High Speed AFM – Contact & Non-contact

Mervyn Miles

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

nanoSpain Conf
NANOSPAIN 2008
Braga (Portugal) April 14-18, 2008
Nanolberian Conference
Portugal
Portuguese Nanotechnology Network
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High Speed AFM

1. Extremely fast - larger areas, high topography

2. Non-contact - single molecules

Why go fast?

Go fast ...

- to follow processes of various types;
- to examine large areas of a specimen;
- to create/manipulate structures over large areas.

Why not go fast?

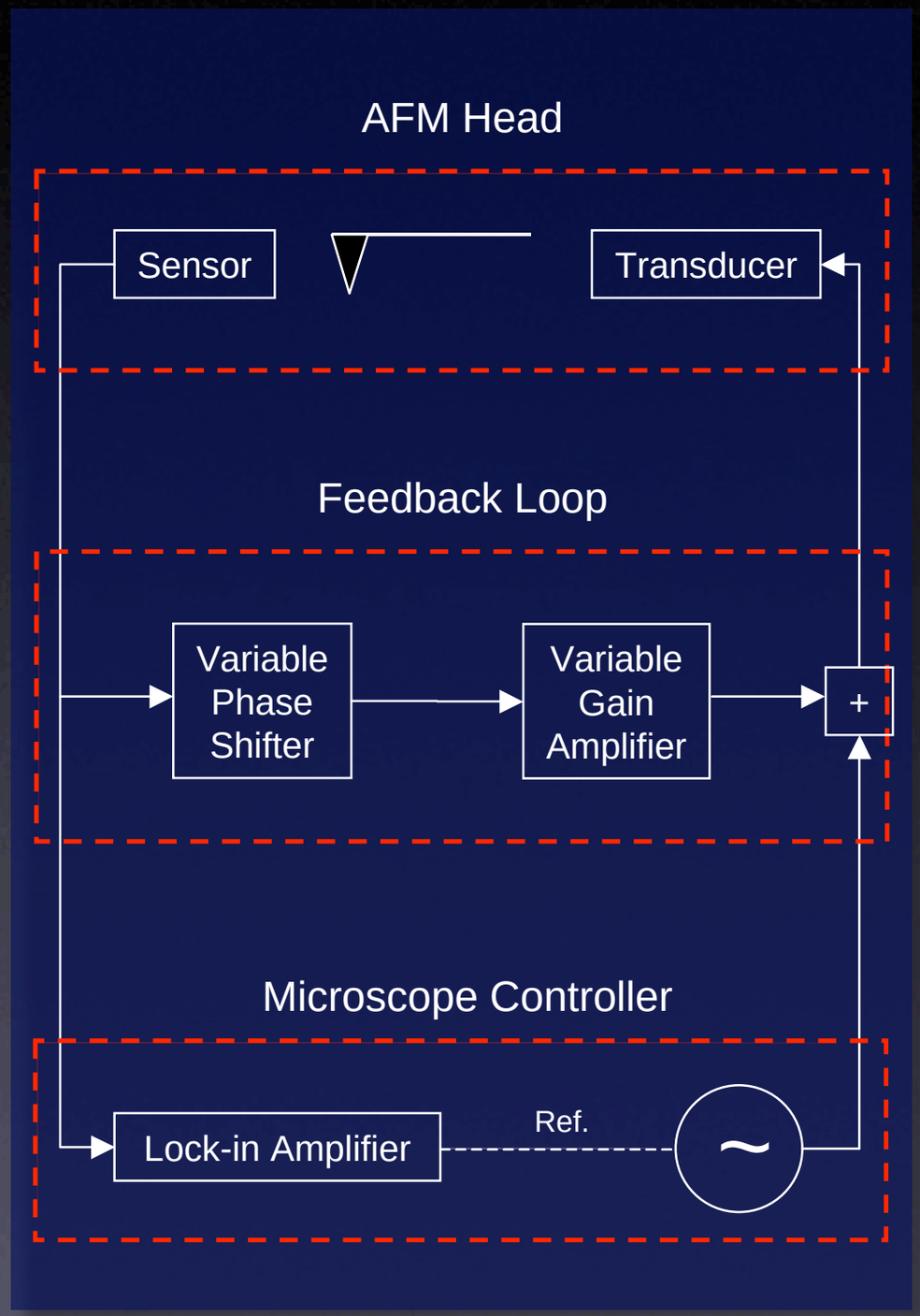
SPM is a Mechanical Microscope

- Inertia - response times
  - scan stage and z feedback
  - AFM cantilever response
- Resonance - positioning
  - scan stage
  - cantilever
  - SNOM probe
- Electronic/software feedback response

How to go faster:

- Parallel imaging with multiple cantilevers
- Strobe imaging of repetitive processes
- Decrease Q in AC modes

Q Control Implementation



$$m\ddot{z}(t) - \alpha\dot{z}(t) - kz(t) = F_0\cos(\omega t) + Ge^{i\phi}z(t)$$

$$\dot{z}(t) = \frac{dz(t)}{dt} = \omega e^{\pm i\pi/2}z(t)$$

$$m\ddot{z}(t) - \alpha_{eff}\dot{z}(t) - kz(t) = F_0\cos(\omega t)$$

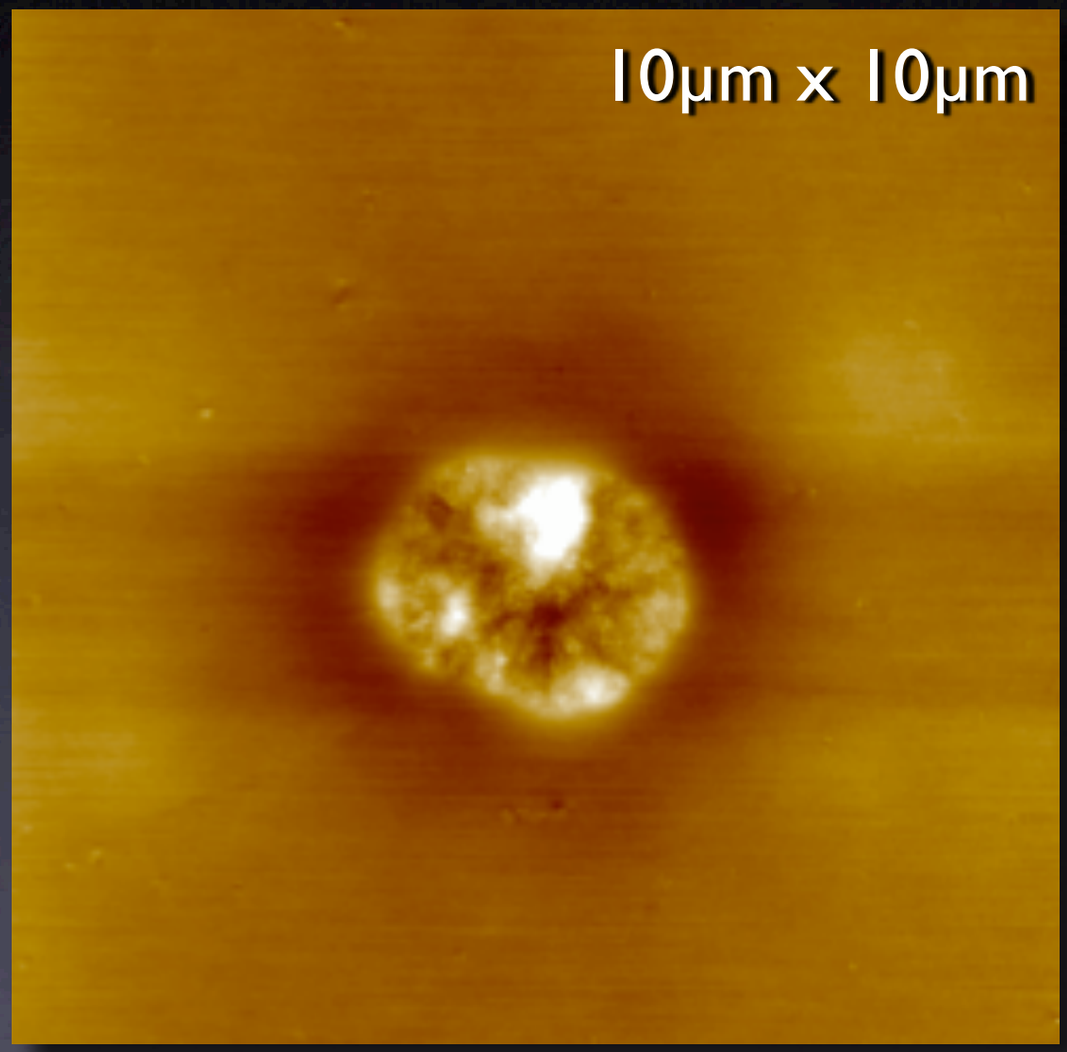
$$\text{where} \quad \alpha_{eff} = \alpha \pm \frac{G}{\omega}$$

J Tamayo, ADL Humphris, MJ Miles, *APPLIED PHYSICS LETTERS*, **77**(4), 582-584 (2000)

The process of Polymer Crystallization

AFM tapping mode - in air

PHB Spherulite Growing
from the Melt



15 minutes sequence
(Q decreased with active Q)

TJ McMaster, JK Hobbs, PJ Barham, MJ Miles

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

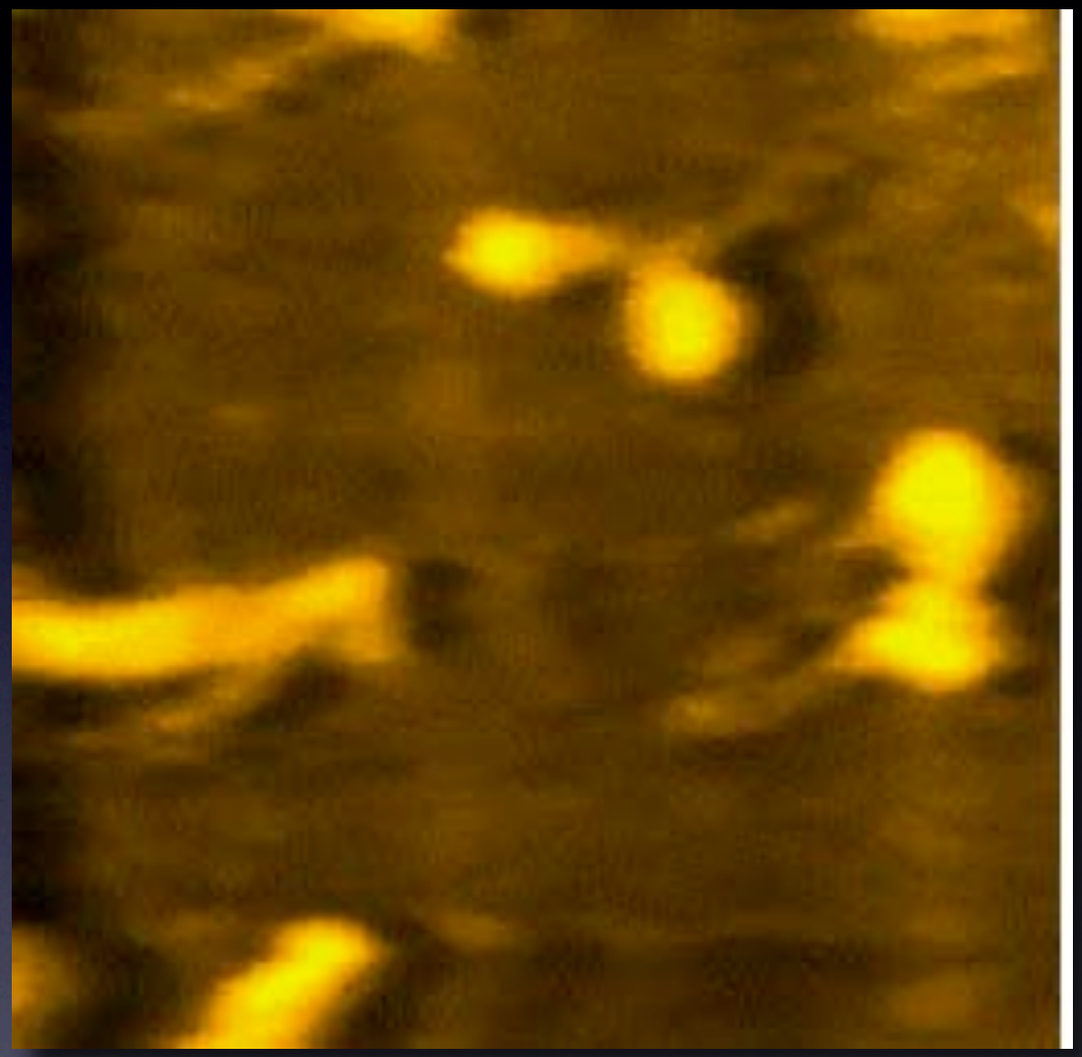
Shifting the timescale

- 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
- Faster feedback electronics & faster data capture

High-speed tapping mode AFM at higher frequencies

Myosin V

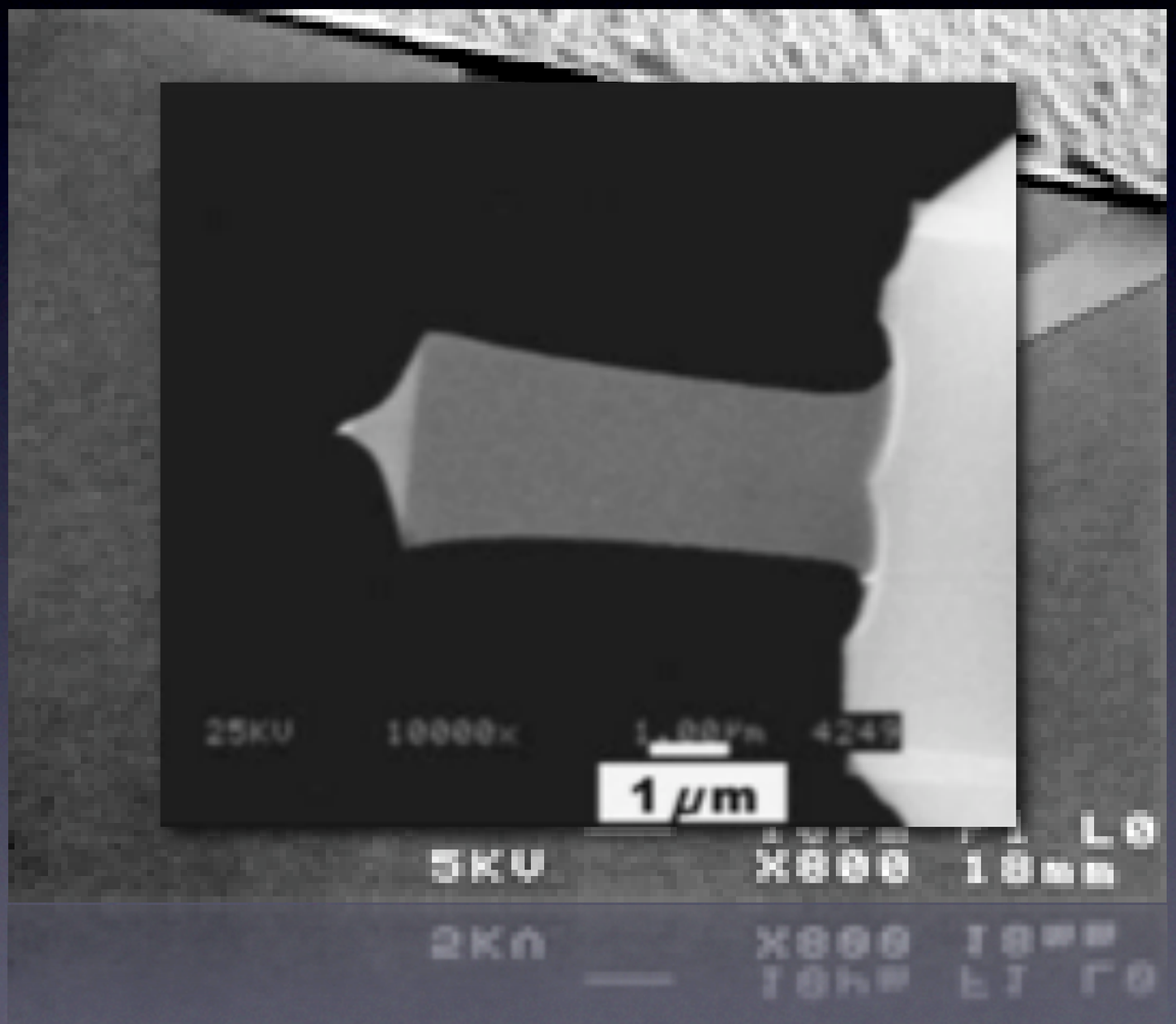


Miniature cantilever

Toshio ANDO (Kanazawa University, Japan)

Scanning faster by changing the time domain

Make higher frequency cantilevers by making them smaller



Difficult to fabricate these small cantilevers reproducibly

Difficult to avoid cantilever substrate contacting sample

A Different Solution

Resonant scan stages

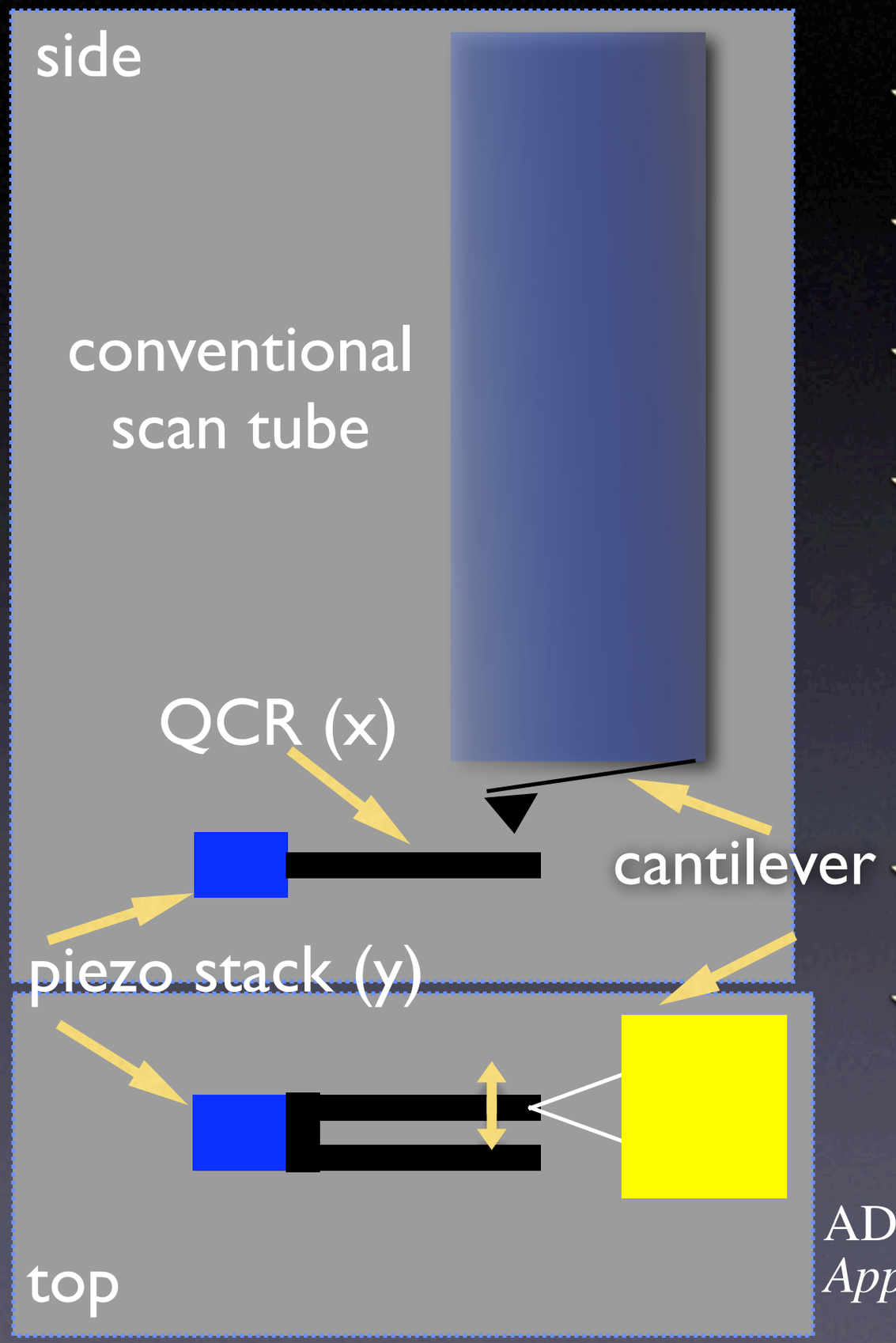
Non-contact SNOM

Superlubrication contact mode

Resonant Scan Stages

- A high Q-factor resonance can provide:
  - high amplitude (scan range)
  - high scan stability
  - small form factor
  - simplicity of construction

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
- ★ Scanned tip mode also possible
- ★ Extra down force (electrostatic)

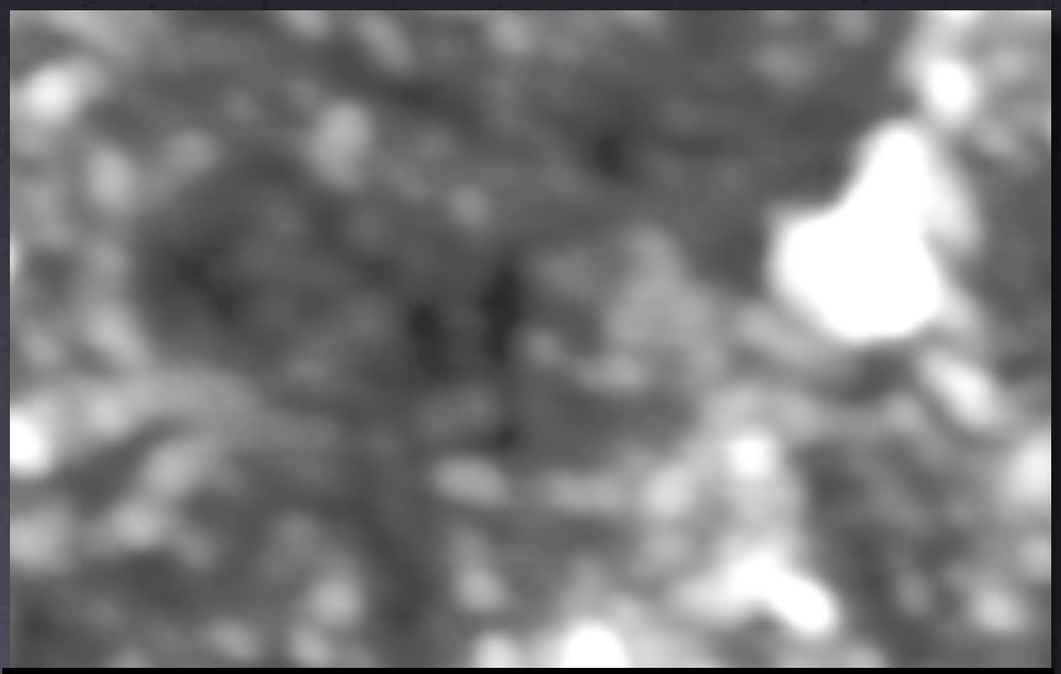
ADL Humphris, MJ Miles, JK Hobbs,
Applied Physics Letters, **86**(3), Art.No. 034106 (2005)

