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Experimental investigation on flexural fatigue strength of graphene oxide modified E-glass epoxy composite beam

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ABSTRACT

Many studies are currently being performed on the complex issue of fatigue of reinforced composite materials. Graphene oxide (GO) is a strong contender for reinforcement, an atomically thin form of carbon with remarkable multifunctional qualities and a perfect surface for interacting with polymer matrices. This report investigates the effect of GO modified epoxy resin on the flexural fatigue life of a composite beam. First, two different weight concentrations of 0.25 and 0.50 % are dispersed by ultrasonication in epoxy resin and composite beams are prepared through a hand layup process. The microscopic analysis confirmed the uniform dispersion. The static bend test resulted in an increase in the flexural strength of the GO-incorporated beam by 33.3 % compared with that of the neat epoxy beam. Flexural fatigue tests were performed for different load levels, and damage evolution at every 4000th cycle was observed. It was noted that under 60 % loading, a significant change in damage initialization was observed between neat epoxy and 0.25 % GO. However, under a higher load level, a negligible effect of crack initialization was observed in all types of beams.

KEYWORDS

epoxy composite • flexural fatigue • graphene oxide

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Introduction

In recent years, industries across the board including the automotive, aerospace, and structural applications have increased their usage of composite materials. Composite materials are even used in electronic equipment's for EMI (electromagnetic interference) [1]. Composites allow for performance levels that are not attainable with traditional materials. Recent research on composite materials is being conducted in labs throughout the world to better understand the behavior of composite structures in order to maximize their use and appropriateness for industrial applications. Different types of loading conditions are acted upon a composite structure like tensile, compressive, bending, shear and torsion so with respect to that the structure is tested according to the load. In order to improve their performance under these loading, various nanoparticles like Carbon nanotubes, Silicon dioxide, Graphene oxide is induced in composite preparation. Some other nano material like Montomorillonite (MMT) is also used by Kirve et al. [2] to improve the strength of bamboo fiber. Liu et al. [3] performed an experiment to study the effect of carbon nanotubes/graphene oxide on the fracture toughness of CFRP laminates. By changing the ratios of carbon nanotubes/graphene oxide, they found out that there was a substantial increase in the fracture toughness of about 151.2 % than the original laminates. A very detailed review has been done by Han et al. [4] where they have highlighted all the

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improvements in mechanical properties of graphene / carbon nanotube reinforced FRP composites. Also, for Glass fiber incorporation of graphene for mechanical properties were characterized by Rathinasabapathi et al. [5]. According to the report, the interface to volume ratio and filler size has a significant impact on the mechanical properties of composites that have had their matrix modified. Icaopo et al. [6] worked-on investigation of GO reinforcement in epoxy resin. From 0-3 wt. % of GO the flexural strength were considered and resulted first in increase of strength up to 0.3 % and sudden decrease from 0.5 to 3.0 % of GO. Hence, nanofillers are essential for the alteration of the polymer matrix in this direction. The effect of functionalized GO was well documented by Pathak et al. [7] where modified the synthesis process of GO and evaluated the flexural strength. A review is carried out by Dias et al. [8] which emphasizes collective research on articles which highlights composite filled with nanoclay and their mechanical behavior. Also, considering the tribological performance, the addition of graphene lead to increase in the tear strength of silicone rubber as observed by Shinde et al. [9].

They are also known for their resistance towards cyclic loading. So, to design a fiber composite to reduce delamination occurring due to fatigue loading, the important concern. To prevent quick fracture and potentially catastrophic failure, composite structures must be designed for either no growth or moderate, steady growth under cyclic fatigue loadings. Through the recent study, the nanotechnology has showed a significant improvement of epoxy composite fibers. Apart from all the nanoparticles, graphene shows promising results. Graphene is a form of carbon possessing a two-dimensional structure. They have extremely high fracture strength and young's modulus and provides large surface area. As compared to other carbon materials graphene contains higher mobility and thermal conductivity with significant strength of 1 TPa. This makes graphene a potential material in sector transistor element, energy harvesting techniques and sensor application. Due to their high surface-to-volume ratio, two-dimensional carbon nanostructures like graphene and its variants are among the most intriguing reinforcements that can be used to enhance mechanical properties.

