Manufacturing and Performance of Carbon Nanotube/High Density Polyethylene Composites

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SUMMARY: This study describes the manufacturing of Carbon Nanotube/High Density Polyethylene composites (CNT/HDPE) and the testing of these materials to determine several key material characteristics (i.e. stiffness, tensile strength, work-to-failure and wear resistance). These nanocomposites are made from untreated, multiwalled carbon nanotubes and HDPE through a process of mixing and extruding. Materials were created with varying weight percentages of nanotubes (0%, 1%, 3%, and 5%) and then molded and machined to form standard test specimens for small punch testing and block-on-ring wear testing. Mechanical tests were then conducted for the various volume percentages of nanotube content with pure HDPE as the control. It was found that each of the measured mechanical properties of the composite increased as a function of increased nanotube content in the range studied.

KEYWORDS: carbon nanotubes, high density polyethylene, wear resistance,

INTRODUCTION

There has been a recent surge of interest in the use of nanotubes as a reinforcing phase in composite materials, in an attempt to improve the electrical and/or mechanical properties of the unreinforced materials. Of specific interest in certain cases is the improvement of the wear behavior. For example, a significant contributing factor to artificial hip implant failure is loosening of the femoral stem due to osteolysis, which is often caused by polyethylene wear debris [1]. Therefore, it is commonly believed that improving the wear resistance of the polyethylene used in these implants would reduce the failure rate. It has been shown that the addition of CNTs can improve the tribological performance of Ni-P coatings, copper, and carbon/carbon composites [2, 3, 4]. The purpose of this study is to examine the effect of the addition of carbon nanotubes (CNT) to high-density polyethylene (HDPE). Specifically, the material and tribological properties of this new nanocomposite are of interest for a variety of s g

MATERIALS AND METHODS

Nanotube Composites

Test samples were made with varying weight percentages of carbon nanotubes in high-density polyethylene. The initial preparation provided enough material for all samples, at each CNT weight percentage, to ensure uniformity of material throughout testing. Multi-walled carbon nanotubes (MWNT) produced by a thermal CVD (chemical vapor deposition) process and virgin HDPE (Marlex® 5502) were used. Appropriate quantities of polyethylene and nanotubes were carefully measured and combined into beakers to produce mixtures of 0%, 1%, 3% and 5% CNT by weight. The beakers were placed in an oven at 200^{o}C for ten minutes to melt the HDPE and then taken out and mixed with a glass rod. The mixtures were pressed into flat wafers, which were then cut into pellets to prepare them for further mixing in a twin-screw small batch extruder (DACA Micro Compounder). The pellets of composite material were then mixed in the extruder at 175^{o}C for 10 minutes, extruded, cut again into pellets and put back into the extruder for 10 more minutes. After the 20 minutes of mixing in the extruder the material was extruded again. Some of the extruded composite was directly formed into small punch samples while the rest was cut into small enough pieces to be compression-molded into wear test samples.

In order to compare mechanical properties of the extruded precursor material, a small punch test [5] was performed as described in Tang et al. [6]. This test gives relative measures of the stiffness, maximum strength, and work to failure for each material. Figure 1 shows a typical Load vs. Displacement graph from the small punch test as well as a comparison of the resulting properties for HDPE with 0%, 1%, 3%, and 5% CNT (normalized to 0% results).

