Fabrication, evaluation of mechanical and DMA properties of basalt and glass fiber reinforced with CNT hybrid composites

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Abstract. Various industrial and medicinal applications have recently exploited composite materials including fibre reinforced composites. Automobile parts must be more secure and rigid in their construction and able to withstand large impact loads. To meet the required applications, the fiber reinforced composites produced with basalt and glass fibers in combination with carbon nanotube nanoparticles are used in this work. The aim of this work is to prepare four different fiber reinforced composites using basalt fiber, glass fiber and carbon nanotube particles in the weight/volume percentage ratio of 0.2%, 0.4%, 0.6% and 0.8 %. These laminates are being cut according to the ASTM standards to obtain specimens. These specimens have been tested for different mechanical tests for instance flexural, impact, transverse, tensile tests and dynamic mechanical analysis (DMA). The tested specimens have also been analyzed under scanning electron microscope (SEM). The test results are being compared on various mechanical parameters. The maximum tensile strength at 0.4%, maximum impact strength and maximum traverse load at 0.2 % and the maximum flexural strength at 0.8 % are observed. From the dynamic mechanical analysis, 0.4 % CNT (multi-walled carbon nanotubes) of basalt and glass fiber composite indicates more loss modulus. SEM images show the dense fiber microstructure of basalt – glass reinforced with CNT composites. The split-up into individual filaments caused improved bonding among fibers and resin as the interfaces were enhanced.

1 Introduction

Fiber reinforced composites have gained substantial consideration in numerous industries due to their excellent mechanical properties and lightweight nature. A composite material is a multiphase substance that is produced artificially and is not drawn naturally. The component phases must be physically and chemically distinct from one another. Composites provide several benefits over traditional materials such as enhanced mechanical properties,

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higher specific strength, lessen the specific gravity, increased specific stiffness, good strength even in hot environments, better impact, thermal shock, toughness, reduced thermal expansion, increased resistance to oxidation and corrosion. The applications of composite materials are in aerospace, automobile, wind turbine blades, and the construction field.

Bulut [1] investigated graphene supported basalt glass fiber for mechanical properties by various stacking by mass proportions (0.1, 0.2, 0.3 wt %) among epoxy and filler. Lee et. al [2] explored the thermal and tensile effects of revised CNT mixed epoxy/basalt fiber composites. They found that these composites have high malleability, twisting and effect strength and the upgrade of warm and mechanical properties is credited to further developed dispersibility and solid interface among the epoxy and CNT's in the basalt fabric. Deak et. al [3] have probed properties and chemical composition of basalt as well as glass fiber. The findings were compared between glass as well as basalt fiber reinforced composites. Kumar et. al [4] considered the influences of graphene nanoparticles on the tribological performance of fiber glass epoxy composites. Alexopoulos et. al [5] fabricated fiber reinforced composites employing entrenched CNT fibers. George et. al [6] carried out an investigational study of silicon particles combined with CNT and graphene nanoparticle coated EDM probes. Lou et. al [7] utilized a blend of fiber and glass powder as blending mediators for ceramifiable silicone rubber composites. These composites have been tested for mechanical properties. Han et. al [8] analyzed the properties of graphene and CNT reinforced epoxy adhesive. Kazemi et al [9] analyzed the impact of extreme velocity retort of basalt fibers or epoxy composites comprising graphene nano particles.

A composite material is a blend of more than two dissimilar metals. These materials of composite have different mechanical and chemical properties and combination of these dissimilar materials leads to a new material called composites and the property of composite material is different from the constituent material properties. Composites have two components. The continuous body component is known as the matrix phase; it gives bulk appearance. Metal, ceramics, or polymers can make up the matrix phase. The placement and direction of the fibers in relation to one more, concentration of the fiber as well as fiber allocation have substantial impact on the characteristics of fiber composites. Basalt fiber exhibits several benefits that make it an excellent substitute for glass fiber as a supporting material. Basalt fibers in specific exhibit mechanical characteristics that are comparable to those of glass fibers. Basalt fibers have excellent chemical stability, are non-combustible and have exceptional resistance to exposure to alkaline and acidic environments. Glass fiber is a substance formed from particularly acceptable fibers of glass. Glass fiber is an extremely tough, low weight, and strong material. Addition of CNT nanofillers to composite materials can improve their mechanical properties, making them stronger. 0.2, 0.4, 0.6 and 0.8 weight percentages of CNT by employing the hand lay-up technique and finally it is cured in a hydraulic press. The mechanical properties viz. impact resistance, flexural, transverse and tensile are evaluated on manufactured hybrid composites through standardized testing methods. This work also focused on comparing four different composite materials fabricated with basalt and glass fibers with CNT nano materials.

