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Graphene coated piezo-resistive fabrics for liquid composite molding process monitoring

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ABSTRACT

In this study, the graphene coated piezo-resistive fabrics have been exploited for liquid composite molding process monitoring. The utility of this novel technique has been demonstrated through compaction and flow monitoring experiments. The coated fabrics are subjected to a series of compaction tests to monitor resistance changes during the compaction cycle. During mechanical compression, the change in resistance has been found to be inversely proportional to the strain associated with the applied load. The repeatability of the change in the electrical resistance is confirmed via a series of vacuum assisted, stepwise and cyclic compression tests. The overall sensitivity of around 30% change in resistance is observed. The results highlight very small differences between the wet and dry compaction cycles when using silicone oil as the test fluid, suggesting that a nonconductive fluid offers negligible interference in the graphene coatings. The fabrics are subsequently used in a full resin infusion cycle to monitor the resistance change during the filling and post-filling stages. A continuous change in the resistance of the fabric is observed during, and after resin infusion process, highlighting the applicability of this novel technique for full process monitoring during the compaction and resin flow stages, in addition to cure monitoring.

1. Introduction

Liquid Composite Molding (LCM) processes are cost-effective Out-of-Autoclave (OoA) manufacturing techniques used for fiber reinforced composite structures. These processes are finding applications in industries such as those associated with the aerospace, automotive, and marine sectors [1]. The term LCM belongs to a family of manufacturing processes that includes vacuum assisted resin transfer molding (VARTM) a.k.a. resin infusion, where the clamping force is applied using vacuum pressure under a flexible tooling. Therefore, controlling the final part thickness is not as straightforward as it is in the resin transfer molding (RTM) process, where the preform is compacted between two rigid mold tools [2]. However, resin infusion is suitable for larger parts, where surface finish is not an issue. The LCM process can be automated, reducing labor costs, production time, and waste [1,3–5]. In a generic LCM process, a low viscosity thermosetting resin is injected under pressure into a mold containing the fiber reinforcement. The applied pressure could be either vacuum or positive pressure or indeed both.

During resin infusion, the reinforcement must be contained in an enclosure where the vacuum can be created. This is achieved by covering the preform with a sealed vacuum bag containing both inlet and outlet vents. Initially, the fibrous reinforcements are laid on the mold to form a “preform”. A consumable item, such as a nylon peel ply, is then laid over the preform and subsequently peeled off after curing, allowing for easy separation of the consumable materials from the part, and a reasonable surface finish on the part that is not in contact with the mold. During the resin infusion process, a complex interaction of preform compaction and component spring-back occurs as the resin flow front advances and creates a pressure gradient across the preform. Once the resin front reaches the end of the preform, the inlet is clamped, excess resin is removed and the resin pressure is allowed to equilibrate within the

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mold. Once the resin is fully cured, the vacuum pressure is released and the part is lifted. Local preform compaction during infiltration involves a combination of wetting and spring-back deformations [6]. Given that the vacuum bag employed during resin infusion provides minimal rigidity, the preform thickness varies in relation to the resin pressure inside the cavity, as does the local permeability. The final part quality or thickness is often not consistent, due to these complex interactions. Govignon et al. [6,7] developed a complex online full-field monitoring system that could track the cavity thickness during mold filling and post-filling.

In the RTM process, there is little information available about the advancement of the flow front, due to the presence of rigid tooling on both sides. Knowledge regarding flow front advancement can help in devising control methods for the manufacturing process and also help avoid race-tracking of resin along the edges of the preform. This race-tracking phenomenon can, in turn, result in the presence of large dry spots and voids within the part. In order to improve the process and ensure part reproducibility, it is important to establish adequate flow monitoring using a control system that can detect flow front progression, whilst giving information on the mold clamping forces and pressure distribution in the mold. Monitoring of resin advancement is a challenging task, since a large number of point sensors are required at different locations [8–12]. The sensors can be used to monitor changes in properties, such as electrical conductivity [13] and heat flux [14]. Changes in these properties can be readily measured and recorded. Visual aids, in the form of still images and videos of the resin infusion process, can also be helpful in this regard [7,15]. All the above-mentioned techniques are very expensive and may add considerable cost to the final part.

