Metrology for Sustainable Nanomaterials

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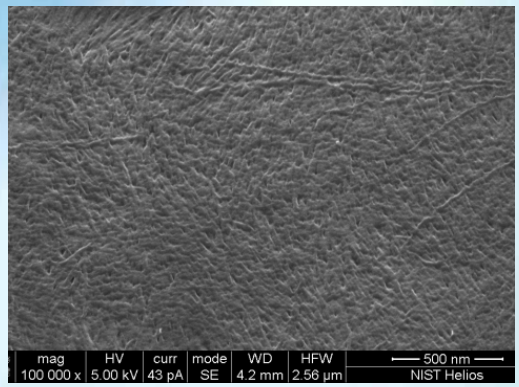
Metrology for Sustainable Nanomaterials

- **NIST is the nations measurement and standards laboratory**

  Research in nanotechnology and the needed nanometrology is found all across NIST

  Work on dimensional metrology of CNCs began in 2006-7 with an early paper and presentation in 2008 employing SEM, HIM and AFM



- **Leverages the knowledge gained from work on other nanomaterials**
- **Requirement for both basic (research and laboratory) and manufacturing (production) metrology.**

Metrology Needs for Sustainable Nanomaterials

- **Challenges in Characterizing Small Particles: Exploring Particles from Nano- to Microscale National Research Council.** National Research Council. 2012

- **Special "Green" Materials Forum** SPIE Instrumentation, Metrology, and Standards for Nanomanufacturing, Optics, and Semiconductors.2011

- **Cross-Industry Issues in Nanomanufacturing** NNI/NIST Sponsored Workshop - A NNI/NIST Sponsored Workshop May, 2008.

- **Interagency Working Group – Instrumentation Metrology and Standards for Nanomanufacturing Workshop,** Gaithersburg, MD (October 17-19, 2006)

- **NNI Interagency Grand Challenge Workshop on Instrumentation and Metrology.** January 27-29, 2004.

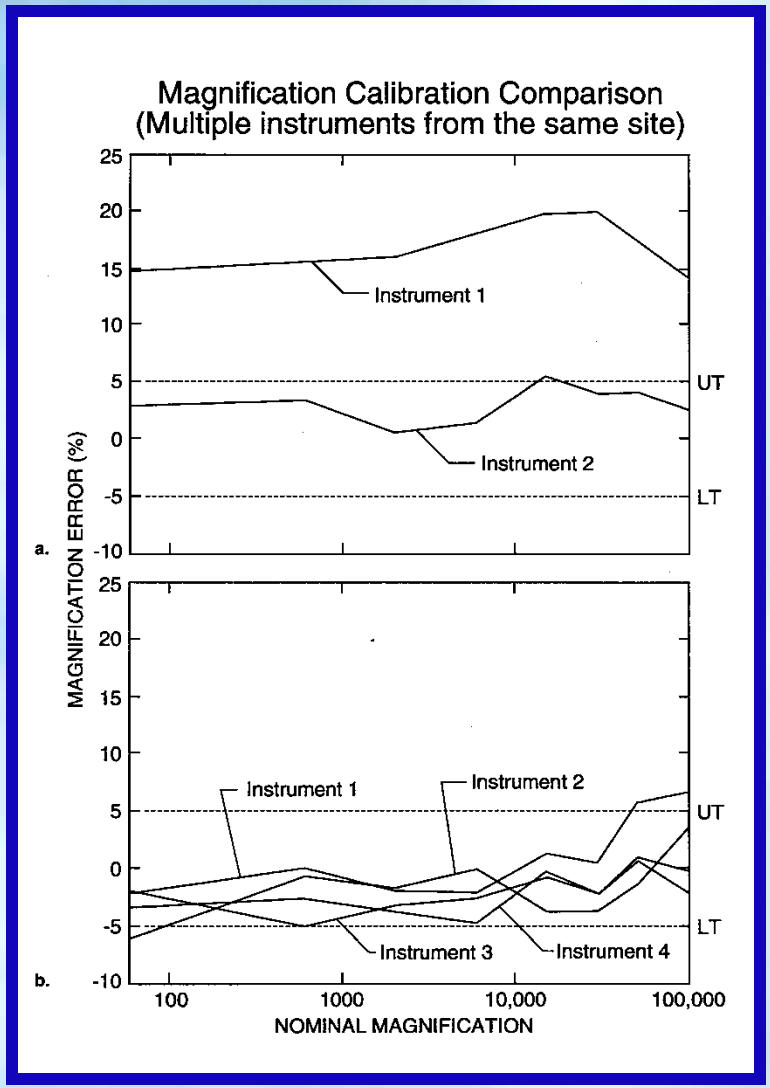
Metrology needs for Sustainable Nanomaterials

