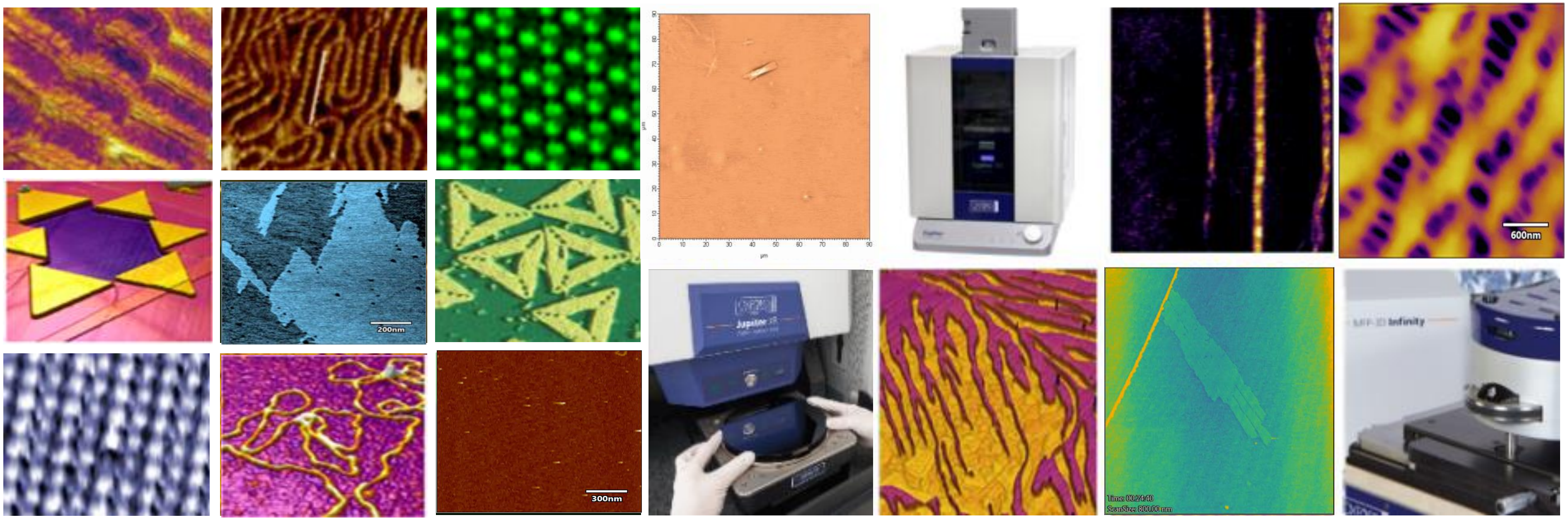
Asylum Research

The Technology Leader in Atomic Force Microscopy



Julien Lopez

AFM beyond topography

GNMEBA Jussieu–2019 December 3rd

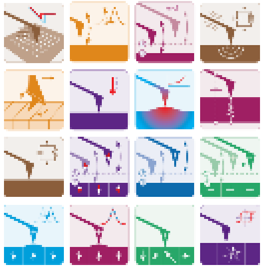
Outline

AFM user?

Goals

Company History

What is an AFM?



AFM Modes

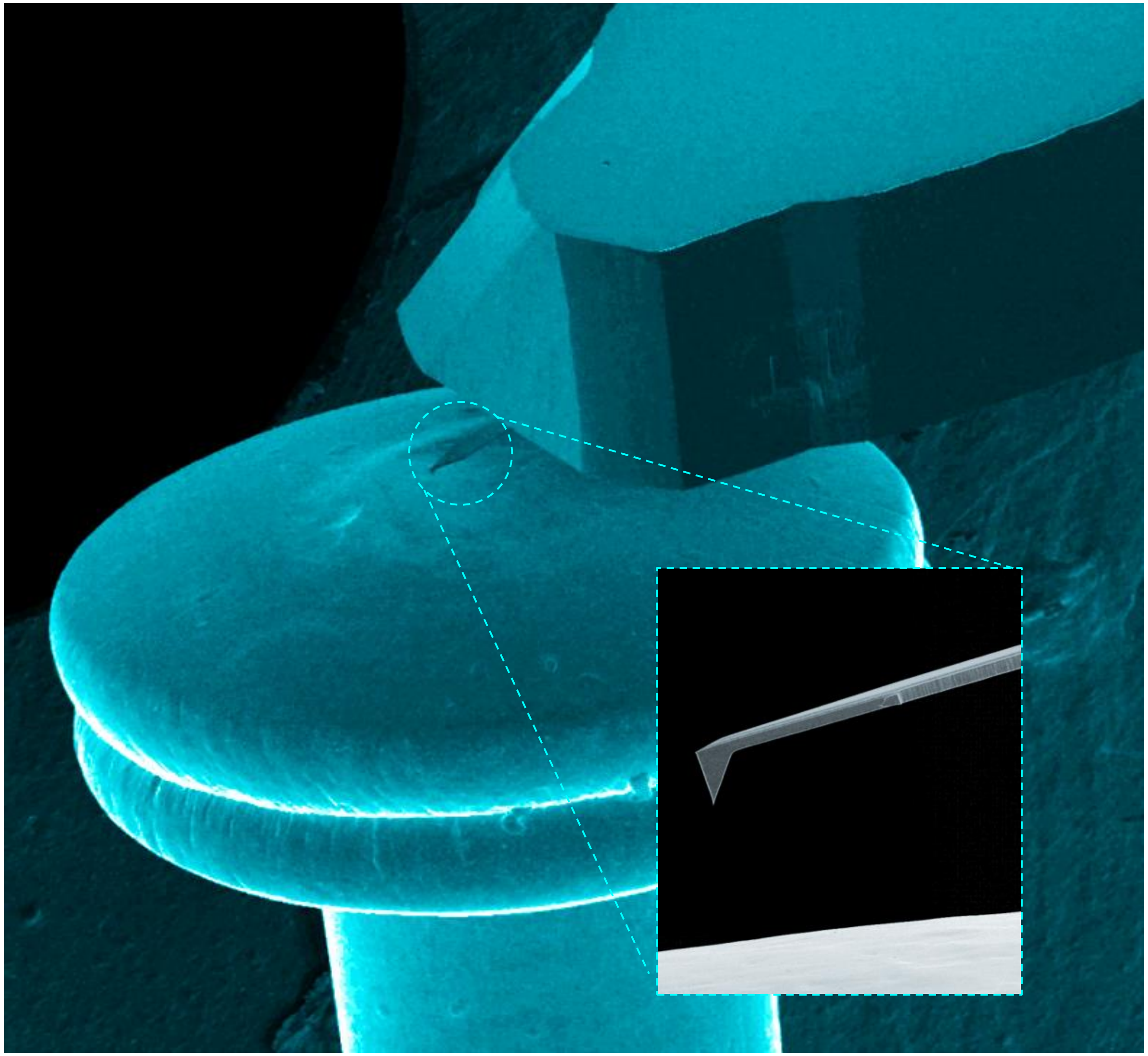
- Basics
- Electrical Modes
- Nanomechanics

MFP3D AFM family

Cypher AFM family

Jupiter XR

Conclusion



Today's goals

YOURS

- Discover or Improve your understanding on AFMs

What can I measure with my AFM? How? ***NOT ONLY TOPOGRAPHY***

Why it's a nice complementary tool to SEM

OURS

- Increase our understanding on what users are doing on their daily basis research

What are you measuring with your AFM? How?

- Understand your future needs

What will be for your the perfect AFM in 10years from now?

- If you're not AFM user, how AFM can help you

Oxford Instruments History

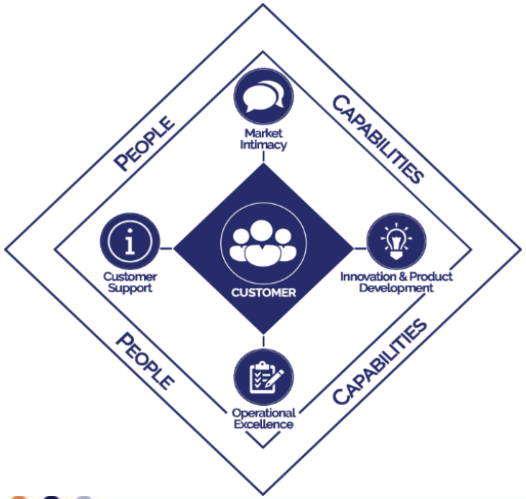
Funded in 1959

1960s OI develops the world's first superconducting magnet

1980s floated on London stock Exchange

>8% revenue invested on R&D

2500+ employees



PEOPLE

CAPABILITIES

Market Intimacy

Customer Support

CUSTOMER

Innovation & Product Development

Operational Excellence

PEOPLE

CAPABILITIES

Fabrication

2D Toolbox

Analysis

Characterisation

Materials growth and deposition

Low temperature and ultra low temperature tools

UHV Scanning Probe Microscopy and spectroscopy, Atomic Force Microscopy, SEM based nanoanalysis and nanomanipulation

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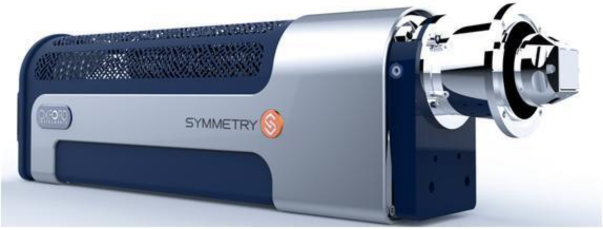
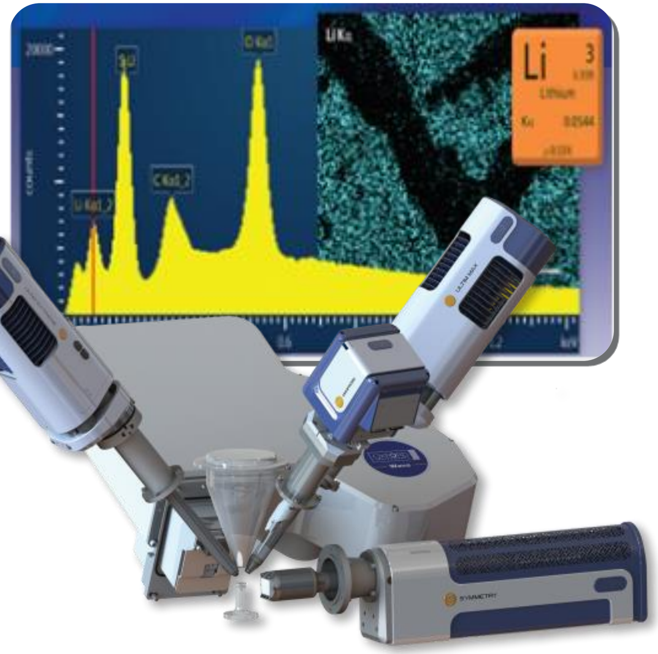


Nanotechnology tools



Ultim Max: grande surface active de 40 mm² jusqu'à 170 mm²
127eV MnKa @ 130.000 cps/s – 56eV CKa @ 130.000 cps/s
Quantitatif à plus de 400.000 cps/s
Cartes X à 1.000.000 cps/s
Jusqu'à 4 détecteurs contrôlés sur la même microscope

Ultim Extreme 100mm² sans fenêtre
Angle solide jusqu'à 0.35 srad
Détection du lithium
Résolution de 10nm @ 1.5kV

Caméra EBSD CMOS Symmetry
CMOS et fibre optique / 5FSD
EBSD et EDS en simultané à plus de 3000 Hz
Haute résolution 1244 x 1024 pixels
Sensible à partir de 100 pA et 5kV
Résolution angulaire meilleure que 0.05°

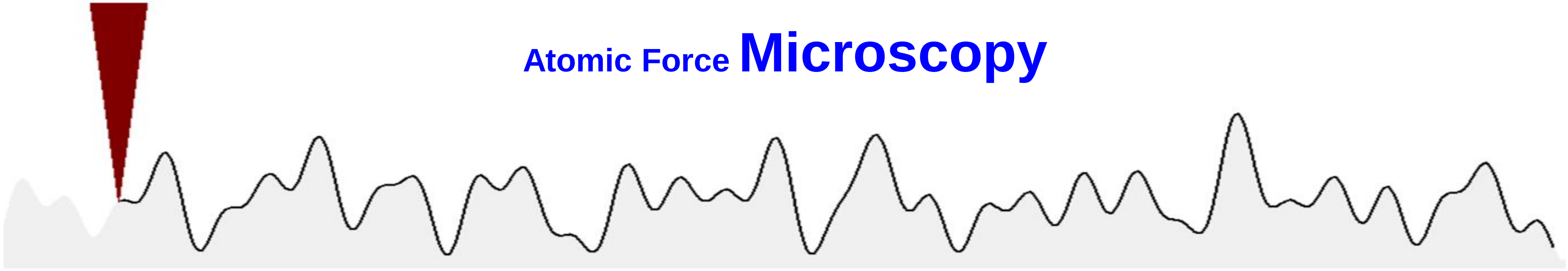
Nouveau logiciel de post-traitement ESBD AZtec ICE
60 millions de pixels traités en quelques secondes

Who are we?

- Founded 1999 by former Digital Instruments employees-self funded, no venture capital
- 120+ employees worldwide today
- >1500+ customers in industry/academia
- Worldwide sales & support network
- Acquired by Oxford Instruments in Dec. 2012
- **2 persons in France for sales and technical support**

Introduction

What is an AFM?



