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Synthesis of Glass Fiber Reinforced Polymer Composites Incorporating MWCNTS and Graphene: A Study on Dynamic Mechanical Behavior and Cole–Cole Representation

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Abstract

Glass fiber reinforced polymer (GFRP) composites were fabricated using multi-walled carbon nanotubes (MWCNTs) and graphene as nanofillers through the hand lay-up technique. Two sets of composites were prepared by incorporating MWCNTs and graphene separately into the epoxy resin matrix at varying weight percentages (2%, 4%, 6%, 8%, and 10%). Mechanical and viscoelastic properties were evaluated following ASTM standards, and Dynamic Mechanical Analysis (DMA) was performed to study their mechanical and thermal behavior. The results indicated that the incorporation of nanofillers enhanced the overall properties of the composites, with optimal performance observed at 4% weight for MWCNTs and 6% weight for graphene. Comparative analysis showed that graphene-reinforced composites exhibited superior mechanical performance, suggesting more effective dispersion and interfacial bonding within the GFRP matrix than MWCNT-based composites. Viscoelastic behavior studied DMA revealed that the storage modulus (stiffness) of graphene-reinforced composites increased across all tested temperatures and frequencies. Additionally, the glass transition temperature (Tg) shifted significantly from 95°C to 110°C, indicating restricted polymer chain mobility due to the presence of graphene nanofillers. Cole–Cole plots derived from storage and loss modulus values demonstrated that the composites followed Debye relaxation behavior. These plots also provided insights into phase heterogeneity and dispersion quality of nanofillers within the epoxy matrix. Moisture absorption tests and X-ray diffraction (XRD) analysis further supported the improved performance and structural characteristics of the nanocomposites. Overall, the study concludes that graphene is a more effective nanofiller than MWCNTs for enhancing the mechanical and viscoelastic properties of GFRP composites. Its superior dispersion and interaction with the polymer matrix contribute significantly to improved strength, stiffness, and thermal stability.

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Keywords: Glass Fiber, Multi-Walled Carbon Nanotubes (MWCNTs), Graphene, Dynamic Mechanical Properties, Cole–Cole Plot.

INTRODUCTION

The main objective was to understand the reinforcing behaviour of Graphene nano Filler and Multi-Walled carbon nano tubes Nano filler in epoxy Glass Fiber Polymer Composites by correlating its mechanical and dynamic properties with the interfacial morphological structure [1]. This work examined the influence of nano fillers on epoxy matrix and properties of epoxy glass fiber polymer composites [2], [4]. The Glass Fiber

Reinforced Polymer composite specimens ultrasonicated with nano fillers namely Multi-Walled Carbon Nano Tubes (2%, 4%, 6%, 8% and 10% by weight) and Graphene (2%, 4%, 6%, 8% and 10% by weight) are Fabricated [3]. The functional properties of nano fillers by various Dynamic Mechanical Analysis tests are investigated [8], [9]. The effectiveness of Graphene and Multi-Walled Carbon Nano Tubes in Glass Fiber Reinforced Polymer composites are determined [12]. Graphene and Multi-Walled Carbon Nano Tubes in the form of Nano Platelets were obtained from a local source and used for the fabrication of Polymer Nano composite. Plain weaved bidirectional type glass fibers were used as fibers for reinforcement [3]. Epoxy resin with controlled amount of Hardener HY951 was used to manufacture the composite specimens [5], [10]. A unique method utilizing an ultrasonicator tailed by different steps was used to mix the nano fillers and resin to improve the dispersion [6]. Two types of glass fiber polymer nano composites based on Graphene and Multi-Walled Carbon Nano Tubes at different percentages of inclusion were fabricated [7]. Ultimately Dynamic Mechanical Analysis test were performed to investigate the effect of functionalization of Nano fillers (MWCNTs and Graphene) [11]. From the review of literature, it is found that the addition of nano filler materials such as graphene and carbon nano tubes enhances the mechanical and thermal properties of Glass fiber polymer composites. Moreover, the nano filler materials occupy larger surface to volume ratio and reduce interlaminar shear to a great extent and can help rectify the drawback of gas permeability in polymer composites [12]. Among the various fibers, glass fibers possess higher strength than carbon, basalt fibers and are easily available at reasonable cost [7]. Glass fibers are more economical than carbon and basalt fibers. Further, nano fillers reinforcement in epoxy resin matrix augments the interfacial adhesion between the matrix and the fiber. From the literature available, it is learnt that all the researchers have fabricated and conducted experiments only with a maximum of 4% weight Nano fillers. Hence in this research, the work has been focused to investigate the effect of nano fillers (Graphene and Multi-Walled Carbon Nano Tubes) upto 10% weight on the properties of Glass Fiber Reinforced Polymer Composites.

MATERIALS AND METHODS

Introduction to Materials and Methods

Synthetic fibers are used as reinforcements in manufacturing polymer composites. Among man-made fibers such as glass, carbon, and basalt, Glass fibers are the best fillers owing to their cost and acceptable mechanical properties. Nano fillers, in addition to glass fibers, have attracted researchers and helped them to improve reinforcement effect in polymers. Graphene and Multi-Walled Carbon Nano Tubes have been incorporated with epoxy resin by academicians and industrialists to overcome the drawback of interlaminar shear that occurs with glass fibers. The main reason for the use of glass fibers and nano fillers as reinforcement is its biodegradability that contributes to a better eco system along with their low and threshold limits.

Materials

After a detailed survey of literature available, glass fiber with nano filler materials (Graphene and MWCNTs) were reinforced with epoxy resin for this work.

Multi-Walled Carbon Nano Tubes Nano Fillers

Multi-Walled Carbon Nano Tubes are powdery in nature and they are chosen with minimum purity of 97% MWCNTs. They are opted with OD<8 μm, 4 to 15 number of walls, density of 0.15 to 0.35 g/cm3 and length of 10 to 30 μm. Multi-Walled Carbon Nano Tubes were used as received from local source: Mahalakshmi Chemicals, Chennai.

Graphene Nano Fillers

Graphene, an allotrope of carbon, is chosen with carbon purity of minimum 99.5%. Graphene is available as powder with APS of 15 microns. The thickness of graphene utilized was about 6 to 8 nm and with surface area of about 150 m2/g. Graphene was used as purchased from a local source: Mahalakshmi Chemicals, Chennai. Table 1 depicts the mechanical properties of the epoxy resin and hardener, and Table 2 presents the mechanical properties of the graphene nanofiller utilized in the fabrication of composite laminates.

Table 1. Properties for epoxy resin LY5561 and hardener HY9511

Properties for Epoxy Resin LY5561	
Elasticity Modulus (kN/ mm^2)	3.2–3.5
Compressive Strength (N/mm^2)	128–146
Tensile Strength (N/mm^2)	85–92
Elongation of Break (%)	7–9.5
Density (g/ cm^3)	1.11–1.32

Table 2. Properties of Graphene Nano Filler

Properties of Graphene Nano Platelet Powder	
Thickness (mm)	6–8
APS (Micron)	15
Carbon (%)	99.5
Surface Area (m^2/g)	150

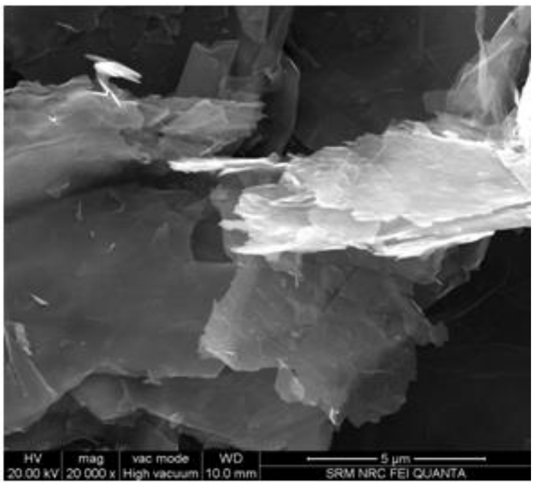
The scanning electron microscopic image of graphene nano filler material with intensity of image ranging from 1µm to 50µm is shown in the figure 1 (a), (b), (c) and (d).

It is obvious from the Scanning Electron Microscopic images, that graphene powders are superimposed and wrinkled together.

