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# Microstructure Analysis of Glass/Epoxy Structure Reinforced with Multi-Walled Carbon Nanotubes

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## ABSTRACT

**This work involves extensive experimental work to analyses the natural frequency of glass/epoxy honeycomb structures reinforced with 0 %, 0.5 %, 1 % and 1.5 % respectively which are prepared by hand layup method followed by vacuum bagging technique. An experimental investigation is carried out using modal analysis technique, to obtain the natural frequencies and the results show significance influence of the carbon nano tube on the natural frequency. Further microstructure analysis were also performed to analyze the surface integrity and dispersion of multi-walled carbon nano tubes in glass/epoxy composites.**

***Keywords*—** MWCNTs, Sonication, Frequency Test, SEM, TEM & OM

## I. INTRODUCTION

The quest for lightweight, high strength, fuel efficient and economic materials for applications in the aerospace and automobile industries have continued to promote enormous research into high performance, lightweight materials. Now a days the outer structure of automobile vehicle and aeronautical components must have excellent energy absorption characteristics to give better resistance to crash loads and better safety to occupants. Because of that reason a lot of importance is given to lightweight structures. Sandwich structures have been widely used to make lightweight components having exceptional mechanical characterizes and energy absorbing capacity such as aviation and transport industry.

Honeycomb is one of the most efficient energy absorbing structures in the modern era on comparison with other energy absorbing structures like metal foams and used in numerous engineering application due to their high stiffness-to weight ratios and durability have wide usages in aeronautical, automotive and civil components. Aluminium sandwich panels with honeycomb core have been widely used as energy absorption structure in lightweight design. Hexagonal honeycomb cores have been studied by numerous researchers subjected to quasi-static and dynamic loads to analyses its properties under various conditions. Honeycomb core has load carrying capacity along different direction of loads. Hexagonal honeycomb is mainly suitable for out of plane compressive loads as it has the maximum load carrying capacity in this direction. Some of the researcher found that the modeling of low velocity/low energy impact, which can lead to a decrease of the structure strength by 50% [1-3]. Figure 2 shows the dimensions of the honey comb structures described herein the length (L), the width (W), and the depth (T) of the structure [4].

In recent times, carbonaceous nanofillers such as carbon nanotubes (CNTs) and graphene play an auspicious role owing to their good geometric, functional properties and extensive range of applications in all field. The first spotting of CNTs in 1991 by Iijima carried the radical changes into the field of polymer nanocomposites (PNCs). Carbon nanotubes (CNTs) are cylindrical shells usually prepared by rolling graphene sheets into a seamless cylinder. It may exist as single-wall nanotubes (SWCNTs), double-wall carbon nanotube (DWCNTs) and multi-walled nanotubes (MWCNTs). Now a days CNTs are very attractive materials for miscellaneous nano technological applications due to their structural characteristics and their extraordinary electrical and mechanical properties. CNTs are an extremely lightweight material.

***Outline of the paper:-***

**Introduction –** In this section brief introduction of honeycomb structures, its fabrication techniques, multi-walled carbon nano tubes (MWCNTs), its properties, vacuum bagging technique and equipment used in microstructure (OM) analysis is presented.

**Methodology -** In this section fabrication procedure of glass/epoxy honeycomb structures reinforced with MWCNTs (0 %, 0.5 %, 1 % and 1.5 %) as per the ASTM standards by using vacuum bagging and hand layup method are presented.

**Literature review -** In literature review section summarization of numerous available previous research work associated with fabrication, application and advantages of glass/epoxy honeycomb structures reinforced MWCNTs are presented and found some gaps which are further implemented to achieve better properties in glass/epoxy composites which are reinforced with MWCNTs.

**Our approach -** From the available literature, found some exceptional properties of MWCNTs which is already implemented in various nanocomposites but till none of the researchers used MWCNTs (0 %, 0.5 %, 1 % and 1.5 %) as a reinforcement in the glass/epoxy honeycomb composites structure to achieve better properties with uniform dispersion by using vacuum bagging technique.

**Results -** In this section analysis and understanding of all the outcomes which is retrieved from modal analysis, optical microscope (OM) investigation are presented.

**Conclusion -** In this section, presented some significant aspect by conducting modal analysis for frequencies evaluation and microstructure analysis of the honeycomb structures, which is very useful in future for the other researchers to prepare novel and unique structures with better properties.

