

SMART GRAPHENE ENABLED PREFORMS

M. Reghat¹, N. Hameed^{1*}, L. Hyde, P. Middendorf², R. Bjekovic³, B. Fox¹
Yehuda (Udi) Weizman¹, Adin Ming Tan¹, Franz Konstantin Fuss¹

¹ Faculty of Science, Engineering and Technology, Swinburne University of Technology, Hawthorn,
Melbourne VIC 3122 Australia

Email: blfox.@swin.edu.au, nisharhameed@swin.edu.au, mreghat@swin.edu.au,
webpage:<http://www.swinburne.edu.au/research/our-research/institutes/manufacturing-futures/>

² Institute of Aircraft Design, University of Stuttgart, Germany

Email: middendorf@IFB.Uni-Stuttgart.de,

³ Hochschule Ravensburg-Weingarten

Email: robert.bjekovic@stw.de

Keywords: Graphene, Coating, Preform, Carbon fiber

Abstract

The carbon fiber (CF) preforming process is one of the rate-limiting steps in composite manufacturing. Lack of control over the raw materials during the preforming process leads to inconsistent production. Batch-to-batch variations are unavoidable as molding environments remain undetected during the preforming process especially during the stacking up of the fabric plies and placing of the fabric preform into the mold cavity. This study aims to create high-quality composites structural parts made from CF preforms and establish an understanding of the use of graphene nano-platelets (GNPs) as a smart fabric coating that allows the sensing and monitoring of the preforming process. In addition to the preform monitoring the mechanical reinforcing by graphene imparts additional functionality into the cured composite part. The ultimate outcome of this study will be an inexpensive, reliable way to manufacture highly uniform CF preforms.

1. Introduction

Aerospace, automotive and electronic industries use CF composites in various applications, exploiting their light weight, high specific strength, and stiffness capabilities [1-3]. The CF preforming process is replacing other techniques as the cost-effective composites manufacturing technology. A preform is a multi-layer reinforcement structure, where the fiber is fixed in layers by a suitable binder that is subsequently impregnated with a matrix system and consolidated to form a composite part. Preforms can be made by spraying discrete chopped fibers combined with a binder over a form. More recently, engineered preforms have been developed through the use of automated knitting and weaving machinery [4]. The fiber forms are subsequently impregnated with resin in a closed mold process, such as resin transfer molding (RTM) or vacuum-assisted resin transfer molding (VARTM). These two and three-dimensional structures are capable of reinforcing high-performance structural composite parts.

The composite structures in high-performance applications are often exposed to external impacts, deformations, and changes in environmental conditions during service. The onset of local damage in such instances is difficult to detect and can have long-term implications on the structural performance. The detection of the structural integrity and the prediction of remaining functional lifespan of components could forecast the failure and prevent unexpected downtime [5-7].

Graphene, a single layer of carbon atoms in a closely packed honeycomb two-dimensional (2D) lattice obtained by the exfoliation of natural flake graphite by sonication. Graphene exhibits outstanding properties including high thermal and electrical conductivity, high mechanical strength, low manufacturing cost as well as high surface area, which bring them to be distinguished nano-reinforcements for carbon fiber reinforced plastics (CFRP) to form multi-functional and multiscale composites [8-10]. Graphene can enhance interfacial bonding between fiber and matrix in the CFRPs. Furthermore, graphene has more sp²-like planes and various edge defects present on the surface and has previously indicated better sensing performance than other carbon nanomaterials such as carbon nanotubes (CNTs) [11, 12]. The GNPs create an electrically conductive network through the composite material, which is susceptible to small dimensional changes. Electrically conductive fiber-reinforced composites can be produced by either adding conductive particles such as GNPs and CNTs in a polymer matrix or coating them on the surface of fabric [13]. High concentration of additives in the resin will result in undesired viscosity. However, incorporating the GNPs in the matrix prior to infusion of the fiber only require the low content of the additive achieving more control over viscosity [14, 15].

