ISOTROPIC PITCH-CARBON NANOTUBE FIBER COMPOSITES


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Introduction

The extraordinary properties of carbon nanotubes [1-7] have provided the impetus for intense research into a wide range of applications. The development of carbon nanotube composites for advanced engineering materials is one such application that seeks to exploit these properties. Nanotubes dispersed in select matrices is one method of transferring their unique properties to other matrices [8-15]. Isotropic pitch derived carbon fibers generally have lower strength and elastic modulus properties compared to other higher-value high performance fibers. Incorporating nanotubes into isotropic pitches to produce isotropic pitch-carbon nanotube fiber composites may be an alternate method of enhancing the properties of isotropic pitch derived carbon fibers [16], enabling their use for high performance applications.

A coal-derived pitch and a petroleum pitch were doped with multiwall carbon nanotubes (MWNT) and the derived composite materials melt spun to produce a continuous filament. These composite pitch fibers were stabilized and carbonized to yield carbon fibers reinforced with carbon nanotubes ranging in concentration from 1 to 4 %. SEM microscopy analysis of the composite fibers showed both excellent dispersion of nanotubes through the fiber and alignment of nanotubes with the axis of the fibers. The physical properties of these MWNT composite carbon fibers were measured. Tensile strength was found to increase about 27 % and elastic modulus was found to increase by about 17 % for a 1 % loading of MWNT in the coal-derived pitch.

The physical properties of the composite fibers have been measured and the results compared to carbon fibers derived from undoped isotropic pitch feed-stocks.

Experimental

Dispersion of the MWNTs in the pitch matrix was achieved by heating the pitch to a temperature above its softening point in a Haake Polylab Rheomix, where high shear mixing could be applied. Tests were performed to determine the optimum conditions (temperature, mixing time and rotor speed) required to adequately disperse the MWNTs and yield a homogenous product, but without unduly degrading the pitch. At the completion of each test, the composite was allowed to cool, recovered from the mixer, and crushed to yield a granular product. Samples were set in resin, sectioned and polished to assess dispersion by optical microscopy.

MWNTs were prepared using a method described previously [17].

The pitch/MWNT composite samples were transferred to a Wayne bench scale extruder fitted with a 6.2mm diameter screw and 0.3mm dia x 1mm capillary die. Tests were conducted to determine the conditions under which the samples could be successfully extruded to produce a continuous thread. Feed size distribution and the temperature profile along the barrel and nozzle were crucial to this task. The extruded thread was attached to a wind-up drum rotating at speeds of up to 1700rpm (12m/s) to draw filament in the range 15 to 30 µm. The shear fields generated within the extruder, capillary die and fiber draw-down result in axial alignment of the MWNTs.

Tows of fiber cut from the wind-up drum were stabilized by heating slowly in air up to 310°C. The stabilized fibers are then rapidly carbonized by heating to 1000°C in nitrogen. The tensile properties of the fibers were measured using a MTS QTest instrument for single filament tests following ASTM D3379.

Results and Discussion

Good dispersion was achieved by operating at a temperature of no more than 5°C above the softening point of the pitch. The pitch is then mobile enough to allow processing while having sufficient viscosity to produce high shearing that promotes dispersion. Uniform dispersion with alignment along the axis of the fiber is evident in SEM images of the composite carbon fibers; Figures 1, 2 and 3 show fracture surfaces for pitch fibers with 1 % and 4 % MWNT loadings. There is no apparent effect on fiber morphology as the loading is increased,
although the MWNT distribution density in the fracture surface noticeably increases, Figures 1, 2 and 3.

The characteristics of the pitch matrix apparently have a pronounced effect upon fiber properties. With the coal-derived pitch there is a significant increase in the tensile strength and elastic modulus, about 27% and 17% respectively for the carbon fibers containing 1% MWNTs compared with those produced from the neat pitch. In contrast, with the petroleum pitch, A500, there is no significant change in either tensile strength or elastic modulus as the concentration of MWNTs is increased, Table 1. This may be attributed to the aromaticity of the two pitches. It can be postulated that the highly aromatic coal-derived pitch should have a greater affinity for the MWNTs due to similarities with the aromatic ring structures of these materials compared to the more aliphatic petroleum pitch. It can be further postulated that the MWNTs greater affinity for the coal-derived pitch matrix leads to the enhanced physical properties, Table 1, for the coal-derived pitch-nanotube fiber composites compared to petroleum pitch-nanotube fiber composites.

References

Figure 1. Pitch-nanotube fiber composite fracture surface: Western Kentucky coal-derived pitch with 1% MWNTs

Figure 2. Pitch-nanotube fiber composite fracture surface: Ashland A500 pitch with 1% MWNTs
Figure 3. Pitch-nanotube fiber composite: Ashland A500 pitch with 4 % MWNTs

Table 1. Physical properties of isotropic pitch-carbon nanotube fiber composites

<table>
<thead>
<tr>
<th>Pitch</th>
<th>MWNT Concentration (%)</th>
<th>Tensile Strength (MPa)</th>
<th>Elastic Modulus (GPa)</th>
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<tbody>
<tr>
<td>Western Kentucky Coal-Derived Pitch</td>
<td>0</td>
<td>400</td>
<td>29</td>
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<tr>
<td></td>
<td>1</td>
<td>510</td>
<td>34</td>
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<td>Ashland A500 Petroleum Pitch</td>
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<td>430</td>
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<td></td>
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