



Measurement Challenges in Nanomanufacturing

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Nanofabrication Research Group



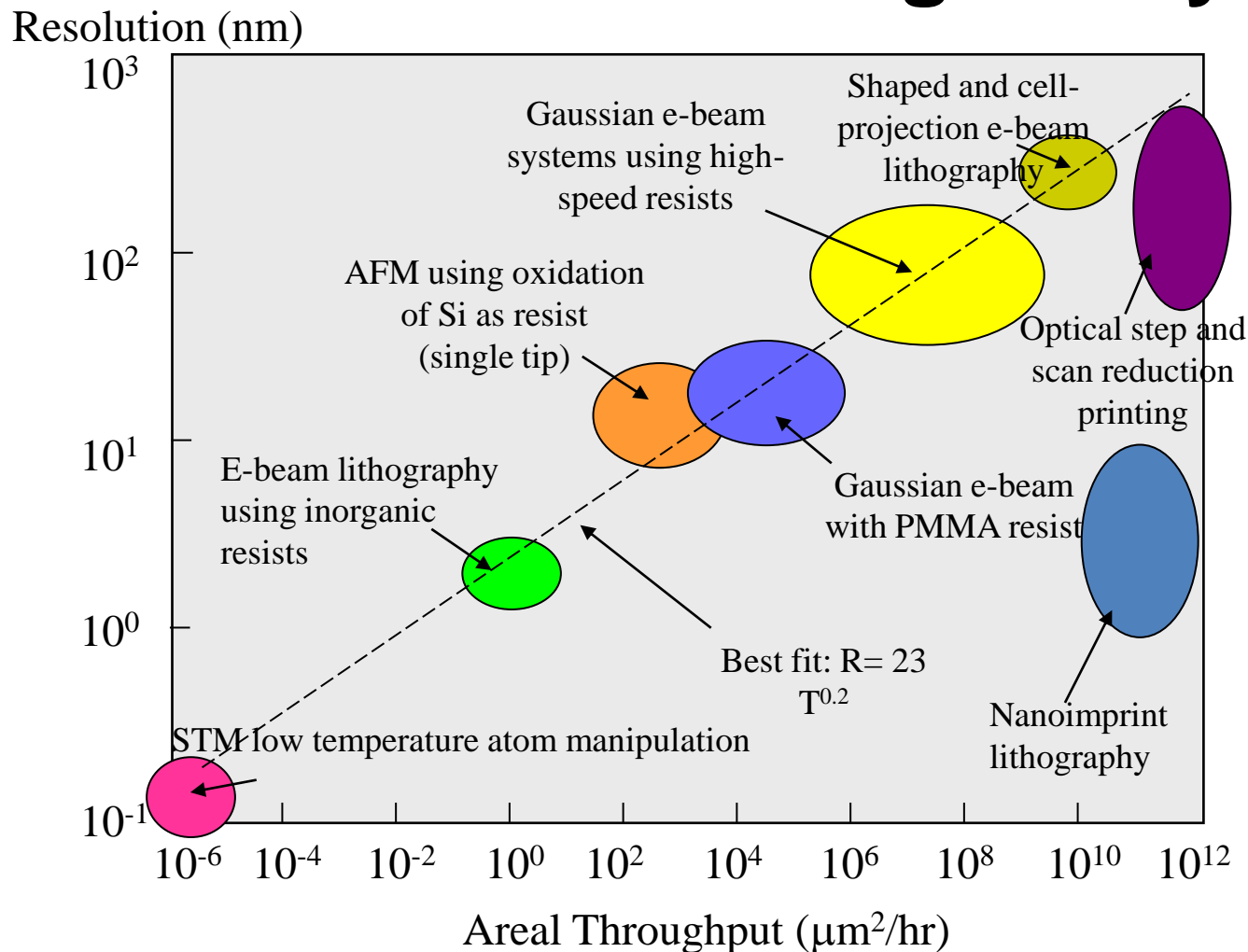
New England Nanomanufacturing Summit 2010

CNST

- Provides the **measurement and fabrication infrastructure** to support all phases of nanotechnology development from discovery to production.
 - Fee-for-use, state-of-the-commercial-art measurement and fabrication capabilities are provided by developing and maintaining a national shared resource, the **NanoFab**.
 - Beyond state-of-the-commercial-art nanoscale measurement and fabrication solutions are developed and provided using a **multidisciplinary** approach that involves partnering with industry, academia, and government.
- Serves as a hub to **link the external nanotechnology community** to the vast measurement expertise that exists within the NIST Laboratories.
- Helps to **educate** the next generation of nanotechnologist.

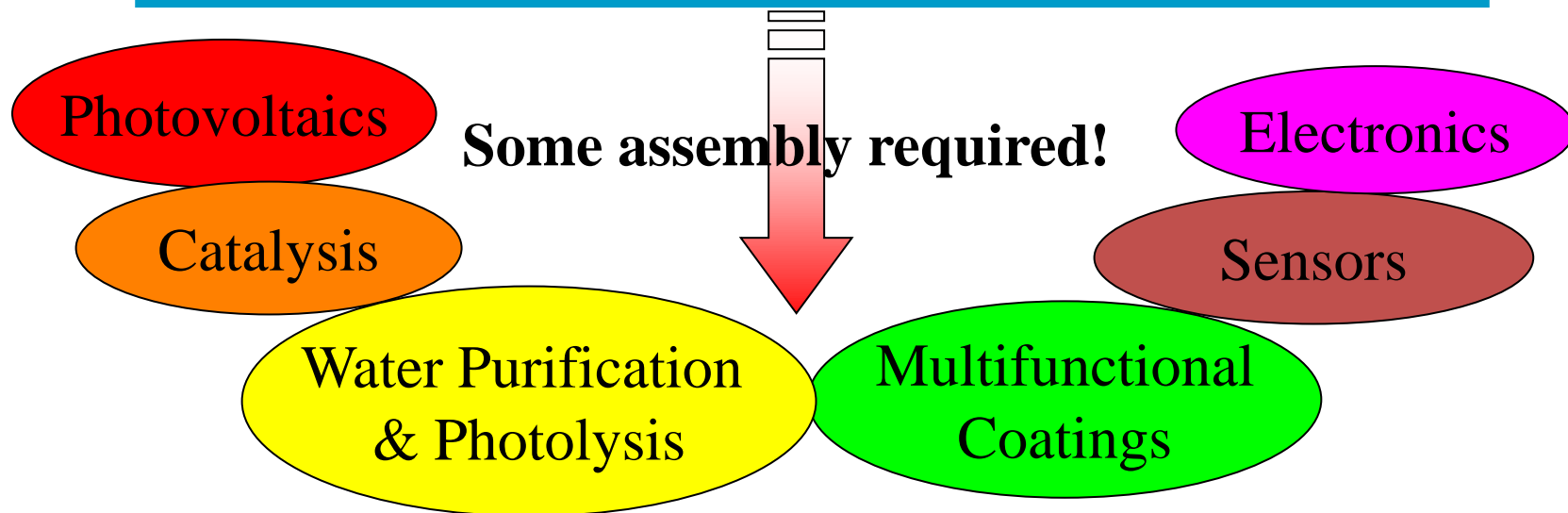
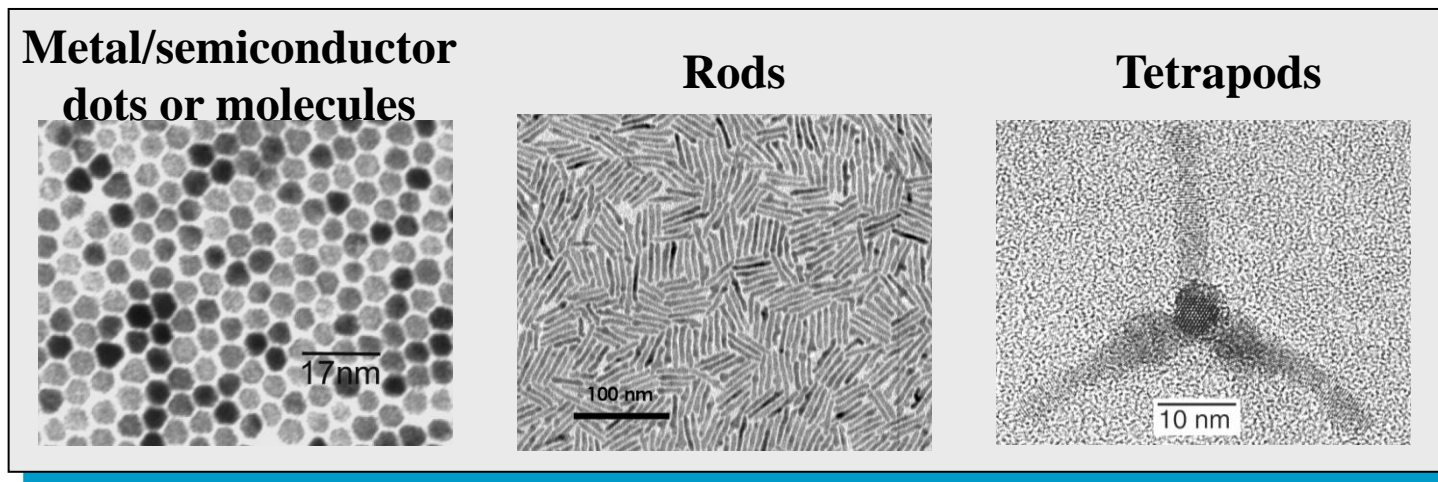
www.nist.gov/cnst

Nanomanufacturing Today



After D.M. Tennant and C.R. Marrian, *J. Vac. Sci. Technol.* (2003)

Nanomanufacturing Tomorrow





Where do we need to go?

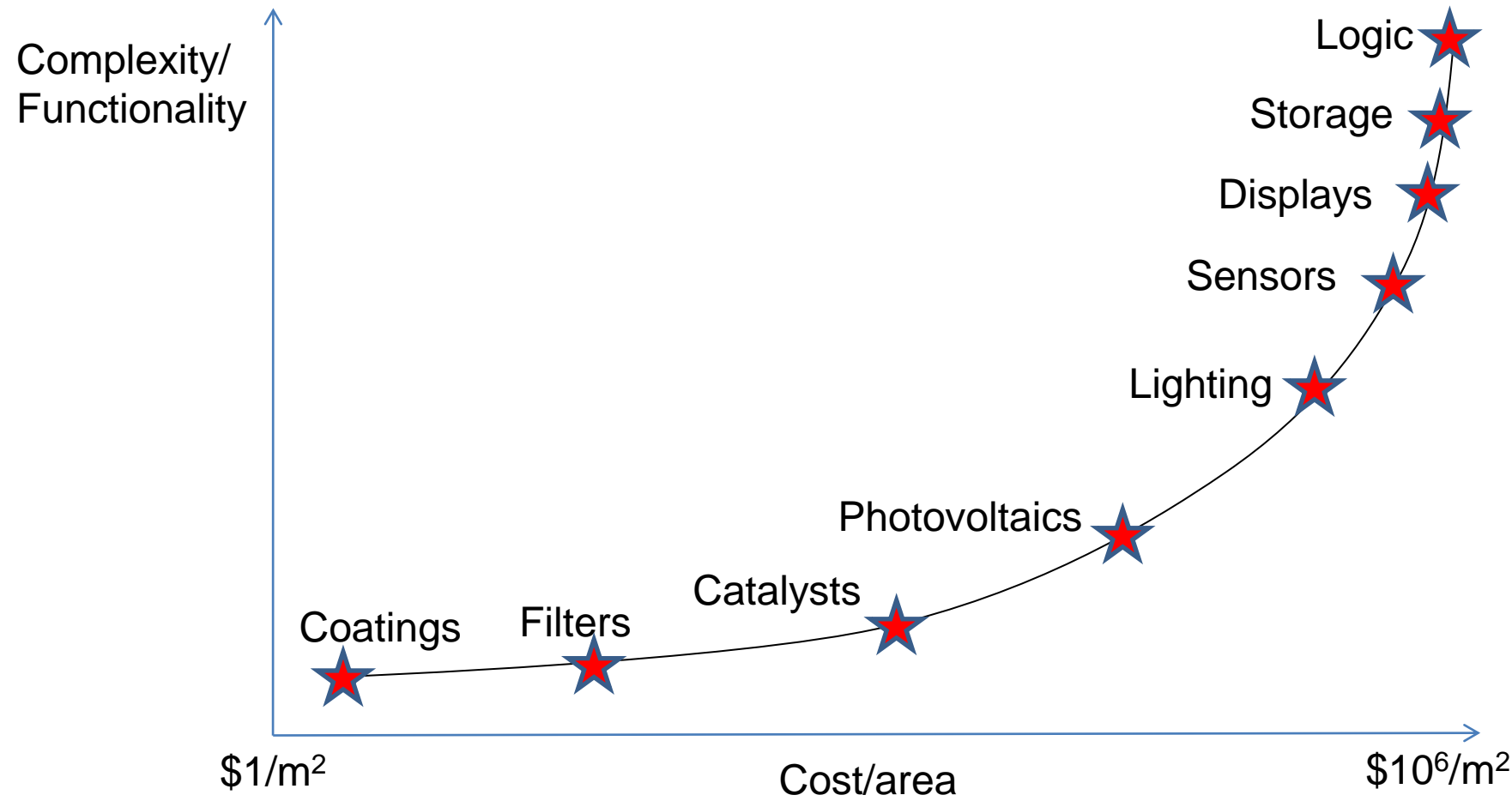
- 193 nm immersion lithography
 - 40 nm pixels, $3 \times 10^{-3} \text{ m}^2/\text{s}$
 - $6.25 \times 10^{14} \text{ pixels/m}^2$, $2 \times 10^{10} \text{ pixels/s}$
- Letterpress
 - 40 μm pixels, $30 \text{ m}^2/\text{s}$
 - $5 \times 10^8 \text{ pixels/m}^2$, $1.5 \times 10^{10} \text{ pixels/s}$
- $10^4 \times$ areal throughput, $10^6 \times$ feature density
- 2×10^{16} 40 nm pixels/s or better

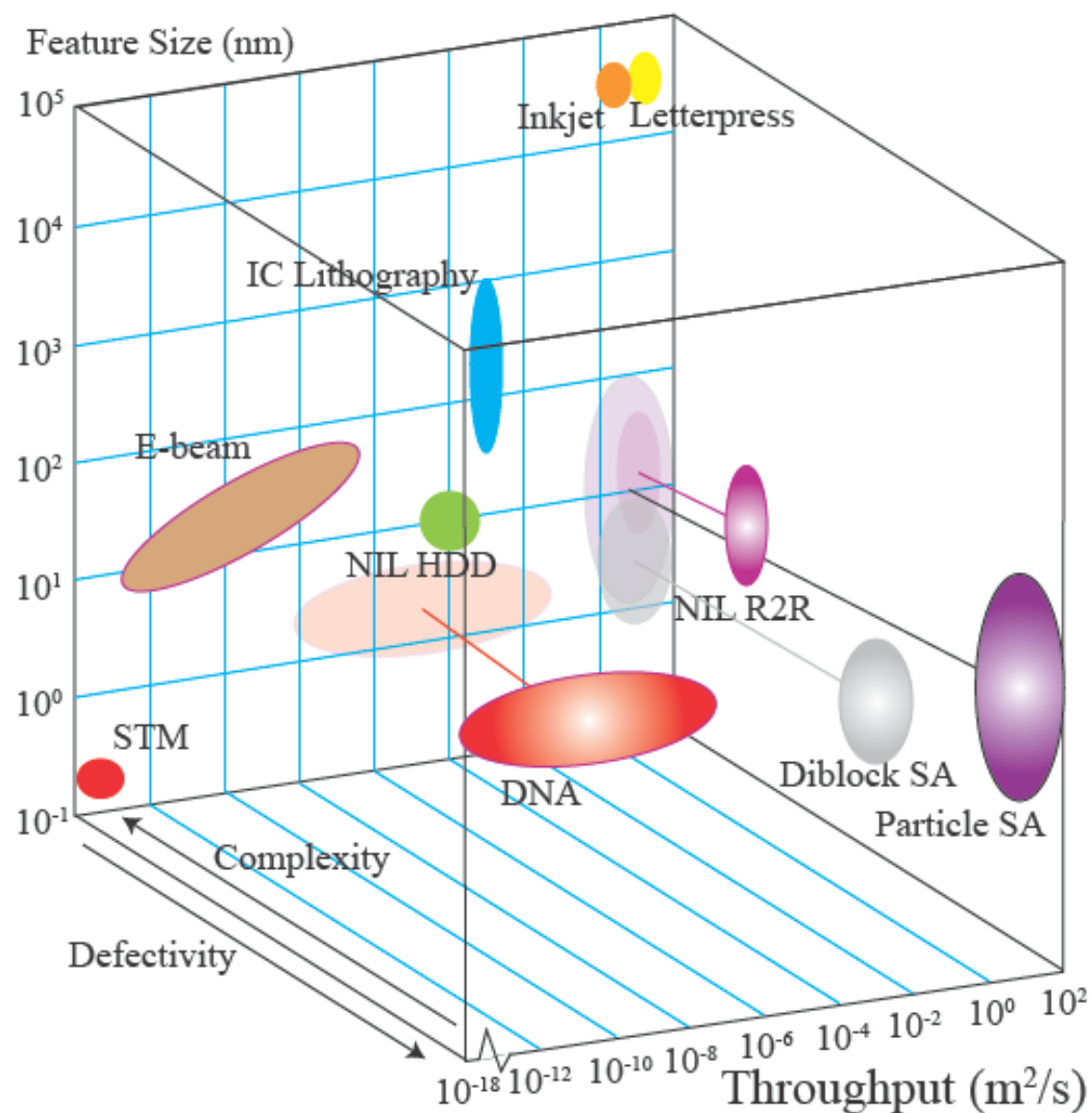
How can measurement help?

