Effect of surface treatments on damping behavior of carbon and glass fiber reinforced friction material

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Abstract

The ability to absorb vibrations in a vehicle during braking conditions depends primarily on the selection of ingredients for a friction material and interfacial adhesion between all these ingredients. In this work, a hybrid brake friction material is developed by combination of carbon fiber (CF), glass fiber (GF), resin and other ingredients. The surfaces of carbon and glass fibers are chemically inert and hydrophobic in nature. Therefore, CF and GF surfaces are modified with surface treatments to increase hydroxyl or carboxyl groups on the surface. An attempt is made to improve the bonding strength between CF, GF, ingredients and polymer matrix. CF surface is modified by oxidation, HNO3 treatment and grafting multi walled carbon nano tubes functionalized (MWCNT-F) on CF. GF surface is modified by HNO3 treatment. Carbon fiber and glass fiber content after surface modifications is mixed with all the ingredients and resin. Friction 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. Sample specimens are cut from the friction composite sheets and damping behaviour of the specimens is evaluated by using FFT analyzer. The best surface treatment method and ingredients are selected to fabricate a friction material to reduce squeal generation at the interface between brake disc and pad.

Keywords: Multi walled carbon nano tubes; carbon fibers, glass fibers, chemical grafting, damping, and Interfacial shear strength

1. Introduction

Now a day's Carbon and glass fiber reinforcements are extensively used in polymer matrix composites for structural applications, aircraft and automotive industries. CF is primarily preferred for 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 expansion and outstanding fatigue characteristics. They are also find applications where high damping chemical inertness and superior wear resistance are important [1-5].GF reinforcements are used mainly in composite applications for reduction of weight and improving the performance, durability and corrosion resistance. CF and GF composites are used widely in pressurized cabin system of an aircraft to sustain the flammability resistance of aircraft, landing gear doors, and 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. In this work, a new hybrid brake friction material is developed by combination of GF, CF, resin and other ingredients to with stand high temperatures generated at the contact between brake disc and pad during braking conditions. 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. [1-6]

The performance of the brake friction material like wear resistance, fading, damping behavior, coefficient of friction, squealing action, reliability, cost and manufacturing considerations primarily depends on the selection of ingredients for the friction material and interfacial adhesion between all the ingredients selected for the friction material. The interfacial

properties of the CF, GF, ingredients and polymer can be improved by modifying the surface of CF and GF 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 [7]. 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 and CF surface. Qiang Song et al [8], Observed that grafting straight carbon nano tubes radially on to carbon fiber surface has improved its mechanical properties. The mechanical properties such as compressive strength and inter laminar shear strength are increased by 275% and 138% compared to pure C/C Composite. S.P.Sharma et al [9] identified that coating carbon fibers by using CVD on CF surface improves its tensile strength to 69% for CF/epoxy/amine polymer matrix composites. Hui Qian et al [10], modified the carbon fiber surface by grafting carbon nano tubes on carbon fibers by CVD and observed that, there is 26% increase in inter laminar shear strength for carbon fiber and poly methyl methacrylate (CF/PMMA) composite. Mohit Sharma, et al [7], observed that, there is an improvement in inter laminar shear strength by 175% for CF/epoxy composite and author also mentioned about , MWCNT deposited on carbon fiber surface by chemical grafting improves inter laminar shear strength by 150% for CF/epoxy composite . But, grafting CNTs on CF surface by using CVD is a costlier process and involves utmost care in 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 for a medium duty application to a greater extent. Therefore, in this work, an attempt is made to modify CF surface by three chemical treatment methods and GF surface by HNO3 treatment. After modifying the surface of CF and GF, these are mixed with all the ingredients in different weight proportions to test the damping characteristics of the friction composite material used in automobile applications. [11-12].

The friction material selected for the design has to withstand the given loading conditions easily and induce less squealing action, maintain better coefficient of friction and less wear rate at the interface between brake disc and pad. Many researchers focused on material properties of the friction materials and analysed its performance by using FEM to minimize vibrations at the contact between brake disc and pad.

