

High-speed AFM and its Applications

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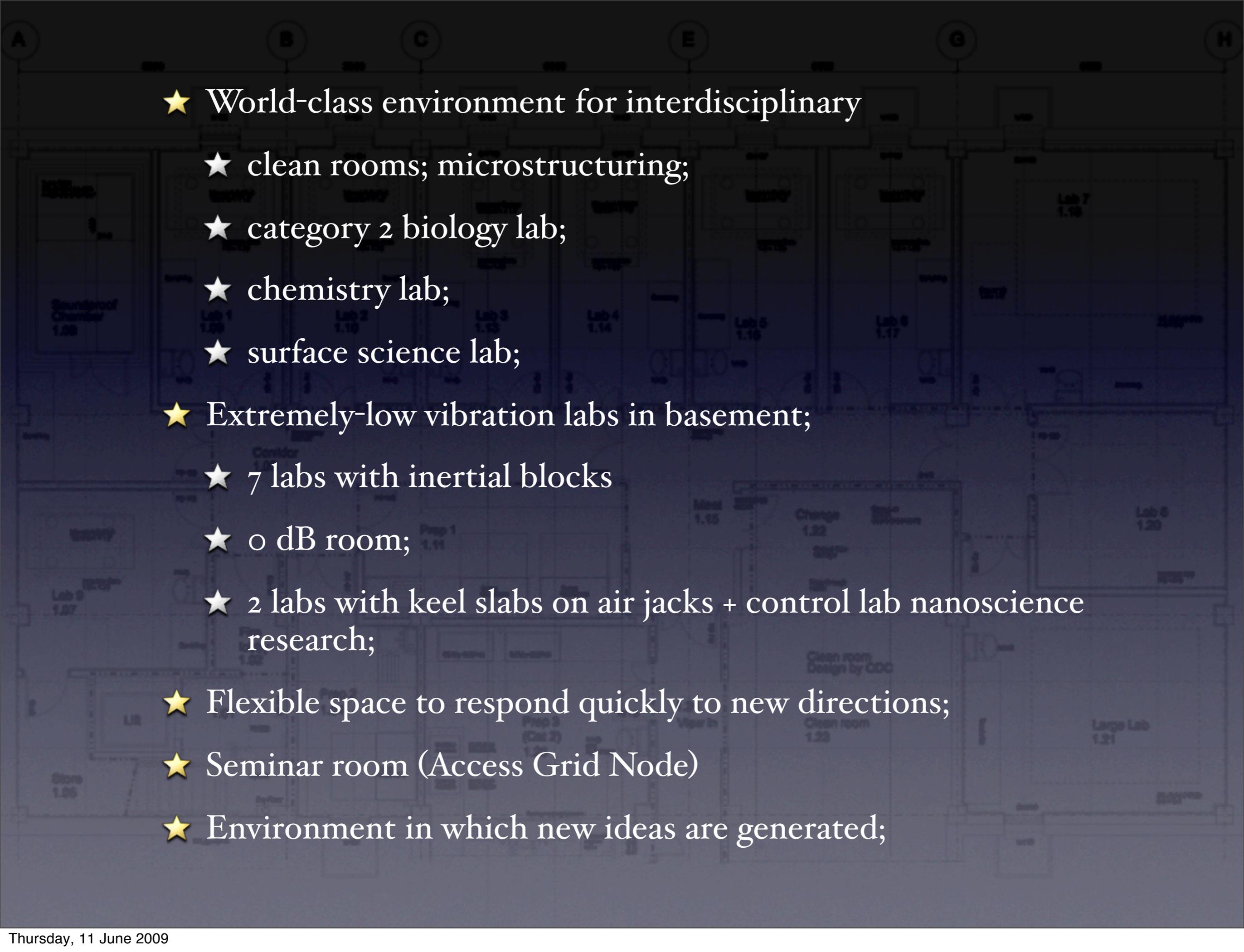










- 
- ★ World-class environment for interdisciplinary
 - ★ clean rooms; microstructuring;
 - ★ category 2 biology lab;
 - ★ chemistry lab;
 - ★ surface science lab;
 - ★ Extremely-low vibration labs in basement;
 - ★ 7 labs with inertial blocks
 - ★ 0 dB room;
 - ★ 2 labs with keel slabs on air jacks + control lab nanoscience research;
 - ★ Flexible space to respond quickly to new directions;
 - ★ Seminar room (Access Grid Node)
 - ★ Environment in which new ideas are generated;

Content

Content

High-speed Force Microscopy

Content

High-speed Force Microscopy

High-speed AFM :

Content

High-speed Force Microscopy

High-speed AFM : contact mode

Content

High-speed Force Microscopy

High-speed AFM : contact mode +

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic
force microscopy :

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic
force microscopy : non-contact
mode

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic force microscopy : non-contact mode +

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic force microscopy : non-contact mode + molecular water layers

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic force microscopy : non-contact mode + molecular water layers

Writing at high-speed :

Content

High-speed Force Microscopy

High-speed AFM : contact mode + superlubrication

Transverse dynamic force microscopy : non-contact mode + molecular water layers

Writing at high-speed : silicon oxidation

EPSRC Nano Equipment Access

High-speed Atomic Force Microscopy:

Why go fast?

Go fast ...

- ★to follow many processes;
- ★to examine large areas of a specimen;
- ★to create or manipulate structures over usefully large areas.

Why not go fast?

AFM is a Mechanical Microscope

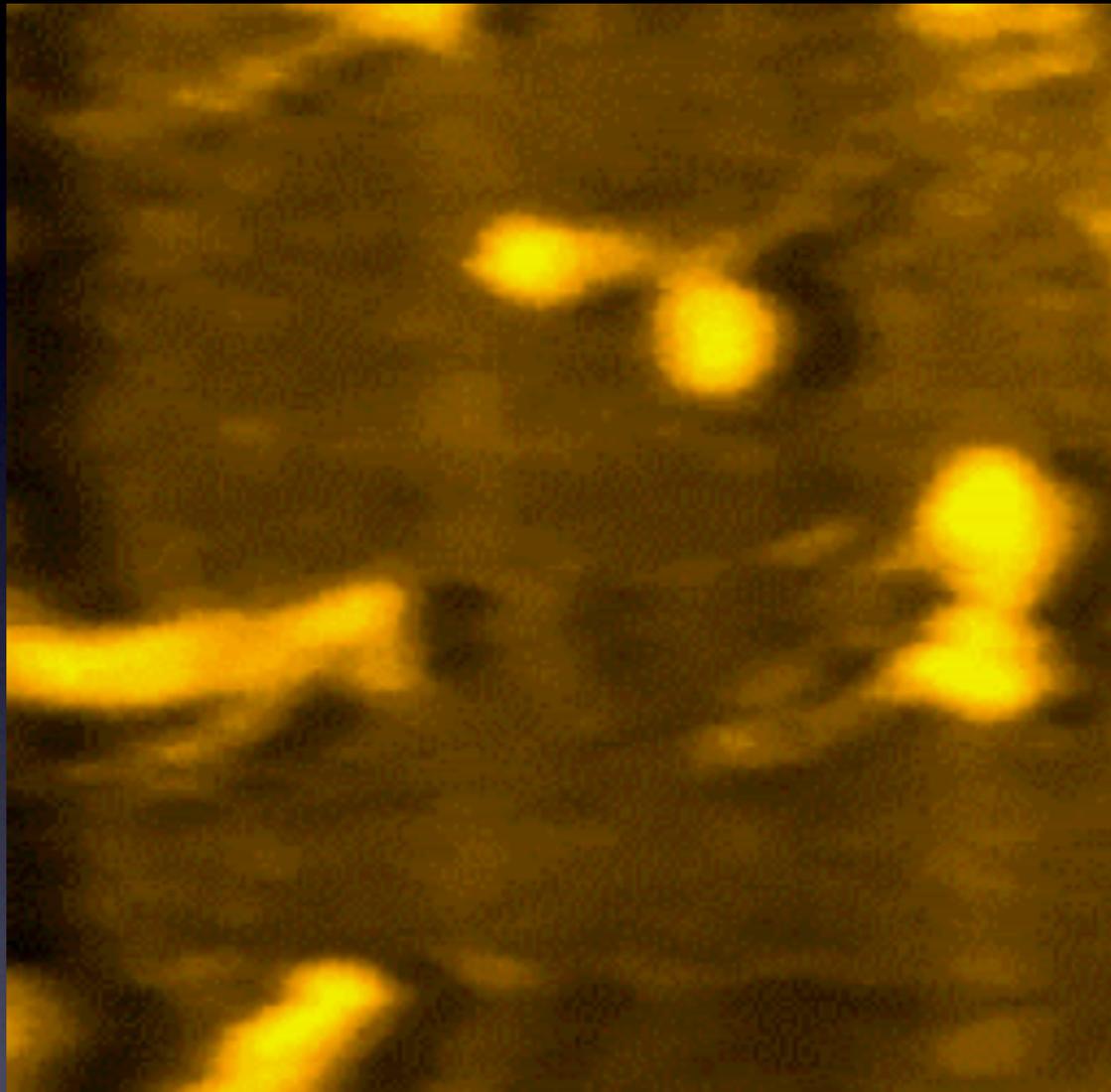
- ★ Inertia - response times
 - ★ scan stage and z feedback
 - ★ AFM cantilever response
- ★ Resonance - positioning
 - ★ scan stage
 - ★ cantilever
 - ★ SNOM probe
- ★ Electronic/software feedback response

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 AFM by operating at higher frequencies

Myosin V



Miniature cantilever



Toshio ANDO (Kanazawa University, Japan)

A Different Solution

A Different Solution

Resonant scan stages

A Different Solution

Resonant scan stages

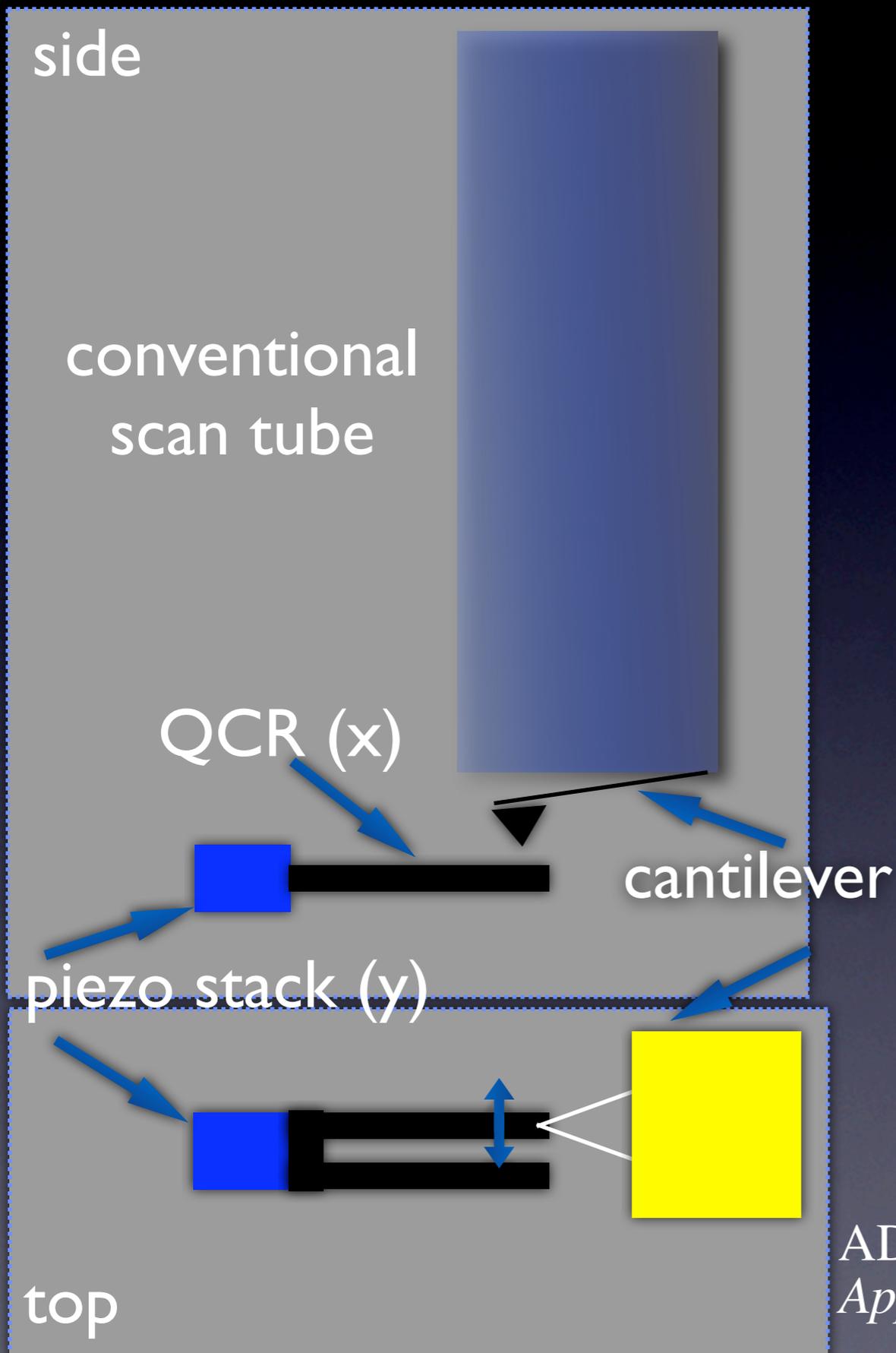
Superlubricity 'contact-mode' AFM

A Different Solution

Resonant scan stages

Superlubricity 'contact-mode' AFM

Resonant scanning AFM



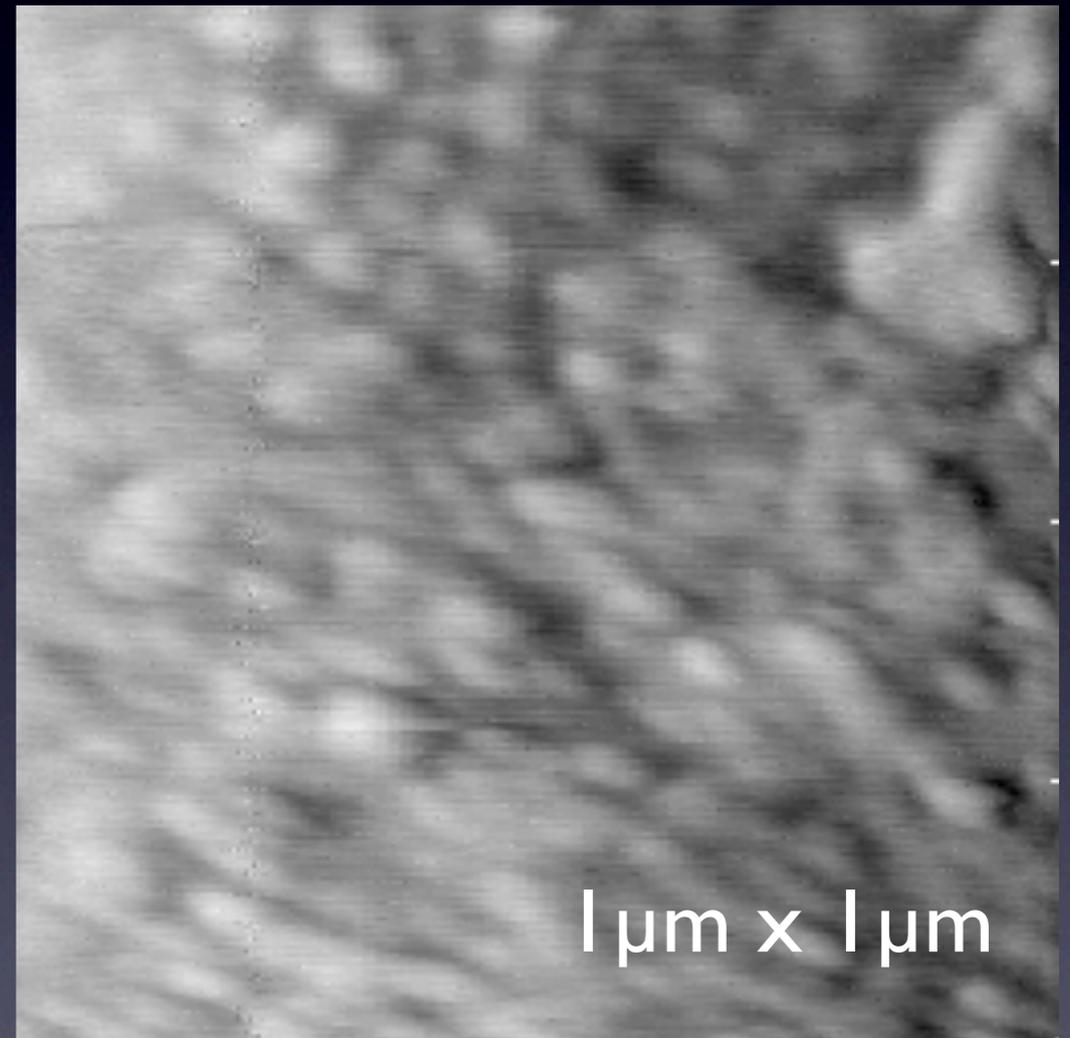
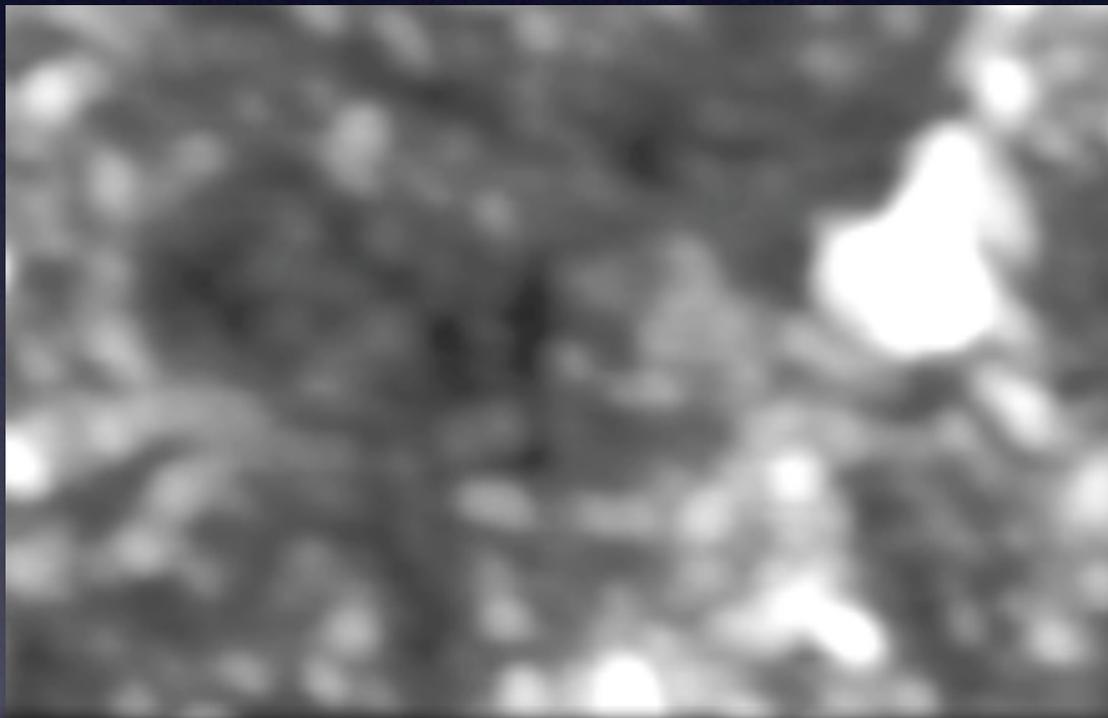
- ★ Resonant sample scan stage (x)
- ★ Piezo stack for 'slow' scan (y)
- ★ No electronic feedback
- ★ Mechanical feedback
 - ★ super lubricity
 - ★ water in confined geometry
- ★ Scanned tip mode also possible
- ★ Extra down force (electrostatic)