- Measurements for fundamental understanding
- Measurements for process/quality control

[illegible]

The Cost of Complexity



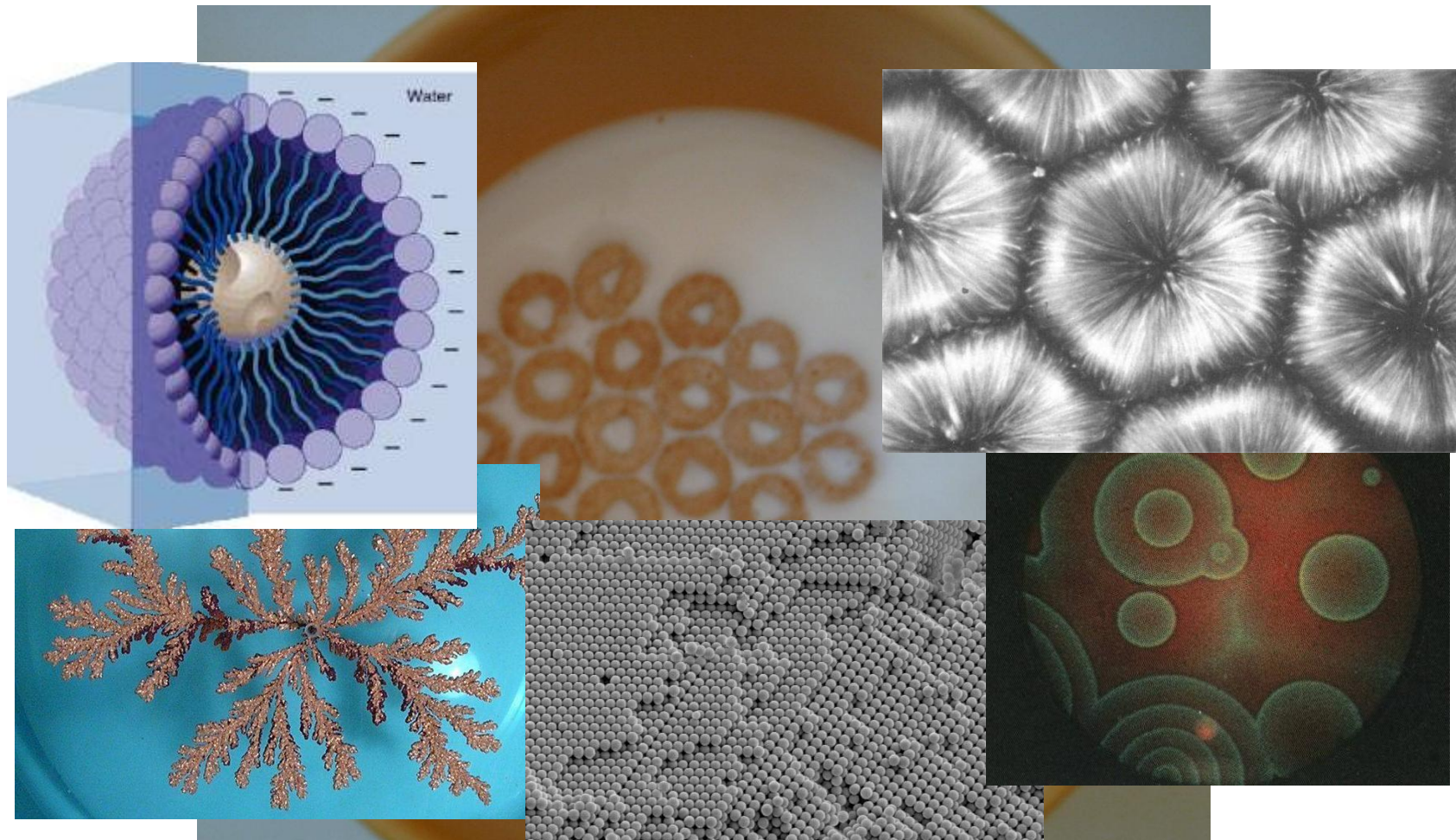




What measurements are needed?

- Measurements for fundamental understanding
 - Slow, expensive, infrequent
 - New measurements needed for novel materials/devices fabrication processes
- Measurements for process/quality control
 - Fast, cheap, periodic or continuous
 - Off-line
 - Real-time

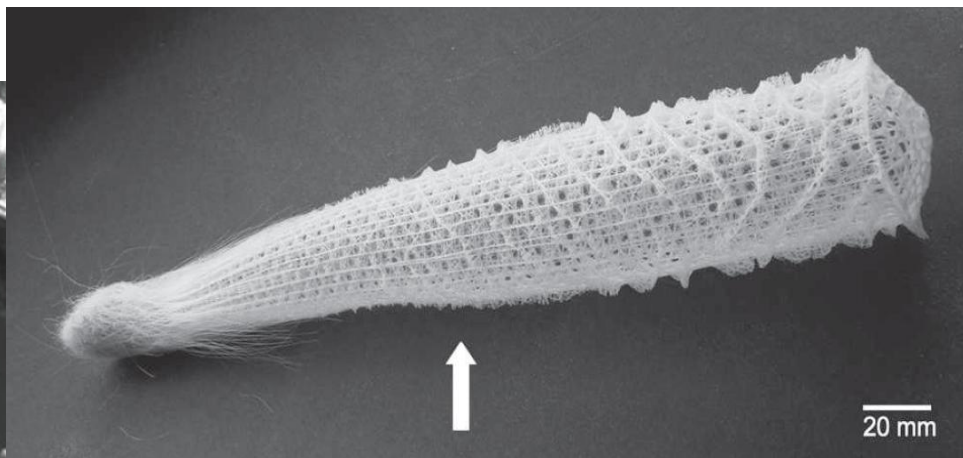
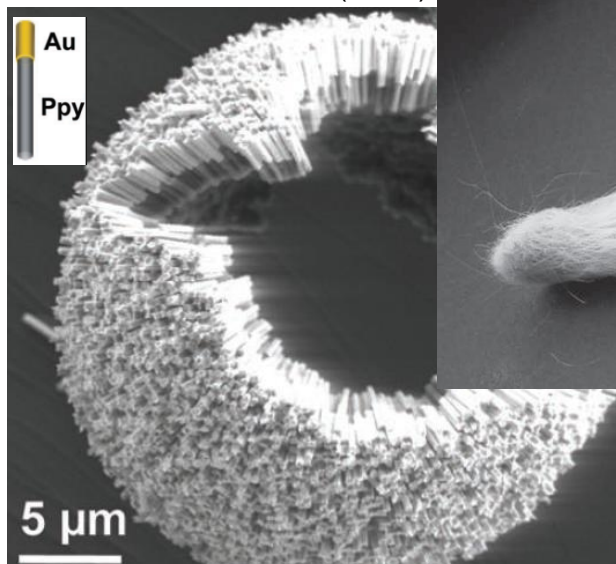
Self Assembly Everywhere



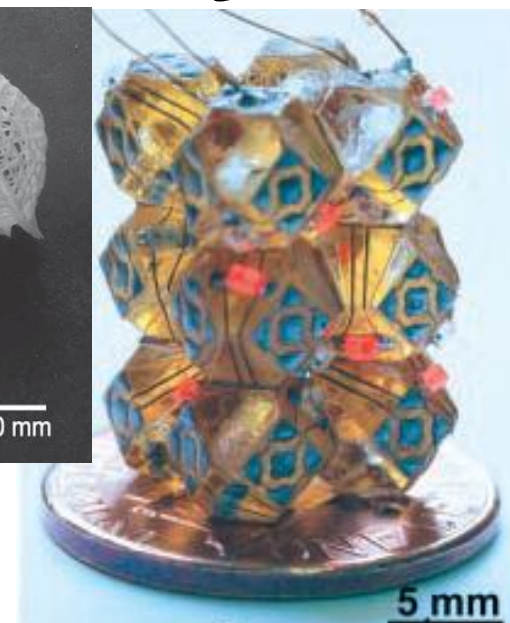
Self-Assembly at All Scales, G.M. Whitesides and B. Grzybowski, *Science* (2002)

Directed Self-Assembly

Park et al., Science (2004)

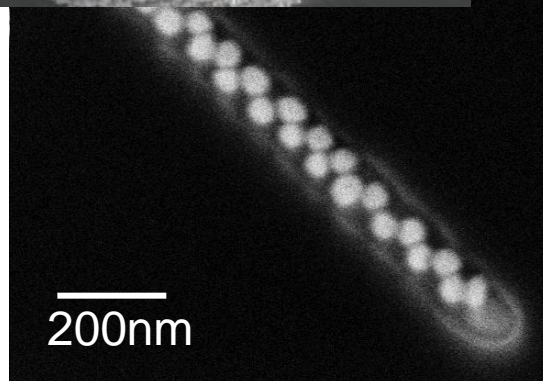


Woesz, J. Mat. Res. (2006)

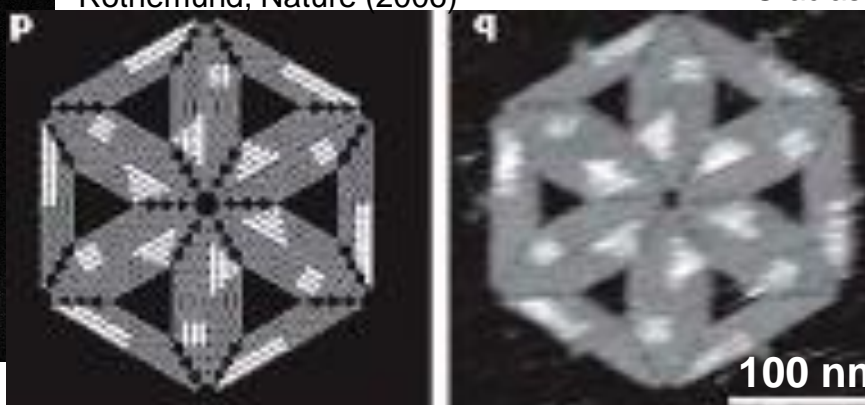


Gracias et al. Science (2000)

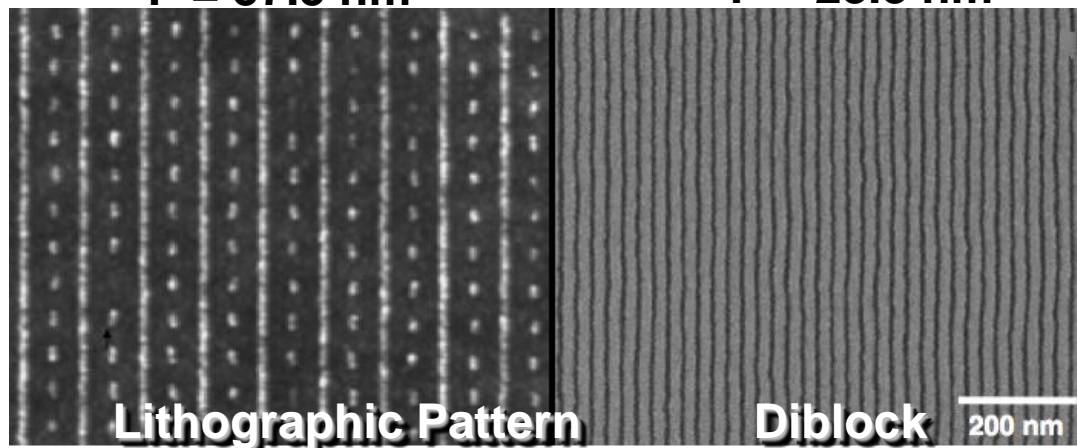
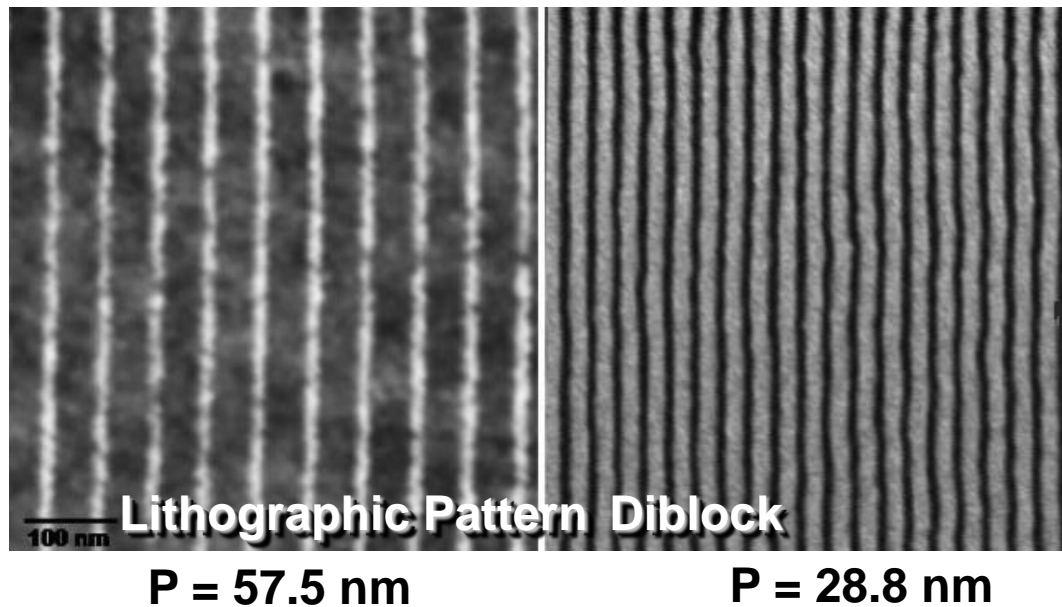
Rothemund, Nature (2006)



Cui et al. Nanoletters (2004)

