

Multifunctional Carbon Fiber–Graphene Epoxy Composites for Structural Health Monitoring under Dynamic Loading

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Abstract

In this work, multi-scale hybrid composite materials were fabricated from carbon fibers and graphene nanoplatelets mixed at different ratios and dispersed in epoxy resin matrix. The main mechanical characteristics (stress-strain) of these composite materials were determined and slight differences were observed with varying mixing ratios. The variation in electrical resistance of the fabricated composite samples was determined as a function of the applied mechanical stress at different frequencies of the multiple loading cycles. Approximately linear behavior was observed for all samples and hence the gauge factor was calculated and found to increase with applied stress and frequency. These results can be successfully used to design multi-functions composite materials for smart and self-health monitoring systems.

Keywords: Carbon fibers; Graphene nanoplatelets; Composite materials; Smart systems

Received: November 2025; **Revised:** January 2026; **Accepted:** February 2026; **Published:** April 2026

1. Introduction

In the last two decades, rapid developments were witnessed in materials science and engineering as a new generation of multifunctional composite materials was invented to satisfy many industries and applications in avionics, space, automobiles, and renewable energy [1]. These carbon fiber reinforced polymers (CFRP) represent the cornerstone in these industries due to their high toughness-to-weight ratio, excellent fatigue resistance, and superior hardness when compared to the conventional materials. However, the different nature of these materials and brittle polymeric matrix make them subjected to complex types of internal damage, such as delamination, micro-cracking, and fiber/matrix debonding, which initiate and develop under the effect of dynamic loading like low-velocity shocks, high-frequency vibrations, or variant periodic loadings [2-4]. These internal defects form a big challenge because they are invisible on the surface but lead to big degradation in the mechanical properties and hardness, and hence cause a catastrophic and unexpected failure. Therefore, an increasing demand to develop smart systems has emerged with the capability for on-line structural health monitoring (SHM) that allows the early detection of damage, diagnosing its site and intensity. Consequently, the functional lifetime of these structures is lengthened, maintenance costs are reduced, and safety and reliability parameters are increased [5-7].

Conventionally, the SHM systems depend on separate techniques such as strain gauges, fiber Bragg gratings (FBGs), or piezoelectric transducers those are mounted on the surface or within the system [8,9]. Despite that these sensors are precise, they suffer from core shortcomings, mainly mechanical incompatibility with the host material, which may lead to deformations in the stresses and weak inter-bonding, in addition to the complexities of connections and additional weakness points proposed to these systems [10]. Accordingly, the concept of smart materials or self-sensing materials has been introduced as a revolutionary alternative aiming to make the structural material capable of sensing its internal state without external devices [11,12]. This can be achieved by the investment of piezoelectric property of the conductors as the stress or mechanical damage lead to change the electrical resistance to allow using the resistance measurements as a direct indicator of the material state [13]. Carbon fibers have moderate electrical conductivity but the crosslinking network they create within the composite material is sensitive to deformations and fractures. This makes the CFRP composites natural and promising candidate for self-sensing applications [14-16].

The dependence on carbon fibers only as a sensing network faces some reasonable challenges, especially under complex dynamic loadings [17,18]. The piezoelectric response of these composites