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

Conventional AFM



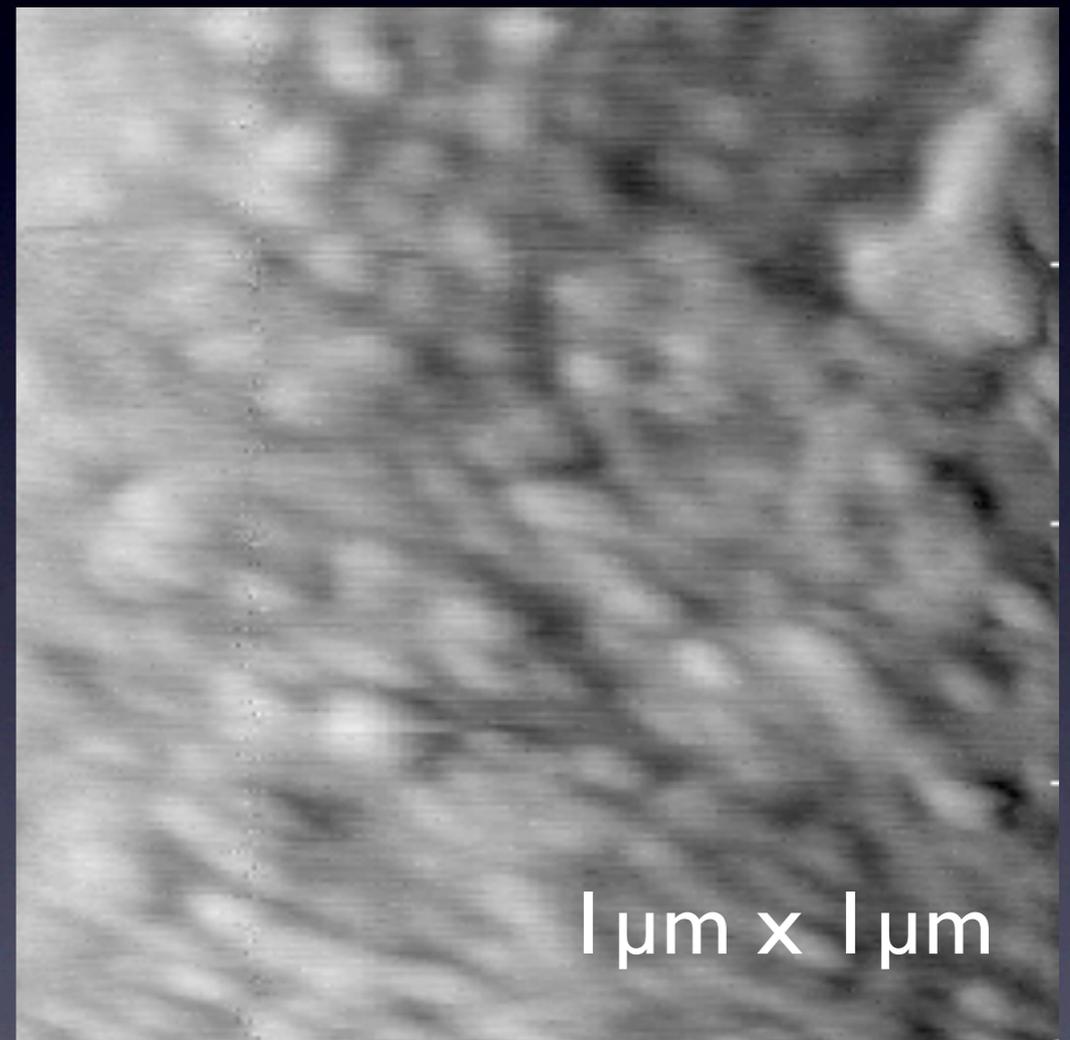
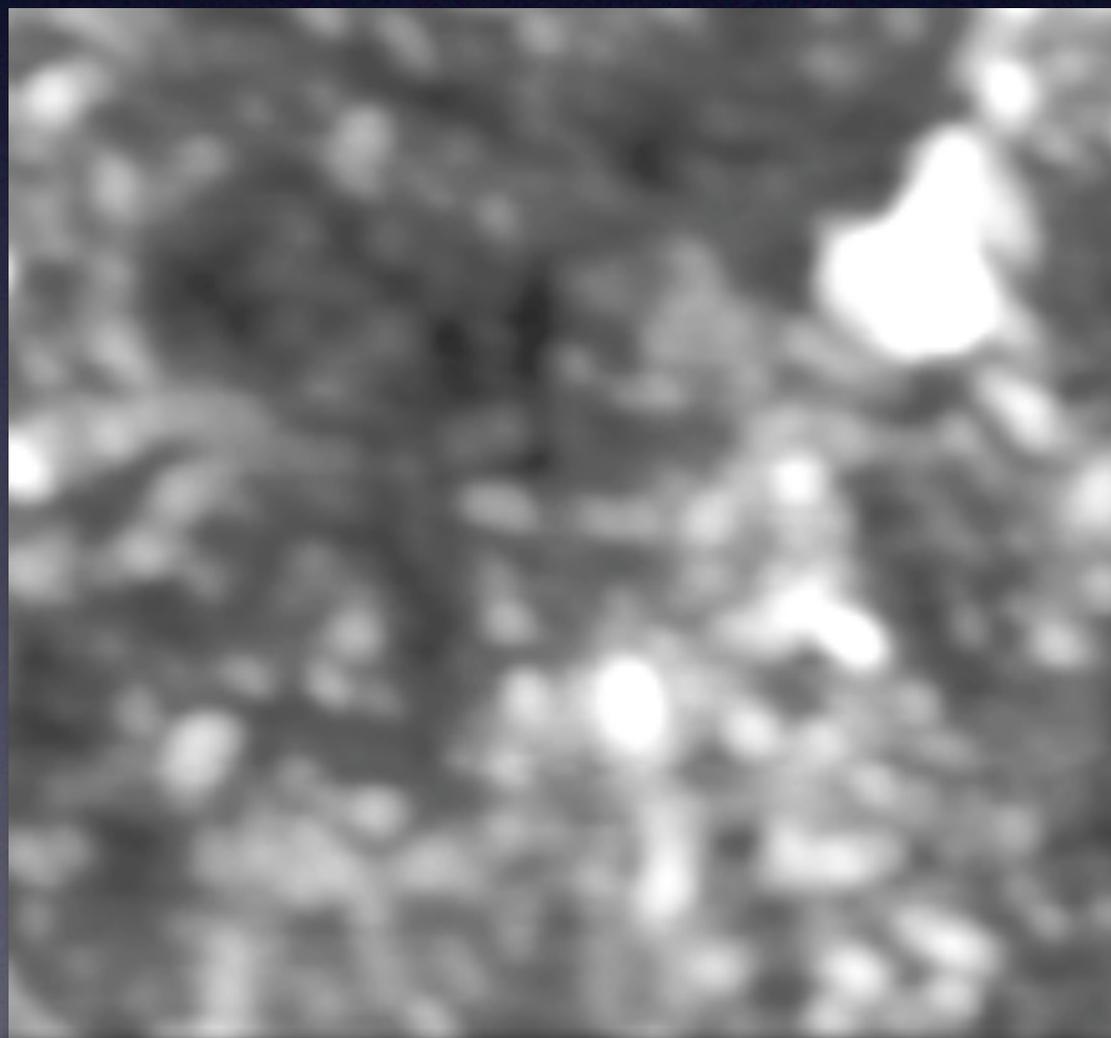
30 frames/s
(Infinitesima vAFM)

Ulcinas, Payne, Heppenstall-Butler

High-speed AFM of Chitosan Film

- tuning-fork resonance scanning

Conventional AFM



30 frames/s
(Infinitesima vAFM)

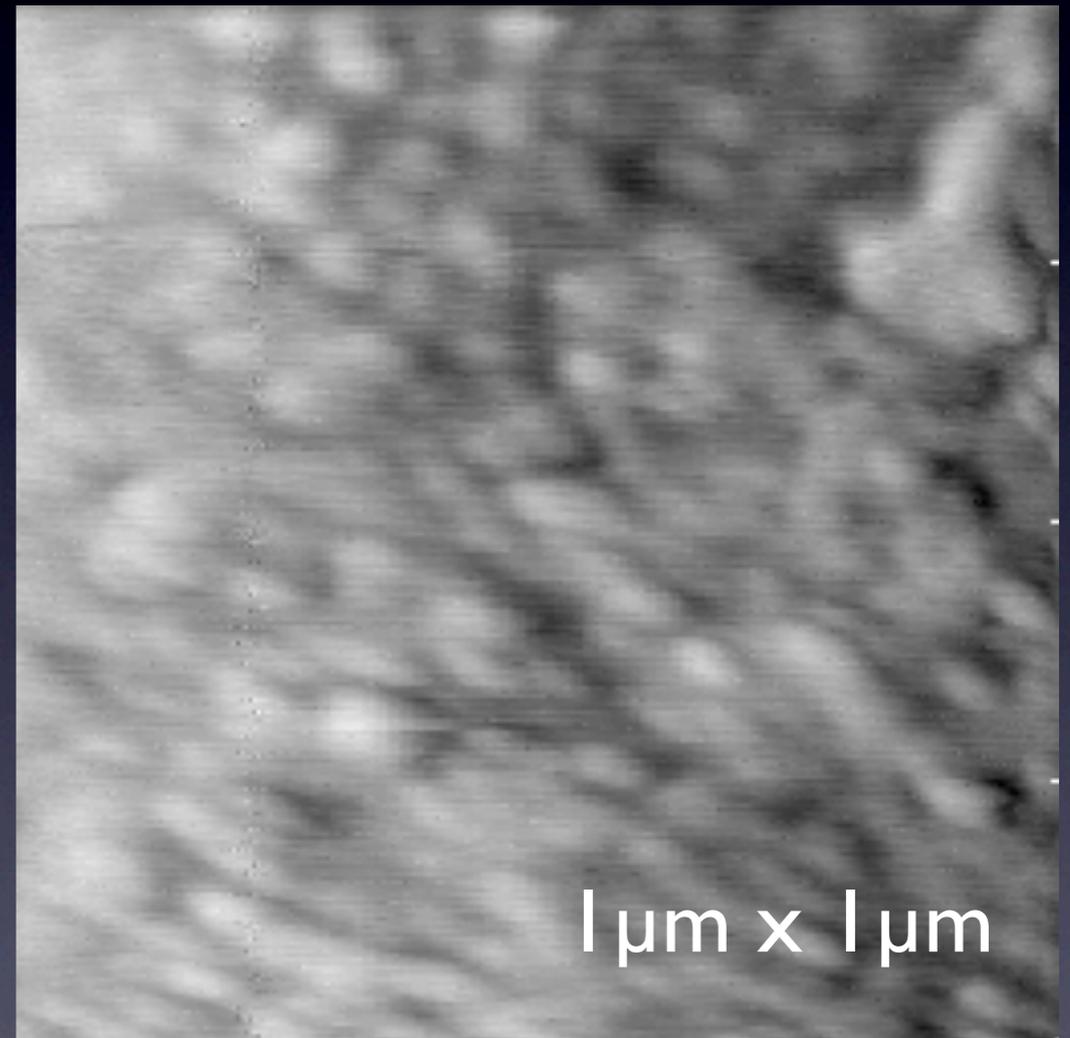
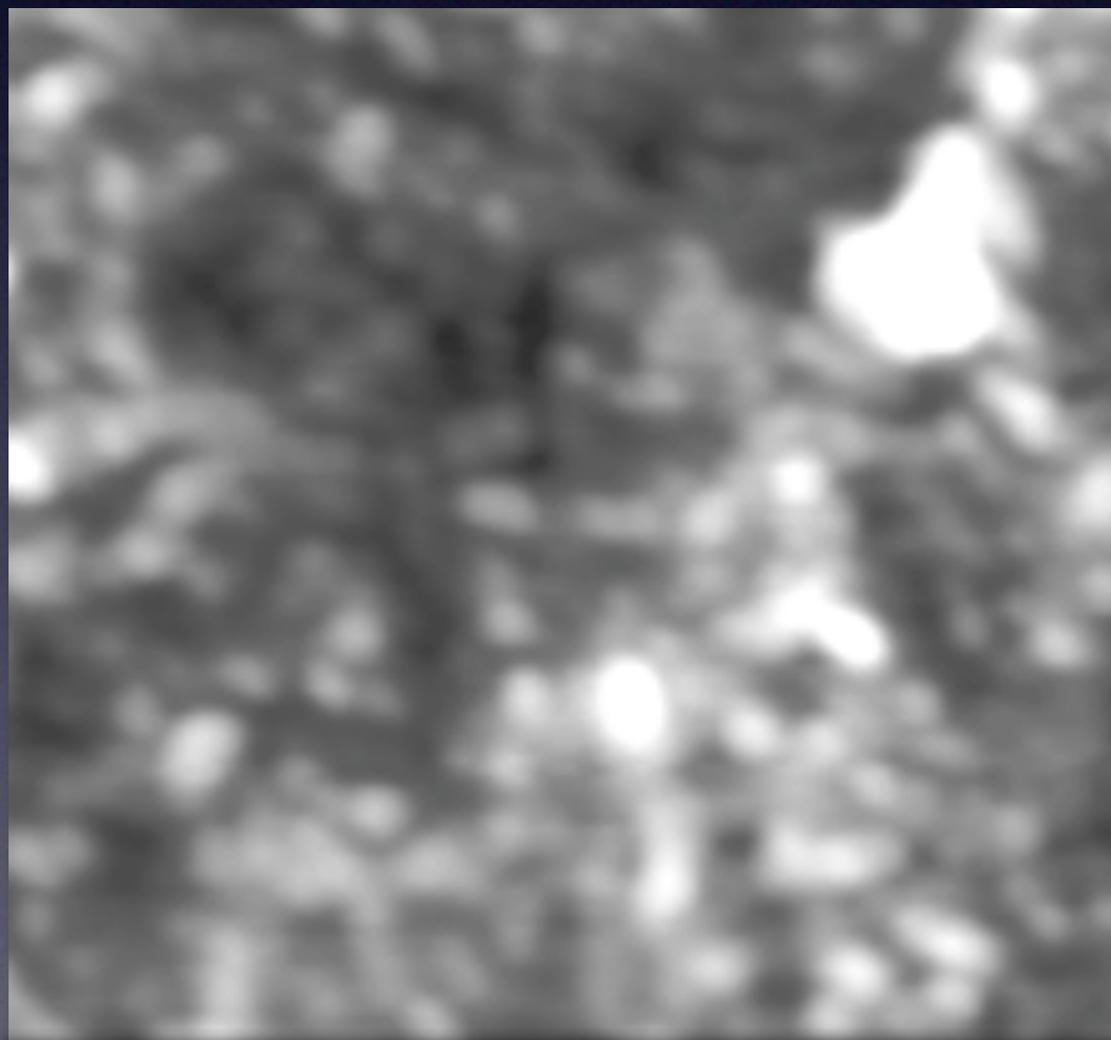
Ulcinas, Payne, Heppenstall-Butler

High-speed AFM of Chitosan Film

- tuning-fork resonance scanning

> 3000 Faster than conventional AFM

Conventional AFM



30 frames/s
(Infinitesima vAFM)

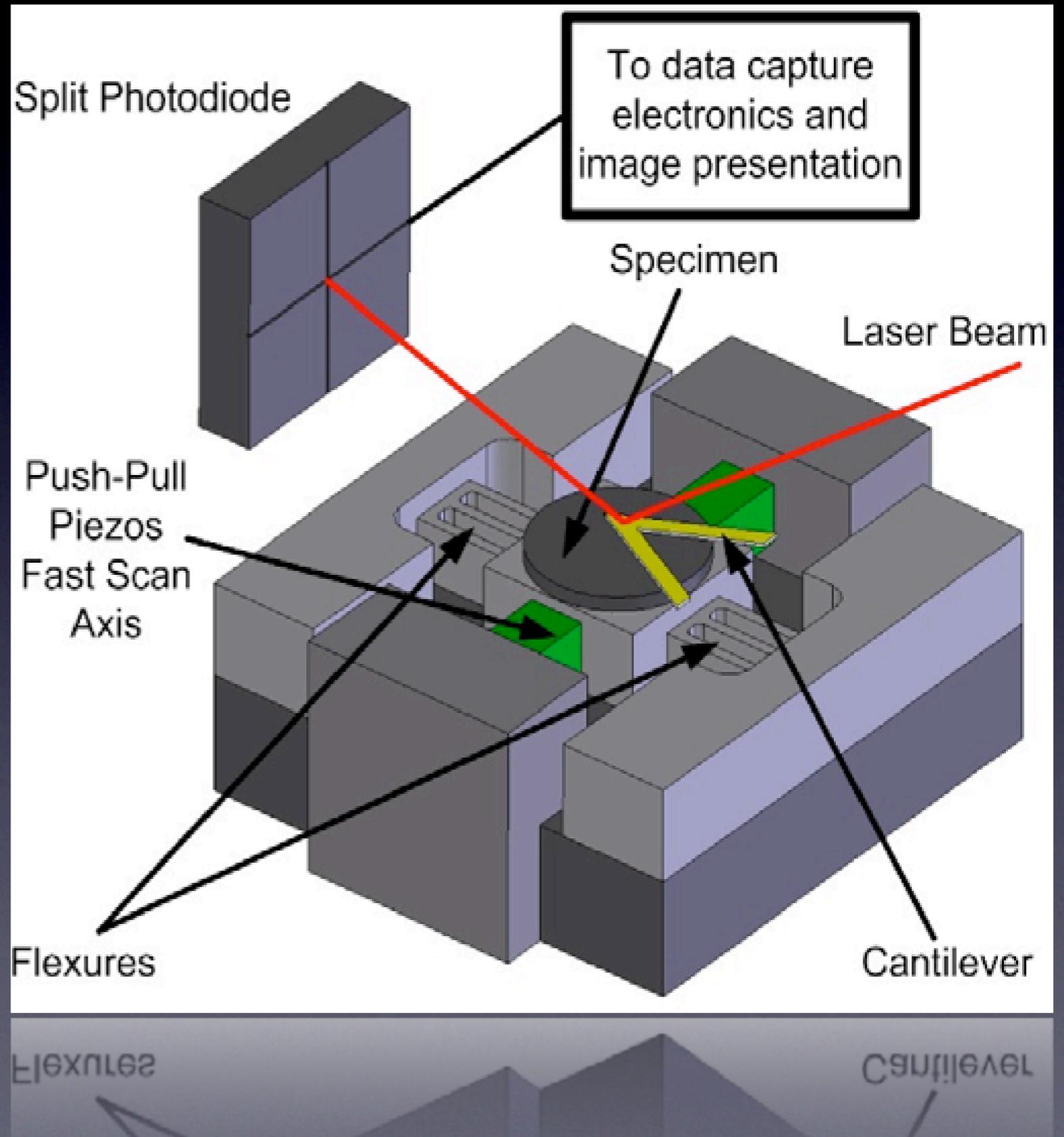
Ulcinas, Payne, Heppenstall-Butler

High-speed flexure stage and cantilever set-up

High speed
Contact Mode

High-speed flexure stage and cantilever set-up

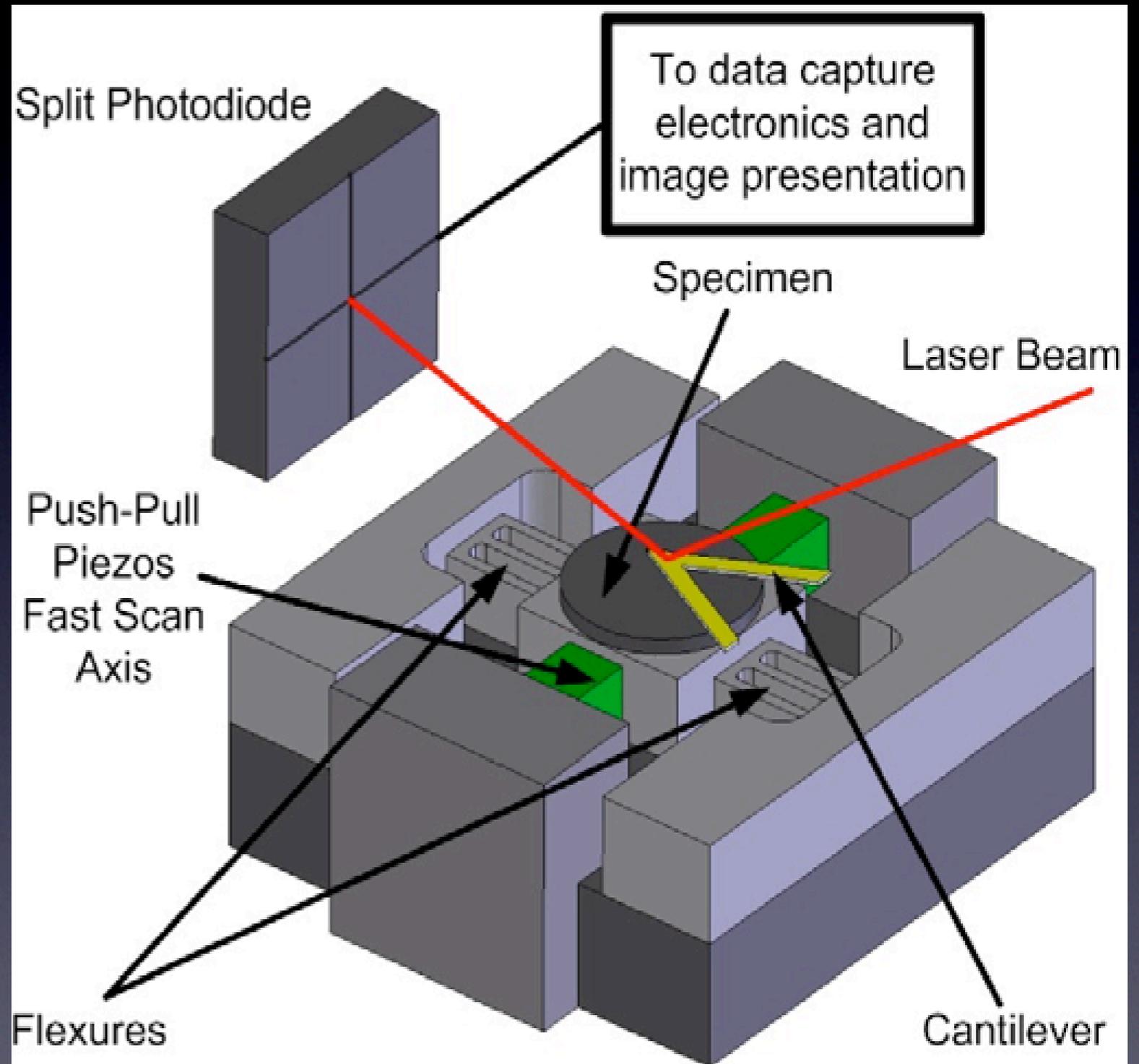
High speed Contact Mode



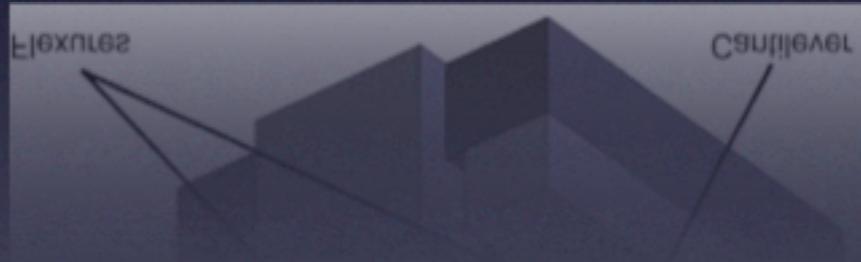
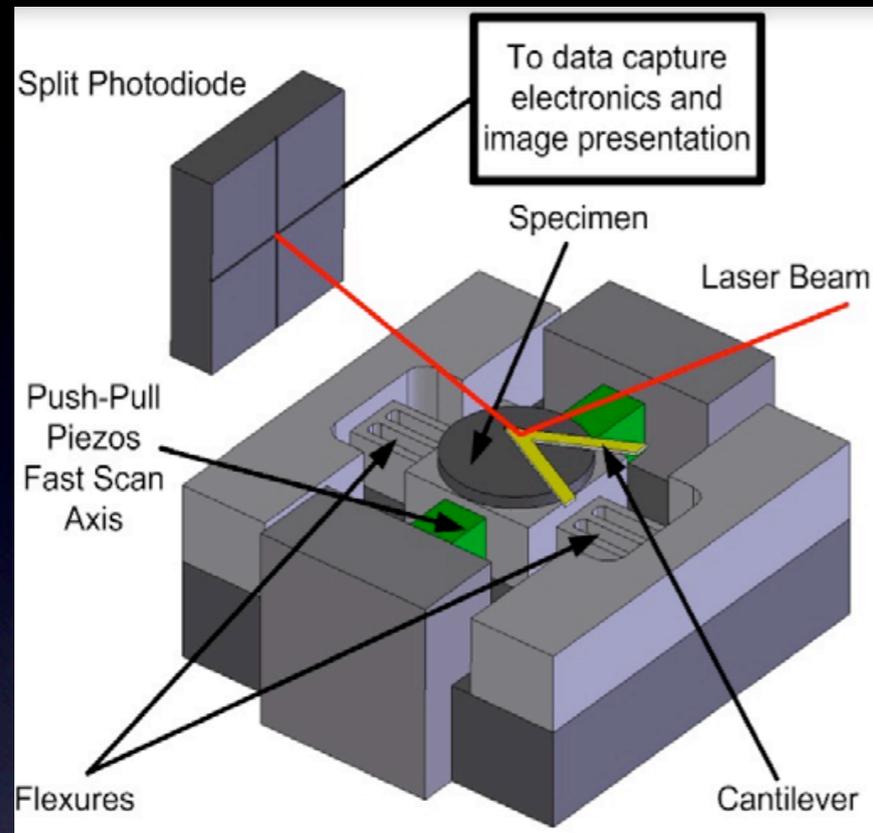
High-speed flexure stage and cantilever set-up