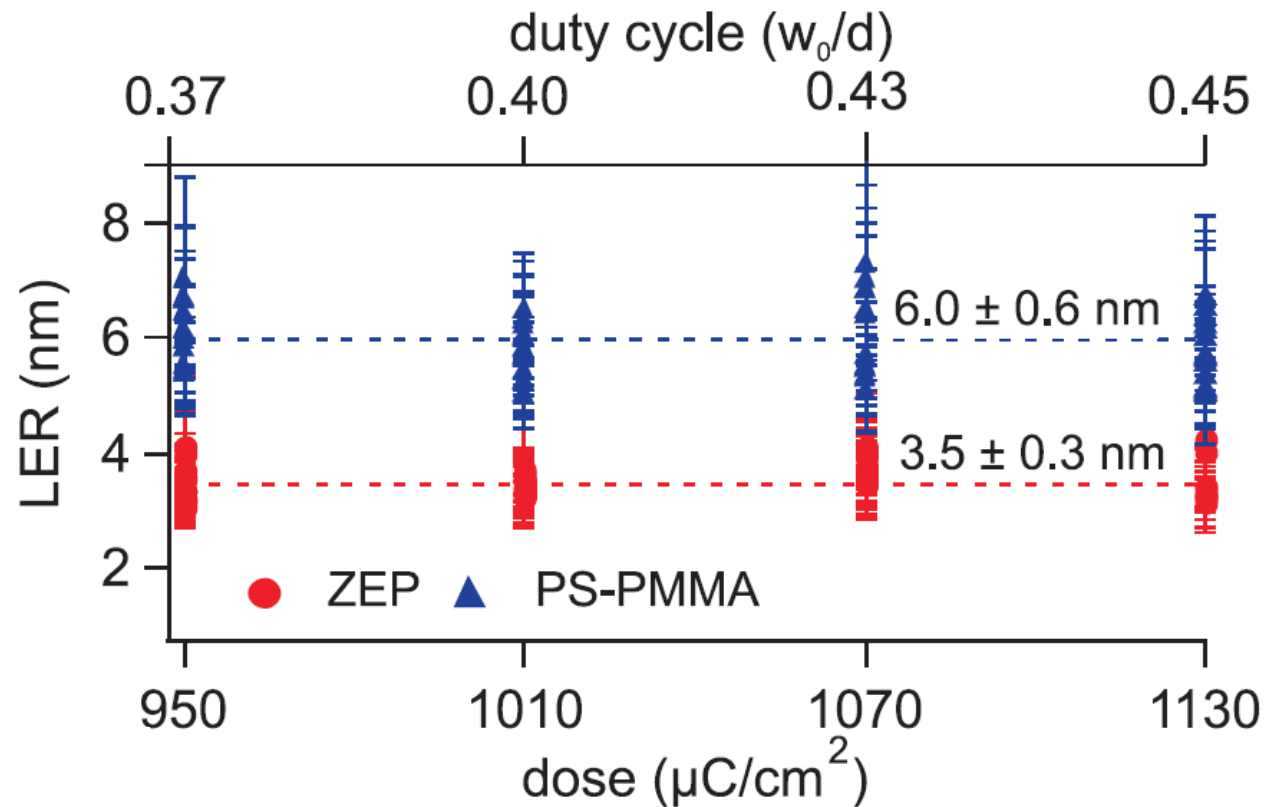
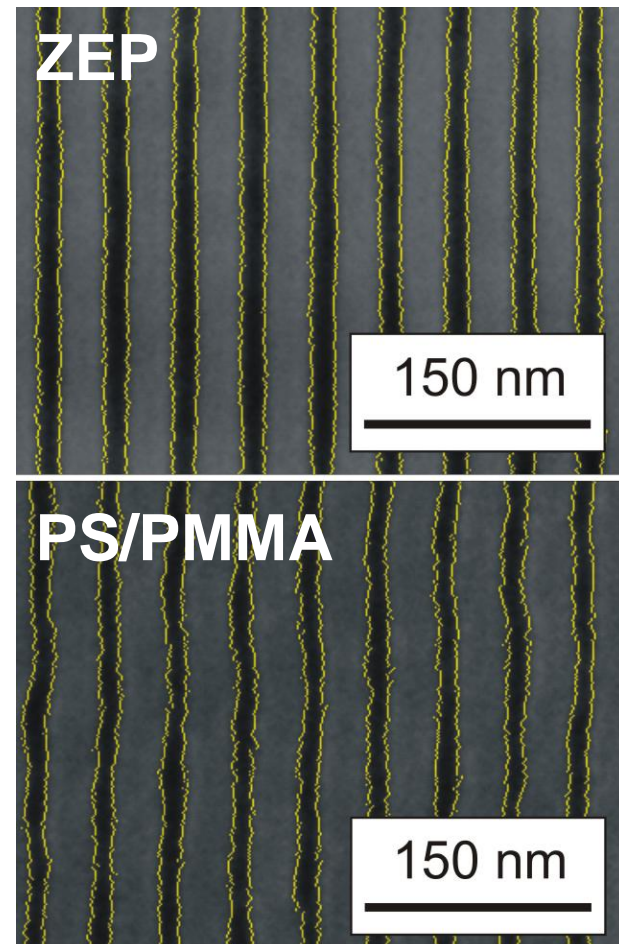


Sub-Lithographic Patterns



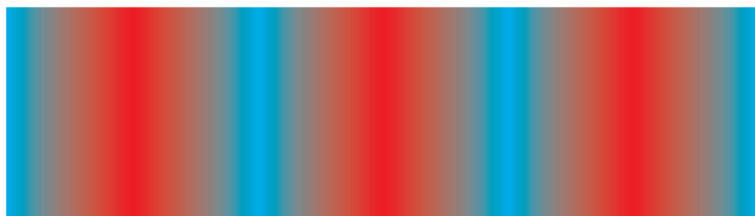
Dense Self-Assembly on Sparse Chemical Patterns: Rectifying and Multiplying Lithographic Patterns Using Block Copolymers, Joy Y. Cheng, Charles T. Rettner, Daniel P. Sanders, Ho-Cheol Kim, and William D. Hinsberg, Advanced Materials, (2008) - IBM

LER from SEM



Resonant X-ray Scattering

Are interfaces sharp, chemically diffuse or rough?



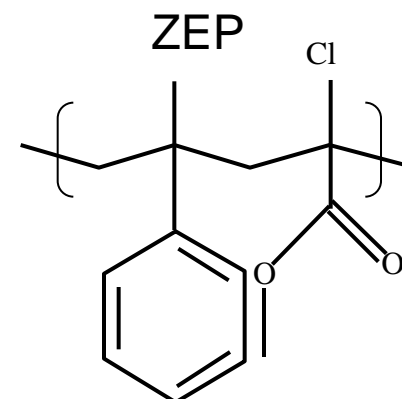
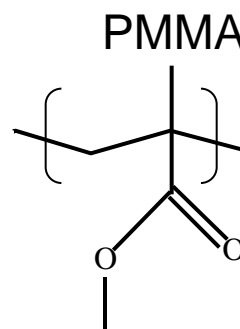
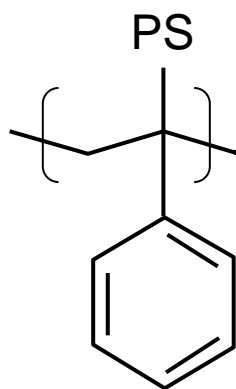
Virgili et al. Macromolecules (2007)

➤ X-ray scattering can measure interfacial width or roughness to sub-0.5 nm accuracy.

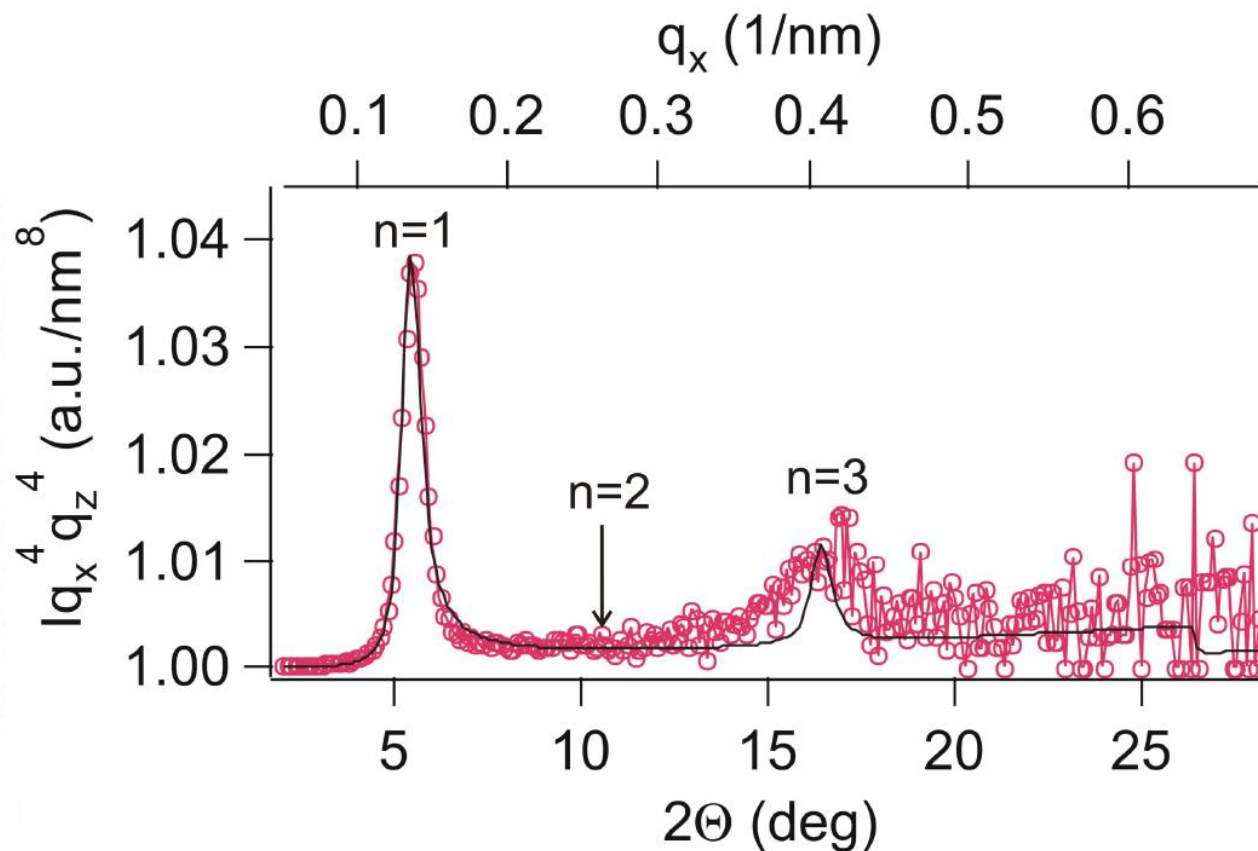
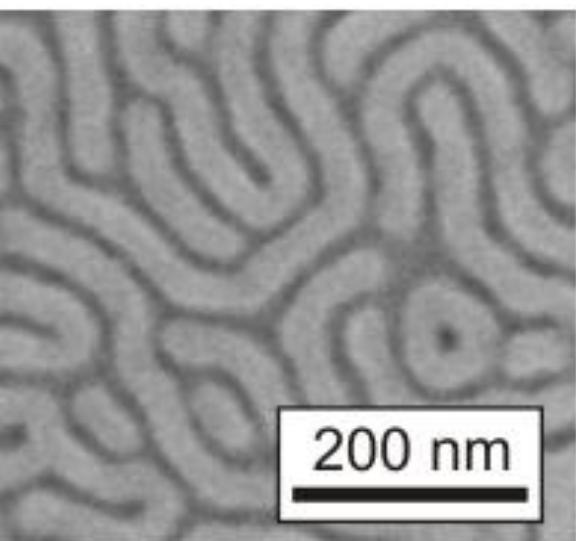
➤ Different chemistries have distinct resonances

➤ Resonant scattering enhances contrast from different chemical domains

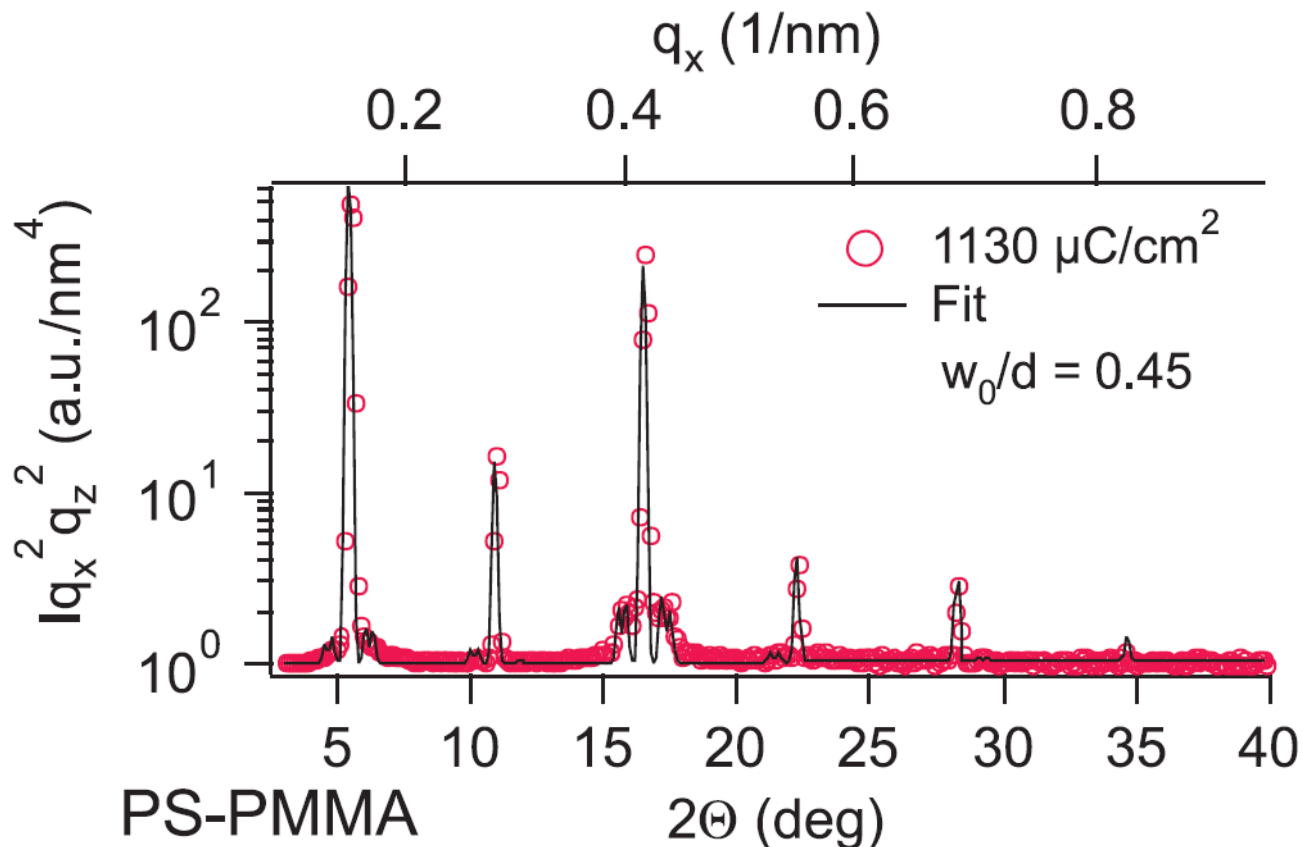
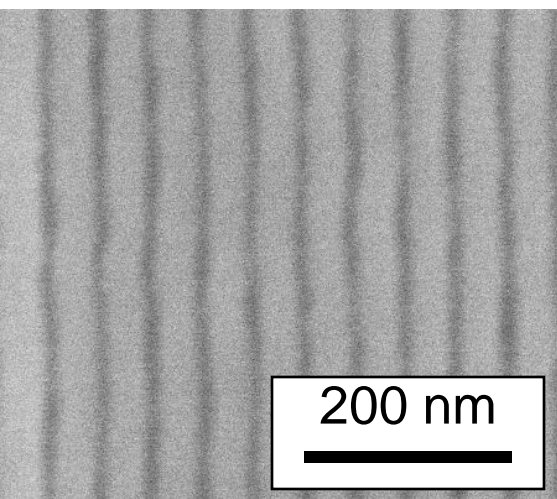
➤ C=C π^* 285 eV, C=O π^* 288 eV, C-O σ^* 293 eV