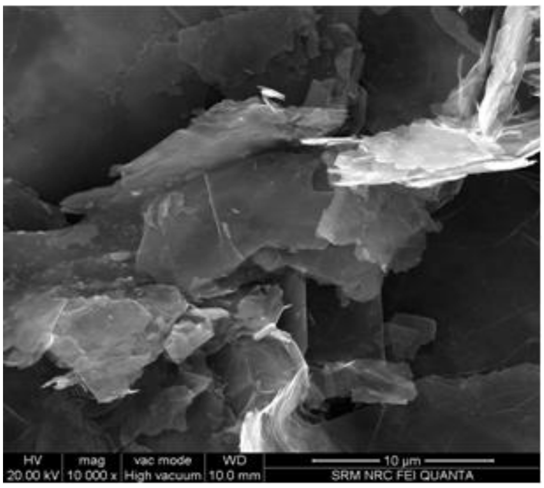
Glass Fibers, Resin, and Hardener

Glass fibers are most reliable among the fibers as they are relatively stiff and having higher tensile strength when used in manufacturing polymers. The glass fibers are woven and oriented directionally as shown in figure 2 for the manufacture of polymer composite specimens.

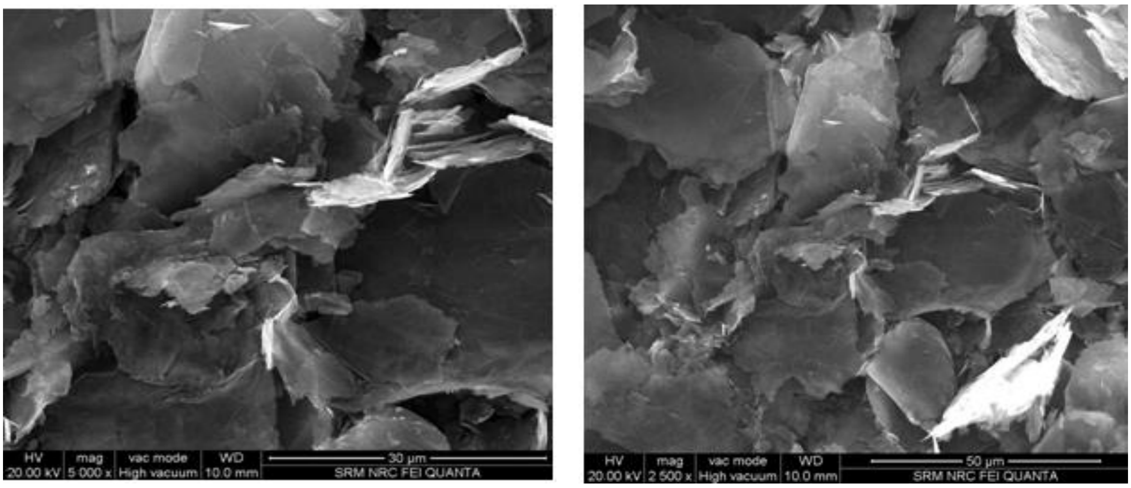
Standard directionally woven glass fibers are purchased from local source: Sakthi Glass Fiber Traders, Chennai, for fabrication. Epoxy resin used for matrix is Araldite LY 5561 bisphenol-A type. With a low viscosity the properties of Epoxy resin LY 5561 are as follows:



(a). Scanning Electron Microscopic image of Graphene powder at 5 µm



(b). Scanning Electron Microscopic image of Graphene powder at 10 µm



(c). Scanning Electron Microscopic image of Graphene powder at 30 µm

(d) Scanning Electron Microscopic image of Graphene powder at 50 µm

Figure 1. Scanning Electron Microscopic Images of Graphene Powder at various intensities

Figure 2. Image of Bidirectional woven glass fiber

Tensile Strength–85 to 92 N/mm2, Compressive Strength–128 to 146 M/mm^2, Percentage of Elongation–7 to 9.5%, Elastic Modulus–3.2 to 3.5 kN/mm^2, Density–1.11 to 1.32 g/cm^3.

Hardener is used for curing and to enable acquitting well for encapsulation. Hardener type Aradur HY951 is chosen for fabrication.

Fabrication of Polymer Composites

The stage by stage fabrication of GFRP composite is depicted in the figure 3.

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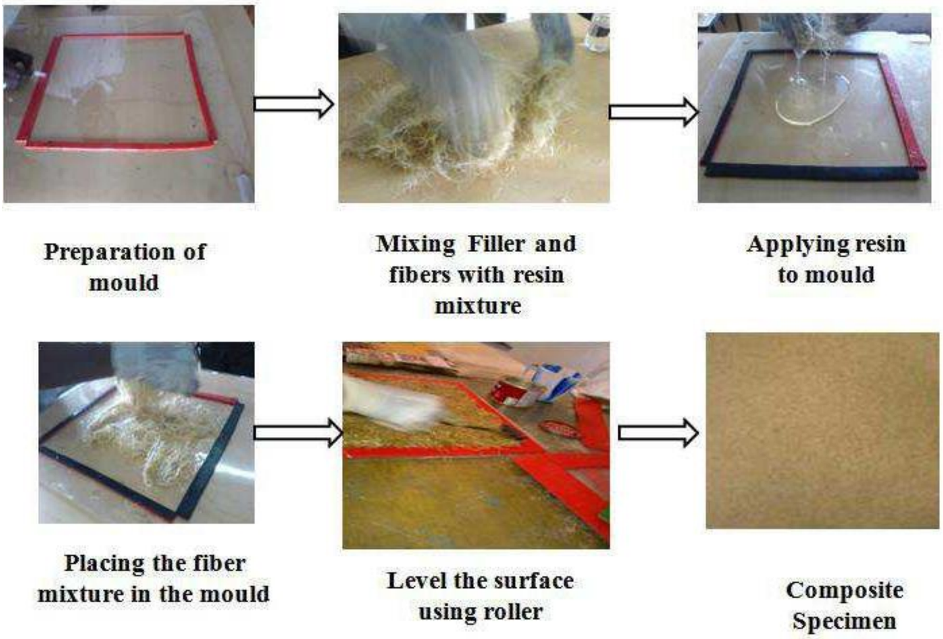

Figure 3. Fabrication of polymer composite

Fabrication of MWCNTS Dispersed GFRP Composites

The sequence of current work (MWCNTs dispersed GFRP) is given in the form of a flowchart in figure 4.

