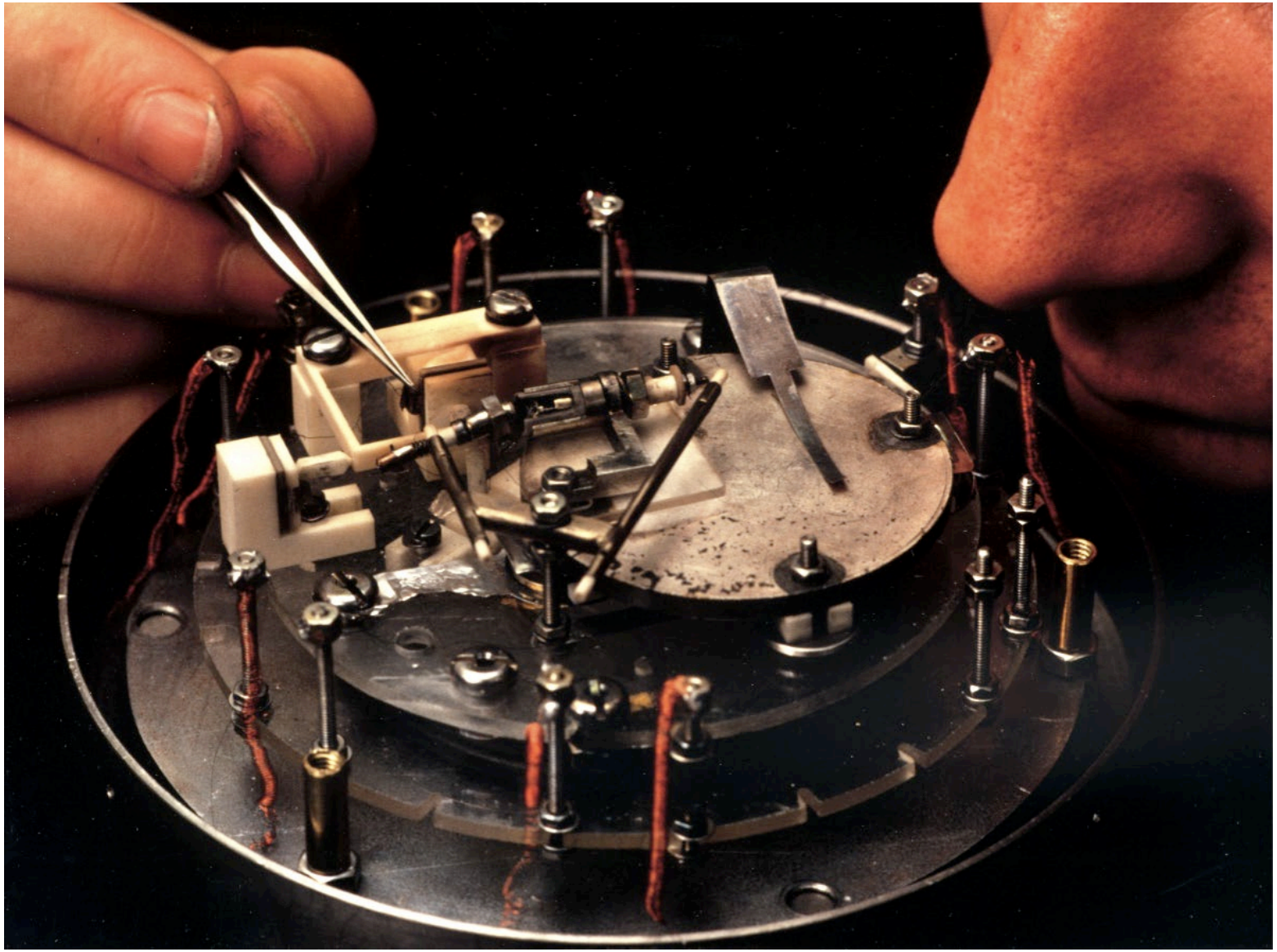
Advances in Scanning Probe Microscopy

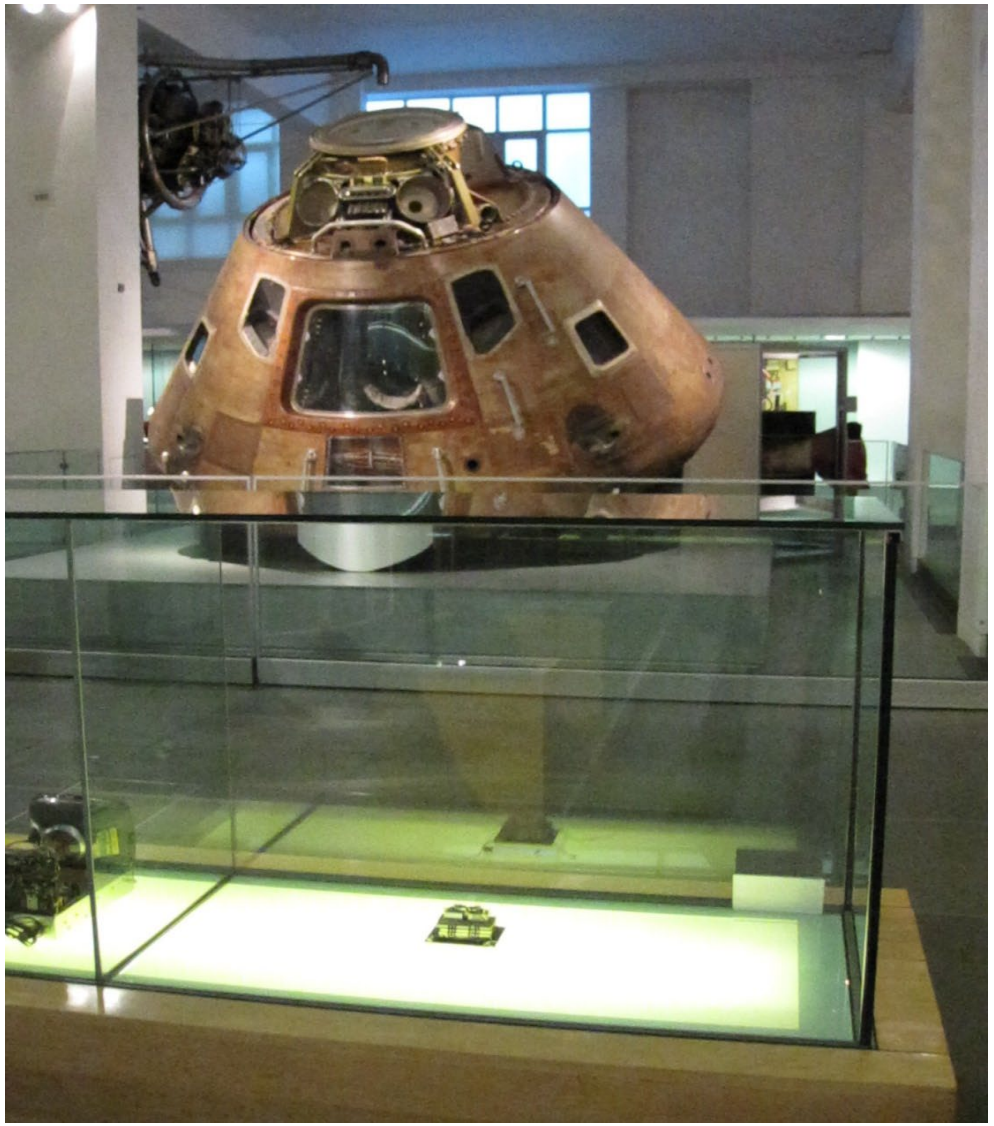
Gajendra Shekhawat
Research Professor
Department of Mat. Sci. & Eng.
SPID Manager, NU*ANCE* Center
Northwestern University


Graphene
on scotch tape !