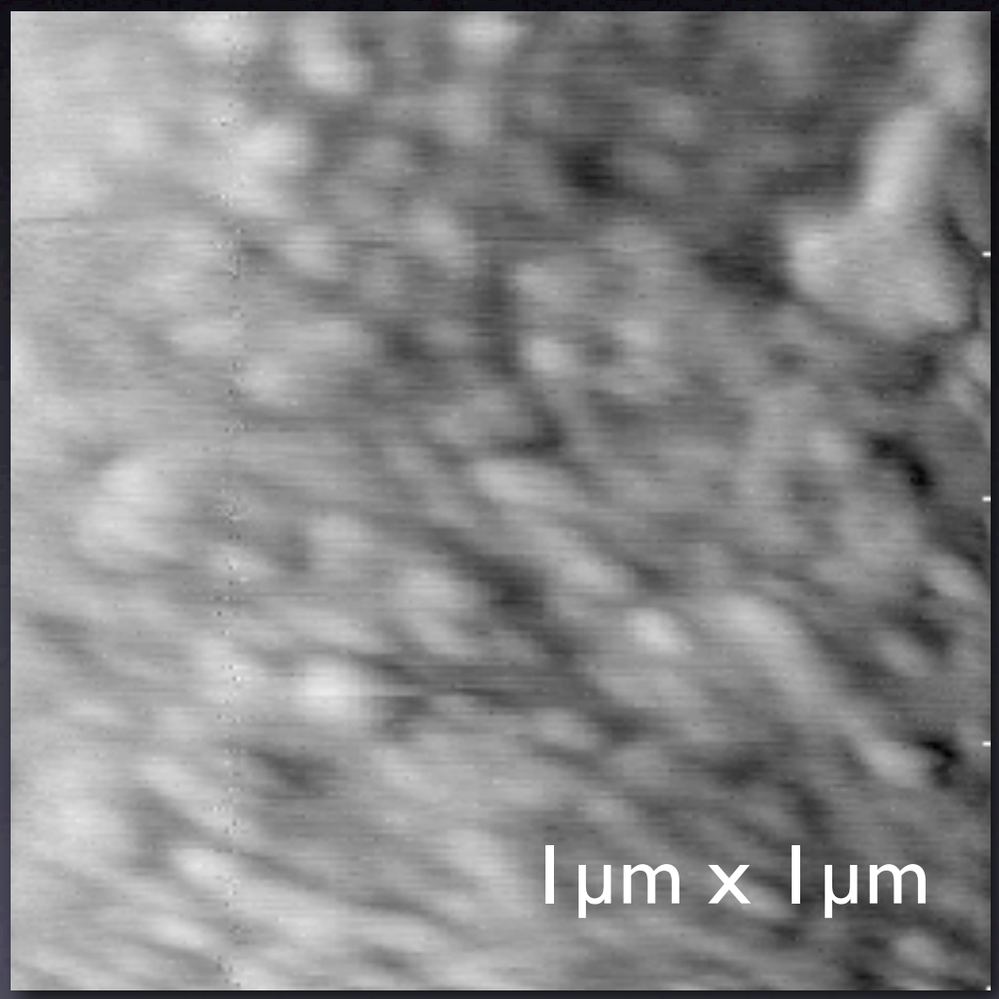
High-speed AFM of Chitosan Film

- tuning-fork resonance scanning

> 3000 Faster than conventional AFM

Conventional AFM



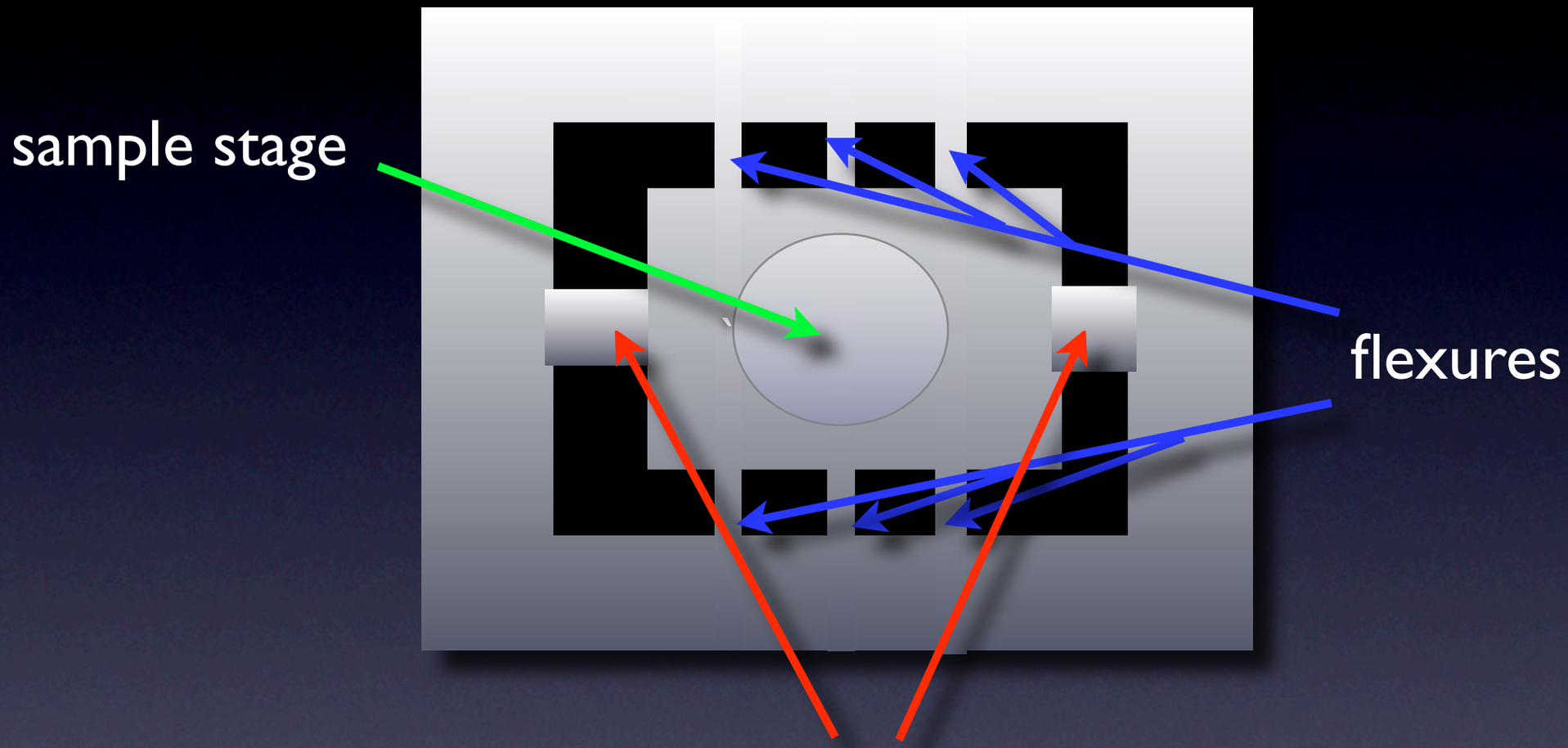


Video AFM 30 frames/s

(Infinitesima vAFM)

Ulcinas, Payne, Heppenstall-Butler

Flexure Stage for high-speed SPM

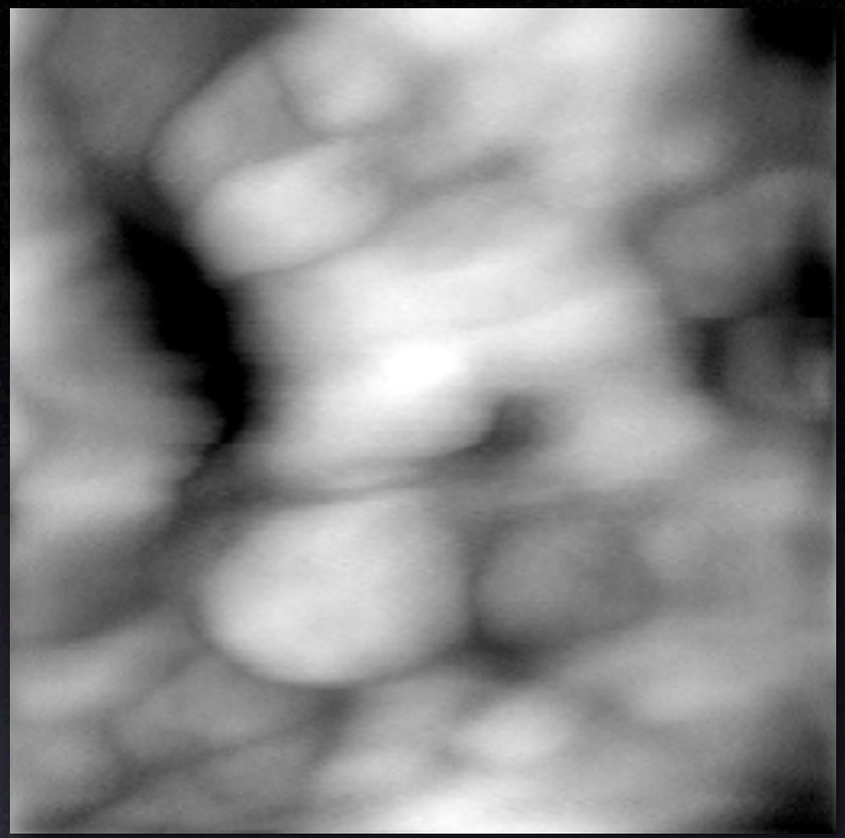


This flexure stage provides the high-speed scan of up to 40kHz with about 3 μm amplitude

Picco, Engledew

PEO video AFM 30 f/s - image reproducibility

0 mins
0 frames

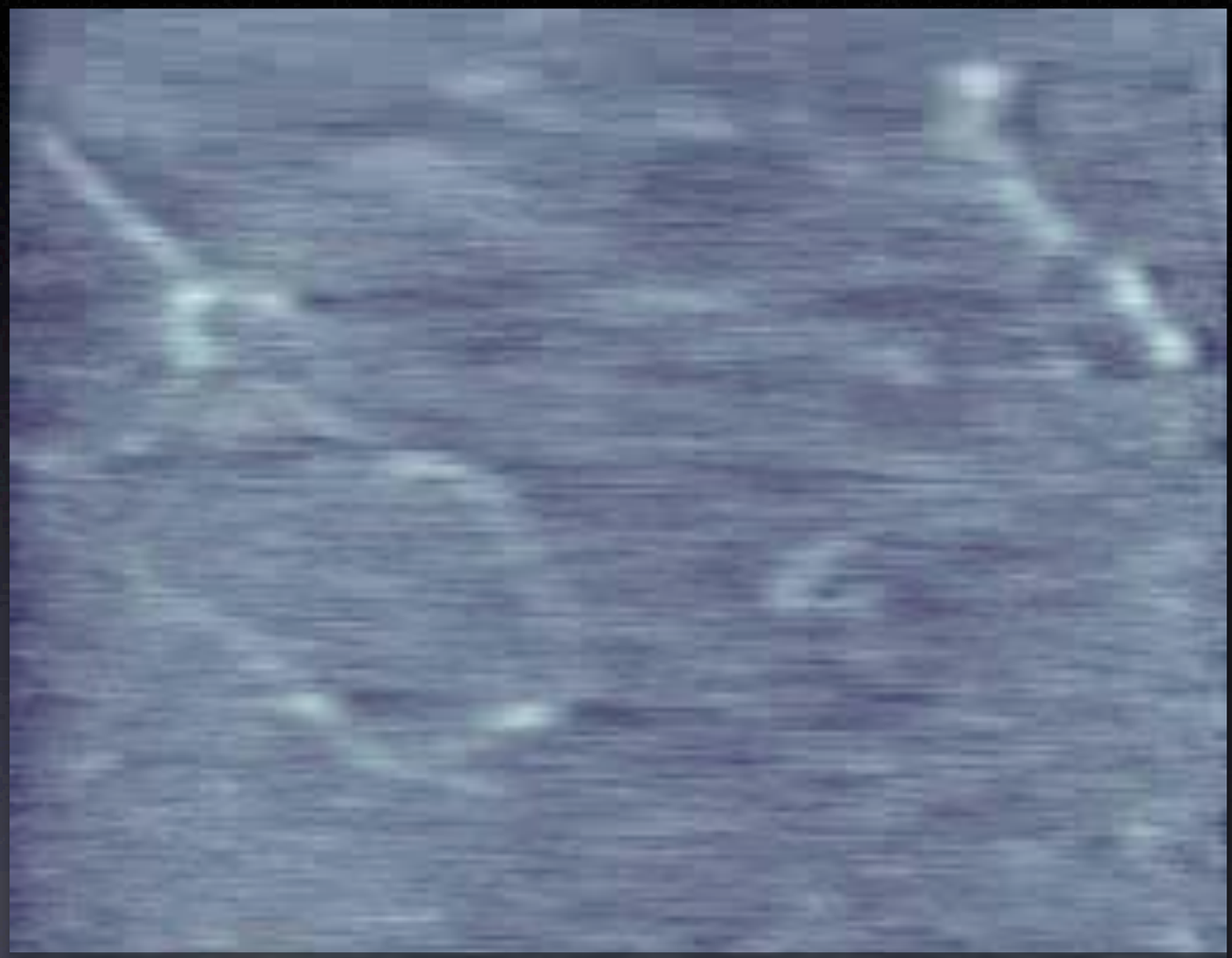


3 mins
~ 6000
frames

7 mins
13,000
frames

10 mins
18,000
frames

High-speed AFM of ‘single molecules’



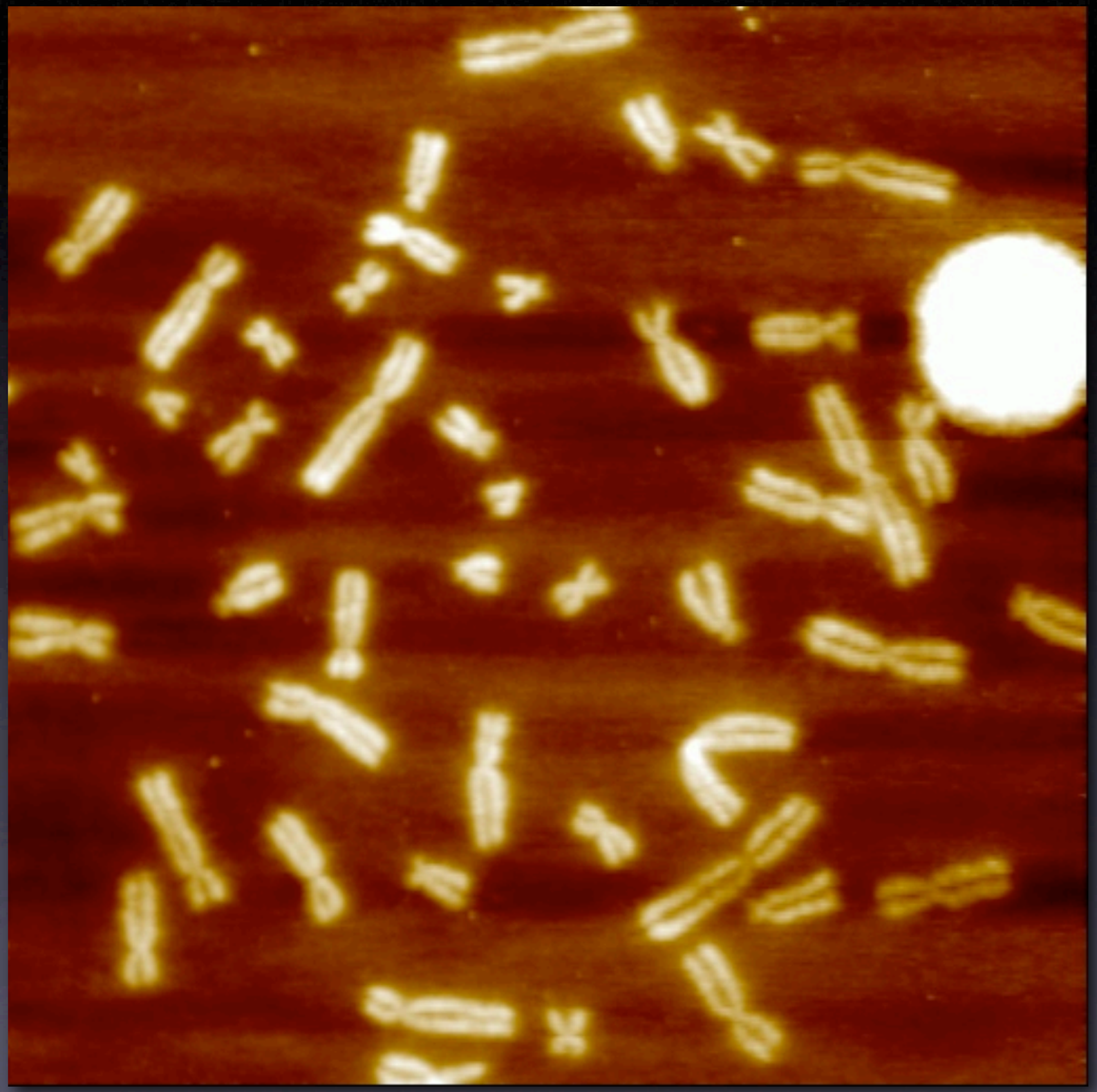
Video AFM in air
partially linearized plasmid DNA
Thomson & Crampton/Infinitesima

