Materials
Science

AIMS Materials Science, 10(4): 693–709.
DOI: 10.3934/matersci.2023038
Received: 12 April 2023
Revised: 16 July 2023
Accepted: 30 July 2023
Published: 17 August 2023

http://www.aimspress.com/journal/Materials

Research article

Mechanical properties of graphene nanoplatelets reinforced glass/epoxy composites manufactured using resin film infusion process

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Abstract: Nanofillers as secondary reinforcement in polymeric composites have shown promising results on improvement of mechanical properties of the polymeric composites. However, due to the in-plane resin path, filtration of the nanofillers through fabric during processing is a major challenge in liquid infusion processes such as resin transfer molding. In resin film infusion process, the resin travels in thickness direction and due the shorter resin path, the possibility of filtration is minimal. In this study, resin film infusion (RFI) process is used to fabricate graphene platelets reinforced glass/epoxy hybrid composites. First, the resin films with 0.4 and 0.8 percent of graphene nanoplatelets (GNPs) were manufactured using solvent casting process. Then, these films were used to fabricate composites using resin film infusion process. Mechanical characterization tests, namely tensile, flexural and short beam, were performed. Tensile strength of the 0.4% and 0.8% GNPs reinforced composites was 36.13% and 22.23% higher, respectively, than that of baseline composites. Flexural strength 0.4% and 0.8% GNPs modified composites was 24.96% higher and 32% lower, respectively, compared to baseline composite. Lastly, minimal change was observed in short beam shear strength due to GNPs reinforcement. Higher void volume fraction of 1.7% and 4.5% as compared to baseline composites observed in 0.4% and 0.8% GNPs modified composites respectively.

Keywords: resin film infusion; graphene nanoplatelets; mechanical properties; UD glass fabric

1. Introduction

Fiber reinforced composites, due to their superior mechanical properties and high strength to weight ratio, have huge potential to replace conventional engineering materials in a variety of structural applications [1]. However, these composites are weak in the thickness direction i.e., in out-of-plane direction, due to the matrix dominated inter laminar region [2]. In recent years, nanofillers have been extensively used to reinforce the polymeric matrix with an aim to improve the mechanical properties of resin, thereby improving overall performance of these materials [1].

Recent research shows that incorporating small quantities of carbon-based nanomaterials in polymers improves their mechanical and electrical properties. This is because nanofillers have a high surface-to-volume ratio, which increases interfacial interactions between the nanofillers and the matrix [1]. The widely used nanofillers are carbon nano-tubes (CNTs) [3], carbon nano-fibers (CNFs) [4], graphene nanoplatelets (GNPs) [3,5] and graphene nano-sheets (GNSs) [6]. Among the resins, the effect of nanomaterials reinforcement on epoxy resin is widely researched. Due to the high cross-linking density, epoxy is inherently brittle [7]. Therefore, it is prone to microcracking. The use of nanofillers has shown improvement in toughness characteristics without any negative effects on processing or mechanical properties [8–10].

Graphene is a 2D nanomaterial that consists of carbon atoms arranged in a hexagonal pattern. This pattern forms a single layer of sp^2-bonded carbon atoms that can be isolated from graphite. Recent research has shown that a small number of layers of graphene stacked on top of each other, like partially exfoliated graphite, can be effectively utilized as a filler in polymer matrices. Nowadays, GNPs are obtainable in the market at a substantially reduced cost when compared to single-layer graphene and have the potential to improve the properties of polymer matrices [6,11]. The mechanical performance of GNPs embedded in an epoxy matrix was reported to be superior to that of CNTs by several researchers, particularly in the aspects of Young's modulus, tensile strength and buckling resistance [10,12].

Liquid infusion methods such as resin transfer molding (RTM) or vacuum assisted resin transfer molding (VARTM) [13,14] are the commonly used methods of researchers to manufacture nanofillers reinforced composites. In both methods, resin is infused into a plain fabric or fiber preforms along the length of the fabric. This fabric acts as a mesh membrane and may filter out the nano fillers [14,15]. The amount of filtration of nano fillers during the infusion depends on the size of nanofiller, distance travelled by resin and fiber volume fractions [16]. However, these shortcomings can be overcome by using resin film infusion (RFI) method to manufacture the nanofiller reinforced composites. In RFI method, b-staged epoxy resin films are interleaved with dry fabric and the layup is then cured under the suitable pressure and temperature conditions [17]. During the curing process, the resin flows across the fabric in thickness direction and thus nanofillers get evenly dispersed in the composite [18]. Also, the b-staged resin is high viscosity resin which reduces the mobility of nano fillers inside the resin [18,19] and this further helps in preventing the agglomeration of the nanofillers [20].

