

INTEGRATED MULTIFUNCTIONAL GLASS FIBER REINFORCED COMPOSITES: TOWARD SMART AND SUSTAINABLE LIGHTWEIGHT STRUCTURES

Palak B. Patel¹, Carolina Furtado², Jeonyoon Lee³, Christopher Dunn⁴, Michael Borgen⁴, Estelle Kalfon-Cohen⁴, Seth S. Kessler⁴, Luiz Acauan⁵, and Brian L. Wardle⁵

¹ Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, MA, USA, <u>palak@mit.edu</u>

² University of Porto, Porto, Portugal

³ Korea Advanced Institute of Science & Technology, Daejeon, South Korea

⁴ Metis Design Corporation, Boston, MA, USA

⁵ Department of Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, MA, USA

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ABSTRACT

Combining multiple functional capabilities within a structure offers system-level benefits, especially in weight-critical applications like air and space vehicles. Extensive research has been conducted on multifunctional composite structures that go beyond their primary structural functions. These structures have demonstrated various additional capabilities, including energy storage, self-health monitoring, ice protection systems, and self-manufacturing attributes. However, the integration of these multiple functionalities into a single engineering solution remains unexplored. Nanoengineering provides an opportunity for integrating functions at the nanoscale without compromising the shape, design, or loadbearing capacity of the structure. This study focuses on demonstrating multiple functionalities in an integrated multifunctional composite (IMC) laminate using Hexcel E-glass/913 unidirectional glass fiber prepreg integrated with multifunctional carbon nanotubes at the interlaminar regions and external surfaces. The IMC, having been tested structurally in a previous study, was tested for multifunctional capabilities here, focusing on electrode-heating multifunctionality through resistive heating as well as structural health monitoring multifunctionality through piezoresistive strain sensing.

1 INTRODUCTION

Innovative aerospace components have been made possible through the utilization of lightweight heterogeneous materials, such as composites. Although these composites offer weight reduction benefits, they can be vulnerable to undesirable failure modes, particularly in weaker regions like the interlaminar region. To address this, nanomaterials, such as carbon nanotubes, have been employed to reinforce the interlaminar region of composites, offering a nano-scale solution to strengthen the weaker areas without increasing interlaminar thickness [1]. Successful reinforcement of the interlaminar region has been achieved using vertically aligned carbon nanotubes (VACNTs) in carbon fiber laminates. These reinforced laminates have exhibited increased shear strength compared to unreinforced counterparts, with insignificant changes in interlaminar thickness [1-8]. Various morphologies of vertically aligned carbon nanotubes, have been explored, demonstrating enhanced strength through nanoscale reinforcement architectures [1-8].

Moreover, the nano-engineering of these lightweight materials offers the opportunity to incorporate multifunctionalities, providing additional advantageous functionalities alongside the primary structural role, without significant weight increase. While the individual exploration of nano-engineered multifunctional capabilities in structural materials has been conducted, such as life-cycle enhancement [1-15], energy saving during manufacturing [9-14], sensing and control [9], in-situ cure monitoring [11-13], and de-icing [15], the integration of these multifunctionalities into a single-engineered composite remains unexplored. This paper presents the demonstration of multiple functionalities in an integrated

multifunctional composite (IMC) by nano-engineering Hexcel E-glass/913 unidirectional glass fiber prepreg. The IMC, having been tested structurally in a previous study, was tested for multifunctional capabilities, and in this study, demonstrated the electrode-heating multifunctionality through resistive heating as well as the structural health monitoring multifunctionality through piezoresistive strain sensing.

2 MATERIALS

The IMC was manufactured by using glass fiber reinforced polymer prepreg with the multifunctional carbon nanotubes. Hexcel E-glass/913 unidirectional glass fiber prepreg was used, which requires an autoclave to be cured. A 16-ply quasi-isotropic layup was used for the tests. Randomly oriented commercial CNT film (Tortech, CNTM4) was placed and integrated into the top and bottom of the laminate. The addition of these CNT architectures to the composite system are the key to the integration of multifunctionalities in composites. The film utilized in this study consisted of non-aligned CNTs with a manufacturer-specified areal density of 4 g/m² and a nominal thickness of 16 μ m. In its original form and when incorporated into a polymer nanocomposite, the film exhibits electrical conductivity, which plays a crucial role in enabling multifunctional capabilities within the laminates. The integration of the composite laminate especially void formation and mechanical properties such as interlaminar shear strength and double-edge notched tensile strength in previous studies [16-18]. The results have shown that the integration of the CNTs occur while maintaining the quality and mechanical properties of the composite laminate [16-18].

