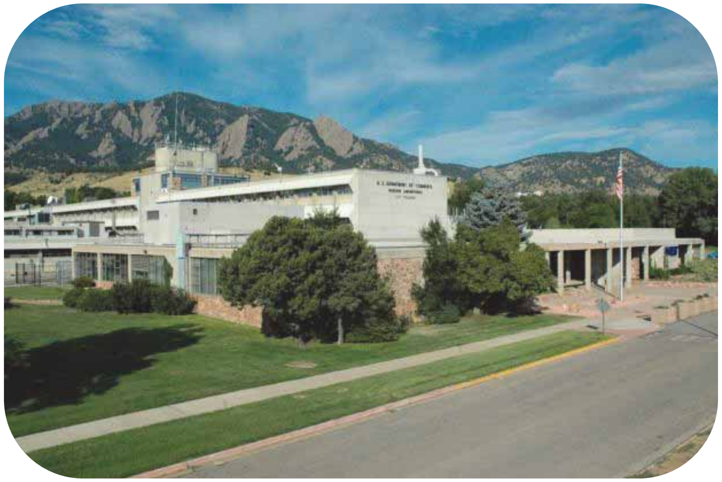
NIST and the Sensor NSI

Steve Semancik*
National Institute of Standards and Technology
Biomolecular Measurement Division
Gaithersburg, MD

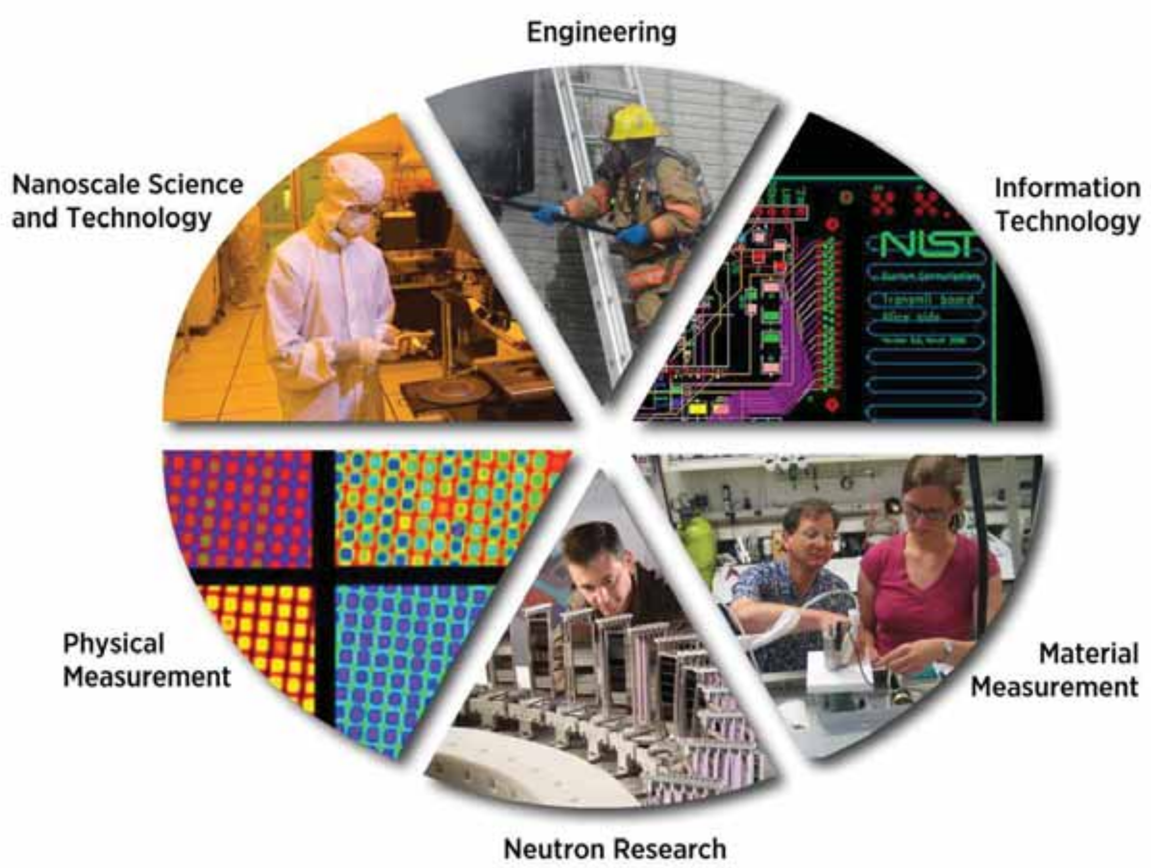
2 campuses:
Gaithersburg, MD
Boulder, CO
~ 3,000 employees
~ 2,600 associates and facilities users