In this experimental study GNPs are used to evaluate the effect of addition of GNPs into the epoxy matrix. The GNPs were dispersed into epoxy using acetone as solvent and tip sonication. The solvent was removed in controlled condition. This GNPs modified epoxy matrix is used to manufacture hybrid composite with unidirectional glass fibers as primary reinforcement. The composite manufacturing method used is RFI. Effects of GNPs percentage on mechanical properties of composite were studied.

2. Materials and methods

2.1. Materials

The two-part resin used in this experimental study was a tetra-functional high viscosity resin supplied by Fine Finish Organics Pvt. Ltd., Mumbai. Epofine®-720 is tetraglycidyl ether of methylene dianiline as resin, and FINEHARD™-976 is an aromatic amine hardener (diamino diphenyl sulfone) was used. Unidirectional (UD) E-glass fiber fabric (900 gsm) was used as reinforcement to manufacture glass fiber reinforced polymer composite (GFRP) panels. Amine graphene nanoplatelets were used as fillers to the matrix. Amine graphene nanoplatelets were supplied by Ad-Nano Technologies, Shimoga, Karnataka, India. Many researchers [21–23] have proved that amine functionalization of GNPs improves its dispersion in epoxy resin. Therefore, amine modified GNPs was preferred in this research instead of bare GNPs.

2.2. Preparation of resin films

The dispersion of graphene nanoplatelets in epoxy is a crucial step in the processing of nanomodified resin films. It has been extensively documented in literature that graphene nanoplatelets (GNPs) can be dispersed in various solvents like ethanol [24–26], acetone [27–29], tetrahydrofuran [30], dichloromethane [31], etc. Solvents like acetone and tetrahydrofuran exhibit favorable adsorption energy for dispersion of GNPs [32]. Additionally, these solvents provide a low-viscosity medium for dispersing nano fillers like graphene nanoplatelets. Furthermore, it is observed that the decrease in viscosity improves the separation of graphene sheets [33].

The process of preparation of film of GNPs reinforced epoxy is described below in a step-by-step manner and is shown schematically in Figure 1.

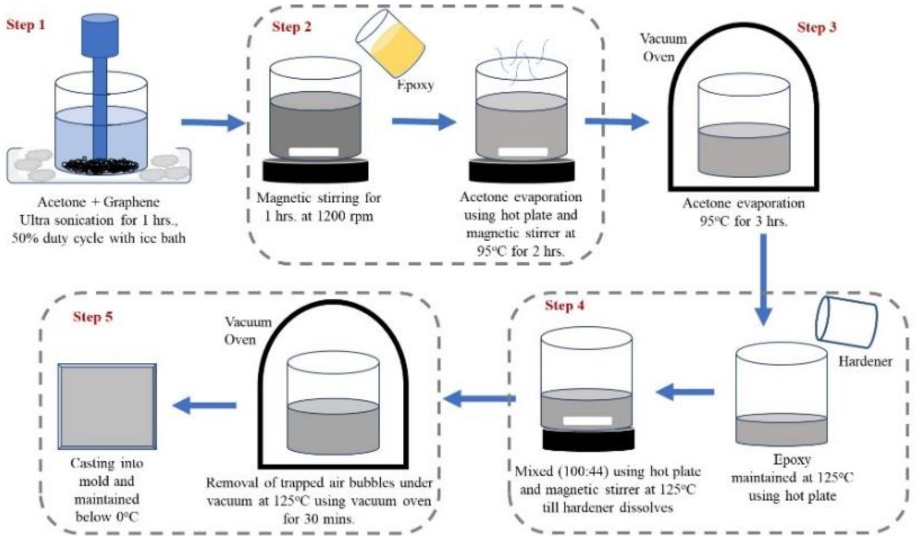

Figure 1. Preparation of GNPs dispersed resin films.

Step 1: The GNPs were dispersed using an ultra-sonicator in acetone (C_3H_6O) for 60 minutes. The process temperature was maintained below 20 °C with an ice bath.

Step 2: The epoxy resin was added to the mixture of GNPs and acetone and mixed manually for few minutes and then the mix was magnetically stirred on a hot plate kept at 95 ℃ until all the acetone was evaporated.

Step 3: The mix was then kept under vacuum pressure for 60 minutes at 95 ℃ to ensure the complete removal of acetone and bubbles.

Step 4: After the removal of acetone, hardener (diamino diphenyl sulfone), which was in powder form, was added to the mix of resin at 125 ℃ for 60 minutes with magnetic stirring. The ratio of resin to hardener was 100: 44, as suggested by the manufacturer.

Step 5: This resin/filler mix was then poured into molds. The mixture was allowed to cool to room temperature. The molds with resin/filler mix were kept in a refrigerator so that the resin solidified and could be used as resin films.

2.3. Fabrication of glass fiber reinforced polymer composite

The required size of glass fabric plies and peel ply were cut and stacked along with resin films. The sequence of stacking is shown in Figure 2.