3 METHODS, RESULTS, AND DISCUSSION

3.1 IMC Quality Characterization

In order to ensure that the laminate integrated with CNTs maintained a comparable quality to the baseline plate, a void characterization study was conducted. Micro-computed tomography (μ -CT), a non-destructive X-ray imaging technique, was employed to assess the presence of voids in the composite after the curing process. In the micro-CT images, the density of the material is indicated by the grayscale value, where lighter regions correspond to denser materials that the X-rays passed through. Therefore, the glass fibers and resin appear lighter, while voids are depicted as darker or nearly black regions.

Figure 1 displays representative images of both the baseline specimen and the specimens with integrated CNTs. The micro-CT scans reveal that the lightest grey areas represent the glass fiber within the medium grey resin matrix. Due to resin infusion, it is difficult to visually distinguish the CNTs in the interlaminar regions from the resin. Notably, no visible voids were observed in any of the specimen configurations, indicating that the quality and void characteristics of the CNT integrated specimens were comparable to those of the baseline specimen.

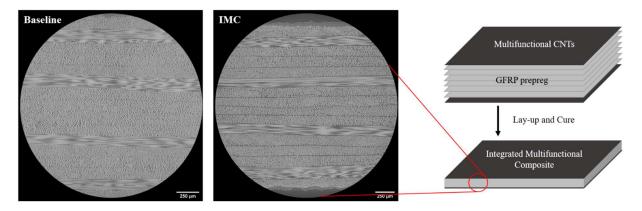


Figure 1: Micro-computed tomographs of the baseline glass fiber laminate and the nanoengineered IMC show similar quality with no voids.

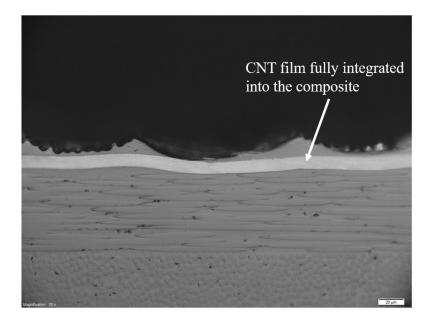


Figure 2: Scanning electron micrograph of the fully integrated CNT film in the IMC laminate showing no voids due to the integration.

3.2 Electrode Heating via Joule Heating

In the IMC, the CNTs act as a resistive heater which can heat the sample when provided current with a power supply. This can be used for various thermal management applications including *in situ* deicing of airplane and jet wings. To demonstrate this multifunctionality, the IMC was tested for heating capabilities.

An IMC sample was prepared by exposing the integrated CNTs, which were fully integrated under the resin of the composite, by lightly grinding the surface to reveal the CNTs. Copper electrodes were attached to the sample using silver epoxy to maintain an electrical connection as seen in Figure 3. The experiment involved applying a fixed current to the sample and measuring the changes in voltage and temperature using a voltmeter and thermocouple. The goal was to observe how the resistance of the CNTs changes with heat and to measure the temperature increase caused by the current. The heating profile of the sample was also recorded using an infrared camera to note the heating uniformity of the sample.

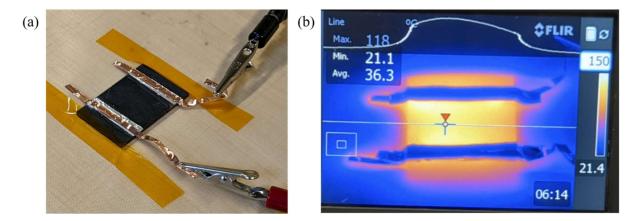


Figure 3: Electrothermal heating IMC demonstration: (a) Electrode heating setup with the sample in black with silver epoxy attached copper electrodes connected to a power supply. (b) Infrared camera image taken when current was supplied to the sample showing uniform heating of the sample.

For the study, different currents ranging from 0.5 A to 1.6 A were provided to the sample for 7 minutes each and the highest temperature the sample reached was noted. The samples were then allowed to cool down until close to room temperature and the test at each current level was cycled 3 times. Figure 4 shows the highest temperature that the sample's surface would reach when provided current for 7 minutes. The results show a close to linear relationship between the current provided and the temperature. For applications such as de-icing of airplane wings, the IMC being capable of reaching over 100 °C shows that it can be utilized for *in situ* de-icing of airplane wings.

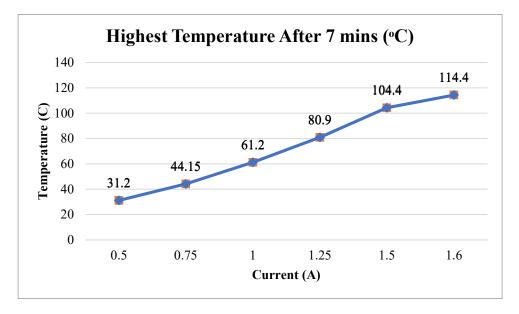


Figure 4: Highest temperature of the surface of the sample as recorded by the thermocouple at the different current values the sample was tested at.

