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# Glass Fiber Reinforced MWCNTs-filled Epoxy Nanocomposites: Morphological and Viscoelastic Characteristics

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## ABSTRACT

This study aims to investigate glass fiber-reinforced multi-walled carbon nanotubes (MWCNTs)-filled epoxy nanocomposites (A, B, and C), with different MWCNTs reinforcement using Dynamic Mechanical Analysis (DMA). Epoxy-E-glass fiber strengthened composites were fabricated with MWCNTs of Ø20-30 nm and <Ø8 nm using hand layup approach. Specimen A (neat epoxy), specimen B (MWCNTs <Ø8 nm) and specimen C (MWCNTs Ø20-30 nm) were fabricated. From DMA, it was found that specimen C has better molecular stability and higher cross-linking density, as seen from its lower tan δ peak (0.301) compared to specimens A (0.455) and B (0.419), even though its glass transition temperature (88.13 °C) is marginally below that of specimen A (92.97 °C). At the glass transition temperature of specimen B, the lowest tan δ (0.265) and loss modulus (398.4 MPa) are observed for specimen C, indicating reduced energy dissipation and heat generation, and thereby improved thermal resistance with lower susceptibility to thermal damage. To assess thermal damage, SEM images at the drilled holes in the inner walls were analyzed, and the observations were found to be consistent with the DMA results, further confirming the thermo-mechanical performance of the specimens.

**Keywords:** Nanocomposites; MWCNTs; Dynamic mechanical analysis; Epoxy-E-glass.

## 1. INTRODUCTION

Compared to traditional monolithic materials, composites, especially polymer matrix composites (PMCs) and metal matrix composites (MMCs), provide a better balance of low density and excellent mechanical performance, making them an attractive choice for lightweight applications. In industries including aerospace, automotive, marine, and defense, where structural weight reduction is a primary concern, these materials are essential because of the need to maintain strength, stiffness, durability, and functional reliability. Polymer matrix composites are a great choice for a material with excellent specific strength, little weight, resistance to corrosion, and design flexibility. Superior strength, adaptability, low density, and long-term durability are just a few of the many benefits of polymer composites. Matrix selection suggests a relationship between important mechanical properties like impact toughness, heat deterioration, and interlaminar shear strength (ILSS) (Painkal *et al.* 2025).

One major goal of adding nanoparticles to composites is to boost performance with minimal additional reinforcement by making the material more efficient. The small size and extremely high specific surface area of nanoparticles allow them to make extensive contact with the matrix at the interface, which improves hardness, stiffness, and strength via load transmission and dislocation motion restriction (Seenivasan *et al.* 2025). Improved wear resistance, fatigue life, and fracture performance are outcomes of their microstructural refinement, reduction of localized plastic deformation, and inhibition of crack initiation and propagation. In addition, nanoparticles can enhance thermal stability, durability against corrosion, and, in certain systems, functional properties including electrical and thermal conductivity (Senthilkumar, 2024). By altering heat transport, thermal stability, and the matrix reaction to temperature, nanoparticle incorporation substantially affects the thermal behaviour of composites. Thanks to their large surface area, strong interfacial interaction with the matrix, and other desirable properties, nanoparticles can increase thermal stability and resistance to heat degradation by limiting the mobility of polymer chains or preventing local matrix deformation. In many systems, they also enhance thermal conductivity by creating more effective heat-conduction pathways, which promotes faster heat dissipation and improves dimensional stability under thermal loading. In addition, nanoparticles can reduce the coefficient of thermal expansion, refine the microstructure, and onset

delay of softening or decomposition, particularly when they are uniformly dispersed and well bonded with the matrix (Prashanth and Keerthi, 2025).