may be nonlinear and low-sensitive at the initial damage course [19]. As well, the fundamental conduction mechanism within the long fibers may not be sufficiently sensitive to the initiating damage at the microscale within the matrix or at the interstitial surfaces [20,21]. To overcome these restrictions, conductive carbon nanofillers, such as carbon nanotubes (CNTs) or graphene, are incorporated in the polymer matrix to form a multi-scale hybrid structure [22]. Addition of graphene – with its exceptional characteristics like drastic surface area, superior electrical conductivity, and amazing mechanical properties – results in a quality leap in the performance of CFRPs [23,24]. Mechanically, graphene enhances the inter-bonding between carbon fibers and matrix, which supports the interlaminar shear strength (ILSS) and increases the resistance to crack propagation, and hence increase the overall toughness of the material [25]. Literature refers to reasonable enhancement in bending resistance and endurance up to 50% when graphene is appropriately incorporated. For electrical and sensing functions, the dispersion of graphene plates within epoxy leads to create a conductive nano-network in parallel to carbon fiber network [26-29]. Under mechanical loading, the inter-distances between graphene plates are changed and the tunneling resistance and inter-fillers contact are changed too, therefore, a strong piezoelectric signal is generated. This unique hybrid structure allows high sensitivity to the tiny deformations and initial damage at the microscale that cannot be detected by carbon fibers only [30]. The synergy between micro-fibers and nano-sheets creates a hierarchical structure with capability of responding to a wide range of frequencies and stress levels. This makes them optimum choice for SHM under various dynamic loading conditions, from low-amplitude vibrations to high-energy shocks. Literature confirmed that these multi-functions composites are capable to simultaneously detect and trace the development of damage during shock and wear tests. Therefore, sufficient information on the system integrity can be provided [31-33].

Most previous studies have focused on the characterization of static properties or sensing behavior under simple loadings, while there is an increasing need to understand the effect of deformation velocity, loading history, effect of stresses on stability and responsivity of graphene nano-network, and hence the reliability of SHM signals [34-37]. The biggest challenge is how to isolate the effect of pure dynamic loading from the effect of damage occurred within the material on the overall electrical signals, and then developing physical or statistical models those are capable of untangling these signals and accurately decoding the state of matter [38-40].

This study presents an experimental and theoretical investigation of the electromechanical behavior of multi-functions hybrid composites fabricated from continuous carbon fibers reinforced with graphene nano-plates dispersed in an epoxy matrix under various dynamic loading conditions including low-energy periodic shocks and loadings. The variations in simultaneous electrical resistance of the samples are analyzed during mechanical tests and compared to the development of damage and internal strain measured by conventional digital image correlation (DIC). This study also aims to investigate the effects of different graphene concentrations and dispersion modes on gauge factor, linearity, and stability under periodic dynamic loading. The results obtained from this study are expected to contribute in designing new generation of smart composite structures with superior capability of self-monitoring in the real time in order to fabricate engineering systems those make the resource consumption much safer, much more efficient and economic.

2. Experimental Part

Fabrication of multi-functions hybrid multifunctional carbon fiber–graphene epoxy composites is an engineering challenging process that requires strict control of process parameters to ensure the formation of the required hierarchical structures that allow self-sensing of SHM under dynamic loadings.

Figure (1) shows a flow chart of the experimental part of this work. The fabrication process is started by the preparation of nano-modified matrix as the graphene nano-platelets (GNPs) is dispersed within the epoxy resin before adding the hardener. Dispersion process is very crucial because graphene tends to re-agglomeration due to strong van der Waals forces among these platelets. This leads to form clusters, which hinder the formation of uniform conductive network and weaken the mechanical properties. In order to achieve effective dispersion, advanced techniques are used to generate high shear stresses to break these agglomerations and then distribute the platelets homogeneously over the matrix. Three-roll milling (TRM) was used as an active method and industrially scalable as it can produce shear stresses much higher than the conventional high shear mixing or probe sonication methods. The TRM enables *in situ* exfoliation of natural graphite to produce graphene micro-platelets inside the epoxy without using solvents or complex chemical treatments. Using TRM has reasonably enhanced the viscosity of the mixture and ensured unified dispersion free of large clusters, as confirmed by the optical

microscopy. After that, the hardener was added at a weight ratio of 6:10 with continuous and slow mixing to avoid trapping air bubbles, which may form origins for future mechanical failure [41].