High speed Contact Mode

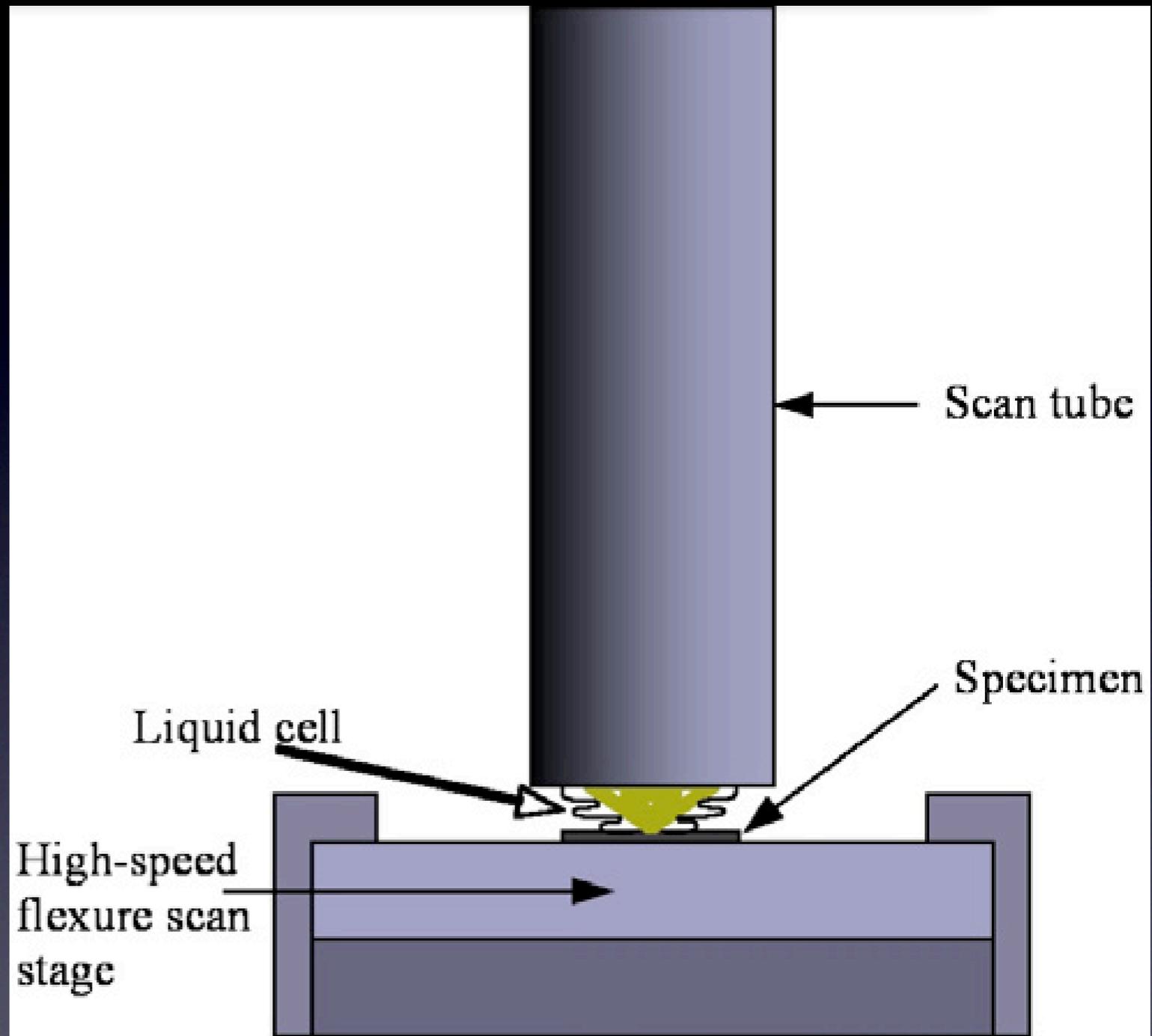
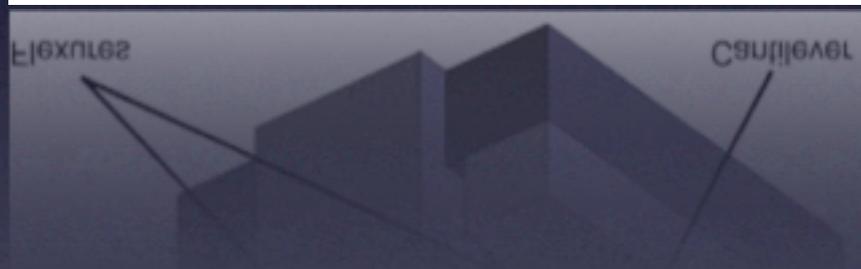
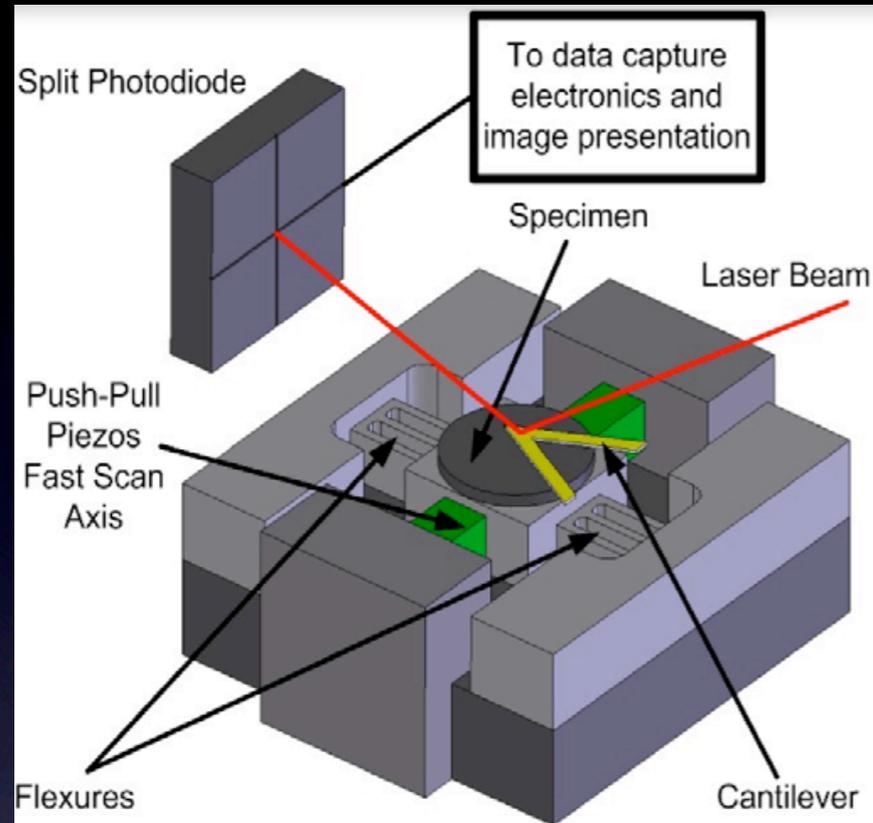
40kHz with
~3 μm amplitude



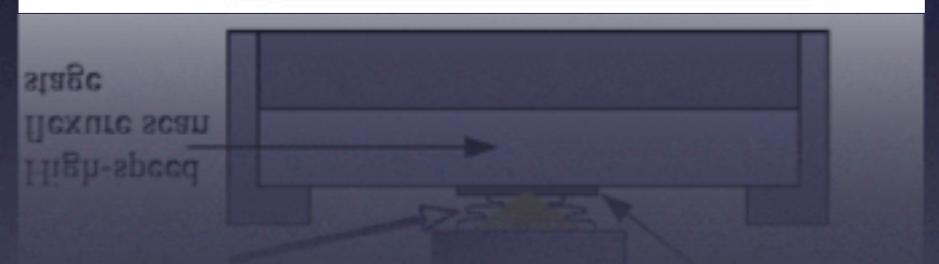
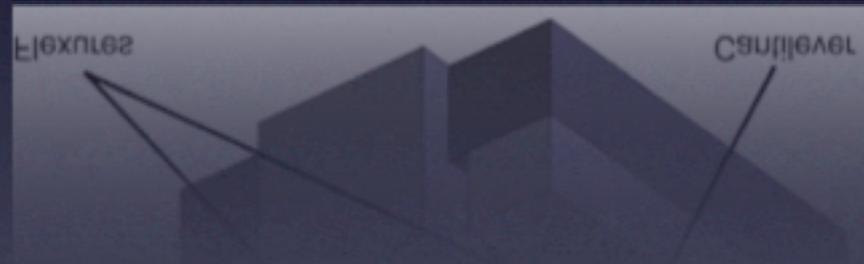
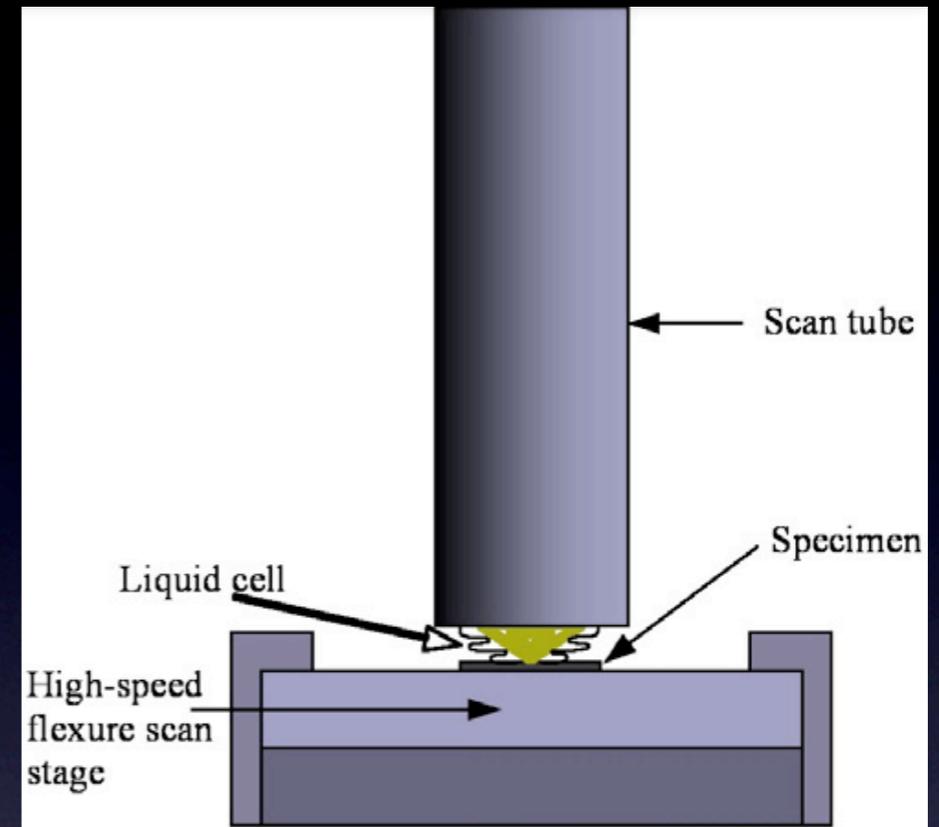
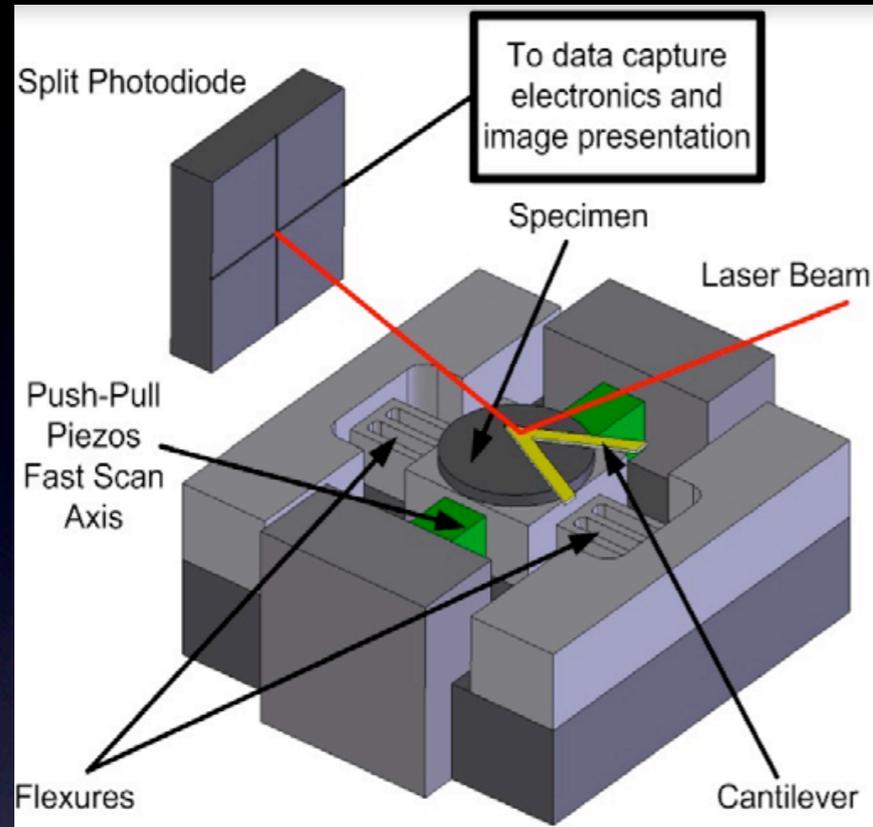
High-speed flexure stage and cantilever set-up



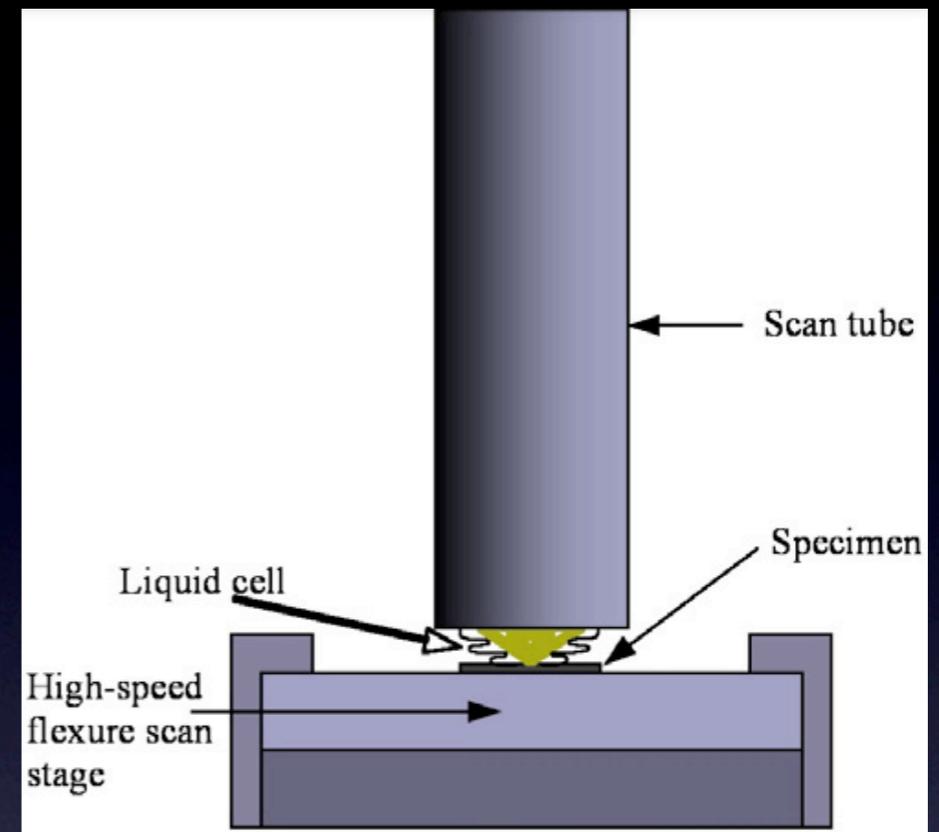
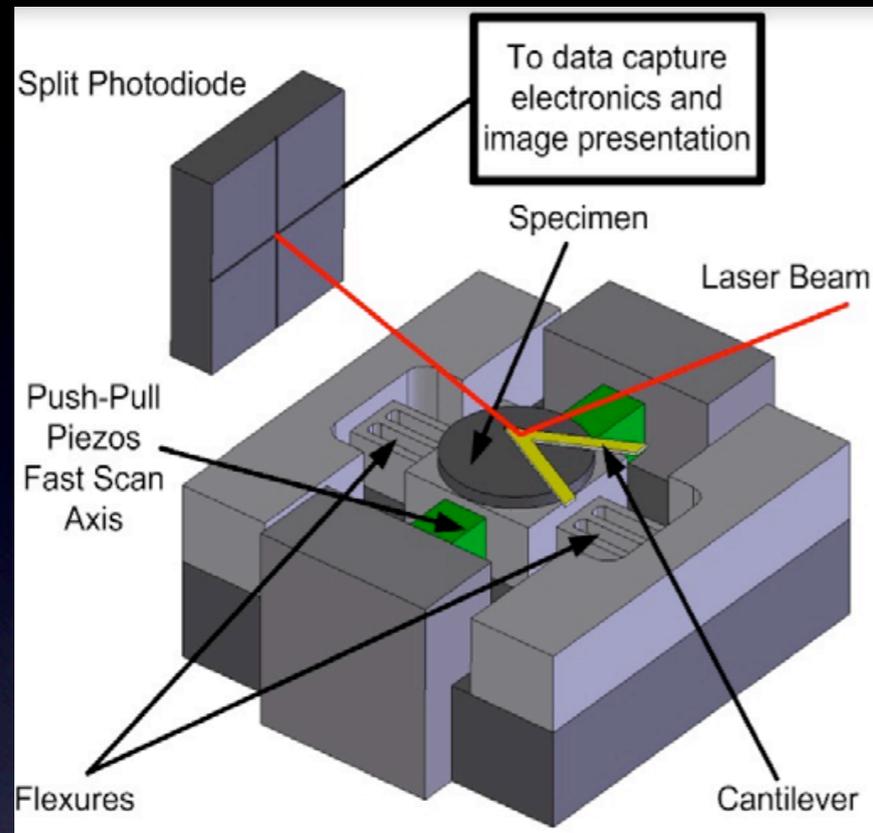
High-speed flexure stage and cantilever set-up