Partially cured epoxy resin (B-staged) films were interleaved between unidirectional glass fiber fabric as shown in Figure 2.

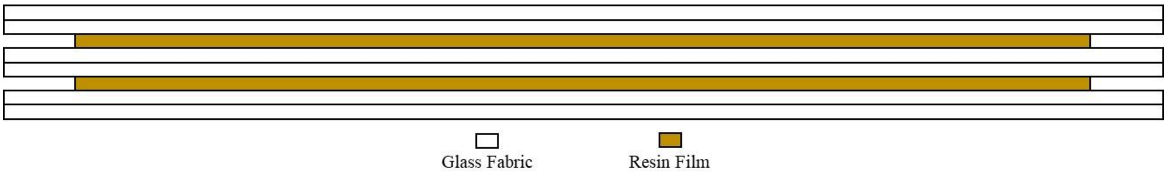

Figure 2. Stacking sequence of fabric and resin film.

The layup was then compressed between two thick glass plates by putting weight on top of the plate. This setup was then kept in an air oven to allow the curing of the composite. The cure cycle adopted for the curing process is 100 ℃ for 1 h followed by 120 ℃ for 1 h and then 150 ℃ for 5 h. Three glass fiber reinforced polymer composite panels were fabricated with 0, 0.4 and 0.8 wt% loading of GNPs in the epoxy matrix. The average thickness of the three panels namely 0, 0.4 and 0.8 wt% loading of GNPs as measured using micrometer screw gauge was 5.00, 5.20 and 5.13 mm respectively. The specimens were cut using waterjet machining from these panels for tensile, flexural and short beam shear strength as per the respective American Society for Testing and Materials (ASTM) standards.

3. Characterization of mechanical properties

In order to understand the effect of GNPs reinforcement on the mechanical properties of composites, tensile, flexural and short beam strength of the composite panels was studied as per ASTM standards. Minimum five specimens were tested to determine the properties. The morphological characterization of the GNPs used in this research, was carried out using TEM (transmission electron microscopy).

3.1. Fiber volume fraction

The fiber volume fraction of the composite specimen was analyzed using the ASTM D-792 [34] and ASTM D-3171 [35] standard. The ASTM D-3171 standard suggests several procedures to remove the resin matrix from the fibers. The matrix burn-off in muffle furnace procedure suggested in standard was used in this work. Three specimens each were tested. Specimen and desiccated crucible were weighed before burning the matrix.

Following formulae were used to calculate the volume fractions.

Reinforcement content, weight percent

$$W_r = \left(\frac{M_f}{M_i}\right) \times 100$$

Reinforcement content, volume percent

$$V_r = \left(\frac{M_f}{M_i}\right) \times 100 \times \left(\frac{\rho_c}{\rho_r}\right)$$

Matrix content, weight percent

$$W_m = \frac{\left(M_i - M_f\right)}{M_i} \times 100$$

Matrix content, volume percent

$$V_m = \frac{\left(M_i - M_f\right)}{M_i} \times 100 \times \left(\frac{\rho_c}{\rho_m}\right)$$

Void volume

$$V_v = 100 - (V_r + V_m)$$

where M_i is initial mass of the specimen in grams, M_f is final mass of the specimen after combustion in grams, ρ_r is density of the reinforcement g/cm^3, ρ_c is density of the specimen g/cm^3 and ρ_m is density of the matrix g/cm^3.

The heating cycle as shown in Figure 3 was used for burning off the resin. After cooling, the burnt specimens were weighed with crucible.

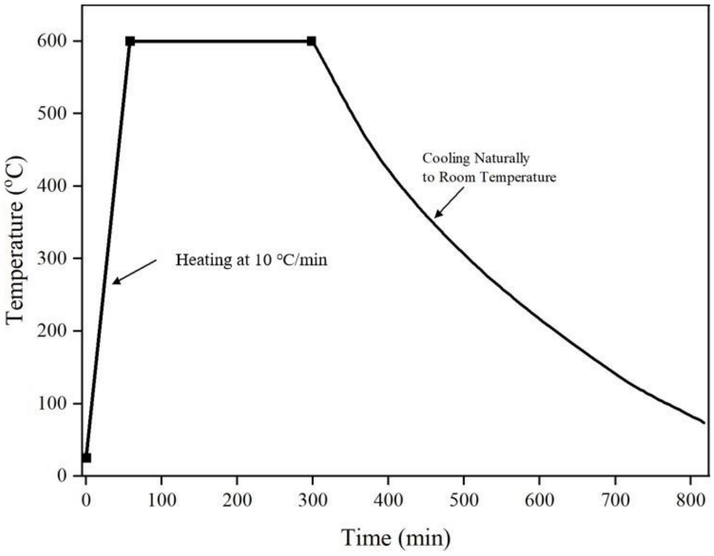

Figure 3. Temperature profile with respect to time of muffle furnace.

