Carbon Nanofiber Composite Structures

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Abstract

This material development project looks into fabricating and testing nanofiber composites, with and without surface treatments, that produce consistent data. Composites made from commercially available Pyrograf-III® carbon nanofiber in molded polypropylene will be studied. Pyrograf-III® is a patented, highly graphitic, low cost, carbon nanofiber produced by Applied Sciences, Inc. Pyrograf®-III is produced with diameters ranging from 70 to 200 nanometers and a length of around 50 to100 microns. Polypropylene is choosen as a matrix material, because of its many applications and widespread use. Two types of Pyrograf-III® nanofiber dispersed in the polypropylene will be tested. The first is a nanofiber with no surface treatment that has been tested before by Applied Sciences, Inc. The second is a fiber with a surface treatment that Applied Sciences, Inc. has not performed extensive tests on.

Project Objectives

Carbon reinforced composite structures have become more and more popular on aircraft and spacecraft because of their light weight, strength, stiffness, and fatigue properties. The major drawback with fiber reinforced composite structures is their poor conductivity and anisotropic physical properties. Carbon nanofiber, on the other hand, makes composite structures that have excellent electrical, thermal, and mechanical properties. They also have an extremely high length to width aspect ratio. However, these carbon nanotube composites are currently limited because of the broad range of electric, thermal, and structural properties that change depending on their positioning within a system. In order to fully use the nanotube composites, ways of dispersing the nanofibers in highly uniform patterns without compromising their high aspect ratio need to be found. In past experiments Burton[1] showed ways to improve electrical and mechanical properties with surface modification and debulking which impact dispersion. Finegan[2] also discussed how surface treatments improve mechanical properties of thermoplastic composites reinforced by carbon nanofiber.

This project looks into testing Pyrograf®-III carbon nanofiber[3]. To test these composites accurately, a repeatable way of fabricating test specimens needs to be specified. Once the experiment is proved repeatable, then the composites will be tested for electrical properties. The electrical properties can then be examined and compared with nanofiber composites that have different surface treatments. These comparisons will be helpful in finding what fiber loads commonly produce optimal resistivity. In addition, these comparisons will show what surface treatments give the best overall resistivity.

Methodology

In order to make comparisons between nanofiber composites, a repeatable method of forming and testing them is needed. To ensure the method of forming and testing the composites was correct, a carbon fiber that had previously been tested by Applied Sciences, Inc. was used as a model. If the tests in this experiment returned matching results, then confidence could be placed in this new test's data.

Tests of resistivity are desired for the nanotube composites. The test composites were made using a MiniMax® injection molder. These first composites were composed of an untreated Pyrograf®-III nanofiber loaded into polypropylene. The loadings were five and ten percent nanofiber by weight. Ten of each percentage were made so average values could be found within each load group.

The resistivity tests on the composites were done using a four point probe. The four point probe used was the same probe that Applied Sciences, Inc. had used in all resistivity tests they previously performed.

This probe measured the resistance in the bars and then this resistance was used to calculate the total resistivity of the bar. A wiring diagram for the four point probe is shown in Figure 1 of the Figures and Tables section. The following equation was used to calculate the resistivity of each sample.

$$\rho = \frac{RA_{cs}}{L}$$

ρ is the resistance of the sample in ohms, R is the measured resistance of the specimen, A_{cs} is the cross sectional area of the specimen, and L is the distance between the points where the probe was applied.

The first test conducted was the resistivity of the five and ten percent by weight nanotube composites. The results obtained showed a lack of repeatability. Resistivity values were near what Applied Sciences had found for this specific weight loading and polymer, but the data is very inconsistent. Figure 2 shows data from the five percent by fiber weight composites that were first made. Under those conditions, specimens made with the MiniMax® molder contained too many visible voids making resistivity testing imprecise. The ten percent fiber specimens were not even tested due to the obvious visible flaws in their fabrication. Methods of improving the quality of the bars tested for resistivity and the method of testing were then improved.

Different techniques of fabricating the resistivity bars were tested. These fabrication changes included packing the fiber and polymer into the MiniMax® molder. This technique helped remove any air that might be trapped in a loosely packed fiber polymer mix. Stirring the nanofiber and the polymer for various lengths of time was also investigated. After packing the fiber and polymer into the molder, these components were heated for two minutes at 250°F. The polymer fiber mixture was then manually stirred. Specimens were stirred for various lengths of time ranging from 10 seconds to 45 seconds. These specimens were then formed, cut apart, and inspected. Stirring the mixture made the resistivity bars visibly more homogeneous. The length of stirring was found not to matter, but whether or not the stirring was done did matter. The amount of fiber and polymer that were injected into the mold was also changed. Since the density of the fiber was different than that of the polypropylene, the hopper on the MiniMax molder would often be to full or not full enough depending upon the percentage of fiber and polymer. This problem was eliminated by simply increasing or decreasing the total weight of fiber and polypropylene that was packed in the hopper.

