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Damping Behaviour of Multi-Walled Carbon Nanotubes Grafting on Carbon Fiber Reinforced Friction Material

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Abstract – *An automobile's braking system plays an important role to control the vehicle at various operating speeds. At present, the brake friction industry is mainly focused on effectiveness of braking in addition to aesthetic considerations of an automobile. In this research, carbon fiber reinforced friction material is developed by grafting multi-walled carbon nanotubes functionalized (MWCNTS-F) on CF surface. The surface of CF is basically chemically inert and hydrophobic in nature and needs to be modified by grafting MWCNTS-F on its surface to increase hydroxyl or carboxyl groups. An attempt is made to improve the bonding strength between CF, polymer matrix and the remaining ingredients. Carbon fiber content after surface modification is varied in weight percentor wt% (2%, 3%, 4% and 5%) and mixed with the remaining ingredients of friction material. Composite sheets are prepared using hand lay-up method and characterized for damping, SEM, TGA and FTIR analysis. It is observed that MWCNTS-F grafted on CF 3 wt% possess good damping results. The results also reveal that, optimum selection of ingredients and surface treatment method on CF is the main reason for improvement of the composite's interfacial adhesion and damping behavior.*

Keywords: Multi walled carbon nanotubes, carbon fibers, chemical grafting, damping, interfacial shear strength

1.0 INTRODUCTION

Carbon fiber (CF) reinforcements have been extensively used in polymer matrix composites for a wide range of applications in mechanical engineering, aviation, nuclear and automotive industries. CF is primarily preferred for composite materials due to its excellent properties, namely 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 (Liu & Kumar, 2012). According to Tiwari and Bijwe (2014) and Soutis (2005), CF reinforcements are also used in applications where high damping and chemical inertness are important. Carbon fiber reinforced polymers have also been found to possess superior wear resistance compared to other fibers (Tanaka, 1977).

The performance and damping characteristics of carbon fiber reinforced composite primarily depends up on the interfacial adhesion between fiber and matrix and ingredients selected for the work as explained by (Chand, 2000; Sharma et al., 2014). In addition, interfacial properties of CF and polymer can be improved by modifying and introducing hydroxyl or carboxyl groups on CF surface. Therefore, various efforts have been carried out to improve CF surface properties by using different treatment techniques including sizing, plasma, chemical oxidation, γ-ray irradiation, electrochemical, dip coating, MWCNT grafting on carbon fiber surface by CVD, etc. (Sharma et al., 2014).

The squeal propensity at the interface between brake disc and pad mainly depends on interfacial adhesion between all the ingredients selected for friction materials, dissipation of heat to surrounding, wear rate and fading phenomena. Many researchers have attempted to minimize vibration generated at the contact region by altering the material properties and performing analysis by using FEM. Nouby and Srinivasan (2009) studied disc brake squeal reduction through pad structural modifications using FEM by complex Eigen values. The researchers identified that, geometric characteristics and chamfer would affect the squeal propensity and Eigen modes. Lu and Yu (2014) focused on brake squeal reduction by considering uncertain parameters like friction coefficient, material properties and thickness of wearing component. It was observed that, a reliability based optimization model for improving the vehicle stability is used based on RSM. Wang et al. (2016), on the other hand worked on disc surface with two types of groove textured surfaces on a pad disc system. They concluded that, cutting a 45-degree groove on the disc surface can significantly reduce friction noise.

The groove textured surface can affect the contact pressure and squeal generation. Nacivet and Sinou (2017), presented a new approach to study the modal amplitude stability analysis of a brake squeal system and identified that, this new method is easy to implement and takes less computing time for complex Eigen values. In addition, Konada and Suman (2017a, 2017b, 2018) performed fluctuating and explicit dynamic analysis on disc braking system by altering the material properties of both disc and pad. They concluded that, material combinations of grey cast iron disc and carbon fiber pad can easily sustain fluctuating loads. Also the researchers identified that, Ti alloy disc and carbon fiber pad combination can easily sustain high speed dynamic loading conditions of an automobile. The properties of materials selected are the main reason to sustain high dynamic loads.