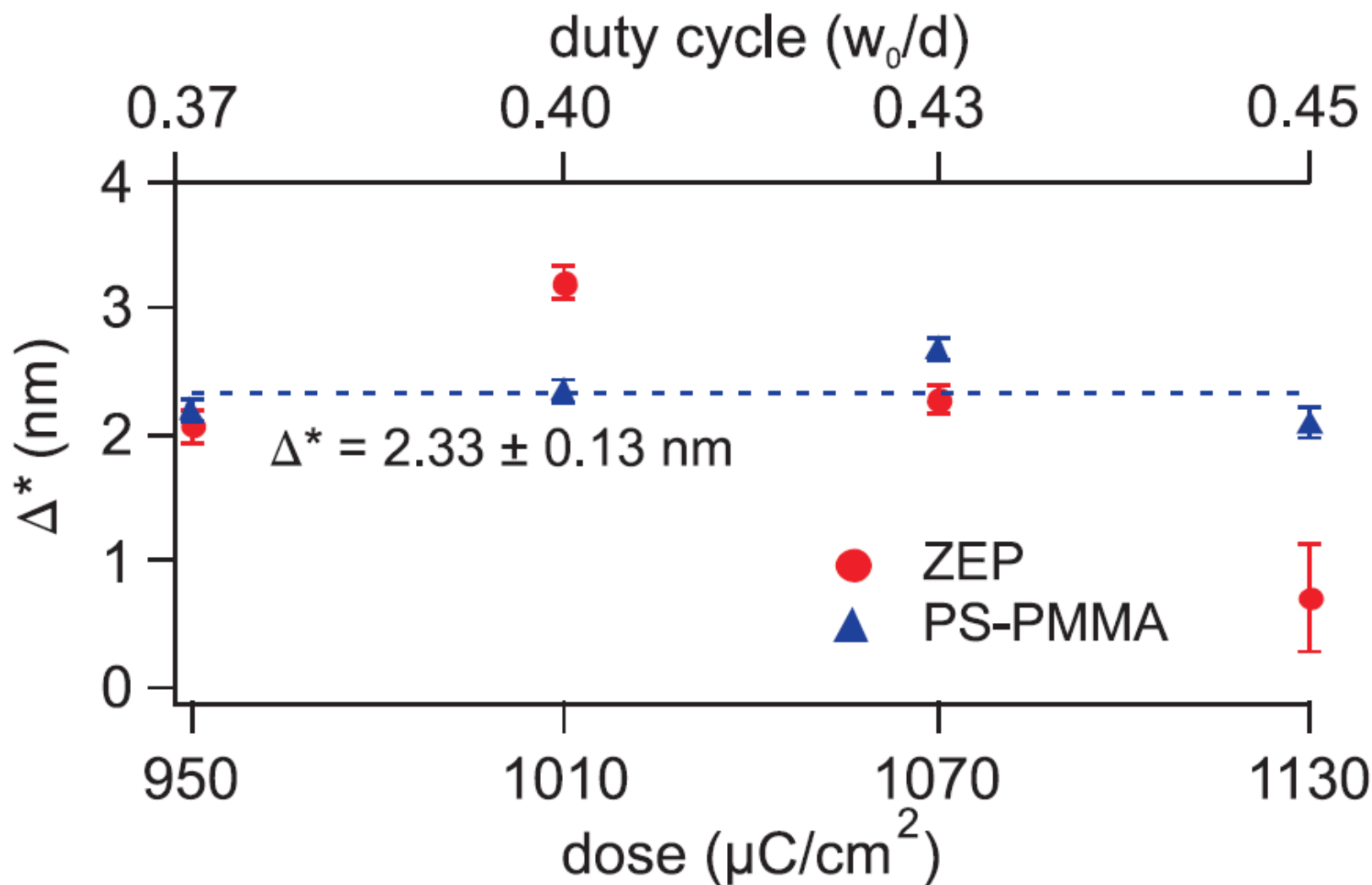
Random Diblock Diffraction



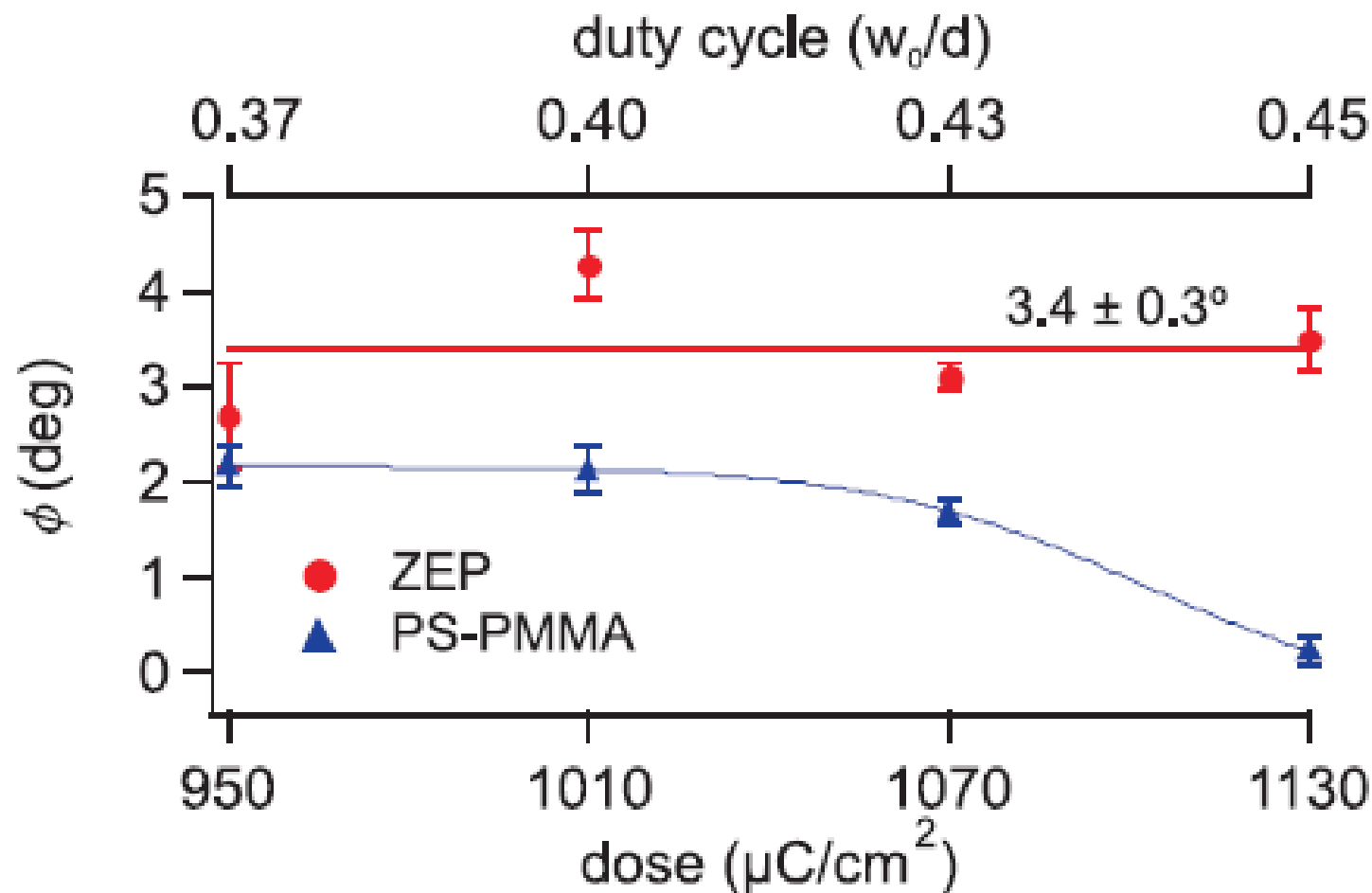
Epitaxial Diblock Diffraction



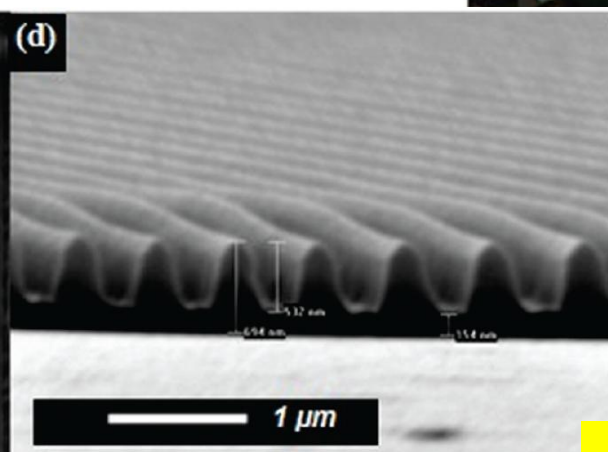
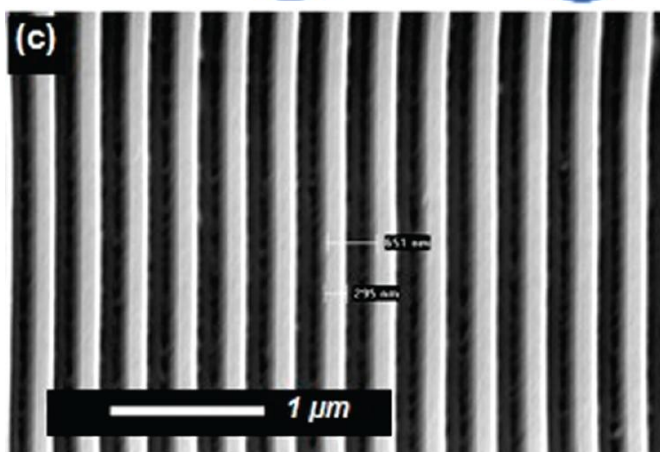
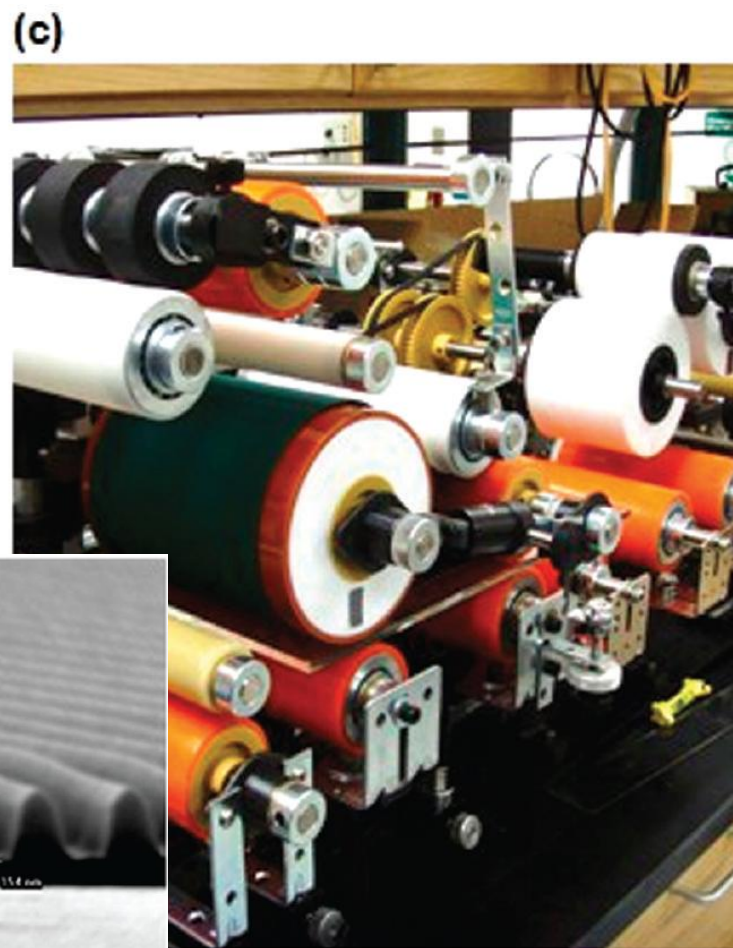
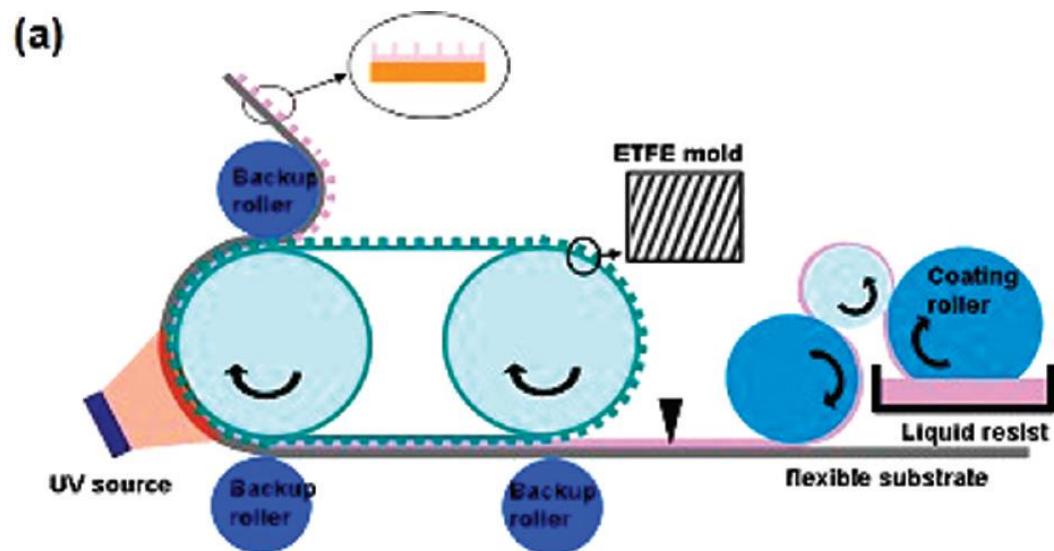
Interface Width from XRD



Sidewall Angle



Roll-to-Roll Imprint





Metrology Requirements for R2R Diblocks

- Determine short range and long range orientation, order, defectivity, thickness, ...
- Challenges:
 - Feature sizes down to a few nm
 - Substrate (“web”) speeds up to meters/second
 - Cost
- Techniques:
 - In-plane polarization analysis
 - Scattered light surface roughness measurement
 - Nano-plasmonic near-field sampling
 - Normal incidence interferometry
- All techniques will require a high level of model development to enable data extraction
- Low-cost/High-speed requirement → Measurement technique must be specific to the type of pattern being measured

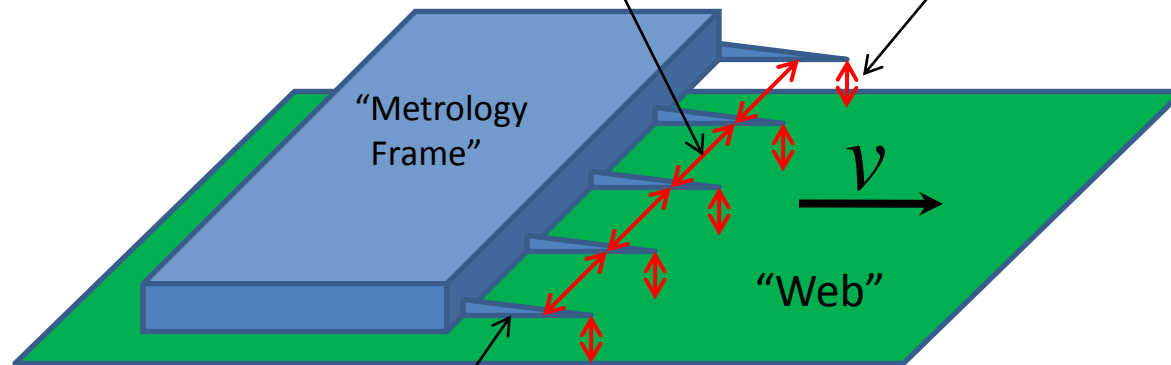


High-Speed Near-field Measurement

- Metrology frame contains “laser gauges”
 - Know spacing between read heads to ~nm
- Read heads operate as in disk drive
 - “fly” above web surface: Height ~ several nm
 - Move up and down: Track surface undulations
- Read heads are “functionalized” with a near field imaging system specific to the pattern being measured
 - ...NSOM, guided modes, plasmonic, interferometric, polarization ...
- Near field sensors give local nm scale resolution
 - Sense Local Order
- Laser gauges allow for correlation of signals from separate sensors
 - Compute Long Range Order

“Laser Gauges” track the relative position of each sensor arm to nm precision
→ Determine long range order.