High-speed AFM of ‘large’ objects

Air

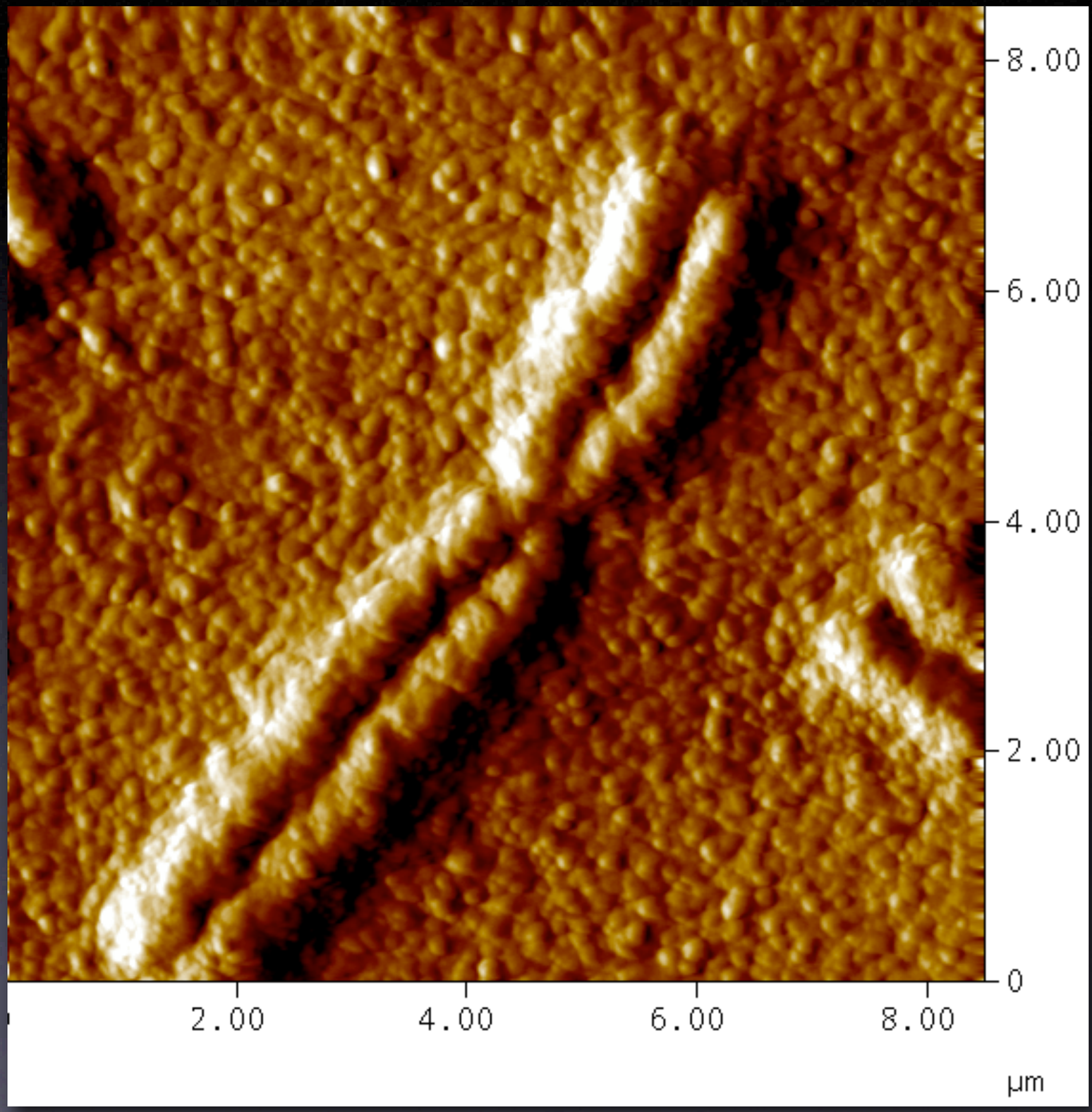
Set of human chromosomes

c AFM



Picco, Miles, Komatsubara, Hoshi, Ushiki

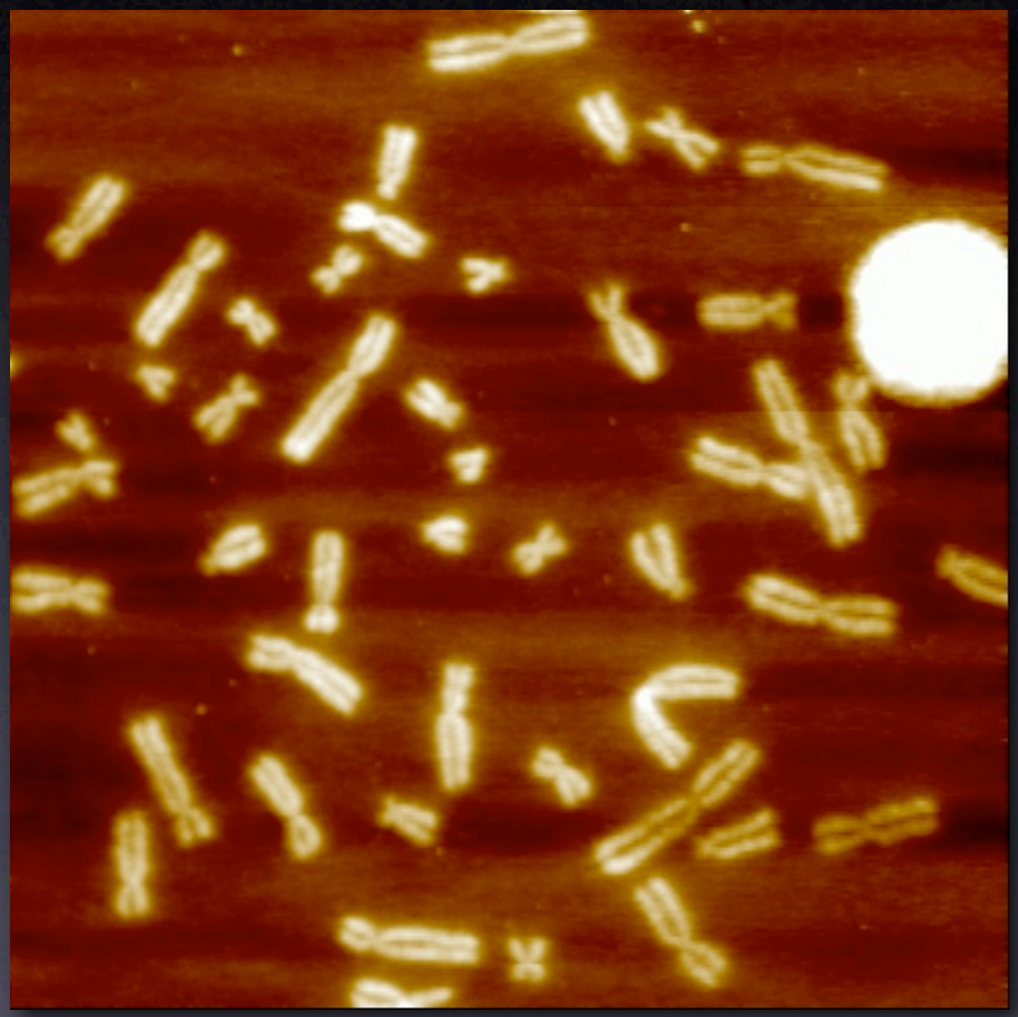
Human Chromosome No. 2



Picco, Miles, Komatsubara, Hoshi, Ushiki

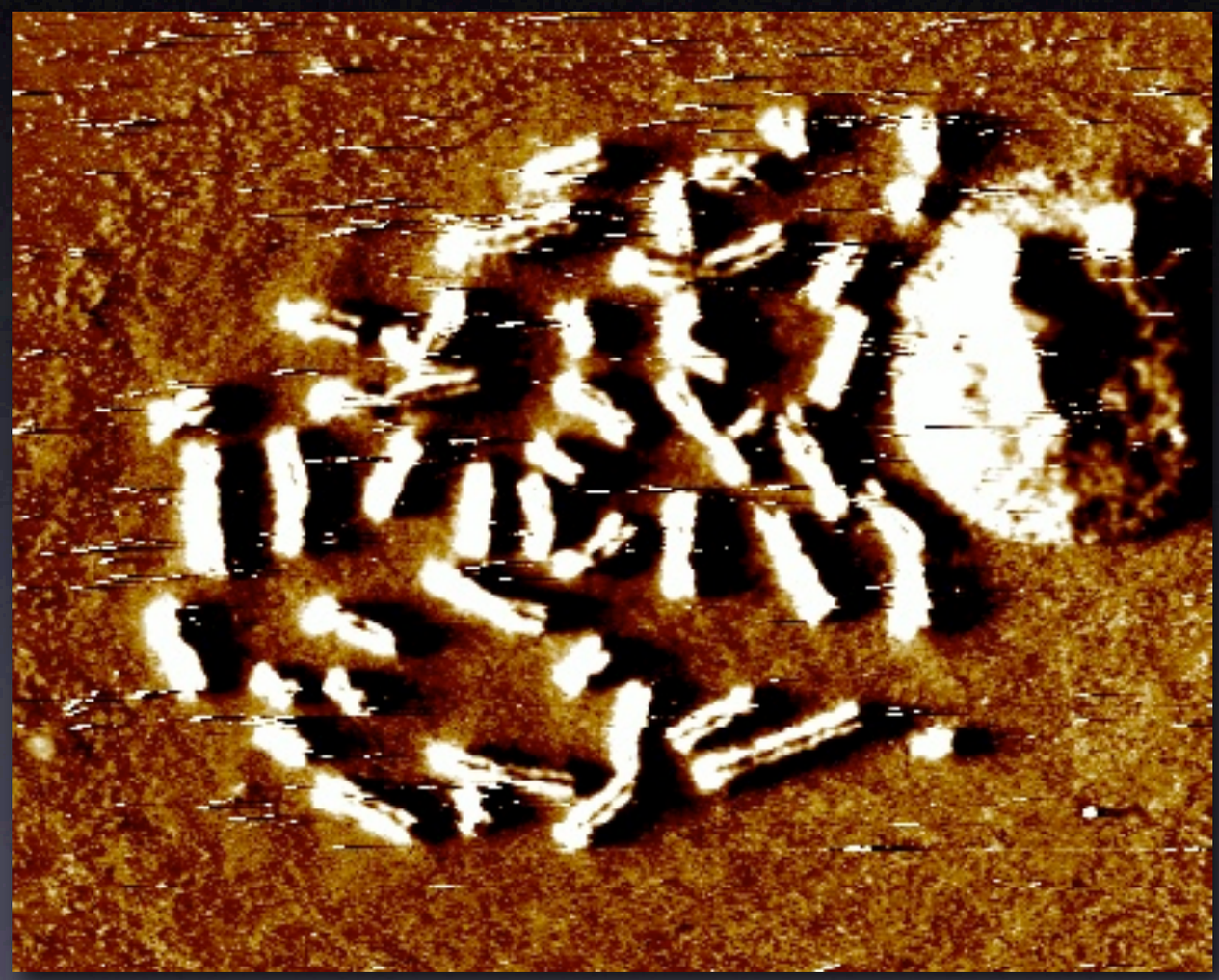
Chromosomes swell >5 times in Height in aqueous Buffer

Air



Chromosome height ~150 nm

2mM NaCl Solution

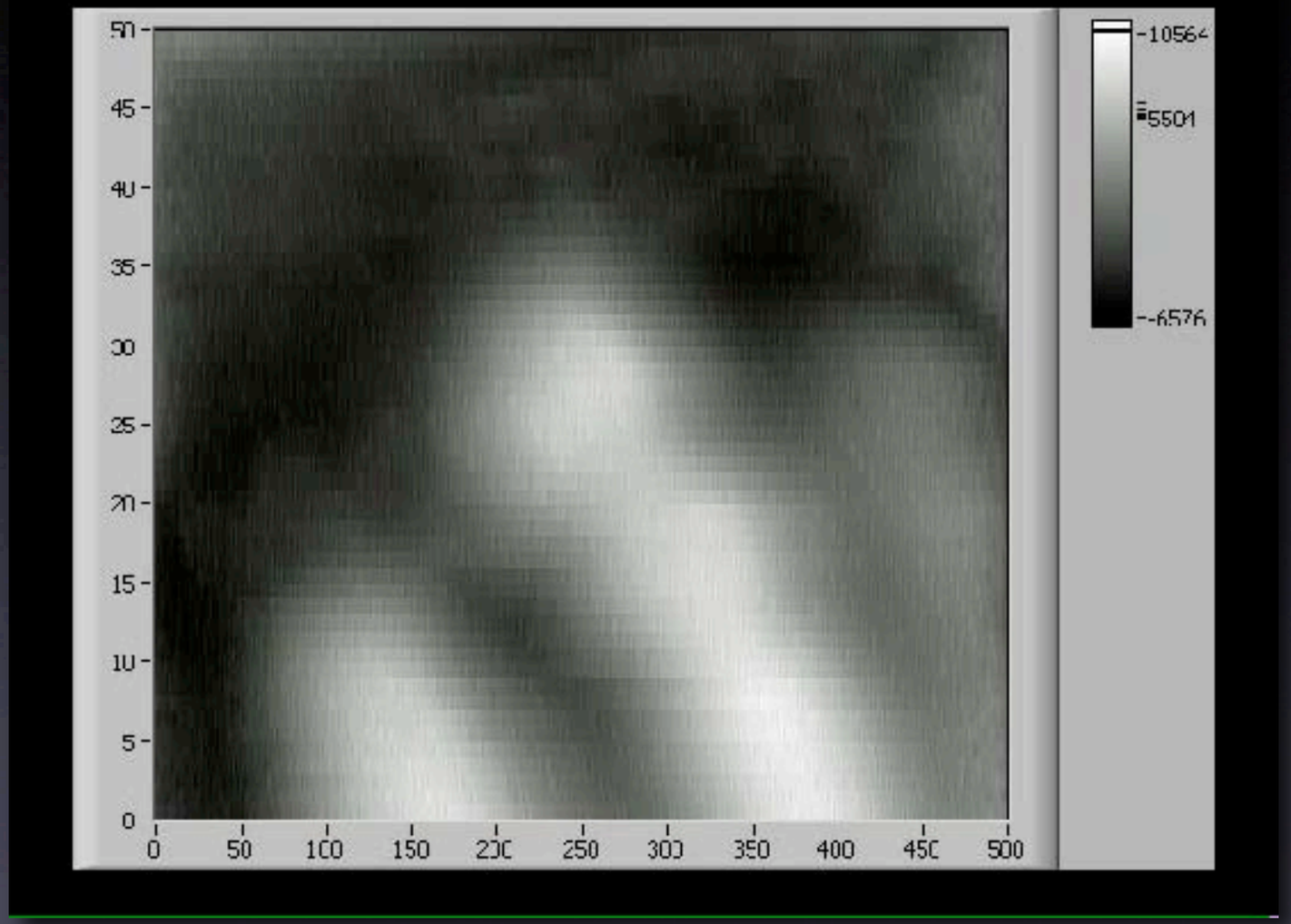


Chromosome height ~700 nm

Picco, Miles, Komatsubara, Hoshi, Ushiki

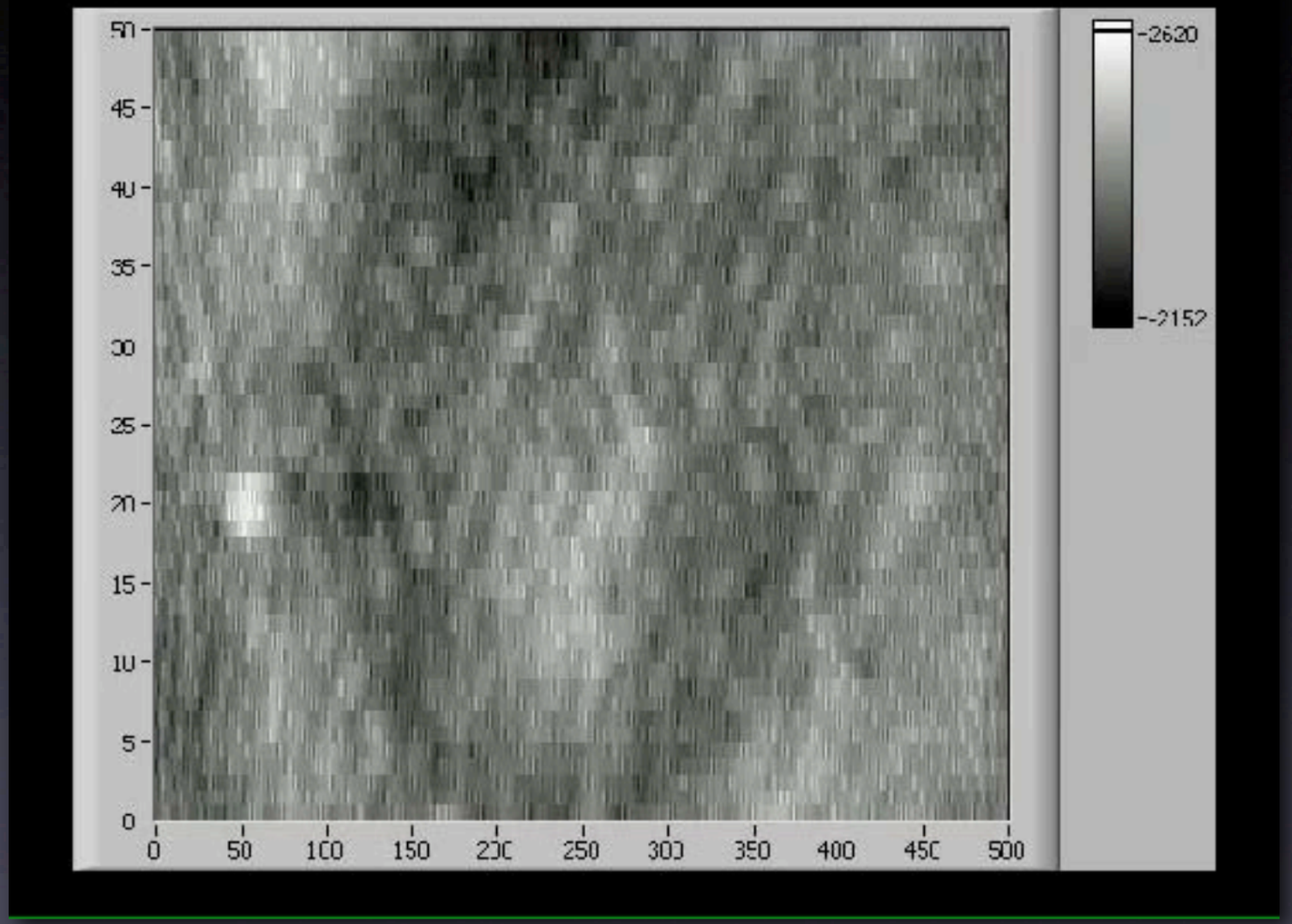
Video AFM of Human Chromosomes in Aqueous Buffer

chromosomes: 1, 2, 7(?), 22



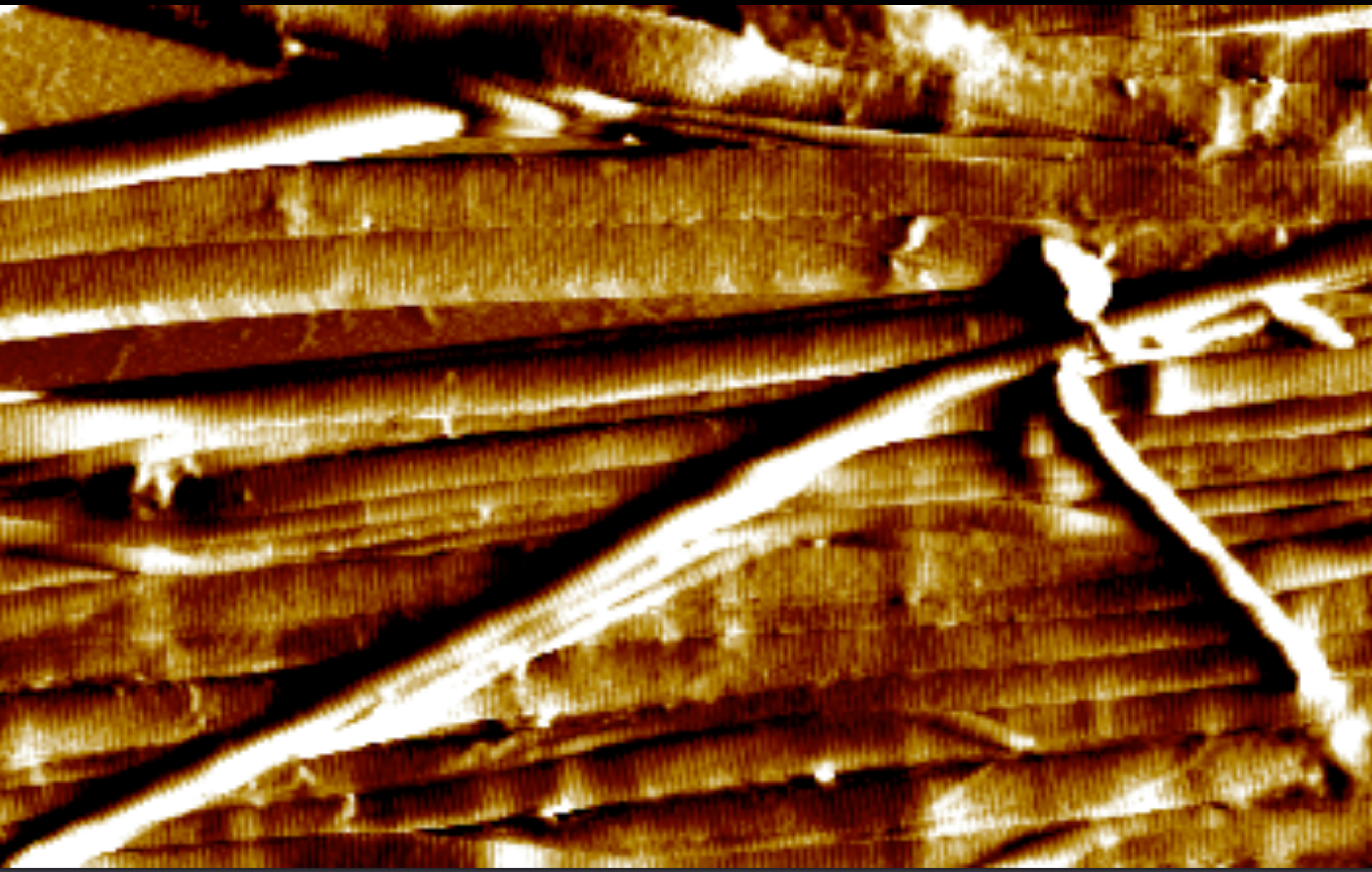
~ 1 µm

Video AFM Human Chromosome - higher mag

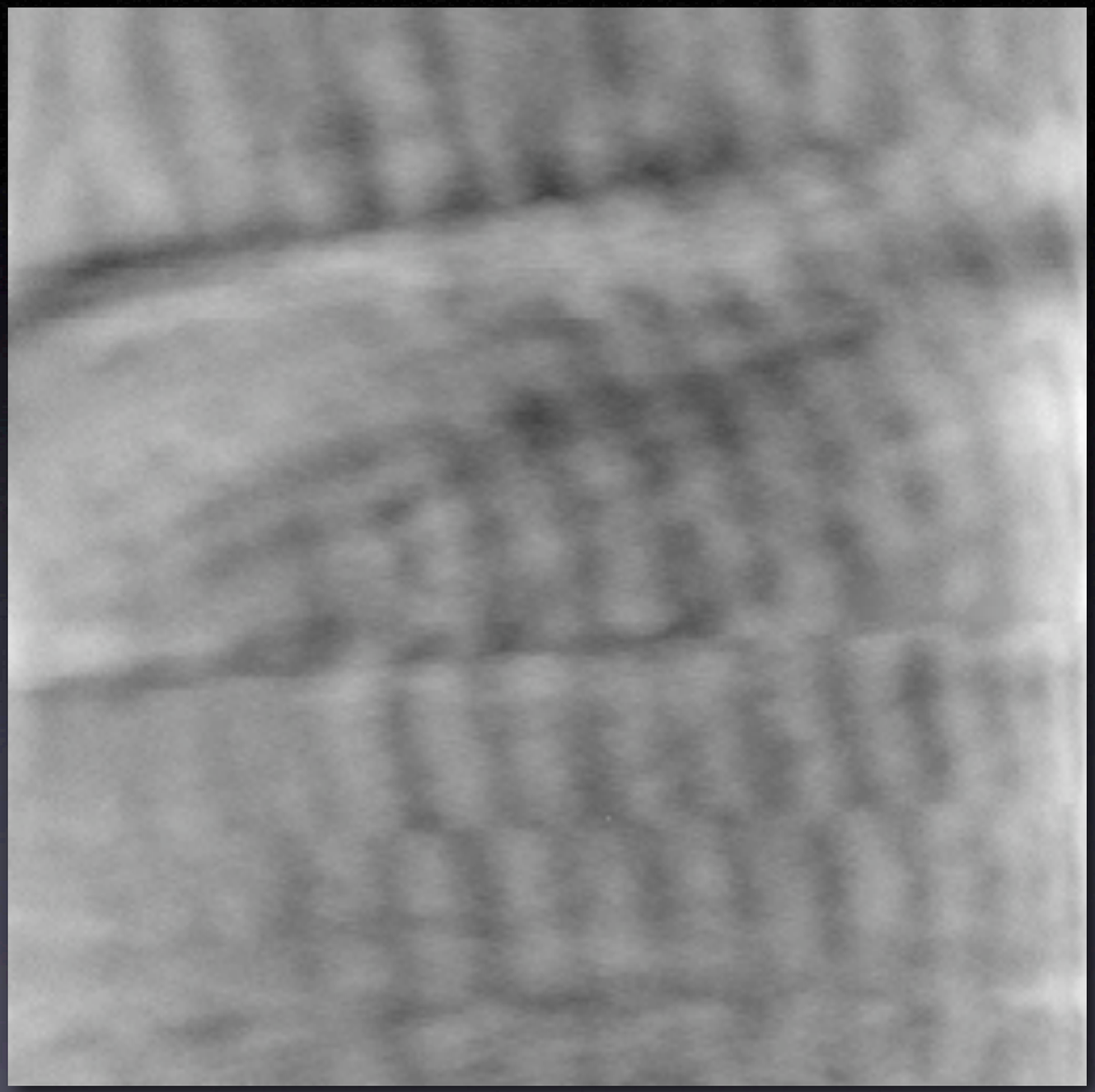


Collagen Micro fibrils

Conventional AFM Image of Collagen Specimen



Collagen Fibres



Banding
periodicity: 67 nm

30 fps

L M Picco, L Bozec, A Ulcinas, D J Engledew, M Antognozzi, M A Horton and M J ***Miles***
Nanotechnology **18** No 4 (31 January 2007) 044030