Influence of matrices on such mechanical properties was reported by Jiang *et al.* (2023). The challenge emerges when these polymer composites are subjected to various machining processes for assembly as these secondary manufacturing processes inevitably enforce thermal damage to the matrices along with fiber pull out. Juon *et al.* (2021) published a review on thermal damage studies in CFRP composites and reported recommendations for future studies of CFRP composites. Drilling is a crucial step in facilitating part assembly across various industries. However, drilling polymer composites presents notable challenges. Jagadeesh *et al.* (2023) reported that dimensional inaccuracies and poor surface finishes are caused during drilling due to bucking, fuzzing, and fiber burning. Polymer matrices are tailored to meet processing and application requirements. Dorigato and Fredi, (2024) remarked on the current fascination in multifunctional materials that can have their performances adjusted to suit specific needs. The behaviour of crystallization may be significantly altered by adding nanofiller to polymer blends. The compatibilization activity of nanofillers in polymer alloys may be significantly impacted by their intrinsic qualities (such as form, size dispersion, aspect ratio, etc.) as well as their synthesis conditions. The shape of nanofillers (NFs) affects the characteristics of nanocomposites. The peak interphase density increases by 6% and 17% as the NF form shifts from nanosphere to nanocylinder and nanofilm, respectively (Wang *et al.* 2024).

Despite the obtainable tailoring potentials of the polymer nanocomposites, the agglomeration is a challenge in ensuring uniform mechanical properties upon processing. The effect of agglomeration in affecting crack propagation mechanism has been reported (Abhiram and Ghosh, 2023). Dynamic Mechanical Analysis (DMA) is commonly employed in rheology to understand phase transitions in polymer materials (Rani *et al.* 2024). Anidha *et al.* (2024) witnessed improved storage modulus (2468 MPa) and glass transition temperature (376.86°C) in 10 wt.% natural rubber reinforced composite indicating better cross-linking density. The authors also conducted X-ray diffraction analysis for better understanding of crystalline phases. According to a recent study investigating the influence of carbon nanopowder on the thermal and wear properties of chopped strand mat E-glass fiber-reinforced epoxy composites, the incorporation of carbon nanopowder significantly affected the wear behavior of the composites (Vaddar *et al.* 2023).

Goswami *et al.* (2015) found that, across a frequency range, the rheology of MWCNT-containing composites showed a moderate decrease in storage modulus. Kumar *et al.* (2017) found that the storage modulus decreased in their DMA investigation after loading the epoxy/MWCNT nanocomposite with 0.75 wt.%. It was hypothesized that this reduction is due to MWCNTs' limited de-agglomeration capabilities. They also found reduction in tan δ with MWCNT addition thereby an augmentation in epoxy stiffness from robust interfacial bonding between well spreader MWCNTs and the epoxy matrix. Rayjadhav *et al.* (2026) conducted DMA studies on polyamide 6(PA6) and polypropylene (PP) nanocomposites and investigated the influence of MWCNT along with graphene. The authors reported that the fabricated test specimens with various graphene-MWCNT ratios shows the highest storage modulus, along with a shift of the transition temperature. By varying the sonication timing and amplitude, a thorough study on thermo-mechanical and toughness property was conducted (Gkikas *et al.* 2012), and an enhancement in the storage modulus was reported with increase in the sonication timing. Her and Lin, (2017) demonstrated that when MWCNTs are included, the storage modulus and glass transition temperature were increased. Improvement in thermal stability was observed (Song *et al.* 2013) in the presence of MWCNTs in carbon fiber (CF)/ epoxy composite (EP) along with lower peak height than CF/EP composite. Therefore, in this study, MWCNT-filled glass fiber reinforced epoxy composites were first fabricated and evaluated using DMA and microstructural examination using SEM micrographs to better understand the resulting material characteristics.

## 2. MATERIAL FABRICATION

Composite materials using epoxy Araldite LY556 and E-glass fiber fabric were fabricated with two types of MWCNTs fillings, each with unlike diameters: 20-30 nm and <8 nm (Ponnuvel and Prem, 2018). Additionally, a reference composite material without any MWCNTs filling was also fabricated. The resulting specimens were designated as specimen A (neat epoxy), specimen B (filled with MWCNTs of Ø <8 nm), and specimen C (filled with MWCNTs of Ø20-30 nm). Ultrasonic bath sonication was utilized as dispersion method for dispersing multi-walled carbon nanotubes (MWCNTs) within polymer matrices. To reduce agglomerations, MWCNTs with 0.3 wt.% were sonicated via an ultrasonic vibrator in a solvent (ethanol) (Chaichan *et al.* 2023; Titarmare *et al.* 2025), and subsequently mixed with the epoxy resin and again sonicated for one more hour. The mixture was stirred at 80°C to remove the solvent. The five-layered composite was made using E-glass coarse cloth, each layer was coated with epoxy Araldite LY556 containing 0.3 wt.% of MWCNTs and hardener HY951 (10:1 ratio). Fig. 1 presents the SEM micrographs of considered MWCNTs. Fig. 2 presents the SEM micrographs of neat epoxy and MWCNTs

mixed with epoxy using ultrasonic agitator and mechanical stirring. Fig. 3 presents the fabrication procedure to develop the composites.

