TRAINING THE ENGINEERING STUDENTS ON NANOFIBER-BASED SHM SYSTEMS

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Abstract

The undergraduate engineering students were trained on the structural health monitoring (SHM) systems for the detections of aircraft composite damages using carbonized electrospun polyacrylonitrile (PAN) fibers. The carbonization process was conducted in two different steps: i) oxidation at 270°C in a furnace for 1 hr, and ii) carbonization in an Argon atmosphere at 750, 850, and 950°C for additional 1 hr. The PAN nanofibers were placed on the pre-preg carbon fiber composites with 0, 45, -45 and 90° stacking sequences, and co-cured in a vacuum oven. The extracted carbon fiber composites associated with the carbonized PAN nanofibers were used as a strain sensor during the loading and unloading of the carbon fiber composites panels. The electrical resistivity values of the nanofibers were changed at different strain conditions. The surface hydrophobicity of the carbonized samples were also measured and the test results were evaluated in detail. During this study, the undergraduate engineering students were involved in the tests to give them hands-on experience in understanding the new technology and stimulate their desire for pursuing advanced studies in engineering fields.

Keywords: Electrospinning, PAN Nanofibers, Pre-preg Carbon Fibers Composites, Conductivity, Surface Hydrophobicity, SHM.

1. Introduction

Carbon fibers have superior stiffness, strength, as well as thermal and electrical conductivities. These fibers are extensively used in the development of high load bearing composite materials. The conventional carbon fibers are prepared from polyacrylonitrile (PAN) due to its high carbon yield (up to 56%) and flexibility of tailoring final carbon fiber products. With the help of rapidly developing electrospinning technique, various fibers can be produced in a cost-effective way. The fibers can be employed for tissue engineering scaffolds, photoactive catalysts, micro or nano actuators, bio and nanofilters, separation membranes, ultrasensitive sensors, and structural health monitoring (SHM) applications [1-4].

Generally, in the electrospinning process, the polymeric solution is charged with the high voltage DC power supply, and electrospun using different process and system parameters. The volume feed rate of the polymer is controlled by using a capillary pump. When the electric repulsive force overcomes the surface tension of the polymeric solution, the solution tends to release from the tip, stays stable for about 2-4 cm and then bending instability takes places for the spiral motions. During which time, the diameters of the fibers are substantially reduced from micro to nanoscale.

Evaporation of the solvent can make a significant impact on the fiber diameter and shape. Finally, the stretched fibers are collected on the metallic collector and dried in a room temperature [3-5].

Zhengping et al. [1] used PAN homopolymer in the electrospinning process to produce nanofibers of 330 nm diameter. The authors varied the final carbonization temperatures from 1000 to 2200 °C to make highly carbonized and crystalline carbon fibers. It was found that with the increase of carbonization temperatures, carbon nanofibers became more graphitic and structurally ordered. Both electrical conductivities and mechanical properties were tested and the test results showed that these properties were substantially increased with the increase of the carbonization temperatures [1].

SHM is an important field of study for safe operation of various structures, such as aerospace, pipelines, tunnels, highways, skyscrapers, bridges, and so on. Since the carbon fibers possess excellent mechanical, tehrmal and electrical properties, they can be ideal materials for aerospace, construction and automotive industries. Conductive PAN-derived electrospun carbon fibers can be used on the surface of the structures to monitor any structural changes during the manufacturing and service, as well as prevention of the moisture ingressions on the composite structures. In addition to the PAN-based nanofibers, there are several other different techniques, such as piezoresistive sensors, and fiber optic ribbon tapes (FORT) were also employed for the same purposes [6, 7].

The wettability of a surface is a significant characteristic of nature for our everyday life. It describes the ability of a liquid to maintain contact with the surface, and this feature can be in the forms of hydrophobic and hydrophilic. A surface of a material is considered as hydrophobic if the water contact angle is in the range of 90 to 150° and hydrophilic if it is in the range of 0 to 90°. It is also defined as superhydrophobic surface when the water contact angle is between 150 and 180°. The wettability of a surface plays a critical role for surface cleaning, corrosion resistance, biosurface, anti-biofouling, structural color, enhancing buoyancy, fuel cell application, anti-contamination, biosensors and transportation of fluid [8-10].

