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Effect of Surface Treatments on Mechanical Properties of Carbon Fiber Reinforced Friction Material

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ABSTRACT

Mechanical properties of a friction material depend mainly on the interfacial adhesion between all the ingredients of a friction material. In this work, a new friction material is developed by combination of carbon fiber (CF), polymer matrix and other ingredients. The surface of CF is chemically inert and hydrophobic in nature and does not possess good bonding behaviour with respect to polymer matrix. Therefore, an attempt is made to improve the bonding behaviour of CF surface with polymer matrix and remaining ingredients of a friction material. CF surface is modified by three different surface treatments to increase hydroxyl or carboxyl groups on the surface. First, surface oxidation treatment, Second nitric acid treatment and third grafting multi walled carbon nano tubes functionalized (MWCNT-F) on CF surface. CF content after surface modifications is varied in (2-5wt %) and mixed with remaining ingredients of a friction material. Composite sheets are fabricated by using hand layup method. The resulting materials are characterized by SEM, TGA and FTIR analysis. MWCNTs-F on CF surface is observed. Twelve composite sheets with varying content of CF and surface treatment method is fabricated. Sample specimens are cut according to ASTM standards to evaluate all the mechanical properties of the composite. It was observed that, surface treatment method (MWCNT-F) grafting on CF surface possess good mechanical properties compared to other formulations. The selection of ingredients also plays an important role for achieving better mechanical properties.

Key words: Multi walled carbon nano tubes; Carbon fibers, chemical grafting and Mechanical properties

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1. INTRODUCTION

Brake friction material present in an automobile converts the kinetic energy of the vehicle to thermal energy by means of friction generated at the contact surface between brake disc and pad. The rate of wear observed in the friction materials depends mainly on the type of friction material used , pressure applied on the pads ,friction material temperature, friction material contact area, friction material surface finish, heat removal rate, squealing propensity, fade resistance and ability to operate over various atmospheric conditions of the automobile. In addition to all these factors, the other factors that affect the friction development are weight transfer, tire and road conditions, and dimensions of brake drum, engine size and drive train gearing [1]. Recently, organic based friction materials with carbon fiber as main reinforcement are widely used in automobile racing cars and aero space industry because of its excellent mechanical and tribological properties. These friction materials possess less wear rate and low vibrations in the braking system of an automobile. CF is primarily preferred for friction composite materials due to its excellent properties, such as high specific strength and stiffness, thermal stability, corrosion resistance, high tensile modulus, self lubrication, lower density, good electrical conductivity, lower linear coefficient of thermal expansion and outstanding fatigue characteristics. They are also find applications where high damping chemical inertness and superior wear resistance are important.CF reinforced composites are widely used in pressurized cabin system of an aircraft to sustain the flammability resistance of aircraft, landing gear doors, floor panels, fan ducts etc. These fibers are also find applications in automotive sector like drive shafts, valve guides, racing brakes, train brakes and clutches etc [2-6]. The performance of carbon fiber reinforced friction material depends mainly on the selection of ingredients for the friction material and interfacial adhesion between all these ingredients [7]. Although, may research works are done from the past to increase the fade resistance, wear resistance of the brake friction material, But, only few works are concentrated on the bonding strength of the friction material. The interfacial properties of the CF, ingredients and polymer matrix can be improved by modifying the surface of CF by introducing hydroxyl or carboxyl groups on the surface. Therefore, many efforts have been carried out from the past to improve the surface