**References -** This section contains all the bibliography (research paper, books, website and other sources which are used in the successful completion of the project) as per APA format.

## II. METHODOLOGY

For accurate and good result, it is essential to fabricate good structured honeycomb with proper dimension. Also well planned experimental plan is required to attain sound results. Very first step of the project is the procurement of all the raw materials such as woven roving glass fiber, MWCNTs, epoxies, araldite, hardener, safety products (hand gloves & mask) etc. for the fabrication of all the specimens. In the second steps fabrication of glass/epoxy honeycomb laminates and corrugated sheets has to be done. Further cutting and joining operation has to be performed with the help of sono-file ultrasonic cutter and araldite-hardener of strip each 10 mm to form honeycomb structure. Finally all the specimen mounted on the flat surfaces to analyse the frequencies, also microstructure analysis has been done to evaluate the surface texture and dispersion of MWCNTs on the glass/epoxy composites.

In this paper frequency evaluation and microstructure analysis of MWCNTs reinforced glass/Epoxy honeycomb structure in fixes boundary condition is carried out. Also the effect of MWCNTs reinforcement on the structure frequency is evaluated in the following steps.

1. Preparation of the specimen by employing hand layup method followed by vacuum bagging technique.
2. Investigations of the frequencies of the honeycomb structures.
3. Microstructure characterization of the composite performed by using OM.

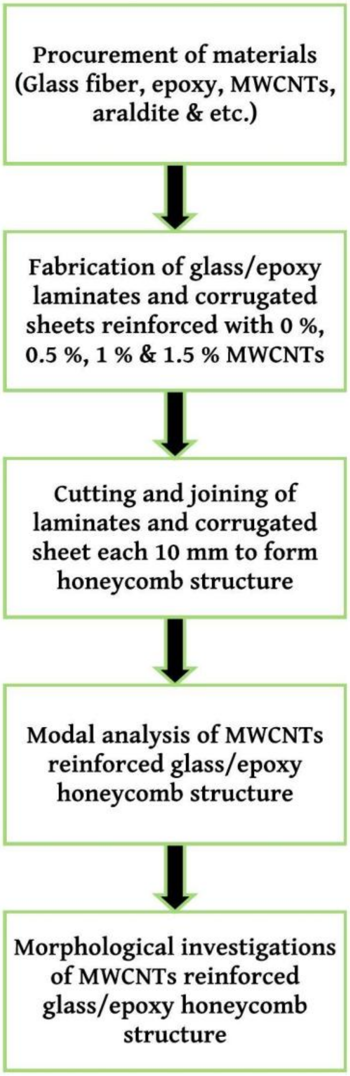

Figure 1. Flowchart depicting the methodology

## III. PRIOR APPROACH

- Mechanical, vibrational, Vibro-acoustic and flexural stiffness [5-7] characteristics of honeycombs structure has been extensively investigated by the large number of research papers. Nilsson et al. [8] presented dynamic characteristics of symmetric sandwich beams with honeycomb cores. By employing Hamilton principle they attain the boundary conditions for free, clamped and simply supported beams and then they also derived six order differential equation governing the apparent bending of sandwich beams. They found that bending stiffness of the structures depend on the frequency and the boundary conditions.