Carbon is a versatile material due to its electrical and thermal conductivity values, porosity, broad potential operating range and low cost. Different promising techniques have been developed to prepare superhydrophobic surface using carbon fibers, carbon nanotubes, and graphene for commercial applications [9]. Zhou et al. [11] used the magnetron sputtering technique to prepare amorphous carbon films with nanostructured surface deposited on silicon and glass substrates at different substrate temperatures. The authors showed that the pure carbon films exhibited different wettability, ranging from hydrophilicity with a contact angle less than 40° to superhydrophobicity with a contact angle more than 152°. It reveals that the surface wettability of amorphous carbon films can be controlled well using nanostructures with different geometrical and carbon state features [12-16].

Inspired by the versatile use of carbon nanofibers, these fibers can be used as a conductive surface for the SHM on composite panels. The composite panels can be made from pre-impregnated (pre-preg) composite with several layers and then electrospun PAN-derived carbon fibers can be applied as top layer. A constant current can be implemented in the axial direction through outer probes of a strain gauge and the corresponding voltage change between inner probes can be

measured for various strains along the axial direction. The voltage changes can be analyzed with different loads. Investigating the surface wettability for self-cleaning and anti-contamination, the contact angle can be studied on the conductive carbon films. Also, involving undergraduate engineering students will give them hands-on experience in understanding the new technology and stimulate their desire for pursuing advanced studies in the engineering fields.

2. Experimental

2.1 Materials

PAN and dimethylformamide (DMF) were purchased from Sigma-Aldrich and used without any further purifications or modifications. Pre-preg carbon fiber composites were purchased from a local store and utilized in these tests.

2.2 Methods

2.2.1 Fabrication of PAN Nanofibers

PAN powder was well dissolved in DMF solvent with 10:90 % weight ratios at 500 rpm on a hotplate for about 1 hr. The polymeric solution was then transferred to a 10 ml syringe with an inside needle diameter of 0.5 mm. One end of a copper electrode having 0.25mm diameter was attached to the needle, while the other end was connected to the power supply of the electrospinning unit. Nanofibers were fabricated at 25kV DC power supply with 1 ml/h feed rate of the polymer solution and a spinneret to collector distance of 25 cm. Figure 1 shows electrospinning process used in the present study.

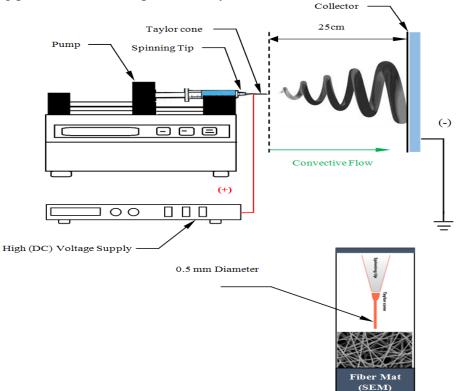


Figure 1: Schematic view of the electrospinning process.

Prior to the carbon fiber composite fabrication, the carbonization process was conducted on the electrospun PAN nanofibers in two different steps: i) oxidation at 270°C in a furnace for 1 hr, and ii) carbonizations in an Argon atmosphere at 750, 850, and 950°C for additional 1 hr. Different temperatures will change the crystallinity and other physical properties of the PAN-based nanofibers.

2.2.2 Fabrication of Carbonized PAN Fibers on Pre-preg Composites

Composite panels were fabricated using pre-preg carbon fibers. Figure 2 shows the pre-preg composite panel model with conductive nanofibers as the top layer. On a flat and smooth Al plate, 10 peel plies of the pre-preg carbon fibers were laid at 0, 45, -45 and 90° stacking sequences. Then carbonized PAN nanofibers were placed on the top of the surface of the pre-preg composite panel to make a conductive surface. In the fabrication process, release film, flat metal sheet, berating film and vacuum bag film were used before sealing with a tacky tape. The debulking procedure was performed under vacuum at 27in Hg overnight. The pre-preg with carbonized PAN nanofiber was cured in four steps: first heating the composite for 30 min at 50 °C, second heating for 1 hr at 120 °C, third heating the for 2 hr at 180 °C, and fourth reduced heating for 30 mins. at 50 °C [13]. The carbonized PAN nanofibers were cohesively bonded on the top surface of the panel without any damage.

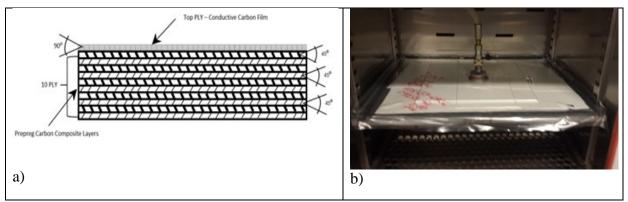


Figure 2: a) The schematic view of a model, and b) curing the pre-preg composite panel with carbonized PAN nanofibers in a vacuum oven.

