



Assessment of the performance of aluminum, copper, and graphene nanometer fillers filled woven glass fiber/epoxy composites

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Abstract

This study embedded graphene nanoplatelet (GNP), nano-sized aluminum and copper in epoxy-reinforced woven glass fiber. The 0.5 wt% nanofillers were incorporated into epoxy resin. Nanofilled glass fiber/epoxy hybrid composite laminates were fabricated using the hand layup technique. Fillers were dispersed in an epoxy matrix via the ultrasonic processor. The influence of the inclusion of these nanofillers on the physical and mechanical performance of glass fiber/epoxy composites was studied. The resulting hybrid nanocomposites illustrated good characteristics when compared to control specimens. The graphene nanophased glass fiber/epoxy composite achieved the maximum improvement of 59.2%, 91.5%, 74.7%, and 100% in in-plane shear, fracture toughness, impact, and interlaminar shear strength, respectively. Additionally, glass fiber/epoxy filled with GNP behaved as the lowest seawater absorption. Control filled with 0.5 wt% GNP showed strong fiber/matrix interfacial bonding in scanning electron microscopy images after being subjected to a fracture toughness test.

Keywords Glass fiber/epoxy · Aluminum · Copper · Graphene nanoplatelet · Mechanical properties · Seawater absorption

Introduction

Polymeric composites have several benefits over traditional materials, including high specific stiffness and strength, good damping properties, good toughness, high impact resistance, low thermal expansion, good corrosion resistance, high wear resistance, ease of shaping, and lower cost than lightweight metals [1–7]. The most popular structural polymer is epoxy, which possesses outstanding bonds, high chemical resistance, high strength and elastic modulus, low shrinkage, ease of processing, good dimensional stability, and good adhesiveness to various substrates [8–10]. For generating lightweight components, which is the primary necessity for industries today, nanofillers are a superior choice to other reinforcements [11, 12].

As a result of the tremendous interfacial area and proximity of the nano-fillers, polymers reinforced with them

have unique properties [13, 14]. Due to their high surface area, nanofillers generally improved the fracture toughness of polymers and fiber-reinforced polymers more effectively than micro-fillers and at significantly lower filler concentrations [15, 16].

Graphene nanoplatelets (GNP) with percentages from 0.1 to 2.0 wt% were distributed within epoxy utilizing a high processor and 3-roll milling. The most notable increase in fracture toughness, 66%, is attained at 0.1 weight% GNP, followed by adding 0.5 weight% GNP [17]. An improvement of 51.2% in the fracture properties was obtained in epoxy nanocomposites using plasma-functionalized GNP at low filler content (0.25 wt%) [18]. Megahed et al. [19] investigated the impact of introducing aluminum (Al) fillers with sizes ranging from 0.2 to 4 wt% to epoxy composites reinforced with glass fibers. The study assesses the increase in mechanical properties by including Al particles with nanometer and micrometer sizes in control composites. Compared to control, the composites displayed improved tensile, flexural, hardness, wear, and impact behavior. Flexural strength, flexural strain, and flexural stiffness were enhanced by 52.2%, 21.4%, and 76.6%, respectively. The effect of copper (Cu) nanopowder on kevlar fiber reinforced epoxy resin composites was investigated [20]. Sample with 15%

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Kevlar/82% Epoxy/3% Cu nanopowder showed the highest results for charpy test. Sample with 15% Kevlar/85% Epoxy/0% Cu nanopowder showed the highest results for the Izod test.

Glass/epoxy composites had organically modified nano-silica added with weight percentages of 0, 0.5, 1, and 3 wt% [21]. Compared to other polymeric composites and nanocomposites, nanocomposite containing 0.5 wt% nano-silica displayed the highest ballistic limit and energy absorption. At 0.5 wt%, the most increased fracture toughness of 1.12 MJ/m³ occurred. Additionally, epoxy was used to embed GNP at weight percentages of 0.1, 0.2, and 0.3 [22]. Compared to pure polymer, the critical stress intensity factor was enhanced by 64% with the inclusion of 0.3 wt% GNP. Additionally, Nayak and Ray [23] observed that seawater diffusivity increased by 15% due to inserting 0.1% of titanium oxide nano particles into the epoxy. The interlaminar shear strength and flexural strength of seawater-aged nanocomposites were increased by 23% and 15%, respectively. However, adding 1.25 wt% multiwall carbon nanotubes to a glass fiber/epoxy composite increased its flexural strength by 13.8% and its interlaminar shear strength by 6.51% [24]. Additionally, adding 2 wt% multiwall carbon nanotubes to glass fiber/epoxy composite increases flexural strength and interlaminar shear strength by 20.34% and 34%, respectively. Glass/epoxy composites filled with 0.5% nanosilica recorded higher ballistic limit, energy absorption and fracture toughness as compared with other nanocomposites [21]. Moreover, with the inclusion of 0.5% weight carbon nanotube, there was improvement of 24.37% in interlaminar shear strength in epoxy based composites and 10.05% enhancement in thermoplastic polyurethane reinforced glass fiber composites [25]. In addition, the most significant improvement of 66% in fracture toughness was obtained by adding 0.1 wt % GNP to epoxy resin followed by adding 0.5wt% GNP [17]. Furthermore, Ravichandran et al. [2] reported that the inclusion of 0.5 wt% halloysite nanotubes has considerably increased the tensile strength, tensile modulus, flexural strength, flexural modulus, fracture toughness, critical strain energy release rate and micro-hardness of the nanocomposites by 45, 49, 46, 17, 125, 134 and 11% respectively.

