Investigation of the Effect of Nano Particles on Visco-elastic Behaviour of CFRP Hybrid Nanocomposites

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Abstract
The aim of the study is to do an experimental analysis and investigation of visco-elastic behaviour of CFRP hybrid nanocomposites, which contains the small percentage of nanoclay and nanoZnO particles mixed for the improvement in the various properties of the CFRP composites. Also there is try to focus on the combined effect of another variable, i.e., fiber angle of orientation along with changing the percentage of nano particles. A hand layup method followed by process of vacuum bagging was used to make the samples of composite. Dynamic mechanical analyzer (DMA) was used to inspect the dynamic mechanical properties of CFRP hybrid nanocomposite specimens with changing temperature. The OVAT analysis is carried out for three mentioned variables and studied effect of variables on the various visco elastic properties like flexural storage modulus, loss modulus, Glass transition temperature and tanδ etc. It is found that due to the presence of nano particles there is tremendous change in the value of storage modulus ,it gets almost doubled, i.e., 200% because of the presence of nano particles. The experimental result also shows the positive effect on the loss modulus because of the nano particle contribution. Similarly there is considerable change in GTT(Glass transition temperature) and change in length in DMA (Dynamic mechanical analysis).

Keywords: Dynamic mechanical analysis (DMA); Visco elastic behaviour; hybrid nanocomposite; nanoclay; nanoZnO

1. Introduction
In the modern duration, fiber reinforced polymer is powerfully used in various components such as brakes, cams, gears and bearings etc, because of their and improved resistance to wear self-lubrication properties, lower friction. Polymer matrix composites are having higher strength to weight ratio, which is useful for various applications in an automobile, aerospace and various appliances. The manufacturing of material for a specific application depends on the variable like material expense, thickness, other working requirements and quality. The deficiency of polymers could be conquer effectively by using various special fillers like nano sized particles. The effects of nanoparticles addition on these properties have been broadly investigated. The addition of nanoparticles by weight of few percent can result in enhancement in mechanical properties significantly. Carbon fibers composites give greater properties such as Young’s modulus, higher strength, better thermal conductivity, and excellent electrical properties than other fibres. Many studies have focused on the incorporation of inorganic nanoparticles, which is a best approach to study the effect on visco elastic properties like storage modulus, loss modulus, GTT, tanδ etc.

Shazed M.A. et al showed that The CNT are also helpful in development of mechanical properties of CFRP composites, when outside layer of CNT applied with different fiber orientations [1]. Rashmi, et al. and Li Chang et al. presented work on the
improvement in the wear resistance of regular size and short carbon fiber composites in addition of nano particles. [2,3]. The astonishing mechanical, thermal and electrical properties of carbon nano tubes and nanoZnO have provoked researches to make use of them as a filler in composite in order to perk up the properties of the parent matrix[4,5]. Also many researchers have enlisted the effect of nanoparticles, fiber orientation etc, on the various carbon fiber composites by using dynamic mechanical analysis [6-11] With addition of nanoclay, the glass transition temperature can be increased as compared to neat CF/PPc composite demonstrating improved heat resistance and improved thermo mechanical properties with addition of organoclay as per the work of Mohamed H. et al [12] In the study by Declan Carolana et al discussed that the nanoparticles are eagerly transferable to a toughness increase in the interlaminar fracture energy of the CFRP composite laminates [13]. M. B. A. Salam et al highlights on the effect of addition of multi walled carbon nano tubes in carbon fiber, epoxy composites, which end results in improvement of the thermo mechanical properties of the composites [14,15]. Sasan Nouranian, et al. has done the optimization of the nano composites for study their consequence in DMA [16]. V.K. Tripathi and N. S. Kulkarni, worked on optimal design comprehensive procedure of a composite laminate taking various parameters [17]U.V Hambire, V.K. Tripathi et al. have done the evaluation and optimization of dental composites for mechanical properties, and Tripathi V.K., et al, enlisted orderly procedure to get best possible optimum condition of composition for friction liners material [18-20]. Likewise it is also studied that carbon nano fibres, natural nano banana particles and nanoclay having stronger effect on the mechanical and viscoelastic properties of the composites as shown by researchers in their respective studies[21-23] Yasser Rostamiyan et al.[24-26] mixed epoxy resin with nanoclay as nanoparticle in the glass fiber reinforcement and studied its effects on mechanical properties, and found that new nanocomposite possesses improved mechanical properties like flexural strength, tensile and impact These studies are carried out using the response surface methodology for the design of experiments. R. Satheesh Raja et al have studied effective improvement in the mechanical properties of GFRP composites with the implementation of micro fly ash [27]. In this paper there is experimental OVAT analysis of the Visco elastic behaviour in the Dynamic mechanical analysis (DMA) of the CFRP hybrid nano composite is presented. The combined effect of the three variables like weight percentage of nano clay and nano Zno particles and orientation angle of fiber is studied in DMA of CFRP nano composites. The OVAT analysis is carried out for three mentioned variables and studied effect of variables on the various visco elastic properties like flexural storage modulus, loss modulus, Glass transition temperature and tanδ etc. It is found that due to the presence of nano particles there is tremendous change in the value of storage modulus ,it gets almost doubled ie 200% because of the presence of nano particles. The experimental result also shows the positive effect on the loss modulus because of the nano particle contribution .Similarly there is considerable change in GTT(Glass transmission temperature) and change in length in DMA(Dynamic mechanical analysis ).