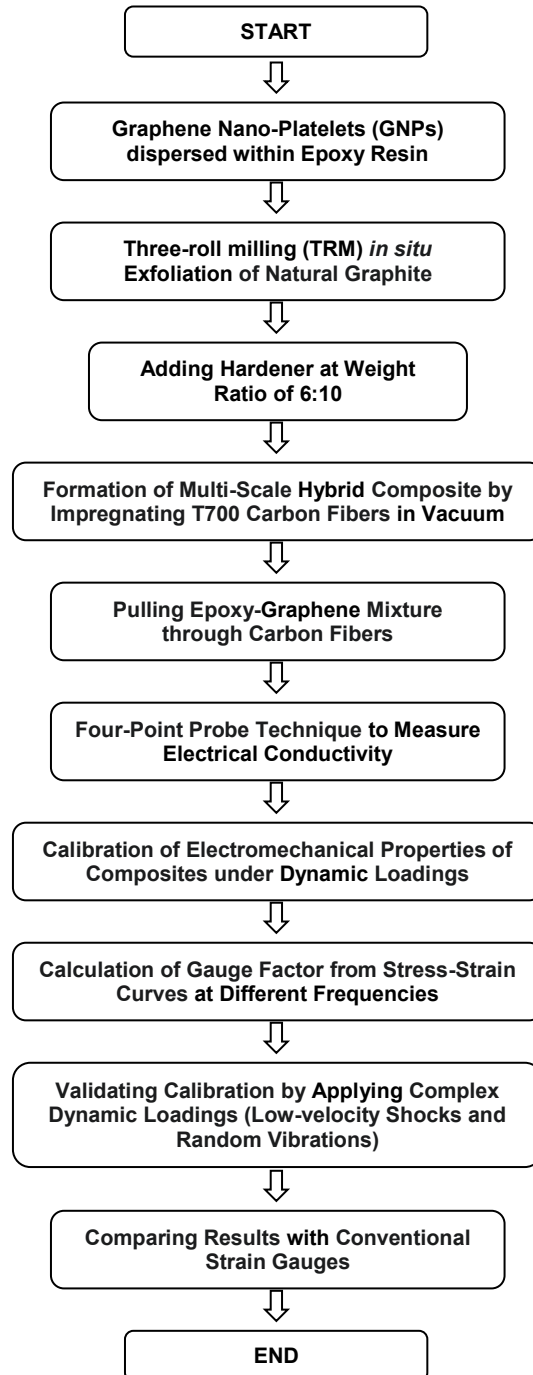


Fig. (1) Flow chart indicating the steps of the experimental part of this work

After the nano-matrix was prepared, the carbon fibers (T700) were impregnated to form the multi-scale hybrid composite. Fibers are mostly highly resistive and non-crimp. They were impregnated with epoxy-graphene mixture by vacuum-assisted resin infusion. In this method, the carbon fabric layers are placed in a single-side mold then the epoxy-graphene mixture is pulled through the fibers due to vacuum pressure (0.1 mbar). This method is effective and allows monitoring of resin flow [42]. This method also requires an accurate investigation of filtration effects as the graphene platelets are filtered by dense

carbon fiber network to cause their agglomeration at the inlet and inhomogeneous properties along the sample. The electrical conductivity was measured along the sample by the four-point probe technique in order to detect the effect of filtration throughout the reasonable reduction of conductivity at the outlet when compared to the inlet.

After solidification, the electromechanical properties of the composites were calibrated to ensure their reliability in the SHM application under dynamic loadings. This characterization aims to determine the relationship between the variations in resistance ($\Delta R/R_0$) and the mechanical stress (ϵ) applied to the sample, this relationship is known as gauge factor (GF) and is determined by performing static or dynamic mechanical tests with synchronized measurements of signals. The calibration was started by coating the contact regions on the sample surface with silver paint to guarantee good electrical contact (i.e., reduce contact resistance). The sample was mounted in the universal testing machine supplied with load cell, extensometer and DIC, then cyclic loadings were applied while the varying resistance was continuously recorded using a picoammeter. The gauge factor (GF) was calculated from the plot of relative variation of resistance versus strain as the slope of the linear part. Further calibration was carried out by applying multiple loading cycles at different frequencies (1-10 Hz) and monitoring both signal stability and drift of the resistance.