- Aminanda et al. [9] analyse the behavior of honeycomb test specimens made of nomex, aluminium alloy and paper under compression test and found that compression load is basically taken by the vertical edges of the hexagonal cell.
- Schwingshackl et al. [10] examine the orthotropic material properties of honeycomb Al core by employing numerous available analytic and experimental methods. They found good agreement between the major theoretical out-of-plane material properties of honeycomb, the experimental ASTM methods, and the presented dynamic approach.
- Luca and Sestieri [11], presented dynamic description of honeycomb cell structures by employing mathematical model based on a multi-scale asymptotic technique which is used to evaluate an equivalent orthotropic model of the honeycomb and also they validate their approach by employing industrial finite-element software. Their results revealed that the two-scale asymptotic perturbation method efficiently calculates the equivalent mechanical properties of the honeycomb cell structures.
- Gaetano et al. [12] presented the theoretical and quantitative design and analysis of a honeycomb panel sandwich structure in which material for sandwich core taken as a HRH-10–1/8–6.0 Aramid Fiber Reinforced Honeycomb. They found that when the stresses in the panel exceed the properties of the materials by any mode failure will occurs.
- Wang [13], Conducted cushioning tests to analyse the dynamic impact behavior of numerous types of paper honeycomb structure by using free drop and shock absorption principle. From the research result he revealed that proper selection of thickness are beneficial to optimize the structure design of paper honeycomb sandwich panel and material selection for packaging design.
- Vyacheslav et al. [14] estimate the influence of polymeric foam free vibration and buckling characteristics of sandwich plates with a hexagonal honeycomb core by using the commercially available finite element code ABAQUS. They found by filling of honeycomb type cores with foam allows to increase the damage resistance to the debonding propagation, but it also alters the structural responses of sandwich structure.
- Chen [15], Investigate the flexural rigidity of a honeycomb consisting of regular hexagonal cells. Their results suggested that the bending deformation of the honeycomb cannot be estimated by using the equivalent elastic moduli attained from the in-plane deformation since the moments acting on inclined walls of honeycomb cell are dissimilar for the in-plane deformation and bending deformation and the validity of the existing analysis is confirmed by numerical results obtained by the FEM.
- Saraswathy et al. [16] conducted analytical formulation for the estimation of frequency of CFRP sandwich beam with debonds by employing split beam theory by considering honeycomb core stiffness and validated the range of debond size through 3D nonlinear transient analysis.
- Crupi [17], Investigate the impact response of aluminium honeycomb sandwich structures with different cell size by experimental Low-velocity impact tests and analytical approach based on the energy balance model. They successfully applied the Simplified collapse models to explain the experimental observations and found good agreement between predicted and experimental limit loads.
- Lu et al. [18] fabricate the carbon fibre/matrix composite honeycomb using carbon fibre and epoxy resin by using compression molding technique. Further mechanical characterisation was performed by finite element analysis (FEA) and three point bending test and found good agreement between them. Their result shows that carbon fibre/epoxy Honeycomb Sandwich has greater bending strength as compared to aluminum and Nomex honeycomb Sandwich.
- Hu et al. [19] designed and fabricate the carbon fiber reinforced composite and lattice truss sandwich panel to get a strong, stiff and weight-efficient structure by employing mould pressing method in which lattice core is made up of orthogonal corrugated lattice trusses. Further shearing and compression and shearing experiments were performed to evaluate the strength and failure modes of the structure.
- Han et al. [20] theoretically examine the natural vibration and buckling response of foam-filled composite corrugated sandwich plates subjected to thermal loading. They validate their findings with the available literature as well as FE simulations and suggested that the proposed theory is simple as well as accurate to predict the free vibration and thermal buckling responses.
- Zhang at al. [21] conducted the experiments by using a pendulum-impact system to investigate the energy absorption property and Indentation response of honeycomb sandwich panels under drop-weight impact subjected to spherical impactor. Further for validation 3 D FEM model with micro-structure was developed. Their results shows that more than 80% of

impact energy is absorbed mostly by top face-sheet and honeycomb core.

- Wang et al. [22] designed the Al alloy honeycomb structures based on origami technology and further fabrication were performed by new fabrication method (press and folding Process) to automate the fabrication process of honeycomb structure. To demonstrate the feasibility of developed device, the honeycomb cores with claws were prepared by this device, which were used to compare the mechanical properties with that bonded by common adhesive. The deformation behaviors and mechanical response of honeycomb structures were examined by the flatwise compressive test and three-point bending test.
- Ivañez et al. [23] studied the crush behaviour and the energy absorption capability of an aluminium honeycomb core. 3 D FE model of a honeycomb-core structure was modelled by using commercial code abaqus. Further Flatwise and edgewise experimental compressive tests were prepared to validate the numerical model. They found good agreement between the numerical and experimental results.
- Duc et al. investigate the nonlinear dynamic behaviours and vibration response of sandwich auxetic composite cylindrical panels [24] and composite double curved shallow shells [25] resting on elastic foundations under mechanical, blast and damping loads by using analytical solution. Composite panels having three no of layers with top and bottom skins are isotropic aluminium materials and the central auxetic core layer – honeycomb structures with negative Poisson's ratio using the same aluminium material. To derive the equations for motion equations Reddy's first order shear deformation theory (FSDT) with the geometrical nonlinear in von Karman and using the Airy stress functions method, Galerkin method and fourth-order Runge–Kutta method are employed.
- Li et al. [26] examine the blasting over pressure distribution and the structural response of sandwich panels with graded honeycomb cores by employing FE software LS-DYNA after validation against the experiments.
- Zhao et al. [27] investigate the impact resistance and deformation/failure modes of composite sandwich panel's titanium alloy plates and aramid honeycomb cores under intense impulse load by using electric gun technique. They suggest that both the deformation/failure modes and the dynamic characteristics of the plates were sensitive to the impact velocity.
- Zhang et al. [28] examine the vibration response of sandwich beams with honeycomb-corrugation hybrid cores by using FE models and modal analysis techniques. For estimation of equivalent macroscopic stiffness of the honeycomb-corrugation hybrid cores method of homogenization was used. Their results shows that frequency parameter has different sensitivity to the geometry parameters, slenderness ratio of corrugated member, the face sheet thickness ratio and the relative density of filling honeycomb.
- Sun et al. [29] conducted experimental as well as numerical analysis on indentation and perforation characteristics of honeycomb sandwich panels with numerous geometric configurations. Four characteristics were considered such as Geometric variables, facesheet thickness, core height, honeycomb core thickness and side length of hexagon cell during experimentation. Their results shows that how the geometric parameters affect the characteristics of indentation and perforation, thereby providing useful guidelines for its potential applications in impact engineering.

