Monthly Progress Report

For the project (CCRP 2002-03):

Nanocomposite Materials

By

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ACCOMPLISHMENTS
We are continuing to develop and test different methods for functionalizing nanotubes. Small changes in the processing conditions are causing significant variations in the elastic properties. We are trying to understand these effects and to optimize the processing.

Task 1: Nanotube Synthesis (~6% effort)
We are trying different acids to purify nanotubes (this is not supported by the OAI grant).

Task 2: Coating and Dispersion of Nanotubes (~47% effort)
We are focusing on three methods to functionalize nanotubes. The progress is explained next.

Solvent free functionalization. We have made a new mold which makes thin rectangular samples. We are trying to improve the samples and are using clear epoxy. MWCNT have been functionalized using the solvent-free method. They will be cast when the properties of the clear samples are repeatable.

Plasma treatment with no monomer. An experiment was performed to see if there is any effect by using the plasma alone. The result is that the properties did not change much due to the plasma treatment with no monomer. The results are being graphed. This test verified the coating on the nanotubes is responsible for the improved dispersion and adhesion.

Nanotube Alignment. The TEM results should be done in two weeks to see if the magnetic alignment of the MWCNT in polystyrene done in France has worked.

Task 3: Fabrication and Characterization of Nanocomposites (~47% effort)
We are making variations in our procedures to see the effects. The main results are discussed below.

Alignment of nanotubes. The cylindrical mold to align nanotubes is being tested and should produce a good sample soon.

MWCNT Epoxy Testing. Variations in the elastic properties seem to occur due to small variations and unknown effects in the processing. In processing the MWCNT, we have used sonication and then mechanical mixing. We have determined that the sonication at the level used was reducing the properties, possibly due to breaking the nanotubes into smaller pieces. Also, the mechanical mixing procedure was improved by mixing in a beaker in hot water. This reduced the viscosity and 9 hrs of mixing or longer was used. This procedure of no sonication and mechanical mixing at elevated temperature has produced an improvement in the elastic modulus of 17%, but the results are not always repeatable.

SWCNT Epoxy Testing. This testing was done using detergent to disperse the nanotubes but no other functionalization. The first experiment using sonication and mechanical mixing with 1% SWCNT by weight produced an improvement in elastic modulus. A second sample was made but without sonication and tested and produced only a small improvement in elastic modulus. Two samples from the same mold for the second experiment were tested and had about equal properties. The first sample that had good improvement was then re-tested and gave the same result. These samples were mechanically mixed for about 9 hours each on different days. The procedure also requires evaporation and heating before the mixing. The time to make these two samples and run the experiments was about four weeks.

We will consider studying the effect of level of sonication for SWCNT and MWCNT. The literature shows the nanotube length reduces with time and level of sonication. Full load transfer from the nanotube to the matrix may require a long nanotube because the bonding is not strong enough for full load transfer in a short nanotube. The critical length of a fiber for load transfer and the rule of mixtures do not take into
account the degree of bonding of the fiber to the matrix. This may be why we are not achieving the properties predicted by the rule of mixtures.

It appears that the sonication level and time, the mechanical mixing level, time and temperature, and the surfactant concentration and temperature to evaporate the water all interplay and the optimization of the process will take significant time. Several experiments have produced moderate improvements in elastic modulus for 1% wt CNT loading. It is likely that further improvement in functionalization, dispersion, and alignment will continue to improve results.

Please send any questions or corrections that should be made to this monthly update to me. Thank you. Mark Schulz.