2 Design and methodology

The following are the details of the materials and fabrication methodology used in this work.

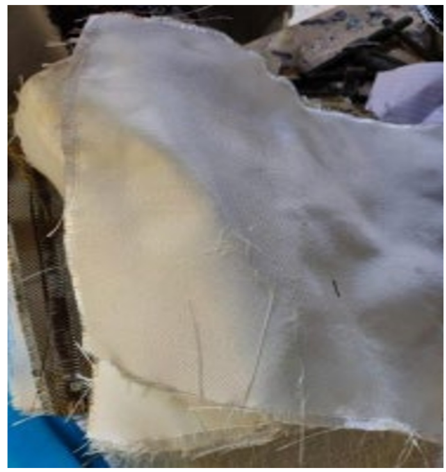
2.1 Materials

The constituents used in the fabrication of fiber reinforced composites are Basalt fiber woven sheets, Glass fiber woven sheets, Epoxy resin-LY556, hardener-HY951, Carbon nanotube nanoparticles. The CNTs used were multi-walled carbon nanotubes (MWCNTs) supplied by Nano Research Laboratory, the length of 10 to 30 microns, outside diameter of 10 to 20 nm and > 95% cleanliness. Upper and lower moulds and transparent covers.

2.2 Fabrication

Four laminates of size 300*300 mm and thickness of 3-3.5 mm are produced by hand lay-up technique and using SVS Hydraulics compression machine to compress. Hand stirring method is used to mix resin and CNT. The CNT wt% varies from 0.2, 0.4, 0.6 and 0.8%.

(a)



(b)

(c)

(d) (e)

Fig. 1. Materials used for the manufacture of basalt – glass fiber with CNT nano particulars
(a) Basalt fiber (b) Glass fiber (c) Lower and upper moulds (d) Applying of CNT mixture to the basalt
(e) Placing the upper and lower moulds and applying hydraulic presser.

Fabrication of 0.2% fiber reinforced composite using basalt fiber and glass fiber with carbon nanotube nanoparticles. In the fabrication of this type of composite material, 8 grams of carbon nanotube nanoparticles and 7 layers of 30x30 cm woven glass fibers and 4 layers of 30x30 cm of basalt fibers, together 11 layers of fibers with 100 grams of hardener-HY951,100 grams of epoxy resin-LY556 added to prepare the 30*30 cm laminate.

Repeat this process until 11 layers of glass and basalt fiber woven fabric sheets are placed with the CNT mixture. Add another transparent sheet with oil placed on it and place the upper mould on it. With the help of the mechanical hydraulic press, apply compression load on the upper and lower mould firmly for one day. The entire laminate is pressed and forms into a fiber reinforced composite. Figure 1 shows substances used for the fabrication of basalt – glass fiber with CNT nano particulars. Figure 2 indicates the arrangement of layers to fabricate the composites.

