Self-assembly and Nanotechnology 10.524

Lecture 6. Nanoscale Characterization and Measurements

Instructor: Prof. Zhiyong Gu (Chemical Engineering
& CHN/NCOE Nanomanufacturing Center)

February 27, 2013

Lecture 6: Nanoscale Characterization and Measurements

Table of Contents

- Useful instruments/tools
- Examples
- Nanometrologies

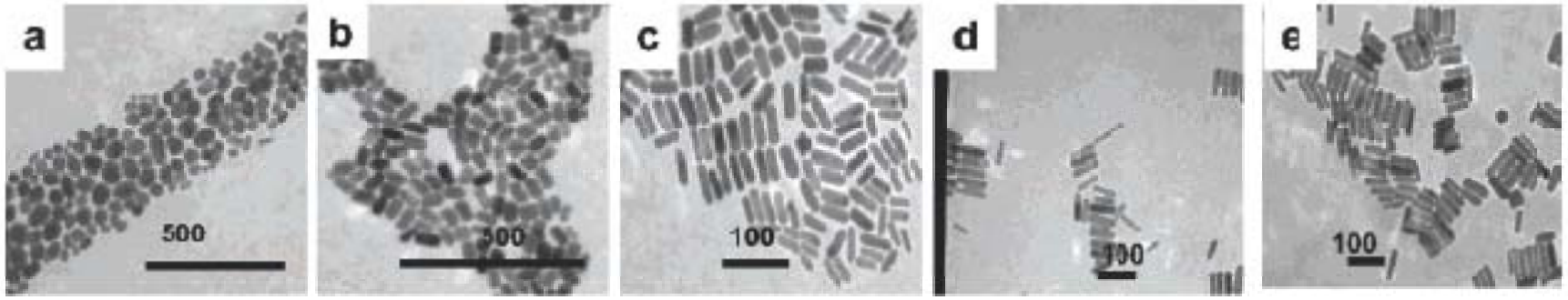
Useful Instruments and Tools

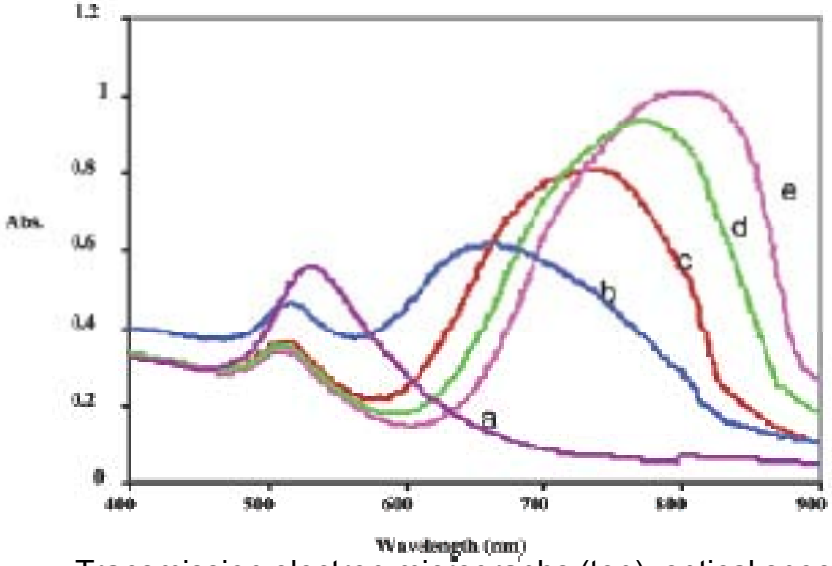
- Spectroscopy
- Electron microscopies
- Atomic force microscopes
- Optical microscopes
- Electrical measurements
- Mechanical measurements
- Thermal measurements, etc.

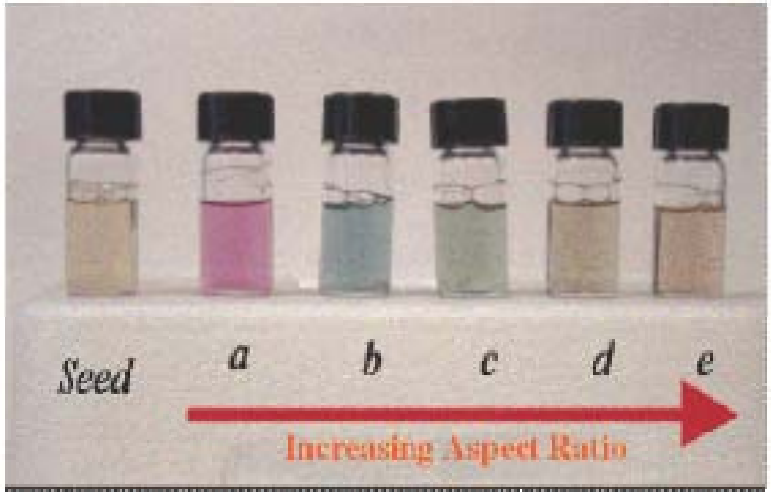
Spectroscopic Techniques

- UV-Vis spectrophotometer
- Fluorescent spectrophotometer
- Mass spectrometer
- ●●●