High-speed flexure stage and cantilever set-up

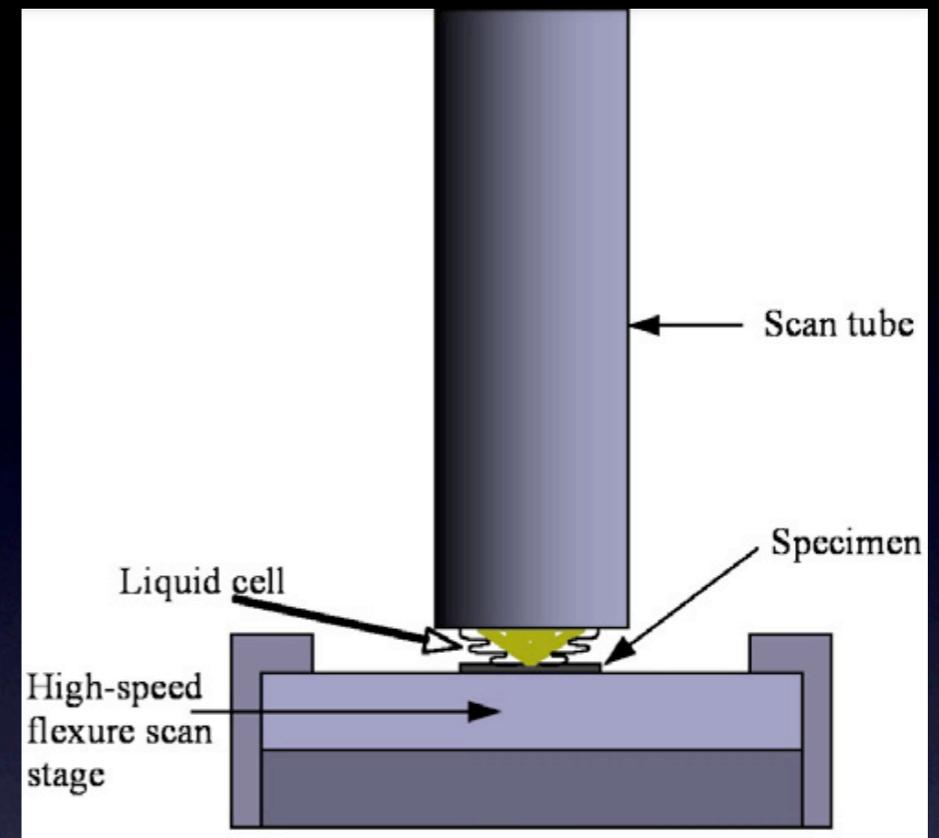
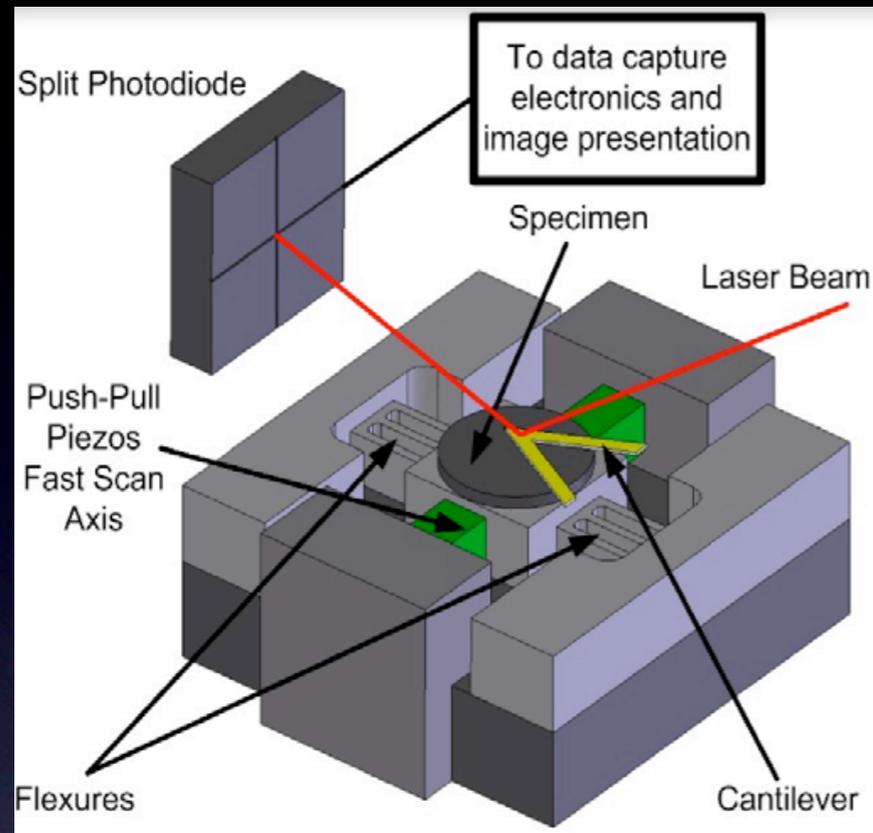


High-speed flexure stage and cantilever set-up



★ Conventional AFM electronic feedback

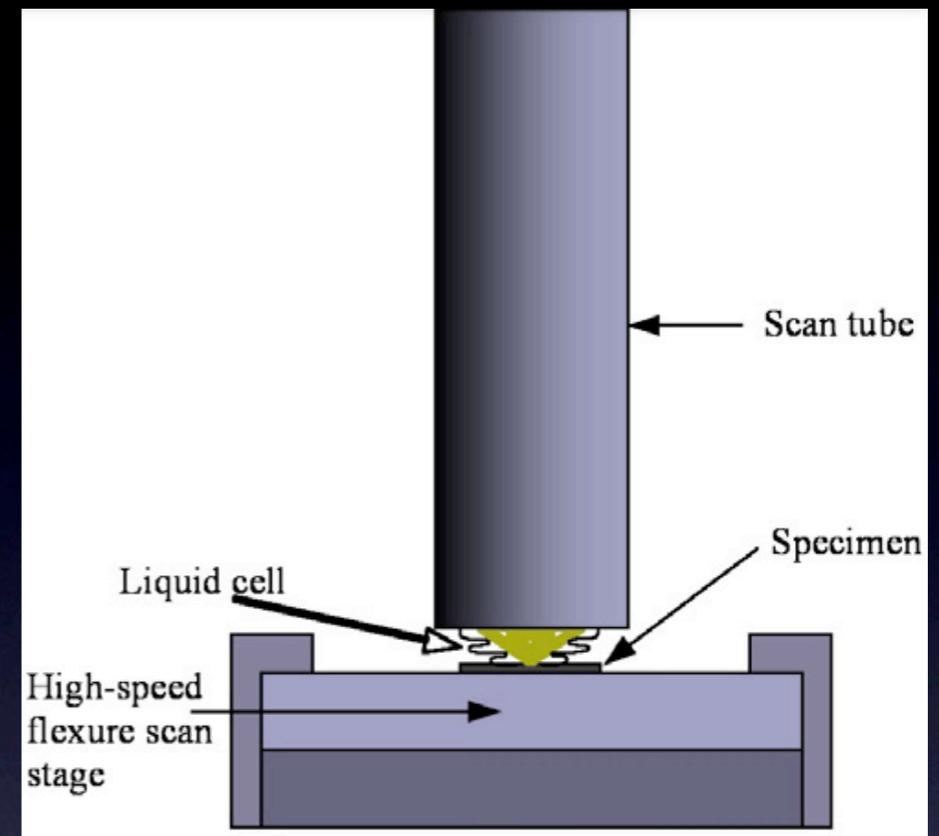
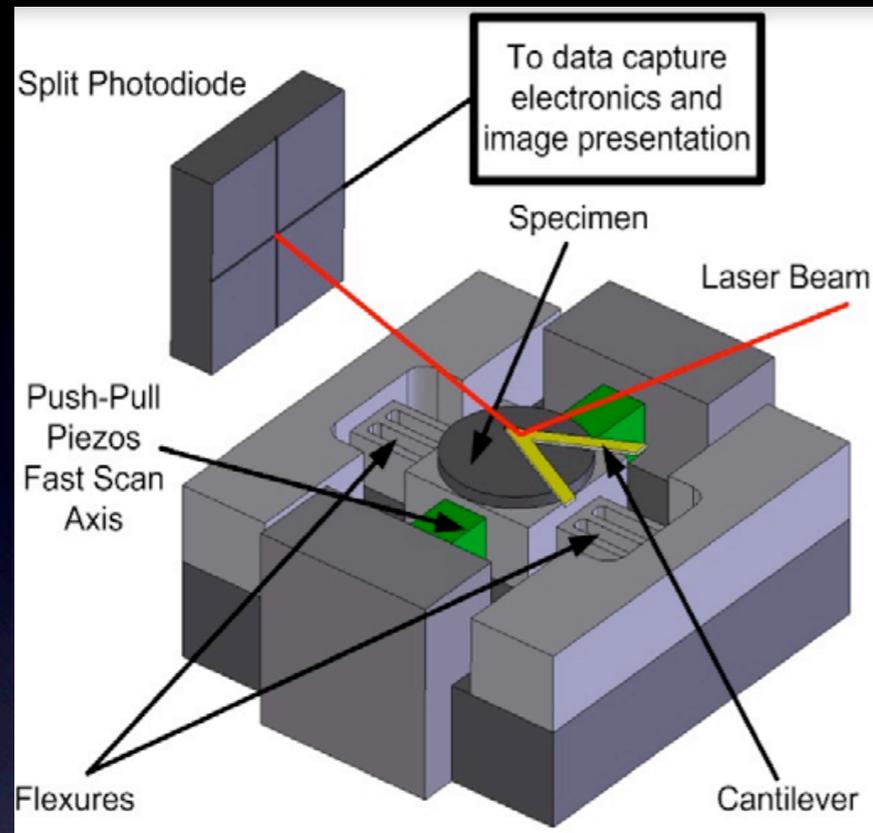
High-speed flexure stage and cantilever set-up



★ Conventional AFM electronic feedback

★ Mechanical feedback

High-speed flexure stage and cantilever set-up

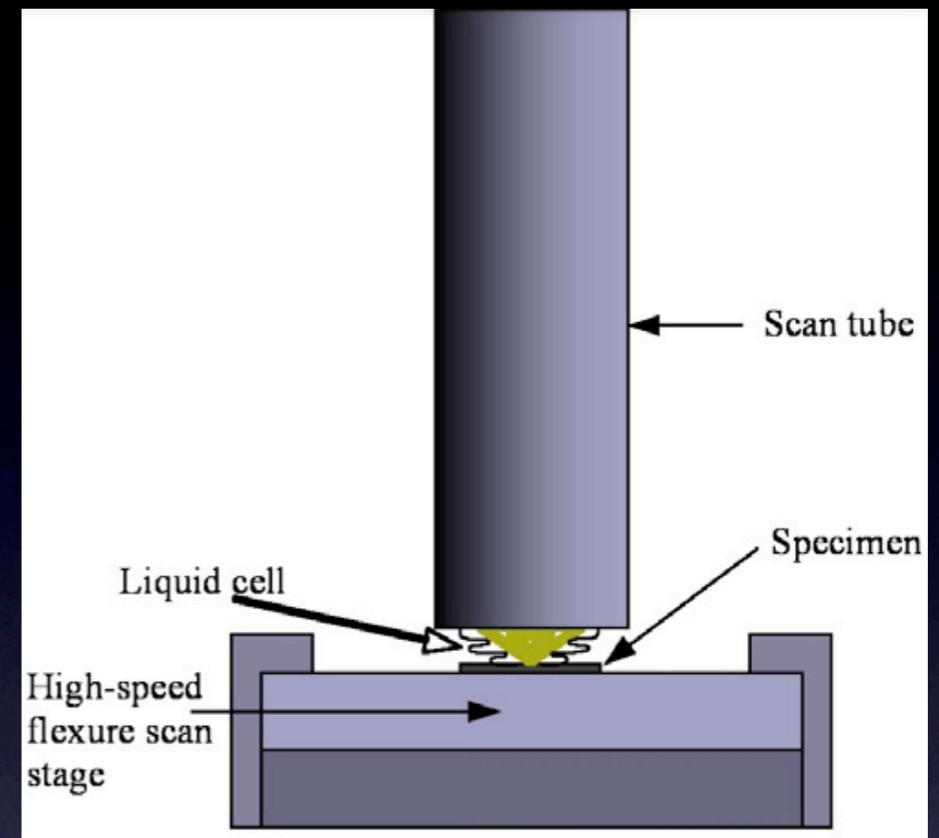
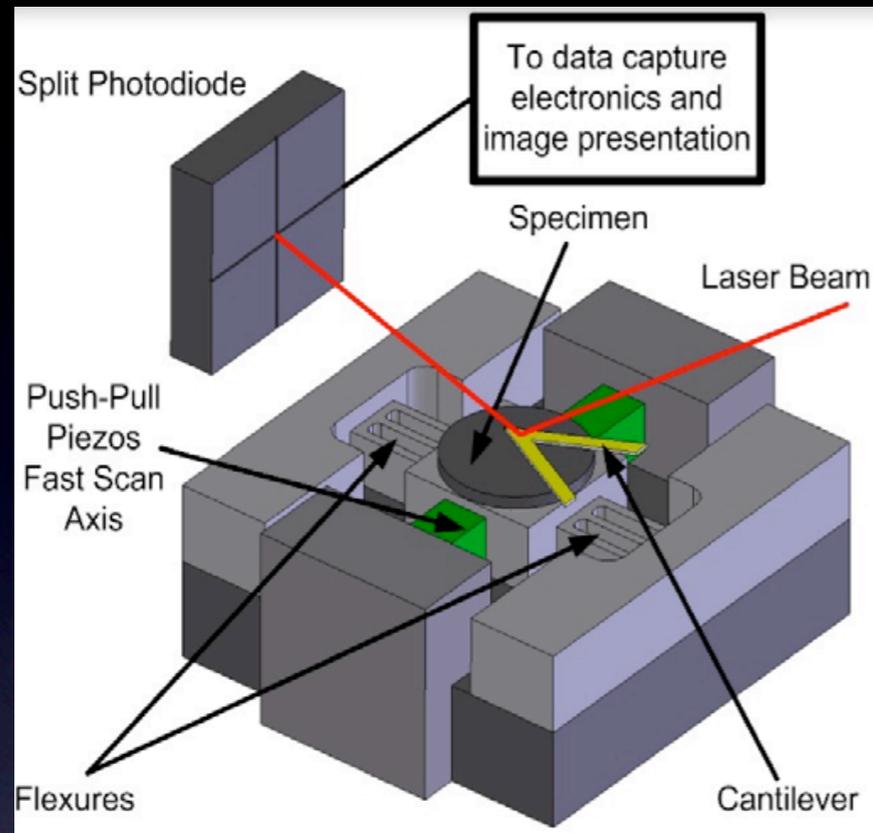


★ Conventional AFM electronic feedback

★ Mechanical feedback

★ super lubricity

High-speed flexure stage and cantilever set-up



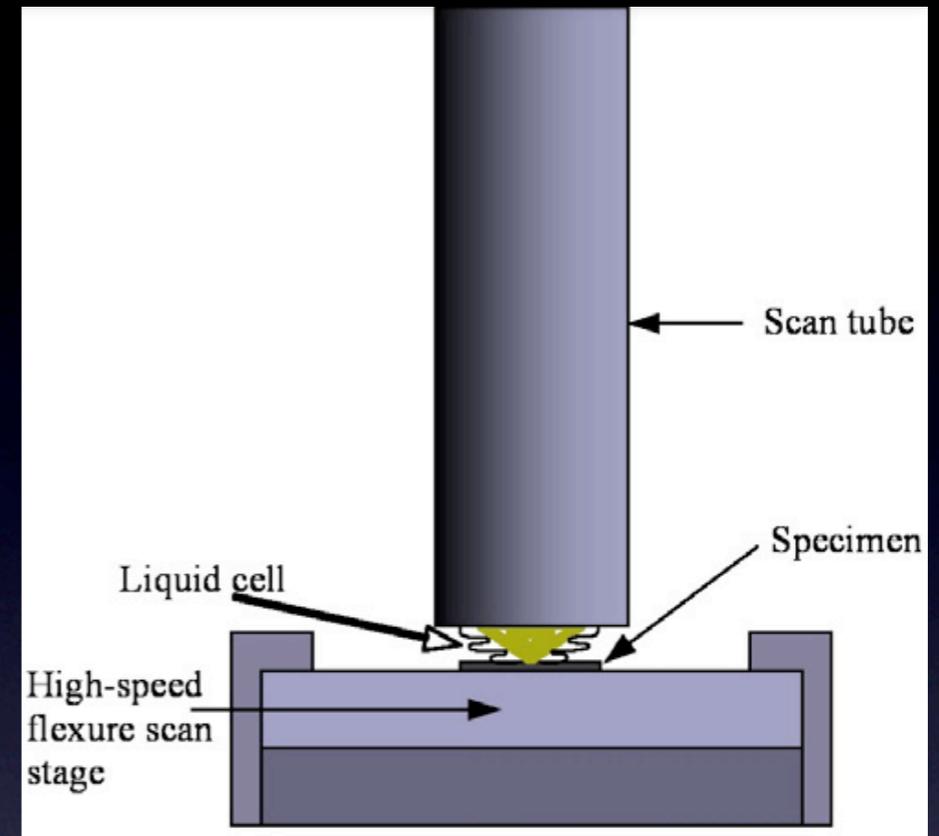
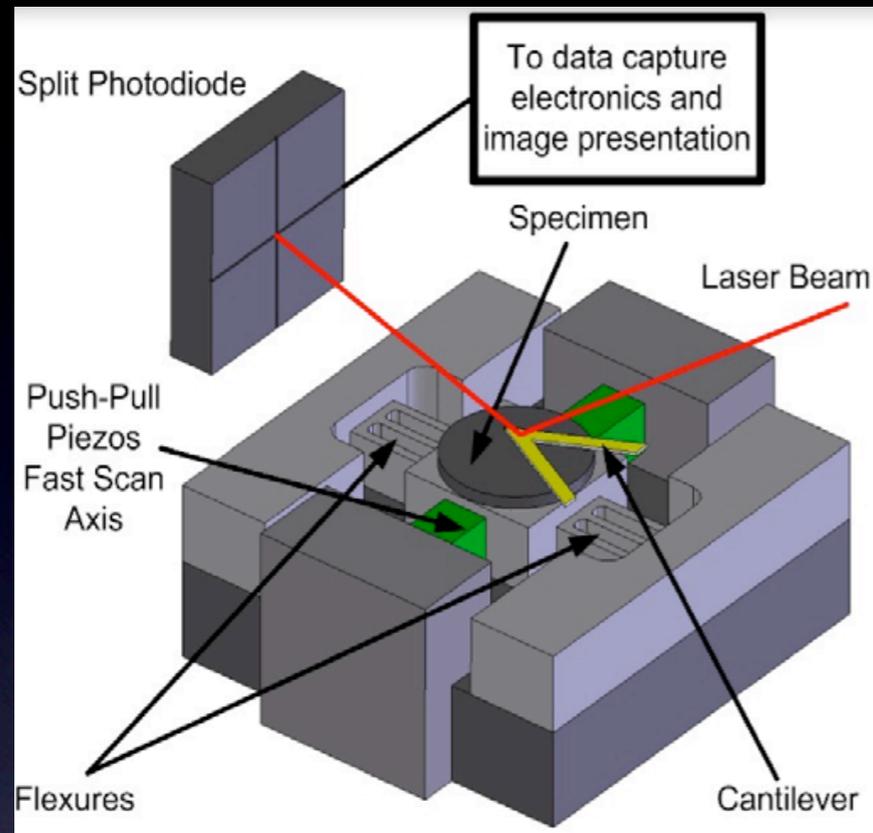
★ Conventional AFM electronic feedback