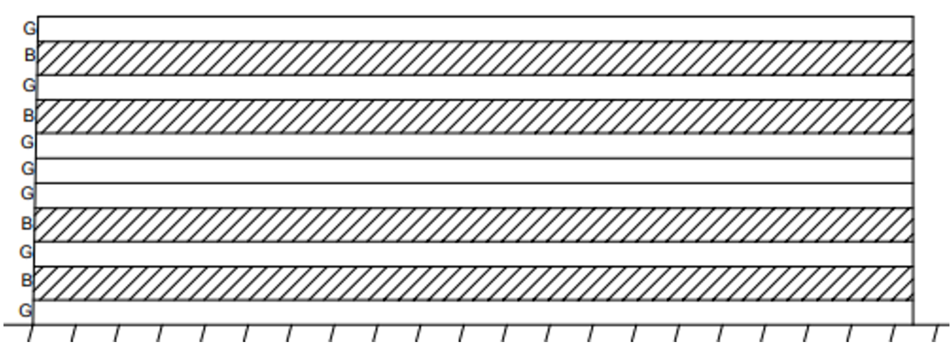
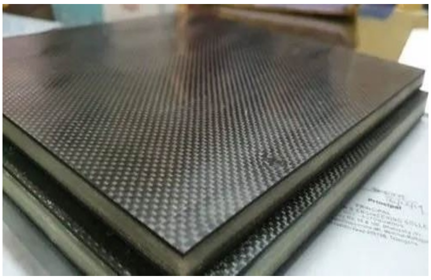
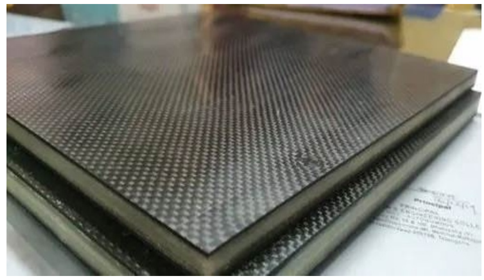


Fig. 2. Arrangement of layers.

The same procedure is followed to fabricate 0.4%, 0.6% and 0.8% fiber reinforced composite using basalt fiber and glass fiber with carbon nanotube nanoparticles of 16, 24 and 32 grams respectively. The fabricated composite laminates are depicted in Figure 3.



(a)



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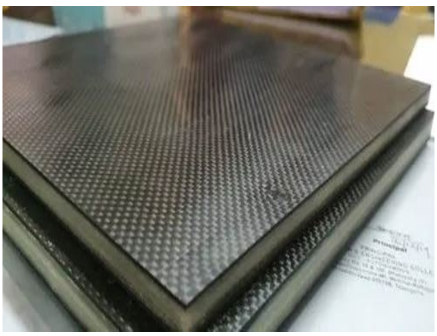

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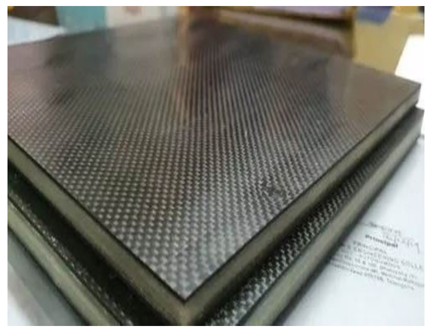


(d)

Fig. 3. Fabricated fiber reinforced composite (a) 0.2 W/V% (b) 0.4 W/V% (c) 0.6 W/V% (d) 0.8 W/V% CNT.

2.3 Calculations of volume fractions

0.2% CNT laminate

1. Total volume of each laminate=3.45*300*300mm^3= 310500 mm^3.
2. Volume of basalt fabric used in each laminate= 4*0.37*300*300mm^3= 133200mm^3.
3. Volume of glass fiber used per laminate= 7*300*300*0.24= 151200mm^3.
4. Volume of resin mixture used per each laminate= Total volume- (Volume of basalt to fabric+ volume of glass fiber) = (310500-133200-151200) = 26100mm^3.
5. % Volume fraction of basalt fabric used per laminate = (Volume of basalt to fabric/total volume) *100= (133200/310500) *100=42.9%.
6. % Volume fraction of glass fiber used per each laminate= (Volume of glass fiber/total volume) *100= (151200/310500) *100=48.7%.
7. % Volume fraction of resin mixture for each laminate= (Volume of resin mixture/total volume) *100= (26100/310500) *100=8.4%.

Similar calculations are carried and prepared the composites for 0.4%, 0.6% and 0.8% CNT laminates.

