University of BRISTOL

IRC in Nanotechnology

# AFM at Video Rate and Beyond


Mervyn Miles

H.H. Wills Physics Laboratory
IRC in Nanotechnology
University of Bristol
Tyndall Avenue
Bristol
U.K.



Frontiers in Scanning Probe Microscopy,
Purdue University, October 2006

# Atomic Force Microscopy: Strengths

- High-resolution 3-D imaging;
- Imaging in liquid, air, & vacuum;
- No staining or coating required;
- No radiation damage;
- Mapping of physical properties;
- Modification of surfaces;
- Non-scanning applications: force spectroscopy, sensors, ...

# Speed Weakness of SPM

- Imaging rate too low:
  - to follow many processes;
  - to examine large areas of a specimen;
  - to create or manipulate structures over usefully large areas.

# Limitations of Conventional SPM

## General to all SPMs

- Scanning system ~ inertia and resonance problems
- Feedback loop response time
- Response of interaction sensor

## Specific to AFM

- Force-sensing cantilever has inertia and resonance

# How to go faster:

- Parallel imaging with multiple cantilevers
- Strobe imaging of repetitive processes
- Decrease Q in AC modes
- Shift the time domain using higher frequencies
- Another method ....

# Solution I

- Decrease the mass of the scanning system
- Increase the stiffness of the scanning system
- Decrease the mass of the cantilever
- Increase the stiffness of the cantilever
- Decrease the Q factor of the cantilever

# A Different Solution

Instead of avoiding resonance,

use a resonating beam

for scanning in the fast direction

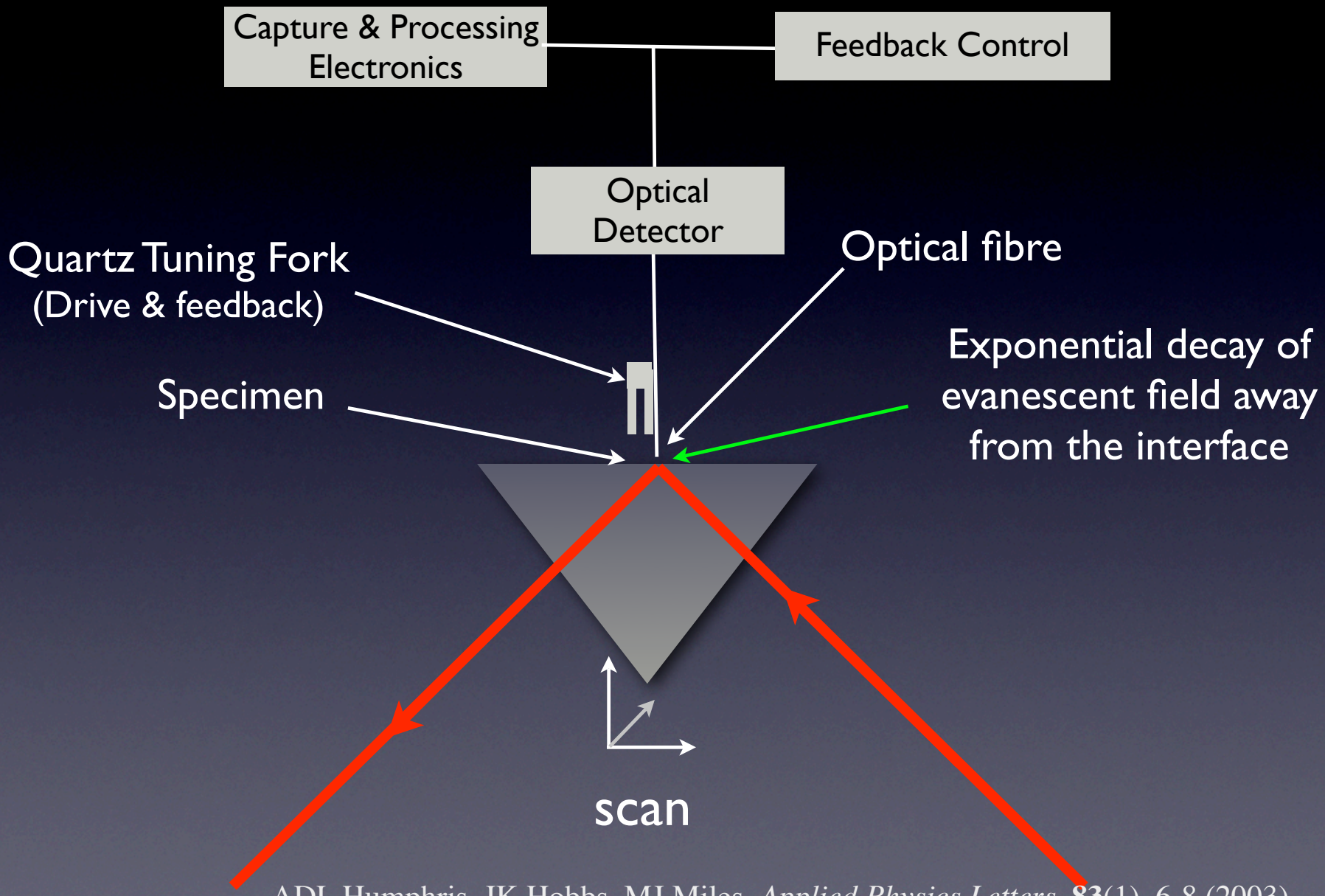
# Solution 2

- Scan by oscillating at resonance with a high amplitude - resonant scanning microscopy (RSM);
- High Q-factor results in high scan stability;
- Data are collected throughout each sweep;

Before high-speed AFM,

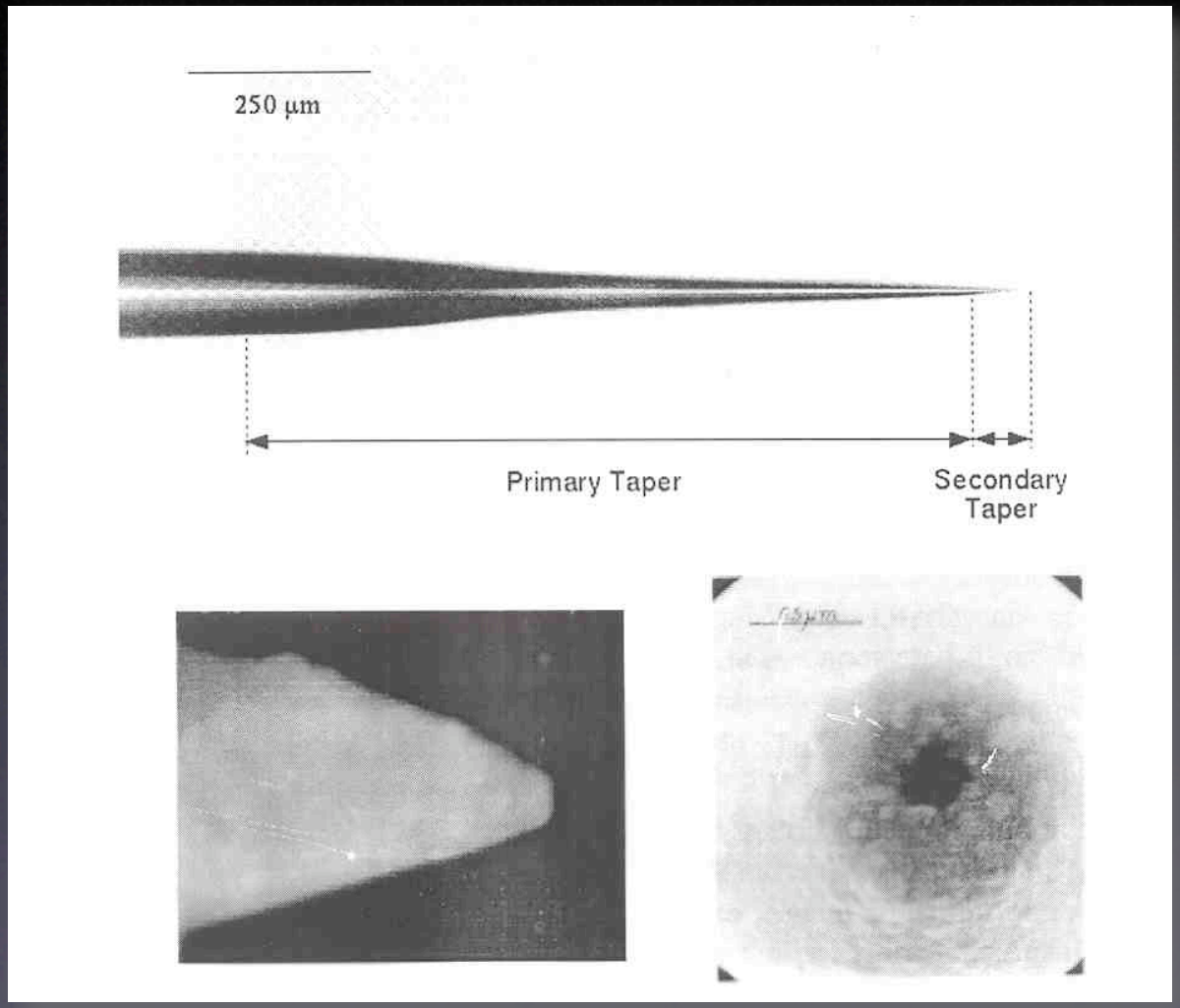
we first built a high-speed SNOM

# Scanning Near-field Optical Microscopy (SNOM)

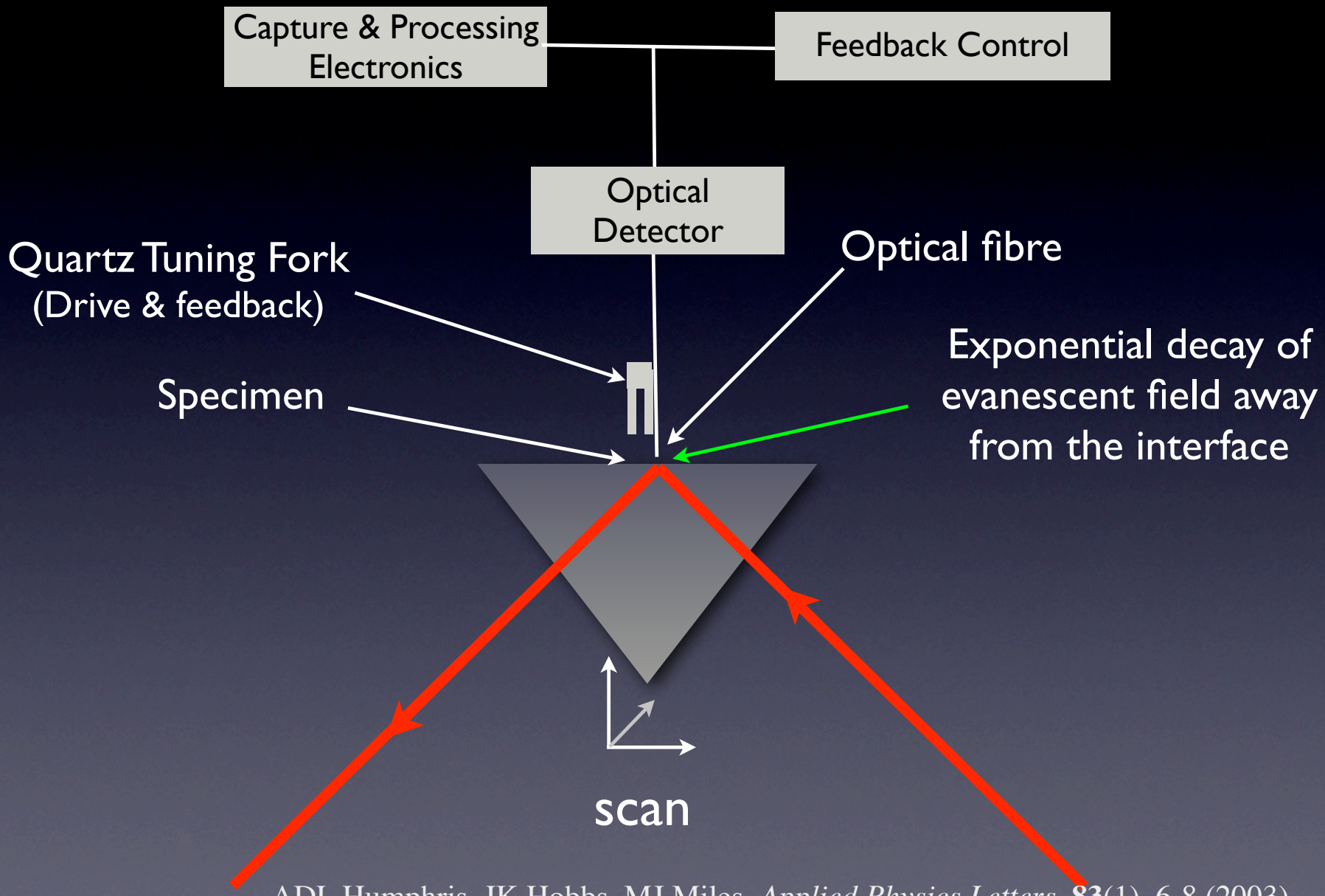



ADL Humphris, JK Hobbs, MJ Miles, *Applied Physics Letters*, **83**(1), 6-8 (2003)

# Melt-tapered optical fibre SNOM probe

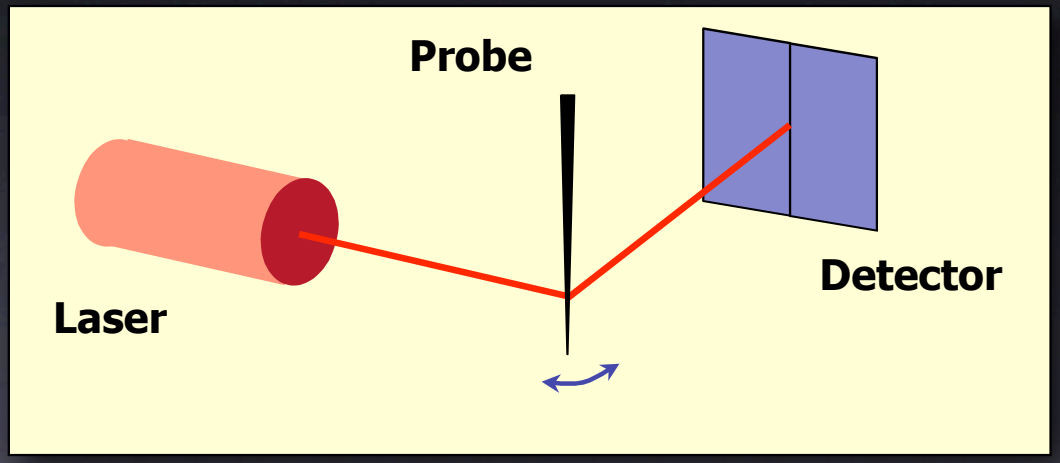


# Scanning Near-field Optical Microscopy (SNOM)

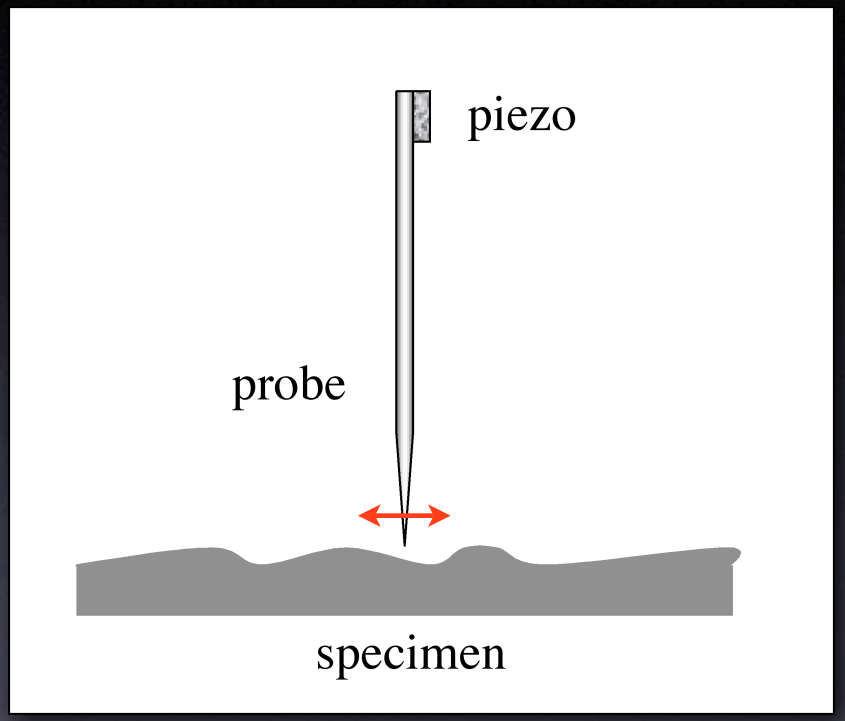



ADL Humphris, JK Hobbs, MJ Miles, *Applied Physics Letters*, **83**(1), 6-8 (2003)

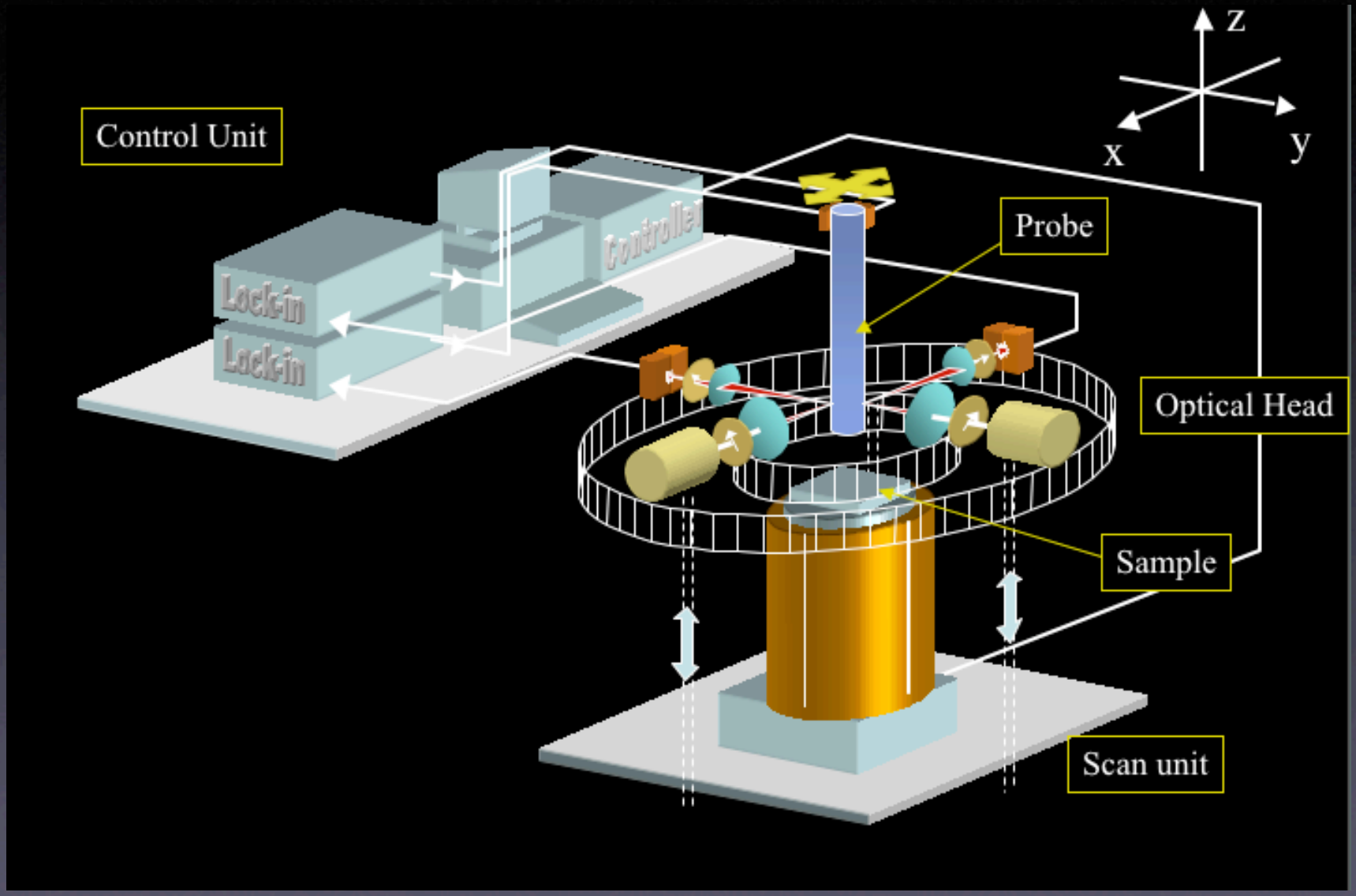
# Transverse dynamic force microscope (TDFM)



