



Influence of temperature on viscoelastic performance of epoxy resin composites reinforced with one dimensional fillers

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Abstract: The viscoelastic behavior of composite materials is dependent on a number of factors such as reinforcement geometry, dimensionality, and continuity. The present article examines the viscoelastic properties of epoxy resin, epoxy matrix composites reinforced with cross-plyed E-glass fibers, and epoxy matrix composites filled with multiwall carbon nanotubes (MWCNTs) with respect to storage modulus, loss modulus, loss factor (tan), and glass transition temperature as examined by dynamic mechanical thermal analysis (DMTA). The findings showed that when the temperature was raised, there is increase in the storage modulus, loss modulus, and loss factor of the pure epoxy resin, cross-plyed E-glass fiber-reinforced epoxy resin polymer matrix (42% fiber volume fraction), and 2% MWCNT-filled epoxy resin polymer matrix composites. In comparison to pure epoxy resin and MWCNT-filled epoxy resin polymer matrix composite, the composite incorporating cross-plyed E-glass fiber epoxy resin polymer matrix composite has a higher glass transition temperature, loss modulus, storage modulus, and loss factor. This shows that the resin molecules are resisted from moving through the cross-plyed glass fibers.

Keywords: Composite, Carbon nanotubes, polymer, viscoelastic

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I. INTRODUCTION

In the past two decades, fiber-reinforced polymer matrix composites, or PMCs, have drawn a lot of interest because of their exceptional qualities, which include high specific strength and good corrosion resistance. However, a change in temperature may have an impact on the characteristics of PMCs. The mechanical, thermal, and structural properties of polymer composites in general are influenced by the reinforcement of carbon nanotubes (CNT) and graphene nanoplate (GnP) in epoxy polymer composites [1]. CNTs are the perfect nanofillers for traditional polymer composites because of their unparalleled strength, vast surface areas, and unique advantages derived from their one-dimensional physical structure. Incorporation of CNT into the polymer matrix significantly improves the carrying capacity of the resultant nanocom-

posite and its mechanical properties [2]; [3]. A combination of elastic and viscous responses to the stress applied to plastics deforms the degree of their strength, which degrades with time until a stable base is reached. Polymer matrices containing nano- or micro-sized fillers have different properties for unique applications. The polymer matrix is used in the form of pastes, which significantly improve their properties by mixing them with reactive or non-reactive additives [4]. The polymer matrix is available in a variety of ranges, and their ratio is identified by the manufacturers and cured at room temperature or at elevated temperatures, achieving favorable mechanical and thermal properties. The properties of fiber-reinforced polymer matrix laminate composites are influenced by the number of layers and fiber directions. The glass fiber-reinforced epoxy resin composite structure exhibits better

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mechanical properties in the fiber direction and is weaker in the transverse direction [5]. The hybrid composite is composed of more than two different materials embedded in a polymer matrix. The benefit of using hybrid composites is that they have the highest mechanical properties of single-phase composites [6]; [7]; [8]. One of the primary causes of environmental contamination is the usage of man-made synthetic fibers, which is reduced when two different materials hybridize [8];[9]. The most commonly used nanofillers are carbon nanotubes (CNT) and carbon nanofibers [10]. The strength, stiffness, aspect ratio, and specific surface area were all increased due to incorporation of CNT filler. [11]; [12]. CNT exhibits unique properties such as high specific strength and specific stiffness. The addition of CNT to composites has its own challenges. The prime challenge is their uniform dispersion [13]; [14] for efficient stress transfer from the weak matrix to the strong nanotubes to obtain superior mechanical properties [14]; [14]. The very high aspect ratio of CNTs is desirable for efficient load transfer but unfortunately the chances of agglomeration are also high due to high attractive forces among CNT molecules. Several methods are suggested in literature for uniform dispersion of CNT in polymer resins, such as sonication, stirring, and calendaring [15]. Dynamic mechanical thermal analysis (DMTA) is basically used to determine the glass transition temperature (T_g) of polymer based materials. When a polymer accumulates sufficient energy from thermal sources, it reaches the T_g , which is the most important physical transition temperature in polymers [13]. A sharp decline in the storage modulus linked to this modification determines the T_g which can also be determined by monitoring increases in the internal friction tangent (\tan) and loss modulus [16]. The DMTA has been used to characterize a variety of polymeric composites and is helpful for a number of polymeric systems, including thermosets and thermoplastics [17]. Glass transitions in fiber-reinforced plastic (FRP) composites indicate their thermal stability, and viscoelastic phenomena including creep and stress relaxation techniques assess the thermo-mechanical performance of these materials. [18]; [19]. Polymer matrix composites often carry less load under constant load vs displacement over comparatively lengthy time periods and are subject to increasing deformation under constant loads (creep). The axial direction of the fiber in fiber-reinforced polymer (FRP) composites is known as the fiber direction because it experiences the least amount of deformation from changes in temperature or time when compared to the transverse direction of the fiber. One of the simplest ways to manage load displacement in both static and dynamic tests of polymeric materials is through the use of