3 Results and discussions

Properties such as tensile strength, impact test, transverse load and flexural strength are evaluated on the fabricated composites. Fractured surface analysis is carried out using SEM. The thermo-mechanical properties like loss modulus, storing modulus, loss tan-delta are also evaluated.

3.1 Tensile test

The tensile strength of the manufactured composite specimen is determined through D3039 ASTM standards. Figure 4 shows the mean tensile strength at 0.2% CNT specimens is 344.3 MPa. The mean tensile strength at 0.4% CNT specimens is 396.5 MPa. The mean tensile strength at 0.6% specimens is 373.1 MPa. The mean tensile strength at 0.8% CNT specimens is 362.7 MPa. The 0.4% CNT specimens displayed the mean maximum tensile strength, while the low mean tensile strength is given at 0.2 % CNT specimens. The percentage change observed in the tensile strength between the maximum to minimum is 13.1%. The CNTs can withstand higher tension loads due to higher mechanical strength compared to other fibers.

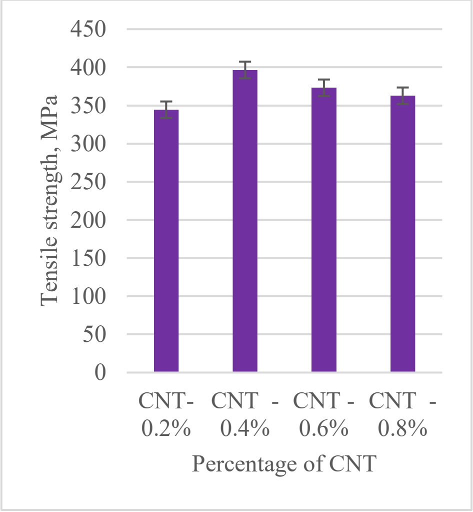


Fig. 4. Tensile strength.

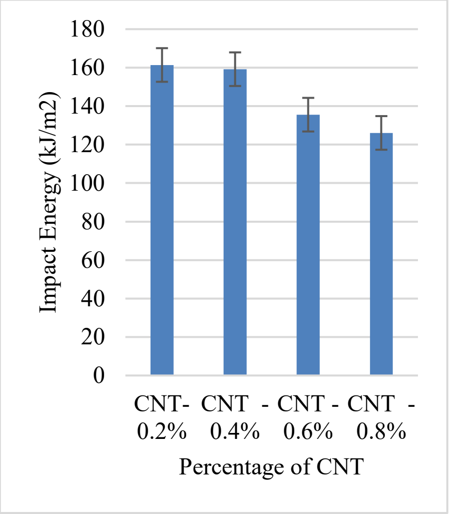


Fig. 5. Impact strength.

3.2 Impact test

Impact strength of the manufactured composite specimen is determined with D256 ASTM standards to evaluate impact resistance of the material with Izod impact test. The altitude the arm blows to later hitting the specimen is applied to calculate the impact strength. To compute impact load, notch sensitivity is generally used. The impact strength is a degree of an energy needed to rupture a test sample mostly found in joules. The impact energy is calculated as IE = mgH, where g is 9.8 m/s^2, m is the mass in kg and H is the load drop height in m. Figure 5 shows that the mean impact energy of 0.2, 0.4, 0.6 and 0.8 weight % of CNT specimens are 161.368 KJ/m^2, 159.198 KJ/m^2, 135.554 KJ/m^2 and 126.078 KJ/m^2 respectively. The maximum mean impact strength stored at 0.2 % weight of CNT specimens and the respective value is 161.3 KJ/m^2. The lowest mean impact energy is stored at 0.8 % weight CNT specimen, and the value is 126.0 KJ/m^2. The mean impact strength reduces by the weight % of CNT rises in laminates. The percentage change observed in the impact strength between the maximum to minimum is 21.88 %. CNT has more impact resistant; therefore, when enough amount of CNTs can improve the impact energy absorption in results.