The development of a cost-efficient sensing technique for flow monitoring will accelerate the technology readiness level of any part manufactured using an LCM process. Recent progress has shown that the graphene-based materials offer enormous potential in electronic and optoelectronic devices, chemical sensors, nanocomposites and energy storage applications [16]. Recent work on piezo-resistive graphene based pressure and strain sensors has identified applications in wearable electronics, human/machine interfaces, soft robotics, MEMS, civil structures such as roadways and bridges, artificial intelligence and beyond [17–25]. Recently, we have developed a simple and scalable application of graphene-coated substrates for pressure sensing [26,27]. The method has been applied to wearable items for real-time health and physical performance monitoring. All the applications have demonstrated both repeatability and scalability. In the current study, using the piezo-resistive nature of graphene, we propose a novel approach for in-situ compaction and fluid flow monitoring in the LCM technique.

In this work, for the first time, we explore the applicability of a graphene-coated piezo-resistive fabric for cost-effective LCM process monitoring. The potential of the measurement process is demonstrated through a number of well-defined experiments. A thin coating of reduced graphene oxide (rGO) is applied on a substrate (such as a glass fabric or a nylon peel ply), rendering the whole fabric electrically conductive. The piezo-resistive effect is a result of changes in the conduction paths within the material and the contact resistance between neighboring conducting regions [28]. The electrical characteristics follow a power law to parameters such as the applied stress/strain. The results show that the use of graphene coated fabric for monitoring and characterizing the LCM process is a novel solution. The cured product also exhibits a measureable conductivity, a characteristic that can be used for in-situ structural health monitoring [29]. The overall objective of this research is to develop a cost effective process monitoring system using graphene coated fabrics for LCM processing. The graphene coatings will help to understand the complex compaction behavior of the reinforcements and will provide information about the flow front advancement and resin curing by means of electrical signals generated due to the change in resistance of the fabric during an LCM process.

2. Materials and methods

2.1. Graphite oxide

Graphene oxide (GO) was synthesized from pretreated graphite powder with a particle size of 20 μm following a modified Hummer's method [30]. This graphite powder was added into concentrated sulfuric acid (H_2SO_4, 98%) in an ice bath. Potassium permanganate $KMnO_4$ was added gradually whilst stirring, and the temperature of the solution was kept below 10 °C. The mixture was then stirred for 2 h at 35 °C and deionized water was added slowly as the graphite oxide further expanded because of the released heat. The solution was stirred for another 30 min to ensure complete expansion of the graphite oxide and the reaction was then terminated by adding 300 ml of deionized water and 5 ml of 30% hydrogen peroxide (H_2O_2) solution. The mixture was centrifuged and subjected to several cycles of washing in 5% HCl solution and then separated by centrifugation. In order to completely remove the metal ions and acids, the graphite oxide was subjected to cycles of washing with DI water and subsequent centrifugation until the pH value of the supernatant reached approximately 6. The graphite oxide was then subjected to ultrasonication for 1 h to obtain exfoliated GO suspended in DI water in the form of a viscous, brown dispersion [26,31]. All of the materials were used directly as supplied by the vendor, without further purification.

2.2. Fabrics

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. The 3D woven fabric was used in order to avoid significant nesting between adjacent layers, an effect that may arise when using 2D fabrics. A commercially available nylon peel ply supplied by Gurit® was also used to demonstrate the effectiveness of the coating process on different substrates [26,32].

2.3. Test fluids

Two types of test fluid were used in this study, a Dow Corning® Silicone oil and Gurit PRIME 20LV® with a fast hardener. Silicone oil was used to avoid any interaction between the dielectric behavior of the resin and its cure behavior on fabric conductivity during processing also, this fluid is widely used in LCM process characterization experiments. The silicone oil has a viscosity of 0.1 Pa s and a density of 1.06 g/cm3 at 23 °C. The resin has a mix viscosity of 0.3 Pa s at 23 °C.

2.4. Coating process

The coating process started by dissolving graphene oxide in distilled water to form a solution (3 g/200 ml). The sheets of glass fabric and peel ply 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 washed thoroughly to remove any