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

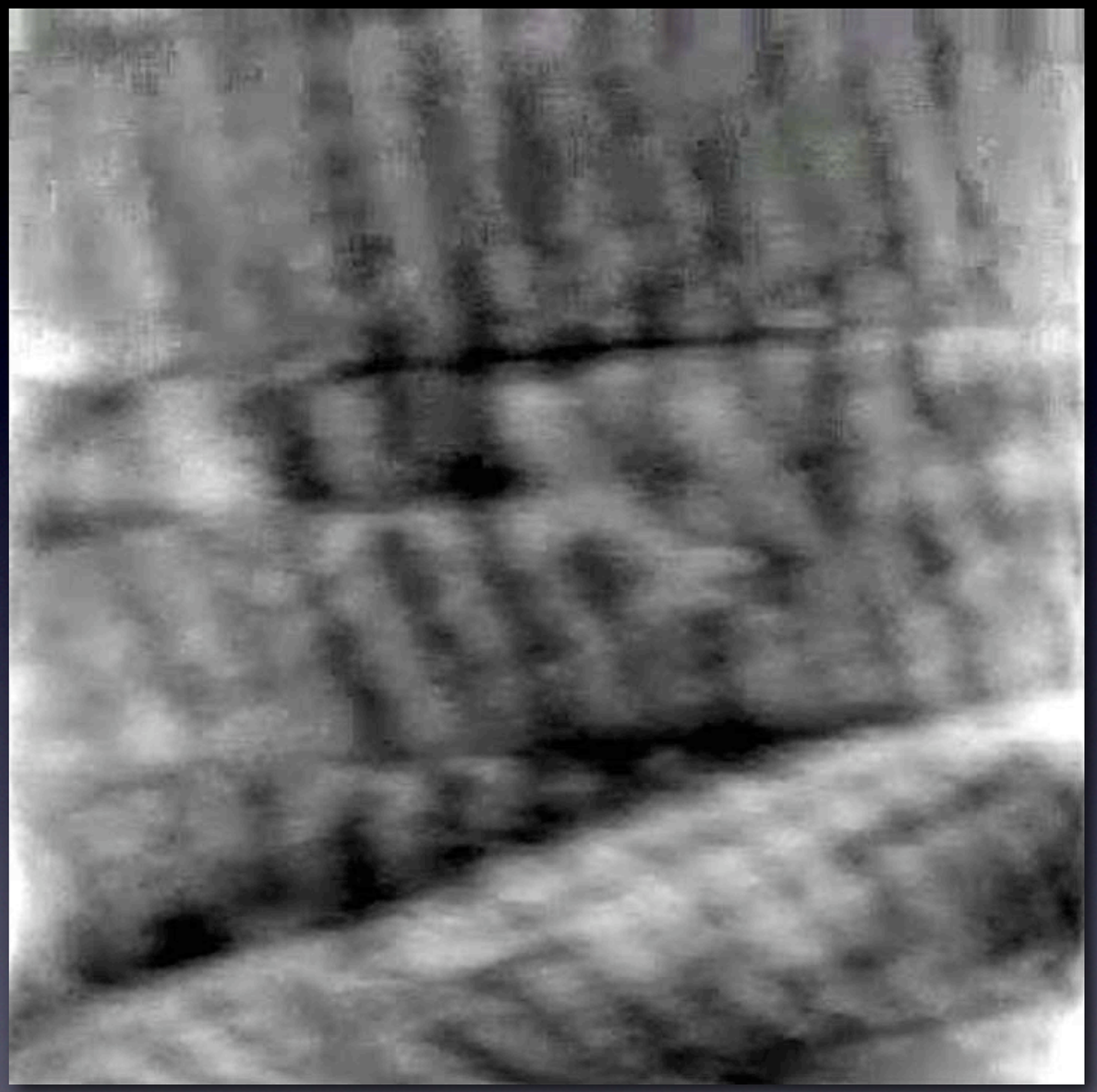
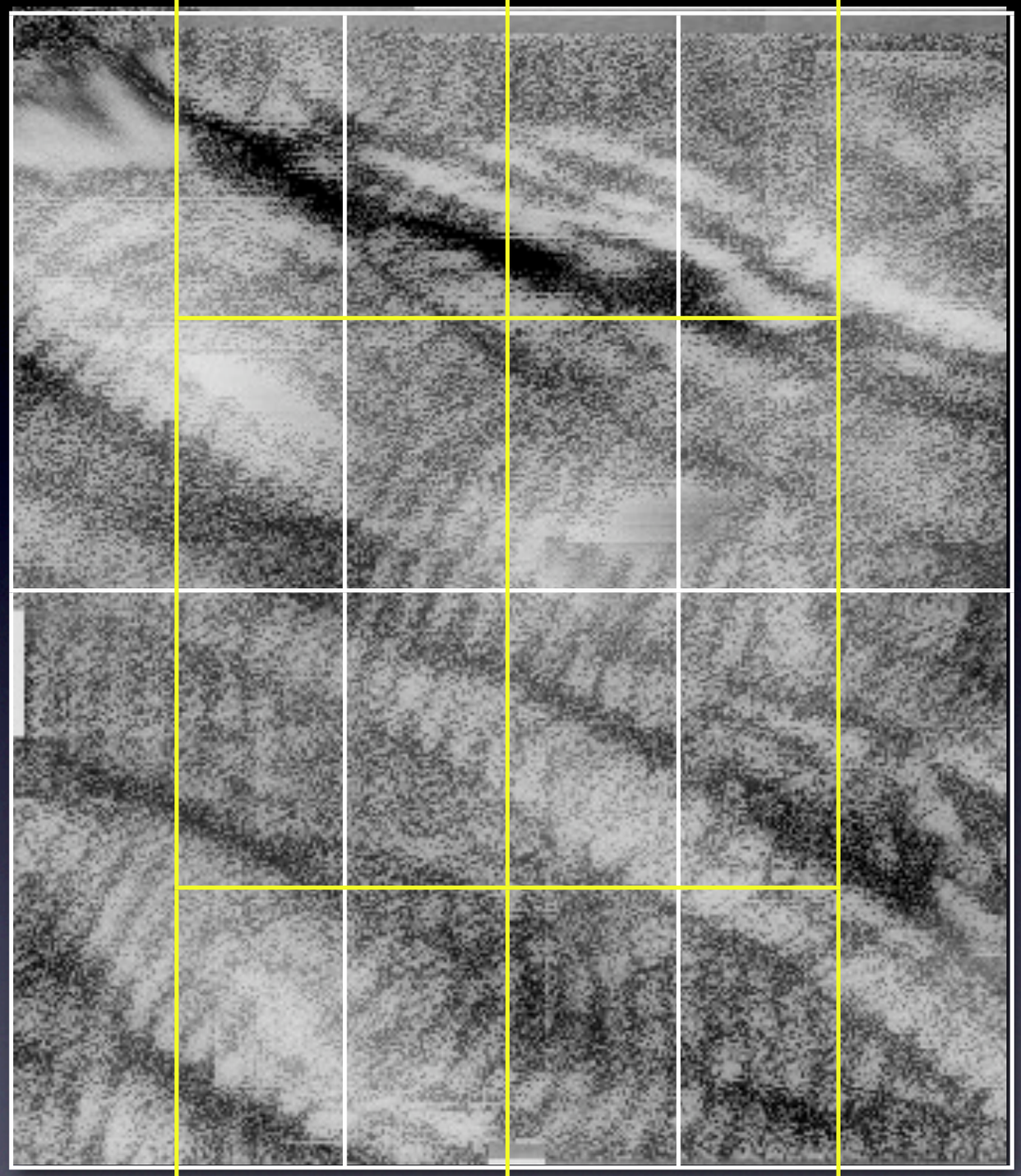


Image acquired in 17 ms

Picco, Bozec, Horton Engledew

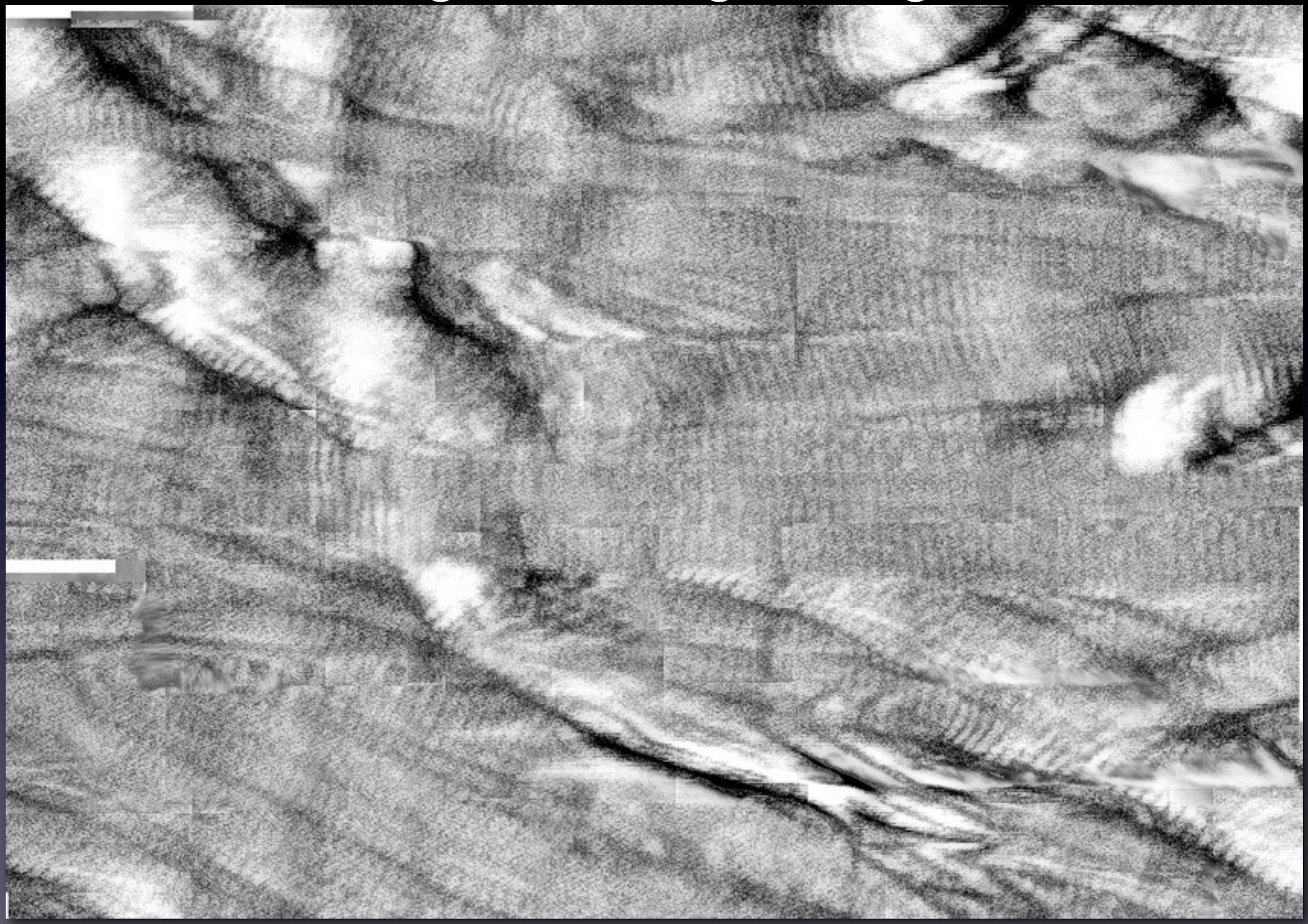
High-speed AFM Collagen Collage



12 images involved - acquired in 200 ms

L M Picco, L Bozec, A Ulcinas, D J Engledew, M Antognozzi, M A Horton and M J ***Miles*** *Nanotechnology* **18** No 4 (31 January 2007) 044030

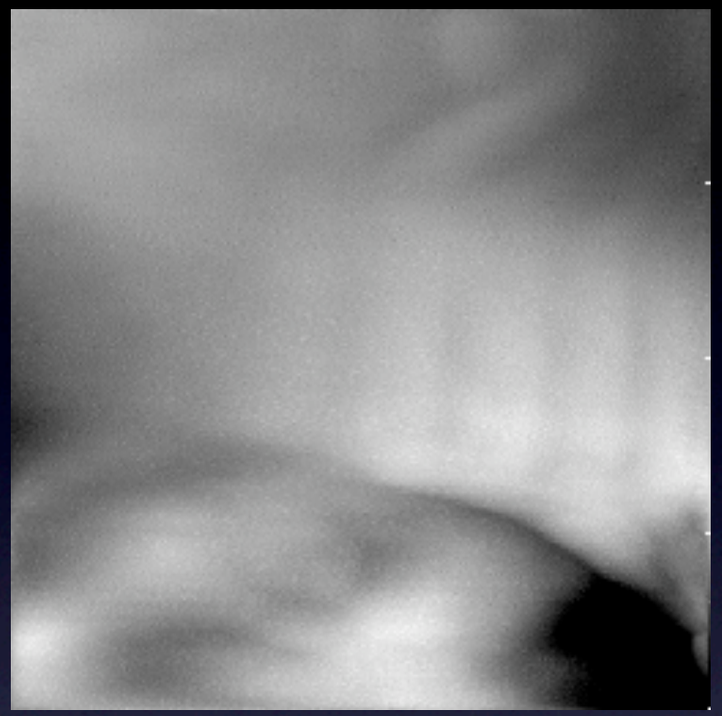
Large area collagen collage



103 images acquired in 1.7 s

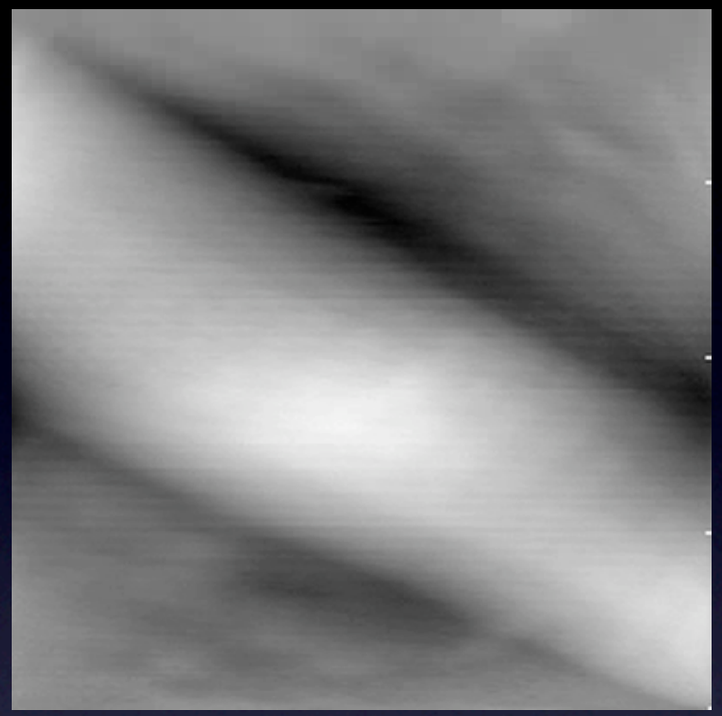
High-speed AFM of Collagen

Bimorph



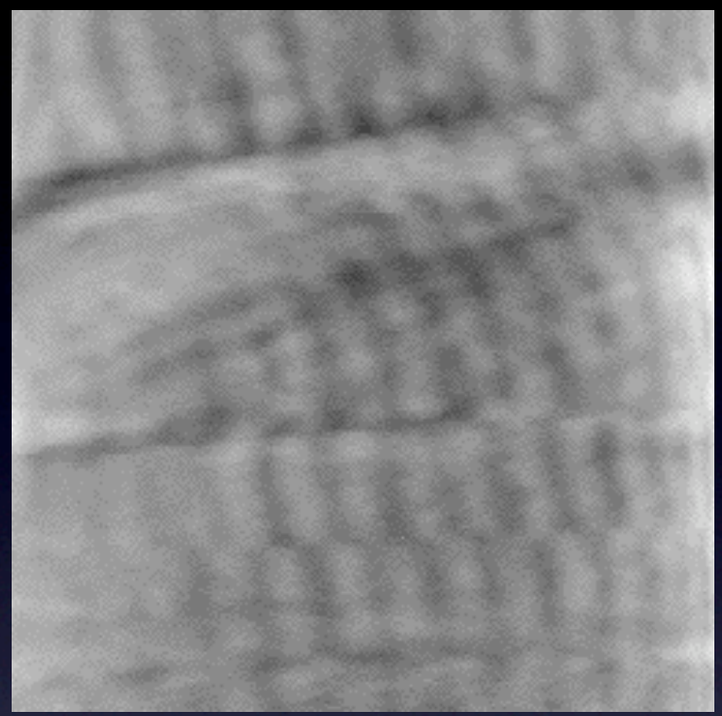
9 fps

Tuning fork



15 fps

Flexure



30 fps

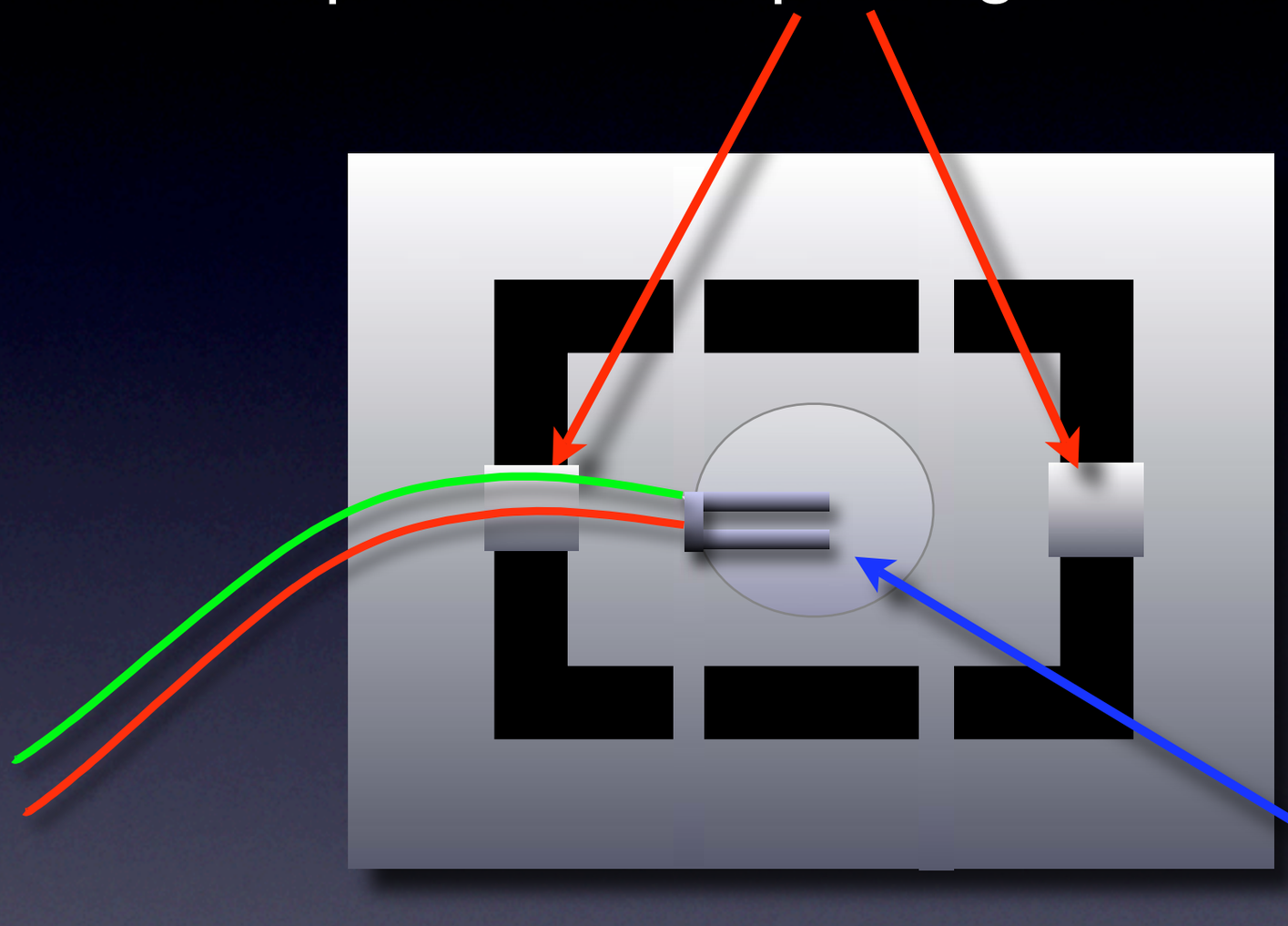
How fast can this system image?

> 1000 fps

AFM images in less than 1 ms

Scanner Arrangement for kilohertz AFM

piezo drivers operating in tandem



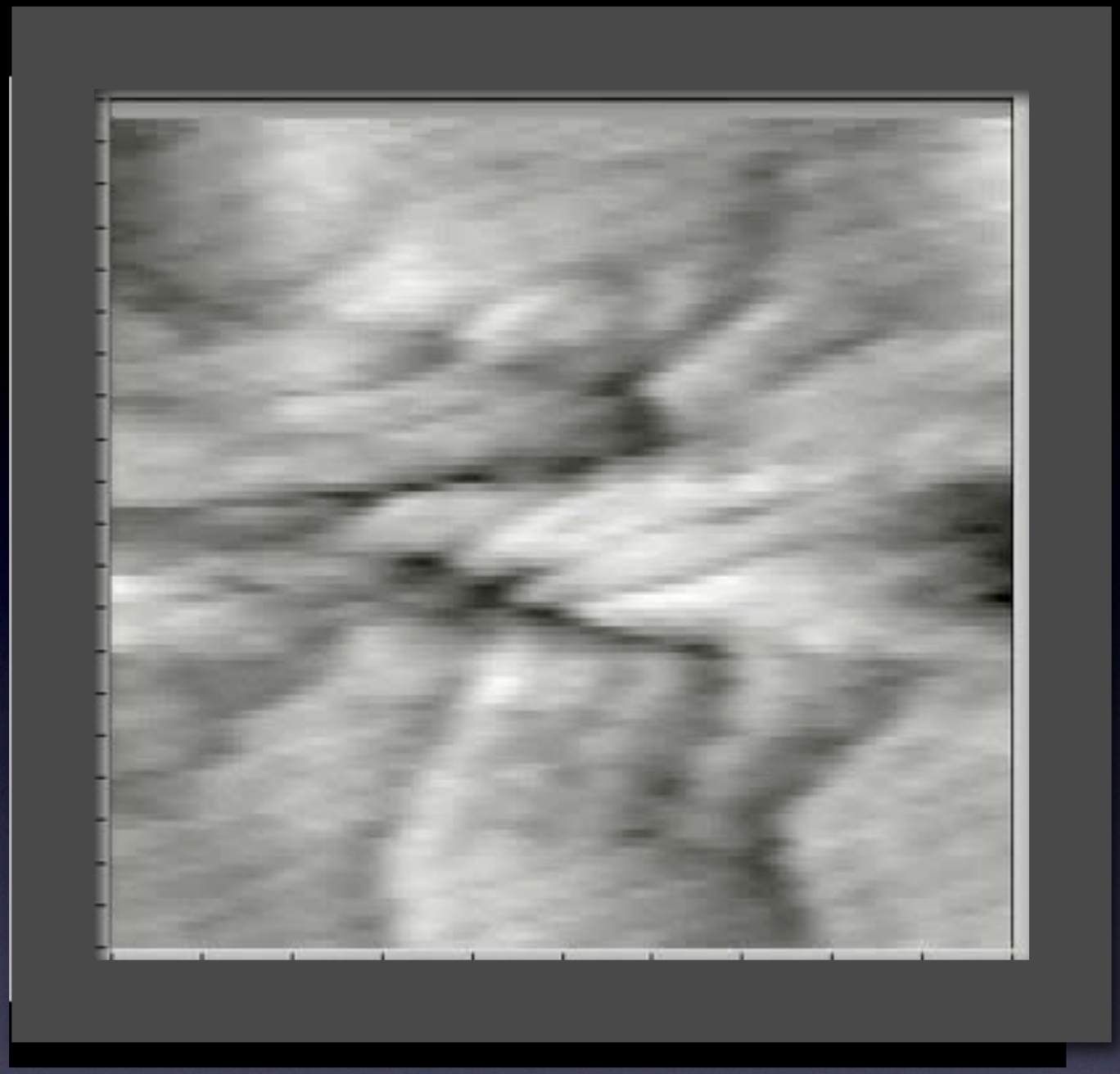
tuning fork scanning

Frame Rate:

1000 fps

Line Rate:

200,000 lps



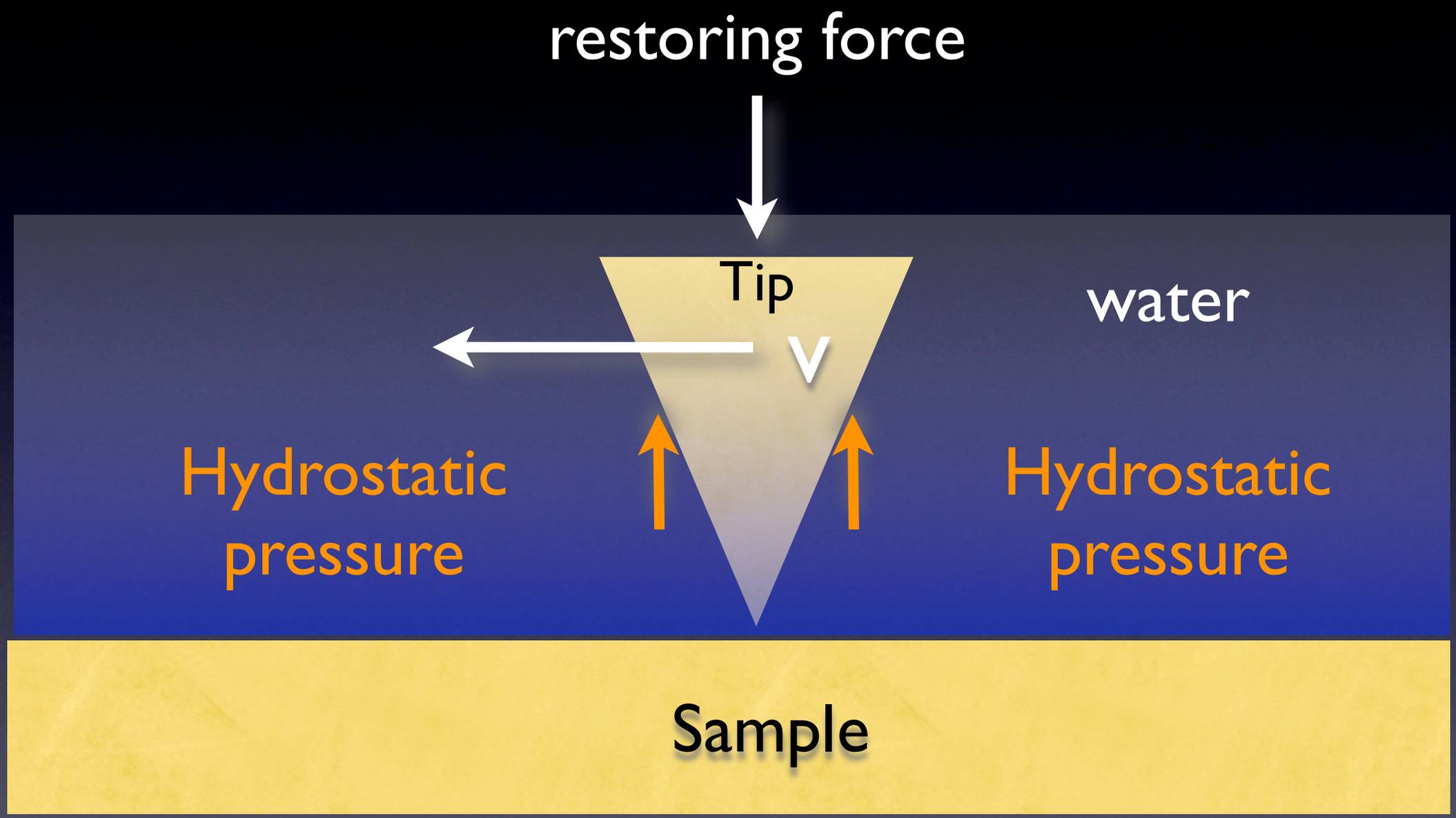
Playback Rate:

25 fps

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

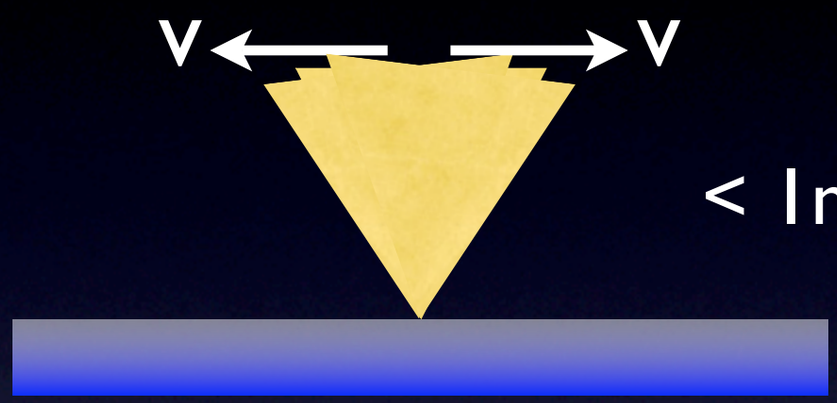
L M Picco, L Bozec, A Ulcinas, D J Engledew, M Antognozzi, M A Horton and M J ***Miles*** *Nanotechnology* **18** No 4 (31 January 2007) 044030

Principle of Superlubrication ‘Feedback’



Torque on Tip

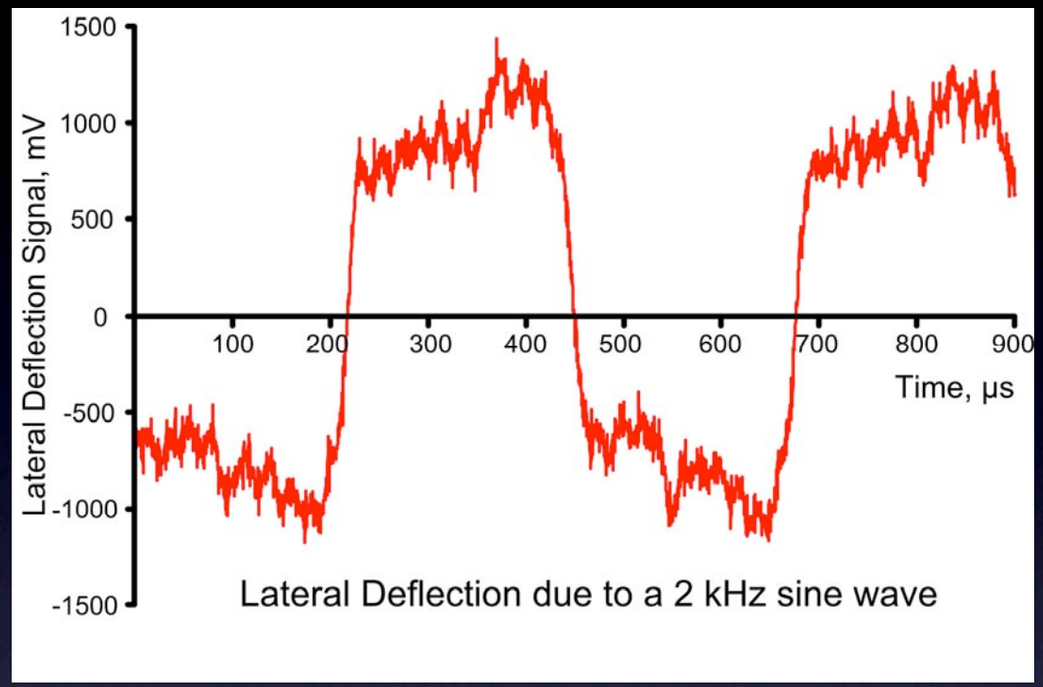
Sine wave displacement of sample



< 1mm/s

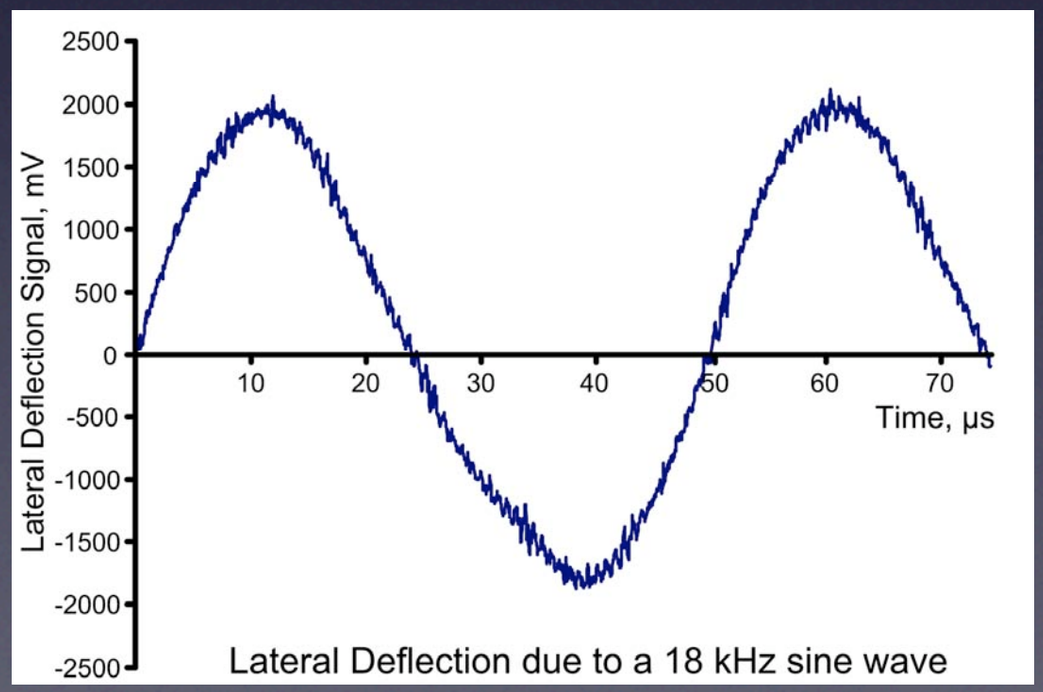
Surface Friction

Velocity Dependent Transition



>> 1mm/s

Hydrodynamic Viscous Force (velocity dependent)

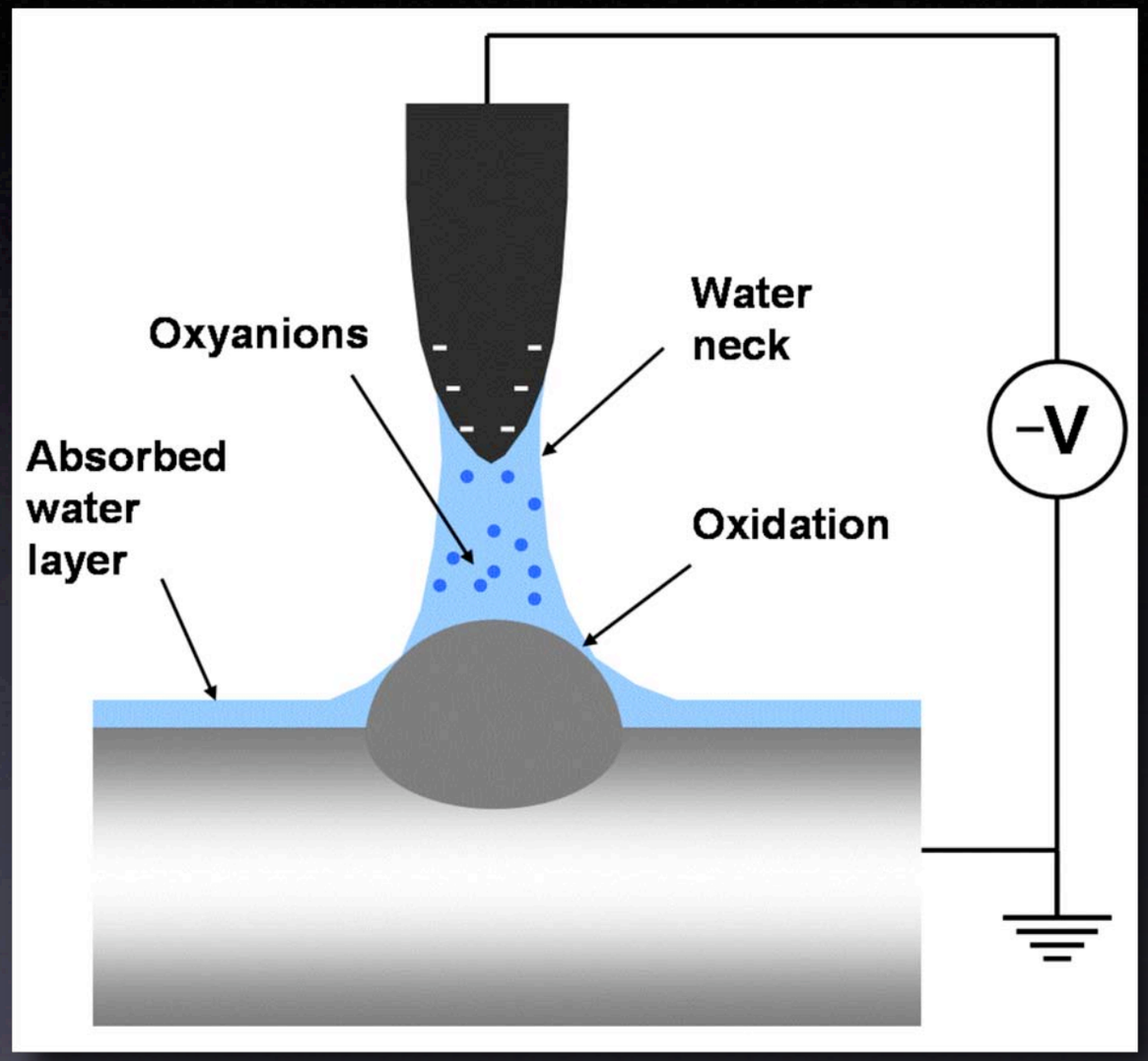


Writing with High-speed AFM

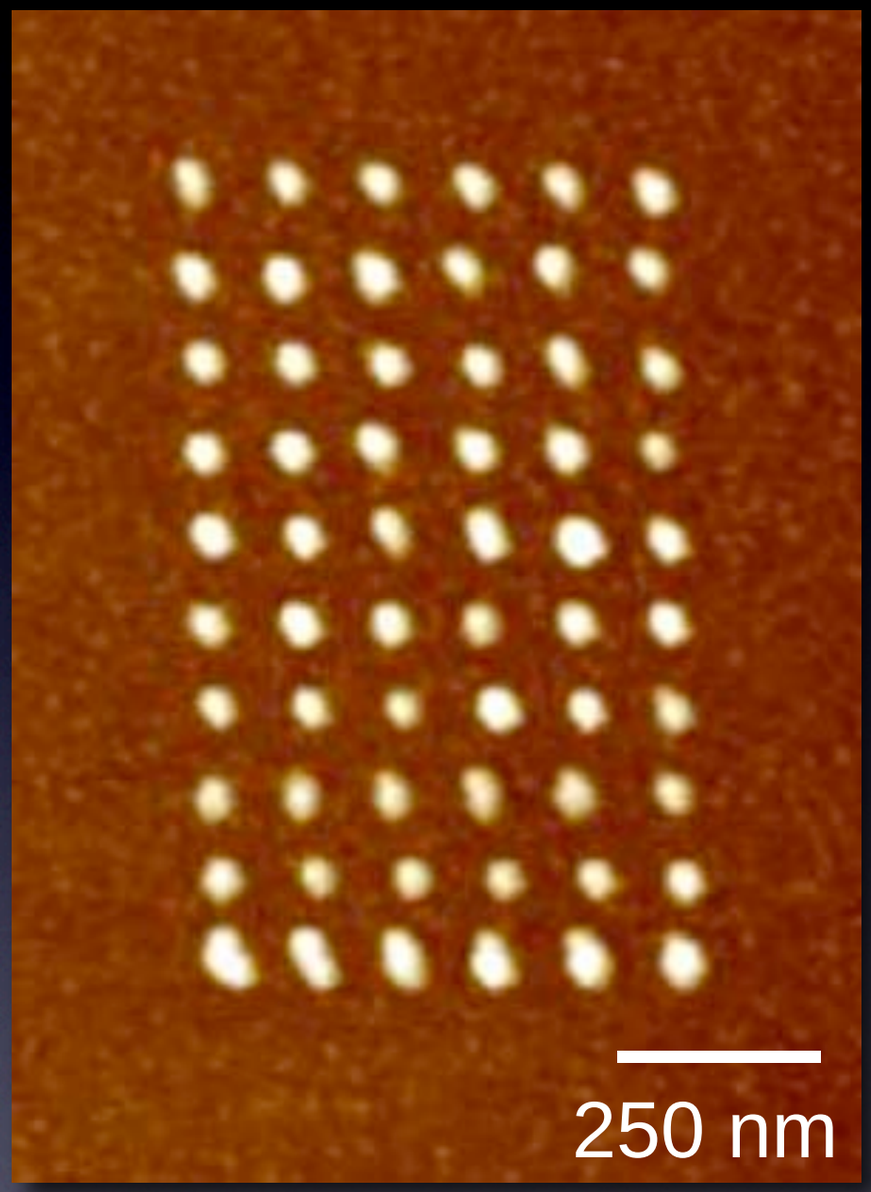
Non-imaging

Electrochemical oxidation of passivated silicon

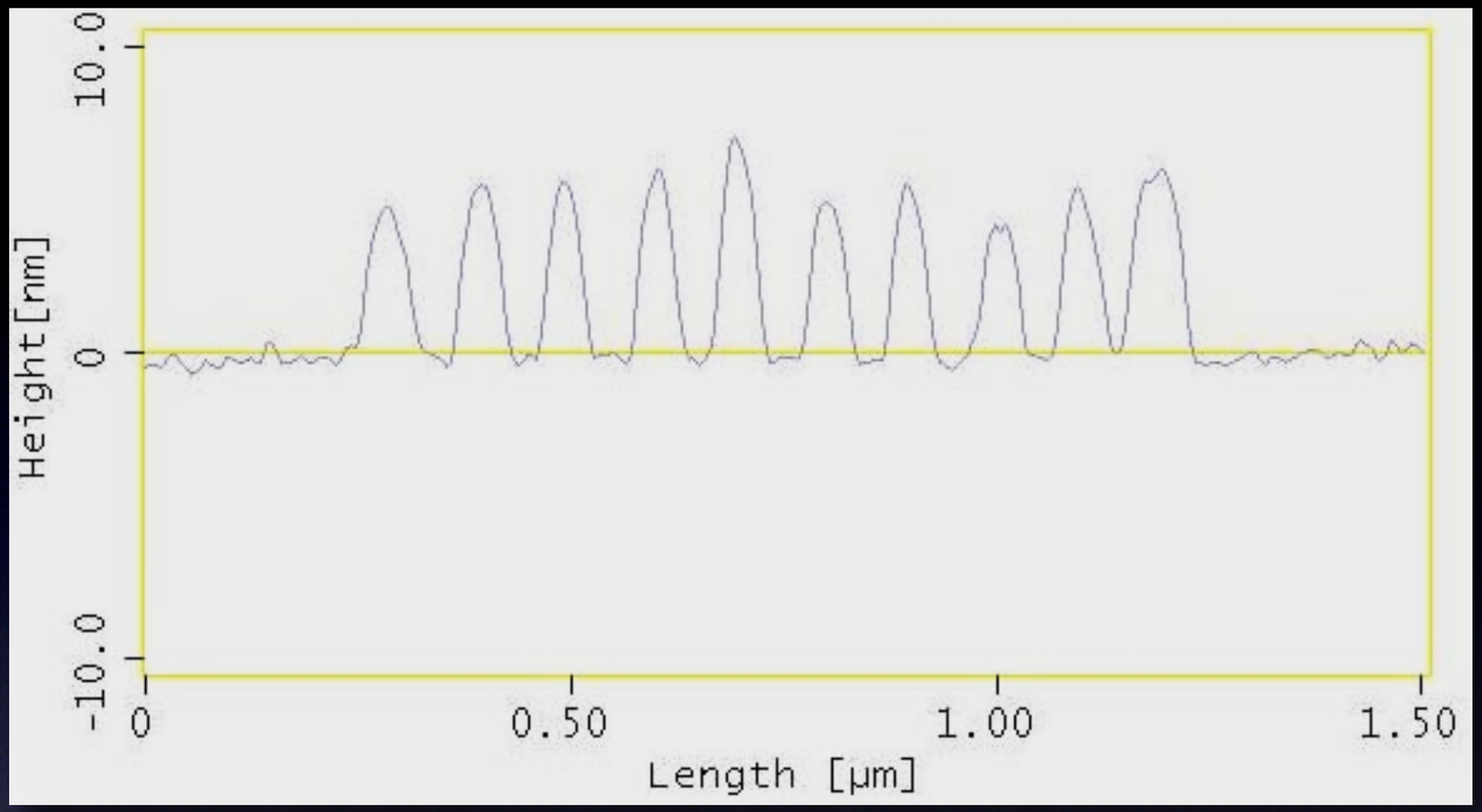
Local AFM electrochemical oxidation

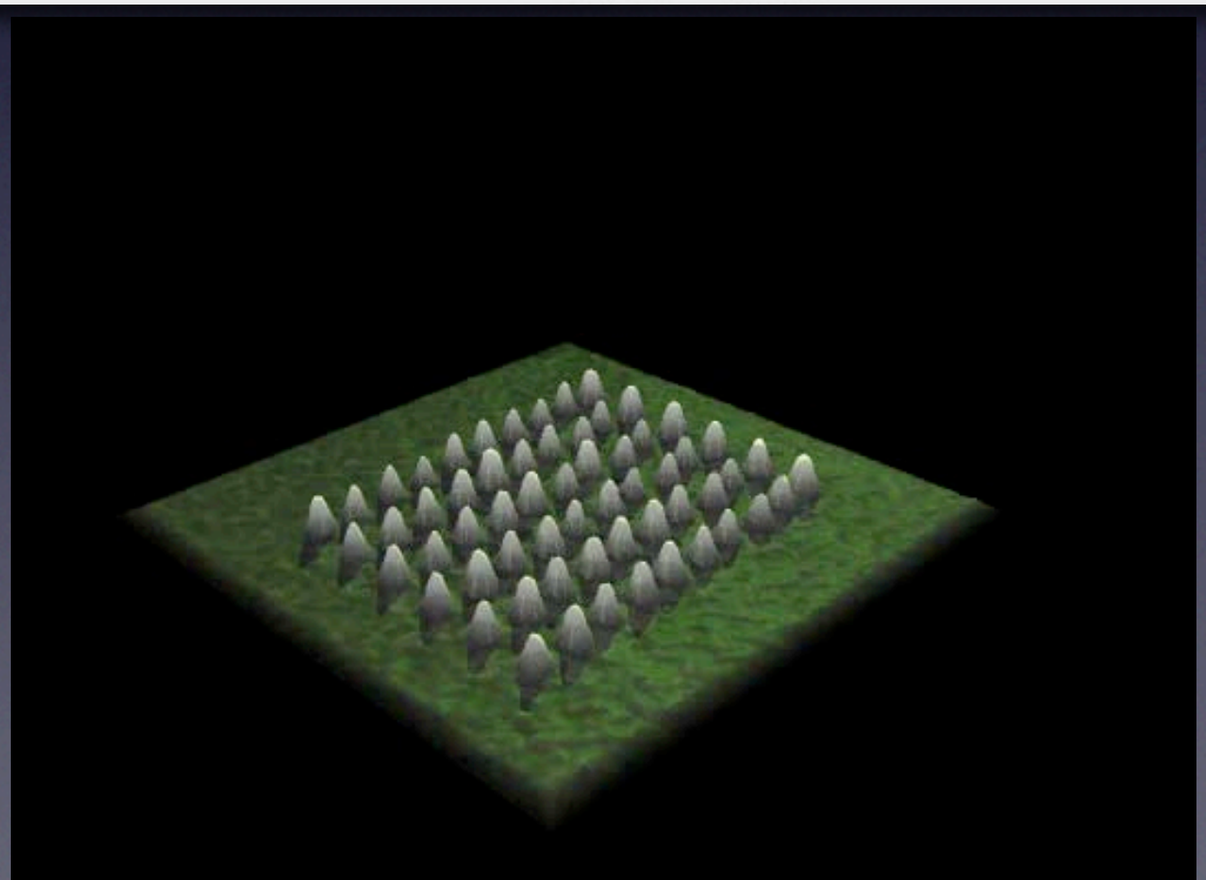


Can be used to pattern surface -
conventional AFM too slow to pattern large areas

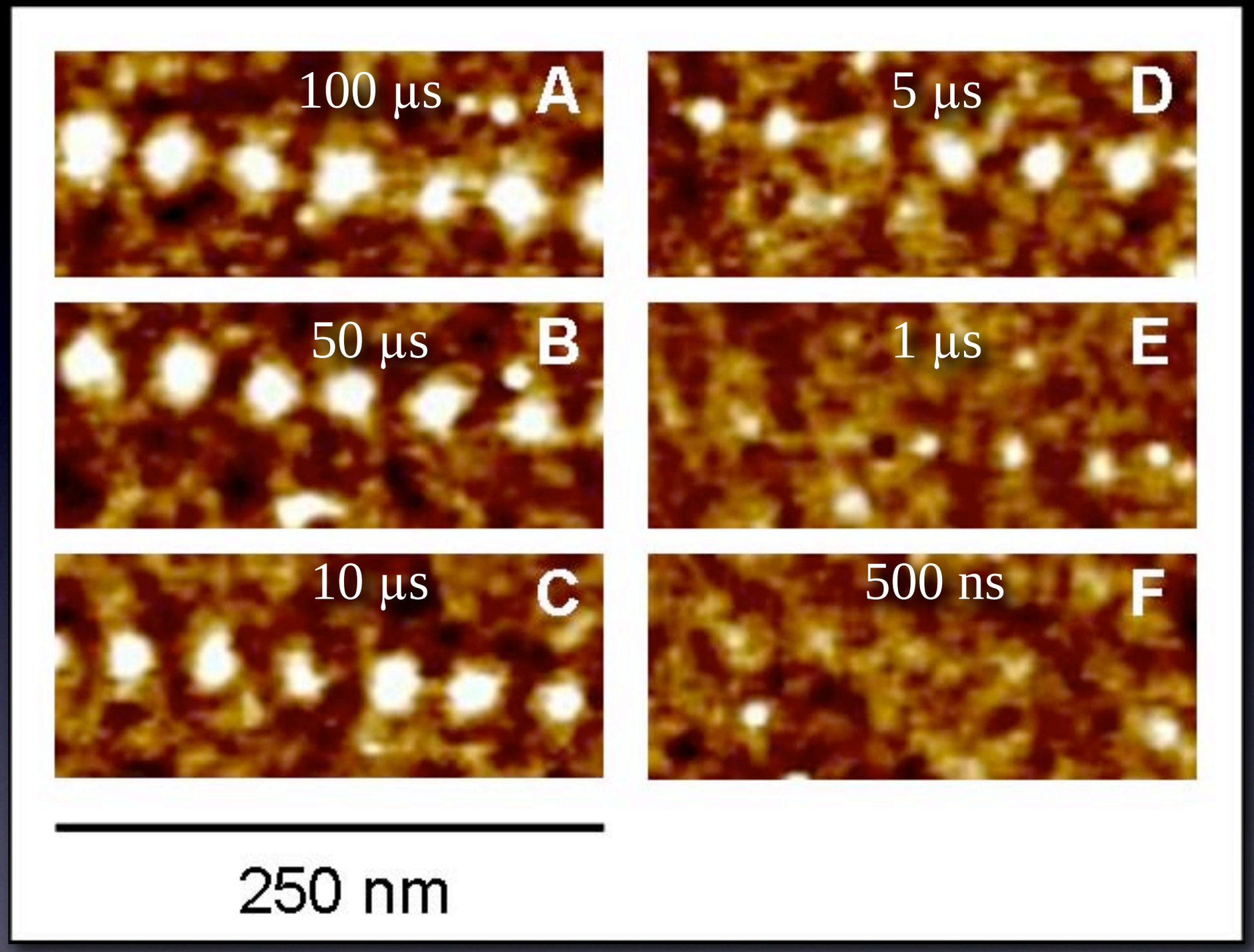


10 ms; -12 V Si tip;
conventional AFM
non-contact





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.