EJ Berger [13] examines the literature related to friction modelling for dynamic simulation. It was concluded from this study that, system model and friction model are fundamentally coupled, and they cannot be chosen independently. The multi scale effects principle can dominate the performance friction contacts. A. Renault et al [14] performed experimental investigations for uncertainty quantification in brake squeal analysis. This paper summarizes and improves the correlation between the experimental and numerical prediction of unstable frequencies for automotive brake systems and concluded that, use of non-deterministic simulations identifies better instabilities of the braking system. A. R. Mat Lazim et al [15]. Identified the squealing characteristics of worn brake pads due to silica sand embedment into friction layers. Author observed that, the tribological properties are more affected by small size silica sand particles and increasing the silica sand particle size leads to damage in the surface of friction material. P.Grzes et al [16] conducted a numerical experiment for the analysis of temperature field in a pad –disc braking system of a rail way vehicle at single braking. Cast iron brake disc and organic composite pad materials are examined. The transient temperatures were verified with numerical experimental values. It was observed that nominal area of contact and the grooves are mainly responsible for heat dissipation and withstanding the given pressure. Hui Lu et al [17] designed a disc braking system with hybrid uncertainties. In his paper author used hybrid probabilistic interval model using RSM. The combination of genetic algorithm and monte- Carlo method is employed to perform the optimization to reduce squeal propensity of the braking system. Ali Belhocine et al [18] carried out a numerical study of mechanical behaviour of dry contact slipping on the disc –pads interface to observe structural deformation and Vonmises stresses due to the contact of slipping between brake disc and pad. The influence of smoothness of mesh, rotation of the disc, materials of pad and friction coeffient, pad groove are primarily important for this study. Amr M. Rabia et al [19] reviewed on experimental studies of automotive disc brake noise and vibration and proved that the experimental data gives good results compared numerical solutions. The author has conducted few investigations to change the cross sections of pad and disc material. M.Nouby et al [20] studied about disc brake squeal reduction through pad structural modifications using FEM by complex eigen values.

K.Naresh Kumar and K.N.S. Suman [21,22] performed fluctuating and explicit dynamic analysis on disc braking system by altering the material properties of disc and pad. It was concluded that material

combinations of grey cast iron disc and carbon fiber pad can easily sustain fluctuating loads. Author, also identified that Ti alloy disc and carbon fiber pad combination can easily sustain high speed dynamic loading conditions of the automobile. The properties of the materials selected are the main reason to sustain high dynamic loads. Hence, all these studies have been reported mainly to reduce squealing action at the contact surface between brake disc and pad. Few studies are concentrated mainly on structural modification of the brake pad material and performing analysis by using FEM software at the beginning of the design. In this work, a new approach has been designed to study the damping characteristics of the brake friction material by variation of surface treatment performed on CF. Surface treatments are performed on CF to improve the bonding strength of the friction composite. The best surface treatment method is selected for the design. The optimum value of CF wt % which possesses good damping characteristics is selected for fabrication of brake friction material.

2. Materials and methods

2.1 Carbon fiber

Polyacrylonitrile (PAN) based chopped carbon fibers were used for this study. Chopped carbon fibers are considered for performing surface treatments easily on fibers. Chopped carbon fibers are used in this study

for performing surface treatments on fiber easily. After surface treatments to increase carboxyl groups on the surface, these fibers are mixed with all the ingredients to formulate friction composite sheets. CF used in this study consists of 95% of CF shown in fig 2.1. The properties of chopped carbon fiber are given in table1.

2.2 Glass fiber

GFs are cheap and possess low weight and less brittleness to use in composites. These fibers have increased surface area and make them much more susceptible to chemical attack and used widely in thermal insulation especially in air craft structures. Commercially available S-2 glass fiber is used for this study. These GFs are purchased from Visakhapatnam, India. The properties of S2- glass fiber supplied by the supplier are given below in table 3.

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 2.3. 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.