Important aspects form the literatures - Glass fiber/epoxy polymer matrix composites being the new age materials for todays advanced world and still a lot more potential to be discovered with analytical and experimentation techniques. Light weight metals with high strength being high on demand in automotive, aerospace and military industries, polymer matrix composites reinforced with CNTs makes it a very good material to meet such demands. Glass/epoxy known for being a lightweight metal, CNT for its stiffness, toughness, low density, hardness and tensile strength makes these a perfect combination to create a light nanocomposites. Also the effects of numerous parameters on the frequencies of the honeycomb structures is an important analysis and there is still an improvisations is required whose properties can be used efficiently in the field of mechanical engineering.

## III. OUR APPROACH

In this project, we deal with the fabrication and characterization of MWCNTs reinforced glass fibre/epoxy honeycomb structures. The following steps are to be taken to fabricate the honeycomb structure.

***Step 1-***

The first step is to Cut the woven fibered glass fiber with [0, 90] orientation in two numbers, dimensions are 140 mm × 140 mm. After cutting we fabricate glass epoxy laminate by applying appropriate amount of resin and hardener by hand layup method. The resin serves as the matrix for the reinforcing glass fibers, much as concrete acts as the matrix for steel reinforcing rods. The percentage of fibre and matrix was 50:50 in weight. Proper mixing of epoxy hardener is mandatory to get uniform distribution of matrix in the fiber. After that we performed vacuum bagging operation to get void free glass epoxy laminate and then leave it for 24 hours in room temperature for the curing operation. The average weight of each laminates was measured by a weighing machine which is come around 0.1 gm.

***Step 2-***

In the second step again cut the glass fiber with [0, 90] orientation in two numbers, dimensions are 140 mm × 140 mm. After cutting we fabricate glass epoxy laminate by applying appropriate amount of resin and hardener. The percentage of fibre and matrix was 50:50 in weight. Further instead of placing glass epoxy laminate into the vacuum bagging samples directly placed it into honeycomb die after open air curing of 20 minutes to get corrugated sheet.

It will take 20 minutes to partially cure then after placed it into the honeycomb die to get an impression on it. After placing we tighten the nut and leave it for minimum 8 hours. Before placing partially cured plate into die we have to clean honeycomb die and apply wax on it by which we easily remove our corrugated sheet with a good impression. After removal of corrugated sheet we measure its weights and thickness for further calculation then we have to remove excess material from the obtained sheet to get the accurate dimension of the final corrugated sheet (c.s).

***Step 3-***

After preparing glass/epoxy plate and C.S. cutting has been performed each 10 mm strip using sonofile ultrasonic cutter (SF-0102) 22 Khz and join them with the help of equal amount of araldite and hardener to form the glass fibre reinforced every ribbon honeycomb and double reinforced every ribbon honeycomb structures structure.

***Step 4 –***

After that all previous steps are repeats to prepare c. s. and plate with 0.5 %, 1. % and 1.5 % MWCNTs solution (solution prepared by mechanical stirring method followed by sonication) respectively and then join them to prepare the MWCNTs reinforced glass/epoxy honeycomb structures.

