MULTI-PHYSICS NANO-ENGINEERED STRUCTURAL DAMAGE DETECTION AND DE-ICING

<u>R. Guzman de Villoria¹</u>*, S.S. Kessler², S. Wicks¹, A. Miravete¹, B.L. Wardle¹ ¹ Department of Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, MA, USA, ² Metis Design Corporation, Cambridge, MA, USA * Corresponding author (rguzman@mit.edu)

Keywords: *Hierarchical Structures, non destructive evaluation, de-icing, carbon nanotube*

1 General Introduction

Catastrophic structural failures are the cause of many physical and personal losses, at a worldwide cost estimated at billions of dollars per year. Nondestructive evaluation (NDE) techniques have been pursued and employed for damage detection of structures to detect cracks and other damage at precritical levels for remediation [1-3]. To address drawbacks with state-of-the-art approaches, a novel multi-physics approach is reported that takes advantage of the effects that damage has on the electrical and thermal transport in a material containing aligned carbon nanotubes (CNTs) to create a new damage detection technique. Another application of the same nano-engineered composites is in thermal applications such as de-icing and antiicing systems. Icing is a serious problem that has caused several aircraft incidents associated with temperatures ranging between -40 °C to 0 °C. Although some technologies have been developed, improved solutions are desirable in order to obtain lighter and more efficient technologies [4].

2. Experimental

2.1 Aligned CNT Synthesis

Aligned CNTs were grown on the surface of alumina fibers (11 μ m diameter) contained in tows that were pre-woven into a cloth. Details of the process can be found in preceding work [5]. Aligned ~30 μ m long multi-walled CNTs were grown uniformly on fiber surfaces in a "Mohawk" morphology [6].

2.2 Composite Fabrication

Composite specimens were made by hand lay-up using a commercial epoxy system (Resin 105 and Hardener 206, West Systems Epoxy) to create 3-ply laminates. The resulting composites have ~50% alumina fiber volume fraction, and ~ 2% CNT volume fraction. [5].

2.3 Non-Destructive Evaluation by Infrared Camera

Damaged specimens were heated via electrical current (Joule-effect heating). Temperature was recorded using an infrared (IR) camera at 160x120 pixels, with a temperature range of -10 to 250°C and a resolution of 0.15° C. In order to apply the current to the sample, copper adhesive tape (Compac Corporation) was placed as electrodes on both ends of the sample (dimensions $\approx 90x25x2.2 \text{ mm}^3$), and connected to a nine volts alkaline battery via alligator tips. Thermal images were taken with a thermal camera (PCE-TC 3, PCE Group) 30 seconds after applying the voltage to allow temperature readings to stabilize [7].

2.4 Non-Destructive Evaluation Using a Thermochromic Ink

Two silver contacts were painted on both ends of the specimen and two alligator clips were attached to the specimen. A ~4mm diameter and ~3 mm deep groove was drilled carefully in one side of the specimen, avoiding damage to the other side, which was coated with thermochromic ink (Chiral nematic sprayable liquid crystal, thermal range 25° C - 30° C ThermometerSite) using an airbrush. A nine volt battery was connected to the alligator clips and the undamaged site of the specimen was filmed with a conventional USB camera.

2.5 Direct De-icing Experiment

For the de-icing test, two silver contacts were painted on both ends of the specimen and two alligator clips were attached to the specimen. The specimen was buried under a layer of snow 2.5 cm thick. Thermal and optical images were taken every five minutes during the voltage application (0.74A, 30V) for half an hour at an ambient temperature of $-10^{\circ}C$.

2. 6 Ice-detection and De-Icing/Anti-Icing System

Once the feasibility of using nano-enginered composites for direct de-icing applications has been demonstrated via Joule heating, an ice-detection system was developed for controlled de-icing and anti-icing applications. Ice-detection here is based on effective heat capacity, where power is applied to a 115×25×2 mm³ nano-engineered specimen for seconds, and the slope of the temperature rise is shown to be well correlated to the thickness of ice present. Silver epoxy vertical electrodes (32) on the top face and horizontal electrodes (8) on the back face were printed and connected to a DC power supply (see Figure 1). Two thermocouples were mounted, one on the center of the specimen and the other ~ 15 cm to the left of the center of the sample. Different amounts of water were poured on top of the sample, cooling it down to -15 °C to cause icing. A vacuum tape frame was assembled around the specimen to prevent water leaks [8].



Fig. 1. Ice-detection sample. The electrodes (perpendicular white lines) were printed by direct writing.