AL is distinguished by its light weight due to its low density, high strength, and ease of machining, as well as its outstanding corrosion resistance [5, 26, 27]. Adding Al to epoxy enhanced hardness and dimension accuracy [28–31]. Graphene nanoparticles have been extensively investigated since they offer a wide range of favorable benefits [32]. Since GNP are well known for their good strength and thermal properties, produce high-performance composite structures at lower costs,

and may improve the interfacial stress transfer, they were chosen as potential alternative fillers in the current work instead of other conductive fillers [33–36]. Composites reinforced with Cu are characterized by their outstanding wear resistance, and good chemical resistance [37].

Studies on the development of innovative composites have been carried out consistently. Most research studied the effect of adding Al and Cu fillers in polymeric composites but in micron size with high weight%. Despite the various benefits of using metal nanofillers as Al and Cu, little research has been done to investigate the addition of Al and Cu in nano size to woven glass/epoxy composites, according to the review of the body of existing literature. Comparing the nanocomposites with composite filled with widely used nanofiller as graphene nanoplatelet filler was the study's objective. This work shows the impact of introducing low-cost nanofillers (Al, Cu and GNP) with a low weight content of 0.5 wt% on the mechanical behavior and corrosion resistance of glass fiber/epoxy.

Experimental work

Materials

The fabricated composite material in this work consisted of eight layers of E-glass woven fiber (300 g/m²) embedded into epoxy. E-glass has an areal density of. Kema-poxy 150 RGL represented the epoxy system and was supplied by CNB Company. Several nano-fillers (Al, Cu, and graphene nanoplatelet) were included into epoxy with 0.5 wt%. Al and Cu nanofillers with a purity of 99.9% and a diameter of 70 nm were provided by US Research Nanomaterials. However, the graphene nanoplatelet was provided by Aldrich Company. The graphene nanoplatelet size was 5 μm with a 6–8 nm thickness and a surface area of 120–150 m²/g. Physical and mechanical properties of the used glass fibers and epoxy as provided by the suppliers are illustrated in Table 1. Table 2 shows the characteristics of the used Al, Cu, and GNP.

Table 1 Properties of fibers and matrix given by the suppliers

Property	Woven glass Fiber	Epoxy resin
Density, g/cm ³	2.5	1.1
Young's modulus, GPa	7.6	1.2
Poisson's ratio	0.22	0.35
Shear modulus, GPa	33	1.4
Elongation, %	1.8–3.2	2.2–2.9
Tensile strength, MPa	3400	55–58

Table 2 Properties of Al, Cu, and GNP

Property	Al nanoparticles	Cu nanoparticles	GNP
Density (g/cm ³)	2.76	8.92	1.1–1.5
Tensile strength (MPa)	60–200	200–400	300–700
Tensile modulus (GPa)	70	117	200
Conductivity (M/m.k)	36	58	100
Thermal conductivity (w/m.k)	200	400	(440–480)
Melting point (°C)	660	1083	4620

Nanocomposite laminate preparation

Nanofillers were added to epoxy and manual mixing. Afterward, this mixture was sonicated for 2 h. Mixing was done at an amplitude of 40% using a Hielscher ultrasonic processor UP 200 S with 0.5s on/off cycle [38, 39]. The blend was cooled by immersing in mixture of ice and water during sonication to decrease the generated temperature from the sonication process [37, 40, 41] as shown in Fig. 1. Then, a hardener was steadily included to the blend with a ratio of 2:1 and was stirred thoroughly. Nanophased epoxy was uniformly spread onto each layer of E-glass fiber by hand layup method. The processed laminate was left to cure for 21 days.

Mechanical and physical testing

Fracture toughness, in-plane shear, interlaminar shear, and impact tests were investigated on the manufactured specimens. Fracture toughness, in-plane shear, and interlaminar shear were done at ambient temperature on a Jinan universal Test Machine WDW 100 kN.