Fig 2.1 Chopped carbon fiber

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 3 Properties of S-2 glass fiber

Fiber Type	Tensile strength(Mpa)	Compressive strength (Mpa)	Density (g/cm^3)	Thermal Expansion(μm/m^{0}C)	Softening temp T(^{0}C)
S-2 Glass	4890	1600	2.46	2.9	1056

Fig 2.3 MWCNT Powder

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%

Carbon fiber surface treatment methods

2.4.1 Acid HNO3 treatment

Carbon fibers (CF) of quantity 180 gms were modified by dipping in a solution of 40% HNO3 i.e. (200ml of HNO3 & 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+HNO3 +Distilled water).The complete mixing operation has to be performed slowly. Initially, 50ml of (HNO3 +Distilled water) is poured on the surface of CF. Allow the sample to rest for 5 min. This operation is continued for four times with interval time gap period of 5 min for allowing the gases to escape to the atmosphere. The collected carbon fibers were allowed to drain out the immersed liquid. Then, CF were washed with distilled water of 3lit qty. First CF was washed with 1 lit of distilled water and wait for 30 mins to allow the distilled water to completely mix with the surface. Then, the liquid is drained out. This process is repeated for 3 times until the CF surface is free from HNO3 liquid gases. Now, CF after washing operation is immersed in NaOH solution of 10% qty (300ml of distilled H_2O +30 gms of NaOH) and allow the sample to completely soak for 30 mins. Finally, CF surface is washed again with distilled water of 2 lit qty, to remove the left over acid gases adsorbed on the CF surface. The collected carbon fibers were dried in an oven at a temperature of 1500C for 2 hrs and followed by drying in atmosphere for 1hr.   The sequential operations carried out in acid HNo3 treatment on CF surface is give below in fig 2.4.1

2.4.2 Surface oxidation treatment on carbon fibers

Chopped carbon fibers are collected in crucible and subjected to surface oxidation treatment in an oven at 600 ^{0} C for 1hr. Firstly temperature is increased from 20^{0}C to 600^{0}C for 30 min under nitrogen gas and in a second step temperature is kept at 600^{0}C for remaining duration time of 30 min under air.

2.4.3 MWCNT surface oxidation treatment

Multi walled carbon nano tubes (MWCNTs) are treated to attach carboxylic acid groups on thier surface to form functionalized MWCNTs called MWCNT-F.In this method 2gms 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% HNO3) (3:1 by volume ratio) .The mixture is sonicated in a bath for 3hrs at a temperature 700C. Now , the reaction mixture is diluted with 200ml of deionised water followed by vaccum filtration process using a filter paper of 2μm porosity. This washing operation is repeated for three times to remove the metal particles adsorbed on MWCNTs .The sample is allowed to dried in an oven at 1000C. After drying operation, the collected powder particles are immersed in 40 ml of acetone solution .Finally, MWCNT powder after vaccum filtration process is added with 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 leave the sample because of low boiling point of acetone having 300 C. Now the sample is dried in oven at 1000C 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. This process of using MWCNT-F on CF surface increase the damping characteristics and reduce the squeal genetation at the contact region between brake disc and pad. Fig 2.4.3 gives the sequential operations carried in functionalising MWCNTs powder .

Fig 2.4.1 Sequence of operations in Acid HNO$_3$ Treatment on CF and GF surface

Fig 2.4.3 Sequential operations carried out in MWCNT surface oxidation to form MWCNT-F

Fig 2.5 Grafting MWCNT-F on CF Surface

2.5 Grafting MWCNT-F on oxidised CF surface

Multi walled carbon nano tubes functionalized (MWCNT-F) of qty 1.5 gms obtained after filtration and drying operation is dispersed in a 20 ml of acetone solvent using an ultra sound bath at 700 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 entitre surface of carbon fiber.The deposition operation is repeated several times with evaporation of the sovent between each deposition.Heat treament process is carried out on the resulting CFs grafted MWCNT-F at 1000C for 2hrs. Fig 2.5 gives the sequential operations carried in grafting MWCNT-F on carbon fiber surface.

3. Preparation of composite sheets

Hand layup method is used to fabricate friction composite sheets because of its ease of control over operating variables and pressure applied on the composite sheets. CF after surface treatment is mixed with varying carbon content wt % ranging from (2% , 3% , 4% & 5%) along with other ingredients using a foculator for 1 hr . After proper mixing of all ingredients , friction composite sheets are fabricated by using hand lay up process. The details of ingredients selected for fabrication of composite sheets are given in table 3.