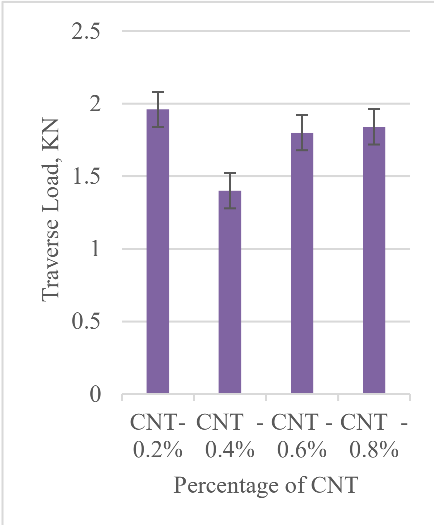


Fig. 6. Transverse load.

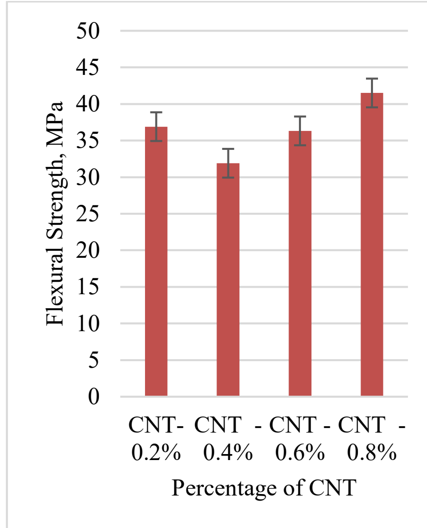


Fig. 7. Flexural strength.

3.3 Transverse load

The transverse load of the manufactured composite specimens is found using D3410 ASTM standards. Figure 6 shows that the average transverse load that can withstand 0.2, 0.4, 0.6, 0.8 weight percentages of CNT samples are 1.96 KN, 1.4 KN, 1.8 KN and 1.84 KN respectively. The average highest transverse load can withstand 0.2 weight percentage of CNT sample. The lowest bearing average transverse load is to withstand 0.4 weight percentage CNT. The percentage change observed in the transverse load between the maximum to minimum is 28.57%. The CNTs can withstand higher transverse load without inducing buckling or other undesirable failure modes.

3.4 Flexural strength

Flexural strength of the manufactured composite specimen is found with D790 ASTM standards. Figure 7 shows the mean flexural strength at 0.2% CNT specimens is 36.9 MPa. The mean flexural strength at 0.4% CNT specimens is 31.9 MPa. The mean flexural strength at 0.6% CNT specimens is 36.32 MPa. The mean flexural strength at 0.8% CNT specimen is 41.5 MPa. 0.8 % weight CNT specimen displayed the mean maximum flexural strength of 41.5 MPa. The least mean flexural strength is displayed at 0.4 weight % CNT samples. The percentage change observed in the flexural strength between the maximum to minimum is 23.13%. CNTs can withstand more loading compared to other fibres.
Similar results were observed in mechanical assessment on basalt/ carbon fiber-reinforced hybrid polymer matrix composite by Razan et al. [10].

3.5 Dynamic mechanical analysis (DMA)

DMA is a basic technique for measuring the behavior of composite under viscoelastic state. From these tests, the stiffness and (the ability of viscosity flow) of the illustration be able to be found out. The viscoelastic behaviour can change with time, frequency and temperature. During DMA, a sample is bent or twisted, and its reaction is recorded. The bending or twisting can be changing, constant, or applied at a fixed rate, and the reaction can be observed with the function of temperature. DMA finds out the stiffness, damping factor and loss stiffness as functions of temperature, time and frequency. Therefore, DMA can give information on behavior of material change and their properties under real time applications. For instance, DMA is applied to find out the glass transition of a material acts when it is loaded and unloaded. The stiffness of the substance can be calculated by the modulus (E), which is the slope of the plot of stress versus strain.

Dynamic testing with DMA instruments means making a sample vibrate with a changing stress, which causes a changing deformation. The connection between the changing stress and deformation is important for finding out the viscoelastic behaviour of the metal. The applied stress can be exhibited by a function of sine. Stress and strain can be written as $\sigma = \sigma_0\sin(\omega t+\delta)$; $y=y_0\cos(\omega t)$, here σo is the highest stress incorporated, ω is cyclic stress change, and t is time. The strain of a system that vibrates with changing stress is also changing, but the distinction in phase among strain and stress depends on how much the material acts like a liquid or a solid.

