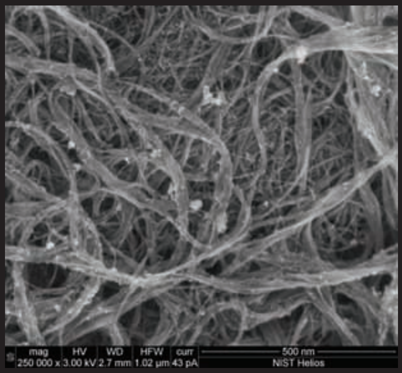
Nanotube Quality Control

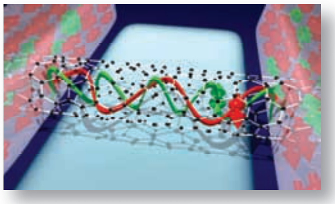
Objective

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.

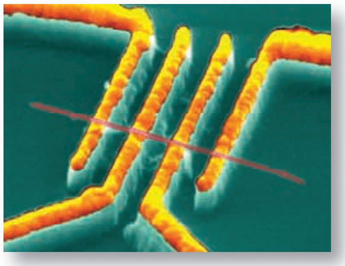


Impact and Customers

- Sales of carbon nanotubes are projected to exceed $4.6 billion by 2015, 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.



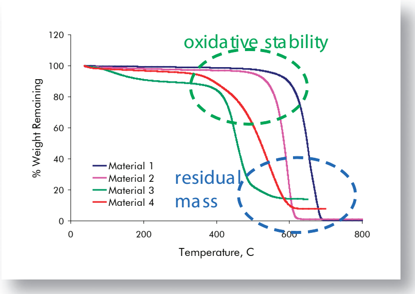
- Presently, characterization of nanotube chemistry requires multiple measurements using different analytical and optical techniques. As production volumes increase, screening tools will become more important for quickly identifying batch-to-batch inconsistencies, enabling quality assurance.



- 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 critical for 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 carbonaceous materials present. The residual mass provides an estimate of the metal fraction, which primarily consists of the catalyst material.

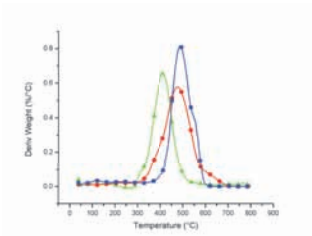


One disadvantage of TGA, however, is the need for relatively large specimen sizes, which is particularly problematic for highly purified materials (where process yields are low). As an alternative to TGA, we developed an elevated temperature quartz crystal microbalance (QCM) technique that interrogates 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.

NANOMATERIALS

Accomplishments

TGA has been used evaluate the compositional differences in different carbon nanotube materials, which arise from the different manufacturing and post-production purification processes. The residual mass and the oxidation temperature data provide information on the material's decomposition.

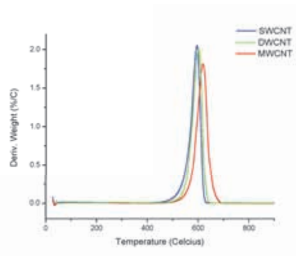


TGA for three manufactured materials

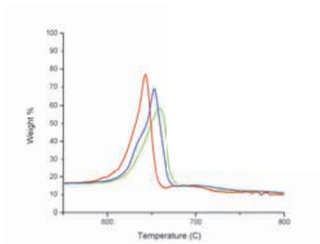
The derivative curve is used to determine the oxidation temperature of the material, as well as defining the mass loss of a single decomposing species (e.g., single peak) or of multiple decomposition events (e.g., double peaks, shouldered peaks). The oxidation temperature represents the thermal stability of the material. A lower oxidation temperature may indicate the presence of amorphous carbons. Higher temperatures can indicate multi-wall carbon nanotubes or other structured carbons may also be present.

TGA is not only a bulk analysis method but can also be used to characterize micro-scale differences within a CNT sample. Differences in CNT chemistry, surface functionality, and purification have been observed with TGA. Increased thermal stability of a CNT sample has also been observed as more layers were added to the nanotube structure (single, double, multi-wall carbon nanotubes).

Length dependency was also measured with the TGA. Single-walled carbon nanotube samples sorted as short (~ 80 nm), medium (~320 nm) and long (~760 nm) were observed to have different oxidation temperatures.



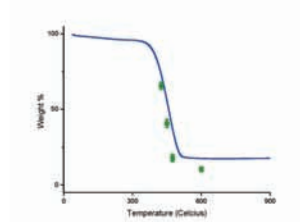
CNTs with different diameters. Single (SWCNT), double (DWCNT) and multi-wall carbon nanotubes (MWCNT) are shown.



TGA of short (green), medium (blue), and long (red) single-wall carbon nanotubes.

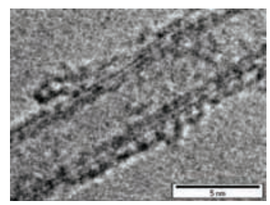
One major disadvantage of TGA analysis for CNTs is the relatively large sample size required, which is especially problematic in the case of costly high-purity samples. In addition to our work with conventional TGA, we have developed an elevated temperature technique based on quartz crystal microbalances which provides TGA-like results for a microgram scale sample or thin film. We validated our elevated temperature QCM method using materials with simple thermal profiles. Once the QCM measurements were shown to closely follow the TGA curve for materials with simple thermal decompositions, we extended the technique to include mixtures of these materials to demonstrate the capabilities of monitoring more complex heterogeneous samples.

For a relatively pure nanotube sample, we were able to closely approximate the TGA data, both in terms of mass loss at temperature and homogeneity. These results confirm that

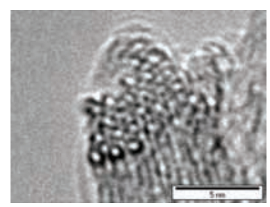


TGA (blue) and QCM (green) measurements of a representative carbon nanotube sample.

the elevated temperature QCM technique can be used to evaluate carbon nanotube quality by manufacturers to test current batch-to-batch production differences.



TEM of a double-wall carbon nanotube



End-on view of a bundle of SWCNTs

TGA results were supported by extensive transmission electron microscopy (TEM) of the carbon nanotube materials.

Learn More

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Publications

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