Investigation of mechanical properties of laminated polymer nanocomposites modified by Graphene Oxide

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Abstract

This paper evaluates the mechanical characteristics of laminated polymer nanocomposites modified by graphene oxide. The nanocomposite samples were fabricated using the traditional hand-layup method (HLM) at three different weight percentages (1%, 2%, and 3%). Tensile and flexural tests analyzed the mechanical behavior of the produced composite. The viscoelastic and thermal properties of the nanocomposite samples are calculated using dynamic mechanical analysis (DMA). Scanning electron microscopy (SEM) reveals the failure processes on the broken surfaces of the nanocomposite specimens. By enhancing the mechanical properties of structural components, the Graphene Oxide (GO) additives in composites make them more efficient than pure samples.

Keywords: hand layup, carbon fiber, graphene, tensile strength, flexural strength.

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1 Introduction

Polymer composites are most widely used in transportation, automobiles, aerospace, defense, sports equipment, infrastructure, etc., due to their lightweight, high corrosion resistance, high strength-to-weight ratio, simple design, and adaptability in the handling process [1]. Composites are arranged by reinforcements or various matrices in which the reinforcements phase plays a load-carrying factor role. However, the matrix material allows them to remain in their preferred position and is the means of load transfer between the matrix and the reinforcement. In various applications, composites are desirable, but their progress due to low toughness is limited [2, 3]. Different fibers are mixed to manufacture toughened composites and form modified hybrid composites, improving mechanical performances over single-fiber composites. For example, Kevlar fibers can be embedded in a plastic resin or shear thickening fluids (STF) to create a composite material that is strong, lightweight, and bullet-proof (like in bulletproof vests) [4–7]. Earlier investigations revealed some excellent benefits of hybrid composites [8–10]. Another example is CNTs and polymers can be combined to form a composite material with significantly improved properties compared to the individual components. Epoxy/CNT nanocomposites offer exciting possibilities due to their unique properties. These composites hold promise for applications in automotive and aerospace materials, packaging (films and containers), high strength flexible deformable channels (which under nanoscale dimensions shows unique flow characteristics [11–14]), EMI shielding, adhesives, and coatings [15]. Also, replacing costly fiber with cheaper ones will reduce costs and provide different mechanical behavior combinations [16–18]. In the fabrication of advanced engineering structures, fiber-reinforced composite polymers are actively used to alter the mechanical efficiency of the assembly. Polymer composites have a high resistance to internal and external corrosion [19]. Combining fibers to create a ply in a thin matrix layer is the first step in creating a composite structure [20–22]. In order to get the appropriate strength in a fiber-reinforced composite, it is standard procedure to lay several layers in a specific sequence and then combine them to form a composite [23]. Different layers in a ply will contain fibers in different directions. Various fibers like glass fiber, basalt fiber, aramid, Kevlar, and carbon fiber are used to create a hybrid laminate [24–26]. Therefore, new hybrid composite materials have advanced and superior features compared to conventional materials and composite structures. Earlier research findings showed efficient ways to fabricate hybrid composites from a combination of synthetic and natural fibers because of their excellent properties. Yusuff et al. [27] investigated a vacuum infusion technique to prepare hybrid composites using a combination of carbon and kenaf fibers into the epoxy resin. The tensile and flexural tests examined the hybridization effect and different piling sequences. The proposed findings demonstrate that introducing carbon fiber hybridization prominently increases the desired mechanical aspects. Amico et al. [28] used various stacking sequences of pure sisal, pure glass, and hybrid sisal/glass to understand the mechanical performances of the developed composites. This work signifies that stacking different fibers and outcomes reveals that pure glass and sisal can significantly improve properties. Arrakhiz et al. [29] used the Luffa and Glass fibers in the polyester matrix to develop hybrid composites. Subsequently, fabrication and mechanical performances were evaluated through the Tensile, three-point flexural, and hardness tests. Through carbon fiber hy-