3.2. Tensile test

The tensile tests were carried out on MTS universal testing machine with a load cell of 100 kN as per ASTM D3039 [36] standard. The details of the specimen are shown in Figure 4. FR4 copper clad plate laminate single-side PCB was used as tabs to improve grip and support during testing. Cyanoacrylate based adhesive (Loctite 415) was used to bond the tabs with the specimens. Wedge grips were used to hold the specimen firmly during the test. The test was conducted at a constant head speed of 2.0 mm/min.

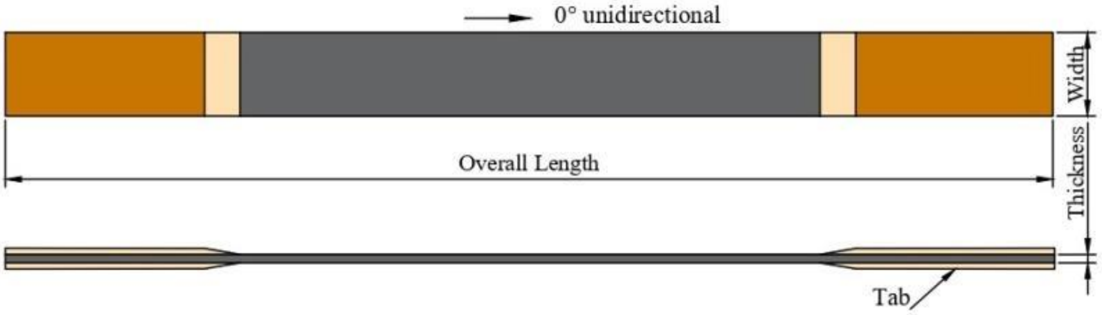

Figure 4. Specimen for tensile test.

3.3. Three point bending test

Three-point loading configuration as mentioned in ASTM D7264 [37] was used to determine flexural properties of the composite test specimen. The schematic of 3-point bending test is shown in Figure 5. The span-to-thickness ratio 32: 1 as recommended in ASTM standard is used. The radius of loading nose and supports was 5.0 mm. The flexural test was conducted at a constant speed of 1.0 mm/min.

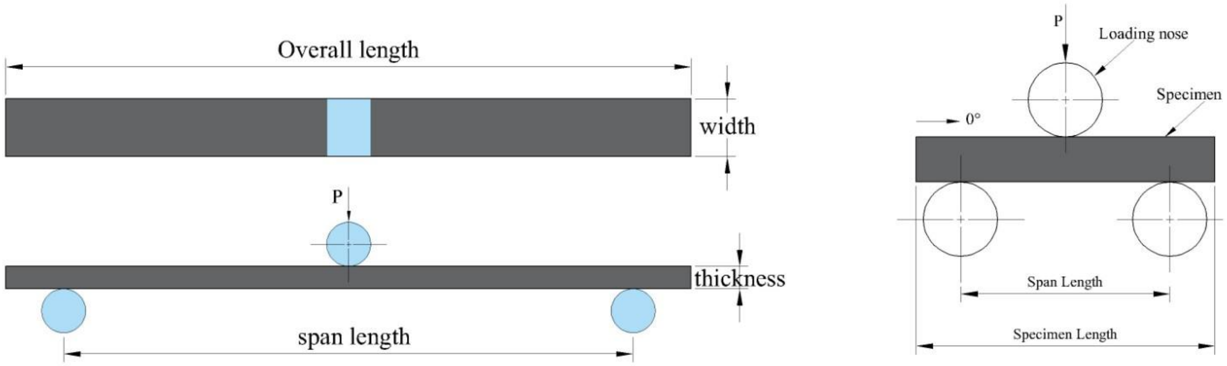

Figure 5. Specimen for flexural and short beam test.

3.4. Short beam strength

Short beam strength was determined using ASTM D2344 [38]. As per the guidelines of the standard, length of the specimen is six times the thickness and width are twice the thickness. At both ends, the overhang was equal to the thickness of the specimen. The rate of crosshead movement for testing was 1.0 mm/min. The short beam strength (SBS) was calculated by

$$SBS = 0.75 \times \frac{F_{max}}{h \times b}$$

where SBS is short-beam strength in MPa, F_{max} is load (maximum) during the test in N, h is specimen thickness in mm and b is specimen width in mm.

4. Results and discussion

The results of effect of addition of 0.4% and 0.8% GNPs to epoxy (matrix) on mechanical properties of the composite is presented in this section.