In-plane shear strength

In-plane shear test was done using an Iosipescu fixture at a 2 mm/min cross-head speed D 5379/D 5379 M – 98 [42]. Coupons were cut with two V-notches at the middle coupon. In-plane shear strength (τ_{xy}) can be considered as:

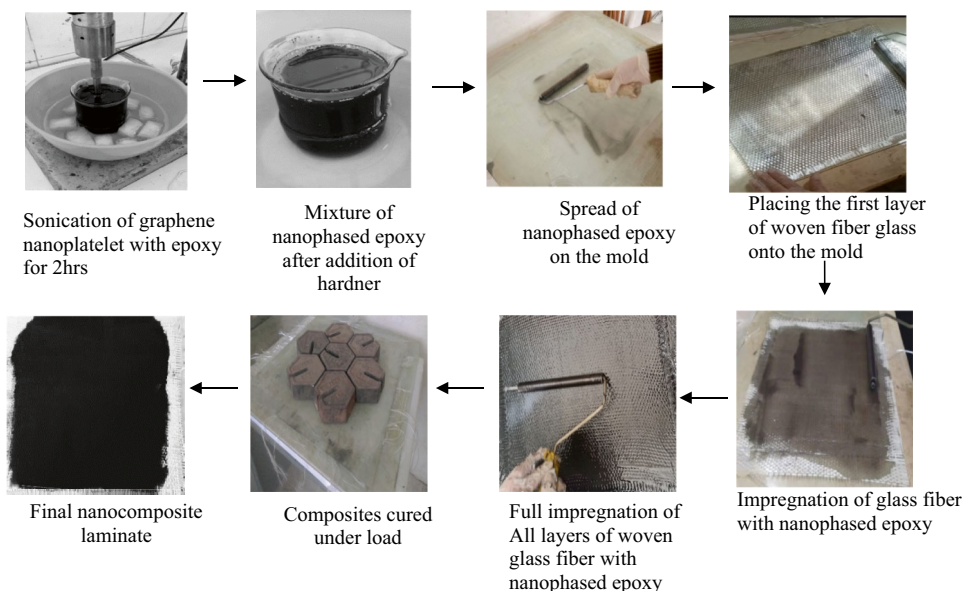
$$\tau_{xy} = \frac{P_1}{v h} \tag{1}$$

where v is the distance between the roots of the V-notches and h is the coupon thickness.

Fracture toughness test

The fracture toughness test (single-edge notch bending (SENB)) was performed according to ASTM D5045-99 [43]. The specimens were tested in the three-point bending tests. The specimens were tested at a displacement rate of 0.05 mm/min. The fracture toughness (K_{IC}) of SENB samples was calculated using the following formulation [44]:

Fig. 1 Manufacturing of nanophased epoxy/glass fiber composites by using the hand layup technique



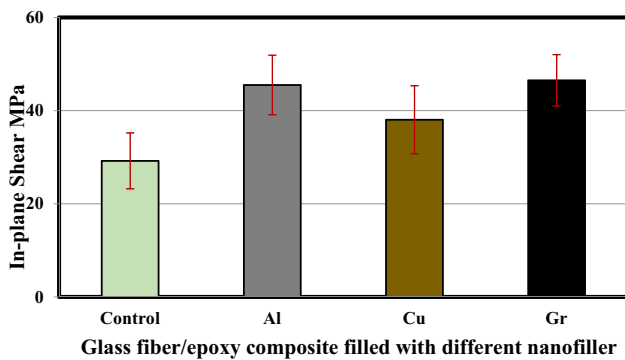


Fig. 2 In-plane shear of nanophased epoxy/glass fiber composites

$$K_{IC} = \frac{P_I}{h\sqrt{w}} f(x) \tag{2}$$

$$f(x) = 6x^{0.5} \frac{[1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2)]}{(1+2x)(1-x)^2} \tag{3}$$

where P_I represents the maximum load, h is the coupon depth, and x denotes to the crack length (a)/coupon width (w).

Impact test

Izod test was performed according to ASTM 256-04 [45]. Flat-wise impact test was conducted on AVERY Denison testing machine. The pendulum of the machine has a falling velocity of 3.65 m/s and an impact energy of 15 J. The impact strength was obtained by dividing the absorbed energy by the original cross-section area of the specimen [3, 46].

Short-beam-shear test

ASTM D2344/D2344M – 00 [47] was used to perform short beam shear test at a rate of 1 mm/min [16, 48, 49]. The apparent interlaminar shear strength (ILSS) was determined by the following formula [50, 51, 52]:

$$ILSS = (0.75P_I)/w h \tag{4}$$

where P_I represents the maximum load, h is the coupon depth, and w denotes to coupon width.

