RATE DEPENDENT ELECTROMECHANCAL CHARACTERIZATION AND MODELING OF GRAPHENE BASED FIBER REINFORCED POLYMER LAMINATES

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Abstract: *Fiber reinforced polymer (FRP) composite laminates are used in numerous structures that require high strength-to-weight ratio. The health status of these laminates is traditionally monitored using point strain gauge arrays, fiber optics etc. installed at critical locations. In this work, a composite laminate capable of sensing its own health has been developed using embedded fabric sensor. In this study, a manufacturing protocol is developed for in-situ reduction of graphene oxide (GO) coated fabric into rGO coated fabric. A vacuum assisted resin transfer molding process was used to fabricate the composite laminates with embedded rGO coated fabric sensors. The piezoresistivity of the composite laminate was measured both before and during fabrication. The in-plane tension tests were carried out at three different loading rates (0.2, 2, 20 mm/min) to determine the rate-dependent piezoresistive response of composite laminates. We recorded both the piezoresistivity and load-displacement data simultaneously to obtain the electromechanical response of the fabricated samples.*

Keywords: Graphene; rate dependency; fiber reinforced polymer; electromechanical; characterization

1. Introduction

The novel applications of graphene associated nanomaterials (GANs) to fabricate multifunctional structures have significantly increased over the years owing to their favorable properties such as higher thermal conductivity, higher electrical conductivity, and better electromagnetic interference shielding, etc. [1–4]. Electrically conductive fiber reinforced polymer (FRP) composite is one of such examples which is used for sensing purposes by introducing GANs into the FRP composite. In such case, the electrical networks formed by the GANs are not static and evolve continuously with external pressure and heat which imparts piezoresistivity to the structure. There are various manufacturing approaches used in the literature to introduce GANs into the FRP composite. One of the most important guidelines to keep in mind is how and where to add nanomaterials to get the desired performance, which ultimately determines the overall manufacturing process. As a prerequisite for scalability, the proposed manufacturing process should be able to be adapted to existing methods of manufacturing [5].

Conductivity of the FRP composite is enhanced by the addition of nanomaterials to the polymer matrix. This approach faces some serious challenges during manufacturing, such as an increase in viscosity that causes difficulties during infusion. Further, nanomaterials can also be filtered by the mesh nature of the fabric reinforcement and distributing media mesh that is generally used as consumables. In addition, nanoparticles in a liquid medium agglomerate over time, impairing

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the even distribution of nanofillers in the matrix and the resulting multifunctionality [6–8]. As a result, serval alternative manufacturing techniques are preferred by depositing the nanofillers on the fabric reinforcement to form continuous electrical networks. These techniques comprise spraying, dipping/solution method, chemical vapor decomposition (CVD), and electrophoretic deposition, etc. [6, 7, 9, 10].

In this work, graphene based FRP composites have been manufactured and electromechanically characterized at various loading rates during in-plane tension. In the as received state, the plain weave E-glass fabric was coated with graphene oxide (GO) which was subsequently reduced to reduced graphene oxide (rGO) for better electrical conductivity. The coated fabric was then embedded within the laminate at a symmetric position. The piezoresistive laminate was manufactured using a vacuum assisted resin transfer molding (VARTM) process. The samples were prepared in accordance with the ASTM standard for FRPs in-plane tension testing. A simultaneous mechanical and piezoresistive response was measured during the mechanical testing at different load rates.

2. Materials

Graphene oxide (GO) paste was supplied by Abalonyx AS, Norway in form of aqueous solution. A Plain woven E-glass fabric with an aerial density of 202 g/m^2 provided by Gurit®, UK was used as reinforcement and substrate for GO coating by solution dipping method. For all the electrical connections commercially available copper tape and wire were used. In the VARTM process, epoxy resin and hardener (GURIT PRIME™ 20LV resin and PRIME™ 20 hardener) were mixed at a ratio of 100: 28 by weight.