Visualization tool

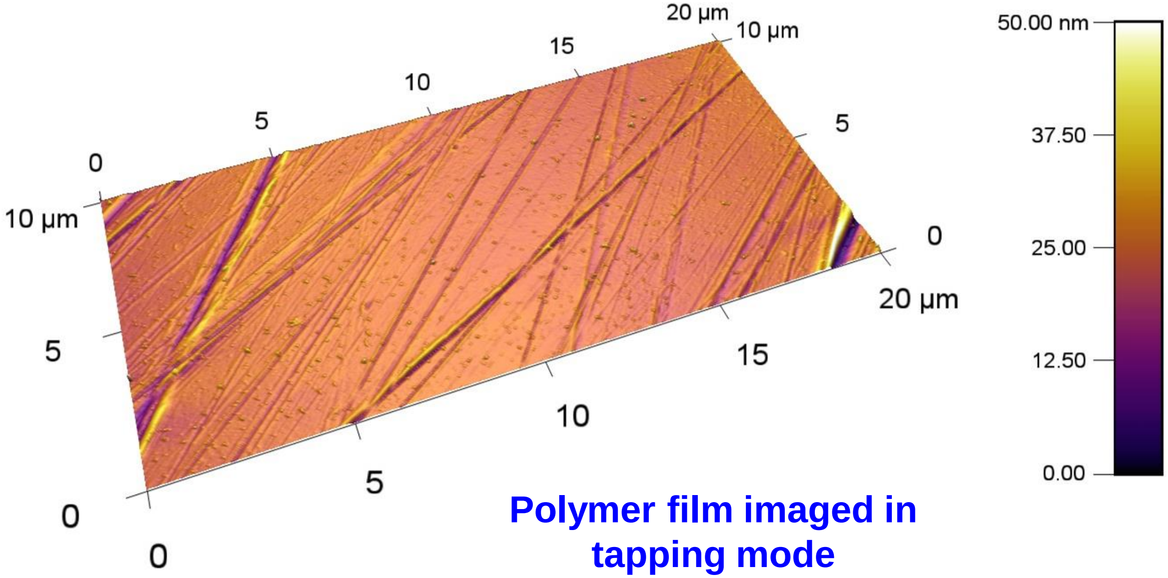
Uses a **sharp stylus**

- Vs. light, electrons
- <10 nm probe radius
- Below diffraction limit

Surface topography

“Touches” the surface

- Contact or proximity to the surface allows us to probe local material properties
- Mapping of electrical, magnetic, mechanical, thermal properties

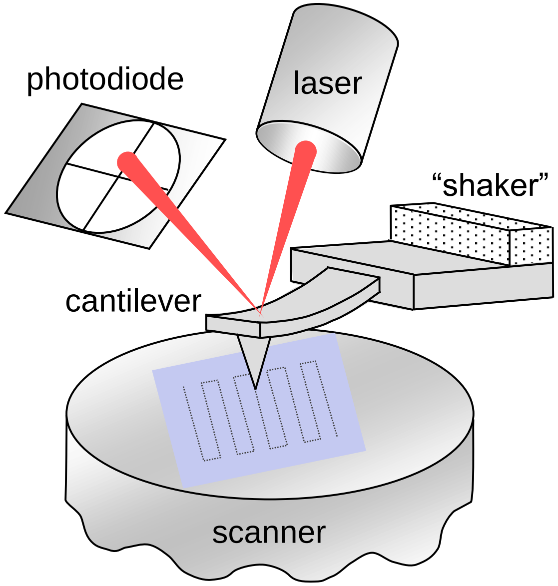


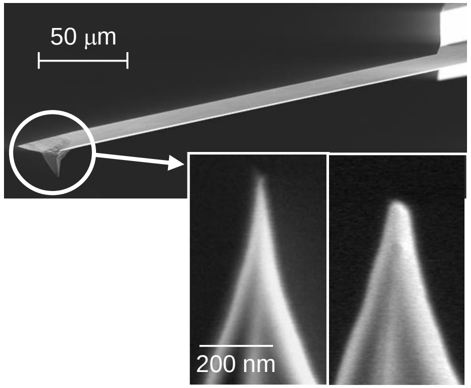
Polymer film imaged in tapping mode

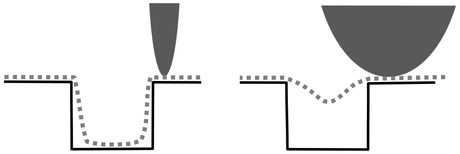
Introduction

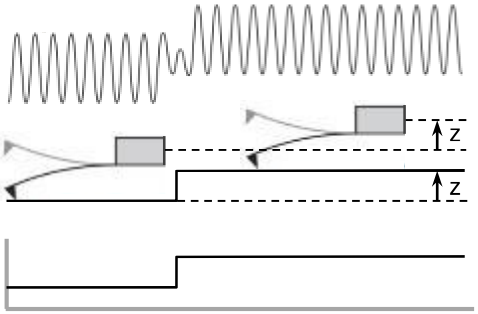
What is an AFM?

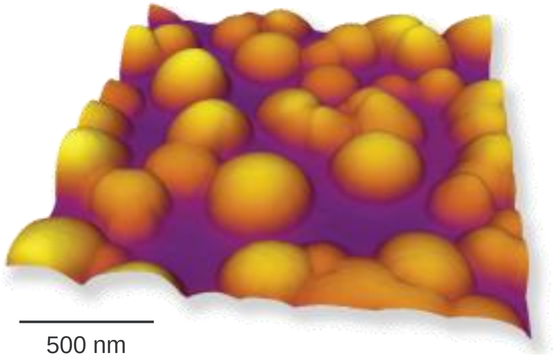
- “Feels” the surface
  - Micromachined cantilever with tip
  - Contact or tapping mode
- Benefits of tapping mode
  - Fast
  - Gentle
  - High spatial resolution
- Spatial resolution determined by
  - Tip size
  - Instrument noise
  - Drift and thermal variations











MDMO-PPV:PCBM solar cell
D. Ginger group, Univ. of Washington

Complementary technics

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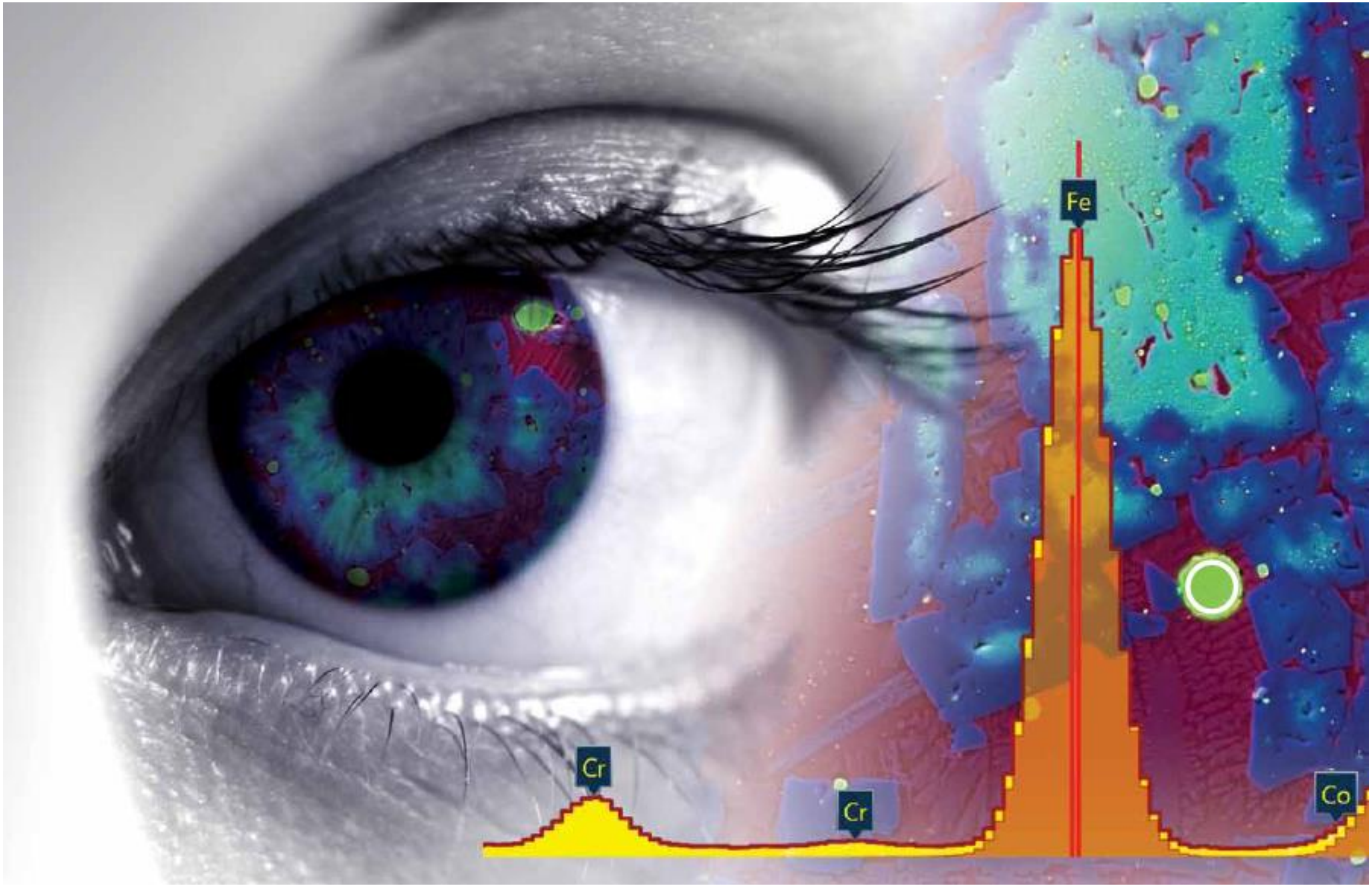

EDS / EBSD / WDS /..

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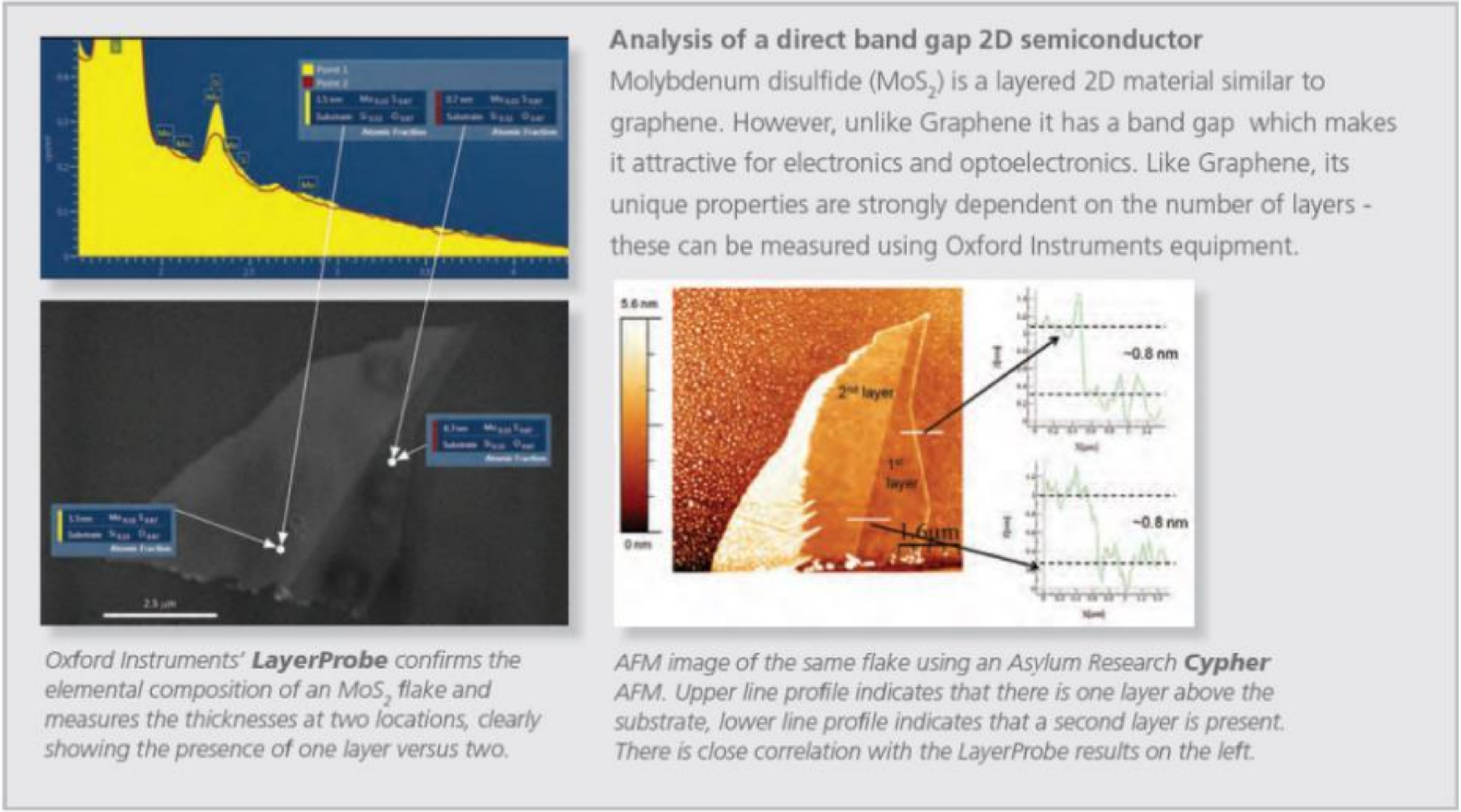


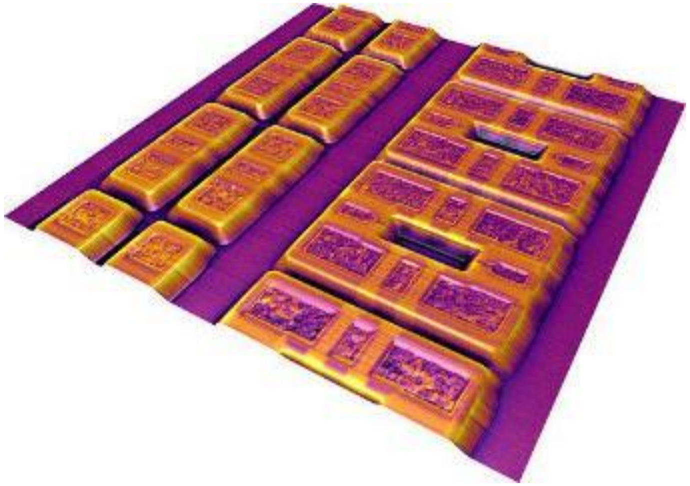
Analysis of a direct band gap 2D semiconductor

Molybdenum disulfide (MoS_2) is a layered 2D material similar to graphene. However, unlike Graphene it has a band gap which makes it attractive for electronics and optoelectronics. Like Graphene, its unique properties are strongly dependent on the number of layers - these can be measured using Oxford Instruments equipment.

Oxford Instruments’ ***LayerProbe*** *confirms the elemental composition of an MoS_2 flake and measures the thicknesses at two locations, clearly showing the presence of one layer versus two.*

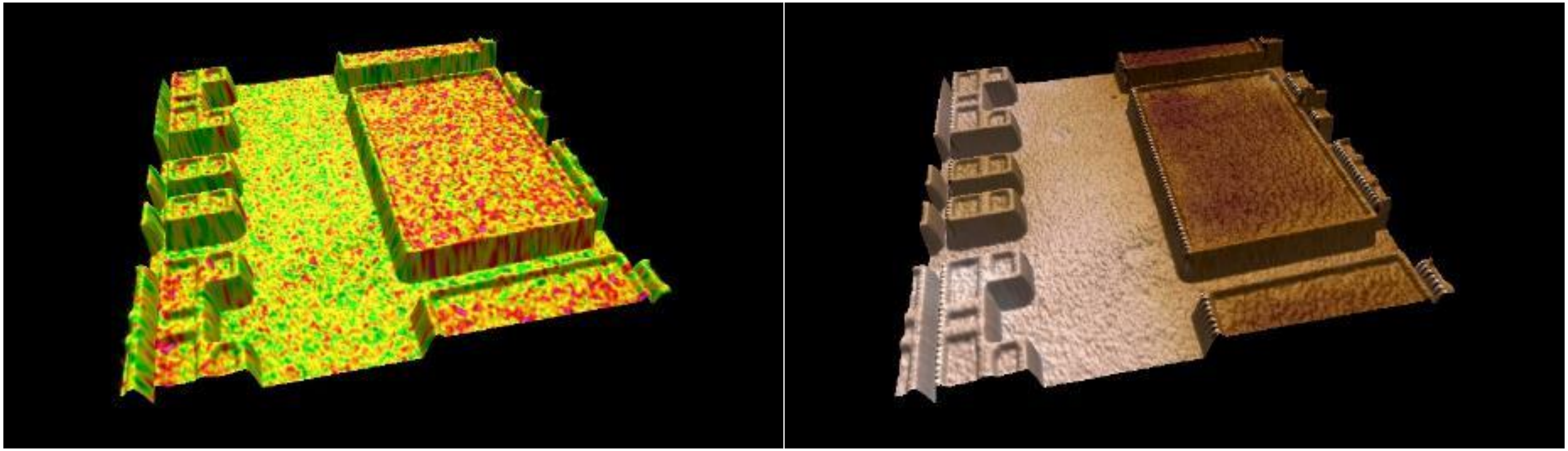
AFM image of the same flake using an Asylum Research ***Cypher*** *AFM. Upper line profile indicates that there is one layer above the substrate, lower line profile indicates that a second layer is present. There is close correlation with the LayerProbe results on the left.*

Fresh news: SRAM EDS + EFM



SRAM 30x30µm.

