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WEAR BEHAVIOR OF CARBON NANOTUBE/HIGH DENSITY POLYETHYLENE COMPOSITES

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Abstract

Carbon Nanotube/High Density Polyethylene (CNT/HDPE) composites were manufactured and tested to determine their wear behavior. The nanocomposites were made from untreated multi-walled carbon nanotubes and HDPE pellets. Thin films of the precursor materials were created with varying weight percentages of nanotubes (1%, 3%, and 5%), through a process of mixing and extruding. The precursor composites were then molded and machined to create test specimens for mechanical and wear tests. These included small punch testing to compare stiffness, maximum load and work-to-failure and block-on-ring testing to determine wear behavior. Each of the tests was conducted for the different weight percentages of composite as well as pure HDPE as the baseline. The measured mechanical properties and wear resistance of the composite materials increased with increasing nanotube content in the range studied.

1. Introduction

The development of composite materials has allowed for the engineering of specific mechanical properties based on various combinations of fibers and resins, microstructural geometries and manufacturing processes. This ability to engineer the desired properties provides control over the design process to meet specific technological needs. Engineering of some properties can best be accomplished through combining materials at smaller and smaller size scales. With the need for increasingly demanding combinations of material properties, nanocomposites have become an area of interest for possible solutions. Nanocomposites are composed of a matrix material and fibers or particles in the nanometer size range. Varying the combinations and architectures of these materials affects the material properties on the nano-, micro- and macro-scales. Thermal, electrical, magnetic, mechanical, and tribological properties can be engineered for specific applications (Fischer 2003).

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The present study was motivated by the need to engineer a wear-resistant bearing material for use in artificial joints. There has been a vast improvement in the success of artificial joints since the original hip implant designs in the 1930's. Most significantly, structural failures of both the device material and the initial fixation of the implant to bone have been reduced. With these two major issues addressed, new areas of concern for improving implant performance have arisen from previously less important problems. Generation of wear debris and the resulting biological response is currently considered the major cause of implant failure (Sochart, 1999; Wright and Goodman, 2001). Over the lifetime of the hip implant, contact and relative motion between the metal and the polyethylene articular surfaces of the implant generates polyethylene wear debris. This wear debris migrates to the tissues around the implant where it can initiate an inflammatory biological response and osteolysis of the bone. Eventually this osteolysis causes loosening and failure of the implant and revision surgery is required (Amstutz et al. 1992). This failure process has led to a search for new implant materials which have better tribological and biological response characteristics. In order to decrease osteolysis, new bearing materials are sought to produce less wear debris over the lifetime of the implant. In addition, the wear debris produced from any new material must have a similar or reduced capacity to induce an osteolytic reaction relative to currently used materials.

Ultra-High Molecular Weight Polyethylene (UHMWPE) is currently the preferred material for the polymer surfaces of the implant. Various methods have been developed to modify its material behavior, most recently, cross-linking techniques. Although cross-linking has been shown to significantly reduce wear, it can have other, detrimental effects on the polymer including reduction of strain to failure and fracture toughness (Baker et al., 1999; Lewis, 2001). Additional research has been focused on the use of metal on metal implants and ceramic on ceramic implants, but to date, none has proven to be consistently superior to UHMWPE. The addition of carbon nanotubes to UHMWPE is seen as a possible way of improving its wear behavior without necessarily compromising other mechanical properties. The cyto-toxicity of carbon nanotubes is currently a subject of debate that warrants further study (Monteiro-Riviere, et al. 2005)

There have been numerous studies performed involving the addition of carbon nanotubes to various matrix materials to improve one or more material properties, but we will focus here on the several that address wear and frictional properties. Lim et al. (2002) added nanotubes to carbon/carbon composites in order to improve the tribological properties. They performed ball on disc wear tests on carbon/carbon composites with 0, 5, 10, and 20 weight % nanotubes added. The tests were run with a loading of 1.5 N and at a rate of 0.5 m/s. It was found that the wear resistance increased with increased nanotube content, with the 20% nanotube material showing a 50% decrease in wear compared to the 0% material. However, friction of the material increased slightly with the addition of nanotubes. This study showed that there was indeed an improvement in wear resistance as a result of the addition of CNTs. However, the testing conditions in this study were not very demanding and the authors concluded that tests should be performed using higher loads and higher rates of loading.