Sonication of CNT is done to promote its dispersion using ultrasonic sound and hence, increase its solubility. Three solutions with varying MWCNTs weight percentage are prepared. Following are the solutions prepared for sonication:

1. 0.5 wt. % MWCNTs +Acetone
2. 1 wt. % MWCNTs + Acetone
3. 1.5 wt. % MWCNTs + Acetone

Further MWCNTs mixed with epoxy to prepare MWCNTs/epoxy solutions by using mechanical stirring. Stirring has been performed till the acetone evaporates completely form the solution. After 4 hours of stirring action at the rotation of 750 rpm negligible % of acetone has been observed.

After preparing glass/epoxy plate and C.S. reinforced with 0.5, 1 and 1.5 wt. % MWCNTs cutting has been performed each 10 mm strip using sonofile ultrasonic cutter (SF-0102) 22 Khz and join them with the help of equal amount of araldite and hardener (50:50) to form the MWCNTs reinforced glass/epoxy every ribbon honeycomb structure.

Summary - Fabrication of the glass/epoxy honeycomb structure reinforced with 0 %, 0.5 %, 1 % & 1.5 % MWCNTs for modal analysis and microstructure analysis was successfully performed as per ASTM standards. Each and every steps for fabrication of specimen described above and composition and % reinforcement of the specimen given below.

- ➢ Glass/epoxy + 0 % MWCNTs reinforced honeycomb structure.
- ➢ Glass/epoxy + 0.5 % MWCNTs reinforced honeycomb structure.
- ➢ Glass/epoxy + 1 % MWCNTs reinforced honeycomb structure.
- ➢ Glass/epoxy + 1.5 % MWCNTs reinforced honeycomb structure.

## IV. RESULT

To obtain the frequency of the structure we have to fix the 80 mm × 80 mm specimen in to flat surface by applying equal amount of araldite and hardener on it. Then as per the guidance manual connections made among data acquisition system (DAS), computer, accelerometer, amplifier, modal hammer and cables to the system as shown in figure 2 & 3. Further fixes honeycomb structure was excited in a selected point by means of a small impact hammer. The subsequent vibrations of the honeycomb structure are measured by an accelerometer. The accelerometer is attached by bees wax. The signal was then subsequently input to the second channel of the analyzer, where its frequency spectrum was also obtained. The response point was held immovable at a precise point and the position of excitation was varied throughout the honeycomb structure. Both input and output signals are investigated by means of FFT and resulting frequency response functions are transmitted to a computer for modal parameter extraction. The output from the analyzer was displayed on the analyzer screen by using DEWESoft 6 software. Various forms of Frequency Response Functions (FRF) are directly measured.