Example: Gold Nanorods





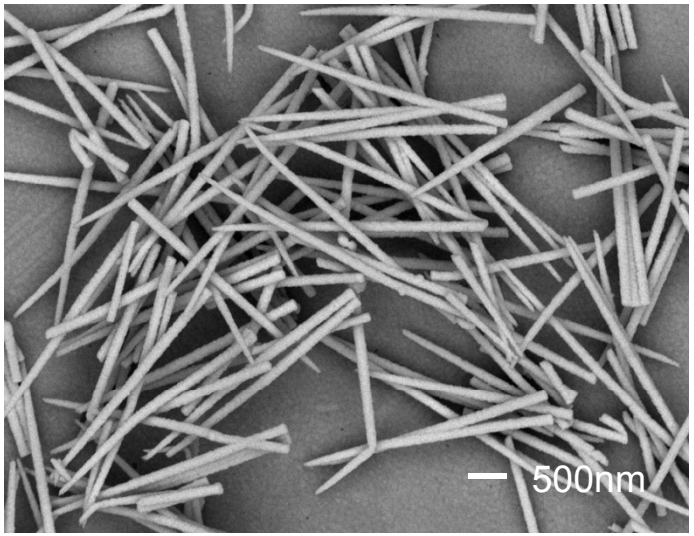


Transmission electron micrographs (top), optical spectra (left), and photographs of (right) aqueous solutions of gold nanorods of various aspect ratios. Seed sample: aspect ratio 1; sample a, aspect ratio 1.35 (0.32; sample b, aspect ratio 1.95 (0.34; sample c, aspect ratio 3.06 (0.28; sample d, aspect ratio 3.50 (0.29; sample e, aspect ratio 4.42 (0.23. Scale bars: 500 nm for a and b, 100 nm for c, d, e.

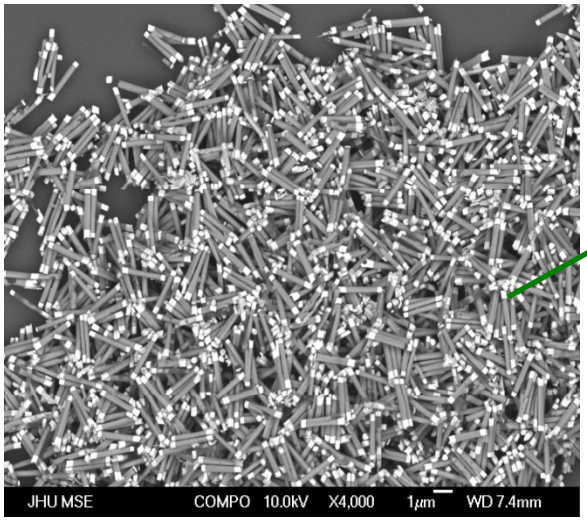
J. Phys. Chem. B **2005,** *109,* 13857-13870

Electron Microscopies: SEM

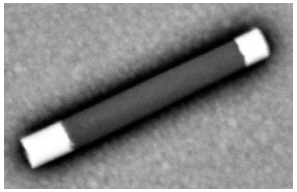
Example: SEM images of multisegment nanowires



One-component nanowires
(gold nanowires, d=50nm)



Multi-component nanowires

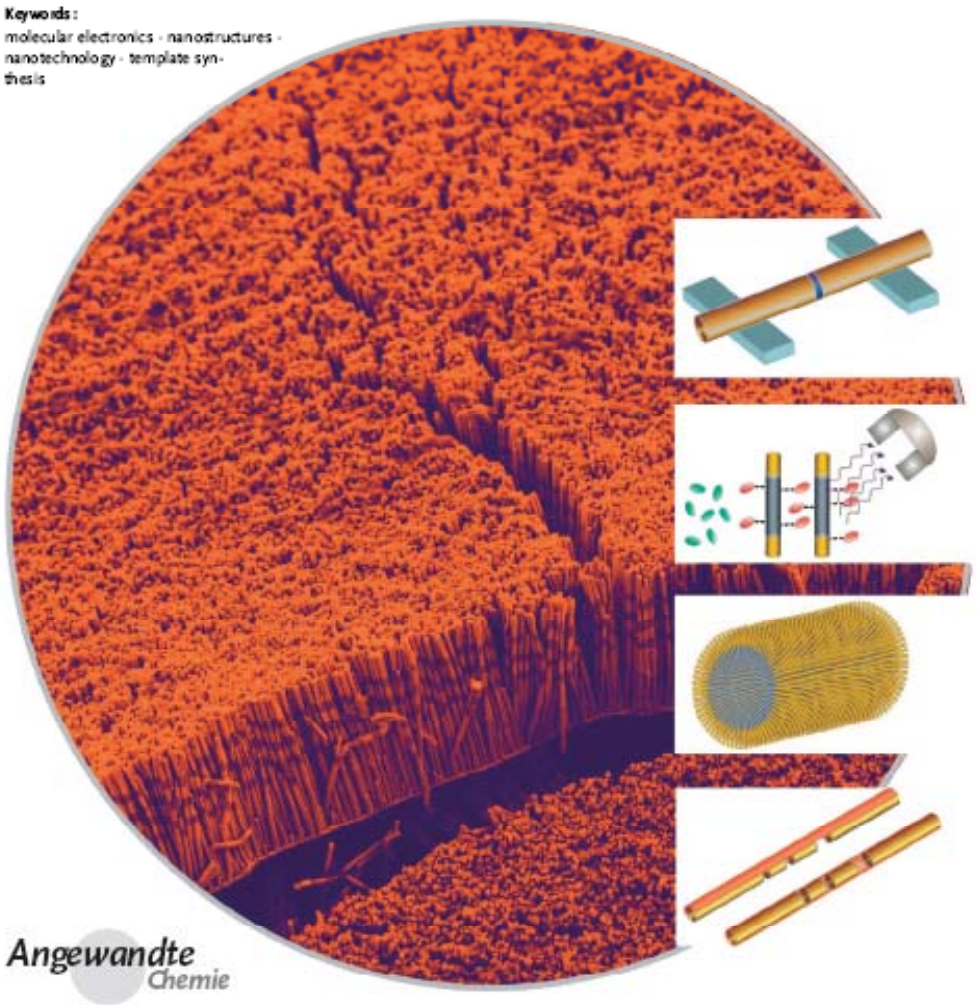


Gold-Nickel-gold
(d=200nm)