Scanning electron microscopy (SEM) was employed to test the fracture surfaces beyond the mechanical tests as well as to relate the microstructure and graphene dispersion to the detected sensing behavior, which can enhance the fabrication process. The calibration was validated by applying complex dynamic loadings (such as low-velocity shocks and random vibrations) and comparing the recorded resistance signals with the readings of conventional strain gauge mounted on the surface to confirm the accuracy and dynamic response of the fabricated hybrid composites.

3. Results and Discussion

Figure (2) shows the behavior of the shear stress as a function of the shear strain over the range 0-2% for the fabricated composite samples at five different mixing ratios of carbon fibers and graphene platelets. This figure indicates the effect of relative composition on mechanical response under shear loadings as the shear stress clearly increases with increasing content of carbon fibers (CFs) in the composite, which leads to stable increase in the mechanical performance. Minimum shear stress of 9 MPa was recorded at minimum shear strain of 0.25% for the sample of 1:3 CFs:GNPs mixing ratio, whereas the maximum shear stress of 58 MPa was recorded at maximum shear strain of 2% for the sample of 3:1 CFs:GNPs mixing ratio. As well, at low strain values, the differences in stresses for the tested samples were very slight but became clearer at moderate and high strains.

These results reveal the crucial role of carbon fibers in enhancing shear resistance of the composite material, which means that the material's ability to hold shear stresses is reasonably enhanced with increasing the content of carbon fibers, especially at the high deformations. Carbon fibers have the ability to form a continuous network that can effectively distribute the shear stresses within the material in addition to their role in resisting crack propagation as well as increasing its hardness [43]. On the other hand, increasing graphene content (as in 1:3 sample) cannot completely compensate the decrease in carbon fibers because graphene – with its excellent properties – is not effective enough – as long carbon fibers – to form continuous supporting structure.

It is interesting that the sample of 1:1 mixing ratio showed moderate values, which got closer to the values exhibited by samples with higher carbon fibers at higher strains. For example, at strain of 2%, the sample with 1:1 mixing ratio showed 51 MPa stress, which is closer to the stress shown by the sample of 2:1 mixing ratio (51 MPa) than that shown by the sample of 1:2 mixing ratio (49 MPa). This refers to a synergetic effect at specific ratios as the two components (CFs and GNPs) are collectively functioning. As well, the uniformly gradient of values with mixing ratios confirms the possibility to control the properties of the composite material by changing the relative contents of its components, which allows the design of materials with specific mechanical properties for specific applications.

Figure (3) shows the variation in resistance ($\Delta R/R_0$) of the composite sample as a function of mechanical stress under multiple loading cycle of different frequencies (1, 3, 6, 8, and 10 Hz). A clear direct relationship between the applied stress and $\Delta R/R_0$, which uniformly increases with increasing stress from 0 to 40 MPa. This behavior reveals the ability of the fabricated composites to work as self-sensing materials as the $\Delta R/R_0$ can be used as a precise indicator for deformation and mechanical stress in the material.

By analyzing the effect of frequency on material's response, when the loading frequency is increased, the measured material's response accordingly increases. For example, at applied stress of 40 MPa, the

value of $\Delta R/R_0$ is about 0.276 at low frequency of 1 Hz, whereas the $\Delta R/R_0$ increases to 0.352 at 3 Hz, 0.47 at 6 Hz, 0.615 at 8 Hz, to reach its maximum of .664 at maximum frequency of 10 Hz. This behavior can be interpreted by the charge transport dynamics within the composite material since the high frequencies may lead to much more effective rearrangement of the electrical conducting network composed of carbon fibers and graphene platelets that enhance the material's sensitivity to the variations in mechanical stress.

