Research Article

Interlaminar Shear Failure Monitoring in GFRPs Using Graphene Nanoplatelets

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ABSTRACT

Components made of glass fiber reinforced polymers often fail under interlaminar shear loading conditions, which can significantly compromise their structural performance. To solve this problem, the piezoresistive effect of graphene nanoplatelets integrated into the glass fiber reinforced polymers is proposed as a technique to monitor and detect interlaminar shear failure. For that, short beam shear specimens were prepared from glass fiber reinforced polymers modified with graphene nanoplatelets, and changes in electrical resistance caused by quasi-static bending forces were recorded to monitor the electromechanical behavior and failure mode. The interlaminar shear strength was obtained at around 30 MPa for both composites (0.75 and 1 wt.% graphene nanoplatelets). Results indicate that the incorporation of graphene nanoplatelets has a minimal effect on the electromechanical behavior curves, and both concentrations (0.75 and 1 wt.%) provide good electrical sensing capability for interlaminar shear failure in glass fiber reinforced polymers, which can be used as an active tool for health monitoring applications across a wide variety of industrial applications, especially where this failure mode may occur.

1. Introduction

Glass fiber reinforced polymers (GFRPs) are now used in a great variety of industrial applications due to their high stiffness, high strength, lightweight, and corrosion resistance [1]. Practical applications in industries such as energy, automotive, and marine include boats, vessels, wind, and tidal turbine blades [2]. However, GFRPs are susceptible to failure under interlaminar shear modes, which significantly affects their mechanical performance and reliability [3,4]. For composite laminates, interlaminar shear failure is considered one of the principal failure modes that may lead to total composite failure. Hence, there is an important opportunity to develop novel strategies to monitor interlaminar shear failure in GFRPs in order to avoid catastrophic failures in structural components. Recently, the integration of carbon nanostructures into GFRPs for sensing purposes and for in-situ structural health monitoring has gained significant attention in the scientific community [5]. In the last few years, a significant amount of research has been reported to evaluate the electrical resistance method based on self-sensing GFRPs modified with carbon nanostructures under different loading conditions [6-8]. For example, Hao et al. [9] reported the piezoresistive effect of composites

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made of glass fiber with a graphene coating. Their results suggested that the graphene coating on glass fibers is sensitive to strain under tensile loading. The piezoresistive response of GFRPs modified with graphene can be affected by the method of incorporation. Moriche et al. [10] found high sensing capabilities of GFRPs when the incorporation of GNPs was performed into the resin matrix. Mahmood et al. [11] reported that the GFRPs modified by reduced graphene oxide (rGO) promoted a good piezoresistive behavior for strain monitoring applications under static and dynamic conditions. In another work, Moriche et al. [12] conducted electro-mechanical measurements on functionalized GNPs-based GFRP composites under 3-point bending and interlaminar shear tests. They found that the multiscale GFRPs had the capacity to detect damage with high sensitivity. Another work by Mahmood et al. [13] investigated the effect of temperature on the piezoresistive response of GFRPs with rGO under flexural loading conditions. They found good piezoresistive behavior in composite laminates with rGO-coated glass fibers under flexural loading at various temperatures. Sanchez et al. [14] proposed the use of epoxy matrix composite laminates doped with GNPs to form conductive networks for monitoring fracture propagation in modes I and II. When the electrical resistance reached a critical value, the failure of the laminate was detected. Reghat et al. [15] reported the use of graphene-coated GFRPs to improve their sensing properties. It was observed that although the integration of graphene into GFRP led to a decrease in mechanical properties (tensile and flexural strength), the electromechanical properties exhibited good piezoresistive behavior under cyclic tensile loadings [15]. Regarding the improvement of mechanical properties, Veerakumar et al. [16] investigated the effect of functionalized GNPs on GFRP composites and noticed enhancements of 16% and 76% in the tensile strength and fracture toughness, respectively. Also, Ramos-Galicia et al. [17] found improvements in interlaminar shear strength (ILSS) of GFRP composites reinforced with graphene derivatives. The influence of the weight percent of GNPs on the electrical and piezoresistive properties of GFRPs has been reported under 3-point flexural tests [18]. Results showed that the piezoresistive behavior of GFRP/GNPs composites was more sensitive at GNP concentrations above the percolation threshold (0.75%) because of an effective electrical network within the composites. A good damage self-sensing capability of GFRPs with GNPs under tensile and flexural loading was also observed in [19]. Batusha et al. [20] evaluated the use of rGO coated onto fibers integrated onto

