SPM Technologies: Past, Present and Future

Qing Tu, MSE & NU*ANCE* Center

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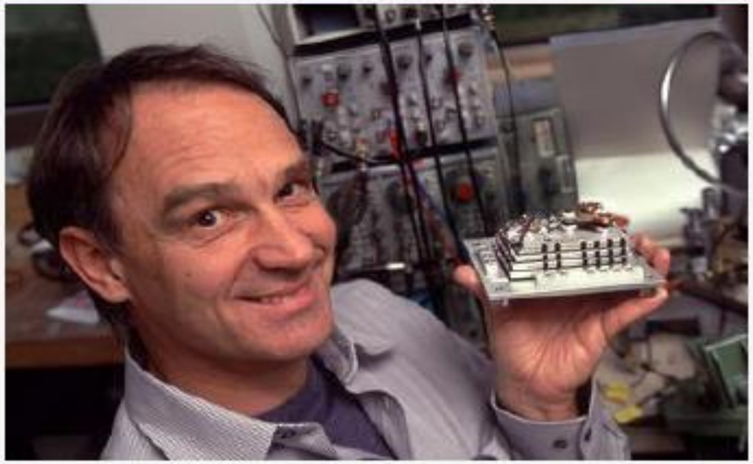
Outline

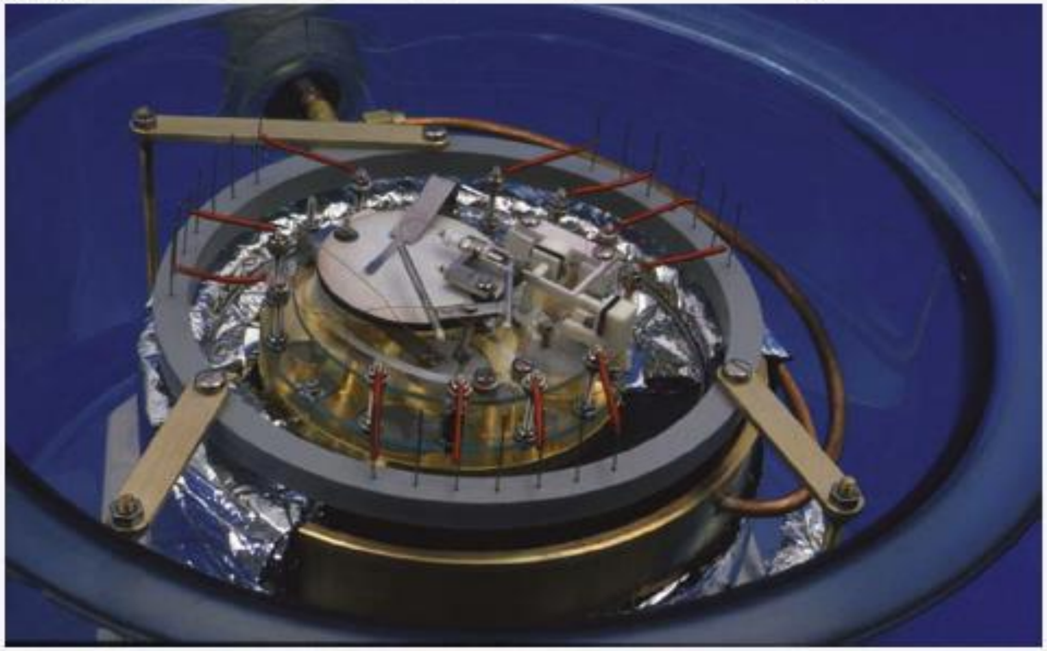
- Overview of AFM history
- Basic Modes
- Advanced Modes

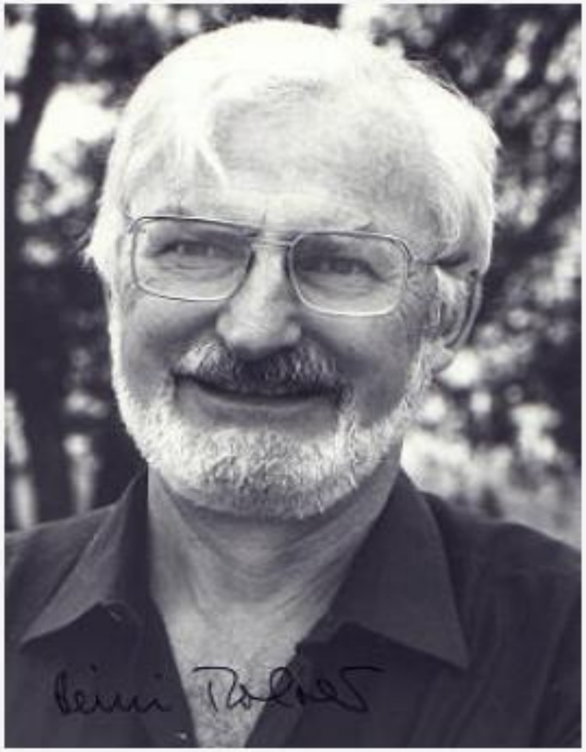
A Revolution in the Nanoworld: *Scanning Tunnelling Microscope*

Nobel Prize 1986



G. Binnig





H. Rohrer

A Brief Moment in the History of STM
Oberlech July 1985
A Giant Step for Nanoscience and Technology

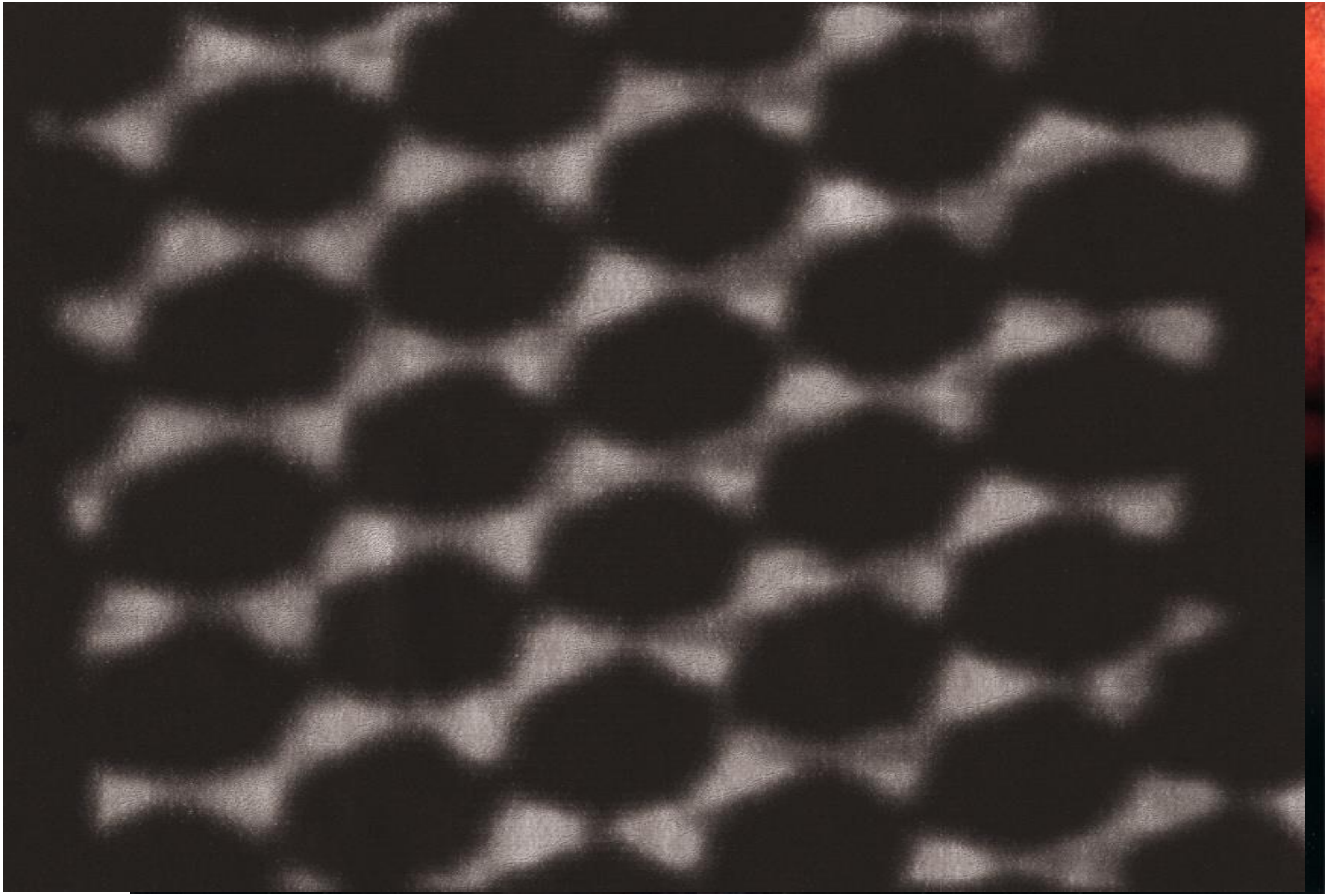
Back row:
Miedema, Baratoff, Quate, Salvan, Feenstra, Kaiser, Welland, Hoesler, Berghaus, Baro, Marti, Vieira,

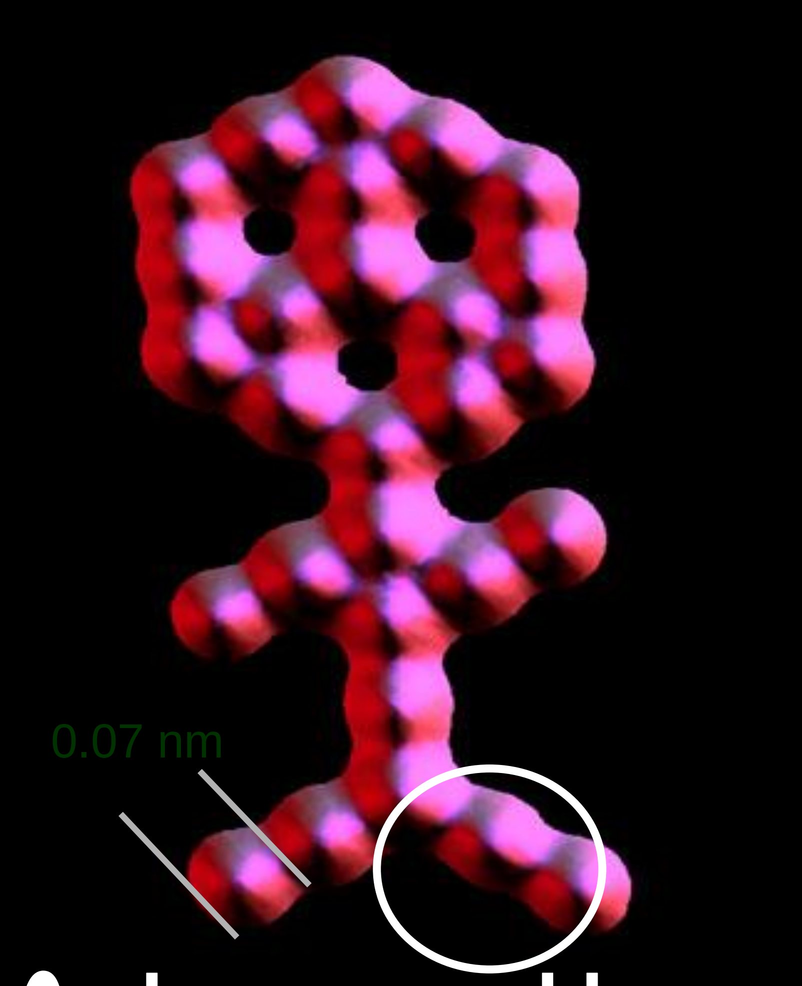
Stoll, Dürig, Muralt, Behm, Hansma, Celotta

Middle row:
Garcia, Neddermeyer, Van Kempen, Ringger, Pohl, Abraham, Chiang, Demuth, Humbert, Gimzewski,
Salemink, Lang, Golovchenko, Güntherodt, Miranda, Fink, Gomez

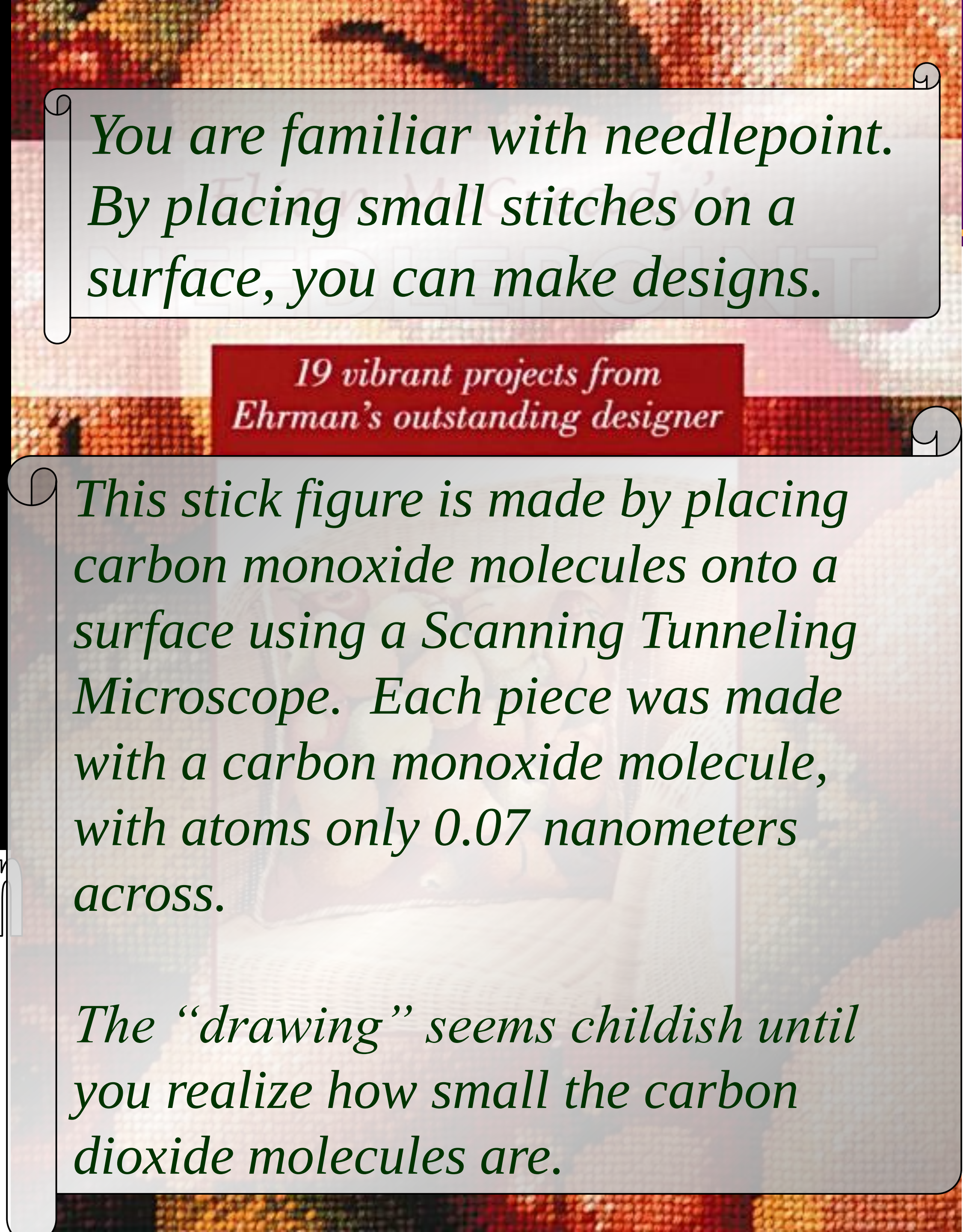
Front row:

Büttiker, Pethica, Baldeschwieler, Rohrer, Wilson, Elrod, Müller, Binnig, Gerber





Carbon monoxide man



You are familiar with needlepoint. By placing small stitches on a surface, you can make designs.