properties of CF by using different treatment techniques like sizing, plasma, chemical oxidation, γ-ray irradiation, electrochemical, dip coating, MWCNT grafting on carbon fiber surface by CVD etc [8]. Among the above mentioned various techniques to improve the interfacial adhesion between fiber and matrix, MWCNT grafting on carbon fiber surface under chemical vapour deposition (CVD) achieved good bonding strength between polymer matrix and CF surface. Qiang Song et al [9], Observed that grafting straight carbon nano tubes radially on carbon fiber surface has improved its mechanical properties. The mechanical properties such as compressive strength and inter laminar shear strength of the composite are increased by 275% and 138% compared to pure C/C Composite. S.P. Sharma et al [10] identified that, coating carbon fibers by using CVD on its surface improves its tensile strength by 69% for CF/epoxy/amine polymer matrix composites. Hui Qian et al [11], modified the carbon fiber surface by grafting carbon nano tubes by CVD and observed that , there is 26% increase in inter laminar shear strength for carbon fiber and poly methyl methacrylate (CF/PMMA) composite. But, grafting CNTs on CF surface by using CVD technique is a costlier process and proper care has to be taken to control operating temperatures. Although, grafting CNTs by CVD on CF surface achieved good results, but due to complexity of operating parameters and cost involved, it cannot be used widely in all the fields of engineering to a greater extent. Hence, in this work, an attempt is made to improve the interfacial adhesion between CF, polymer matrix and ingredients by modifying the CF surface using three chemical methods namely oxidation, HNO_3, and MWCNT-F grafting on CF .Friction composite sheets are developed to evaluate mechanical properties of all the specimens [12-14].

2. MATERIALS AND METHODS

2.1 Carbon Fiber

Polyacrylonitrile (PAN) based chopped carbon fibers were used for this study. CF used in the present study consists of 95% of CF in order to obtain good mechanical properties when mixed with remaining ingredients. This is shown in fig 2.1. The properties of chopped carbon fiber are given in table1.

Fig. 2.1 Chopped carbon fiber

Fig. 2.2 MWCNT Powder

2.2 Multi Walled carbon nano tubes (MWCNT)

Multi walled carbon nano tubes (MWCNT) used in the present study is produced by using chemical vapour deposition method (CVD) shown in fig 2.2. The properties of MWCNT supplied by the supplier for this study are given in table 2. Chemicals such as NaOH, HNO_3, H_2SO_4, acetone solutions used in the present study is procured from chemical laboratories, Visakhapatnam, India.

2.4 Carbon Fiber Surface Treatment Methods

Surface treatment methods are performed on CF to improve the surface roughness and decrease chemical inertness of the surface by introducing carboxyl or hydroxyl groups on the surface. The following three treatment methods are performed to modify the CF surface.

2.4.1 HNO_3 Treatment

Carbon fibers (CF) of quantity 180 gm were modified by dipped in a solution of 40% HNO_3 i.e. (200ml of HNO_3 & 300ml of distilled water) for 48hrs at room temperature. The fibers were allowed to immerse completely in the nitric acid solution and distilled water. Few gases will generate during mixing process of (CF+HNO_3 +distilled water). CF surface was diluted with distilled water of 3lit qty. This process is repeated for 3 times to remove HNO_3 liquid gases adsorbed on CF surface. Finally, CF surface is immersed in NaOH solution of 10% qty (300ml of distilled H_2O +30 gm of NaOH) and allow the sample to completely soak for 30 min. Then, CF surface is diluted with distilled water of 2 L quantity to remove left over acid gases adsorbed on its surface. This process of dilution, initially with HNO_3 and finally with NaOH makes the surface completely free from adsorbed gases on CF surface. Collected carbon fibers after complete dilution process were allowed dry in an oven at a temperature of 150 °C for 2 hrs followed by drying in atmosphere for 1hr. The sequence of operations carried in HNO_3 treatment is shown in fig 2.4(a).

2.4.2 Surface Oxidation treatment

Chopped carbon fibers collected in the crucible are subjected to surface oxidation treatment in an oven at 600 °C for 1hr. First, temperature is increased from 20 °C to 600 °C for 30 min under nitrogen gas and in a second step temperature is kept at 600 °C for remaining duration time of 30 min under air.