Each sensor head is functionalized to measure precisely the type of pattern(s) being generated on the “web”
→ Determine short range order

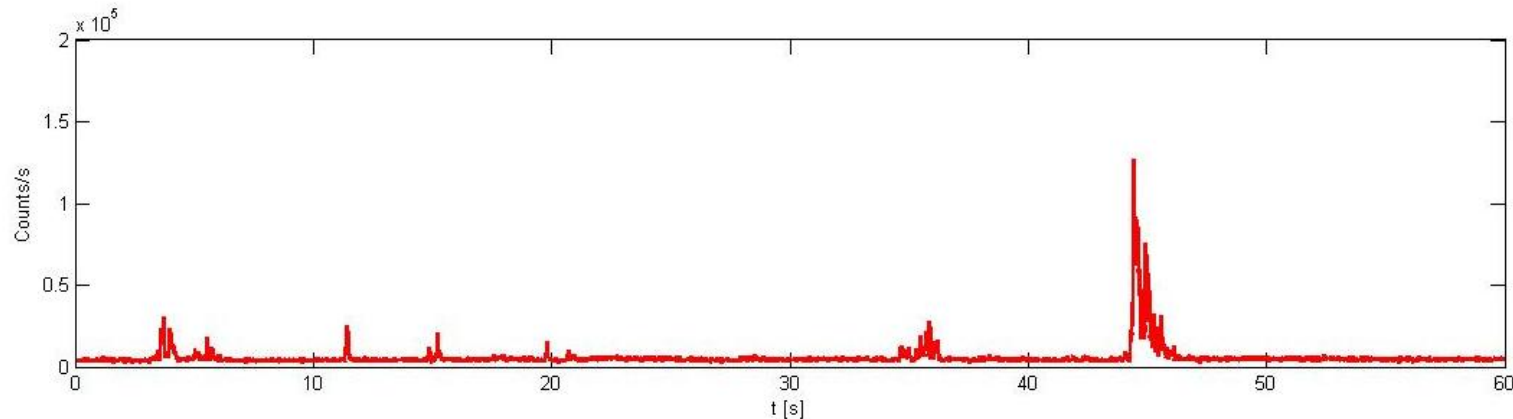


“Flying Height Sensor” arms from hard disk read heads.
• “Float” a few nm above the “web”
• Automatically (aerodynamically) move up and down to track vertical deviations in the “web” top surface position



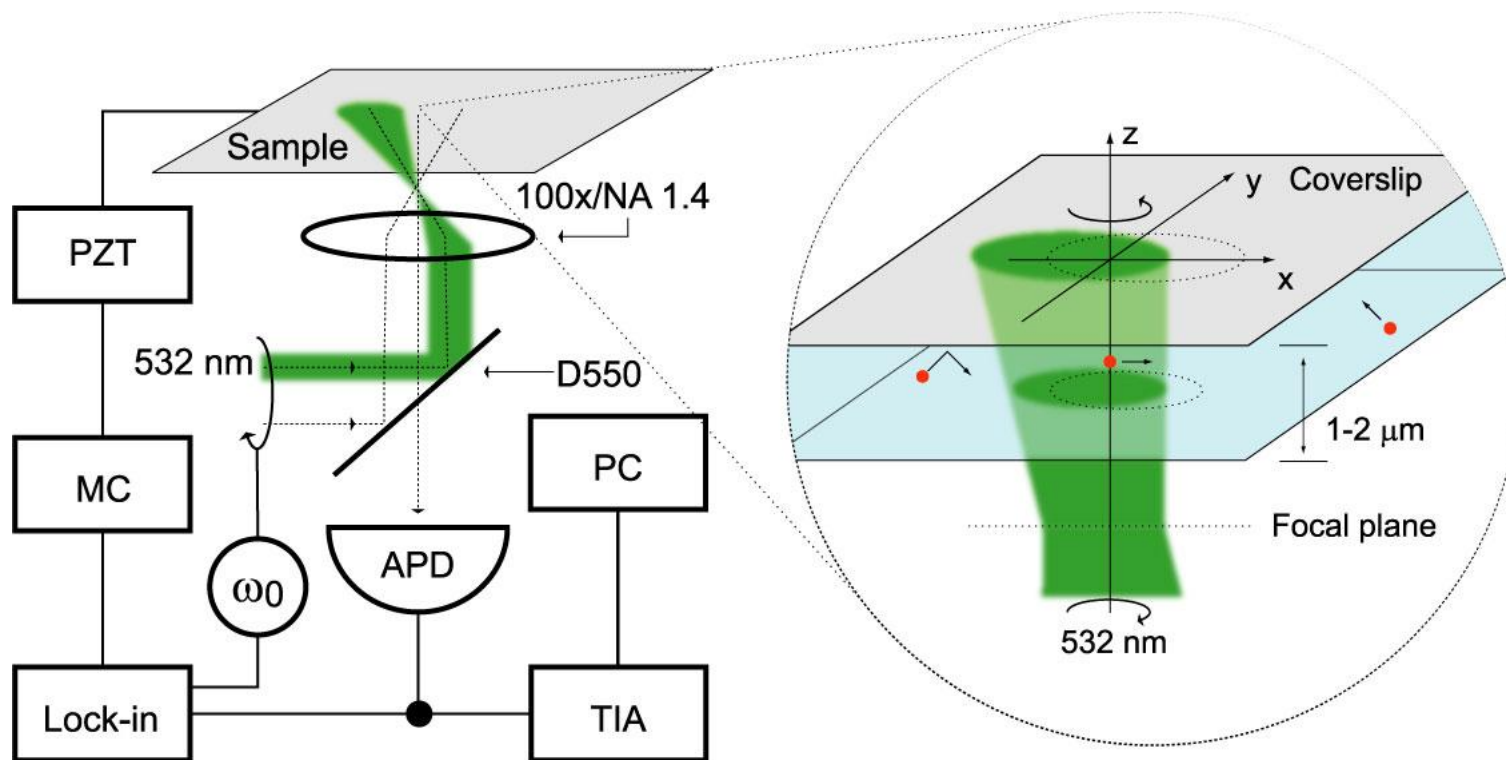
Single-Molecule/Nanoparticle Behavior

- Brownian motion limits the dwell time of single molecules in a diffraction-limited microscope observation volume
- **Tracking-FCS** uses feedback control to combine the spatial resolution of single-particle tracking with the temporal resolution of fluorescence correlation spectroscopy, all with a single molecule or nanoparticle
- The method is sensitive to rotational motion, conformational changes, binding/unbinding – anything that affects fluorescence intensity



- In open-loop configuration, 60-nm diameter particles diffuse across the beam in 100 ms – 300 ms

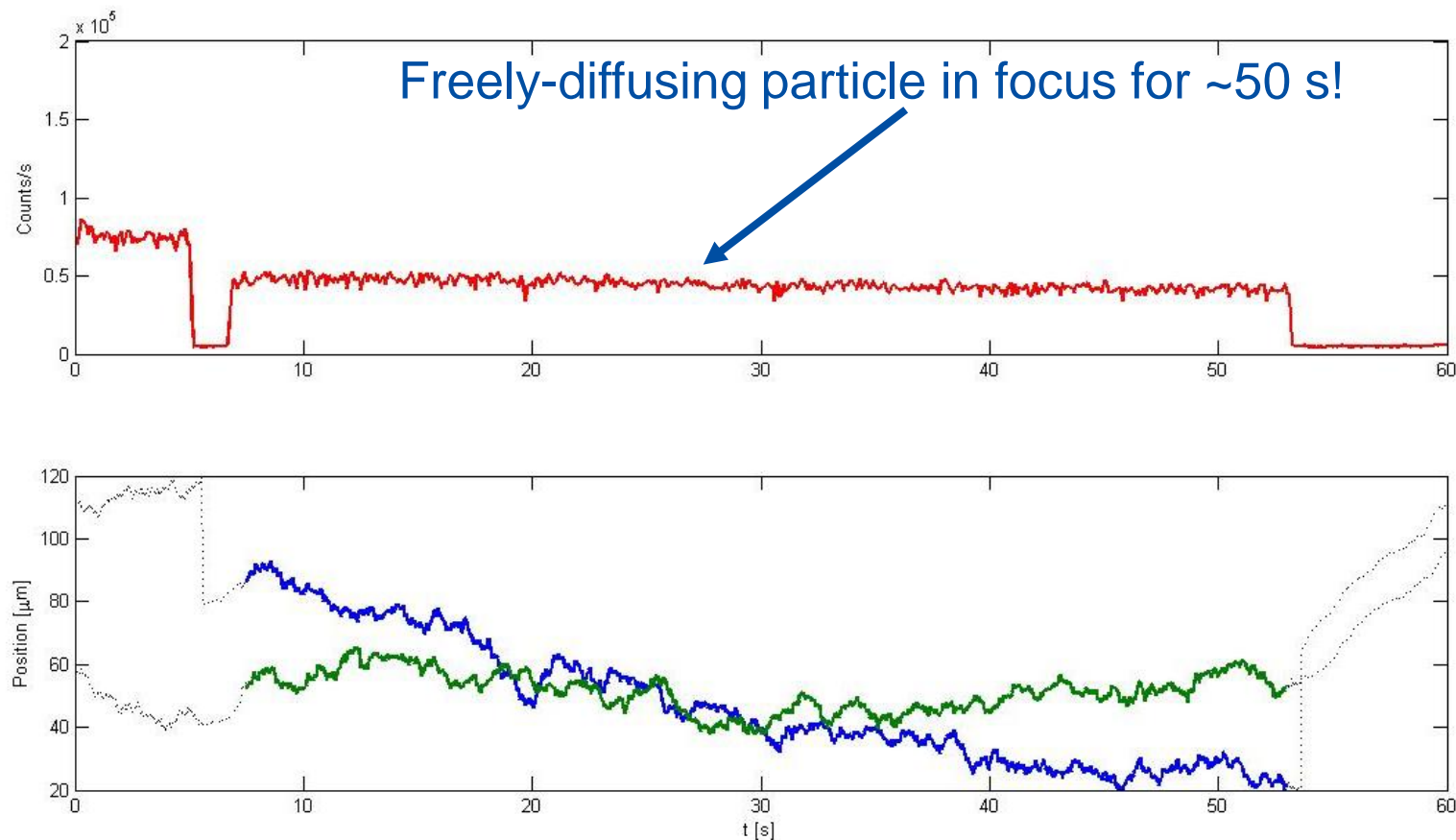
Controlling Brownian Motion



- A. J. Berglund and H. Mabuchi, "Tracking-FCS: Fluorescence correlation spectroscopy of individual particle," *Optics Express* **13**, 8069-8082 (2005)
 A. J. Berglund, K. McHale and H. Mabuchi, "Fluctuations in closed-loop fluorescent particle tracking," *Optics Express* **15**, 7752-7773 (2007)
 K. McHale, A. J. Berglund, and H. Mabuchi, "Quantum dot photon statistics measured by three-dimensional particle tracking," *Nano Letters* **7**, 3535-3539 (2007)

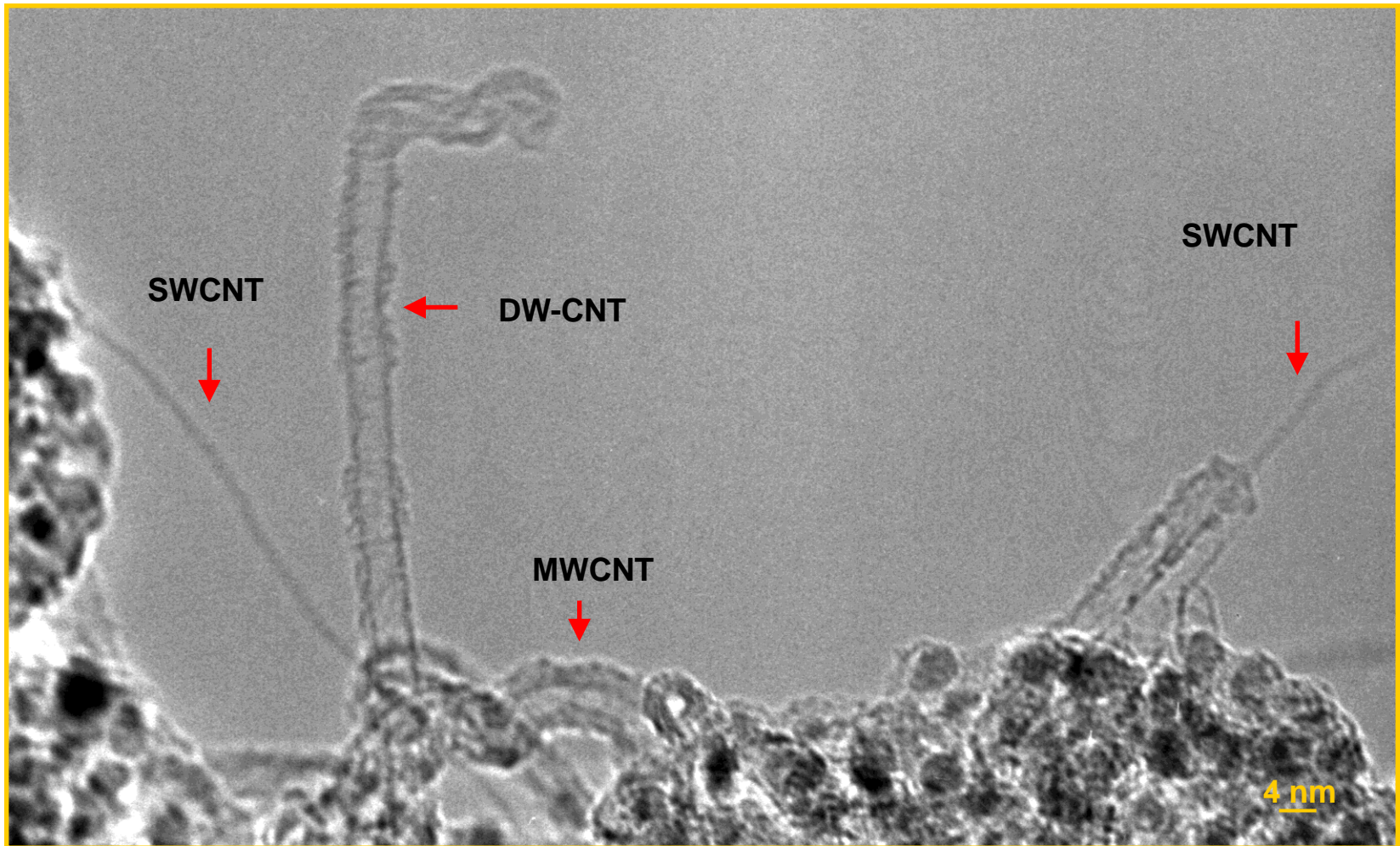
- Rotating the laser focus encodes a single particle's position
- Translation of the sample locks a particle to the observation volume

Controlling Brownian Motion



- In closed-loop mode, single-photon fluorescence signal (top) and particle position (bottom) are collected simultaneously for a single particle

Morphological Diversity in CNTs

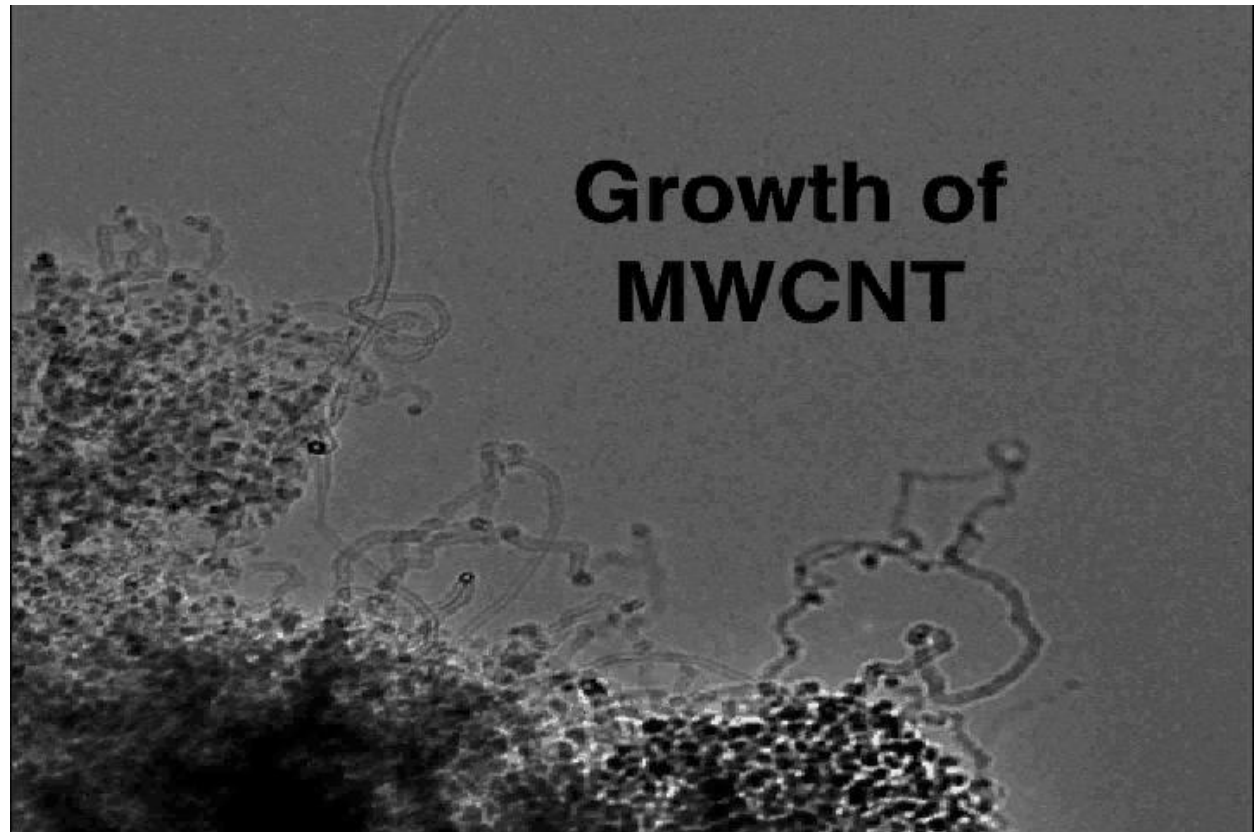


Growth at 500°C in 300 mTorr of C_2H_2

Factors Controlling the Catalytic CVD Synthesis of CNTs

- Catalyst: Ni, Cu, Co, Fe, Mo and bimetallic catalysts
- Support: SiO_2 , MgO , TiO_2
- Temperature: 500°C – 1000°C (400°C - 700°C)
- Precursor: Hydrocarbons (CO , CH_4 , C_2H_4 , C_2H_2 etc.)
- Pressure: 760 Torr (1 - 300 mTorr)