This stick figure is made by placing carbon monoxide molecules onto a surface using a Scanning Tunneling Microscope. Each piece was made with a carbon monoxide molecule, with atoms only 0.07 nanometers across.

The "drawing" seems childish until you realize how small the carbon dioxide molecules are.

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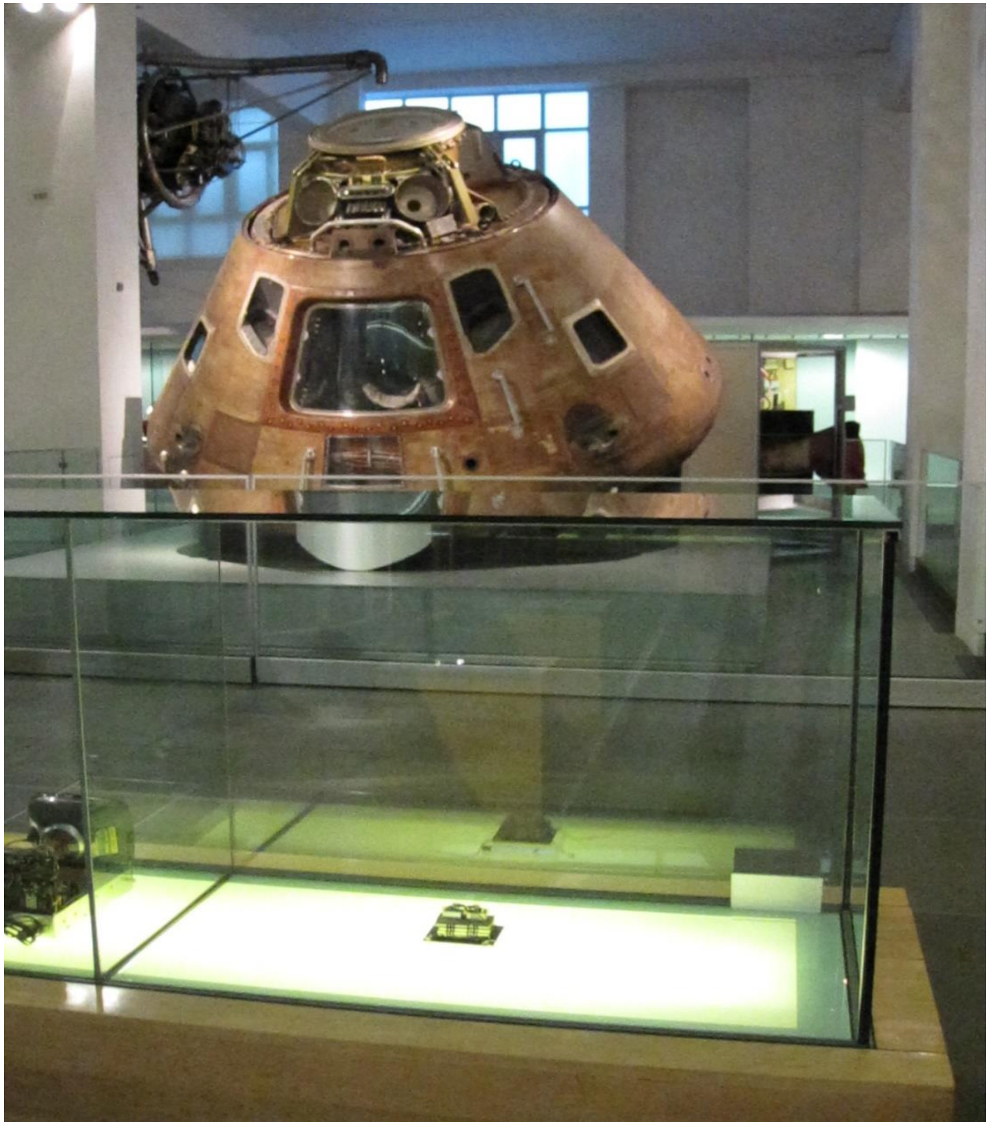
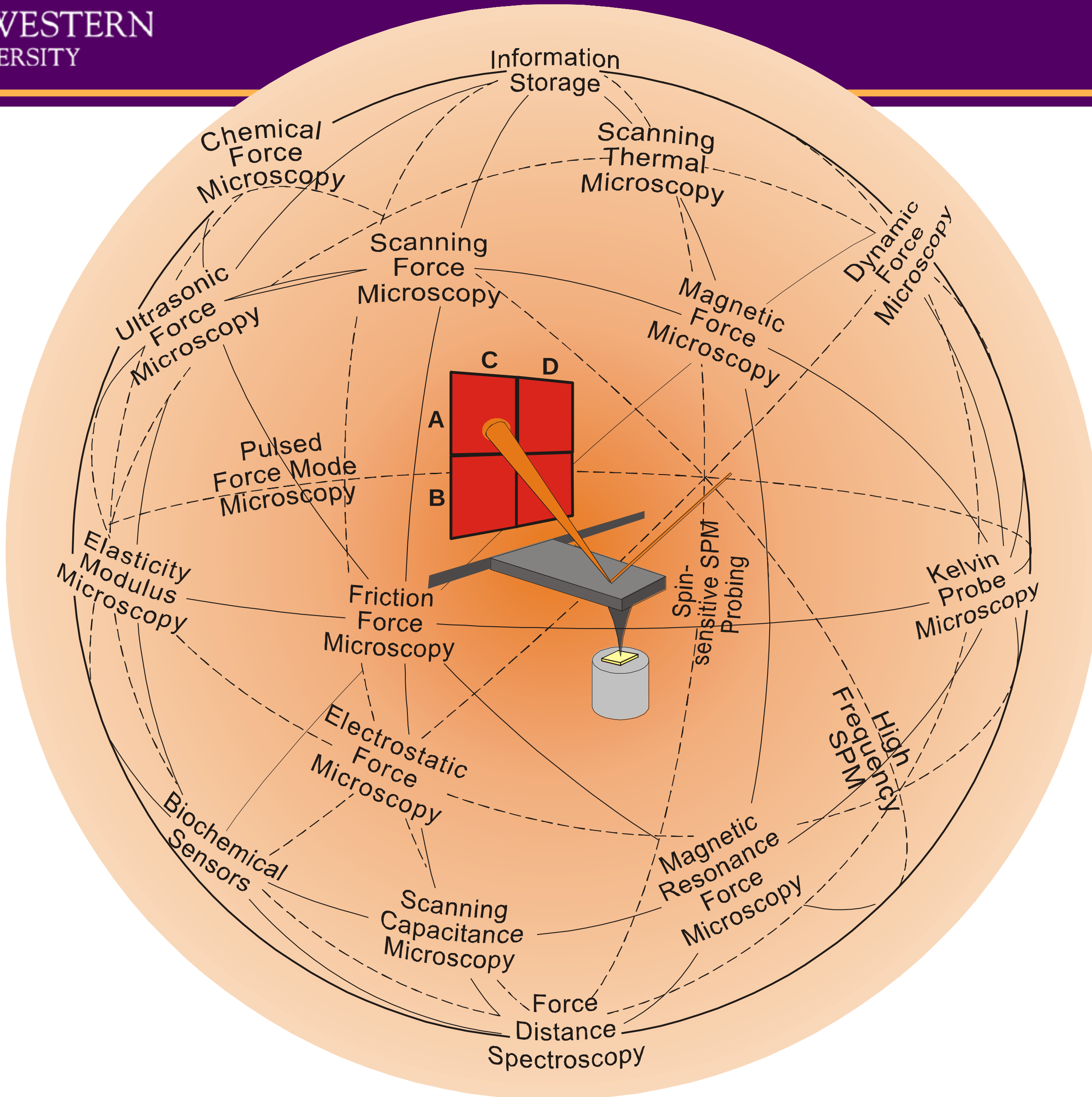
Science Museum London

„The Making of the Modern World“



Original AFM

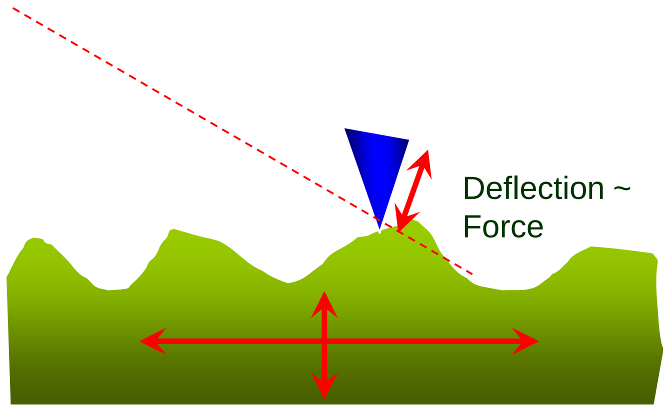
AFM UNIVERSE



Atomic Force Microscope

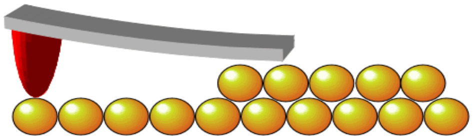
- Instead of using light or electrons to probe the sample, the AFM uses a tip suspended above the surface.
- The attractions or repulsions between the tip and the surface cause the tip to deflect.
- A laser senses the deflection.
- Scanning the tip across the surface generates the image.

a. **Nonconducting Surface – No bias voltage.**
b. **Sensing tip is cantilever <u>force</u> sensor.**
c. **Relies on "van der Waals" forces between atoms and molecules**

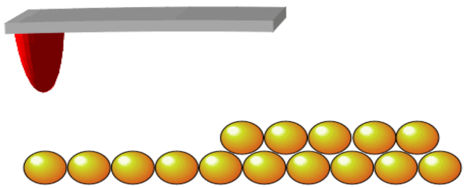


Piezoelectric translators

contact mode



non-contact mode



Tip angstroms from surface (repelled)

Constant force

Highest resolution

May damage surface

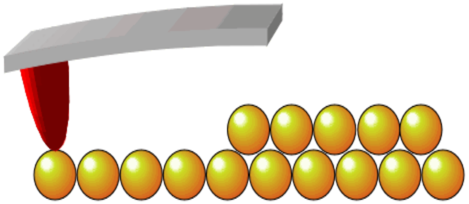
Tip hundreds of angstroms from surface (attracted)

Variable force measured

Lowest resolution

Non-destructive

tapping mode



Intermittent tip contact

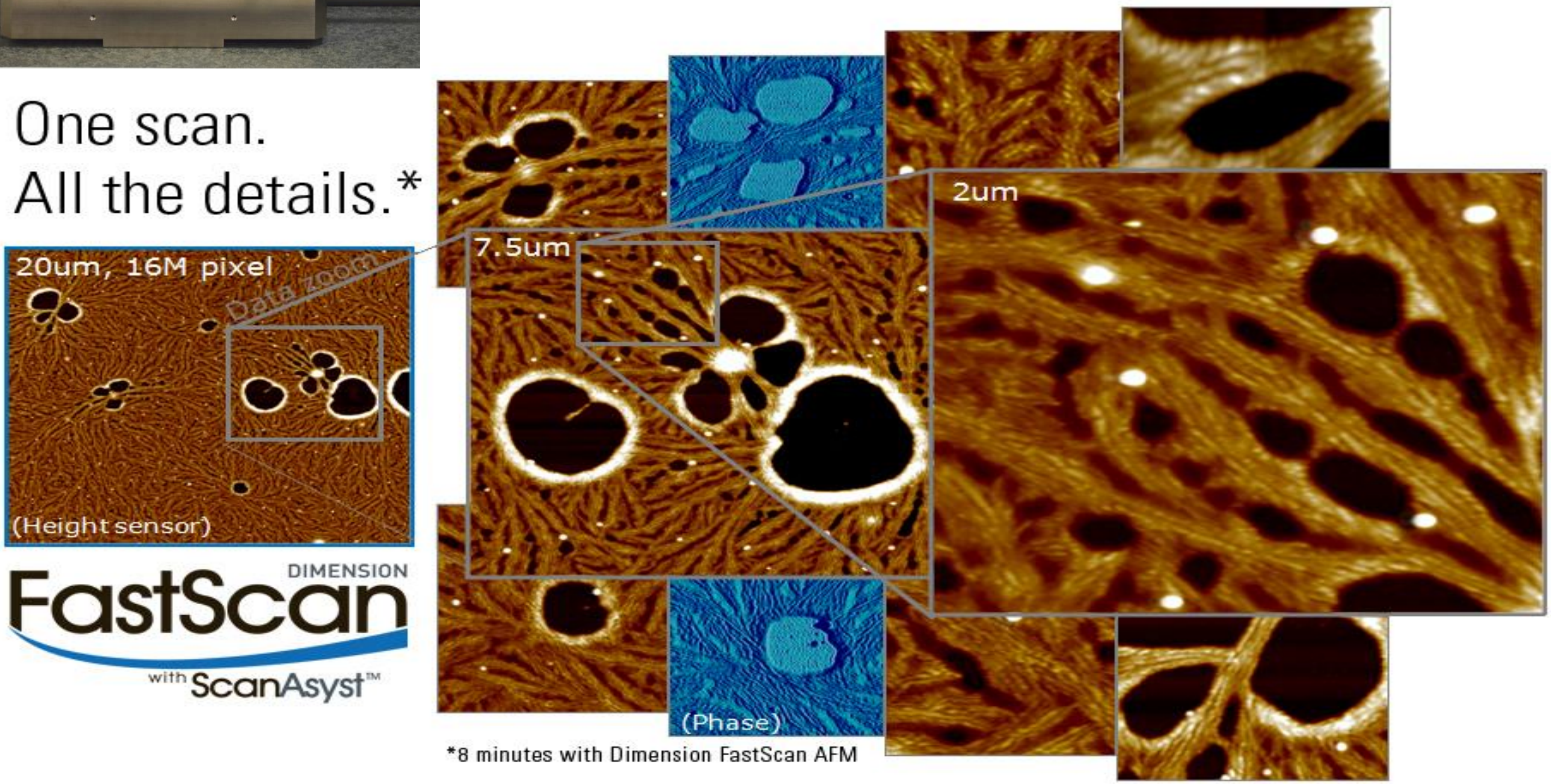
Variable force measured

Improved resolution

Non-destructive

- High bandwidth enables exceptional force control and high scan rates with closed-loop accuracy to surpass efficiency of any other commercial AFM system
- 20Hz Tapping Mode scan rates provide excellent quality images, matching that typically seen at 1Hz and maintaining good quality even at scan rates >100Hz
- Higher speed ScanAsyst delivers superb quality images at 6Hz and a surveying capability up to a 32Hz scan rate

