Metrology for Nanomanufacturing

Key Elements for the Future of Nanomanufacturing: Instrumentation, Metrology, and Standards

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Nanotechnology and Nanomanufacturing

• Nanotechnology is the frontier of innovation and is one of the most dynamic growth areas in the U. S.
  – Predicted by NSF to become a trillion dollar business
  – Large U. S. Federal government investment

• Nanometrology is needed by nanotechnology and nanomanufacturing more than any other prior technology.
  – NIST has been tasked by the NNI as the lead agency for Instrumentation, Metrology and Standards
  – NIST is co-lead with NSF on Nanomanufacturing.
Approximate 2005 Nanotechnology Spending

**World-wide government spending has been (~$4.6B):**
- $1.7 billion (36%) in North America, almost entirely accounted for by the U.S.
- $1.7 billion (36%) in Asia, dominated by Japan
- $1.1 billion (26%) in Western Europe, led by Germany
- $100 million in the rest of the world

**Established corporations spent ($4.52B):**
- $1.9 billion (42%) was in North America
- $1.7 billion (38%) was in Asia
- $850 million (19%) was in Europe
- $70 million (2%) was in the rest of the world

To date the spending is likely doubled
Nanotechnology and Nanomanufacturing

- Nanomanufacturing is the primary bridge over which these substantial investments can be recovered.
- That bridge is just being put on the drawing board
- ...and you are the architects
Nanotechnology and Nanomanufacturing

- Measurement science (metrology) is critical for successful Nanomanufacturing.
- Metrology has proven that it is value added.
- Semiconductor industry is the prime example because it is so well documented.
RTI International report:
Economic Impact of Measurement in the Semiconductor Industry

• NIST plays a leading role in developing SRMs, and most SRMs are either sold directly by NIST or are traceable to NIST standards.

• Many instrument and tool providers develop their own in-house standards to calibrate their equipment.

• These vendor-supplied standards are also usually NIST traceable.

• SRMs are used by most of the semiconductor supply chain and include the following:
  – Front-end processing
    • – thin film for transmission electron microscopy, or TEM (NIST SRM 2063a)
    • – scanning electronic microscopy, or SEM, performance (NIST SRM 2069b, 8091, and 2800)
    • – optical microscope linewidths (NIST SRM 475 and 476)
    • – implantation standards (NIST SRM 2133–2137)
    • – ellipsometry (NIST SRM 2531 and 2534)
    • – microscale dimensional measurement (NIST SRM 5001)
RTI International report: Conclusion: Measurement innovations add up to big savings for semiconductors.

RTI estimates that for every $1 spent on measurement, the industry as a whole saw a $3.30 return.

On average of 14 similar studies the ratio is $1:$44.
Metrology for Nanomanufacturing

• Critical to the realization of robust nanomanufacturing is the development of the necessary instrumentation, metrology, and standards.
• Integration of the instruments, their interoperability, and appropriate information management are also critical elements that must be considered for viable nanomanufacturing.
Metrology for Nanomanufacturing

• Advanced instrumentation, metrology and standards allow the physical dimensions, properties, functionality, and purity of the materials, to be measured and characterized.
• Enables production to be scaleable, controllable, predictable, and repeatable to meet market needs.
• Metrology need must be close to the critical processes
  – on-line vs. off-line
• Highly precise processes taking advantage of the economy of scale is a must.
“Flat-world” Nanotech Activities

- Fundamental knowledge gaps emerging
  - Materials
  - Measurements
  - Instrumentation
  - Standards
  - Modeling
    - Insufficient data
    - Inaccurate data
  - Environmental Health and Safety (EHS)

- Primary needs are: instrumentation, metrology and standards
  - Data, Data, Data
Instrumentation - Problem

- Much of the current measurement infrastructure currently used by nanotechnology/industry is only evolutionary.
- New potentially revolutionary metrology is needed for many applications.
- Automated, operator independent instrumentation adapted to nanomanufacturing.

- NNI Grand Challenge Workshop on Instrumentation and Metrology.
- IWG Instrumentation Metrology and Standards for Nanomanufacturing Workshop.
Why is this a Hard Problem?

- Much of the measurement infrastructure currently available for nanotechnology/nanomanufacturing is only evolutionary:
  - Optics
  - Transmission Electron Microscope
  - Scanning Electron Microscope
  - Force Microscopy, etc.

- New potentially revolutionary metrology is needed for many applications:
  - Helium Ion Microscope

- Automated, operator independent instrumentation adapted to nanomanufacturing

- Much of this was underscored in the NNI Grand Challenge Workshop on Instrumentation and Metrology and the recent NIST Nanomanufacturing Workshop.
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Research into Basic Contrast Mechanisms Involved with Image Formation Needed

Once that is resolved, the same issues limiting the SEM accuracy need to be tackled.