3. Experimental

3.1 Specimen preparation

The samples were manufactured according to the ASTM D3039 standard. Accordingly, the in-plane tension test sample length and width were 200 mm and 20 mm respectively. The rGO-coated fabric was placed in a symmetrical position within the laminates consisting of eight plain woven glass fabrics. After that, VARTM process was used to fabricate the required laminates. The final laminate average thickness was measured as 1.7 mm. Throughout the VARTM process, resistance change was measured on each sample. E-glass tabs having a thickness of 1.7 mm each side were also attached with the help of cold compression to the samples for firm gripping and transfer of load during in-plane tension experiments. Finally, the samples were painted appropriately with speckle patterns to acquire a complete strain field using digital image correlation (DIC). Figure 1 illustrates the steps involved in fabricating in-plane tension test samples.

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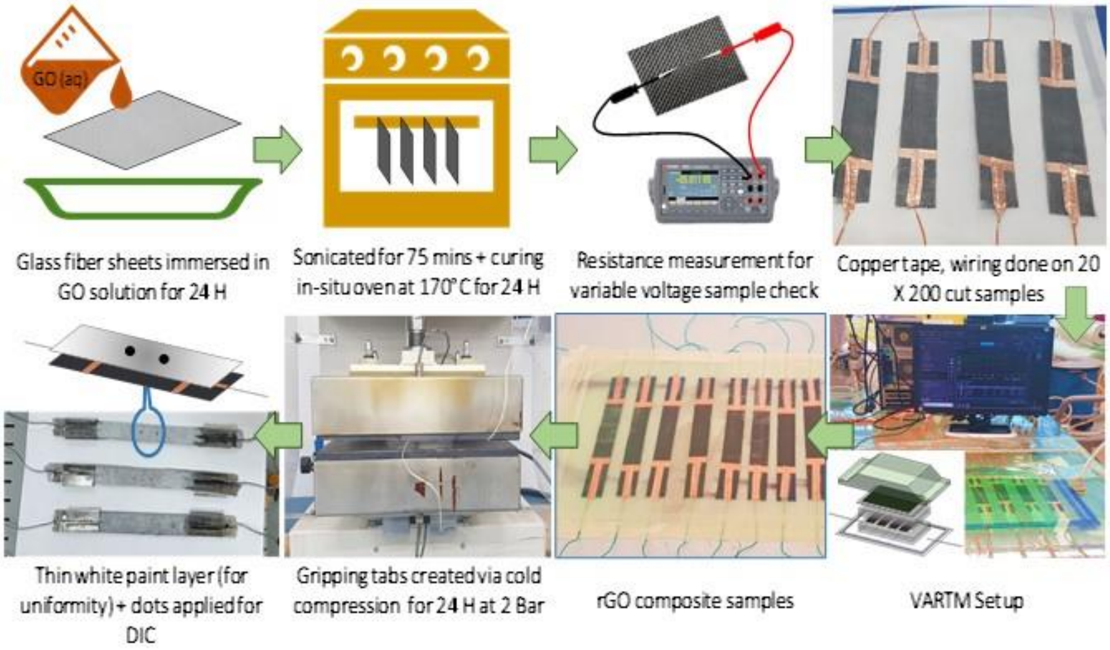

Figure 1. Steps followed in the fabrication of samples: starting from the raw materials until the final specimens.

3.2 Experimental testing procedures

3.2.1 In-situ piezoresistive measurement

Piezoresistivity is expressed as a fractional change in resistance (FCR) which was monitored during the various stages of the VARTM process including vacuum bag compaction, resin flow, and finally curing and post curing. For this purpose, BenchVue acquisition software was used along with the Keysight DAQ970A data acquisition system (DAQ) to display real time readings. It measures the instantaneous resistance change caused by the compaction of the vacuum and resin flow pressure in each sample placed transversely to the resin flow, as shown in Figure 1. The instantaneous resistance R is normalized by the characteristic resistance R_o measured prior in the absence of any external excitation (stress or strain) to compute FCR as given by Eq.(1):

$$FCR = \left(\frac{R-R_0}{R_0}\right) \times 100 \tag{1}$$

3.2.2 Electromechanical in-plane tension test

For all tension tests, the Instron 5969 with load cell capacity of 50/5 kN equipped with built-in DIC system was used. The in-plane tension tests were carried out at three different loading rates (0.2, 2, 20 mm/min) to determine the rate-dependent piezoresistive response of composite laminates. We tested three samples at each load rate. The electromechanical characterization test setup is shown in Figure 2 which records both the mechanical load vs. displacement data and resistance, simultaneously.