Hence, all these studies have been carried out mainly to reduce squealing action at the contact surface between brake disc and pad. However, few studies are done on structural modification of the brake pad material and performing analysis by using FEM software at the beginning of the design. This research attempts to study the damping characteristics of the brake friction material by grafting multi-walled carbon nanotubes on CF. The optimum value of CF wt% which possesses good damping characteristics is selected for fabrication of brake friction material.

2.0 MATERIALS AND METHODS

2.1 Carbon Fiber

Commercially available, Polyacrylonitrile (PAN) - based chopped carbon fibers are used for this study. Chopped carbon fibers having a carbon content of 95% is used as shown in Figure 1. The properties of the carbon fiber are shown in Table 1.

Figure 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%	1.54×10^{-3}	95%

2.2 Multi-Walled Carbon Nano Tubes (MWCNT)

Multi-walled carbon nanotubes (MWCNT) used in the study is produced using chemical vapour deposition method (CVD) as shown in Figure 2. The properties of MWCNT supplied for the study are given in Table 2. Chemicals such as NaOH, HNO_3, H_2SO_4, acetone solutions as used in the study is procured from the Visakhapatnam chemical laboratory, India.

Figure 2: MWCNT powder

Table 2: Properties of multi-walled carbon nanotubes

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%

2.3 Carbon Fiber Surface Treatment Method

2.3.1 MWCNT surface oxidation treatment

The multi- walled carbon nano tubes (MWCNTs) are treated to attach carboxylic acid groups on the surface to form functionalized MWCNTs called MWCNT-F. In this study, 2gms of MWCNT is added to 100 ml of concentrated sulphuric acid and 35ml 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 hours at a temperature of 70°C. The reaction mixture is then diluted with 200ml of deionised water followed by vaccum filtration process using filter paper of 2µm porosity. This washing operation is repeated three times to remove metal particles adsorbed by MWCNTs. The sample is allowed to dry in an oven at 100°C. After drying operation, the collected powder particles are immersed in 40ml of acetone solution.