Various methods have been used to coat graphene on to the CF including chemical vapor deposition (CVD), electrophoretic deposition (EPD), dipping and spraying. Among the many techniques available, dip coating is more common, effective, low-cost and used in large-scale processes. Several studies have focused on piezoresistive composites. Shazed et al. [16] investigated the coating of CNTs on CF using CVD followed by fabricating CNTs coated CF reinforced polypropylene (CNTs-CF/PP) composites. These hybrid composites indicated enhanced young modulus and tensile strength when compared to the neat-CF/PP composite. MORICHE et al. [17] compared the electrical response of nano-reinforced epoxy matrices and dip coated fabrics and concluded that composites with dip coated fabrics were more sensitive to fiber breakage and showed higher sensitivity under tensile loads. Recently, Kwon et al. [18] studied graphene oxide/CNTs hybrid materials prepared and coated on CF surfaces by EPD method. The results showed the enhancement of both mechanical and electrical properties of CFRP.

This proof of concept study aims to test the feasibility of a uniform interconnected graphene coating network to be used as a sensor material in composites. This study aims to fabricate a uniform, interconnected graphene network within composites that is highly sensitive to strain and deformation. Initial stages of this study have focused on creating graphene-coated CF using dip coating followed by infusing them into a composite part. Surface morphologies of coated CF were examined using optical microscopy. The electrical response of the coated composite was simultaneously monitored during a three-point bending test using electromechanical testing Instron series 5569. Strain monitoring results showed the sensing effect of the graphene.

2. Materials and methods

Plain weave CF was supplied by Play with Carbon PTY Ltd, Sydney Australia. The coating of the fabrics was performed using the dip coating method with graphene dispersions supplied by Imagine (Imagine X3). Composite laminates were prepared using standard hand lay-up process followed by vacuum assisted curing. The epoxy resin and hardener (PRIME™ 20LV and PRIME™ 20 slow hardener) supplied by Gurit Company, were mixed at a weight ratio of 100/ 26 for 2 min. The four piles of fabrics were layered up while resin was spread within the fabrics using a brush. The laminate was vacuumed at -700 mmHg for 24hours at 25 °C followed by post-curing in an oven at 65 °C for 6hours. Finally, the composite was cut into the dimensions of 15 cm× 4 cm× 0.5 cm using a diamond cutter. The prepared composite laminates were cut into different sizes for further testing, including three-point bending tests. Copper electrodes and silver paint were coated on both sides of the sample as shown in Fig.1 to minimize the electrical contact resistance.



Figure.1 As prepared graphene coated carbon fabric composite

3. Results and discussion

The morphology of coated CF was investigated using an optical microscope (Olympus Stream Motion BX61). Fig.2 shows the images of 1x and 3x times graphene coated CF. The images indicate that multiple coating of the fabric result in better uniformity and coverage.