Gu, Ye, Gracias. *Journal of Materials (JOM)* **2005**, *57*, 60-64

Electron Microscopies: SEM

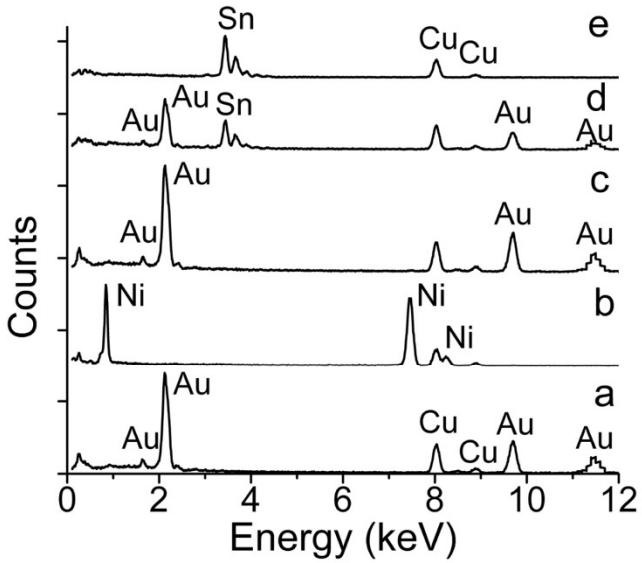
Example: SEM images of multisegment nanowires

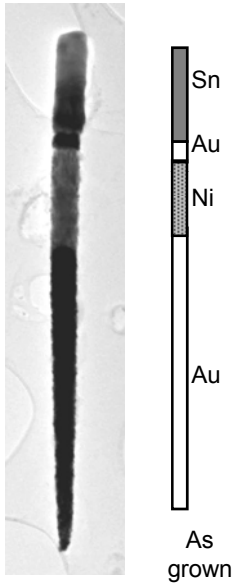


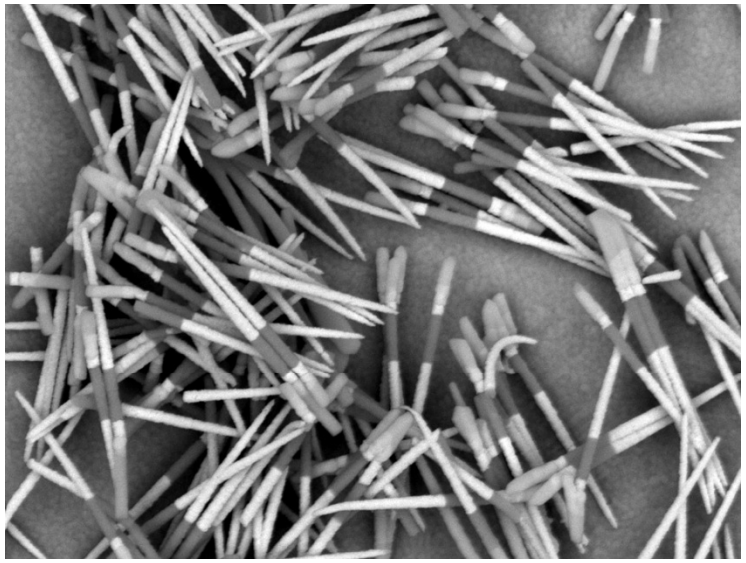
Hurst, et al. Angew. Chem. Int. Ed. 2006, 45, 2672 –2692

Electron Microscopies: TEM

Example: TEM images of solder reflow
and element analysis (EDS: Energy Dispersive X-Ray Spectroscopy)







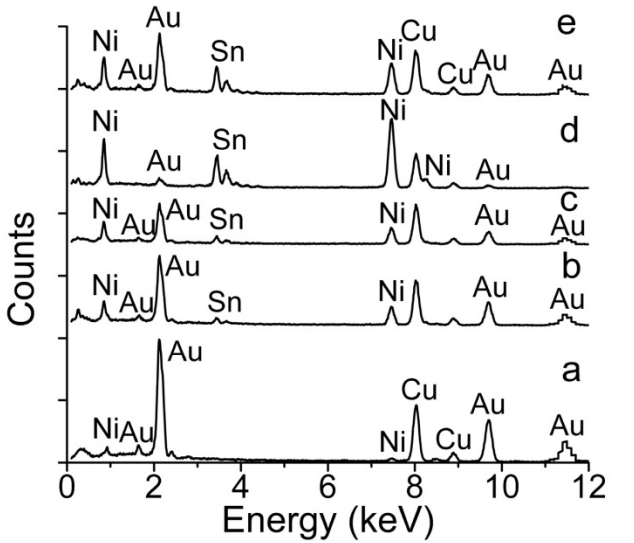
Before reflow

SEM images

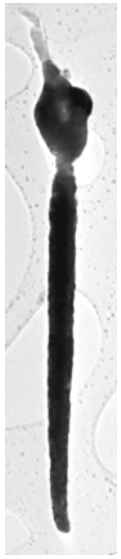
Gu, Ye, Smirnova, Small, Gracias, *Small* **2006**, *2*, 225-229