4.1. Transmission electron microscopy (TEM)

The morphological characterization of the GNPs was first carried out using TEM and is shown in Figure 6. The graphene is multilayer (10–15 layers) and has sharp corners and are clearly visible. The selected area electron diffraction (SAED) confirms the crystallinity of the graphene nanoplatelets. As per the literature, single sheet of graphene shows a hexagonal SAED pattern as it is crystalline in nature [39]. Whereas multilayer graphene shows a rotationally misaligned hexagons which in turn looks like a circle and confirms its polycrystalline nature [40]. Thus, SAED confirms that graphene is multilayer and thus can be referred as graphene nanoplatelets.

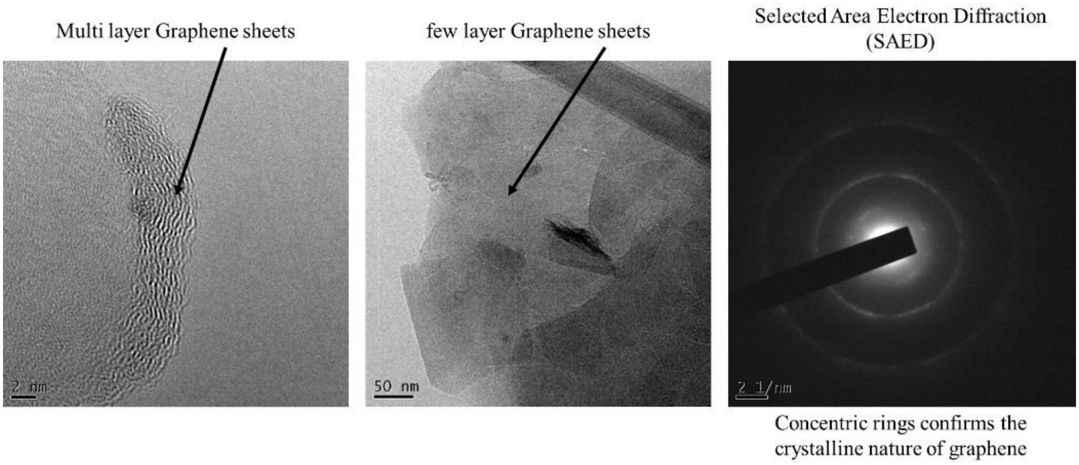

Figure 6. TEM of amine graphene nanoplatelets.

4.2. Volume fraction and void content of constituent of composite

While working with high viscosity resin like epoxy, it is very difficult to remove all the voids and air bubble from the resin [41]. Voids are defect in materials, and thus affect the properties and performance of composites. The void content of composites of baseline, 0.4% and 0.8% GNPs composite panels are shown in the Figure 7. The fiber volume fraction in three panels is almost same in all the three panels. The void fraction is 2.66%, 4.35% and 7.15% respectively in baseline and 0.4% and 0.8% GNPs composite panels.

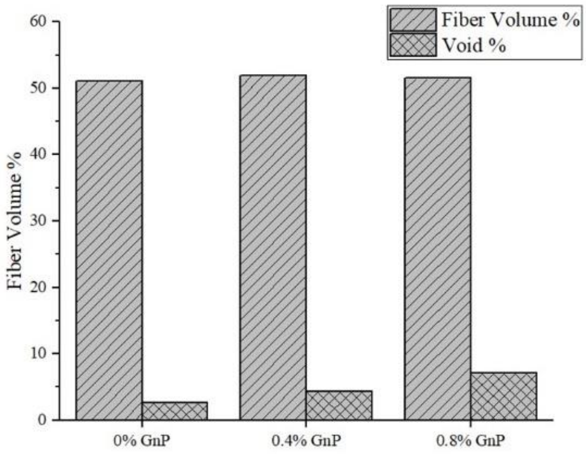

Figure 7. Volume fraction and void content.

The reasons for the increase of void percentage at higher loading of GNPs could be attributed to three main reasons as per the published research. First, due to adsorption of GNPs onto the epoxy resin molecules, there is possibility of increase in viscosity of resin [42]. The higher viscosity resin does not flow easily and therefore void pockets are created. Second, GNPs can block the escape of air bubbles from the resin during the degassing process [43]. These trapped air bubbles then turn into void in the composites. Lastly, the functional group on GNPs with oxygen attached to it, is decomposed at high temperature and can creates voids. The void percentage observed in this work is consistent with work published by others [44].

4.3. Microscopy

Figure 8 shows the morphology of fractured surface of baseline, 0.4% and 0.8% GNPs composite panels. Figure 8a–c shows the SEM micrographs of failed specimens of composite panels with 0%, 0.4% and 0.8% GNPs reinforcement respectively. The higher surface roughness owing to the presence of residue of matrix suggests improvement in adhesion of matrix and fibers [44] in case of GNPs reinforced composites. The magnified view of GNPs reinforced composites (Figure 8d) clearly shows that matrix adheres to the fibers better due to addition of GNPs. The improved fiber-matrix adhesion could be the possible reason for improvement of the material properties of GNPs reinforced composites [44]. Figure 8b,c also show uniform dispersion of GNPs in the composite materials. The presence of GNPs may have resisted crack propagation [45] resulting in improved mechanical properties of the GNPs reinforced composites. Figure 8c shows agglomeration of GNPs in few places, which may have acted as a source of crack propagation [46] causing lower mechanical properties of 0.8% GNPs reinforced composites as compared to that of the 0.4%.