composite laminates to monitor their fracture behavior. The multiscale composite laminate with rGO coated on its fibers demonstrated good sensing capabilities for in-situ crack monitoring under in-plane and out-of-plane loading. Agnihotri et al. [21] reported the influence of the weight percentages of nanoclay on the ILSS of GFRPs. They observed that the ILSS of neat GFRPs increased by 34.31% due to the addition of 2.5 wt.% nanoclay. Thakur and Singh [22] evaluated the ILSS of GFRPs and the effect of GNPs on damage caused during drilling. It was found that the ILSS increased by 18.49% in 0.25wt% GNP-embedded GFRP composite. In another work, Thakur and Singh [23] examined the ILSS of GFRPs fabricated with different wt.% of GNPs. Their results showed that the ILSS of GFRP composites was enhanced by 18.5% at a 0.25 wt.% GNP concentration because of the optimum dispersion of the GNPs in the epoxy matrix. Singh and Thakur [24] studied the influence of carbon nanotubes on the abrasive water jet machining response of GFRPs. The authors found that the incorporation of carbon nanotubes into GFRPs improves their machining performance. In another study by Thakur et al. [25] investigated the effects of various wt.% GNPs on drilling-induced damage of carbon fiber reinforced composites. They found that the incorporation of 0.25wt.% GNP into the composite enhanced their mechanical and machining qualities. Very recently, Ahmad et al. [26] reported a computational approach for modeling the electromechanical response of graphene-coated laminates under quasi-static tensile loading. Their research provided a valuable contribution to monitoring the strain in these laminates. Taymaz et al. [27] fabricated carbon FRP composites with MXene fibers for in-situ strain sensing. The integration of MXene fibers into laminates provided excellent piezoresistive strain sensing capabilities for repetitive tensile deformations and low-velocity impact loading. Compared to tensile and flexural tests and the type of carbon nanostructure, less effort has been performed in the evaluation of GFRP modified with GNPs and subjected to the interlaminar shear test. One of the most recent studies related to interlaminar shear strength of GFRPs manufactured by spray coating of GNPs onto glass fibers is the one by Li et al. [28]. They tested short beam shear specimens made of these manufactured GFRP/GNPs. Their results showed that the short beam shear strength of GFRPs modified with GNPs increased by 8% compared to laminates without GNPs. However, the electromechanical response of GFRP/GNPs for damage monitoring was not reported. Therefore, this work aims to evaluate the interlaminar shear failure of GFRPs using GNPs as piezoresistive

sensing elements. Interlaminar shear failure of GFRPs with integrated GNPs was characterized using the short beam shear method, evaluating the influence of GNPs on the electromechanical behavior and sensing capabilities during damage progression.

2. Materials and Methods

2.1. Materials

The commercial graphene nanoplatelets (GNPs) were provided by Strem® Chemicals Inc. (USA) and had a carbon content of 99.5 wt. %. The XRD pattern of GNPs is presented in Fig. 1. A characteristic diffraction peak was observed at approximately 26.37°, which corresponds to the (002) plane, confirming the crystalline nature of GNPs [29].
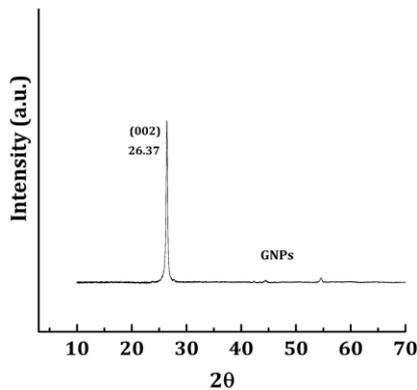

Fig. 1. XRD diffraction patterns of GNPs

Composite was fabricated by unidirectional E-glass fibers with a density of 1 g/cm^3 and epoxy resin (Epolam 2040 with amine hardener Epolam 2047). Both materials were purchased from Axon Composites (USA). The GNPs solution was prepared using ethanol (from J.T. Baker, USA). The electrode contacts (copper wires) were made using silver paint from Ted Pella Inc. (USA).