Science
Museum London

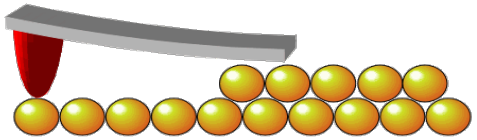
„The Making of
the
Modern World“



Original AFM

Conventional Scanning Probe Microscopy

contact mode



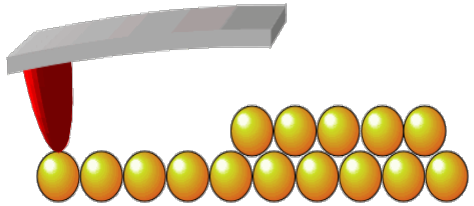
Tip angstroms from surface (repelled)

Constant force

Highest resolution

May damage surface

Tapping mode



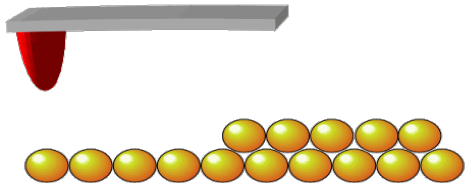
Intermittent tip contact

Variable force measured

Improved resolution

Non-destructive

non-contact mode



Tip hundreds of angstroms from surface (attracted)

Variable force measured

Lowest resolution

Non-destructive

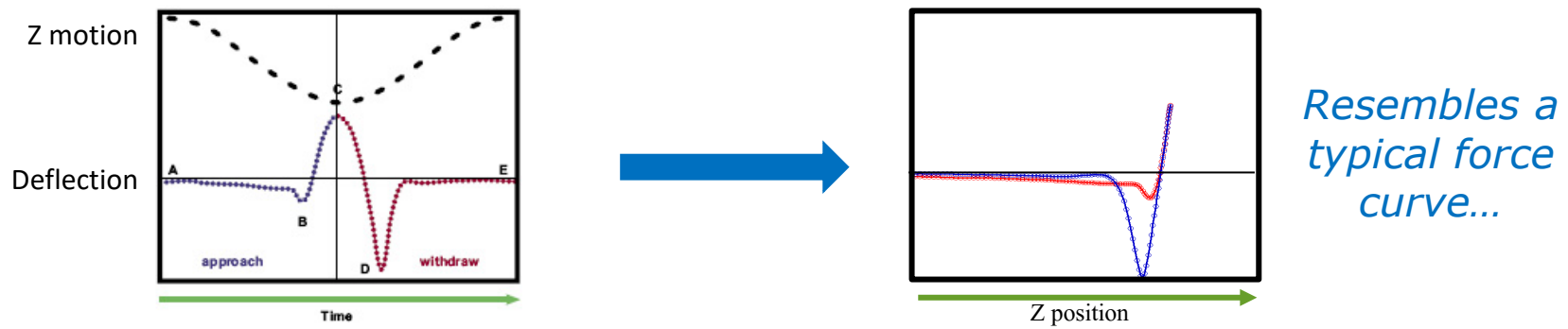
Functional Advances in Scanning Probe Microscopy

Not Only Topography

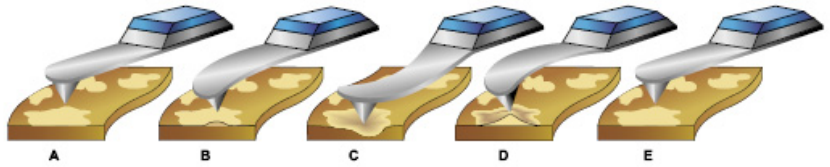
Electrical Properties	Magnetic Properties	Mechanical Properties	Thermal Properties	Chemical Properties
• Conductivity • Charge • Resistivity • Impedance • Photo-conductivity • Piezo-electricity • Workfunction	• Magnetic domains	• Friction • Adhesion • Stiffness • Modulus • Visco-elasticity • Hardness	• Thermal conductivity • Temperature • Melting point	

Peak Force Tapping Technology

Controls and measures force as feedback



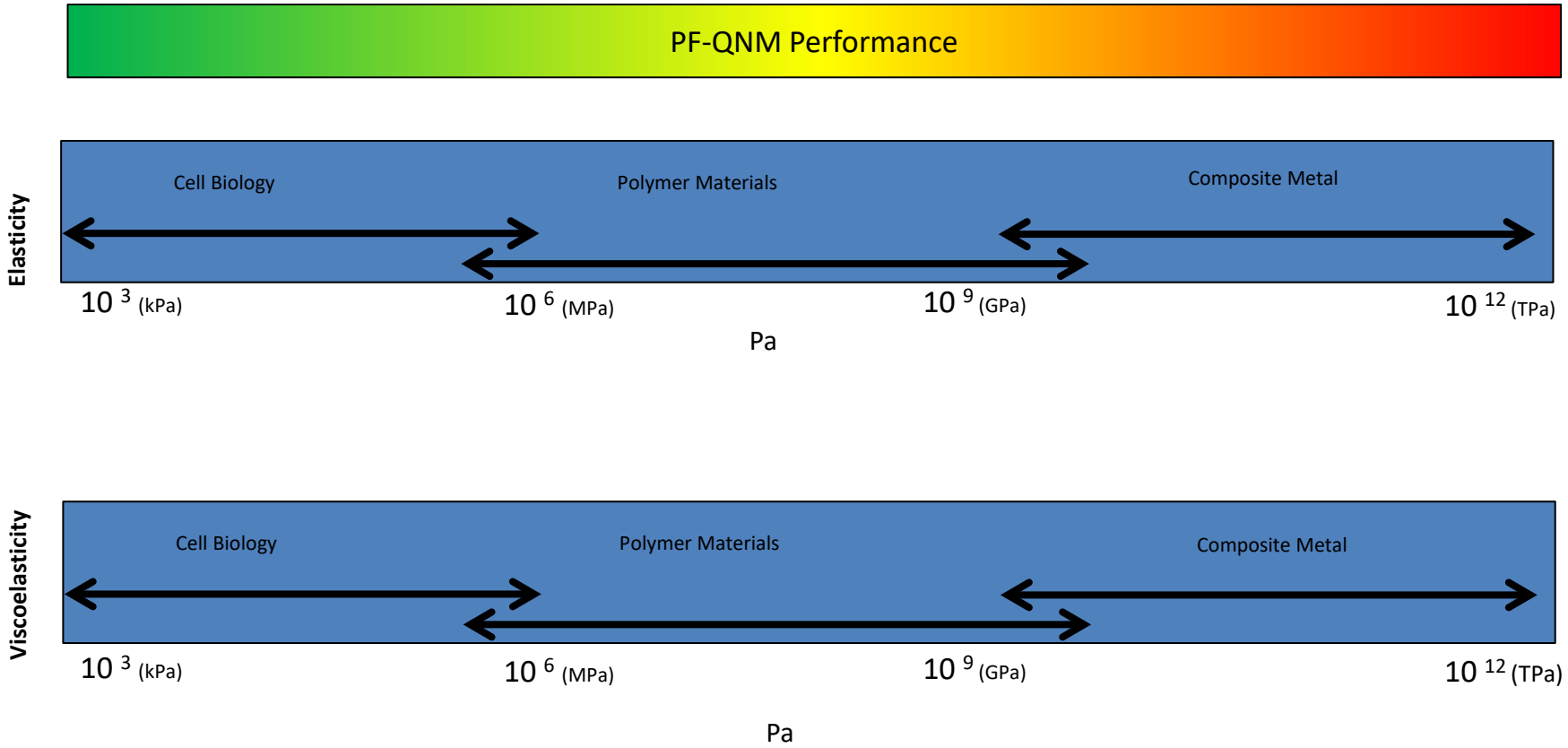
Resembles a typical force curve…



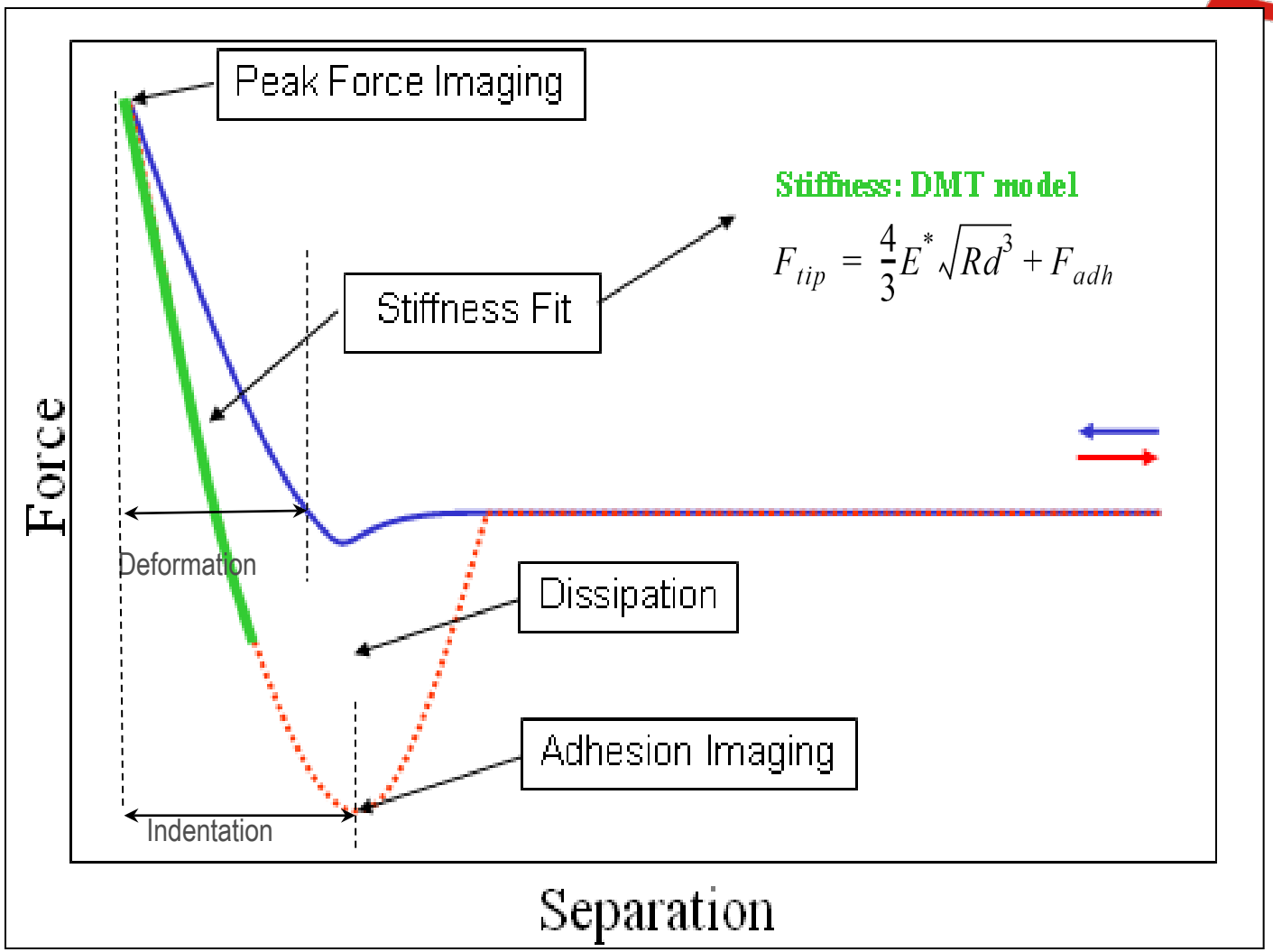
Peak Force Tapping Mode:

- Probe modulated at small amplitudes at low frequency (1-2kHz).
- Feedback signal is peak force between tip and sample.
- Direct control of imaging forces with ultra-low set points (<100pN).
- Images acquired at typical scan rates (1000's force curves/sec).

Peak Force QNM: Complete Solution for Nanomechanical Characterization



PeakForce QNM

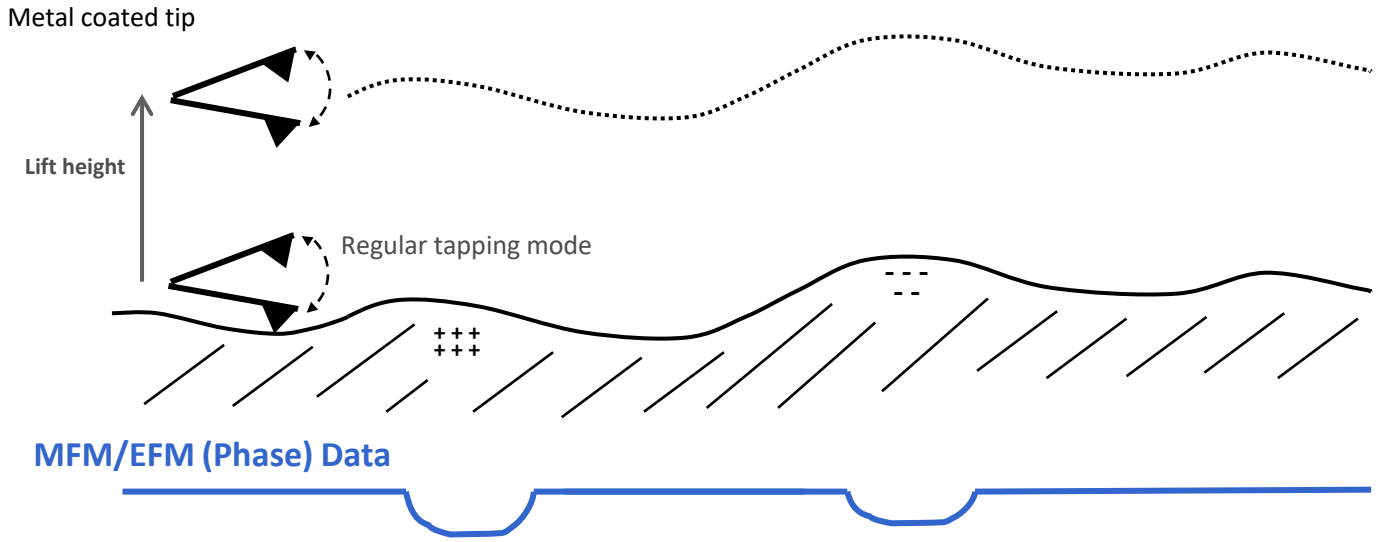


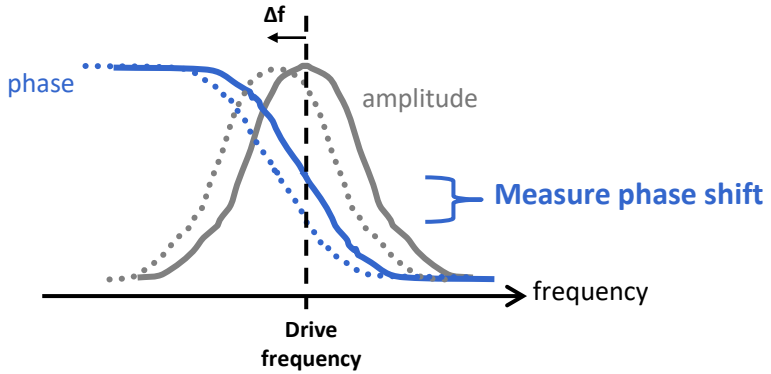
Simultaneously obtain quantitative data:

- Topography
- Modulus
  - <10kPa–100GPa
- Adhesion
- Energy Dissipation
- Deformation
- Indentation

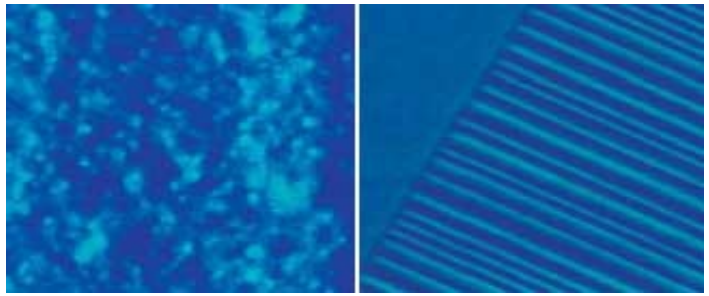
Magnetic Force Microscopy

Lift Mode





Topography & Magnetic Phase of floppy disc (100 µm scan)



Electrical Properties


Tapping mode


Contact mode


Peak Force mode


Fast Force Volume

electric fields charges surface potential work function

Electric Field Microscopy (EFM)

Kelvin Probe Force Microscopy (AM-KPFM & FM-KPFM)

HV-KPFM

conductivity resistivity

Conductive AFM (C-AFM)

Tunneling AFM (TUNA)

Photoconductive AFM (PC-AFM)

impedance capacitance resistance

Scanning Capacitance Microscopy (SCM)

Scanning Spreading Resistance Microscopy (SSRM)

Scanning Microwave Impedance Microscopy (sMIM)

piezo-electric

Piezoresponse Force Microscopy (PFM)

HV-PFM

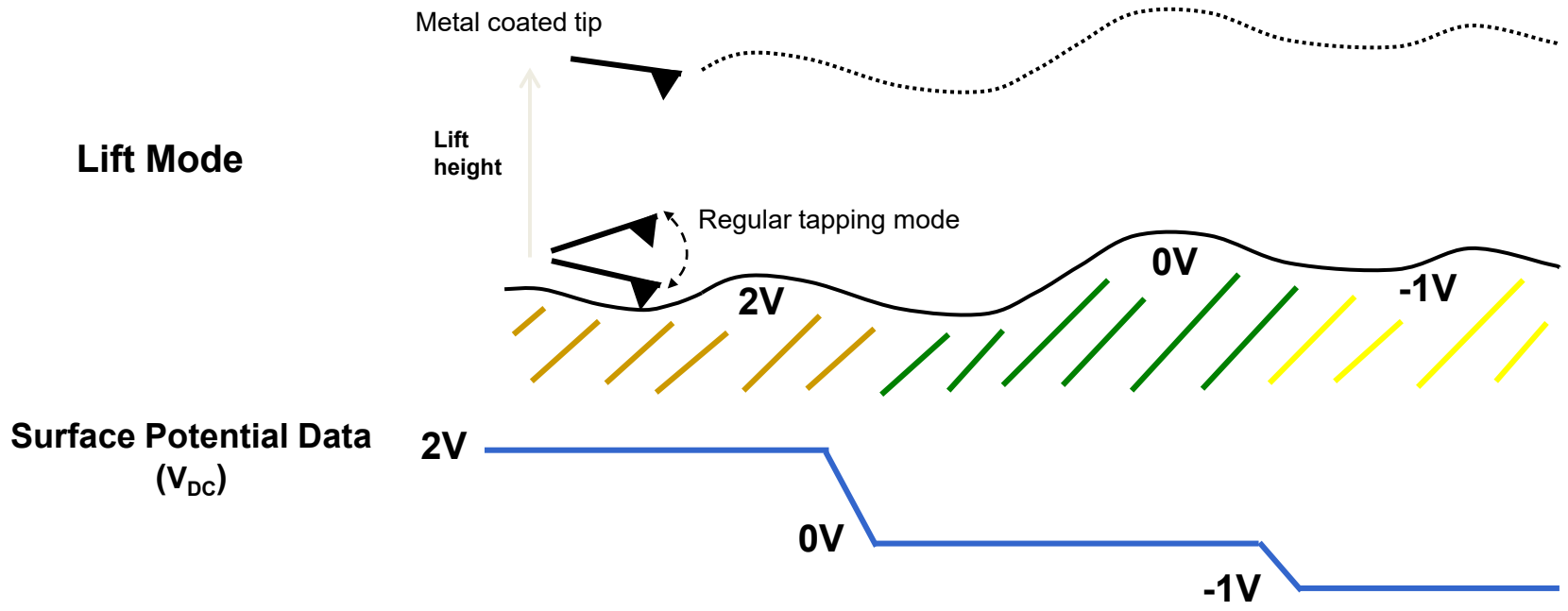
Dual Frequency Res. Tracking PFM (DFRT PFM)