# Low-amplitude (~1 nm) Oscillations

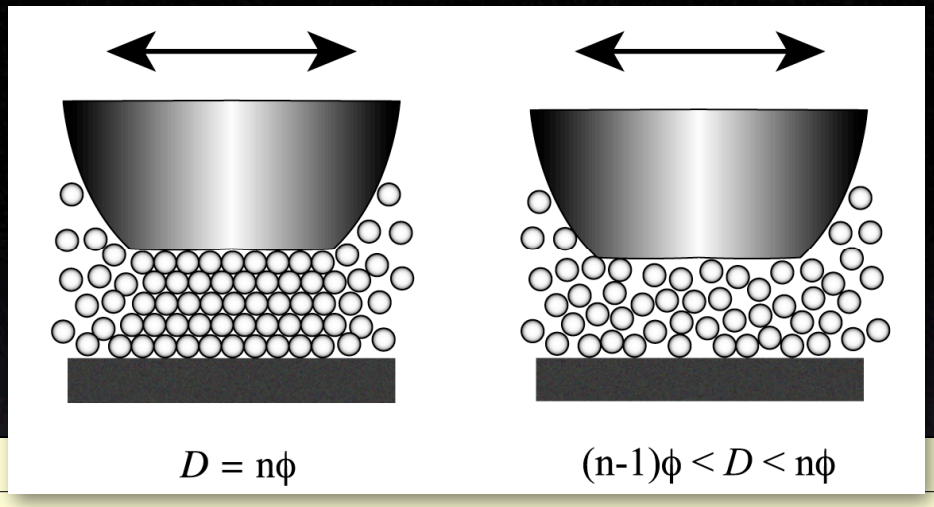


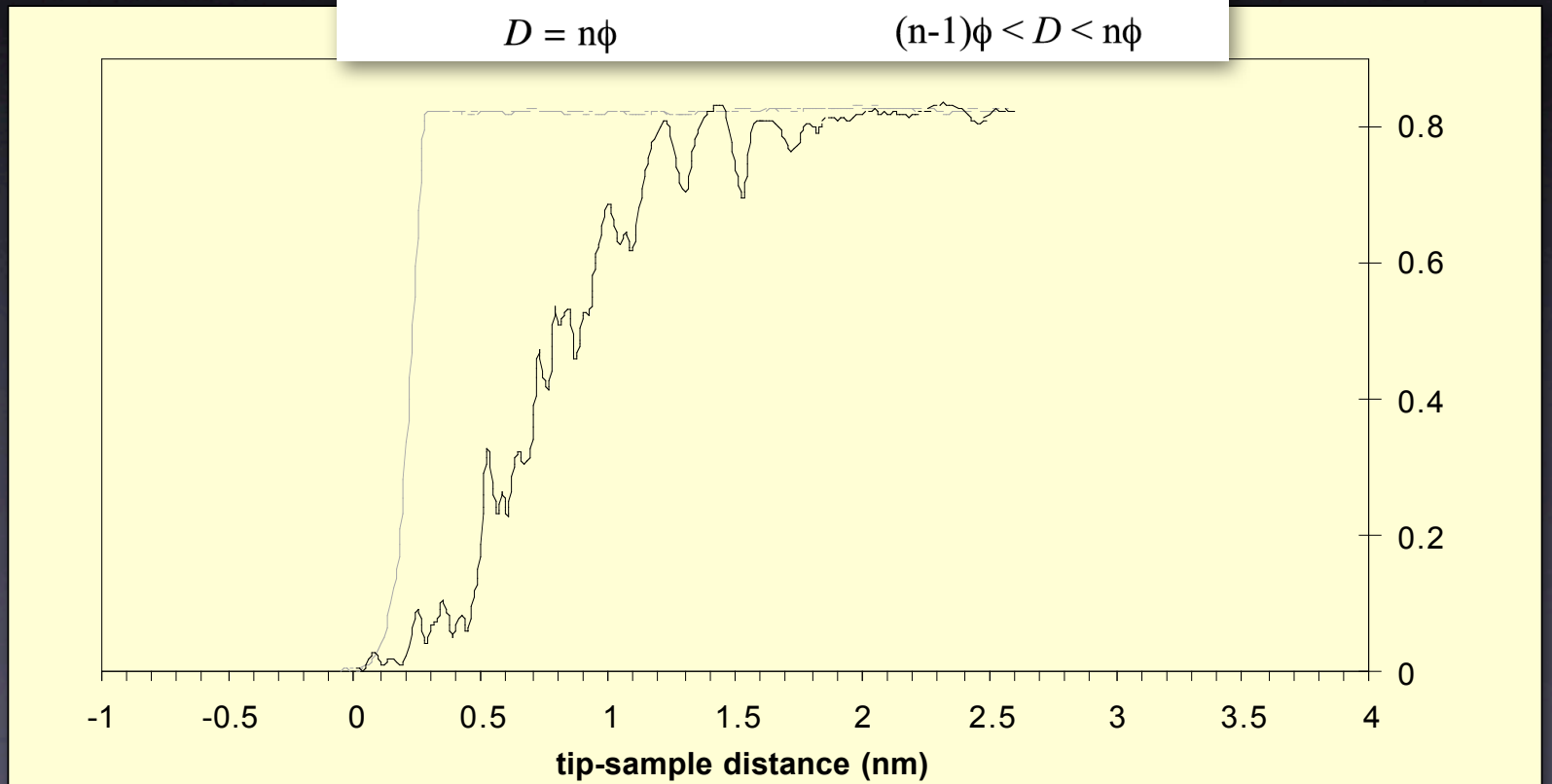
# Transverse Dynamic Force Microscope, TDFM




M Antognozzi

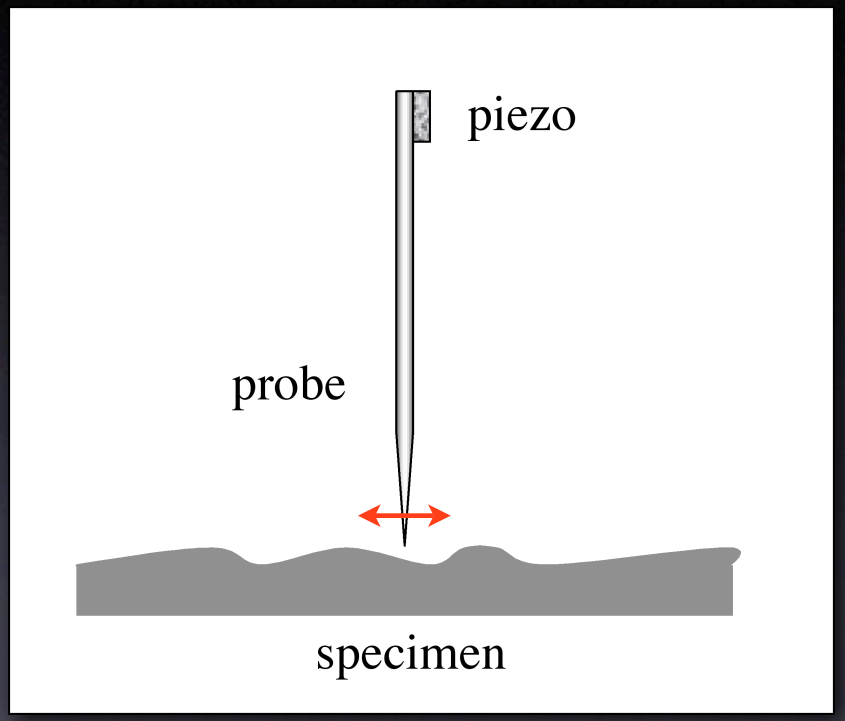
# Surface detection via confined water layer



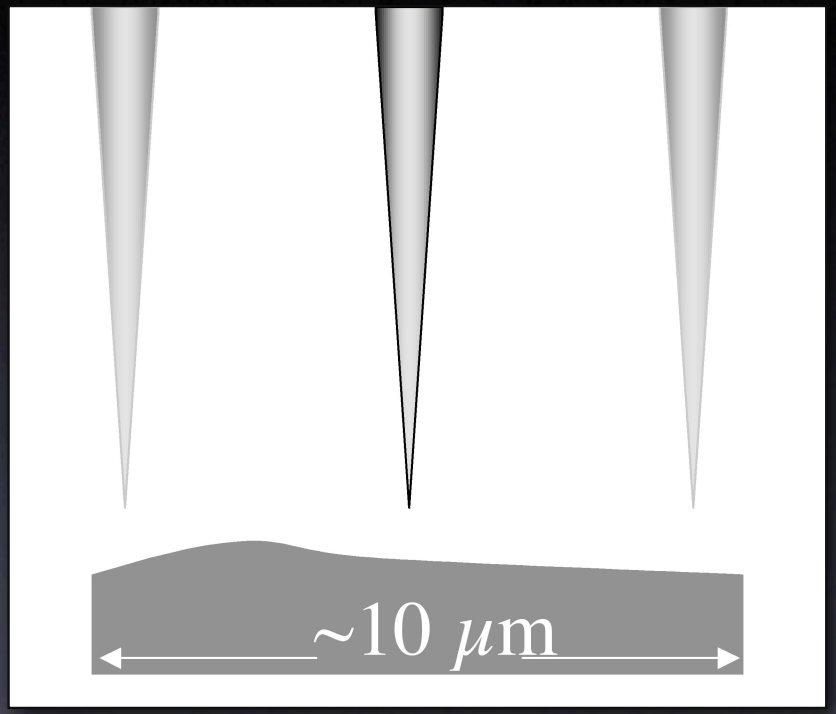

M Antognozzi, ADL Humphris, MJ Miles, *APPLIED PHYSICS LETTERS*, **78**(3), 300-302 (2001)

# Low-amplitude (~1 nm) Oscillations



## For high-speed SNOM

High-amplitude (~ 5 μm) for fast axis scanning



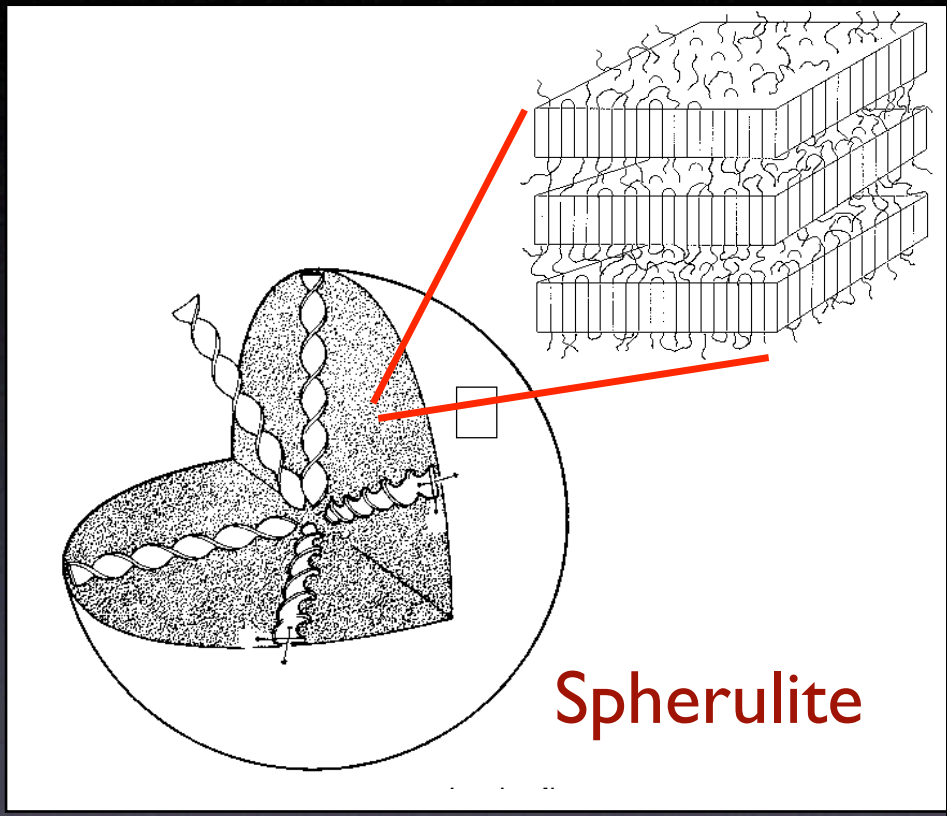

Tip scanning in fixed plane set by average optical intensity

ADL Humphris, JK Hobbs, MJ Miles, *Applied Physics Letters*, **83**(1), 6-8 (2003)

Several crystals may nucleate together and grow to form a spherical structure known as a spherulite.

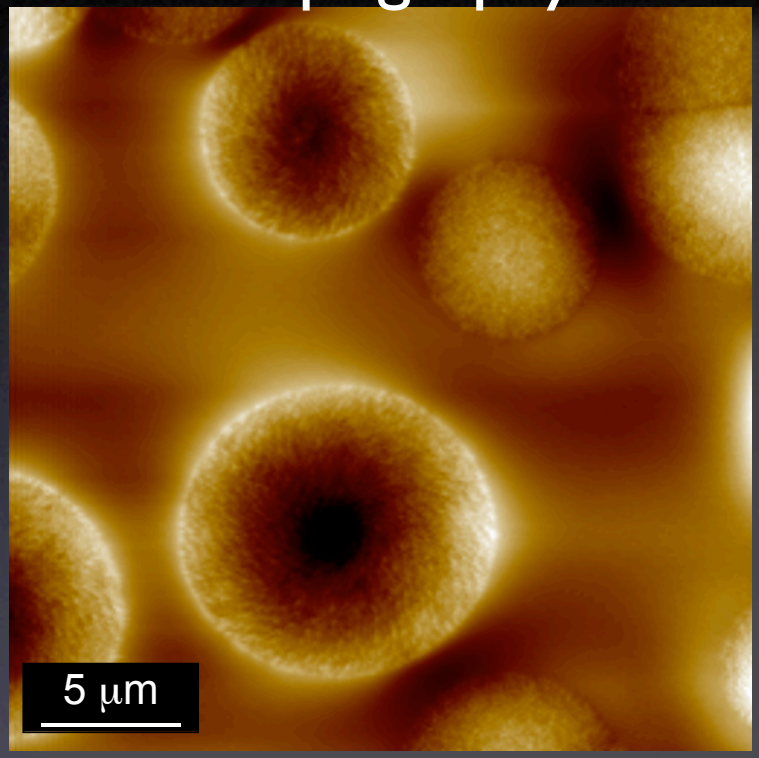
Within the spherulite, the polymer crystals grow radially from the centre in a ribbon morphology

These ribbon crystals often twist as they grow with the same pitch and remain in phase.

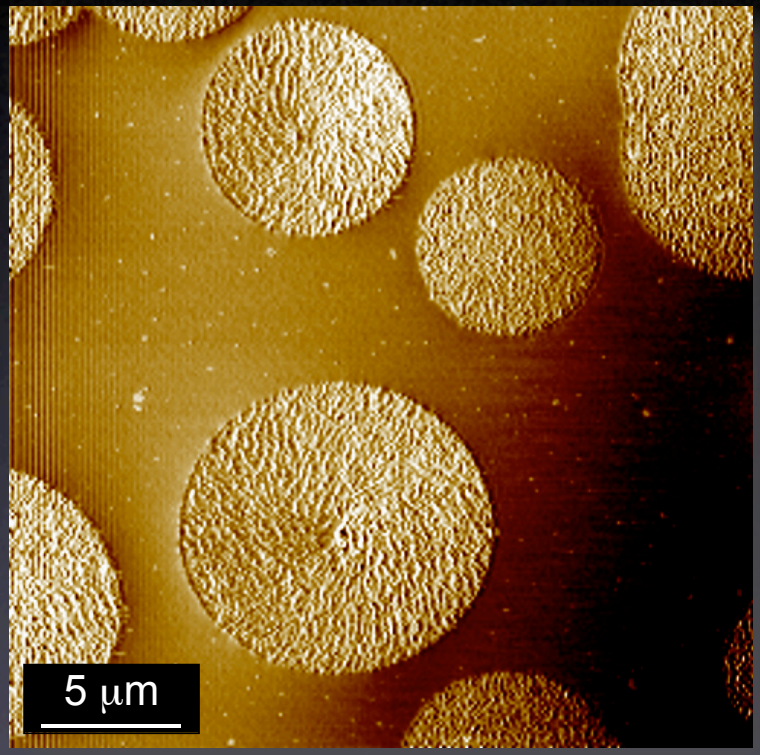



In a thin film, the spherulite is a disk-like structure in which the coherent twisting of the ribbon crystals results in concentric rings with the ribbons alternately flat on and edge.

Topography



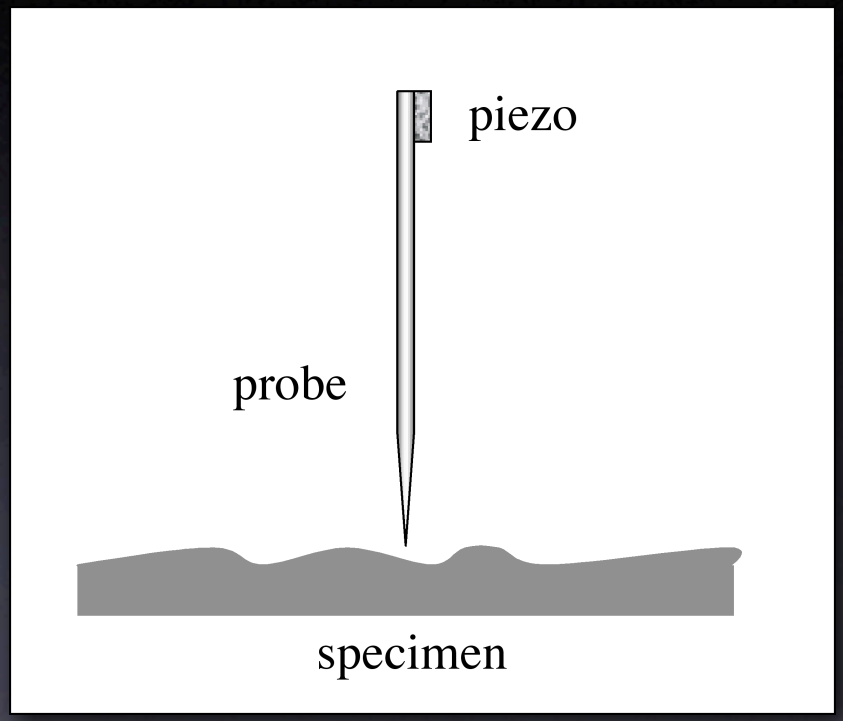

Phase

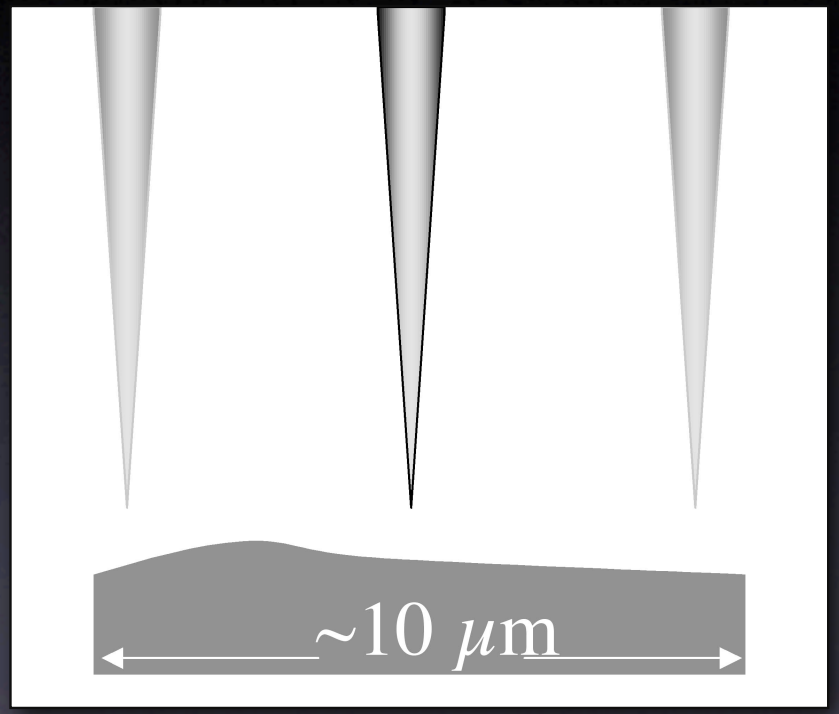



AFM

Quality factor has been reduced from 270 to 90.
Tip velocity was 403μm/s, line rate 8Hz

