GRAPHENE COATED SMART FABRICS FOR VARTM PROCESS MONITORING

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ABSTRACT

The flow of resin must be monitored online using different types of sensing techniques in liquid composite moulding process. In this work, the piezo resistive nature of graphene coating is exploited for the flow monitoring and pressure measurements. The utility of the measurement process is demonstrated through several experiments. A non-conductive substrate, a 3D woven glass fabric, has been coated with conductive reduced graphene oxide and was subjected to mechanical compression, vacuum compaction and an actual VARTM cycle. The coated substrates become conductive with a finite resistance which changes under the applied loads and during the infusion process. The sensitivity of the coating is quite advocate for the monitoring the whole process. The use of graphene for monitoring an LCM process was successfully demonstrated. From the mechanical compression of the reinforcement to resin impregnation process, each step can be monitored using coated glass fabric.

1 INTRODUCTION

Liquid composite moulding (LCM) process involves impregnation of the resin into the fibrous reinforcement with the goal to saturate all the empty space between the fibres with the resin before the resin starts to cure. Usually thermosetting resins are used and the reinforcement impregnation is achieved through pressure driven flow, with either positive or negative applied pressure, or even both. These processes provide good control over harmful volatiles generated by thermoset resins, thus complying by environmental regulations and standards. The final fibre volume achieved can be higher and more consistent as compared to with the traditional open mould techniques. The LCM process can be automated reducing the labour cost, production time and waste produce. Based on minor differences, LCM processes are categorized as Resin Transfer Moulding (RTM), Vacuum Assisted Resin Transfer Moulding (VARTM), Seemann's Composite Resin Infusion Moulding Process (SCRIPM), Structural Reaction Injection Moulding (S-RIM), and so on [1].

RTM processes consist of a mould cavity that is in the shape of the part to be manufactured. The fibre preform is placed in the cavity. The mould is closed and clamped or held under pressure in a press. The resin is injected into the compressed preform through one or more gates from a pressurized container. Once the mould is full, the injection is discontinued and the resin is allowed to cure. The mould is open once the cure is complete or the part is sufficiently hardened to be demoulded. The process is explained in Figure 1. VARTM and SCRIMP are slight modifications of the RTM process where the top half of the mould is replaced by a vacuum bag. In VARTM, the reinforcement must be contained in an enclosure where vacuum can be created. This is achieved by covering the preform with sealed vacuum bag containing inlet and outlet vents. Initially, the fibrous reinforcements are laid on the mould to form the preform. A consumable item such as Nylon® fabric, is then laid over the preform which is peeled off after curing, allowing for easy separation of the consumables from the part, and a reasonable surface on the side of the part not in contact with the mould. Distribution media can be laid over the peel ply to enhance resin flow if the preform has low in-plane permeability. Once inlet and vent tubes are placed, the mould is closed using a vacuum bag sealed with sealant tape. With the enclosure sealed, the inlet is clamped and vacuum is applied to the outlet vents. Once full vacuum is achieved, the inlet is opened and the resin penetrates the preform. Once the resin front reaches the

end of the preform, the inlet is usually clamped. Excess resin is removed and the resin pressure is allowed to equilibrate within the enclosure. Once the resin is fully cured, the vacuum is released and the part is lifted [1]. The process is explained in Figure 1.