Switching Spectroscopy PFM (SS-PFM)

Scanning Kelvin Probe Microscopy KPFM

Generates a map of electric potential of surface

AM-KPFM
TappingMode AFM with Amplitude Modulation potential detection – Standard version

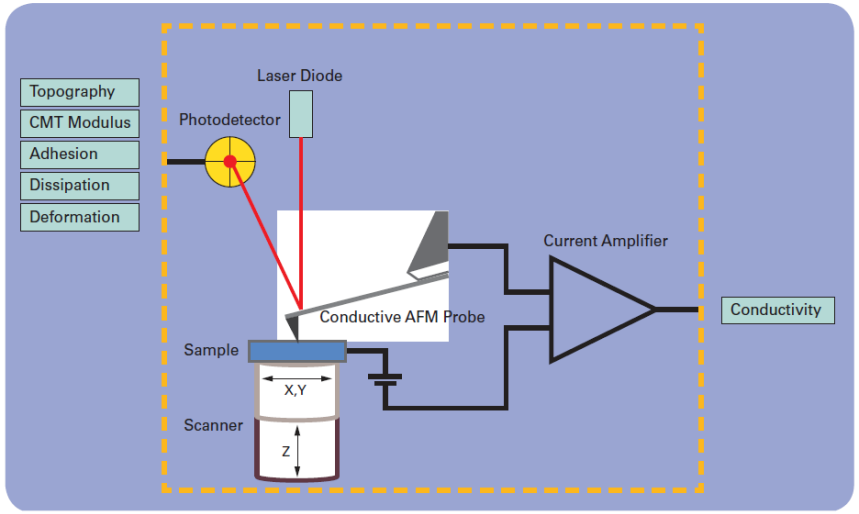


Cantilever only driven with electrical forces during lift line (V_{DC} + V_{AC} applied to tip).

When $V_{DC} = V_{sample}$ cantilever does not oscillate.

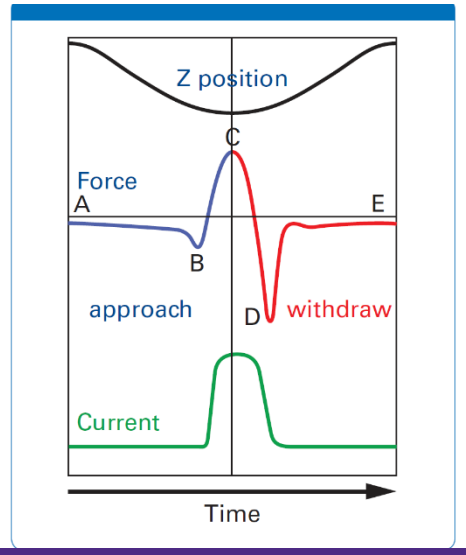
Use feedback to maintain this condition; record V_{DC}.

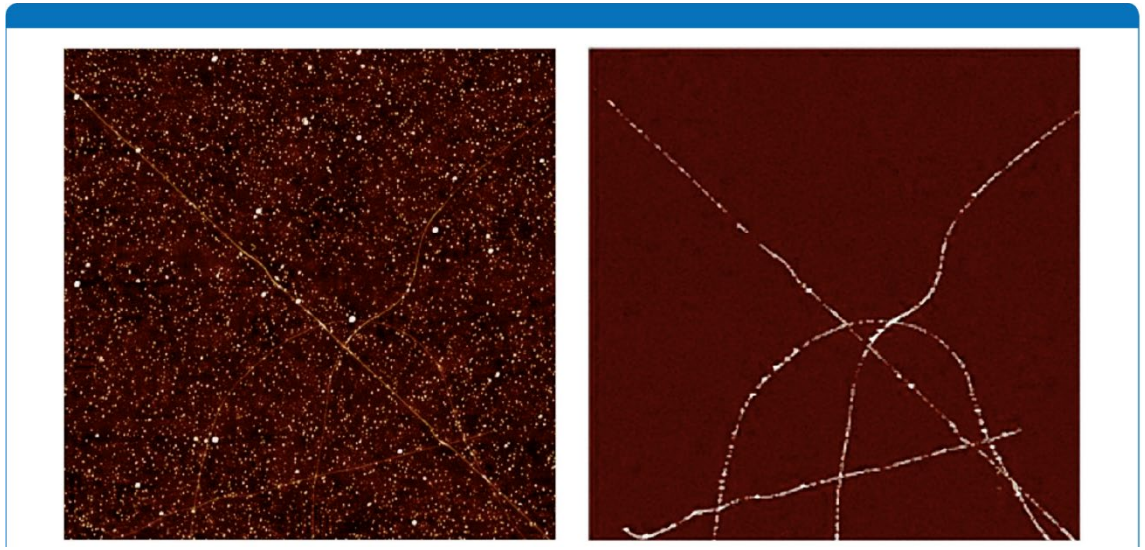
Peak Force TUNA



Peak Force TUNA SPM Platform:

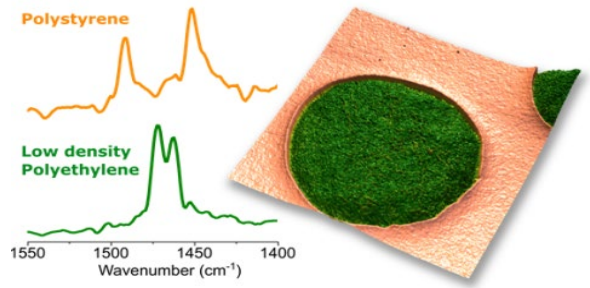
- ➔ **Imaging mode**
  - ➔ **Maps the electrical current and mechanical Properties**
- ➔ **Current Spectroscopy Mode**
  - ➔ **Current voltage curves**



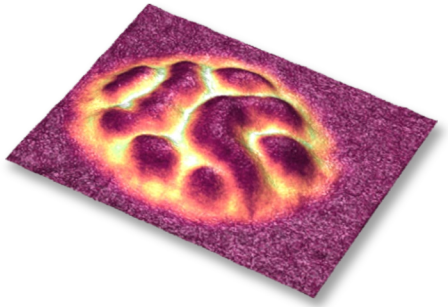


AFM Infrared Spectroscopy & Imaging: *Chemical ID*

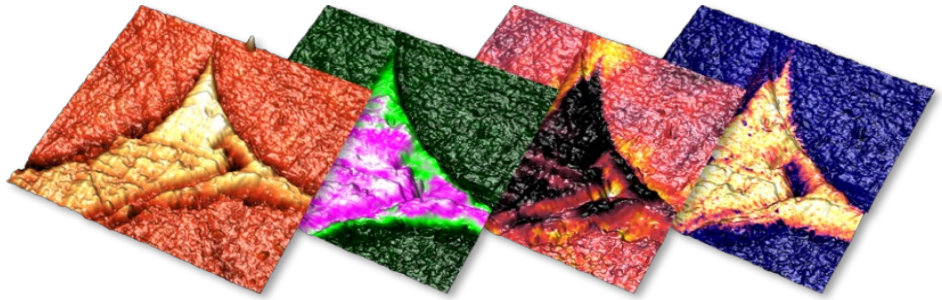
- Highest performance spectra with FTIR correlation
- AFM-IR monolayer sensitivity and surface sensitive measurements
- The most advanced correlative microscopy with PeakForce Tapping modes
- Broadest range of AFM modes and accessories