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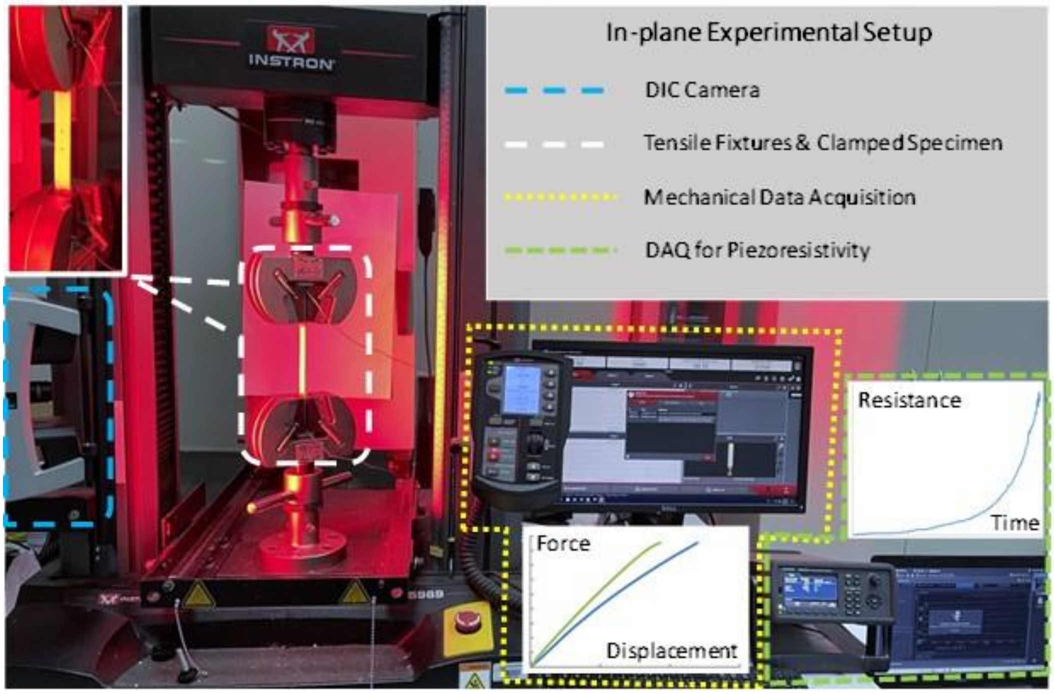
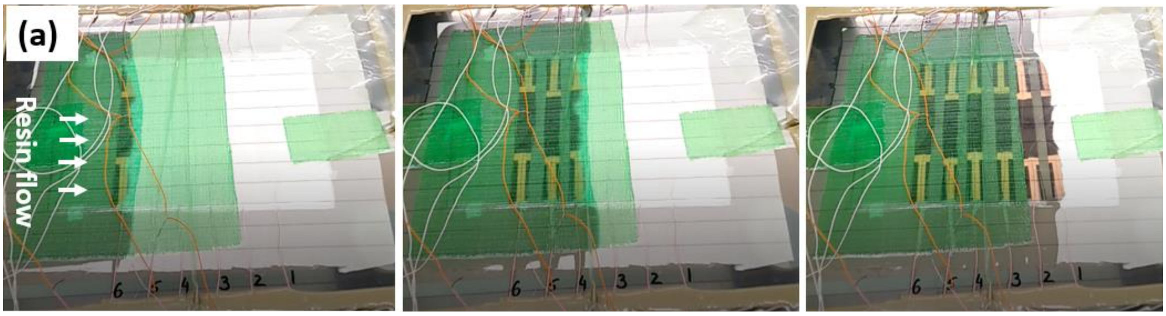


Figure 2. Electromechanical in-plane characterization test setup.