bridization, the mechanical efficiency improvement of natural fibers was compared with that of one of the strongest fibers, flax, by different flax carbon hybrids in low-volume fractions of the interlayer [30]. The results indicate that hybrid binding morphology may influence the stiffness of fibers. Papa et al. [31] used a vacuum environment to develop the stacked composites using glass and carbon fiber and performed three-point bending and low-velocity impact tests. Consequently, the flexural and toughness test results revealed that the stacking sequence significantly impacts flexural strengths and failure mode. [32] modified conventional hand layup method for Glass/carbon fiber polymer composites. The varying load was applied to the mold during the curing of composite material, and the effects of these loads on tensile strength, modulus, and wear resistance were examined. It has been identified that the tensile strength, modulus, and wear resistance decrease when the molding load increases. Al-Hajaj et al. [33] studied the impact characteristics of a hybrid composite using an epoxy matrix comprised of carbon and flax fibers. Rolfe et al. [34] investigated the blast resistance of modified composite sandwich panels against a full-scale explosive charge. The samples were created to recognize how the layout order of the skin affects blast reaction. The sandwich panel deflection was captured during the explosion using 3D high-speed digital correlation. Combining glass and carbon fiber layers in sandwich panel hybrid laminate skins reduced normalized deflection by up to 41% and 23%, respectively, compared to GFRP and CFRP panels. Park and Jang [35] integrated the epoxy matrix and laminate of polyethylene (PE) fibers to manufacture hybrid composites. PE fibers were used because of their high break elongation, basic strength, and hardness. The mechanical test finding reveals that the hybrid composite depends on the fiber's reinforcing position, like the highest flexural strength located at the outermost layer. Carbon fiber recently emerged as one of the most promising materials for producing hybrid composites. Carbon fibers are highly regarded for their excellent mechanical strength, elasticity modulus, low density, and high flame resistance [36]. Carbon fiber is irreplaceable in vast engineering technology sectors such as cars, helicopters, vessels, buildings, and the sports industry [37, 38]. However, carbon fiber is more expensive than other fiber. The researcher observed the trend of substituting some carbon fiber layers with ductile fiber to overcome the drawbacks of carbon fiber reinforced polymer (CFRP) [39, 40]. This process is termed hybridization, which can cause considerable advantages and mechanical and physical property changes [41]. Glass fiber is a favorable reinforcement for manufacturing hybrid composites. The glass fiber has good modulus, strength, and strain to failure with a wide operating temperature range over the carbon fiber with additional chemical resistance features, ease of processing, and low cost. As a result, glass fibers are gaining popularity as a reinforcing medium for hybrid and composite laminates [42–45]. However, it is not enough in a fast-growing industry, necessitating the development of cost-effective materials with improved material characteristics. Therefore, the properties of polymer composite (structural materials) can be modified by adding nanomaterial. Nevertheless, nanomaterials present a unique challenge during manufacturing, and many different strategies have been proposed to avoid agglomeration [46]. Furthermore, functionalizing the surface of CNMs is the most effective strategy for addressing this issue [47]. The interaction between CNMs and the polymer matrix is also essential for efficient load transfer to reinforcement. In recent years, many methods, such as covalent side-wall functionality, non-covalent functionalization incorporation, etc., have been developed to increase the

usefulness of CNMs. Incorporating CNMs into the resin without compromising polymer integrity is critical for producing laminated polymer nanocomposite. Better adhesion linkages are needed to successfully transfer a large amount of load between the CNMs and matrix interface. Laminated polymer nanocomposites (LPNC) comprise alternating fiber layers and modified polymer infused with nanomaterials. These materials combine the desirable properties of polymers and nanoparticles, enhancing mechanical, electrical, and thermal properties. Several methods like Layer-by-Layer (LbL) Assembly [48], Solvent Casting [49], In-situ Polymerization [50], Electrostatic Layer-by-Layer (ELbL) Assembly [51], Hand layup, and Vacuum assisted resin transfer molding (VARTM), etc., are commonly used for manufacturing laminated polymer nanocomposites. The hand layup method is the most common for developing laminates because it allows manufacturing finished products with minimal investment in specialized equipment. As a result, many low-cost manufacturing approaches are commonly utilized in industrial practice, enabling a higher level of fiber supersaturation while reducing the likelihood of voids and pores. As a result, this paper presents a detailed examination of the characteristics (Tensile, flexural, and dynamic mechanical) analysis of LPNC by the hand layup method.

2 Fabrication and characterization method

Hand layup is one of the oldest and most conventional methods for fabricating composite structures. Carbon fabric of 400 GSM, bidirectional plane woven (reinforcement) GO with 99% pure CNMs (reinforcement), Epoxy (matrix), and hardener-D (binder) was employed to produce the nanocomposites. The high-purity acetone deagglomerates the nanomaterial particles sonicate at 45 Hz frequency, 25^{o}C for 90 minutes. Next, a magnetic stirrer mixes the epoxy matrix with GO for two hours at 80^{o}C to