Electron Microscopies: TEM

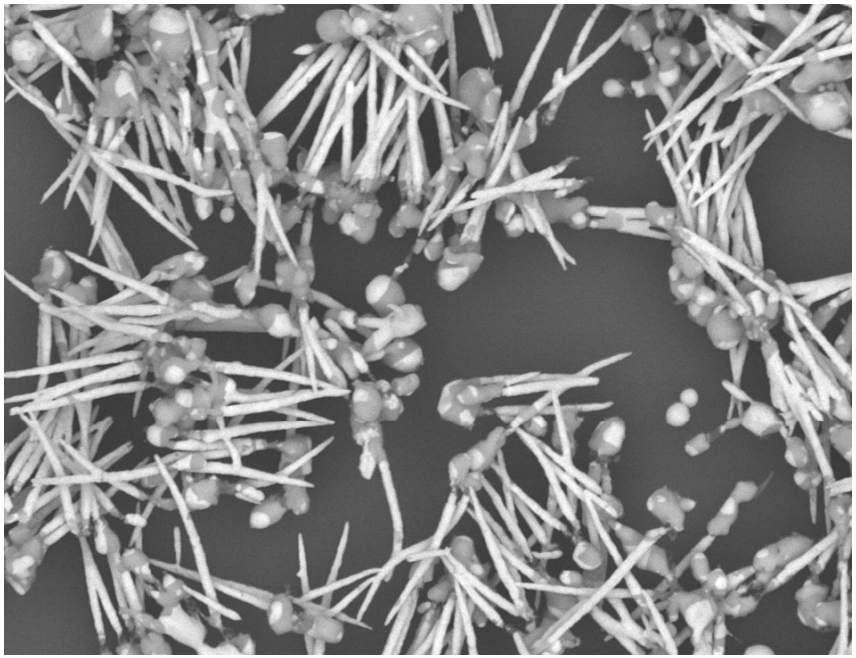
Example: TEM images of solder reflow
and element analysis (EDS: Energy Dispersive X-Ray Spectroscopy)