2.2. Manufacturing Process

In order to prove the concept, glass fiber reinforced polymers (GFRPs) modified with GNPs were manufactured following the method established in our previous work [18], which consists of a combination of a spray coating technique and the vacuum-assisted resin infusion (VARI) process. Fig. 2 presents the manufacturing method of the GFRP with GNPs. The first part of the process aims to deposit GNPs onto the glass fiber surface using the spray coating technique. For that, a certain amount of GNPs was mixed in 40 mL of ethanol, and then the solution was subjected to ultrasonic dispersion using an ultrasonic bath for 1.5 h at room temperature. Two different GNP concentrations (0.75 and 1.0

wt.%) were used to prepare the solutions. Each solution was sprayed onto the glass fiber fabric using an airbrush operated at an air pressure of 30 psi. After 24 h of solvent evaporation, samples of glass fiber with GNPs were analyzed using a scanning electron microscope. Composite laminates were fabricated by VARI. Four fabrics with GNPs were stacked in a [0]$_4$ configuration, and then the prepared mixture of epoxy resin (100:30) was infused into the vacuum bag molding under an applied pressure of -85 kPa. The GFRP-GNP laminate was cured on a hot plate for 4 h at 80 °C. Specimens were finally extracted from the cured laminates. Specimen dimensions were determined according to the ASTM standard D2344 [30].
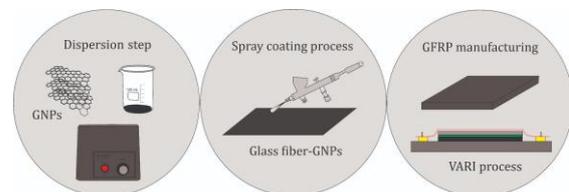

Fig. 2. Manufacturing process of GFRP-GNP laminate

2.3. Short Beam Shear Test

The electromechanical tests were conducted using short beam shear (SBS) specimens with dimensions of 40 mm x 13 mm x 3.2 mm. All SBS specimens were instrumented using copper wires as electrodes. The electrodes were attached at both ends of the SBS specimen using silver paint to reduce contact resistance, as shown in Fig. 3. All SBS tests were conducted following the recommendations described in the ASTM D2344 [30]. Five samples were tested for each laminate condition. The tests were performed using an Instron testing machine with a 10 kN load cell and a three-point bending fixture. The span-to-thickness ratio was set to 4, and the loading speed was 1 mm/min. During the test, the changes in electrical resistance ($\Delta R/R_0$) were recorded by means of a Keysight 34465A digital multimeter. The obtained $\Delta R/R_0$ values were used to create a load-displacement-electrical resistance graph for interlaminar shear failure monitoring of GFRPs using GNPs.
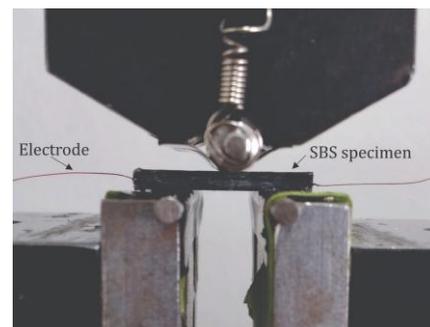

Fig. 3. Electromechanical test set-up of SBS specimen

2.4. Scanning Electron Microscope

Glass fiber fabrics coated with GNPs at different concentrations (0.75 and 1.0 wt.%) were examined under a scanning electron microscope (JEOL JSM-6610LV) operated at 15 kV and 5000× magnification.

3. Results and Discussion

3.1. SEM Observations

In order to examine the surface morphology of the unidirectional glass fiber before and after the GNP spray deposition, a SEM analysis was performed on samples with 0, 0.75, and 1 wt.% GNP concentrations. Fig. 4 shows SEM images of glass fibers analyzed after the spray coating of GNPs onto their surfaces.
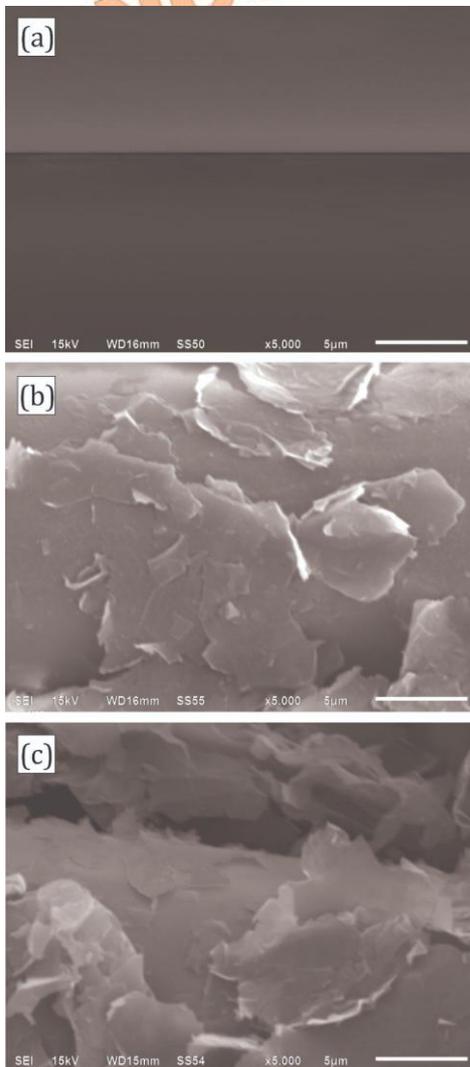