Chen et al. (2002) added nanotubes to Ni-P based composite coatings in an attempt to improve the friction and wear behavior. They performed ring-on-block wear tests under oil lubricated conditions. An initial period of wear lasting 20 minutes was run with a loading of 200 N to allow for a wear-in period of the sample. Following the wear-in period, testing was performed for a 6 hours with loads of 800 N and loading rates of 800 rpm. The authors did testing for the Ni-P-CNT composite material as well as Ni-P, Ni-P-SiC, and Ni-P-graphite materials for comparison. Their results showed that the Ni-P-CNT material had an 80% decrease in wear compared to the Ni-P material. The friction coefficient was also smaller for the Ni-P-CNT compared to all the other materials tested. With loads of 800N and rates of 800 rpm, this study provides a much more severe condition of wear than the study by Lim et al. (2002).

Dong et al. (2001) added nanotubes to copper and studied the resulting wear properties. Comparisons were made between nanotubes/Cu composites, carbon fiber/Cu composites and pure Cu. Wear tests were performed for each sample and it was found that the coefficient of friction as well as wear volume decreased significantly for the carbon nanotubes/Cu composite materials relative to the other materials. The authors also found that there was an optimum weight percentage content of nanotubes, below and above which the properties decreased. They speculate that the improved wear properties with nanotubes could be the result of the creation of a carbon film at the surface of the material which may act as a lubricant and prevent oxidation of the Cu prolonging the integrity of the material.

Zoo et al. (2004) added nanotubes to UHMWPE to determine the impact this had on tribological properties. Samples of 0.1%, 0.2%, and 0.5% carbon nanotubes by weight in ultra high molecular weight polyethylene (UHMWPE) were fabricated for ball-on-disc wear testing. After testing of each sample, SEM imaging was used to compare the worn and unworn surfaces. The results of testing showed that the wear loss of the 0.5% CNT material decreased by about 90% compared to the plain UHMWPE. The investigators also concluded that the coefficient of friction increased slightly as the weight percentage of nanotubes increased. Zoo et al. hypothesized that the improvement in properties was due to increased shear strength of the UHMWPE caused by addition of CNTs. They also stated that the increase of friction coefficients could be due to the change of morphology of the surface caused by CNT addition as well as the increased shear strength.

The current investigation is motivated by a desire to produce a material for use in artificial joints, which has the capability of reducing the production of wear debris. As described above, carbon nanotubes have proven to be a possible source of improved wear characteristics when added to a variety of materials. Therefore, we have developed a new nanocomposite material comprised of high-density polyethylene and multi-walled carbon nanotubes. If this study shows that the wear behavior of HDPE can be improved by the addition of nanotubes, without a critical reduction in other relevant structural properties, the technology can be extended to the production of CNT/UHMWPE composites. Small Punch testing has been performed to compare other material properties and SEM imaging has been used to qualitatively assess the dispersion of CNTs.

2. Nanocomposite Material Manufacturing

2.1. Manufacture of Precursor Composite Material

Multi-walled carbon nanotubes (MWNT) produced by thermal CVD (chemical vapor deposition) processes and HDPE pellets were used in the composite material. The CNTs were purchased from ILJIN Nanotech. The ratio of nanotubes to polyethylene used was determined by the selected composite weight percentages of 1% 3% and 5%. Figure 1 is a schematic showing the manufacturing procedure used for production of the composite material. Steps a–e were performed by hand and no additional chemicals were used in the process. In step f, a DACA® small batch compounder/extruder was used in order to obtain further dispersion of the nanotubes. This compounder/extruder was a twin screw driven machine, which mixes materials at a set rate in a heated chamber. A die is fixed to the outside of the chamber to extrude material into either a thin rod or films of specific thickness. A total mixing time of 20 minutes, found in previous investigations to yield good nanotube dispersion (Tang et al., 2003), was used for all the materials created for this study.

2.2. Molding and Machining of test Samples from Composite Material

Extruded CNT/HDPE composite films, prepared as described above, were used to make test specimens. Punch test samples were pressed directly from the extruded materials using a simple

mold. The mold was an aluminum plate with wide grooves machined to the desired sample thickness and a surfaced aluminum plate was used for the mold top. After the molding, disc-shaped specimens were cut from the film. Contoured wear samples were produced from pellets cut from the extruded films and compression molded in accordance to the geometric specifications for use with Falex® Block-on-ring wear and friction tester (ASTM G 77). First, four-inch long prismatic bars of material were molded from the precursor material pellets using compression molding techniques. The bar was made for slicing into multiple samples much like slices of bread are cut from a loaf. The bar of material was then machined and sliced into individual samples, meeting the geometric tolerances.