Figure 1: (a) Hand Lay up of CFRP,(b) Vacuum Bagging
2. Experimental

2.1. Details of material

For the experiment in this assessment CFRP epoxy composites treat as controlled sample. The unidirectional carbon fabrics having standard modulus $3K$ of 200 GSM, code HCU200. The resin named Hinpoxy C, which is Bisphenol -A based liquid resin beside with the amine modified hardener known as Hinpoxy hardener-B is a colorless, and small viscosity liquid with stoichiometric ratio of A:B=10:3 was used. Resins, hardener with carbon fiber, supplied from the Hindoostan Mills Ltd. Mumbai, India. The nanoclay was montmorillonite, modified surface with layers of Aluminoisillicate (approximately thickness of 1 nm), and SpGra. Zinc oxide -ZnO nanoparticles, with Purity of 99.9%, SSA: 20-60 m2/g, with Particles Size: 30-50nm, Morphology: nearly spherical, True density: 6 g/cm3 density for Bulk: 0.28 - 0.48 g/cm3 were used, supplied from Nano Research Lab. Jamshedpur, (Jharkhand), India.

2.2. Fabrication of composites

CFRP hybrid nanocomposites laminates were made-up by using hand layup method (as shown in Fig.1.(a) followed by process of vacuum bagging (as shown in Fig. 1.(b) ). The epoxy resin system is a Diglycidyl ether of Bisphenol A (DGEBA) mixed with 1,3-phenylenediamine hardener at a ratio by weight of 100:30. The carbon fibers unidirectional of 200 gsm were used as main reinforcement in the matrix. The Montmorillonite nanoclay and Zinc Oxide nanoparticle was dried out for overnight at 75°C in an oven previous to use for compose suitable for spreading in epoxy resin. The nano particle content was preferred in between the 1% to 5% for mixing in the epoxy resin and hardener. For the making of specimens, 14-16 nos. plies were used according to ASTM standards; accordingly the sub specimens are cut of the required size. For executing the mixing of the particles uniformly in the resin mixture, the ultra sonication of the mixture was done for 40 minutes at 40°C temperature, and then this mixture is magnetically stirred with 800 rpm for one and half hour. After completing the mixing treatment he hardener is mixed and this mixture is used for the preparation of the CFRP hybrid nanocomposites laminates by hand layup succeeded by process of vacuum bagging as shown in Fig.1. For process of vacuum bagging, pump was used for the period of 5 hours with a stable of 720 mm Hg vacuum pressure. And the specimen was put in the bag for 16 hours duration of curing at room temperature. After the curing, specimen was kept in an oven for process of post curing for five hours at 80°C.