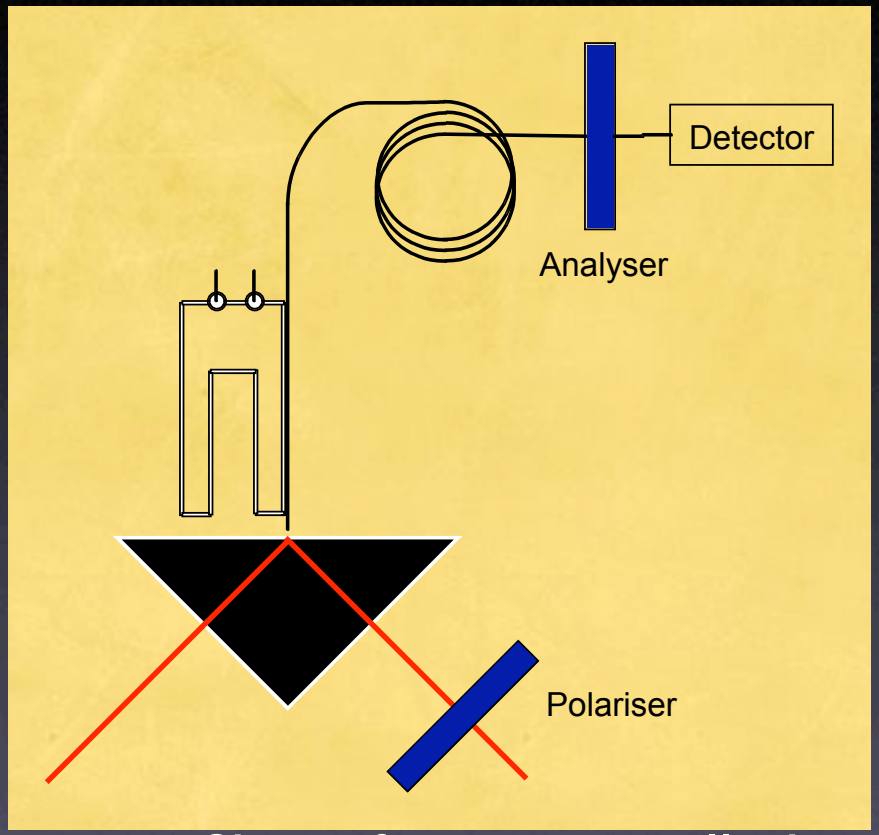
# High Amplitude Oscillations



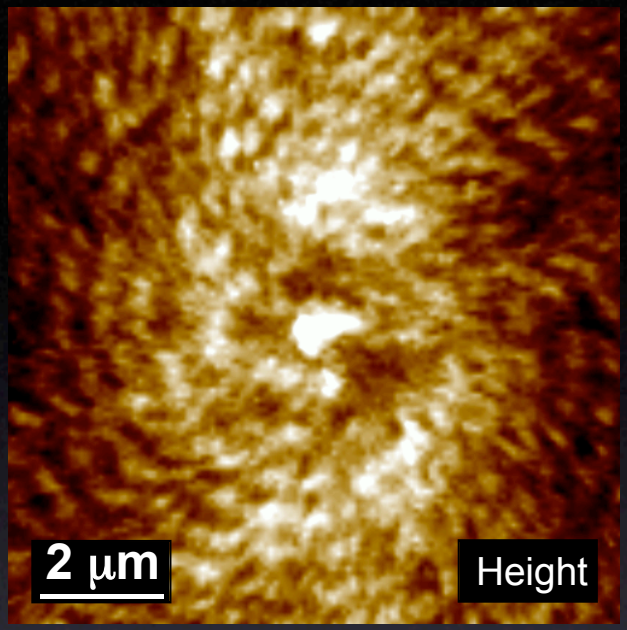



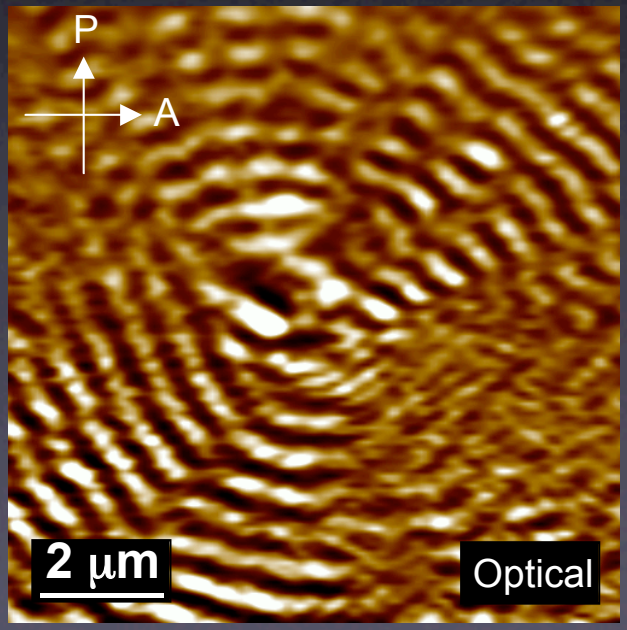
# Birefringent PSTM

Slow scanning - 20 minutes/image
assisted with active Q



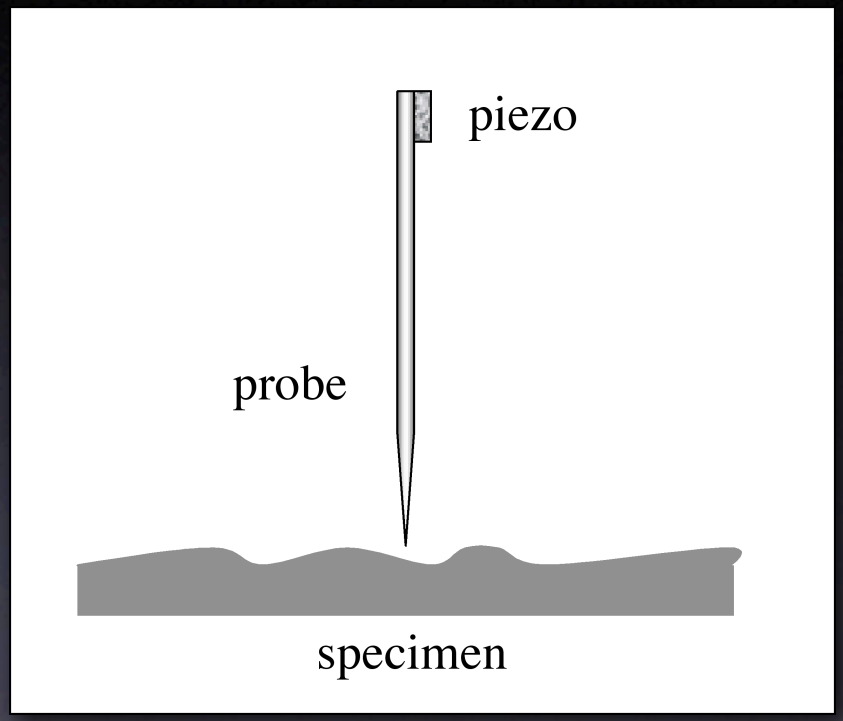

Shear-force controlled
via tuning fork

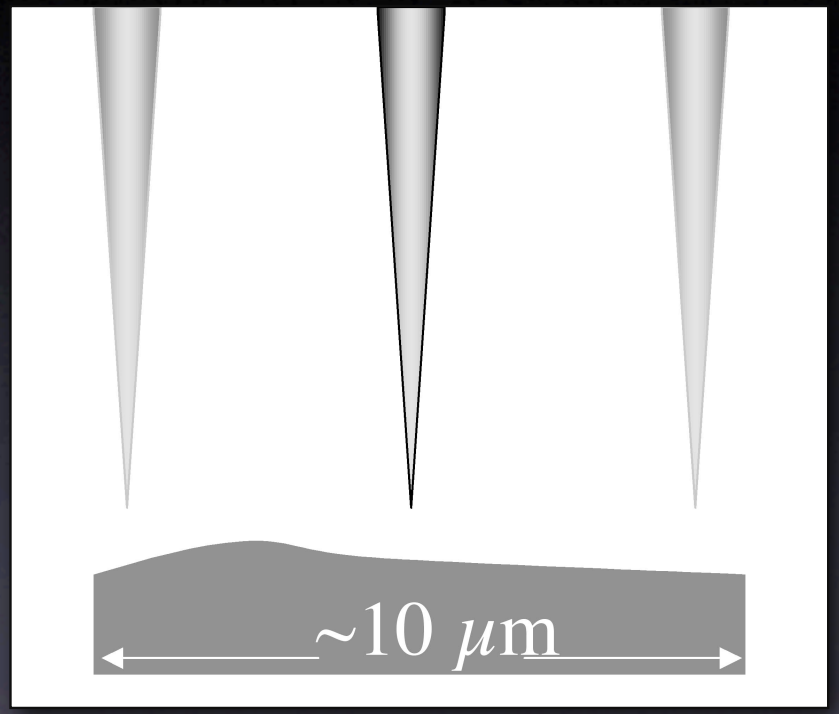



ADL Humphris/JK Hobbs
see, e.g., RL Williamson, MJ Miles, *JOURNAL OF VACUUM SCIENCE & TECHNOLOGY B*, **14**(2), 809-811 (1996

# High Amplitude Oscillations



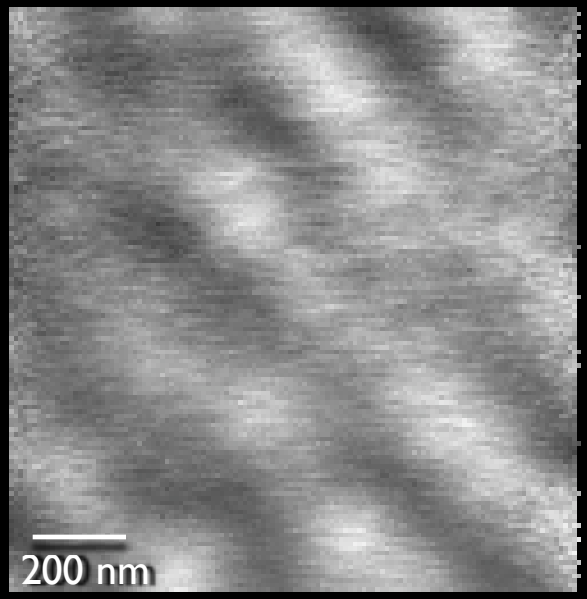
# Comparison: Conventional SNOM & RSM SNOM

Conventional SNOM

1 frame in 1000 sec

RSM SNOM

120 frames/sec

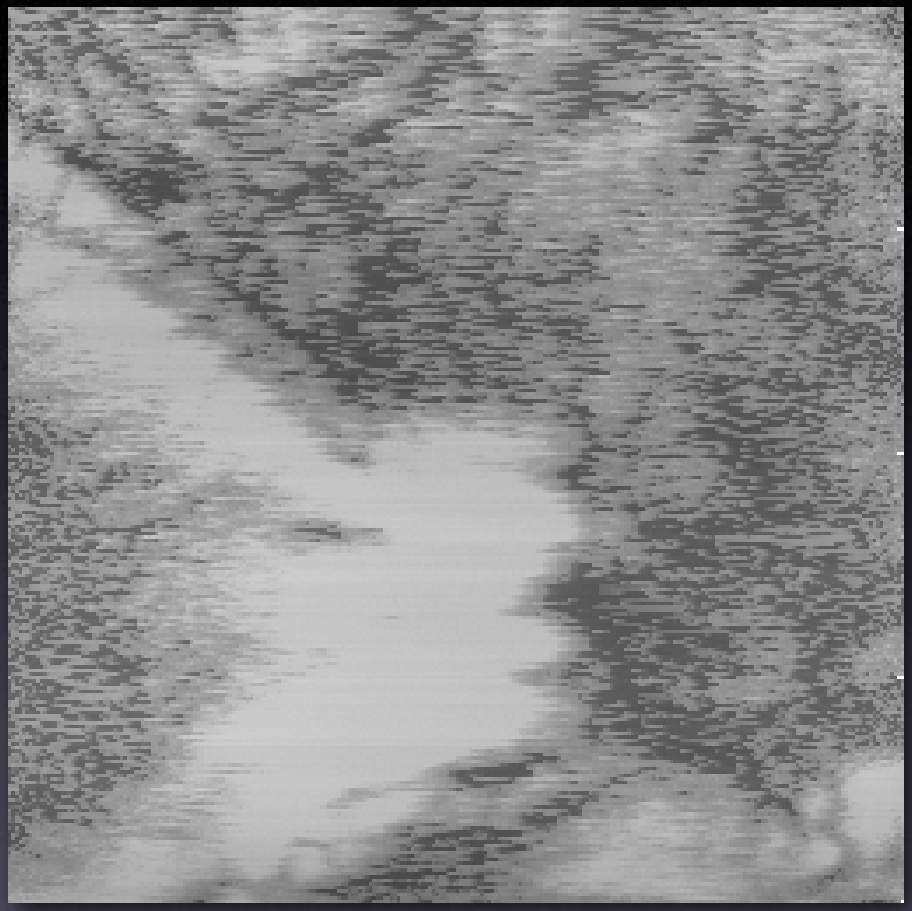



**> 100,000 Faster than existing SNOM**

PHB spherulite between crossed polars

ADL Humphris, JK Hobbs, MJ Miles, *Applied Physics Letters*, **83**(1), 6-8 (2003)

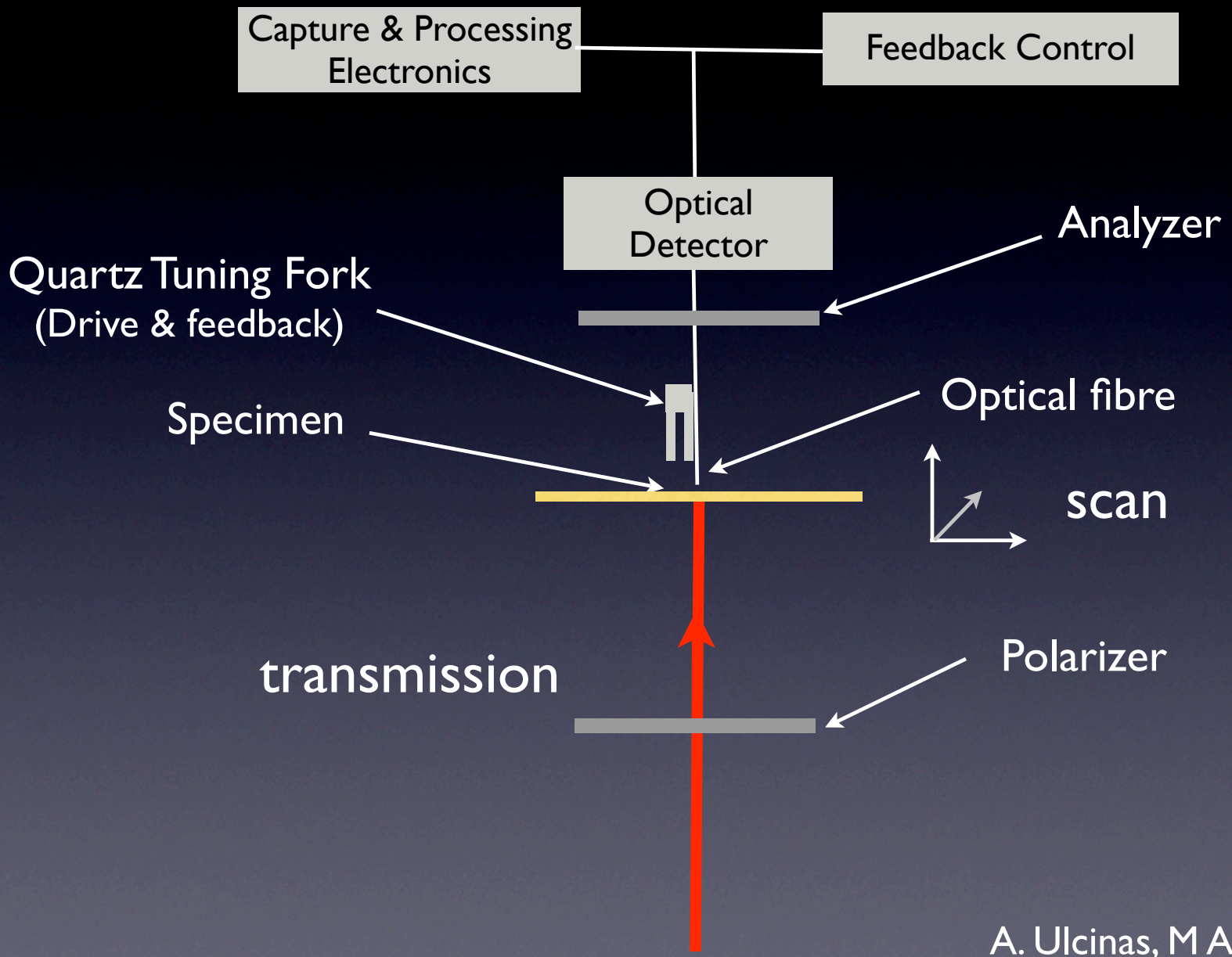
# High-speed SNOM of collagen



Intensity related to height -
67 nm repeat of collagen visible

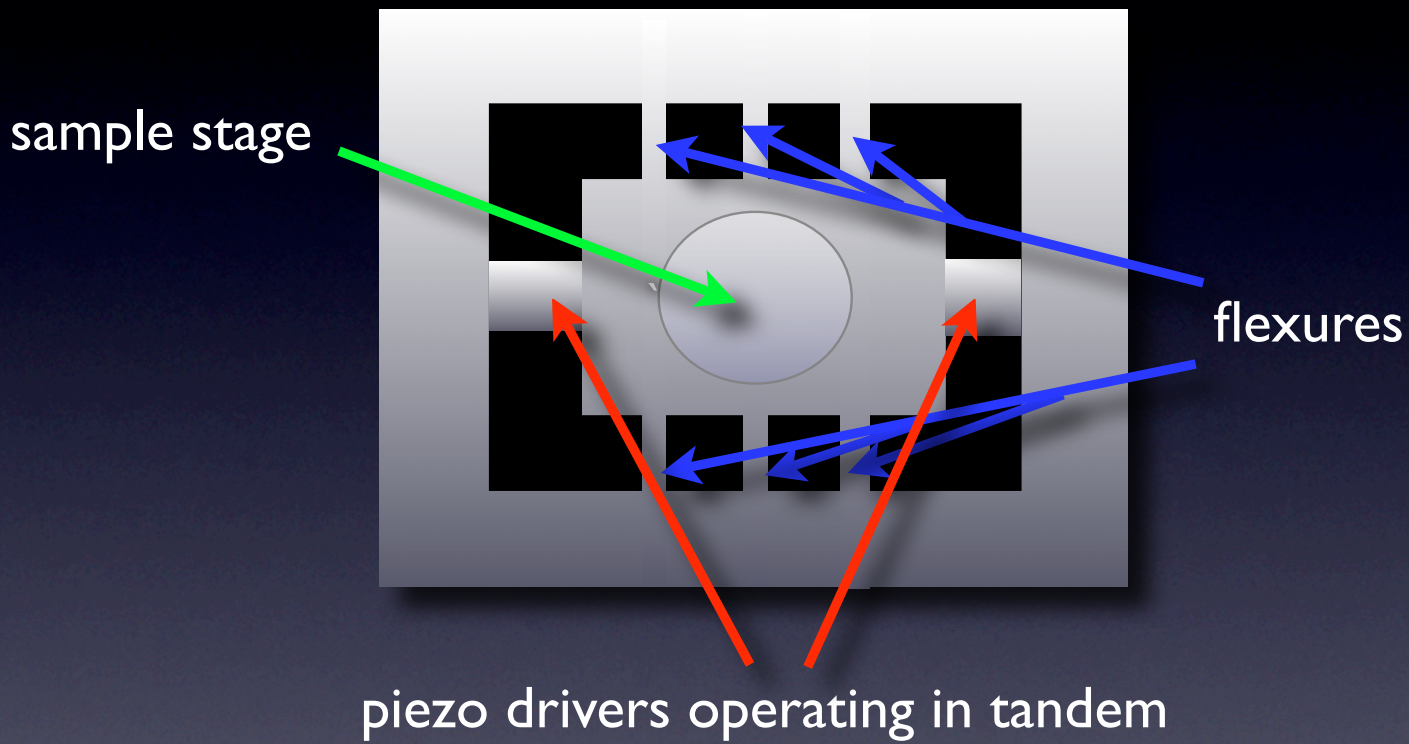
A Major (Uni Wien, ), L Bozec, MA Horton, Miles