2.2.3 Mechanical Testing of PAN Reinforced Composites

A four circumferential ring probe was used to measure resistance on the surface of the composite panels. Through outer probes, a constant current applied in the axial direction, and the corresponding voltage drop was measured between the inner probes at various axial strain conditions. The specimen was subjected to tension and compression cycles in the servo-hydraulic test frame. Figure 3 shown the experimental setup for testing the strain sensing response of composite panels with carbonized PAN nanofibers. The test specimen was subjected to a constant 1mm/min loading and unloading cycles. The input current was maintained constant at the outer probes during the measurements of a potential difference between the two inner probes and strains from the strain gauge. The displacement due to loading was used to calculate strain rates under quasi-static loading. Data was acquired using the Lab View program.

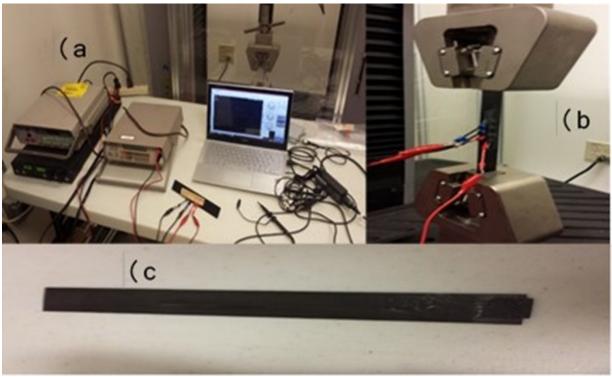


Figure 3: Experimental setup: a) equipments used for testing of the strain sensing response, b) the specimen with 4 probes to measure voltage changes in a load frame, and c) the test specimen with a length of 177.8 mm, width of 50.8 mm and thickness of 1.26 mm.

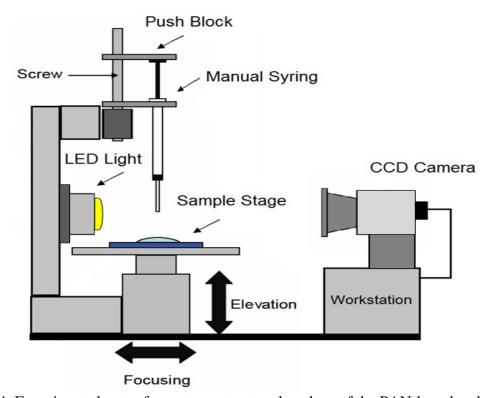


Figure 4: Experimental setup for water contact angle values of the PAN-based carbon fiber reinforced composites.

2.2.4 Hydrophobicity of Carbonized PAN Nanofibers on Pre-preg Composites

A goniometer (KSV CAM 100 Contact Angle Meter) was used to determine the surface wettability of the composite panels. A small droplet of DI water was placed on the surface of the compaste panles by means of a small syrange to measure the water contact angle values. Figure 4 shows the experimental setup for water contact angle values of the PAN-based carbon fiber reinforced composite panels.

3. Results and Discussion

Figure 5 illustrates the plot of stress vs strain and strain vs time values of the test specimens of the composite panels. The stress vs strain plot shows almost a linear relationship during the loading because of the brittle structures of the composite panels. The plot of strain vs. time also showed a linear relationship. Figure 6 depicts the plot of a voltage difference across the two inner probes in the four circumferential ring probes as a function of time. There was a sudden increase in voltage difference at the beginning of the test. After 25 seconds of operation time, voltage difference increased gradually with the same trend. A linear relationship found between the change in voltage and change in strain. It is believed that the change in the voltage between the two inner probes was because of change of dimensions of the carbon fiber specimens, and it can be attributed to the change of resistance of the composite panel surface.

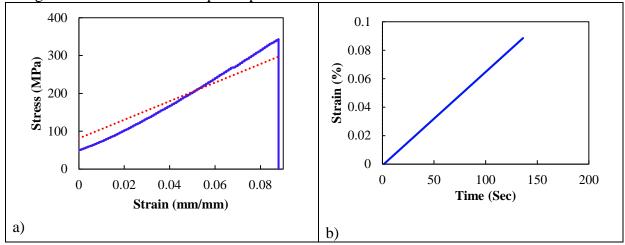


Figure 5: The plots showing a) the stress vs strain and b) the strain vs time for the composite panels.