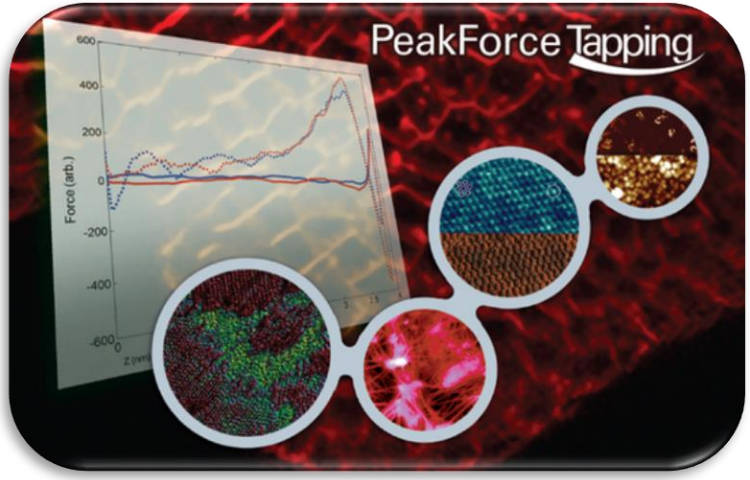
One scan.
All the details.*

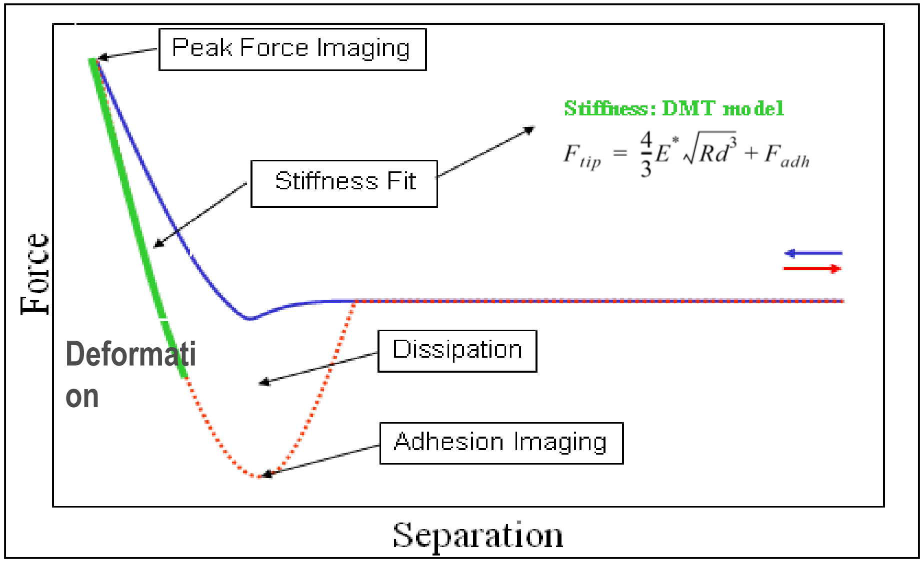


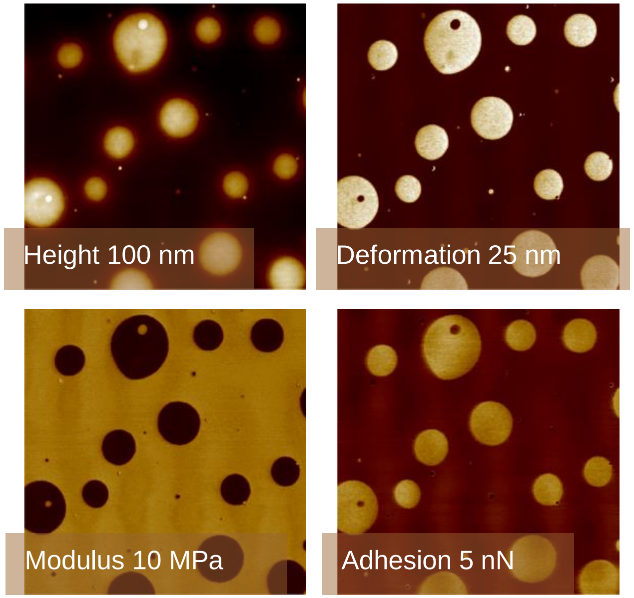
DIMENSION FastScan with ScanAsyst™

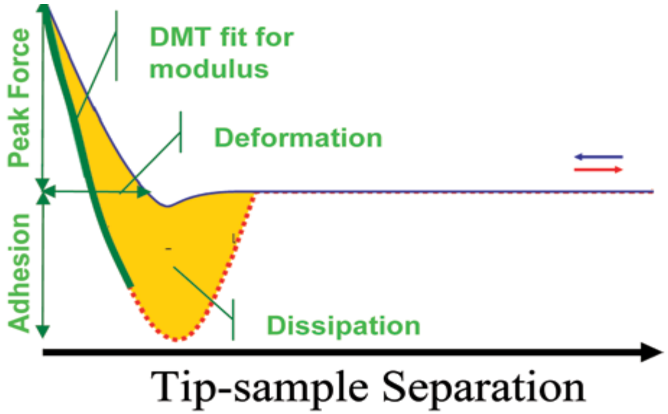
*8 minutes with Dimension FastScan AFM

ADVANCES in SPM:
Quantitative Nanomechanics





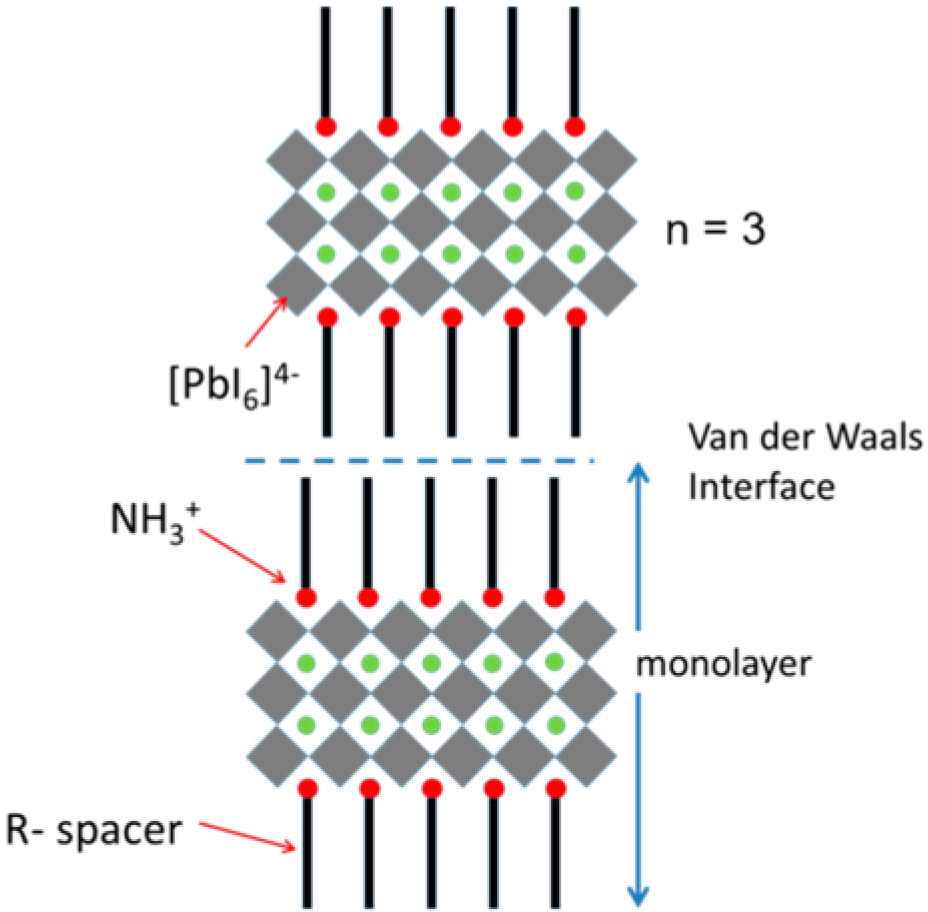


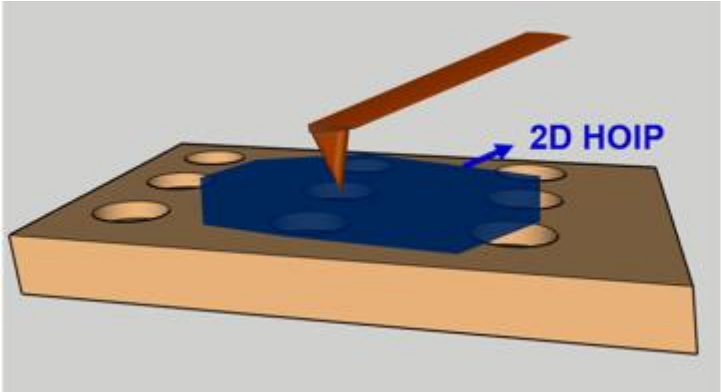


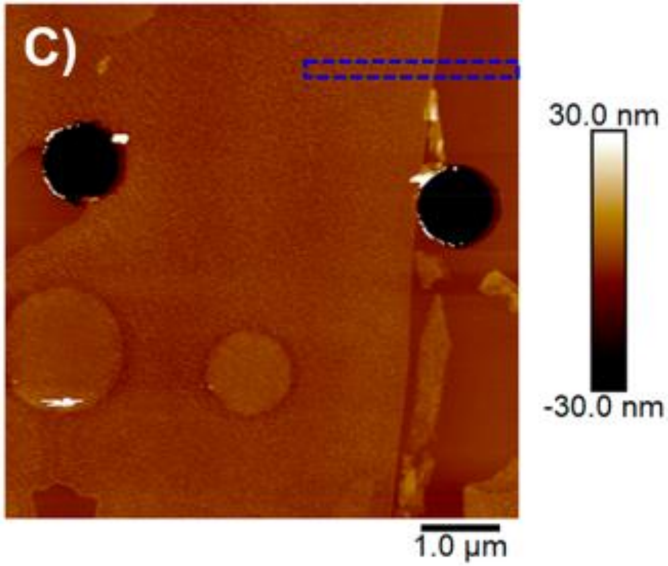
ADVANCES in SPM: *Quantitative Nanomechanics*

- 2D Ruddlesdon-Popper HOIPs:$(C_mH_{2m+1}NH_3)_2(CH_3NH_3)_{n-1}Pb_nI_{3n+1}$

  Here m = 4, n = 3

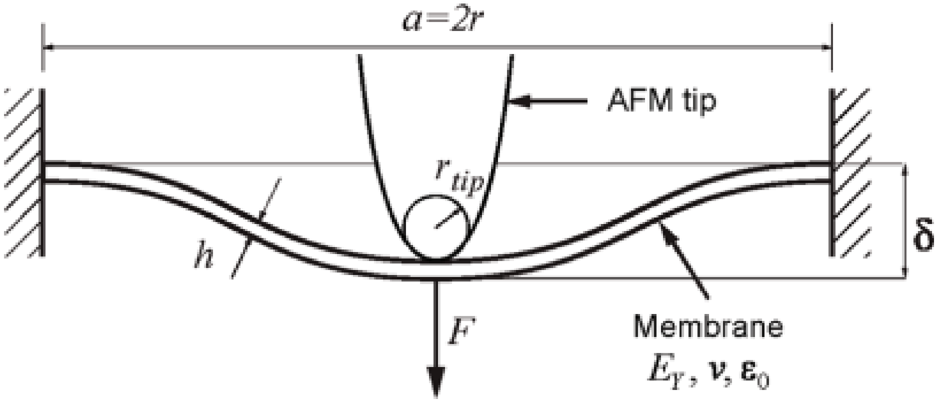






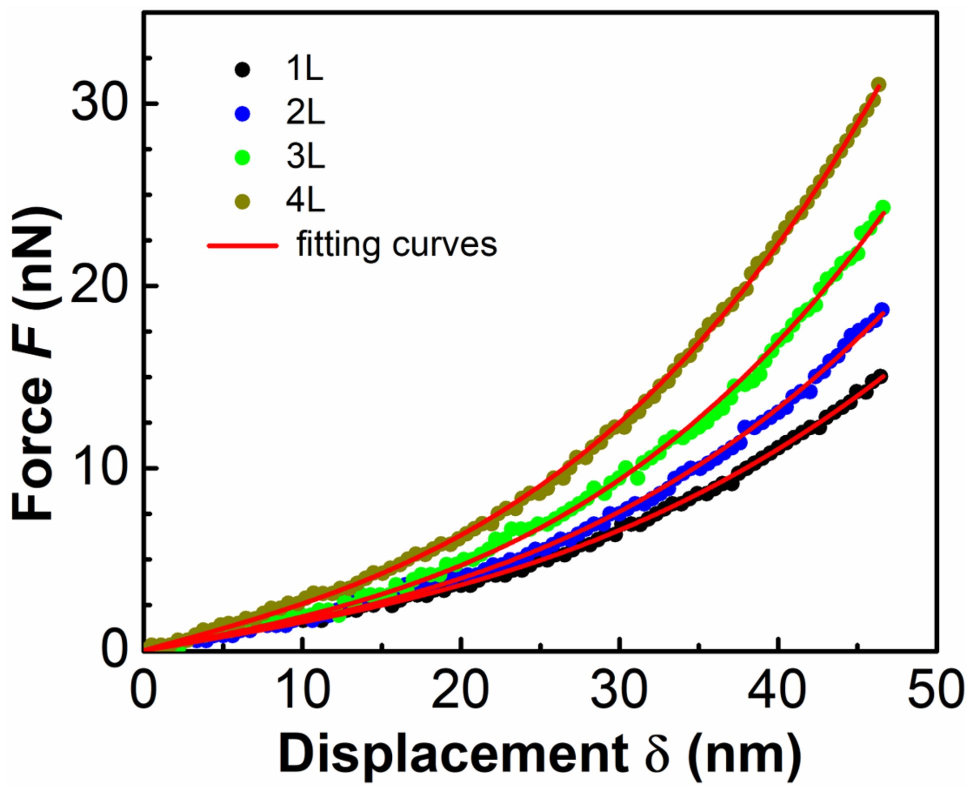
Tu et al., ACS Nano, 2018, 12(10), 10347 – 10354