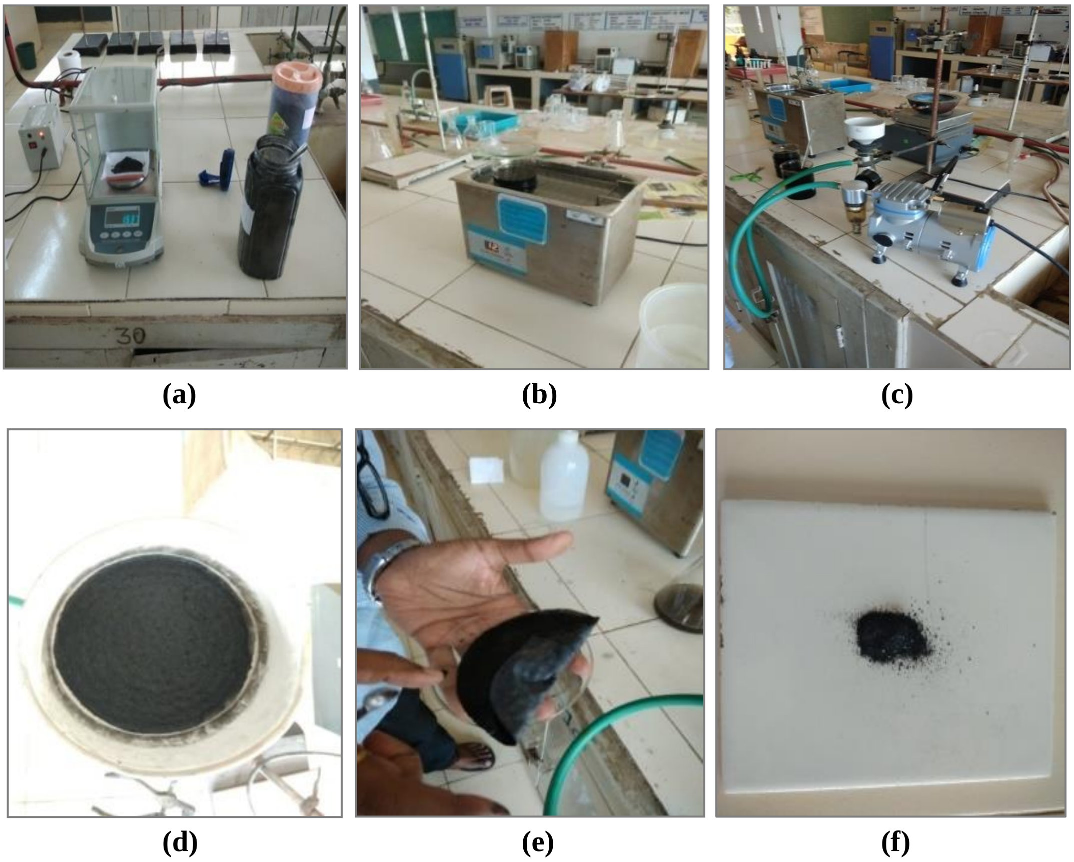

Figure 3: Sequential operations in MWCNT surface oxidation to form MWCNT-F

Finally, MWCNT powder after vaccum filtration process is added with acetone solution and placed on ultra sound bath sonicator to allow the acetone solution to completely mix with MWCNTs. During sonication process, acetone gases will leave the sample because the low boiling point of acetone is at 30°C. The sample is later dried in oven at 100°C for 4 hours followed by drying in the open air to form MWCNT-F powder. This process will completely remove metal particles present in MWCNT powder in order to use MWCNT-F more effectively in polymer matrix composites. The process of using MWCNT-F on CF surface increases the damping characteristics and reduces squeal generation at the contact region between brake disc and pad. Figure 3 (above) provides the sequential operations carried out in functionalizing MWCNTs powder.

2.3.2 Grafting MWCNT-F on oxidised CF surface

Multi-walled carbon nano tubes functionalized (MWCNT-F) weighing 1.5gms obtained after filtration and drying operation is dispersed in a 20-ml acetone solvent using an ultra sound bath at 70°C for 1 hour. Complete mixing operation is carried out using an ultra sound bath sonicator. Chopped carbon fibers are then placed on a glass substrate and suspension containing MWCNT-F is deposited drop by drop using a droppler over the entire carbon fiber surface. The deposition operation is repeated several times with evaporation of the solvent between each deposition. Heat treament process is then carried out on the resulting CFs grafted MWCNT-F at 100°C for 2 hours. Figure 4 illustrates the sequential operations carried out in grafting MWCNT-F on carbon fiber surface.

(g) **(h)** **(i)**

Figure 4: Grafting MWCNT-F on CF surface

3.0 PREPARATRION OF COMPOSITE SHEETS

Hand lay-up method is used to fabricate friction composite sheets for the ease of fabrication and control of all operating variables. CF after grafted with MWCNTs-F is varied in wt% ranging from (2%, 3%, 4% and 5%) along with other ingredients. All the ingredients are mixed using a foculator for 1 hour. After proper mixing of all ingredients, friction composite sheets are fabricated using hand lay up process. Details of ingredients selected for fabrication of composite sheets are given in Table 3.