# Scanning Near-field Optical Microscopy (SNOM)




A. Ulcinas, M Antognozzi

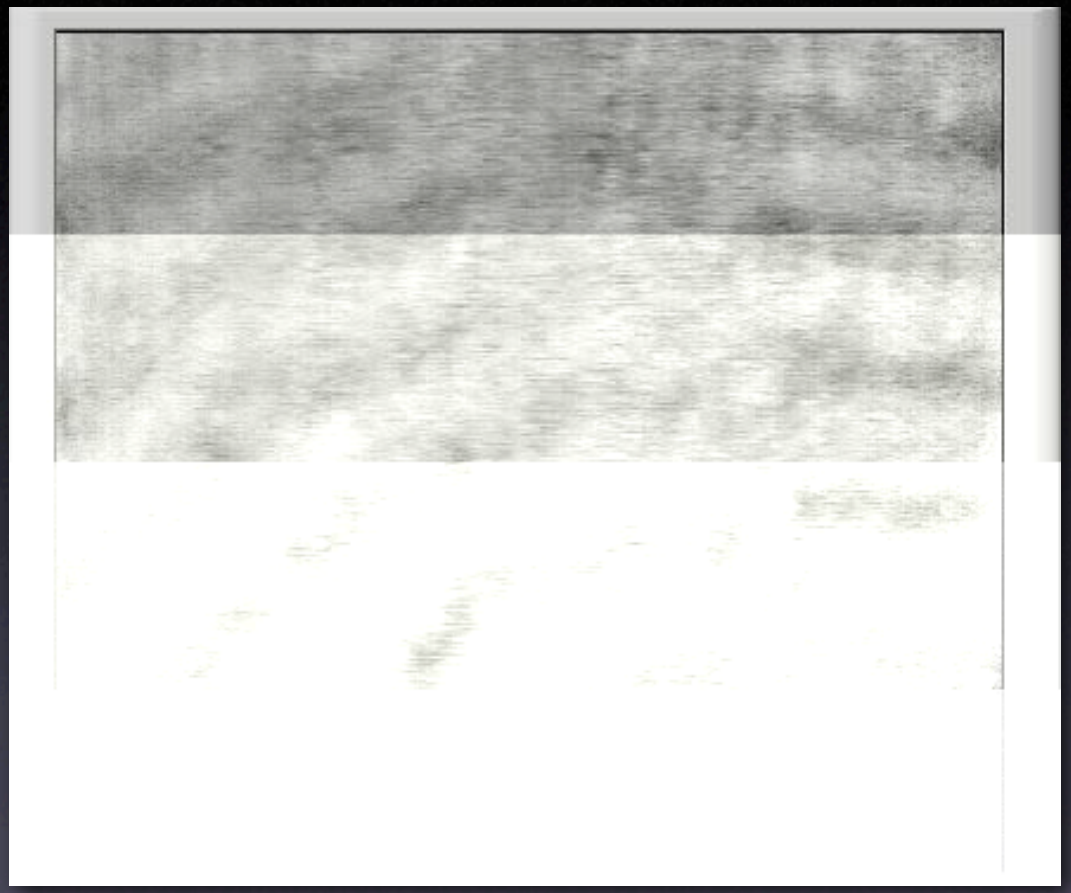
# Flexure Stage for high-speed SPM




This flexure stage provides the high-speed scan of up to 40kHz with about 3 μm amplitude

Picco, Engledew

# High-speed SNOM of polymer spherulite of PHB



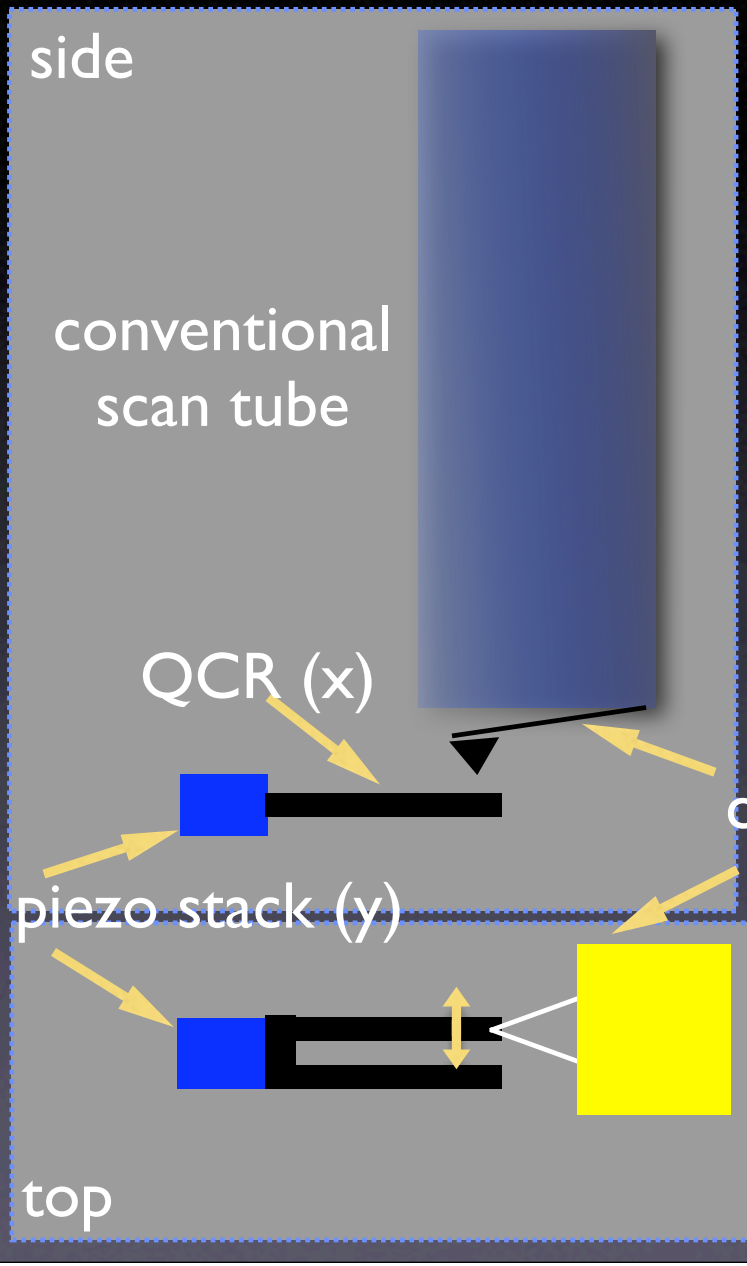
Flexure stage scanner - 3 μm x 3 μm image

Transmission SNOM with crossed polars

A. Ulcinas, M Antognozzi, Picco, Engledew

# High-speed AFM

# Resonant scanning AFM




- Resonant sample scan stage (x)
- Piezo stack for 'slow' scan (y)
- No electronic feedback
- Mechanical feedback
  - super lubricity
  - water in confined geometry

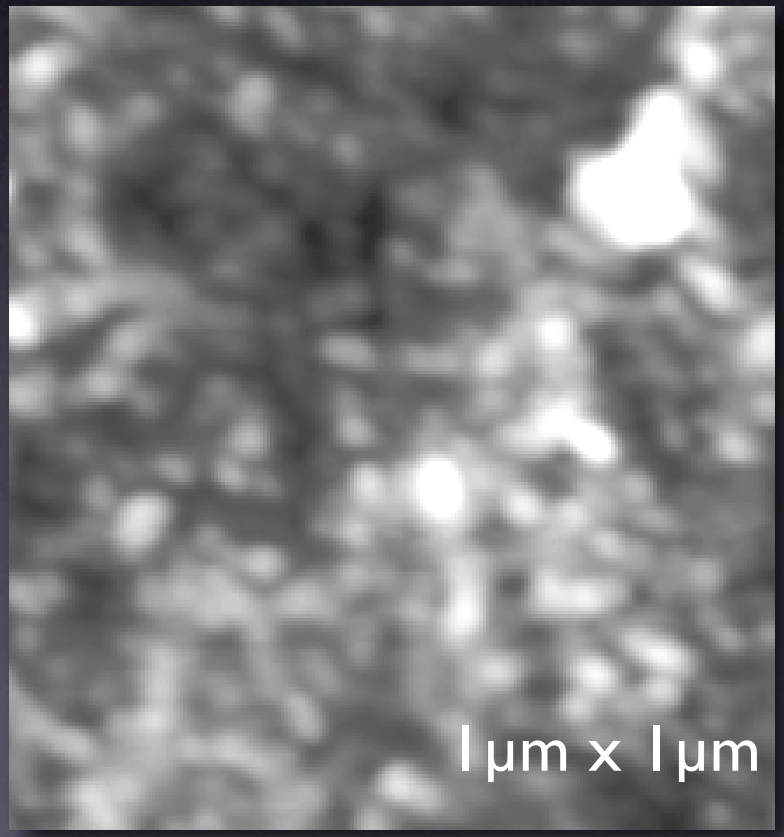
ADL Humphris, MJ Miles, JK Hobbs,
*Applied Physics Letters*, **86**(3), Art.No.

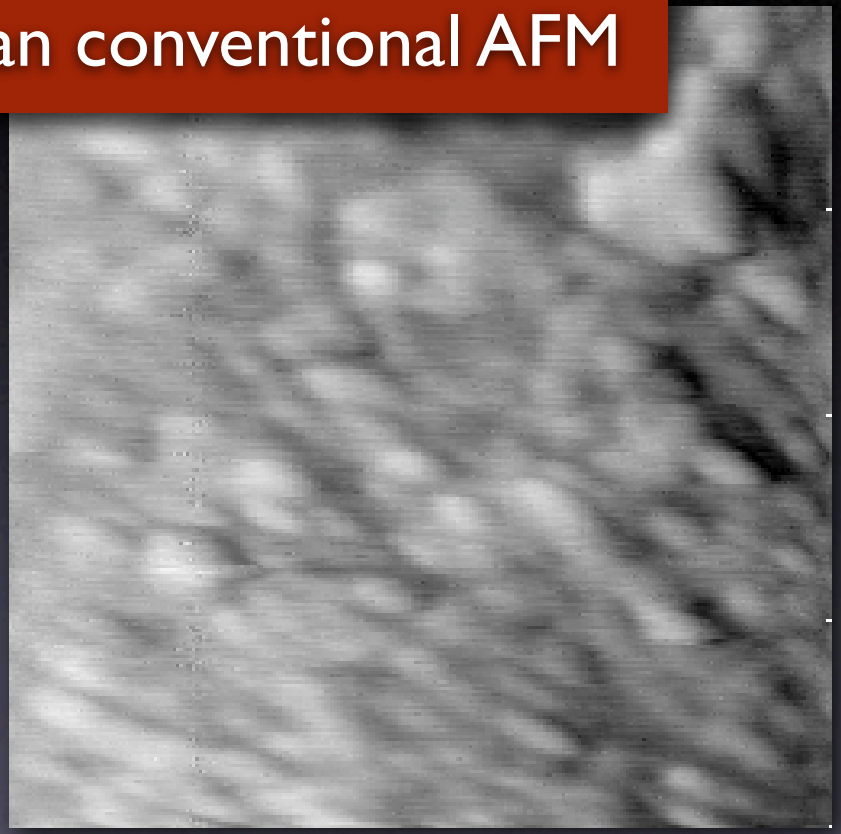
# High-speed AFM of Chitosan Film

- tuning-fork resonance scanning

**> 3000 Faster than conventional AFM**

Conventional AFM

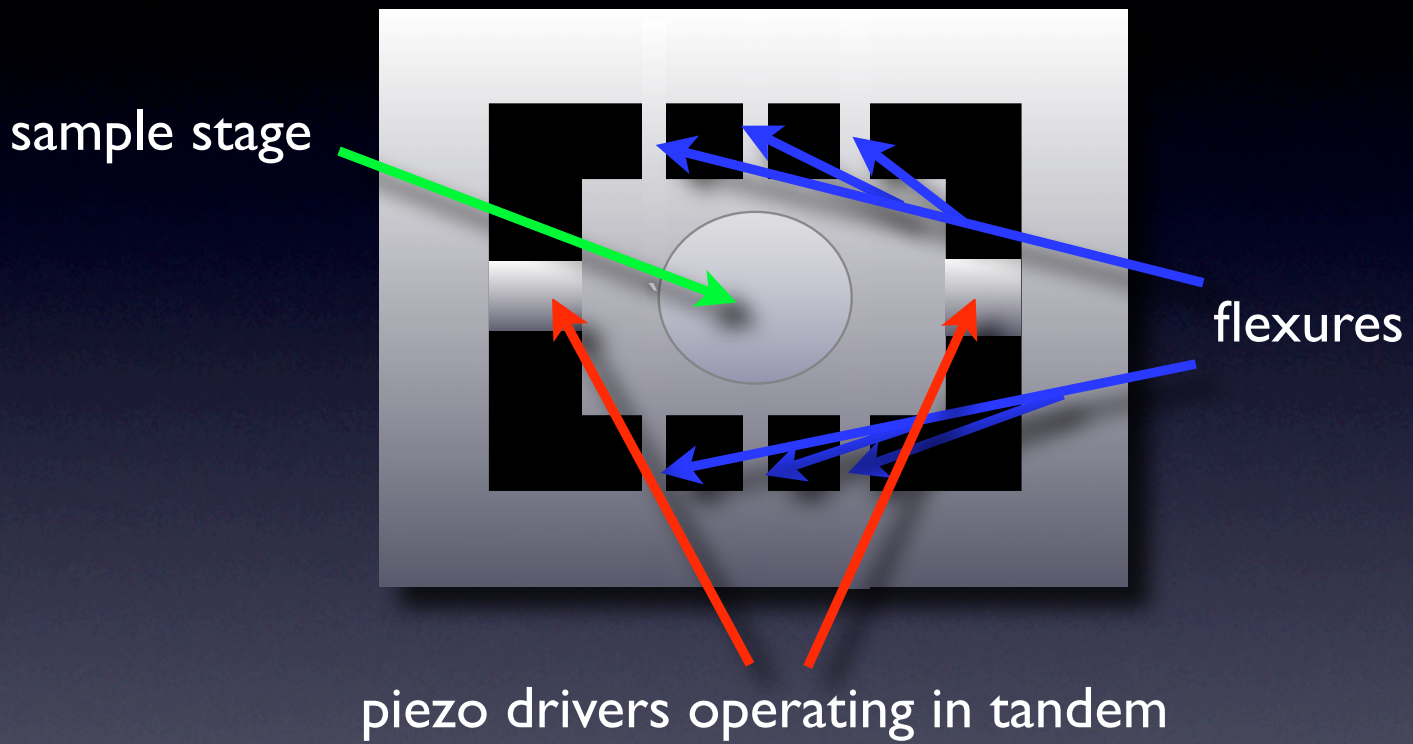


30 frames/s
(Infinitesima vAFM)

Ulcinas, Payne, Heppenstall-Butler

# Flexure Stage for high-speed SPM

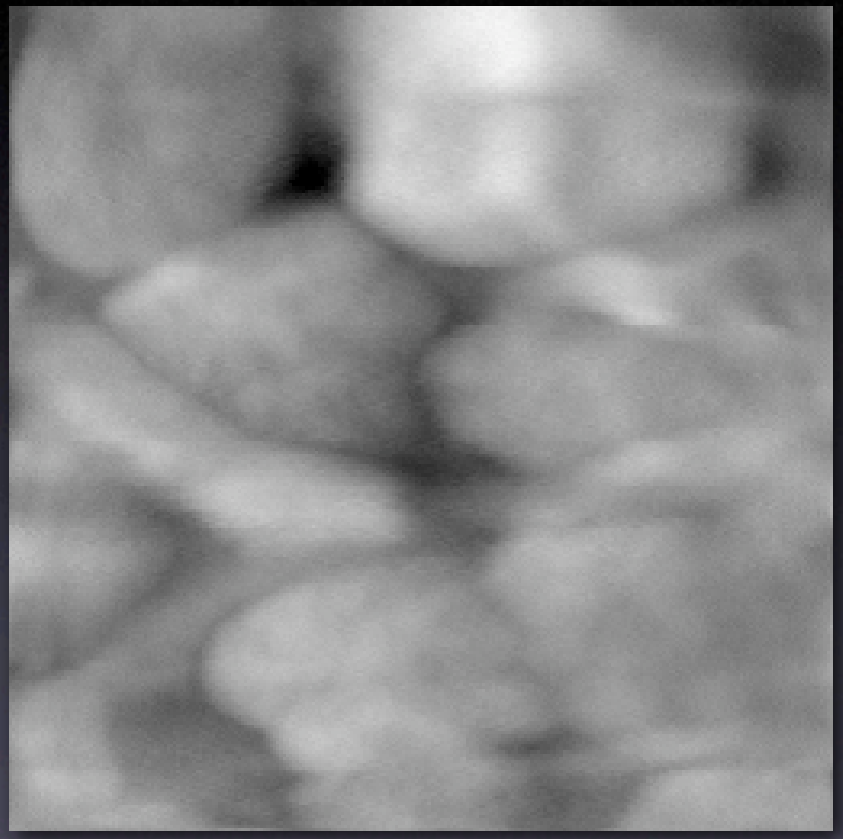



This flexure stage provides the high-speed scan of up to 40kHz with about 3 μm amplitude