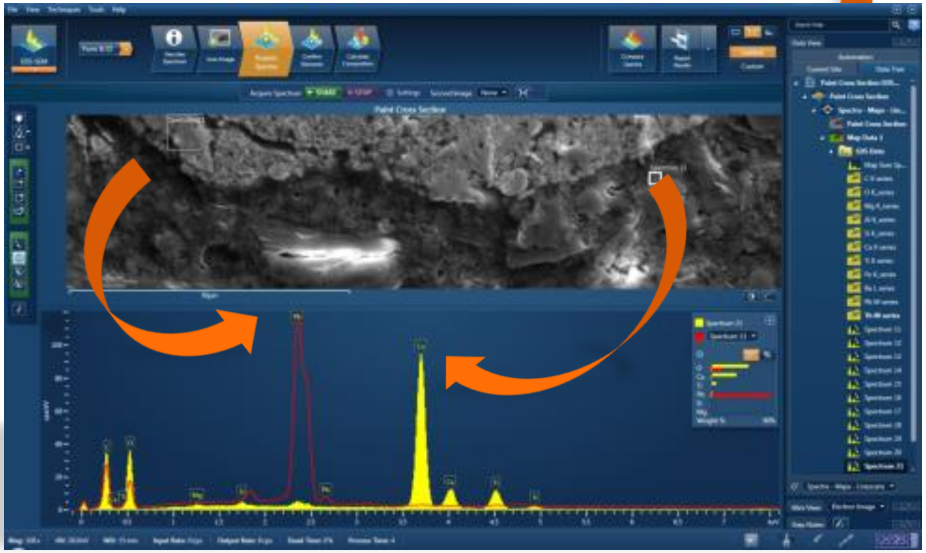
The sample is a SRAM standard sample. The red color is As dosed in Si (2x10^20 cm^-3). The second is SKPM result.



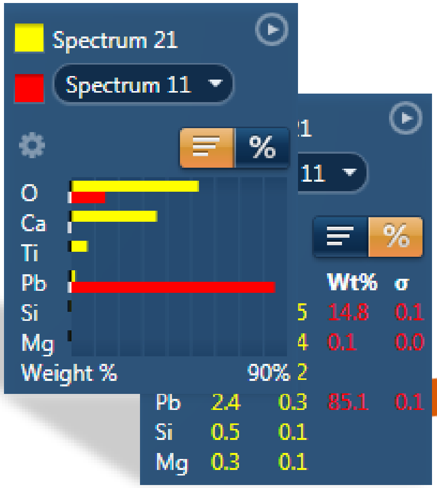
Next step: Same software

⇨ Identification / quantification

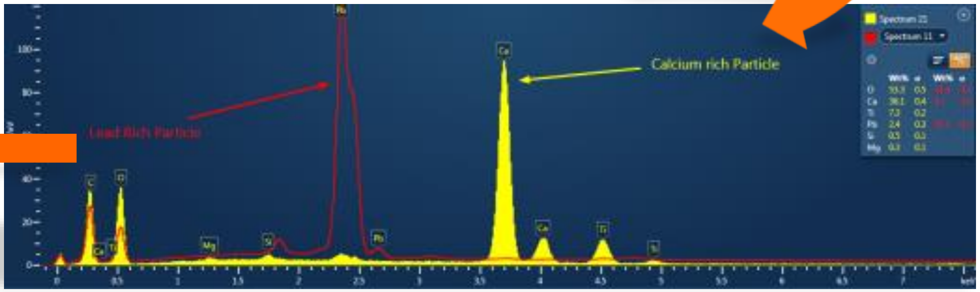
Acquisition



Quantification

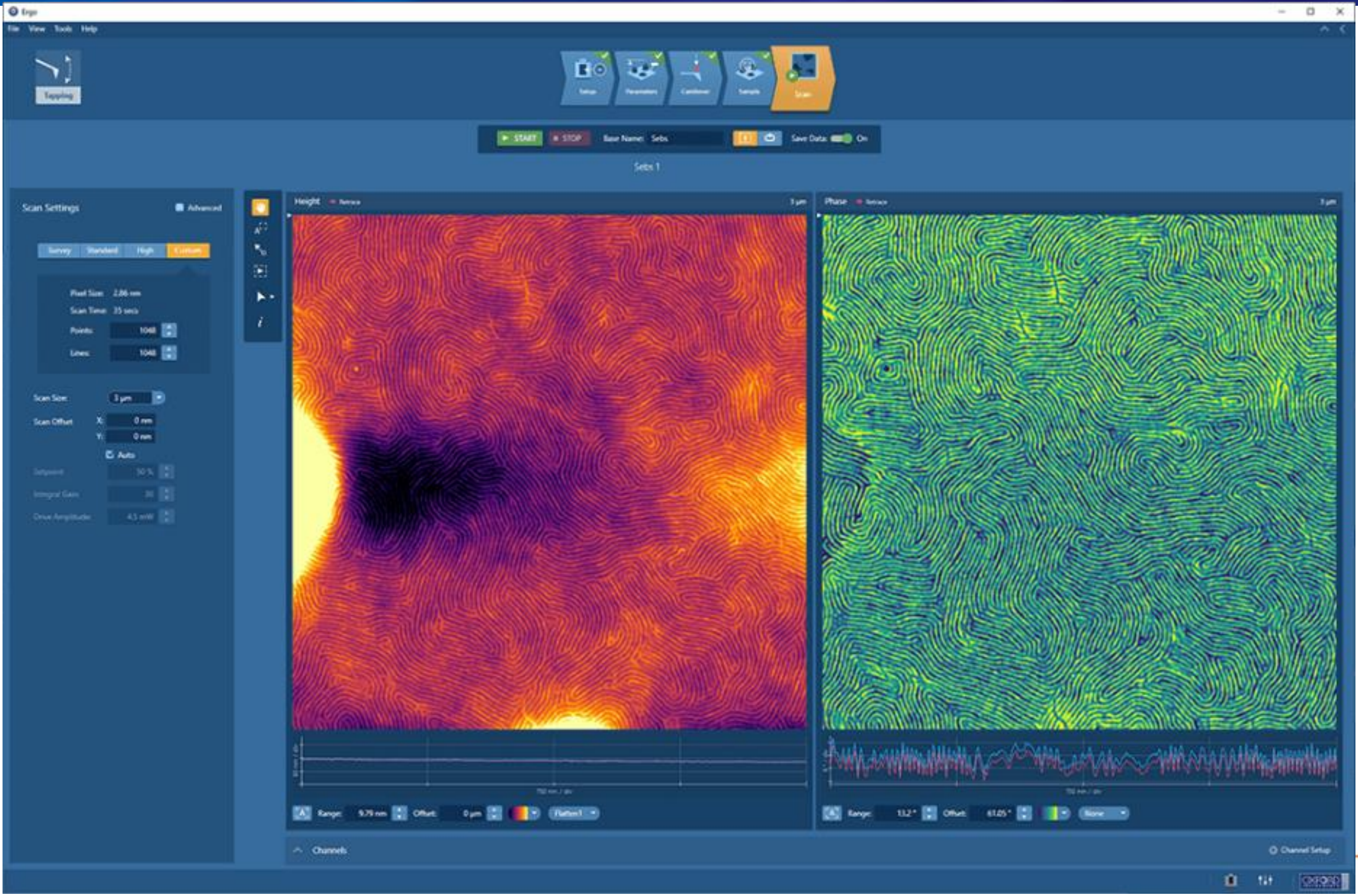


Comparer/documenter



"Sortir" un résultat/une analyse

ERGO = Aztec



Base Name: Sebs

Save Data: On

Sebs 1

Scan Settings

☐ Advanced

Survey | Standard | High | Custom

Pixel Size: 2.86 nm

Scan Time: 35 secs

Points: 1048

Lines: 1048

Scan Size: 3 µm

Scan Offset X: 0 nm

Y: 0 nm

☑ Auto

Setpoint: 50 %

Integral Gain: 30

Drive Amplitude: 4.5 mW

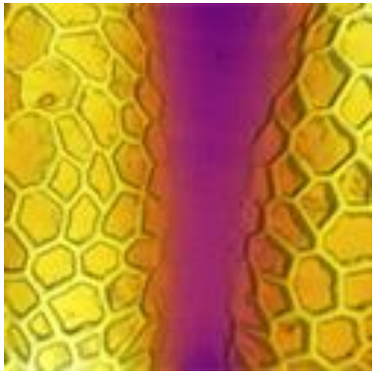
Range: 9.79 nm Offset: 0 µm Flatten1

Range: 13.2 ° Offset: 61.05 ° None

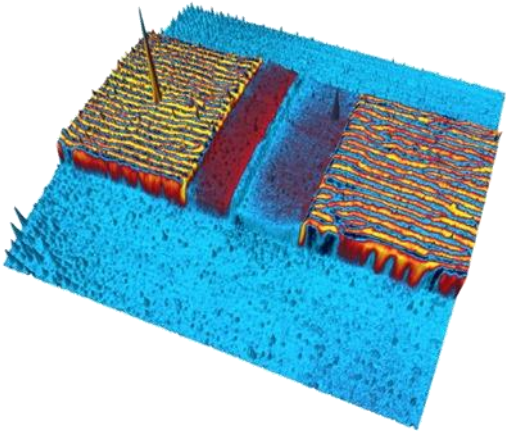
Introduction

A large number of applications

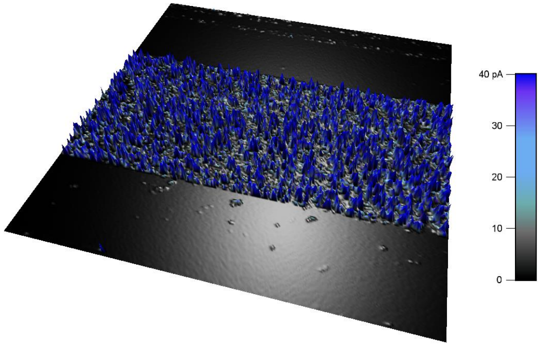
- Materials science / polymers
- Physics and chemistry
- Biomaterials / biophysics
- Molecular and cell biology
- Magnetics and data storage
- Photovoltaics / solar / battery / energy research
- Semiconductors



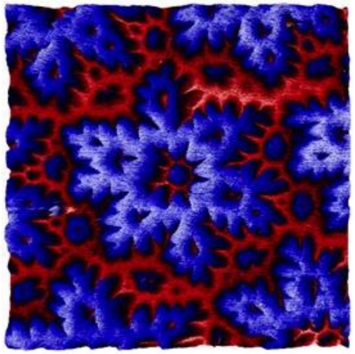
Pollen



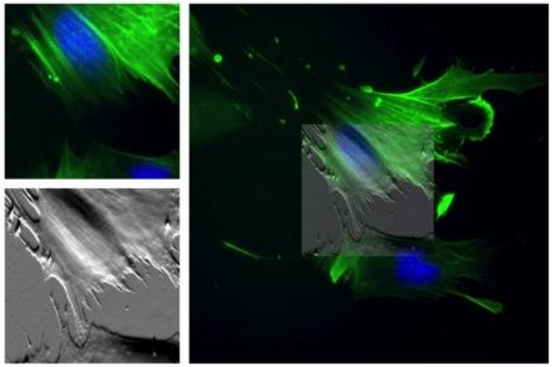
Read write head



Photoconductivity



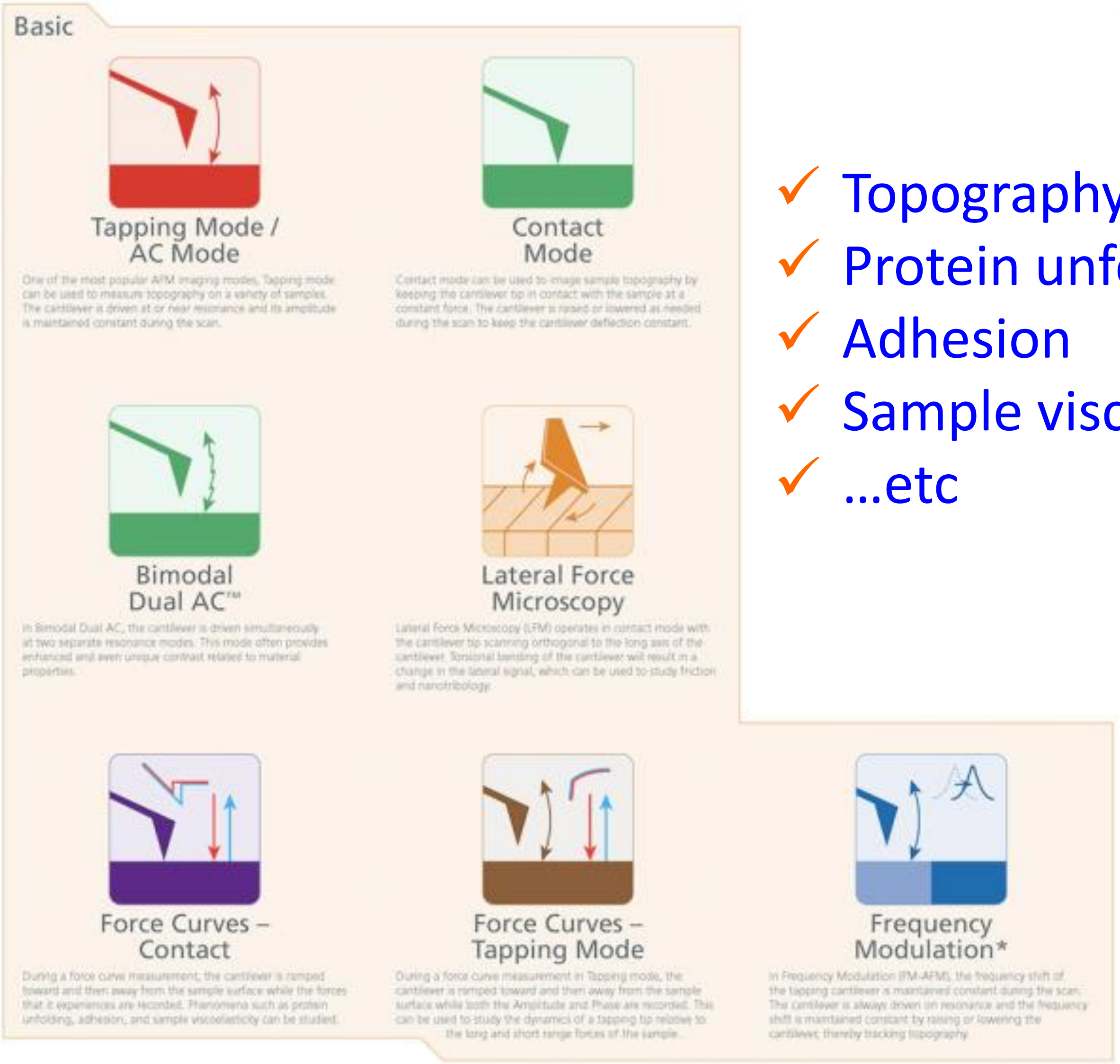
Garnet



Cell biology

AFM modes

Back to basics…



Basic

Tapping Mode / AC Mode

One of the most popular AFM imaging modes, Tapping mode can be used to measure topography on a variety of samples. The cantilever is driven at or near resonance and its amplitude is maintained constant during the scan.

Contact Mode

Contact mode can be used to image sample topography by keeping the cantilever tip in contact with the sample at a constant force. The cantilever is raised or lowered as needed during the scan to keep the cantilever deflection constant.

Bimodal Dual AC™

In Bimodal Dual AC, the cantilever is driven simultaneously at two separate resonance modes. This mode often provides enhanced and even unique contrast related to material properties.