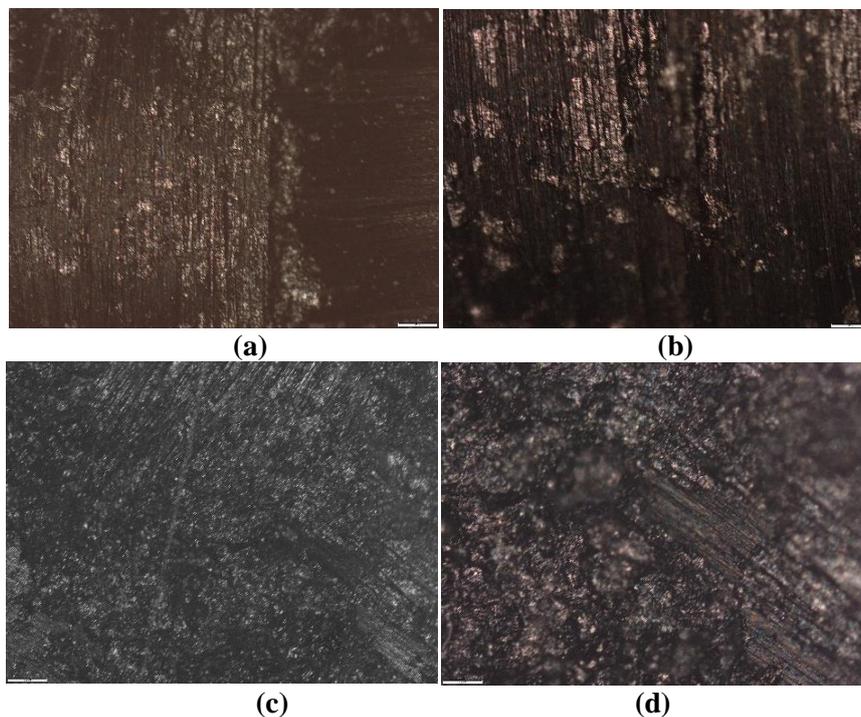


Figure. 2 Optical microscopy images of coated CF a) 1X time coating with 5X magnification b) 1X time coating with 10X magnification c) 3X time coating with 5X magnification d) 3X time coating with 10X magnification

Three-point bending test was carried out using an electromechanical testing machine (Instron 5569, Massachusetts, USA) as shown in Fig.3. Ten loading cycles at a rate of 45 mm/min with a maximum force of 40N were applied to the sample. The electrical data from the sensor was recorded using a USB DAQ module (USB-2404-10, Measurement Computing, Norton MA, USA) and recorded with Tracer DAQ Pro (Measurement Computing, Norton MA, USA) to monitor the change of voltage over time at a sampling frequency of 100 Hz.

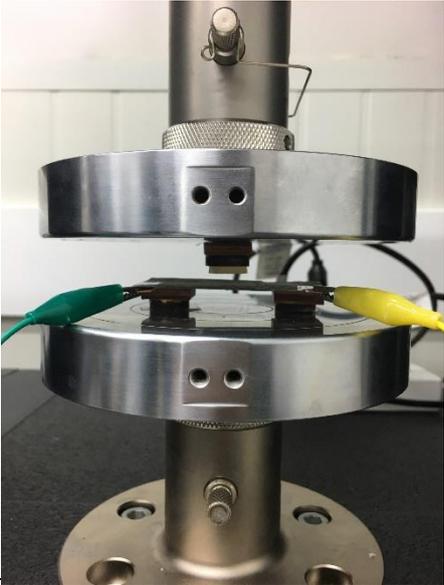
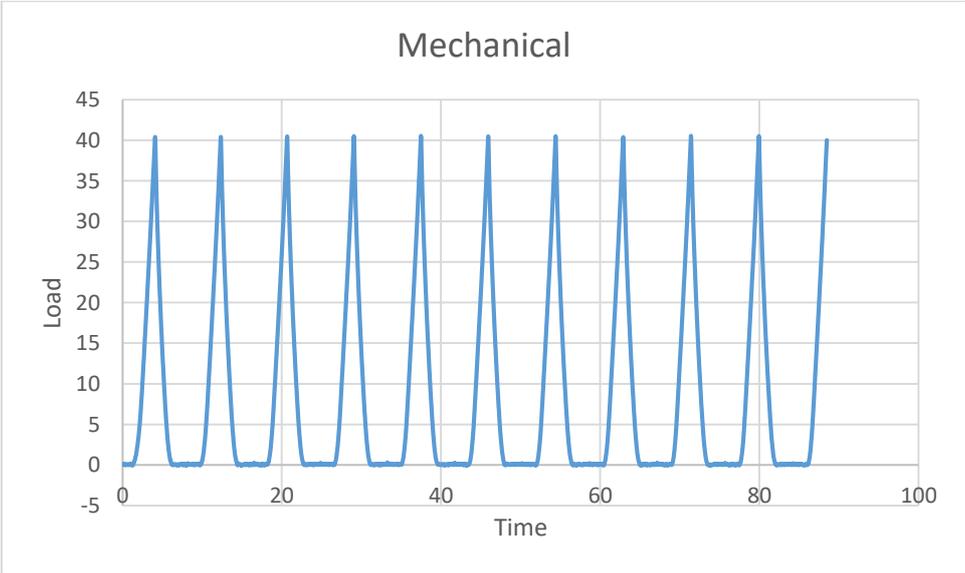