- **NEED: Measurement instrument validation standards and procedures**
  - Calibration
    - Chemical Composition
    - 3-D Physical Properties
      - Size/Length, Strength, etc.
  - Misuse of standards
    - Standards are often developed for a specific use
    - Use for other purposes can invalidate them
      - latex spheres
- **NEED: Standardized measurement procedures**
  - Do instruments of the same class provide the same answers to the question: "How XXX* is it"
    - Interlab Study data from the same lab

XXX*- fill in the blank: big, composition, etc.

Instrument Calibration:
Interlaboratory SEM Study

- **Over 60 instruments studied**
- **Calibration error varied between 10 % and 60 %.**
- **“X” scan to the “Y” scan was not 1:1**
- **Gross differences in data from instruments from the same laboratory**



Reference:
Postek, M. T. , Vladar, A. E. , Jones, S. and Keery, W. J. 1993. Inter-laboratory study on the lithographically produced scanning electron microscope magnification standard prototype. NIST J. Res. 98:447-467.

Metrology needs for Sustainable Nanomaterials

- **NEED: Fundamental, scientifically validated, material property measurement databases.**
  - **National labs have been a good source for this type of work**
    - **NIST, Sandia, Oak Ridge**
  - **Needed for input into a variety of predictive models**
  - **Accurate models can answer many questions economically:**
    - **EX: What is the proper loading of CNC to achieve a particular strength?**
    - **EX: What would happen if a different resin is used?**

Accurate models can result in tremendous savings to industry

Metrology needs for Sustainable Nanomaterials

- **NEED: Performance standards and procedures**
  - **Characterizing nanomaterials requires the instruments to function optimally at all times**
    - **Need to know how well an instrument should run and monitor that performance (or lack of it)**
      - **Does the instrument performance change with time?**
      - **When is maintenance needed**
- **NEED: New methods for imaging and characterization need to be developed**
  - **Helium ion microscope**

Metrology needs for Sustainable Nanomaterials

- **NEED: Fundamental understanding of methods divergence and 3-D hybrid metrology issues:**
  - **Use the right tool for the right purpose**
  - **Why different types of instruments provide different answers.**
    - **EX: Gold nanoparticle standard**

Table 1. Reference Value Mean Size and Expanded Uncertainty [(a)]
Average Particle Size (Diameter), in nm

Technique	Analyte Form	Particle Size (nm)
Atomic Force Microscopy	dry, deposited on substrate	8.5 ± 0.3
Scanning Electron Microscopy	dry, deposited on substrate	9.9 ± 0.1
Transmission Electron Microscopy	dry, deposited on substrate	8.9 ± 0.1
Differential Mobility Analysis	dry, aerosol	11.3 ± 0.1
Dynamic Light Scattering	liquid suspension	13.5 ± 0.1
Small-Angle X-ray Scattering	liquid suspension	9.1 ± 1.8

Metrology needs for Sustainable Nanomaterials

- **Why is methods divergence a big problem?**
  - **Multiple studies using different instruments reporting different data**
- **Reasons for methods divergence**
    - **Instrument issues:**
      - **SEM – electron beam interaction**
      - **Optics – diffraction**
      - **Scanned probe – tip size**
      - **Etc.**
    - **Sampling differences**
      - **Not all techniques measure the same measurand**
  - **Need to know what you are measuring, how it is being measured and why it is being measured.**
    - **What is the role of accuracy?**

Metrology needs for Sustainable Nanomaterials

- **NEED: Manufacturing Metrology**
  - **How do you characterize a boatload of nanomaterials with no or little human intervention with high throughput?**
    - **What automated equipment will be needed?**
  - **Instrument manufacturers need to be directed and put into the loop early**
    - **ITRS**
    - **Semiconductor industry a good example but it has taken a long time and much investments in R&D**
    - **Knowledge can be leveraged**

Conclusion

- **In the nanoworld, we have no first-hand experience so we must rely on “well calibrated” instruments to provide measurements for any conclusions.**
  - **Instruments are fallible**
- **Role of accurate measurements more important today than any other time because the error budget is so small and we cannot rely only on past data since properties might change**
- **Metrology is often the last thing considered, but often turns out to be a limiting factor.**

If you can’t measure it, you can’t manufacture it

Thank you