ADVANCES in SPM: *Quantitative Nanomechanics*



$$F = \sigma_0^{2D}\pi\delta + E^{2D}\frac{q^3\delta^3}{r^2}$$

$$\sigma_m = \frac{1}{h}\sqrt{\frac{F_{max}E^{2D}}{4\pi r_{tip}}}$$



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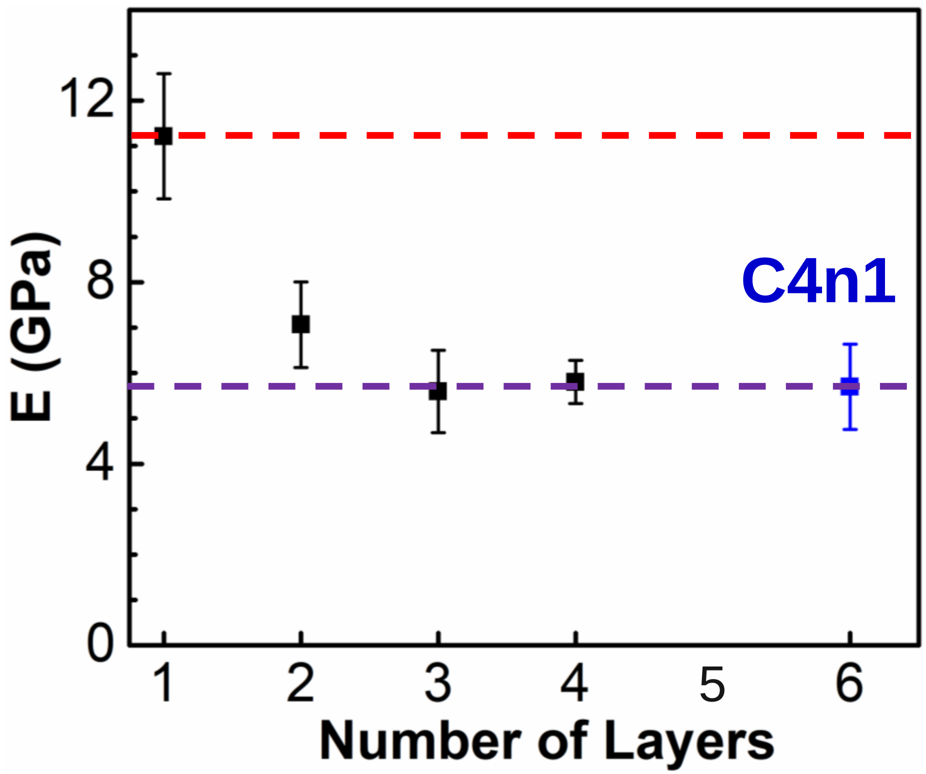
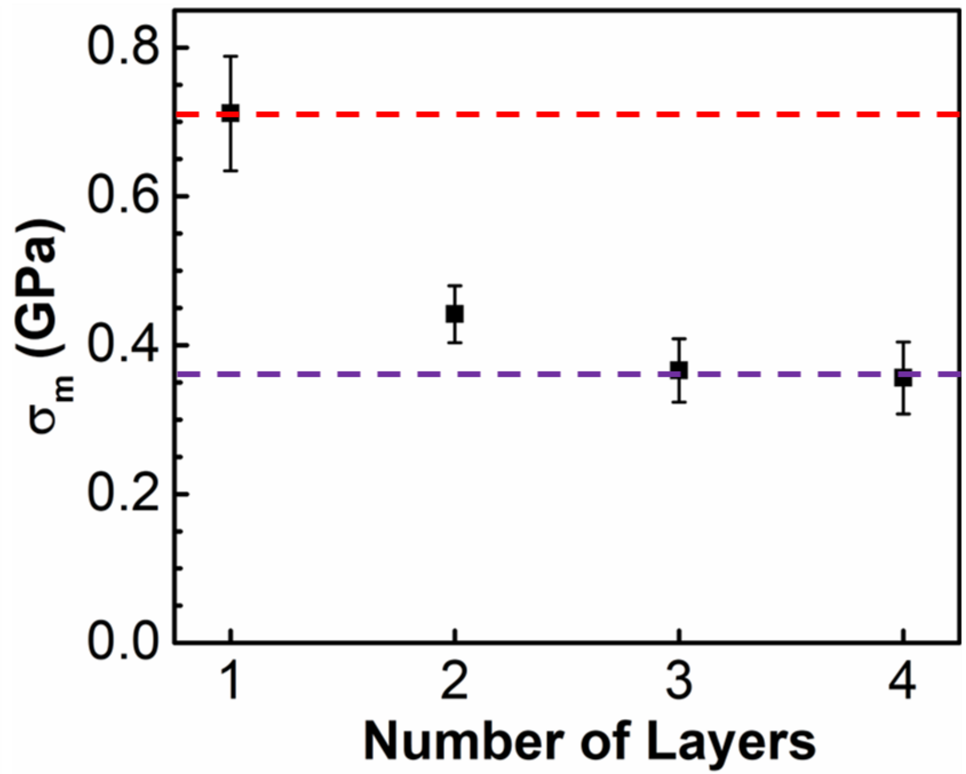
ADVANCES in SPM: *Quantitative Nanomechanics*

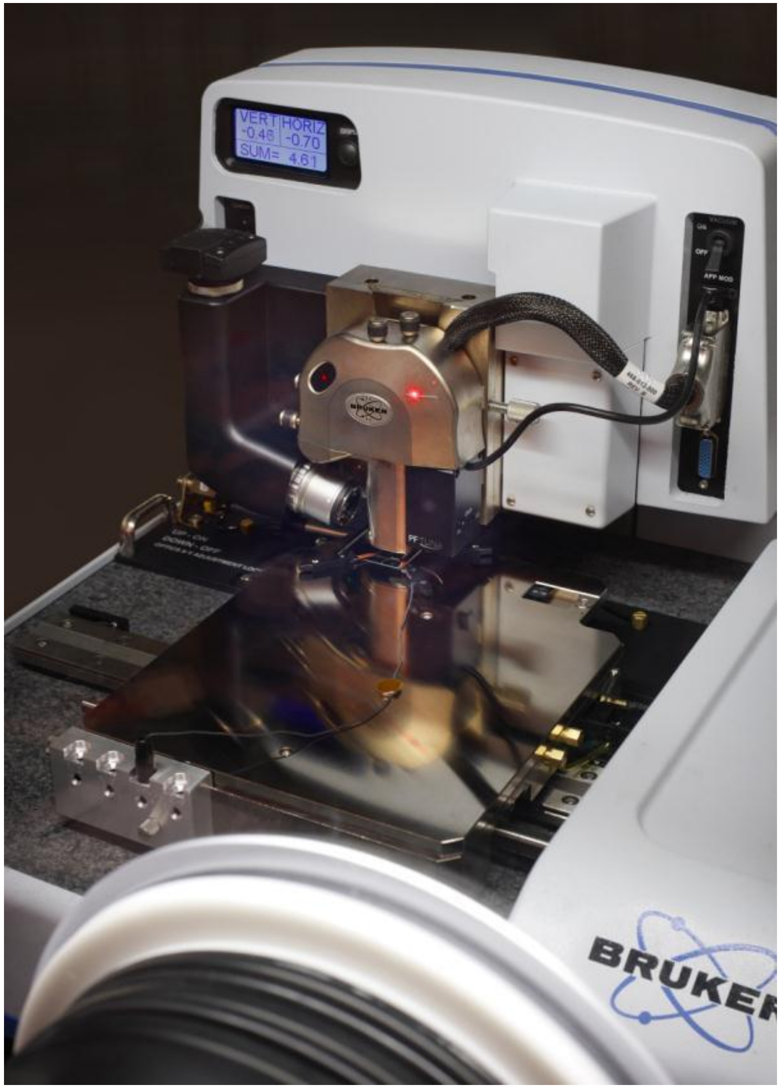




Tu et al., ACS Nano, 2018, 12(10), 10347 – 10354

Photoconductive AFM

- Measure OPV conductivity under illumination
- Unravel conduction mechanisms
- Combine with PeakForce TUNA & 1ppm environmental control



Life Science Imaging System

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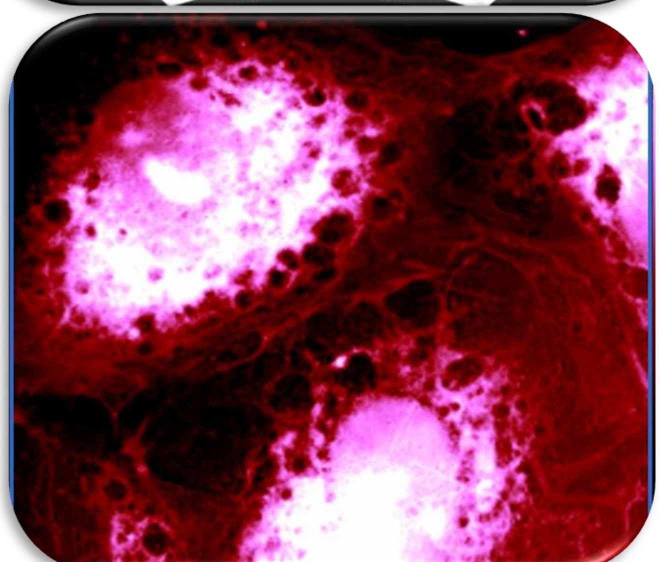
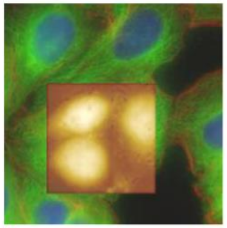


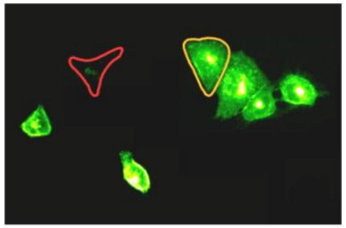
- ***Uncompromised Performance***
  - High-resolution imaging capability
  - PicoForce-quality force measurements
  - Supports standard light microscopes for uncompromised optical performance
- ***Most Complete Integration of AFM and Light Microscopy***
  - MIRO software allows optical images to guide AFM imaging and force measurements
  - Create correlated AFM and optical datasets with flexible offline analysis features
  - MIRO makes the AFM a natural extension of the optical microscope
- ***Easiest to Use and Most Productive Life Science AFM***
  - ScanAsyst automatically optimizes imaging parameters for expert-quality results
  - Probe exchange and laser alignment is made easy with EasyAlign™ accessory
  - "Experiment Selector" automatically configures the software for common modes
- ***Simple, Effective Solutions for Biological Samples***
  - Easy mounting for common sample types, including slides, cover slips, and petri dishes
  - Micro-volume perfusion accessory is ideal for applications that utilize expensive reagents
  - Petri dish perfusion accessory with heating capability allows long-duration live cell studies

MIRO software enables the AFM to be used as a natural extension of the light microscope

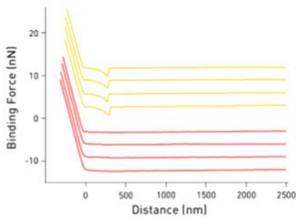
- Optical images are directly imported and registered to AFM calibration
- Images can be used to guide AFM imaging and force measurements
- Images can be overlaid and adjusted for presentation