Lateral Force Microscopy

Lateral Force Microscopy (LFM) operates in contact mode with the cantilever tip scanning orthogonal to the long axis of the cantilever. Torsional bending of the cantilever will result in a change in the lateral signal, which can be used to study friction and nanotribology.

Force Curves – Contact

During a force curve measurement, the cantilever is ramped toward and then away from the sample surface while the forces that it experiences are recorded. Phenomena such as protein unfolding, adhesion, and sample viscoelasticity can be studied.

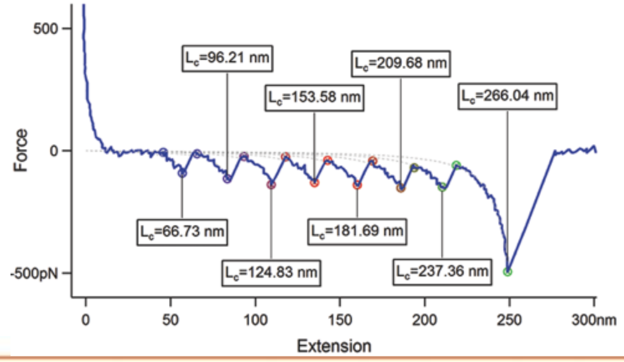
Force Curves – Tapping Mode

During a force curve measurement in Tapping mode, the cantilever is ramped toward and then away from the sample surface while both the Amplitude and Phase are recorded. This can be used to study the dynamics of a tapping tip relative to the long and short range forces of the sample.

Frequency Modulation*

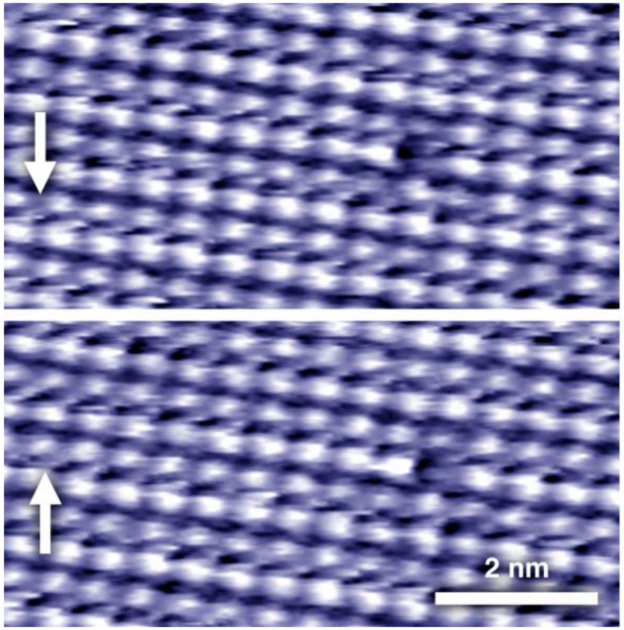
In Frequency Modulation (FM-AFM), the frequency shift of the tapping cantilever is maintained constant during the scan. The cantilever is always driven on resonance and the frequency shift is maintained constant by raising or lowering the cantilever, thereby tracking topography.

- ✓ Topography
- ✓ Protein unfolding
- ✓ Adhesion
- ✓ Sample viscoelasticity
- ✓ …etc

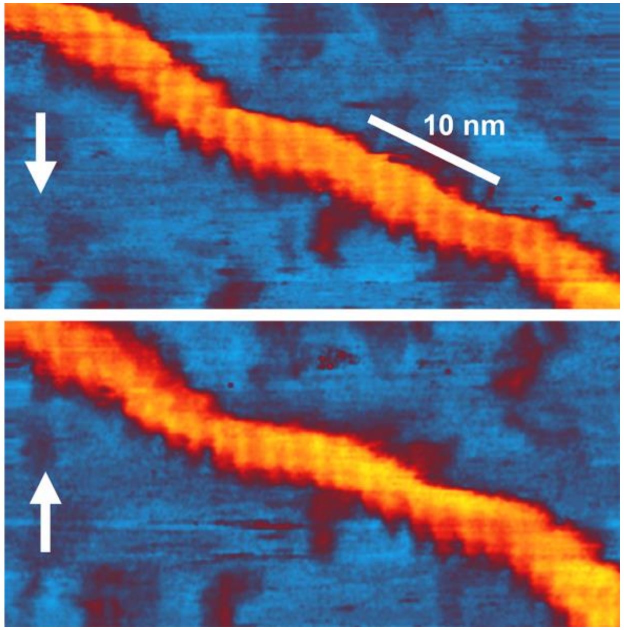


AFM modes

Highest Resolution Imaging

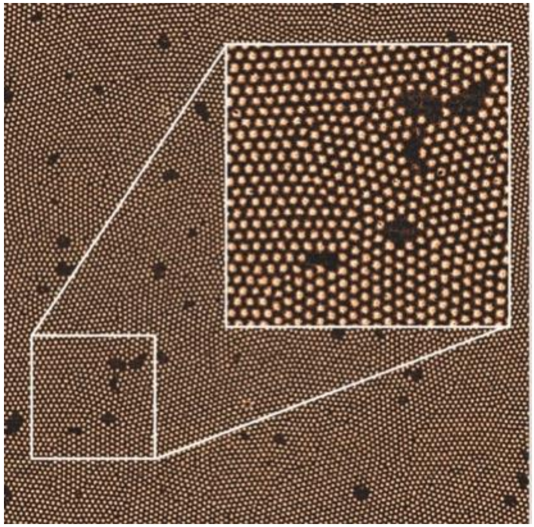


Calcite with repeatable, single isolated atomic point resolution (in water)



DNA helix

Imaged Routinely!



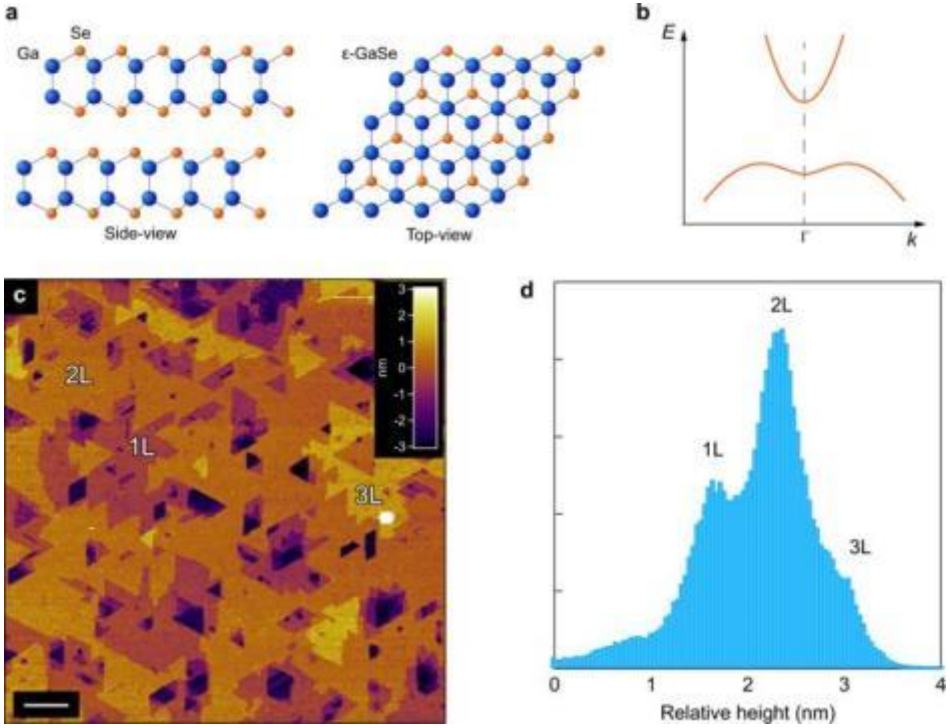
Patterned silicon with zoom. Courtesy O. Gang, Brookhaven Nat. Lab

Every Cypher must demonstrate point defect resolution during testing… So Easy ☺

Some AsGa papers

Particle Analysis

CAFM conductivity image of GaAs quantum dots, 700 nm scan. Courtesy of T. Jones, U. Warwick.



AFM image of ultrathin GaSe grown on the surface of GaAs using MBE. The scale bar is 500 nm long. Labels 1L, 2L, and 3L refer to monolayer, bilayer, and trilayer regions of the film

EPFL, Andras' Kis Lab

Process status

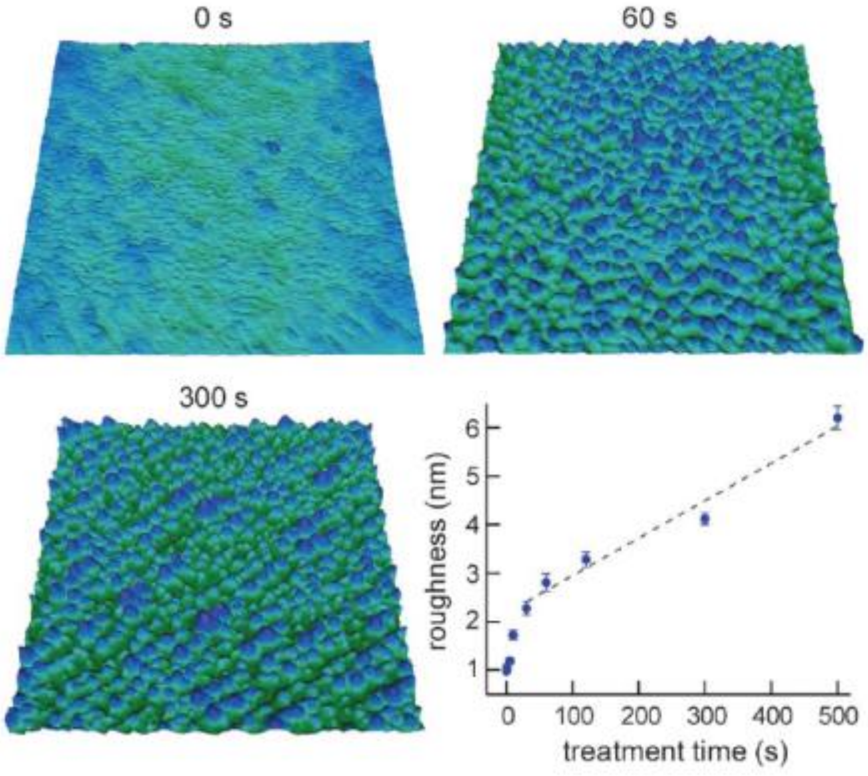


Figure 1: *Oxygen plasma treatment of polyethylene terephthalate (PET) films. PET fibers coated with a conducting polymer such as polypyrrole could be used in "smart" electronic textiles. However, achieving good coating-to-fiber adhesion remains a key challenge. Images of PET films exposed to oxygen plasma show that RMS surface roughness increased with exposure time. Films processed longer than 60 s displayed surface etching and uniform nanoscale features. The graph reveals a linear dependence of roughness on treatment time after ~30 s. Combined with data on surface chemistry, the results can be used to optimize treatment parameters for improved coating adhesion and conductivity. Scan size 1 µm; height scale 35 nm. Imaged with the Cypher S AFM. Adapted from Ref. 2.*

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AFM modes

Electrical properties

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Electrical

Conductive AFM*

Conductive AFM (ORCA™) scans in contact mode while measuring any current flowing through the sample into the conductive cantilever tip. Additionally, this mode allows for localized I-V measurements which can be made at specific user-defined points.

Scanning Kelvin Probe

Scanning Kelvin Probe Microscopy (SKPM) is a two-pass imaging mode where a conductive cantilever tip images the surface in Tapping mode in the first pass, and then lifts above the surface by a constant height to acquire a quantitative measure of the sample's potential.

Electrostatic Force Microscopy

Electrostatic Force Microscopy (EFM) is a two-pass imaging mode where the sample's longer-range electrostatic forces are qualitatively imaged in a secondary pass after an initial surface pass. Variations in trapped charge, potential, or the sample's conductivity or permittivity can be imaged.

Fast Current Mapping*

Fast Current Mapping is a mode that collects current continuously during an array of force curves taken at high speed. Current maps of the sample can be generated with the benefit of minimal tip-wear since the tip is not scanning the surface.

Scanning Tunneling Microscopy*

Scanning Tunneling Microscopy (STM) provides tunneling current imaging at constant current or constant height, and I-V measurement capability.

Scanning Microwave Impedance Microscopy*

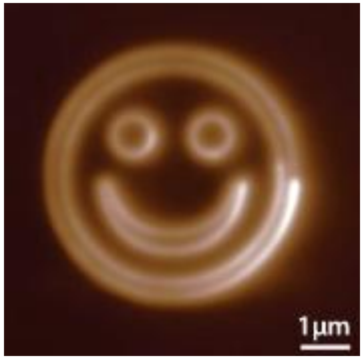
Scanning Microwave Impedance Microscopy (sMIM) is a near-field technique that uses a microwave source to distinguish local sample variations in resistance, capacitance, as well as dC/dV and dR/dV responses and can be operated in contact or tapping mode.

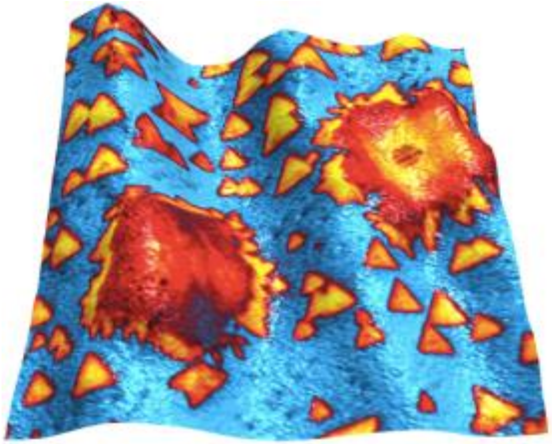
Nano Time Dependent Dielectric Breakdown*

Nanoscale Time Dependent Dielectric Breakdown (NanoTDDB) is a point measurement mode that is used to study dielectric breakdown at the nanoscale. A high voltage bias ramp is sent to the cantilever tip until the breakdown event in the sample occurs.

- ✓ Local charges
- ✓ Current leakage
- ✓ Surface potential
- ✓ Dielectric breakdown
- ✓ …etc

KPFM of a charge pattern written on PMMA





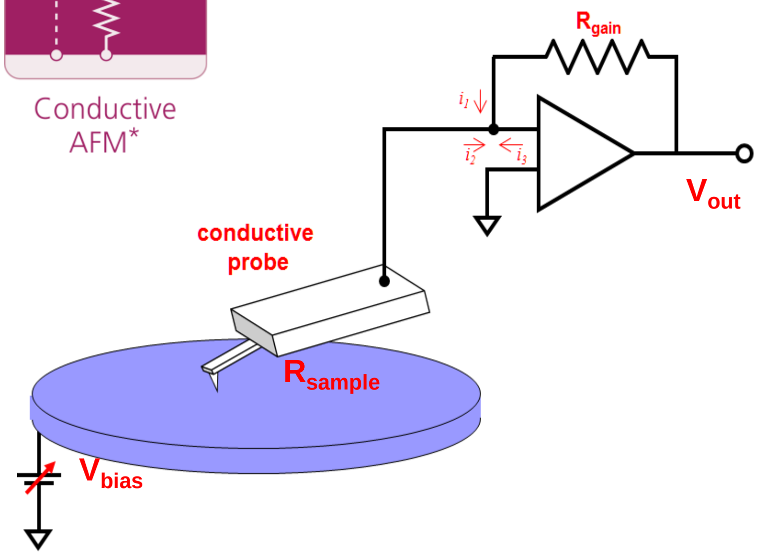
KPFM distinguishes Graphene and Boron nitride on a copper substrate.