2.4.3 MWCNT-F grafting on CF

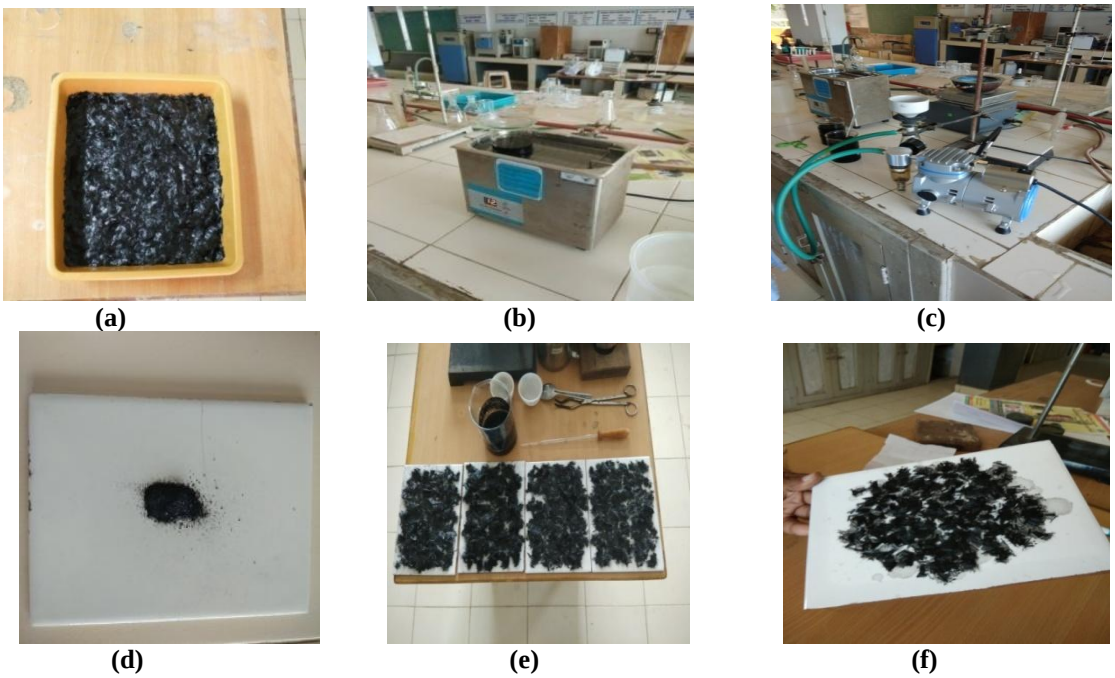
Multi walled carbon nano tubes (MWCNTs) are treated to attach carboxylic acid groups on their surface to form functionalized MWCNTs called MWCNT-F. In this method 2gm of MWCNT are added to 100 ml of concentrated sulphuric acid and 35 ml of nitric acid (Purity of 98.08% H_2SO_4 and 70% HNO_3) (3:1 by volume ratio). The mixture is sonicated in a bath for 3 hrs at a temperature 70 °C. Now, the reaction mixture is diluted with 200ml of deionised water followed by vacuum filtration process using a filter paper of 2µm porosity. This dilution process is repeated for three times to remove the metal particles adsorbed on MWCNTs. Then, the sample is allowed to dry in an oven at 100 °C. After drying operation, the collected MWCNT powder particles are immersed in 40 ml of acetone solution and placed on a ultra sound bath sonicator to allow the acetone solution to completely mix with MWCNTs. During sonication process, acetone gases will escape to the atmosphere because of having low boiling point of acetone i.e 30 °C. Now the collected sample powder is dried in oven at 100 °C for 4 hrs followed by drying in atmosphere to form MWCNT-F powder. This process will completely removes metal particles present in MWCNT powder to use MWCNT-F more effectively in polymer matrix composites. Multi walled carbon nano tubes functionalized (MWCNT-F) of qty 1.5 gm obtained after filtration and drying operation is dispersed in a 20 ml of acetone solvent using an ultra sound bath at 70 °C for 1 hr. Complete mixing operation is carried out using a ultra sound bath sonicator. Now, chopped carbon fibers are placed on a glass substrate and suspension containing MWCNT-F is deposited drop by drop using a droppler over the entire surface of carbon fiber. The deposition operation is repeated several times with evaporation of the solvent between each deposition. Heat treatment process is carried out on the resulting CFs grafted MWCNT-F at 100 °C for 2 hrs. Fig 2.3 (b-f) gives the sequential operations carried in grafting MWCNT-F on carbon fiber surface.