3.3 Structural Health Monitoring via Piezoresistivity Strain Sensing

Structural health monitoring can be performed on the IMC by sensing the change in resistance in the CNT network. When the IMC undergoes strain, the resistance across the CNTs integrated in the composite will change due to their piezoresistive properties.

IMC samples were prepared for a 3 point bending test with CNT strain sensors and strain gages on both the tension and compression side. The CNT strain sensors comprised of two electric wires each connected to the exposed CNTs at a fixed distance to measure the resistance change in the CNTs between the electric wires. The CNT strain sensors and strain gages were placed at an equal distance from the testing rig rollers in order to be able to compare the strain data. The samples were tested to 1000 µstrains, 2000 µstrains, and 3000 µstrains for 10 cycles to test the structural health monitoring capabilities of the CNTs under fatigue. In Figure 5, the average relative change in resistance in the CNT network for the different strain values can be seen. A clear trend of increasing relative change in resistance with an increase in strain has been noted which demonstrates that the CNTs provide a structural health monitoring multifunctionality to the IMC. The difference in tension and compression can be attributed to difference in the length of the CNT strain sensor that were sensing the strain in the region of the sample under tension and compression.

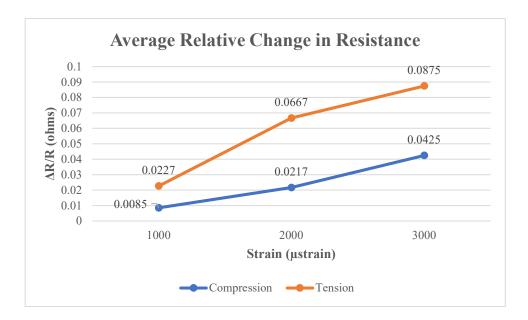


Figure 5: The average relative change in resistance in tension and compression for 1000, 2000, and 3000 µstrain.

4 CONCLUSION

Multifunctionalities of composite laminates, which have been demonstrated individually to date, are demonstrated in the same composite laminate for the first time. The IMC multifunctionalities demonstrated include electrode heating *via* Joule heating as well as structural health monitoring *via* piezoresistivity strain sensing. Integration of these functionalities improves the efficiency of the system and reduces the mass and volume of the total structure, towards sustainable and smart composite structures. While the in-service multifunctionality of life-cycle enhancement through increased mechanical properties has been demonstrated in previous studies on the same IMC, additional multifunctionalities related to manufacturing such as energy savings during curing and cure sensing will be demonstrated on the same IMC system in future work.

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REFERENCES

- [1] E. Kalfon-Cohen, R. Kopp, C. Furtado, X. Ni, A. Arteiro, G. Borstnar, M. N. Mavrogordato, I. Sinclair, S. M. Spearing, P. P. Camanho, and B. L. Wardle "Synergetic Effects of Thin Plies and Aligned Carbon Nanotube Interlaminar Reinforcement in Composite Laminates." *Composites Science and Technology*, Vol. 166, pp. 160–168, 2018.
- [2] J. Lee, S. S. Kessler, and B. L. Wardle "Void-Free Layered Polymeric Architectures via Capillary-Action of Nanoporous Films." Advanced Materials Interfaces, Vol. 7, No. 4, p. 1901427, 2020
- R Guzman De Villoria, P Hallander, L Ydrefors, P Nordin, and BL Wardle. In-plane strength enhancement of laminated composites via aligned carbon nanotube interlaminar reinforcement. Composites Science and Technology, 133:33–39, 2016