Fig. 3 Fracture surface for specimens after subjecting to in-plane shear for a control, b AL, c Cu, and d Gr

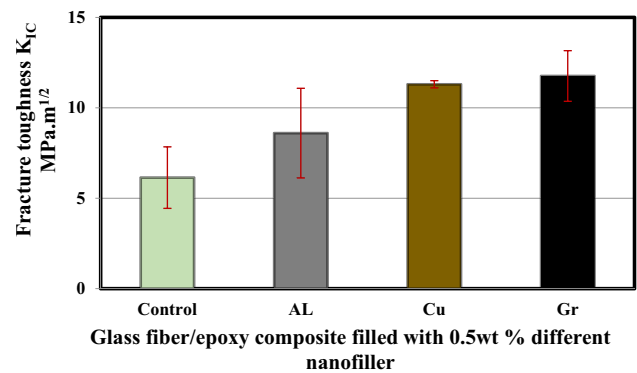
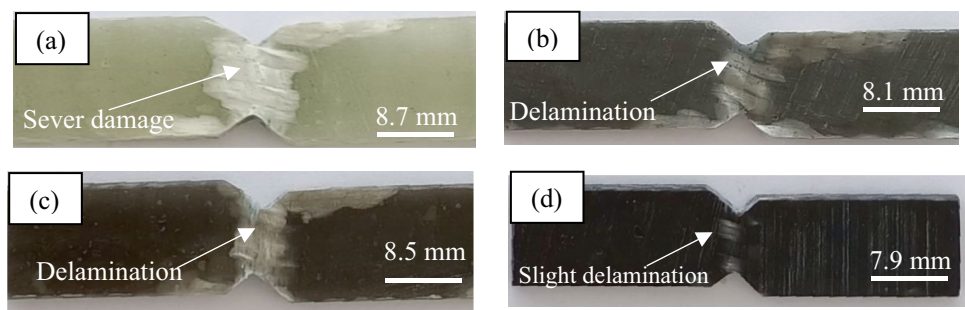


Fig. 4 Fracture toughness of nanophased epoxy/glass fiber composites

Water absorption test

Water absorption test was performed according to ASTM D5229/D5229M-14 [53]. Specimens were immersed in artificial seawater at ambient temperature. The manufactured coupons were periodically drawn from seawater and then wiped to eliminate seawater. The wiped coupons were then weighed to determine the change in weight during adsorption. Coupons were submerged for 85 days in seawater. The seawater absorption percentage $M(t)$ was determined as [54]:

$$M(t) = \left(\frac{w_t - w_0}{w_0} \right) \times 100 \tag{5}$$

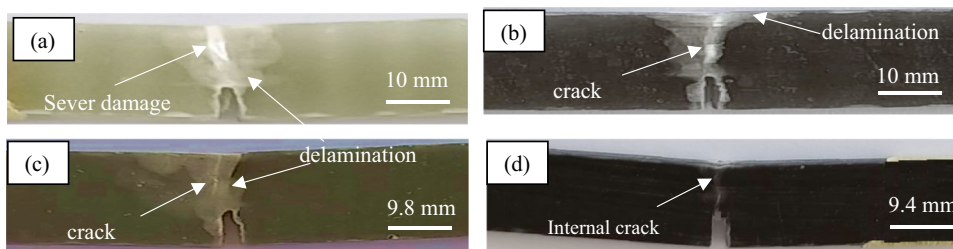
where w_t is the coupon weight after time (t) and w_0 is the initial weight.

Results and discussions

In-plane shear strength

The average in-plane shear strengths of the control specimen and glass fiber/epoxy reinforced by the three different nano-fillers were determined. Glass fiber/epoxy filled with GNP demonstrated the maximum improvement of 59.2% in in-plane shear strength as compared with the control specimen. Moreover, 30.2% and 55.9% increased in in-plane

Fig. 5 Failure of specimens after subjecting to fracture toughness test. **a** Control, **b** AL, **c** Cu, and **d** Gr



shear strength was recorded for glass fiber/epoxy filled with Cu and AL, respectively (as shown in Fig. 2). Figure 3 shows the failure mechanism in the control specimen and specimen reinforced with nano-fillers under in-plane shear load. Severe damage grew at the V-notch roots in the control specimen center. However, moderate delamination at the V-notch roots was attained in glass fiber/epoxy laminates filled with Al nanoparticles and control filled with Cu nanofiller. Graphene nanocomposite laminate is perfectly bonded with slight delamination, indicating improved adhesion compared to the control specimen. Insignificant delamination was detected between the layers of nanocomposites. This revealed the excellent bond between graphene nanofilled epoxy and glass fibers, which improved the in-plane shear resistance compared to control specimens. The addition of nanofiller to polymer offers a more efficient stress transfer that decreases the local stress concentration around the interlayer of fiber/epoxy and enhances the interfacial adhesion, thus, the properties of the manufactured laminates [55].