3. Material Characterization of Composite Materials

3.1. Small Punch Test

A small punch test technique was used to compare the general mechanical properties of the newly created composites and unreinforced HDPE. This testing technique, created by Kurtz et al. (1997) and adapted by our research group, uses small disc-shaped samples to find material stiffness, maximum load, and work to failure. The test, as developed, bases material property calculations on the assumption that data extracted from extremely small deformations allows for elastic property evaluations. In our tests, we found these calculations gave inconsistent results due to the limited sample size of the data at such small deformations. Therefore, we based our comparisons on the data throughout the range of loading and considered these results as a relative measure of the material behavior and not as a means to evaluate the actual elastic properties.

The punch samples made were 0.5 mm thick and 6.35 mm in diameter. Punch testing was performed using a hemispherical indenter apparatus on a materials testing machine (Instron MicroTester, Canton MA). The testing machine provided position measurement resolution of around 20nm. Figure 2 shows the small punch test setup.

Punch tests for the different CNT percentage composite materials showed that there were changes in material properties such as stiffness, maximum load, and work to failure. As can be seen in Figure 3, these material properties increase with the increase of CNT weight percentage. In this figure, the properties are normalized with respect to the values for the unreinforced HDPE (i.e. all the 0% CNT results are shown as the value one). Since the testing included plastic as well as elastic deformation the stiffness values are not to be interpreted as elastic moduli. In fact, the slope of the load deflection curve was evaluated for the loading from 0–75N. Therefore, the stiffness measured is an indication of the relative stiffness of the materials and includes plastic as well as elastic deformation.

3.2. SEM Imaging

Scanning Electron Microscopy (SEM) was used to qualitatively assess the dispersion of CNTs throughout the composite material. Samples of the materials were dipped in liquid nitrogen and cracked to produce a brittle fracture surface then imaged under SEM. Figure 4 shows the results of this imaging and reveals that there is fairly good dispersion of CNTs compared to other manufacturing processes tested. However, the clumping of nanotubes still remains a major issue (Fan et. al., 2004). The small lighter spots in Figure 4b are the nanotube clumps. Currently, new steps in the manufacturing process, such as oxidizing the CNTs prior to mixing, are being tested to find a way to better disperse the CNTs and reduce clumping.

4. Wear Testing

4.1. Materials and Methods

Wear testing was conducted using a Falex® block-on-ring wear and friction tester. A diagram of the test configuration with a photo of the chamber is shown in Figure 5. The experimental procedures were adapted from ASTM G 77 and D 2714 test methods.

The testing conditions selected, provided an approximate simulation of a severe case of hip joint loading. Loading in the hip is typically several times body weight and cyclical. Depending on the activity of the person (walking, running, climbing, etc), this load varies considerably and can reach up to eight times the total body weight. The amount of loading used in testing was calculated based on the ratio of the surface area between an average hip implant and the contoured wear test specimen. A 45 kg (100 lb) load, applied to the contoured wear test specimen was chosen as a convenient loading for the wear test setup. This load corresponds roughly to 6 times bodyweight loading, on a 28 mm diameter hip, for a 90 kg (200 lb) person.

De-ionized water was used in the chamber for lubrication and to maintain temperatures at around 40 Celsius. In more aggressive tests, bovine serum should be used to simulate the synovial fluid in the joint; however, for these initial tests, we chose to simplify the procedure and limit the number of variables. LabView® software was used for data acquisition to record friction, displacement, number of cycles, and the temperatures of the test chamber and the wear sample. The tester ran at a rate of 200 rpm and data was recorded every minute, over a period encompassing 500,000 cycles, which is approximately equivalent to 6 months of walking for an average person.

The test rings were purchased from Falex® Corporation and were made of ASTM #UNS-S31603 (Low Carbon Stainless Steel) with a highly polished surface finish. Prior to testing, isopropyl alcohol was used to clean off the test rings and all the metal surfaces in the test chamber. After a test was completed, the water was drained from the chamber and any abnormal aspects were noted, such as cloudy or oily water or extensive wear debris. Each new sample run in the tester used a new, unused stainless steel wear ring.

