AC 2007-2475: DEVELOPMENT OF A NANO-FILLED COMPOSITE EXPERIMENT FOR A FRESHMAN CLASS

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Development of a Nano-filled Composite Experiment for a Freshman Class

Abstract

Projects that help first-year engineering and science students become more familiar with concepts of nanomaterials may become an important vehicle for increasing student interest and understanding of the potential of technologies that manipulate materials at the molecular level. The authors received a National Science Foundation NUE award entitled "Infusing Nanomaterials into Undergraduate Science and Engineering Curricula" for the development of an undergraduate Certificate in Nanomaterials within the colleges of engineering and science. As part of the NSF-supported project, but separate from the certificate program, we are developing a series of experiments that will be introduced in the first-year engineering program at a large, research-intensive university. The goal of the experiments is to help students determine how differences in concentrations of nano-sized particles, which are added to common materials, can change physical properties. In this exercise, student teams will fabricate composite films filled with different concentrations of carbon black in two different types of polymers: 1. latex-based composite with poly (vinyl acetate), and 2. solution-based composite with poly (vinylpyrrolidone). The concentrations vary from about 2 wt% to 15 wt%. For mechanical properties, the students will determine the tensile strength and the strain to failure using an Instron test machine with a 1000 lb_f load cell. For electrical properties, the students will determine the electrical resistivity for the different carbon black concentrations.

Preliminary experiments show relatively little change in the tensile strength. However, there is a three to five order change in the composite's resistivity. A select group of freshman will run the experiment during spring 2007. Students will be asked to propose hypotheses to explain why changes in the concentrations change or fail to change physical properties of the materials.

Introduction

Currently, there is a substantial interest in the behavior of materials that are in the nanometer size range.[1] The National Science Foundation has had a nano initiative for a number of years. The size range for nanomaterials is from 1 to 100 nm. At this scale, the physical, chemical, and biological properties of material are different from their bulk material counterparts. For example, if a metal has a grain size of 1 nm, and the grain boundary thickness is assumed to be 0.2 nm and the grains are considered as squares, then the boundary area becomes approximately 50% of the cross-sectional area. Since grain boundaries have different characteristics than does the matrix, the materials properties may be substantially changed. According to the www.nano.gov website there are seven programs in the USA that have degrees in nanotechnology and 15 additional universities with programs and courses in nanotechnology.

The objective of this paper is to describe the development of an experiment that uses carbon black particles in polymeric matrix to form composites with varying amounts of carbon black.[2] The students measure mechanical and electrical properties of the specimens, and from the information collected, they design a sensor for detecting various vapors.

Development of Activity

The authors were awarded an NUE from the National Science Foundation entitled, "Infusing Nanomaterials into Undergraduate Science and Engineering Curricula."[3] The primary purpose of the project is to develop a certificate in nanotechnology and science that students' at Texas A&M University would be able to earn. The current plan is for the students to take five courses in nano related areas and then do a two semester research project that is nano related. Since the program is joint between the Colleges of Engineering and Science, an introductory course has been developed and was taught for the first time during spring 2007. The course is intended to be open to both engineering and science students, so there will be basic mechanics for the science students and basic quantum mechanics and kinetics for the engineering students. This builds on a prior NSF grant that was concerned with issues in nanoscale manufacturing.[4]

As an additional part of the project, we are developing an experiment that is intended to be used in the freshman year as a means of getting students excited about the potential of nanotechnology. The current incarnation of freshman engineering is project oriented and the intent would be to use the experiment in one of the two classes required.

Description of the Experiment

The experiment has three major components: 1. preparation of carbon black containing composite, 2. measurement of electrical and mechanical properties, and 3. development of a vapor sensor.

The current plan is to use emulsion and solution based polymers. The emulsion based polymer is a latex made of polyvinyl acetate (PVAc), while the solution based composite will use polyvinylpyrrolidone (PVP). Carbon black is added to the mixture to form a composite with the highest concentration. The remaining five or six combinations are made by diluting with more polymer and water. For the emulsion based composites 15 wt % will be the maximum, while for the solution based composites approximately 35 wt% will be the maximum.

Once the composite solution contains the correct amount of carbon black the composites are cast into plastic molds about 50 mm square. The molds are made of polystyrene. The thickness is less than 1 mm and the specimens take about one week to dry. Some care must be taken in removing the sheets form the molds as they are relatively brittle and will fail easily. The sheets are cut into 25 mm by 50 mm specimens for testing. The uniformity of the specimens may be determined from the resistivity measurements.

The mechanical properties are determined from a four-point bending for the modulus of elasticity[5] and a tensile test for the tensile strength and the elongation at failure. Electrical resistivity was measured using a multimeter and a caliper to measure the dimensions. Both sets of measurements were to be plotted against the fraction of carbon black in the composite.

Finally with the information from the measurement of resistivity or conductivity, the students were to develop a sensor for detecting different vapors. If possible the sensor is intended to be quantitative.

