Influence of carbon nanotubes/ graphene nanoparticles on the mechanical and morphological properties of glass woven fabric epoxy composites

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Abstract: *An epoxy matrix reinforced with multi-walled carbon nanotubes (MWCNTs) and graphene nano particles (GNPs) is used to fabricate woven glass fabric epoxy composites using Hand Layup and compression moulding technique. Three types of composites are fabricated using 7-mill plain weave glass fabric, +45°/-45°, 0°-90° multi axial glass woven fabrics as reinforcements and epoxy as matrix. Mechanical characterization is performed on the fabricated composites.0°-90° GWFE composites are fabricated with 0.5 wt%, 1.5 wt% MWCNTs, and 0.5 wt%, 1.5 wt% graphene nano particles (GNPs).The results from mechanical and morphological characterization revealed that specimens containing 0.5 wt%, 1.5 wt% MWCNTs, and 0.5wt %, 1.5 wt% graphene nanoparticles (GNPs) are altered when compared to the glass woven fabric epoxy composites without nanoparticles. Glass woven fabric epoxy (GWFE) composites with 1.5 wt% of MWCNT possess the highest hardness of 90.33 which is 6.27% greater when compared to other composites. The tensile strength of composite specimens containing 1.5 wt% graphene nanoparticles (GNPs) increased by 14.5% over glass woven fabric epoxy (GWFE) composites. The mode of fiber failure in tensile fractured surfaces of GWFE composites is apprised through SEM images.*

Key Words: *Tensile strength, epoxy resin, nano reinforcements, Glass woven fabric epoxy (GWFE), multi-walled carbon nanotubes (MWCNTs), graphene nanoparticles (GNPs), fiber pull out*

1. INTRODUCTION

Aerospace, construction, transportation, automotive, sports goods, employ fiber-reinforced composite materials [1]. Amid the fiber reinforced composite materials, glass fiber polymer composites find place in enormous applications because of their high corrosive resistance, high stiffness to density ratio, high endurance limit, and greater manufacturing ability when compared to other engineering materials [2]. The mechanical properties of nano polymer composites are improved by the addition of CNTs which were exposed in 1990s [3]. The flexural modulus, toughness and strength of the epoxy based nano polymer composites are enhanced through the incorporation of CNTs by altering the epoxy structures [4]. Glass woven fabric reinforced epoxy composites are fabricated by hot press technique with epoxy to hardener ratio at 10:3 [5]. Epoxy composites with woven fabric are manufactured by hand lay

up technique followed by compression moulding with resin and hardener to ratio as 100:12 [6]. Oil cake- filled glass woven fabric epoxy composites are prepared by epoxy, hardener in the weight ratio of 100:38 using hand layup followed by curing at a pressure of 0.0965MPa for 24 hours using H-type press [7]. Woven mat glass fiber epoxy composites with fabrics at various weaving angles are studied and it was found that the energy absorption increases with decrease of weaving angle between interlacing yarns and accordingly [0º/20º] woven composite exhibits better energy absorption than [0º/90º] woven composite when subjected to impact test [8]. The effect of orientation on glass fiber polymer composites with chopped strand and rovings at three different orientations such as 0º, 45º, 90º is studied and the tensile strength for the composite with 90º, fiber orientation is found to be maximum, decremental impact strength due to short fibers [9]. E-glass woven fabric polyester composites are incorporated with (CNF) carbon nano fillers at 0.1, 0.2, 0.3, 0.4 weight percentages. 0.2wt% CNF filled composites show maximum mechanical properties when compared to other CNF filled composites due to the good dispersion and interfacial interaction between fiber and matrix [10]. Woven fabric composites possess 15% less compressive strength over non-crimp laminate [11]. The shear strength in the laminates of woven E- GF reinforced epoxy composites with modified and unmodified epoxy is observed and the modified composite i.e. with 0.5wt% MWCNTs and 10phr butyl glycidyl ether (BGE) epoxy composite exhibit 25.4% increment in interlaminar shear strength than unmodified composite [12]. Glass fiber epoxy composites in three different orientations i.e. [0º/90º], [0º/+60º/-60º], [0º/+45º/-45º] are investigated by performing low velocity impact tests and it was found that [0º/90º] GF-reinforced epoxy composite possesses/has low saturation energy when compared to other composites [13]. Epoxy composites with chopped strands of E-glass fiber having various fiber volume fractions are subjected to TGA and it was found that the composites with 60% fiber volume fraction possess enhanced thermal stability, shift in degradation temperature from 357ºC to 390ºC and the addition of nanoparticles in epoxy matrix improves the mechanical properties of the composite [14]. Woven fabric E-Glass reinforced epoxy composites are subjected to immersion in four different liquid media such as distilled water, saturated salt solution, 5- molar NaOH solution and 1- molar hydrochloric acid solution and there is lower damage for the composites immersed in distilled water, which did not affect the mechanical properties [15-16].

