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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Production and Collection

Arc Discharge Method

Pulsed Laser Vaporization

Wall Deposit

Webs

Collarette

Cathode

Arc Production Conditions:

3.92 kV/18.1T. Pressure: 50 Torr. Voltage: 38.2 V. Current: 101.5 A. Electrode Distance: 3 mm. Automated

Laser Production Conditions:


JSC Characterization Protocol for TEM

TEM Analysis

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 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.

JSC Characterization Protocol for TGA

Information extracted from TGA data:

1. Average residual mass Mᵣ (in %): Shows fraction of residual metals in the specimen.
2. Temperature Tₚ of the maximum in the burning rate dM/dT: Shows thermal stability of the specimen.

Analysis Results Summary Table for Laser Material

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>Inner</th>
<th>Cottar</th>
<th>Sleeve</th>
<th>Main</th>
<th>Filter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Residual Mass</td>
<td>0.4%</td>
<td>1.3%</td>
<td>12.2%</td>
<td>14.3%</td>
<td>10.4%</td>
</tr>
<tr>
<td>Thermal Stability</td>
<td>430°C</td>
<td>485°C</td>
<td>650°C</td>
<td>650°C</td>
<td>650°C</td>
</tr>
<tr>
<td>D/Band Position</td>
<td>1380 cm⁻¹</td>
<td>1280 cm⁻¹</td>
<td>1280 cm⁻¹</td>
<td>1280 cm⁻¹</td>
<td>1280 cm⁻¹</td>
</tr>
<tr>
<td>Small Diameter %</td>
<td>8.5%</td>
<td>22.6%</td>
<td>8.1%</td>
<td>9.1%</td>
<td>27.5%</td>
</tr>
</tbody>
</table>

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.