The mixing operation of all the ingredients is carried out in a foculator. Initially resin is taken in a foculator and all the ingredients are added slowly one after the other with an interval time gap period of 10 min. Total mixing operation for all the ingredients was carried out for 30 min for each sample sheet. Twelve composite sheets of varying carbon content along with other ingredients are fabricated. Samples of size 190mm x 20mm are cut from the sample sheets to test the damping characteristics of each specimen. The sequence of operations involved in fabrication of friction composite sheets and cutting the specimens are given in fig 3.

4. Thermo gravimetric analysis (TGA)

TGA is a method of thermal analysis in which the mass of   a   sample   is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption and desorption, chemical phenomena including chemisorptions, thermal decomposition and solid gas reactions (eg. oxidation or reduction). 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 analysis is performed for all the samples. The behaviour of friction composite specimen S3 with increase in temperature (HNO3 treated GF wt34% and MWCNT-F grafted on CF wt4%) is shown in fig4. The temperature is increased slowly from 00C to 10000C for an ending time period of 112.6 min. The oxidative mass loss is observed with increase in temperature. The mass loss is observed to be very less. The weight loss for the sample is observed to be 0.202 mg for the sample S3. The mass loss for the sample S4 is very less compared to other formulations .The selection of ingredients for the sample S4 can easily sustain high temperatures. This analysis reveals that, ingredients selected for the friction composite specimen for the sample S3 can exhibits good temperature with standing capability with increase in temperature compared to all other formulations.

Table 3. Material Selection

Material	Sample S1	Sample S2	Sample S3	Sample S4
CF [MWCNT-F grafted]	2%	3%	4%	5%
GF(HNO$_3$ treated)	36%	35%	34%	33%
Phenolic Resin	40%	45%	50%	55%
Zirconium Silicate (ZrSiO4)	8%	6%	5%	4%
Graphite	5%	4%	2%	1%
Barium sulphate (BaSO4)	4.5%	3%	2%	1%
Rubber Powder	4.5%	4%	3 %	1%
Total	100%	100%	100%	100%

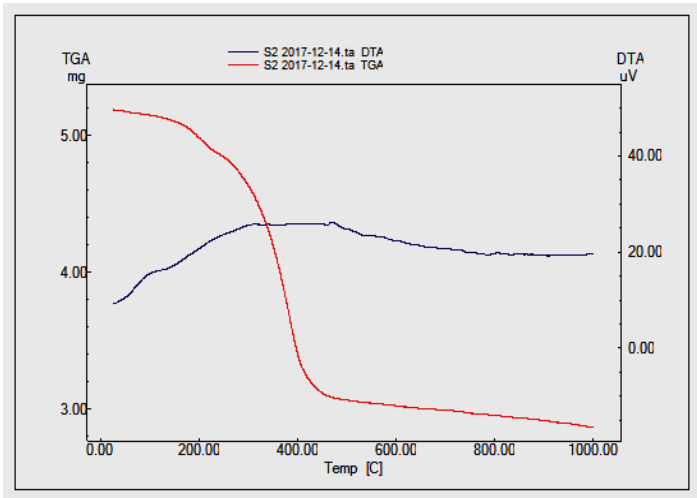**Fig 3**. Sequence of operations carried in fabrication of composite sheets

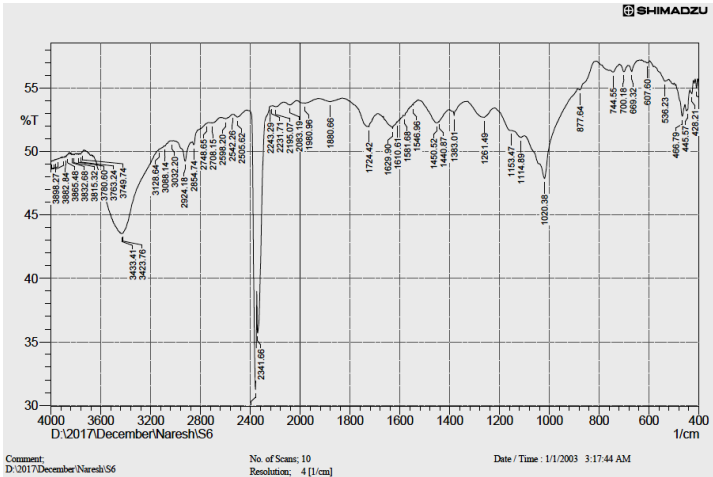**Fiq4.**TGA analysis on powder sample of S3 specimen