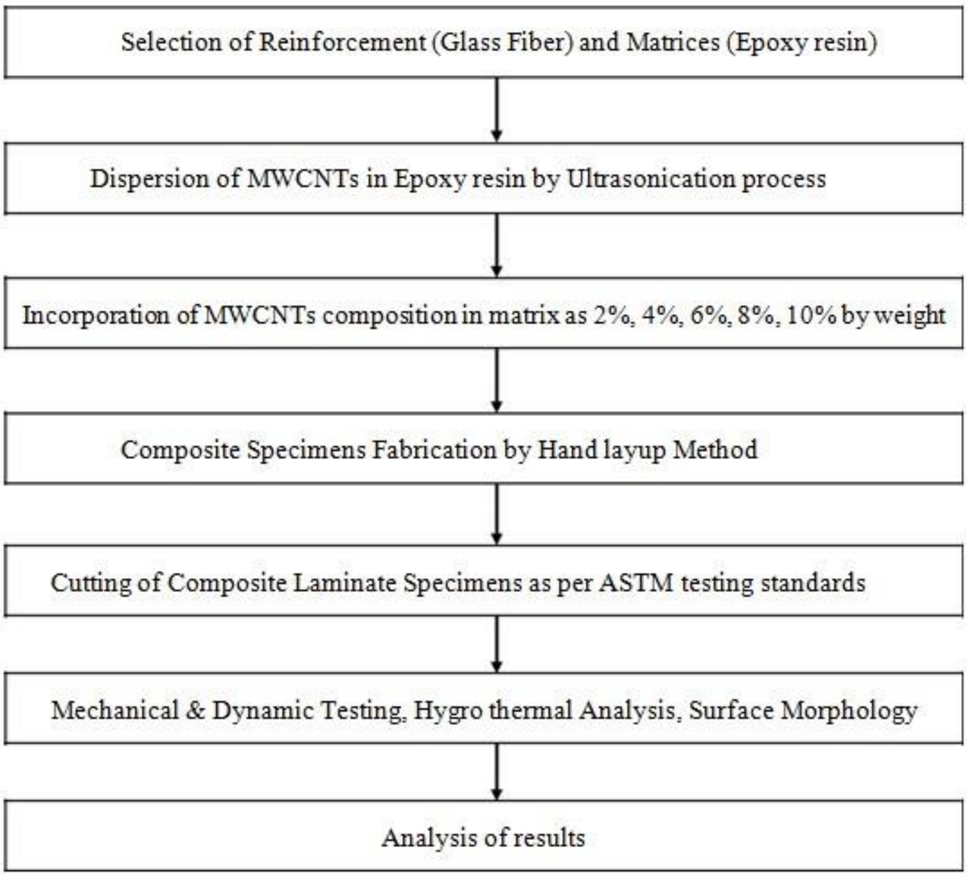

Figure 4. Fabrication of MWCNTs dispersed GFRP

Figure 4 shows the Fabrication flow process of MWCNTs/GFRP Composite Multi-Walled Carbon Nano Tubes nano fillers were used along with epoxy resin. The Nano filler material was amalgamated into the epoxy resin matrix with varying weight percent (2, 4, 6, 8, & 10%). An ultrasonicator was utilized to stir the MWCNTs and epoxy resin matrix for 60 minutes continuously to achieve better dispersion and uniform distribution. MWCNTs were incorporated with epoxy resin matrix at suitable weight percentages and composite specimens pertaining to suitable weight percentage of MWCNTs prepared using Hand–Layup method. Composite specimens for different mechanical testing following ASTM standards were fabricated.

The silicon rubber mold was used for the fabrication of composite plates. Rigid plates covered the top and the bottom of the mold. Initially, a known amount of filler resin matrix was poured into the mould. Then bidirectional woven glass fiber was spread and a compressive force of 500 N was applied to fabricate the composite laminate.

Fabrication of Graphene Dispersed GFRP Composites

The sequence of current work (Graphene dispersed GFRP) is given in the form of a flowchart in figure 5.

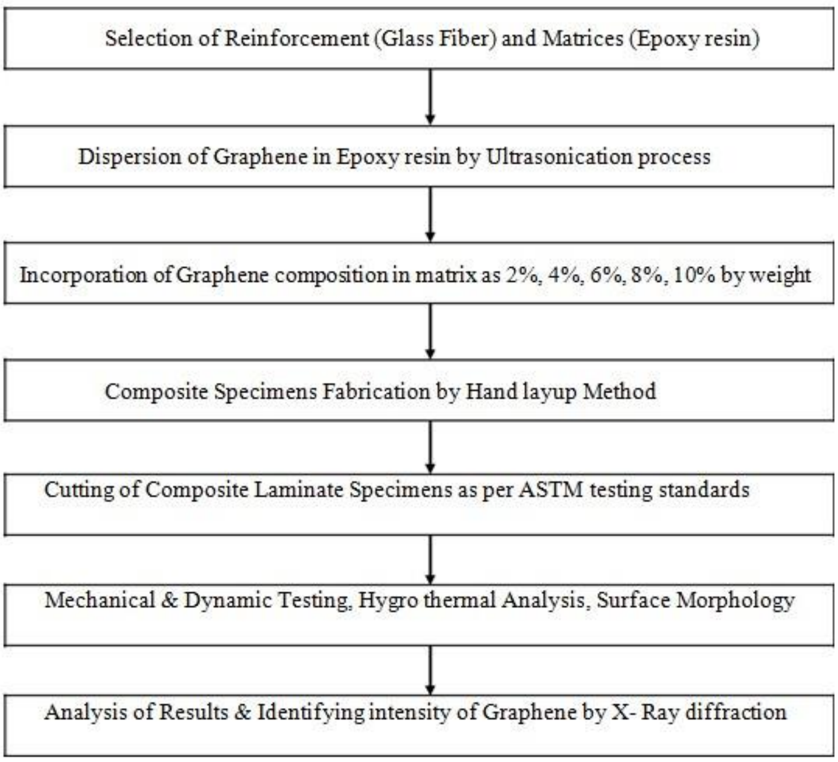

Figure 5. Fabrication of Graphene dispersed GFRP

Figure 5 shows the Fabrication flow process of Graphene/GFRP Composite Composite Laminates incorporating Graphene nano fillers were manufactured by Hand Lay-up method tailed by compression after curing. The composite laminates were fabricated. In this present work, epoxy resin was used as matrix and graphene as nano filler material. Graphene was extracted from carbon in the form of powder. The fine graphene in suitable eight percentages (2, 4, 6, 8, & 10%) were incorporated with epoxy resin matrix using ultrasonicator for better dispersion and distribution. The epoxy resin matrix incorporated with graphene is poured into the mould made of silicon rubber. The mixture was allowed to cool with the help of hardener at room temperature for 6 to 8 hours. The effects of graphene on mechanical properties were investigated and studied. Visco Elastic Properties were also analysed using Dynamic Mechanical Analyser.

Dynamic Mechanical Analysis

Dynamic Mechanical spectroscopic analysis is a technique used to study the Visco–Elastic properties of polymer composite samples resulting from changes in variables, namely time, temperature, frequency, force and strain. ASTM standard D5023 was followed to experimentize DMA with specimen dimensions as 50 x 12 x 3 mm. A sinusoidal force was applied with a controlled temperature and frequency in DMA to measure sinusoidal deformation i.e., visco-elastic properties of GFRP composite samples.

Standard dynamic mechanical analyzer MetraVib machine shown in the figure was utilized with frequencies varying as (0.5, 1, 2, 5, & 10 Hz) with the temperatures ranging from 20^0 to 150^0 C. in DMA samples being clamped between two arms placed in parallel. The distances between the arms are adjusted by means of precise slide to accommodate sample length varying from 1 mm to 65 mm. An electro mechanical motor attached to the arm drives the sample to a selected frequency or amplitude. The composite sample undergoes deformation when it is displaced. As a function of stress applied, the samples response, strain and frequency is measured using a linear variable differential transformer (LVDT) and the feedback of the same is provided. The different parameters of interest in this work are Loss modulus, storage modulus, Damping factor, and Glass Transition temperature. The stiffness of the material is regarded as Storage modulus (E') and the Loss modulus (E") is the energy lost in internal motion.

The damping property of composite sample can be measured by damping factor using Tan δ = E"/E'.

RESULTS AND DISCUSSION

Dynamic Mechanical Analysis on GFRP Composite Incorporated With MWCNTS

Storage Modulus (E')

The study on Visco–Elastic properties of GFRP composites is of great significance as filler reinforced fiber polymers undergo various dynamic stresses on application of loads. The temperature effects of nano filler loading (2%, 4%, 6%) on storage modulus of GFRP composites at altered frequencies are revealed in figures 6, 7 and 8.

