Carbon Nanotube Material Quality Assessment

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Abstract

The nanomaterial activities at NASA-Johnson Space Center focus on carbon nanotube production, characterization and their applications for aerospace systems. Single wall carbon nanotubes are produced by arc and laser methods. Characterization of the nanotube material is performed using the NASA-JSC protocol developed by combining analytical techniques of SEM, TEM, UV-VIS-NIR absorption, Raman, and TGA [1]. A possible addition of other techniques such as XPS, and ICP to the existing protocol will be discussed. Changes in the quality of the material collected in different regions of the arc and laser production chambers is assessed using the original JSC protocol. The observed variations indicate different growth conditions in different regions of the production chambers.

Abstract
The nanomaterial activities at NASA-Johnson Space Center focus on carbon nanotube production, characterization and their applications for aerospace systems. Characterization of the nanotube material is performed using the NASA-JSC protocol developed by combining analytical techniques of SEM, TEM, UV-Vis-NIR absorption, Raman, and TGA. A possible addition of other techniques such as XPS, and ICP to the existing protocol will be discussed.

NASA-JSC Protocol for SWCNT Characterization
- Thermogravimetric Analysis (TGA), (TA SDT 2960)
  3 runs using 3-4 mg of material in 100 sccm air at a 5 °C/min heating rate from room temperature to 1000 °C.
- Transmission Electron Microscopy (TEM) & Energy Dispersive X-ray Spectroscopy (EDS), (JEOL 2010 FA)
  Small quantity dispersed in methanol, sonicated then placed on TEM grid.
- Scanning Electron Microscopy (SEM) & EDS (Phillips XL40 FEI)
  Image 3 locations at 20 and 50kX with corresponding EDS spectra at same locations.
- Raman Spectroscopy (Renishaw RM 1000, Horiba Yvon Jobin)
  Spectra collected in three different locations with 780, 633 and 514 nm excitation sources.
- UV-Visible-NIR spectrometry (Perkin-Elmer Lambda 900)
  0.1 mg sample placed in 10 mL Dimethyl Formamide (DMF) and sonicated until well dispersed.

Production and Collection
Arc Discharge Method
Pulsed Laser Vaporization

Wall Deposit
Collarette
Cathode

Arc Production Conditions:

Laser Production Conditions:
- 3/4" diameter target, 15°C ±1%N, Pressure: 500T, Air Flow Rate: 100 acbm, Pulse Separation: 50 ns, Power Density: 1.6x10^4, Oven Temperature: 1200 °C, Laser Sequence: Green-IR.

JSC Characterization Protocol for TGA
Information extracted from TGA data:
1. Average residual mass M_r (in %): Shows fraction of residual metals in the specimen.
2. Temperature T_m of the maximum in the burning rate dM/dT: Shows thermal stability of the specimen.
3. Standard deviation of M_r and T_m: Shows homogeneity of the specimen.

JSC Characterization Protocol for TEM
TEM Analysis:
1. Images: Qualitative information about non-nanotube carbon impurities ("schems") and graphitic particles and their distribution within a sample.
2. Images: Qualitative information about metal content.

JSC Characterization Protocol for SEM
SEM Analysis:
1. Images: Qualitative information about impurities, general morphology of the sample and its homogeneity.
2. EDS: Qualitative information about metals, silicon and chlorine impurities.

JSC Characterization Protocol for Raman Spectroscopy
Analysis of Raman Spectra:
1. Nanotube protonation state from the C-C stretch mode shift.
2. Possible information about impurities and disorder in the sample from the 1340 cm⁻¹ disorder peak position and width.
3. Qualitative information about sample homogeneity from the variability in the spectra.

Additional Techniques:
- ICP can provide a better quantitative measure of metal impurities with as produced and purified materials (digestion of metals major issue).
- XPS can provide information on the chemical states of non-carbon impurities to assist with TGA and ICP analysis.