2. MATERIALS AND METHODS

2.1 Materials

Plain woven 7-Mill glass fabric +45º/-45º, 0º-90º multi-axial woven glass fabric, Epoxy Araldite LY 951 and hardener HY556 are collected from M. S. industries located in Kakinada, A.P., India. Multi-walled Carbon-Nanotubes (MWCNTs), Graphene nano particles (GNP) with 10-30 nm OD, length of 1-10µm, 90% purity and Carbon – 90.26%, Oxygen – 8.49% multi layered with thickness approximately 3-10 nm, diameter of 20-60µm, purity greater than 98% respectively, are procured from nano wings centre in Khammam, Telangana, India. Hardener and epoxy are mixed in the ratio of 1:10 as per the specifications.

2.2 Methods

Ethanol is added to as received MWCNTs which are in agglomerated form, ultrasonic vibrator is utilized for sonication of 1 hour at 40 KHz [17]. The MWCNTs, GNP are introduced in epoxy, mixed uniformly with the help of magnetic stirrer rotating at 800 RPM for 120 minutes.

The fabrication of composites mainly involves cutting of plain woven 7-Mill glass fabric, +45º/-45º, 0º-90º glass woven fabrics according to the mold dimensions which are shown in Figure 1.

Fig. 1 - Cutting woven glass fabric in to required mould/ mold dimensions

Applying epoxy resin on the glass fabric and laying the fabric samples one above the other along with ramming accompanied by hydraulic pressing at 100ºC for 2 hours gives laminates with desired thickness. The number of layers to be placed depends on the type of woven glass fabric i.e. 6 layers for 7-Mill glass fabric, 3 layers for 0º-90º, 3 layers for +45º/-45º glass woven fabric. The fabricated GWFE composites are shown in Figure 2 (a, b, c). Mechanical characterization is performed on the fabricated composites. MWCNTs and GNPs at 0.5 and 1.5 wt% are incorporated in to the epoxy resin using magnetic stirrer. MWCNT/ GWFE composites and GNP/ GWFE epoxy composites are fabricated through hand layup and compression molding technique.

Fig. 2(a, b, c) - Fabricated 7 M, 0º-90º, +45º/-45º GWFE composites

GWFE, MWCNT/ GWFE and GNP/ GWFE composites are subjected to mechanical tests such as tensile, compression, flexural and impact tests. Adithya UTM equipment (model UTE - 40KN, which is shown in Figure 3 (a)) is utilized to find out the tensile and compressive properties of the samples as per ASTMD3039 and ASTM D695 standards. Specimens of 3mm thickness, 25mm width, 250mm gauge length are subjected to tensile load with a span length of 150mm,and cross head travel of 2mm/min. Compressive load is applied to samples of 3/12.7/25.4 mm (thickness/ width/ gauge length) dimensions with cross head moving at 2mm/min. Figure 3 (b)) depicts the United UTM (model STM-50KN) equipment which is used to evaluate the flexural properties of the samples. The scale of the specimens tested for

flexural properties is approximately 3/ 10/ 50 mm (thickness/width/gauge). Five samples of each composite are at least examined. The Izod impact strength of the notched samples are evaluated on a KI-1.4 instrument depicted in Figure 3(c) as per ASTM D256.

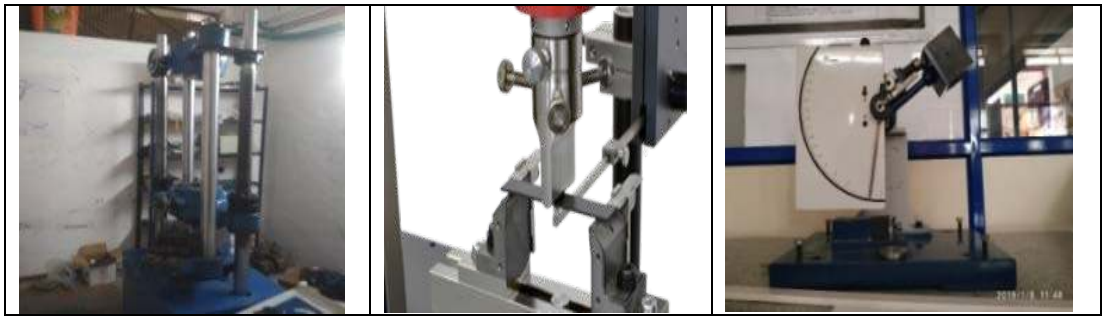


Fig. 3(a, b, c) - Experimental setup of Tensile, Compression, Flexural and Impact tests