Picco, Engledew

# PEO Spherulite video AFM

- flexure stage (non-resonance) scanning



30 frames/second
1.5 μm x 1.5 μm

Engledew, Picco, Miles

# PEO Spherulite video AFM

30 frames/second

- flexure stage (non-resonance) scanning

Engledew, Picco, Miles

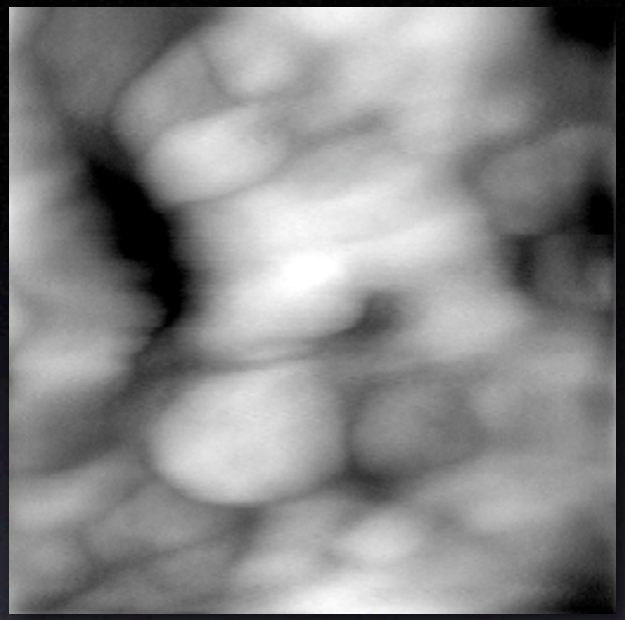
# PEO Spherulite video AFM

30 frames/second
1.5 μm x 1.5 μm

Engledew, Picco, Miles

# PEO video AFM 30 f/s - image reproducibility

0 mins
0 frames



3 mins
~ 6000
frames

7 mins
13,000
frames

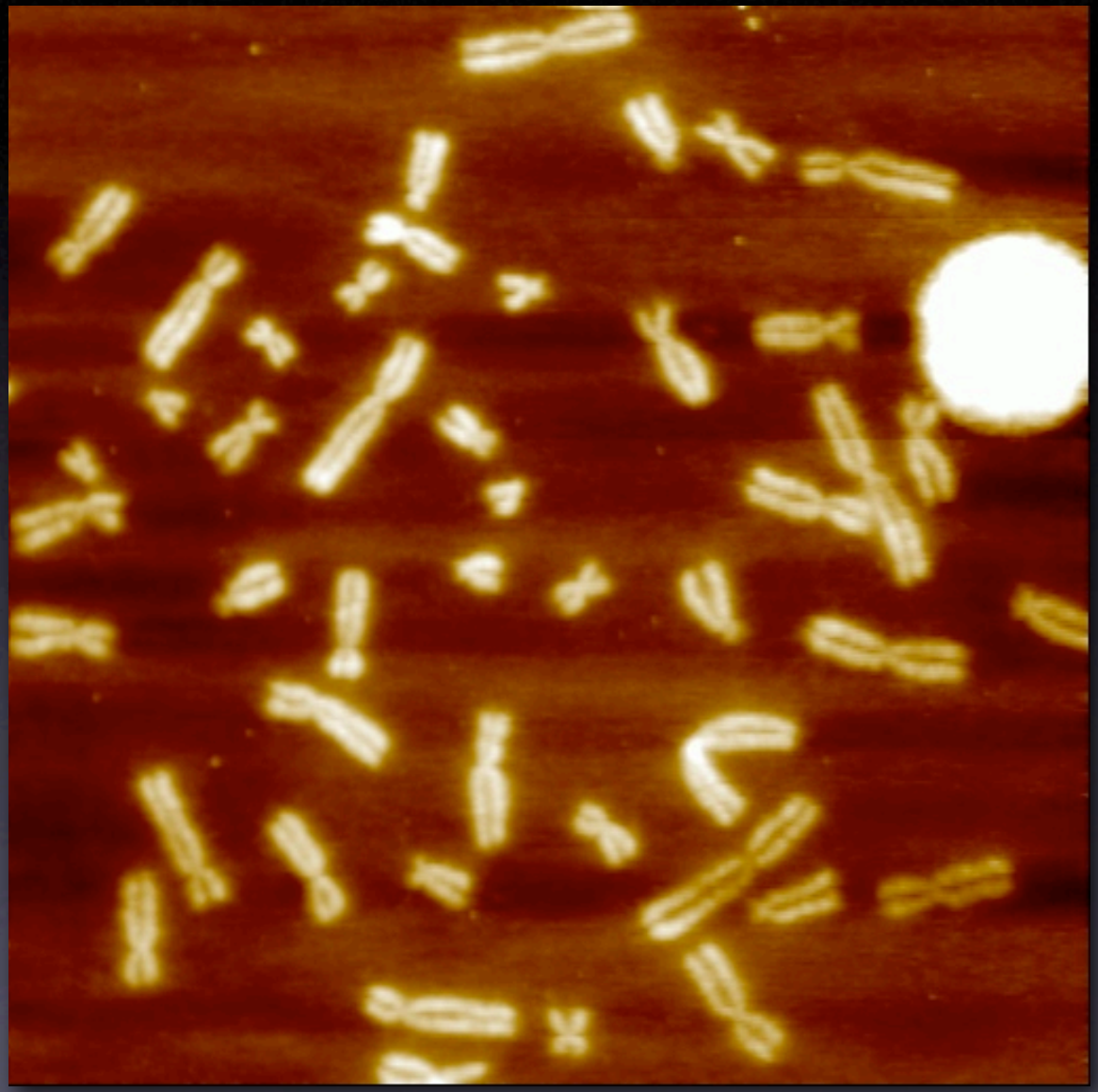
10 mins
18,000
frames

# High-speed AFM of ‘large’ objects

Air

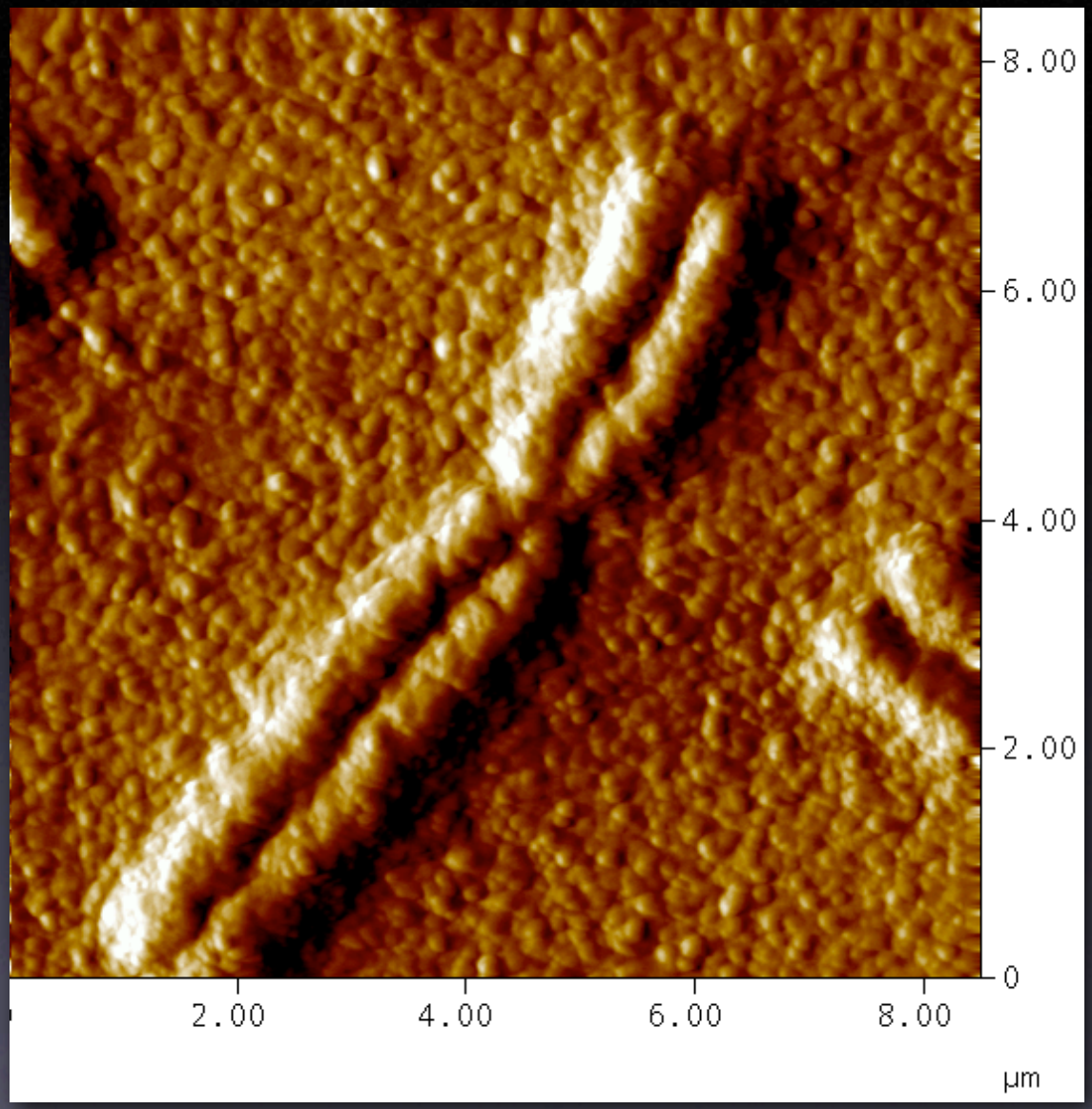
Set of human chromosomes

c AFM

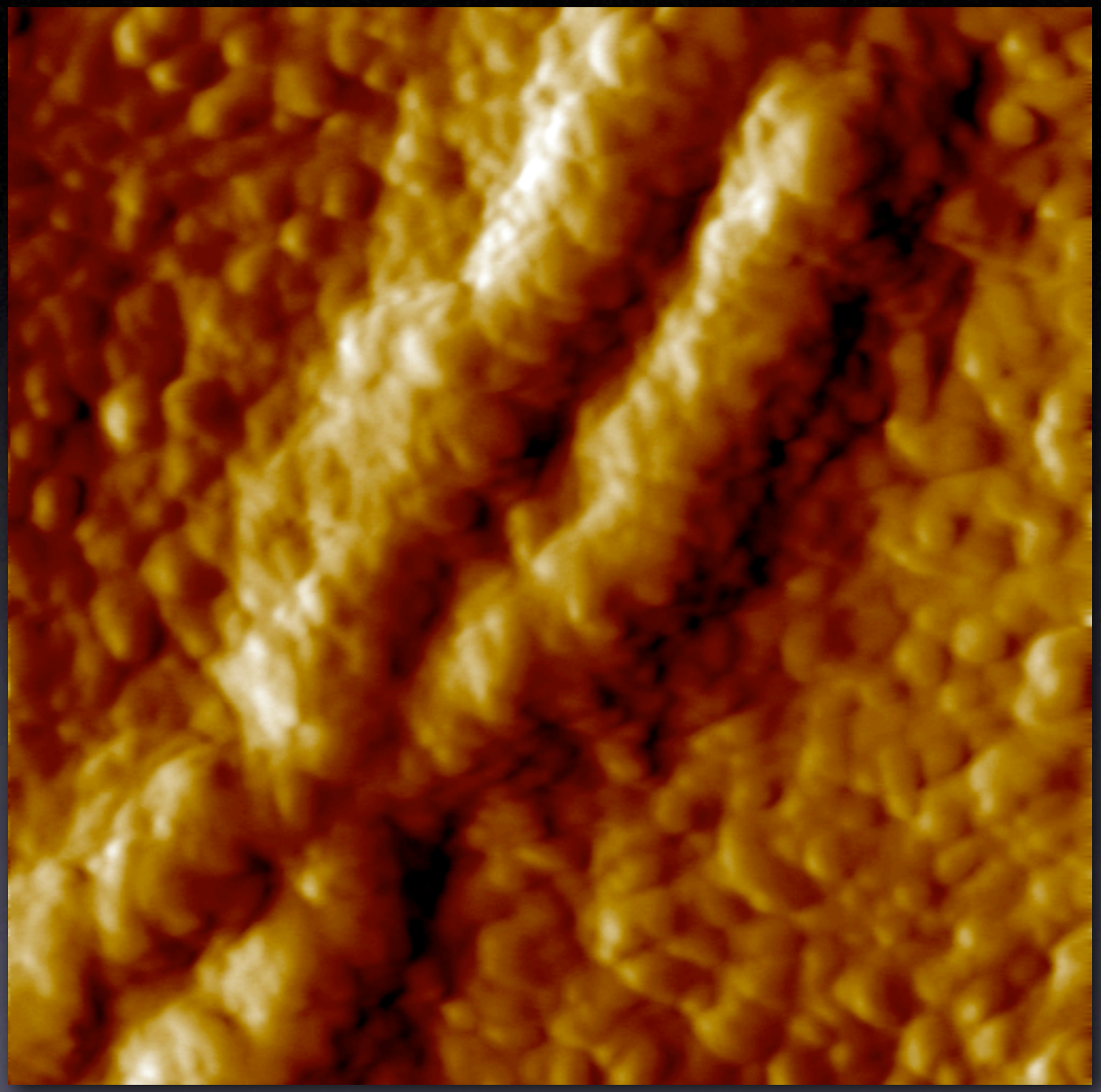


Picco, Miles, Komatsubara, Hoshi, Ushiki

# Human Chromosome No. 2



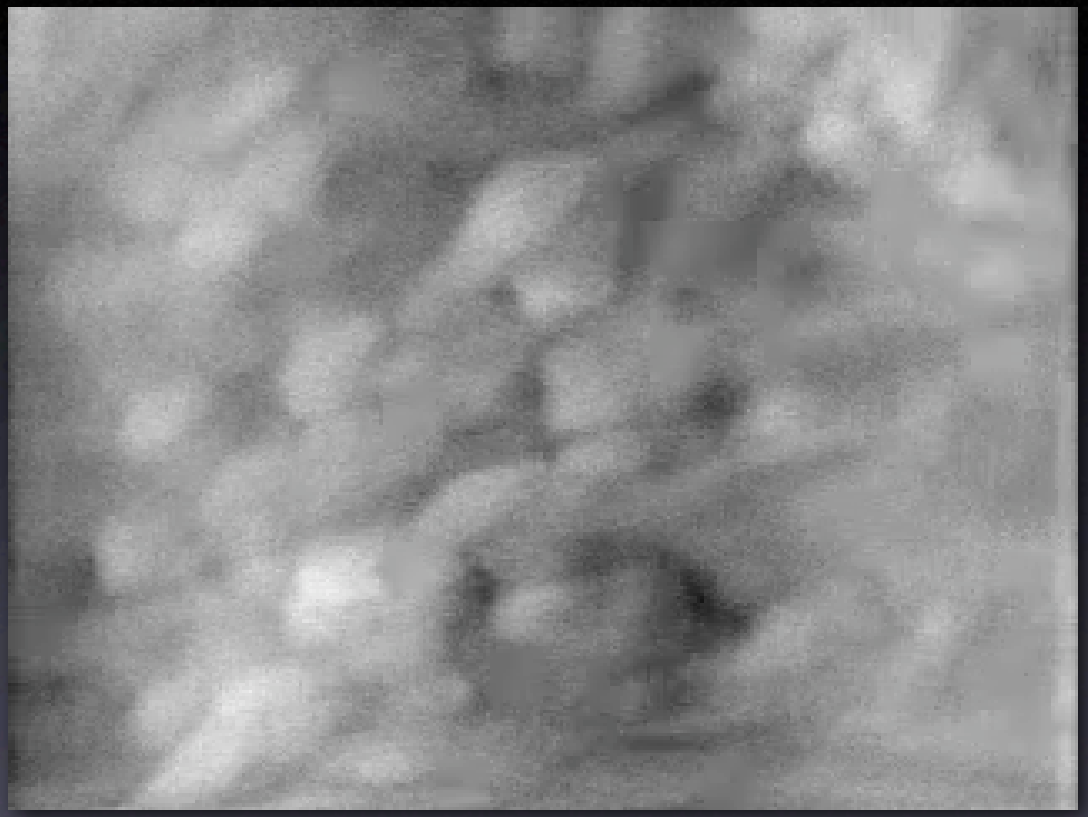

Picco, Miles, Komatsubara, Hoshi, Ushiki



Picco, Miles, Komatsubara, Hoshi, Ushiki

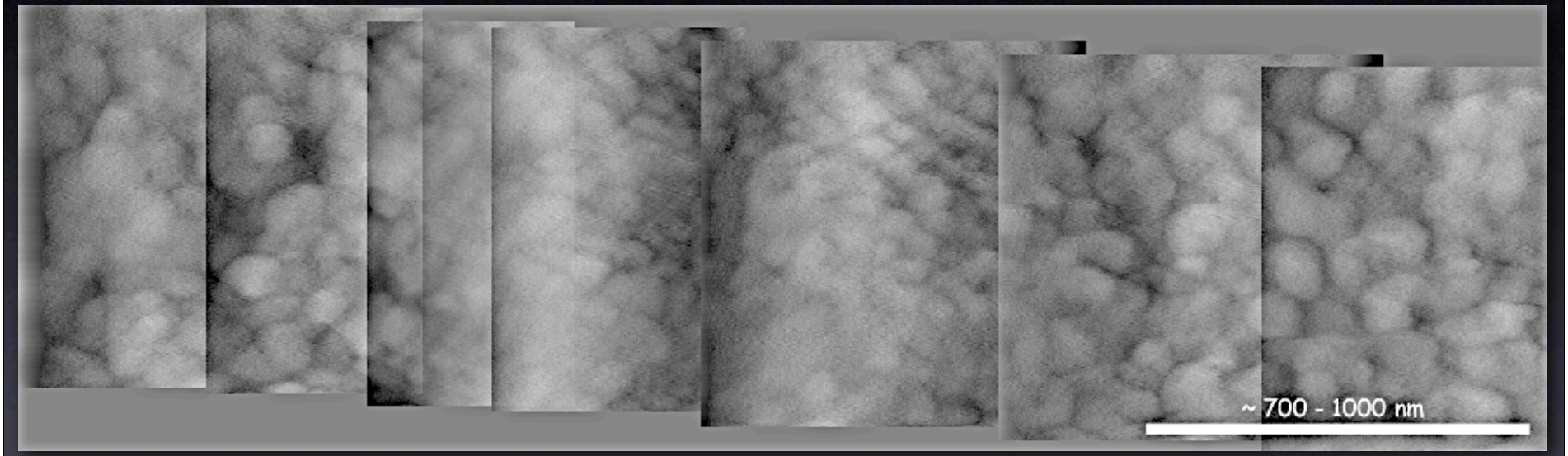
# High-speed AFM of parts of human chromosomes

Air

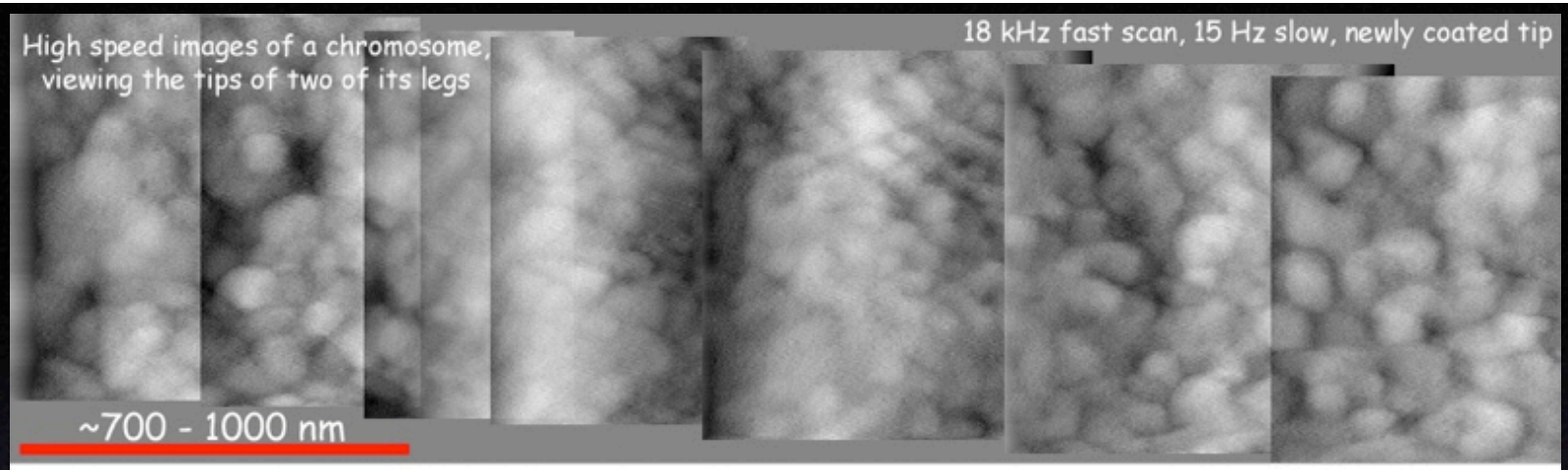


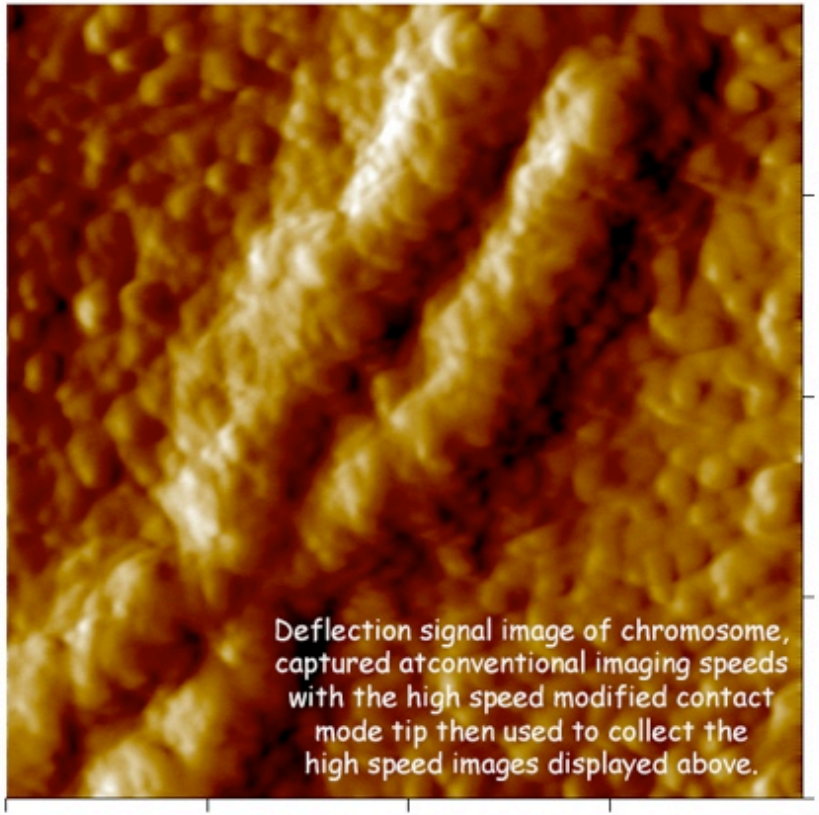
Picco, Miles, Komatsubara, Hoshi, Ushiki

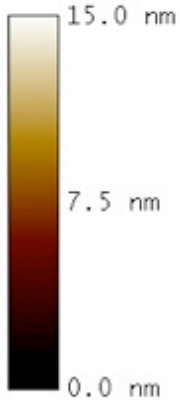
# Montage of high-speed AFM images of part of human chromosome No. 2