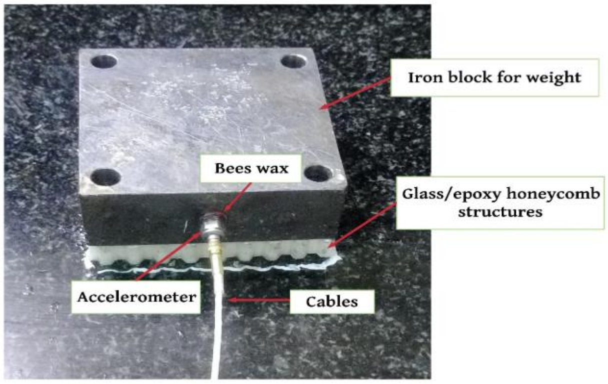

Figure 2. Mounted specimen

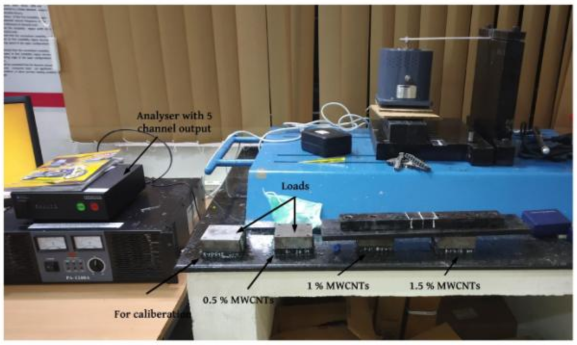

Figure 3. Mountings of all the MWCNTs reinforced specimen for modal analysis

| % CNT | S No. | Frequency/TF | |
|---|---|---|---|
| | | L | R |
| 0 % CNT (Reinforced every ribbon) | 1 | 887.50 Hz /0.411 | 916 Hz /0.405 |
| | 2 | 1193.750 Hz/0.441 | 1206.06/0.445 |
| | 3 | 887.500 Hz /0.393 | 990.500 /0.391 |
| 0 % CNT (Double reinforced every ribbon) | 1 | 918.7050 Hz /0.410 | 931.250 /0.330 |
| | 2 | 1325.00 Hz/0.394 | 1062.500 Hz/0.420 |
| | 3 | 916.50 Hz /0.38 | 918.75 Hz /0.410 |
| 0.5 % CNT (Reinforced every ribbon) | 1 | 1526.875 Hz /0.261 | 1524.375 Hz /0.262 |
| | 2 | 1946.250 Hz /0.301 | 1932.500 Hz/0.299 |
| | 3 | 1538.750 Hz /0.247 | 1522.500 Hz/0.251 |
| 1 % CNT(Reinforced every ribbon) | 1 | 1533.125 Hz /0.259 | 1525.000 Hz /0.262 |
| | 2 | 1952.500 Hz /0.243 | 1933.750 Hz/0.243 |
| | 3 | 1594.370 Hz/0.258 | 1533.625 Hz /0.273 |
| 1.5 % CNT(Reinforced every ribbon) | 1 | 1538.750 Hz /0.247 | 1528.125 Hz /0.262 |
| | 2 | 1960.000 Hz /0.265 | 1933.350 Hz/0.299 |
| | 3 | 1608.750 Hz/0.254 | 1637.260 Hz /0.283 |

Table 1. Frequencies/TF

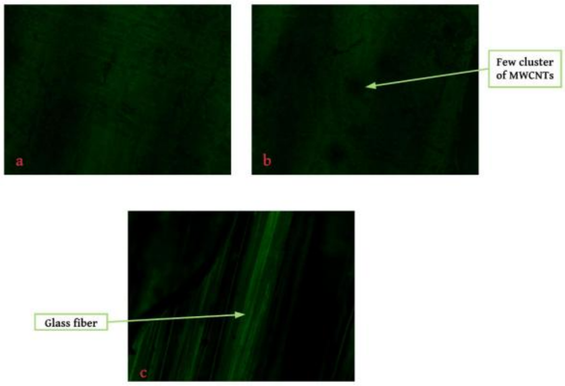

Figure 4. Optical images glass/epoxy plates (a) 10 X (b) 20 X (c) 50 X.

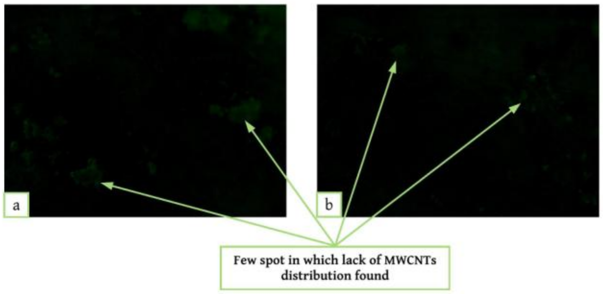

Figure 5. Optical image of MWCNTs reinforced glass/epoxy plates (a) 50 X (b) 20 X

After studying, comparing and understanding all the data retrieved from modal and microstructure analysis (OM tests) which were successfully done on the samples, observation shows void and impurities free samples. Also found that the natural frequency of structures increases with the increasing of weight % of MWCNTs reinforcement and in next chapter various points can be concluded to discuss the advantages of adding MWCNTs as reinforcement in the glass/epoxy honeycomb structures.

## V. CONCLUSION

1. Preparation of the 0 %, 0.5 %, 1 % & 1.5 % MWCNTs reinforced glass fibre/epoxy honeycomb structures based samples for determination of their frequency and microstructure analysis was successfully done using DAS and vacuum bagging method respectively.
2. The Glass/ Epoxy honeycomb structure reinforced with 0 %, 0.5 %, 1 % and 1.5 % reinforced with CNT are prepared and modal testing is done by using Data acquisition System. Frequency Response Functions are obtained by FFT. Quantitative results i are presented to show the effects of MWCNTs reinforcement. It is

found that the frequency of structures increases with the increasing of weight % of MWCNTs reinforcement.

3. Sonication and shear mixing of MWCNTs before adding it into the glass/epoxy by using vacuum bagging process assures good particle distribution in the fabricated samples which can be clearly seen in the OM images.
4. The dispersion of CNTs in the fabricated samples was enhanced significantly through sonication and shear mixing process as visible in the OM images.

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