Figure3. Three-point bending test arrangements with Instron 5569



a

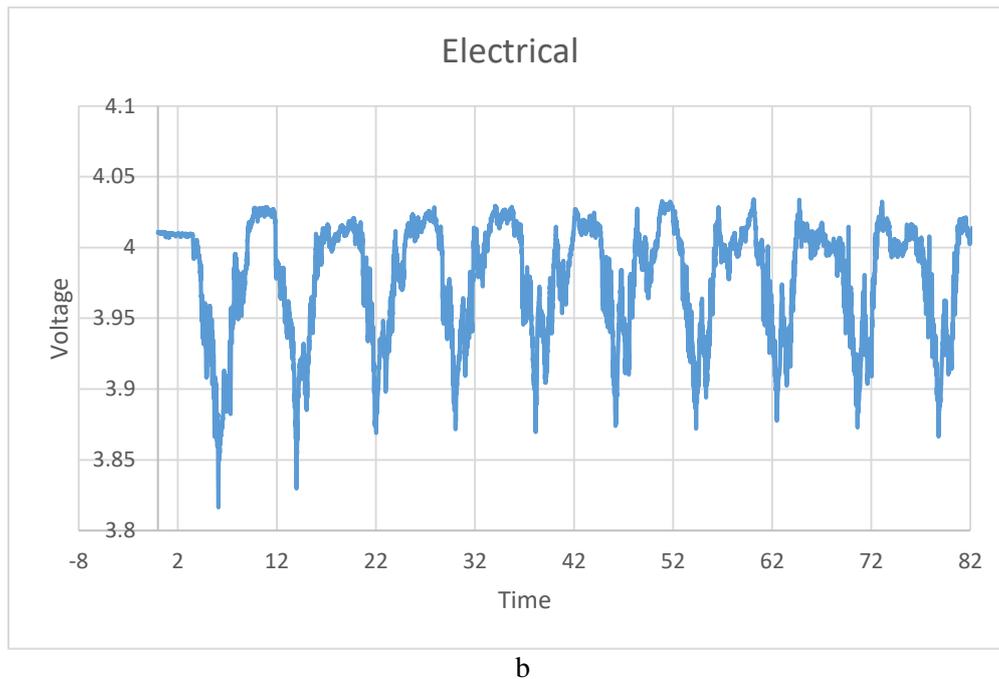


Figure 4. a) Change of load over time b) Change of voltage over time during cyclic three-point bending tests

The sample was cut in half. The top and bottom layer of epoxy removed to expose the conductive carbon particles and subsequently coated with silver paint. The electrical response of the coated composite during bending test was measured. The electrical current flows were measured in z-direction, or the vertical plane. Fig.4 illustrates the change of load and voltage over time. The preliminary results showed change in the electrical data upon mechanical loads, indicating that the sample is electrically sensitive to bending. Graphene can create an electrically conductive network through the composite material, which is susceptible to dimensional changes. When strain or damage are induced into the system, the electrical resistance of the material changes due to modifications in distances between the GNPs or in overlying areas.

4. Conclusions

The proof of concept of using graphene as a sensor material to create smart and functional composite was established. Preliminary results showed that the graphene coating is suitable for monitoring the electrical performance of composites despite some inconsistencies. Optical microscopy showed that three-time coated fabrics have a complete coverage compared to the one-time coating. The three-point bending test results showed that the coated CF composite was electrically sensitive to bending and deformation, as a change in electrical signals were obtained when an external force was applied. Further tests need to be conducted to ensure the repeatability of the results. Future study will be focused on integrating strain gauges for quantitative analyses, comparing the mechanical-electrical results of uncoated and coated sample and electrical – mechanical calibration (conductivity – force).

Acknowledgments

The authors acknowledge the Faculty of Science, Engineering and Technology of Swinburne University, the Centre for Design Innovation (Swinburne University) and Imagine Intelligent Materials. MR

acknowledges RTPS scholarship support and acknowledges ARC for DECRA fellowship (DE170101249).