3 Results and Discussion

3.1 Non Destructive Evaluation by Infrared Camera

When a potential difference was applied to an impact damaged nano-engineered composite (Figure 2), electric field lines concentrate in the vicinity of cracks as electrons flow around the damage, causing "hot spots" via Joule heating. This effect was

amplified because the heat flow was impeded in areas of damage. These changes of temperature can be locally visualized through a conventional IR thermal camera. Low-power operation (a nine volts standard battery is exemplary, providing a 15 °C rise at 1 Watt) and high spatial resolution was demonstrated that is beyond state-of-the-art in nondestructive evaluation [7].

Multiple applications have been identified and investigated using this Nano-Engineered Thermal NDE (NET-NDE) method. In typical engineering structures where composite components are joined by metallic rivets or bolts, cracks and other defects may form during the structure's operational life in the vicinity of the attachment. Inspecting such a region (5.06 mm diameter hole) using the NET-NDE directly with two electrode tips that apply 12.3 V on the surface of the composite component, allows damaged areas to be identified and later verified by careful visual inspection. A significant drawback of applving advanced composites in aerospace components is detecting internal damage, as exemplified by "barely visible impact damage" (BVID) that is caused when the composite structure is damaged internally with little or no visual evidence on the external (visible) surface. Impact damage has been detected by applying a 0.11 A, 0.34V between two surface electrodes in prior work [7].



Fig.2. Power and resolution characteristics of nanoengineered non-destructive evaluation (NDE)

technique: (a) optical image and (b) thermograph of a composite specimen powered by a 9V battery.

3.2 Non Destructive Evaluation Using a Thermochromic Ink

Thermochromic inks are made of substances, in our case liquid crystals, that display different colors at

different temperatures. When a potential difference is applied to a specimen with a non visible damage (Figure 3), Joule heating increases the temperature of the specimen. The electric field lines concentrate around the damaged area, producing an increment of temperature, which can be detected as a red color on the non visible damage region 25 seconds after the power is applied (Figure 3). If the voltage is applied for longer times, the whole sample heats above 30 °C, changing its color completely.



Fig 3. Optical damage detection: (a) Optical image of a composite specimen with non visible damage in

the center: (b) When a difference of potential is applied (9V battery), a change of color is produced in the damaged region.

Compared to a thermal camera, which is sensitive to the infrared radiation generated by the sample, a thermochromic coating changes its color when the heat is transmitted to the surface by thermal conduction. Thermochromic paint can also measure a smaller range of temperatures than thermal cameras.

3.3 De-icing

The nano-engineered composite material is investigated for another multifunctional application: de-icing and anti-icing. Icing, in the flight environment with a temperature range from ~ -40° C to 0°C, is a serious problem that has caused several aircraft incidents. The nano-engineered composite has demonstrated the capability to reach high temperatures to melt ice (see Figure 4) using very low power: a composite specimen (9.6 cm³ in volume) buried under 25-mm-thick snow (ambient temperature -10°C) melted in 15 minutes at 22W power.



Fig.4. De-icing experimental setup of the heated (22.2W for 15min) composite material embedded in snow (-10°C, 2.5cm of snow thickness).

3.4. Ice-detection and De-Icing/ Anti-icing System

First, the amount of ice on the specimen was calculated [8]. It has been estimated from the ice detection algorithm that the ice thickness on the specimen is correlated with the rate of temperature rise of the ice when the specimen is heated (Equation 1):

$$\frac{dT_{Heating}}{dt} \propto m_{ice} \tag{1}$$

Where $T_{Heating}$ is the temperature of the sample when it is heated with a constant power supply, *t* is the heating time, and m_{ice} is the mass of the ice. As the ice area is kept constant by the vacuum tape enclosure, the mass of water is proportional to the thickness of ice.

This algorithm is in agreement with the experimental values of several specimens at -15 °C ambient temperatures with varied thickness (0, 1, 2, 3 mm of water) of ice on top of them (see Figure 5).

Once the amount of ice was determined, a closedloop system was performed for anti-icing (0 mm of water) and de-icing (to melt the iced stored on the sample). To determine the power required, 2.8 g of water was poured on the sample placed in a freezer at -15 °C. The most effective power to avoid ice formation was 2W for the size of samples tested [8]. For the de-icing test, several water thicknesses were evaluated (0.1, 2, 3 mm with start temperatures of - 5, -10 and -15 °C). Low power was applied for a short time $(5kW/m^2 \text{ applied for less than } 2 \text{ min}, \text{ starting temperature of -5°C) [8].}$



Figure 5. Heating rates of several specimens with water on the surface placed at -15 °C.