Mission: to promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life

* Chemical & Biochemical Microsensor Project Leader
stephen.semancik@nist.gov

NIST Palette of Technical Efforts



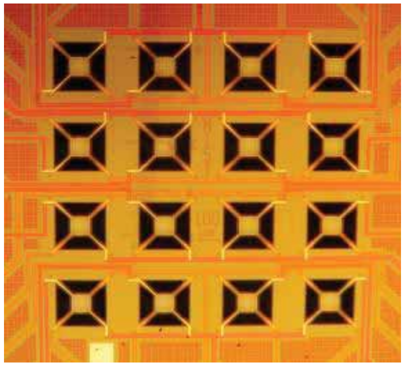
The Sensor NSI at NIST

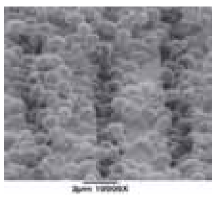
- Nanomaterials-Enabled Sensor Research
- Nano EHS
- Center for Nanoscale Technology Facilities/Activities

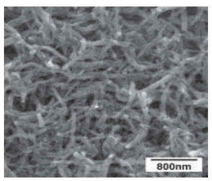
NIST

Sensing Nanomaterials Integrated with Functional Platforms

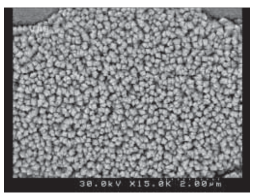
Chemiresistive Microhotplate Array Gas Sensors

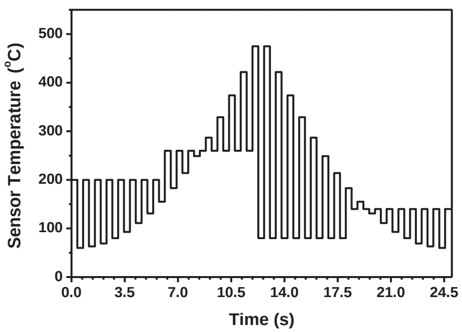






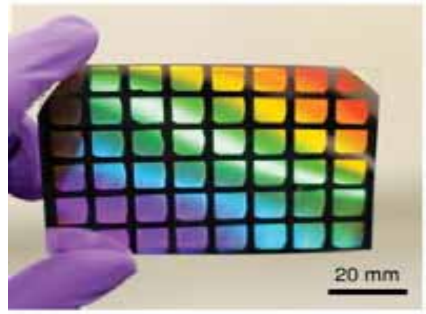
features
10s – 100s nm





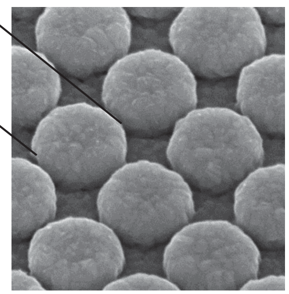
multiple types of nanomaterials and rapid T programming for selectivity

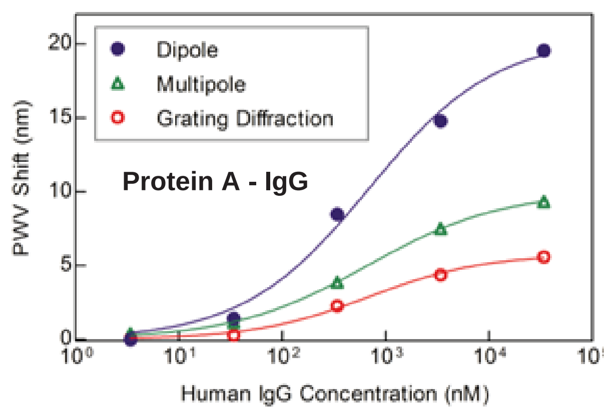
Nanodome Nanomaterials for Flexible Photonic Biosensing