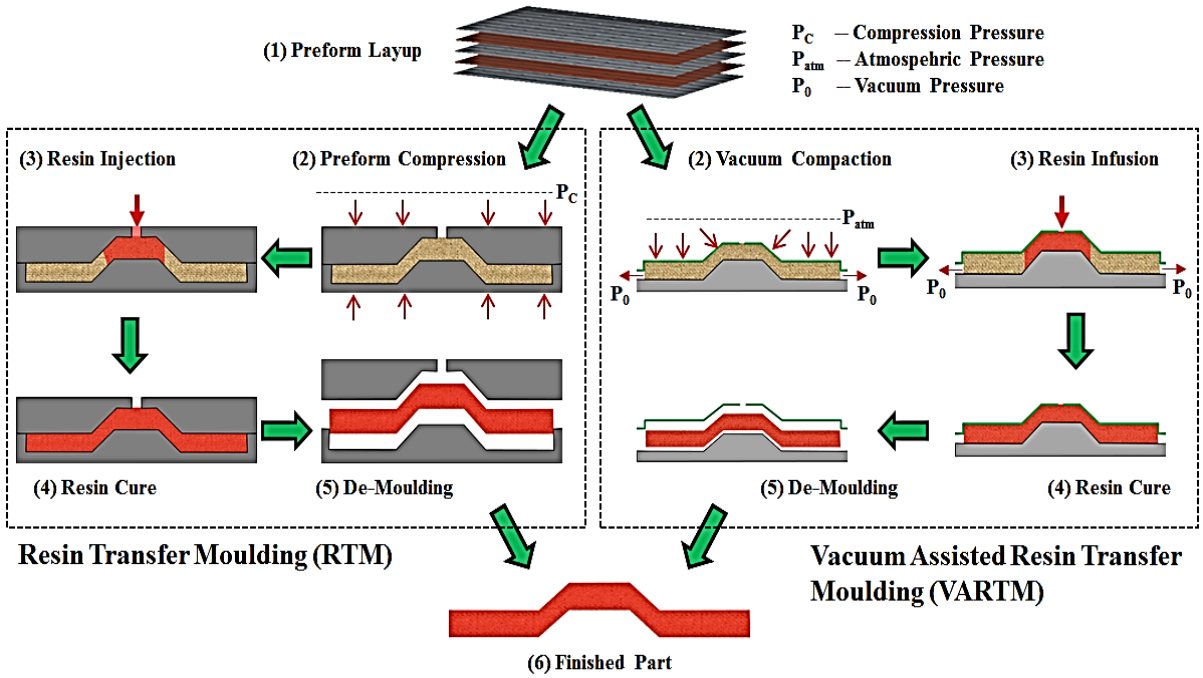

Figure 1: An illustration of RTM and VARTM manufacturing processes.

The quality of the finished product is heavily dependent on the flow process. During processing there is a little information available about the advancement of the flow front. Knowledge about the flow front advancement can help in devising control methods for the process. The pressure variation within the enclosure needs to be known to assess the whole process. The monitoring of the resin infusion process is a challenging task as we need a large number of sensors at various locations. Visual aids in the form of still images and videos can be helpful in this regard. Three-dimensional digital image correlation technique can not only monitor the thickness evolution but also can give a full-field distribution of the thickness change of the laminate [2-5]. The sensors are based on changes in properties, such as electrical conductivity [6], acoustic emission [7] and heat flux [8].

Here we present the use of graphene coating for monitoring an LCM process through a series of experiments. Graphene, graphite, carbon nanotubes and fullerenes have proven extraordinary mechanical, electrical, optical, thermal and magnetic properties. Recent progress has shown that the graphene-based materials can have a profound impact on electronic and optoelectronic devices, chemical sensors, nanocomposites and energy storage [9-14]. The high pressure sensing gauge of graphene has made it highly desirable in wearable electronics, human/machine interfaces, soft robotics, MEMS, artificial intelligence and beyond. Recently, we have developed a simple and scalable application of graphene coated substrates for pressure sensing [15-17].

A thin coating of conductive reduced Graphene Oxide (rGO) has been applied on a substrate as the sensor making the whole substrate electrically conductive. From the mechanical compression of the fibres to the resin infusion process, each step is monitored using the graphene coating. The processes are characterized in terms of the change in the relative resistance of the coated material used. Development of a cost efficient sensing technique for the flow monitoring will accelerate the technology readiness level of VARTM. The results showed use of graphene coating for monitoring and characterization of an LCM process is a novel solution. From the mechanical compression of the fibres to the resin infusion process, each step can be monitored using the graphene coating. The sensitivity of the coating is quite advocate for the monitoring of the whole process.