The test data acquired for each sample had to meet certain criteria, which determined if they were reportable. The criteria for acceptable test data included the following:

- The quality of each sample was visually inspected before and after testing. Air bubbles or other defects were noted and in cases where they clearly affected the wear test, the data was not used.
- The tests were all run for a minimum of 400,000 cycles with the standard cutoff being 500,000 cycles. Therefore, any test that did not run a minimum of 400,000 cycles was not included. The few data sets that cutoff between 400,000 and 500,000 due to equipment issues were still used and the data scaled to account the fewer number of cycles.
- A logarithmic, best-fit line was fitted to the data. If the correlation of this trend line was above 80% the data was deemed acceptable. Otherwise, the data was discarded.
- Visual inspection of the wear test chamber was done after every test. If there was an obvious flaw with the conditions of the interior of the chamber the test data was discarded. Flawed conditions included: excess material disintegration or presence of foreign substance in water such as oil or corrosion.

4.2. Wear Test Results and Discussion

From the test data, wear rates and friction coefficients were compared for nine samples of pure HDPE 0% CNT, four samples of 1% CNT, four samples of 3% CNT, and five samples of 5% CNT composite material. The first step of the analysis was to determine how best to characterize the wear-versus-cycles data. As stated above in the criteria, a logarithmic best-fit curve was fitted to the data. In our tests, this function most accurately describes the wear-versus-cycles data, even after larger numbers of cycles. This became evident when attempting to describe the steady state wear portion of a test. Figure 6 shows test data from a typical sample with a best-fit logarithmic curve.

The 97% correlation of this curve is typical of the tests conducted, and by visual inspection it is apparent that the wear data does indeed follow a logarithmic curve. The beginning portion of sample testing (the highly curved portion from 0 – 250,000 cycles) is considered to be a wear-in period, or run-in period for the sample. During this period, the wear surfaces are newly surfaced and any minor defects in the sample are being worn down. After a number have cycles have passed, steady state wear is assumed. From the data, 250,000 cycles was chosen as the wear-in period after which the surface abnormalities should have smoothed out. The wear data was then analyzed between 250,000 and 500,000 cycles to determine a steady state wear rate. Figure 7 shows test data for the steady state wear portion of the same test with a linear best-fit line.

Typical long-term wear rates for hip implants are reported as if wear was a linear function in time. Standard clinical testing determines the amount of material loss over the lifetime of the implant and wear rates are reported in units of mm/10⁶ cycles, approximating mm of wear per year (with mass wear rates in mg/10⁶ cycles) (Kaddick and Wimmer, 2001). As described above and seen in Figure 6, the typical data from the contoured block-on-ring wear fit a logarithmic curve. During steady state, correlations between the data and linear trend lines were extremely low, ranging from 30% to 60%. This implies that in the experiments conducted, wear was not a linear function of cycles. However, it is generally assumed that steady state wear of hip implants follow a linear trend, so the linear best fit lines in steady state periods of testing, as shown in Figure 7, are reported as well as the logarithmic wear rates. It is important to remember that the data being reported is not directly comparable to clinical wear rates or the more aggressive wear testing in hip simulators. However, the data does report very relevant comparisons between new potential implant materials giving a good preliminary study on the potential wear behavior of materials in artificial joints.

Figures 8–10 show comparative data for wear and wear rates. Most of the data collected followed the expected trend; decreasing wear rates with increasing nanotube content. However, there was some discrepancy with a few tests. Some wear tests performed, yielded data that did not match with the typical results. The majority of these samples had disintegrated in the wear test chamber during testing causing these differences. A few runs seemed to be physically intact and still had very different wear rates or wear versus cycle plots that were not monotonic or logarithmic. These anomalous results were generally attributed to equipment error and the appropriate repairs were made, including recalibration of the sensors.

The test data confirmed the hypothesis that the addition of nanotubes to HDPE, decreases the wear rate of the material. Table 1 shows average wear rates and the decrease in wear rates for increased CNT by weight content. Table 2 shows the average friction coefficients and their comparison to the 0% CNT material.