4. Results and discussion

4.1 In-situ VARTM piezoresistivity measurement

The instantaneous resistance R was measured for the rectangular sensors during the different stages of VARTM process such as vacuum compaction, resin flow and final curing as given in Figure 3. The positions of all six samples and resin flow fronts can be seen in Figure 3(a) where sample 6 is nearest to the resin inlet. The FCR evolution of all the samples showed a sudden drop in the percentage change upon vacuum application and resin flow over the rGO coated sensors. The vacuum application and resin flow compressed the rGO coated fabric sensors which in-turn decreased the inter-yarn gaps. Hence, this compaction enhanced the electrical network and decreased the resistance R. The maximum decrease was noted for the sensor located near the inlet having a value of 63.5%. This may be associated to the position of the sample in mold. After completing saturation of the fabric with resin and entry of the excess resin into the outlet duct, the resin entry was stopped by clamping at approximately 26 min. The differences in the FCRs of all samples in Figure 3 may be due to the minor variation in rGO coating.



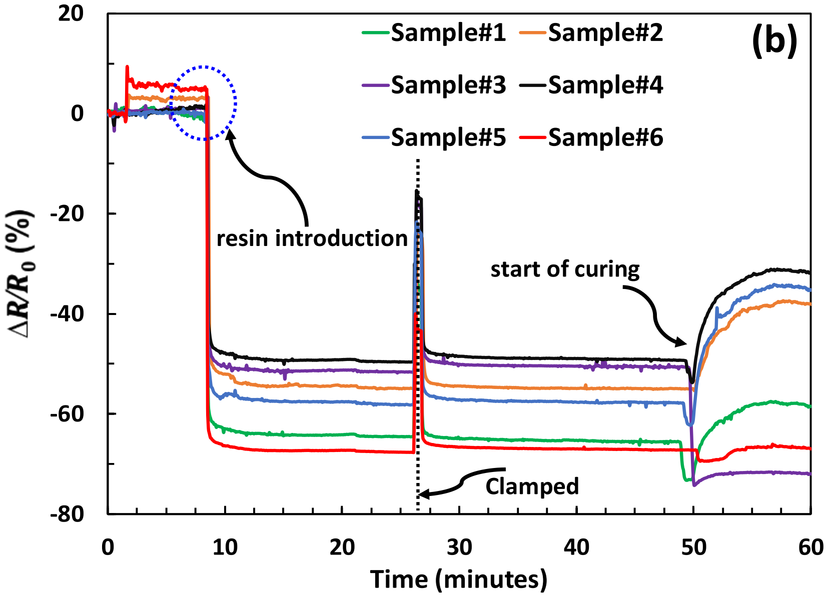


Figure 3. In-situ piezoresistivity measurement during the VARTM process, (a) resin flow fronts at sample 6, sample 4 and sample 1, (b) FCR measurement vs. time for six sensors.

4.2 In-plane tensile electromechanical characterization

Three samples per load rates (0.2, 2, 20 mm/min) were tested to record the scatter in the piezoresistivity of the developed sensors. The average stress (σ) vs. average strain (ε) in the fiber direction and the respective changes in the resistance are compared for the three load rates, as shown in Figure 4 (a). As the load rate is increased, the stress-strain behavior demonstrated the strain-hardening behavior which is very well documented for metals as well as FRP composites.

The piezoresistive behavior in the in-plane tension experiments were found in agreement with other researchers in similar experimental setup [10]. The FCR graphs started to rise with low slope within the strain of 0.5%. After that, a significant increase in FCR was observed as the strain increased beyond 0.5% in the elastic strain limit of the rGO coated fabric sensor. There is also a possibility of microscale partial localized fibers damage causing discontinuation in the electrical network. The end points of each stress-strain plot denote the catastrophic brittle fracture of the number of fiber yarns or the complete ply known as macro-level damage (see fractured samples in Figure 4-b). Upon this macro-level damage, a substantial increase in the FCR can be seen which is expected showing the overall discontinuation of the conducting network in the continuous rGO coated fabric sensor. Such piezoresistive behavior can be expressed with simplified exponential growth functions.