In marine, construction, aerospace, and automobile industry's composite structure undergoes flexural loading. Considering the marine environmental conditions, Romina et al. [10] studied the effect of graphene nanoplatelets on the flexural properties of fiber metal laminates. They observed 128 % improvement in flexural strength was by 0.25 wt. % of nanoplatelets which was due to the metal/polymer adhesion. Also, carbon nanomaterials show improvements in mechanical and thermal properties of composites [11]. Flexural test simulation is performed by Joshi et al. [12] which showcase the improvement of epoxy fibers with varying range of graphene content. Filipe et al. [13] understood the behavior of hybrid composites under tension-tension fatigue loading and observed that carbon/carbon composites showed better resistance towards damage. The fatigue damage to composite is occurred in different stages, in order to prolong its effect nanoparticles are dispersed through various processes. A remarkable improvement of flexural fatigue of epoxy composite were observed by Shoriekh et al. [14] by combination of graphene nanosheet. 3-point bending fatigue tests were conducted on aluminum composite with honeycomb core by Mzad et al. [15]. They performed first 3-point static tests on the specimens and then fatigue test at three different loading conditions. Also, to improve interfacial bonding, Dabbagh et al. [16] investigated the effect of GO-NH_2

epoxy composite on the fatigue loading. By varying the loading conditions, about 72-241 % improvement was noted in fatigue life. The synergy effect of graphene oxide sheets and carbon nanotubes was studied by Yuanqing et al. [17]. They observed 950 % increment in fatigue life with respect to pure epoxy. Ashutosh et al. [18] performed experimentation to evaluate the effect of surface roughness on fatigue strength of epoxy composited single lap joint. They used modified adhesive with graphene oxide and observed enhancement of 30 % in fatigue strength. The effect of aluminum oxide, magnesium oxide and copper oxide (CuO) nanoparticles on the fatigue behavior of woven composite was carried out by Ergün et al. [19]. They observed that among different weight concentrations, 0.5 % CuO was the stiffest.

Various processes are involved for graphene oxide to get induced in the epoxy resin. In this the study, a simple synthesis is followed, mentioned in [20]. By using injection molding techniques, Sen-Sen et al. [21] reported the effects of graphene oxide coating on glass fiber. To improve interfacial strength, graphene oxide (GO) is used, which leads to better epoxy and fibers bonding. Most of the research is focused on the static improvement of the composite structure. Fewer researches are carried out on the flexural fatigue performance. The present experimental work details the characterization of epoxy/GO composites and studies the effect on the flexural fatigue life of the composite beams.

Materials and Methods

Materials

Research Grade graphene oxide was used to incorporate in epoxy resin (EPOFINE) and its hardener. For preparation of specimen, glass fiber of 700 GSM was used. All the materials came up with the properties as shown in Tables 1 and 2.

Table 1. Specifications of glass fiber

Parameters	Specification
Weight, Gram/Sq.m	740.0
Width, mm	1020.0
Thickness, mm	0.7
Weave Pattern	Plain

Table 2. Specifications of nanomaterial

Parameter	Specification
Thickness, nm	0.8-2.0
Average Lateral Dimension(X&Y), µm	10
C:O	55:45

Dispersion of graphene oxide with epoxy resin

There are two different weight concentrations of GO i.e. 0.25 and 0.50 % used in this study. As per the E-glass fibre sheet to be made of 300 × 300 mm^2, we require 0.625 gm of graphene oxide (0.25 % GO). Essentially, at a concentration of 2 mg·ml^{-1}, GO was dissolved in acetone. For proper mixing of acetone and GO, its mixture was kept into an ultrasonic bath for 2 hours. This mixture was then poured into a beaker containing epoxy resin required for the sheet. The whole mixture of resin/nanoparticles was kept on magnetic stirring for about 10 hours (Fig. 1). This process was referred from [20].