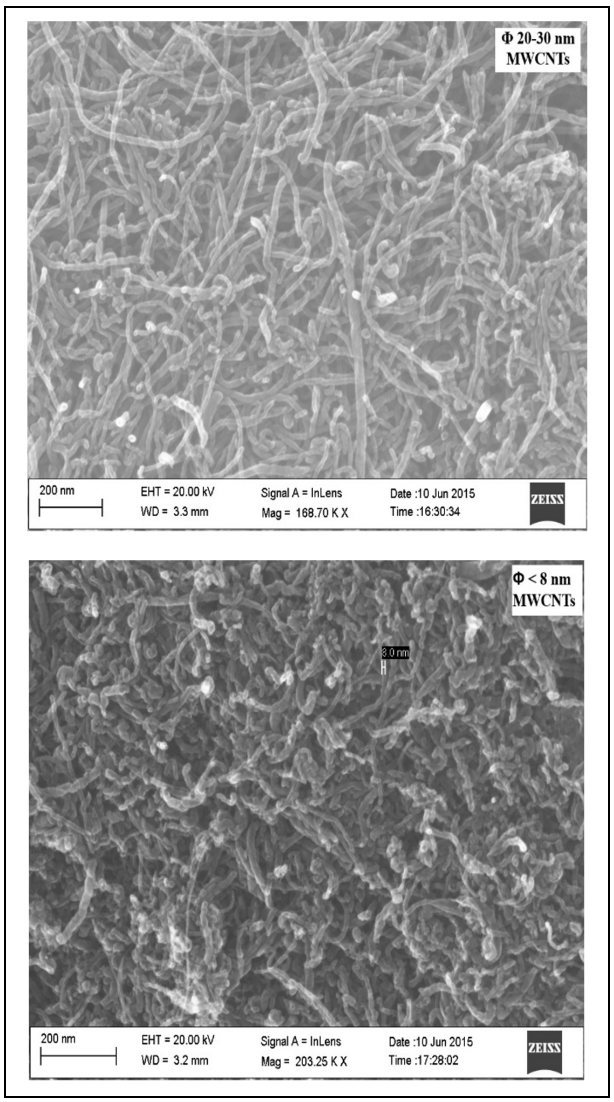



**Fig. 1: SEM micrographs of MWCNTs (a) Ø20-30 nm (b) Ø<8 nm**

## 2.1 Experimental Methodology

Dynamic mechanical analysis was conducted using a DMA Q800 V20.6 Build 24 analyzer to study the heat dissipation behaviour of the fabricated specimens under varying temperature and loading conditions. In this method, a small oscillating force is applied, and the resulting deformation is measured. The test provides storage modulus (E′), representing stiffness, and loss modulus (E″), indicating heat dissipation. The tan δ (loss factor) reflects the damping behaviour of the material. DMA is also used to determine the glass transition temperature (Tg) and to evaluate cross-linking and filler–matrix interactions in composites. (Kehrer *et al.* 2018).