Results

While we have not actually done this with a class, a student has developed the experiment and proofed the concept. The variation in mechanical properties was not very much. If we examine the work of Grunlan et al.[7], they showed a variation in tensile strength as a function of increased carbon black. This is illustrated in Figure 1 a and b. Our results were not nearly so clear cut as Grunlan's.

However, the electrical resistivity results were much more interesting, and they showed a clear dependency on the amount of carbon black in the composite. These results are shown in Figures 2 and 3, where an estimate of the percolation limit is about 5% for the PVP and about 2% for the PVAc. On the log scale used, there is a three to five order of magnitude change in the resistivity as a function of carbon black.

We have done no detailed development work on the sensor. We have shown with the samples produced that the resistivity changes in the presence of a vapor. Previously, researchers at the California Institute of Technology have developed sensors using very similar systems.[6]

Part of the idea of this experiment is that it is to be open ended so that the students may explore different ideas that they might have. One of the concerns is safety, and there needs to be careful instruction with regards to good safety practice for the students.

Conclusions

We have developed a set of experiments that demonstrate the effect of carbon black on the properties of composites that are intended to be used in a freshman engineering program. We were able to produce composites, make mechanical and electrical property measurements. We did not directly produce a sensor, although the concept was shown to work[6].

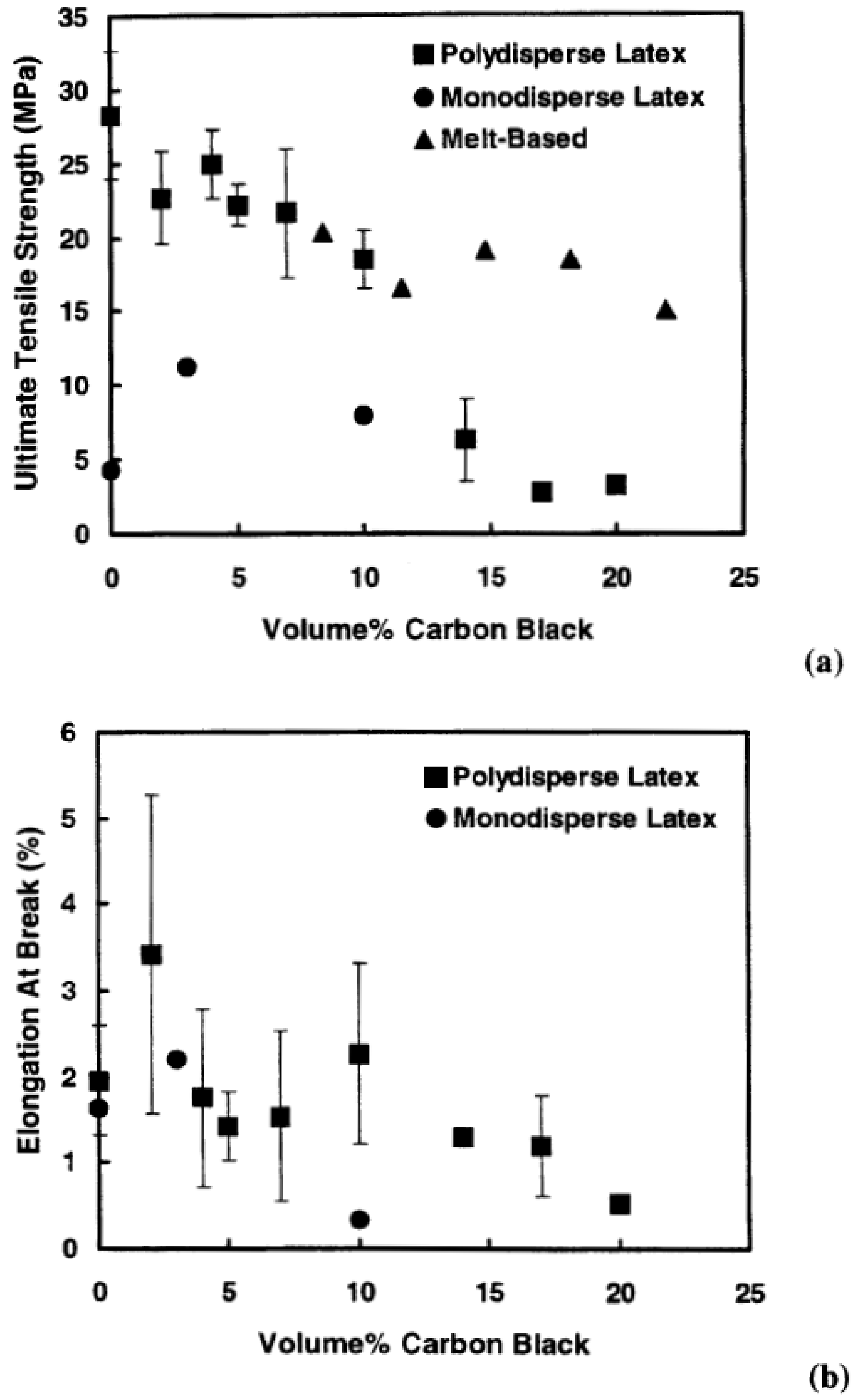

Figure 1. Volume % carbon black versus (a) tensile strength and (b) final elongation.[7]