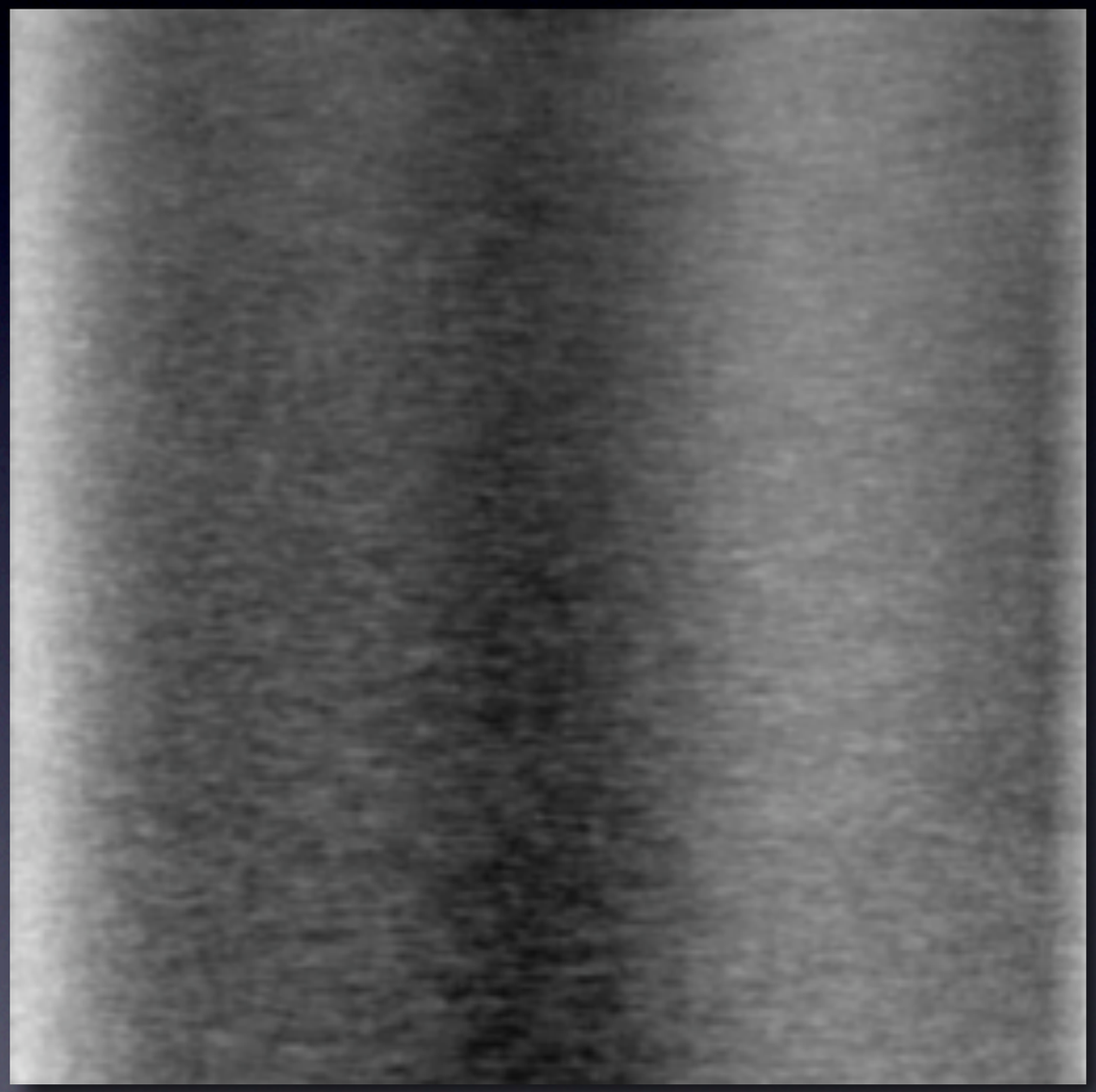
Picco, Vicary

High-speed AFM

Simultaneous writing (oxidation) and imaging

30 fps

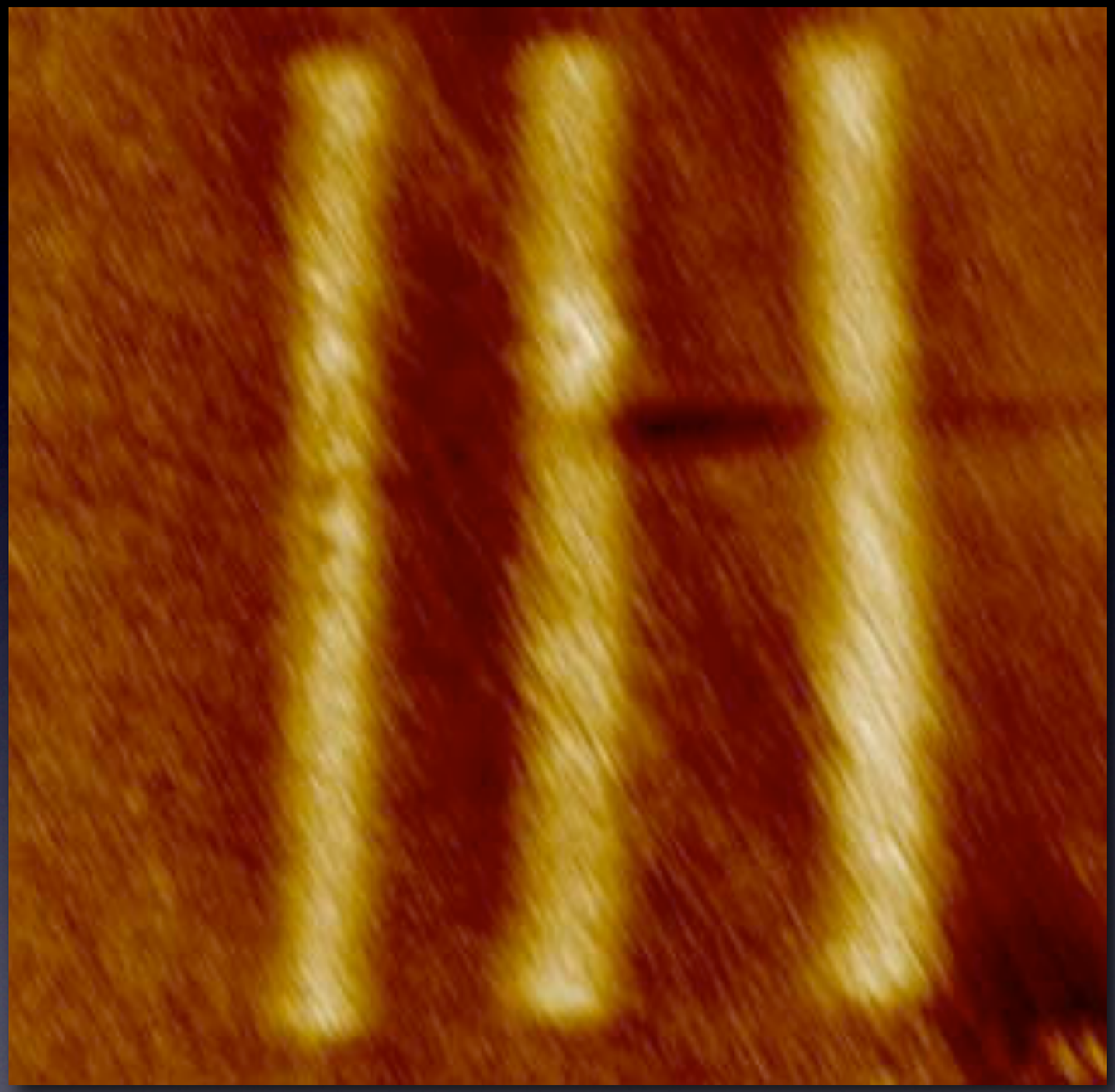
Read-Write



3 voltage pulses synchronized to fast line scan

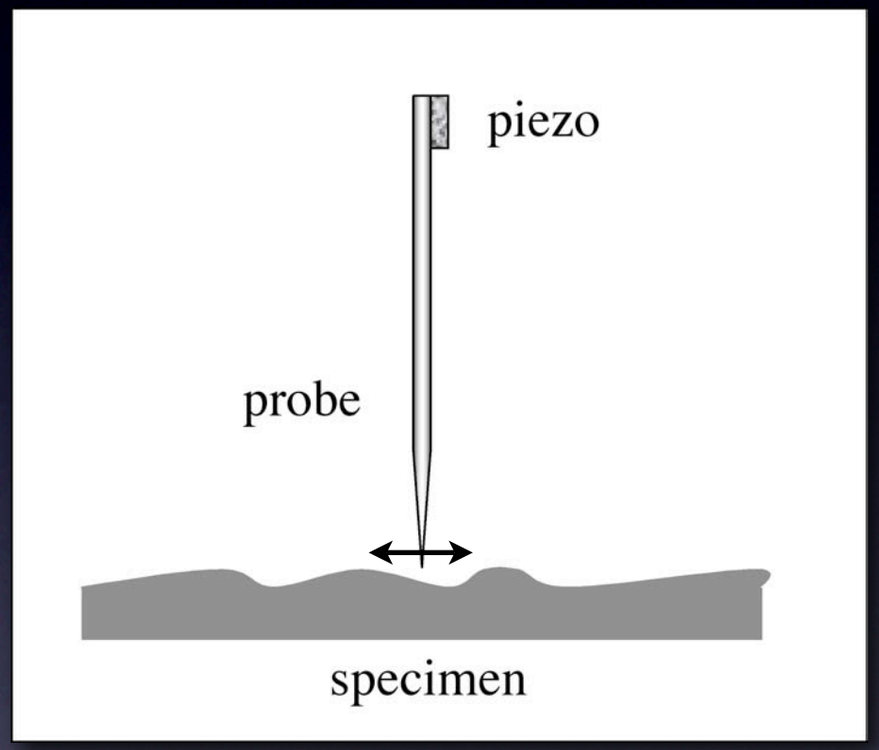
Picco, Vicary

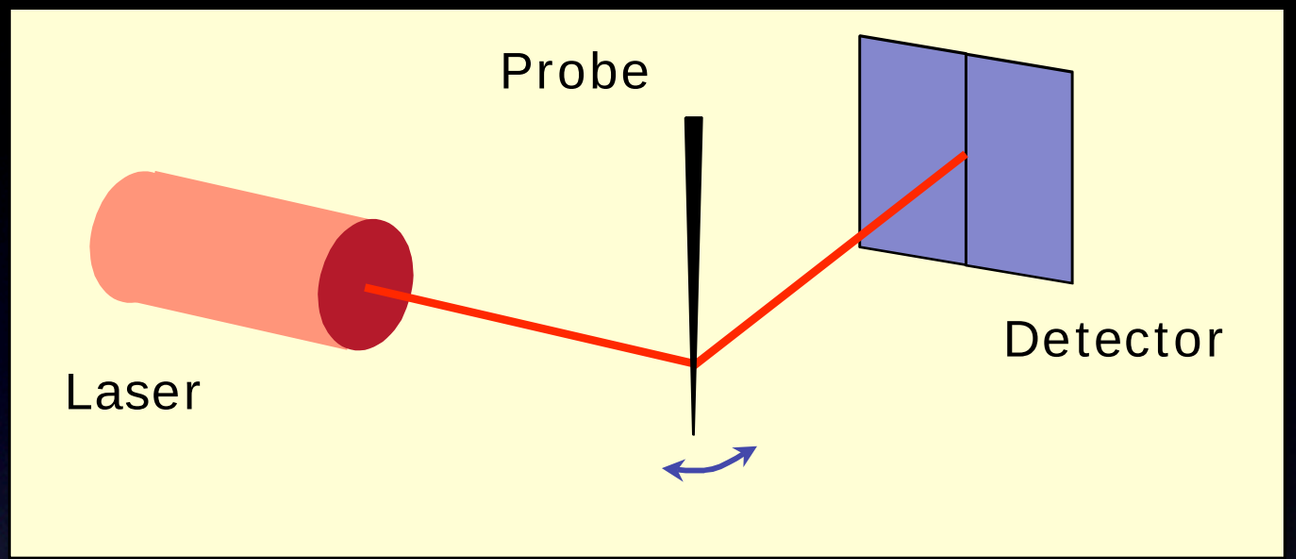
Conventional AFM of oxidized lines on Si

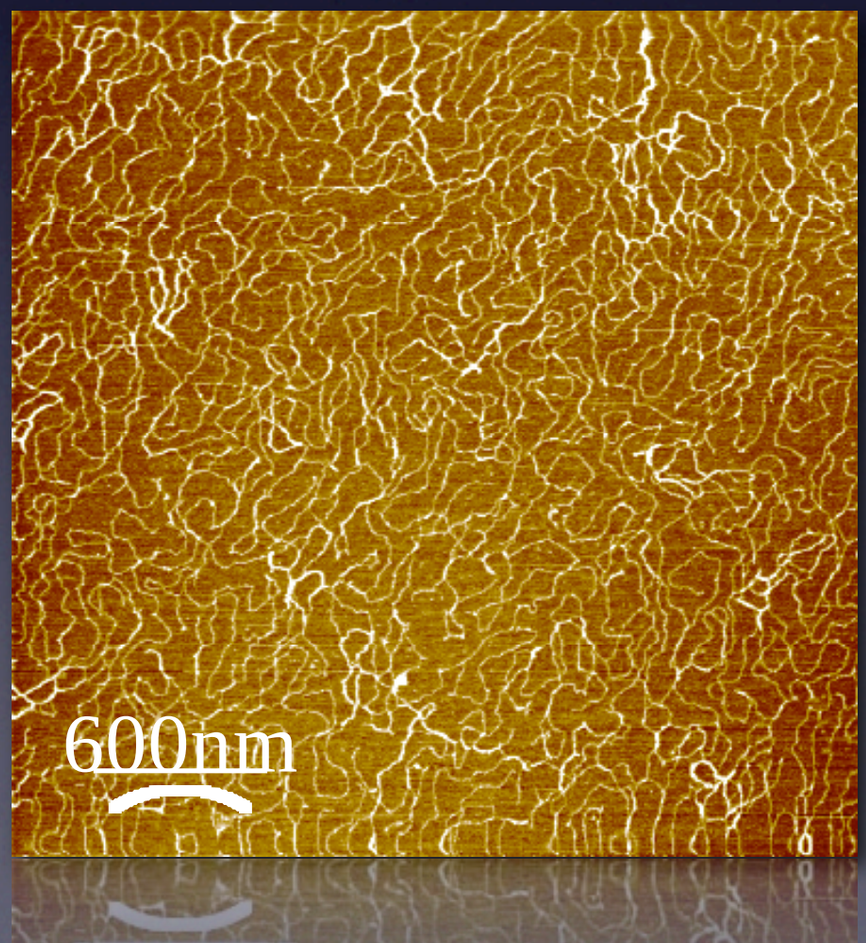


High-speed Transverse Dynamic Force Microscope (TDFM)

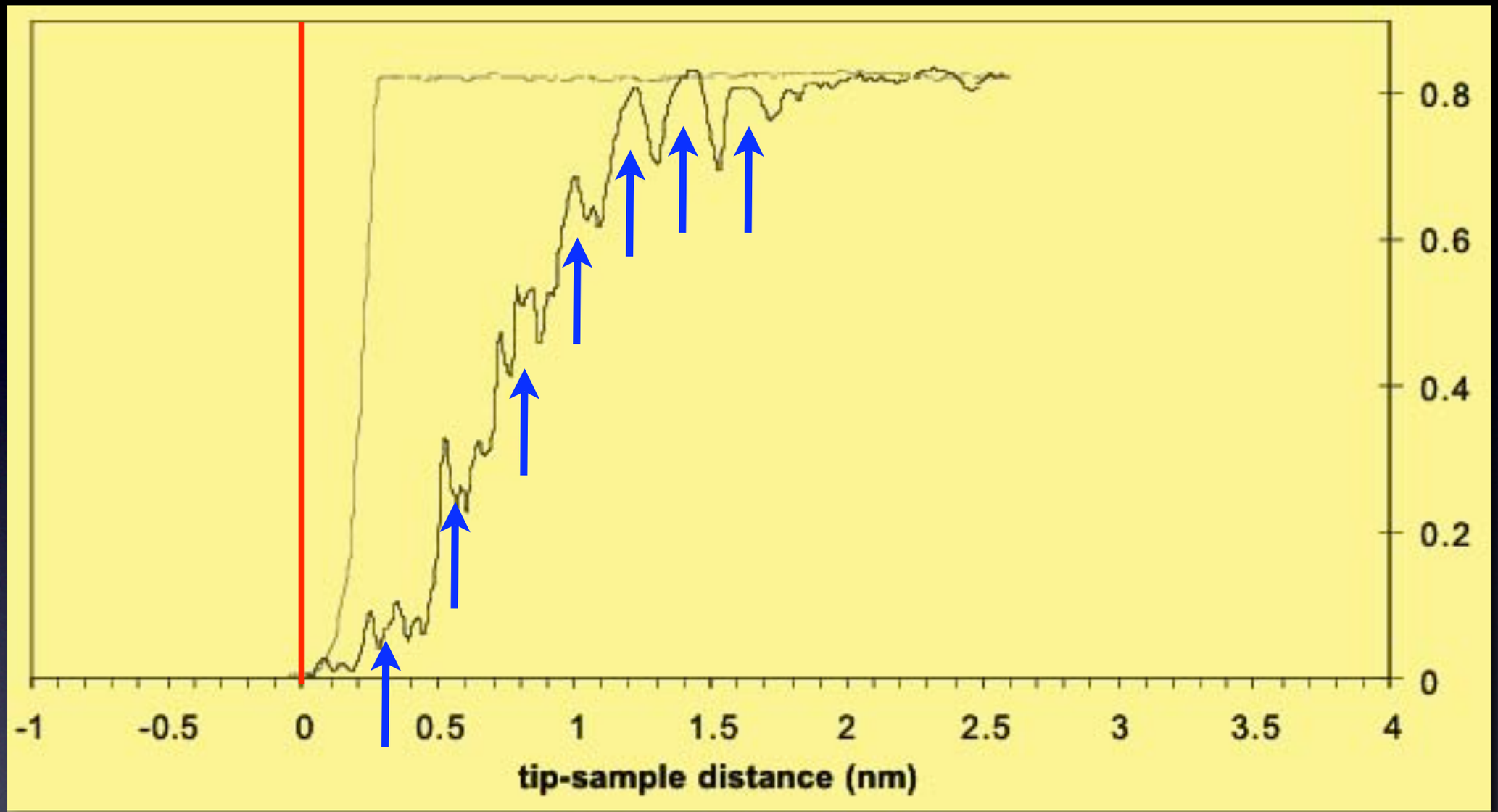
Conventional Transverse Dynamic Force Microscopy