Table 3: Material selection

Material	Sample S2	Sample S3	Sample S4	Sample S5
CF (MWCNT-F Graft]	2%	3%	4%	5%
Phenolic Resin	70%	70%	70%	70%
Zirconium Silicate (ZrSiO4)	20.5%	20%	19%	20%
Graphite	1.5%	1%	1%	0.50%
Barium sulphate (BaSO4)	2.5%	3%	2.50%	1.50%
Rubber Powder	3.50%	3%	3.50%	3%
Total	100%	100%	100%	100%

Fabrication of composite sheet starts with an initial step of preparing a die with dimensions of 42cm x 22cm x 1.2cm made of mild steel material. The die is finished to remove unwanted material of 2cm thickness on all sides by using gas welding. Mixing operation of all the ingredients is done in a foculator. Initially resin is taken in a foculator and all the ingredients are added slowly one after another with an interval time of 10 minutes. Total mixing operation for all the ingredients is carried out for 30 minutes for each sample sheet. Eight composite sheets of varying carbon content for two surface treatments are fabricated. Samples in the size of 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 is shown in Figure 5.

Figure 5: Sequence of operations carried in fabrication of composite sheets

4.0 THERMO GRAVIMETRIC ANALYSIS (TGA)

TGA is a method of thermal analysis where the mass of a sample is measured over time as 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 (e.g. oxidation or reduction). A thermo gravimetric analyser continuously measures mass while the temperatures of a sample undergo changes 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.

In this study, an analysis is performed on powder sample collected from the friction composite sheet with CF (wt5%) grafted by MWCNT-F. The temperature is increased slowly from 0°C to 1000°C for an ending time period of 112.6 minutes. The oxidative mass loss is observed to be minimal and varies with an increase in temperature for the Sample 4 as shown in Figure 6. The weight loss for the sample is observed to be 2.11mg for the Sample 4 (CF wt5%) of CF grafted with MWCNT-F. This indicates that the ingredients selected for the friction composite specimen for Sample 4 with CF wt5% (MWCNT-F grated on CF) exhibits good temperature with standing capabilities with increase in temperature. The selection of ingredients for the Sample 4 (CF wt5%) of CF grafted with MWCNT-F possesses good temperature resistance with increased temperature.

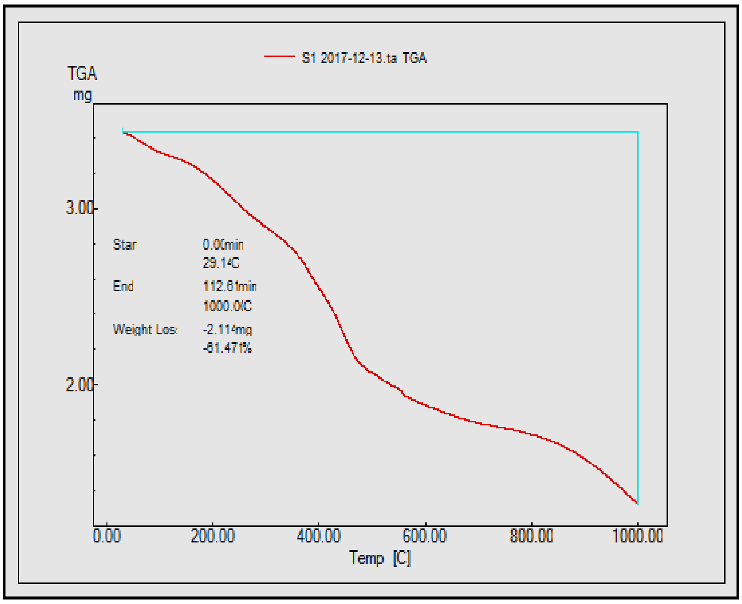

Figure 6: TGA analysis on CF grafted with MWCNT-F composite sheet

5.0 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 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 Shimadzu make 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^{-}.

It is observed that the intensity of peak begins at 44.416 and stops at a value of 40.824 for the Sample 4 (CF wt5%) for CF grafted with MWCNT-F. This indicates that Sample 4 (CFwt5%) of CF grafted with MWCNT-F specimen has good bonding strength compared to other formulations. The intensity of peak value displays fewer fluctuations with increased wavelength and the inter laminar shear forces between all the ingredients are high and this sample possess good bonding strength with respect to resin. The absorption spectra and intensity of peaks with respect to wavelengths is shown in Figure 7.