The solid-like response of a material can be compared to the storage of energy in a spring, while its liquid-like response can be seen as a reason of energy loss. The most frequent DMA test for solid materials is the temperature sweep. In this test, the temperature goes up slowly while the frequency and changing stress is constant. The temperature can go up in steps, with higher gaps (e.g., 5ºC) between measurements, or non-stop (1-2ºC/minute). When a polymer gets hotter, it changes Tγ and Tβ because it gets bigger, which also makes its stiffness change. The glass transition of polymers (Tg) is behavioural change in the temperature limit of 140-160ºC. There are different ways to find out the glass transition temperature, viz with complex modulus, storage modulus, and tan δ (vs temperature) based on the situation. Since these ways give different values, it is important to say which way was used for finding out. For example, while using the storage modulus, the Tg is found by the temperature when E' begins to go down. Also, peaks in loss modulus E" and tan δ display the glass transition and whichever the start or the highest number can be used to find out Tg. Figure 8 to 11 shows the samples used for DMA analysis.

The effect of reinforcing basalt and glass nanofibers is studied by analyzing the influence of various weight % (0.2, 0.4, 0.6, and 0.8 wt. %) of nano fiber reinforcement on a neat composite using DMA. Table 1 shows composite material changes with increasing temperature using CNT.

Fig. 8. Specimen of CNT 0.2 wt %.

Fig. 9. Specimen of CNT 0.4 wt %.

Fig. 10. Specimen of CNT 0.6 wt %.

Fig. 11. Specimen of CNT 0.8 wt %.

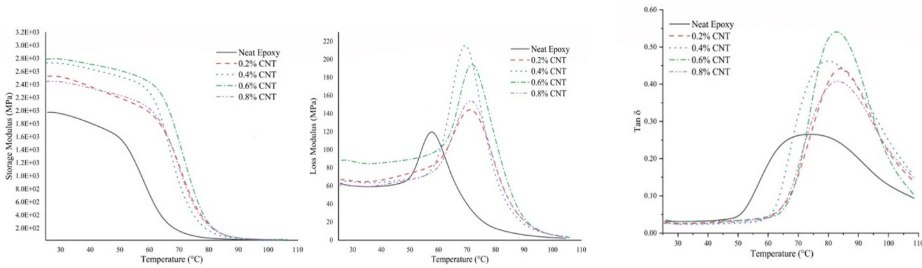
Table 1. Composite material changes with increasing temperature using CNT.

Temperature (°C)	Neat Epoxy (MPa)	Temperature (°C)	0.2% CNT (MPa)	Temperature (°C)	0.4% CNT (MPa)	Temperature (°C)	0.6% CNT (MPa)	Temperature (°C)	0.8% CNT (MPa)
25.625	62.958	25.777	67.222	27.307	65.111	26.145	88.374	25.105	61.038
27.648	60.775	28.159	66.399	29.496	65.067	28.175	88.999	27.591	61.031
31.536	59.564	32.234	64.442	33.464	63.075	32.025	85.155	31.603	59.154
36.308	59.080	37.072	64.913	37.824	63.458	36.259	84.228	36.033	58.862
41.361	59.681	42.102	67.397	42.884	64.789	40.768	85.790	41.081	61.750
46.552	62.283	47.360	72.030	48.161	66.950	45.375	87.177	46.376	64.559
51.866	68.583	52.517	76.044	53.345	68.460	50.202	89.460	51.551	67.869
57.579	146.39	57.607	81.265	58.464	74.854	55.316	92.631	56.646	72.324
64.286	63.892	62.778	93.427	63.689	129.12	60.442	99.476	61.727	82.705
74.313	19.402	68.459	146.48	69.659	270.52	65.589	118.89	67.366	142.09
81.464	11.093	74.361	148.27	77.415	75.549	71.327	229.13	73.261	171.06
87.013	7.155	82.567	46.961	87.216	19.978	78.142	131.62	81.130	55.195
92.820	4.502	91.454	14.219	94.578	9.607	86.818	28.885	90.824	15.181
98.881	2.907	98.268	6.408	100.30	5.639	94.944	9.714	97.746	6.967
104.52	2.039	103.75	3.557	106.13	3.399	100.741	5.021	103.438	3.849