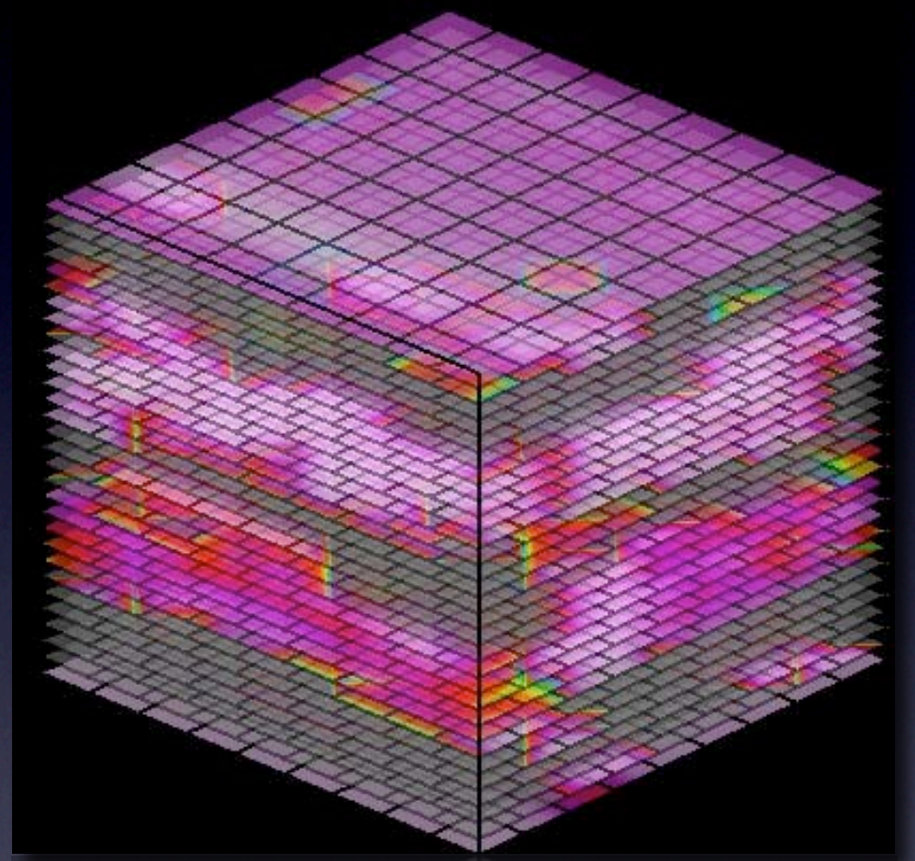




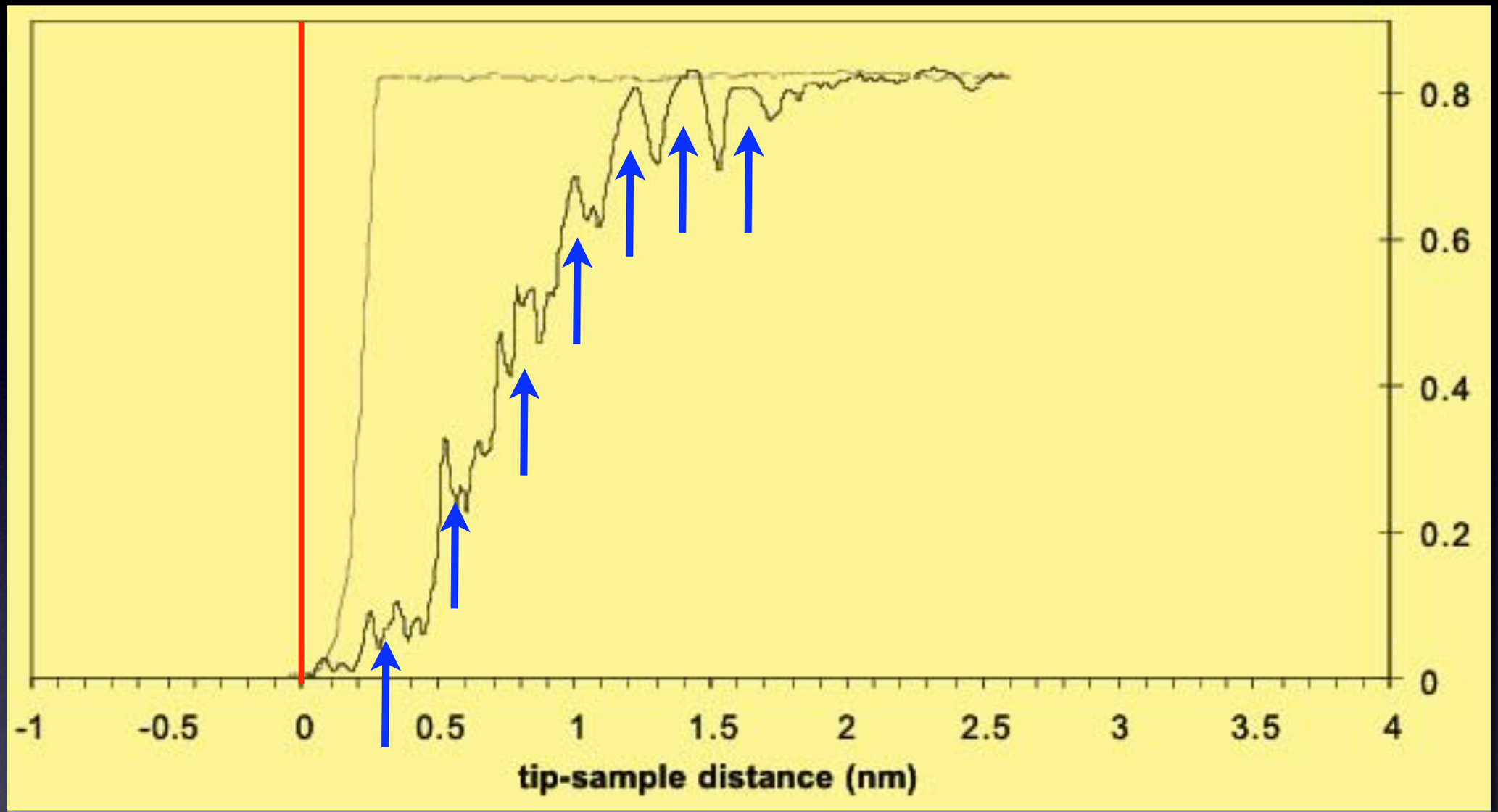
TDFM approach curve - water layers



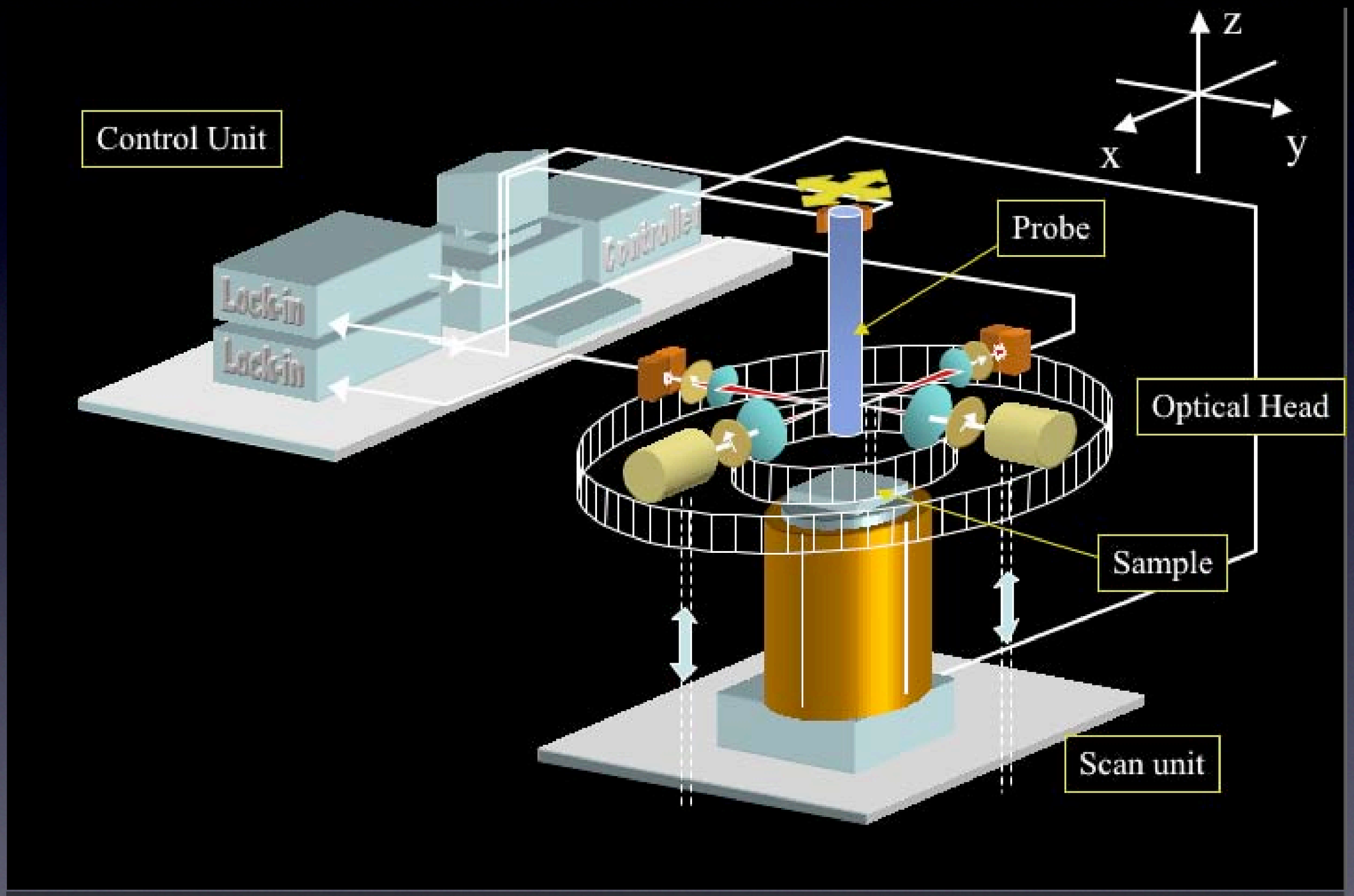
3D Mapping of Water Layers above a Surface



TDFM approach curve - water layers



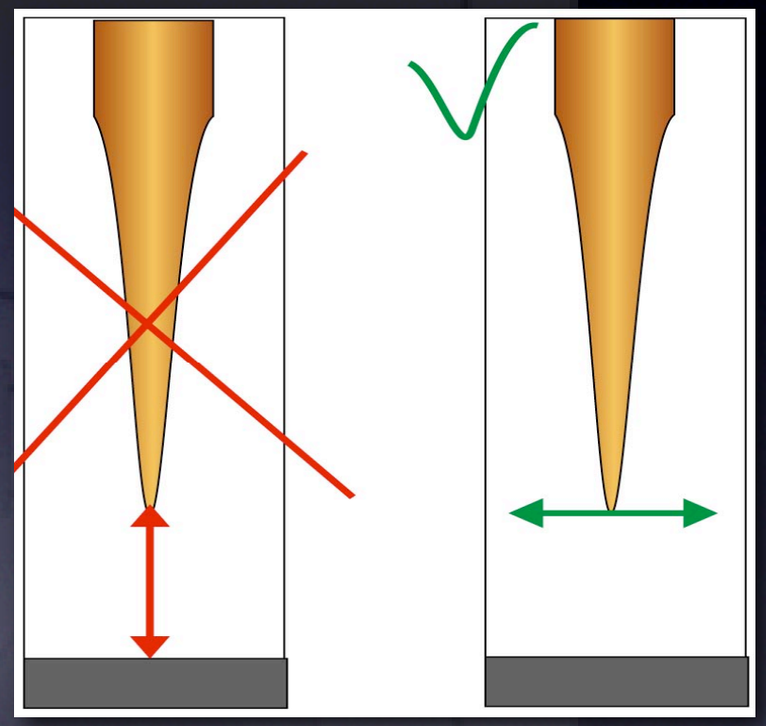
Conventional TDFM



Conventional TDFM

Typically, the probe will be a piece of glass fibre, pulled to produce a tapered tip of radius ~ 50 to 100 nm

The probe is mounted perpendicularly to the sample and oscillated parallel to the surface at its fundamental frequency



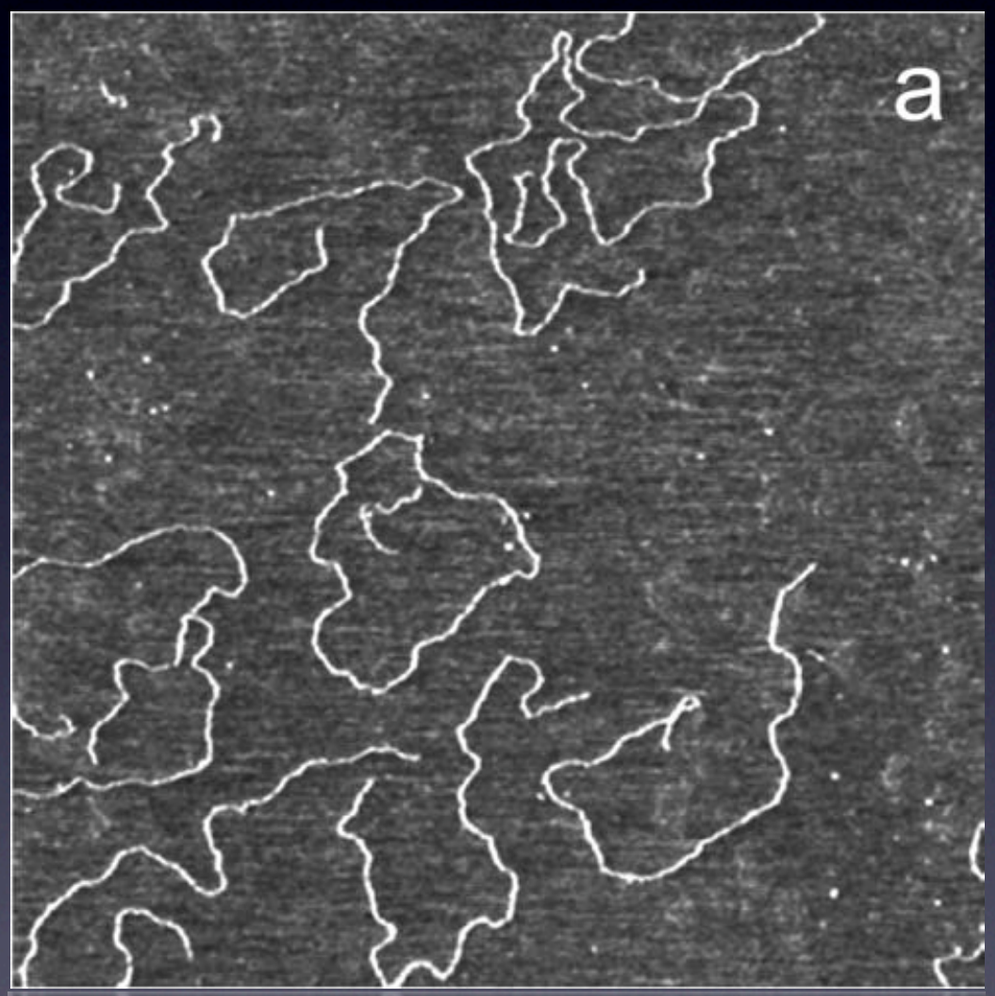
In order to achieve a high force sensitivity, the fibre will be several mm long, resulting in a low resonant frequency of ~ 5 – 15 kHz

At small probe-sample separation distances the dynamic response of the probe is changed by a shear force interaction with the surface

Ordering of the water layers confined between the probe and the sample is responsible for the shear force contrast mechanism

TDFM – A low damage, non-contact technique

2 µm x 2 µm image of 4331 bp DNA fragments deposited onto mica. Image taken in air with TDFM [4].



TDFM minimises probe-sample interaction forces, resulting in low sample distortion compared to Tapping Mode or Contact Mode AFM [4]

Measured height of DNA by TDFM: 1.1 ± 0.2 nm
Measured height of DNA by TM-AFM: 0.6 ± 0.1 nm

The probe is very stiff in the direction perpendicular to the surface, this prevents jump-to-contact events

[4] M. Antognozzi *et al*., *Single Mol*. **3** (2002) 2-3, 105-110

High-speed Non-contact TDFM

Why is TDFM a good candidate for High-speed?

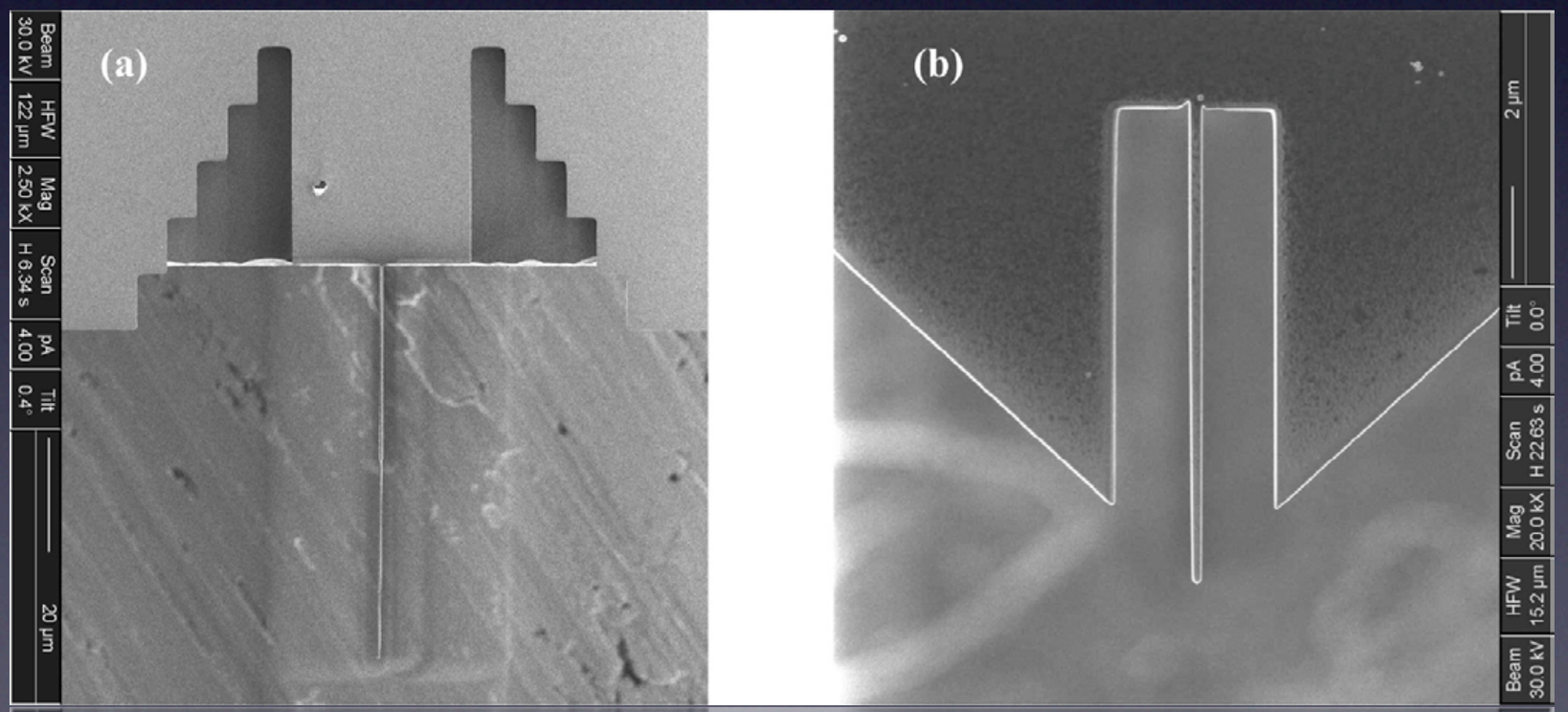
- ★ Small high-frequency probes are easy to make
- ★ TDFM is a non-contact force microscopy
- ★ High vertical rigidity of probe
- ★ Circular symmetry of probe - choice of oscillation
- ★ Probe carrier away from sample
- ★ Wide choice of materials for probes

Tips: etched; cut; FIB; CNT; etc. from silicon, glass, metal, polymer, nanowires, ...

Main Advantages of using TDFM for High Speed

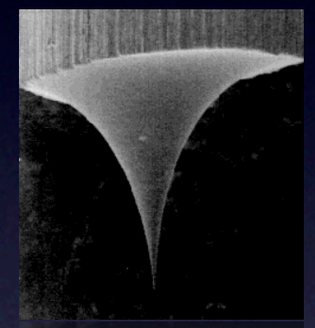
★Small high-frequency probes are easy to make

TM cantilever FIB'd to give a 1.8 MHz resonant frequency probe

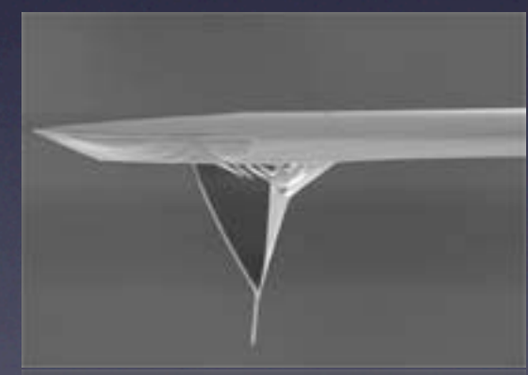


pulled glass probe

etched W tip



CNT tip



A New Detection Arrangement

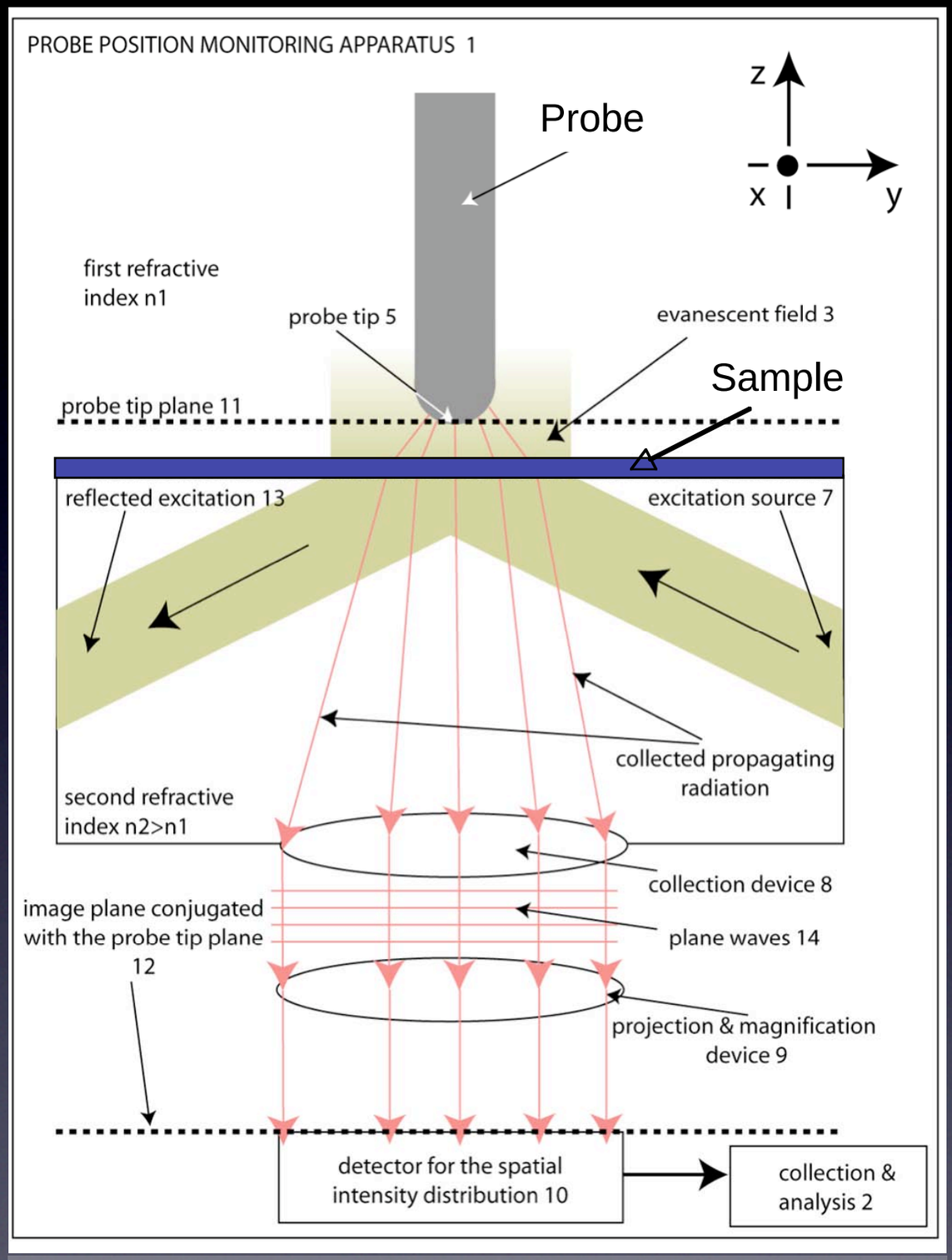
An evanescent wave is generated above the sample surface

Only the very end of the vertically mounted probe enters the evanescent wave and is illuminated