High performance, sensitive, AFM-IR spectroscopy with correlation to FTIR

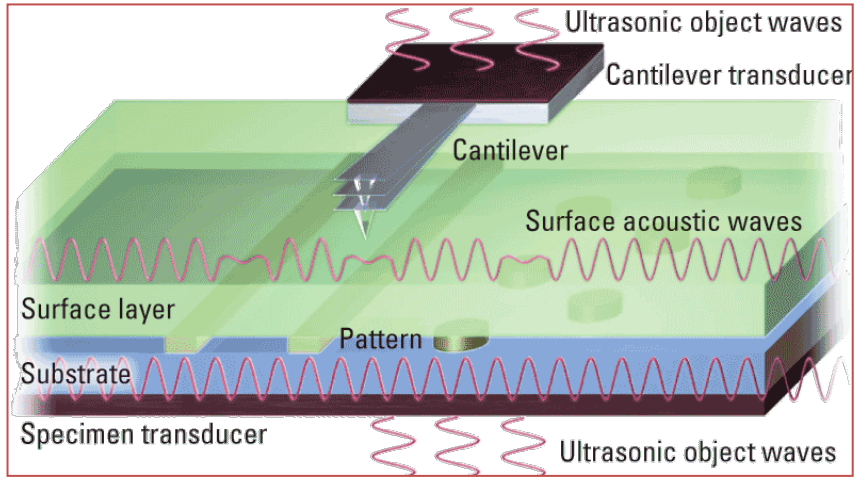


<10 nm chemical imaging spatial resolution & monolayer sensitivity



nanoscale property mapping and correlative microscopy

Advances in Functional SPM: Non-Invasive Imaging using Ultrasonic Waves



Near-Field SPM Platform:

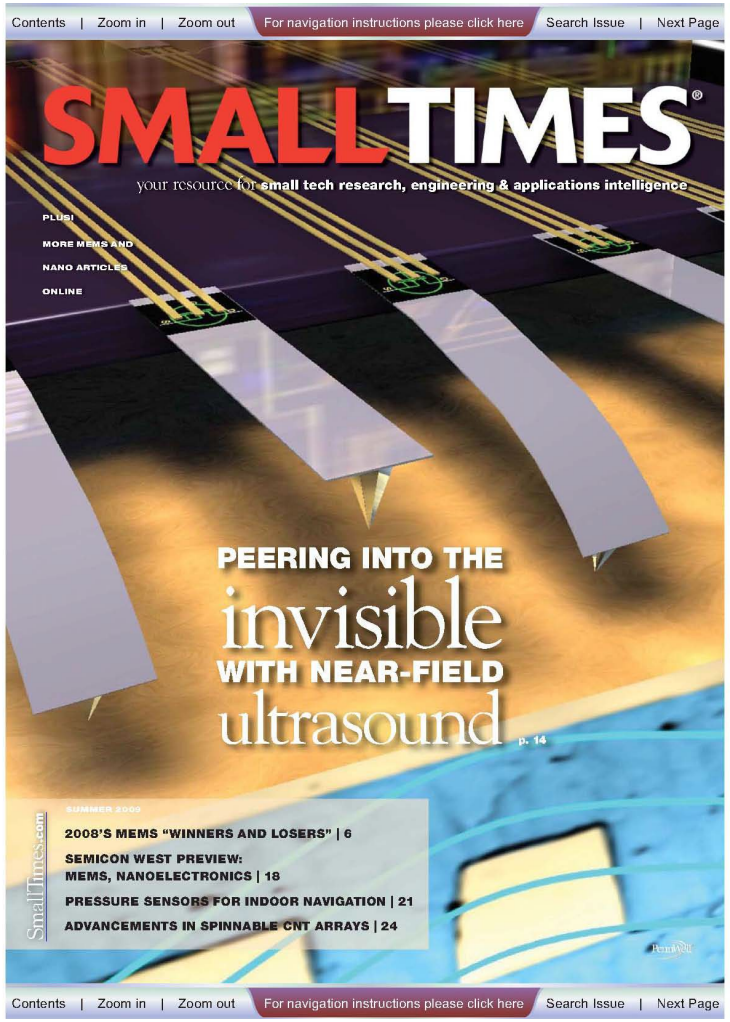
➔ **Excellent Lateral Resolution**

Ultrasound source:

➔ **Non-destructive and Depth-Sensitive**

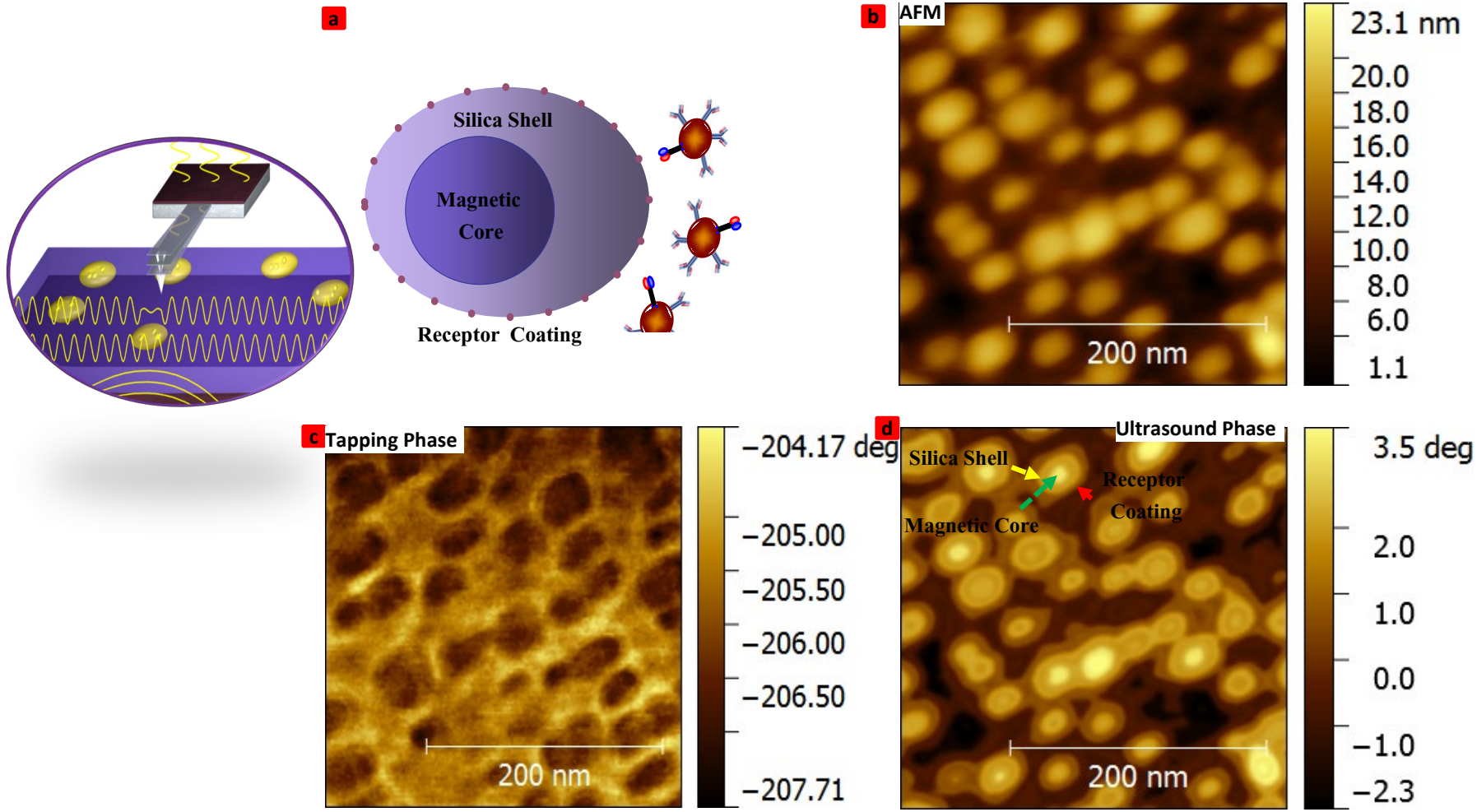
Holography Paradigm:

➔ **Sensitive to "*Phase*" Perturbations**



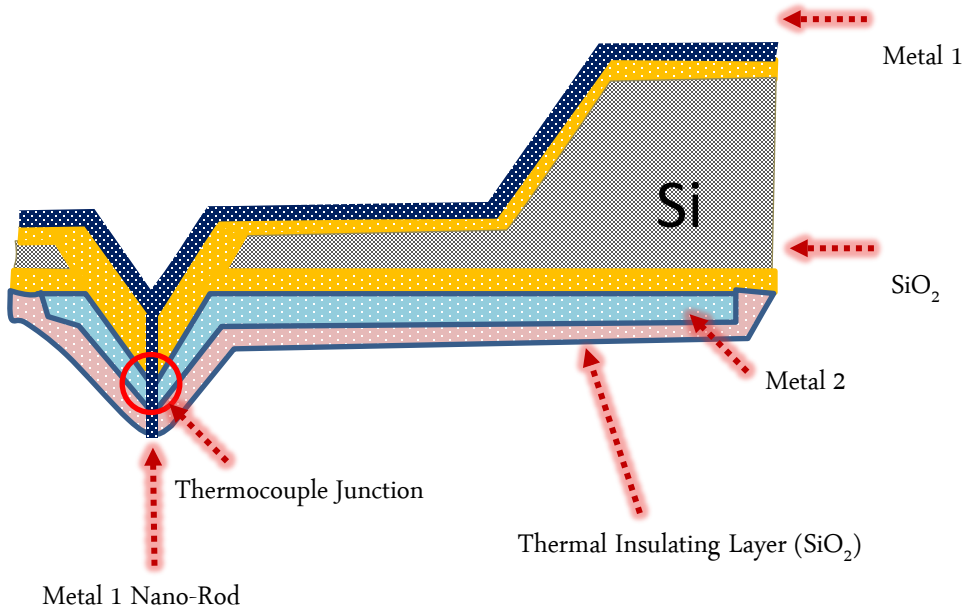
Science 310, 89 (2005), Nature Nanotechnology (2008 and 2009)