Table -1 Properties of carbon fiber

Material	**Properties**						
	Diameter (µm)	**Length (mm)**	**Tensile strength (MPa)**	**Tensile modulus (GPa)**	**Sizing**	**Resistivity Ω/cm**	**Carbon content**
Carbon fiber	6.9	6	4810	225	1-1.2%	$1.54x10^{-3}$	95%

Table -2 Properties of multi walled carbon nano tubes

Material	**Properties**						
	Diameter (nm)	**Length (microns)**	**Metal particles**	**Amorphous Carbon**	**Specific surface area(m^2/g)**	**Bulk density g/cm^3**	**Nano tubes purity**
MWCNT	10-30	10	<4%	<1%	330	0.04-0.06	>95%



(a) **(b)** **(c)**

(d) **(e)** **(f)**

Fig. 2.3 (a-f) Surface treatment methods performed on CF

3. PREPARATION OF COMPOSITE SHEETS

Preparations of friction composite sheets are carried out by using hand layup process. CF after surface treatment method is mixed with other ingredients using a foculator for 30 min for each sample sheet. Initial step of fabrication involves preparing a die with the dimensions of 42cmx22 cmx1.2 cm made of mild steel material. The die is finished to remove the unwanted material of thickness 2cm on all the sides by gas welding operation. Initially resin is taken in a foculator and all the ingredients are added slowly one after the other with an interval time period gap of 10 min. Total mixing operation for all the ingredients was carried for 30 min for each sample sheet. Twelve composite sheets with variation of surface treatment performed on CF are fabricated. Samples are cut from the friction composite sheets as per ASTM standards to evaluate mechanical properties of all the specimens. The details of ingredients

Selected for fabrication of composite sheets are given in table 3 and the sequence of operations involved in fabrication of friction composite sheets is given in fig 3.

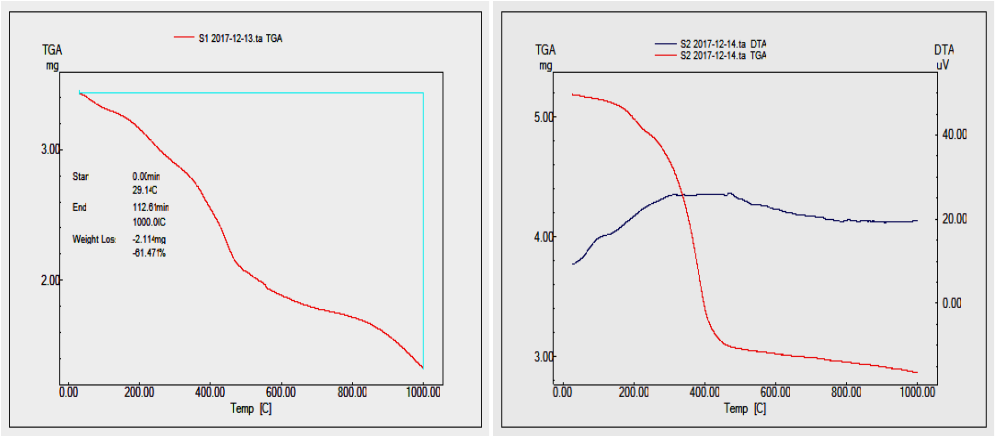
Fig. 3 Sequence of operations performed in fabrication of composite sheets

Table -3 Material Selection

Ingredient	Vol %
Phenolic Resin	70
Carbon fiber	2-5% (S2,S3,S4,S5)
Zirconium silicate	15
Graphite Powder	1.5
Barium Sulphate	2.5
Rubber Powder	3
Molybdenum disulphide	Balance

4. THERMO GRAVIMETRIC ANALYSIS (TGA)

A thermo gravimetric analyzer continuously measures mass while the temperature of a sample is changed over time. Mass, temperature, and time in thermo gravimetric analysis are considered as base measurements while many additional measures may be derived from these three base measurements. The present analysis is performed on all the specimens powder samples collected from the friction composite. The temperature is increased slowly from 0 °C to 1000 °C for an ending time period of 112.6 min. The oxidative mass loss is continuously observed with increase in temperature. It was observed that the specimen S5 with (MWCNT-F grafted on CF wt5%) possess low weight loss compared to other formulations of the material (i.e. 2.11 mg). The optimum selection of ingredients for the sample S5 (MWCNT-F grafted on CF wt5%) able to sustain high temperatures easily and there is no considerable mass loss is observed after TGA analysis. Fig 4 gives TGA performed on sample S5 (MWCNT-F grafted on CF wt5%) of sample powder with increase in temperature.