2 EXPERIMENTAL SETUP

2.1 Materials and Methods

Graphene oxide (GO) was synthesized from pre-treated graphite powder with a particle size of 20 µm following a modified Hummer's method [18]. The coating process started by dissolving graphene oxide in distilled water to form a solution (3g/200 ml). The sheets of glass fabric were soaked in the solution overnight and then dried in a controlled environment at 80 °C. After the GO was deposited on the samples, it was reduced with HI acid (57 % wt. distilled). The samples were rinsed thoroughly to remove any excess HI acid. The samples were then dried again in a controlled environment at 80 °C. The coating process is repeated until uniform coating throughout the fabric is achieved; usually 1 to 2 times repeat is sufficient. The whole coating process is explained in Figure 2.

The purpose of these experiments is to demonstrate the suitability of graphene-coated fabrics as piezo-resistive sensors for LCM process monitoring. A 3D woven orthogonal glass fabric (TG54N60C) supplied by Texonic®, Canada, was used as a reinforcement to demonstrate the applicability of the concept. The 3D woven fabric has an areal density of 1790 g/m^2 and a thickness of 2 mm. Dow Corning® Silicone oil (viscosity = 0.1 Pa.s, density =1.06 g/cm^3) was used as a test fluid to avoid any interaction between the dielectric behaviour of the resin and its cure behaviour on fabric conductivity during processing.

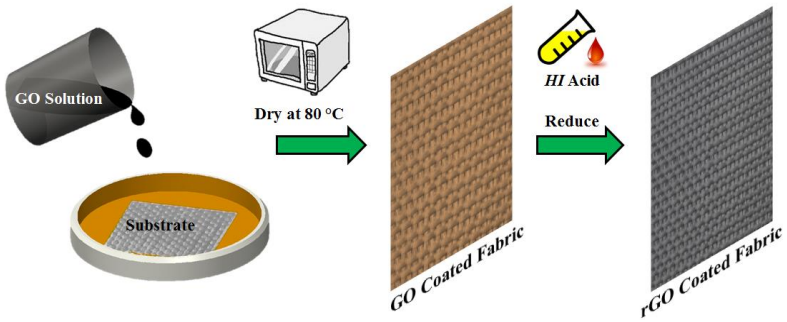

Figure 2: Schematic of rGO coating process on pristine fabric.

2.2 Setup and Procedure

An Instron 5969 universal testing machine with a 5 kN static loading capacity, was used for the mechanical compression tests. A 25 mm x 100 mm rectangular rGO coated glass fabric sample was placed on a 150 mm diameter round compaction fixture. The sample was placed on the bottom stationary platen of the testing frame. Two electrical connections were taken from the longer side of the sample. A linear force profile with an increment of 2 N/min was applied to the samples to give a maximum force corresponding to a stress of 100 kPa (equivalent to full vacuum). After reaching the target force, the unloading cycle was initiated. The total resistance change was recorded using data acquisition hardware (NI ELVIS II) in LabVIEW, and the load vs. deflection curve was recorded using the Instron data acquisition system. The experimental set-up is shown schematically in Figure 3.

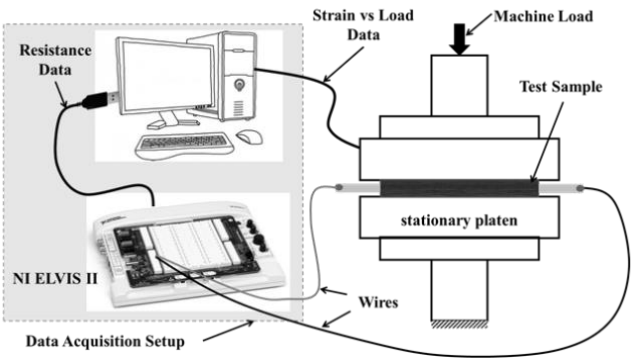

Figure 3: Schematic diagram of the mechanical compression test setup.