References

- [1] L.G. Tang, J.L. Kardos, A review of methods for improving the interfacial adhesion between carbon fiber and polymer matrix, *Polymer composites* 18:100-113, 1997.
- [2] G. Kalaprasad, S. Thornas, Hybrid fibre reinforced polymer composites, *Int Plast Eng Technol* 1: 87-98, 1995.
- [3] S.-Y. Fu, B. Lauke, Y.-W. Mai, *Science and engineering of short fibre reinforced polymer composites*, Elsevier, 2009.
- [4] A. Mills, Automation of carbon fibre preform manufacture for affordable aerospace applications, *Composites Part A: Applied science and manufacturing* 32: 955-962, 2001.
- [5] D. He, B. Fan, H. Zhao, X. Lu, M. Yang, Y. Liu, J. Bai, Design of electrically conductive structural composites by modulating aligned CVD-grown carbon nanotube length on glass fibers, *ACS applied materials & interfaces* 9: 2948-2958, 2017.
- [6] R. Zhang, B. Gao, W. Du, J. Zhang, H. Cui, L. Liu, Q. Ma, C. Wang, F. Li, Enhanced mechanical properties of multiscale carbon fiber/epoxy composites by fiber surface treatment with graphene oxide/polyhedral oligomeric silsesquioxane, *Composites Part A: Applied Science and Manufacturing* 84:455-463, 2016.
- [7] E. Bekyarova, E. Thostenson, A. Yu, H. Kim, J. Gao, J. Tang, H. Hahn, T.-W. Chou, M. Itkis, R. Haddon, Multiscale carbon nanotube– carbon fiber reinforcement for advanced epoxy composites, *Langmuir* 23:3970-3974, 2007.
- [8] Y. Geng, S.J. Wang, J.-K. Kim, Preparation of graphite nanoplatelets and graphene sheets, *Journal of colloid and interface science* 336:592-598, 2009.
- [9] P.-y. Hung, K.-t. Lau, B. Fox, N. Hameed, J.H. Lee, D. Hui, Surface modification of carbon fibre using graphene–related materials for multifunctional composites, *Composites Part B: Engineering* 133:240-257, 2018.
- [10] B.Z. Jang, A. Zhamu, Processing of nanographene platelets (NGPs) and NGP nanocomposites: a review, *Journal of Materials Science* 43:5092-5101, 2008.
- [11] X. Lee, T. Yang, X. Li, R. Zhang, M. Zhu, H. Zhang, D. Xie, J. Wei, M. Zhong, K. Wang, Flexible graphene woven fabrics for touch sensing, *Applied Physics Letters* 102:163117, 2013.
- [12] X. Chen, X. Zheng, J.-K. Kim, X. Li, D.-W. Lee, Investigation of graphene piezoresistors for use as strain gauge sensors, *Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena* 29:06FE01, 2011.
- [13] A. Markov, B. Fiedler, K. Schulte, Electrical conductivity of carbon black/fibres filled glass-fibre-reinforced thermoplastic composites, *Composites Part A: Applied Science and Manufacturing* 37:1390-1395, 2006.
- [14] M. Kim, Y.-B. Park, O.I. Okoli, C. Zhang, Processing, characterization, and modeling of carbon nanotube-reinforced multiscale composites, *Composites Science and Technology* 69:335-342, 2009.
- [15] E. Bekyarova, E.T. Thostenson, A. Yu, M.E. Itkis, D. Fakhruddinov, T.-W. Chou, R.C. Haddon, Functionalized single-walled carbon nanotubes for carbon fiber– epoxy composites, *The Journal of Physical Chemistry C* 111:17865-17871, 2007.
- [16] M. Shazed, A. Suraya, S. Rahmanian, M.M. Salleh, Effect of fibre coating and geometry on the tensile properties of hybrid carbon nanotube coated carbon fibre reinforced composite, *Materials & Design* (1980-2015) 54: 660-669, 2014.
- [17] R. MORICHE, M. SÁNCHEZ, S.G. PROLONGO, A. JIMÉNEZ-SUÁREZ, A. UREÑA, Structural health monitoring in multiscale composite materials: nanoreinforced epoxy matrices and coated fabrics, *8th European Workshop on Structural Health Monitoring, EWSHM*, 2016.
- [18] Y.J. Kwon, Y. Kim, H. Jeon, S. Cho, W. Lee, J.U. Lee, Graphene/carbon nanotube hybrid as a multi-functional interfacial reinforcement for carbon fiber-reinforced composites, *Composites Part B: Engineering* 122: 23-30, 2017.