380 nm

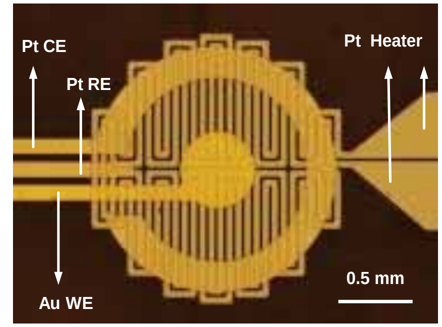
Ag domes
18 nm gaps

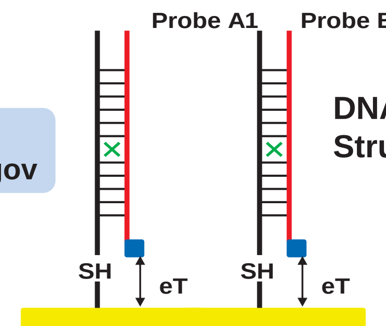




enhanced sensitivity in localized plasmonic measurements

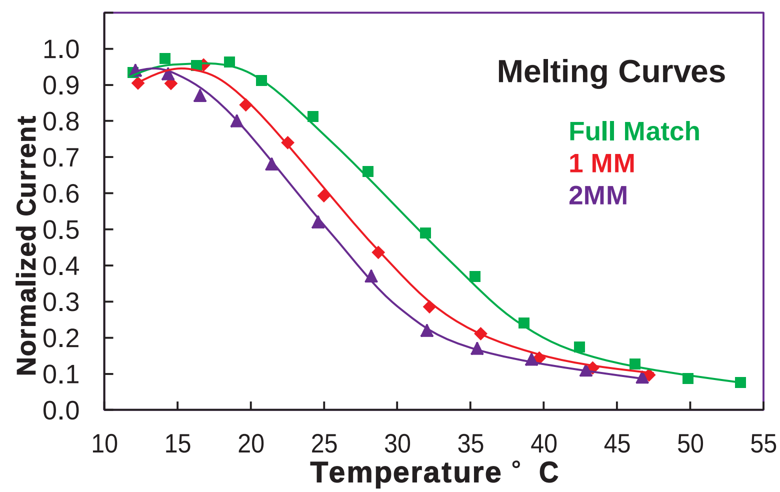
Microscale EC Cell for Biomolecular Measurements





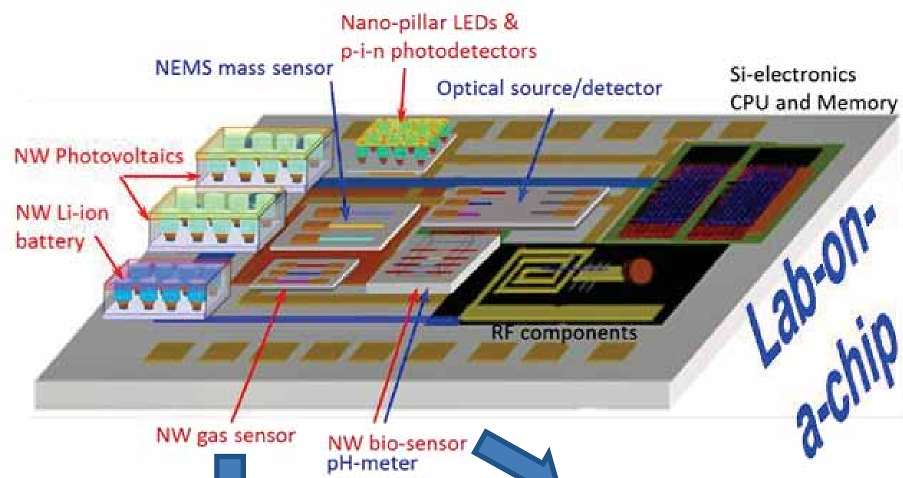
DNA Structures

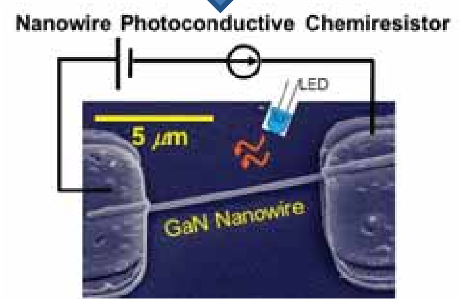
Contact:
stephen.semancik@nist.gov

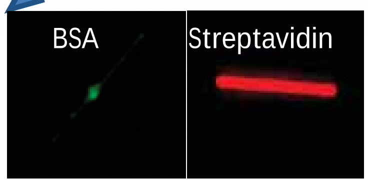


single nucleotide polymorphism detection on small sample volumes

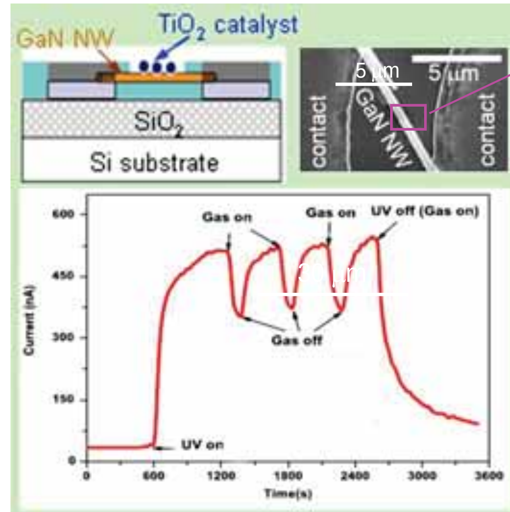
Semiconductor Nanowire Materials for Sensing