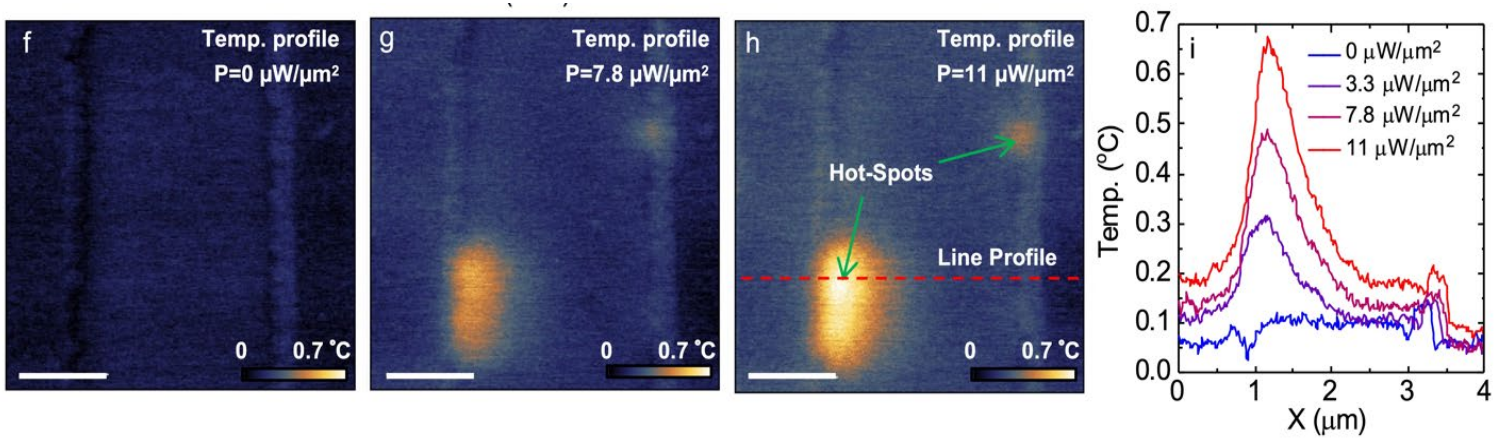
Sub-Surface Nanomechanics



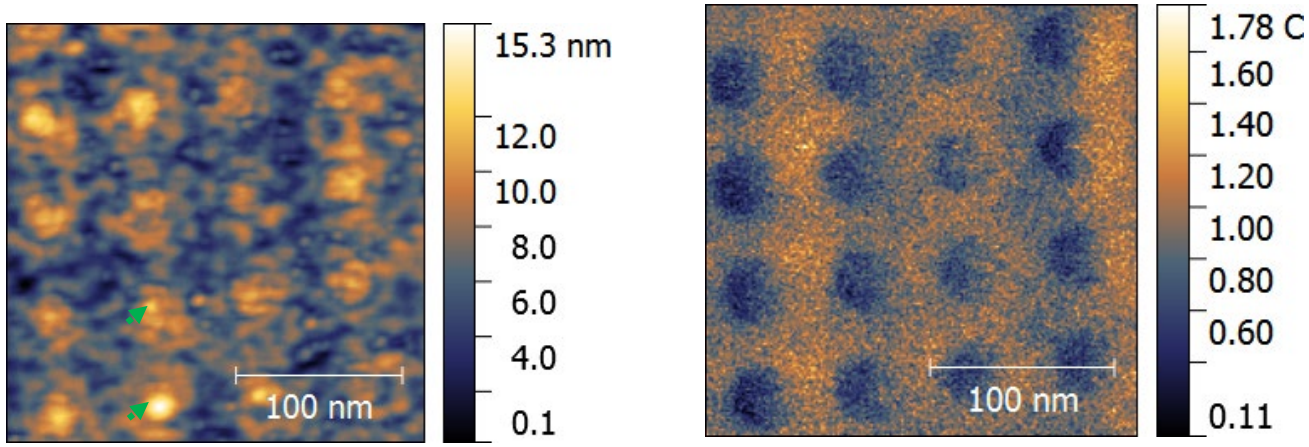
Science Advances 2017: 3;e1701176, Nature Scientific Report 8 (1) 1002 (2018), Nature Scientific Report 7, 14152 (2017)

Thermal Imaging using Nanomechanical Probe



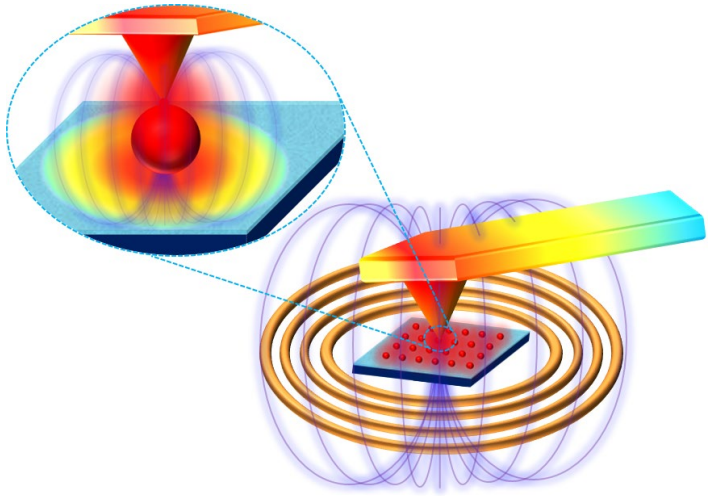


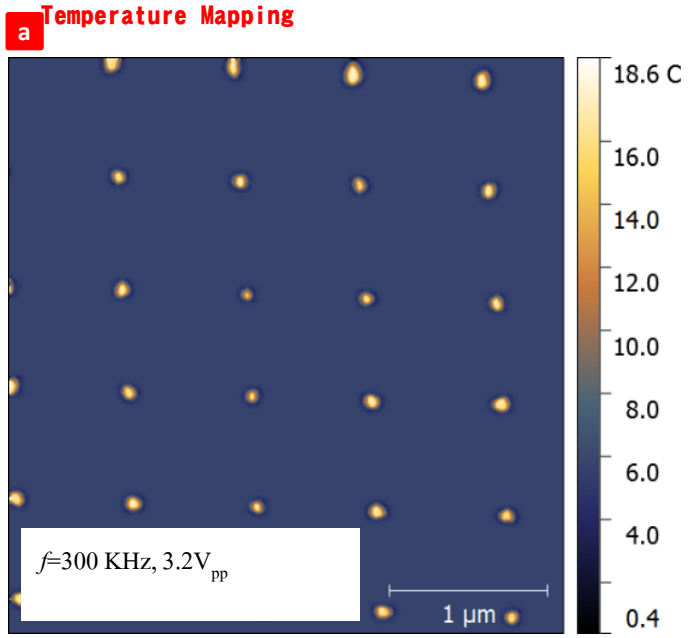
Advanced Materials 31, 24 (2019)

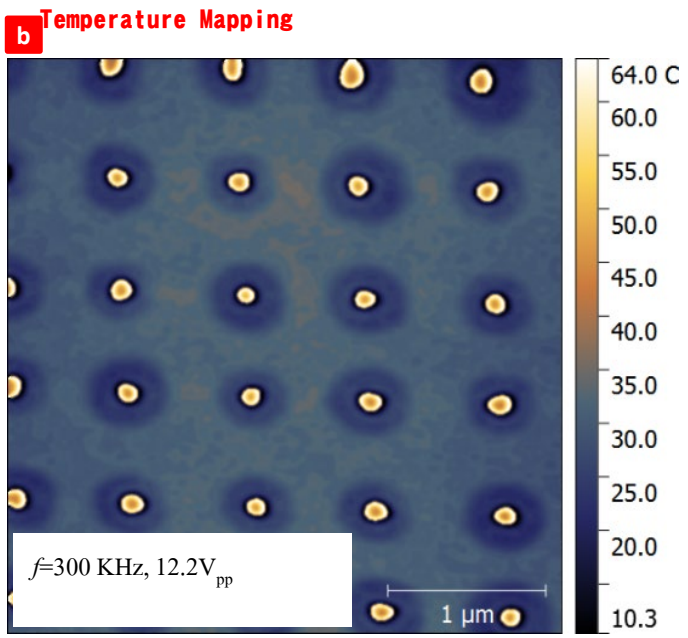


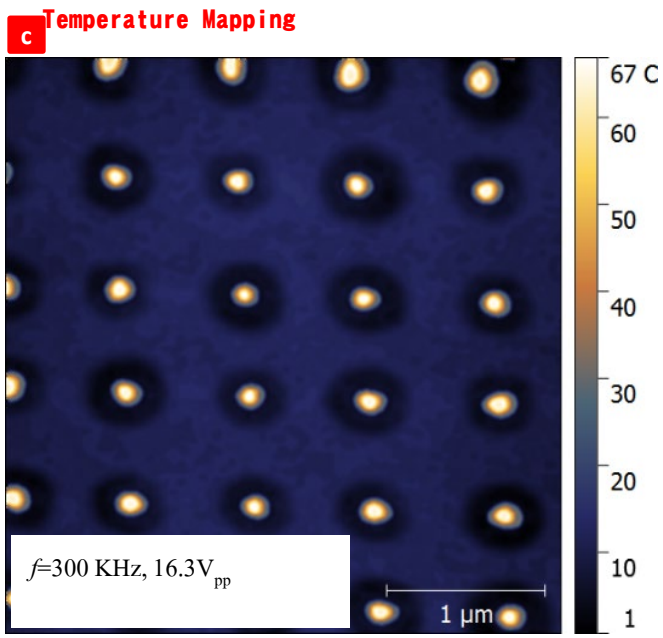
ACS Nano 12, 2 (2018)