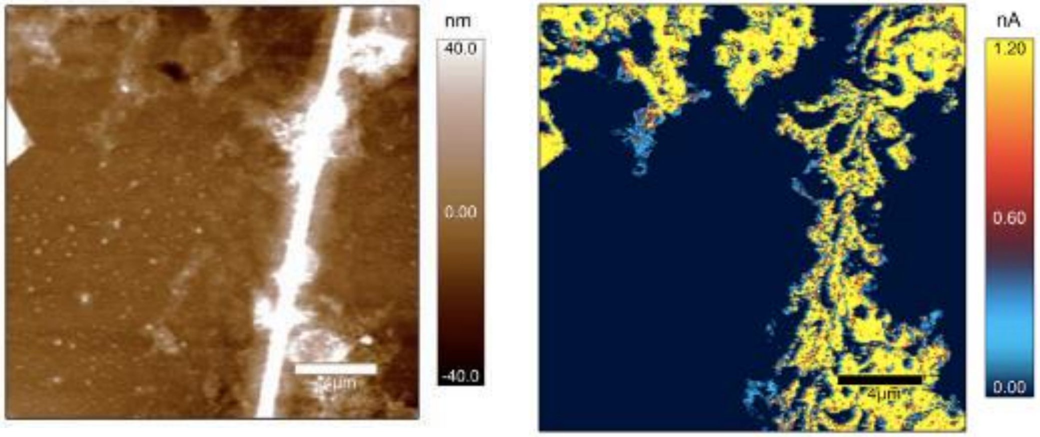
CAFM

Conductive AFM (ORCA™*)

Asylum Research
an Oxford Instruments company

Conductive AFM*

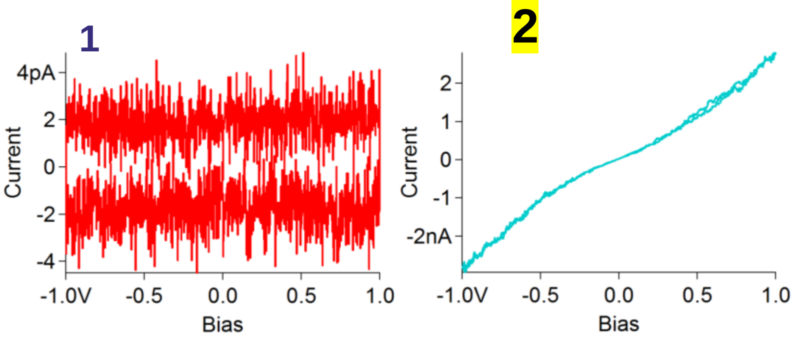




CNT surounded by graphene on glass surbstract

Conductive AFM (CAFM) looks at current passing through the tip and sample. It generates a current map or current-voltage spectra of the surface.

Current is measured with a **transimpedance amplifier**, which converts the current signal to a voltage signal. The amplifier **gain** is defined by the gain resistor.



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*Optimized Resistance Conductance Amplifier

Datasheet Image

PV general (AR CdSe/ITO Test Sample)

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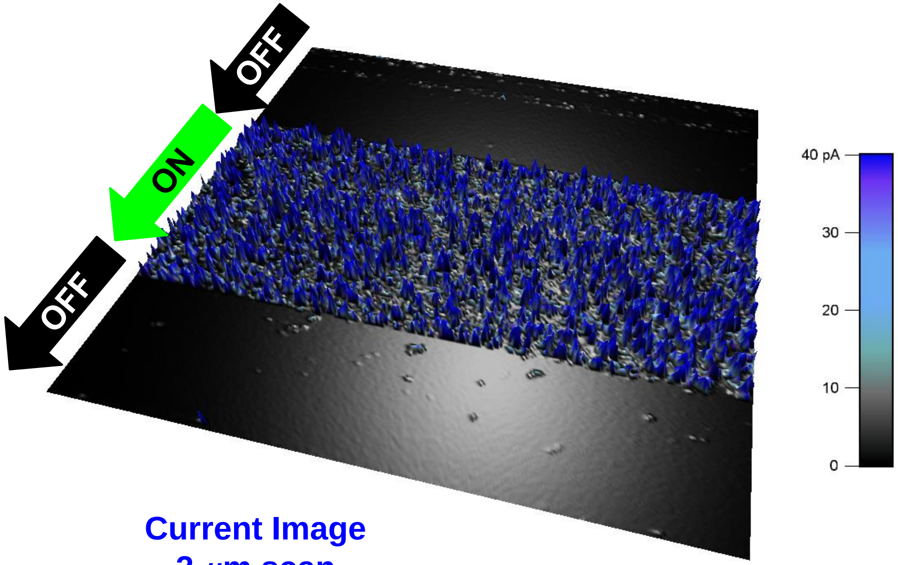
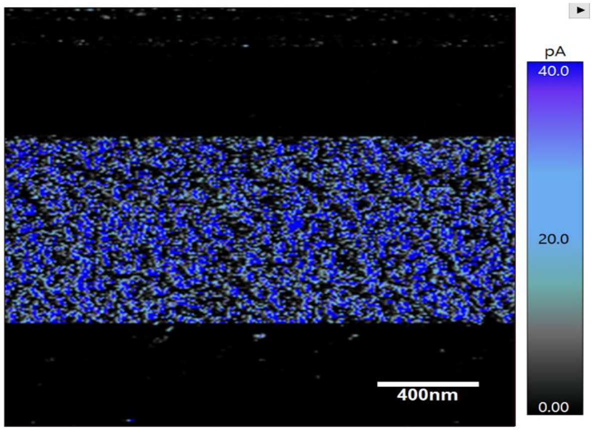


Current Image
2-μm scan

CdSe on ITO imaged with ORCA™ conductive AFM at 1V sample bias. The Infinity PV Package used an included 530nm was used to irradiate the sample with up to 0.9W/cm2 intensity. Illumination was turned off for only the middle portion of the scan.



MRS OnDemand® WEBINAR SERIES

Presented by: OXFORD INSTRUMENTS
Asylum Research AFMs

AFM Characterization of Emerging Photovoltaics

Host: Donna Hurley
Lark Scientific LLC

Speakers
F. Ted Limpoco
Oxford Instruments Asylum Research

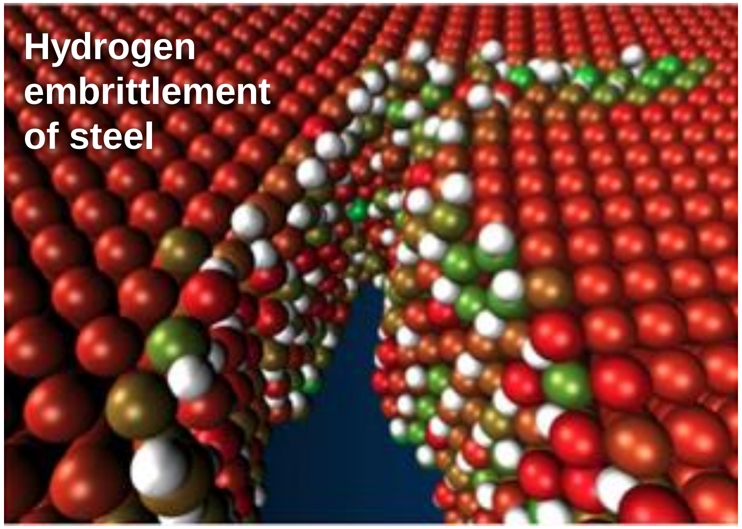
Rajiv Giridharagopal
The Ginger Group Lab, University of Washington-Seattle

MRSBulletin

Emerging Photovoltaics

Corrosion

Tags: Electrochemistry, Oxidation, Embrittlement



Corrosion's impact

- Losses due to corrosion costs \$2.5 T annually
- Impacts **environment** and **personal safety**, especially from **catastrophic failures**
- Estimated 15-35 % could be saved if best practices and technologies are applied

Jobs to be done:

- Corrosion potential of materials (metals)
- Nanoscale topographic changes of bias-dependent reactions
- Real time changes at the charge transfer interface
- Fundamental understanding of corrosion mechanisms

EC-AFM Imaging of Li-O$_2$ Battery Solid-Electrolyte Interface/Interphase Dynamics

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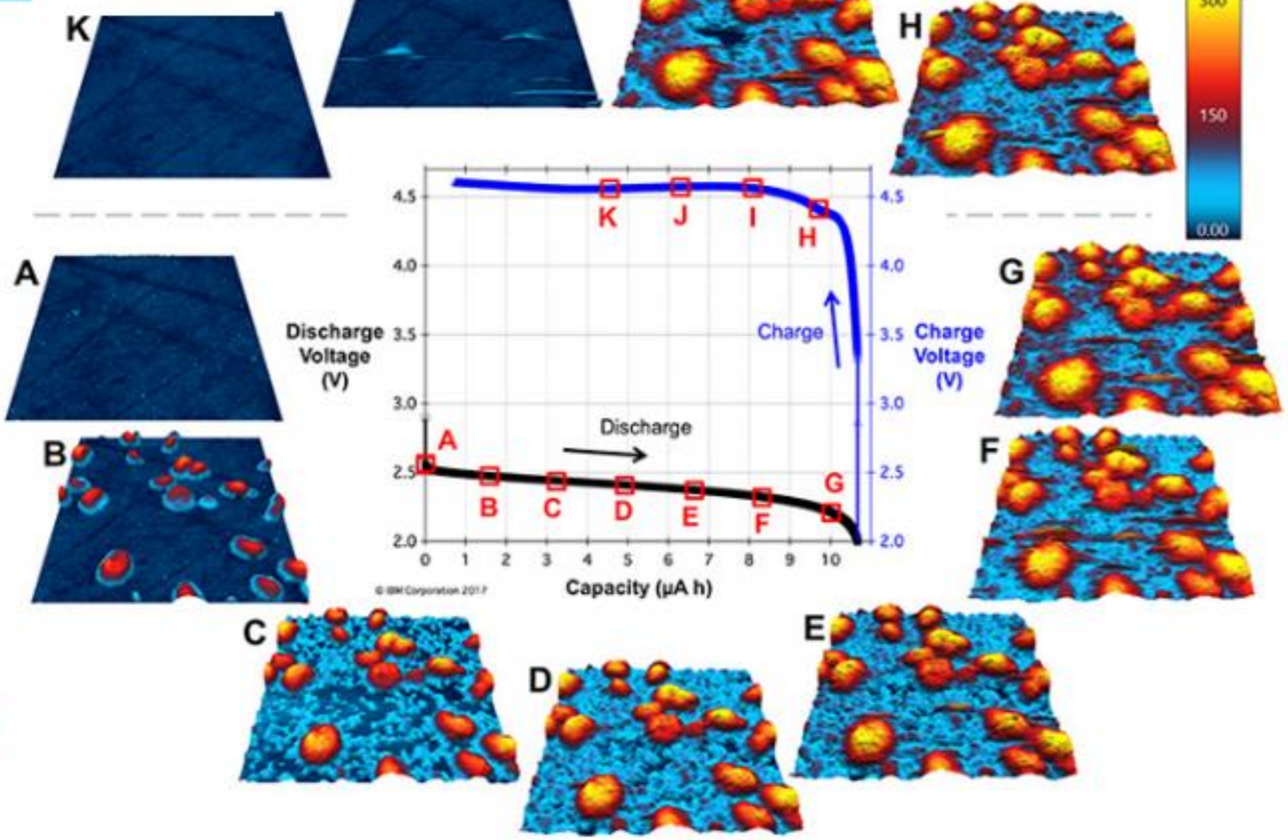
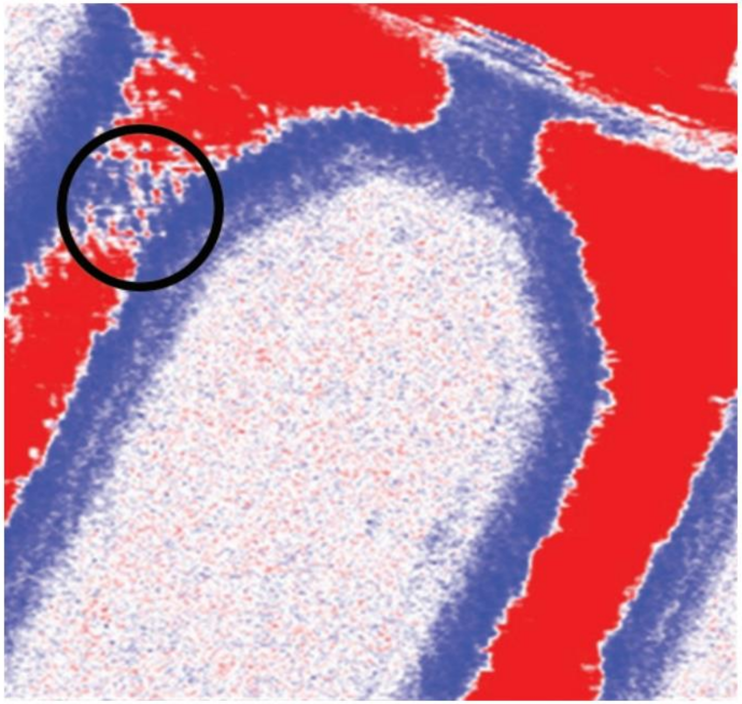
what structural changes happen inside the battery during charging or discharging?



SRAM Memory Device

Example: Identifying unexpected dopant variations



dC/dV phase shows possible failure on an **SRAM** device

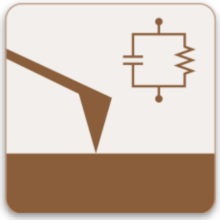
Jobs to be done:

- Semiconductor devices consists of segregated regions of **p-type** and **n-type** silicon
- Failure occurs when there is an unexpected variation in dopant type
- **Goal:** Map out **dopant type variation** to determine failure in devices

Solution: sMIM dC/dV Mapping

(Scanning Microwave Impedance Microscopy)

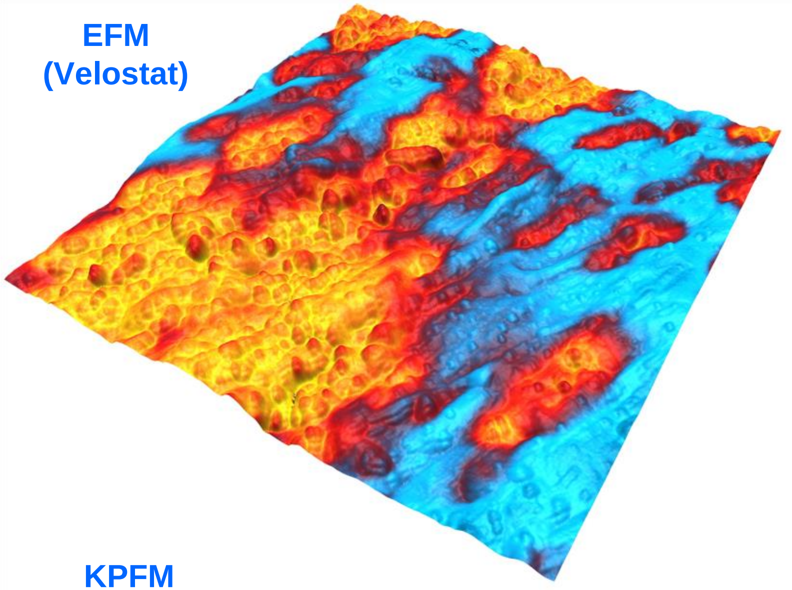
- dC/dV Phase image gives charge carrier type
- Strong contrast (signal) indicates doped regions, e.g., red is p-type, blue is n-type
- Image shows a leak of the n-type region into the p-type region between two devices, which may indicate failure