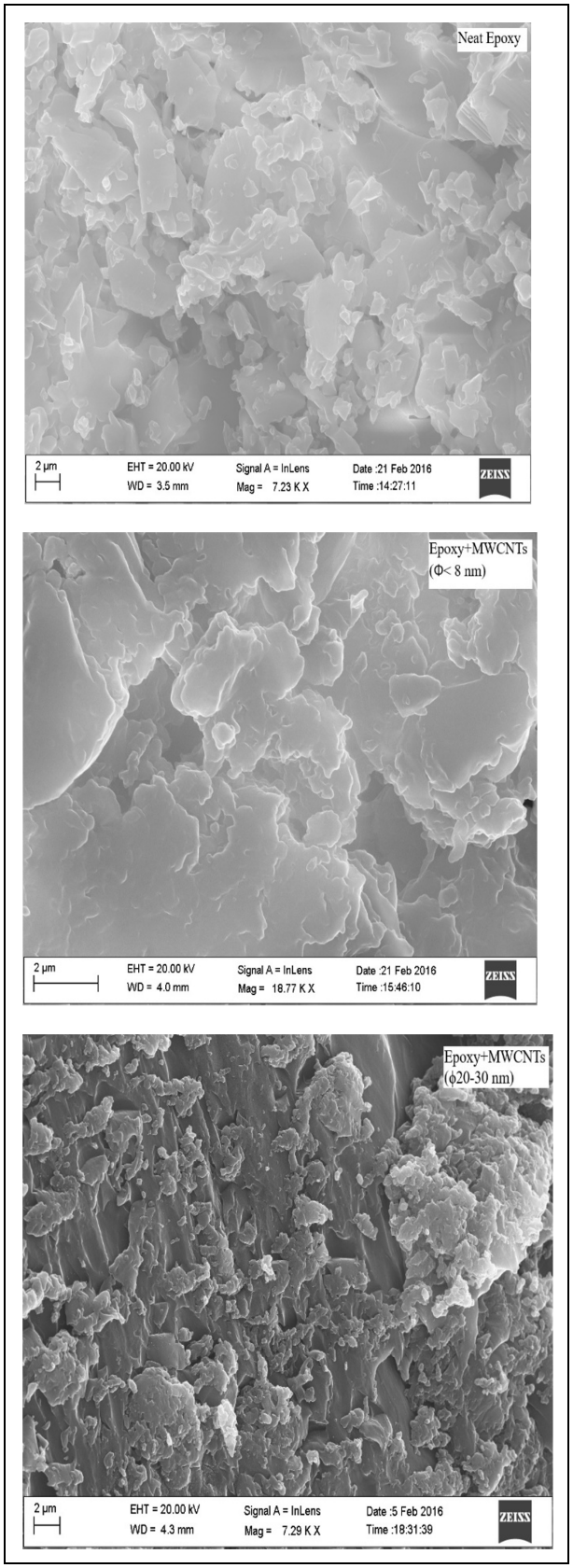



**Fig. 2: SEM images of (a) Neat epoxy (b) Epoxy + MWCNTs (<8 nm) and (c) Epoxy+ MWCNTs (20-30 nm) composites**

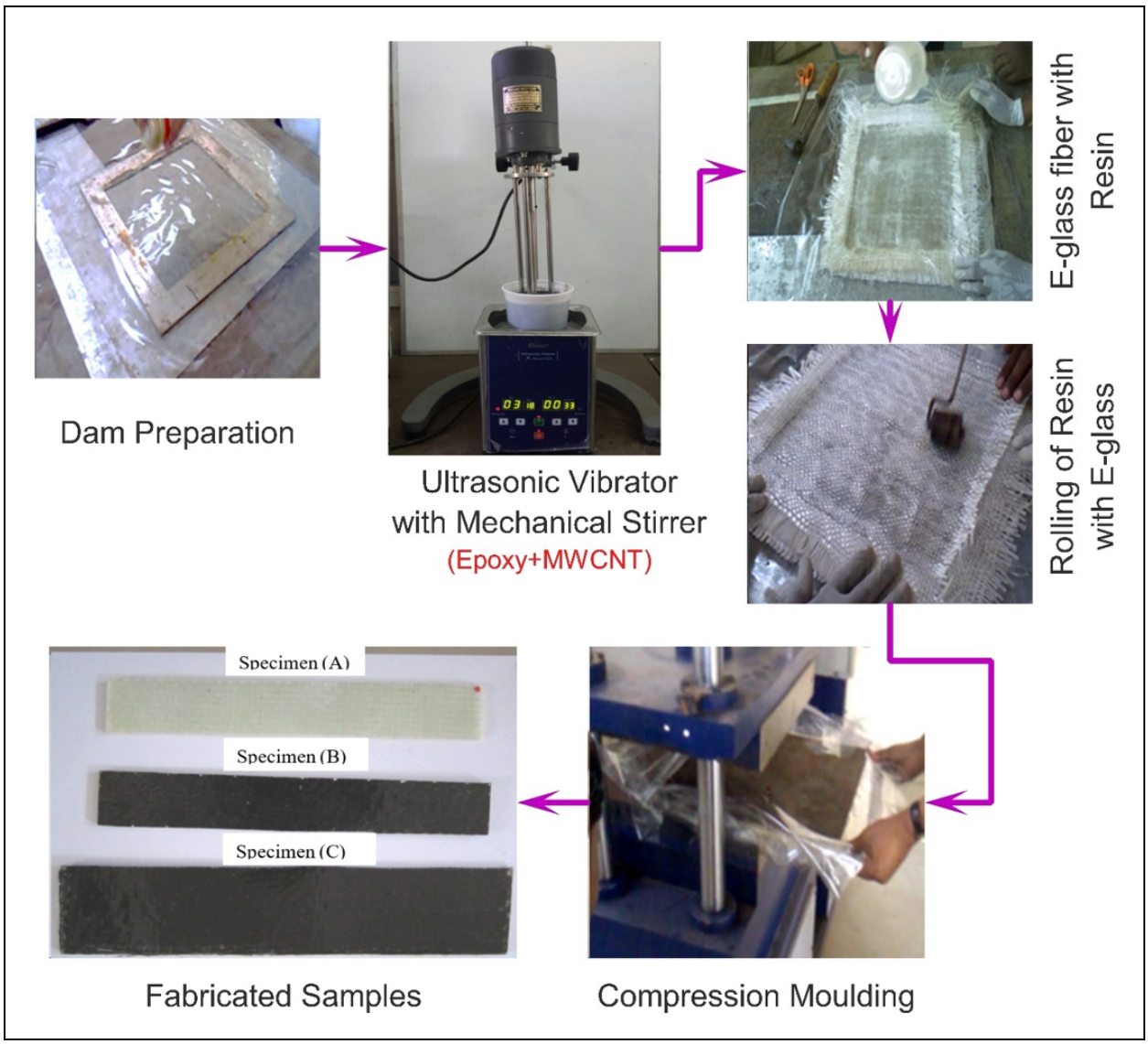
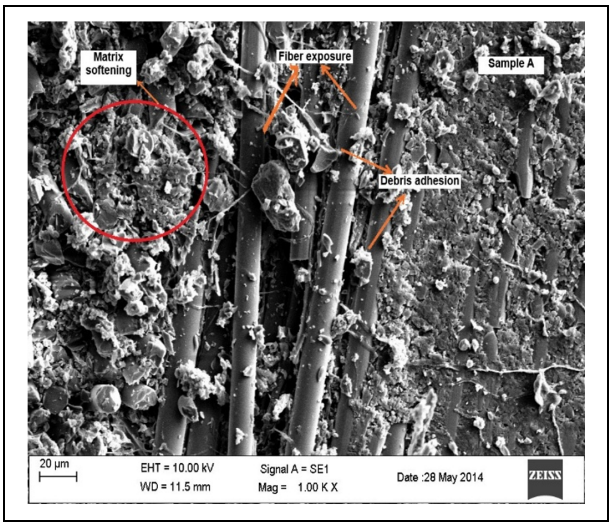



**Fig. 3: Fabrication procedure**

## 3. RESULTS AND DISCUSSION

### 3.1 Characterization of Fabricated Composites

The images of inner wall of the drilled holes of sample A, B and C are shown in Fig. 4. Noticeable fiber pull outs were observed in all samples indicating interlaminar debonding during the drilling process. Sample A shows more severe matrix softening, fiber exposure with scattered debris adhesion. Sample B shows better matrix adherence with reasonable fiber pullouts, while sample C shows smooth surface with minimal debris in comparison with sample A and sample B. The above observations suggest that detailed drilling studies and dynamic mechanical analysis of the samples will help better explain the performance and damage behaviour of the specimens.