Fracture toughness

Fracture toughness is an essential material property determining the stress value required to propagate an existing crack [56]. Figure 4 displays the average fracture toughness values of the control and nanofilled glass fiber/epoxy

composite. A 91.5%, 84%, and 40% enhancement in fracture toughness was achieved with glass fiber/epoxy filled with GNP, Cu, and Al nanofillers, respectively, compared with the control composite. A 51.5% and 7.5% enhancement occurred in fracture toughness by adding GNP to glass fiber/epoxy, as compared to Al and Cu nanoparticles. Similarly, the fracture toughness rose to 66% compared to neat epoxy, containing 0.1 weight% GNP. Conversely, when GNP were added, the flexural modulus increased steadily from 0.1 to 2.0. As a result, the applied stress was successfully transferred to the matrix-containing particles, improving the Al₂O₃ nanocomposites' flexural strength, modulus, and fracture toughness by 26.4%, 12.0%, and 24.0%, respectively [57]. Moreover, GLARE loaded with Al₂O₃, SiO₂, Al, and Cu nanoparticles increased fracture toughness by 3.7%, 4.2%, 0.92%, and 2.75% [46]. Gouda et al. [35] developed a micro bamboo filler in an epoxy resin hybrid composite with varied weight percentages of GNP [35]. The existence of nanofiller minimizes the crack propagation and decreases the brittleness character of the hybrid composite material. The interlaminar fracture properties of the developed materials were enhanced by incorporating different types of graphene [15]. In addition, an increasing waviness of graphene can improve mechanical performance by blocking the debonding region at interfaces between graphene and epoxy [33]. Because of the exceptionally high surface

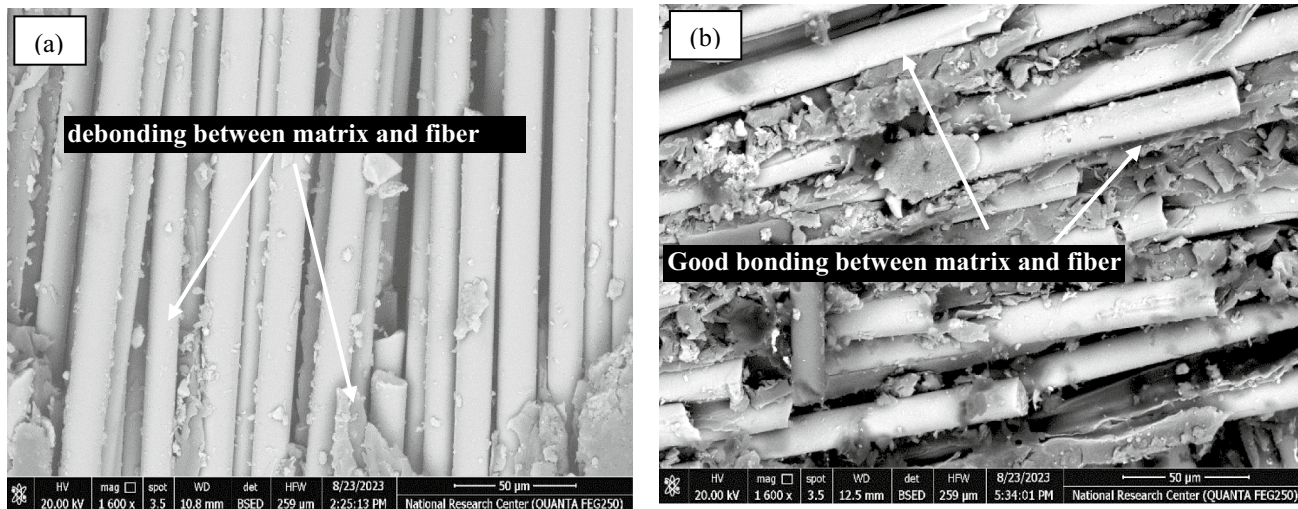


Fig. 6 SEM of the fracture surfaces subjected to fracture toughness test for **a** Control **b** Gr

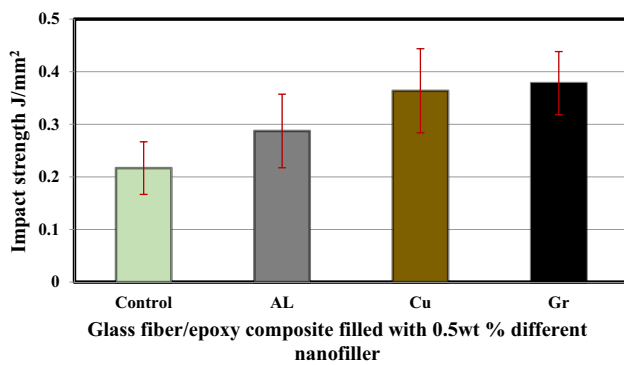


Fig. 7 Impact strength of nanophased epoxy/glass fiber composites

area, as compared to other graphite derivatives, an enormous improvement in properties is observed for graphene nanoplatelet-filled composites [34].