Figure 7 shows the plot of the surface resistance of test specimen as a function of time. It was observed that there was a sudden decrease in resistance of the composite coupons at the beginning of the test. After 60 seconds of operation time, the resistance remained almost consistent about 1.9 m Ω for the rest of the operation time. The specimen resistivity might be due to the change of dimension of the test specimen while applying the loads. After the certain dimensional changes on the composite panels, the permanent structural damages can be observed.

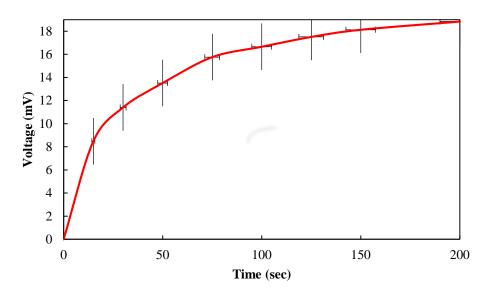


Figure 6: The plot showing the voltage differences across the inner probes of the composite panels as a function of time.

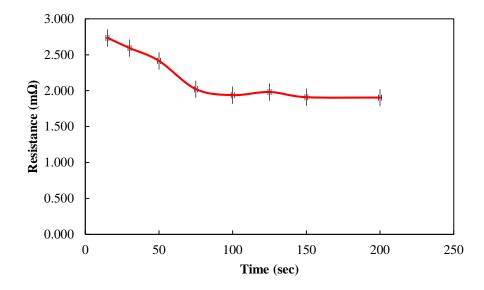


Figure 7: The plot of the surface resistance of test specimen as a function of time

Figure 8 illustrates the water contact angle values on the surfaces of the composite panels covered with PAN nanofibers carbonized at 750°C, 850°C and 950°C. The water contact angle studies showed that the PAN fibers carbonized at 750°C, 850°C and 950°C provided 155°, 157.8°, and 154.3° contact angles, respectively. Highest water contact angle was observed at 850°C carbonization temperature. The test results also indicate that all the water contact angle values are in superhydrophobic range and will significantly repel the water molecules from the surface, which may be useful for the service life of the SHM systems developed in this study. These superhydrophobic surfaces may also prevent the moisture ingression of the composites panels.

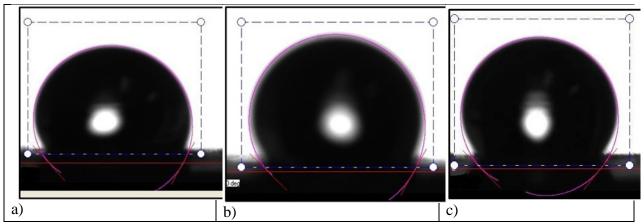


Figure 8: The images showing the water contact angle values on the surfaces of the composite panels co-cured with PAN nanofibers carbonized at a) 750°C, b) 850°C and c) 950°C.

4. Benefits of Studies for Undergraduate Students

Undergraduate students in the Department of Mechanical Engineering at WSU were involved in every step of the present study, learned many new technologies (nanotechnology and composite technology) and gained a lot of skills and knowledge about fabrication, characterization, and evaluation of the data produced during the studies. The students realized that the new SHM systems could be used in various industries, such as aircraft, automobile, energy, transportation, constructions, and so on. The students mentioned about their experiences to their peers, and affected them towards advanced technology and hands-on experiences. Because of these discussions, new students came and joined our group for the similar studies. The students joined our group used these research activities as their own Engineer of 2020 requirements. One of the students (Mr. Omar Alsaiari) is also a co-author of the present study. We believe that these activities will motivate many BS students to do further studies in these fields for their MS and PhD degrees.

5. Conclusions

Electrospinning technique was used to prepare PAN nanofibers. These fibers were carbonized at 750°C, 850°C and 950°C, and then placed them on the pre-preg carbon fiber composites prior to the vacuum oven curing. The test panels were extracted from the composite samples and tested at different stress vs strain, and strain vs time to determine the electrical property changes during the loading and unloading. The test results showed that the electrical resistance values were considerably increased at higher loadings, indicating that some structural damages took place during the loads. The surface of the samples were found to be superhydrophobic, which may be useful for the service life of the composites and prevention of the moisture ingressions on the composite structures. The undergraduate students were involved in every steps of these work and grained a lot of new technologies, skills and experiences to share with their peers.