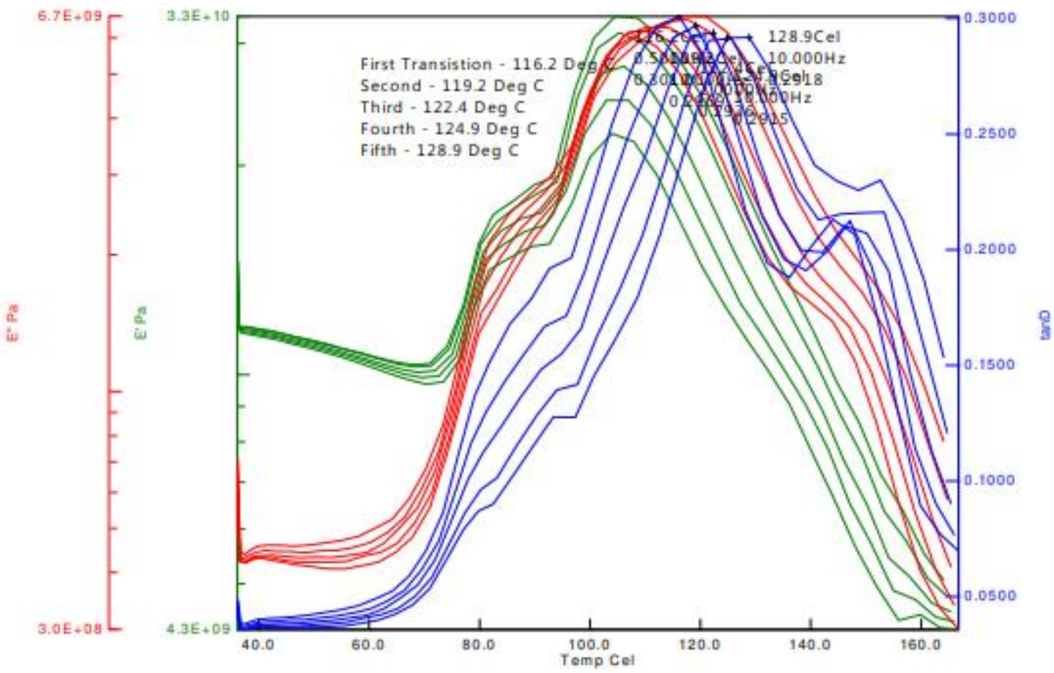

Figure 6. Influence of Temperature on Visco-elastic Properties with 2% Weight MWCNTs reinforcement in GFRP

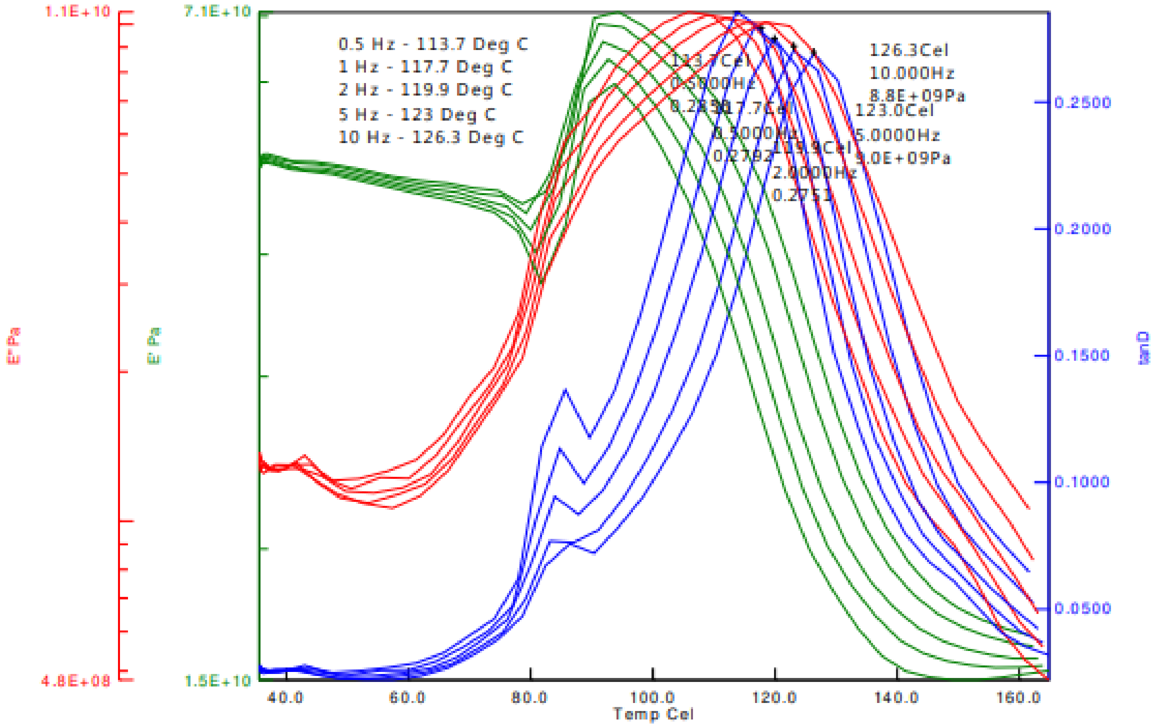

Figure 7. Influence of Temperature on Visco-elastic Properties with 4% Weight MWCNTs reinforcement in GFRP

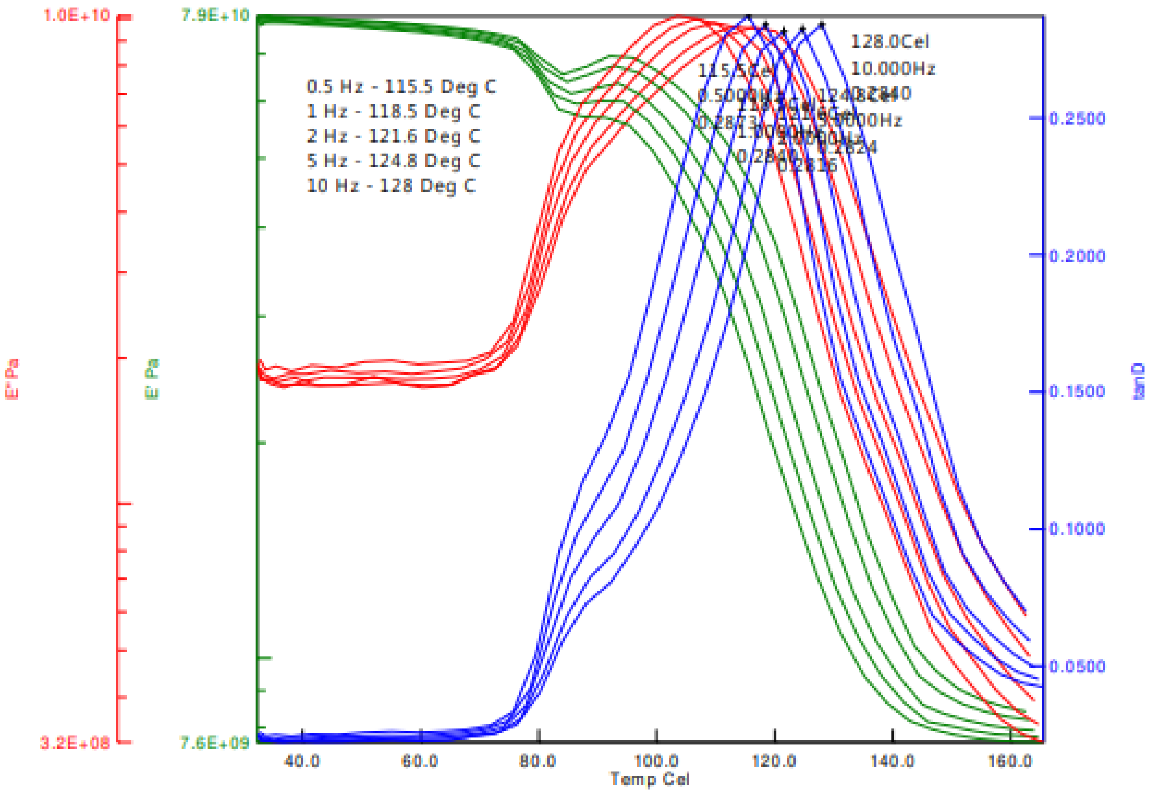

Figure 8. Influence of Temperature on Visco-elastic Properties with 6% Weight MWCNTs reinforcement in GFRP

The figures show that the epoxy resin with lesser nano filler loading acquires lower storage modulus among all the GFRP composites due to its lower stiffness compared with the other composite specimens. The incorporation of larger amount of MWCNTs reveals improvement in absorption energy in the composite. The energy storing capacity is increased on accumulation of nano fillers. The storage modulus values of all the figures show a flat region at lower temperatures and then the curve drops in a steep manner causing the value of modulus to decrease gradually. This implies that the composite transforms from glassy state to softened rubbery flow region with increase in temperature.

At lower glass transition temperature, the GFRP with MWCNTs causes free movement of molecules in the polymer chain initializing the physical cross links. At greater glass transition temperature, the stiffness of the GFRP composite incorporated with MWCNTs is enhanced restricting the free molecular crusade in the polymer chain when heated. The addition of nano filler increased storage modulus value in both rubbery and glass regions. An observation shows that the storage modulus is high when measured at higher frequency (short time), whereas when these composites are exposed at lower frequencies (Long time), it resulted in lower storage modulus values. This is due to the localized stresses being minimized by the rearrangement of molecules in the GFRP composite.

