NANOTUBE CARBON-CARBON COMPOSITES

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Introduction

The discovery of carbon nanotubes has spawned a tremendous level of activity in carbon research, since the practical realization of their extraordinary properties can open numerous possibilities for new materials [1]. Carbon nanotubes can form a foundation from which to develop generations of advanced materials with unique electrical, thermal, mechanical, and other properties [2]. Here we show that single walled carbon nanotubes (SWNTs) when dispersed in isotropic petroleum pitch matrices lead to nanotube carbon composites and nanotube reinforced carbon fibers with enhanced mechanical and electrical properties. The aromatic character of commercial pitches derived from coal or petroleum suggests that there should be good compatibility with pristine nanotubes, and may even offer prospects for the utilization of unpurified or raw nanotubes. Moreover, pitches can be generated with a range of composition and properties, allowing latitude to match the matrix characteristics with that of the dispersed phase. Composite fibers can be produced by extrusion in which it is possible that mechanical shear can be used to orient the nanotube bundles, much as it effects molecular orientation in the formation of fibers from mesophase pitch. The fiber form is particularly appealing as the steps to the commercial development of fibers for matrix reinforcement, in which the fibers themselves consist of a matrix - nanofiber composite, can be readily envisaged.

Experimental, Results and Discussion

A study has been made of the synthesis and properties of carbon nanotube composites in the form of carbon fibers and sintered discs produced via the addition and dispersion of purified SWNTs in an isotropic pitch precursor.

As-prepared SWNT bundles obtained from Carbolex, Inc. were purified by the hydrothermal method [3]. Ashland A500 pitch was first dissolved in quinoline for 2 hours at 60°C. Purified nanotubes were then added to the hot pitch solution and dispersed using an ultrasonic dismembrator wand (Fischer Scientific) before being transferred to a vacuum distillation apparatus to remove quinoline under reduced pressure. Suspensions with up to 10 wt % SWNT additions were prepared. After cooling to room temperature the pitch-nanotube mixtures were crushed to -150μm and either compressed in a mold at 100°C and 15GPa to produce a porous sintered disc or transferred to a single hole fiber spinning apparatus (Fig. 1). In the spinnerette the feedstock was heated to ~310°C and extruded under 80psi nitrogen pressure through the 300μm orifice. The thread was drawn down to produce fibers with an average diameter of ~18μm by winding onto a rotating drum.

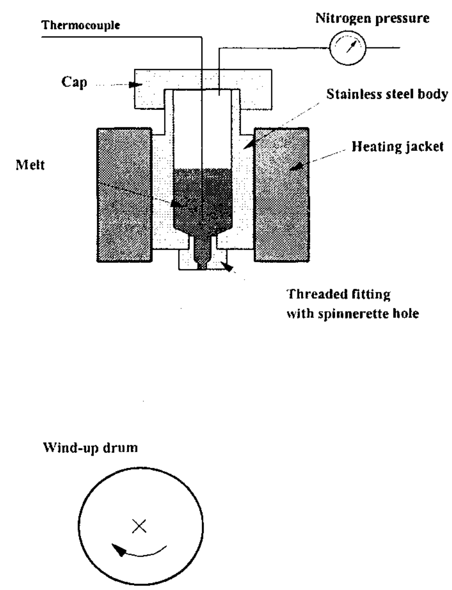

Fig. 1: A single hole spinnerette

The pitch/nanotube composites were then oxidatively stabilized by heating at a rate of between 0.1 and 1°C/min to 310°C under 1 L/min air flow. The stabilized products were carbonized under 2 L/min nitrogen flow in a tubular reactor at 20°C/min to 1100°C.

For the carbonized composite fibers, samples were mounted onto fiberboard testing plaques using colloidal graphite (Ted Pella, Inc.) as the adhesive. Tensile strength and modulus of elasticity were measured (ASTM D638M modified for fiber geometry) on a QT/1L tester (MTS Inc.) with a 150g full-scale load cell. The slope of the stress-strain curve and the value of stress at which the fiber breaks determine, respectively, the modulus of elasticity ($E_{composite}$) and the tensile strength ($\sigma_{composite}$). In Fig 2, the tensile strength is plotted against the elastic modulus for 0, 1 and 5 wt % SWNT additions (filled symbols) along with the corresponding resistivity data (open symbols). Uniform fibers could not be spun from the nanotube-pitch composites containing higher concentrations of SWNT apparently due to the formation of bridges across the spinnerette orifice, as well as significant changes in the rheology of the melt, making it difficult to draw fibers at these high loadings [4]. The solid and dotted traces in the figures serve respectively, as a "guide to the eye" for the behavior for the tensile strength and electrical resistivity as a function of the elastic modulus. These results show that pitch composite fibers prepared with a few wt % loading of purified SWNTs yield novel materials with enhanced tensile strength, modulus and electrical conductivity compared to the original pitch fibers. In particular, we find that the tensile strength, modulus and conductivity of a pitch composite fiber with 5 wt % loading of purified SWNTs are enhanced by 90%, 150%, and ~340% respectively, as compared to the corresponding values in unmodified isotropic pitch fiber.

In continuing work, we will investigate the effects of the carbon nanotube addition to the physical properties of the sintered discs. Given the apparent difficulty in spinning fibers with high nanotube concentrations, the direct production of nanotube containing artifacts may prove to be an even more productive field of investigation.

Acknowledgements
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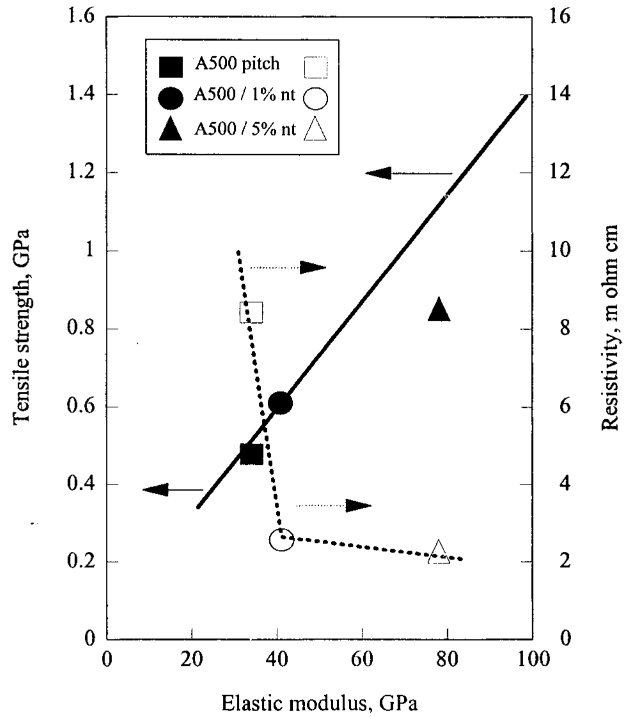

Fig. 2 Experimental values of properties of nanotube-pitch composite carbon fibers.