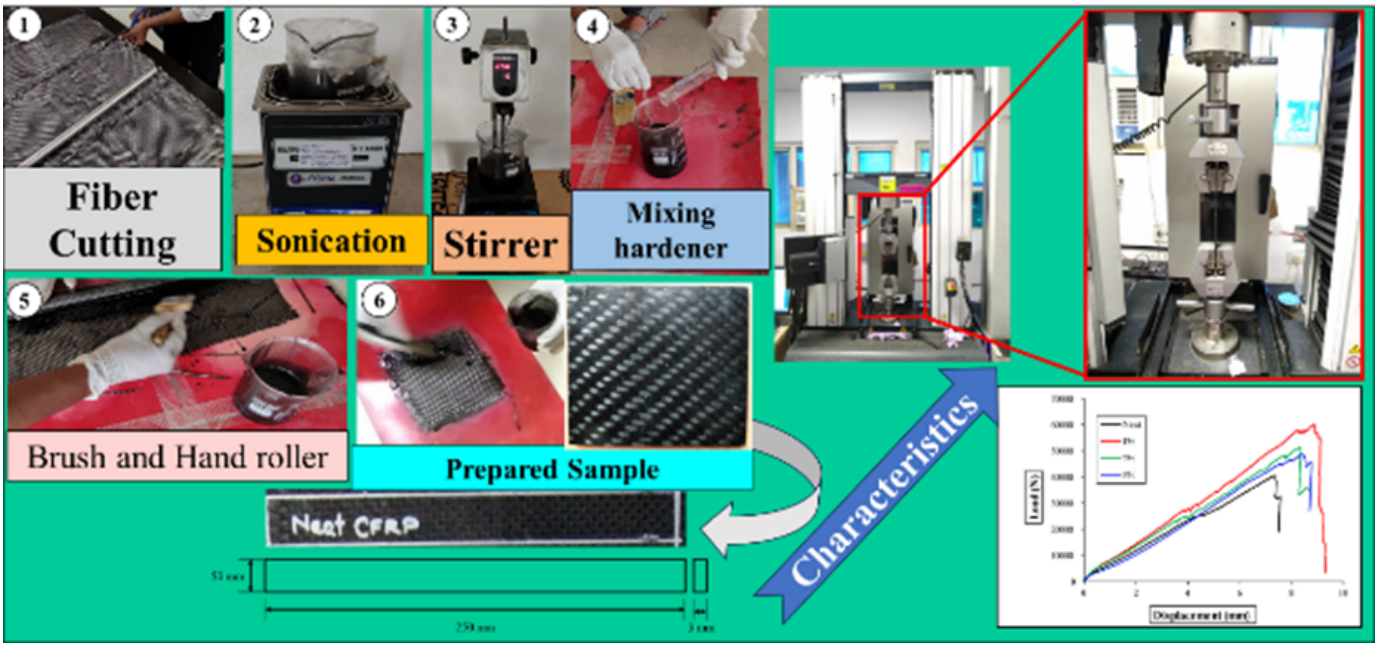

Figure 1: Hand layup preparation

improve dispersion. The impregnated fibers squeegee to manually distribute the resin throughout the layers of reinforcement using brushes and rollers. This process is repeated until the desired thickness and fiber content are achieved. The layup part is covered with a release film and peel ply to provide a smooth surface finish and to facilitate the removal of excess resin and air bubbles. The curing process takes place at room temperature. Once the resin has fully cured, the part is removed from the mold, excess flash or trim is trimmed, and sanding is performed. Hand layup offers flexibility in part geometry and can produce a wide range of composite components, including flat panels, curved surfaces, and more complex shapes. The hand layup process is demonstrated in Figure 1. The effectiveness of the developed novel material in withstanding tensile and flexural strength is evaluated following ASTM-D3039 and ASTM D790, the uniaxial stress, and the 3-point bending test (Figure 1).

3 Result and discussion

3.1 Tensile strength

As a reference, the stress-strain performance of plain CF epoxy is assessed for GO filler concentrations of 1, 2, and 3 wt.%. Figure 1 depicts the load-displacement curve of the sample. The tensile strength of neat CF/Epoxy is found to be 625.79 MPa. Adding 1 wt. % of GO increases the tensile strength by 33.02% (i.e., 934.35 MPa). Tensile strength improves by 20.61% (i.e., 788.26 MPa) after adding 2 wt.% GO, and 774.01 MPa after adding 3 wt.% GO over neat CF/Epoxy composite. The findings indicate that adding more GO reduces the tensile strength of laminated polymer nanocomposites. Because of the fillers' propensity to agglomerate together, excessive reinforcing makes structures brittleness [52]. The tensile strength of developed composites was determined to be 625.79 MPa for neat CF/Epoxy and 934.35 MPa after adding 1 wt. percent GO to the epoxy. The neat CF/Epoxy is having an 8.643 GPa young modulus and 1 wt.% GO increases the young modulus by 10.727 GPa, incorporating GO 2 wt.% reduce of modulus, i.e., 9.779 GPa and further incorporation of GO, i.e., 3 wt.% GO minimum the tensile modulus 9.416 GPa. Due to polymer chain motion restriction after high filler loading, the addition of GO at high concentrations may cause a minor reduction in the tensile curve and elongation at the feature [53]. Modifications are influenced by a number of parameters, including processing method, GO type, GO aspect ratio and GO material [54]. As observed from the reported literature, a hand layup processing technique is feasible for cost-effective [55].