AFM image overlaid on fluorescence image

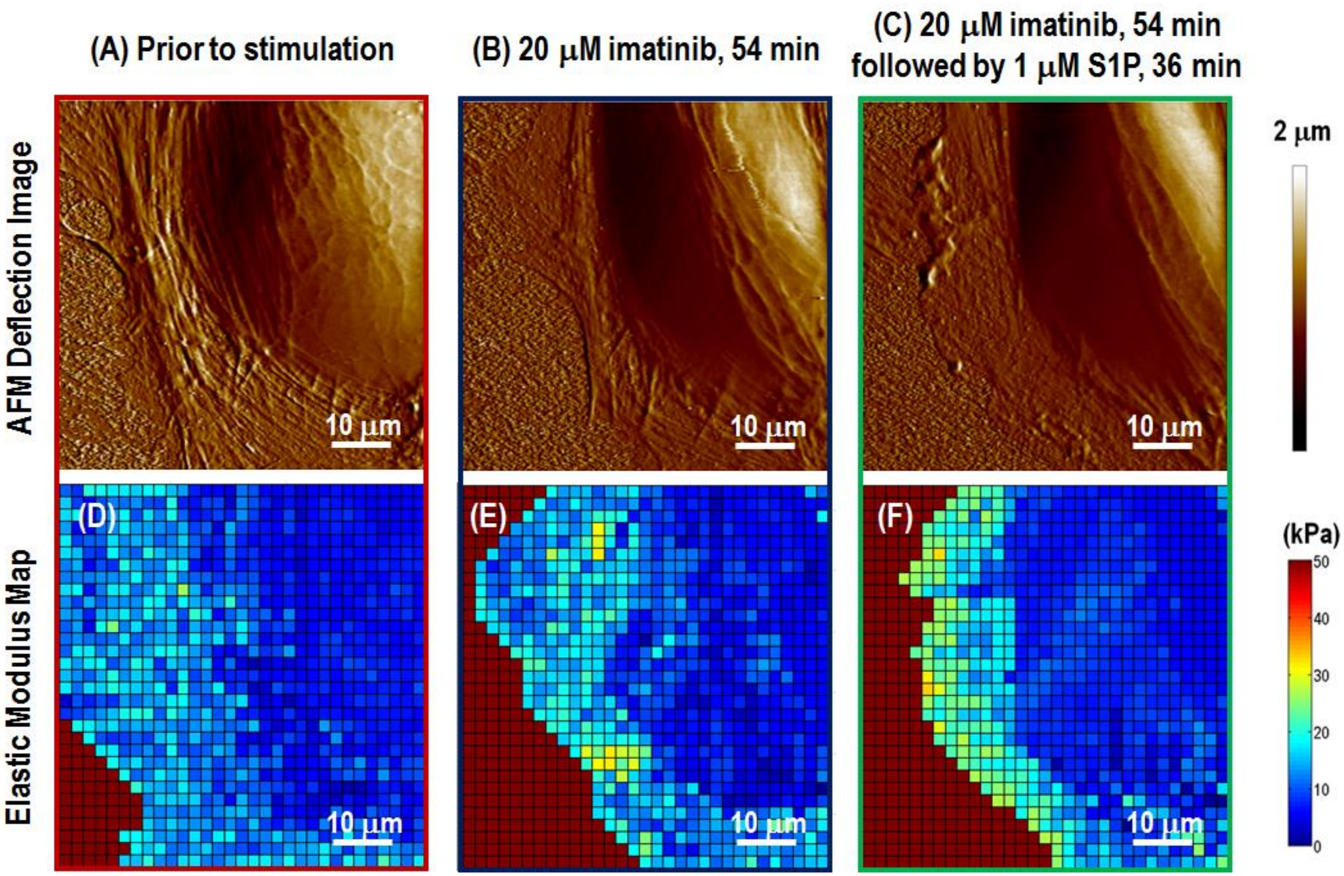


Fluorescence intensity corresponds to expression level of a tagged protein on the cell membranes



AFM binding measurements made with a functionalized probe correlate strongly with the fluorescence results

Endothelial Live Lung Cell Nanomechanics

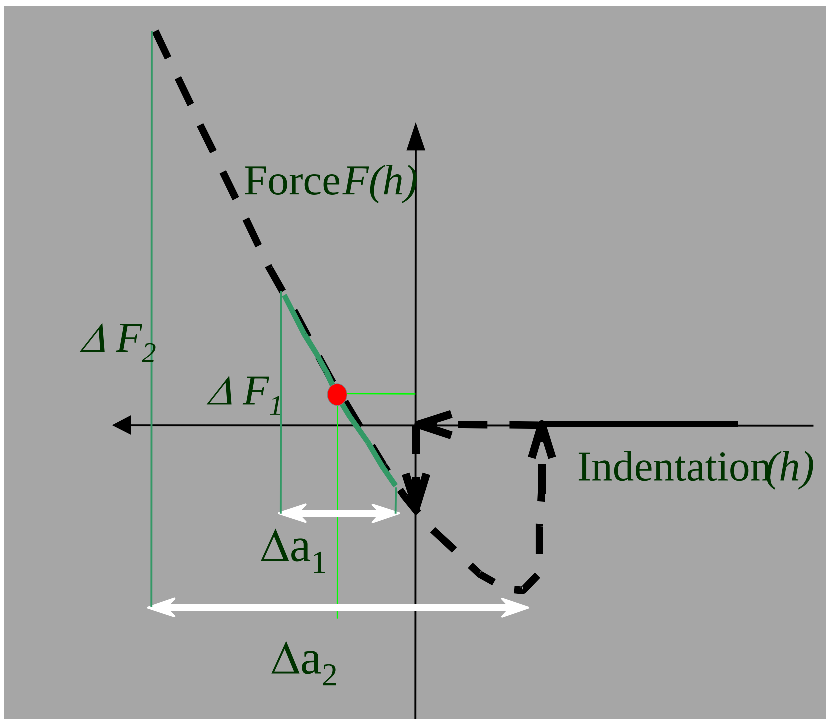


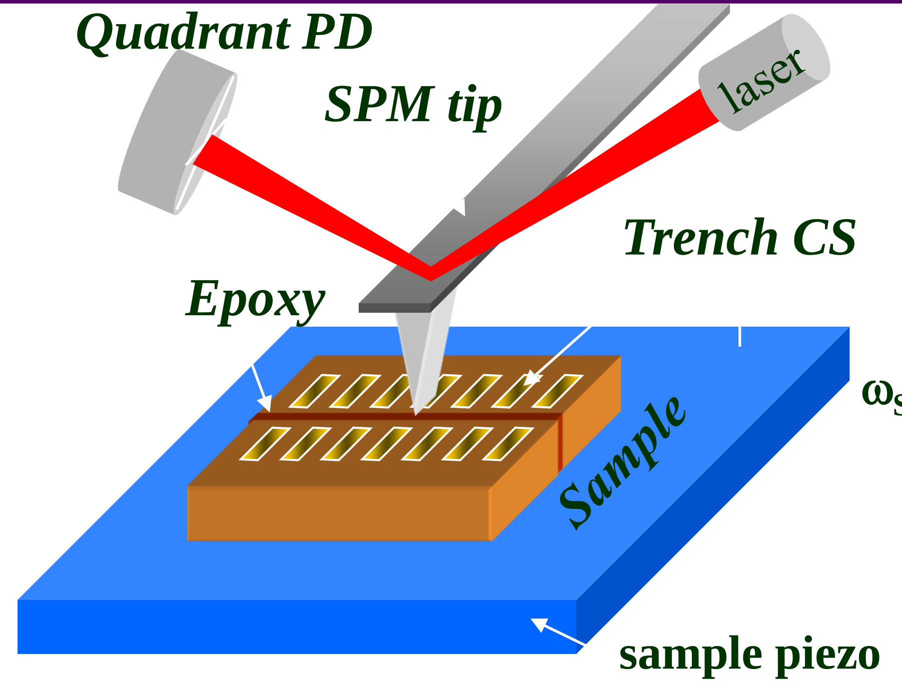
Nature Scientific Report 5, 11097 (2015)
Nature Scientific Report 8 (1) 1002 (2018),
Nature Scientific Report 7, 14152 (2017)
Nature Materials 15(4), 469 (2016)

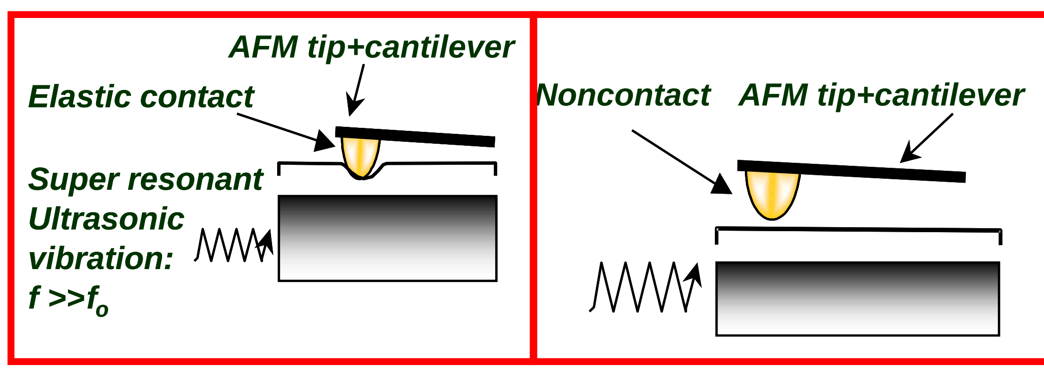
AFM deflection images of live EC prior to any simulation (A); in response to 54 min after treatment with 20 mM imatinib (B) followed by 36 min treatment with 1 mM S1P (C). The mechanical measurements were carried out by acquiring arrays of 32 × 32 loading-unloading curves in the force-volume map.. The time-lapse elastic modulus maps prior to any simulation (D); in response to 54 min after treatment with 20 mM imatinib (E); followed by 36 min after treatment with 1 mM S1P (F). Each pixel indicates the localized sub-cellular elastic modulus.

Ultrasonic Force Microscopy

1. Introduce ultrasonic (RF) vibration to sample in contact AFM
2. *Cantilever essentially rigid (inertially damped): fsample>> fcantilever*
3. Ultrasonic cantilever oscillation amplitude proportional to sample elasticity

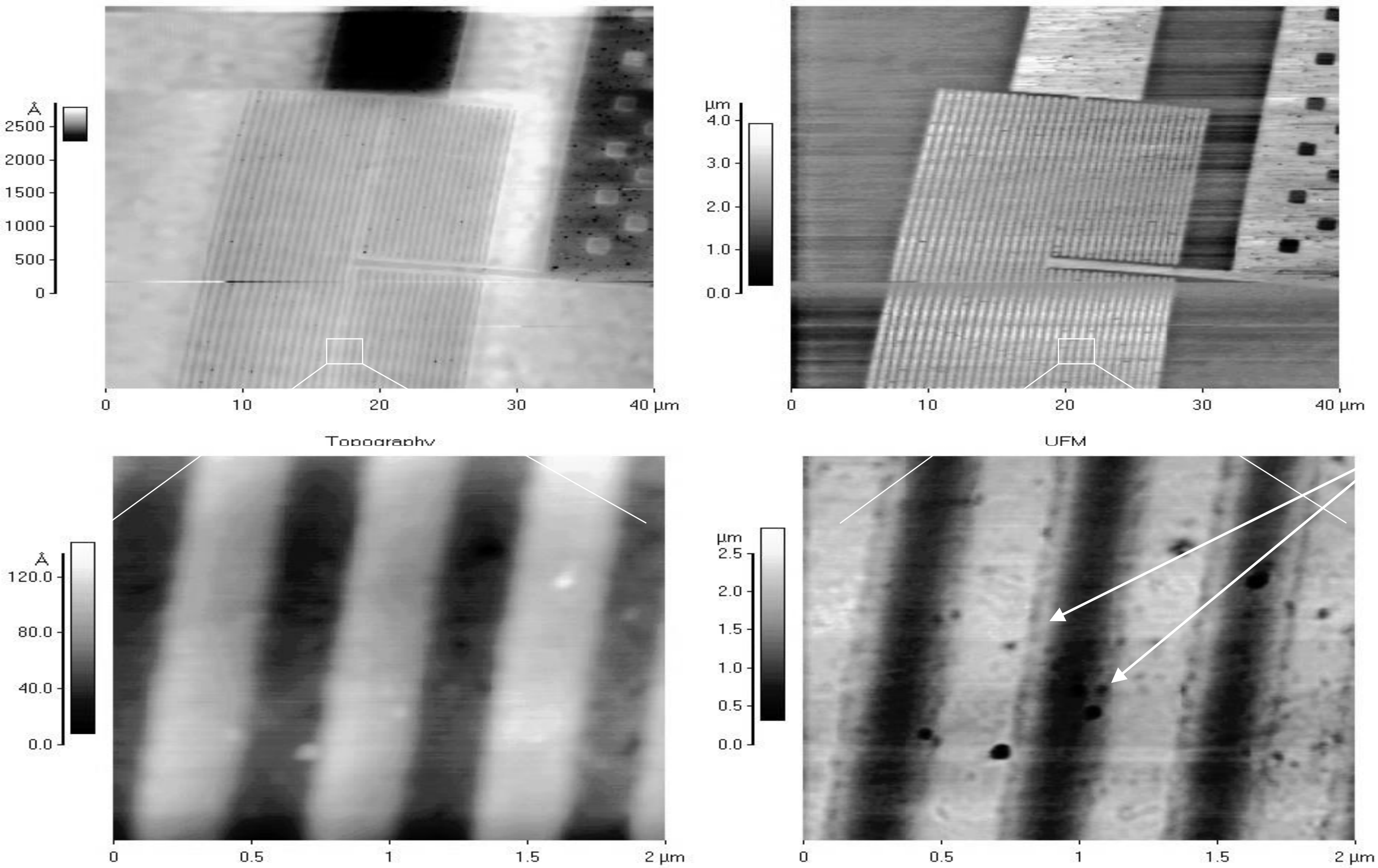






Elastic Mapping (Depth)