Loss Modulus (E")

The measure of viscous response of a polymeric material is the Loss modulus and the energy dissipated as heat is measured. The overall resistance to deformation i.e., the inelastic state of polymer is termed as Loss modulus or viscous modulus. Figure shows the Loss modulus values of Glass Fiber composite reinforced with different weights of MWCNTs. It is observed that at lower temperatures the value is less and at higher temperatures & nano filler loadings the loss modulus values attain a peak followed by a drop. The resistance to deformation is due to entanglement and occlusions as the polymer chains encounter each other while undergoing changes to get a new shape of polymer material.

Damping Factor (Tan Δ)

The ratio of Loss modulus (E") to the storage modulus (E') of the composite is the damping factor or loss factor or shift factor. It is the energy that is dissipated in the composite specimen during the loading of nano fillers. Damping factor is dimensionless one and it is the ability of the composite material to absorb vibrational energy. The figures show damping factor (TAN δ) of MWCNTs filled GFRP composite at various frequencies (0.5, 1, 1.5, 2, 2.5 Hz). The damping factor for low MWCNTs weight composite is high and on addition of nano fillers, the tan δ value decreases, and then on further addition, shows a slight increase in the damping factor value with higher temperature. The molecules in the polymer start mobilizing at glass transition temperature causing a higher tan δ value. Upon increasing the temperature and weight of nano fillers (MWCNTs), the molecular movement inside the polymers are interrupted resulting in a lower tan δ value. The energy is dissipated when the nano filler contents are added beyond a threshold limit causing agglomeration in the matrix and thereby decreasing the volume of matrix.

Dynamic Mechanical Analysis on GFRP Composite Incorporated with Graphene Nano Filler

Storage Modulus (E')

The response of composite, i.e., deformation of sample on application of sinusoidal stress was monitored as a function of temperature and time.

In DMA technique, the storage moduli (E') exemplify the stiffness of composite material. The stiffness of composite or storage modulus is directly proportional to the stored energy as a function of temperature and time. Graphene, an allotrope of carbon remains to be a trending nano filler material opted by researchers in recent years due to its exceptional properties.

The phase shift analysis was made between the response and the deformation. The sample responses for elastic, viscous, and visco elastic behaviours are shown in figure 9.

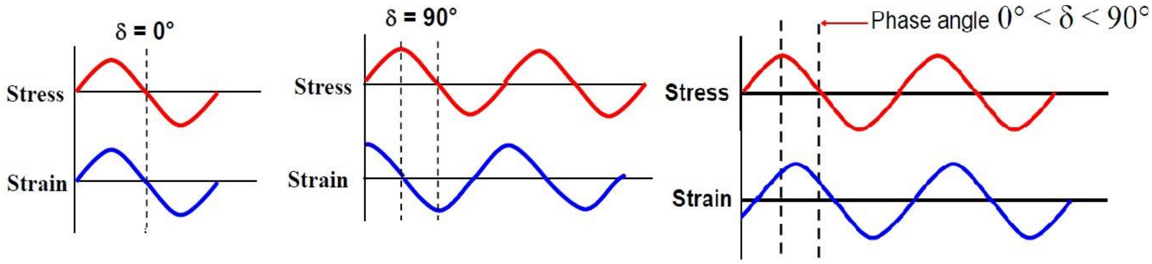

Figure 9. (i) Elastic Response, (ii) Viscous Response (iii) Viscoelastic Material Response

The molecular structures with difference in composition were measured using DMA and the physical molecular structure and chemical composition of polymer composites were indicated. The figures 10, 11 and 12 shows the value of storage modulus obtained with different nano filler compositions.

The value of storage modulus dropped after 80°C and there was deep fall with increase in the temperature. The storage modulus or stiffness of the composite increased with increase in graphene weight.

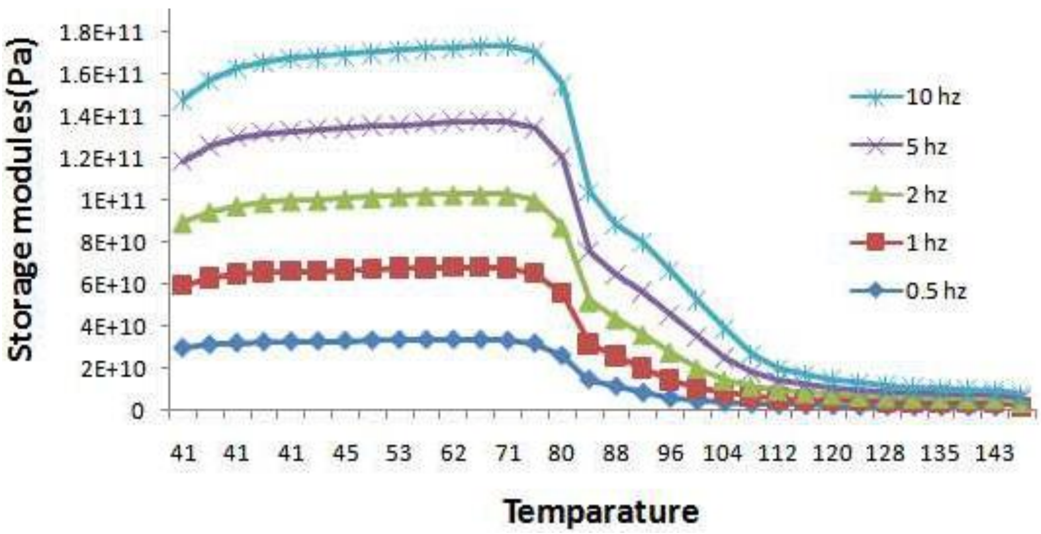

Figure 10. Influence of Temperature with 2% Weight Graphene enhancement in GFRP on Storage Modulus

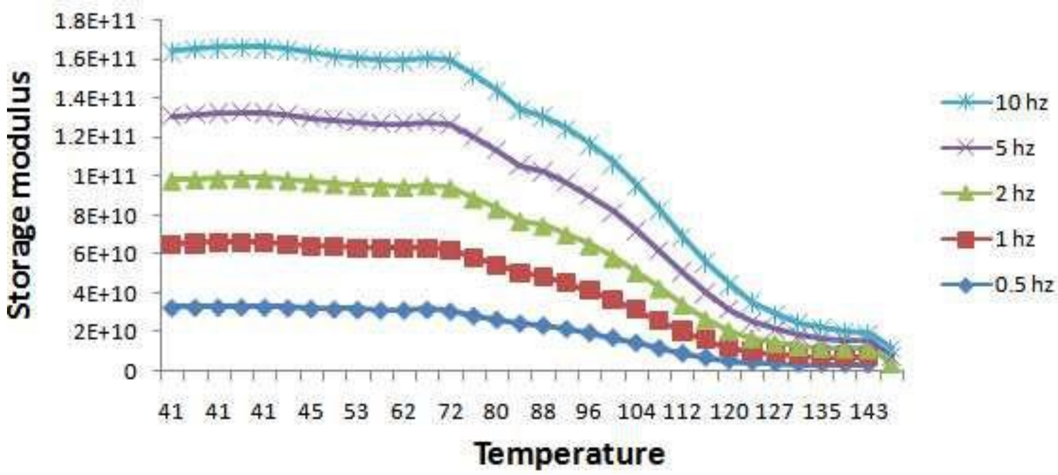

Figure 11. Influence of Temperature with 4% Weight Graphene enhancement in GFRP on Storage Modulus