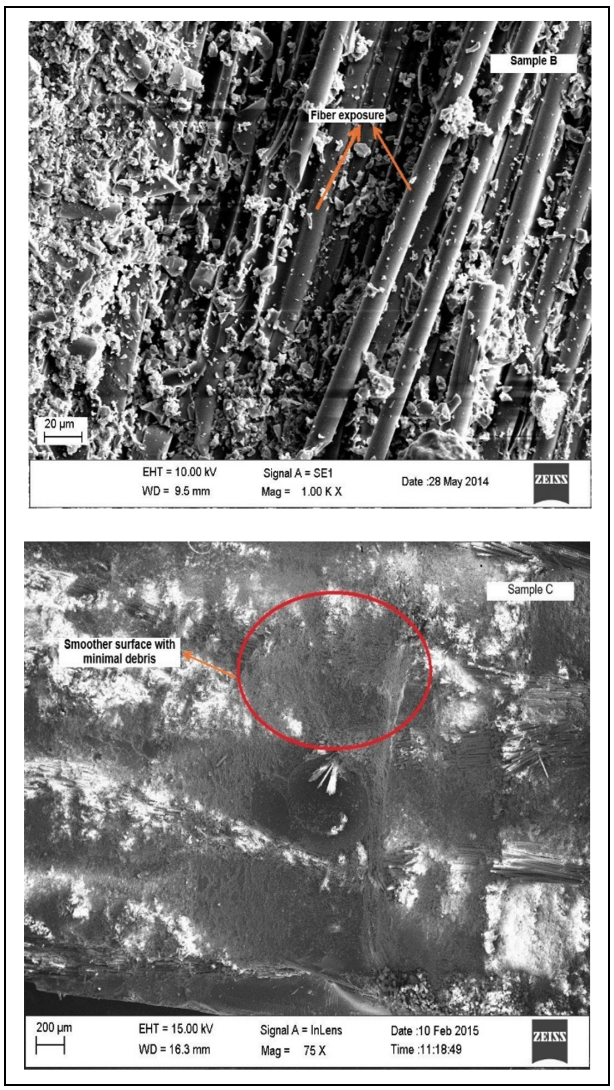



**Fig. 4: Drilled hole wall surface (a) SEM image of sample A (b) SEM image of sample B and (c) SEM image of sample C**

## 3.2 Dynamic Mechanical Analysis

From DMA results, tan delta (δ) peaks of 0.455, 0.419, and 0.301 were observed for specimen A, B, and C, respectively, as revealed in Fig. 5, 6 and 7. The tan δ peak decreases as the matrix cross-linking density increases (Mittal, 2005). From the obtained tan delta values of the specimens, it can be inferred that the cross-linking density of specimen C is better than specimens A and B. Also, it is obvious that the existence of MWCNTs restricts the molecular mobility of the epoxy matrix. The tan δ values of all the specimens at the glass transition temperature of specimen B were examined to understand the energy dissipation ability of the specimens in the form of heat. At the glass transition temperature of specimen B, tan δ values of specimen A, B, and C are 0.454, 0.419, and 0.265, respectively. Their corresponding loss moduli are 1211 MPa, 688.9 MPa, and 398.4 MPa, respectively. Specimen C, reinforced with larger diameter MWCNTs (20–30 nm), exhibits a lower tan δ compared to specimen B with smaller diameter MWCNTs (<8 nm), indicating reduced damping and energy dissipation, and thereby improved stiffness and interfacial bonding. The higher loss factor observed in specimen B can be related to the higher specific surface area of the smaller MWCNTs, which promotes stronger van der Waals interactions, increased agglomeration, and consequently greater energy dissipation in comparison with specimen C. Challenges of agglomeration due to high surface energy and predominance of van der Waals forces in nano fillers were reported earlier (Mohammadi *et al.* 2025). Since loss modulus is a measure of release of thermal energy, it is evident that specimen C dissipates less heat energy than specimens A and B. This suggests that the thermal damage due to the generation of heat during the drilling process is less in specimen C than in specimens A and B.