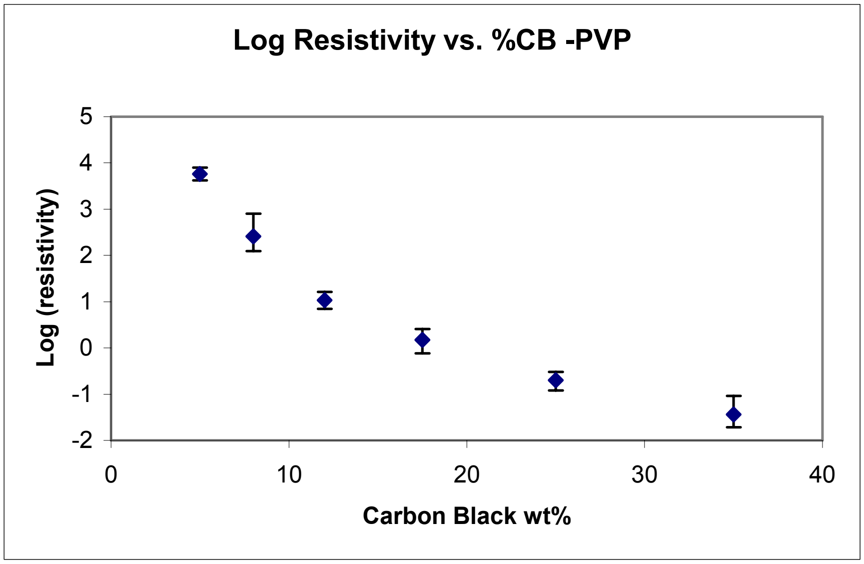

Figure 2. Log Resistivity vs. % Carbon Black concentration- solution based PVP polymer. Percolation limit appears to occur at 5% Carbon Black concentration

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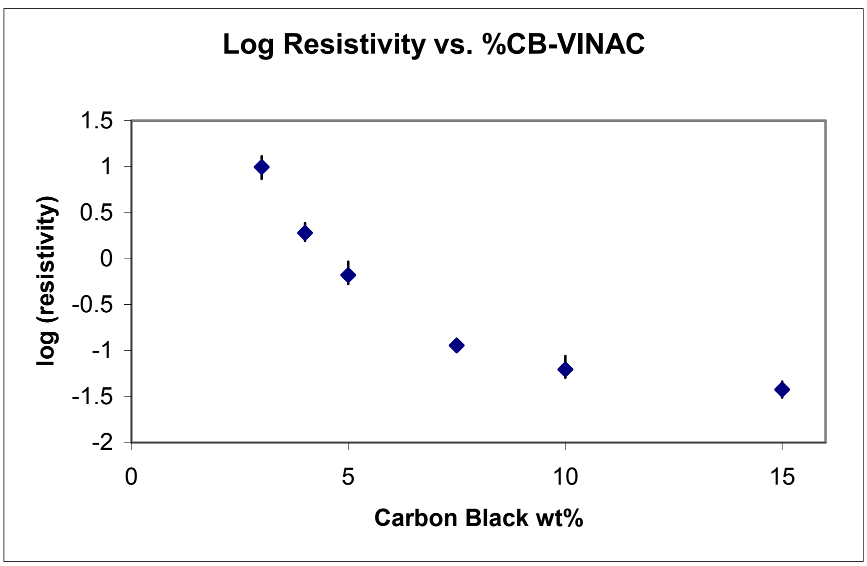

Figure 3. Log Resistivity vs. % Carbon Black concentration- emulsion based Polyvinyl Acetate polymer. Percolation limit appears to occur at 2% Carbon Black concentration

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[1] www.nano.gov

[2] T. S. Creasy, J. C. Grunlan, and R. B. Griffin, "An Undergraduate Laboratory: the Effect of Nanoparticle Microstructure on the Electrical Properties of Polymer Nanocomposites," Proceedings of IMECE06, ASME International Mechanical Engineering Congress and Exposition, November 5-10, 2006, Chicago USA, IMECE2006-14408.

[3] J. Froyd, R. Griffin, I. Karaman, and W. Teizer NUE: Infusing Nanomaterials into Undergraduate Science and Engineering Curricula Grant Number: EEC- 0532576, 2005.

[4] Froyd, J., T. Creasy, I. Karaman, W. Teizer, and R. Caso. Undergraduate Educational Components for Nanoscale Issues in Manufacturing. American Society for Engineering Education. Salt Lake City, Utah June 20-23. Unnumbered, 12 pages.

[5] ASTM Standard D6272.

[6] B. J. Doleman, M. C. Lonergan, E. J. Severin, T. P. Vaid, and N. S. Lewis, "Quantitative Study of the Resolving Power of Arrays of Carbon Black-Polymer Composites in Various Vapor-Sensing Tasks," *Anal. Chem.*, 1998, vol70, 4177-4190.

[7] J. C. Grunlan et al., *Polymer Eng. Sci.*, **41**, 1947 (2001)