Figure 5 demonstrates the failure mechanism in control and composite laminates incorporating different nanofillers under a fracture toughness test. The control composite has distinct delamination and cracks between the plies of the composite laminate in the higher support region, extending across the thickness of the control specimen. On the other hand, a small amount of delamination and cracking among composite laminate plies developed on the area of the top support through the thickness of the control specimen in glass fiber/epoxy loaded with Al nanoparticles. There is a minor delamination in the laminate layers of the nanocomposite. A good adhesion in the glass fiber/epoxy loaded with graphene nanoparticles suggested a better bonding between the nanocomposite than in the control. Compared to the control, this showed good adhesion between the woven glass fibers and nanophase epoxy, increasing the fracture toughness. Figure 6 displays the SEM pictures of the control and control filled with GNP fracture surface after fracture toughness testing. The control specimen exhibited poor adhesion, leading to a fracture toughness drop. However, the control loaded with GNP showed strong interfacial bonding. The fracture progression path through the nanophase epoxy is oriented by an excellent interfacial connection. Compared to the control specimen, the crack's long traveling route improves the fracture toughness, which uses considerable energy [58]. A similar observation was obtained with Hashim and Jumaha [22]. They attributed their results to incorporating resistant and stiff nanofillers such as

GNP, inducing the formation of fracture mechanisms to deviate the crack propagation. The energy absorbed during the crack propagation led to the construction of several fracture ditches. Furthermore, the improvement in fracture toughness in composites filled with GNP is attributed to the presence of nano-fillers, inhibiting crack propagation and is responsible for the increase of the acquired fracture toughness values. The crack stops upon meeting the nano-fillers and then bifurcates on both sides [15].

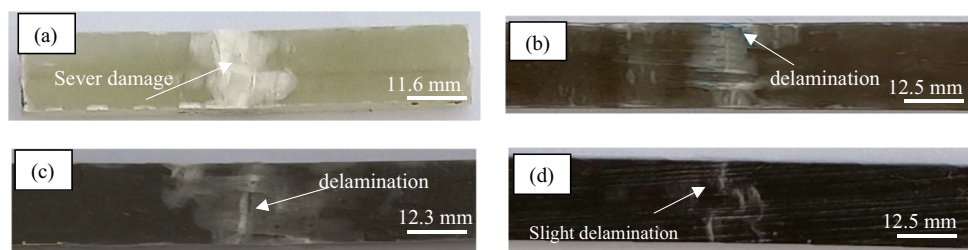
Impact strength

Figure 7 indicates the average impact strength from filling glass fiber/epoxy with different nanofillers. The figure shows an enhancement in the impact strength by incorporating all nanofillers into the polymeric composite material. When glass fiber/epoxy was filled with 0.5 weight% GNP, the impact strength increased to its maximum value. Compared to the control composite, the impact strength increased by 74.7%. The control composites' impact strength is increased with additional nanofillers. Using nanofillers in epoxy matrices prevents cracks from forming and creates a winding channel for them to travel along, increasing impact energy [46]. An enhancement of 32.6% and 67.8% in impact strength was obtained with glass fiber/epoxy loaded with Al and Cu nanoparticles, respectively, compared to the control composite. Similarly, adding three wt% hollow site nanotubes in the nanocomposites increased impact energy values by 119.3% compared with the same weight% of Al nanofillers [2]. The impact resistance relies on the complex energy dissipation mechanisms; thus, the interface properties and the difficult stress concentration of the nanoparticles and milled fibers affect the control of the composite fracture [59].

Under compression or impact of metals, metallic structures buckle and/or fold with significant plastic deformation. However, fiber-reinforced composites fail due to a series of damage mechanisms, including fiber breakage, matrix cracking, fiber-matrix debonding, and delamination, which take place due to their brittleness [60].

Figure 8 shows the failure mechanism in control and nanofilled glass fiber/epoxy composite subjected to impact loading. Severe delamination and cracks occur in the control composite after striking with the hammer. On the contrary,

Fig. 8 Failure of specimens after subjecting to impact test **a** Control, **b** AL, **c** Cu, and **d** Gr