AFM ***UFM***



Note structure within polymer trench wall Width of high modulus region ~ 120 nm

Scanning Near Field Ultrasound Holography (SNFUH): *Seeing the Invisible!*

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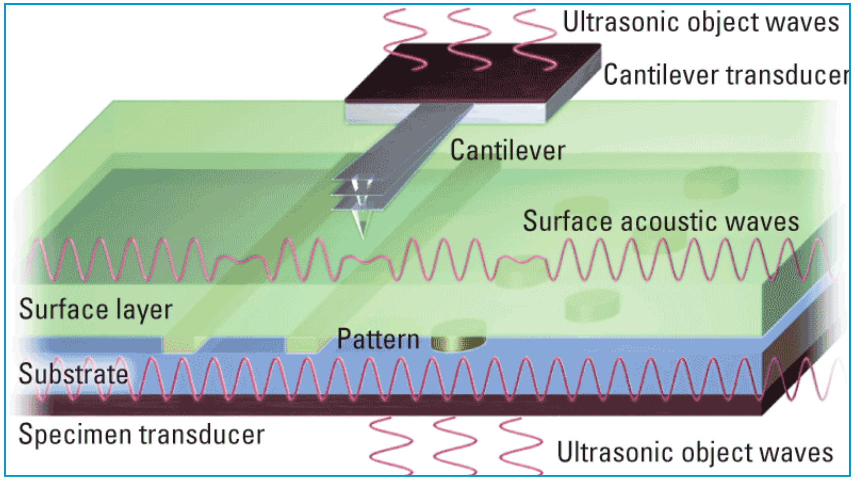
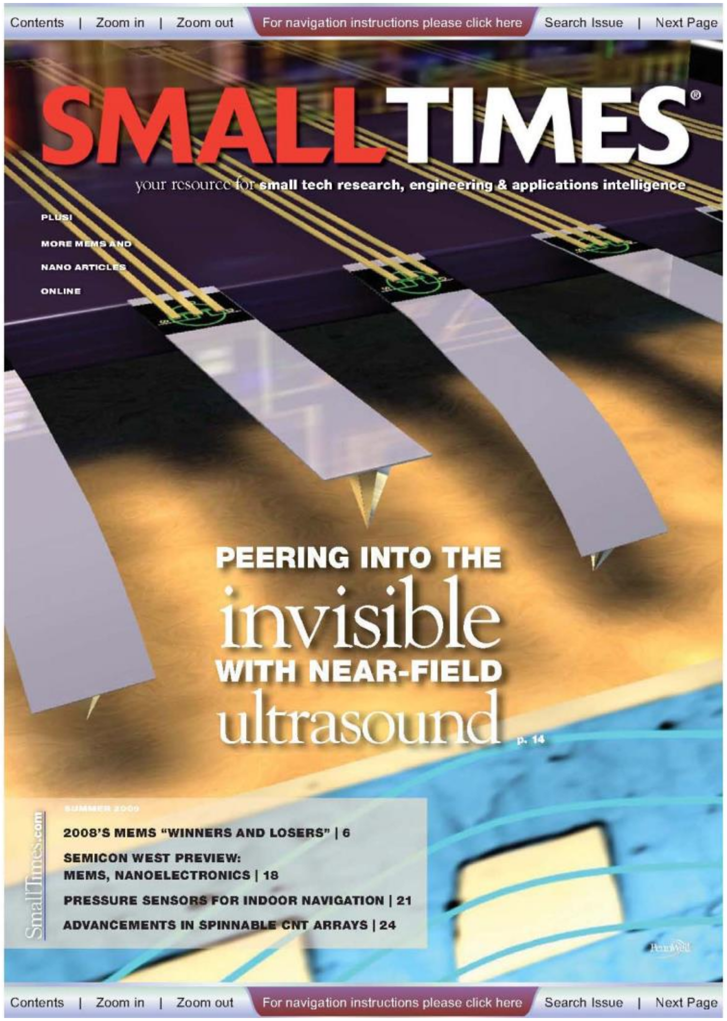
Science 310, 89 (2005), Nature Nanotechnology 3,501 (2008)



Near-Field SPM Platform:
➔ **Excellent Lateral Resolution**

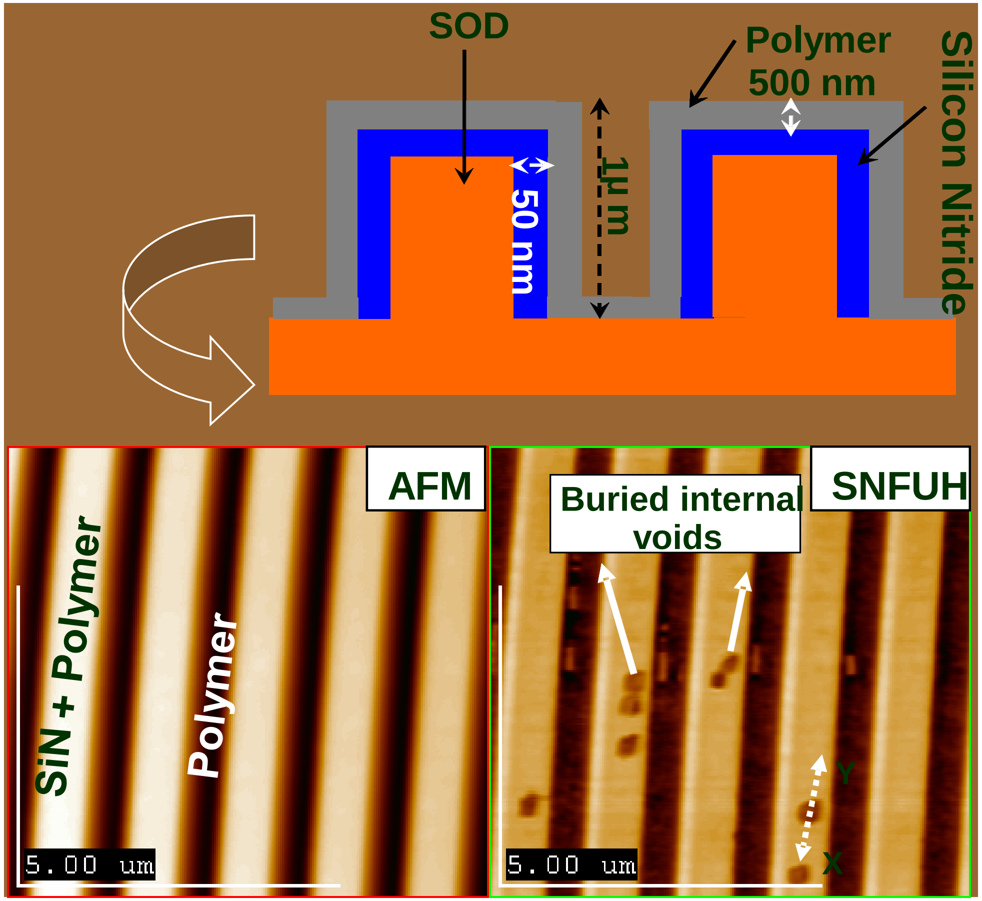
Ultrasound source:
➔ **Non-destructive and Depth-Sensitive**

Holography Paradigm:
➔ **Sensitive to "*Phase*" Perturbations**



Direct Application in Failure Analysis

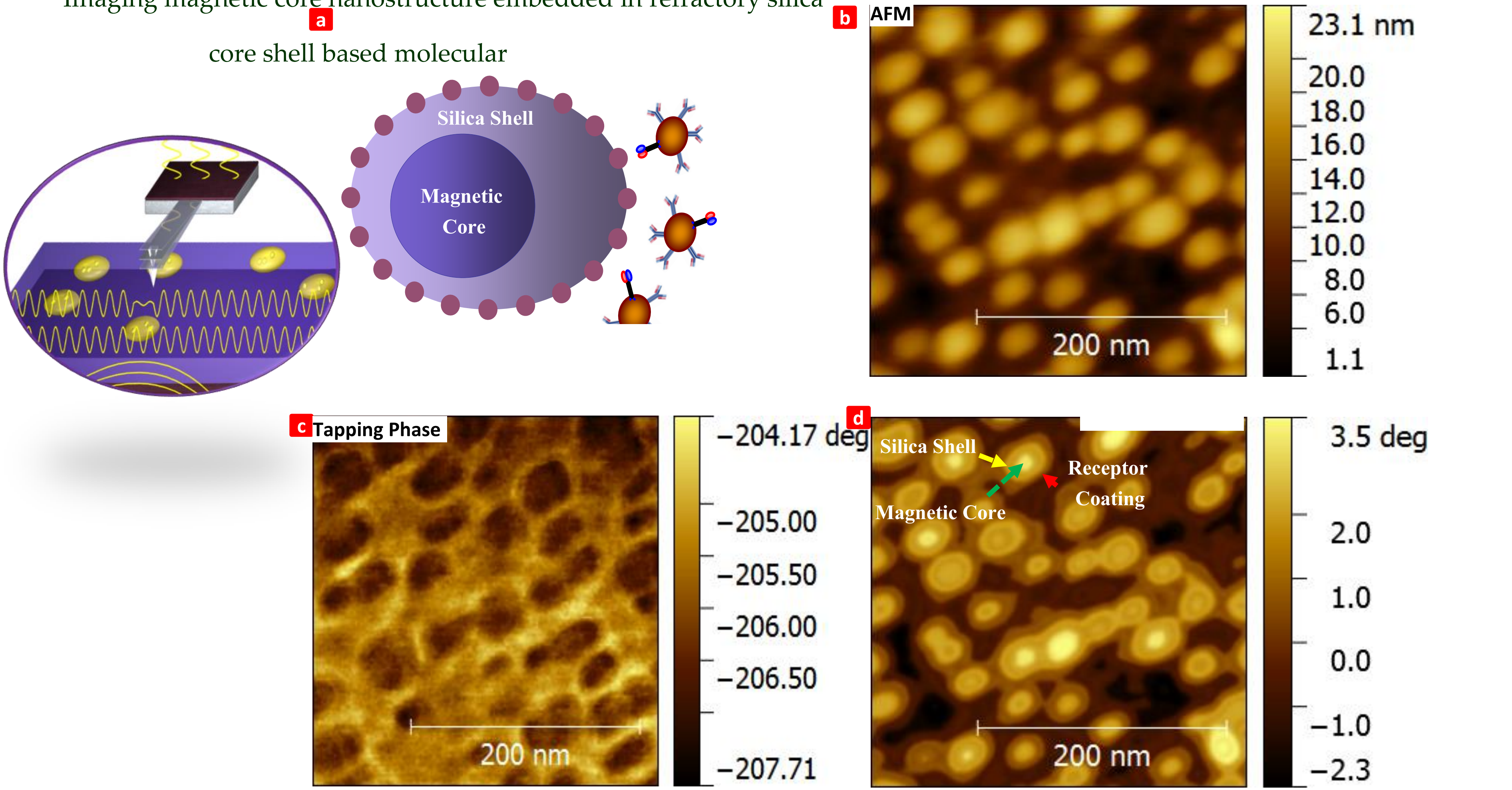
- Scanning Near Field Ultrasound Holography in Semiconductors
  - Nanoscale Imaging of embedded features/defects
  - Quantitative modulus imaging of metal-low K dielectrics
  - Non-invasive monitoring of molecular markers
  - Nanoscale non-invasive 3D tomography
  - Failure analysis and 3D Interconnects
  - Voiding, delamination with nanometer scale resolution



Science 310, 89 (2005)

Ultrasound Bioprobe for Nanomechanical Analysis

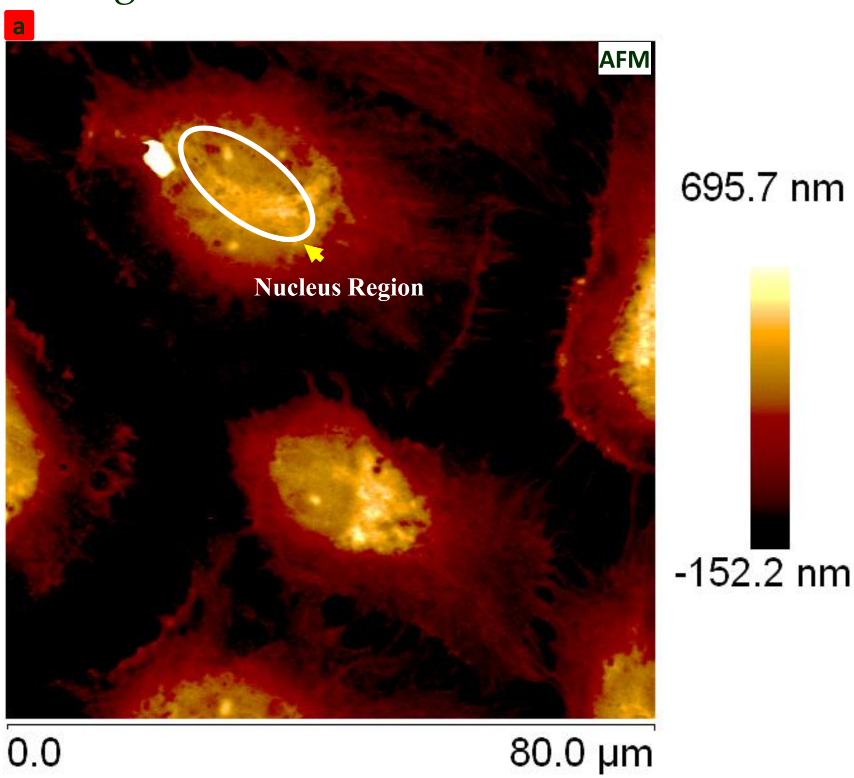
Imaging magnetic core nanostructure embedded in refractory silica

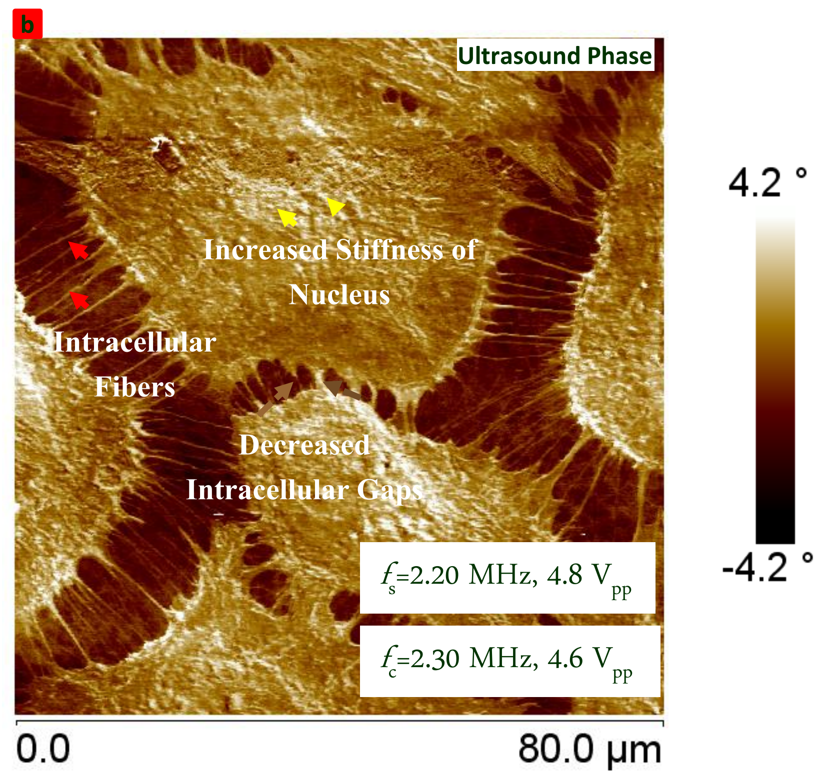


Science Advances 2017: 3;e1701176, Nature Scientific Report 8 (1) 1002 (2018), Nature Scientific Report 7, 14152 (2017)

Ultrasound Bioprobe for Nanomechanical Analysis

AFM topographical image EC cells altered by addition of thrombin and ultrasound bioprobe phase image demonstrates remarkable contrast from intra-cellular fibers. Intracellular fibers are predominantly seen in the ultrasound phase image along with stretched gaps and sub-cellular phase contrast on the nuclei region of the cells.