A 400 mm x 100 mm rGO coated glass fabric sample was compacted under vacuum. The purpose of this test was to obtain resistance values corresponding to vacuum levels observed during an actual LCM cycle. The reinforcement was laid on a glass workbench that had been cleaned using acetone. A vacuum bag was used to cover the sample which was sealed using tacky tape. Two electrical connections were taken from the shorter side of the sample. The overall resistance change was measured continuously by applying the vacuum pressure in four increasing steps, starting from 0% vacuum to 100% vacuum and backwards, and holding the vacuum at each step for 2 minutes, so that pressure stabilizes to a steady state value. The applied stepwise vacuum compaction is shown in Figure 4 (a). Multiple loading-unloading cyclic compactions were also applied to the fabric, starting from atmospheric pressure to full vacuum and back, shown in Figure 4 (b). The pressure was held constant during each step for approximately 2 minutes. In resin infusion, during pre-filling, a vacuum is applied, and as the pressure differential between the mould cavity and atmospheric pressure increases, the reinforcement is subjected to a 'dry compaction' (the dry preform is compacted to a volume fraction higher than the volume fraction at zero stress) [19]. A similar procedure was adopted using a 'wet' reinforcement that had been previously soaked in silicone oil.

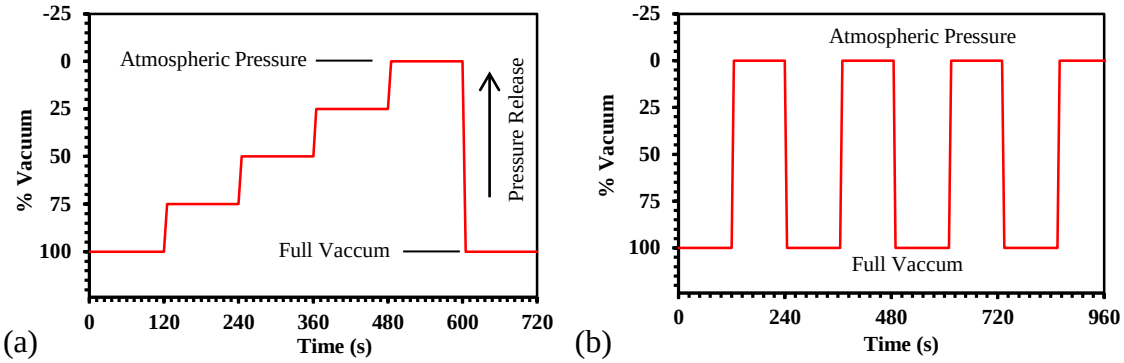

Figure 4: Time history of the applied vacuum for the (a) stepwise and (b) cyclic compaction.

The same sample geometry and experimental set-up used for vacuum compaction was utilized for the infusion process. The rGO coated glass fabric sample was infused with silicone oil using the resin infusion manufacturing technique. Electrical connections were attached at each end of the fabric to measure the electrical resistance. In order to ensure a uniform distribution of fluid across the width of the fabric, a distribution medium was used at both the inlet and outlet gates. The silicone oil was de-gassed for about 5 minutes to remove any entrapped air bubbles. The vacuum pump was operated continuously to maintain the desired pressure all the time. The fabric was covered with a vacuum bag and sealed with tacky tape. A schematic of the experimental arrangement for the compaction calibration and resin infusion process is shown in Figure 5 (a). The resulting change in the electrical resistance of the sample was monitored continuously using a National Instruments ELVIS II data acquisition board interfaced with an impedance analyser in LabVIEW.