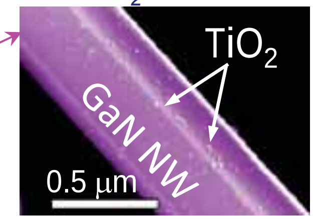




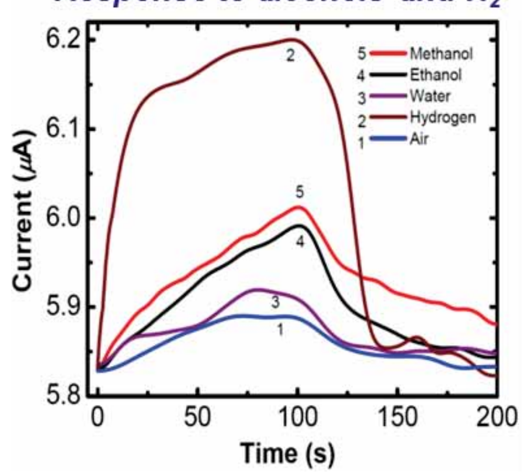
GaN-NW / TiO_2-nanocluster sensor & its response to toluene exposure



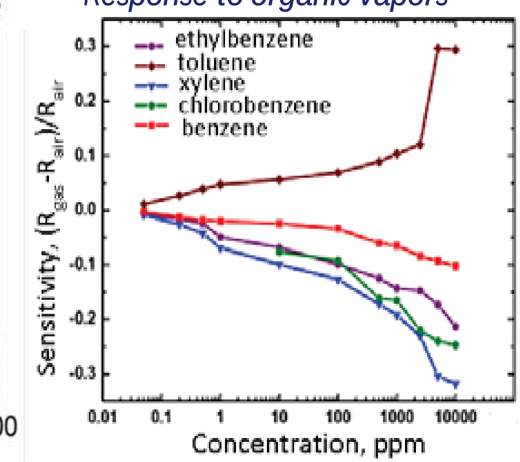
GaN NW decorated with TiO_2 clusters



Response to alcohols and H_2



Response to organic vapors



Nanowire Sensors Advantages:

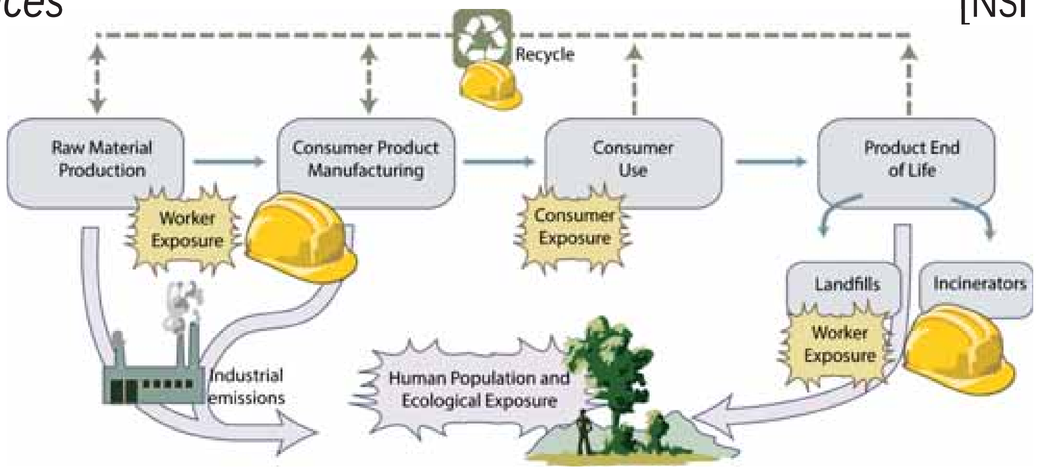
- Tunability based on nano-catalyst used
- Broad sensing range: sub-ppm to 1 %
- High sensitivity: ~ 59 ppb to benzene and related environmental pollutants
- Room temperature operation
- UV-light "turn on" sensing
- Fast recovery time

GaN nw Contact:
albert.davydov@nist.gov

NIST

Sensors for Nanotechnology/NanoEHS (debra.kaiser@nist.gov)

Develop tools, methods, and devices to detect, identify, and quantify engineered nanomaterials (ENMs) across their life-cycles in a variety of environmental and biological matrices [NSI White Paper]



NIST-relevant activities

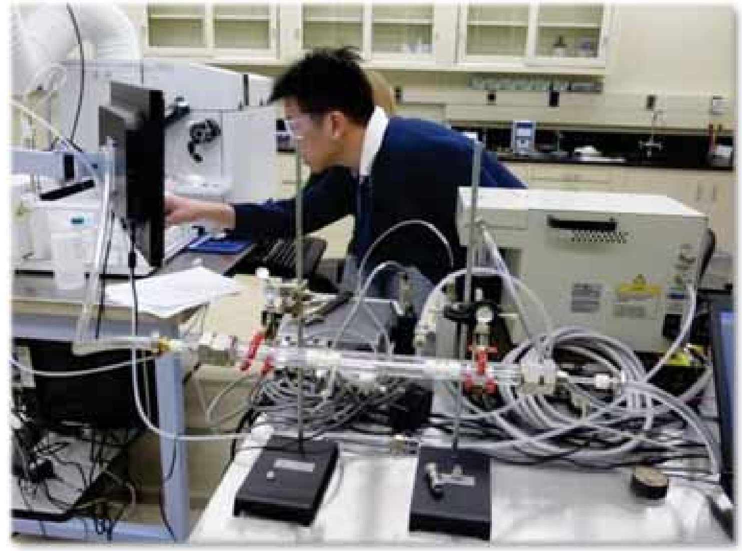
- Hyphenated *instruments* and *methods* to simultaneously measure two or more properties of ENMs in liquid media
- Nanoscale *reference materials* to ensure accurate and reproducible measurements of ENM properties
- *"Tagged" ENMs* to track environmental pathways: fluorescent silicon nanocrystals
- *Methods and protocols* to detect and quantify ENMs in consumer products using broadly available and applicable instruments:
  - Multi-wall carbon nanotubes (MWCNTs) in polymer-based products
  - Silver nanoparticles (NPs) in textiles