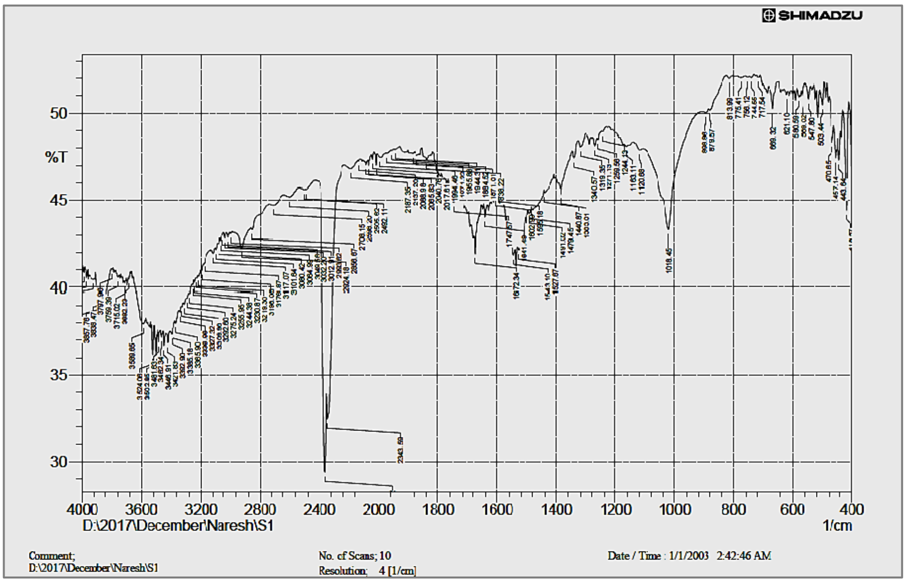

Figure 7: FTIR analysis on CF grafted with MWCNT-F composite sheet

6.0 SCANNING ELECTRON MICROSCOPY RESULTS

Scanning electron microscopy results are taken to observe grain distribution and bonding behaviour of each fabricated composite specimen. Figure 8(a) and 8(b) represent the scanning electron microscopy images of composite sheets fabricated with untreated and MWCNT-F grafted composite sheets.

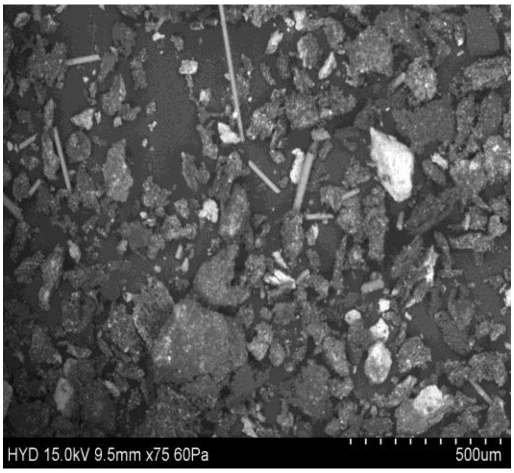

Figure 8(a): Untreated composite

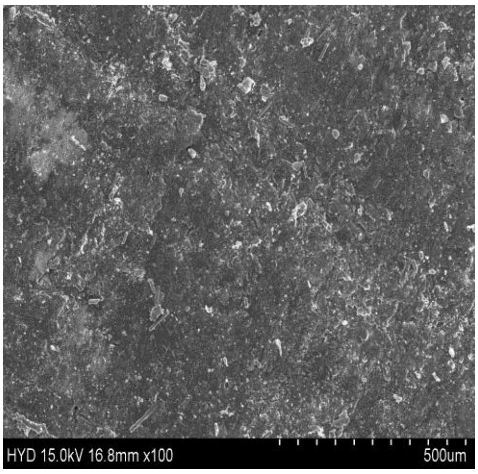

Figure 8(b): MWCNT-F deposited on CF composite

It is clearly observed in Figure 8(a), that the bonding strength is very weak for untreated CF composite sheets and inter laminar shear forces between all the ingredients is minimal. The composite specimen containing CF grafted with MWCNT-F from Figure 8(b) exhibits high bonding strength and good inters laminar shear forces between all the ingredients compared to the remaining treatment methods.