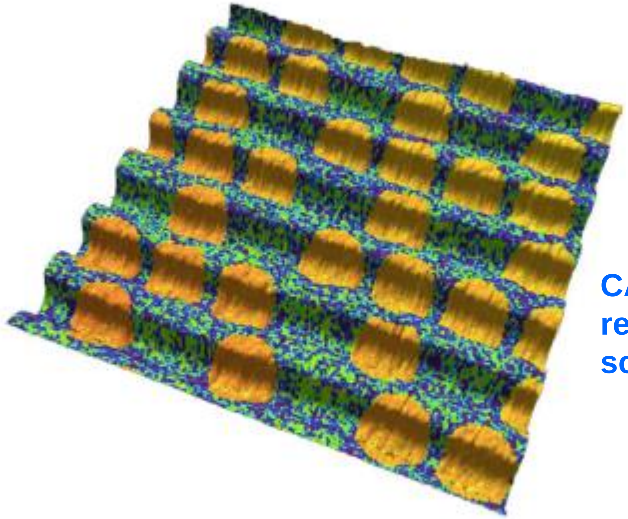


EFM, KPFM

Probing Long-Range Forces

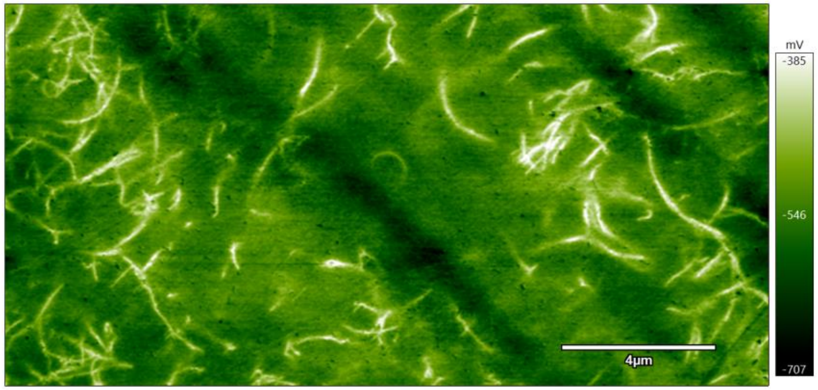
EFM (Velostat)

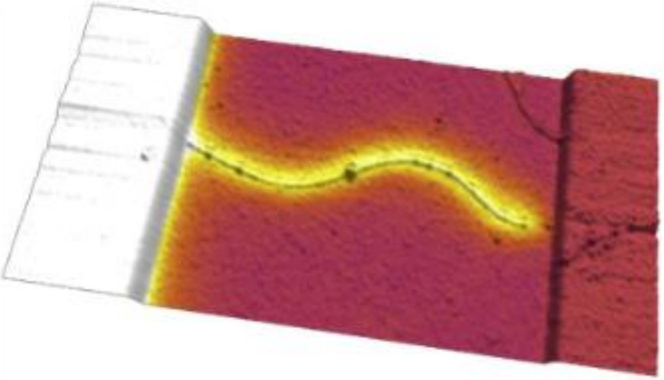




CAFM image DVD recorded marks, 5 µm scan

KPFM (CNTs in Polyimide)

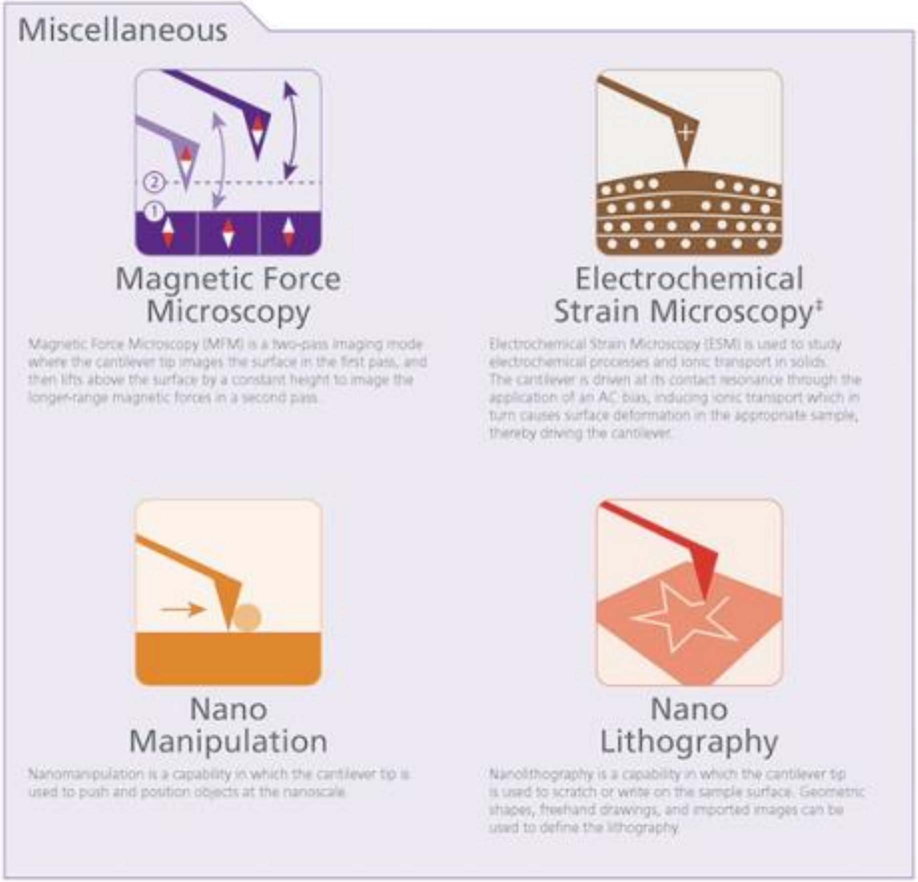




EFM image of CNT attached to an electrode, 5x2,5 µm scan

AFM modes

And much more….



Miscellaneous

Magnetic Force Microscopy

Magnetic Force Microscopy (MFM) is a two-pass imaging mode where the cantilever tip images the surface in the first pass, and then lifts above the surface by a constant height to image the longer-range magnetic forces in a second pass.

Electrochemical Strain Microscopy‡

Electrochemical Strain Microscopy (ESM) is used to study electrochemical processes and ionic transport in solids. The cantilever is driven at its contact resonance through the application of an AC bias, inducing ionic transport which in turn causes surface deformation in the appropriate sample, thereby driving the cantilever.

Nano Manipulation

Nanomanipulation is a capability in which the cantilever tip is used to push and position objects at the nanoscale.

Nano Lithography

Nanolithography is a capability in which the cantilever tip is used to scratch or write on the sample surface. Geometric shapes, freehand drawings, and imported images can be used to define the lithography.

- ✓ Magnetic domains (variable magnetic field IP & OP)
- ✓ Charge carrier measurement
- ✓ Nanomanipulation
- ✓ Nanolithography
- ✓ Thermal (melting point)
- ✓ …etc

Thermal

Local Themal Analysis*

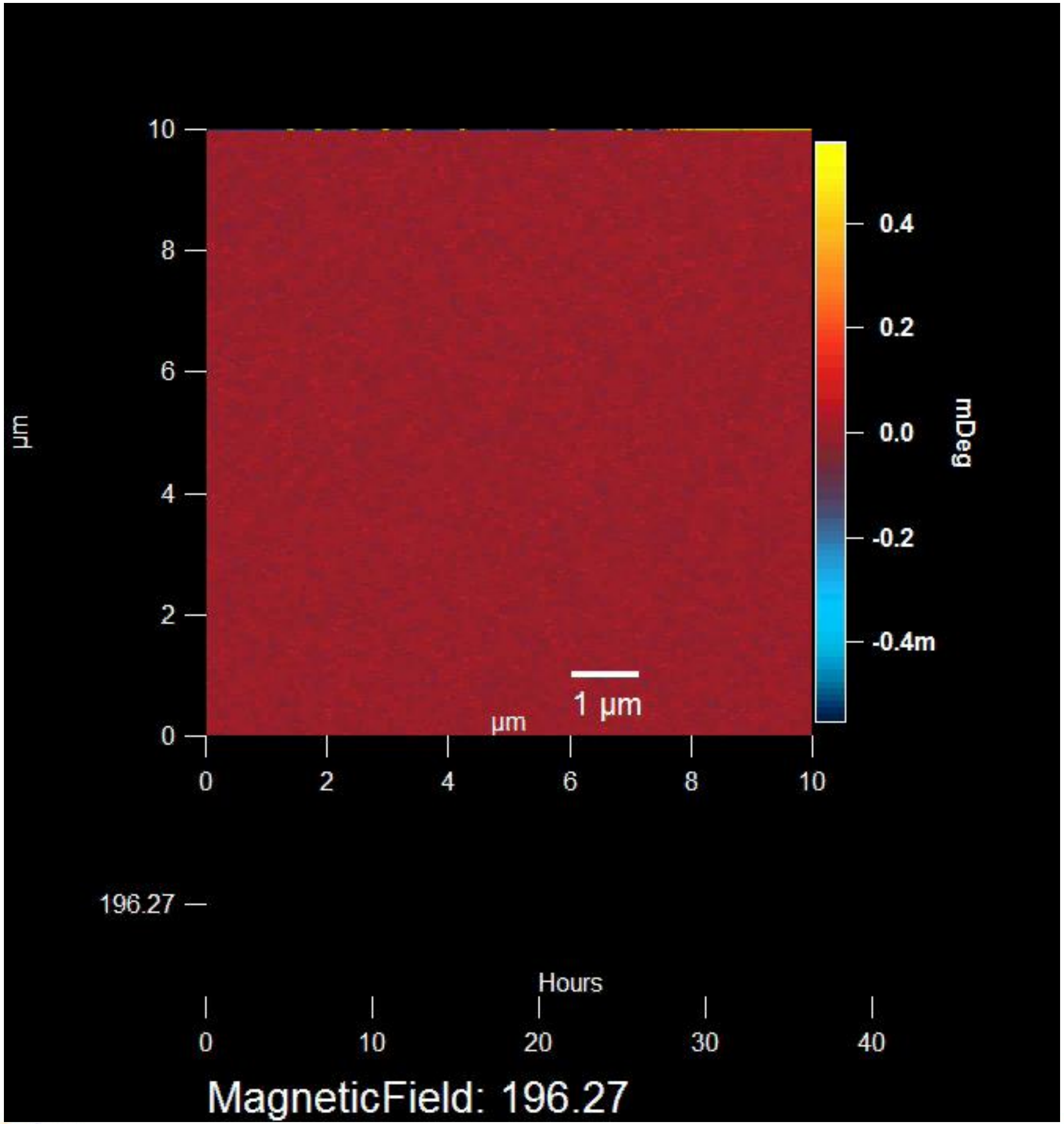
Local Thermal Analysis (Ztherm™) is a nanoscale thermal analysis measurement used to locally measure melting and glass transition points. The temperature of the AFM cantilever tip is ramped up while the cantilever is in contact with the sample, initially causing the sample to thermally expand and then melt.

Scanning Thermal Microscopy*

Scanning Thermal Microscopy (SThM) is a mode used to measure the temperature of the sample. For isothermal samples, SThM can alternatively be used to map variations in the sample's thermal conductivity.

MFM

Probing Long-Range Forces



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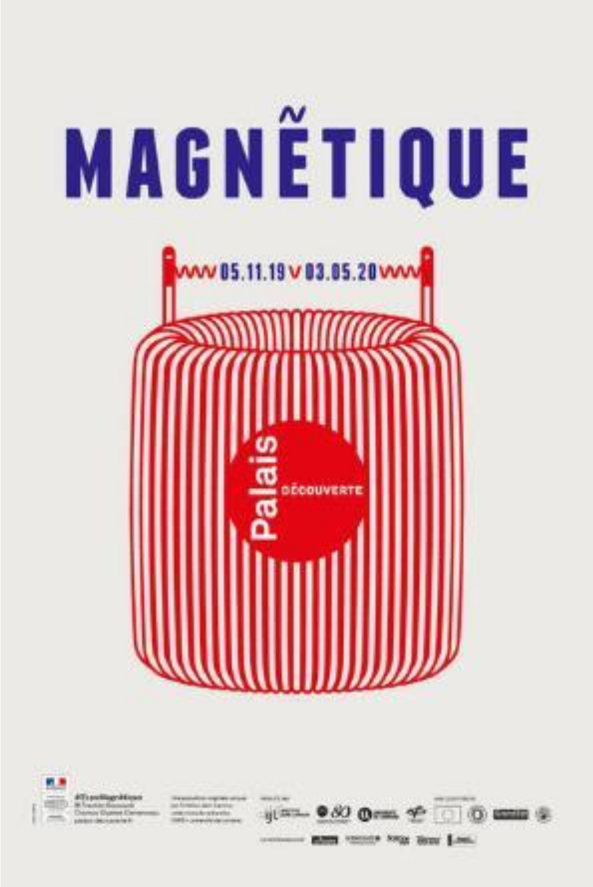


Advanced AFM Modes

AFM as a Materials Characterization Tool

NanomechPro™

AM-FM Viscoelastic Mapping*

AM-FM is an imaging mode used for viscoelastic mapping. The cantilever is driven simultaneously at two separate resonance modes. For the higher mode, changes in frequency are related to sample stiffness and elasticity, whereas changes in amplitude are related to sample dissipation and loss.

Calibrated Loss Tangent

This technique maps the sample's loss tangent, which is the ratio of the energy dissipated to the energy stored as the cantilever taps the surface.

Contact Resonance Viscoelastic Mapping*

Contact resonance provides quantitative imaging of elastic and loss modulus. The cantilever is scanned in contact while simultaneously being excited at the tip-sample contact resonance. This resonance is tracked with various techniques (e.g. DART or Band Excitation).

Force Maps

A Force Map is simply an array of force curves. Quantities like sample height, sample modulus, and tip-sample adhesion can be extracted from this array of force curves, in addition to a number of other preset and even custom calculated quantities.

Fast Force Mapping*

Fast Force Mapping is simply an array of force curves taken at high speed. Quantities like sample height, sample modulus, and tip-sample adhesion can be extracted from the array of force curves, in addition to a number of other preset and even custom calculated quantities.

Force Modulation

In force modulation, the cantilever tip is driven below resonance while in contact with the sample. The amplitude response of the cantilever is indicative of the sample stiffness.

Cantilever Indentation

The cantilever can be used to indent the surface for qualitative mapping of the sample's mechanical properties. While various mechanical models can be applied, the results are only qualitative due in part to the lateral movement of the tip while in contact.