NIST

Hyphenated Instruments and Methods

Objective

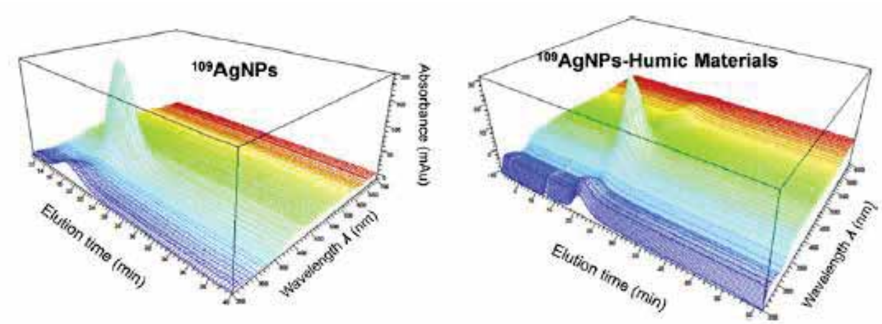
- Develop, validate and apply novel measurement instrumentation and methods based on coupling of size discrimination with multiple detection modes
- High resolution size analysis with real-time quantification of bulk and surface composition, analyte concentration, isotope speciation, agglomeration; particle number, z-average and mass size distributions
- Utilize ENM stable ENM isotopes (*e.g.*, Ag^{109}/Ag^{107}) to interrogate transformations and fate



Examples of hyphenated instruments

- Couple electrospray dynamic mobility analysis (ES-DMA) with inductively coupled plasma mass spectrometry (ICP-MS) for ultra-high resolution size discrimination of aerosolized ENMs combined with quantitative analysis
- Couple asymmetric-flow field flow fractionation (A4F) with optical detection (MALS, UV/Vis, fluorescence, DLS) and ICP-MS for separation and analysis of ENM populations in complex media

3D A4F-UV fractograms for AgNPs with and without Suwannee River Humic Acid



Contact: Vincent Hackley, NIST

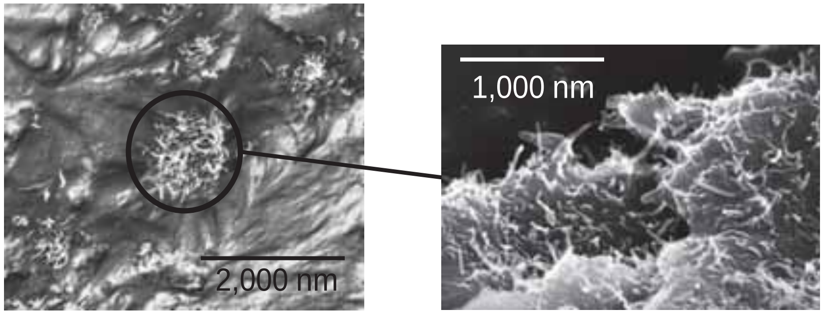
Detection, Quantification, and Release of ENMs in Products

Motivation and Objective

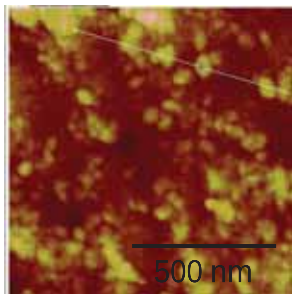
- ENMs are incorporated in over 1,600 consumer products
- Questions concerning the presence, form, and concentration of ENMs in such products
- Concerns about the release of ENMs from products into the environment and human exposure
- Develop measurement protocols, based on broadly available and applicable instruments, to detect and quantify ENMs in two large classes of products and to assess release of ENMs from products