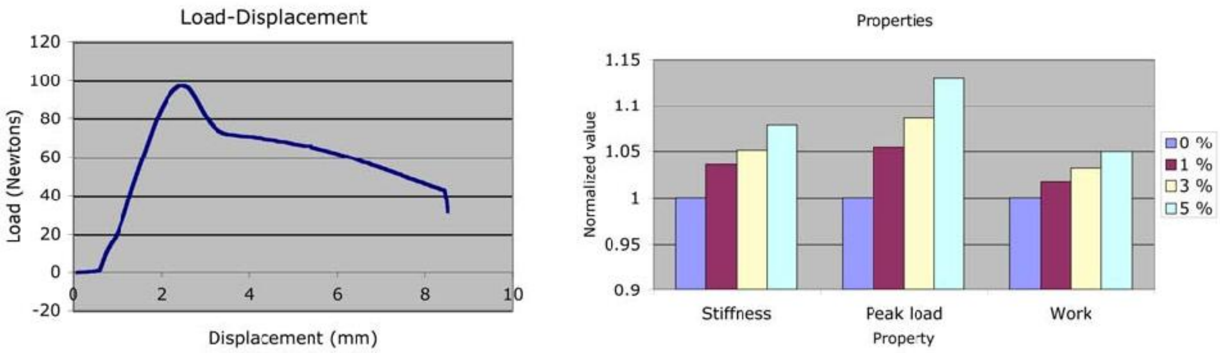

Fig. 1 Typical Load vs. Displacement curve and material property comparison data.

Wear Samples

The various percentages of CNT/HDPE composite materials, prepared as described above, were used to make wear test specimens. Contoured wear samples were produced in accordance to the geometric specifications for use with Falex Block-on-ring wear and friction tester. The samples made of CNT/HDPE material only used a small amount of composite on the wear test surface, with the rest being pure HDPE.

This minimized the amount of composite material used. First a “loaf” of samples was made using a simple compression molding technique. The mold was preheated in a 225°C oven for 20 minutes. A layer of approximately 4g of test material pellets was placed in the mold, covered with 7g of HDPE pellets. This was put back into the oven for 20 minutes with the mold top on and weight applied. The mold was then removed and additional HDPE material was added and placed in the oven for another 20 minutes. Finally, the mold was removed from the oven and allowed to cool prior to removal of the material. The “loaf” was then machining into individual samples (slices).

Wear Testing

Wear testing was performed using a Falex block-on-ring wear and friction tester. A diagram of the test configuration and a photo of the chamber are shown in Figure 2. The experimental procedures were adapted from ASTM G 77 and D 2714 test methods [7]. A load of 200 lbs weight was used and the ring was set to run at a rate of 200 rpm. De-ionized water was used in the chamber for lubrication and to allow for dissipation of heat produced from friction between the wear ring and test sample. Labview software was the acquisition interface used to collect data for friction, displacement, number of cycles, and the temperatures of the test chamber and the wear sample. Data was recorded each minute, over a period encompassing 500,000 cycles. After a test was completed, the water was drained from the chamber and any out-of-the-ordinary aspects were noted such as cloudy or oily water or extensive wear debris.

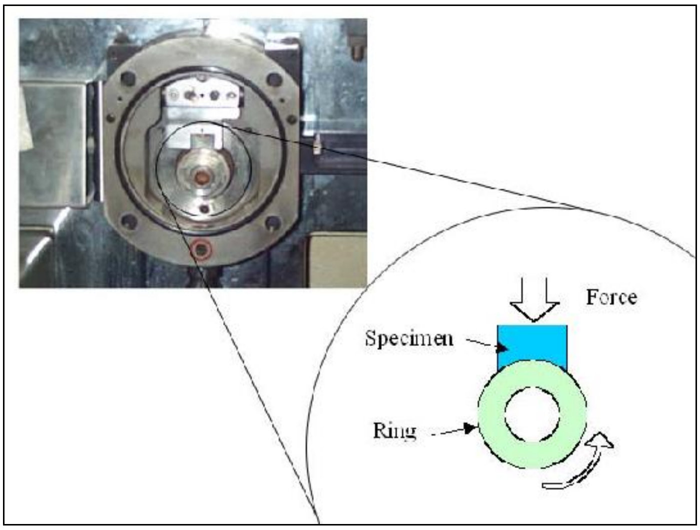

Fig. 2 Test chamber of Falex Wear Tester and diagram of ring and block with rotation and force directions.