Temperature Mapping

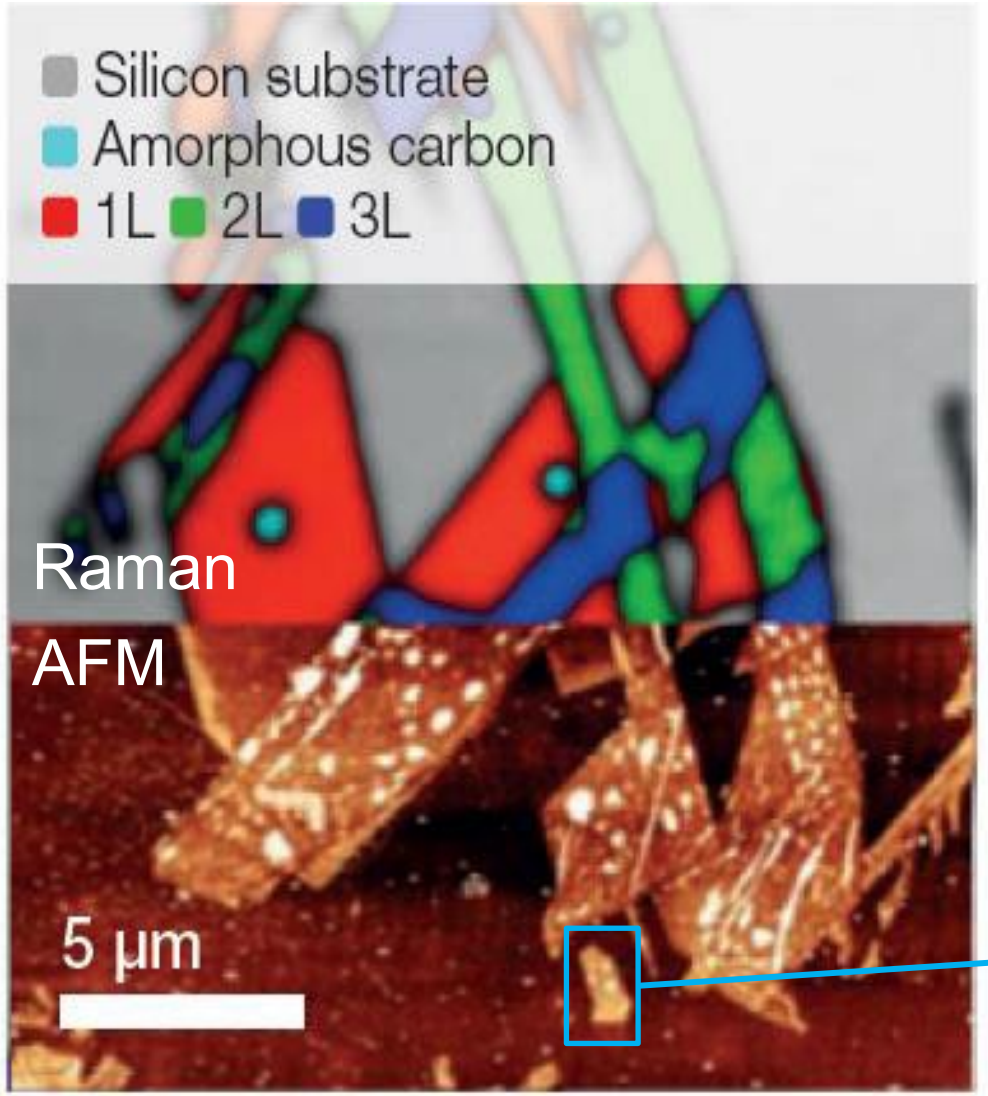




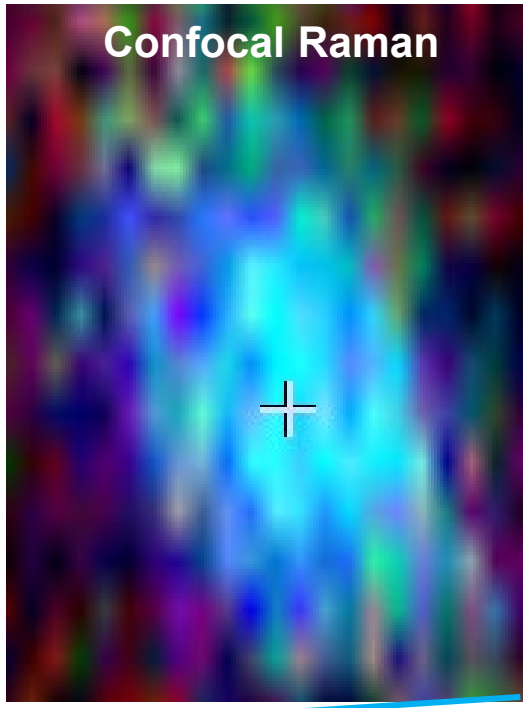


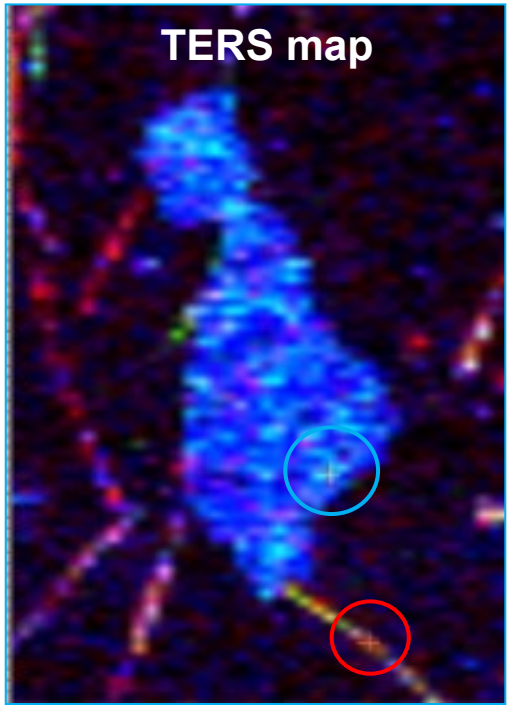


Bringing AFM together with Raman/PL



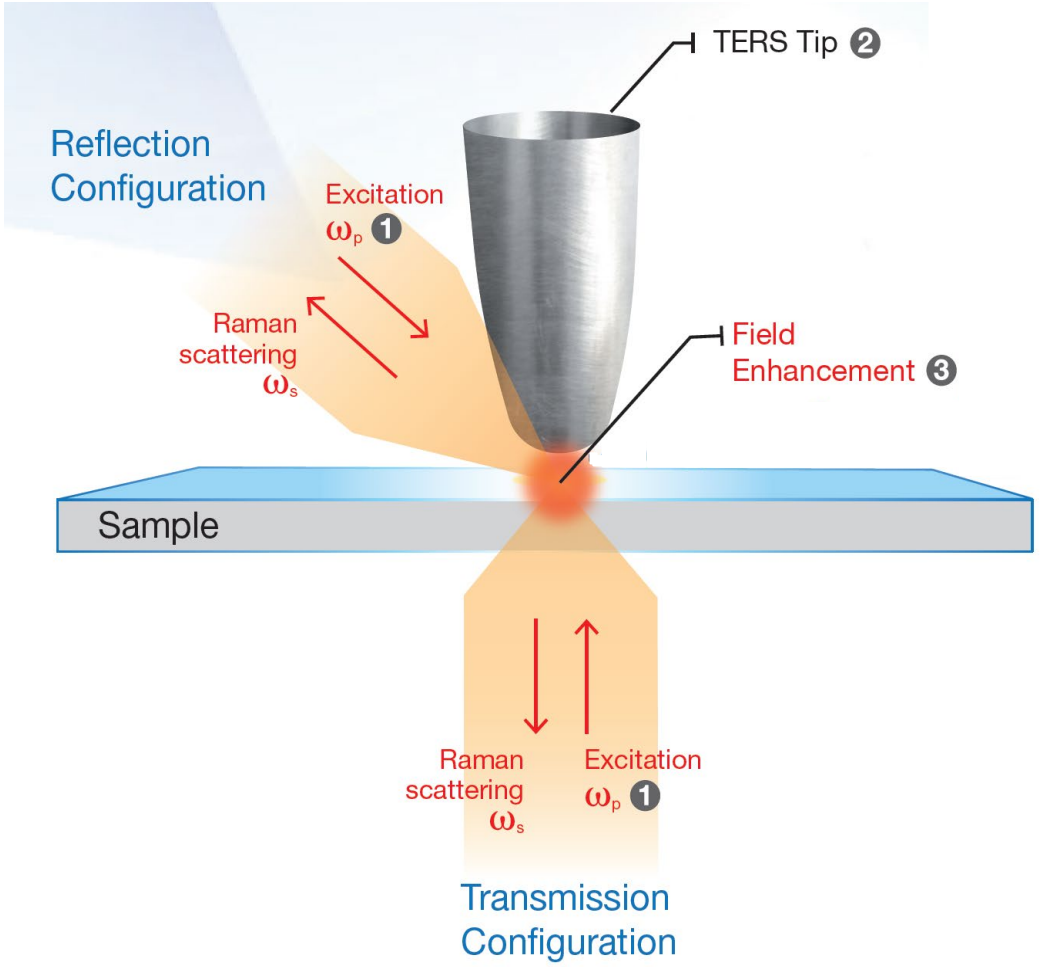
Confocal Raman and TERS of the same area, graphene oxide and CNTs



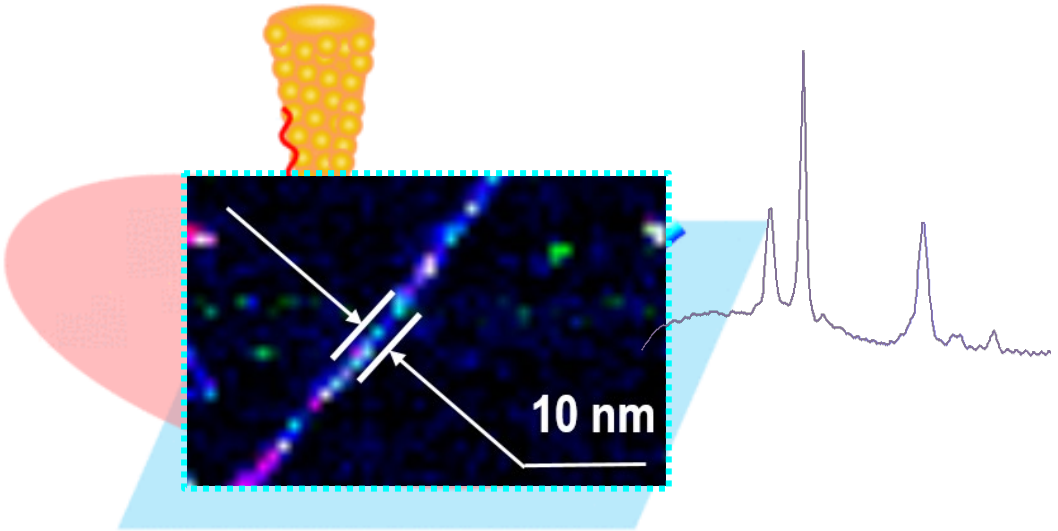


How is it possible?...

Tip-enhanced Raman Spectroscopy (TERS)



- Combine AFM, Raman, and LSPR effect to get nanoscale spatial resolution and surface analyte detection!
- Confine plasmon resonance effect to nano-sized "hot spot" at tip-sample junction.
- Requires right instrument configuration, noble metal tip and substrate, and very thin (1-2 nm preferable) sample.



Amplification of Raman signal by 10^{5-7} in TERS

J. Phys. Chem. B 110, 6692, 2006
Optics Express 21, 25271, 2013

TERS and TEPL for materials research

AFM provides:

- Topography
- Adhesion / stiffness
- Surface potential
- Conductivity
- Capacitance (charge carrier concentration)
- Photocurrent