After reflow



d=50nm



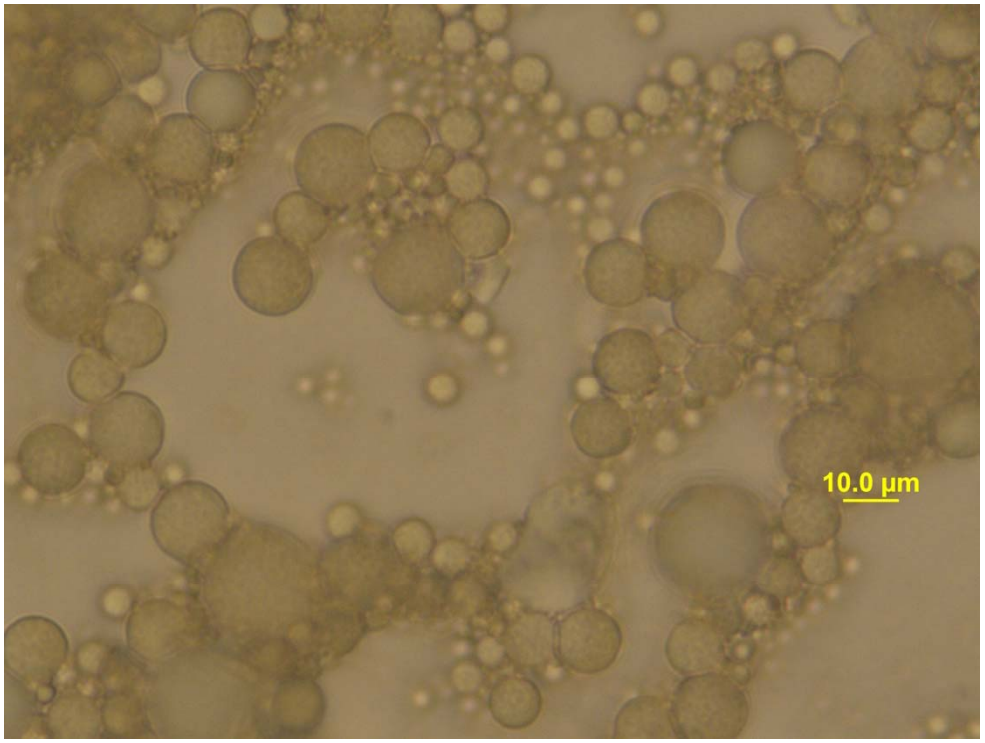
SEM images

Gu, Ye, Smirnova, Small, Gracias, *Small* **2006**, *2*, 225-229

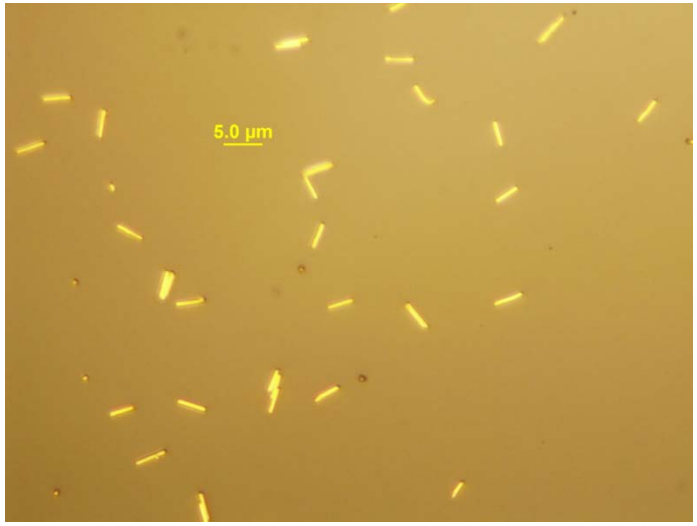
nano manufacturing center at UML

Optical Microscopies

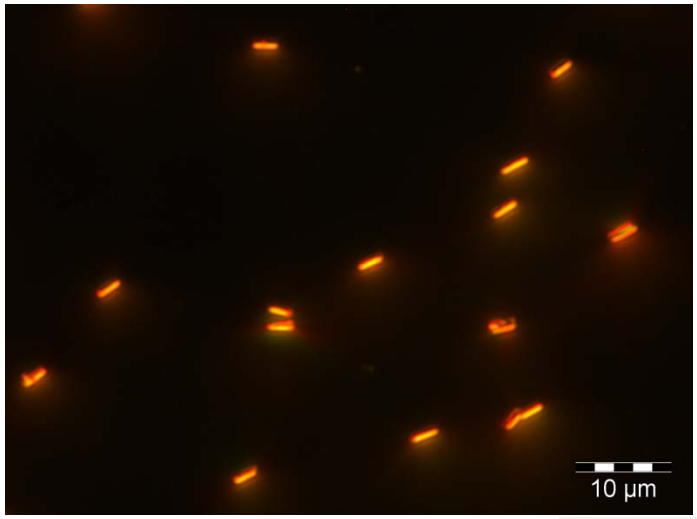
- Regular light microscopies
  - Bright field: sub-micron, x1000
  - Dark field: might see smaller features