★ Mechanical feedback

★ super lubricity

★ water in confined geometry

High-speed flexure stage and cantilever set-up



★ Conventional AFM electronic feedback

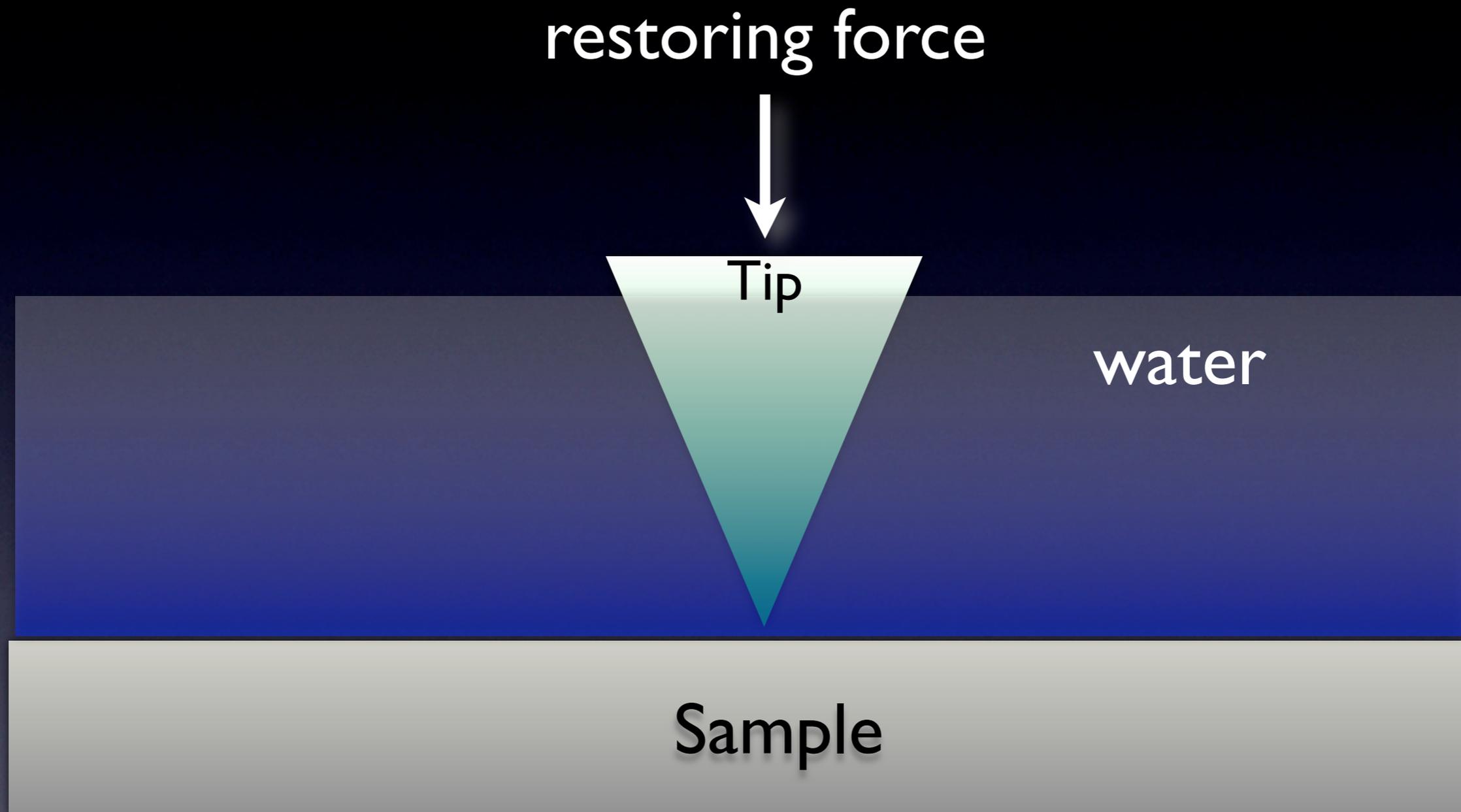
★ Mechanical feedback

★ super lubricity

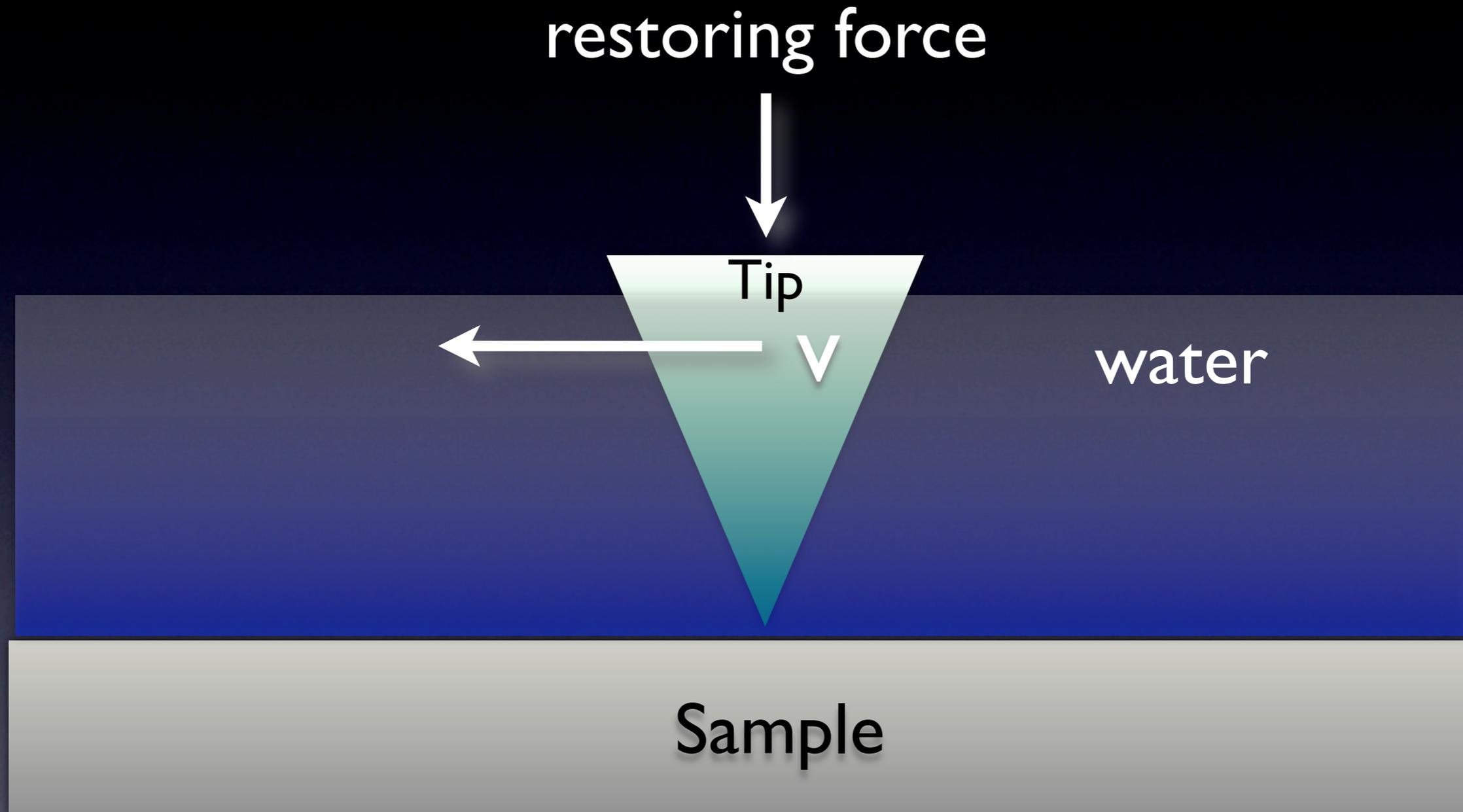
★ water in confined geometry

★ viscoelastic behaviour of specimen

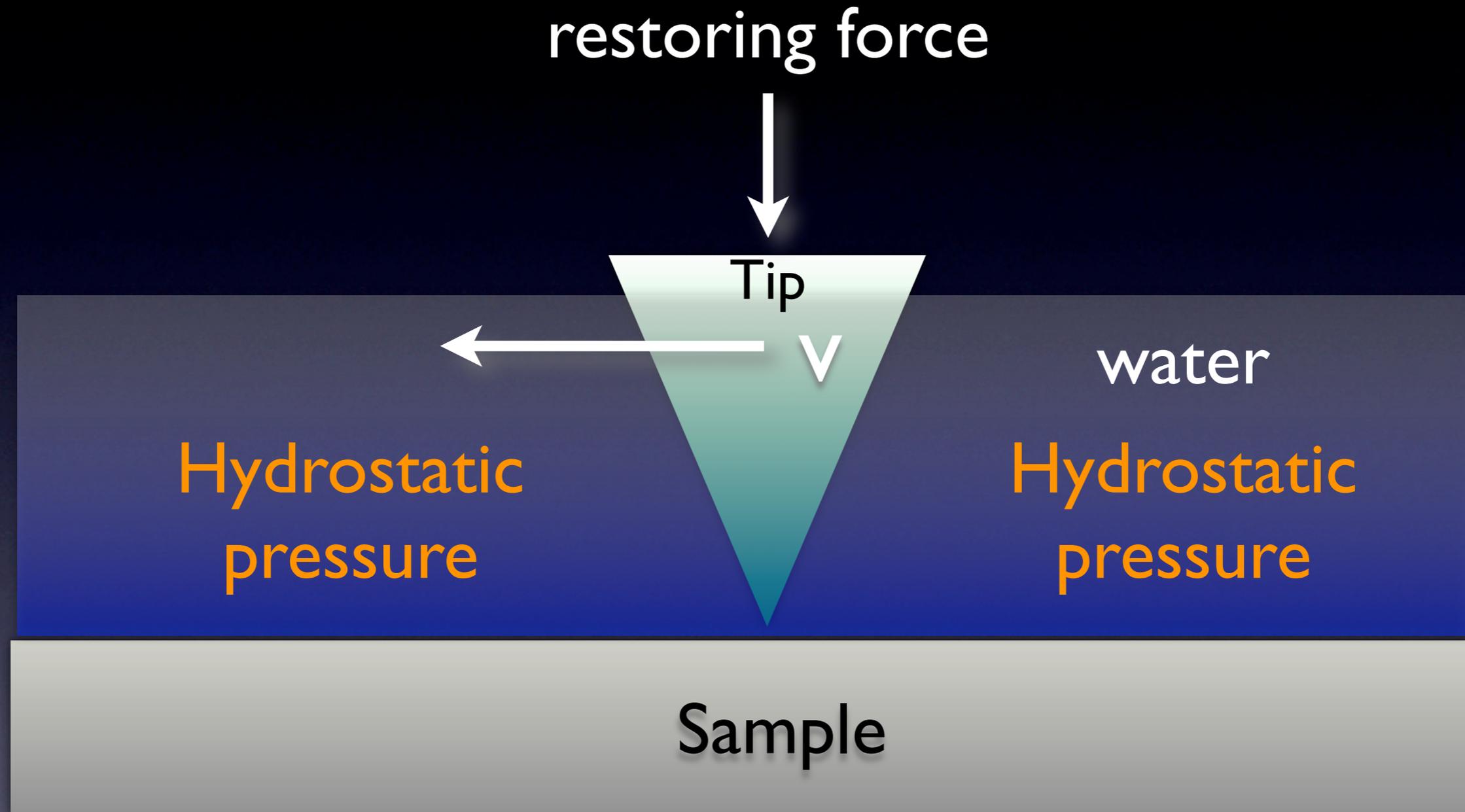
Principle of Superlubrication 'Feedback'



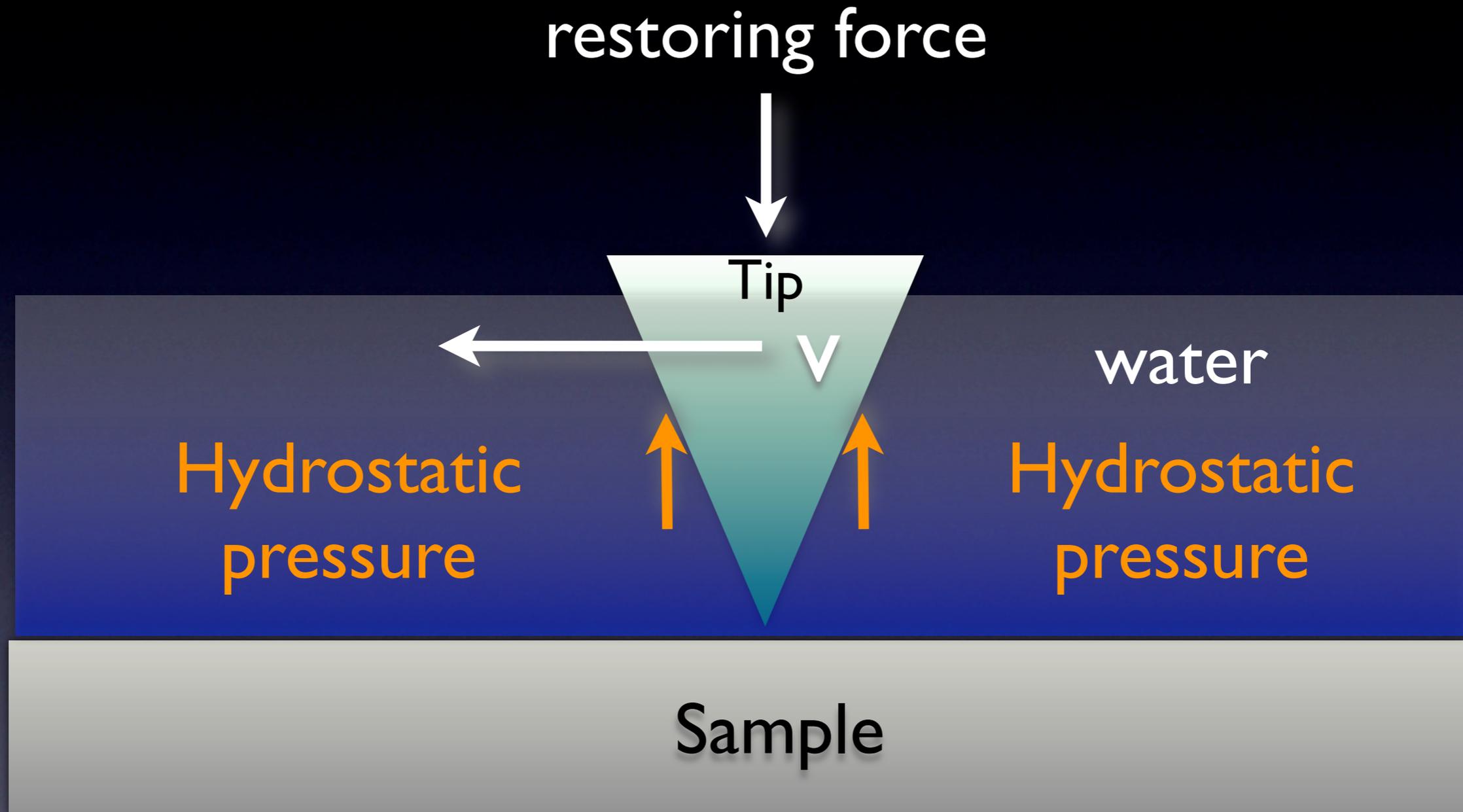
Principle of Superlubrication 'Feedback'



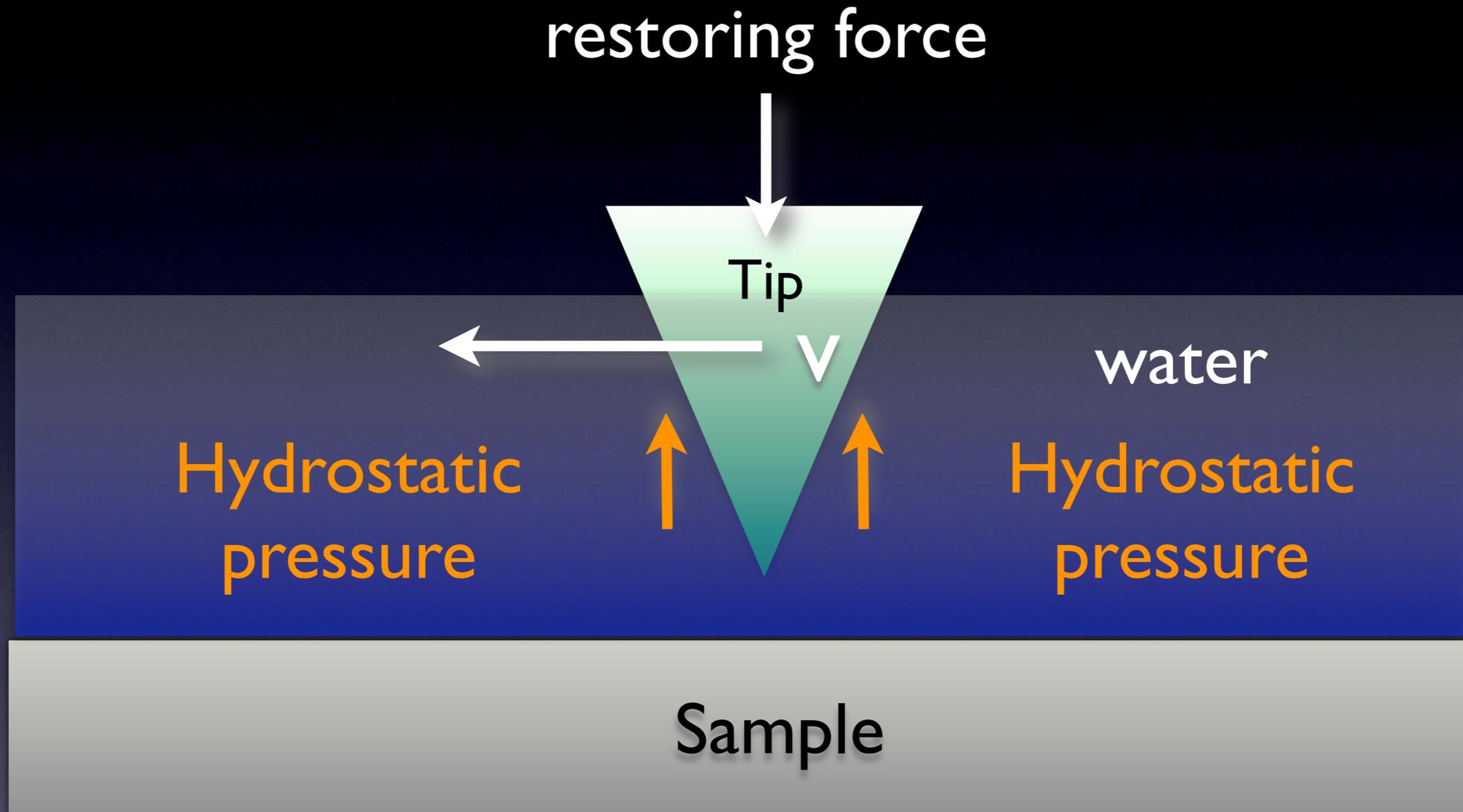
Principle of Superlubrication 'Feedback'



Principle of Superlubrication 'Feedback'



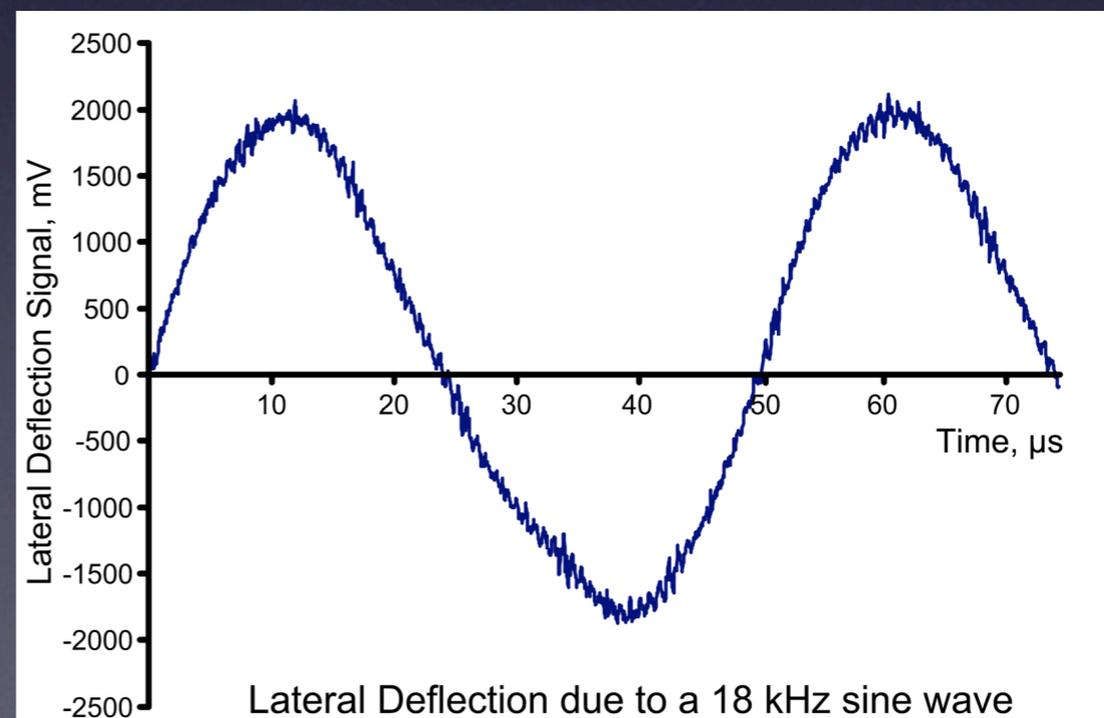
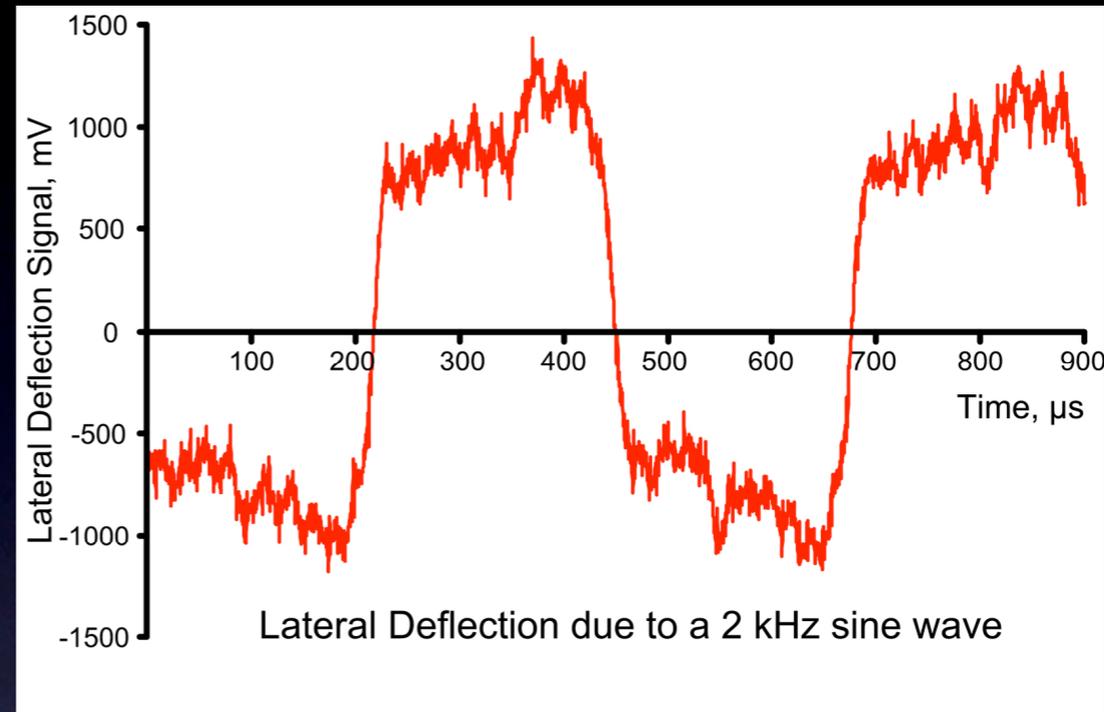
Principle of Superlubrication 'Feedback'