The measurements are performed at ambient conditions of 23°C temperature and 40% relative humidity with 3.2/ 12.7/ 64 mm dimensioned samples. Impact strength is calculated by dividing impact energy with sample width. Durometer is used to examine the Shore D hardness of the samples as per ASTM D 2240 standard. In certain standard conditions of force and time, the indenter penetration into the sample is noted. Surface morphology of GWFE composites and tensile fractured specimens are studied through scanning electron microscope (SEM; TESCAN, VEGA 3 SBH, CZECH Republic) at 10 kV acceleration voltage.

3. RESULTS AND DISCUSSIONS

3.1 Tensile properties

The tensile strengths presented in Figure 4 indicates highest tensile strength for +45°/-45° multi axial glass woven fabric epoxy composite. This is due to the stronger opposing force from the interlaced fibers aligned in an angle of 45° to the direction of tensile load. 1.5wt% GNP/0°-90° GWFE composite led to 14.5% increase in tensile strength without any reduction in elongation when compared to 0°-90° GWFE composite. 0.5 MWCNT/ 0°-90° GWFE and 1.5 MWCNT/ 0°-90° GWFE showed reduction in tensile strength, which might be due to the entrapping of solvent and also due to increase in viscosity of the matrix [18]. Compared to MWCNTs, GNPs possess rougher nanoscale surface that enables stronger fiber/ matrix interface interlocking.

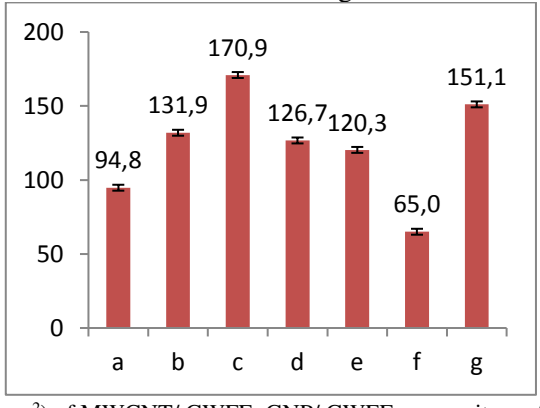


Fig. 4 - Tensile strength (N/mm^2) of MWCNT/ GWFE, GNP/ GWFE composites, a-7M GWFE, b-0°-90° GWFE, c - +45°/-45° GWFE, d - 0.5 MWCNT/0°-90°GWFE, e - 1.5 MWCNT/0°-90°GWFE, f- 0.5 GNP/0°-90° GWFE, g - 1.5 GNP/0°-90°GWFE

3.2 Compressive properties

It is evident from results in Figure 5, that the compressive strength of 0º-90º GWFE composite is highest when compared to 7M GWFE and +45º/-45º GWFE composites. This may be due to the fibers parallel to the loading direction (90º plies) and perpendicular to the loading direction (0º plies). As the load increases, there is a strong opposing force from both the parallel and the perpendicular direction, resulting in crack initiation and progressive starting, which eventually leads to fiber buckling.

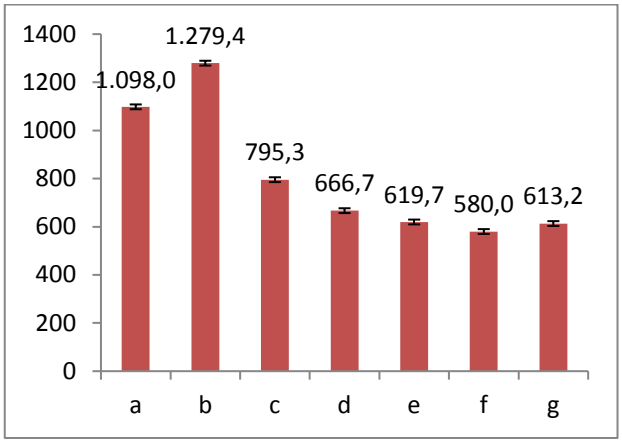


Fig. 5 - Compressive strengths (N/mm^2) of MWCNT/ GWFE, GNP/ GWFE composites, a - 7M GWFE, b - 0º-90ºGWFE, c - +45º/-45º GWFE, d - 0.5 MWCNT/0º-90ºGWFE, e - 1.5 MWCNT/0º-90ºGWFE, f - 0.5 GNP/ 0º-90º GWFE, g - 1.5 GNP/ 0º-90º GWFE.