3.5.1 Storage modulus, loss modulus and loss tangent

The stiffness of a viscoelastic material is denoted by the storage modulus E′, which displays how much energy is accumulated during a loading cycle. It also depends on the temperature of the composites. Basalt - glass fiber nanocomposites with different amounts of weight were experimented using the three-point bending approach, and graphs that compare the modulus of storage are exhibited with experimental values. Figure 12 demonstrations the storage modulus with temperature of the different weight percentage of basalt fiber nano composites for CNT. The outcomes indicate that the storage modulus of the nanocomposites goes up a little bit when nanofibers are added. The composite samples had a big drop in storage modulus between 52 – 68°C for CNT, where the glass transition occurs. The Tg of the composite without anything added was 52°C, while that of the basalt and glass fiber nanocomposites was between 60 –70°C. In both the solid and soft regions, the stiffness of basalt - glass fiber nanofiber-reinforced composites went up with more fiber content.

This observation could be attributed to the communication among the epoxy's hydroxyl group and the hydroxyl clusters present on harmed sites on the surface of basalt - glass nanofibers or due to atomic scale defects.



(a) (b) (c)

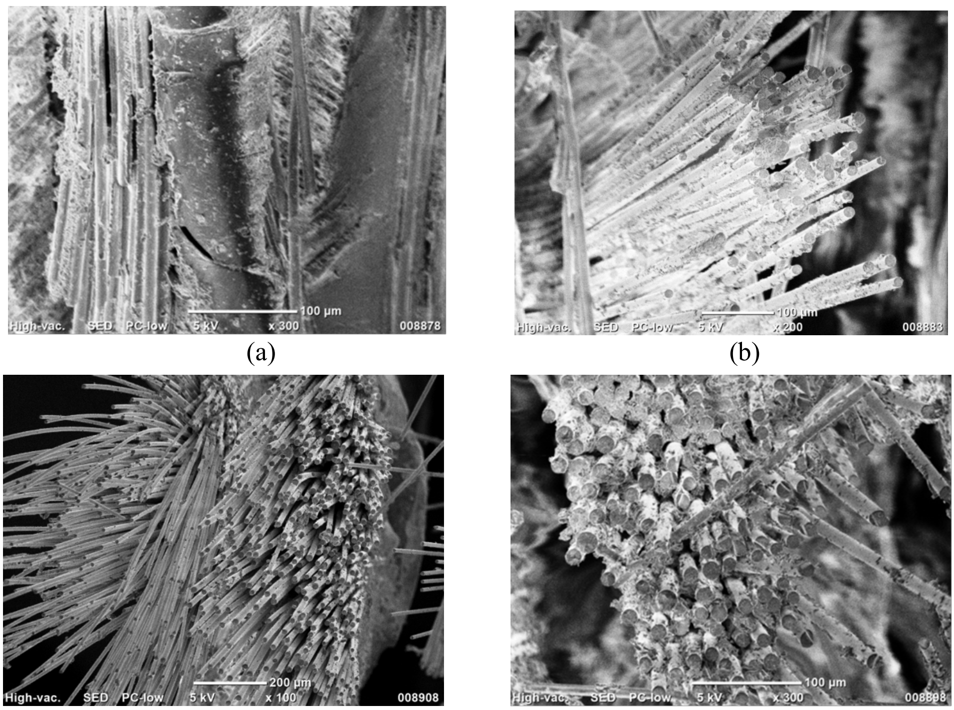
Fig. 12. Variation of DMA parameters with different weight percentage of CNT nano composites (a) Storage Modulus (b) Loss Modulus (c) Loss tan δ.