Approach

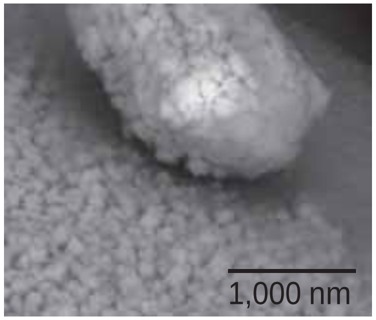
- Two classes of products and methods studied:
    - MWCNTs in epoxy: *electron microscopy (SEM, TEM), atomic force microscopy (AFM), Raman spectroscopy, and X-ray photoelectron microscopy (XPS)*
    - silver NPs in textiles: *SEM, AFM, XPS, inductively-coupled plasma mass spectroscopy*
- Develop methods and protocols for sample preparation and measurements by each method
- Establish ENM detection limits for each method
- Initiate work on methods to determine the release of ENMs in environmental media



SEM image showing typical clumping of MWCNTs in epoxy composite



AFM image of silver NPs in a cotton thread



SEM image of silver NPs in a commercial textile

NIST Nano-scale Reference Materials (RMs)

Gold Nanoparticles: RMs 8011, 8012, and 8013

- Nominal diameter measurements by six techniques
- 10 nm, 30 nm, and 60 nm particles in liquid suspension
- Contact: Vince Hackley, vhackley@nist.gov

Single-Wall Carbon Nanotubes (Raw Soot): SRM 2483

- Elemental composition measurements (carbon and soot)
- Contact: Jeffrey Fagan, jfagan@nist.gov

Polystyrene Spheres: SRMs 1963a and 1964

- Nominal diameter measurements
- 100 nm and 60 nm spheres
- Contact: Michelle Donnelly, mdonnelly@nist.gov

Titanium Dioxide Nanomaterial, SRM 1898

- Specific surface area by BET; dry powder
- Contact: Vince Hackley, vhackley@nist.gov

Single-wall Carbon Nanotubes, RM 8281

- Three length-sorted populations of SWCNTs in liquid dispersions
- Contact: Jeffrey Fagan, jfagan@nist.gov

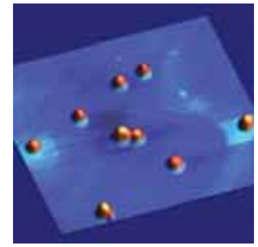
Fluorescent Silicon Nanocrystals, RM 8027 (Winter 2014)

- Nominal diameter homogeneity, silicon mass fraction
- 2 nm particles in liquid suspension
- Technical contact: Vytas Reipa, vreipa@nist.gov

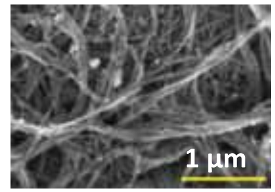
Silver Nanoparticles, RMs 8016 and 8017 (Fall 2014)

- Nominal diameter measurements: 10 nm and 75 nm, freeze dried
- Technical contact: Vince Hackley, vhackley@nist.gov

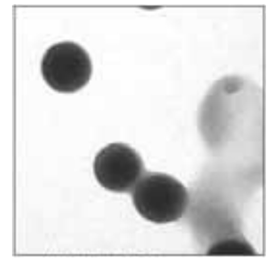

Gold RMs

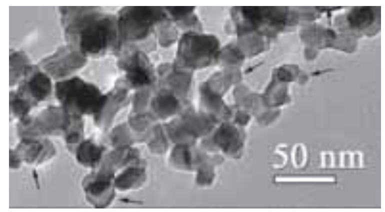

AFM image, 30 nm gold RM


SRM 2483



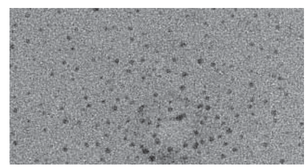
SEM image, SRM 2483


TEM image ,SRM 1963a

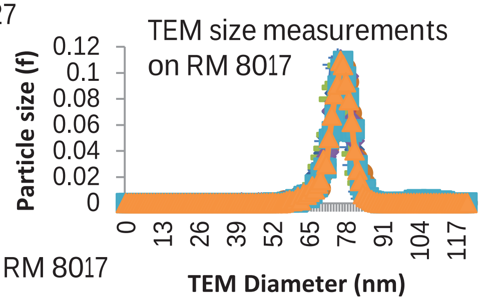


TEM image, SRM 1898

RM 8281


SEM image, SRM 8027


RM 8017

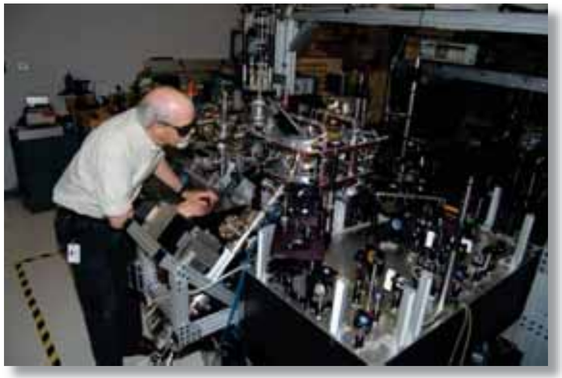


The CNST in Brief

Established in 2007 to develop nanoscale measurement and fabrication methods *specifically* for advancing nanotechnology "*from discovery to production*"