Resistivity testing techniques also changed. Conductive coatings that electrically connected the four point probe to the composite specimens were altered. The conductive silver paint was allowed to dry before testing the specimen instead of testing when still wet as done in prior experiments. These changes in fabrication and testing soon made resistivity bars that were close to being visibly flawless. Results produced when resistivity testing was done varied little when compared to data from previously done tests. Figure 3 shows resistivity data for composites tested after the fabrication and testing modifications were made.

With confidence in resistivity testing, the next step in experimentation could begin. This involved finding a percolation curve for a specific silver metalized Pyrograf-III® nanofiber. This percolation curve gives researchers information on what fiber loadings produce the best resistivity values in composite specimens. The curve was found by first loading the nanofiber into polypropylene at 5% by weight. Five of each of these specimens were made. The number of test specimens was chosen based upon advice from Applied Sciences, Inc.'s experience in performing percolation tests. The starting point of 5% loading was based upon previous tests done on similar Pyrograf-III® fibers. Resistivity at this point was greater than the four point probe could measure. The fiber loading was then increased by 5% to bring the resistivity down into a range that could be read. This process of increasing fiber load by 5 % was repeated until resistivity could be found for the silver metalized nanofiber composite tested. Once resistivity could be measured, then fiber loading was increased in intervals of 2.5% until the resistivity value leveled off at a minimum. The percolation curve is shown in Figure 4 of the Figures and Tables section.

Results Obtained
The first resistivity test samples showed little promise of any consistent and repeatable data. After the technique of fabricating these samples was examined and modified, reliable data was obtained. Resistivity test specimens now produce resistivity data that is much more precise than before this experiment took place. There is still work to be done in order to eliminate as much error as possible in composite nanofiber fabrication and testing, but this experiment shows that simple changes in fabrication and test procedure can make a big difference in test results.

Fiber loading in various polymers shows much promise. From the percolation curve data, the effect of silver metalized fiber weight loading on the resistivity of polypropylene is known. Figure 2 in the Figures and Tables section shows a sharp drop in resistivity for the silver metalized fiber composite. This drop occurs between 25% and 30% fiber loading. This percolation curve can now be compared to other percolation test results done on other fibers. These comparisons help Applied Sciences, Inc. choose what nanofiber surface treatments give polypropylene composites the best conductivity values.