Silica microparticles



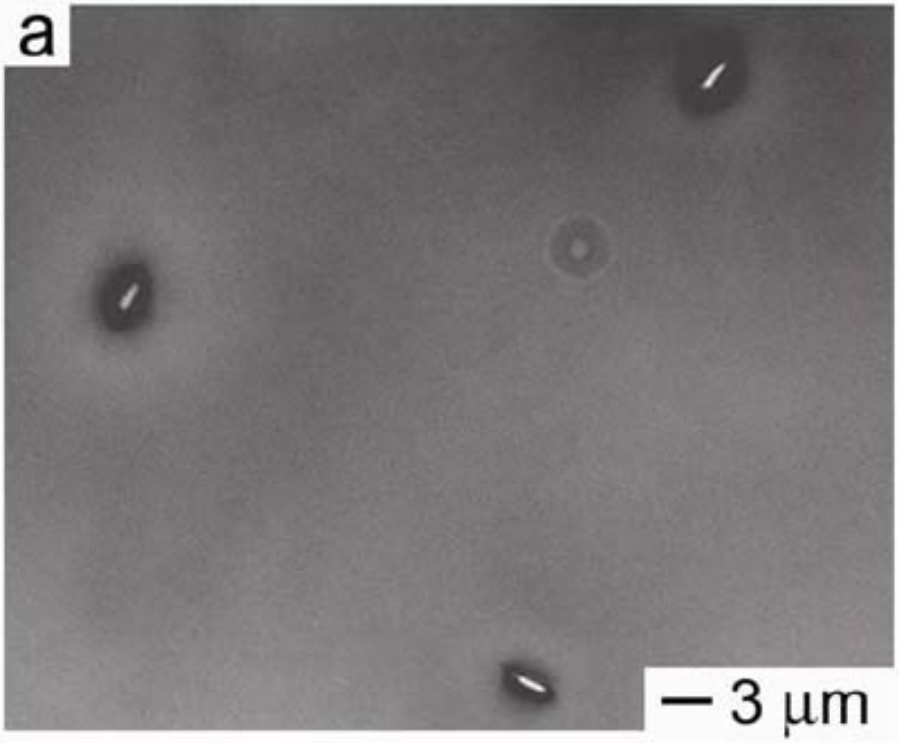
Au nanowires: bright field



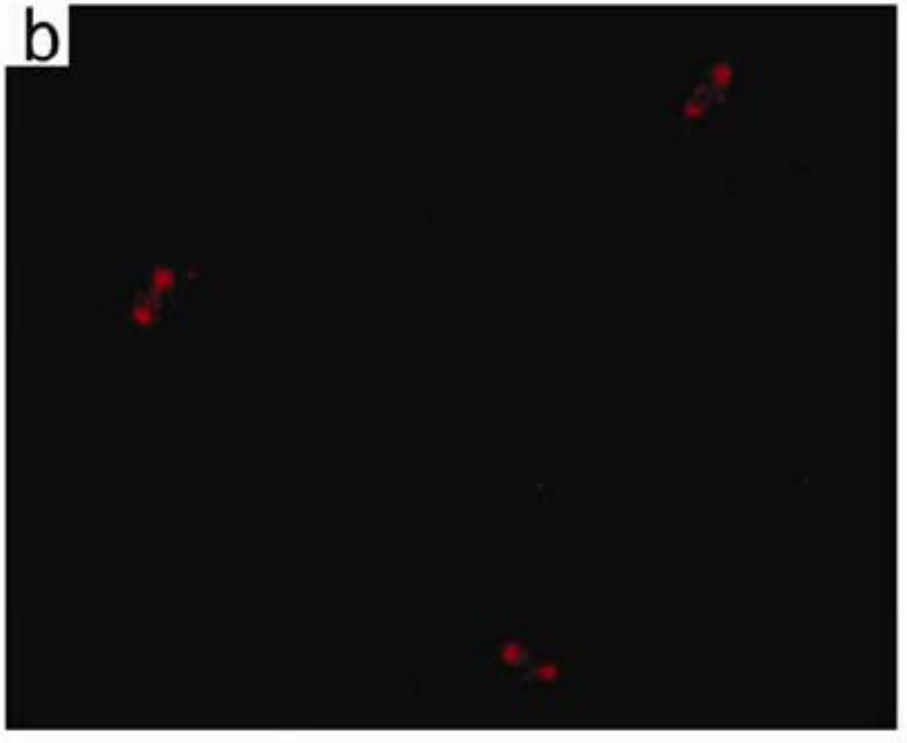
Au nanowires: dark field

Optical Microscopies

❖ Fluorescent microscopes



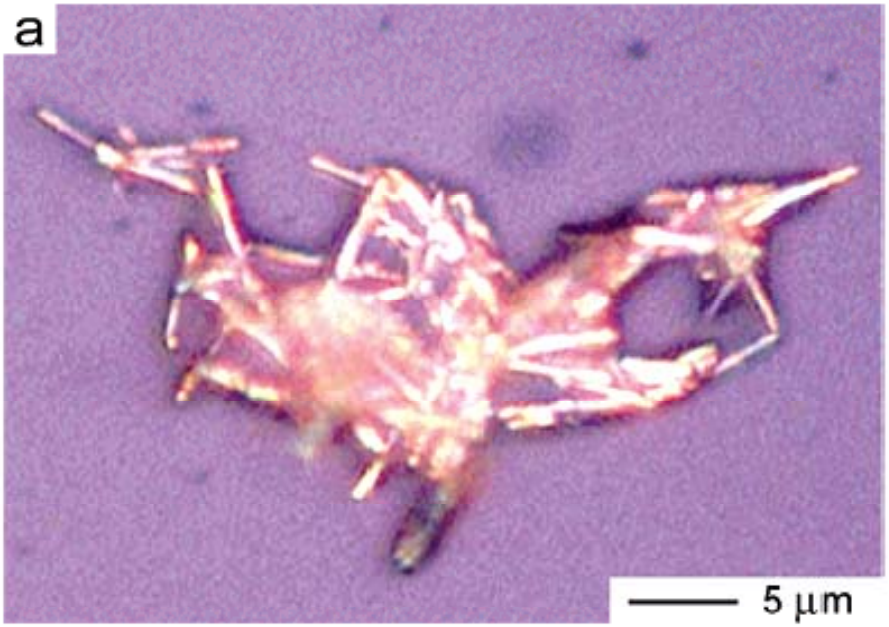
Optical image of Au-Pt-Au



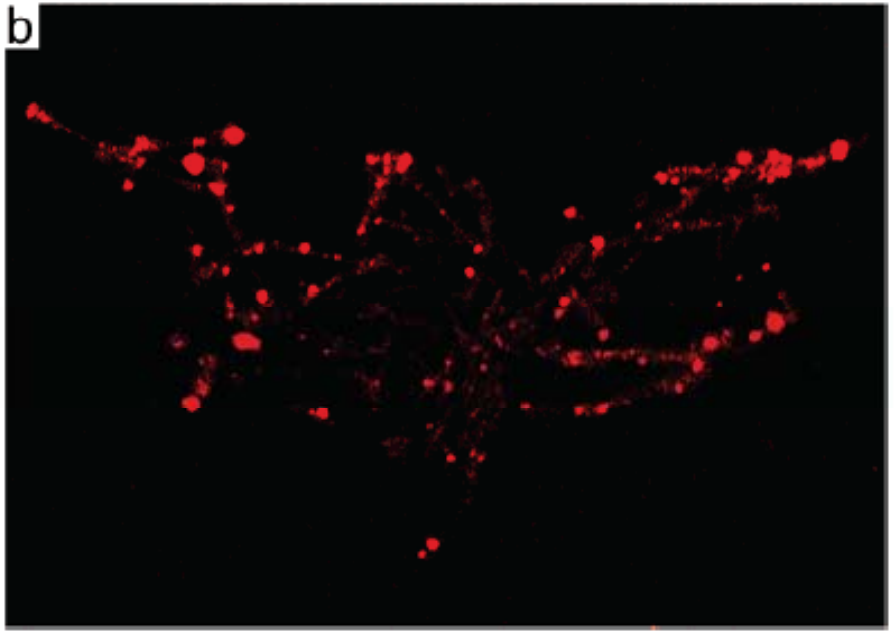
Fluorescent image of Au-Pt-Au

Optical Microscopies

❖ Scanning laser confocal microscopes



Optical image of Au-Pt-Au



Confocal image of Au-Pt-Au

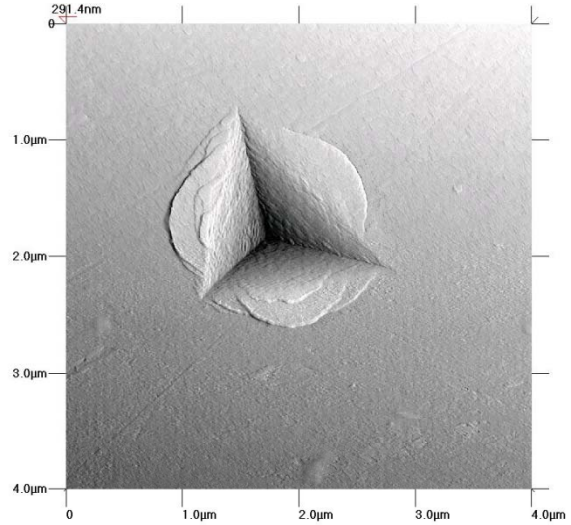
Nanoscale Measurements