Environmental TEM

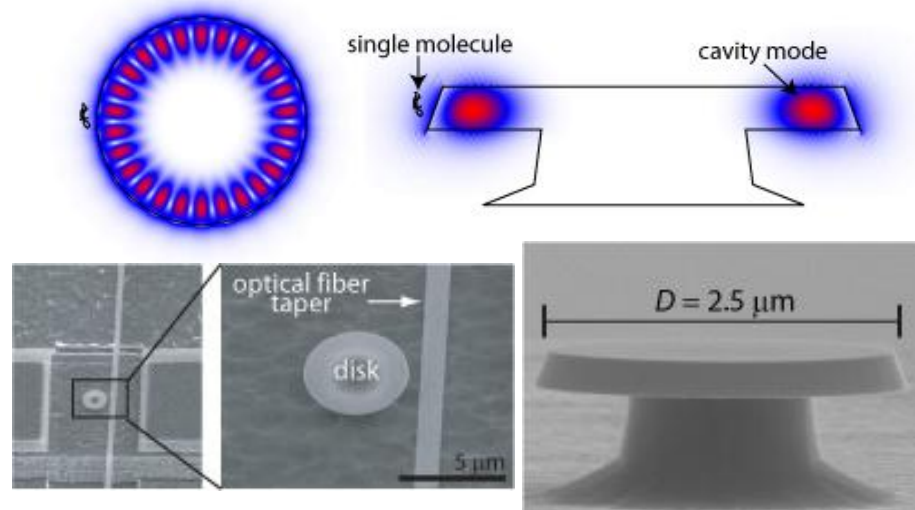


**THANK
YOU!**

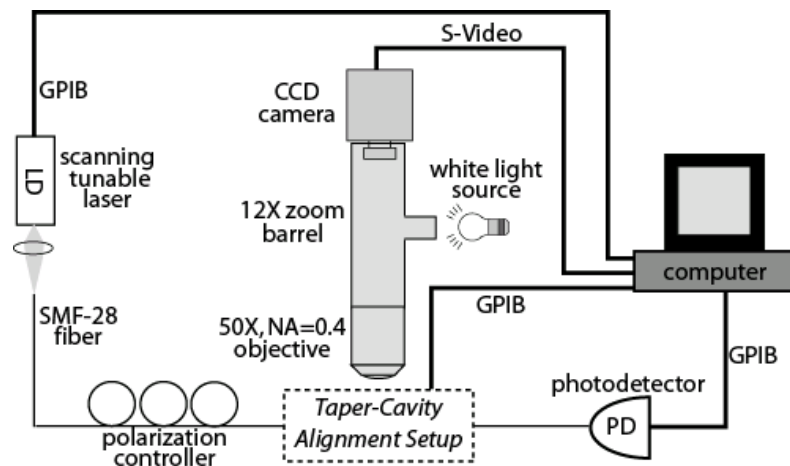
Nanophotonic cavities for single molecule detection

- **Label-free, single molecule sensitivity due to:**
 - High quality factors (long photon interaction time)
 - Wavelength-scale light confinement (strong light-matter interaction)

- First demonstration: Armani et al, *Science*, 2006 – Single molecule detection of interleukin-2 in a 80 nm diameter, toroidal glass microcavity



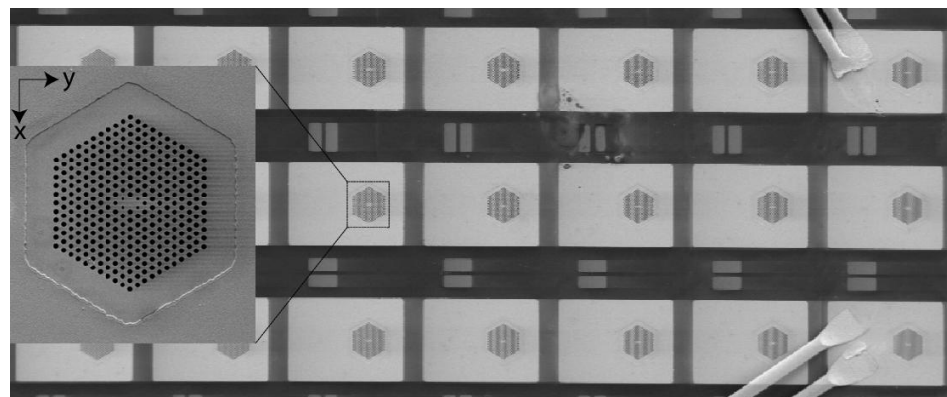
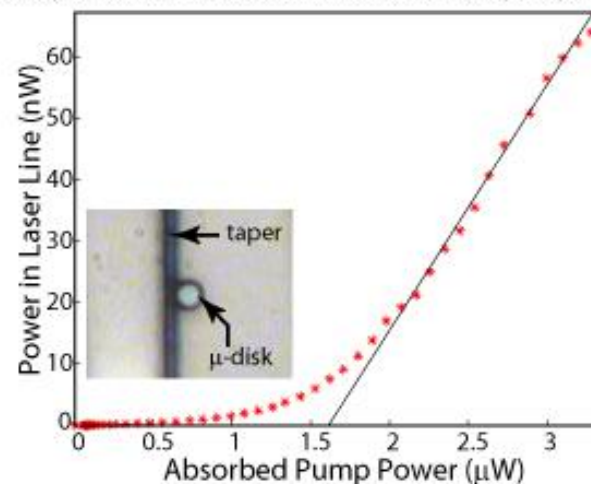
(K. Srinivasan and O. Painter, *Nature*, 2007)



Nanophotonic light sources for chip-level spectroscopy

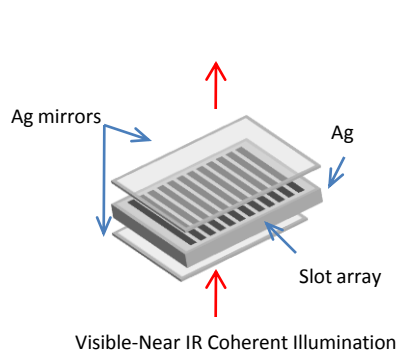
- **Nanofabricate optical resonators in light-emitting media to create on-chip lasers and LEDs for spectroscopy applications**
 - 2D arrays of devices on a chip
 - Tune resonator geometry across array for multi-wavelength device
 - Optimized device geometries for in-plane or vertical emission, depending on application
 - Can be integrated with microfluidics for sample delivery to sensing region

Fiber-coupled microdisk lasers (K. Srinivasan et al, *Opt. Express*, 2006)

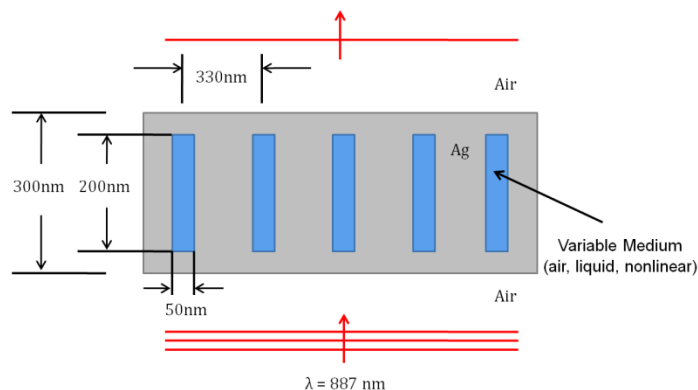


Nanoplasmonic Optical Resonators For Biosensing

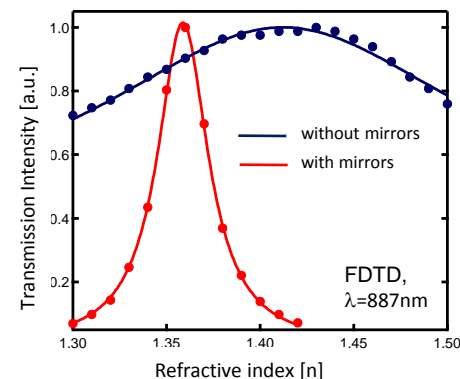
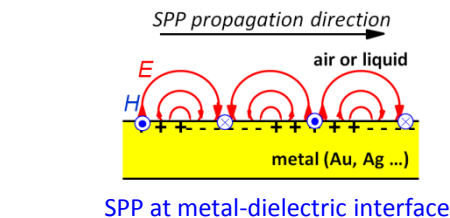
- Plasmons confine light to nanoscale dimensions
- Array of slots gives high-Q resonator
- Transmission very sensitive to index in slots



a) Device Perspective

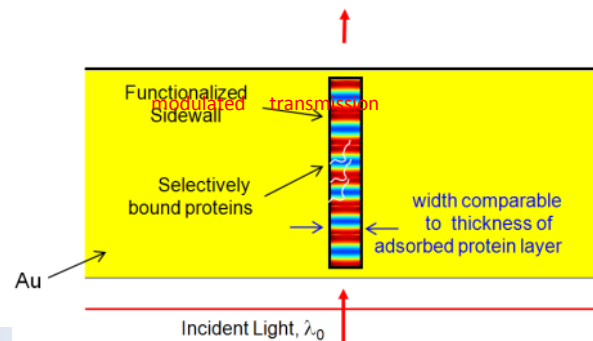


b) Device Cross Section



c) Simulated transmission v.s. index of cavity medium

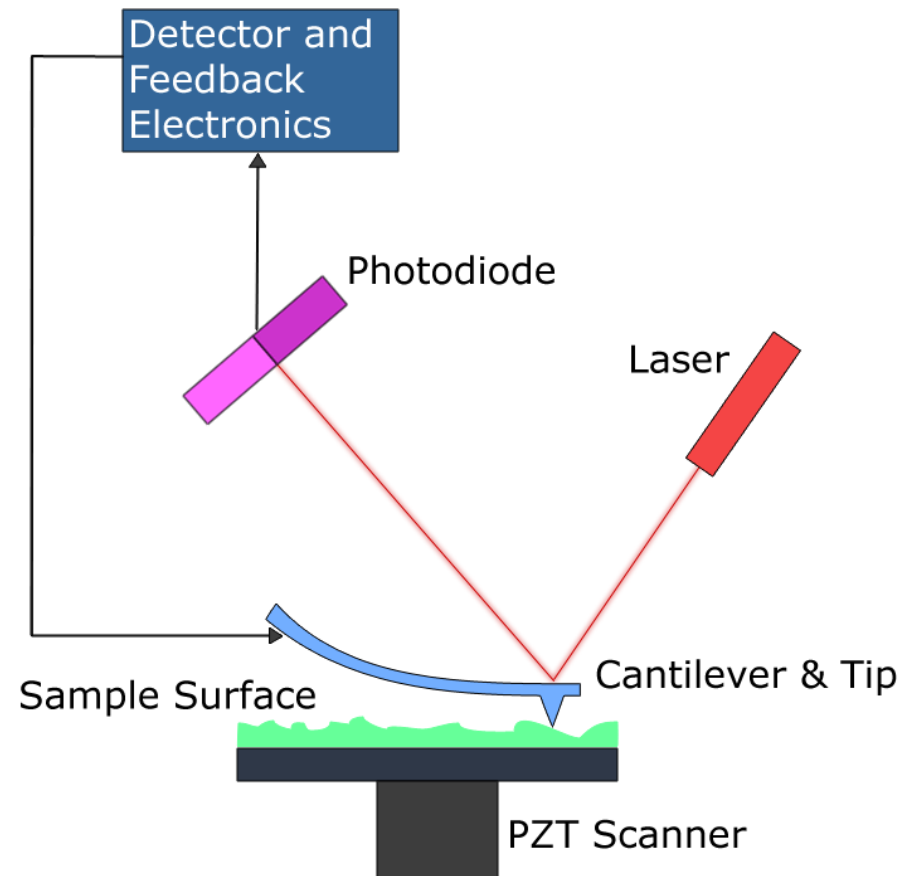
- Nanoscale volume and sensitivity can yield label-free sensing of molecules selectively bound to cavity walls



Pacifici et al., Nature Photonics (2007)

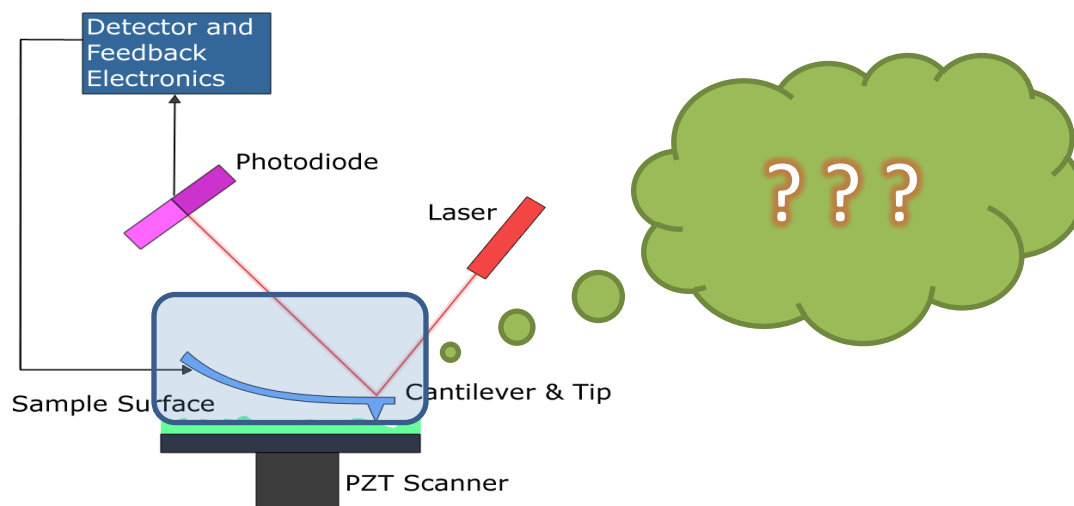
Atomic Force Microscopy

- Proven utility for Nanoscience
- Optical readout works well
- Free space optics:
 - cm-scale beam path
 - stray light
- Bulk external actuators
- Stability, vibration issues
- Precision is typically limited by optical readout noise
 - off-resonance
 - in low to modest Q situations (air, liquid)
- Bandwidth is limited by cantilever and readout