- A User Facility with a unique, hybrid design

- The **NanoFab** is a shared resource with commercial state-of-the-art tools open to all [like NSF-supported university nanocenters]
  - 60,000 ft^2 (5600 m^2) of labs and cleanroom for nanofabrication
  - 19,000 ft^2 (1800 m^2) cleanroom;
  - 8,000 ft^2 (750 m^2) at class 100
  - ~ 100 major tools, including advanced lithography (e-beam x2, ASML stepper), microscopy (FE-SEM, FIB, TEM)

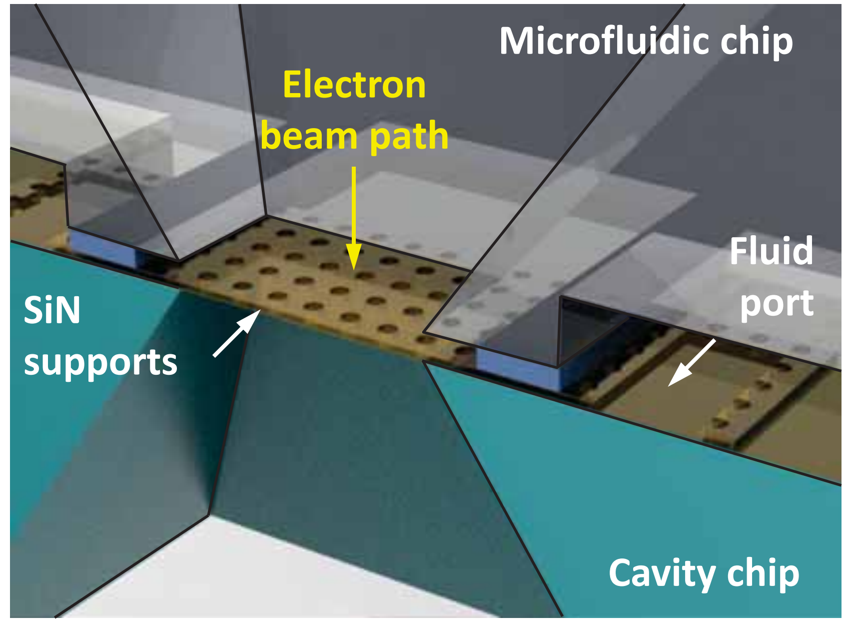


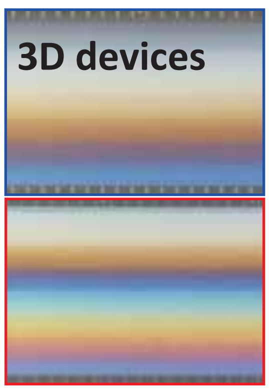
- The **NanoLab** advances nanotechnology by developing new measurement solutions, and supports the NanoFab with expert consultation [like DoE nanocenters]

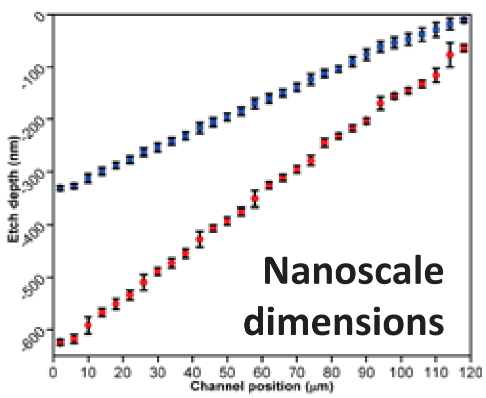
Nanofabricated Fluidic Nanoparticle Sensors

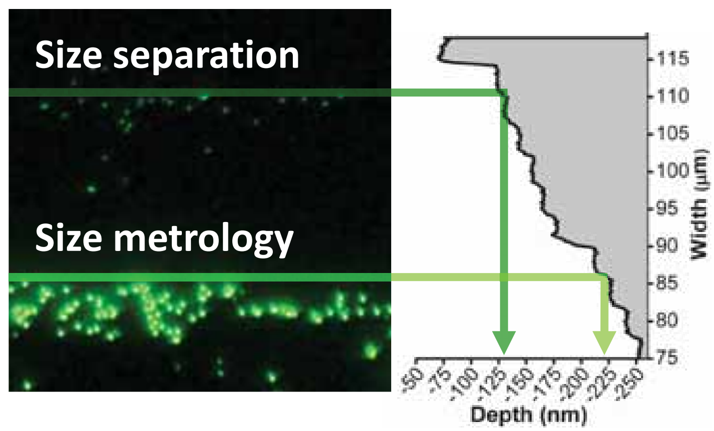
- Nanoscale confinement and transport of nanoparticles dispersed in fluids
- Measurement by electron microscopy, energy loss spectroscopy, optical microscopy
- Size separation by 3D nanofluidic size exclusion
- Towards nanofabricated fluidic nanosensors for integrated separation and characterization of nanoparticles