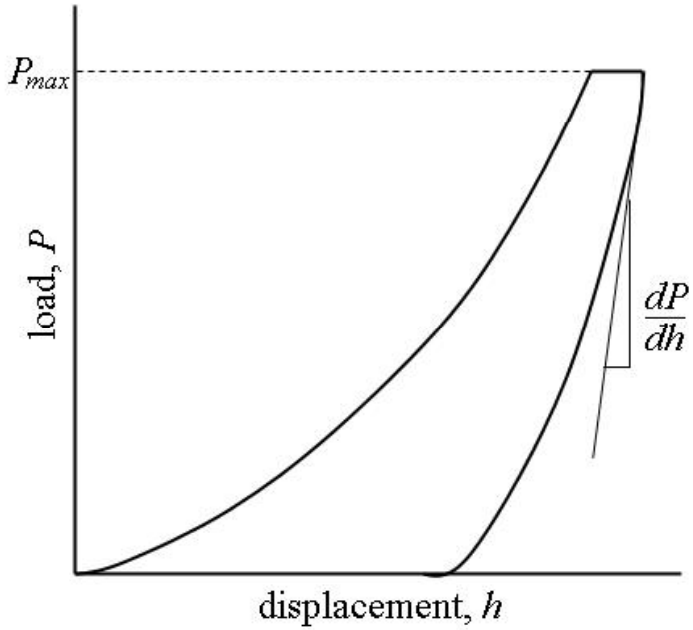
- Optical
- Electrical
- Mechanical
- Thermal
- ●●●

Mechanical Measurements: Nanoindentation

Nano-indenter (**TI 700 Ubi™**)

http://hysitron.com





Schematic of load-displacement curve for an instrumented nanoindentaion test

Nanometrologies

- Metrologies are needed in order to image and measure the properties of nanoscale objects

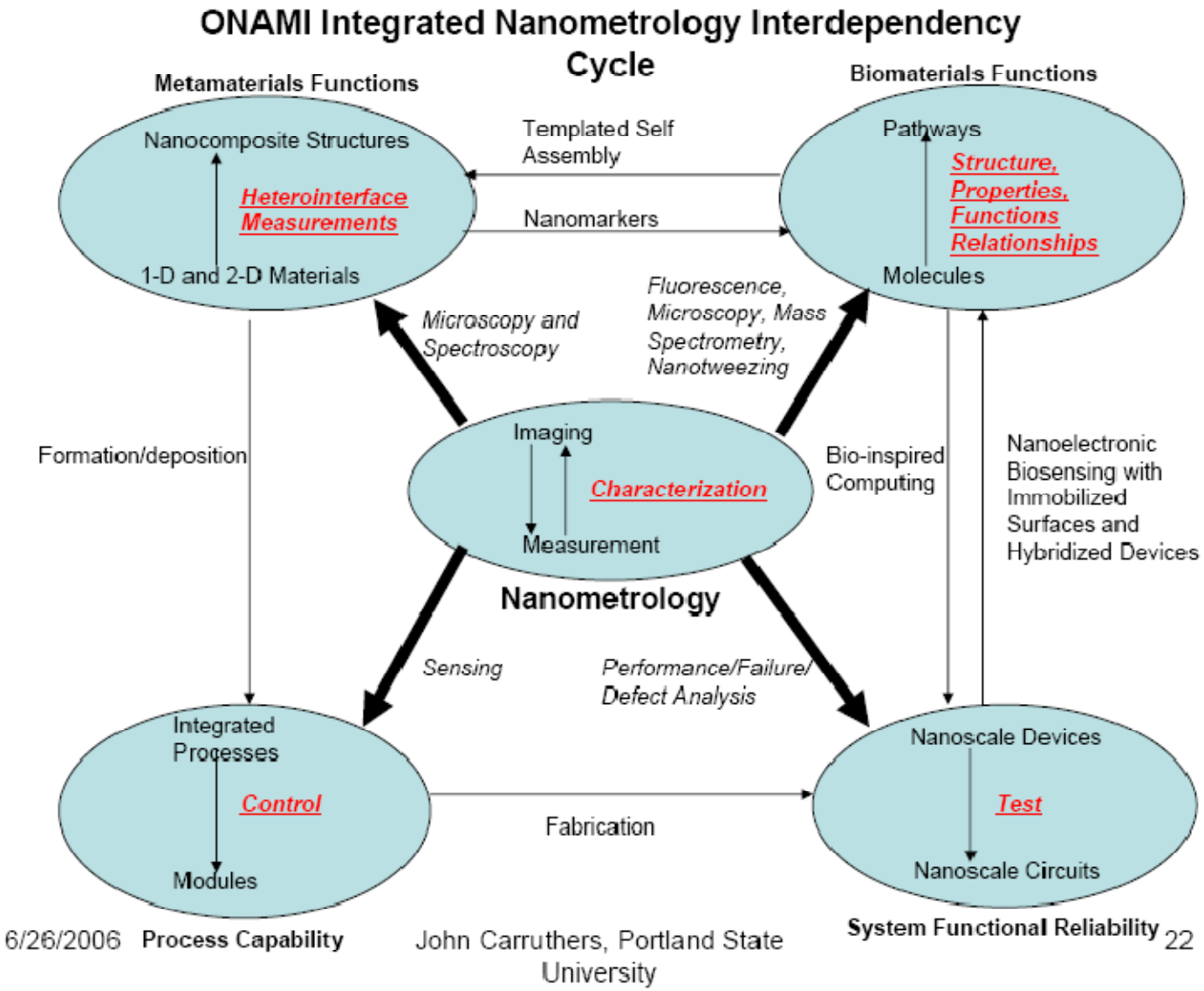
- Without capabilities such as electron microscopes, we would not have the microscale technologies we have today