Fig. 4. SEM micrographs of glass fibers with GNPs; (a) 0 wt.%, (b) 0.75 wt.% and (c) 1.0 wt.%

It can be observed in Fig. 4a that, because of the absence of GNPs on the fiber, a smooth morphology of the surface is observed. When GNPs are deposited onto the fibers (Fig. 4b and c),

a different surface morphology can be noted. Both 0.75 and 1.0 wt.% GNPs concentrations present a relatively homogeneous distribution of GNPs on the fibers. It is also observed that the GNPs cover the glass fiber in a random orientation and connect among themselves. The results indicate that spray coating allows a good and direct deposition of GNPs onto the surface of the fibers and confirms that it is a viable technique to obtain the final GNP-coated fiber for multiscale composite laminate manufacturing.

3.2. Interlaminar Shear Failure Monitoring

In order to investigate the sensing capability of GNPs for interlaminar shear failure monitoring in multiscale GFRPs, the short beam shear (SBS) test and in-situ measurement of the electrical resistance were simultaneously performed on instrumented samples (see Fig. 3). Fig. 5 presents the relationship between the load-displacement and the changes in electrical resistance obtained from the specimens during the SBS test.
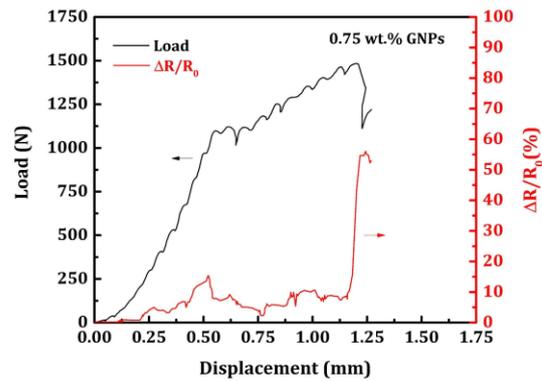

Fig. 5. Representative load vs. displacement curve and electrical resistance change during the SBS test of GFRP with 0.75 wt.% GNPs

It can be seen in the graph that the load increases linearly with the growth of displacement until it reaches 1100 N. On continuation of the test, the load increases again until the final failure is reached, where it suddenly drops at 1.25 mm displacement (see Fig. 5). This refers to the interlaminar shear failure of the GFRP composite. The obtained value of ILSS for 0.75 wt.% was 29.2 MPa. By comparing the changes in electrical resistance obtained with the load-displacement curve, it is possible to observe a good correlation between both mechanical and electrical responses, confirming that the behavior of the specimen under bending and the interlaminar shear failure can be monitored and detected due to the sensing capability of GNPs. It is also important to note that the change in the electrical resistance remains small in the early stage of bending loading but changes abruptly at the moment of

the specimen failure, see Fig. 5. This can be explained by the fact that in the early loading phase, the GNP network retains electrical conductivity as microcracks have not yet developed enough to disrupt conductive pathways. On the other hand, the fact that the electrical resistance shows a significant increase and the load drops means that the specimen has failed, and hence it can be correlated to the failure mode.