Figure 2: (a) DMA 242 E Artemis set up, (b) Specimen Holder in DMA setup, (c) Specimens of coded units
3. Dynamic Mechanical Analysis (DMA)

DMA is an extensively used method to determine the effect of temperature on the properties of damping and various moduli of materials by application of sinusoidal force. Modulus is calculated in 2 parts, these are storage modulus, $E'$ and loss modulus, $E''$. The relation of loss modulus to the storage modulus is known as mechanical damping (given by $\tan \delta$ is a dimensionless number) it gives the energy lost or can also be expressed in form of recoverable energy. The higher $\tan \delta$ value is indicates that material has higher non elastic strain, while lower $\delta$ shows that material is more elastic. Storage modulus elaborates the elastic behavior of the CFRP and which is proportionate to the energy stored in one cycle, while loss modulus gives the viscous properties of material, which is proportionate to energy dissipated in one cycle. The phase angle, $\delta$ which is the phase difference between the dynamic stress and strain of viscoelastic material under the effect of sinusoidal oscillation. This dynamic mechanical test is used to derive the viscoelastic and elastic behaviors of CFRP hybrid nanocomposites sample with the sinusoidal force application. The thermal analyses were completed by using a DMA242E analyzer (Netzsch, Germany) in a mode of three-point-bending with the oscillation of 1 Hz (as shown in Fig. 2.(a) ). The storage modulus ($E'$), damping factor ($\tan \delta$) and loss modulus ($E''$) of each CFRP hybrid nanocomposite sample were determine under the subsequent conditions: a temperature range from 40°C up to 150°C at rate of heating of 3°C/min, as a cooling agent the liquid nitrogen is used by putting in the machine fixture (as shown in Fig. 2.(b). The samples were sized in 40 mm in length and 4 mm in width, while the thickness were around 3.2 mm(as shown in Fig. 2.(c).

4. Experimental OVAT analysis and results

The DMA test is carried out to study the various changes in thermal behaviour or viscoelastic properties of the CFRP hybrid composites. This experimental study highlights the effect of variable quantity of nano particles along with variation of the fiber orientation angle on the CFRP hybrid nano composites properties like storage modulus, GTT, Loss modulus etc. In the OVAT=I analysis by varying the weight percentage of nano zinc keeping nano clay percentage and fiber orientation angle constant the effect is studied. Then simultaneously OVAT-II and OVAT-III is carried out by varying the weight percentage of nano ZnO and fiber orientation angel. The results of DMA test are along with the controlled sample ie for plain CFRP composites as shown in Table 1 and Table 2.

Table 1: OVAT analysis values and controlled sample values of storage and loss modulus along with temp at loss modulus

| Sr. No | Fiber Angle | % of Clay | % of ZnO | Storage Modulus MPa | Peak Loss Modulus/Temp MPa/°c |
|--------|-------------|-----------|-----------|---------------------|-------------------------------|
| I-A    | 0°          | 2         | 2         | 7122                | 1549/67.5                    |
| I-B    | 0°          | 2         | 3         | 4800.93             | 1128/70.9                    |
| I-C    | 0°          | 2         | 4         | 18197               | 3218/65.4                    |
| II-A   | 0°          | 2         | 2         | 5386                | 1265/66.2                    |
| II-B   | 0°          | 3         | 2         | 11899.54            | 1905/65.8                    |
| II-C   | 0°          | 4         | 2         | 5279.62             | 973/65.7                     |
| III-A  | 0°          | 2         | 2         | 7122                | 1549/67.5                    |
| III-B  | 45°         | 2         | 2         | 10282               | 2135/67.7                    |
| III-C  | 90°         | 2         | 2         | 3877                | 708/65.8                     |
| PC     | 0°          | 0         | 0         | 6428                | 2798/76.6                    |
Table 2: OVAT analysis values and controlled sample values of Tan δ and Glass transition temperature (GTT)