3.2 Flexural strength

The flexural test measures the stiffness of the proposed nanocomposite sample. A small percentage of GO was enough to alter the flexural characteristics of CF/Epoxy significantly. The neat CF/Epoxy has a 13.807 GPa flexural modulus and 1 wt.% GO increases the flexural modulus by 19.241 GPa, incorporation of GO 2 wt.% reduce of flexural modulus, i.e., 15.57 GPa and further incorporation of GO, i.e., 3 wt.% GO minimum the flexural modulus 11.73 GPa. The flexural characteristics of CF/Epoxy modified by GO wt.%, with a little addition of GO, induced significant alterations. The

Material	Tensile properties		Flexural properties	
	Strength (MPa)	Modulus (GPa)	Strength (MPa)	Modulus (GPa)
Neat	625.79	8.644	289.95	13.807
1 wt.% GO	934.35	10.727	442.55	19.241
2 wt.% GO	788.26	9.779	363.70	15.575
3 wt.% GO	774.01	9.416	342.67	11.732

Table 1: Mechanical properties of the developed nanocomposites

flexural strength of developed composites found that neat CF/epoxy has 289.95 MPa flexural strength and adding 1 wt.% GO into the epoxy improves the flexural strength by 442.55 MPa. Nanofillers in the composite matrix also provide certain challenges in processing and handling from an engineering perspective. Therefore, obtaining homogeneous characteristics across the composite matrix largely depends on improved interphase interaction between nanofillers and matrix [56]. Matrix structures made of polymers provide a promising approach to this issue. Polymer nanocomposites, a contemporary grade of the composite family, are constantly expanding the properties of the current composite matrix. The nanoscale dimension results in a large interface area per volume between the nanoelement and the polymer matrix. Enhancing the aspect ratio positively impacts the matrix, such as self-healing, ternary systems, changing film morphology, and carrier mobility. Mechanically robust materials, particularly well suited to multipurpose items, are characterized by their durability and rigidity [57]. The properties (Tensile and flexural), strength, and modulus of fabricated nanocomposites are specified in Table 1.

3.3 Breakage analysis after tensile and flexural testing

After the tensile and flexural test, the specimen's breakage is displayed in Figure 2. The developed composite fails in uniaxial tensile and 3-point bending tests at the load point of the span length's center. A crack goes through on the compressive side of the CFRP laminate, but there is no crack on the other side surface. This indicates that the compressive side of the laminate fractured severely during bending, leading to the catastrophic failure of the entire laminate. The 1 wt.% GO had significantly fewer tensile and compressive cracks than the neat epoxy laminates, indicating different failure modes. This is due to the better dispersion of nanomaterials in the matrix and proper fiber-matrix bonding. The fiber and matrix bonding in multilayer carbon/polymer composites is more likely to rupture through different failure modes. Another crucial problem associated with breaking modes is degradation, which subsequently influences tensile strength and stiffness. Polymer matrix bonding can be affected by the inertness of the fiber surface. Inadequate fusion at the interphase of two different matrices leads to substantial damage and failure of the composite structure [58].

The crack propagation faces had "generally" large areas of protruding ruptures (ductile) in the case of the one neat specimen tested. During the tensile tests, the fracture face of the specimen containing 1% GO had areas of textured rupture, indicating the presence of some ductility. SEM fractography