Torque on Tip

Velocity Dependent Transition

Sine wave displacement
of sample



Torque on Tip

Velocity Dependent Transition

Sine wave displacement
of sample

$< 1 \text{ mm/s}$



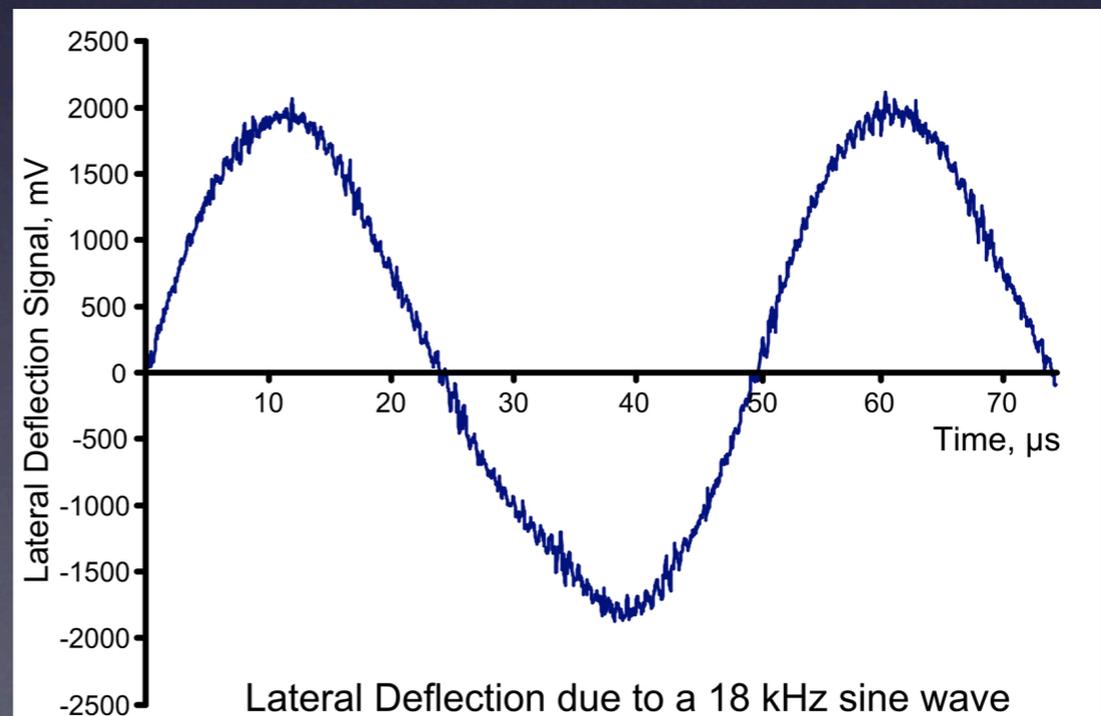
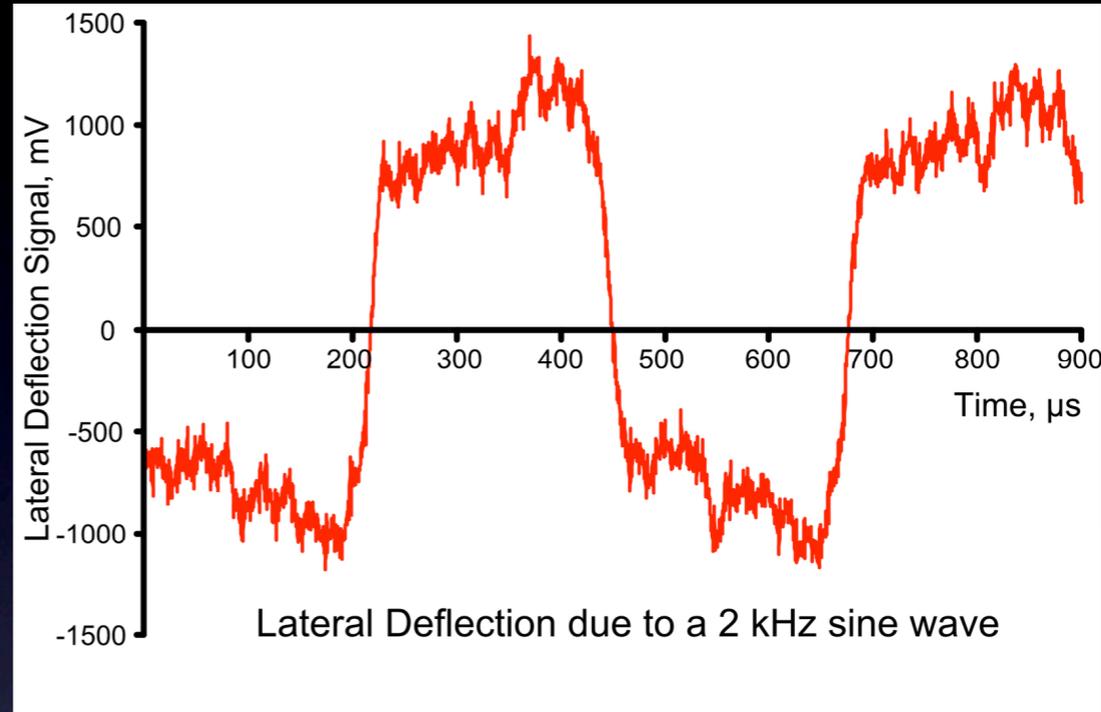
Surface Friction



$>> 1 \text{ mm/s}$



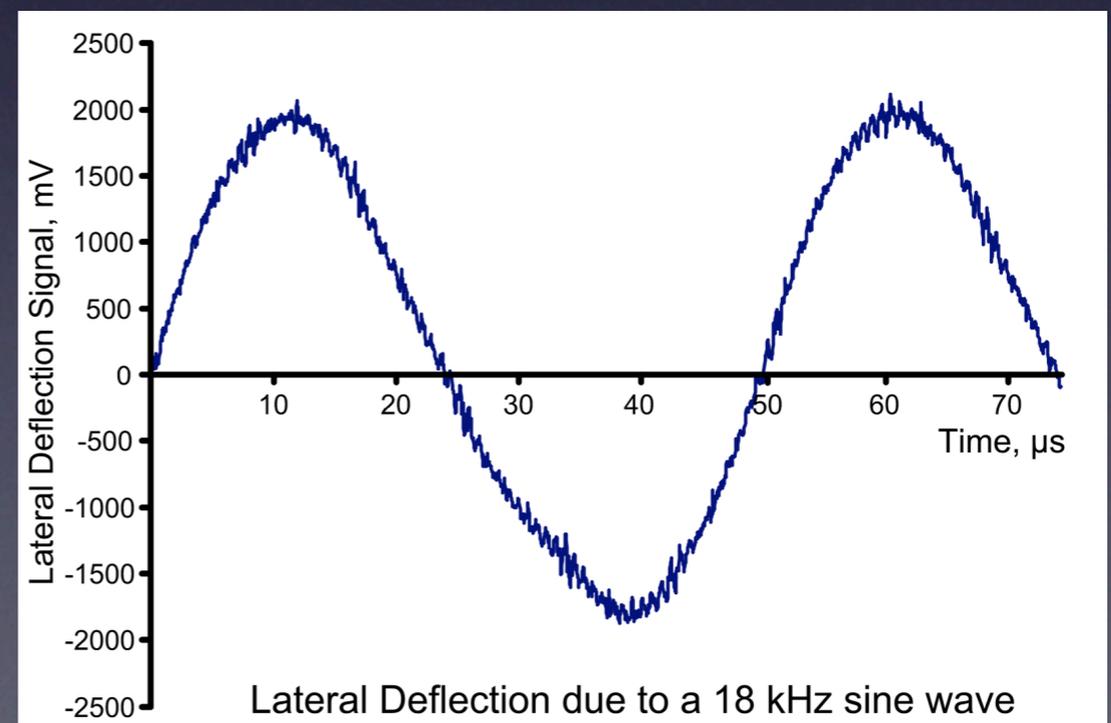
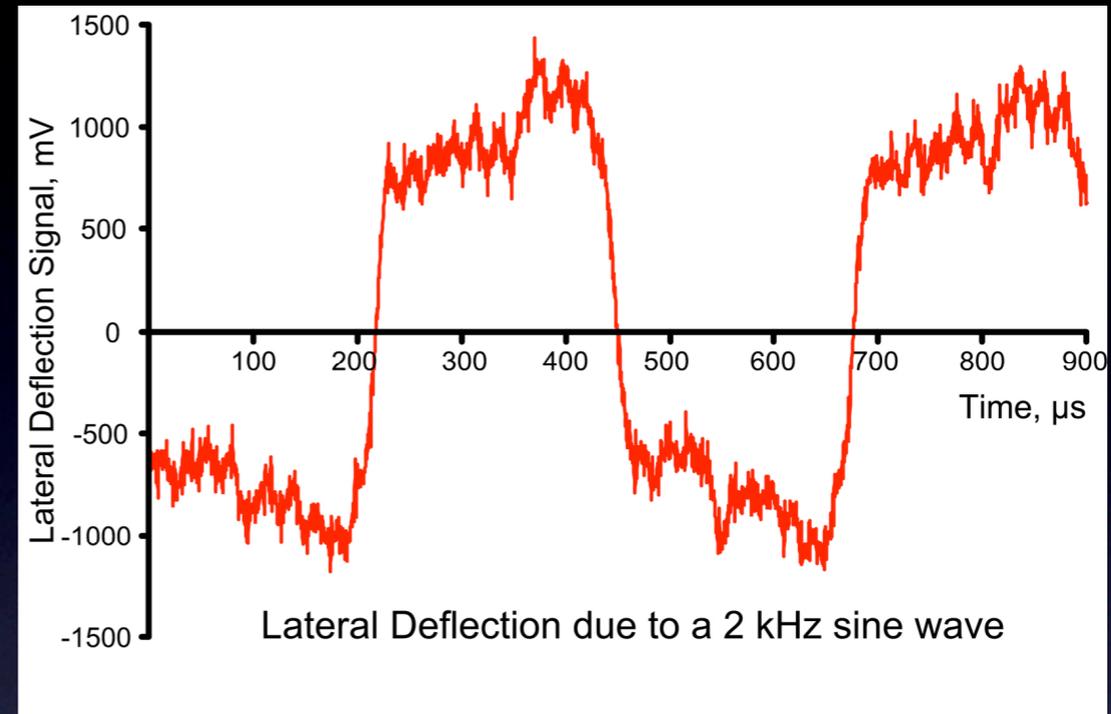
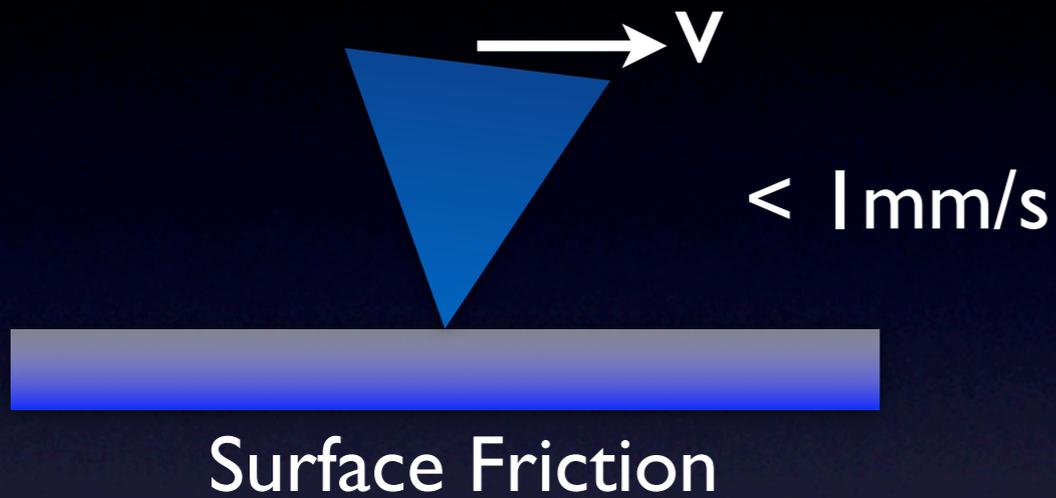
Hydrodynamic Viscous Force
(velocity dependent)



Torque on Tip

Velocity Dependent Transition

Sine wave displacement
of sample



Torque on Tip

Velocity Dependent Transition

Sine wave displacement
of sample

$< 1 \text{ mm/s}$



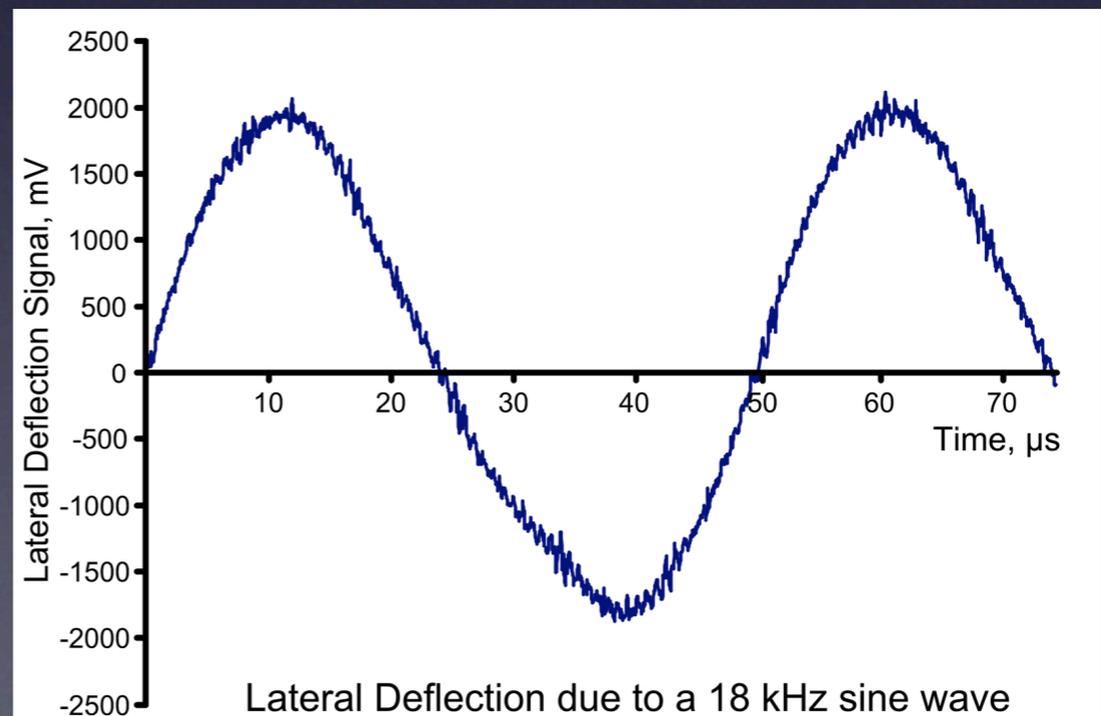
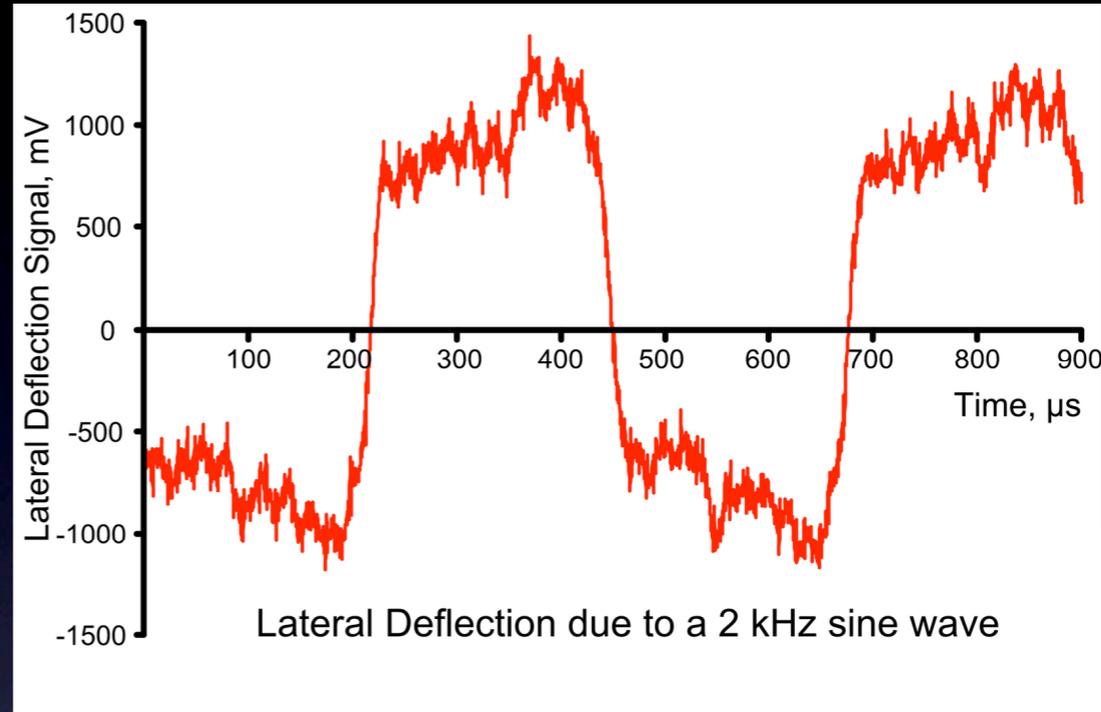
Surface Friction



$>> 1 \text{ mm/s}$



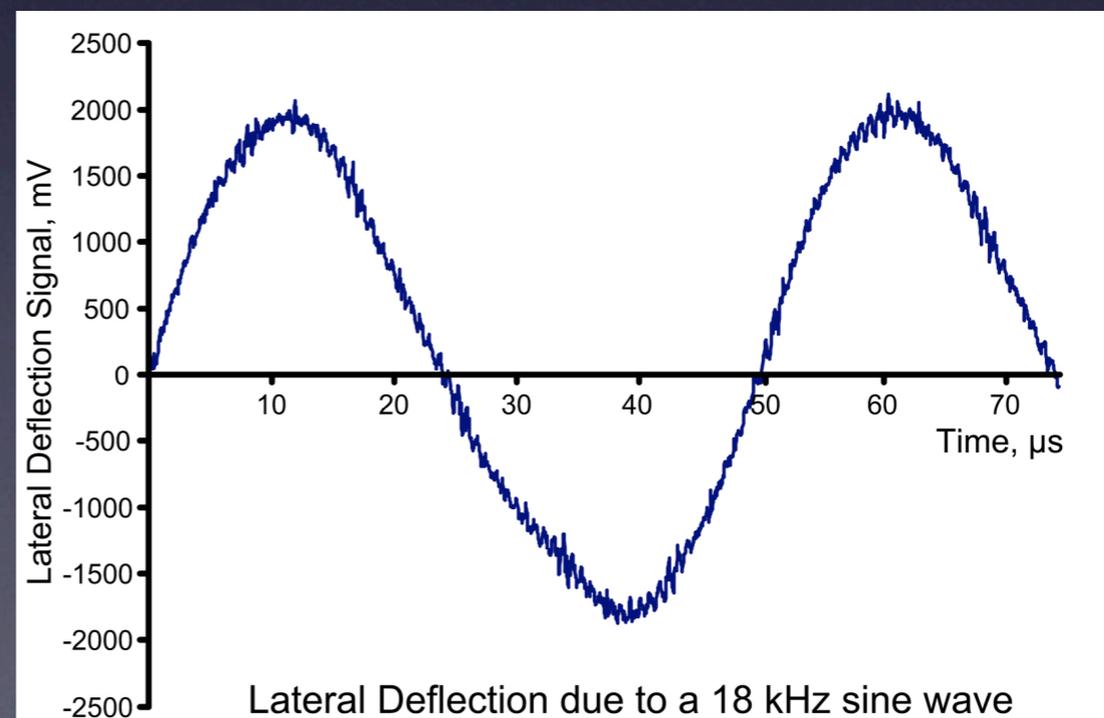
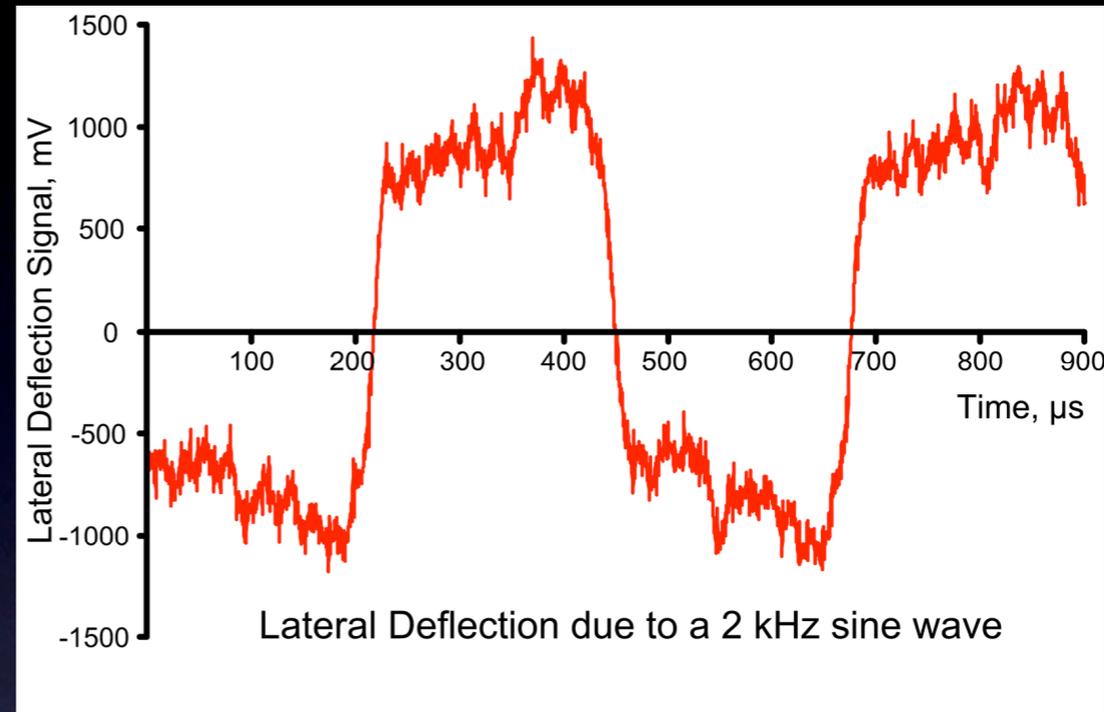
Hydrodynamic Viscous Force
(velocity dependent)