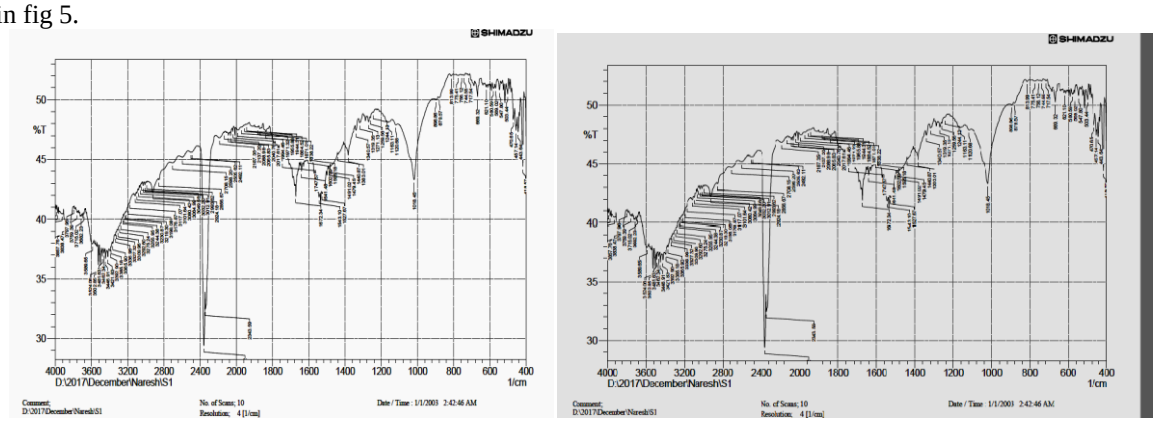


(a) **(b)**

Fig. 4 Thermo gravimetric analysis on sample S4 and Sample S3

5. FTIR ANALYSIS

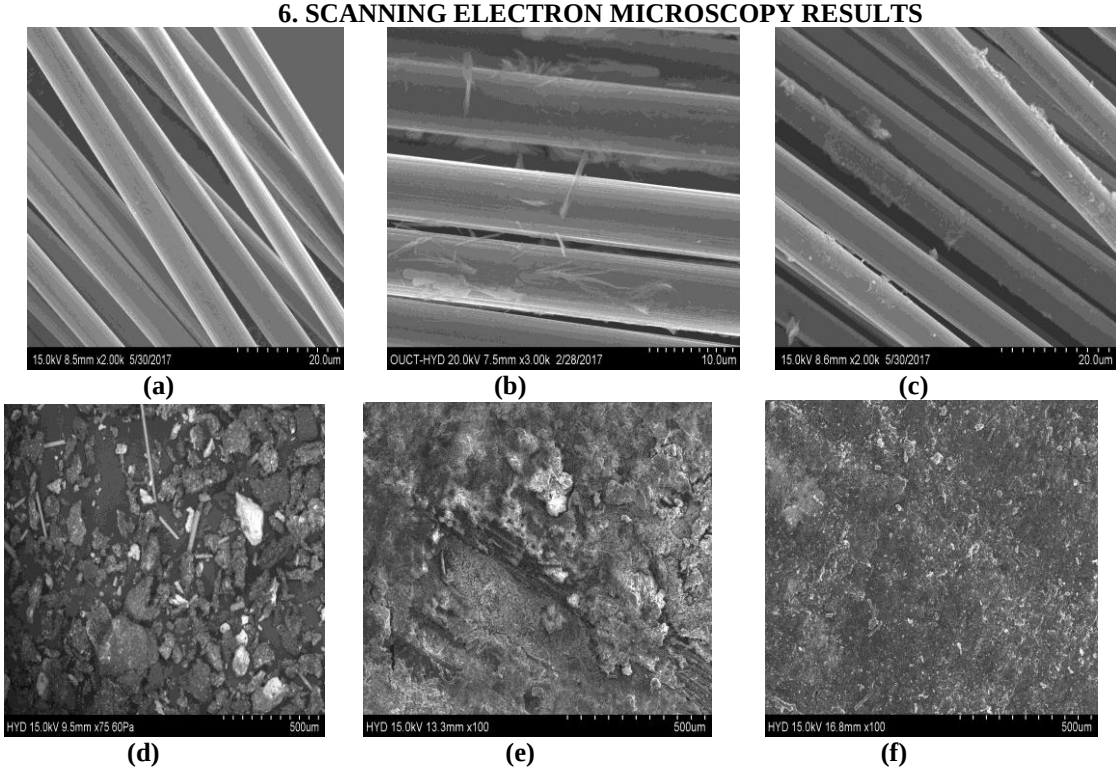
FTIR is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high spectral resolution data over a wide spectral range. This converts the raw data in to the actual spectrum. The main aim of FTIR is to observe how well the sample absorbs light at each wavelength. FTIR spectroscopy of make shimadzu is used for this purpose. All the samples are subjected to infrared monochromatic light and intensity of peaks at each wavelength varying from 4000cm^{-} to 400 cm^{-} is observed. It was observed that, the intensity of peak begins at 44.416 and stops at a value of 40.824 for the sample S5 (CF wt5%) for CF grafted with MWCNT-F. The intensity of peak value is having fewer fluctuations with increase of wavelength and the variation between all the values of intensity of peaks is very less. The inter laminar cohesive forces between all the ingredients are greatly improved after surface treatment methods, especially with grafting MWCNT-F on CF surface. The sample specimen S5 (CF wt5% and CF grafted by MWCNT-F) possess good bonding strength with resin compared to other formulations of the material. The absorption spectra and intensity of peaks with respect to wavelength for sample S5 is given in fig 5.



(a) **(b)**

Fig. 5 FTIR analysis on sample S4 and S3

6. SCANNING ELECTRON MICROSCOPY RESULTS



(a) **(b)** **(c)**

(d) **(e)** **(f)**