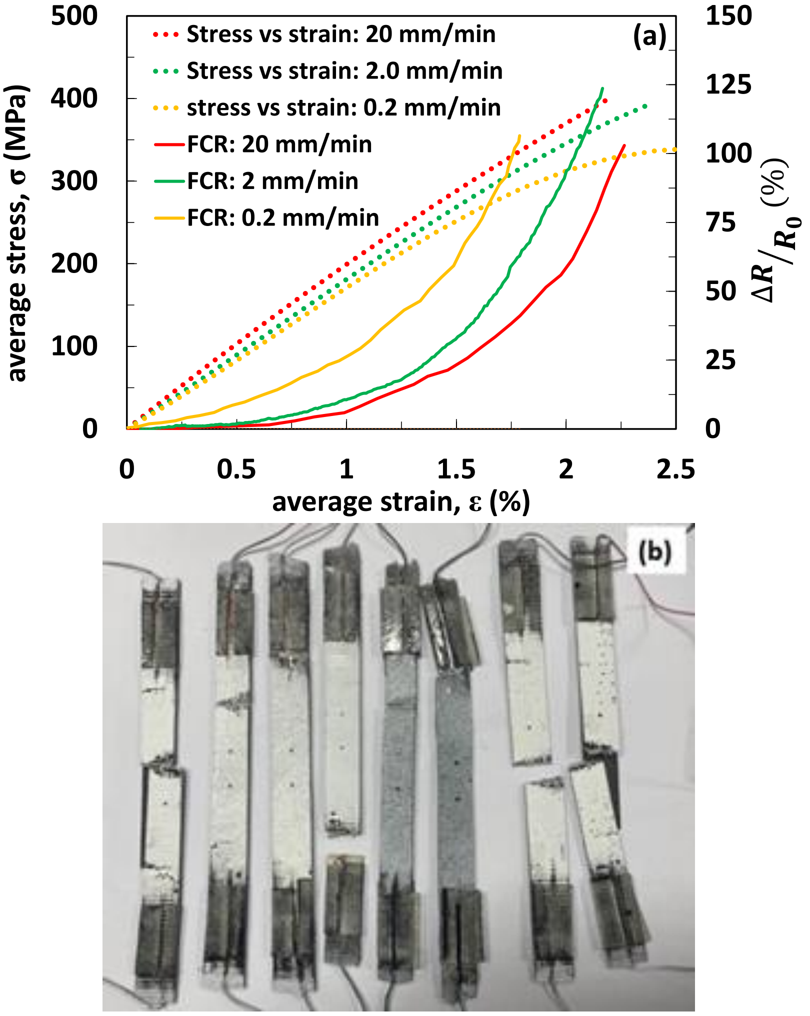


Figure 4. Simultaneous recordings of in-plane stress-strain in loading direction and FCR (a) comparison of the three load rates of 0.2, 2.0, and 20 mm/min, (b) some of the post-test fractured sensors.

The piezoresistive sensitivity for the three load rates were compared by determining the gauge factors: $k = (\Delta R/R_0)/\varepsilon$. These gauge factors (k) were calculated for the three load rates in the moderate slope region. Tangent lines were drawn to the exponential functions fitted in between the experimental upper and lower bounds given in Figure 5 (a) at strain of 1%. The percentage error based on the upper and lower bounds of the failure stress and FCR are shown in Figure 5 (b). The fitted functions describing the average FCR is given by Eq.(2).

$$FCR = (a + be^{\beta\varepsilon}) \times 100 \tag{2}$$

The corresponding constants and gauge factors are summarized in Table 1. Eq.(2) was constrained to $a = -b$ during fitting such that the initial conditions of FCR at $\varepsilon = 0$ must be satisfied. The slowest load rate of 0.2 mm/min resulted into a maximum gauge factor of 53 with respect to the other two cases. It can be noticed that the load rate is in inverse relation with the gauge factor.