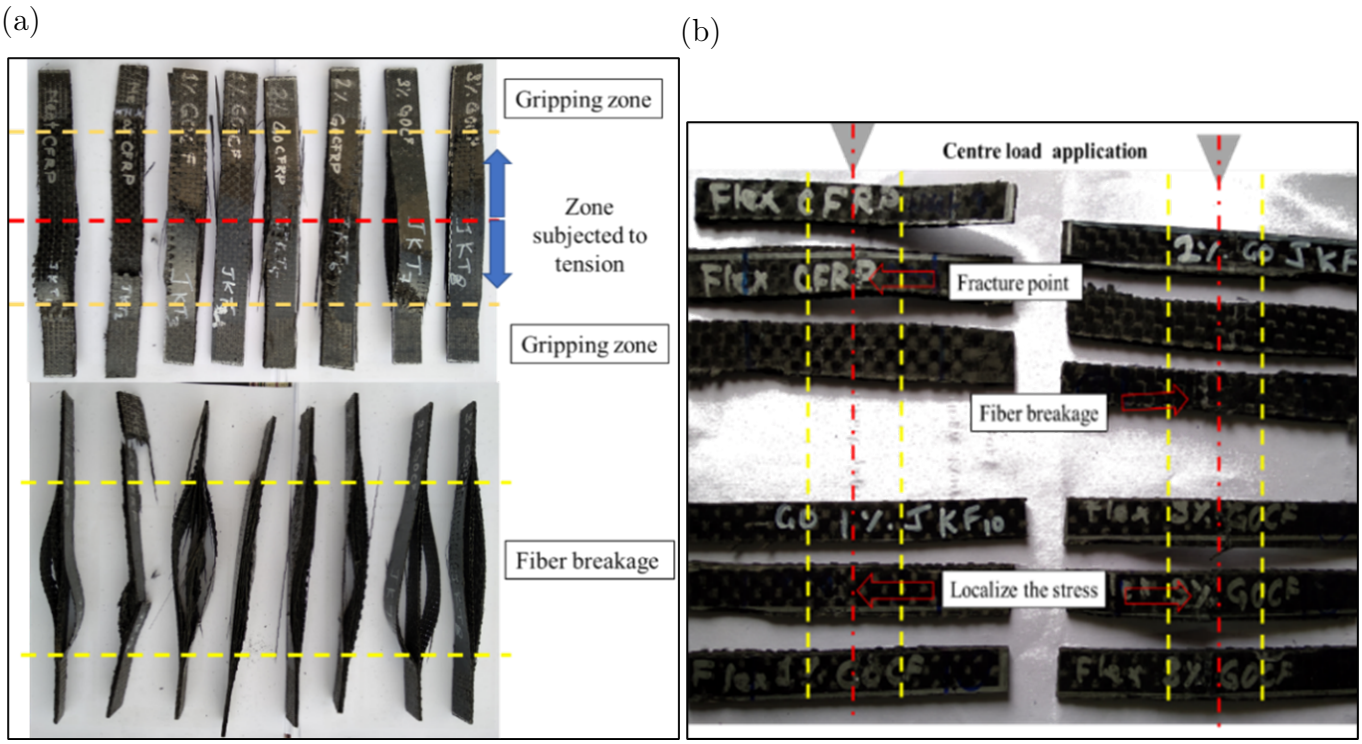

Figure 2: (a) Tensile and (b) flexural breakages after test

analysis was used to characterize the developed LPNC's fracture and prove the relationship between microstructural damage evolution and fracture mechanism. The polymer deformations, fiber pull-out, and voids can be found in Figure 3. As a result of matrix failure, a maximum load was transferred to fibers. As a result of interfacial shear and crack connections, the matrix still offers a mechanism for load transfer even after matrix cracking has occurred. It can also be seen that the fibers are being pulled out from the matrix. This phenomenon may occur due to agglomeration of nanofiller content, high fiber breakage, and poor stress transfer between fiber and matrix. A brittle failure with fewer fiber pull-outs is shown in Figures 3 and 4 for tensile and flexural tests, respectively. Modifying fibers with 1% GO could be the reason for improved bonding and dispersion. Figure 3 indicates the SEM image of the composite specimen's fracture surface after the flexural test. A brittle failure with less fiber pull-out was also noted during the test.

3.4 Dynamic mechanical analysis (DMA)

Dynamic mechanical analysis (DMA) was performed on a TGA-55 flexural analysis (3-point bending) mode with a 1.00 Hz oscillation frequency. The responses were extracted at a 5 K/min screening rate from room temperature to 300 °C. Figure 4 exemplifies the output graphs of DMA for storage modulus vs. temperature concerning energy stored per cycle is proportional with the function of inclusion of GO nanofiller. The storage modulus of pristine CFRP and GO-modified CFRP composite decreases significantly with increasing temperature due to epoxy matrix detachment from reinforcement. With the addition of GO in the first segment of the graph, i.e., the glassy zone (below T_g), the storage modulus of the modified CFRP composite over the entire temperature domain (30 to 100 °C)