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Figures and Tables

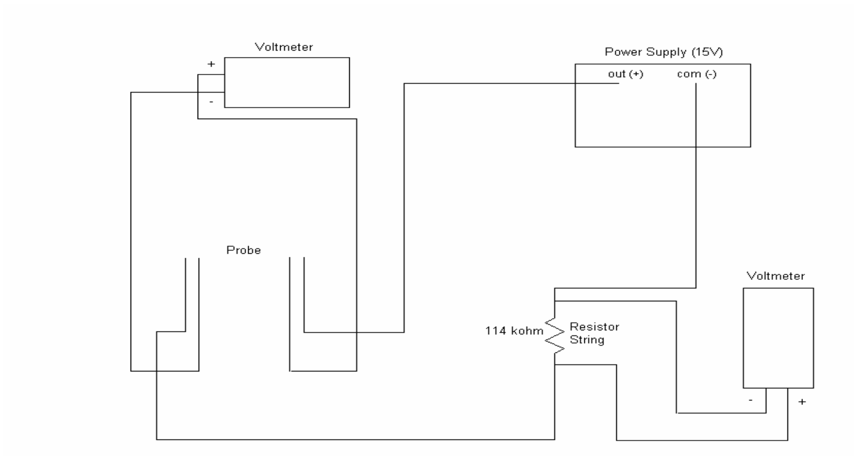

Figure 1. Wiring diagram of four point probe testing apparatus

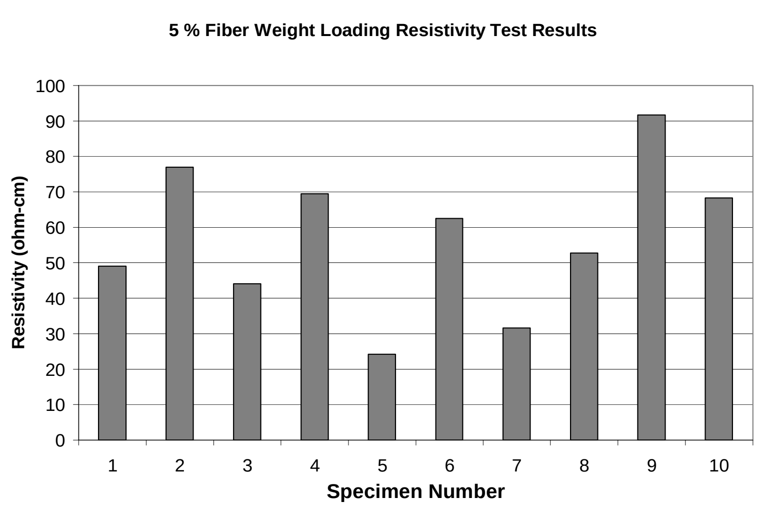

Figure 2. Five percent by fiber weight composites that were made before fabrication and testing techniques were modified.

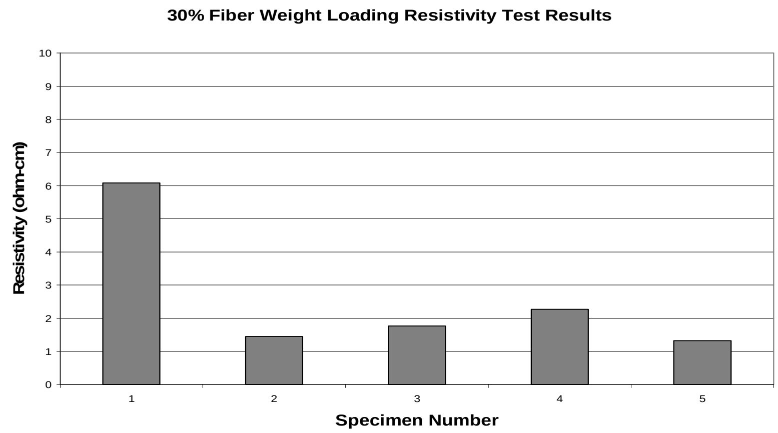

Figure 3. Thirty percent by fiber weight composites that were made after fabrication and testing techniques were modified.

The resistivity graphs Figure 2 and Figure 3 are shown on different scales for ease of reading. Note the number of specimens fabricated and tested at each weight loading was reduced in later testing because of both time and expense. Five specimens were determined to be satisfactory for finding resistivity values. Note the large difference of resistivity values in Figure 2 and the much smaller difference in Figure 3. Work is still being done to eliminate even more of the variation that is shown in Figure 3. The graphs in Figure 2 and Figure 3 are of different fibers and weight loadings, but neither of these factors should effect how precise the resistivity can be read in a specimen.

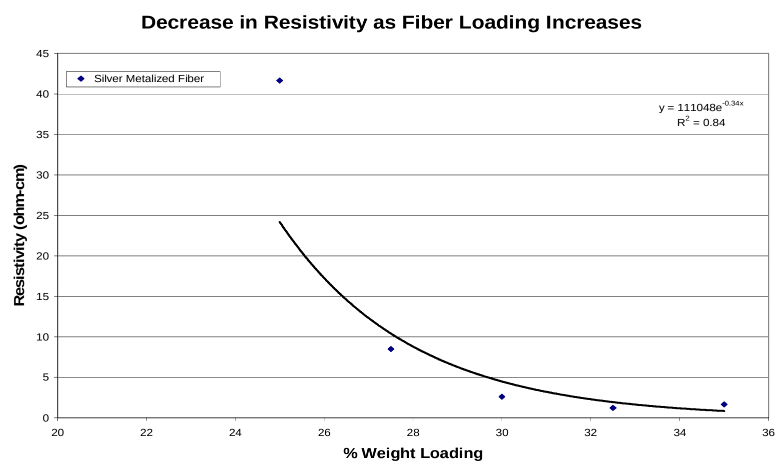

Figure 4. Plot of resistivity vs. percent weight loading for silver metalized fiber composite.

The data shown in Figure 4 was fitted with an exponential curve. Applied Sciences, Inc. is continuing to test this fiber in order to obtain more data points and a better curve fit.

References

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