Additionally, the results show that the friction coefficients decreased with the addition of nanotubes. However, unlike the wear rates, there was not a clear, monotonic trend of decreasing friction coefficients for increased percentage of CNTs. In other studies involving the addition

of nanotubes to matrix materials (Chen et al., 2002; Dong, 2001; Lim, 2002; Zoo, 2004) sometimes friction increased, sometimes it decreased indicating that the friction mechanism is complex and that there are probably competing factors associated with it. Due to nanotubes' extremely low frictional properties (Cummings and Zettl, 2000) it is possible this decreased friction could be due to worn off CNTs acting as a lubricant, much like powder graphite lubricants. However, the CNTs also change the wear behavior and possibly the adhesion of water molecules to the articular surface. It is important to remember that these friction coefficients are under lubricated conditions so are not equivalent to friction coefficients of the actual materials.

After testing of various CNT percentage materials, preliminary samples of Ultra High Molecular Weight Polyethylene (UHMWPE) were tested for comparison. Samples were made using a hot press technique, which supplied the extremely high pressures required to polymerize the UHMWPE particles via diffusion. These samples were more wear resistant than unreinforced HDPE but comparable to the 5% CNT composite.

5. Conclusions

The results of punch tests demonstrate that the addition of CNTs to HDPE improves certain material properties compared to those of unreinforced HDPE. With the addition of 5% by weight of CNTs, the overall material stiffness increases by 8%, the maximum load to failure by 13% and the work to failure by 5%. The wear resistance and frictional properties of HDPE are also improved by the addition of CNTs. The wear tests showed that the addition of 5% by weight of CNTs decreased the overall wear rate by up to 50% and friction coefficients decreased by at least 12%. It was also seen that the addition of CNTs to HDPE could bring wear rates down to the level seen in UHMWPE, the material currently in common use for artificial joint implants.

These results show promise for the use of nanotubes as a reinforcing material to improve the wear resistance of HDPE and possibly UHMWPE. Furthermore, the preliminary results show that the addition of a few weight percent of CNTs does not negatively affect other material properties and actually improves certain ones. The CNTs that were used in this study were not of extremely high quality. As discussed, they were MWNTs produced by CVD process. There are currently higher quality nanotubes available, including SWNTs. However, since these lesser quality CNTs improved the material as shown in this study, it is reasonable to assume that higher quality CNTs might have an even greater impact on the material properties.

Noting the affects of CNTs on HDPE and the structural similarities between HDPE and UHMWPE, it is likely that the addition of CNTs to UHMWPE will result in similar improvements. This is important because improving the current UHMWPE material in hip replacements is a key factor for increasing the lifetime of artificial joint implants. Adding CNTs to UHMWPE is the next logical step for future studies with more direct clinical application. Once material processing techniques are developed for good dispersion of CNTs in UHMWPE and molding techniques are perfected, other potential material improvements could be investigated. Future tests will include fracture testing, fatigue testing, tensile testing for yield stress and Young's modulus, and additional tests to determine material surface characteristics, wear debris sizes and uniformity, and biological reactivity of the wear debris.

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Figure 1.
Manufacturing of precursor material (a–f show each manufacturing step)

Figure 2.
Punch Testing Setup

Figure 3.
Comparison of material properties (stiffness, work to failure, etc.) for HDPE with varying CNT % content by weight

Figure 4. SEM images of fractured material surface (a) Pure HDPE and (b) 5% CNT/HDPE composite material

Figure 5.
Falex Wear Tester chamber and diagram of ring and block with directions of rotation and force

Figure 6.
Typical wear test data with logarithmic best fit line

Figure 7.
Typical steady state portion of wear test data

Figure 8.
Wear comparison for varying CNT% content by weight

Figure 9.
Wear rate comparison with standard deviations, for varying CNT%

Figure 10.
Steady state, linear wear rate with standard deviation (from 250,000 to 500,000 cycles)

Table 1

Wear rates for varying %CNT content

Material	Average Wear Rates(mm/10 ⁶ cycles)	% Decrease of wear rates compared to 0%CNT	
		Logarithmic Wear	Linear Wear
0%CNT	0.1546	0%	0%
1%CNT	0.0840	25.80%	45.70%
3%CNT	0.0798	29.70%	48.40%
5%CNT	0.0637	38.10%	58.80%

Table 2

Frictional data for varying CNT% content materials

Material	Avg. Friction Coefficient	% Change
0% CNT	0.0506	0.0%
3% CNT	0.0300	-40.7%
5% CNT	0.0443	-12.5%