Instrumented Nanoindentation*

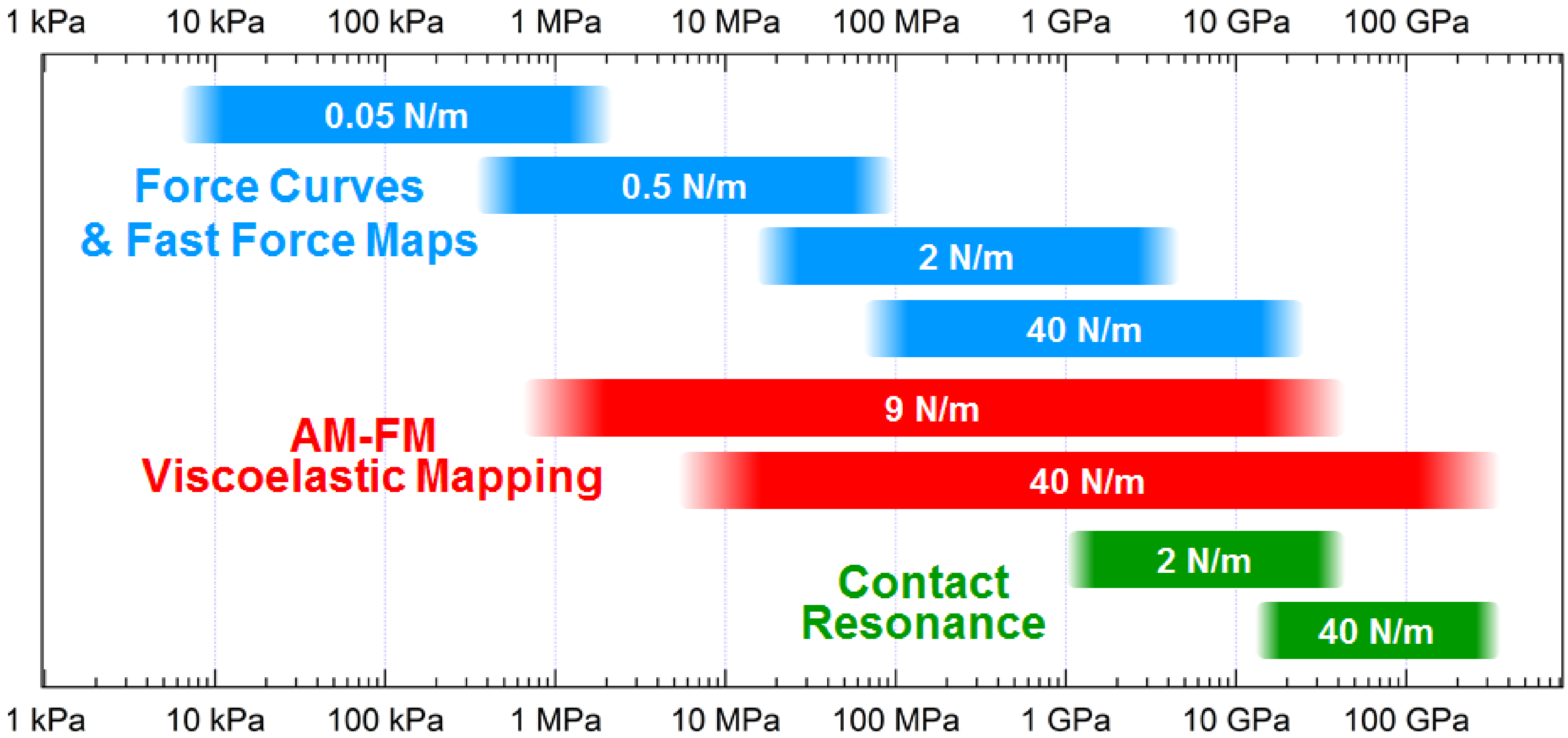
Instrumented indentation provides quantitative mechanical characterization of the sample at the nanoscale.

QUANTITATIVE:

- ✓ Loss Tangent (dissipation/stored)
- ✓ Adhesion
- ✓ Sample viscoelasticity
- ✓ Stiffness
- ✓ Young modulus
- ✓ …etc

Spring Constant

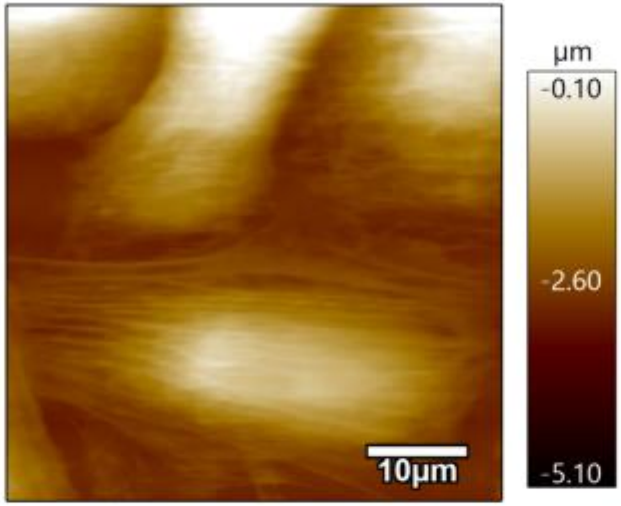
Selection Guide: Nanomechanics



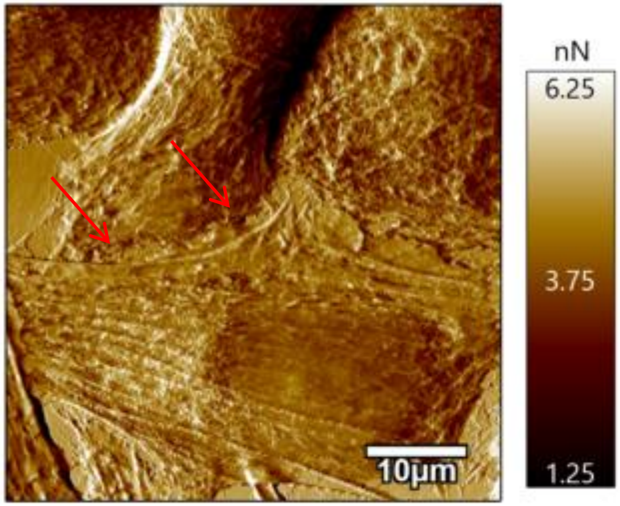
Fast Force Maps of Live Bone Cancer Cells (U2 OS)

- 50µm scan, 512x256, Z Rate = 100 Hz, Setpoint of 4nN, Force distance = 2µm
- The data was acquired in approximately 27 minutes.
- We can see incredible detail of the cell extension.
- In the Fmax channel there isn't any sign of the tip disturbing the fine cell structures (red arrows).

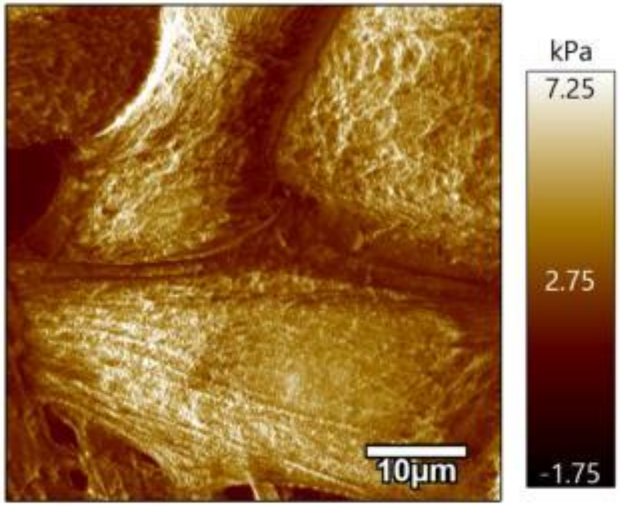
ZSensor



Fmax



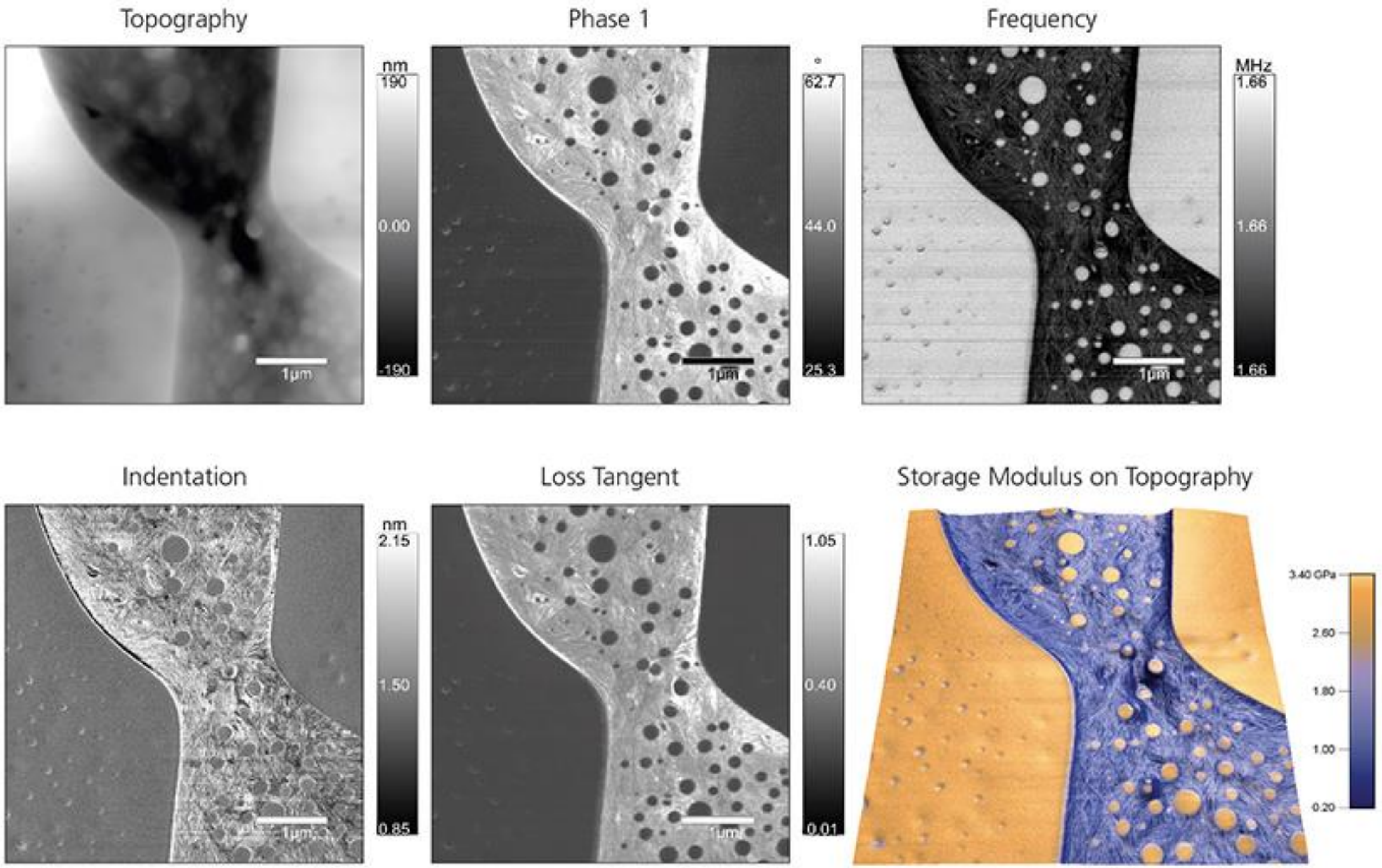
2P Hertz Fit



Sample courtesy of MD Dong, Aarhus University

AMFM

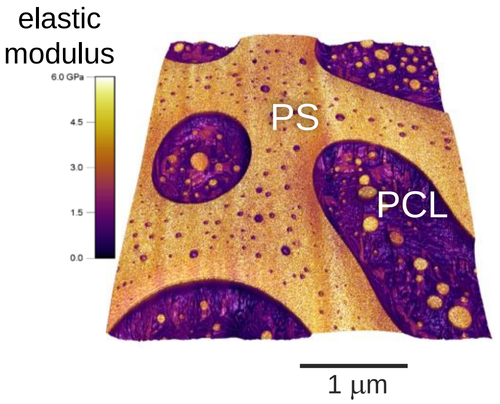
Example: Polystyrene/Polycaprolactone Blend

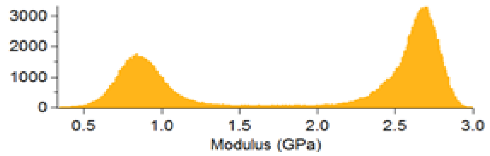


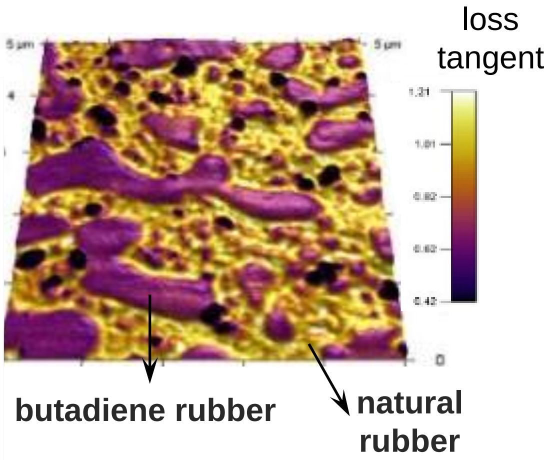
Images of a thin polymer film composed of polystyrene (E~3 GPa) and polycaprolactone (E~0.5 GPa).

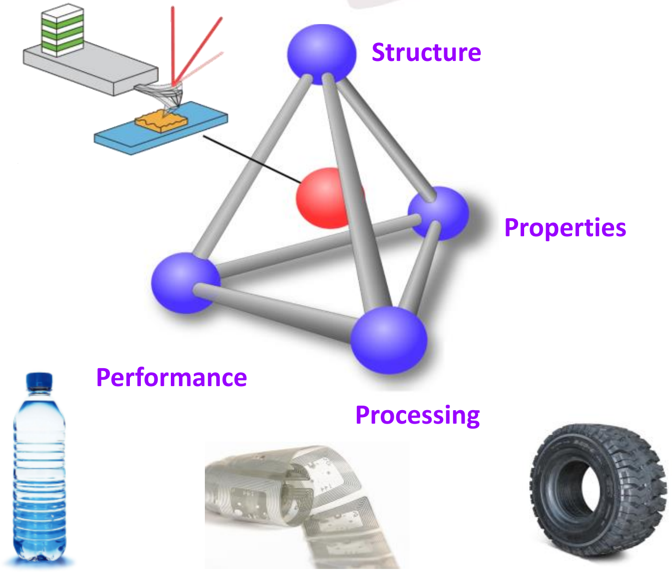
Nanomechanical Characterization

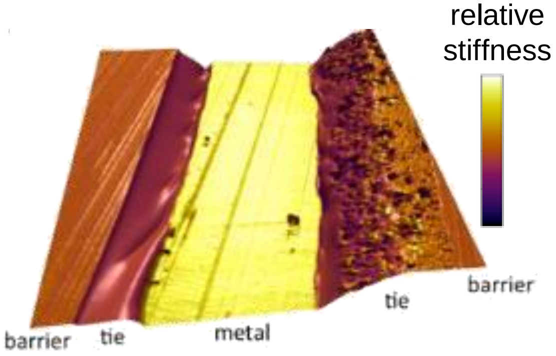
- New AFM techniques for nanomechanics have many new capabilities
  - Wide modulus range (~10 kPa to 100$^+$ GPa)
  - Fast qualitative imaging (down to ~10 s per image)
  - Quantify elastic and viscoelastic behavior