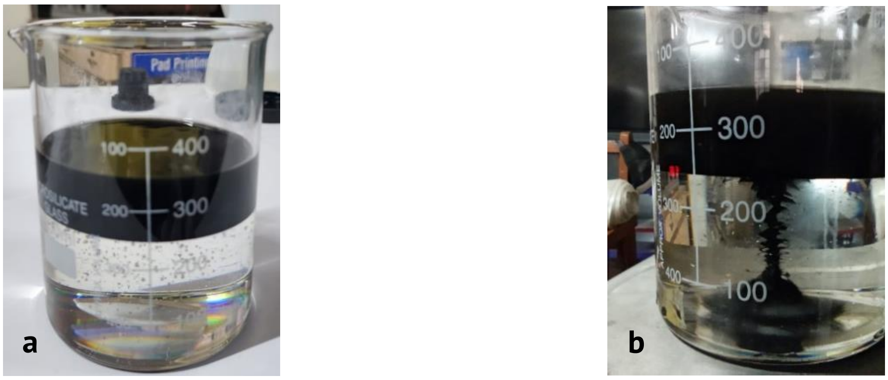

Fig. 1. (a) GO mixture with epoxy resin and (b) magnetic stirrer action of GO and resin mixture

Composite preparation

The composite was fabricated by hand lay-up process followed compressed curing with orientation of $[0/45/0]_{s.}$ Three different sheets of 300 × 300 mm^2 were fabricated for net epoxy, 0.25 and 0.50 wt. % of GO. Testing of specimen were carried out according to ASTM D790 [22] (Fig. 2).

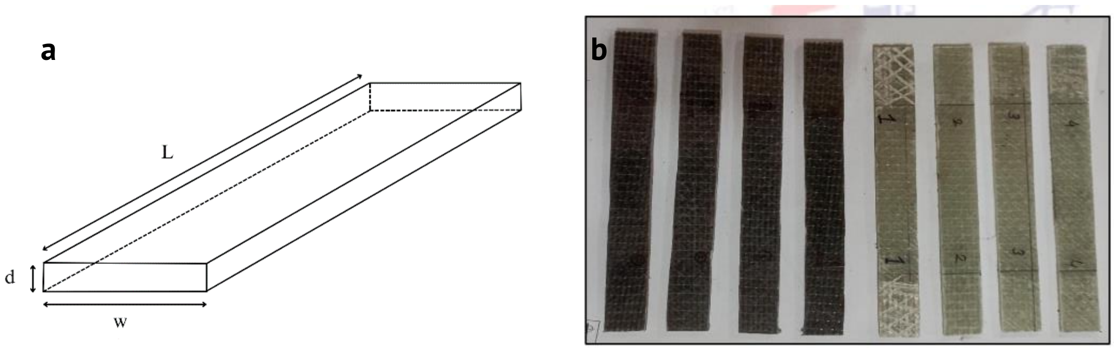

Fig. 2. (a) Schematic of test specimen and (b) actual test specimens

Static bending test

The test was carried out on universal testing machine (UTM) with capacity of 50 kN in accordance to ASTM D790 standard (Fig. 3). The ram speed for the tests was 5 mm/min. For each test, 3 samples were used. The bending stress was calculated: $\sigma = \frac{3FL}{2wd^2}$, where F is the maximum load sustained, L is the supporting span length, w is the width of specimen and d is the thickness of specimen.

Fig. 3. Schematic of three-point bend test

Flexural fatigue test

The flexural fatigue test is carried out for specimens under different loading conditions using an axial fatigue testing. The stress ratio (R) was 0.1 and frequency was 3.5 Hz for all tests. The fatigue test was carried out at 80, 60, 40 % of the maximum bending force they could withstand at static test (F). The test was terminated when the upper surface was delaminated.