Picco, Miles, Komatsubara, Hoshi, Ushiki



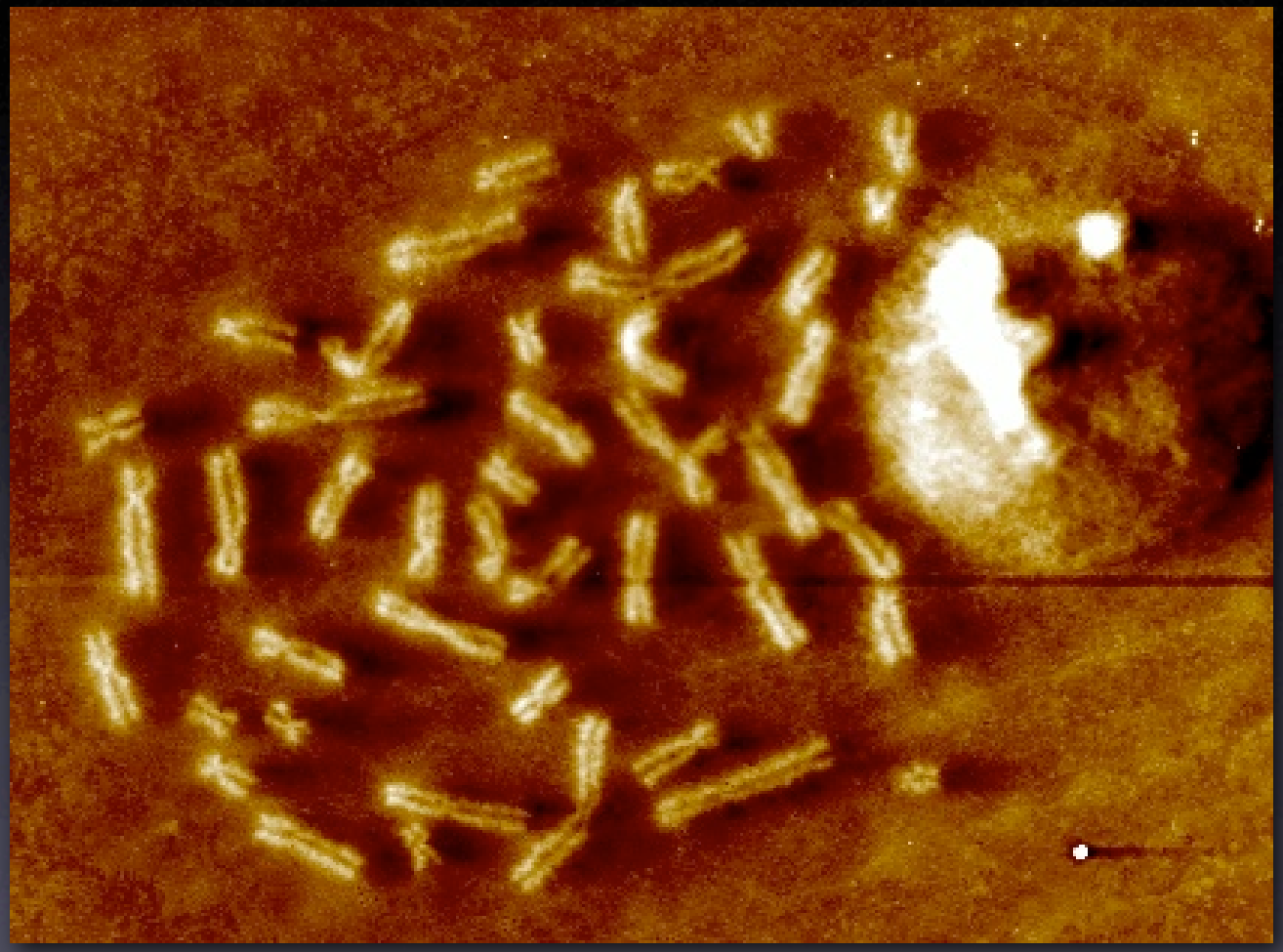




Note the similarities with the above high speed image - the small features on the surface of the chromosome are accurately imaged in both modes and are distinctive against the larger surface features.

| | |
|---|---|
| Digital Instruments NanoScope | |
| Scan size | 3.959 µm |
| Scan rate | 0.4994 Hz |
| Number of samples | 512 |
| Image Data | Deflection |
| Data scale | 15.00 nm |

Air



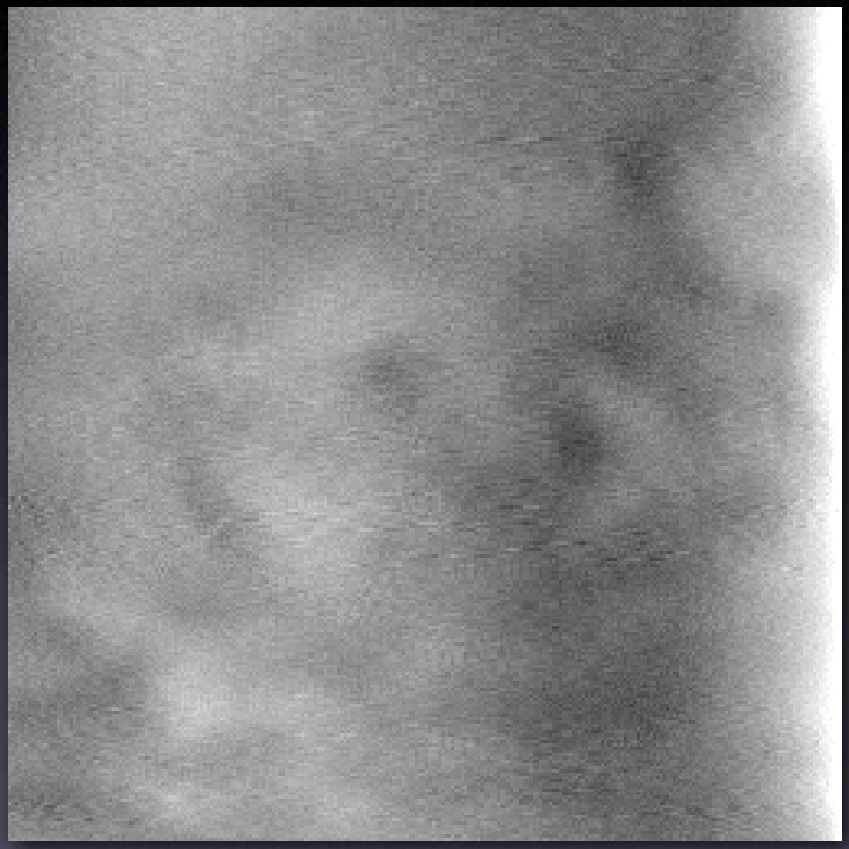
Picco, Miles, Komatsubara, Hoshi, Ushiki

2M NaCl

Picco, Miles, Komatsubara, Hoshi, Ushiki

# Human chromosomes in liquid

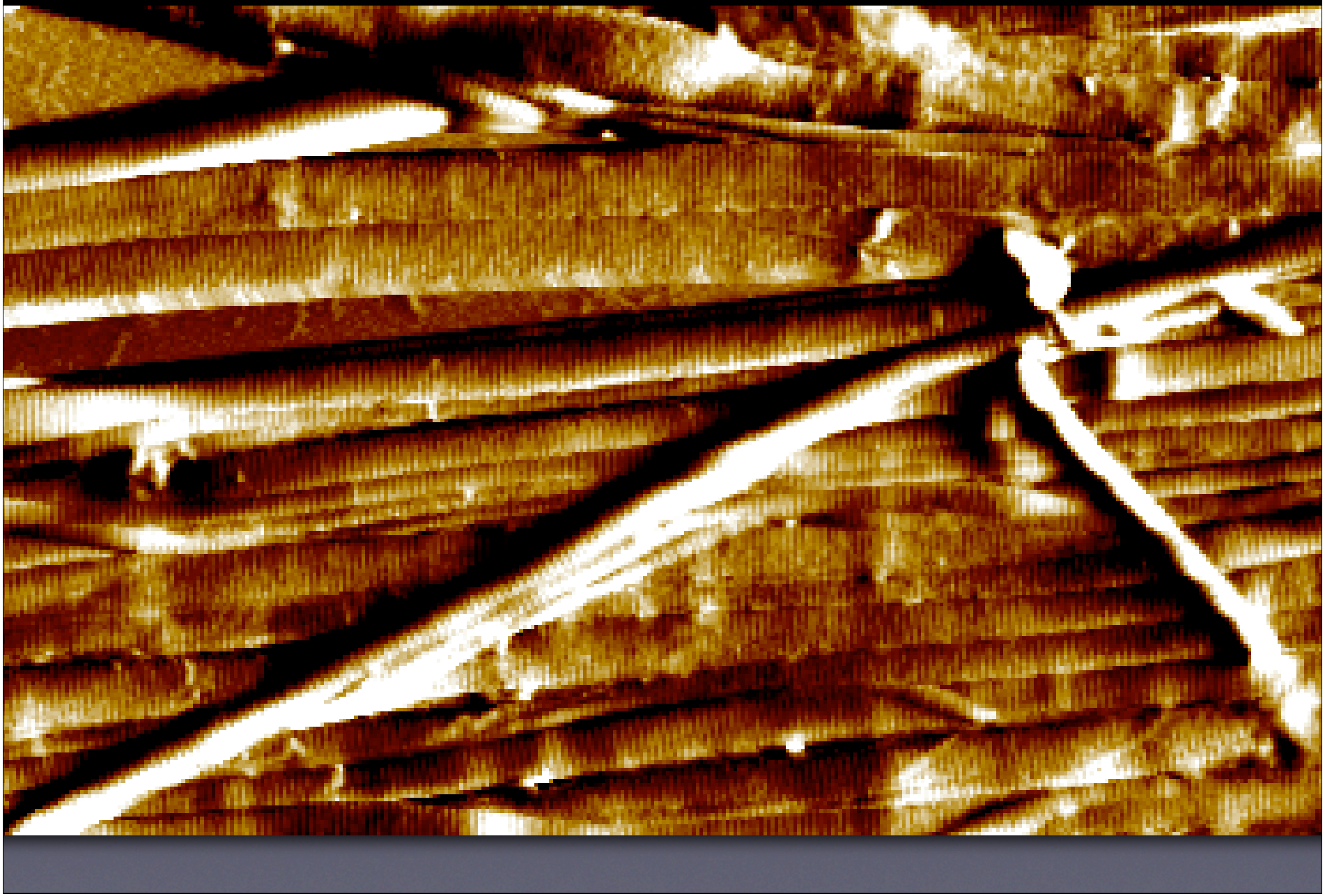
2M NaCl



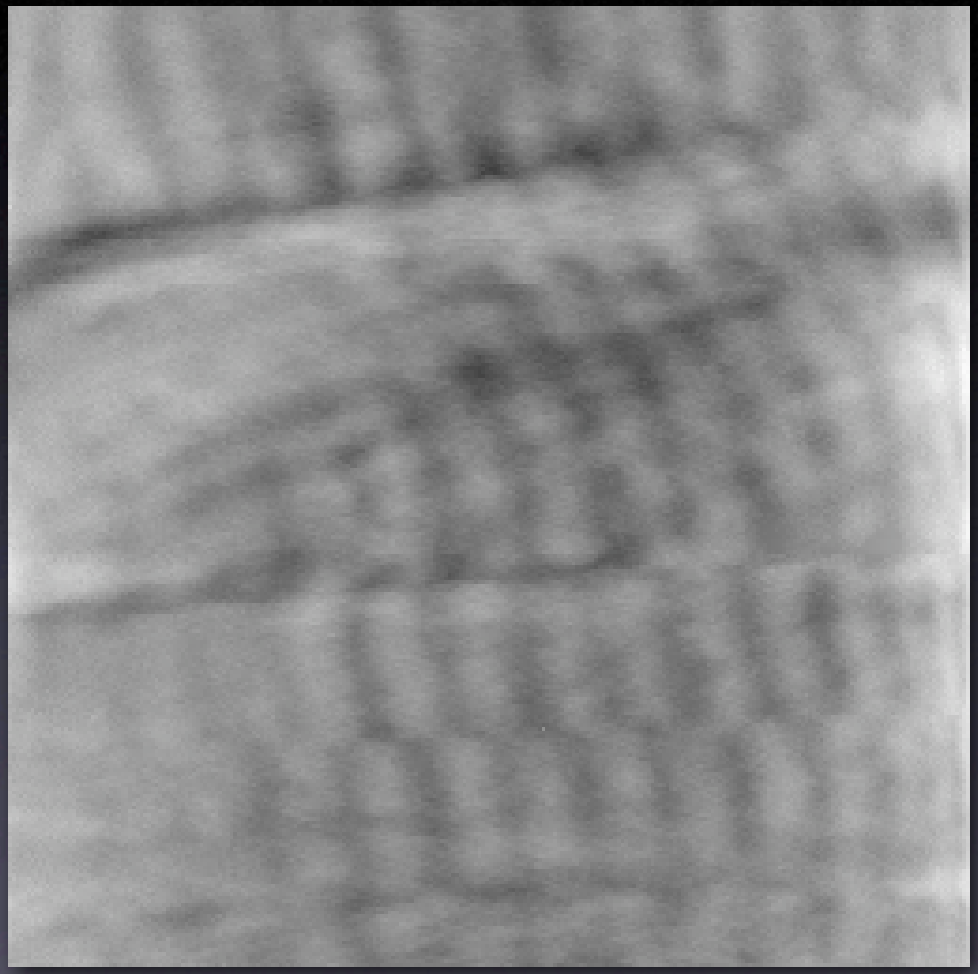
Height range: ~0.5 μm

Picco, Miles, Komatsubara, Hoshi, Ushiki

# Conventional AFM Image of Collagen Specimen



# Collagen Fibres



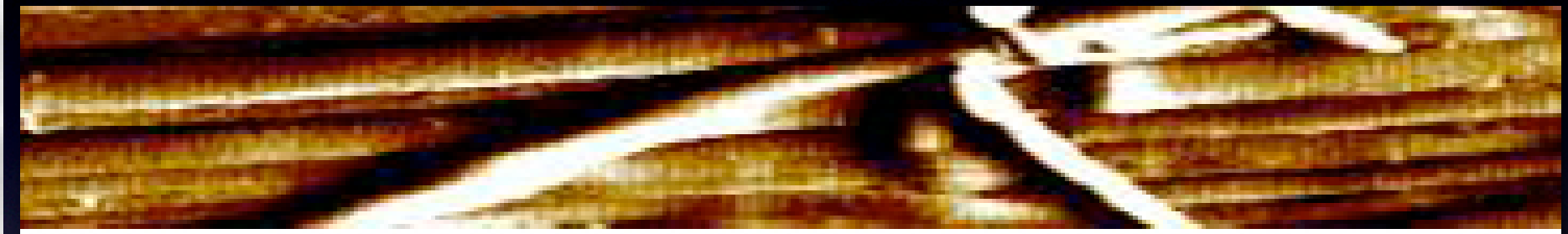
Banding
periodicity: 67 nm

30 fps

Picco, Bozec, Horton Engledew

# Linear Collage of High-speed AFM Images of Collagen Fibre

Conventional AFM



High-speed AFM

All the high-speed images were taken in 0.7 s

# Single frame from collagen movie

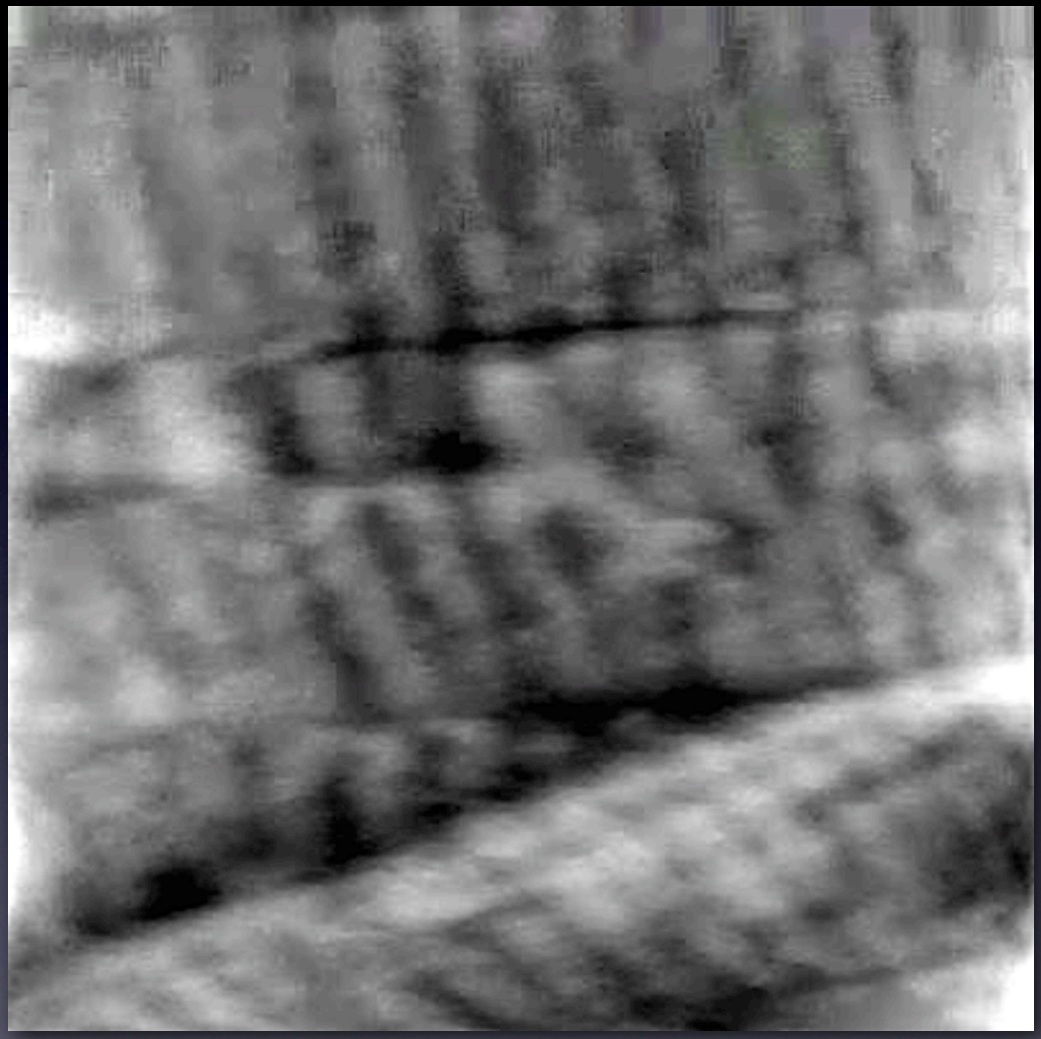
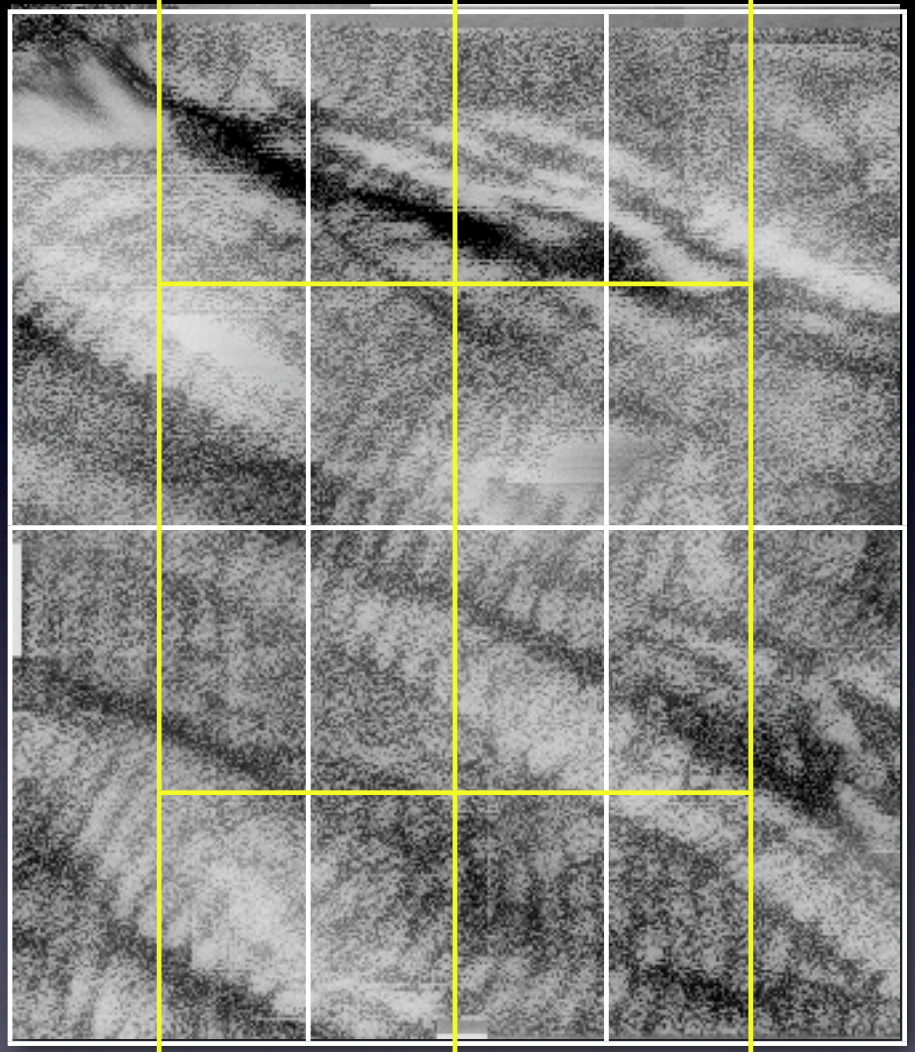