4 Conclusions

Multiple applications have been identified using the enhanced electrical properties of the nanoengineered FFRP laminates, such as crack detection in composite components joined by metallic fasteners, crack detection in structures impacted with internal damage, and *in-situ* progressive damage monitoring during a tensile strength test [7]. The thermal nano-engineered NDE technique demonstrated here can provide a new and effective inspection route for monitoring future generations of safer infrastructure materials. Different detection techniques have been used, including thermal cameras or thermochromic coatings. Further research will analyze the differences and advantages of both techniques, as well as their sensitivity to different kinds of damage. It must be emphasized that aligned-CNT containing composites have the primary advantage of enhanced structural properties, with the multifunctional aspects of NET-NDE and or de-icing/anti-icing being realized as multifunctional benefits.

A novel method for ice-detection, de-icing and antiicing has been performed, demonstrating benefits over current systems. Lower power operations were obtained compared to conventional resistive heating blanket approach for de-icing systems. In this case, the mechanical structure is the sensor and the heater itself, enabling closed-loop operation and lighter structural systems. Nano-engineered composites constitute a truly multifunctional material [9], with higher mechanical performance than conventional advanced composites [5], and the potential for sensing and heating applications among others, all implemented by the material itself.

Acknowledgments

This research was sponsored by Air Force Office of Scientific Research, under contracts FA9550-09-C-0165 and FA9550-11-C-0002, and by the Department of the Navy, under contract N68335-10-0227 where work was performed by Metis Design Corporation in collaboration with the Massachusetts Institute of Technology in Cambridge, MA. Nanoengineered composites utilized in this work and other technologies were developed with support from Airbus S.A.S., Boeing, Embraer, Lockheed Martin, Saab AB, Sprit AeroSystems, Textron Inc., Composite Systems Technology, Hexcel, and TohoTenax through MIT's Nano-Engineered Composite aerospace STructures (NECST) Consortium.

References

- M. Kupke, K. Schulte, and R. Schuler, "Nondestructive testing of FRP by d.c. and a.c. electrical methods," *Composites Science and Technology*, vol. 61, no. 6, pp. 837-847, May. 2001.
- [2] E. T. Thostenson and T.-W. Chou, "Carbon Nanotube Networks: Sensing of Distributed Strain and Damage for Life Prediction and Self Healing," *Advanced Materials*, vol. 18, pp. 2837–2841, 2006.
- [3] C. Li, E. T. Thostenson, and T.-W. Chou, "Sensors and actuators based on carbon nanotubes and their composites: A review," *Composites Science and Technology*, vol. 68, no. 6, pp. 1227-1249, May. 2008.
- [4] S. Thomas, R. Cassoni, and C. Mac Arthur, "Aircraft anti-icing and deicing techniques and modeling," AIAA 34th Aerospace Sciences Meeting and Exhibit, 1996.
- [5] S. Wicks, R. Guzman de Villoria, and B. L. Wardle, "Interlaminar and Intralaminar Reinforcement of Composite Laminates with Aligned Carbon Nanotubes," *Composite Science and Technology*, vol. 70, no. 1, pp. 20-28, 2010.
- [6] N. Yamamoto, A. J. Hart, S. Wicks, E. J. Garcia, B. L.Wardle, and A. H. Slocum, "High-yield atmospheric-pressure growth of aligned carbon nanotubes on ceramic fibers for multifunctional

enhancement of structural composites," *Carbon*, vol. 47, no. 3, pp. 551-560, 2009.

- [7] R. Guzman de Villoria, N. Yamamoto, A. Miravete, and B. L. Wardle, "Multi-Physics Damage Sensing in Nano-Engineered Structural Composites," *Nanotechnology*, vol. 22, p. 185502 (7pp), 2011.
- [8] S. S. Kessler, C. T. Dunn, S. Wicks, R. Guzman de Villoria, and B. L. Wardle, "Carbon Nanotube (CNT) Based Aerosurface State Awareness for the BAMS UAS," in 8th International Workshop on Structural Health Monitoring, Stanford, CA, 2011.
- [9] H. Cebeci, R. Guzman de Villoria, A. J. Hart, and B. L. Wardle, "Multifunctional properties of high volume fraction aligned carbon nanotube polymer composites with controlled morphology," *Composites Science and Technology*, vol. 69, no. 15-16, pp. 2649-2656, 2009.