Dynamic Mechanical Thermal Analysis (DMTA). Generally, the viscoelastic behavior of polymer composites explains phenomena such as creep and stress relaxation, especially when the DMTA method is utilized, as numerous researchers have demonstrated. [20]; [1]; [21]. The primary reason of viscoelastic deformations in FRP composites is the rheology of the polymeric matrix. This property enables the molecular chains within the matrix to extend under thermo-mechanical loads by moving against each other, unfolding, and straightening [21]. Movements at the molecular scale contribute to the creeping, deformation, or relaxation of stress at the macroscopic scale. The incorporation of carbon fibers significantly improves the mechanical properties of polymers and boosts their resistance to viscoelastic deformation. To prevent slippage, stiff nanofillers are added to the polymer matrix of FRP composites [18];[17]. The percolation limit is crucial for nanofillers with strong van der Waals-like forces, including carbon nanotubes (CNTs). Agglomerated CNT reduces the efficiency of CNT as reinforcement since it is not only weakly coupled to the matrix but also causes stress concentration [20]. The dynamically charged polymer matrix material stores deformation energy elastically at high temperatures for thermal analysis, a portion of which is dissipated. The amount of stored energy is dissipated with the frequency of the applied load. The storage modulus (E') depends on the frequency, and the loss modulus (E'') is the elastic storage of energy in the imaginary part for the dissipation of dynamic energy. Furthermore, the loss factor is the ratio of the loss to the storage modulus. The storage modulus, loss modulus, and loss factor are used to represent the dynamic modulus materials [?]; [14].

The present investigation discusses and compares the influence of temperature on the viscoelastic properties of cross-plyed E-glass fiber-reinforced epoxy matrix composites, MWCNT-filled epoxy resin, and unfilled epoxy resin.

II. METHODS

A. Preparation of MWCNT Filled and Unfilled Epoxy Resin Specimen

The resin (LY-556) and hardener (HY-917) were used in a 10:1 ratio and sourced from Ciba Geigy Ltd., Mumbai, India. The MWCNT particles, with a purity of 99.5 percent, were obtained from J.K. Impex Company, Mumbai. Both pure epoxy resin (unfilled) and MWCNT-filled epoxy resin plates were prepared manually. Initially, the 2% MWCNT-filled epoxy resins and unfilled epoxy resin were mixed using a mechanical stirrer. Once the MWCNTs were uniformly dispersed in the resin, the hardener

was added to the mixture of MWCNT-filled and unfilled epoxy resin. This mixture was stirred for a few seconds before being poured into a mold. The mixture was then left to cure for 24 hours at room temperature.