Fig 5. FTIR analysis on sample S3

5. Fourier transform infrared spectroscopy analysis (FTIR)

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 55.001 and stops at a value of 49.235 for the sample S3 with (HNO3 treated GF wt 34% and MWCNT-F grafted on CF wt4%). It is also observed that, sample S4 is having good bonding strength compared to other formulations. The intensity of peak value is having fewer fluctuations with increase of wavelength for the sample S3 compared to other formulations. The ingredients selected for fabrication of friction material for the sample S3 with (HNO3 treated GF wt 33% and MWCNT-F grafted on CF wt5%) possess good bonding strength and high inter laminar vanderwalls forces with respect to resin. The absorption spectra and intensity of peaks with respect to wavelengths is given in fig 5.

6. Scanning Electron Microscopy Results

Scanning electron microscopy results are taken to observe the grain distribution for the entire samples specimen after fabrication. The bonding strength and behaviour of the friction composite sheet can also be visualized based on the images. Fig 6(a) represents the SEM image of powder collected from the sample S3 with all the ingredients. Fig 6(b) represents sample S4 image and Fig 6 (c) represents the SEM images of S3 sample composite sheet.

From Fig 6(b), it was observed that, sample S4 composite specimen exhibits partial bonding strength and inter laminar shear force distribution between all the ingredients are weak and does not possess good absorptive characteristics with resin. The composite specimens containing S3 with (HNO3 treated GF wt 34% and MWCNT-F grafted on CF wt4%) , shown in fig 6(c) exhibits high bonding strength and good inter laminar shear forces between all the ingredients compared to other formulations of materials selected for the fabrication. The surface structure of sample 4 and grain distribution throughout the surface is uniformly distributed across the sample and all the ingredients are greatly bonded to the resin. This sample S3 with (HNO3 treated GF wt 34% and MWCNT-F grafted on CF wt4%) with remaining ingredients can exhibit good mechanical and tribological properties and good bonding strength in automobile applications to use as a friction material.

Fig 6(a) S3 sample Specimen powder
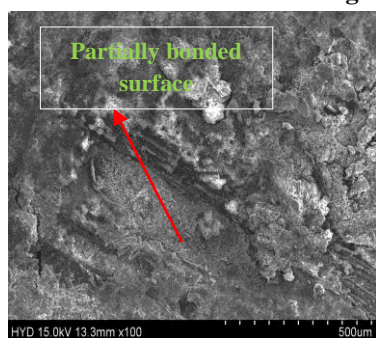
Fig 6(a) S4 sample composite sheet
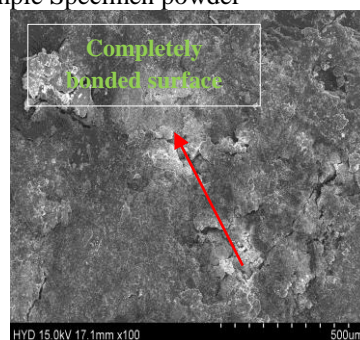
Fig6 (b) S3 sample composite sheet

7. FFT Results

7.1 FFT Analyzer

The FFT or Fast Fourier Transform spectrum analyzer uses digital signal processing techniques to analyzer a wave form with Fourier transforms to provide in depth analysis of signal waveform spectra. The advantages of FFT Spectrum analyzer is to fast capture of wave form, non repetitive events, analyze signal phase and wave form can be stored easily.

Fast Fourier Transform converts continuous domain in to continuous frequency domain including both magnitude and phase information.