7.0 FFT RESULTS

7.1 FFT Analyser

The FFT or Fast Fourier Transform spectrum analyser uses digital signal processing techniques to analyse wave form with Fourier transforms to provide in depth analysis of the signal waveform spectra. The advantages of FFT Spectrum analyser are to quickly capture wave form, non-repetitive events, analyse signal phase and wave form and easily store them. Fast Fourier Transform converts continuous domain into continuous frequency domain including both magnitude and phase information as shown in Figure 9(a).

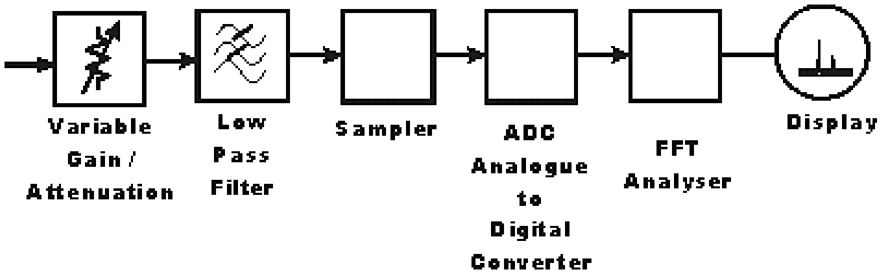

Figure 9 (a): Spectrum analyser block diagram

The apparatus used in the experiment as shown in Figure 9(b) consists of impact hammer, accelerometer, multi-channel vibration analyser, 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 analyser as well as connecting cables for the impact hammer and accelerometer. The equipment specifications are given as COCO80 crystal instruments, sensitivity 10mv/LBF, sensor 96mv/g, resolution 1024/450 and the frequency range is linear varying from 0 to 360Hz as shown in Figure 9(b).

The first step in using FFT analyser is to prepare a cantilever set up by considering different nodes on the specimen. Specimen of dimensions 190mm x 20mm x 10mm is cut from the composite sheets. Different nodes on the specimen with equal distances (i.e. 19mm consisting of 10 equal nodes) are marked. The specimen at a distance of 76mm from the fixed end (i.e. at the 4 node) is later fixed.

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 the better value of peak intensity is taken as the time reference for that sample. Values of three different frequencies for each sample with respect to the given impact force for each sample is noted. This enables calculation of the damping factor of each sample as well as identification of vibration absorptive characteristics.

Figure 9(b): FFT Analyser

7.2 Damping Ratio Measurement

Damping ratio of all the samples are calculated using half wave band width technique by calculating the Q factor. Q factor is calculated by taking reference band width of 3dB below the peak value as shown in Figure 10(a). Damping ratios of all the samples are calculated by using the formula and values of damping ratios of all the samples are represented in Table 4.

$$\text{Q factor is given by } Q = f_c / (f1 - f2)$$

where f_c is the central frequency. The damping ratio is calculated as follow:

$$\text{Damping ratio } \zeta = 1 / (2Q)$$

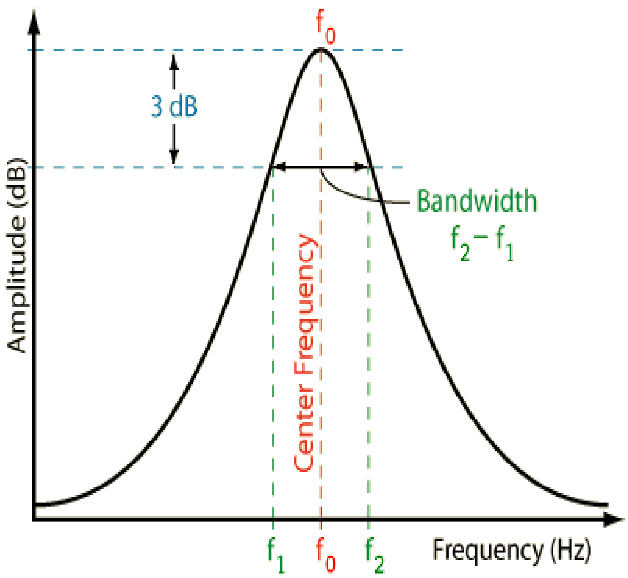

Figure 10(a): Half wave band width technique

Table 4: Natural frequencies of different composite specimens

Type of Composite	Natural Frequencies (Hz)		
	Mode 1	Mode 2	Mode 3
UT2	21.88	.....	.....
UT3	31.25	39.84	50.78
UT4	24.22	53.91	76.56
UT5	31.25	37.50	39.84
M2	40.63	53.71	79.60
M3	31.25	39.06	51.50
M4	31.25	39.06	58.59
M5	32.03	39.80	52.34

Table 5: Damping ratios of different composite specimens

Type of Composite	Damping Ratio
UT2	0.591
UT3	0.107
UT4	0.185
UT5	0.075
M2	0.194
M3	0.032
M4	0.172
M5	0.112

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 identified that, the damping behaviour of all the samples is less than 0.5 except for untreated sample UT2. Specimens M3 and UT5 display good damping characteristics compared to the other formulations. This is mainly due to selection of optimum ingredients and rubber powder particles in the specimen, which are responsible for absorbing the vibration, induced in vehicle and reduce squeal propensity at the contact between brake disc and pad. The amplitude *vs* frequency response for specimens M3 and UT5 are given in Figure 10(b) and 10(c).