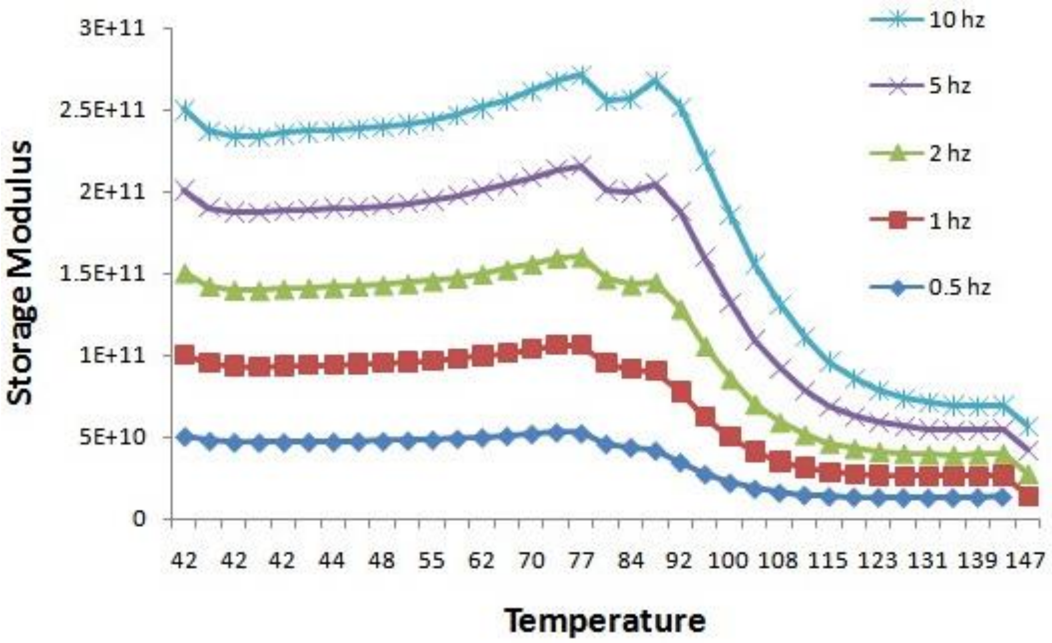

Figure 12. Influence of Temperature with 6% Weight Graphene enhancement in GFRP on Storage Modulus

The values of storage modulus increased upto 100°C followed by a drop with 6% weight of Graphene composite. The deterioration of polymer composites occurred on addition of graphene nano filler exceeding a certain extent. DMA was utilized to analyse where and how the mechanical properties occured and varied with respect to temperature and time. Various visco-elastic properties such as loss modulus, storage modulus, damping factor, and glass transition temperature were determined as a function of time and temperature and the same for different nano filler loadings is shown in figures 13, 14 and 15.

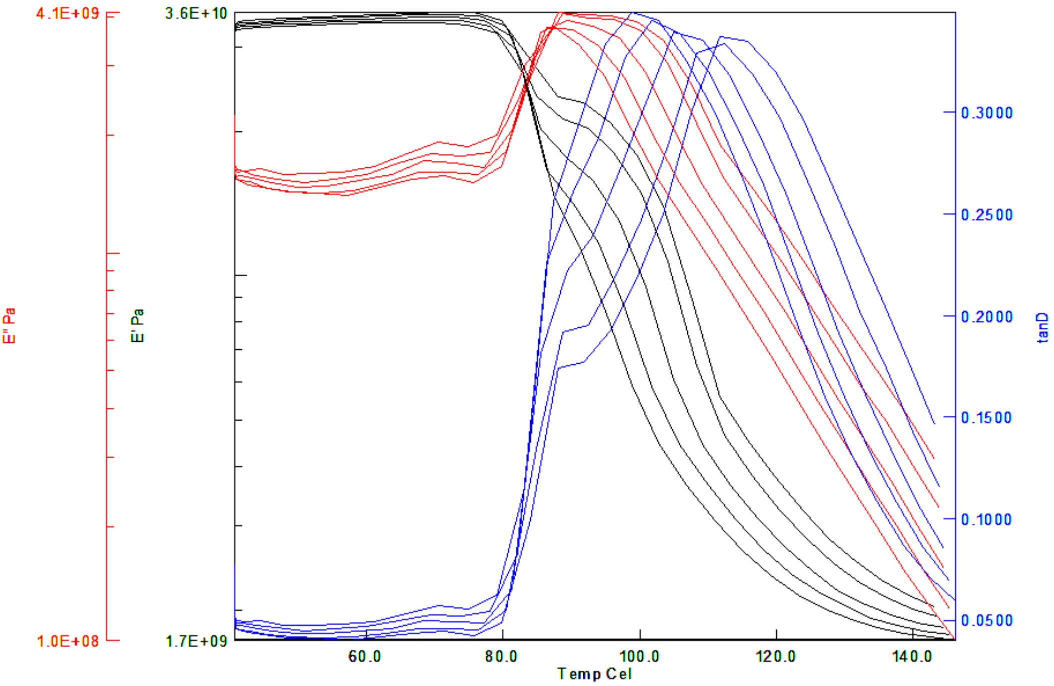

Figure 13. Influence of Temperature on Visco-elastic Properties with 2% Weight Graphene reinforcement in GFRP

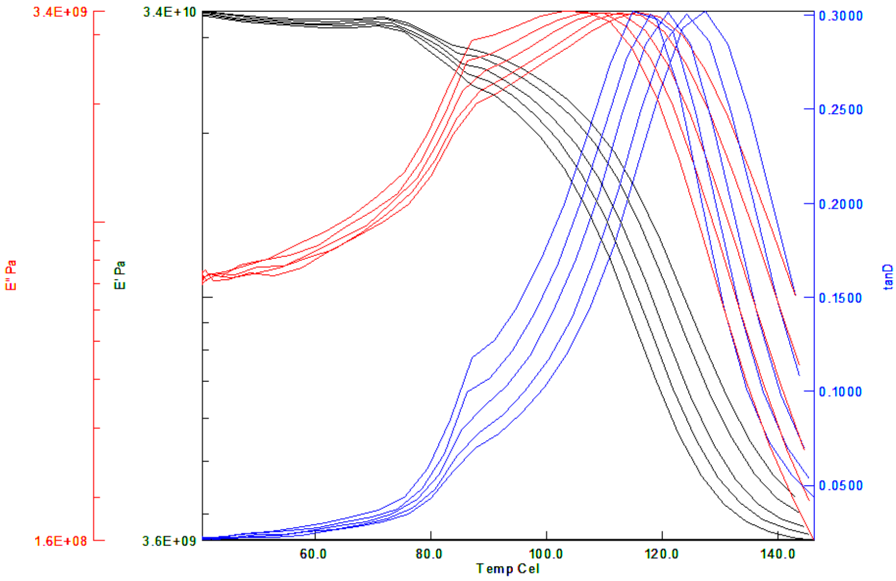

Figure 14. Influence of Temperature on Visco-elastic Properties with 4% Weight Graphene reinforcement in GFRP

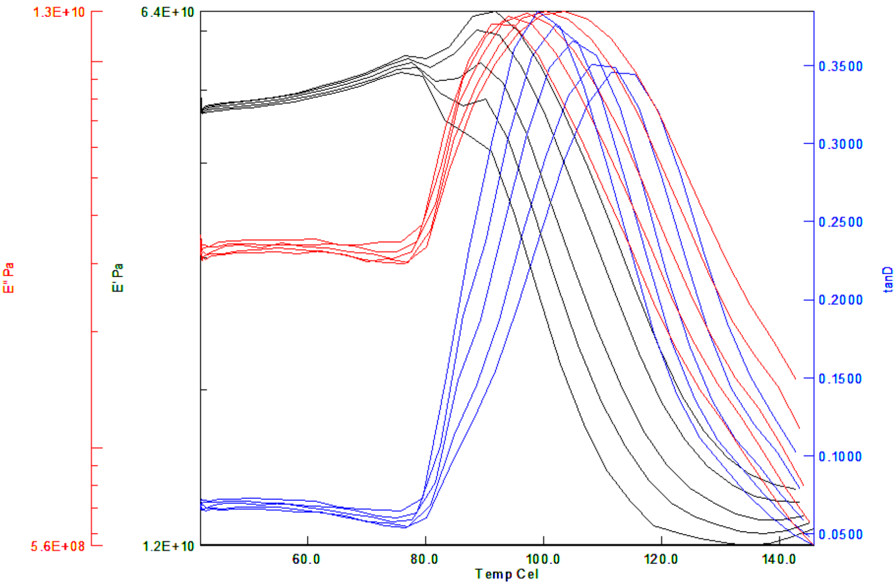

Figure 15. Influence of Temperature on Visco-elastic Properties with 6% Weight Graphene reinforcement in GFRP

Loss Modulus (E”)

The energy loss in medium or internal friction is regarded as Loss modulus or viscous behaviour. The dynamic and mechanical behaviour of composites are reliant on temperature and time. There are four regions in viscous elastic gamut as shown in figure 13 namely glassy, glass transition, rubber plateau and liquid flow regions that exhibit amorphous nature of polymer composites.