Results and Discussion

3-point bend test and bending fatigue were carried out on the ready specimens to evaluate the bending strength and fatigue life of the specimens.

Static bend test

According to the standard, the test was carried out at 5 mm/min ramp speed on UTM at MITWPU. For each percentage of GO, there were three specimens tested on UTM and average result for load vs cross head travel are plotted in the Fig. 4(a). It is observed that the flexural strength modified epoxy composited increases from 255 to 322-343 MPa. The maximum increase in strength at 0.25 wt. % of GO corresponds to a hike of 25.65 % compared to net epoxy resin (Fig. 4(b)). This result develops that even at lower concentrations, the strength of the composite beam can be improved. It is because of interfacial bonding between the layers is enhanced due to the incorporation of GO [23].

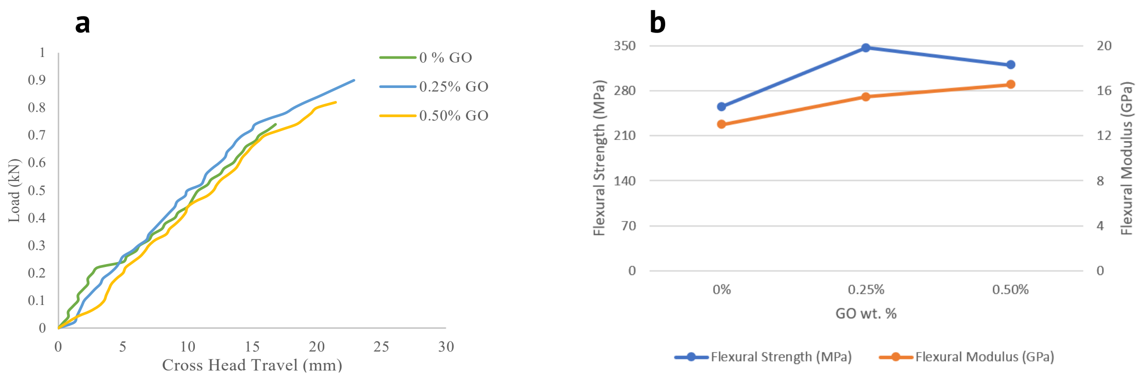

Fig. 4. (a) Load vs displacement; (b) variation of flexural strength and flexural modulus

Flexural strength at 0.50 wt. % of GO results in significant rise of 20.3 % compared to net epoxy. But increasing the weight content of GO has led to a small amount of decay in the flexural strength. In the graph as shown in Fig. 4(b), plotted for flexural strength vs flexural modulus, the increase in flexural strength with increase in weight percentage of GO has been observed, whereas flexural modulus shows similar trend but slight decline for 0.50 wt. % of GO. Such behavior might be linked to nanofiller agglomerations that form inclusions from which microcracks can propagate during testing, resulting in a decrease in the mechanical characteristics of the epoxy resin. Furthermore, these agglomerates yield a reduced usable section, causing the specimens to fail prematurely [6].

Fatigue damage evolution

The fatigue testing is done by varying loading conditions. For analyzing the damage propagation, the 60 % loading condition is discussed here. Fatigue testing have been halted at various points during the loading to provide information about damage development and to comprehend how fatigue cycling impacts composites. At every 4000th cycles observations were noted. Transverse matrix cracks typically begin at the specimen's edge, owing to the effects of microdamage during sample cutting. These damages are highlighted in the yellow boxes in Fig. 5. From 6000th cycles onwards the crack density rises as the cyclic load increases, followed by crack coupling and interfacial deboning. The upper layer starts to de-bond and delaminate at further increase in cycles. Hassan et al. performed similar experimentation and conferred that during this phase, crack propagation and buildup in a number of stress concentration areas are the main causes of a rapid decay of modulus. The complete surface gets delaminated at 16232nd cycle. The size of the yellow box indicates that the damage is increasing in that area. It is observed that growth of the crack is in the dispersed way. Reifsnider et al. [23] also studied the behavior of cracks on the fiber surface and inferred that the transverse cracking is formed in regular spacing, which can also observed here.