The rise in glass transition temperature indicates an increased level of interface between polymer chains and the surface of basalt fabric nanofibers. The presence of nano filler may restrict segmented chain motion and create an interphase region, leading to a rise in Tg of the soft phase. The storage modulus rises with the existence of basalt nanofibers, which can be attributed to a combination of nanofibers inserted in a viscoelastic matrix constraint.

At extreme focuses, fibers reduce matrix mobility and deformation, allowing stress to be transmitted from the epoxy matrix to the basalt and glass nanofiber reinforcement. The loss modulus, E´´ denotes energy lost as heat and is proportionate to energy dissipated through one loading cycle. It measures vibrational energy transformed through vibration and may not be retrieved. The loss modulus exhibits interesting behaviour and is temperature - dependent for basalt fiber nanocomposite specimens. The loss factor tan δ, signifies damping or friction in a viscoelastic arrangement and is the relation between loss modulus and storage modulus. It assesses energy exhaust in relations of recoverable energy and is communicated as a dimensionless quantity. An elevated tan δ value specifies a material with a great non-elastic strain constituent, whereas a low number specifies that high elastic.

3.6 Scanning electron microscope (SEM) analysis

A JEOL JSM-6480LV scanning electron microscope is exploited to look at the surface shape of the composite. The composite specimens are prepared well, desiccated in the air, and coated with platinum. The fracture face form of the composite samples is studied using SEM. The SEM is a commonly utilized tool with a large depth of focus, making it ideal for topographical imaging. SEM was employed to examine the surface morphologies and representative SEM phase images of fibers. Figure 13. shows the SEM images of different samples. Alkali-treated fibers exhibited significant changes in surface characteristics. Raw fiber surfaces have an uneven phase, as seen in SEM images. Figure 13 indicates a dense fiber microstructure. The morphology of basalt – glass reinforced with different percentages of CNT similar structure. 0.2% CNT composite shows more resin content than others. From SEM micrographs, it is straightforward to observe that the chopped glass fibers and basalt fibers were clearly disconnected and consistently dispersed throughout the specimens. The split into distinct filaments lead to improved bonding among fibers and resin as the borders were enlarged. Accordingly, the bond relating the fibers and the resin was retrieved to be intense, and these fibers affected abnormalities during the cracks in the matrix, needing additional power to fracture the samples.



(a) (b) (c) (d)

Fig. 13. SEM images of different CNT samples (a) 0.2 wt % (b) 0.4 wt % (c) 0.6 wt % (d) 0.8 wt %.

4 Conclusions

Four laminates of 0.2, 0.4, 0.6 and 0.8 weight percentage of CNT hybrid composites are fabricated using hand layup technique and the curing is done using hydraulic press. The mechanical properties viz. tensile strength, flexural strength, transverse load and impact energy are determined. The subsequent conclusions are drawn from the current work.

1. The 0.4 weight % sample exhibits the best tensile strength of 396.52 MPa. 0.2% of CNT has the lowest average tensile strength, measuring 344.396 MPa. The maximum average impact energy noted at 0.2 weight % specimens is 161.386 KJ/m^2 and minimum average is 126.078 KJ/m^2 at 0.8 weight %.
2. The 0.4 weight % of CNT specimens shown the lowest mean transverse load of 1.33 KN and the highest value is 1.806 KN at 0.2 weight %. The average maximum flexural stress is obtained at 0.8 weight % samples and the value is 41.52 MPa and the lowest flexural strength is noted at 0.4 weight % and corresponding value is 31.95 MPa.
3. The maximum tensile strength at 0.4%, maximum impact strength and maximum traverse load at 0.2 % and the maximum flexural strength at 0.8 % are observed.
4. From the dynamic mechanical analysis, 0.4 % CNT of basalt and glass fiber composite

indicates more loss modulus and 0.6% CNT has more mechanical damping system.

5. From SEM images, the chopped glass fibers and basalt fibers were clearly disconnected and consistently dispersed throughout the specimens. The fragmented and distinct filaments lead to improved bonding among fibers and resin.

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