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.

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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. Characterization of the nanomaterial 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. Material in the production chambers is assessed using the original JSC protocol. The observed variations indicate different growth conditions in different regions of the production chambers.

NASA-JSC Protocol for SWCNT Characterization

- Thermogravimetric Analysis (TGA), (TA SDT 2960): 3 runs using 3-4 mg of material in100 cc min 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)
- Scanning Electron Microscopy (SEM) & EDS (Phillips XL40 FEQ)
- Raman Spectroscopy (Rashinshaw RM 1000, Horiba Yvon Jobin)
- UV-Vis-NIR spectrometry (Perkin-Elmer Lambda 900)

JSC Characterization Protocol for SEM

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 TEM

1. Images: Qualitative information about non-nanotube carbon impurities ("schmutz" and graphitic particles) and their distribution within a sample
2. Images: Qualitative information about metal content

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

Production and Collection

Arc Discharge Method
Pulsed Laser Vaporization

JSC Characterization Protocol for Laser Material

- Residual Mass: 0.45% - 0.85% - 0.63% - 0.25% - 0.50%
- Thermal Stability:
  - Inner: 492°C
  - Wall: 475°C
  - Tube: 465°C
  - Main: 467°C
  - Sleeve: 467°C
  - Filter: 462°C
- D/G Ratios
  - 0.268 0.986 0.867 0.986 0.750
- Dispersions:
  - Zone 1: 1300 cm⁻¹
  - Zone 2: 1300 cm⁻¹
  - Zone 3: 1300 cm⁻¹
  - Zone 4: 1300 cm⁻¹
  - Zone 5: 1300 cm⁻¹

Additional Techniques:
- ICP can provide a better quantitative measure of of the metal impurity levels 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.