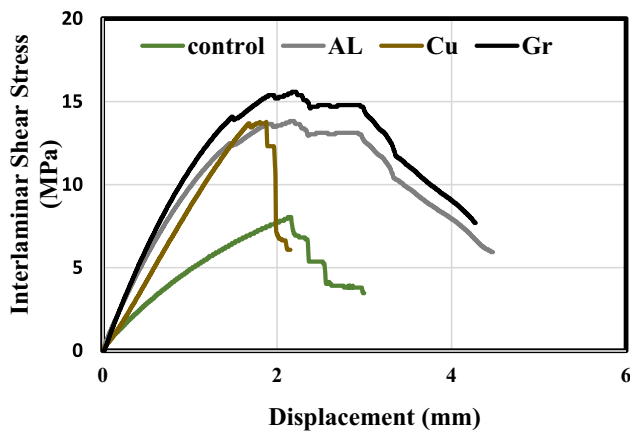
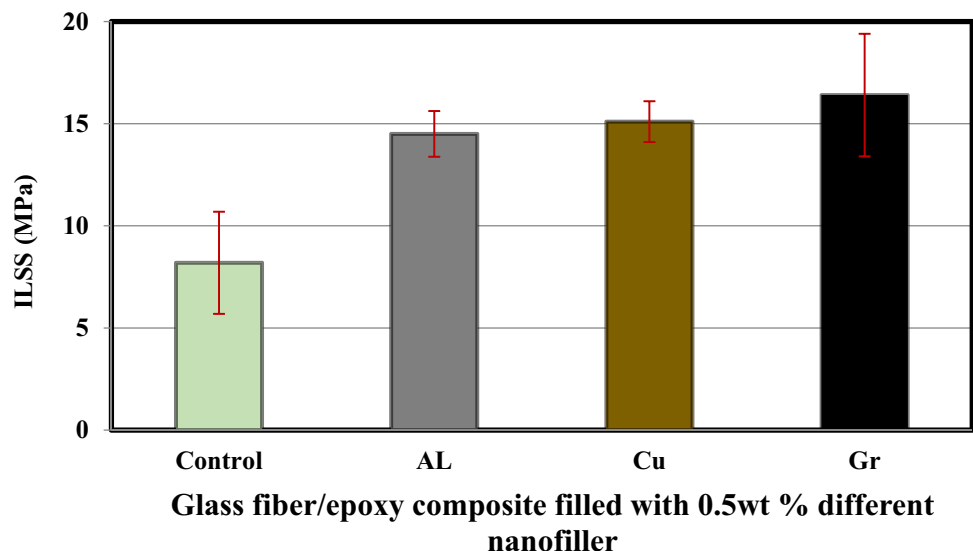


Fig. 9 Interlaminar shear stress-displacement of nanophased epoxy/glass fiber composites

the glass fiber/epoxy filled with GNP sustains good bonding between nanocomposite layers, resulting in crack arresting. This improves the impact resistance of glass fiber/epoxy filled with GNP. Slight delamination was observed with glass fiber/epoxy filled with other nanofillers.

Heaven Dani and Venkateshwaran [61] reported that impact strength improvement with nanocomposites is due to microload-sharing fine nano-silica particles in an epoxy matrix. The presence of nano-silica particles hindered the crack propagation, thus increasing the impactor penetration resistance. In addition, Alam and Chowdhury [62] indicated that the strength of adhesion between the matrix and nanofiller, as well as the interface molecules' flexibility, causes the dispersing of additional energy, thus avoiding the early development of cracks with greater efficiency in composites with CuO filler materials.

Fig. 10 Interlaminar shear strength of nanophased epoxy/glass fiber composites



Interlaminar shear strength

Figure 9 indicates the interlaminar shear stress with displacement of control and nanofilled glass fiber/epoxy composite exposed to a three-point bending force in a short beam test. The interlaminar shear stress descends when the displacement increases. The glass fiber/epoxy filled with GNP achieved the highest peak interlaminar shear stress among other nanocomposites, followed by glass fiber/epoxy filled with Al nanoparticles. A pronounced increase in interlaminar shear stress was observed with glass fiber/epoxy filled with Cu nanofillers. In comparison to the control specimen, it showed less displacement. Figure 10 indicates the ILSS of the control specimen and nanocomposites. The highest ILSS value was obtained for the control specimen filled with a GNP nanofiller with an enhancement of 100%. A 77.1% and 84.4% enhancement in ILSS was presented by glass fiber/epoxy loaded with Al and Cu nanofillers, as compared to the control specimen. This could be explained by adding nanoparticles to epoxy to enhance the adhesion between the glass fibers and the epoxy that contained nanofillers.

Figure 11 shows the failure of specimens after being subjected to interlaminar shear tests of control and nanocomposite laminates. Short span length in the interlaminar shear test under a three-point flexural load designates the interfacial bonding strength of the manufactured plate. However, Al nanofillers, Cu nanofillers, and GNP in the epoxy matrix enhanced the ILSS of glass fiber/epoxy composites. Nanomodification of the epoxy led to improved interfacial bonding between the nanophase epoxy and woven glass fibers in addition to shear stresses between the layers showing relatively weak bonding at the interface, debonding between the composite layers causes failure in the control laminate.