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References

- [1] Zhou, Z., Lai, C., Zhang, L., Qian, Y., Hou, H., Reneker, D. H., & Fong, H. "Development of carbon nanofibers from aligned electrospun polyacrylonitrile nanofiber bundles and characterization of their microstructural, electrical, and mechanical properties." *Polymer* 50.13 (2009): 2999-3006.
- [2] Nataraj, S. K., K. S. Yang, and T. M. Aminabhavi. "Polyacrylonitrile-based nanofibers—a state-of-the-art review." *Progress in polymer science* 37.3 (2012): 487-513.
- [3] Park, Jun-Seo. "Electrospinning and its applications." *Advances in Natural Sciences: Nanoscience and Nanotechnology* 1.4 (2010): 043002.
- [4] Chen, Q., Zhang, L., Rahman, A., Zhou, Z., Wu, X. F., & Fong, H. "Hybrid multi-scale epoxy composite made of conventional carbon fiber fabrics with interlaminar regions containing electrospun carbon nanofiber mats." *Composites Part A: Applied Science and Manufacturing* 42.12 (2011): 2036-2042.
- [5] Cramariuc, B., Cramariuc, R., Scarlet, R., Manea, L. R., Lupu, I. G., & Cramariuc, O. "Fiber diameter in electrospinning process." *Journal of Electrostatics* 71.3 (2013): 189-198.
- [6] Salvado, R., Lopes, C., Szojda, L., Araújo, P., Gorski, M., Velez, F. J., & Krzywon, R."Carbon Fiber Epoxy Composites for Both Strengthening and Health Monitoring of Structures." *Sensors* 15.5 (2015): 10753-10770.
- [7] Charlaftis, P., Loutas, T. H., Koimtzoglou, C., & Kostopoulos, V. "Structural Health Monitoring of Aerospace Components with Optical Fibre Ribbon-Tapes."
- [8] Shih, H. R., Walters, W. L., Zheng, W., & Everett, J. "Course Modules on Structural Health Monitoring with Smart Materials." (2008).
- [9] Meng, L. Y., & Park, S. J. "Superhydrophobic carbon-based materials: a review of synthesis, structure, and applications." *Carbon Letters* 15.2 (2014): 89-104.
- [10] Bharathidasan, T., Narayanan, T. N., Sathyanaryanan, S., & Sreejakumari, S. S. "Above 170° water contact angle and oleophobicity of fluorinated graphene oxide based transparent polymeric films." *Carbon* 84 (2015): 207-213.
- [11] Zhou, Y., Wang, B., Song, X., Li, E., Li, G., Zhao, S., & Yan, H. "Control over the wettability of amorphous carbon films in a large range from hydrophilicity to superhydrophobicity." *Applied Surface Science* 253.5 (2006): 2690-2694.
- [12] Czél, G., Pimenta, S., Wisnom, M. R., & Robinson, P. "Demonstration of pseudo-ductility in unidirectional discontinuous carbon fibre/epoxy prepreg composites." *Composites Science and Technology* 106 (2015): 110-119.
- [13] Alarifi, I. M., Alharbi, A., Khan, W. S., and Asmatulu, R. "Electrospun Nanofibers for Improved Electrical Conductivity of Fiber Reinforced Composites," SPIE Smart Structures/Non-Destructive Evaluation Conference, March 8-12, 2015, San Diego, CA, 8 pages.
- [14] Asmatulu, R., "Nanotechnology Safety," Elsevier, Amsterdam, The Nederland, August, 2013.
- [15] Kececi, E., and Asmatulu, R. "Effects of Moisture Ingression on Mechanical Properties of Polymeric Laminate Composite Structures and Its Prevention by Hydrophobic Barrier Films," *International Journal of Advanced Manufacturing Technology*, 73(2014): 1657-1664.

[16] Zhang, B., Asmatulu, R., Le, L.N., Kumar, S.S.A., and Soltani, S.A. "Mechanical and Thermal Properties of Hierarchical Composites Enhanced by Pristine Graphene and Graphene Oxide Nanoinclusions," *Journal of Applied Polymer Science*, 131 (19) (2014), DOI: 10.1002/app.40826

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Dr. Asmatulu received his Ph.D. degree in 2001 from the Department of Materials Science and Engineering at Virginia Tech. After having the postdoc experiences, he joined the Department of Mechanical Engineering at WSU in 2006 as an assistant professor, and received his tenure and promotion to be associate processor in 2012. He is currently working with 13 M.S. and 8 Ph.D. students in the same department. Throughout his studies, he has published 76 journal papers and 166 conference proceedings, edited two books, authored 33 book chapters and 4 laboratory manuals, received 35 funded proposals, 15 patents and 34 honors/awards, presented 91 presentations, chaired many international conferences and reviewed several manuscripts in international journals and conference proceedings. To date, his scholarly activities have been cited more than 1050 times, according to the web of science.