The compressive strength of 0º-90º GWFE nano composites is drastically reduced to 55% due to the voids in CNTs enriched matrix and the instigation of early failure of CNTs/ epoxy interface. Deterioration in mechanical properties is observed due to the presence of MWCNT agglomerates or GNP [19].

3.3 Flexural properties

0º-90ºGWFE composite exhibits better flexural strength when compared to 7M GWFE and +45º/-45º GWFE composites. Figure 6 indicates that 0.5 MWCNT/ 0º-90º GWFE composites showed 37% improvement in flexural strength when compared to other composites which may be due to the presence of carbon nano particles in the layers of glass fabric along with epoxy which acted arresters for slippage and by preventing the crack propagation with 1.5 wt% in the matrix. Due to the possible agglomeration of MWCNTs, MWCNTs in epoxy matrix hindered the flexural strength of the composites [20].

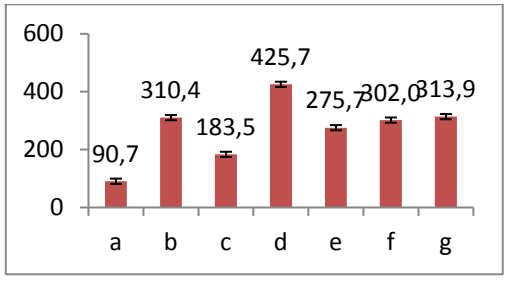


Fig. 6 - Flexural strengths (N/mm^2) of MWCNT/ GWFE, GNP/ GWFE composites, a - 7M GWFE, b - 0º-90º GWFE, c - +45º/-45º GWFE, d - 0.5 MWCNT/0º-90º GWFE,e- 1.5 MWCNT /0º-90º GWFE, f - 0.5 GNP/ 0º-90º GWFE, g - 1.5 GNP/ 0º-90º GWFE

3.4 Hardness

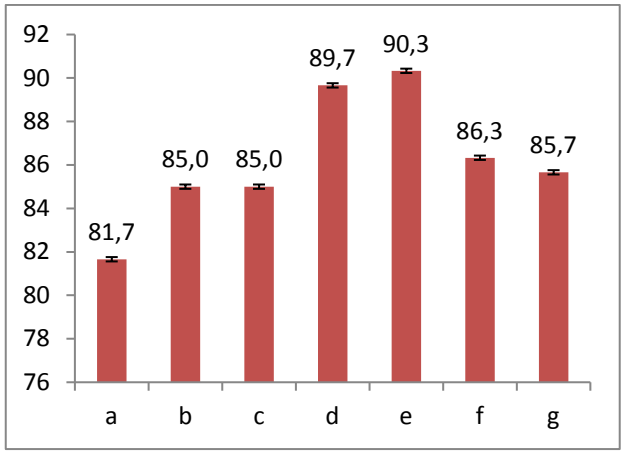


Fig. 7 - Hardness of MWCNT/ GWFE, GNP/ GWFE composites, a -7M GWFE, b - 0º-90º GWFE, c - +45º/ -45º GWFE, d -0.5 MWCNT/ 0º-90º GWFE, e - 1.5 MWCNT/ 0º-90º GWFE, f - 0.5 GNP/0º-90º GWFE, g - 1.5 GNP/ 0º-90º GWFE

Figure 7 depicts the effect of MWCNT and GNP content on the hardness of glass woven fabric epoxy composites. GWFE composites with 1.5 wt% of MWCNT possess the highest hardness of 90.33 which is 6.27% greater over other composites due to the strong interfacial bonding between fiber and matrix [21]. No significant change is observed between 0.5 and 1.5wt% MWCNTs in epoxy matrix.

3.5 Impact strength

Izod impact strengths are shown in Figure 8. With GNP addition to GWFE composites, the impact strength increased from 46.51 to 67.8 KJ/mm^2. The impact strength of GWFE composites incorporated with GNP at 1.5wt% is appreciable when compared to other composites, and it may be due to the geometry (larger surface area) of GNP which facilitates in good matrix-reinforcement bonding.