The apparatus used in the present experiment consists of Impact hammer, Accelerometer, multi channel vibration analyzer , A PC or a Laptop loaded with software for modal analysis, test-specimen (A cantilever held in a fixture) , power supply for the PC and vibration analyzer and connecting cables for the impact hammer and accelerometer. The equipment specifications are given as COCO80 crystal instruments, sensitivity 10 mv/LBF , sensor 96mv/g , resolution 1024/450 and the frequency range is linear varies from 0 to 360 HZ shown in fig 7(b) . The first step in using FFT analyzer is to prepare a cantilever set up by considering different nodes on the specimen. Specimen of dimensions 190mmx20mmx10mm is cut from the composite sheets. Mark different nodes on the specimen with equal distances (i.e 19mm consisting of 10 equal nodes) . Fix the specimen at a distance of 76mm from the fixed end (i.e at the 4 node). After connecting all the cables, impact hammer force is applied at the free end of the specimen. Three strikes are considered for each specimen and take the better value of peak intensity as the time reference for that sample. Note the values of three different frequencies for each sample with respect to the given impact force for each sample. This enables to calculate the damping factor of each sample and identify the vibration absorptive characteristics of each sample.

7.2 Damping ratio measurement

Damping ratio of all the samples are calculated by half wave band width technique by calculating Q factor. Q factor is calculated by taking reference band width of 3dB below the peak value as shown in fig 7.2 (a). Damping ratios of all the samples are calculated by using the formula and the values of damping ratios of all the samples are represented in table 4.
Q factor is given by $Q = fc / (f1-f2)$
Where fc is the central frequency
Damping ratio $\zeta = 1 / (2Q)$

Frequency vs amplitude response for all the samples is determined and the damping ratios of the samples are calculated in table 5. From Table 5, it is observed that, the damping behaviour of all the samples is less than 0.5 and possesses good damping characteristics. The sample specimen S3 Possess good damping characteristics compared to other formulations. The sample specimen S4 also possesses good damping characteristics slightly equal to sample S3. The composite specimens S3 and S4 exhibit good damping behaviour mainly due to selection of optimum ingredients and rubber powder particles in the specimen, which are responsible for absorbing the vibrations induced in the friction composite. These formulations of materials can extend its usage in automobile applications to reduce squeal propensity at the contact between brake disc and pad .The amplitude vs frequency response for the samples S3 and S4 specimens are given in fig7.2 (b) and fig 7.2(c).
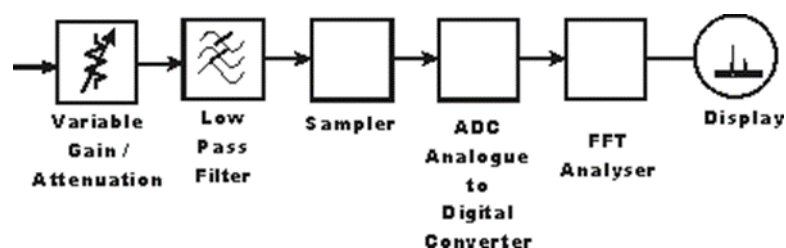

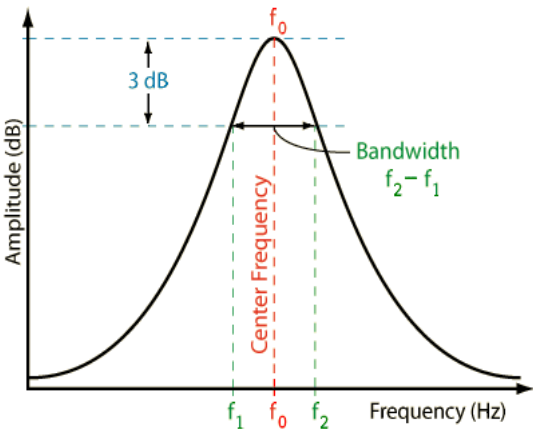**Fig 7.1 (a)** Spectrum analyzer block diagram

Fig 7.1 (b) FFT Analyzer


Fig 7.2 (a) Half wave band width technique

Table 4. Natural frequencies of different composite specimens

Type of composite material	Natural frequencies (Hz)		
	Mode 1	**Mode 2**	**Mode 3**
S1	31.25	39.06	52.34
S2	31.25	39.06	39.84
S3	31.25	39.06	52.34
S4	31.25	39.84	53.13