B. Preparation of Glass-Fiber-Composite Specimen

The most fundamental method for processing composites is the hand lay-up technique. To prevent the polymer from adhering to the mold surface, a release agent is first applied. Thin plastic sheets are then placed at the top and bottom of the mold to ensure a smooth surface for the final product. After the plastic sheet is positioned on the mold surface, the E-glass fiber woven mat is trimmed to fit the mold dimensions. The thermosetting polymer (resin LY-556, hardener HY-917) is thoroughly mixed in a 10:1 ratio with the hardener and poured onto the surface of the mat in the mold. A brush is used to evenly distribute

the polymer. The second layer of mat is then applied on top of the polymer surface, and a roller is gently used to remove any excess polymer and trapped air. This process is repeated for each layer of mat and polymer until the required number of layers is achieved. Once the layers are stacked, a plastic sheet is placed on top, pressure is applied, and release gel is sprayed on the inner surface of the top mold plate, which is then positioned on the stack. The composite part, with a fiber volume fraction of 42%, is removed from the mold and further processed after curing for 24 hours at room temperature.

The fabricated glass fiber-reinforced plastic (GFRP) composite, pure (unfilled) epoxy resin, and MWCNT-filled epoxy resin plates were prepared with the specified dimensions for DMTA test samples; thickness = 2 mm, width = 10 mm, and length = 40 mm. The properties of materials are listed in Table 1.

TABLE 1
PROPERTIES OF USED MATERIALS

Material	Geometry	Elastic Modulus(GPa)	Density (g/cm ³)
MWCNT, 99.98%	Length= 2 μ m Average inner diameter= 6.5 nm Average outer diameter= 40 nm	1000	1.7
Epoxy Resin	—	2	1.2
E-Glass Fiber	Single fiber diameter = 10 μ m	89	2.53

C. Dynamic Mechanical Thermal Analysis

A rheometer (Anton Paar Co., Ltd., Germany) was used to determine the storage modulus, loss modulus, and loss factor of cross-plyed E-glass fiber-reinforced epoxy resin polymer matrix composites, MWCNT-filled epoxy resin polymer matrix composites, and pure epoxy resin with variation of temperature. All samples were analyzed at elevated temperatures. range of 30-180 OC with 2°C/min heating rate and 1 Hz constant frequency. From the generated data, the peak loss modulus (E'') can be considered as Tg for the composites. The proportional contribution of the viscous elastic characteristics is represented by the loss factor term, which is defined as the ratio of the loss modulus (E'') to the storage modulus (E'). There isn't a single glass transition for these materials; rather, Tg is better understood as a range of temperatures when the material experiences abrupt changes in its thermomechanical characteristics. Tg can be calculated

using the temperature curve of the storage modulus (E'), the peak temperature of the loss modulus (E''), and the peak temperature of the loss factor after scanning the test sample [21].

III. RESULTS AND DISCUSSION

One technique that is frequently used for material characterization is dynamic mechanical analysis (DMA). It measures the material's reaction to an oscillating force after it is applied. It assesses the stiffness, which is related to the sample's viscosity and elasticity, as well as the inclination to flow, in contrast to standard tensile testing. The storage modulus, loss modulus, and loss factor of the pure epoxy resin, cross-plyed E-glass fiber reinforced epoxy resin polymer composite, and MWCNT-filled epoxy resin samples were carefully measured with temperature variation, and the results are plotted in graphical form in Figs. 1, 2, and 3.

