

**CONTROLLED CARBON NANOTUBE SYNTHESIS
FOR QUANTIFICATION OF POLYMER-NANOTUBE
COMPOSITE MICROMECHANICS**

By

Justin Bernard Bult

An Abstract of a Thesis Submitted to the Graduate

Faculty of Rensselaer Polytechnic Institute

in Partial Fulfillment of the

Requirements for the Degree of

DOCTOR OF PHILOSOPHY

Major Subject: Materials Science and Engineering

The original of the complete thesis is on file
in the Rensselaer Polytechnic Institute Library

Examining Committee:

Linda Schadler, Thesis Adviser

Pulickel Ajayan, Thesis Adviser

Rahmi Ozisik, Member

Richard Siegel, Member

Brian Benicewicz, Member

Rensselaer Polytechnic Institute
Troy, New York

November 2007
(For Graduation December 2007)

Abstract

Conventional experimental approaches to the understanding of nanotube-polymer micro-mechanics have struggled to produce reproducible data due to the inherent difficulty in physically manipulating the nanotube in-situ. To avoid the problems scale represents in nanotube-polymer composites a novel approach of using Polarized Raman spectroscopy was developed. The Raman spectroscopic technique has the advantage of using non-invasive analysis to compute the composite micro mechanical properties of interfacial shear stress and critical length.

Composites with nanotubes of defined length were needed in order to use the Raman technique. To satisfy this requirement a new thermal Chemical Vapor Deposition (CVD) tool capable of reproducibly growing aligned length uniformity with large mass yield was designed and built. The course of developing these furnace capabilities led to the investigation of nanotube growth mechanics. It is shown herein that a stable passivation barrier is required for nanotube growth. Using X-Ray Photoelectron Spectroscopy (XPS) depth profiling of metal substrate growth conclusively shows the presence of a stable catalyst layer on the outer surface of stable oxides of greater than 100 nm. By analyzing the diffusion profile represented in the XPS data it is shown that a critical thickness for the passivation oxide can be calculated as a function of time and temperature. For the growth parameters used in this study the critical thickness was found to be between 10 nm and 30 nm depending on the diffusivity value used for iron in chromia. This value agrees well with experimental observation.

Uniformly grown carbon nanotubes with lengths of 4, 14, 17, 22, 43, 74, and 116 μm were incorporated into a polycarbonate matrix polymer via solvent-antisolvent processing. The nanotube composites of varied length were tested in tensile strain while Raman spectra were taken concurrently to deduce the load transfer to the nanotube due to composite strain. It is found that the stress slope of the nanotube varies with length and gives conclusive evidence for an interfacial shear stress of 10.5MPa in a polycarbonate matrix and a critical nanotube length of 147

μm (aspect ratio of 4500).

With the development of new chemical vapor deposition capabilities there are opportunities for technical applications. The chemical vapor deposition system is demonstrated to have the ability to selectively control growth to lengths of over 5 mm and produce such samples across eight 100 mm silicon wafers per growth run. Extending the understanding of the growth mechanics allows for switch contact electrodes to be fabricated using noble metal infiltrated aligned nanotube arrays grown on metal substrates. The switch performance is found to be superior to conventional low-force switches with the array resistance maintained at 0.5Ω over the entire 100,000 cycle test at loads less than $500 \mu\text{N}$.