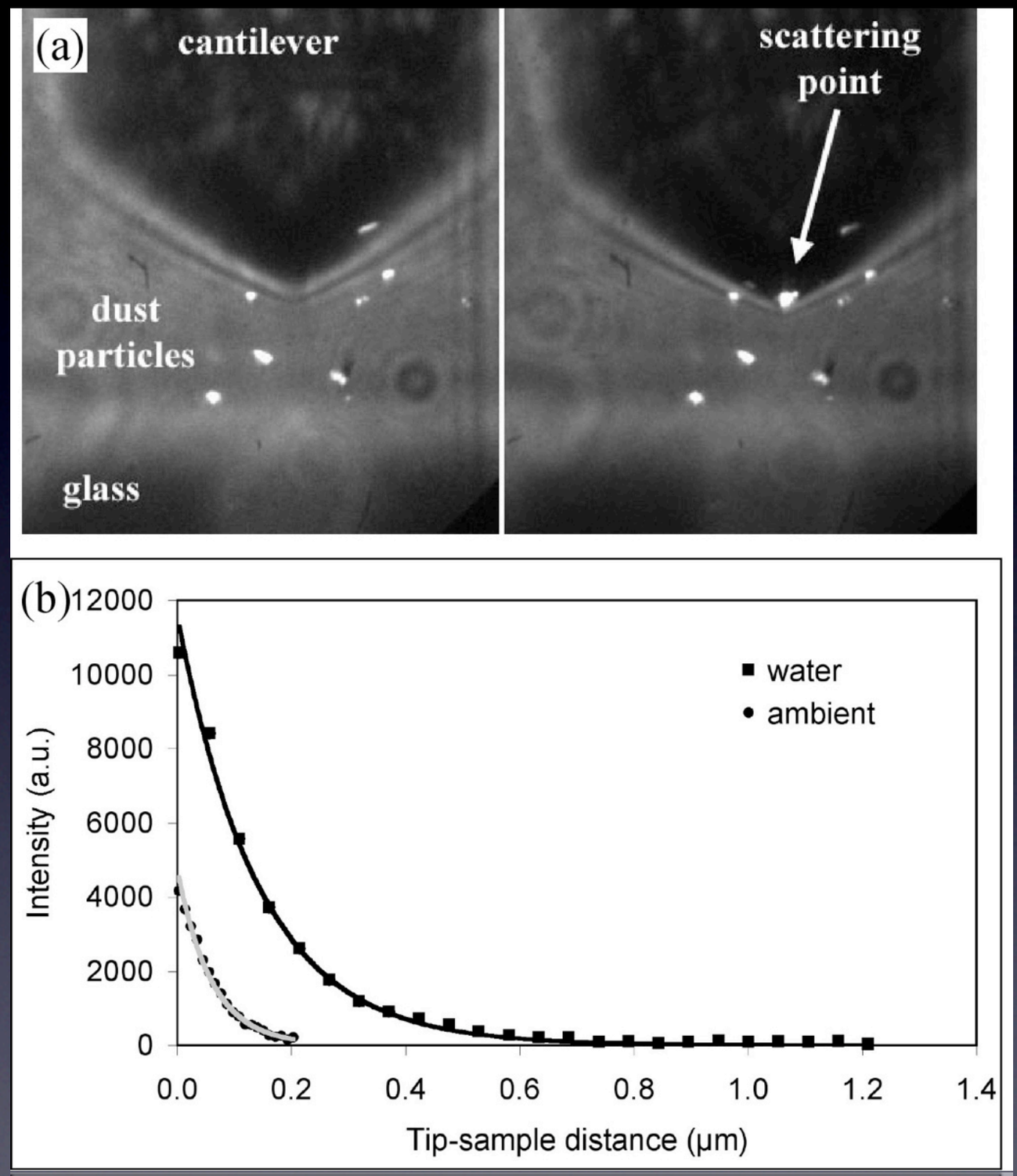
The light scattered by the tip of the probe is collected and directed to a multi-segment photodetector

The x and y position of the tip is then monitored directly

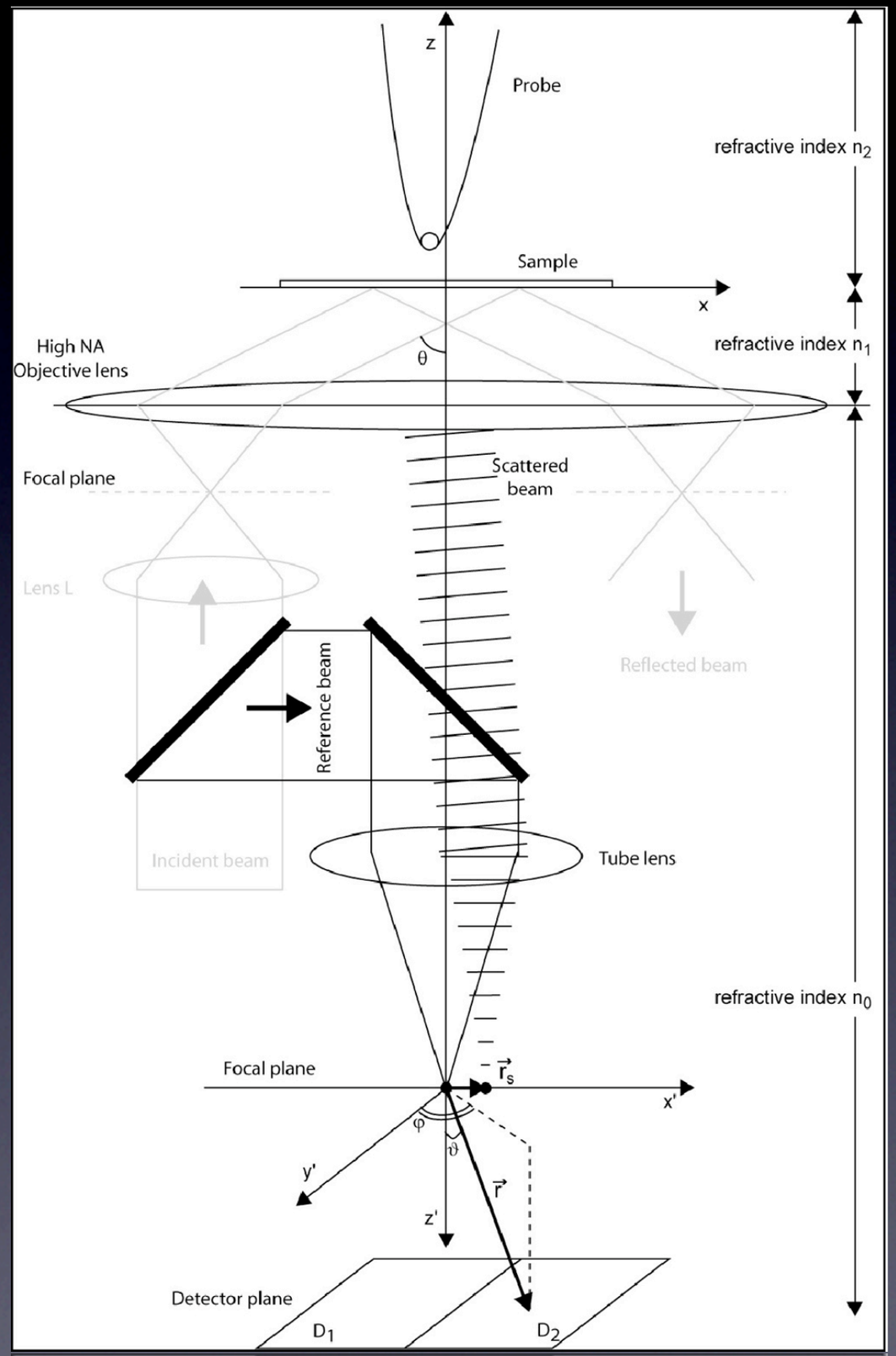
The evanescent field is present under both ambient conditions and in liquid environments



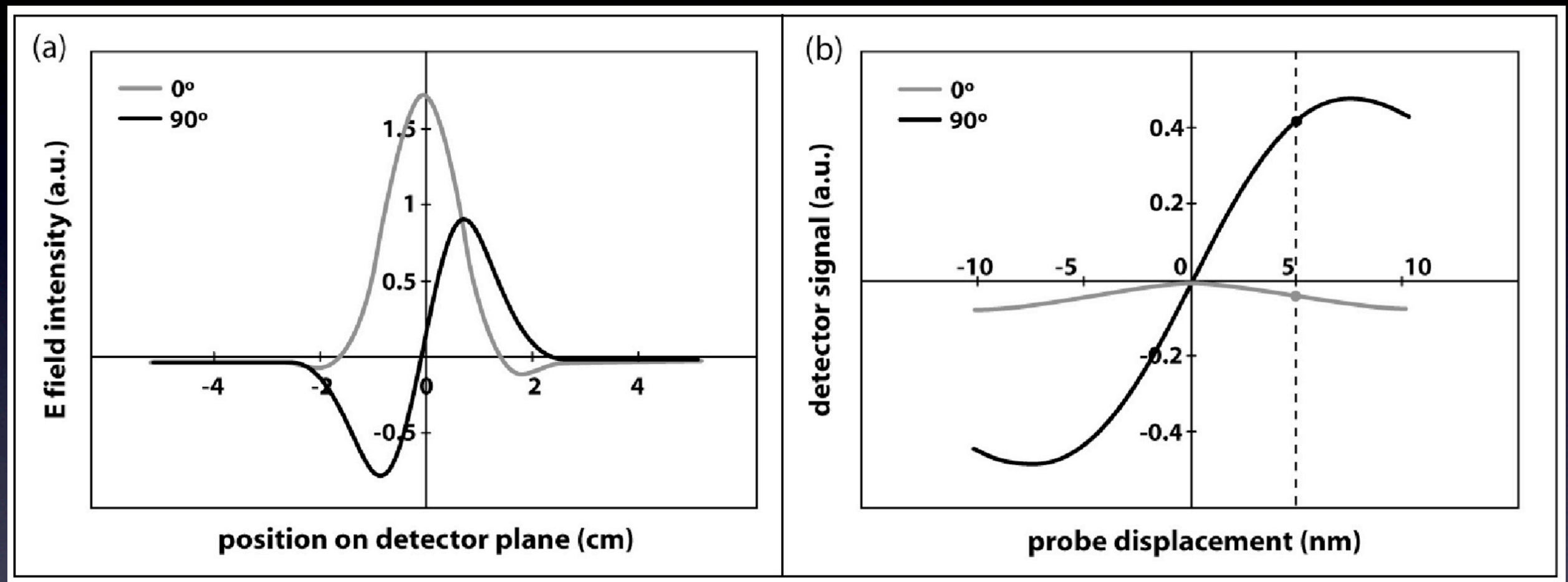
Evanescent Field - decay length



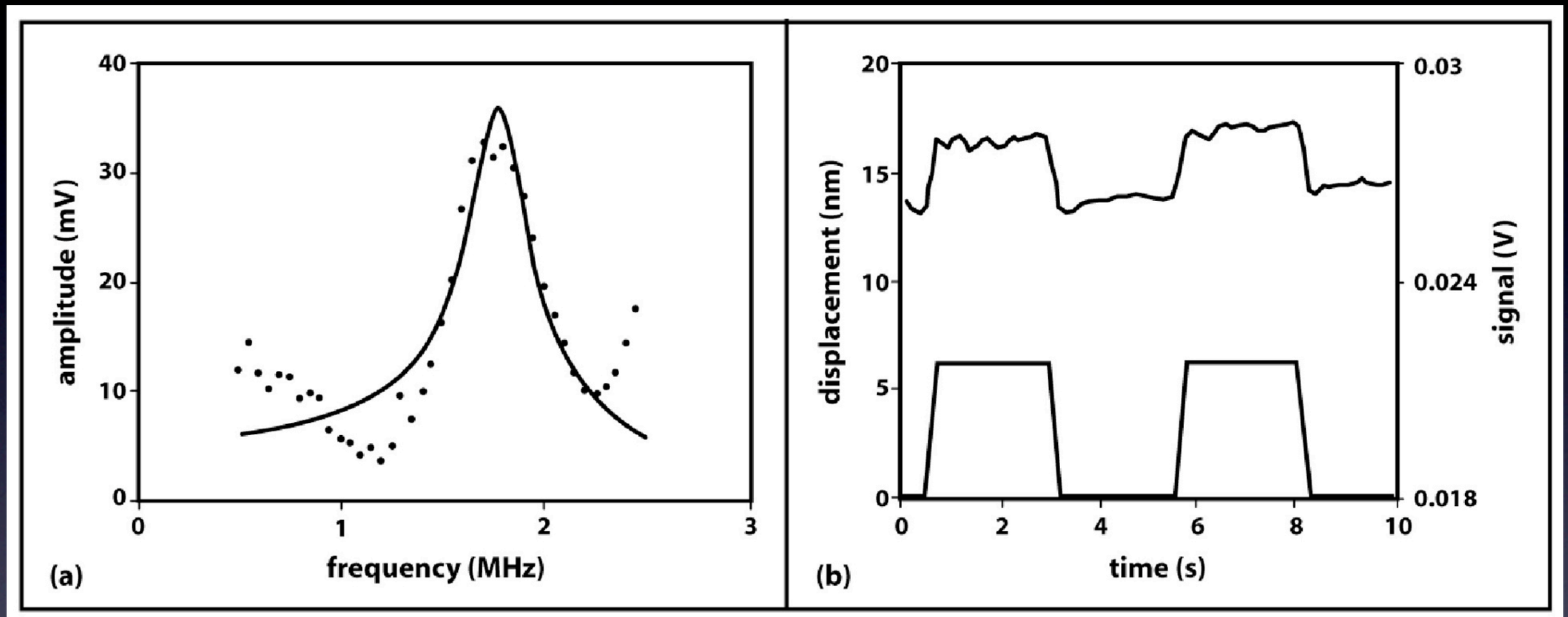
Interferometry - tip motion detection

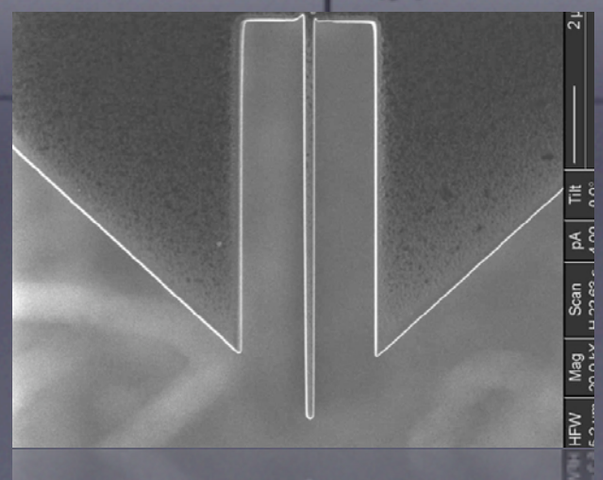


Sensitivity enhancement with interferometry



Response of 1.8 MHz probe

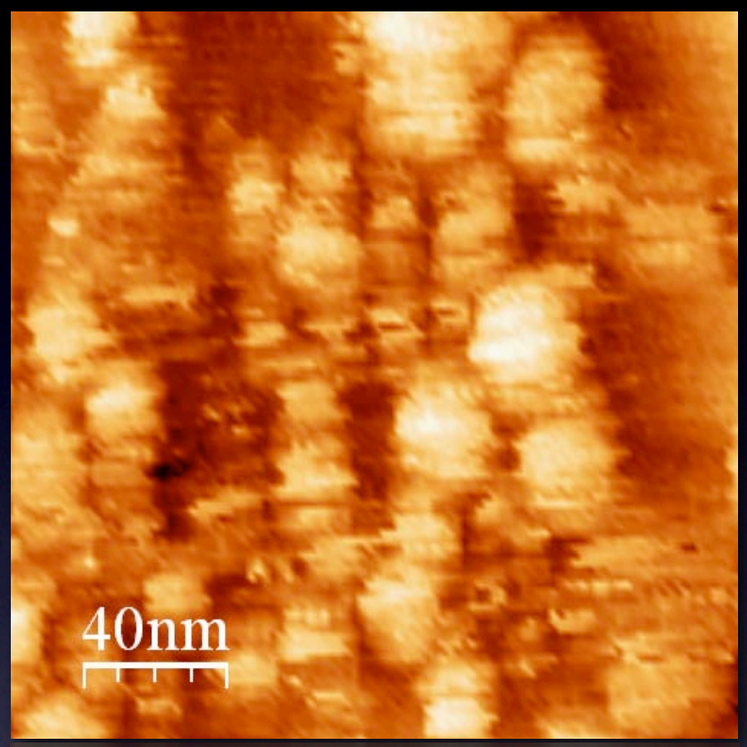




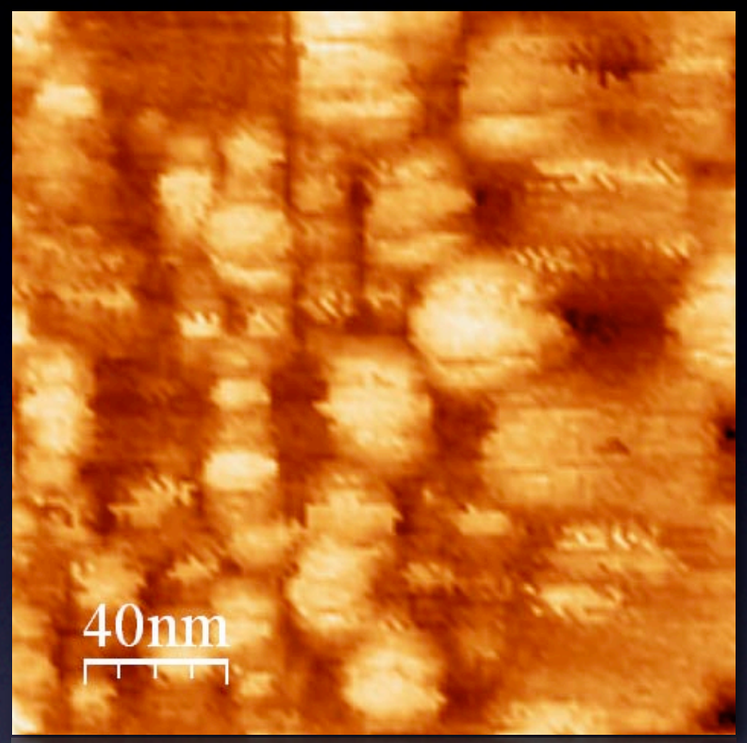
Imaging Rates

Scans performed on a 20 nm thick layer of gold, evaporated onto glass coverslips

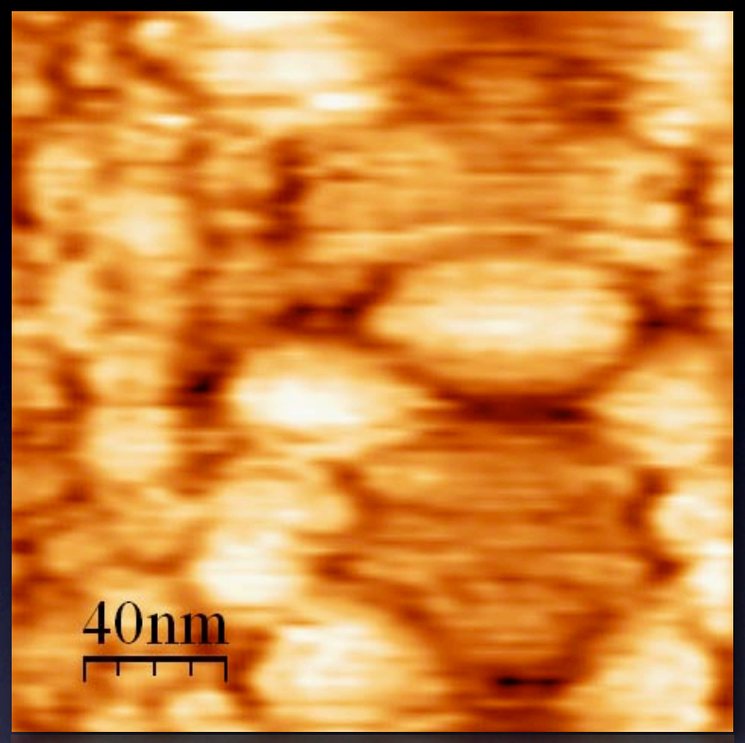
5 Hz



10 Hz



20 Hz

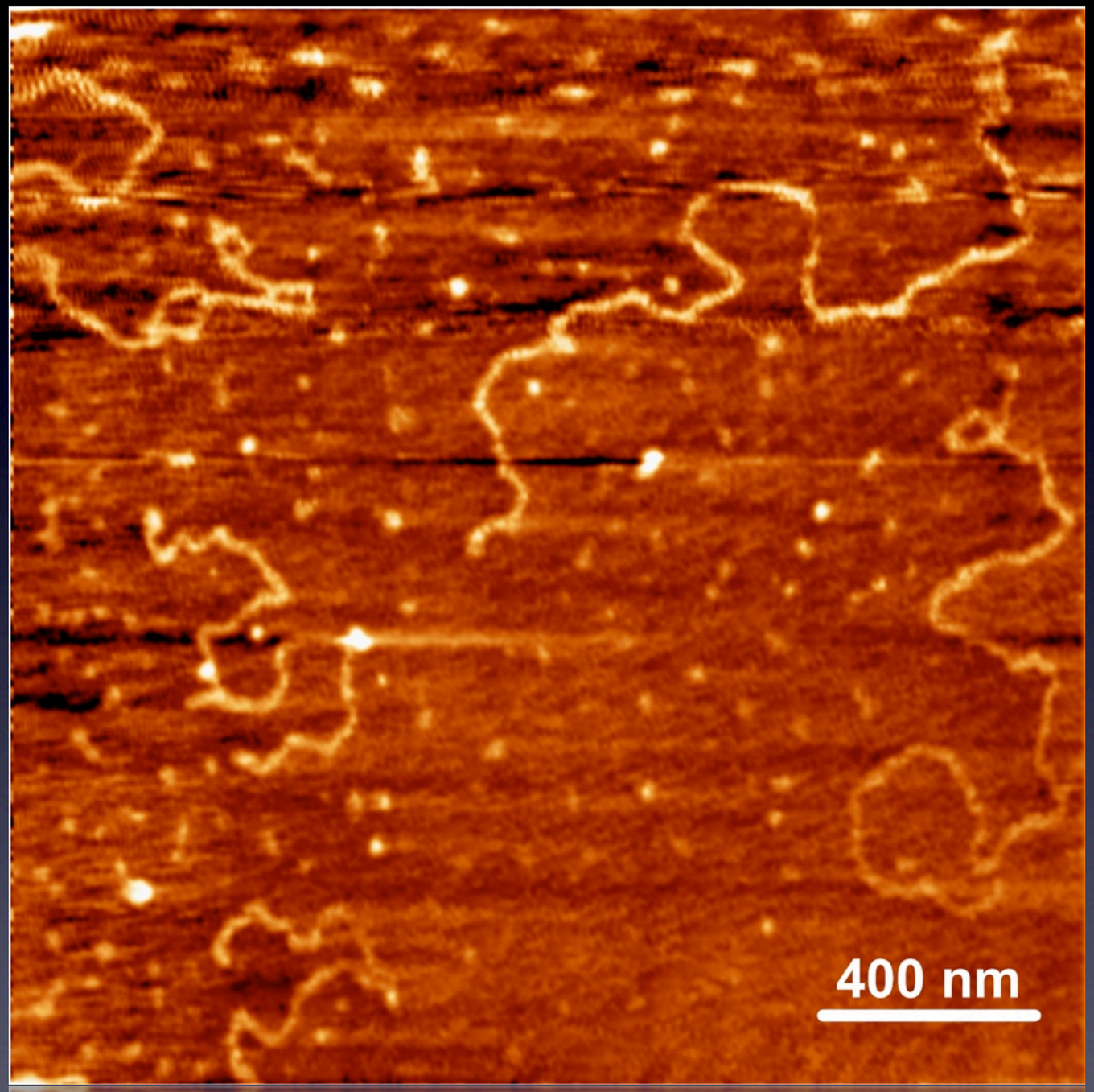


The response of the scan stage is seen to be substantially distorted by a 20 Hz fast scan signal

The tip tracks the surface without noticeable loss of resolution

245 x 256 pixels at 20 lines/second → 100 x 100 pixels gives 2 secs per image

Image of -DNA molecules deposited onto mica and observed in a 5 mM $NiCl_2$ solution.



Summary

- High-speed ‘contact-mode’ AFM - most useful for:
  - large areas & high specimens
  - very high speeds
- Non-contact TDFM - proof of concept:
  - single molecule imaging
  - low forces
  - water structure
  - easy probe fabrication from a wide range of materials

and all members of the Nanophysics Group

Dr. Loren Picco

Dr. Arturas Ulcinas

Dr. Massimo Antognozzi

Dr. Peter Duncon

Dave Engledew

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

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