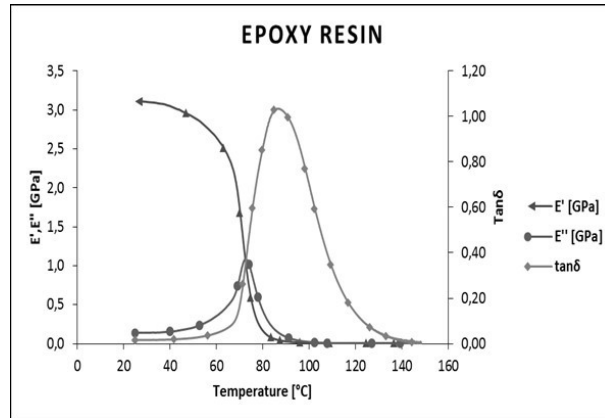


Fig. 1. Flow diagram for the roll forming process design correction

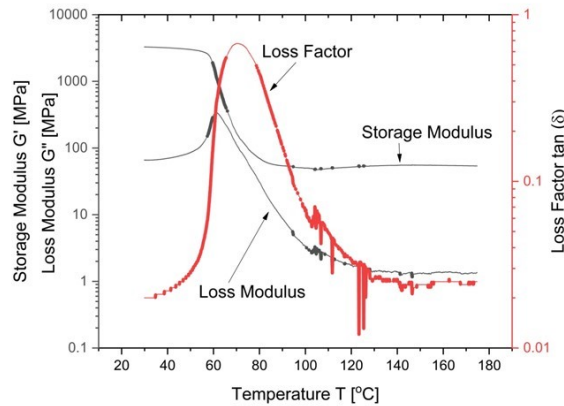


Fig. 2. Variation of storage modulus, loss modulus and loss factor of E-glass fiber reinforced epoxy resin composite with temperature.

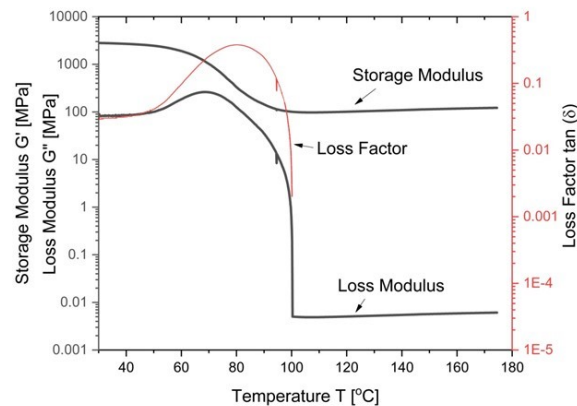


Fig. 3. Variation of Storage Modulus, Loss Modulus and Loss Factor with the increase of Temperature of MWCNT-filled Epoxy Resin polymer Composites.

In general, storage modulus (E' or G') and loss modulus (E'' or G'') are used to distinguish material phases of

variation. The viscoelastic behavior of pure epoxy resin indicates that the storage modulus (E') decreases rapidly

as the temperature rises to 32° , and the loss modulus (E''), and loss factor increase abruptly as the temperature increases from 50°C to 90° , and then decreases with the increase in temperature, as shown in Fig.1. The storage moduli of the cross-plyed E-glass fiber reinforced epoxy resin composite, pure epoxy resin, and MWCNT-filled epoxy resin decrease rapidly with temperature increase after 25°C and then stabilize after 60°C with further temperature increases up to 90° , as shown in Figures 1, 4, and 5. However, the loss modulus and loss factor of pure epoxy resin, cross-plyed E-glass fiber-reinforced epoxy resin, and MWCNT-filled epoxy resin specimens increase with the increase of temperature from 70° to 85° and then decrease with the increase of temperature. This peak temperature indicates the transition temperature of the

pure epoxy resin, MWCNT-filled epoxy resin, and cross-plyed E-glass fiber-reinforced epoxy resin polymer matrix composite. The values of loss modulus and loss factor of GFRP composite are higher than those of pure epoxy resin and MWCNT-filled epoxy resin due to the interference of the epoxy resin molecules' movements with the axial and transverse directions of the glass fibers. The storage modulus of GFRP composite is nearly the same as that of pure epoxy resin. Figures 4 and 5 show the gradual increase and decrease in loss modulus, and loss factor with the increased temperature up to 60° to 90° of multiwalled carbon nanotube-filled epoxy resin and cross-plyed E-glass fiber-reinforced epoxy resin polymer matrix composites.

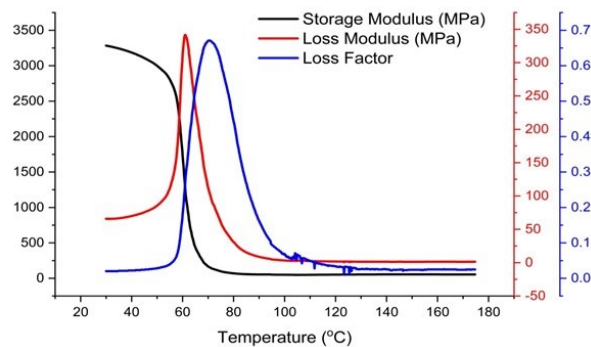


Fig. 4. Variation of Storage Modulus, Loss Modulus and Loss Factor with the increase of Temperature of MWCNT-filled Epoxy Resin polymer Composites.