In the case of GFRPs with 1.0 wt.% (Fig. 6), both mechanical and electrical curves follow a similar trend with intermittent drops during the SBS test, as observed in GFRPs with 0.75 wt.%. This indicates that the changes in electrical resistance of GNPs integrated into GFRPs are sensitive to the interlaminar shear loading, and the results also confirm that the concentration of GNPs has a minimal effect on the piezoresistive response of GFRPs subjected to shear loading. These results are consistent with our previous work [18] in GFRP/GNP laminates subjected to flexural loading conditions, where a good electrical sensing capability is obtained at GNP concentrations above the percolation threshold (0.75%). The obtained value of ILSS for GFRPs with 1 wt.% GNPs were 30.3 MPa, which was similar to 0.75 wt.%. These ILSS results agree with the results obtained by Thakur and Singh [22,23].
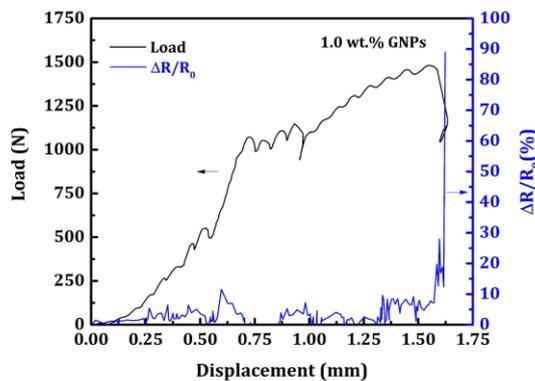

Fig. 6. Representative load vs. displacement curve and electrical resistance change during SBS test of GFRP with 1.0 wt.% GNPs

3.3. Failure Analysis

The analysis of the failure under shear loading conditions can be observed in Fig. 7. More specifically, Fig. 7a presents a photograph of the failure of an SBS specimen with 0.75 wt.% GNPs and Fig. 7b shows the failure of an SBS sample with 1.0 wt.% GNPs. It can be stated that during short beam shear mechanical testing, the specimens experience a state of normal and shear stresses, which in turn results in an interlaminar shear failure, as shown in Fig. 7a and b. For both

cases, the failure experimentally observed in SBS specimens was in reasonable agreement with the behavior of the electromechanical curve (Figs. 5 and 6), where the abrupt drop observed in the electromechanical curve marks the final process of damage generated in GFRPs during the SBS test and confirms that GNPs are sensitive for monitoring and detecting interlaminar shear failure.
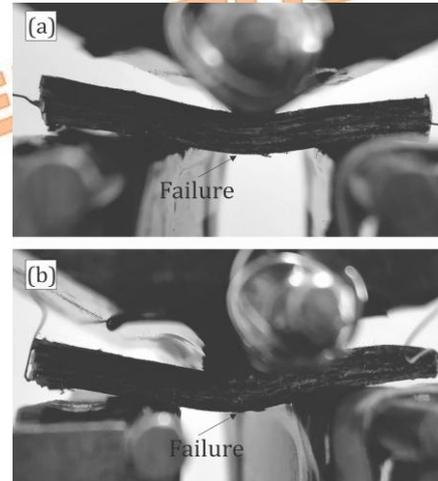

Fig. 7. Interlaminar failure observation in SBS specimens made of GFRP with GNPs; (a) 0.75wt.% and (b) 1.0 wt.%

4. Conclusions

Glass fiber reinforced polymer was modified with graphene nanoplatelets (GNPs) to obtain multiscale composite laminates with electrical and piezoresistive properties. For that, glass fibers were first spray-coated with GNPs (0.75 and 1.0 wt.%), and then the laminates were fabricated by the vacuum-assisted resin infusion process. After the spray coating, the SEM analysis revealed the presence of GNPs on the fiber surface for both concentrations (0.75 and 1.0 wt.%), with a homogeneous distribution of GNPs to ensure the generation of electrically conductive paths. Short beam shear was used as a viable test method for evaluating the interlaminar shear failure of GFRPs. In this sense, SBS specimens were instrumented to obtain electrical resistance measurements during the mechanical test. The results of the electromechanical test confirm the sensing capability of GNPs for in-situ monitoring and detection of interlaminar shear failure of GFRPs. The changes in electrical resistance recorded during the SBS test correlate well with the load-displacement curve. The abrupt drop observed from the mechanical test represents the interlaminar shear failure of the specimen, which aligns well with the electrical resistance signal. The ILSS value of the GFRP composite reinforced with 0.75 and 1 wt.% GNPs were around 30 MPa and were found to be minimally affected by the

weight percentage of GNPs. Therefore, the use of GNPs as sensing elements is a simple, practical, and viable tool for interlaminar shear failure monitoring in GFRPs.

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Funding Statement

Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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