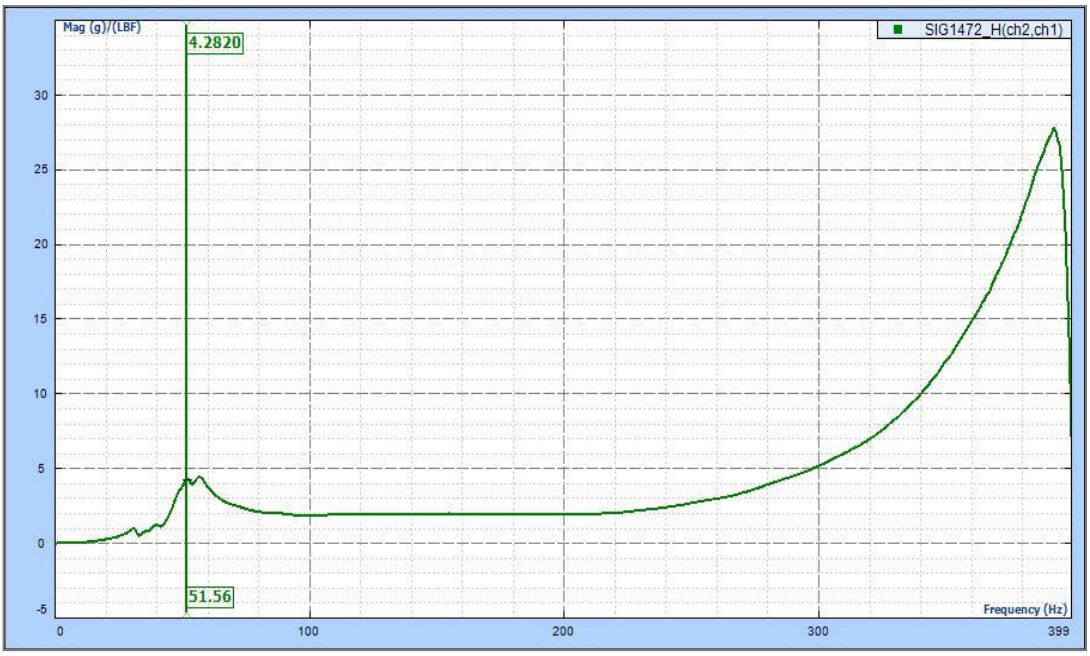

Figure 10(b): Amplitude vs. frequency curve for M3 specimen

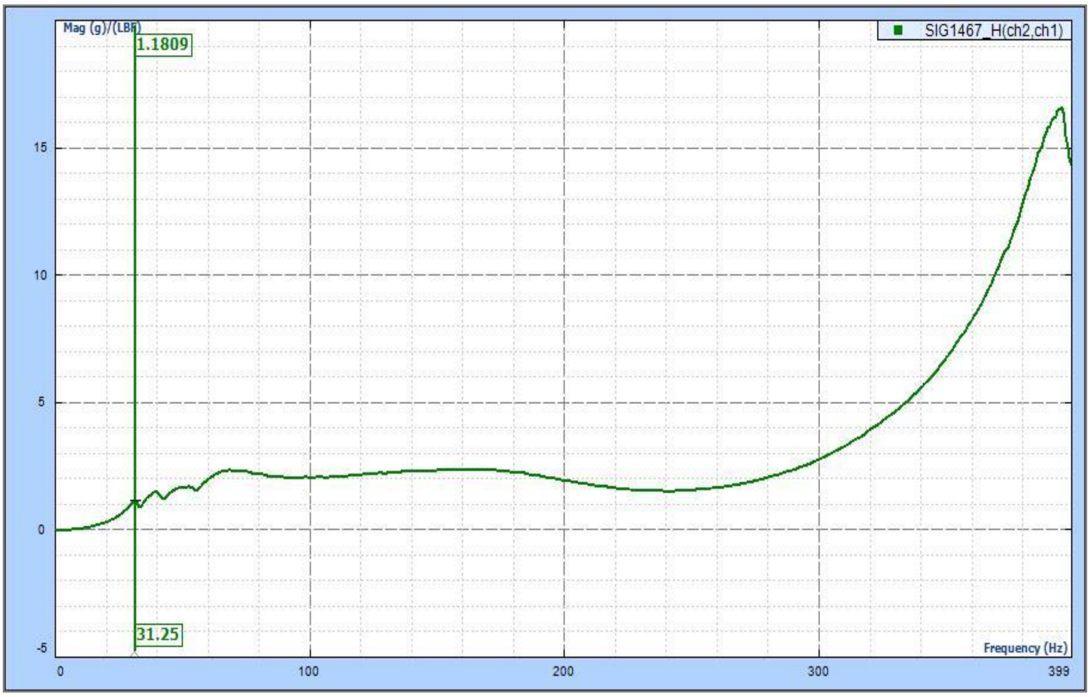

Figure 10(c): Amplitude vs. frequency curve for UT5 specimen

8.0 CONCLUSION

Damping behaviour of the brake friction material is determined by grafting multi-walled carbon nanotubes on CF. Damping ratios of all the fabricated samples is evaluated by using FFT analyser. In addition, weight loss and bonding strength of all the samples are determined by using thermo gravimetric and FTIR analysis. Based on thermo gravimetric and FTIR analysis performed on each sample powder of friction composite sheet, it was observed that

MWCNT-F grafted on CF with CF (wt5%) registers low weight loss with an increase in temperature and has better interfacial adhesion between all the ingredients compared to other formulations.

Based on FFT results, it was observed that, MWCNT-F grafted on CF specimen with CF (wt3%) (Sample M3) possess good damping characteristics compared to other formulations. Hence, CF (wt3%) grafted with MWCNT-F (Sample M3) has greater ability to absorb vibration and reduce squeal generation at the contact region between brake disc and pad. Optimum selection of ingredients and surface treatment method on CF is the main reason for better properties in terms of damping, interfacial adhesion and thermal stability of the composite.

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