Optical spectroscopy:

- Structure, defects (Raman)
- Electronic band structure (PL)
- Mechanical strain (Raman peaks shift)
- Doping (PL and Raman)
- Photocurrent

TERS/TEPL

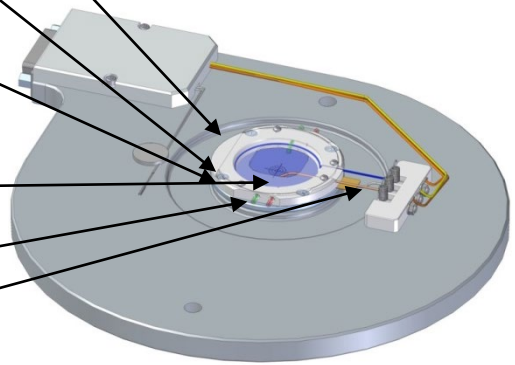
all this at the nanoscale

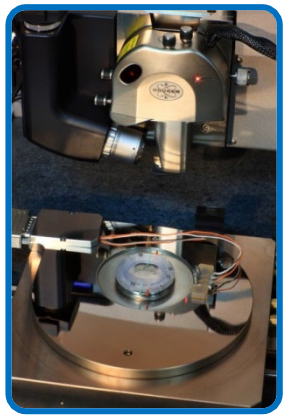
Available lasers for TERS & TEPL:
532, 473, 633,785

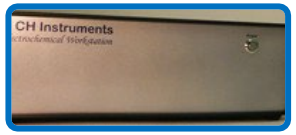
Electrochemical Techniques (EC-AFM

- Complete solution, highly chemically inert, RT to 60C
- Bruker unique SECM capability for sub-100nm local EC and electrical measurements in liquid

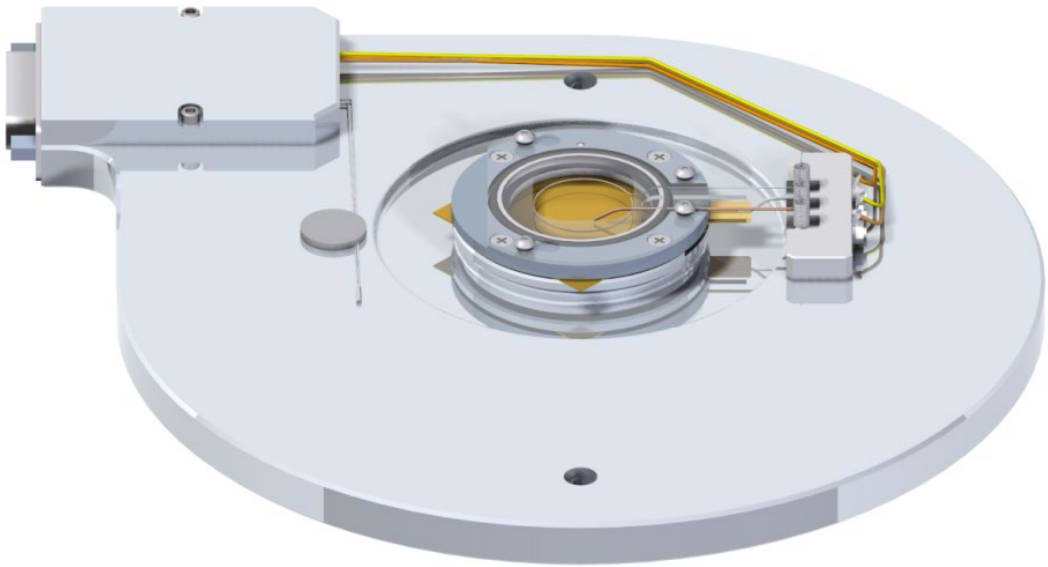
- Integrated sample heater and thermocouple for temperature control
- Kel-F body for Li battery research
- Teflon body for GP EC research
- Vapor seal ensuring solvent compatibility and instrument protection
- Perfusion option for bio applications
- Flexible electrode connections

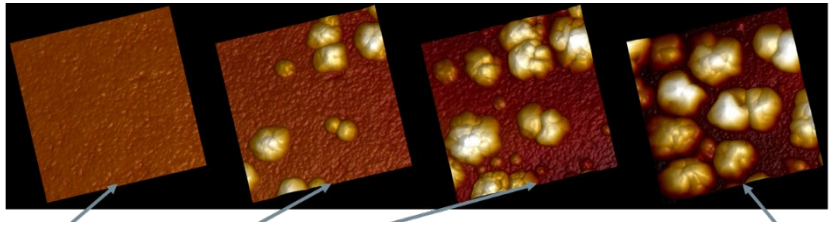






Model 760D Bipotentiostat





Cu Deposition on Glassy Carbon In $CuSO_4$ solution