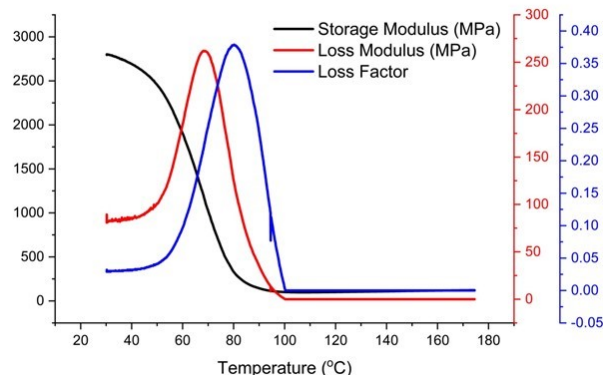


Fig. 5. Variation of Storage Modulus, Loss Modulus and Loss Factor with the increase of Temperature of MWCNT-filled epoxy resin polymer composites.

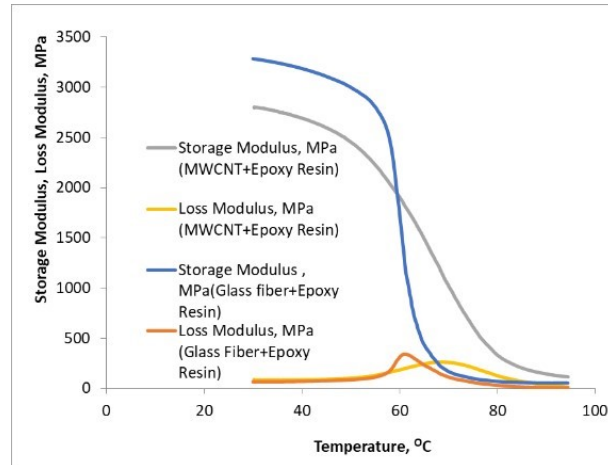


Fig. 6. Comparison of Storage Modulus, Loss Modulus of cross-plyed E-glass Fiber-reinforced epoxy resin polymer composite and MWCNT-filled epoxy resin polymer composites with the temperature.

Fig. 6 clearly indicates that at 55°, storage modulus, loss modulus, and loss factor decrease very rapidly with an increase in temperature. These values are lower than those of pure epoxy resin and cross-plyed E-glass fiber-reinforced epoxy resin composites because of the uniform dispersion of MWCNT particles in the epoxy resin polymer matrix. Figures 1 and 6 illustrate how pure epoxy resin, cross-plyed E-glass fiber-reinforced epoxy resin composites, and MWCNT-filled epoxy resin differ in regard to viscoelastic characteristics (storage modulus, loss modulus, and loss factor). The major differences between the loss modulus temperatures of, pure epoxy resin, cross-plyed E-glass fiber reinforced epoxy resin

(62°), and MWCNT-filled epoxy resin (75°), show the variation in the transition temperature phases of all the materials above. However, because the mobility of epoxy resin molecules is restricted along fiber directions, the storage modulus of cross-plyed E-glass fiber-reinforced epoxy resin is higher than that of pure epoxy resin and MWCNT-filled epoxy resin composites.

IV. DESCRIPTION

The micrograph clearly shows that the MWCNT fillers were uniformly distributed in the epoxy resin as shown in Fig. 7(a) which maximizes the toughness of materials compared to epoxy resin composites.