Torque on Tip

Velocity Dependent Transition

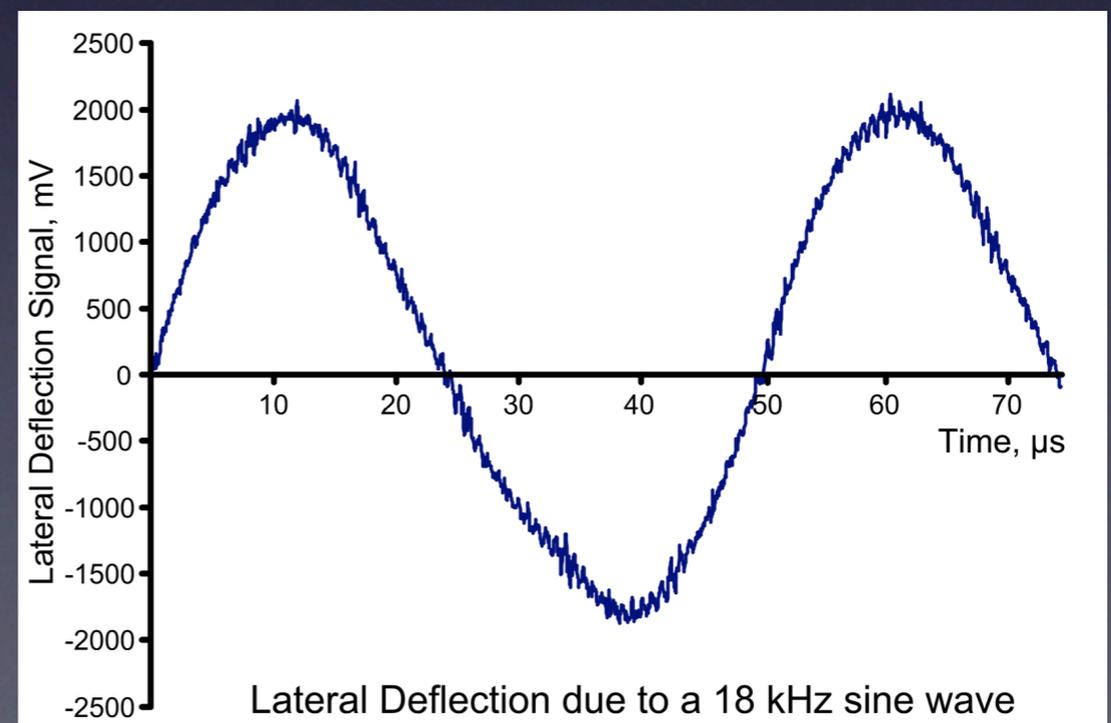
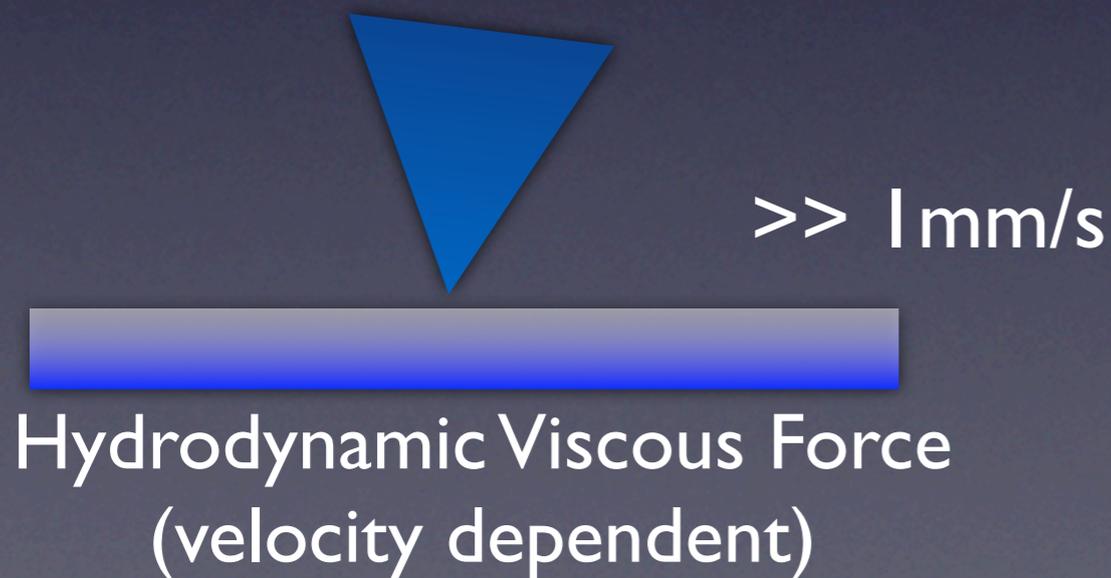
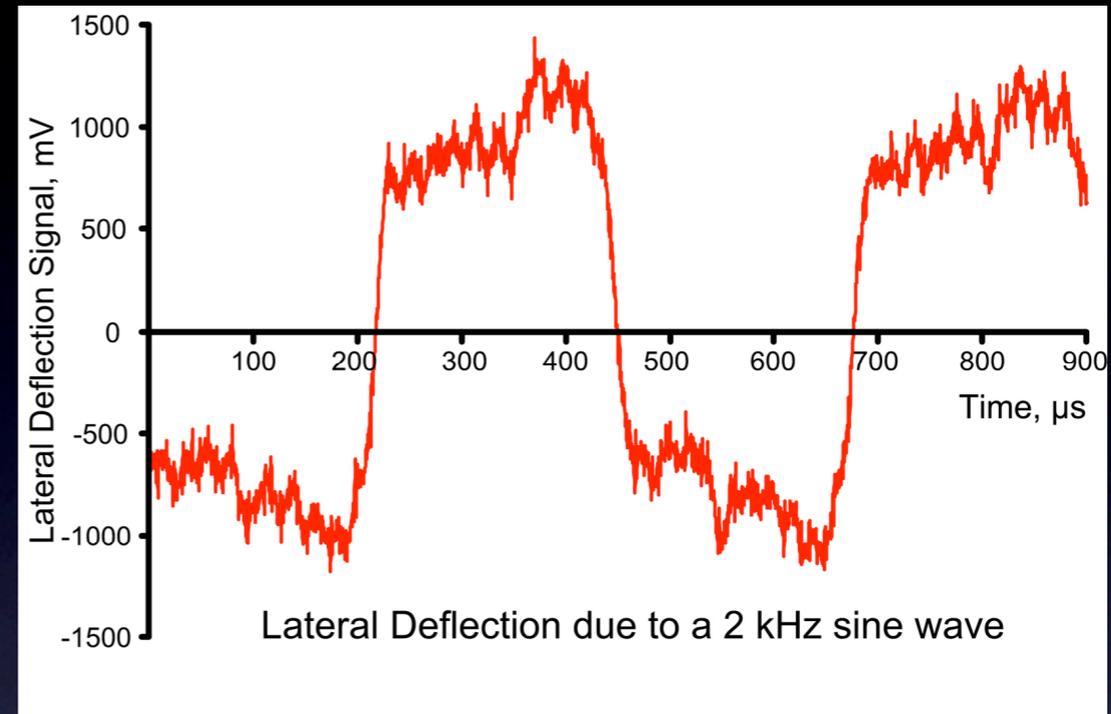
Sine wave displacement
of sample



Torque on Tip

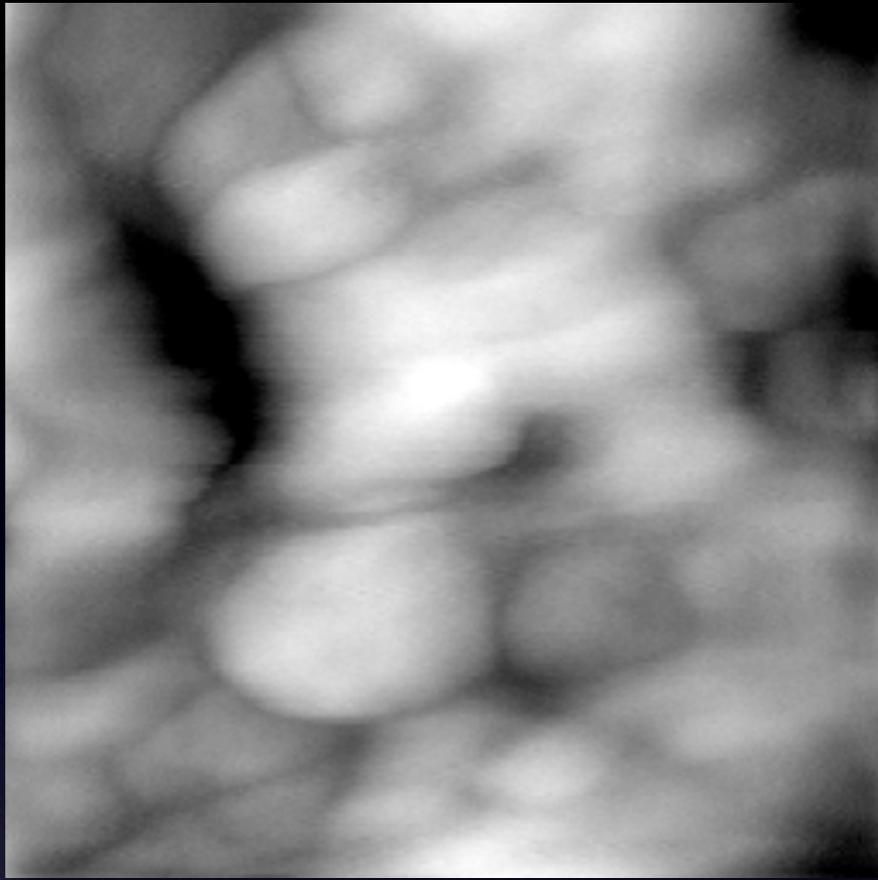
Velocity Dependent Transition

Sine wave displacement
of sample



PEO video AFM 30 f/s - image reproducibility

0 mins
0 frames



3 mins
~ 6000
frames



7 mins
13,000
frames

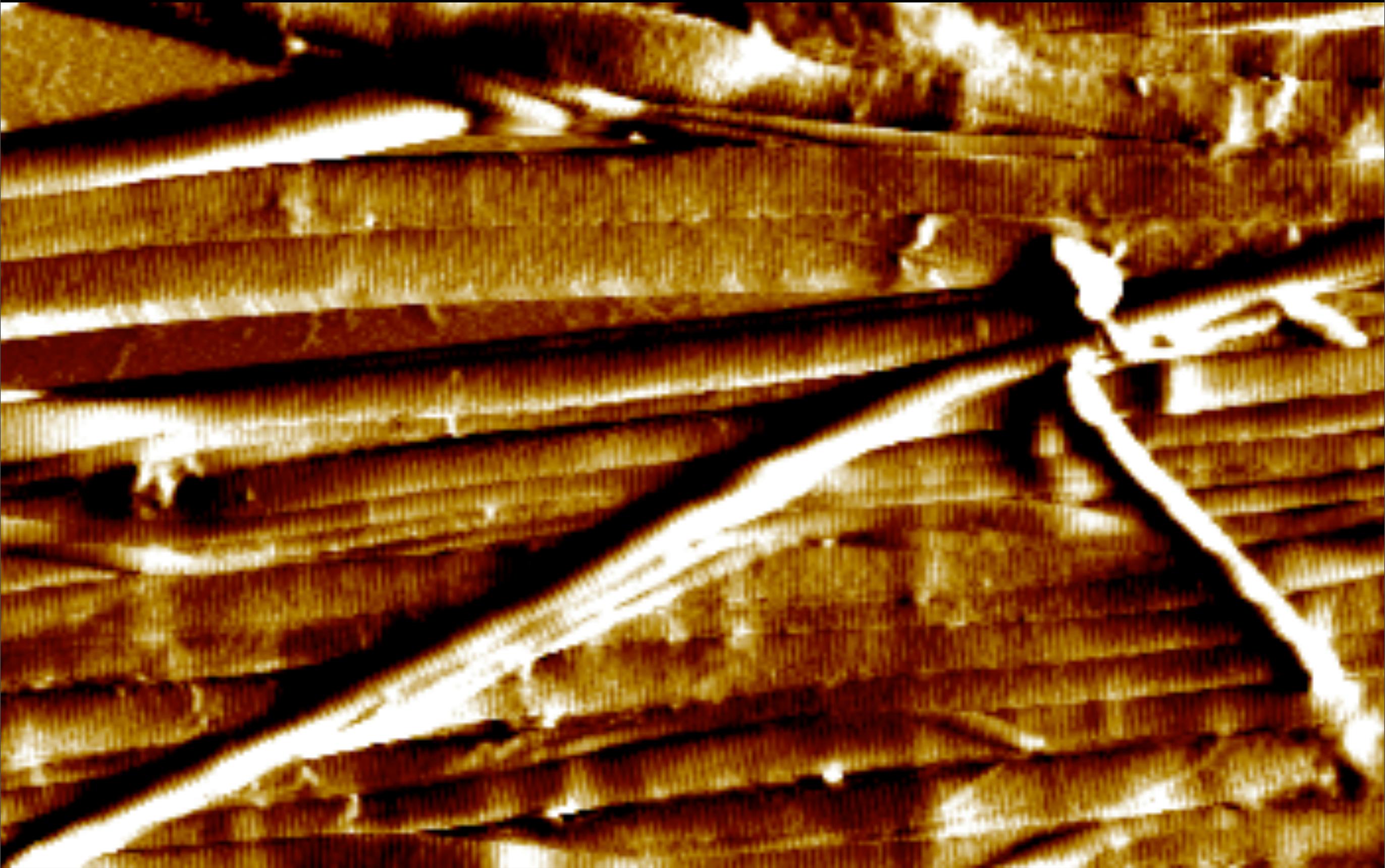


10 mins
18,000
frames

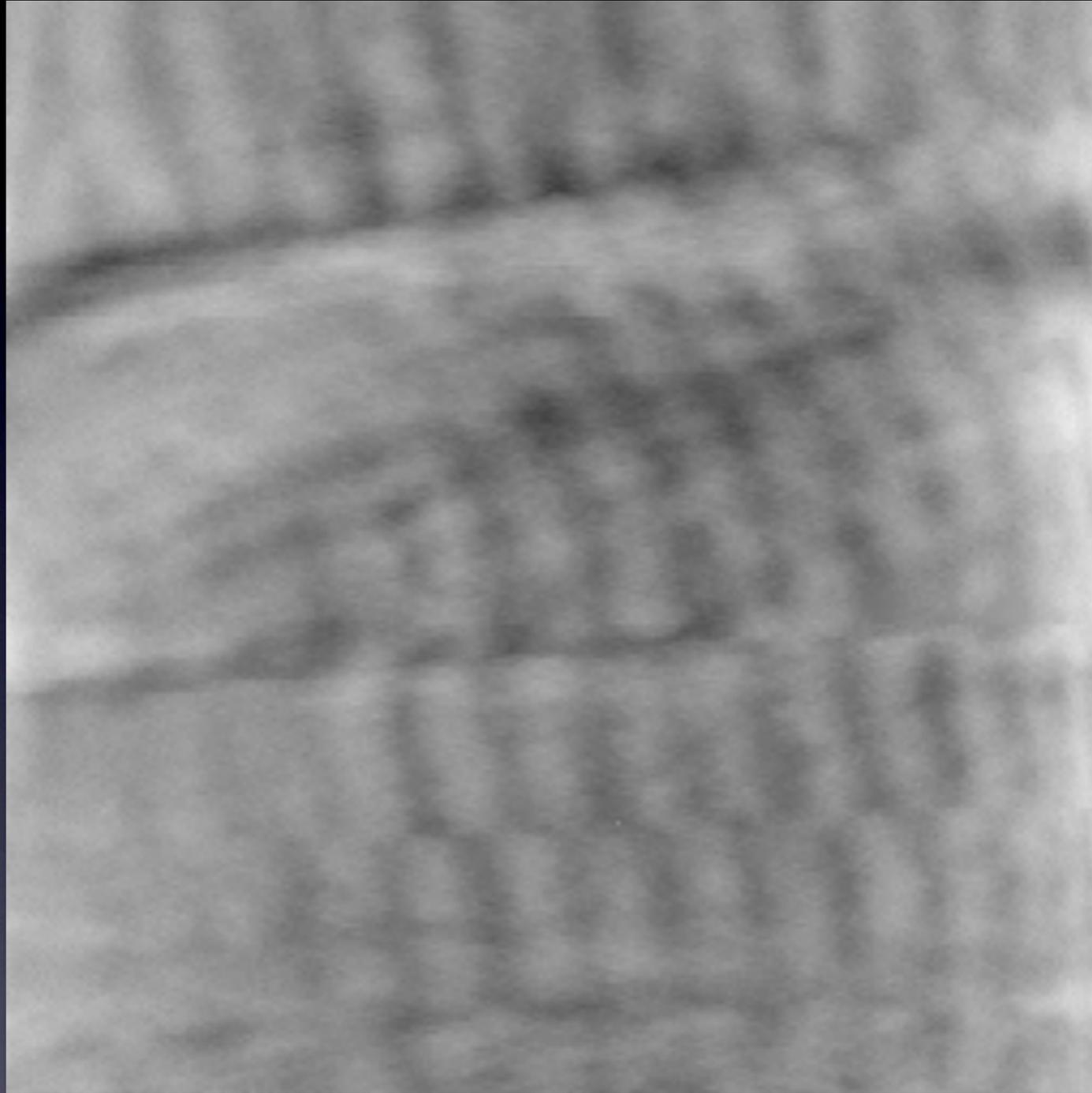


Collagen Micro fibrils

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

Linear Collage of High-speed AFM Images of Collagen Fibre

Linear Collage of High-speed AFM Images of Collagen Fibre

All the high-speed images were taken in 0.7 s

Linear Collage of High-speed AFM Images of Collagen Fibre

Conventional AFM

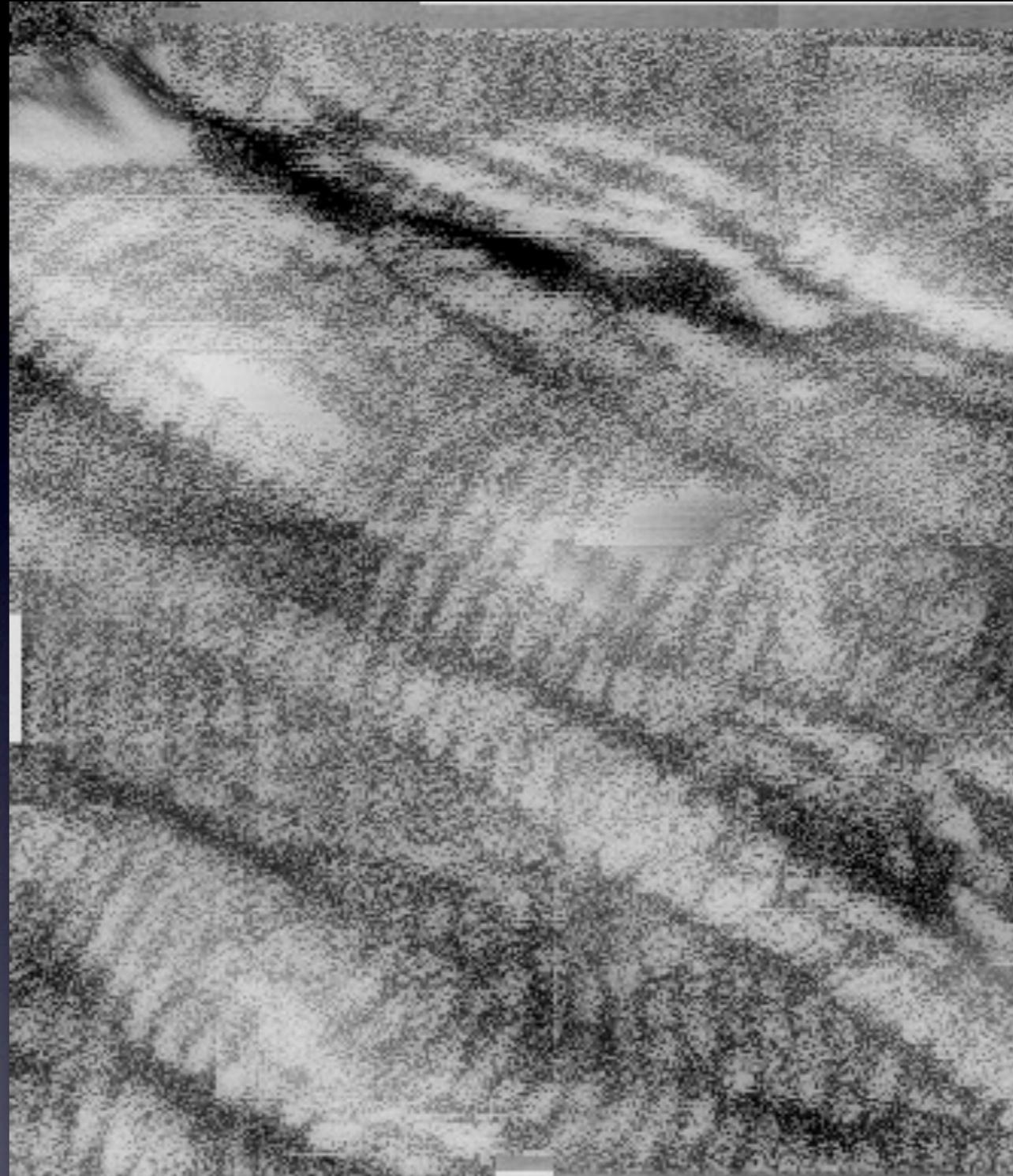


High-speed AFM



All the high-speed images were taken in 0.7 s

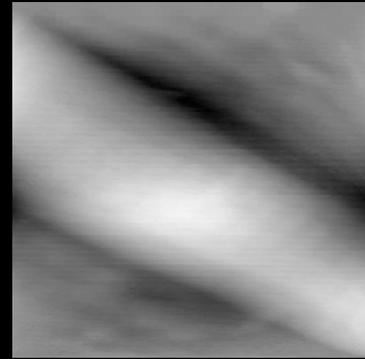
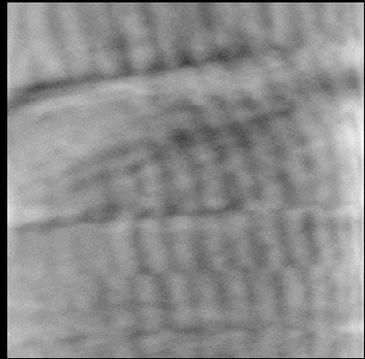
High-speed AFM Collagen Collage



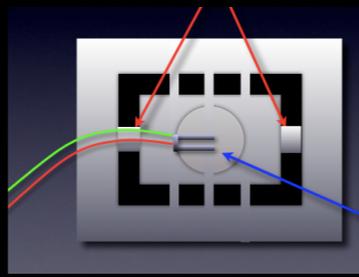
12 images involved - acquired in 200 ms

L M Picco, L Bozec, A Ulcinas, D J Engledew, M Antognozzi, M A Horton and M J Miles
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How fast can this system image?