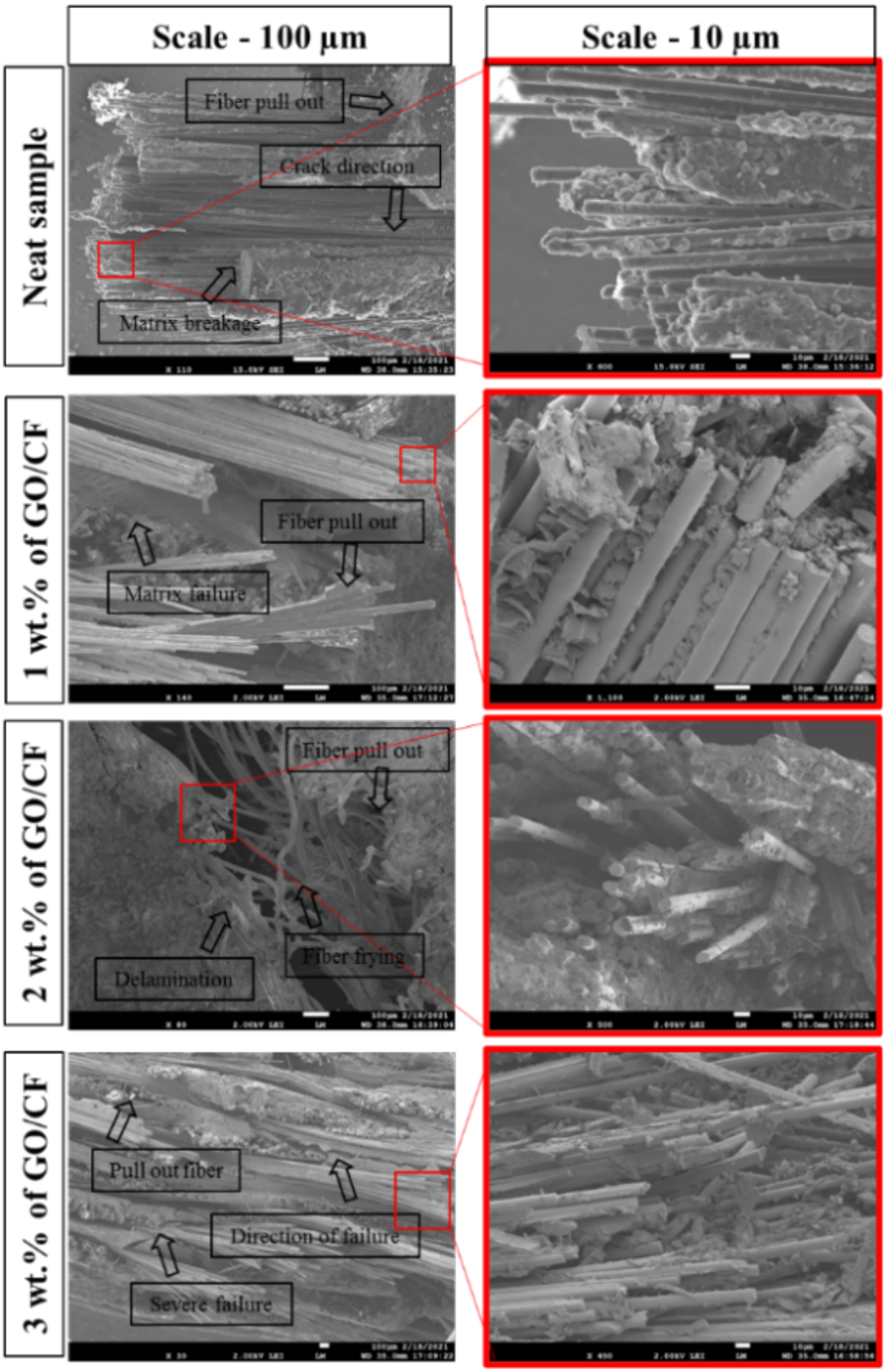

Figure 3: Microstructure of post-analysis of tensile test specimen

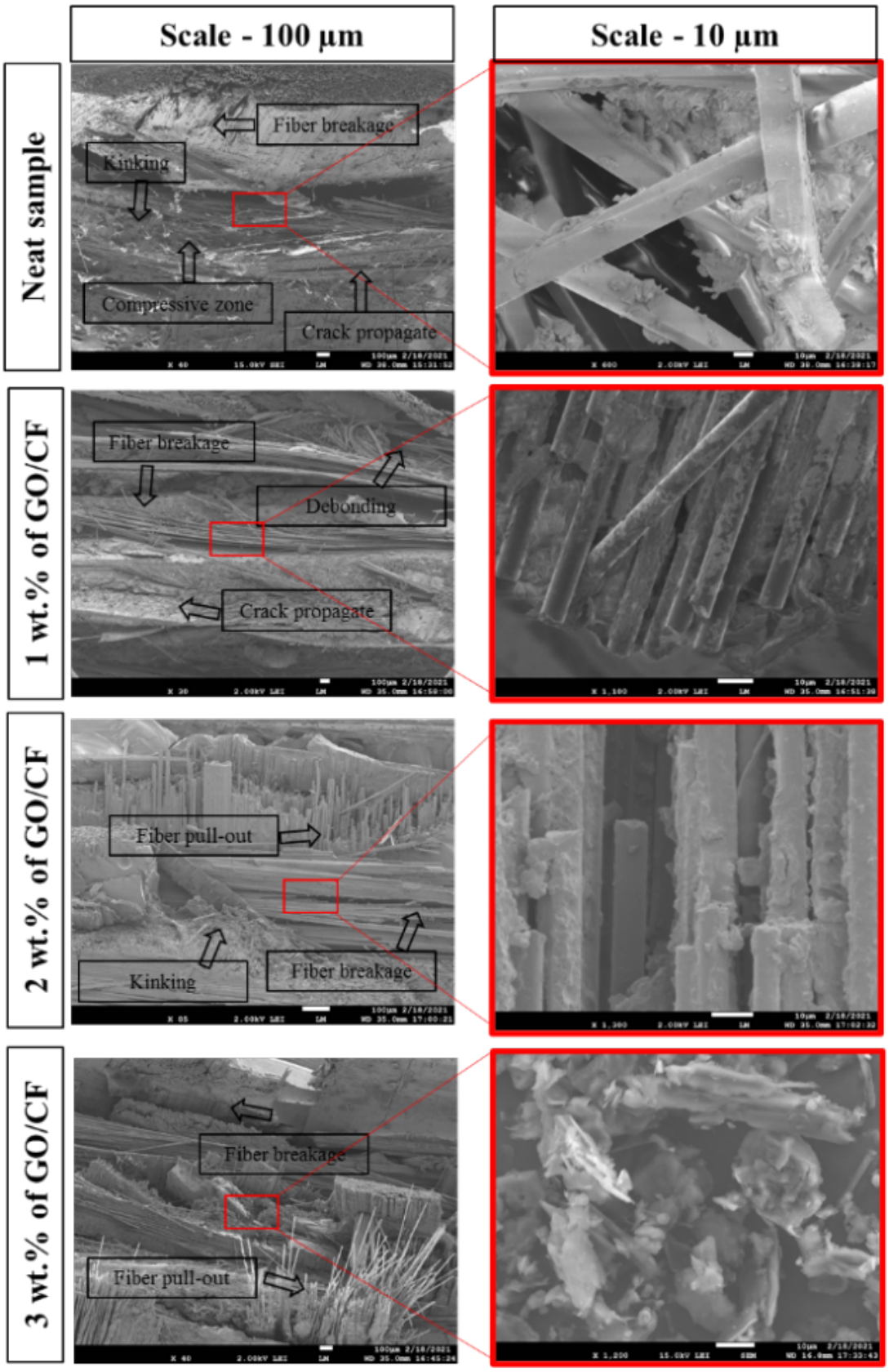

Figure 4: Microstructure of post-analysis of flexural test specimen

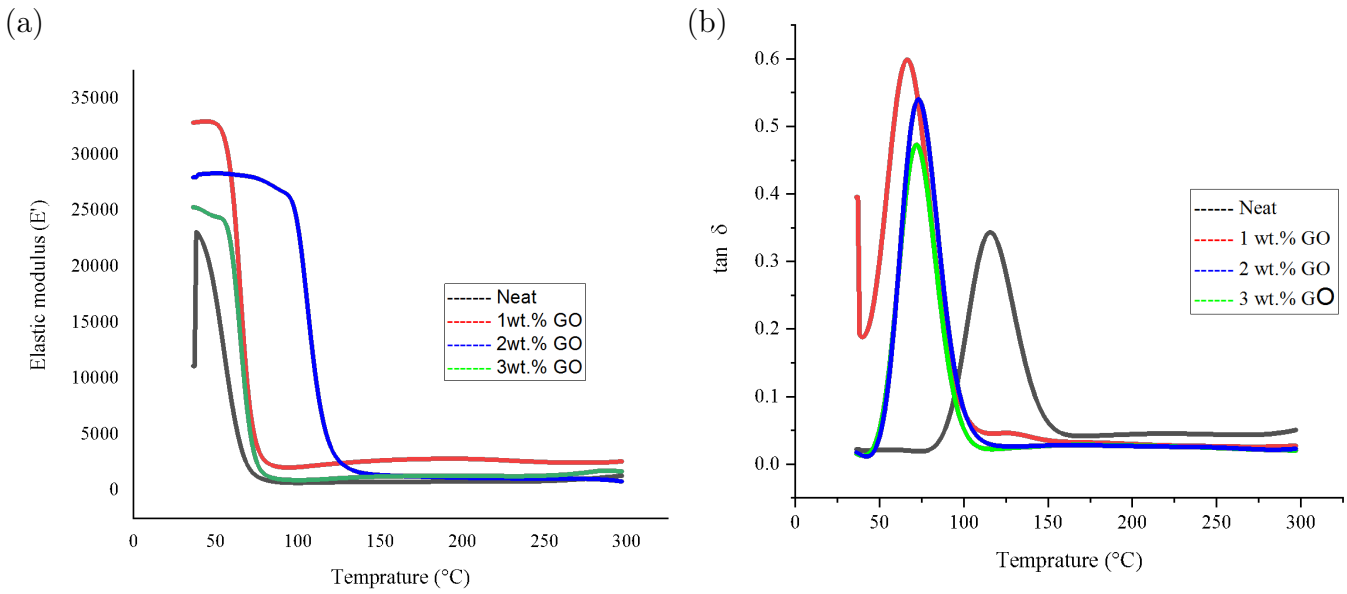

Figure 5: (a) Storage modulus vs. temperature (°C) and (b) tan vs. temperature (°C) for GO/CF epoxy nanocomposite

is raised [59]. The improved stiffness of modified CFRP composites is due to the rise in storage modulus. The storage modulus of the modified CFRP composites increases at 1 weight % of GO in the glassy zone. The storage modulus of 1 wt.% GO integrated modified CFRP composites are 17.47% improved than pristine CFRP at 35 °C. The improved storage modulus of GO-modified CFRP composites is induced by incorporating the influence of nanofillers, which limit polymer-chain mobility [60]. It increases the load-bearing capability of modified composite materials.

Furthermore, the reinforcing impact of GO in the epoxy polymer is restricted to a specified quantity of GO. Figure 5 depicts the loss factor, , the curve of the pristine CFRP, and its 1 wt.% GO incorporated modified CFRP nanocomposites and evaluated them with the help of DMA. The enhanced T_g, determined from the peak position of , was observed at 1 wt.