Fig. 6(a-f) Surface treated fiber and composite sheets

SEM images presented in Fig 6(a), Fig 6(b) & Fig 6 (c) demonstrate the three different surface treatments performed on CF. The grain distribution and uniformity of each fabricated composite specimen is observed from SEM images. Fig 6(a) represents the CF surface subjected to oxidation treatment. It was clearly observed from fig. 6 (a), that after this

treatment method, the chemical inertness of the CF surface is not greatly improved. This can be visualized in fig. 6(a) with shiny colour CF. Scanning electron microscopy images are also taken on CF treated with HNO_3 treatment and MWCNT-F on CF surface. It is observed from fig. 6(b) that, a little improvement in the surface roughness takes place .But, MWCNT-F grafted on CF surface shown in fig. 6(c) exhibits high surface roughness and great improvement in the chemical inertness of the surface. SEM images are also taken on composite sheets having CF treated with (surface oxidation, HNO_3 and MWCNT-F grafting on CF). It was observed from SEM image fig. 6(f) that, the composite specimen containing CF grafted with MWCNT-F exhibits high bonding strength and good inter laminar attractive forces between all the ingredients of the composite compared to remaining surface treatment methods and formulations of the materials. This friction composite shown in fig 6(f) have a chance to possess high mechanical strength and good damping and tribological properties compared to all other formulations.