Image acquired in 17 ms

Picco, Bozec, Horton Engledew

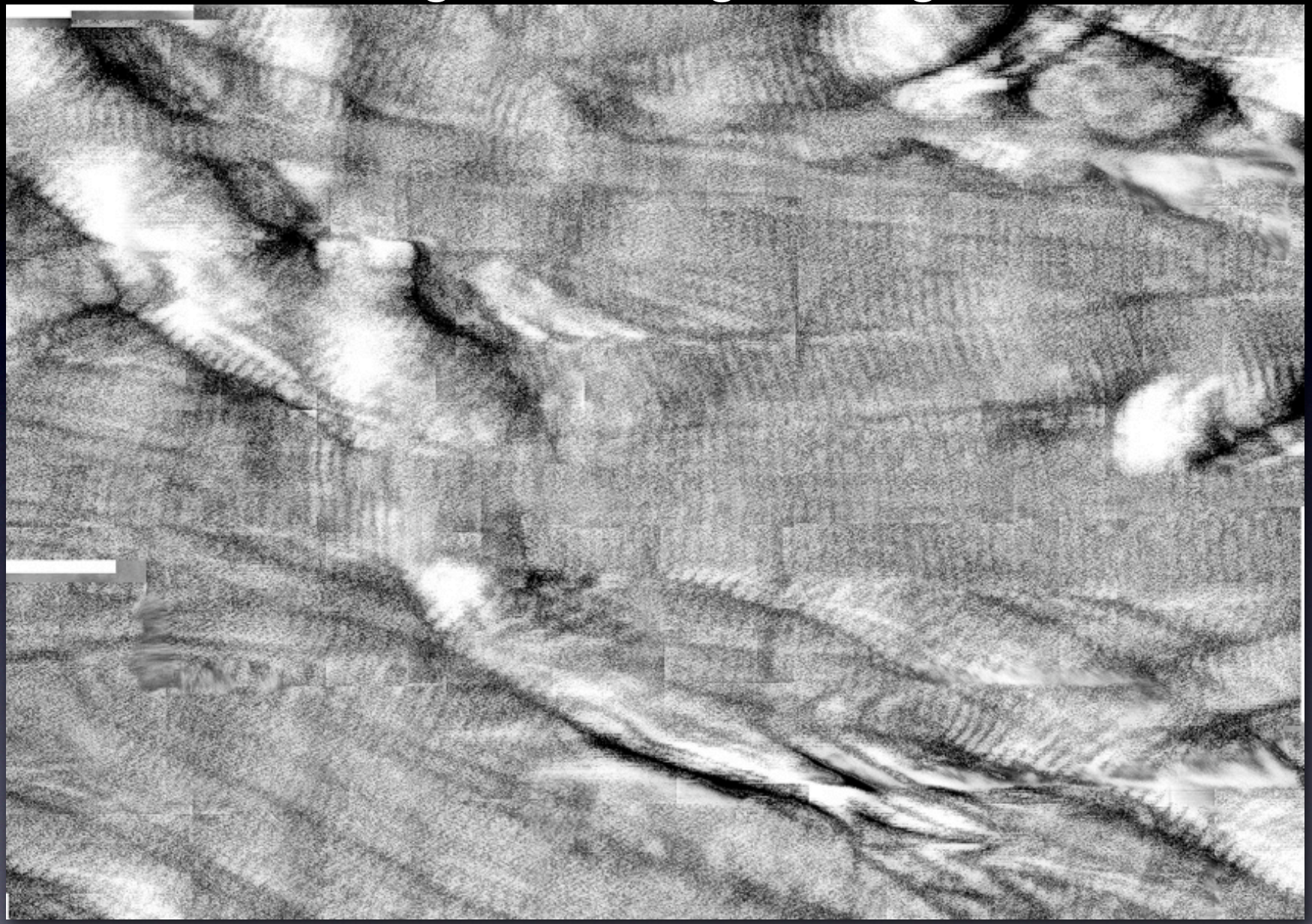
# High-speed AFM Collagen Collage



12 images involved - acquired in 200 ms

Picco, Bozec, Horton Engledew, Miles

# Large area collagen collage



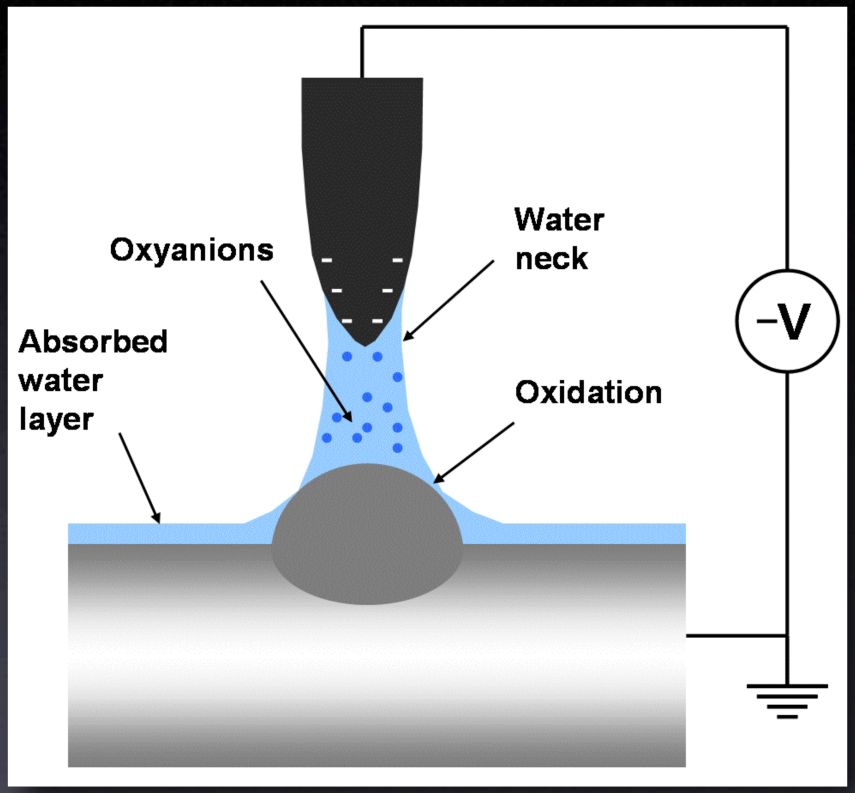
103 images acquired in 1.7 s

Non-imaging:

High-speed writing

Electrochemical oxidation of passived silicon

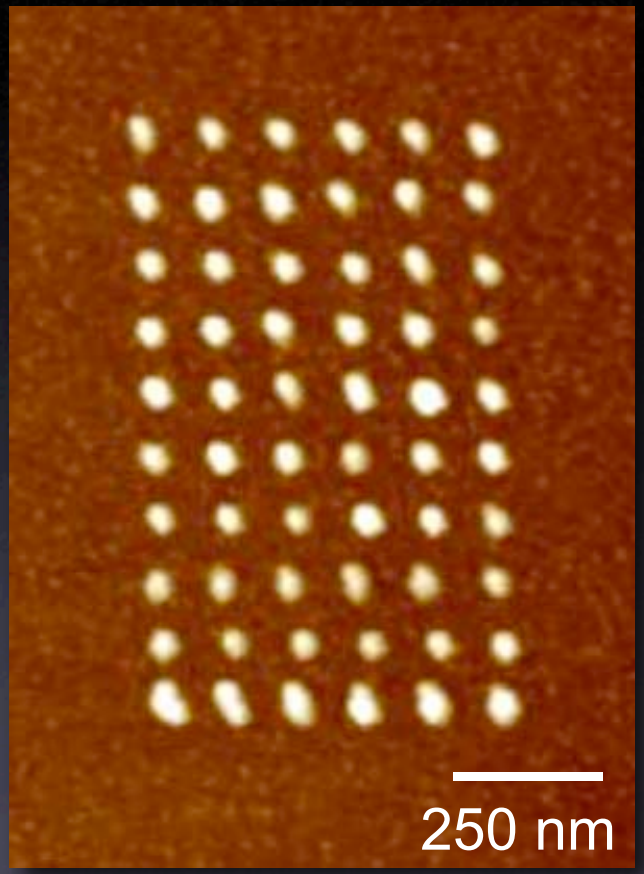
# Local AFM electrochemical oxidation

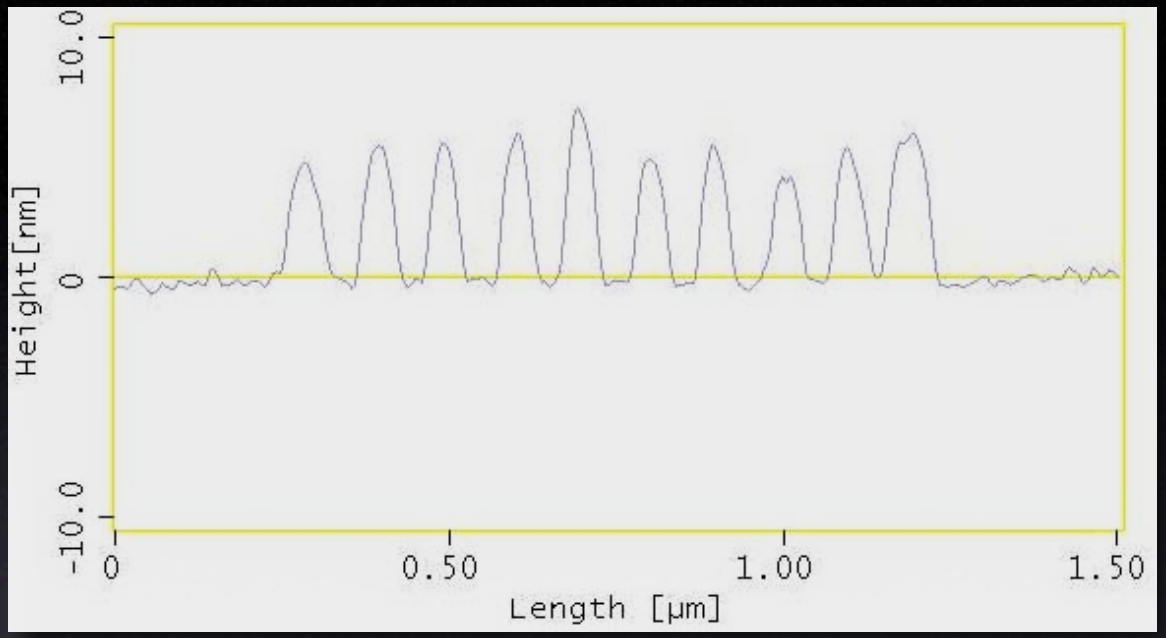



Can be used to pattern surface -
conventional AFM too slow to pattern large areas

# Ti Oxidation

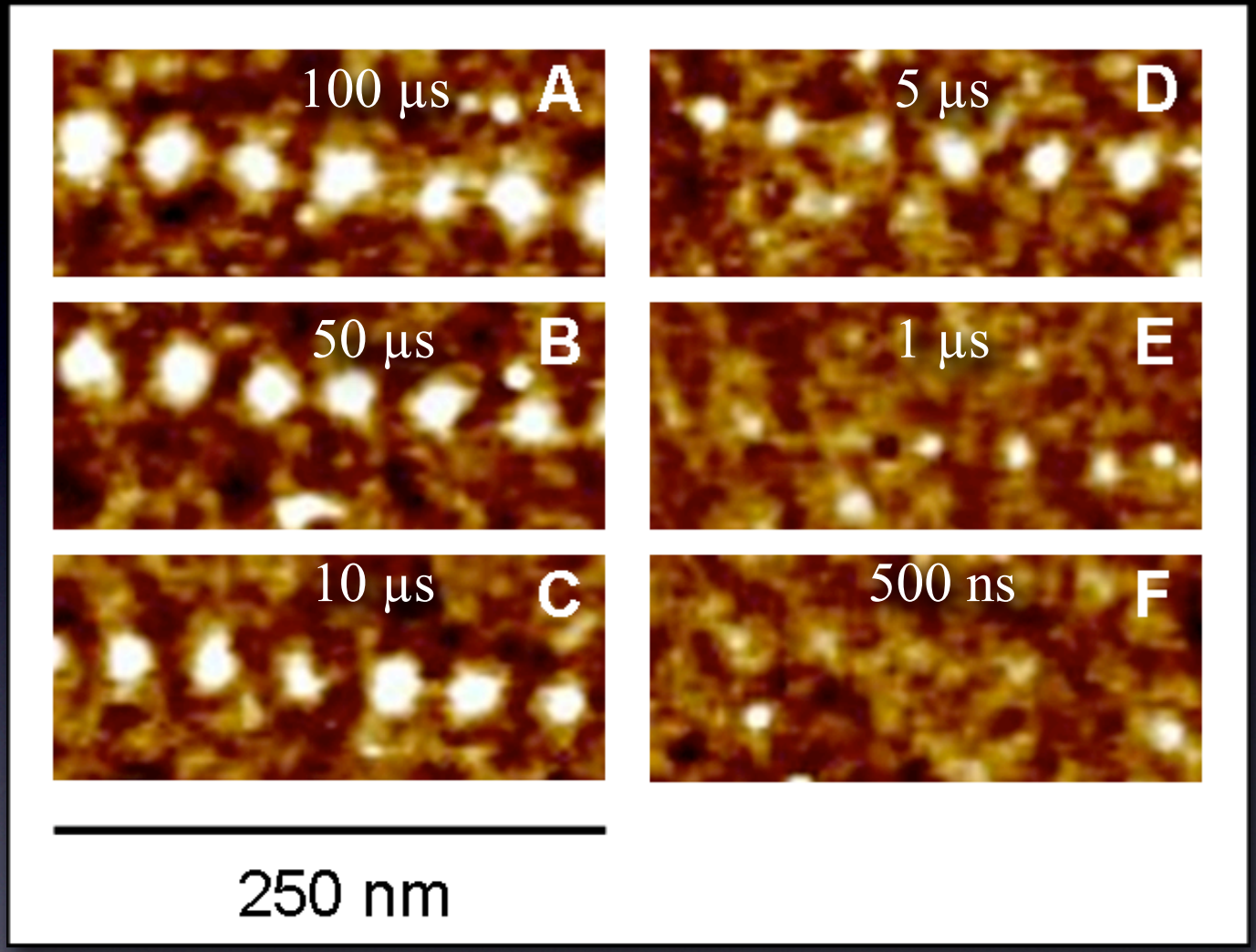
James Vicary




10 ms; -12 V Si tip;
conventional AFM
non-contact

# Oxidation of Silicon with high-speed AFM




Topographic AFM images of oxide nanostructures created with a -12 V tip bias for pulse times. Height range: 2 nm.