% GO reinforcement 43 °C enhancement in T_g has been found. In Figure 4, the peak height of the loss factor decreased with an increase in GO reinforcement, but the peak width of is indifferent to the GO nano-filler. On the high-temperature side of the DMA profile, the nanocomposites had a broadened peak. The larger-peak factor for the nanocomposites implies a lesser crosslink density and higher heterogeneity, suggesting epoxy network intercalation into the GO nanomaterials [61].

4 Conclusions

This paper highlights the significance of CNMs and fiber in polymer nanocomposite development and their progressive characteristics. The mechanical characterization of Tensile, Flexural, and DMA analysis shows that the opted fabrication method has a good dispersal state of CNMs into epoxy resin. Based on the acquired results and discussion regarding the synergistic effect of nanomaterials and their properties, the following conclusion can be drawn:

- The tensile strength of the epoxy matrix used in this study increased from 625.79 MPa to 934.35 MPa, and the tensile modulus increased from 8.643 GPa to 10.727 GPa, respectively.
- The flexural strength of developed composites found that neat CF/epoxy has 289.95 MPa flexural strength, adding 1 wt.% GO into the epoxy improves the flexural strength by 442.55 MPa. The neat CF/Epoxy has a 13.807 GPa flexural modulus and 1 wt.% GO increases the flexural modulus by 19.241 GPa.
- The one wt.% GO/CF laminates had significantly fewer tensile and compressive cracks than the neat epoxy laminates, indicating different failure modes.
- According to DMA test results, the peak strength of tan delta curves decreased with the add-up of GO contents and CF into the epoxy phase. The finding of the curves shows shifting to a higher feature value. These curves shifted to the curve of tan delta peak increases due to enhanced interfacial adhesion between the reinforcing materials.

This paper pertains to fabricating epoxy nanocomposite comprising Graphene oxide/carbon fiber layers. The proposed manufacturing method shows the feasibility and application potential for developing multifunctional end products. The finding indicates that 1.0 wt.% of GO was the optimum addition to improve the mechanical properties of the composites.

Conflicts of interest

The authors declared no potential conflicts of interest.

Data availability statement

All data generated and analyzed during this study are included within this manuscript.

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