The fatigue life data for the specimens with and without graphene oxide are presented in Table 1. For 0.25 wt. % GO, Fig. 6 represents the damage evolution due to flexural fatigue loading. The results show that the addition of graphene oxide significantly delays in the damage initialization under fatigue loading of the epoxy composite. Daniel et al. [24] reported that modest but considerable stiffness and strength improvements were made, especially at low graphene oxide weight fractions. GO has many groups on the surface and edge which are reactive. Due to the incorporation of nanoparticles led to increase in the interfacial bonding [25]. It is due to the fact that graphene acts like a filler between the growing fractures. Using these findings as a basis, we suggested the following interpretation: In the experiment, it was observed that 0.25 wt. % GO sustained maximum number of cycles to failure at 60 % of loading condition. The table illustrated fracture images at loading of 60 % at every 4000 cycles. For 0.5 GO wt. % it is observed that the delamination is more even at early stages. This indicates that more weight leads to agglomeration.

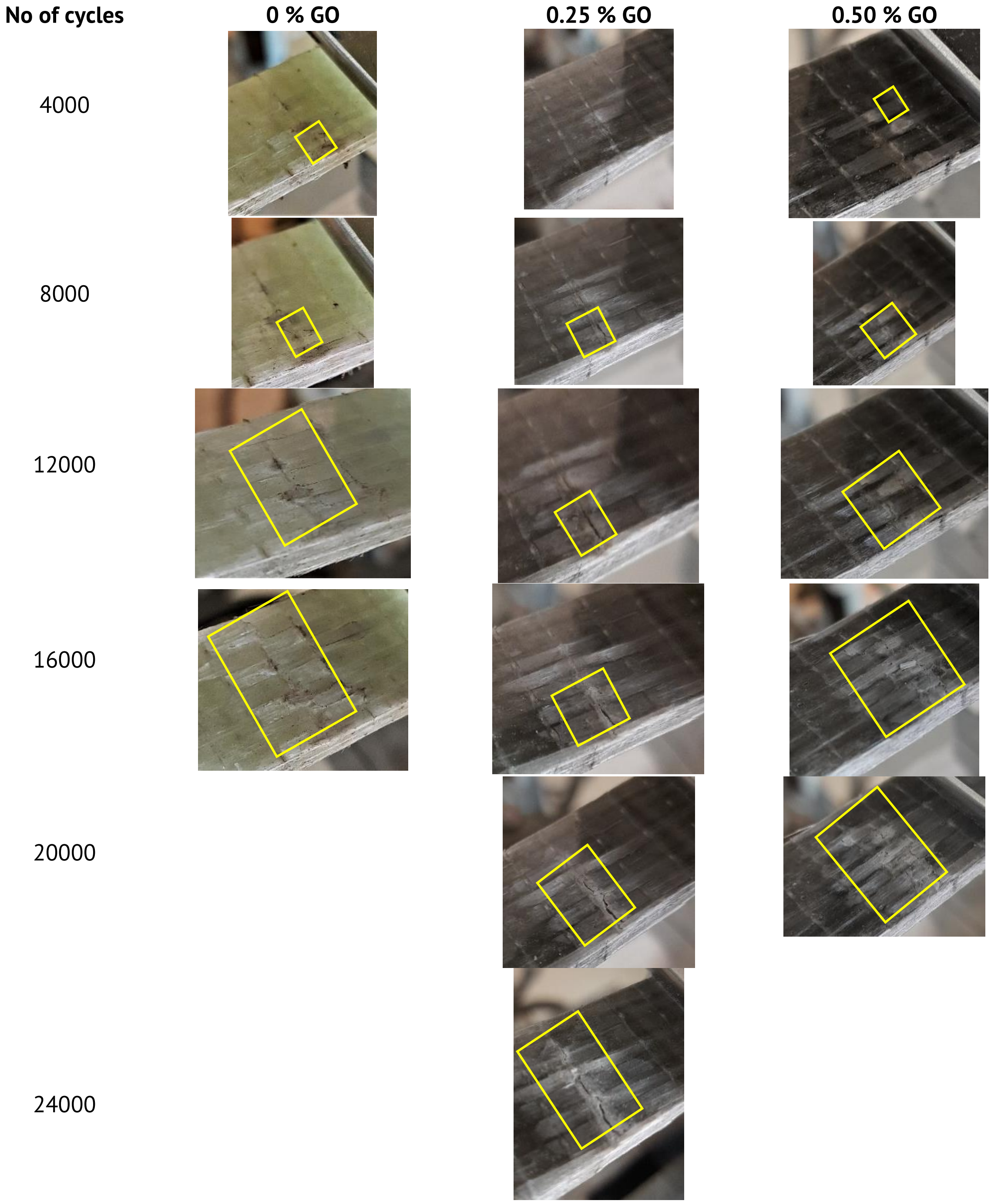

Fig. 5. Damage evolution of all type's specimens under fatigue loading

Similarly, for the rest of the tests this pattern is observed and a table is formulated. At high levels of load, there is no significant increase in number of cycles to failure. Figure 6 illustrates the S-N curve for the tested specimens.

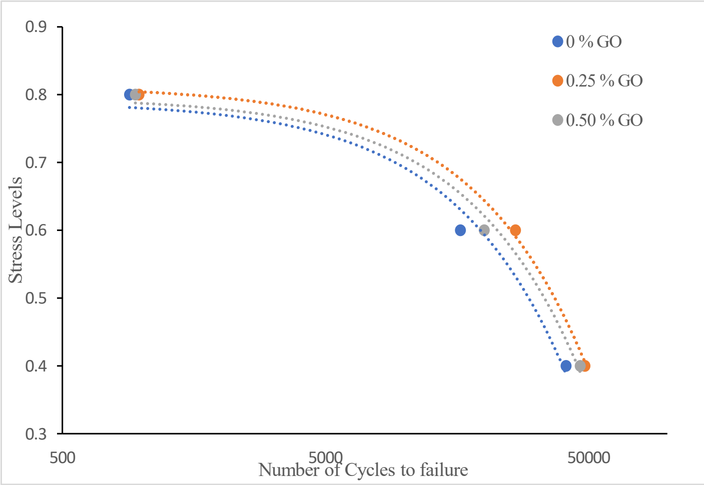

Fig. 6. Variation of fatigue loading cycles to failure for different stress levels

Conclusions

In the present research, the influence of different weight concentrations of GO on the damage evolution under flexural fatigue loadings of the composite beam. These were the key findings from this experimental investigation. Synthesis of GO dispersion is time-consuming but also was well dispersed. The static results showed a significant increase in the flexural strength of 0.25 % GO than the net epoxy composite beam. Due to incorporation of GO, the failure of its beam has showed less debonding between the layers of lamina which also indicated a better interfacial strength. Fatigue results showed that at 0.25 wt. % of GO, the crack initialization is postponed by 4000 cycles than the neat epoxy. Also, 0.5 wt. % of GO caused agglomeration and resulted in early failure, with more delamination observing than that of 0.25 % GO. The incorporation of functionalized GnP into the polymer matrix increased the matrix phase's and the fibre interfaces' fracture toughness. The continuous matrix phase's continual nanoparticle dispersion caused microcracks to deflect, lowering the crack-tip energy. As a result, composites showed extended fatigue life before final fracture and the beginning of matrix cracking and delamination under cyclic loading was delayed.

This research will guide for designing and improving glass fiber composites under cyclic loading. However, research opportunities are open to understand the effect of functionalization of GO for better interfacial bonding. Moreover, composite preparation techniques like VARTM can also be used for uniform dispersion of modified epoxy.

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