Table 5. Damping ratios of different composite specimens

Type of composite material	Damping ratio
S1	0.185
S2	0.115
S3	0.064
S4	0.067
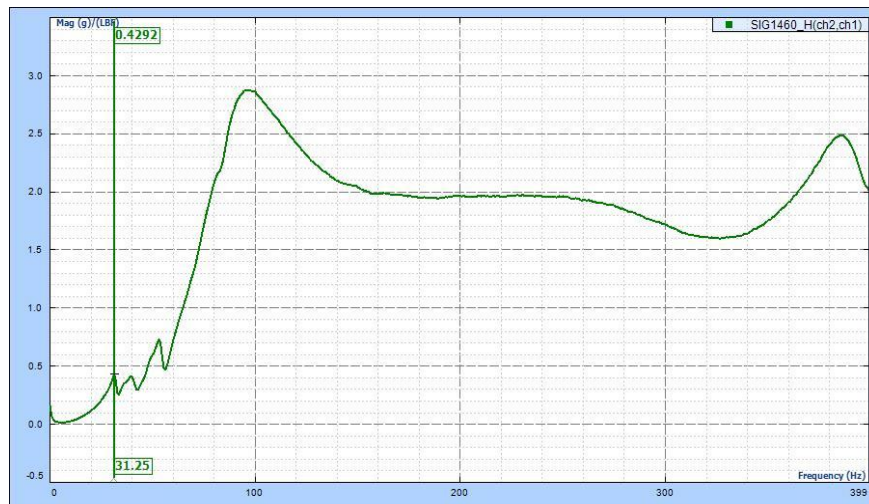
Fig7.2 (b) Amplitude vs Frequency curve for S3 Specimen
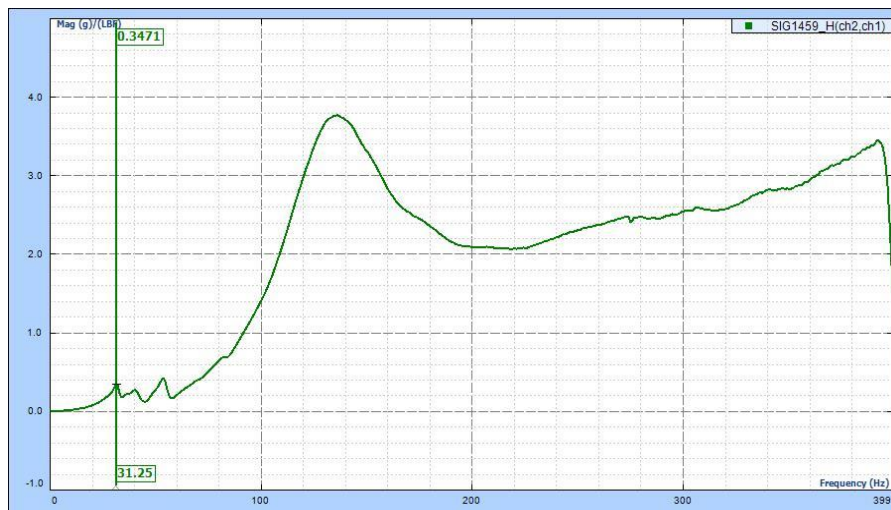
Fig7.2 (c) Amplitude vs Frequency curve for S4 Specimen

Conclusion

The damping behavior of the brake friction material is evaluated by varying the surface treatment on CF and GF. The surface of CF is grafted with MWCNT-F and GF is treated with HNO3 treatment .After surface modifications, these fibers are mixed with ingredients and friction composite sheets are fabricated with varying content of CF and GF using hand layup method. Damping ratios of all the fabricated samples is evaluated by using FFT analyzer .The weight loss and bonding strength of all the samples are determined by using thermos gravimetric and FTIR analysis. Based on thermos gravimetric and FTIR analysis of all the samples, it was observed that

sample S3 with (HNO3 treated GF wt 34% and MWCNT-F grafted on CF wt4%) gives low weight loss with increase in temperature and high bonding strength compared to other formulations of material. The damping behavior of sample S3 specimen is also greatly improved compared to other formulations. The selection of optimum ingredients in the S3 sample specimen of friction composite and surface treatment performed on CF and GF are mainly responsible for increasing the inter laminar shear forces between all the ingredients for enhancing the damping characteristics and bonding strength. This material combination of sample S3 can extend its usage in automobile brake friction industry for reducing the

squeal generation at the interface between brake disc and pad.

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