james.liddle@nist.gov
samuel.stavis@nist.gov





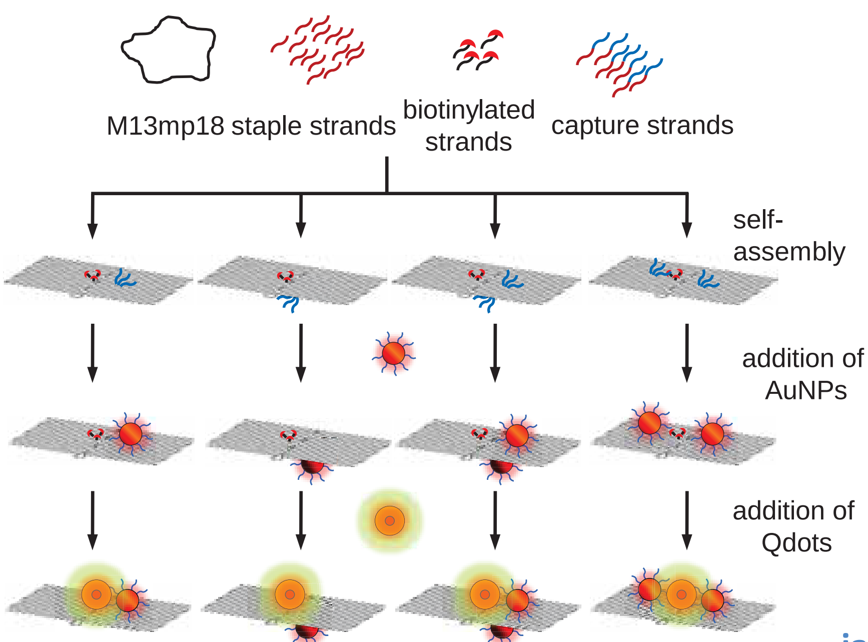




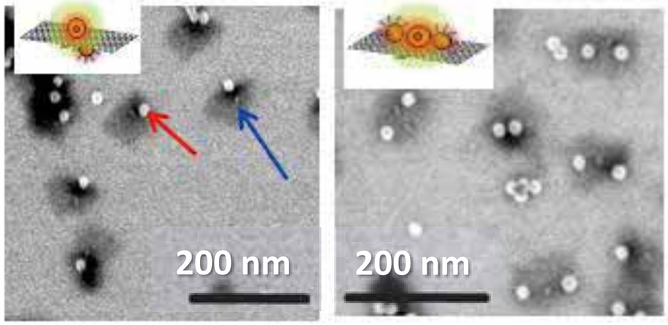
Nanomanufactured DNA Optical Sensors

- DNA origami as a nanomanufacturing platform for hybrid nanostructures
- Nanoassembly with molecular precision enables control and measurement of nanoscale optical phenomena
- Towards nanomanufactured optical nanosensors for nanoparticles

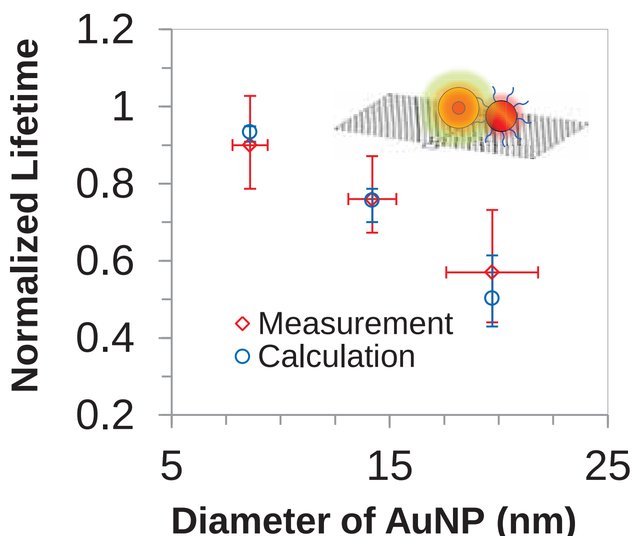
AuNP-Qdot nanoassembly process



Electron micrographs of hybrid nanostructures



Qdot lifetime modulated by AuNP size



james.liddle@nist.gov

Smart Sensor Standards

- Smart sensor and actuator communication interface standards – IEEE 1451 (ISO/IEC 21451)
  - Support self-identification and self-description of sensors via standardized electronic datasheets
  - Define metadata, physical units, and messaging protocols
  - Support wired and wireless networks, and secure Internet access
- Clock synchronization standard (for sensor and measurement systems) – IEEE 1588 (IEC 61588)
  - Define a time synchronization protocol for networked measurement and control systems
  - Support various industries (test and measurement, power and utility, telecom, semiconductor, …), which can benefit from precise synchronization of measurements to a nanosecond.
- For more information
  - Contact: kang.lee@nist.gov or 301-975-6604

NIST