7. MECHANICAL PROPERTIES RESULTS

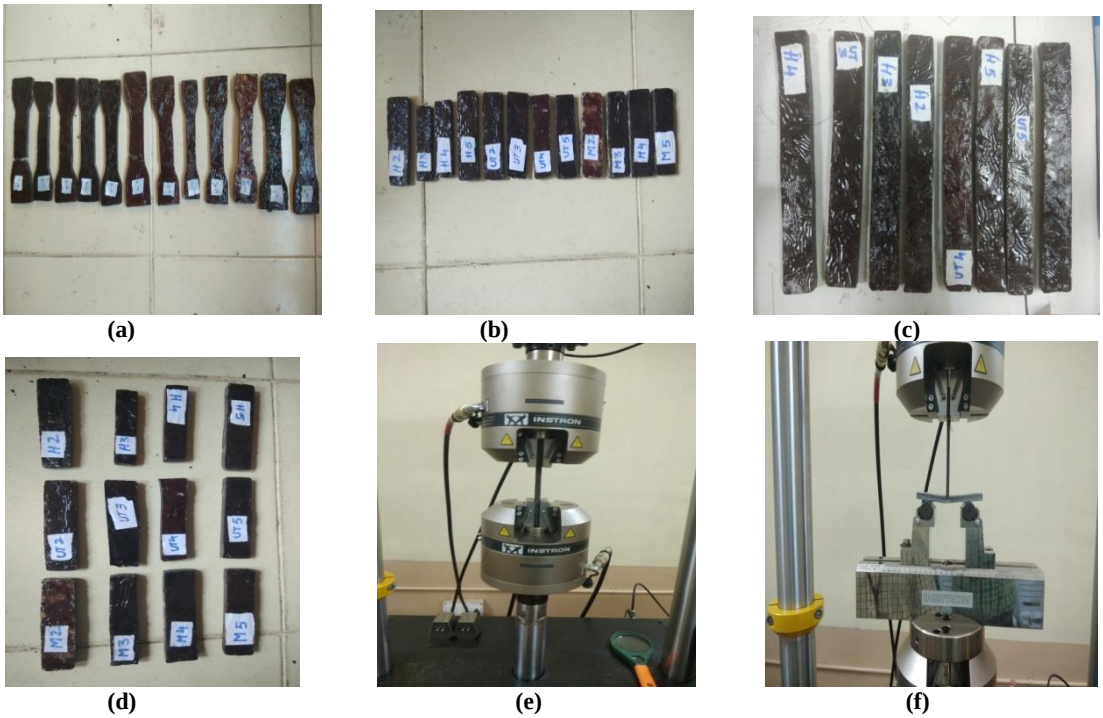


Fig. 7 (a-f) Specimens and Experimental set up

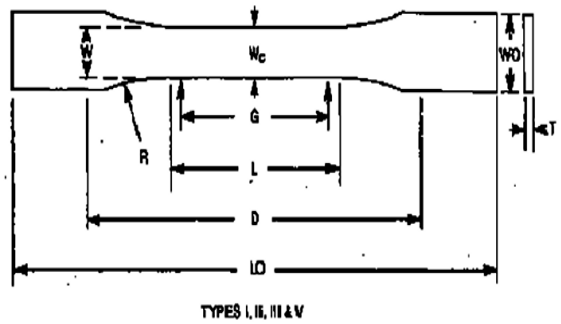


Fig. 7(g) Dimensions of tensile test specimen

Mechanical properties are the basic important properties to evaluate the strength of any material. Mechanical properties of all the samples are evaluated with varying surface treatment method performed on CF. Tensile, compressive and flexural tests are conducted by using instron testing machine of model No 8801, shown in fig 7.1 (a). The machine is equipped with advanced load cell technology for faster testing and reduction of inertia errors. Tensile test is performed based on ASTMD638-02a standard of (Type1). The specimens for all the tests to be conducted are shown in fig 7(a-d). The tensile test is performed by fixing the specimens between two clamping jaws firmly and loaded gradually with incremental load until failure of the specimen takes place. The values of deformation against each load value are noted and tabulated. The breaking load for each specimen is noted and ultimate tensile strength values of all the samples with varying surface treatments are calculated. The compressive and flexural stresses of all the samples are determined by mounting the specimen with alteration of fixture arrangements. The values of tensile, compressive and flexural strengths of all the materials with variation of surface treatment methods are given in table 3. Impact, hardness and surface

roughness values of all the fabricated samples are determined and tabulated in table 4. Impact test is performed as per ASTM D 256 and specimens of size 55x10x10 mm are cut from the samples to determine the impact strength of the materials.