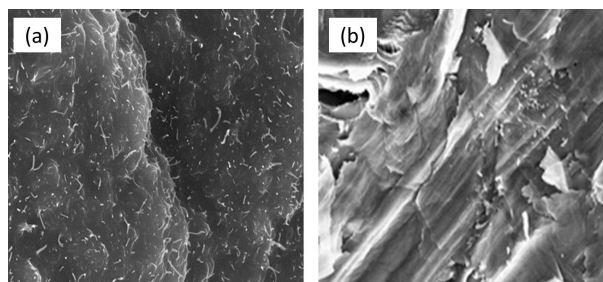


Fig. 7. SEM micrograph clearly indicates that (a) the uniform dispersion of MWCNT -filled epoxy resin and (b). debond of cross-plyed E-glass fiber from matrix in the polymer matrix.

The crack deflection occurs in the vicinity of MWCNT fillers which requires more energy for propagation of crack which means more energy can be stored elastically in the material. Thus storage modulus of MWCNTs filled epoxy was higher as compared to epoxy resin. However storage modulus of glass fiber-reinforced epoxy resin composites was higher as compared to MWCNTs filled epoxy resin composites. This may be mainly attributed to the continuity of glass fibers which is missing in MWCNTs. In case of glass fibers reinforced poly-

mer composites, firstly, the polymer matrix fractured in a river pattern, and then the crack propagated through the direction of the glass fiber that debonded from the matrix, as shown in Fig. 7(b). The viscoelastic behavior of pure epoxy resin, E-glass fiber-reinforced epoxy polymer composites, and MWCNT-filled epoxy polymer composites is obtained in terms of loss modulus, storage modulus, and loss factor, as shown in figures 1, 2, and 3. The findings show that the presence of MWCNTs and cross-plyed E-glass fiber on the epoxy polymer ma-

trix's surface enhanced the material's energy dissipation mechanisms, which in turn affected the loss factor of pure epoxy resin, the composite of cross-plyed E-glass fiber epoxy resin polymer matrix, and MWCNTs on the epoxy resin polymer matrix. The increase in storage modulus, loss modulus, and loss factor is attributed to the presence of cross-plyed E-glass fiber, which hinders the movement of polymer matrix molecules along the fiber directions, thereby increasing friction between the polymer matrix and the glass fiber. Conversely, the decrease in storage modulus, loss modulus, and loss factor in the MWCNT-filled polymer matrix suggests that the uniformly dispersed CNT particles do not influence the movements of polymer matrix molecules. Figure 7(b) shows the glass fiber debonding from the matrix. The loss tangent exhibits nonlinear frequency-dependent behavior for both composite samples. The mechanisms of energy dissipation under viscoelastic deformation at the glass fiber/polymer matrix interfaces are activated by relatively small applied strain levels (0.05%). The material depreciation behavior is represented by the loss factor, or $\tan \delta$, which is calculated by dividing the loss modulus by the storage modulus. A larger area under the $\tan \delta$ peak indicates higher energy dissipation. The area under the $\tan \delta$ peak is smaller in the epoxy matrix containing uniformly dispersed MWCNTs compared to pure epoxy resin, cross-plyed E-glass fiber reinforced epoxy resin polymer matrix composite, and MWCNT-filled epoxy resin composite, as shown in Figures 1–6. This is explained by the interfa-

cial interaction between the surrounding polymer matrix and MWCNT particles. As a result, the damping peak decreases at MWCNT-matrix interfaces due to a decrease in molecular chain mobility. There is a noticeable rise in the loss tangent of glass fiber composite [6]. This indicates that further growth promotes the increase of the loss factor. Fig. 7 shows the uniform distribution of CNT particles throughout the polymer matrix. At higher magnification (20X), the SEM micrograph (Fig. 7-a.b.) shows the homogeneous dispersion of MWCNTs and the debonding of glass fiber in the epoxy polymer matrix. The matrix used in this study is a slow-curing matrix with a relatively low viscosity [14]. This allows for gravity segregation of the MWCNTs, resulting in the deposition of CNT on the polymer matrix surface and the formation of small clusters within the matrix system as seen in Fig. 7(a). The formation of clusters leads to non-uniform stress transfer bonding at the MWCNTs-matrix interface. However, polymeric matrix molecules move easily from corner to corner as a result of an imperfect bond between each nanoparticle [12]. This allows easy movement of polymer matrix molecules and changes the higher phase transition temperature in relation to glass fiber polymer matrix composites. Fig. 8(a) shows that the initial stress wave propagated easily before the interface of the glass fiber, and then the stress wave jumped over the glass fiber, with energy absorbed by the polymer matrix at the applied load and temperature.