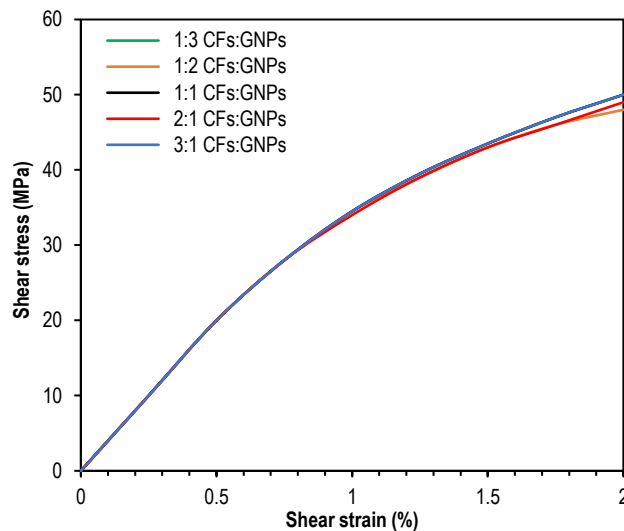


Fig. (2) Shear Stress-strain curves for the prepared samples with different mixing ratios of CFs:GNPs

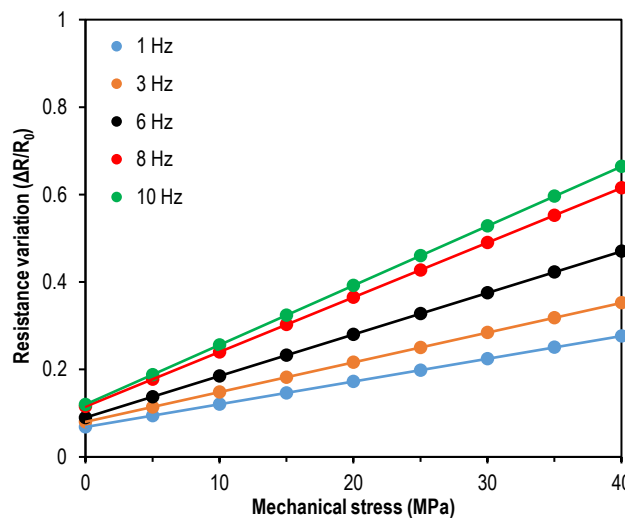


Fig. (3) Variation of electrical resistance with mechanical stress applied to sample at different frequencies of multiple loading cycles (the slope is the gauge factor, GF)

The slope calculated at each frequency is the most indicative value to express the rate of resistance variation with stress (i.e., material's sensitivity). This value increases with increasing frequency that quantitatively confirms that the material's sensitivity is enhanced with increasing loading frequency. This increase is not completely linear as a specific leap is observed when the frequency is increased by 40% (from 3 to 6 Hz) then continued at high rates. This refers to a critical frequency at which, the electrical response of the material may start to change.

In practical applications, these results presents valuable information to design smart sensing devices based on the fabricated composites as the selection of suitable loading frequency can reasonably enhance the sensing performance. If the application requires as high as possible sensitivity, operating at a frequency of 10 Hz may be the optimum as it produces the best response, while low frequencies may be suitable for applications requiring higher stability and lower energy consumption. With an

approximately linear relationship between the mechanical stress and variation in resistance at certain frequency, the calibration of these composites as sensing materials would be easier. This reliable linear behavior and controllable high sensitivity via variable frequency make these hybrid composite materials promising candidates for the SHM and smart system applications requiring precise sensing with real-time mechanical stresses under various dynamic loading conditions.

4. Conclusions

In concluding remarks, increasing the content of carbon fibers in the composite leads to an observable enhancement in shear resistance and shear stress particularly at high strains due to their ability to form a continuous network that support the distribution of stresses and limit the propagation of cracks within the material, whereas increasing graphene content solely does not compensate the reduction in fibers' content. A synergetic effect can be observed with balanced mixing ratio that allows the precise control of mechanical properties throughout the modification of structure. A linear relationship between stress and relative variation in resistance can also be observed with consequent increase in sensitivity with frequency of the multiple loading cycle, which confirms the feasibility of these composites for smart sensing and structural monitoring applications.

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