The DMA results show that the thermo-mechanical responses of all the specimens are different. With a glass transition temperature of approximately 92.97 °C, Specimen A indicates a rigid polymer network. On the other hand, a tan δ peak of around 0.455 indicates higher molecular mobility and energy transfer, which in turn suggests weaker bonding between surfaces and less productive cross-linking. In a reported work, the interfacial capacity of glass fiber filled polyester showed a negative correlation with tan δ at the glass transition temperature (Chua, 1988). Sample B shows balanced behaviour with better interfacial interaction than Sample A, with intermediate values of Tg (around 92 °C) and tan δ (about 0.419). Although there is a small decrease in Tg (around 88.13 °C), specimen C shows the lowest tan δ peak (about 0.301), which suggests less chain motion, greater filler-matrix interfacial attachment, and improved effective cross-link density. Specimen C had the best thermo-mechanical performance, while specimens A through C revealed an increasing reduction in tan δ, indicating an improvement in interphase stiffness and stress transfer efficiency.

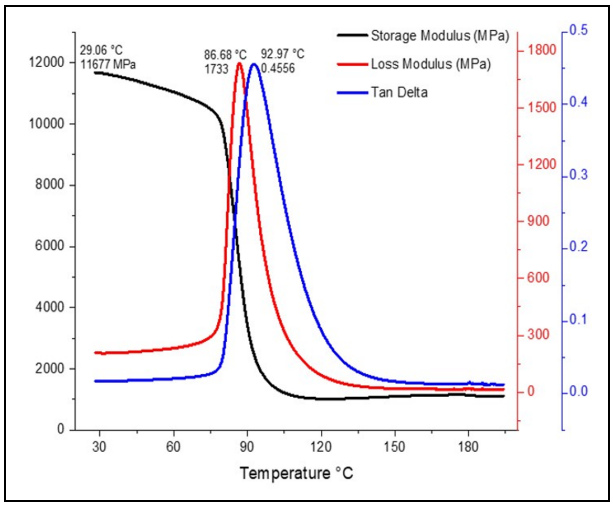



**Fig. 5: DMA results of sample A**

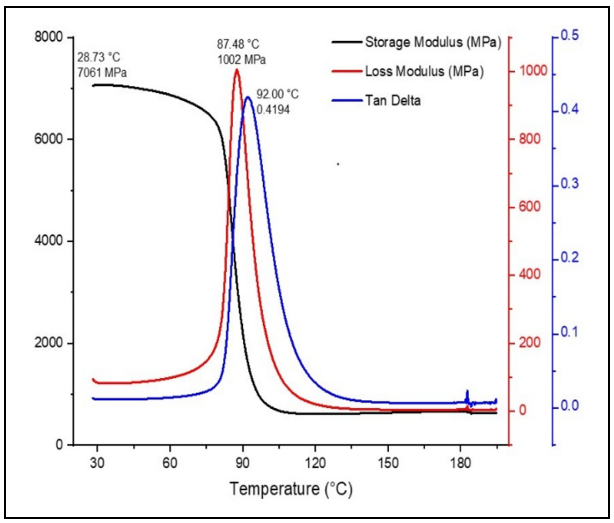



**Fig. 6: DMA results of sample B**

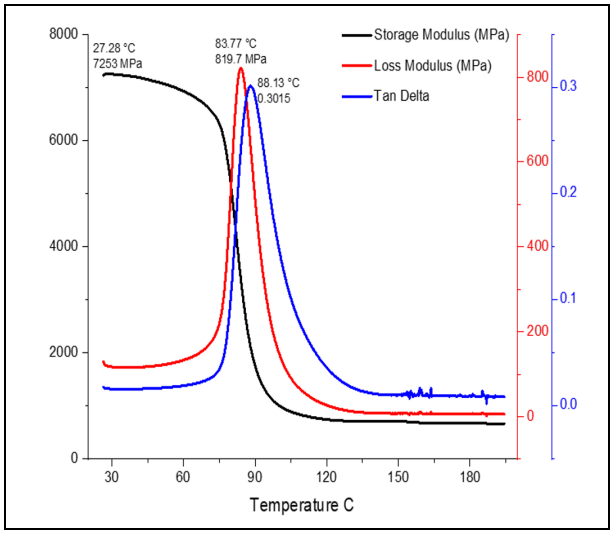



**Fig. 7: DMA results of sample C**

Specimen A exhibits a storage modulus of 2666 MPa and a tan δ of 0.454 at 92 °C, suggesting increased damping and high stiffness. With a tan δ of 0.419 and a storage modulus of 1643 MPa, specimen B demonstrates considerable stiffness and minimal energy loss. In comparison, specimen C exhibits the lowest storage modulus of 1498 MPa along with the lowest tan δ of 0.265, indicating lower stiffness but minimal energy dissipation and damping. The reduction in the storage modulus in specimens B and C is also attributed to the possible agglomeration of MWCNTs and to the presence of traces of ethanol used in the fabrication process. The reduction in tan δ indicates a shift from a more damping-dominated behaviour