- [2] Estelle Kalfon-Cohen, Reed Kopp, Carolina Furtado, Xinchen Ni, Albertino Arteiro, Gregor Borstnar, Mark N Mavrogordato, Ian Sinclair, S Mark Spearing, Pedro P Camanho, et al. Synergetic effects of thin plies and aligned carbon nanotube interlaminar reinforcement in composite laminates. Composites Science and Technology, 166:160–168, 2018.
- [3] Xinchen Ni and Brian L Wardle. Experimental investigation of interlaminar fracture micromechanisms of aligned carbon nanotube-reinforced aerospace laminated composites. In AIAA Scitech 2019 Forum, page 1201, 2019.
- [4] Xinchen Ni, Carolina Furtado, Nathan K Fritz, Reed Kopp, Pedro P Camanho, and Brian L Wardle. Interlaminar to intralaminar mode I and II crack bifurcation due to aligned carbon nanotube reinforcement of aerospace-grade advanced composites. Composites Science and Technology, 190:108014, 2020.
- [5] Xinchen Ni, Estelle Kalfon-Cohen, Carolina Furtado, Reed Kopp, Nathan K Fritz, A Arteiro, G Valdes, T Hank, G Borstnar, M Mayrogordato, et al. Interlaminar reinforcement of carbon fiber composites using aligned carbon nanotubes. In submitted to the 21st International Conference on Composite Materials (ICCM), Xi'an, China, 2017.
- [6] Xinchen Ni, Carolina Furtado, Estelle Kalfon-Cohen, Yue Zhou, Gabriel A Valdes, Travis J Hank, Pedro P Camanho, and Brian L Wardle. Static and fatigue interlaminar shear reinforcement in aligned carbon nanotube-reinforced hierarchical advanced composites. Composites Part A: Applied Science and Manufacturing, 120:106–115, 2019.
- [7] Xinchen Ni, Luiz H Acauan, and Brian L Wardle. Coherent nanofiber array buckling-enabled synthesis of hierarchical layered composites with enhanced strength. Extreme Mechanics Letters, 39:100773, 2020.
- [8] Xinchen Ni and Brian L Wardle. Aerospace-grade advanced composites with buckling-densified aligned carbon nanotubes interlaminar reinforcement. In AIAA Scitech 2020 Forum, page 0156, 2020.
- [9] Jeonyoon Lee, Itai Y Stein, Seth S Kessler, and Brian L Wardle. Aligned carbon nanotube film enables thermally induced state transformations in layered polymeric materials. ACS applied materials & interfaces,7(16):8900–8905, 2015.
- [10] Jeonyoon Lee, Xinchen Ni, Frederick Daso, Xianghui Xiao, Dale King, Jose Sánchez Gómez, Tamara Blanco Varela, Seth S Kessler, and Brian L Wardle. Advanced carbon fiber composite out-of-autoclave laminate manufacture via nanostructured out-of-oven conductive curing. Composites Science and Technology, 166:150–159, 2018.
- [11] Jeonyoon Lee, Frederick Daso, Seth S Kessler, and Brian L Wardle. Carbon fiber prepreg composite laminates cured via conductive curing using nanoengineered nanocomposite heaters
- [12] Jeonyoon Lee and Brian L Wardle. Nanoengineered in situ cure status monitoring technique based on carbon nanotube network. In AIAA Scitech 2019 Forum, page 1199, 2019.
- [13] Jeonyoon Lee, Itai Y Stein, Erica F Antunes, Seth S Kessler, and Brian L Wardle. Out-of-oven curing of polymeric composites via resistive microheaters comprised of aligned carbon nanotube networks. 2015.
- [14] Jeonyoon Lee, Seth S Kessler, and Brian L Wardle. Void-free layered polymeric architectures via capillary-action of nanoporous films. Advanced Materials Interfaces, 7(4):1901427, 2020.
- [15] Jeonyoon Lee, Christopher J Brampton, Christopher R Bowen, Brian L Wardle, and Hyunsun A Kim. Investigation of aligned conductive polymer nanocomposites for actuation of bistable laminates. In 23rd AIAA/AHS Adaptive Structures Conference, page 1725, 2015.
- [16] PATEL, P., FURTADO, C.F., COOPER, M., ACAUAN, L., LOMOV, S., AKHATOV, I., ABAIMOV, S., LEE, J. and WARDLE, B., 2021. Nanoengineered Glass Fiber Reinforced Composite Laminates with Integrated Multifunctionality. In Proceedings of the American Society for Composites—Thirty-Sixth Technical Conference on Composite Materials.
- [17] Patel PB, Furtado C, Lee J, Cooper M, Acauan L, Lomov SV, Akhatov IS, Abaimov SG, Wardle BL. Building Life-Cycle Enhancement Multifunctionality into Glass Fiber Reinforced Composite

Laminates via Hierarchical Assemblies of Aligned Carbon Nanotubes. In AIAA SCITECH 2022 Forum 2022 (p. 0501).

[18] Palak B Patel, Carolina Furtado, Megan Cooper, Jeonyoon Lee, Keiran Ball, Yeajin Lee, Albertino Arteiro, Marta Majkut, Lukas Helfen, Luiz Acauan, Mark Spearing, Iskander S Akhatov, Stepan V Lomov, Sergey G Abaimov, and Brian L Wardle. Synchrotron radiation 3d computed tomography study on in-situ mechanical damage progression of nanoengineered glass fiber reinforced composite laminates with integrated multifunctionality. European Conference on Composite Materials (ECCM).