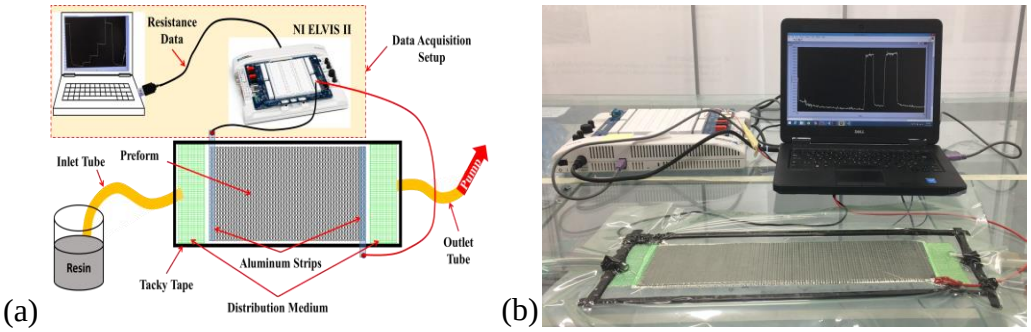

Figure 5: Vacuum compression and resin infusion set-up (a) schematic and (b) actual.

3 RESULTS AND DISCUSSION

The results of the four experiments are discussed here. The relative change in resistance ($\%\Delta R/R_0$) was adopted as a measure of the piezo-resistivity of the fabric sensor. The change was calculated with an initial value R_0 as a reference.

3.1 Mechanical Compaction

As the load increased, the electrical resistance of the reinforcement started to drop. When the load reached the maximum value, the total drop in resistance was approximately 22.5%, relative to the initial resistance of 44.8 kΩ. The relative change in resistance has been found to be inversely proportional to the strain resulting from the applied load, as shown in Figure 6 (a). When the load was removed, the resistance reverted back to a value close to that at the outset. A hysteresis loop is observed during the unloading phase, due to the fact that the fabric was not allowed to relax. Full recovery was not possible, due to some permanent deformation or de-bulking, wherein fibre tows collapsed due to an inter-tow nesting effect. The observed behaviour is purely piezo-resistive in nature and can be explained in terms of the mechanical deformations in the fibre resulting from the applied load. The piezo-resistive effect is a result of changes in the conducting paths within the material and the contact resistance between neighbouring conducting regions [17].

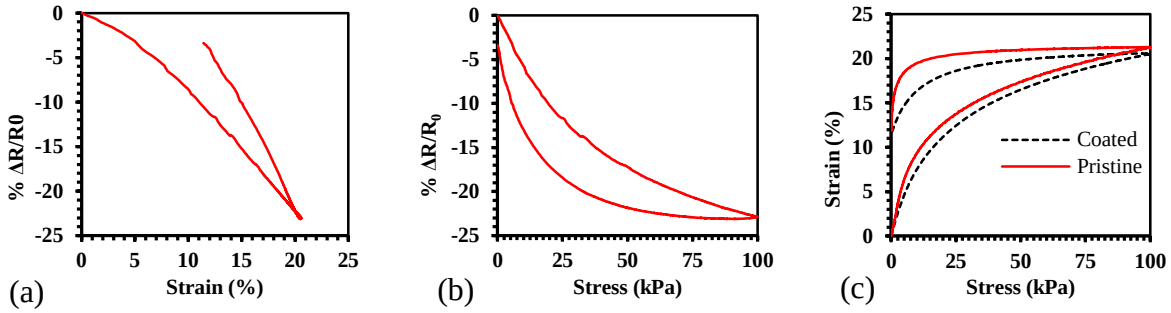

Figure 6: Mechanical compression test results (a) relative resistance change vs. strain, (b) relative resistance change vs. stress and (c) strain vs. stress of both coated and pristine fabrics.

3.2 Vacuum Compaction

The results from the stepwise vacuum compaction tests are shown in Figure 7 (a). A release in pressure results in a change in resistance. When the pressure was released from 25% (25 kPa) vacuum to 0% vacuum (atmospheric pressure), the change in relative resistance was significant (a jump from 15% to 28% in relative resistance), when the pressure was released; the resistance instantly goes to its initial value. This can be explained in terms of the relaxation capability of the fabric at different pressure levels. The repeatability of this change in resistance with release of vacuum pressure is shown in Figure 7 (b). The results highlight a very small difference between the wet and dry compaction responses, suggesting that the oil has a negligible interference in the internal structure of the coated fabric.