Loss modulus or internal friction exemplifies the lost energy due to heat that is proportional to dissipated energy. The graphene reinforced GFRP composites with various compositions show a rise in loss modulus after the glassy region. A peak is attainted during the glass transition region and then upturns with increase in temperature. This shows the molecular motion between graphene nano filler and the matrix as a possibility of increase in loss modulus. The effective dispersal of graphene nano filler into the resin matrix exhibits the mobility of polymer molecules upto the glass transition region.

Journal of Polymer & Composites
Volume 14, Special Issue 2
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Figures 16, 17 and 18 shows the viscous properties comparison graphs with respect to 2%, 4%, and 6% weight addition of graphene nano filler.

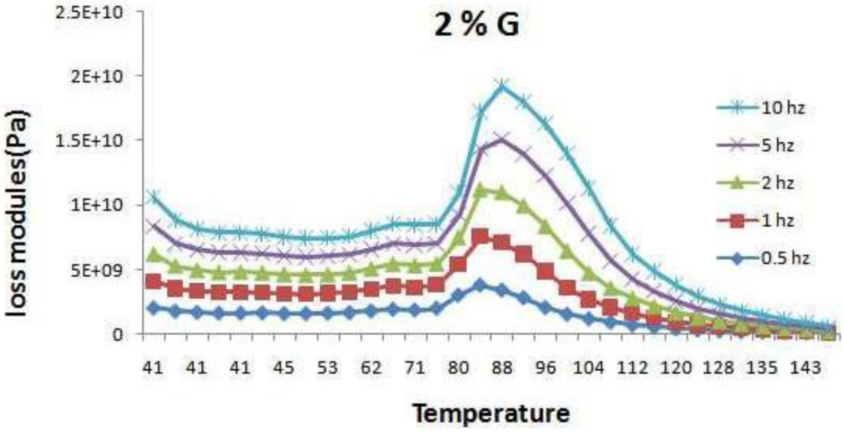

Figure 16. Influence of Temperature with 2% Weight Graphene enhancement in GFRP on Loss Modulus

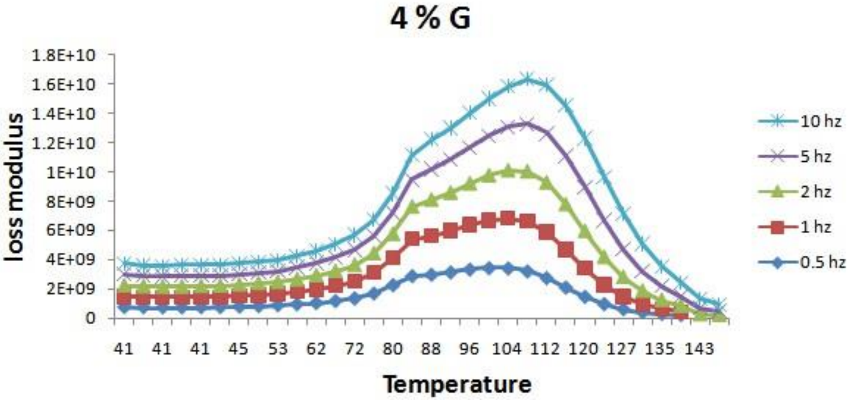

Figure 17. Influence of Temperature with 4% Weight Graphene enhancement in GFRP on Loss Modulus

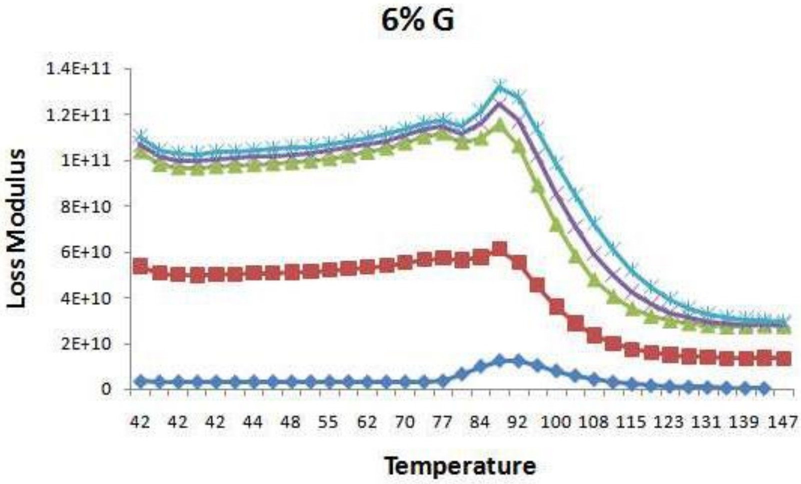

Figure 18. Influence of Temperature with 6% Weight Graphene enhancement in GFRP on Loss Modulus

The loss modulus dropped at 100°C with 6% weight of graphene to GFRP whereas glass transition temperature was between 85°C and 125°C.

Damping Factor (Tan Δ)

Damping factor is the phase shift or loss factor that is denoted by tan δ.

Tan δ = Loss Mudulus (E")/ Storage Modulus (E')

It exemplifies the dynamic mechanical damping behaviour in visco elastic system that is in either way regarded as the measurement of energy loss with an increase in temperature and also expresses the recovered energy. The shift factor tan δ for 2%, 4%, and 6% weight of graphene is shown in figures 19, 20 and 21.

A lower peak in the curve indicates elastic strain and a higher peak in the curve indicates the inelastic strain. The homogeneous state of the polymer chain was identified using width of tan δ value or shift factor. When the width of the curve is broader, it indicates the polymeric chain structure had significant motion in viscous action on increase in temperature. The peak is narrower for 6% wt graphene GFRP composite indicating narrow distribution i.e., homogeneous dispersion of graphene in molecules of polymer composites. The damping co- efficient curves for 2% and 4% wt of graphene have a broader peak indicating agglomeration of nano filler.

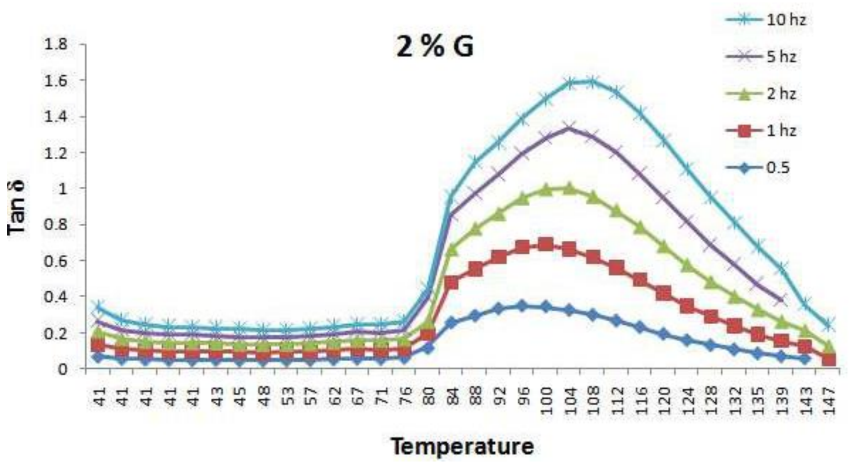

Figure 19. Influence of Temperature and 2% Weight Graphene enhancement in GFRP on Damping Factor

