0.5 Wt % Mwcnts and (e) 5 % KH560 Treated Carbon fiber.

fiber. FESEM analysis is performed to study the morphological characteristics of the laminates and are explained as follows: -

0.5wt % functionalised MWCNTs integration along with KH560 silane coupling agent (surface treatment) composite laminates are fabricated using Vacuum bagging technique and are designated as five modules (M1, M2, M3, M4&M5).

Surface treated agent (KH-560) showed as effective modification technique for good interphase of composite laminate indicating (high tensile strength, flexural and inter laminar shear strength with 39 %,55 % and 20 %) respectively.

FE-SEM images show bonding of fiber/resin fig.9. The magnification of 10 and 50 μm describes a clear understanding of fiber and resin content in the laminate.

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CONTRIBUTORS

Mr Soppari Bhanu Murthy is a research scholar from Osmania University. His research area of interest is Composites and robotics.
In the current study, he has contributed from studying literature and found out the gaps developed a new method of surface treatment for any lamia, fabricated the sample as per ASTM standards and finally development of enhanced mechanical properties required are achieved.

Dr N. Kishore Nath obtained PhD degree and presently working as Scientist 'G' & Project Director-VEDA, in DRDO-ASL.
His contribution in the current study include from the raw material till the completion of work everything has been suggested and monitored.

Dr P. Ramesh Babu obtained PhD degree from IIT - Kharagpur and working as a Senior Professor, Osmania University Hyderabad. Area of interest -FEA, MCM, CAMDA, CIM, Machine drawing, material science and metallurgy.
For this current study, he is co guide for my project. His contribution in this work is guided in all aspects from choosing of material to machining process and writing the research paper.