RESULTS AND DISCUSSION

As found by Tang et al. [6] material properties determined using the small punch test changed as a function of CNT percentage. As the weight percentages of CNTs were increased the stiffness, maximum strength, and work to failure all increased. Comparing the 0% to the 5% CNT samples the stiffness, maximum strength, and work to failure showed improvements of approximately 8%, 13%, and 5% respectively. In addition to showing that the CNTs improve material properties of HDPE this also confirmed that the materials produced were acceptable for use in further testing to determine tribological properties.

The wear data collected was approximated by a logarithmic curve-fit which gave a value for the wear rate (mm) vs. log-cycles. For each sample, the logarithmic curve was required to meet a minimum correlation (R-squared) value of 90%. The standard deviations of each sample grouping were also calculated. It is apparent from the data that wear decreases with increased weight percentage of CNTs. An improvement of 26% decrease in wear was seen from 0% to 1% samples and an improvement of nearly 40% was seen between the 0% and 5% samples. Figure 3 shows two different plots of the comparison of wear rates for the different samples. It was determined that standard deviations present within each sample group are primarily caused by variation of sample manufacturing processes. Manufacturing steps were strictly followed, however at the micro level there are still deviations in material structure such as polymer chain alignment, branching, and crosslinking. Also, as discussed in Tang et al., the dispersion of the CNTs becomes an important issue both on micro and nanoscale levels, which causes variation of material structure throughout the sample's volume. However, even with these small deviations, the overall trends of the data show that there is improved wear characteristics with increasing weight percentages of nanotubes. Wear tests are currently being run for the 3% samples and data will be reported in the near future.

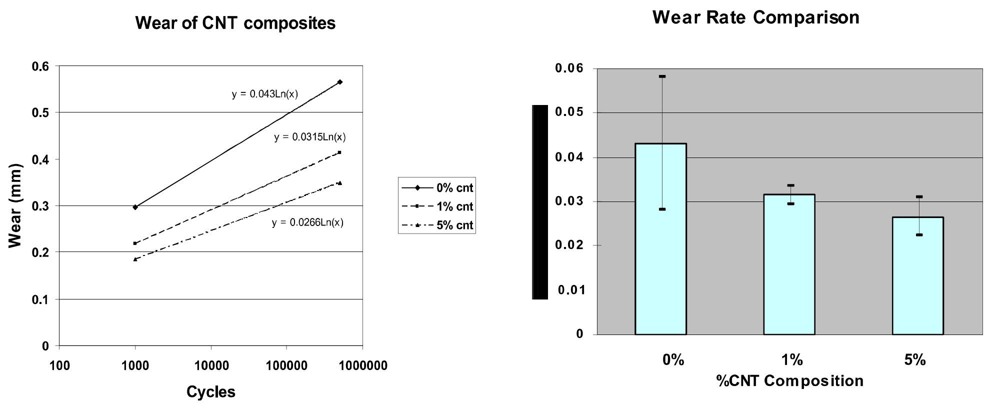

Fig. 3 Wear Rate Comparison for varying CNT%

In some cases after a test was run the water from the test chamber was cloudy due to contamination from a small amount of oil, these occurrences were attributed to small amounts of oil leaking from the spindle housing into the test chamber and were corrected by checking the oil level in the spindle housing and changing it to the appropriate level. In some other instances, samples wore at an extremely high rate and large amounts of shredded polymer material were present in the water and the test chamber. These results were thrown out based on the assumption that there was some type of material defect in the sample that caused it to rapidly fall apart.

CONCLUSIONS

Varying weight percentages of CNTs were added to HDPE in an attempt to improve material properties of HDPE including the wear resistance. As measured by the small punch test, the addition of CNTs improves the material properties compared to the virgin HDPE material. The addition of CNTs to HDPE also decreases the wear rate of the material and this value decreases as the percentage of CNTs is increased. In addition to improvements in stiffness, tensile strength, work to failure and the tribological properties, the addition of CNTs is also likely to change other material properties such as fracture strength and fatigue crack resistance. The authors are currently conducting a series of fracture tests to evaluate these effects as well as additional wear tests on different %CNT samples and hope to report on these in the near future.

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