Gold Nanoparticle Reference Material

<table>
<thead>
<tr>
<th>Field Of View</th>
<th>Detector</th>
<th>Dwell Time</th>
<th>Date</th>
<th>Time</th>
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<td>2:31 PM</td>
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<td>Acceleration V</td>
<td>Image Size</td>
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</tbody>
</table>
Standards:

- Google Search: Standards+nanotechnology+needed
- Returned hundreds of references:
  - PISCATAWAY, N.J., USA, 22 Dec. 2003 The rapid pace of nanotechnology research and the promise of the many applications emerging from it have prompted a broad initiative at the IEEE to create the standards needed to foster this field.
  - The American National Standards Institute's Nanotechnology Standards Panel (ANSI-NSP) serves as the cross-sector coordinating body
- Numerous standards bodies and government laboratories working on this issue
- Numerous NNI workshops discussing standards for Nanotechnology especially for Environment Health and Safety
Standards:
Real World Situation

- Carbon nanotube – two words for many different possible materials
- At least 50 different CNT species have been identified
- Only half of these species are semiconducting
- Current manufacturing processes do not simply make one type of CNT
  - Inherently produce a mixture of CNT species along with 3-60%+ unwanted chemical impurities

MWCNTs 270 tons/yr (245 000 kg/year)
SWCNTs 7 tons/yr (6 350 kg/year)
8 000 US$/kg to 500 000 US$/kg

## Matrix: Purity & Structural Properties, SWCNTs

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Category</strong></td>
<td><strong>Method</strong></td>
</tr>
<tr>
<td>Morphology</td>
<td>SEM/EDX (Lead: USA)</td>
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<tr>
<td></td>
<td>TEM (Lead: USA, Co-lead: Japan)</td>
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<tr>
<td></td>
<td>Raman Spectroscopy (Lead: USA)</td>
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<td>UV-Vis-NIR Absorption (Lead: Japan)</td>
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<td></td>
<td>NIR-PL/Fluorescence (Lead: Japan)</td>
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<tr>
<td></td>
<td>TGA (Lead: USA, Co-lead: Korea)</td>
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<tr>
<td></td>
<td>TG-MS (Lead: Japan)</td>
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<tr>
<td><strong>Purity</strong></td>
<td>Non-carbon impurities</td>
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<tr>
<td></td>
<td>Tube surface cleanliness</td>
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<tr>
<td></td>
<td>Nanotube and non-nanotube carbon</td>
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<tr>
<td></td>
<td>Carbonaceous content (Quantitative)</td>
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<tr>
<td></td>
<td>Non-carbon content (Quantitative)</td>
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<td><strong>Length and Diameter</strong></td>
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<td></td>
<td>Tube diameter, metal cluster size</td>
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<td>Diameter (Lead: Japan)</td>
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<td>Diameter (Lead: Japan)</td>
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<tr>
<td><strong>Tube Type</strong></td>
<td>Metallic/ Semiconducting</td>
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<tr>
<td></td>
<td>Metallic/ Semiconducting (Lead: USA, Co-lead: Korea)</td>
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<tr>
<td></td>
<td>Chirality (Semi conducting tubes)</td>
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<tr>
<td><strong>Dispersability/Solubility</strong></td>
<td>Tube bundling</td>
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<tr>
<td></td>
<td>Tub bundling or separation (solution)</td>
</tr>
<tr>
<td></td>
<td>Tube bundling</td>
</tr>
<tr>
<td><strong>Additional</strong></td>
<td>Oxidation/transition temperatures</td>
</tr>
<tr>
<td></td>
<td>Oxidation/transition temperatures</td>
</tr>
</tbody>
</table>

**Other Participants**
- China
- China, Korea
- Korea
- Korea
- USA
- TBD: Canada and Germany
- China
Residual Catalyst Content in Carbon Nanotube (CNT) Bearing Material

- Cross NIST Laboratory effort
- Collaboration between NIST, NASA, NIOSH, NRC, Univ. of Maryland and Lehigh University
- Product of commerce - as received sample from the manufacturer.
  - Main requirement was minimum of 200 grams of material made in one single process run.
  - Single-walled, closed-ended, carbon nanotubes, made by an evaporative/arc method
  - Packaged in 0.35-0.5 gm
Residual Catalyst Content in Carbon Nanotube (CNT) bearing Material

- Residual catalyst concentrations measured by instrumental neutron activation analysis (INAA) and cold neutron prompt gamma-ray activation analysis (CNPGAA)

Material proved to be too inhomogeneous to be useful as an RM
A new material has been identified and is being pursued

A great many lessons were learned by this work
The data obtained will be published in the near future

- UV, Vis and NIR
- AFM
- etc....
Collaboration between the NCI, FDA, and NIST
- Perform pre-clinical characterization of nanomaterials as drug delivery systems
- Accelerate the use of nanoparticles for drug delivery, as image contrast agents and for diagnosis.

NIST is working to develop quantitative, reproducible measurement methods and protocols for nanotechnology/nanoparticles measurements.
Gold Nanoparticle Size Standard

Reference Material has been released
Gold Nanoparticle Size

Standard RM

RM 8011 - 10 nm
RM 8012 - 30 nm
RM 8013 - 60 nm

Report of Investigation available at:

http://ts.nist.gov/measurementservices/referencematerials/index.cfm
Strategic Alliances Imperative

- Developing effective instrumentation, nanometrology, and nanostandards is not a simple problem to solve.
- Success requires strategic alliances between: Governments, University and Industry.
- Nanotech strong fit for strategic alliances:
  - Multi-disciplinary nature
  - Enabling technology
  - Broad industry implications
  - Federal funding opportunities
  - Commercialization challenges
- Cannot be done alone which leads to the multidimensional nature of nanotechnology…
- ..and the reason for this workshop!
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