in specimen A to a more elastic and stable response in specimen C, likely due to better interfacial bonding and restricted molecular motion. Strong filler-matrix interactions restrict local chain motion and cause a noticeable reduction in tan δ, reflecting improved interfacial bonding and effective cross-linking the nano-fillers also disturb the uniform packing of polymer chains and generate small regions of free volume (Senthilkumar *et al.* 2024). This is shown in Fig. 8 for samples B and C. This interphase effect allows segmental motion to begin at slightly lower temperatures, leading to a modest drop in Tg even as load transfer improve. Wang *et al*. (2018) had also reported similar kind of observations in their study. The simultaneous decrease in tan δ and slight shift in Tg indicates the development of a more rigid yet structurally modified polymer network, typical of well-dispersed nanocomposites with enhanced thermo-mechanical performance.

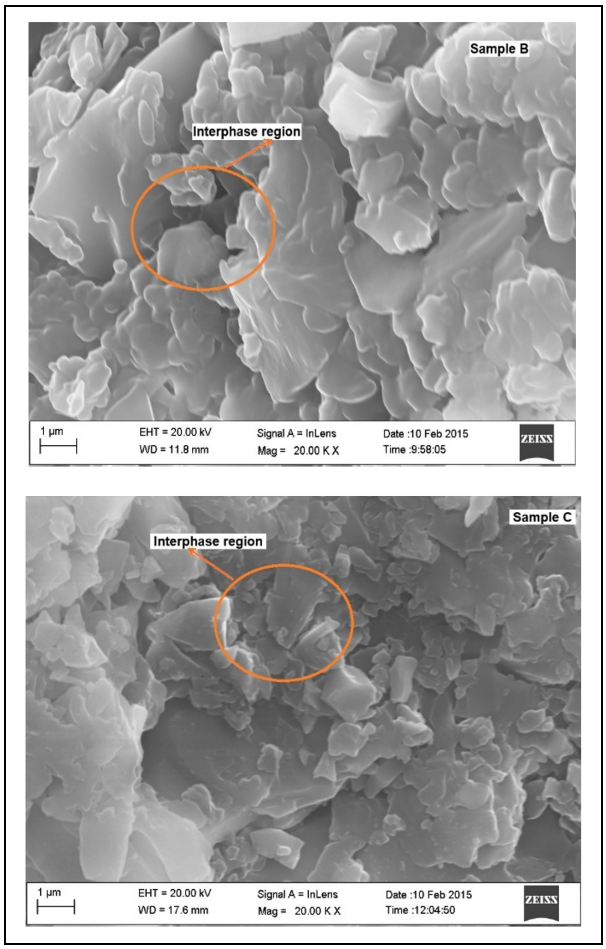



**Fig. 8: Interphase regions (a) sample B and (b) sample C**

## 4. CONCLUSION

The study carried out fabricating different composites with and without MWCNTs in different dimensions (<Ø8 nm, and Ø20-30 nm) using hand layup approach. The properties of nanocomposites were compared based on Dynamic Mechanical Analysis. The MWCNTs (Ø20-30 nm) have a lower surface area compared to the smaller (<Ø8 nm) ones, leading to better dispersion and reduced agglomeration. In contrast, the high surface area of <Ø8 nm MWCNTs, because of the strong van der Waals forces, tend to cluster together, leading to uneven dispersion and the formation of micro-

voids. The DMA tests reveal that specimen C exhibited greater molecular stability and higher cross-linking density, lower heat generation with tan δ peak of 0.3015 (C), as compared with 0.4556 (A) and 0.4194 (B) making it more resistant to drilling-induced thermal damage compared to samples A and B.

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## CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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