| Sr. No | Fiber Angle | % of Clay | % of ZnO | Tan δ | GTTemp °C |
|--------|-------------|-----------|----------|-------|-----------|
| I-A    | 0°          | 2         | 2        | 0.755 | 70.8      |
| I-B    | 0°          | 2         | 3        | 0.798 | 75.2      |
| I-C    | 0°          | 2         | 4        | 0.758 | 69.9      |
| II-A   | 0°          | 2         | 2        | 0.808 | 69.8      |
| II-B   | 0°          | 3         | 2        | 0.767 | 70.5      |
| II-C   | 0°          | 4         | 2        | 0.661 | 71.1      |
| III-A  | 0°          | 2         | 2        | 0.755 | 70.8      |
| III-B  | 45°         | 2         | 2        | 0.787 | 70.6      |
| III-C  | 90°         | 2         | 2        | 0.634 | 71.5      |
| PC     | 0°          | 0         | 0        | 0.599 | 82.1      |

Figure 3: Showing the Storage modulus and GTT and Tanδ values of OVAT-I compared with plain CFRP sample

From the above figure No 3. This is the output of given directly by Dynamic mechanical test analyser. It can be understood that how there is drastic improvement in the value of storage modulus in the flexural test and also here is increase in the inflection temperature with the small decrease in GTT (Glass transmission temperature), as compared with the CFRP controlled sample. Similarly in the Figure 4. Showing the Loss modulus and temperature of OVAT-I, (in which the wt. % of nano clay is kept constant) compared with plain CFRP sample, the loss modulus is also increasing than the controlled sample but it is desirable with due increase in the storage modulus.
As shown in the figure 5, it can be seen that the in OVAT-II, (in which the wt. % of nano ZnO is kept constant), the flexural storage modulus is almost doubled as compared with plain CFRP sample, with the small decrease in the GTT and considerable increase in the value of Tan δ. And as reflected in the figure 6, showing the Loss modulus and temperature of OVAT-II, compared with plain CFRP sample, the loss modulus is also increasing than the controlled sample but it is desirable with due increase in the storage modulus.
Figure 6: Showing the Loss modulus and temperature of OVAT-II compared with plain CFRP sample

Figure 7: Showing the Storage modulus and GTT and Tanδ values of OVAT-II compared with plain CFRP sample

After the OVAT-II, There is variance of the fiber orientation angle in the OVAT-III, by keeping the wt. Percentage of nano clay and nano ZnO constant. It is obtained from the dynamic mechanical analysis/test of CFRP hybrid nano composites with variance in the orientation angle and keeping the nanoparticle wt percentage at the value of 2% of both nano clay and nano ZnO. The graph presents that more than double improvement in the storage modulus due to flexure as compared the controlled sample of CFRP. Also it
shows that there is increase in the inflation temperature and the value of \( \tan \delta \) (damping factor).

According to the graph in figure 8, which gives the value of loss modulus with the change in temperature it can be seen that the peak value of temperature and loss modulus is high.

![Figure 8: Showing the Loss modulus and temperature of OVAT-II compared with plain CFRP sample](image)

For the controlled sample of CFRP as compared with the CFRP hybrid nanocomposite samples. Hence it is proved from the experimental analysis that along with the nano particle presence there is also effect of the change in fiber orientation angle on the visco elastic properties of CFRP composites.

5. Conclusion

Carbon fibre reinforced composites with and without nano particles were fabricated and tested for the dynamic mechanical analysis and visco elastic properties. For dispersing nanoparticles in the epoxy system, a combination of sonication and magnetic stirring methods was used. For fabricating laminate composite, combination of hand layup and vacuum bagging methods was used. From the results of the tests it is concluded that the presence of the nano particles with varying their wt. percentage in epoxy system of CFRP composites are having greater value around more than 200% is increased in the value of storage modulus due flexure and hence its having high loss factor too. There is also moderate change in the GTT and peak temperature of the CCFRP with nano particles. From the results of OVAT-III, it is proved that there is also effect of change in the fiber angle orientation on the visco elastic behaviour of the CFRP hybrid nano composites. Hence it is observed that there is definite enhancement in the CFRP composite because of the mixing of controlled wt. Of nano particles. Also fiber orientation angle is also having considerable effect on thermo mechanical properties of CFRP along with nanoparticles.
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