Table 1: Fitted parameters and computed gauge factors

Load rate	(a)	(b)	(β)	Gauge factor

mm/min				(k)
0.2	-6.8	6.8	1.6	53
2.0	-2.3	2.3	1.9	29
20	-2.7	2.7	1.6	21

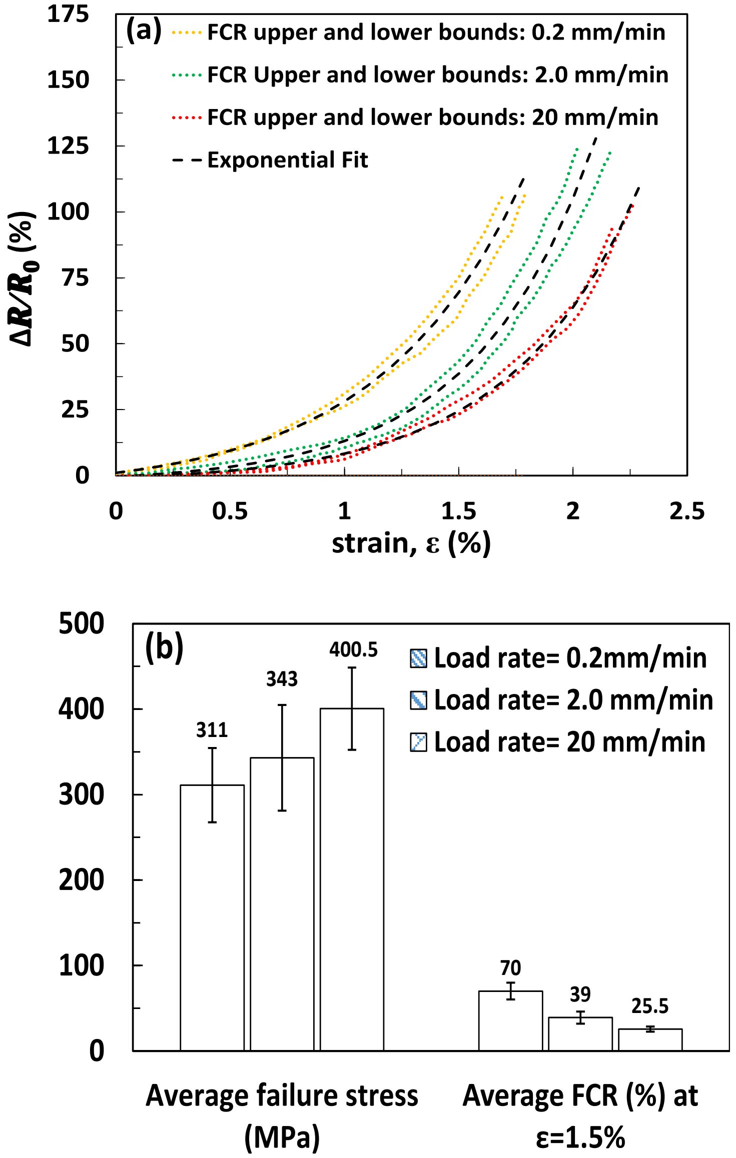


Figure 5. (a) Exponential growth model representation, (b) Fractured strength and FCR with percentage error.

5. Conclusions

Graphene enhanced coated fabric is inherited with obvious advantages to be embedded as sensor in advanced multifunctional fiber reinforced polymers composites as compared to the mixing of nanofillers in the matrix polymer material. Complete fabrication process of such multifunctional FRP composite was demonstrated by manufacturing samples for in-plane tension tests using the VARTM process. The rGO coated fabric sensors were found capable to capture the various stages of the VARTM for process monitoring such as vacuum compaction, resin entry and flow, curing, and post-curing. Then, electromechanical characterization in in-plane tension tests were completed at three load rates of 0.2, 2.0 and 20 mm/min to evaluate the load rate dependency of piezoresistivity. The patterns of the piezoresistivity observed during the experiments were represented with exponential growth model. Besides, gauge factors were computed for each load rate in the elastic region. During in-plane mechanical tension tests, it was concluded that piezoresistivity and gauge factor are inversely proportional to the load rate.

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