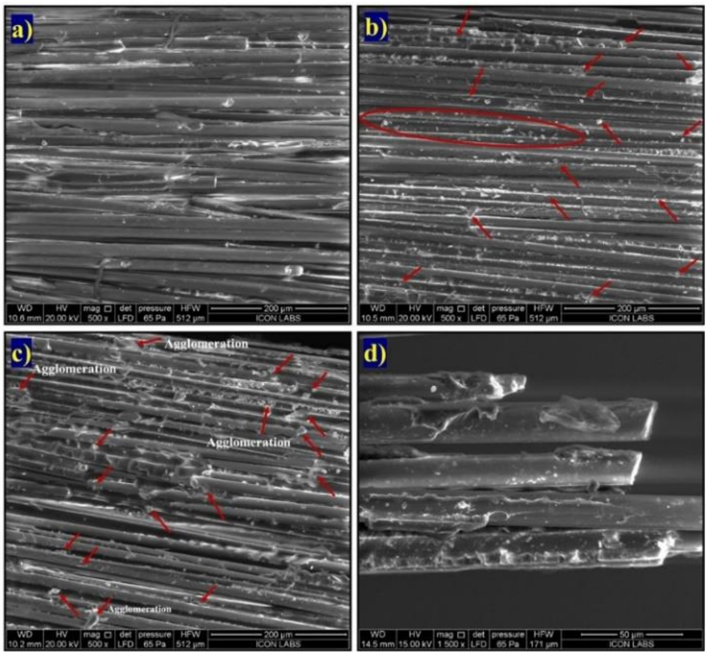

Figure 8. SEM micrograph of fractured surface (a) 0% GNPs, (b) 0.4% GNPs, (c) 0.8% GNPs, (d) 0.4% GNPs fractured fibers.

4.4. Tensile failure analysis

The tensile test was conducted in accordance with ASTM D-3039. The test coupons were cut such that orientation of fibers is along the length of coupons. Most of the specimen showed explosive gage middle (XGM) type failure mode as described in ASTM 3039 [36]. This XGM (broom-like) failure is very common in unidirectional glass fiber polymer composite (GFRP) because of the de-bonding of fibers from the matrix resulting in spitting the specimen along the length [47,48].

Figure 9 shows the load vs displacement curves for one the coupons of 0%, 0.4% and 0.8% GNPs composites. It can be observed that, addition of GNPs improves the tensile properties of the composite. The improvement in tensile strength for 0.4% and 0.8% GNPs composite 36.13% and 22.3% respectively.

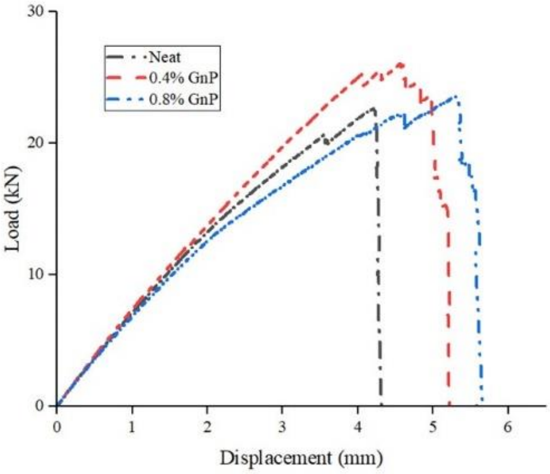

Figure 9. Load vs displacement.

As per the results shown in Figure 10, it is evident that reinforcement of GNPs increases tensile strength of glass/epoxy composite. However, the improvement in tensile strength of 0.8% GNPs composite was lower than that of the 0.4% GNPs composite. This trend may be because of agglomeration of GNPs and increase in void volume percentage at higher loading of GNPs. Rafiee et al. [49] also observed decrease in mechanical properties at higher nanofiller weight percentage [50]. Additionally, it is an established fact that when high surface area GNPs are coated on the fiber surface, it leads to improved interfacial adhesion between fiber and matrix, which consequently result in improvement in tensile strength [46,51].