Science Advances 2017: 3;e1701176

Scanning Thermal Imaging System

(Joint Development with APP*NANO*)

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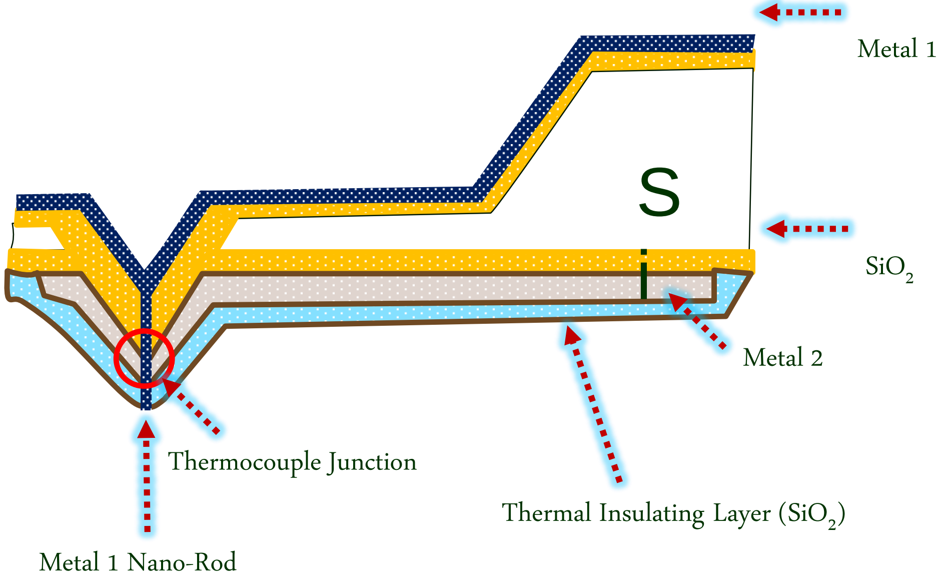
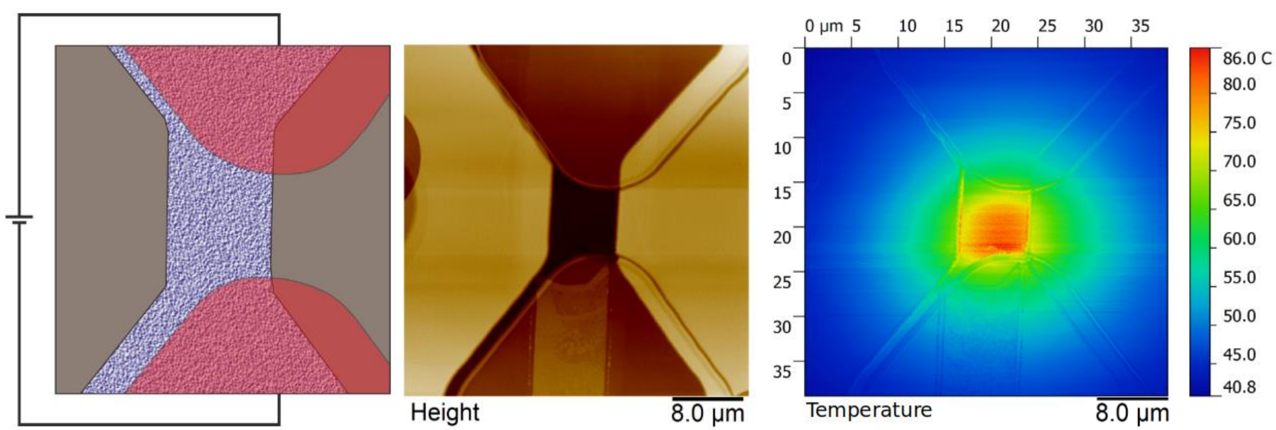


In conventional thermocouples, junction is directly in contact with the sample. The the size of the junction determines the resolution. In this current innovative design of the Thermal Probe the resolution is determined by the diameter of the metal -1 nano-rod and not by the size of the junction. Using modern microfacbrication techniques, one can easily create nano-rods of less than 20nm diameter. The smaller size, however, may have impact on the response time of the probe. The nanorod is positioned at the apex of the tip. This brings the nanorod in direct contact with the sample and as a result the thermal sensitivity of the probes is significantly improved. The extended length of the nanorod (length beyond the thermal junction) helps achieving long operational life of the probes.



Surface temperature mapping of a silicon micro heater. *Left panel*: schematic of the silicon micro-heater showing different degrees of ion implanted areas. Gray is plain silicon, blue is low dose implant and pink is high dose implant overlying plain silicon and low dose areas. *Middle panel:* topography and *Right panel:* Temp image. The temperature image captures the point-to-point variations in the surface temperature due to joule heating at the center and diffusion of heat by the underlying silicon.

ACS Nano, **2018**, *12* (2), pp 1760–1767

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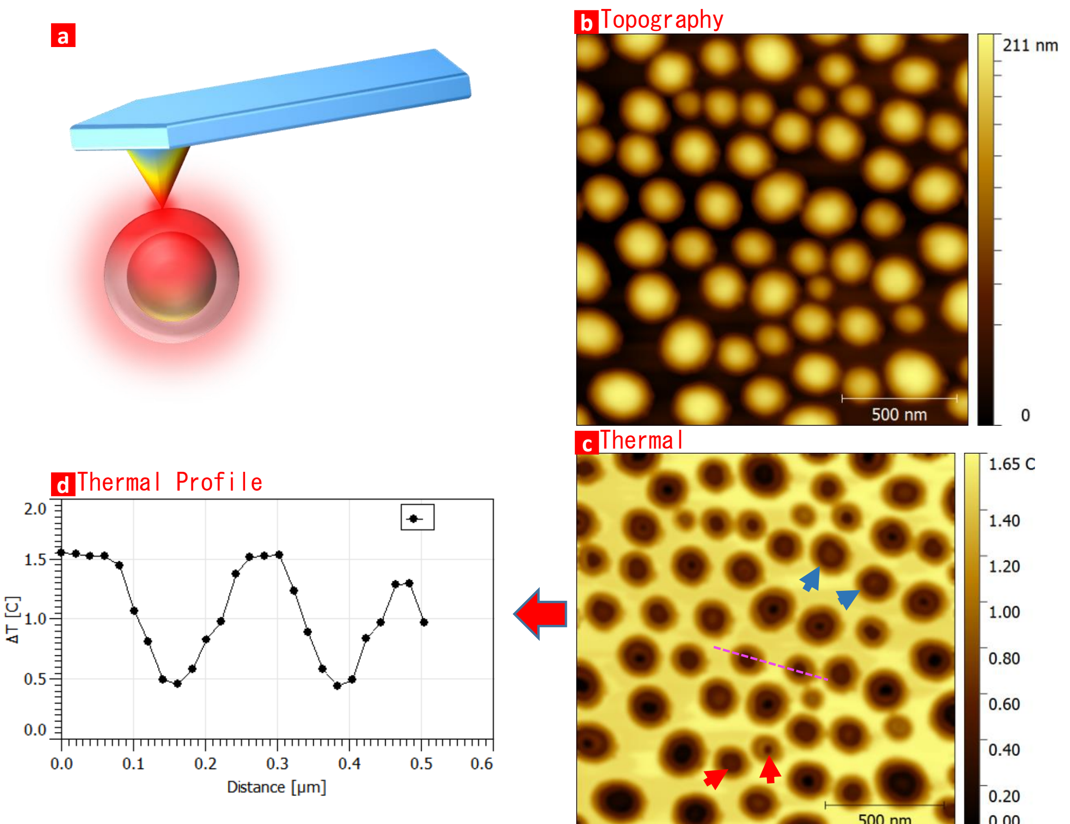
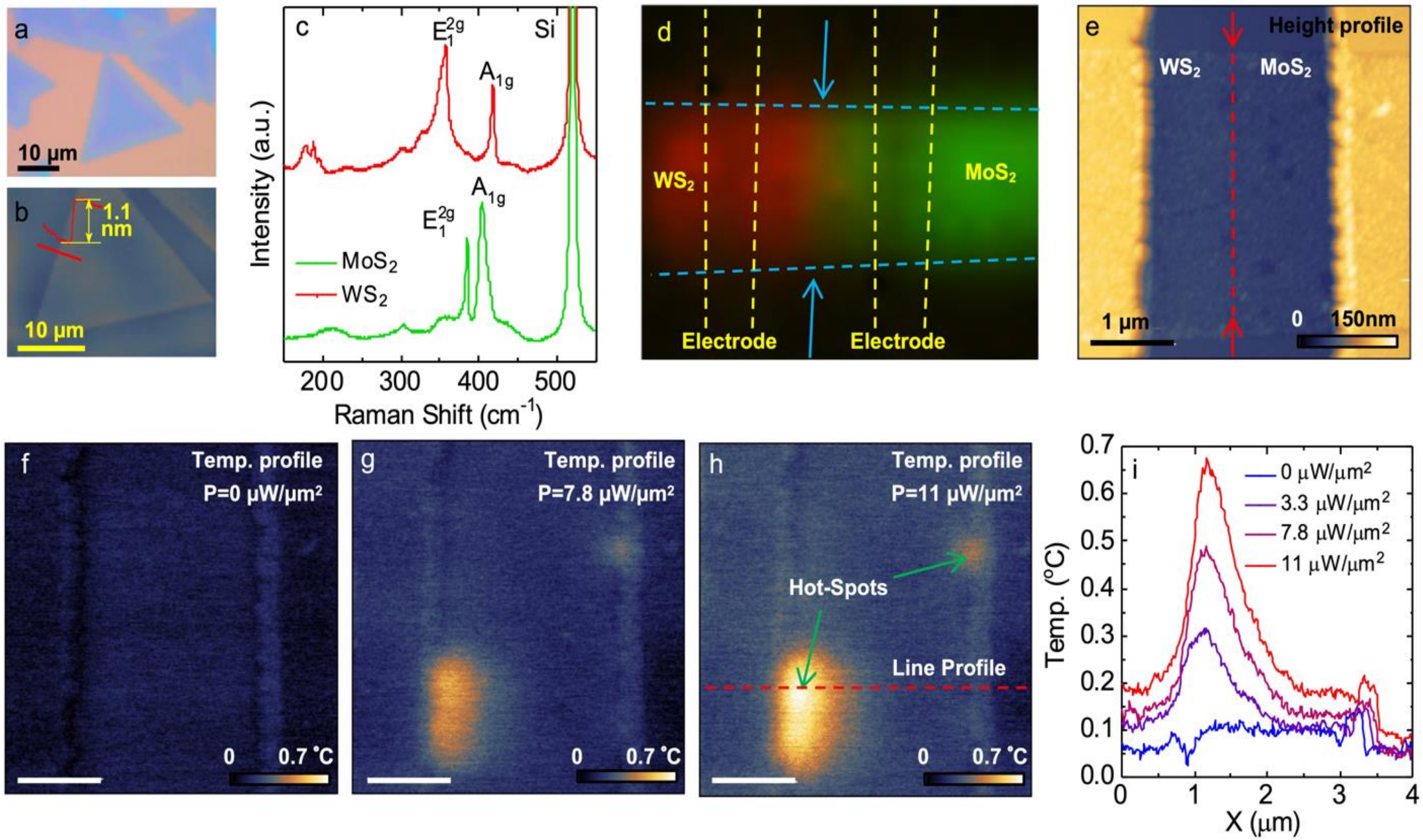
Sub-Surface Thermal Contrast

a) Schematic illustration of thermal probe interaction with gold nanoparticles (GNP) encapsulated in silica shell. b) Shows AFM topography image and (c) shows a remarkable thermal contrast from embedded GNP in silica. It clearly reveals a high thermal sensitivity, lateral resolution and contrast. The thermal image showing the difference in heat transfer from the tip to the silica shell and silicon substrate. d) Shows the cross-sectional profile where temperature change (ΔT) from 0.8-0.9°C was recorded across the particle



ACS Nano, **2018**, *12* (2), pp 1760–1767

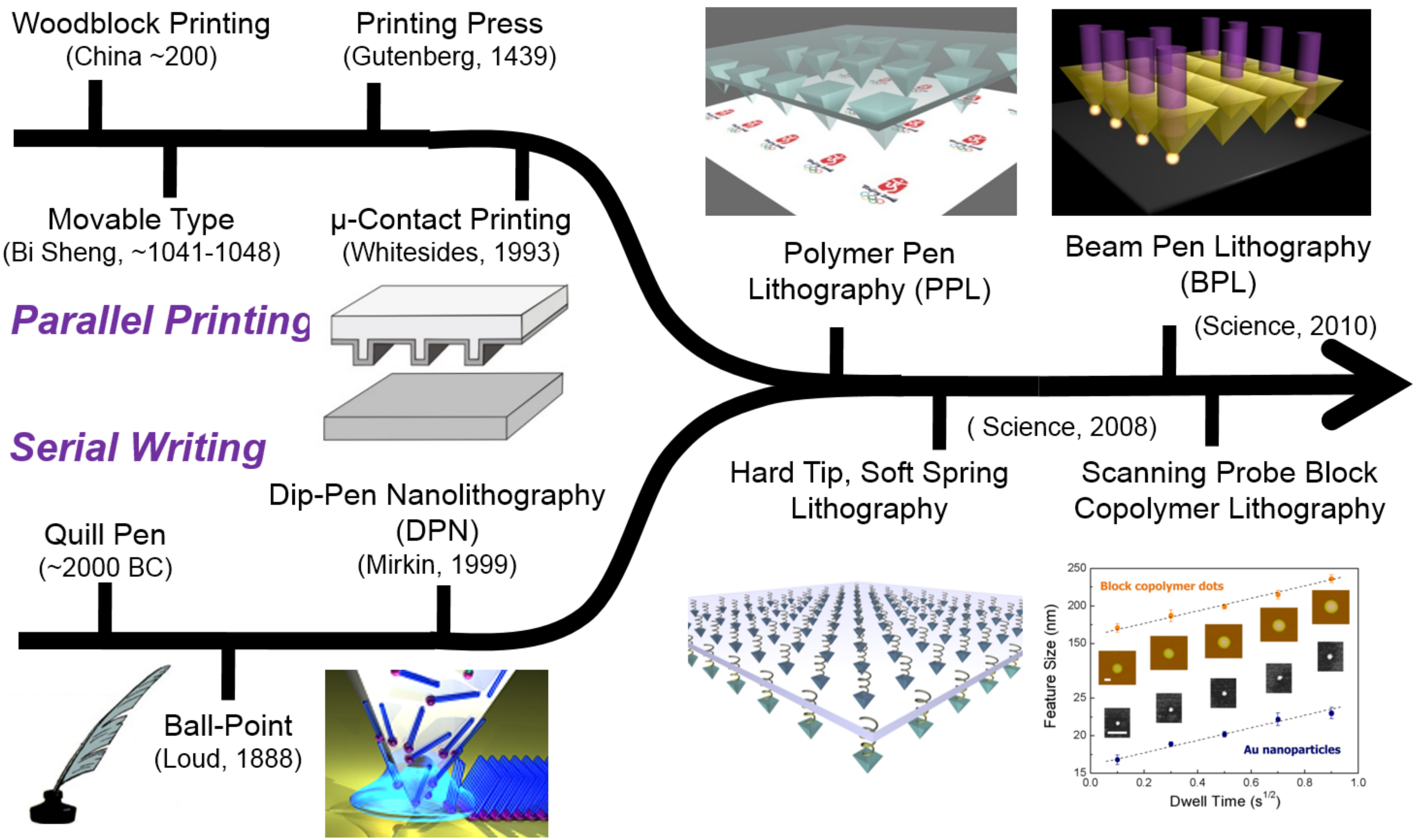
Mapping Hot Spots in Layered Materials



(a) Optical and **(b)** AFM images obtained from a MoS_2-WS_2 heterostructure. **(c)** Raman spectra obtained from MoS_2 and WS_2 regions. **(d)** Raman map of the MoS_2-WS_2 heterostructure device. **(e)** AFM topography image of the same device. **(f-h)** Temperature rise profiles of the device at different dissipated electrical power at V_G = +60V. The heating predominantly takes place on the WS_2-metal vertical junction and the lateral interface does not contribute to localization of the heat. The green arrows in (h) shows the position of the formed hot-spots. **(i)** Temperature line profiles on the dashed red line in (h) at different applied powers.