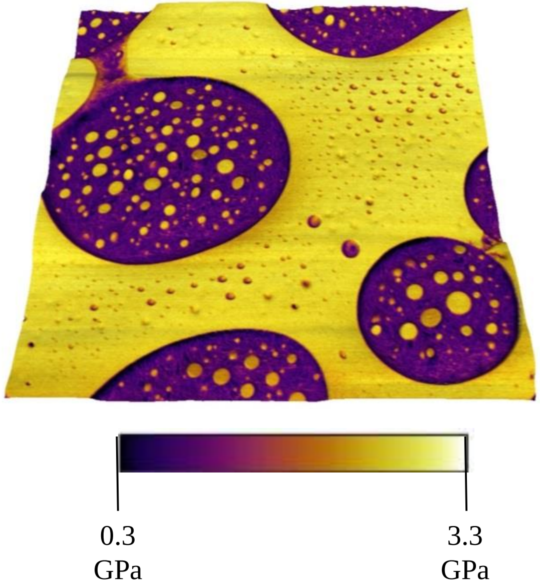




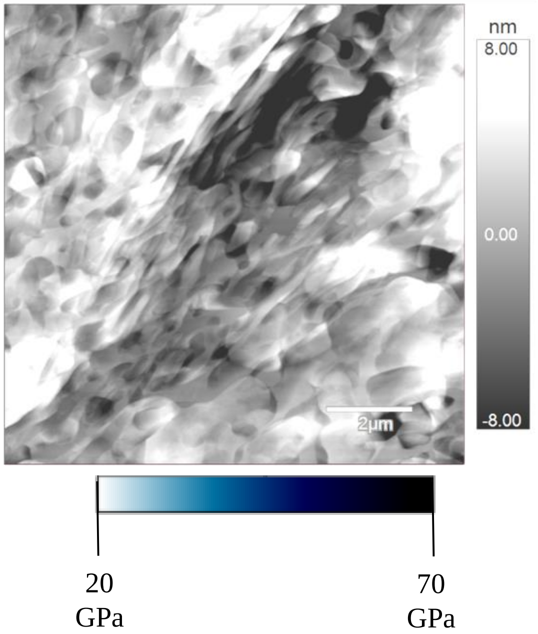
Wide range of moduli with AM-FM

- Bimodal AM-FM imaging covers a wide range of moduli
- All samples measured with the same (~30N/m) type of cantilever

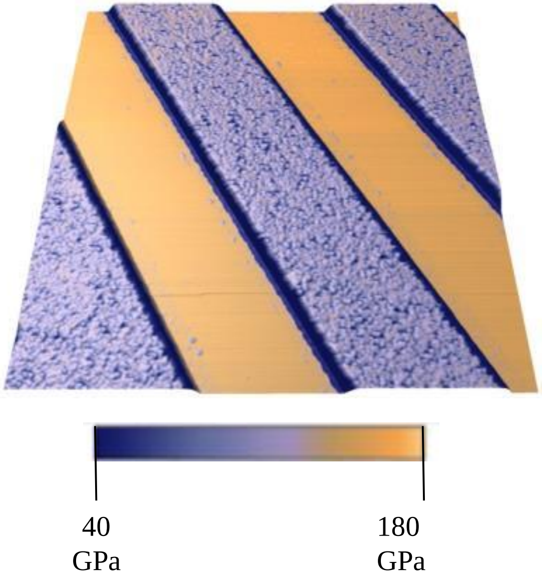
Polystyrene/ Polycaprolactone



Tin/Lead



Titanium/ Silicon

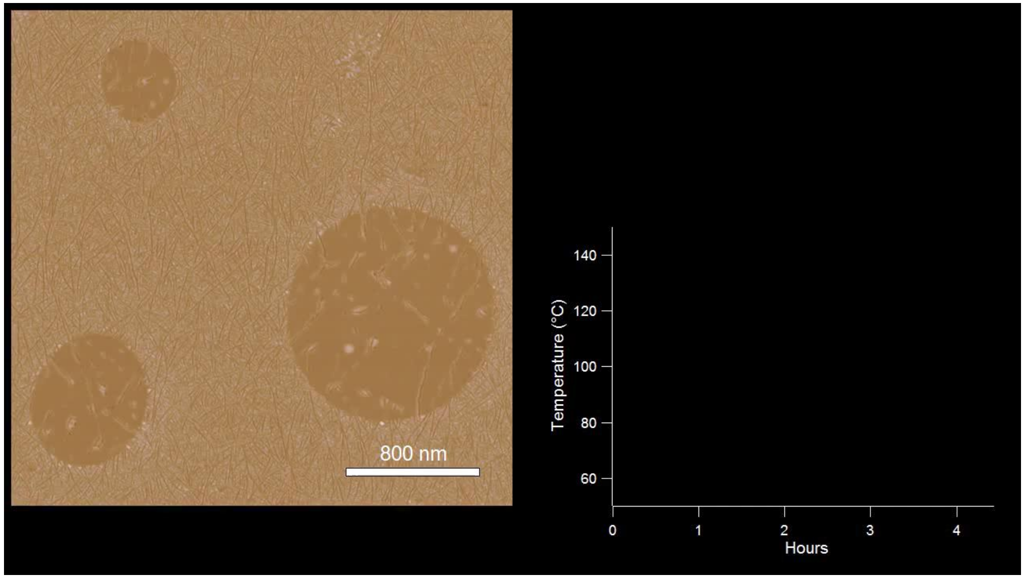


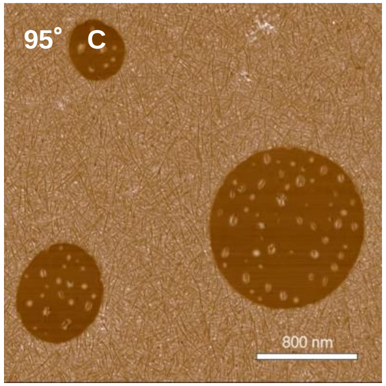
← 3 orders of magnitude →

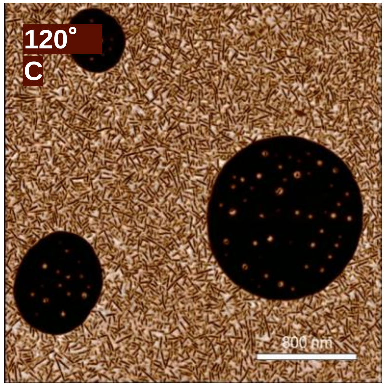
Environmental Control

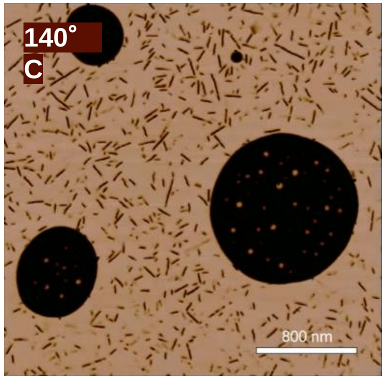
Example: Polymer Melting (Temperature)

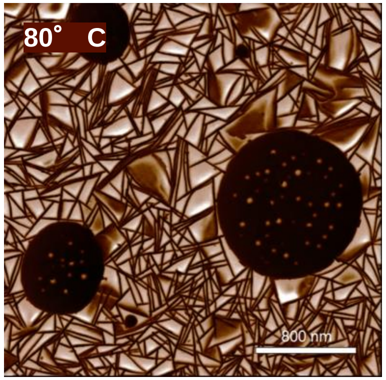
Melting and recrystallization of a sPP-PS polymer film



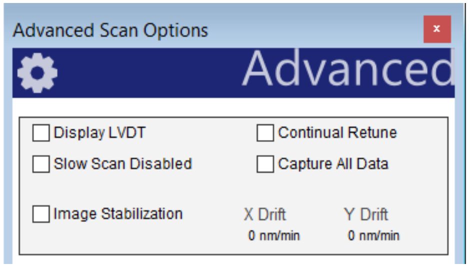








- blueDrive™
- heating, cooling



☐ Display LVDT
☐ Continual Retune
☐ Slow Scan Disabled
☐ Capture All Data
☐ Image Stabilization
X Drift 0 nm/min
Y Drift 0 nm/min

MFP3D Platform

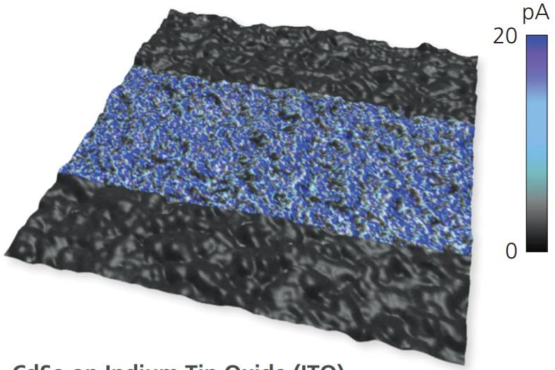
The best place to start with atomic force microscopy

- Easily the best AFM at this price point
- Supports large samples—up to 80 mm (100mm special)
- High performance—single scanner achieves atomic resolution but also
  allows huge 120 µm scans
- Simple to use without sacrificing experimental flexibility

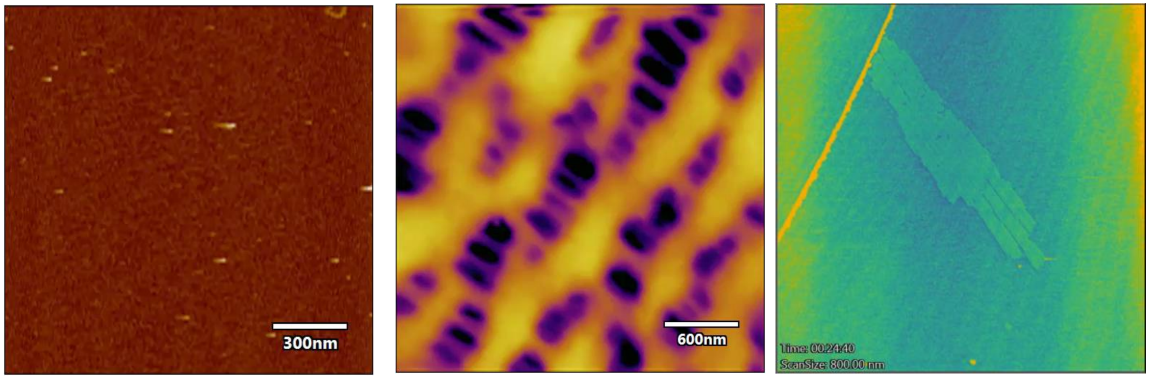
Asylum Research AFM family

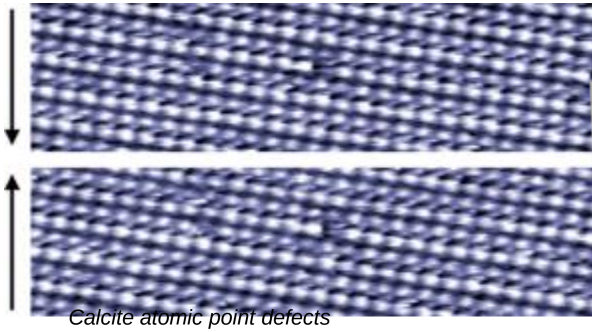
- **MFP-3D Family**
  - High performance
  - Greatest versatility
  - Mid-size samples

- **Cypher Family**
  - Ultra-high resolution
  - Fastest scanning
  - Ease of use
  - Small samples



CdSe on Indium Tin Oxide (ITO)





Calcite atomic point defects

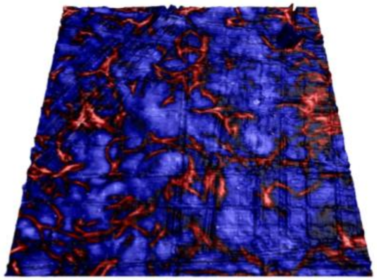
Jupiter joins the Asylum AFM family

- Jupiter is ideal for:
  - Fast scanning and automated data acquisition
  - Ease of use
  - 200mm samples
  - Automated measurements

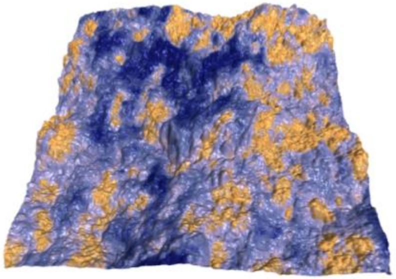
- Asylum Research commitment
  - Provide highest possible AFM performance
  - Support advanced research with innovative measurement modes and accessories
  - Deliver highest level of customer support!

NEW!

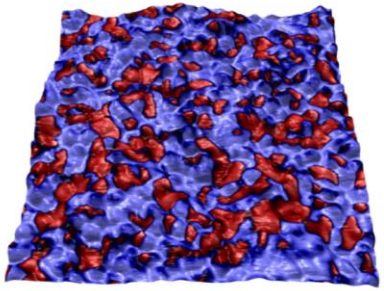
Jupiter XR AFM



Carbon nanotubes in polyimide



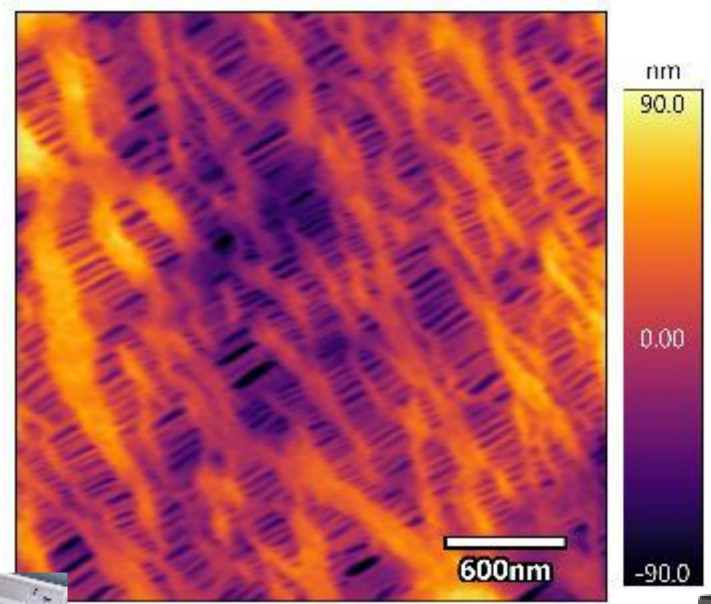
Carbon black-filled polymer film



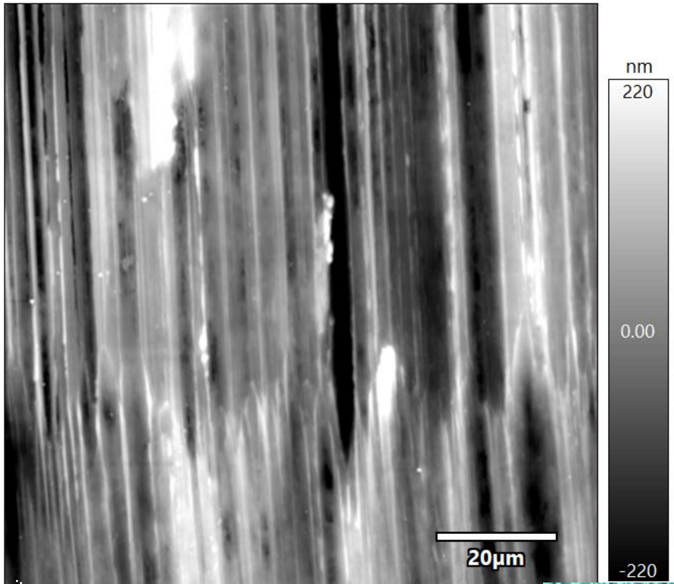
Sol-gel piezoelectric thin film

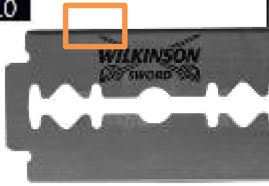
Performance = High Resolution + High Speed

- High Speed
  - Improved productivity
  - Scan rates 5-20 Hz are routine
    - images in 5-100 seconds
  - Large scans, rough samples
- Specifications
  - Laser spot size
    - compatible with small levers for fast imaging
  - blueDrive for force control and tracking



Celgard membrane imaged at 40 Hz
(**3 µm scan 26 seconds**)





Razor blade edge imaged at 2 Hz
(**100 µm scan in 130 seconds**)

Conclusion

- Tapping Mode – Fast, easy, high resolution
- Perfect complementary technics to SEM
- Different Physical properties investigation: Mechanical, Electrical….
- Watch our free online Webinar

Innovation Day 2019

10 - 11 December

INNOVATION DAY | 10 - 11 DEC | INSA, LYON

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Thank you for your attention