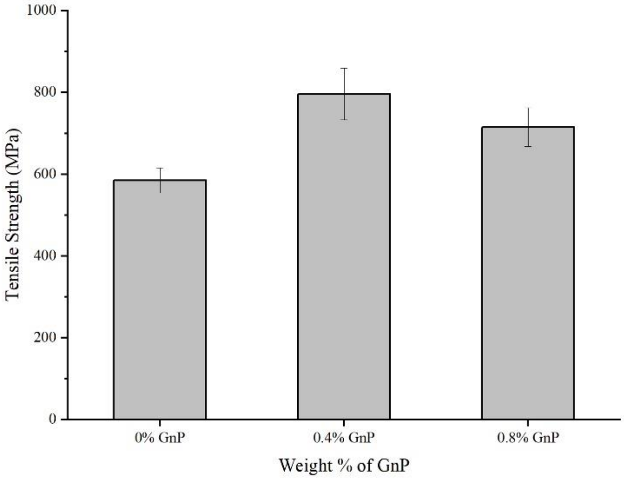

Figure 10. Tensile strength of composite.

Reinforcement of epoxy with 1% GNPs by weight fraction could not be achieved during this research due to the very high increase in viscosity of epoxy resin. Adsorption of nano filler [42] onto the resin molecules could be the reason for increase in viscosity.

Composite with 0.4% GNPs shows 8% increase in the tensile modulus and composite with 0.8% GNPs shows 28% increase in tensile modulus compared to neat epoxy/glass fiber composite.

4.5. Flexural test analysis

The flexural properties were determined as per ASTM D7264. The behavior of composite material under flexural test (3-point) is shown in Figure 11.

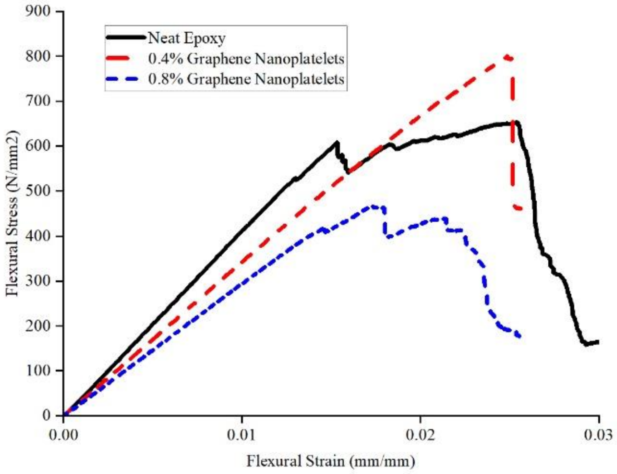

Figure 11. Flexural stress vs strain.

The addition of graphene to the epoxy has improved flexural strength and it can be seen from Figure 12.

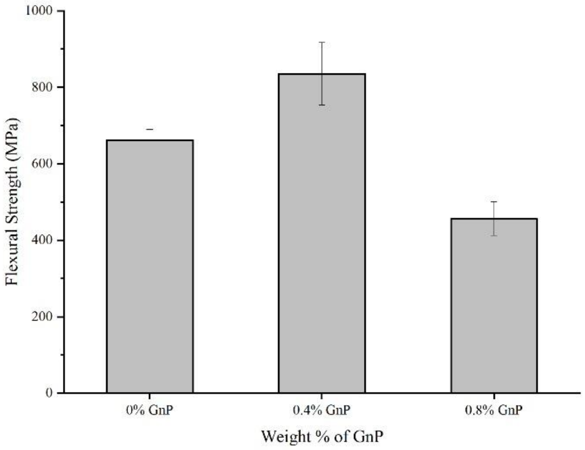

Figure 12. Flexural strength vs GNPs weight%.

Improvement of 24.96% and reduction of 32% was observed for GNPs 0.4% and 0.8% GNPs composites respectively as compared to baseline composite. Matrix reinforcement with GNPs may have led to effective load transfer among the fibers [52,53], resulting in improved flexural strength in 0.4 % GNPs composites. However, agglomeration of GNPs could be the reason for deterioration of flexural strength in 0.8% GNPs composites. To conclude, the GNP's loading percentage is very critical factor in deciding the performance of the composite.

4.6. Short beam strength (inter-laminar shear strength)

The inter-laminar shear strength (ILSS) properties were determined as per ASTM D-2344. As can be observed from Figure 13, not much change was observed in ILSS of composites with 0.4% and 0.8% GNPs as compared to the baseline composites. Minimal improvement of about 3% in ILSS was observed in 0.4% GNPs composites, whereas 0.8% GNPs loading did not show any change in ILSS of the composite.