Adcanced Materials (Under Review)

Soft Nanopatterning



Courtesy: Chad Mirkin

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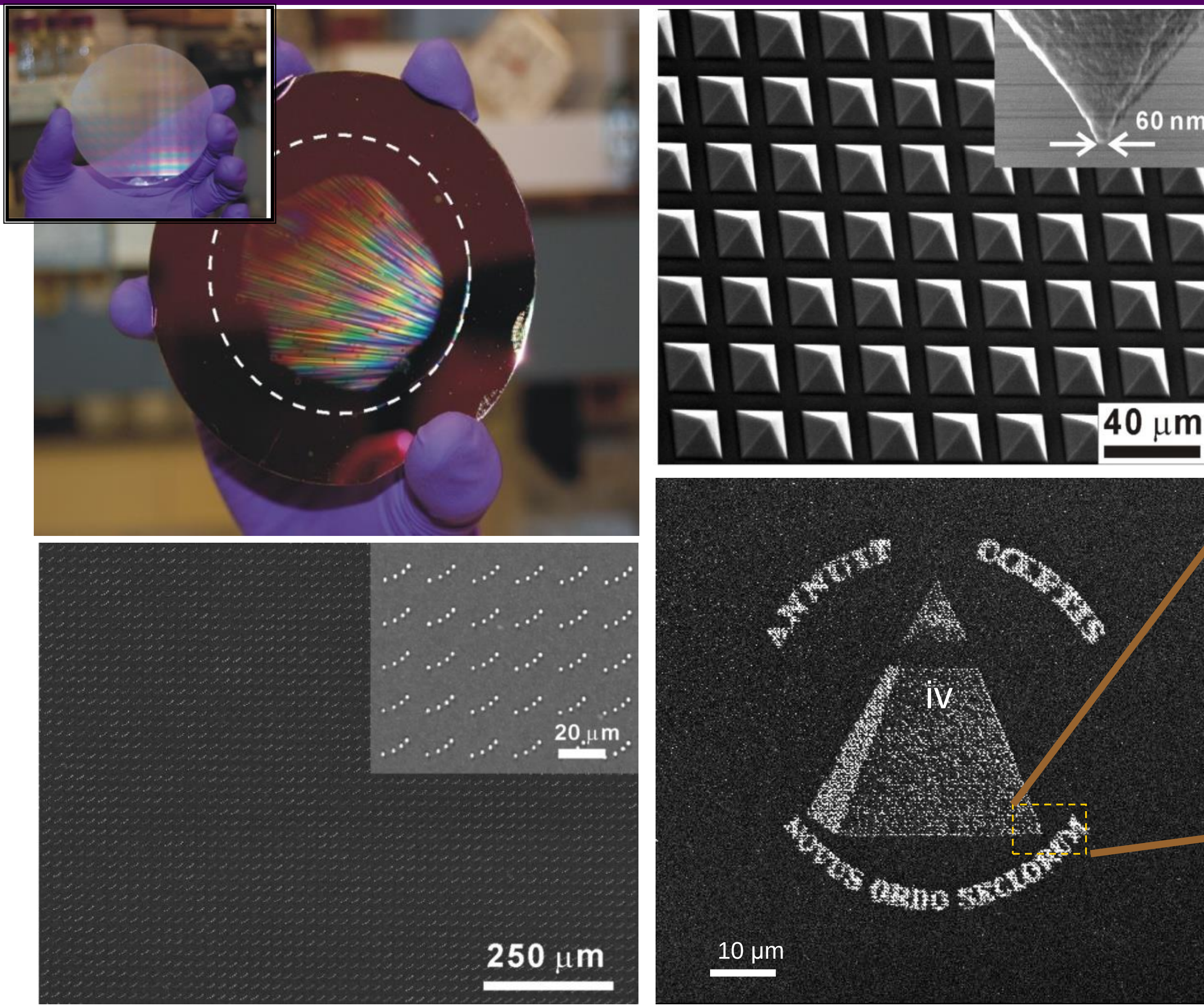
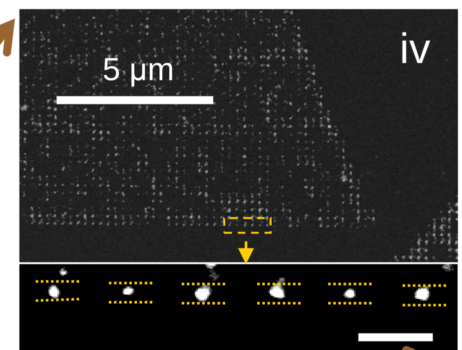
11 Million Polymer Pen Array



Average Feature Size = 42 nm



Courtesy: Chad Mirkin

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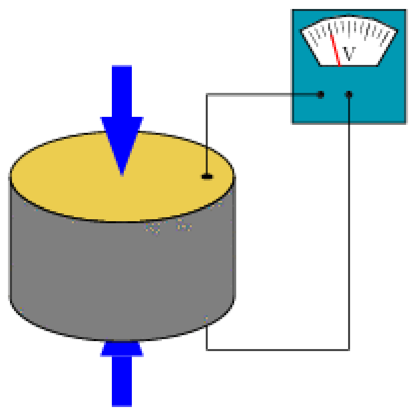
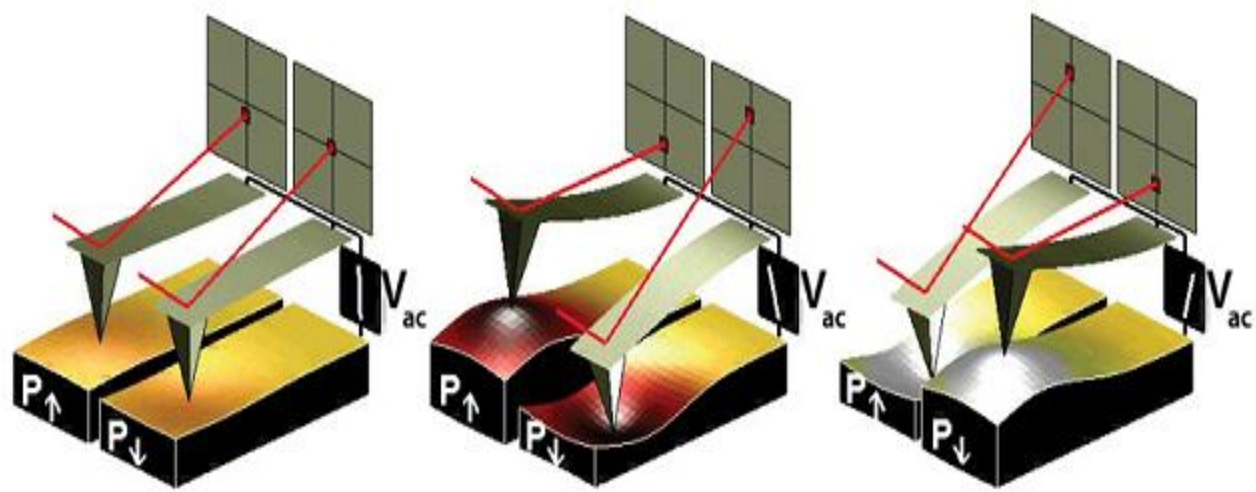
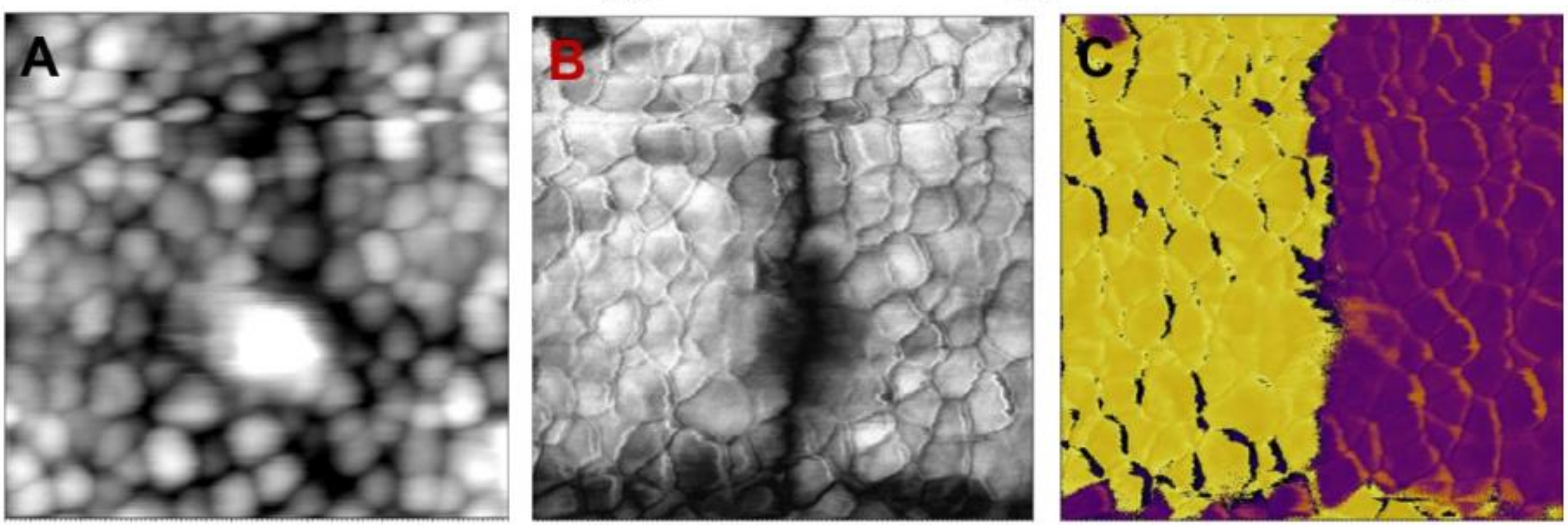
Piezoresponse Force Microscopy

- Piezoelectric Materials



- Piezoresponse Force Microscopy





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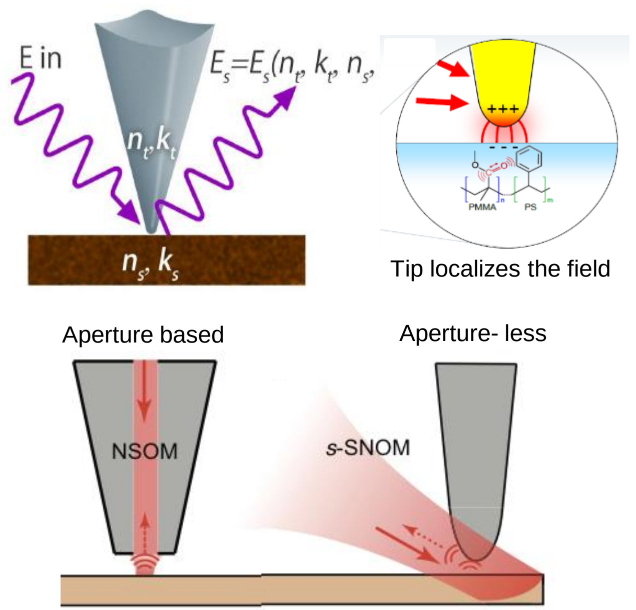
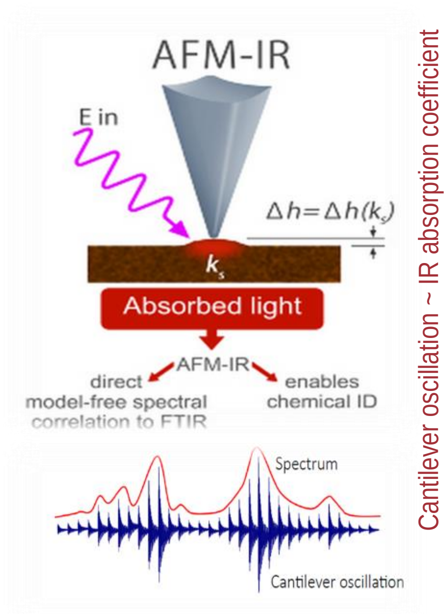
Nano IR System Integrated with AFM and sSNOM (Coming soon!)

Chemical Analysis at nanoscale resolution



Scattering near field optical microscope (sSNOM). This aperture-less system collects scattered energy form the near field resulting in sub-20 nm resolution. In comparison, aperture based traditional NSOM system resolution is limited to 100-150 nm.



- 10's of nanometer optical and sub-eV spectral resolution
- Near-field spectroscopy (nano-FTIR)
- High speed optical near-field imaging
- Simultaneous optical amplitude (reflection) and phase (absorption) measurements
- VIS-IR-THz spectral range.