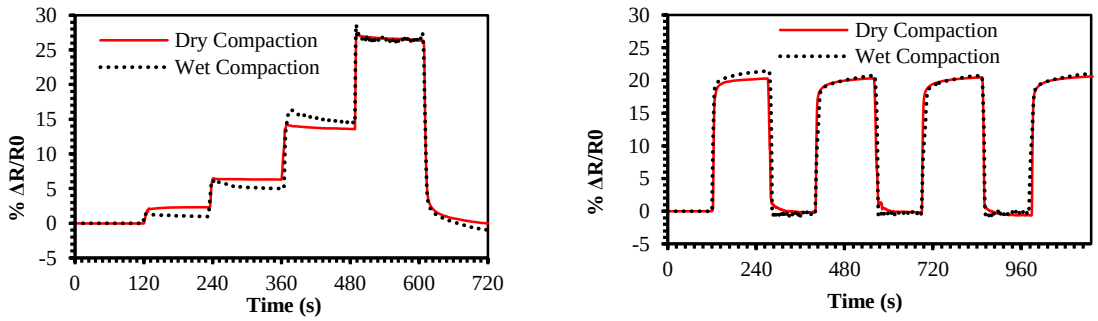

Figure 7: Response of rGO coated glass fabric during (a) stepwise and (b) cyclic vacuum compaction.

3.3 Oil Infusion Process

The infusion process started with preform compaction, after which silicone oil was infused under full vacuum. As soon as the fluid enters the reinforcement, a change in resistance is noted. As the flow front progresses with time, the resistance keeps increasing. The pressure gradient inside the reinforcement changes from full vacuum to atmospheric pressure, which is called the spring-back phenomena of the reinforcement. Figure 8 shows the resistance evolution as the pressure distribution inside the preform changes from full vacuum to one atmosphere. Once the preform was filled with fluid, the injection line was clamped, as a result, the resistance started to fall. This is called the post-infusion phase, during which the pressure gradually decreased. After some period, the pressure stabilized, as did the resistance. Since the silicone oil has negligible dielectric properties hence, the change in resistance is purely a result of the interaction of the fluid and the fibers.

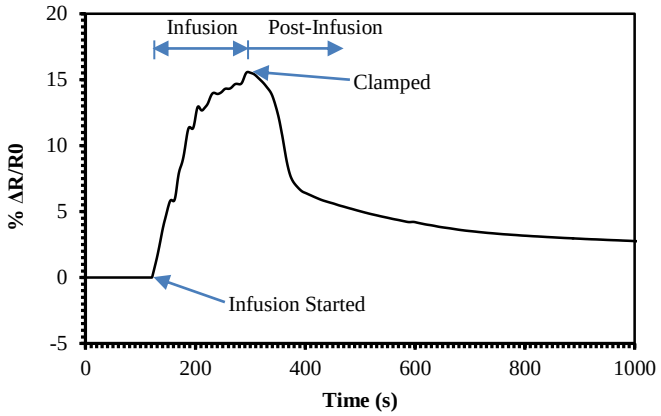

Figure 8: Resistance change during the infusion process of rGO coated glass fabric with silicone oil.

4 CONCLUSIONS

As compared to traditional point sensing technique, the use of graphene coatings on fabrics for monitoring the LCM process has been successfully demonstrated. A non-conductive substrate, a glass fabric has been coated with reduced graphene oxide and subjected to mechanical and vacuum compaction tests. The coated fabric was used in an infusion process using electrically inert injection fluid. The coating has been shown to be sufficiently sensitive for monitoring the entire LCM process. The graphene-coated fabrics exhibited an electrical resistance that changed under applied loading and during mold filling. Here, mechanical compression of the reinforcement and the subsequent oil infusion process were monitored using the graphene-coated “smart” fabric.

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