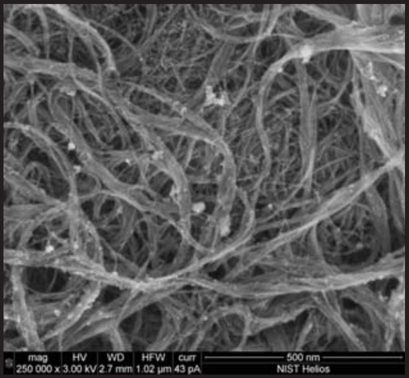
Nanotube Quality Control

NANOMATERIALS

Objective

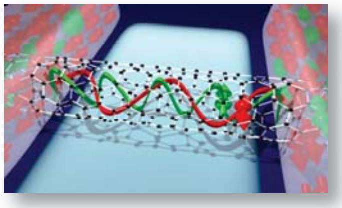
Our goal is to develop new methods to rapidly screen bulk carbon nanotubes for chemical purity and homogeneity. The different synthesis routes used to produce these materials generate different mixtures of tube geometries, along with varying amounts of carbonaceous and metallic impurities. New inspection techniques are needed to enable material benchmarking, process optimization, and quality control. Ensuring material quality is the first step toward widespread commercialization of these materials.

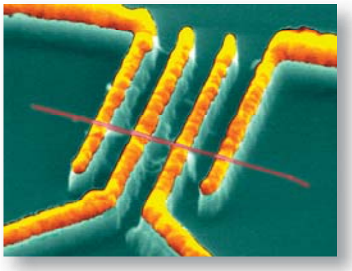


Impact and Customers

- Sales of carbon nanotubes are projected to exceed $1.9 billion by 2010, with the materials finding applications in lightweight composites, microelectronics, and biomedical products. The highest growth is projected for single-walled carbon nanotubes, where performance is directly related to chemical purity.

- Presently, characterization of nanotube purity requires multiple measurements using different analytical and optical techniques. As production volumes increase, screening tools will become more important for quickly identifying batch-to-batch inconsistencies, enabling quality assurance.

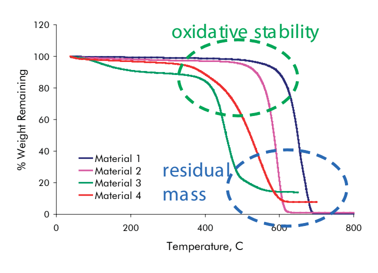




- Subtle changes in nanotube chemistry can have a dramatic impact on device performance and reliability. For example, the presence of residual catalyst particles can alter the current-carrying capacity of a single isolated nanotube. Understanding raw material quality is the first step to predicting product reliability.

Approach

Thermogravimetric analysis (TGA) is widely used to gather data on nanotube chemistry. By monitoring weight loss as a function of temperature, one can determine decomposition kinetics and use this data to closely approximate the distribution of impurities present in a few milligrams of material. Oxidative stability provides an indirect measure of the types of carbons present. The residual mass provides an estimate of the metal fraction, which primarily consists of the catalyst material.

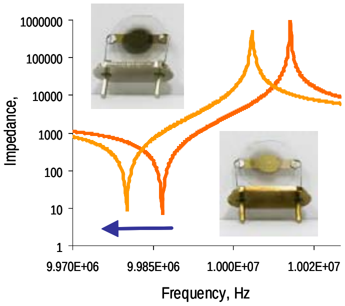


One disadvantage of TGA, however, is the need for relatively large specimen sizes, which is particularly problematic for highly purified materials (where process yields are low). As an alternative to TGA, we developed an elevated temperature quartz crystal microbalance (QCM) technique that interrogates samples on the order of 1 microgram or less. A variety of coating techniques can be used to deposit the nanotube material, including drop casting, spin coating, and spray deposition.

Accomplishments

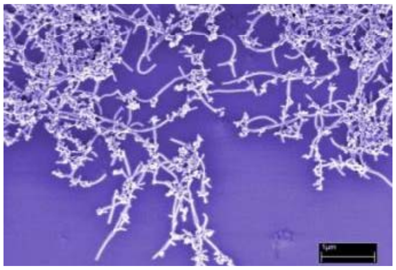
We validated our elevated temperature QCM method using several commercial-grade carbon nanotube materials with different degrees of chemical purity. We first characterized the bulk materials by TGA, identifying their oxidation temperature. Using this temperature as a guide, we then treated multiple crystals over a 75 °C temperature range and compared mass loss and homogeneity data with the results from TGA.

To determine mass changes in the nanotube coating, we monitored the resonance frequency of the quartz crystal using an impedance analyzer. During coating deposition, the resonance frequency decreases, with the shift in frequency directly proportional to the change in mass. On heating, the coating mass decreases due to oxidation of the carbon material, resulting in an increase in resonance frequency.



Shift in resonance frequency with mass

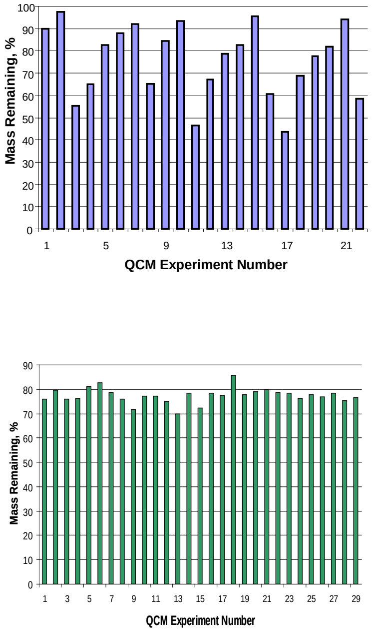
For one nanotube material, we found a high metal content, exceeding 40 %. Amorphous carbon content was also high, resulting in a relatively low percentage of carbon nanotubes. However, the homogeneity of the material appeared quite good by TGA, with 25 successive measurements producing a variability in oxidation temperature of less than 2 %.



SEM micrograph of the nanotube material

QCM experiments were then performed using this same material, which was dispersed in chloroform and spray deposited to form an ultra-thin coating. The average mass change at the oxidation temperature (~400 °C) determined by QCM was consistent with TGA results (n=25 crystals). However, the individual data proved inconsistent, with variability exceeding 20 %. Because of the high metal content, we postulate that each sample deposited from the ultra-fine spray nozzle contained a different amount of metal particles. Taken together, the 25 coatings represented only a small fraction of the TGA sample, illustrating that the homogeneity can vary with sample size.

Characterization of a material with far less metal content confirmed our hypothesis. For a relatively pure nanotube specimen, we were able to closely approximate the TGA data, both in terms of mass loss at temperature and homogeneity. The figures below illustrate the degree of inconsistency from coating to coating, depending on the chemistry of the nanotube specimen. Because the presence of impurities can influence processing (e.g., ease of dispersion, nozzle clogging), it is imperative that sample preparation be standardized to avoid further variations.



Coating variability determined by QCM

Learn More

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Publications

Hooker SA, Geiss R, Kar A, Schilt, R, *Rapid Inspection of Carbon Nanotube Quality*, ACerS IntlConfAdvCeram&Comp, Daytona Beach, FL (2007)

Standard Test Methods for Measurement of Electrical Properties of Carbon Nanotubes, IEEE Standard 1650 (2005)

Freiman S, Migler K, and Hooker S, *NIST Recommended Practice Guide on Carbon Nanotube Characterization*, in press – draft document at: http://www.msel.nist.gov/Nanotube2/Carbon_Nanotubes_Guide.htm