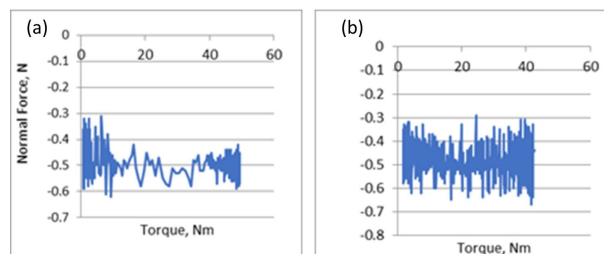


Fig. 8. Variation of normal force with torque of (a) cross-plyed E-glass-fiber-reinforced epoxy resin polymer matrix and (b) MWCNT-filled epoxy resin polymer matrix.

Figure 8 (b) indicates that the stress wave easily propagated in the MWCNT-filled polymer matrix at the applied load and temperature due to the uniform dispersion of nanoparticles, as can also be identified by Figure 7 (a). The primary cause of the damping observed in MWCNT-epoxy nanocomposites is the interfacial sliding that occurs between the MWCNT and the surrounding epoxy matrix. Interfacial slippage between MWCNT and epoxy matrix is extremely low when there is strong interfacial adhesion between them.

The aggregation of MWCNT caused by an increase in MWCNT content in the epoxy causes an increase in interfacial slippage [18]. Therefore, the percentage of filler, critical shear stress, structural features, and crosslinking of nanofillers with the polymer matrix are the elements influencing the damping characteristics of polymer nanocomposites. The large aspect ratio and surface area of CNTs significantly improve the damping properties of polymer nanocomposites [19]. Better dispersion of CNTs in polymers is facilitated by the lower viscosity at higher

temperatures. According to reports, the mechanical properties of polymer matrices are enhanced by the addition of CNTs up to a specific proportion of loading [18]; [17]. At a higher load of CNT-filled polymer matrix, there is a premature failure of nanocomposites that is due to the non-uniform distribution of leads around the agglomeration of CNTs and the creation of voids. The important components of a high storage module with a low loss factor are the uniform dispersion and good interaction of CNT with the epoxy matrix. [19].

V. CONCLUSION

The damping properties of the pure epoxy resin, cross-ply E-glass fiber reinforced epoxy resin, and MWCNT-filled epoxy resin composite was assessed at increasing temperatures and frequencies. The storage modulus, loss modulus, and loss factor curves that resulted from the extra time required to mobilize the epoxy resin polymer molecules can be used to calculate the glass transition temperature of pure epoxy resin, cross-ply E-glass fiber reinforced epoxy resin, and MWCNT-filled epoxy resin polymer matrix. The results also indicate that the storage modulus, loss modulus, and loss factor of the pure epoxy resin, cross-ply E-glass fiber epoxy resin polymer matrix, and MWCNT-filled epoxy resin polymer matrix composites are severely affected by the variation in temperature. Because the movements of the epoxy resin polymer matrix molecules are significantly affected by the cross-ply E-glass fiber directions compared to the MWCNT-filled epoxy resin polymer matrix and a pure epoxy resin matrix. As a result, the storage modulus, loss modulus, and loss factor values of the pure epoxy resin and cross-ply E-glass fiber reinforced epoxy resin polymer matrix composite is higher than those of the MWCNT-filled epoxy resin polymer matrix composite and the pure epoxy resin matrix. Hence, the phase transition temperature of the cross-ply E-glass fiber polymer matrix (62°) is less than that of the MWCNT-filled epoxy resin composite (72°) and pure epoxy resin.

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