- Similarly, we need measurement capabilities at the nanoscale, otherwise we will not be able to manufacture terascale circuits or validate their operational performance and reliability

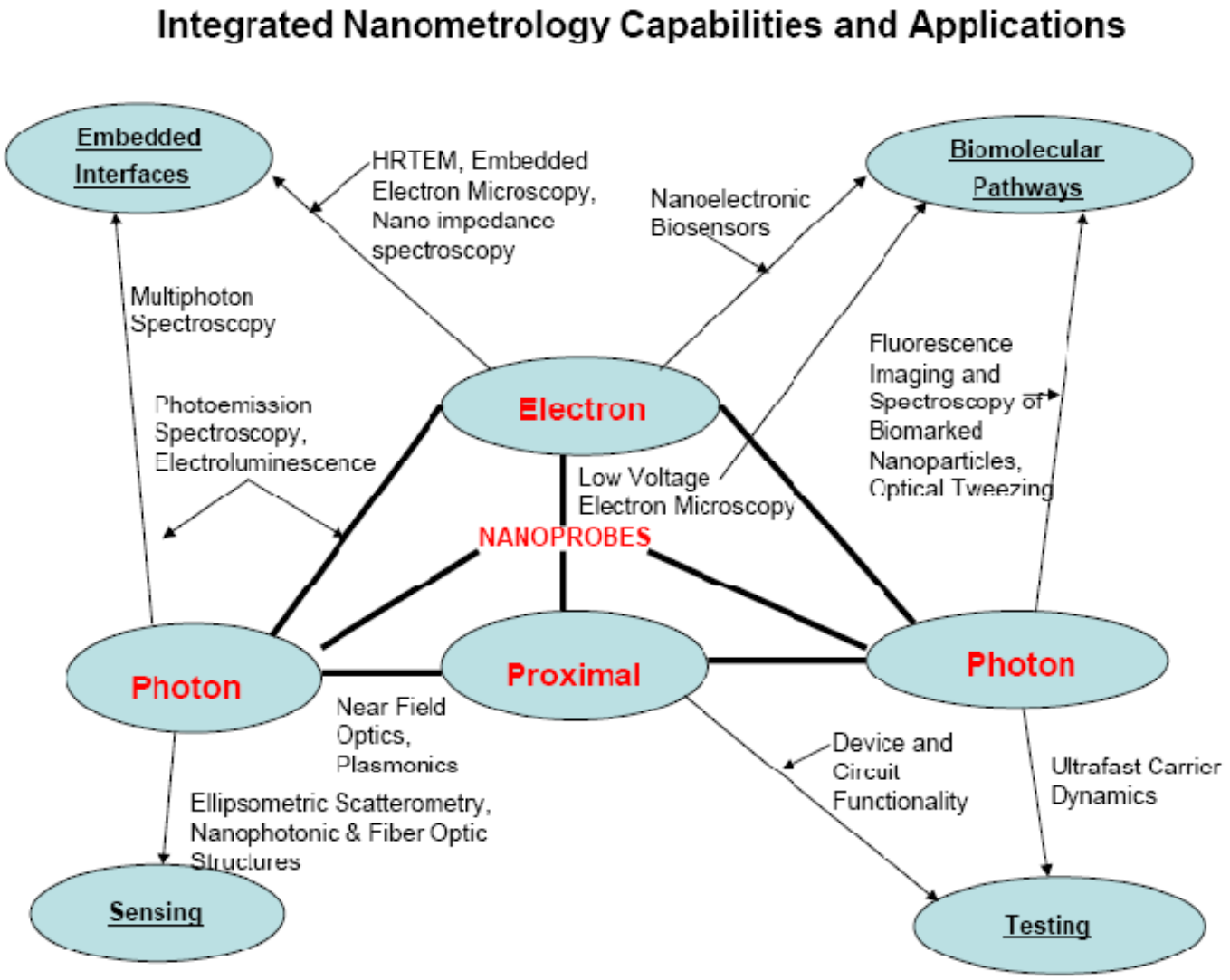
- Capabilities can be separated by the probes used:
  - –Charged particle probes (TEM, SEM, Ion probes)
  - –Proximal probes (STM, AFM, SPM)
  - –Electromagnetic probes (NSOM, Confocal microscopy)

John Carruthers, Portland State University

Nanometrologies



Nanometrologies



Nanometrologies

ISO Survey of Standardization Needs by National Body Members in the Next 1-3 years

Standard Methods for Toxicological Screening of Nanomaterials
Standard Methods for Determining Relative Toxicity/Hazard Potential of Nanomaterials
Standard Guide for Controlling Occupational Exposures to Nanomaterials
Standard Template for Material Safety Data Sheet (MSDS) for Products Containing Nanomaterials
Nanomaterial Product Information For Use In Determining Health & Safety Precautions
Standard Method for Selection of Personal Protective Equipment (PPE) for Use With Nanomaterials
Standard method for determining physical hazards of nanomaterials (i.e. explosive, flammability, water reactivity, etc.)
Standard Method to Establish Occupational Exposure Limits for Nanomaterials
Standard Methods to Assess Exposure to Nanomaterials During Consumer Product Use
Standard methods for determining nanoparticle concentrations in air and water