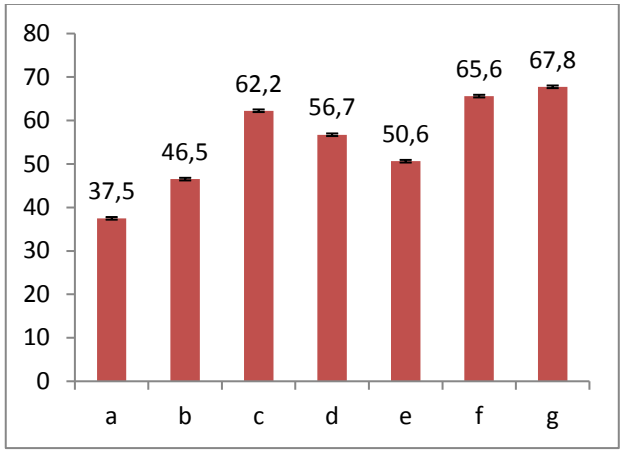


Fig. 8 - Impact srengths (KJ/mm^2) of MWCNT/ GWFE, GNP/ GWFE composites, a - 7M GWFE, b - 0º-90ºGWFE, c - +45º/-45º GWFE, d - 0.5 MWCNT/ 0º-90º GWFE, e - 1.5 MWCNT/ 0º-90º GWFE, f - 0.5 GNP/ 0º-90º GWFE, g - 1.5 GNP/ 0º-90º GWFE

GNPs' ability as reinforcing materials comes from the more residual groups of hydroxyl and epoxides on their surface. The interfacial adhesion between fiber and matrix was improved by micromechanical interlocking and covalent bonding with epoxy matrices, resulting in superior mechanical efficiency of GNP-containing composites [22].

Crack propagation by GNP is effectively retarded when compared to MWCNTs due to their geometry.

Failure during mechanical tests occurs when there is a weak interface between woven glass fabric and matrix in polymer laminate.

In this case, the increment in mechanical properties is due to the presence of MWCNT and GNP may not be transferable to the laminates. In case of failure mechanism associated to interfacial debonding, the incorporation of nanoparticles further deteriorates the bonding between the matrix and the woven glass fabric [23].

3.6 Morphological Characterization

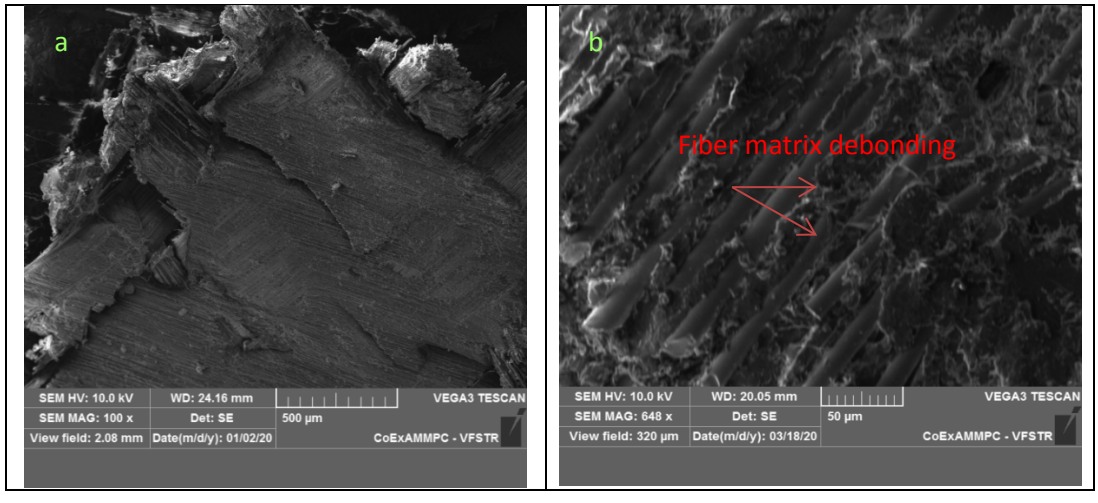
Figure 9(b, c, d) displays the SEM images of tensile fractured surfaces of GWFE composites with MWCNT and GNP.

Figure 9(a) presents the glass woven fabric completely embedded in epoxy matrix which indicates a good interfacial adhesion between the matrix and the reinforcement.

Not only do the MWCNTs and GNP have improved dispersion, but they also increase the chemical interaction and wettability with the matrix, both of which improve the interface between fiber and matrix.

Hydroxyl groups can react with epoxy resins, while epoxide groups can react with amine-curing agents and reactive carboxyl groups on GNP surfaces, both of which further improve the interfacial adhesion of the fiber/matrix [24-25].