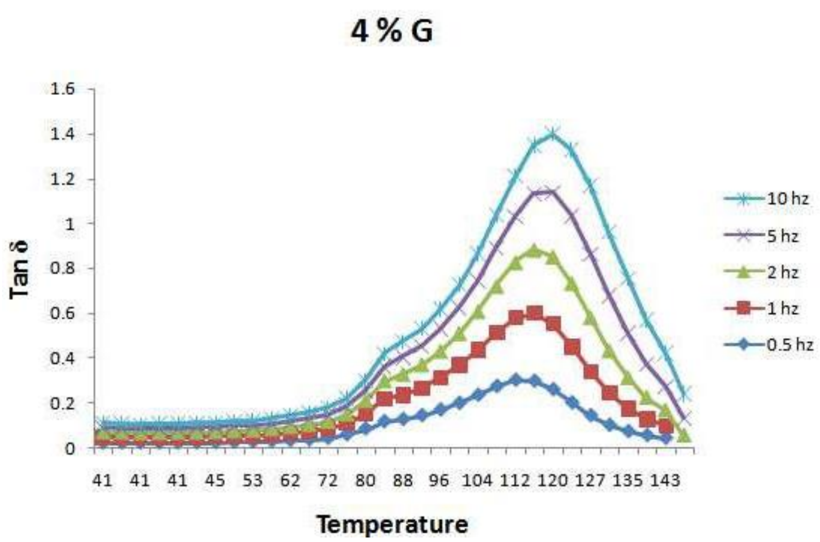

Figure 20. Influence of Temperature and 4% Weight Graphene enhancement in GFRP on Damping Factor

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Volume 14, Special Issue 2
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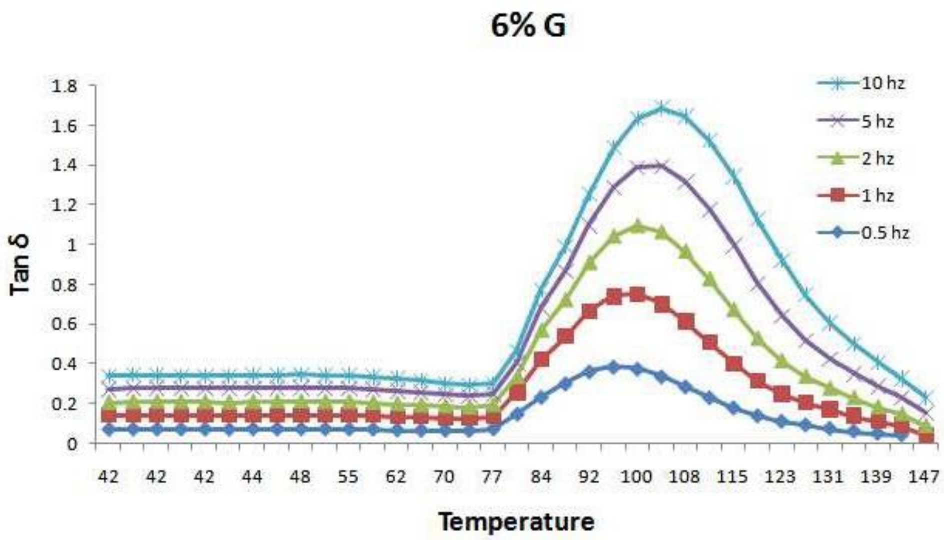

Figure 21. Influence of Temperature and 6% Weight Graphene enhancement in GFRP on Damping Factor

Cole–Cole Plot

Cole–Cole Plot is the representation of complex dielectric frequency dependent functions namely Loss modulus and storage modulus in graphical form. Cole–Cole plot is drawn only for polymeric material that obeys Debye relaxation process. It has one or more processes relaxed with comparable frequencies. The Cole–Cole plot is manipulated as a semi circle for best polymers by plotting storage modulus values in horizontal axis and Loss modulus values along vertical axis.

The dispersal of graphene nano fillers in glass polymer composites is characterized by drawing a cole–cole plot using the experimental datas obtained between storage and loss modulus.

The linear behaviour of visco elastic properties of graphene reinforced GFRP composites with the glass transition temperature is addressed by cole–cole plot. The molecular structural changes on strengthening of nano filler materials in polymer composites are exposed. The cole–cole plot drawn using experimental data is shown in figure 22.

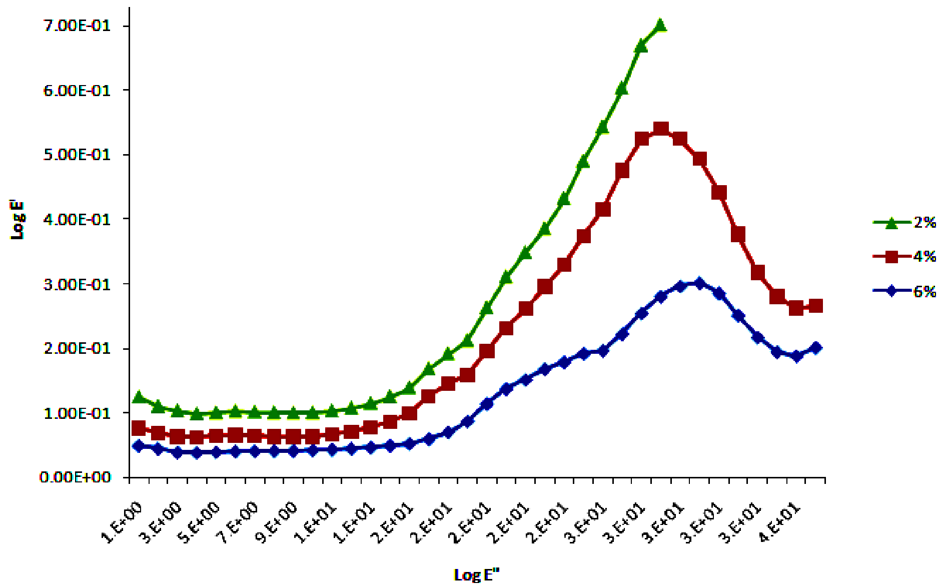

Figure 22. Cole-Cole Plot of Graphene reinforced GFRP Composite

The curve for 6% weight of graphene is semi-circular in shape indicating the homegenic nature of polymeric composite that obeys Debye relaxation process. The curves for 2% wt and 4% wt of graphene reinforced GFRP are not in the shape of semi-circle denoting heterogenic nature of composite. It can be inferred that the dispersion of nano fillers with epoxy resin matrix is better with 6% wt graphene reinforced GFRP composite.

CONCLUSIONS

The effect of nano fillers (Multi-Walled Carbon Nano Tubes and Graphene) on the dynamic properties of epoxy–glass fiber composites were analysed by conducting experiments. In addition, optimum percentage addition of nano fillers in epoxy nano composites was evaluated along with bidirectional orientation of glass fibers. The following conclusions were derived from this work: Nano composites were fabricated with 2%, 4%, 6%, 8% and 10% weight of MWCNTs and Graphene in the epoxy resin matrix individually.

Visco–Elastic properties were investigated by Dynamic Mechanical Analysis to evaluate the influence of MWCNTs nano filler loading on GFRP Nano composites. It showed that the glass transition temperature (Tg) shifted from 85°C to 110°C due to addition of MWCNTs. Further, the damping factor was found to decrease with the addition of nano filler. This showed that the MWCNTs filler restricted free molecular movement at high temperatures, thereby increasing dissipation of energy.

The influence of Graphene on the Visco–Elastic behaviour of GFRP composites was studied by using DMA technique. The stiffness of the composite (Storage Modulus) had increased by the addition of Graphene at all temperatures and frequencies. The Glass transition temperature was found shifted from 95°C to 110°C interlocking the mobility of polymer molecules by the addition of Graphene Nano filler.

The nature of Graphene reinforced GFRP composite was characterized by drawing Cole–Cole plot obeying Debye Relaxation process. Cole–Cole plot was drawn with the experimental values of storage modulus and Loss modulus. The curve for GFRP composite with 6% weight Graphene was semi–circular in shape indicating the homogeneity in the composite, whereas Cole–Cole plot of the GFRP composite with 2% and 4% weight Graphene Nano filler had shown a deviation from semi–circle indicating heterogeneity nature of composite. It was clearly evident from the mechanical property values that Graphene Nano filler had an effective incorporation with GFRP composite compared with MWCNTs and it could be concluded that Graphene Nano filler is optimum filler that can be incorporated with fibers to increase its strength thereby having an effective dispersion into the matrix.

From this study, the most important factor in the incorporation of nano fillers is that it can be added only to a certain extent and when these Nano fillers are added beyond a threshold limit, deterioration in the properties occur due to various defects such as nano void formation, agglomeration of fillers, fiber pull out and interlaminar crack propagation, etc.

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