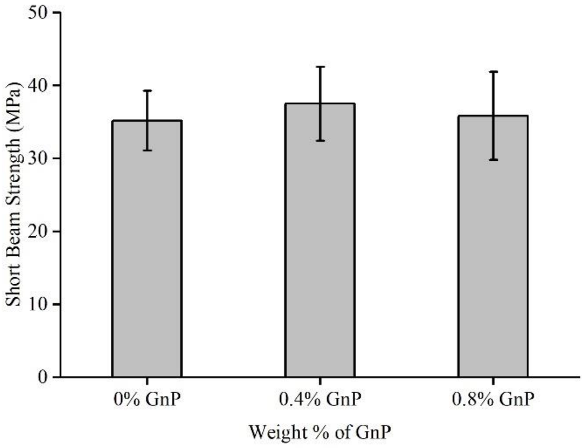

Figure 13. Short beam strength vs GNPs weight%.

4.7. Improvement due GNPs reinforcement vs degradation due to voids

In fiber reinforced composites, the role of matrix is to bind the fibers together and to transfer the load among the fibers. Voids act as barrier to load transfer among the fibers, resulting in degradation of performance of composite. As per the void content analysis presented earlier, it is observed that void fraction is higher in 0.4% and 0.8% GNP composites as compared to baseline composite. It is established through research that the increase in voids fraction causes degradation in mechanical properties of the composites. Whereas, many researchers have observed improvement in mechanical properties of composites due to addition of GNP. Therefore, in this research, an attempt is made to understand synergetic effect of degradation due to increased voids fraction and improvement due to GNP reinforcement on mechanical properties of the composites. Hagstrand et.al [54] suggested simple equations mentioned below for predicting the degradation of tensile strength due to increase in void fraction

$$\sigma_{void} = \sigma_0(1 - V_{void})$$

where E_{void} is elastic modulus of composite considering the effect of voids, E_0 is elastic modulus of composite without any voids, σ_{void} is tensile strength of composite considering the effect of voids, σ_0 is tensile strength of composite without any voids and V_{void} is voids fraction in the composite specimen.

Figure 14 shows the comparison of experimental tensile strength of the composite with varying GNPs content and corresponding predicted tensile strength using the above equation. Considering the effect of degradation due to void, we can see there is effective improvement of tensile strength by 36.13% and 22.23% for 0.4% and 0.8% composites respectively.

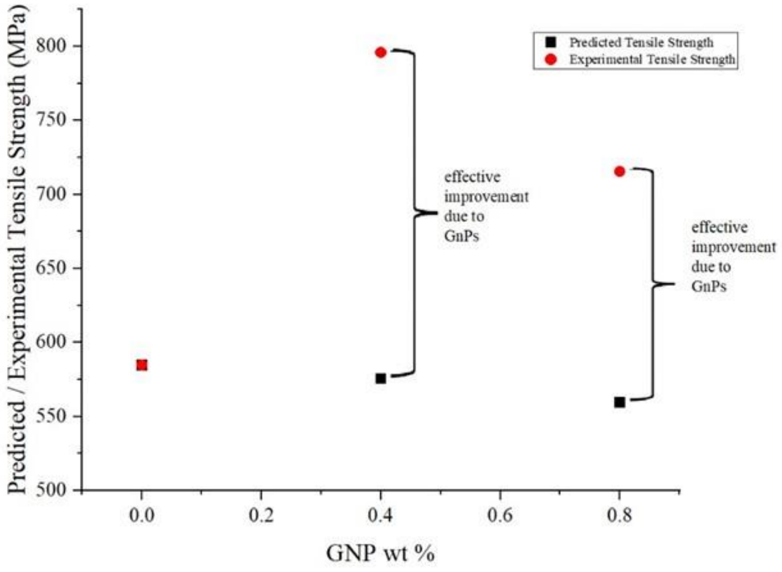

Figure 14. Comparison of predicted tensile strength vs experimental tensile strength.

5. Conclusions

In this work, GNPs reinforced glass/epoxy multiscale composites were manufactured using RFI process. It is observed that RFI process is suitable for development of GNPs reinforced multi-scale composites due to its ability to give uniform dispersion of GNPs. Overall, the results of the experimental characterization showed improvement in the mechanical properties due to GNPs reinforcement. The tensile strength and flexural strength have improved at low GNPs (0.4%) loading weight percentage with 36% and 25% respectively. As the GNPs loading weight percentage increases, the properties do not show improvement. The effect of GNPs was not seen on the short beam strength. Also, other popular composite fabrication processes like VARTM, which filters out nanofillers, and RFI have shown positive results. Thus, low GNPs loading weight percentage can improve the properties of composites without considerable increase in the weight of composite.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

Acknowledgments

Authors are thankful to Technical Education Quality Improvement Programme (TEQIP-III) at Veermata Jijabai Technological Institute (VJTI), Mumbai, India for providing financial assistant to the author. Also, authors are thankful to IIT-Bombay for allowing the use of facilities at Sophisticated Analytical Instrument Facility (SAIF) and Advanced Mechanical Testing Facility (AMTF) lab.

Conflict of interest

The authors declare no conflict of interest.

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