The notations used in Fig 7 (g) for tensile test are indicated with the dimensions Lo – Overall length 165(6.5) mm,D-Distance between grips 115(4.5) mm, L- Length of narrow section 57 (2.25) mm, G- Gauge length 50(2) mm, R- Radius of fillet 76(3.0) mm, Wo- Width overall 19 (0.75) mm, Wc- Width of narrow section 13(0.50) mm, T – Thickness of the specimen 10 (0.50) mm. Compression test is conducted as per ASTM D3410 and specimens of dimensions 125mmX 20mmX 10mm are cut from the sheets to evaluate compression strength of the materials. The dimensions of specimen for flexural test are taken as 190mmX20mmX10mm and flexural strength of the specimen is determined against bending load. Hardness values of all the samples are determined by using brinell hardness tester and surface roughness of the samples are determined by using taly surf instrument. All these properties of friction composite specimens helps to estimate the mechanical properties of the composite specimens and surface roughness values enables to estimate the coefficient of friction and wear rate of the friction material for a suitable application. The selection of ingredients and surface treatment method is mainly responsible for the improvement of mechanical properties and bonding strength of the material.

Table -3 Tensile, Compressive and Flexural properties of friction composite materials

Type of composite	Brinell hardness number	Imapct strength (J)	Surface Roughness (Ra)
UT2	10.33	7	11.94
UT3	12.02	6	7.733
UT4	13.6	7	4.708

Table -4 Hardness, Impact and surface Roughness values of friction composite materials

Type of composite	Tensile Load at Break (KN)	Ultimate Tensile Strength (Mpa)	Maximum Compressive load (KN)	Ultimate Compressive strength (Mpa)	Maximum flexural load (KN)	Flexural stress (Mpa)
UT2	0.93	7.11	32.67	43.44	0.097	5.49
UT3	0.56	4.57	20.67	24.69	0.731	41.14
UT4	0.79	6.48	30.27	34.39	0.22	12.42
UT5	0.98	5.19	32.31	40.79	0.613	34.51
H2	0.94	7.67	15.39	23.64	0.09	5.49
H3	1.54	12.44	24.75	39.16	0.18	10.57
H4	0.48	3.96	33.45	46.45	0.097	5.49
H5	2.28	14.79	25.08	29.33	0.191	10.79
M2	8.01	44.84	14.94	14.94	1.261	70.965
M3	7.06	45.65	18.24	19.82	0.942	53.022
M4	10.17	51.52	23.79	27.82	2.247	126.43
M5	0.93	72.86	17.4	18.12	3.095	174.11

8. CONCLUSION

The focus of this work is to develop a carbon fiber reinforced friction material for automobile and aerospace applications. Three surface treatments are performed on CF namely oxidation, HNO_3, MWCNT-F grafted on CF to use CF more effectively in friction materials. TGA, FTIR and mechanical properties of all the samples are determined .Based on thermo gravimetric analysis, it was observed that MWCNT-F grafted on CF sample M5 (CF 5wt%) possess low weight loss with increase in temperature from 100^{0} C to 1000^{0} C . FTIR analysis reveals about bonding behaviour of the friction composite. It was observed that, MWCNT-F grafted on CF sample M5 (CF 5wt%) possess good bonding strength . The intensity of peaks for the sample S5 is having fewer fluctuations with varying wavelength compared to remaining formulations of the materials and surface treatment method. Based on mechanical results, it was observed that, the sample specimen M5 (MWCNT-F grafted on CF5wt %) exhibit high tensile, impact and flexural strength values compared to remaining formulations. The compression strength of friction composite is more for HNO_3 treated sample specimen H4 (CF 4 wt%) and hardness and surface roughness values are more for MWCNT-F grafted on CF for 2wt% and 3 wt%).Hence, it was concluded that the surface treatments performed on CF is the main reason to have better mechanical, thermal and good bonding behaviour of the friction composite. Further, it also concludes that, grafting MWCNT-F on CF yields good bonding behaviour of the composite compared to remaining treatment methods.

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