When the samples are prone to tensile fracture, Figure 9 (b, c, d) indicates the process of failure of glass fabric initiated by interfacial crack propagation. These interfacial cracks may stop transmitting force properly to produce matrix de-bonding that eventually pulls glass fibers out of the matrix. Figure 9(d) indicates fiber breakage of composite due to the presence of nano particles which inhibits crack growth. Henceforth the tip of the crack is deviated in multi directions which continue to progress and facilitate the load to the glass fibers which are brittle in nature. Unlike the above, the woven glass fiber composites with aligned CNTs –the reinforced layer of matrix facilitated the crack travel very easily through the mid plane without any fiber/ epoxy de-bonding or fiber bridging [26-27].



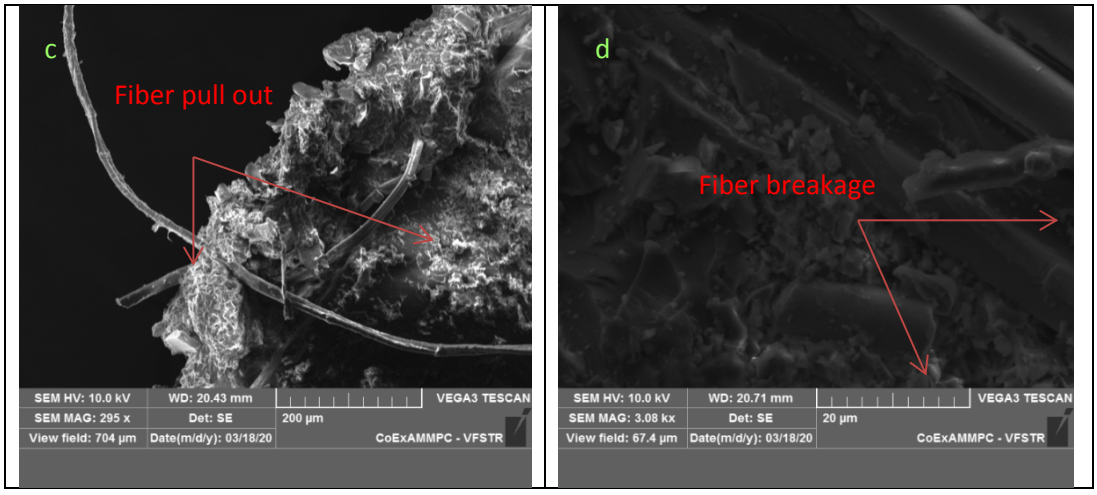


Fig. 9 (a, b, c, d) - SEM images of Tensile fractured specimens of GWFE composite

5. CONCLUSIONS

This paper presented the manufacturing process and effect of MWCNTs and GNPs on the mechanical and morphological properties of GWFE composites. The experimental results inferred that the mechanical properties vary with type of woven fabric used and concentration of MWCNTs and GNPs in matrix.

The failure modes of fiber-matrix are observed under SEM. The highest tensile strength of 170.9 N/mm^2 is noticed for +45º/-45º multi axial glass woven fabric epoxy composite.

1.5wt% GNP/0º-90º GWFE composite led to 14.5% increase in tensile strength without any reduction in elongation when compared to plain 0º-90º GWFE composite.0.5 MWCNT/ 0º-90º GWFE and 1.5 MWCNT/ 0º-90º GWFE showed reduction in tensile strength, which might be due to increase in viscosity of the matrix.

The compressive strength of 0º-90º GWFE composite is 1279.4 N/mm^2 which is highest over 7M GWFE and +45º/-45º GWFE composites.

The compressive strength of 0º-90º GWFE nano composites is drastically reduced to 55% due to the voids in CNTs enriched matrix and the instigation of early failure of CNTs/epoxy interface.

1.5wt% MWCNTs in epoxy matrix hampered the flexural properties of the composites due to potential agglomeration of MWCNTs.

GWFE composites with 1.5wt% of MWCNT possess highest hardness of 90.33 which is 6.27% greater over other composites due to strong interfacial bonding between reinforcement and matrix.

No significant change is observed in the hardness of composites with 0.5 and 1.5wt% MWCNTs in epoxy matrix.

With GNP addition to GWFE composites, the impact strength increased from 46.51 to 67.8 KJ/mm^2.

The SEM images of tensile fractured GWFE nano composites showed failure through interfacial crack propagation, fiber pull out and fiber de-bonding.

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