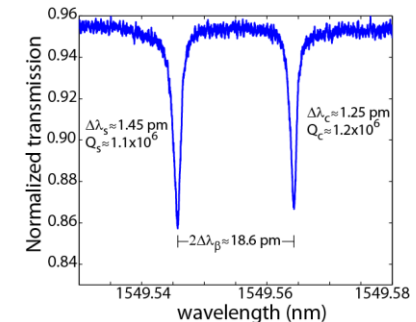
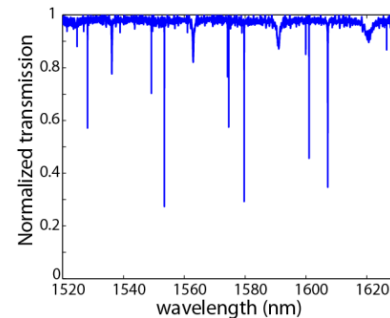
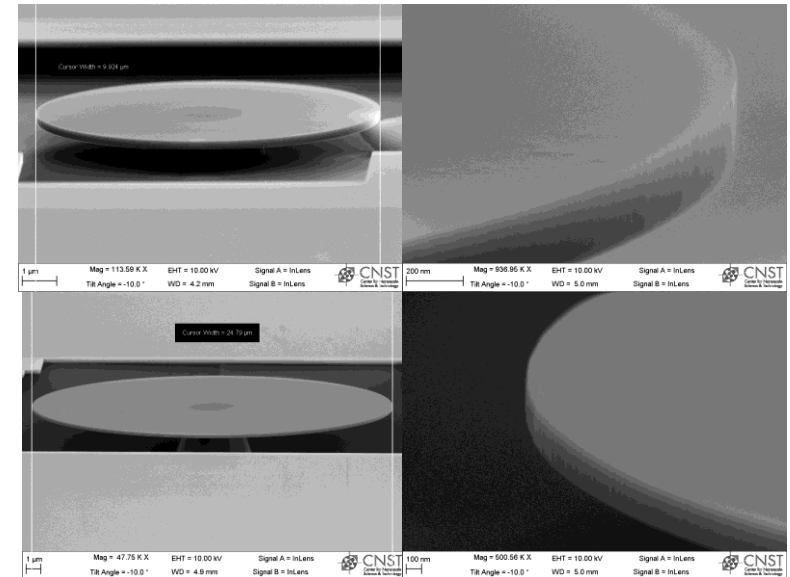
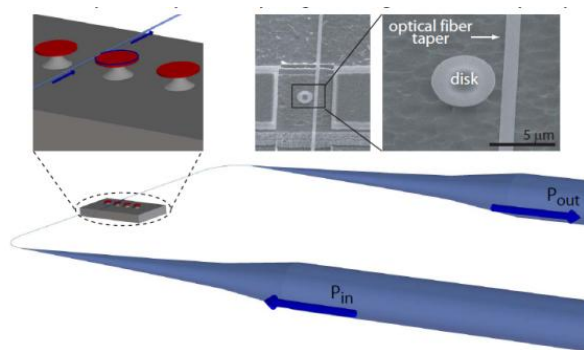
Motivation

- Integrated Optical Sensing of Mechanical Motion
 - Optical Interferometric Readout
 - High Q and High Bandwidth Mechanical Probe
 - Internal MEMS Actuation
 - Fiber Based Excitation and Readout
 - Applications in Real Life Problems i.e. AFM



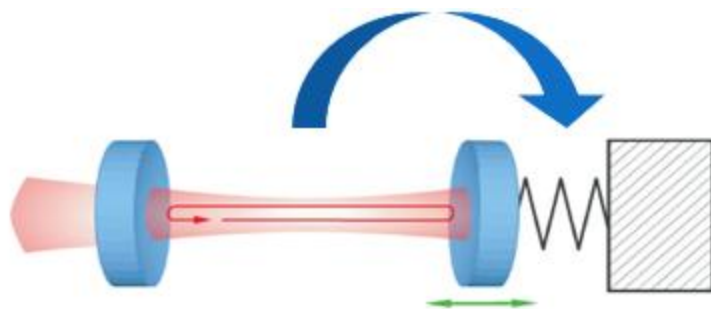
Nanophotonic Resonators

- Microtoroid ($Q \sim 10^8$)
- Microdisk ($Q \sim 10^6$)
 - 10 μm diameter Si disk $Q = 1.2 \times 10^6$
 - 25 μm diameter SiN disk $Q = 1.7 \times 10^6$
- Microring ($Q \sim 10^6$)
 - 60 μm diameter SiN ring $Q = 1 \times 10^6$
- Photonic Crystal ($Q \sim 10^5 - 10^6$)
 - L3 Cavity $Q = 5 \times 10^4$
 - MH Cavity $Q = 2.9 \times 10^5$

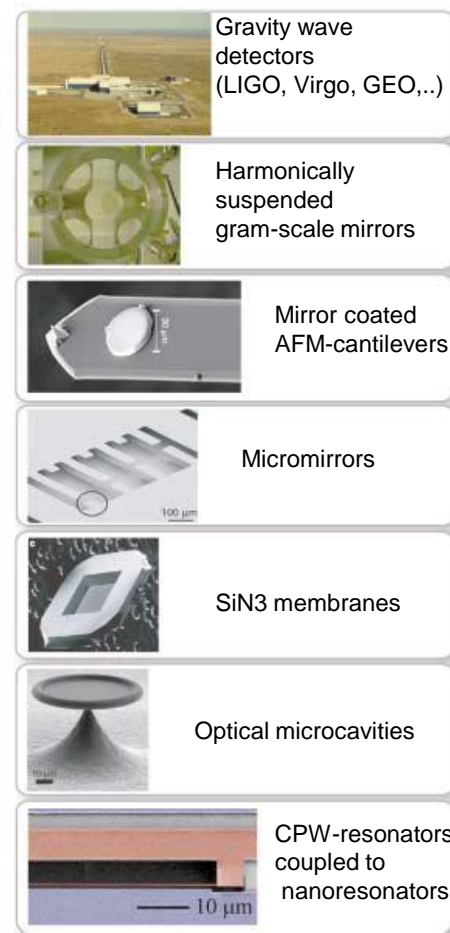


Cavity Optomechanics

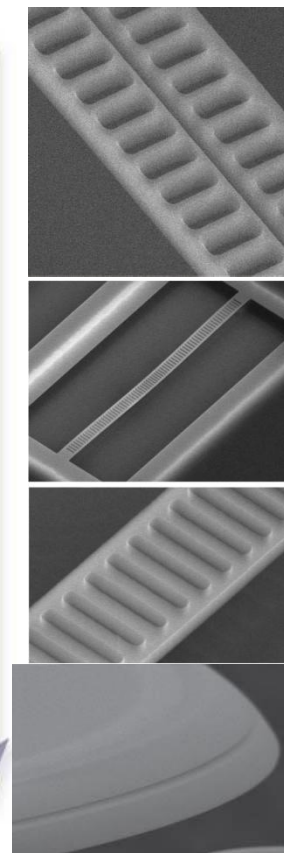
- **Optical shot noise limited mechanical measurements with noise floors of order 10^{-18} m/Hz^{1/2}.**
- **Opto-mechanical cooling reduces thermal noise floor by orders of magnitude.**
- **Optically excited regenerative oscillation for precise mechanical frequency shift measurements.**



Coupling of high Q mechanical and high Q optical modes



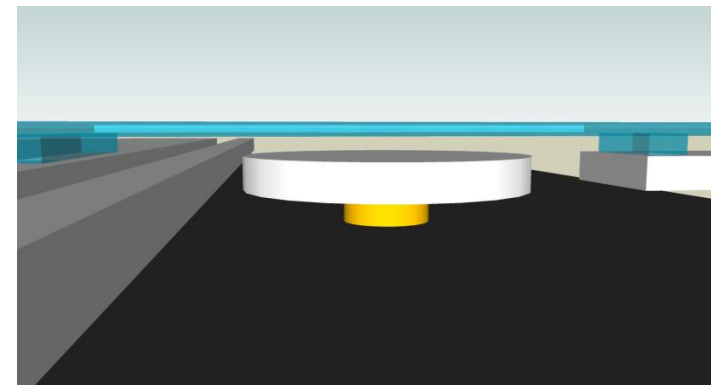
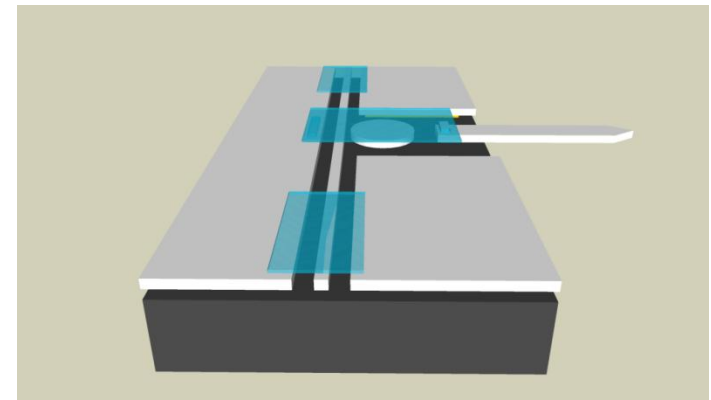
T. J. Kippenberg, Science (2008)



O. Painter's group

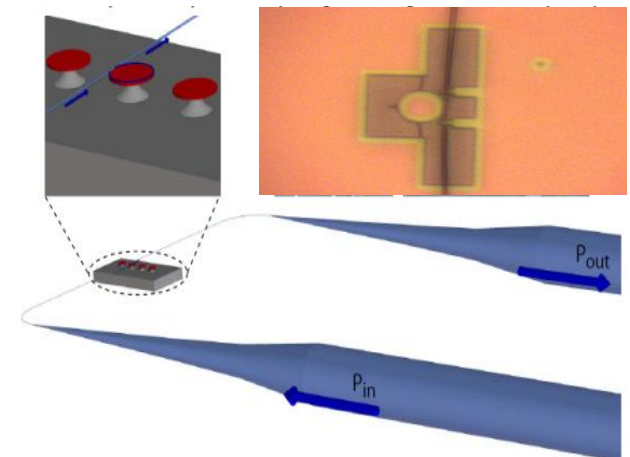
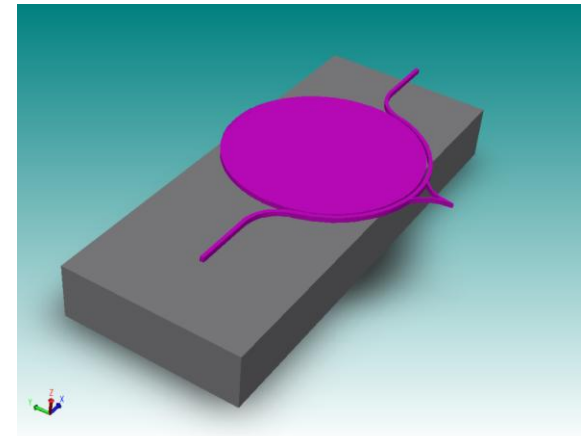
Integrated MEMS Tunable High Q Optical Cavity

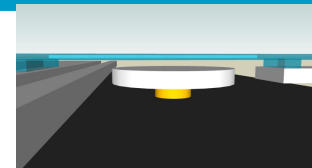
- MEMS Actuated Mechanical Resonator
 - Single crystal Si, Si nitride
 - Electrostatic actuation for
 - Tuning
 - Positioning
- High Finesse Interferometry
 - Si Microdisk resonator ($Q > 1M$)
 - Integrated optical excitation
- Evanescent Field Optical Coupling
- Independent Optimization of Optical and Mechanical Components



Simplified In-plane Opto-Mechanical Probe

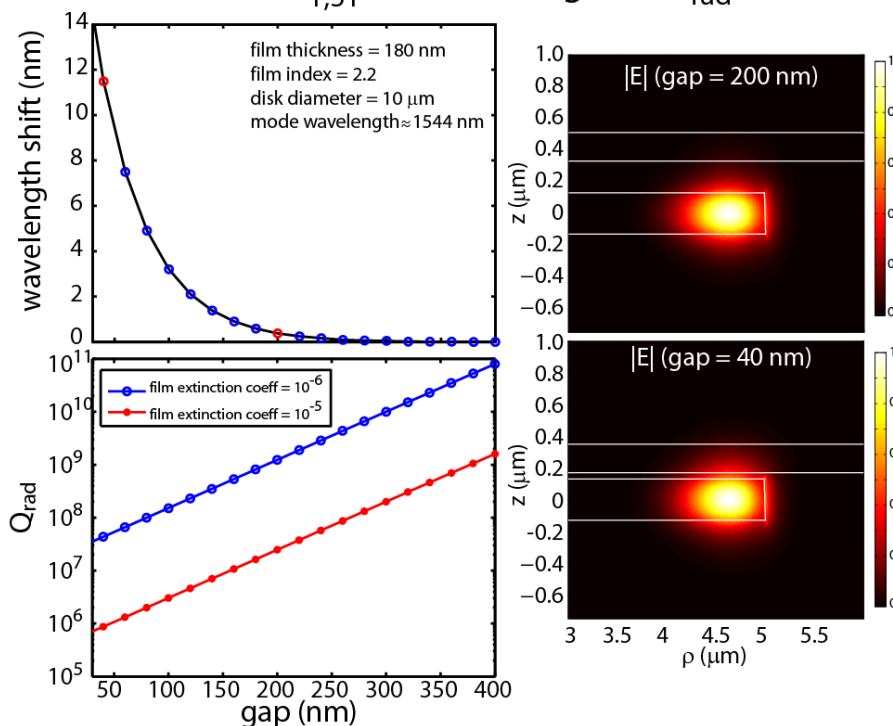
- Mechanical Resonator
 - Single crystal Si
 - Small, high bandwidth
- High Finesse Interferometry
 - Si Microdisk resonator
 - External excitation (tapered fiber)
- Evanescent Field Optical Coupling