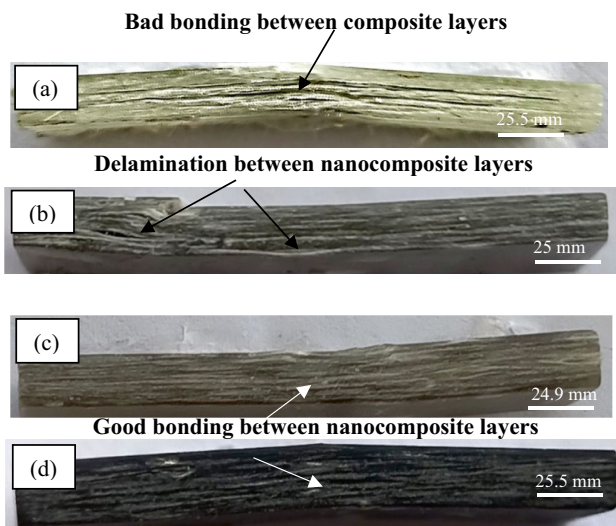


Fig. 11 Failure of specimens after subjecting to interlaminar shear test. **a** Control, **b** AL, **c** Cu, and **d** Gr

Water absorption

Figure 12 shows the water absorption of glass fiber/epoxy composites reinforced with nanofiller under seawater conditions. The figure indicated that the curve exhibited two regions. The first region represented the high rate of water uptake, with a second zone signifying the sluggish rate of water absorption coming next. The water uptake percentage increased as the process by which water molecules diffuse into microcracks and defects created between glass fiber and polymeric matrices and are absorbed by the polymer chains in polymeric composites is known as water uptake [63]. Through the interaction of the polar molecules of the absorbed saltwater, plasticization happened as the seawater infiltrated into the epoxy matrix. Molecules of absorbed seawater push polymer chains apart. Consequently, the free volume rises [64, 65]. The figure shows that incorporating 0.5 wt% of nanofiller to epoxy reduced the water uptake percentage in seawater compared to control. This might be because, compared to

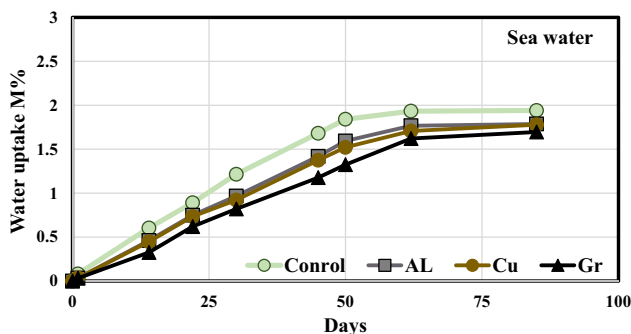


Fig. 12 Water absorption of nanophased epoxy/glass fiber composites

the control specimen, the nano-sized inclusion resulted in a lower rate of saltwater absorption. These plasticized composites loaded with nanofillers behaved better because of their barrier qualities [66]. In addition, the presence of nanofillers reduced seawater permeability in nanocomposites by delaying the relaxation of the epoxy chains surrounding the nanofiller. Because of nanofillers' hydrophobicity and epoxy resin's hydrophilic nature, incorporating nanofillers into glass fiber/epoxy composites decreased seawater uptake.

It was suggested that the high aspect ratio of the nanofiller, which forms winding routes for the water molecules, was responsible for improving seawater uptake resistance [67]. By blocking the holes inside the epoxy matrix, nanofillers increase the density of epoxy cross-linking, decrease free volume, and allow for interconnection with molecular chains [54]. Nanofillers can create free volume in the polymeric matrix, which occurs when these nanofillers start to agglomerate due to a high specific interfacial area (fillers area per composite volume) where water may be collected. Thus, water molecules penetrate the epoxy easily without restriction [68]. The epoxy chain's segmental mobility was reduced by the nanofiller's consistent dispersion throughout the epoxy matrix [69]. Previous works reported similar results [70–72].

Conclusions

This work carried out an experimental investigation regarding the in-plane shear, barrier behavior, fracture toughness, impact, and interlaminar shear strength of control and different nanocomposites. Various nanofillers were added to the glass fiber/epoxy composite with 0.5wt% of epoxy. Nanocomposites, including Al, Cu, and GNP, were prepared using the hand layup technique into a glass fiber/epoxy composite. The addition of all nanofillers positively affected the barrier behavior, in-plane shear, fracture toughness, impact, and interlaminar shear strength properties compared to the control specimen. At 0.5 weight%, GNP produced the best results. Compared to the control composite, glass fiber/epoxy loaded with graphene nanoplatelet, Cu, and Al nanoparticles increased fracture toughness by 91.5%, 84%, and 40%. Nonetheless, glass fiber/epoxy loaded with Al and Cu nanoparticles improved impact strength by 32.6% and 67.8%, respectively.

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Data availability The datasets used during the current study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest The authors declare no conflict of interest.

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