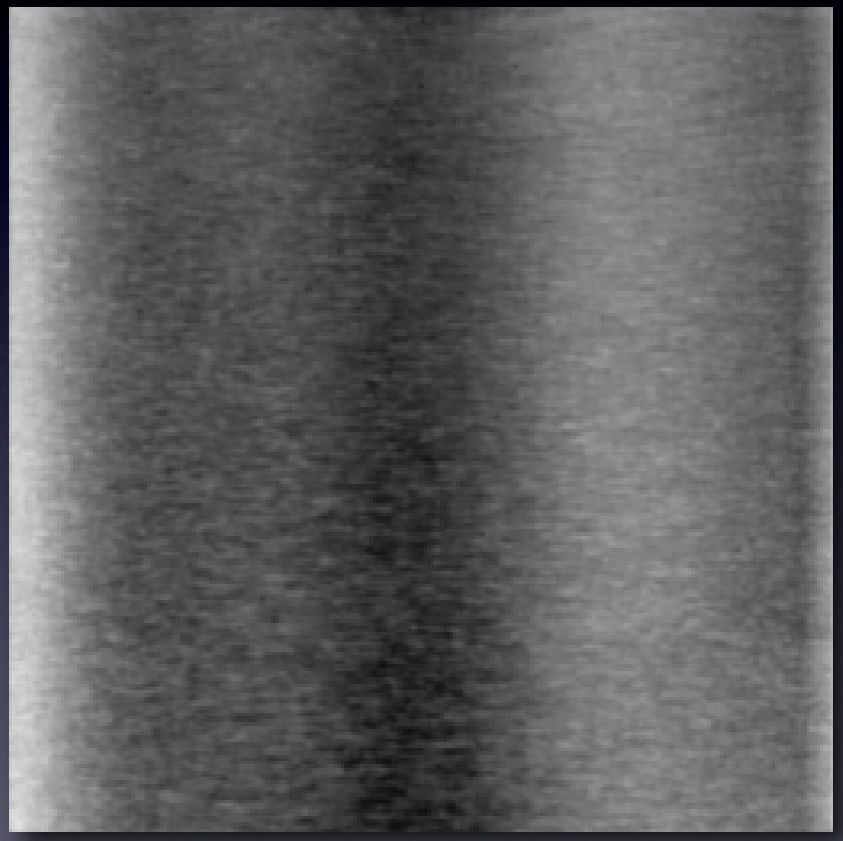
Picco, Vicary

# High-speed AFM

Simultaneous writing (oxidation) and imaging

30 fps

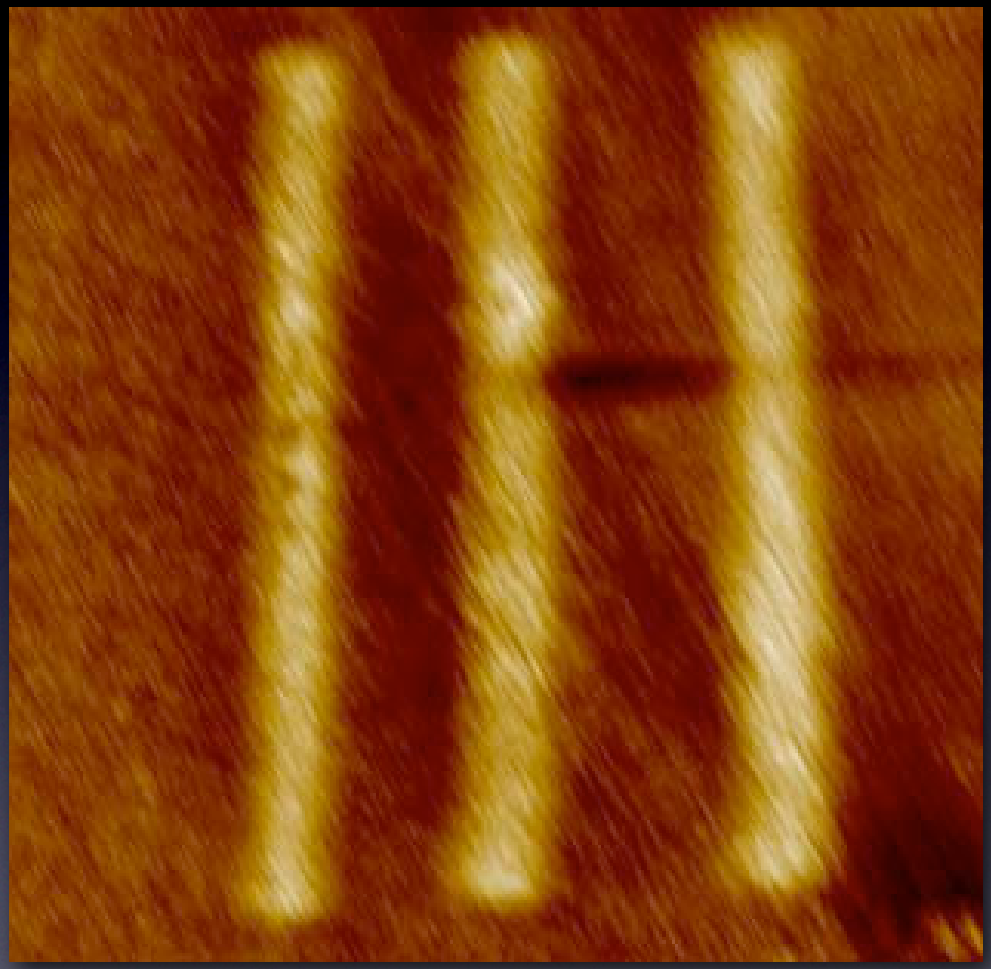
Read-Write



3 voltage pulses synchronized to fast line scan

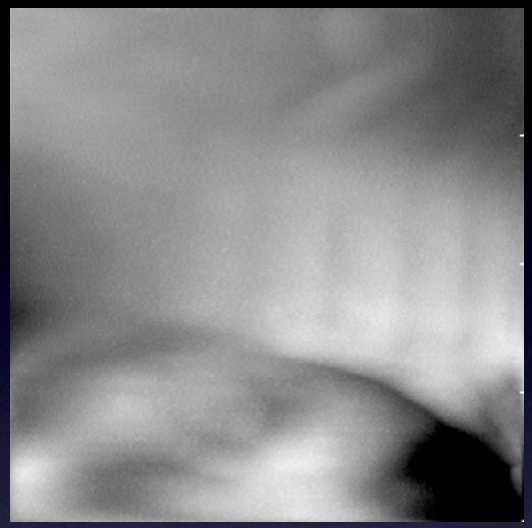
Picco, Vicary

# Conventional AFM of oxidized lines on Si



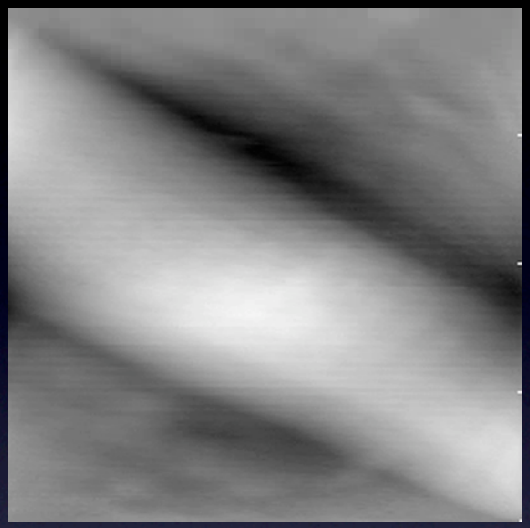
# High-speed AFM of Collagen

Bimorph



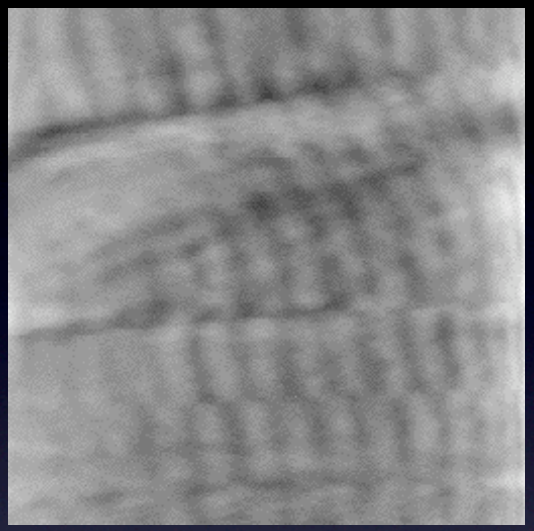
9 fps

Tuning fork



15 fps

Flexure



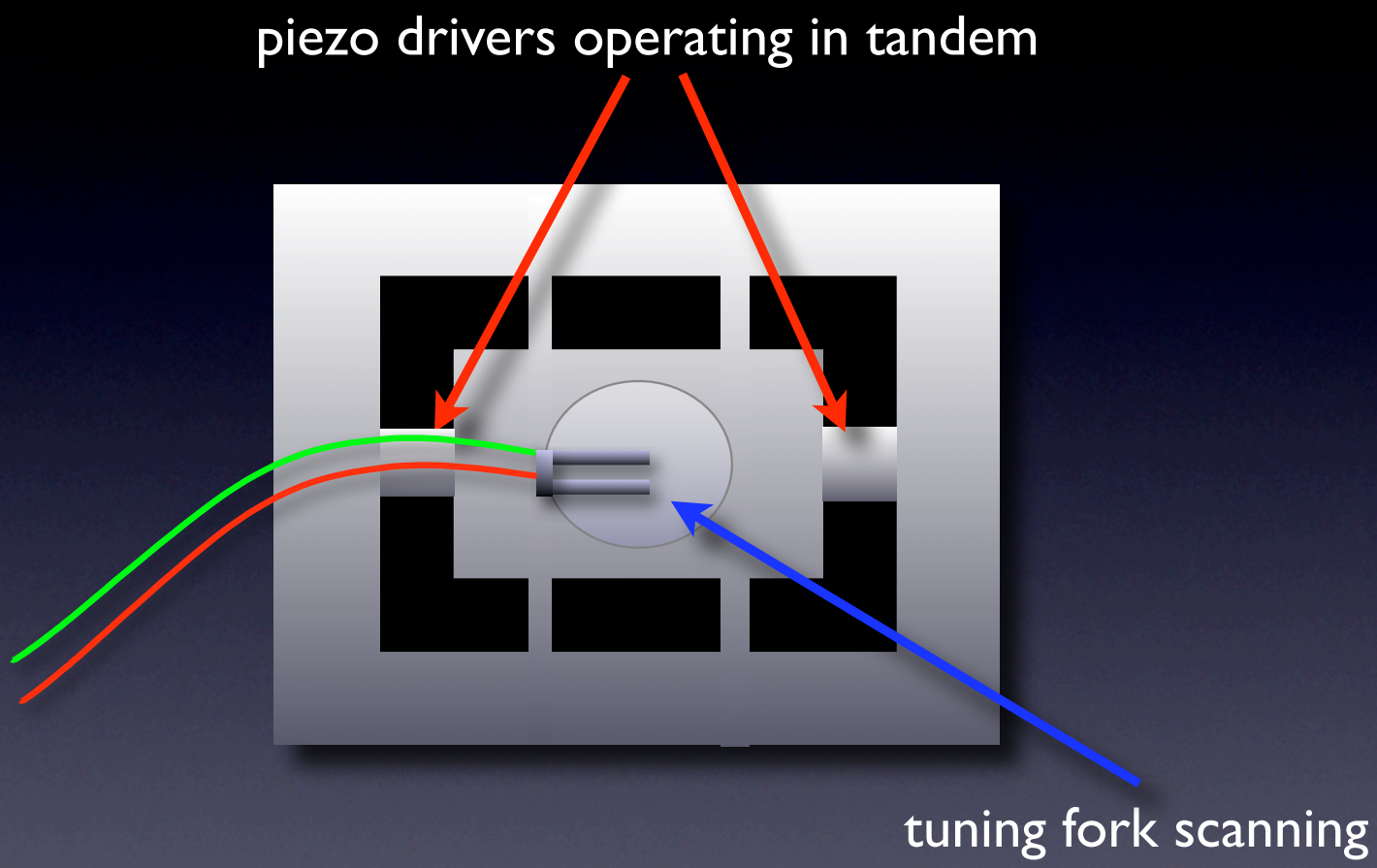
30 fps

How fast can this system image?

>1000 fps

AFM images in less than 1 ms

# Scanner Arrangement for kilohertz AFM

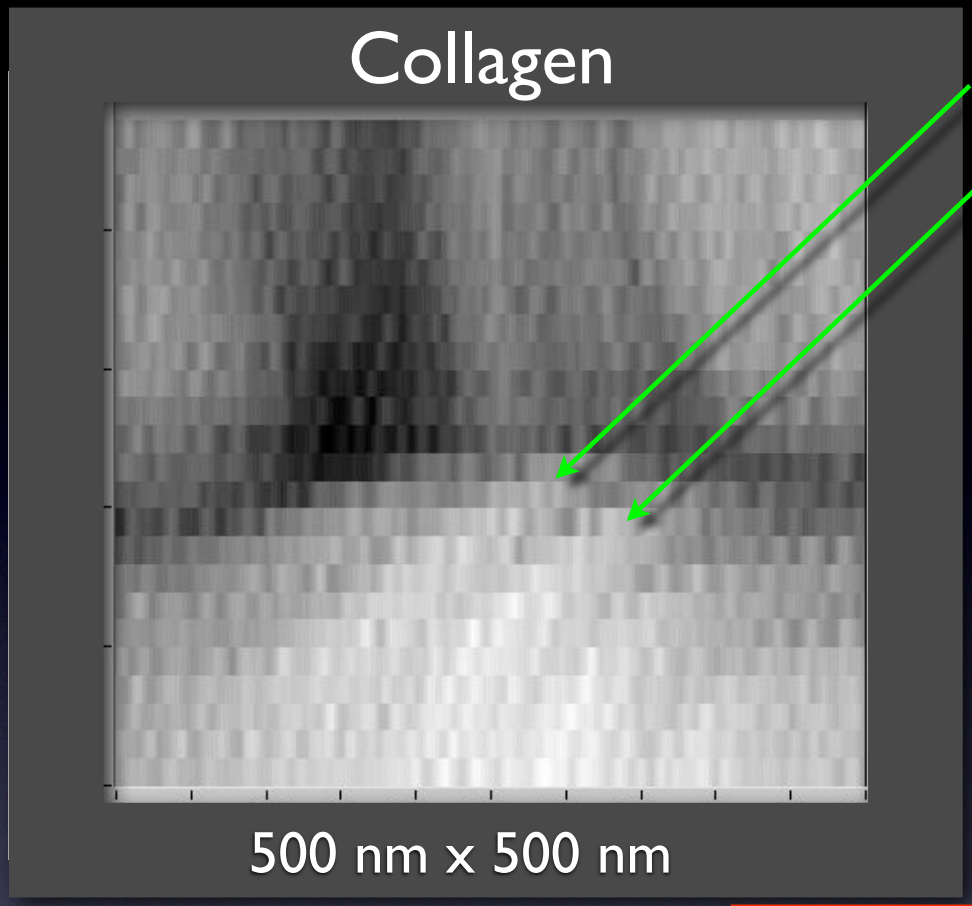


*Frame Rate:*

1300 fps

*Line Rate:*

64,000 lps




Collagen banding 67 nm

*Playback Rate:*

*25 fps*

Each frame acquired in: 750 µs

Playback slowed down by > 40 times!

1 Million Images generated in ~15 minutes!

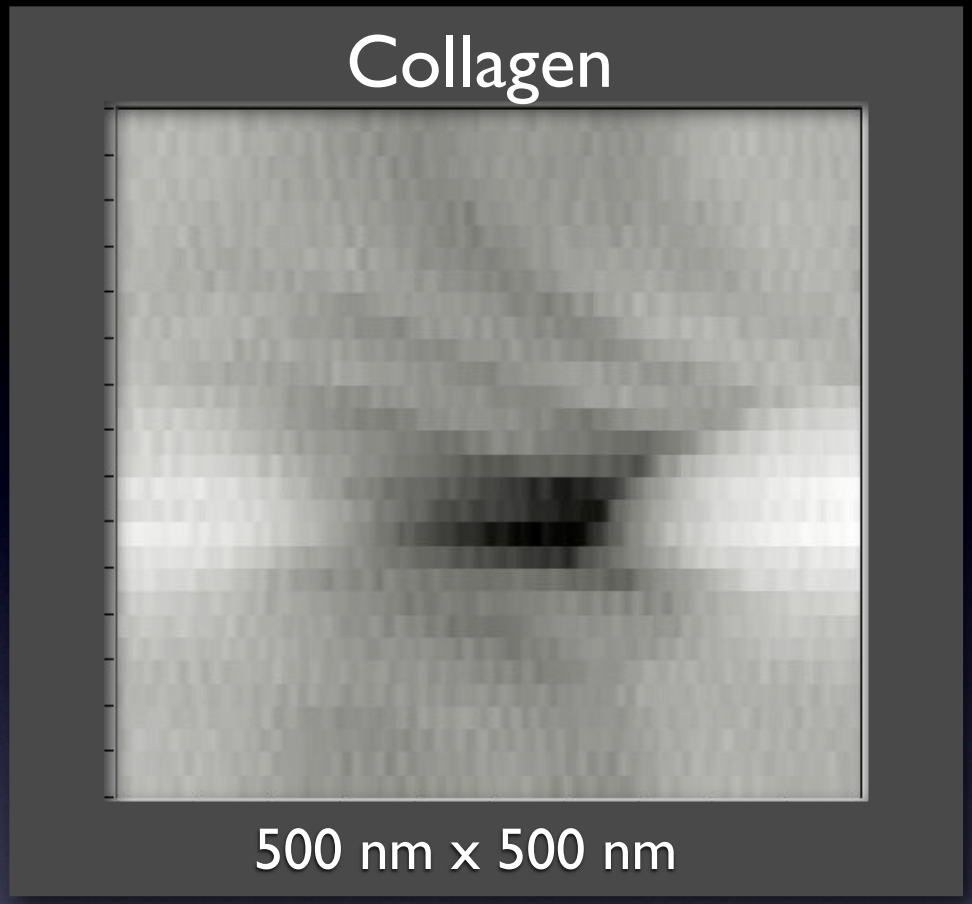
Picco, Ulcinas, Antognozzi, Engledew, Horton, Bozec, Miles

*Frame Rate:*

1200 fps

*Line Rate:*

64,000 lps




*Playback Rate:*

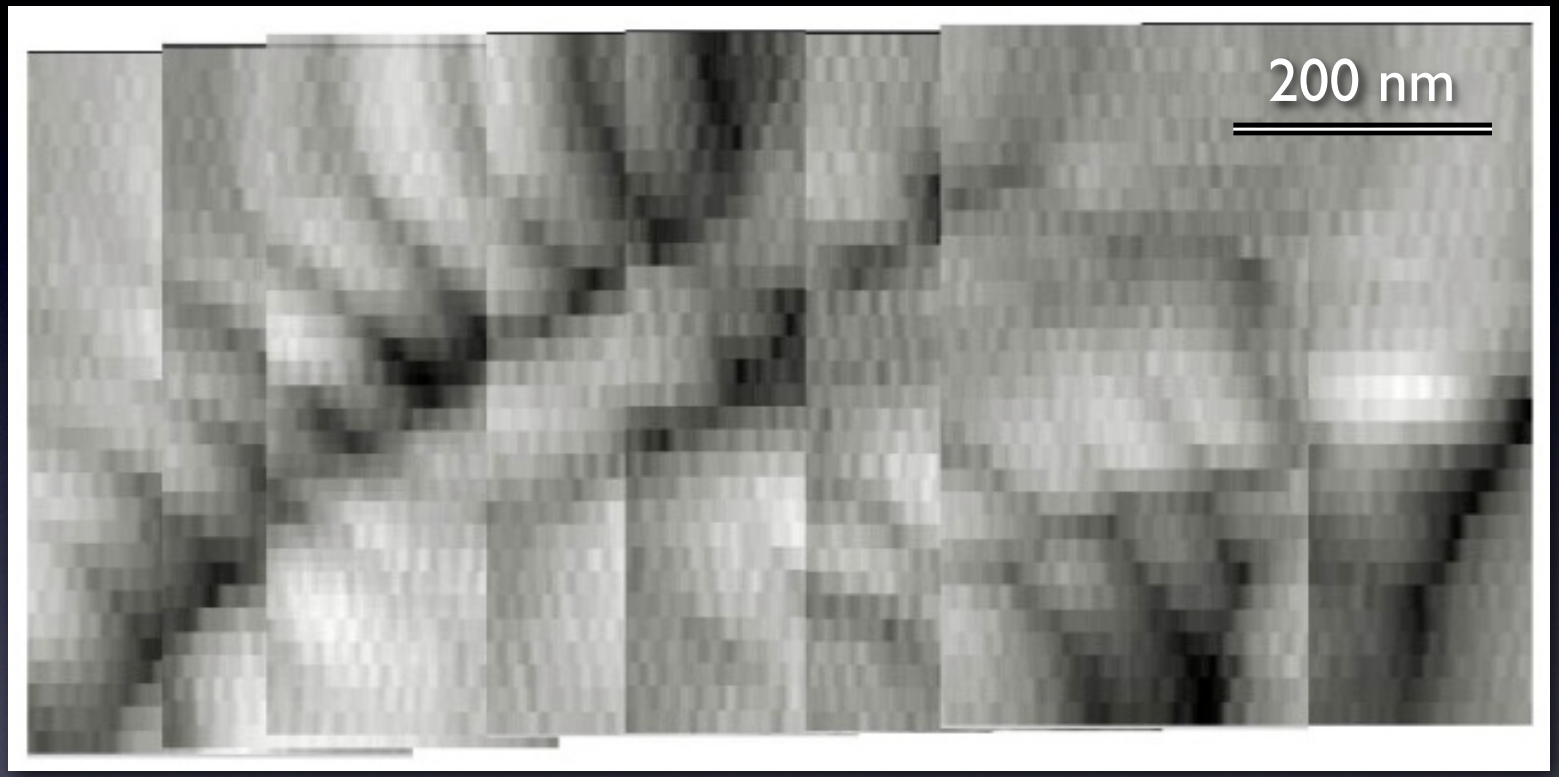
*25 fps*

Specimen being oscillate by 50 nm

Collagen banding = 67 nm

Picco, Ulcinas, Antognozzi, Engledew, Horton, Bozec, Miles

# Composite image from 1000 fps AFM of Collagen



So far, the movies were rather pixellated ....

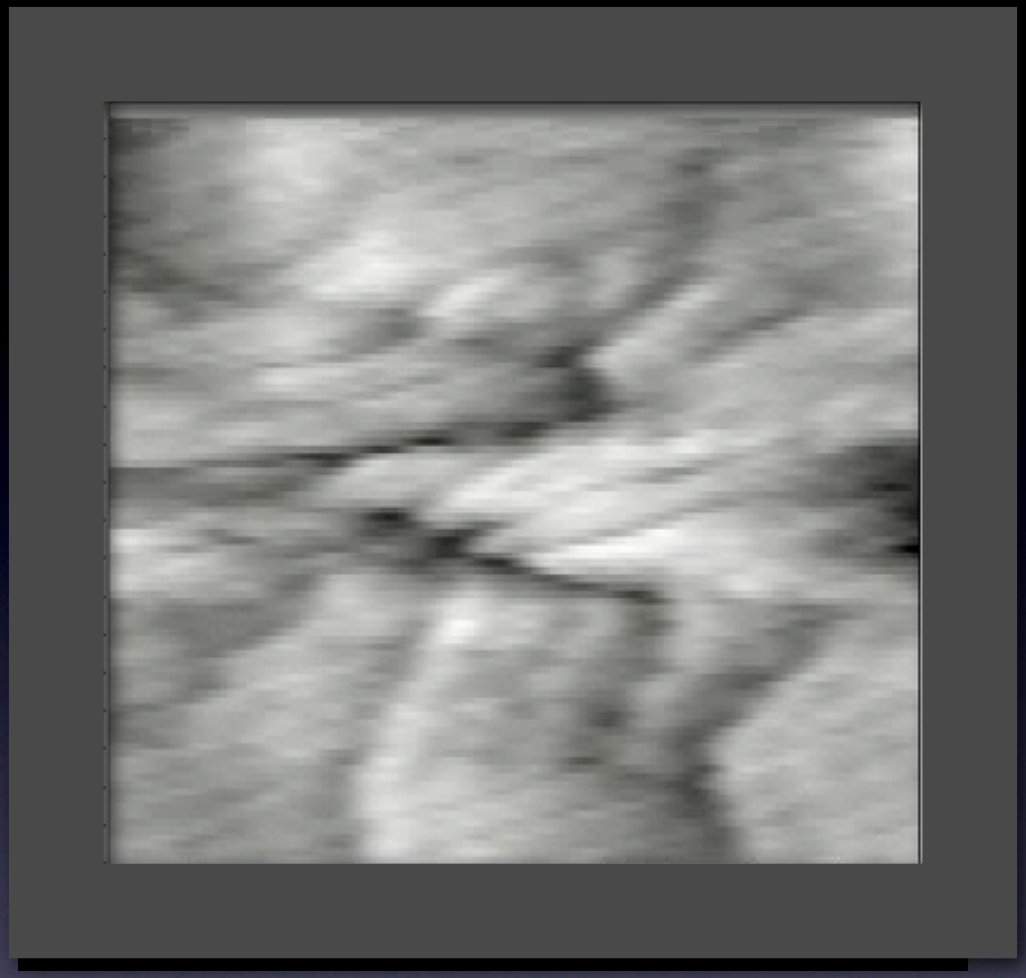
... this can be fixed by increasing the line rate further ....

*Frame Rate:*

1000 fps

*Line Rate:*

200,000 lps



*Playback Rate:*

*25 fps*

200 x 100 pixels; ~ 25 ns/pixel (on average)

Picco, Ulcinas, Antognozzi, Engledew, Horton, Bozec, Miles

Nanoscience and QI Centre, Bristol
Infinitesima
Thanks to...
Peter Dunton
Massimo Antognozzi
Monica Berry
Debra Brayshaw
David Engledew
Simon Haward
Jon Hayes
(Jamie Hobbs)
(Andy Humphris*)
Terry McMaster
Niigata University:
Tatsuo Ushiki
Osamu Hoshi
Nae Komatsubara
UCL, London :
Michael Horton
Laurent Bozec
(Andras Major)
Sheila Morris
Loren Picco
Andy Round
Mark Szczelkun
Arturas Ulcinas
James Vicary
Craig Williams
Alex Wotherspoon
Thank you for your attention
*www.infinitesima.com