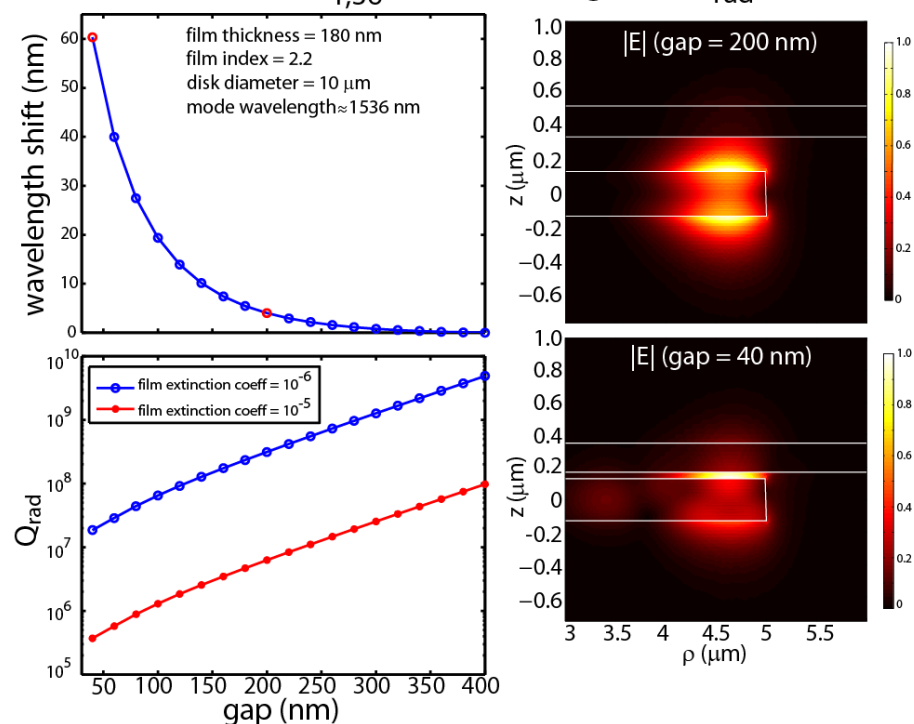


FEM Simulation

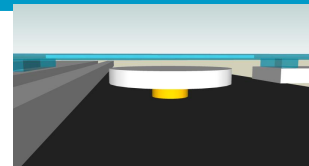
TE_{1,51} mode tuning and Q_{rad}



TM_{1,36} mode tuning and Q_{rad}



Device Design, Fabrication and Mechanical Testing



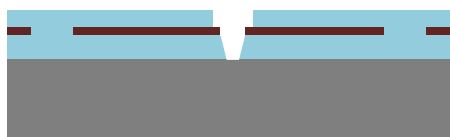
SOI: 260 nm Si, 1 μm Oxide



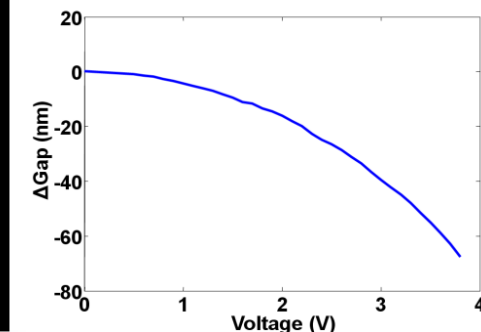
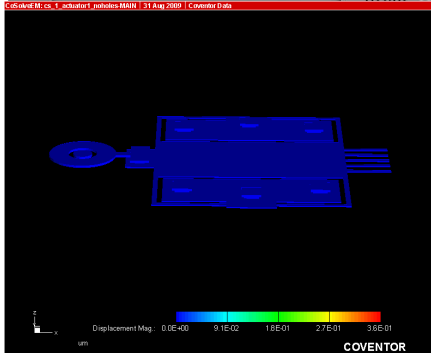
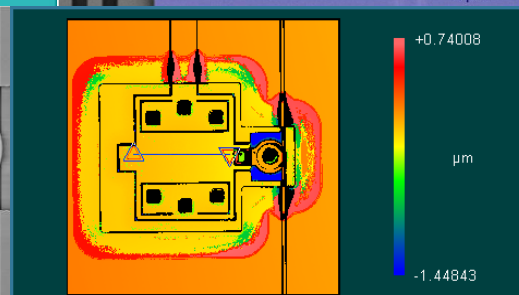
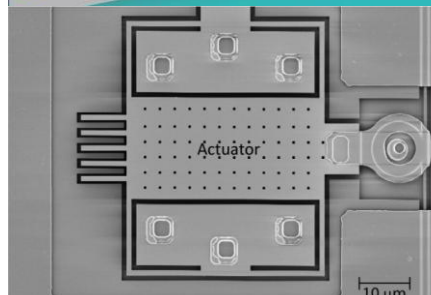
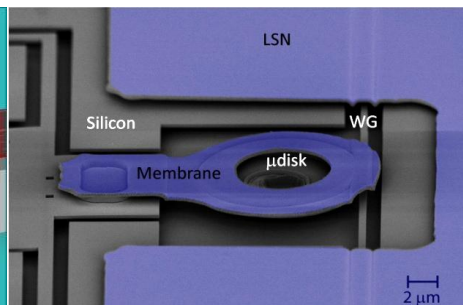
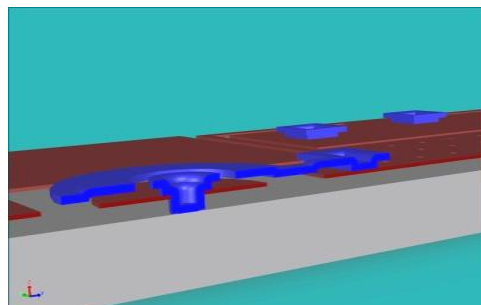
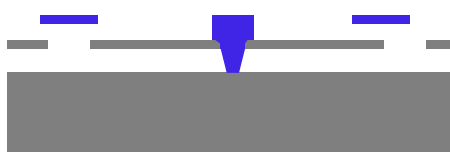
Si layer patterned with E-beam and dry etched



Si Dioxide deposited, patterned and etched

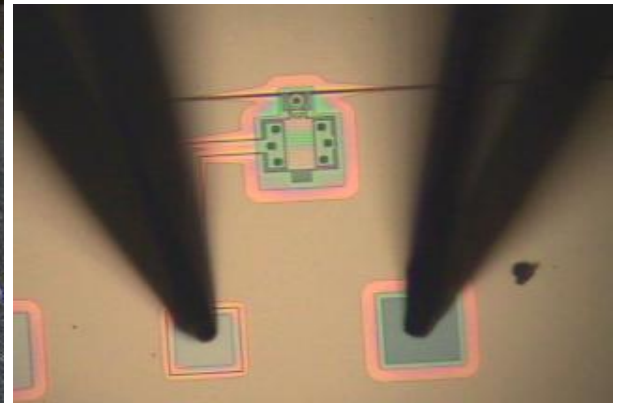
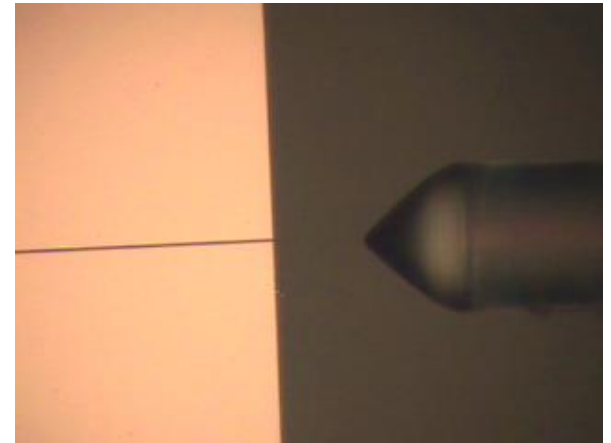
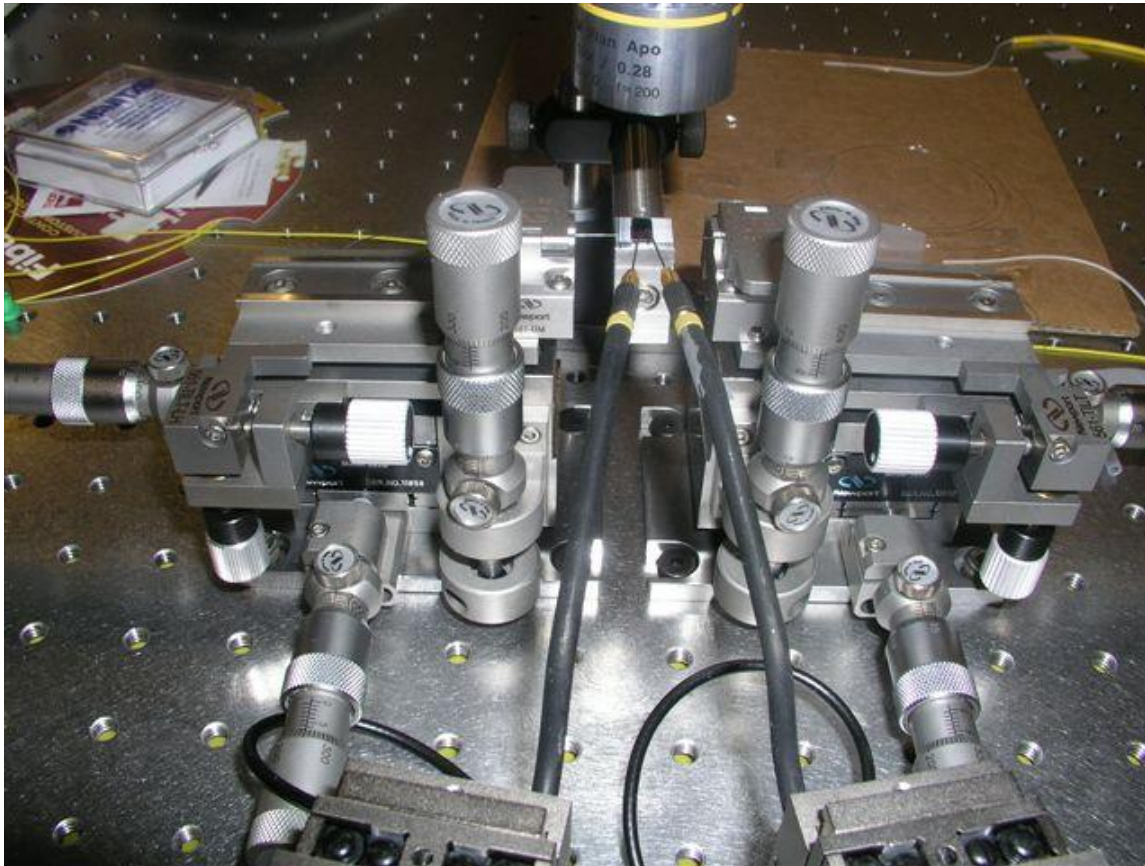
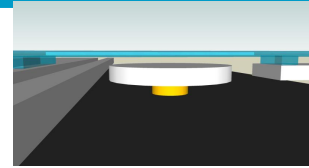


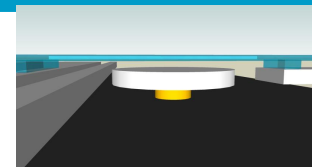
Si Nitride deposited, patterned and etched; Si Dioxide etched



**3.8V:
69 nm**

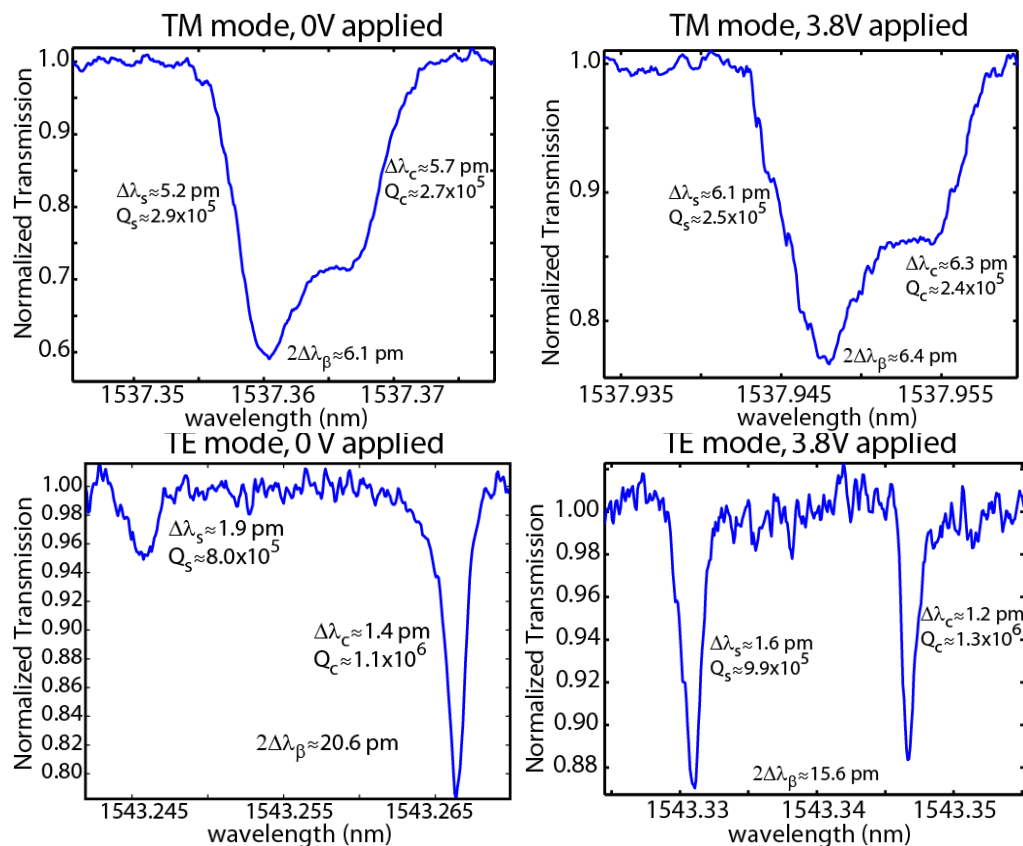
Testing Setup



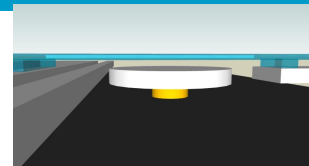


Experimental Results

Resonant spectra at various gaps

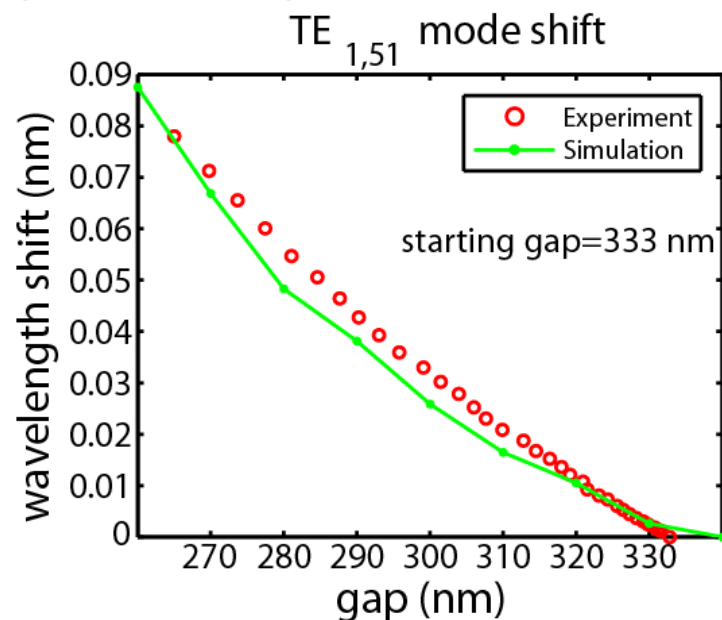
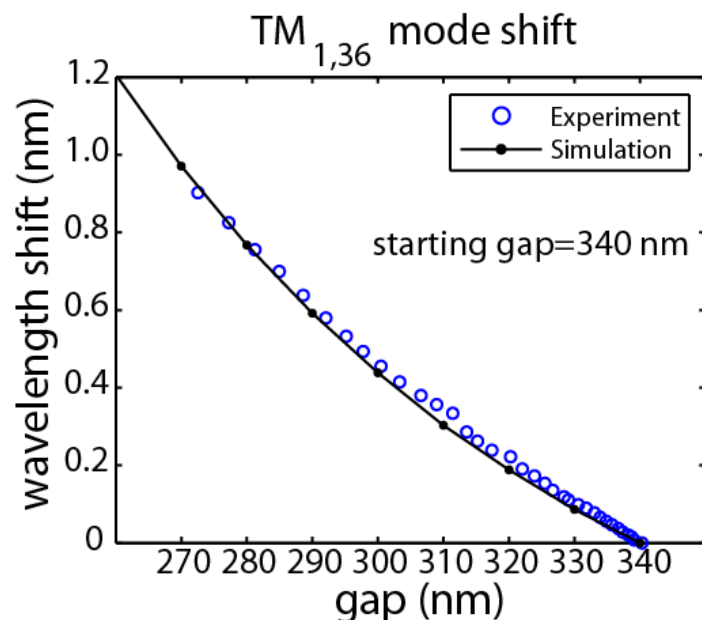


- 5.2 pm linewidth (TM, $Q \sim 3.0 \times 10^5$)
- 1.2 pm linewidth (TE, $Q \sim 1.3 \times 10^6$)
- 69 nm mechanical displacement
- No obvious optical Q degradation



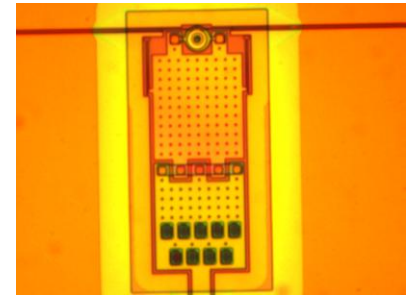
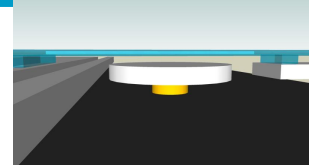
Experimental Results

■ Resonant frequency tuning vs. gap change



- Optical resonance tuning: ~ 150 linewidth (TM); ~ 50 linewidth (TE)
- Mechanical Displacement/resonance shift: 0.5 nm/linewidth (TM); 1.3 nm/linewidth (TE)
- Implication for displacement sensing: 0.5 pm sensitivity assuming a 1000 SNR

Integrated MEMS Tunable Cavity Summary



New Design

**Optical resonance tuning:
15 nm ~ 750 linewidth (TM)**

- Optomechanical Transduction
 - Optical Cavity Linewidth: 1.2 pm (TE, $Q \sim 1.3 \times 10^6$)
 - Optical resonance tuning: ~ 150 linewidth (TM)
 - Mechanical Displacement/resonance shift: ~ 0.5 nm/linewidth shift (TM)
 - Implication for displacement sensing: < 0.5 pm sensitivity