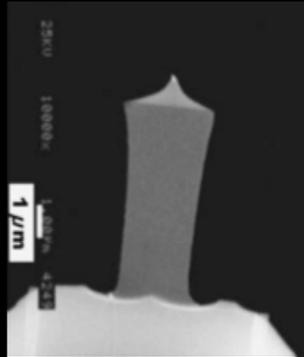
1000 fps



Resonant/Flexure contact AFM

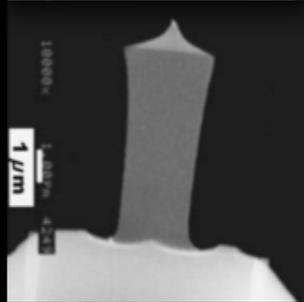
How fast can this system image?

100 fps

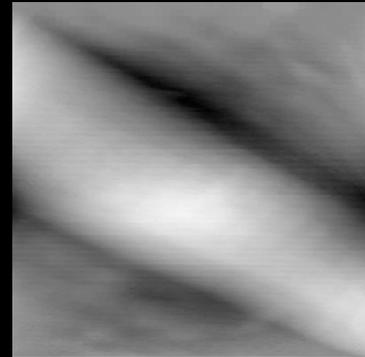
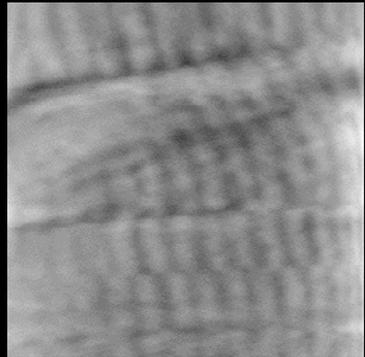


High-speed small-cantilever AFM

10 fps



1.0 fps



0.1 fps

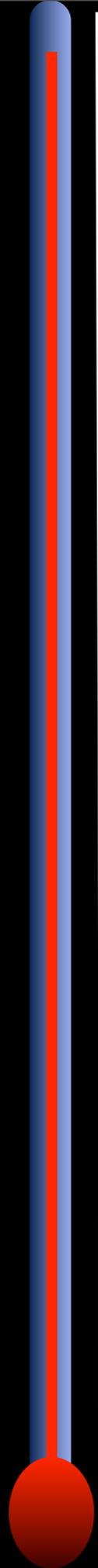


AFM + Active Q control

0.01 fps

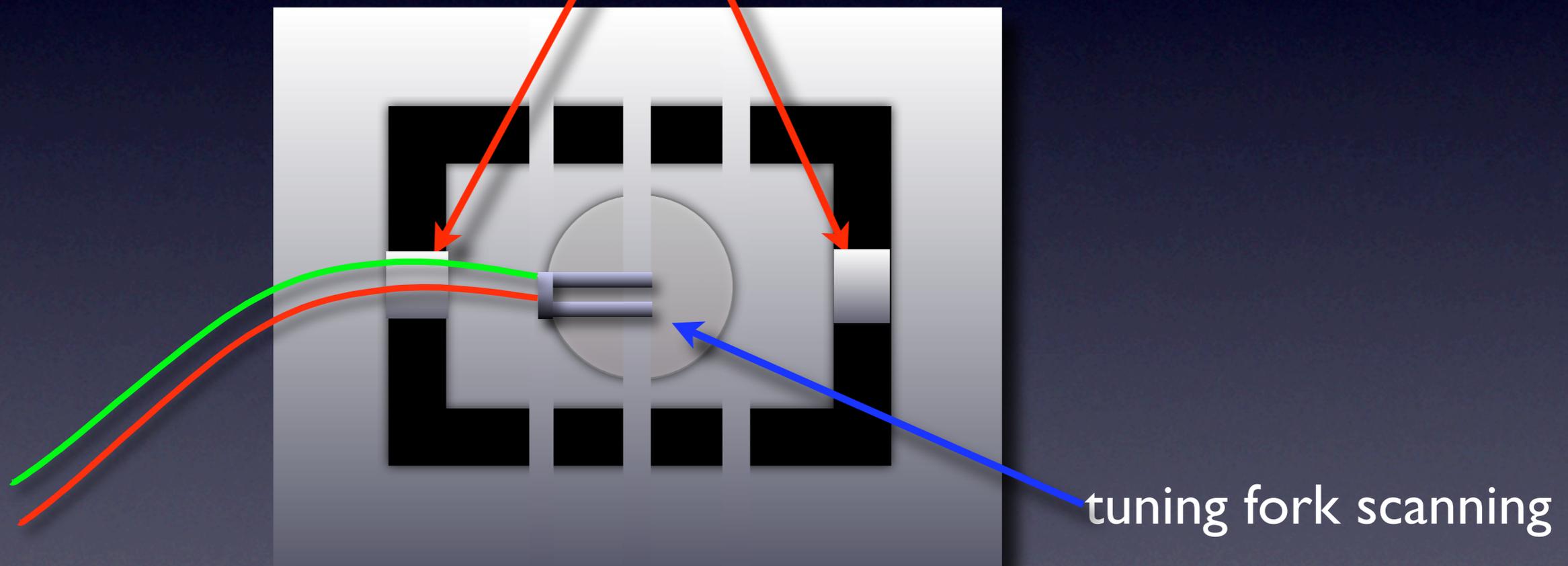


Conventional AFM



Scanner Arrangement for kilohertz AFM

piezo drivers operating in tandem



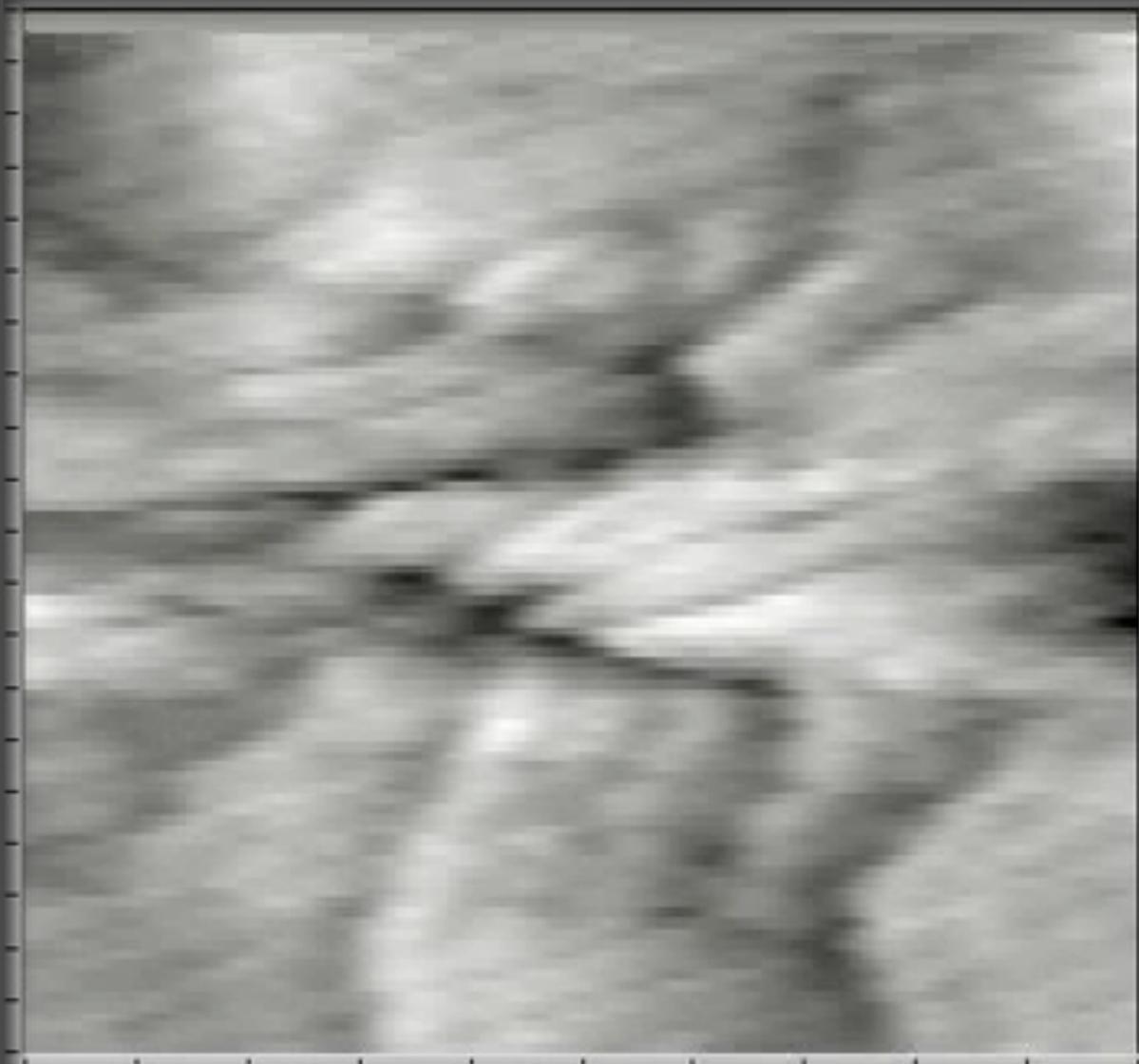
L M Picco, L Bozec, A Ulcinas, D J Engledew, M Antognozzi, M A Horton and M J Miles
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Frame Rate:

1 000 fps

*Line
Rate:*

200,000 lps



*Playback
Rate:*

25 fps

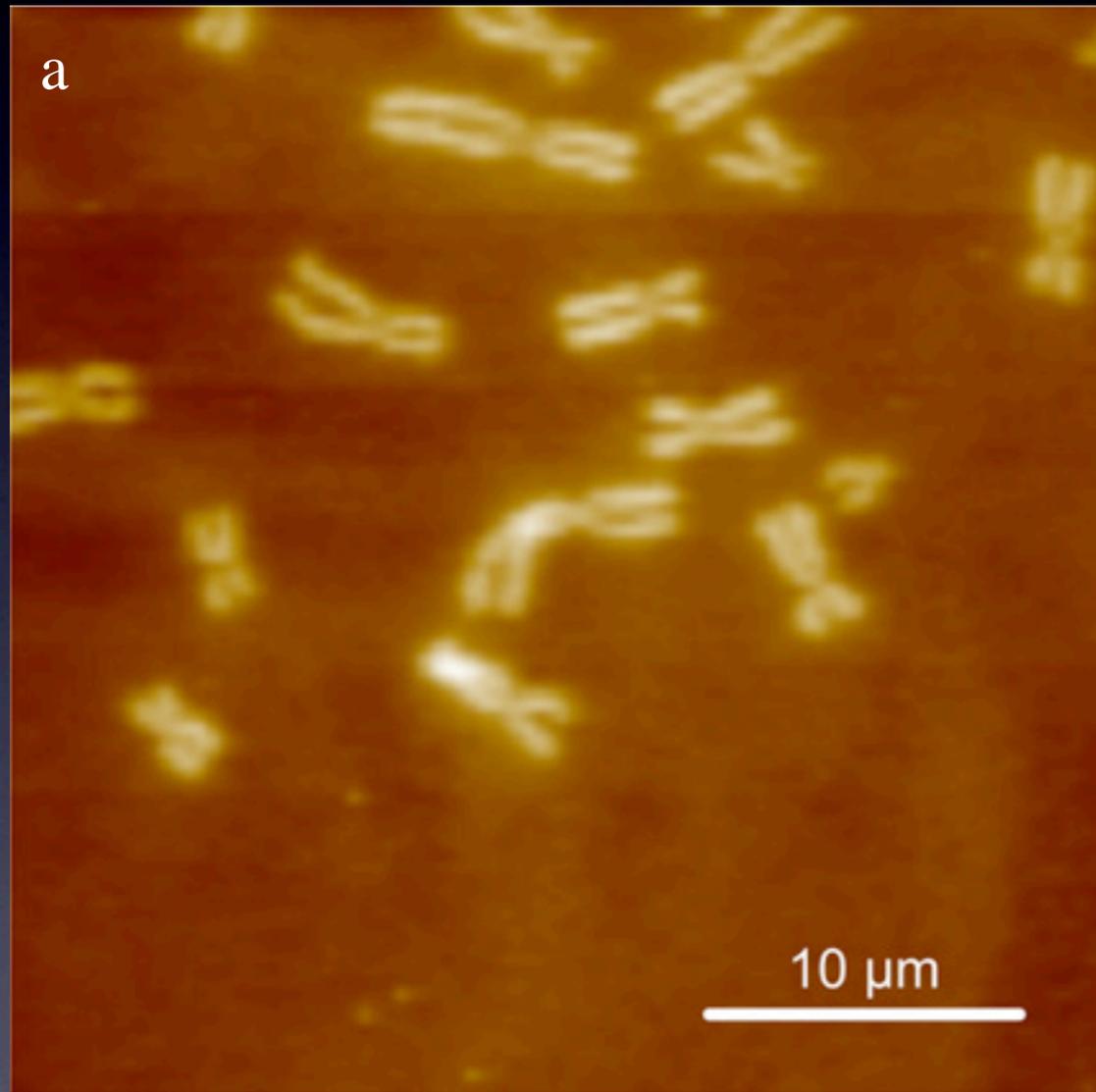
200 x 100 pixels; ~ 25 ns/pixel (on average)

*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*

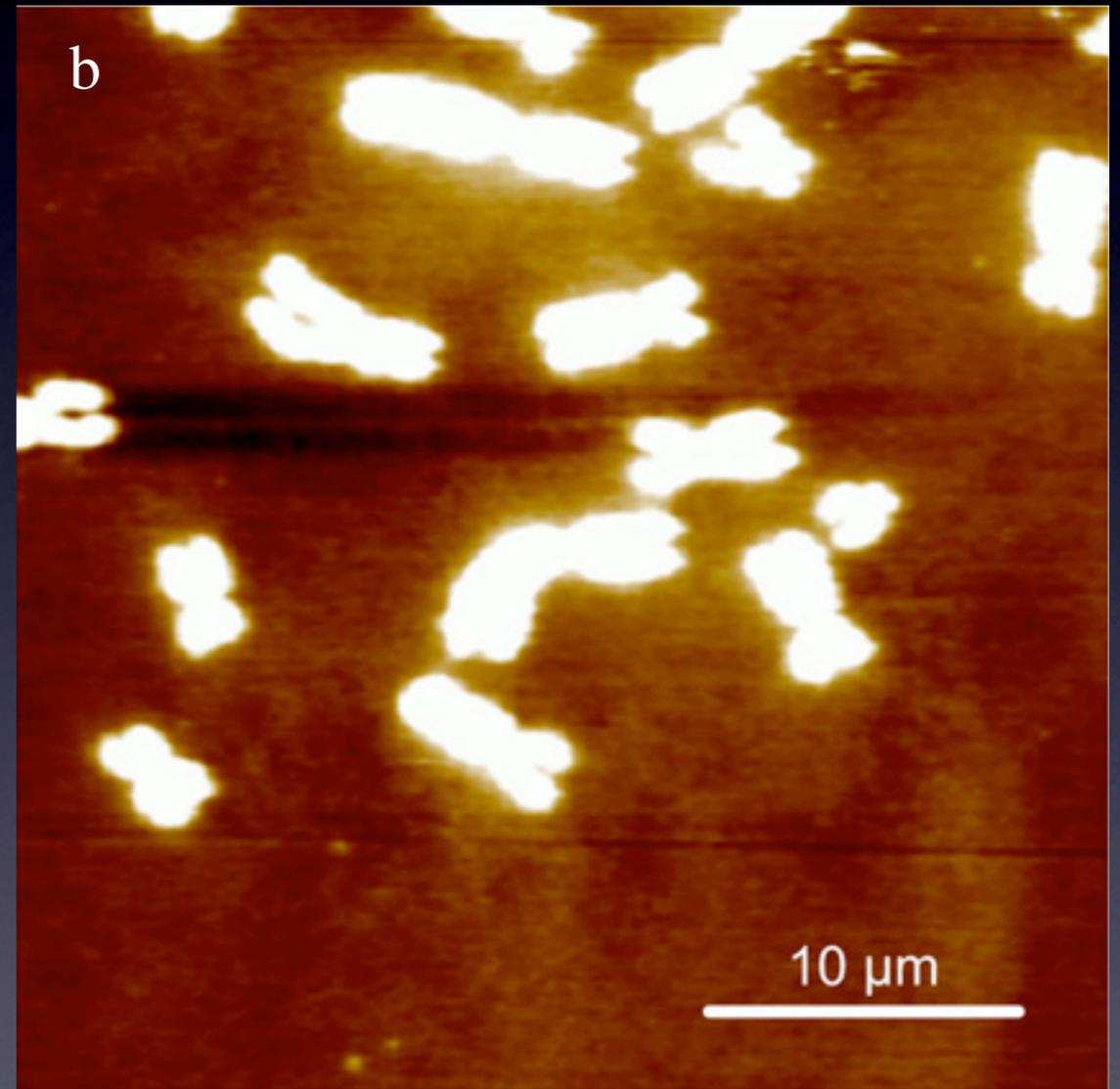
High-speed AFM of 'large' objects

Human Chromosomes

Air



2mM NaCl



Picco, Miles, Komatsubara, Hoshi, Ushiki

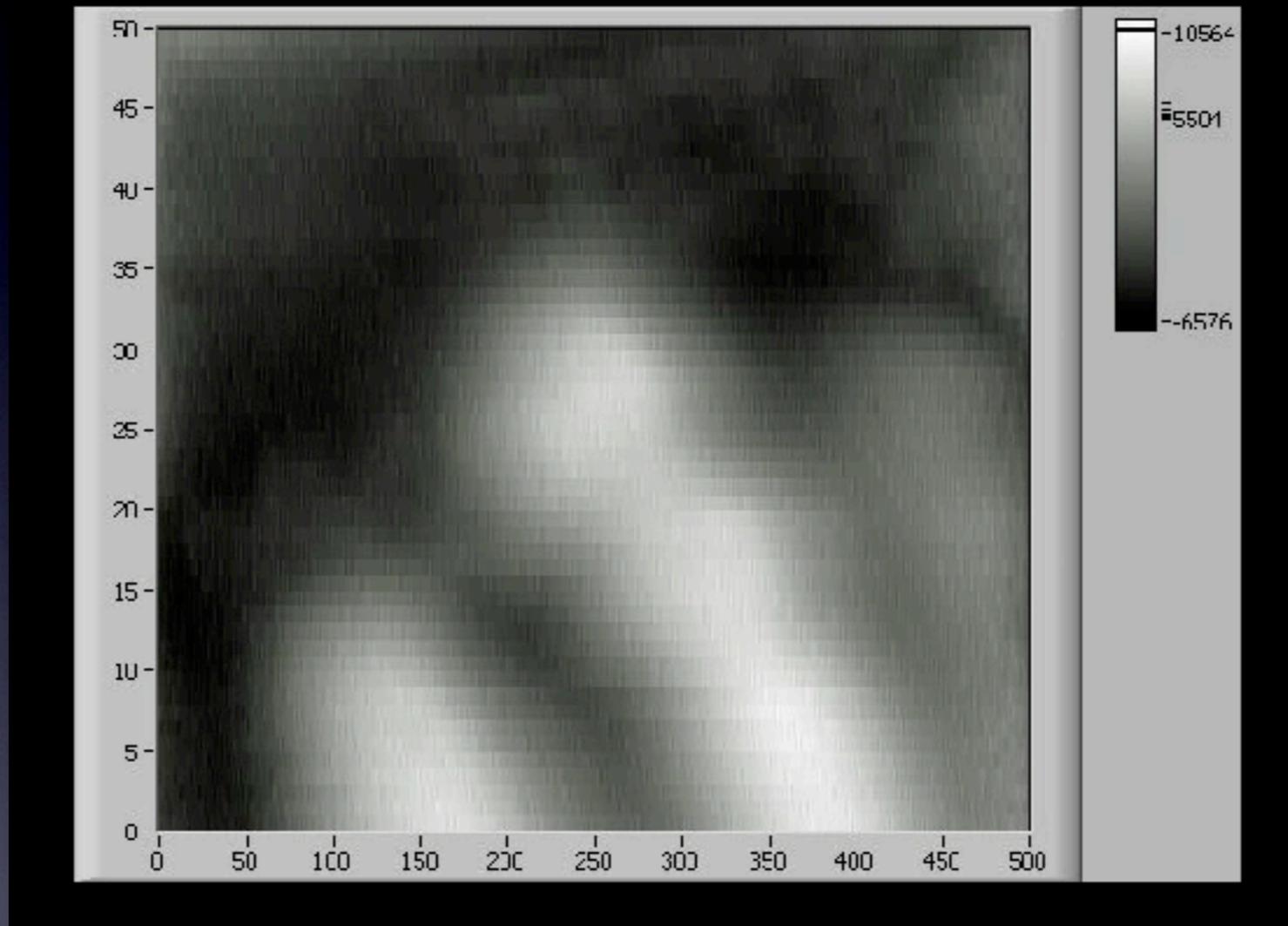
Human Chromosomes in 2mM NaCl buffer

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

~ 1 μm

Human Chromosomes in 2mM NaCl buffer

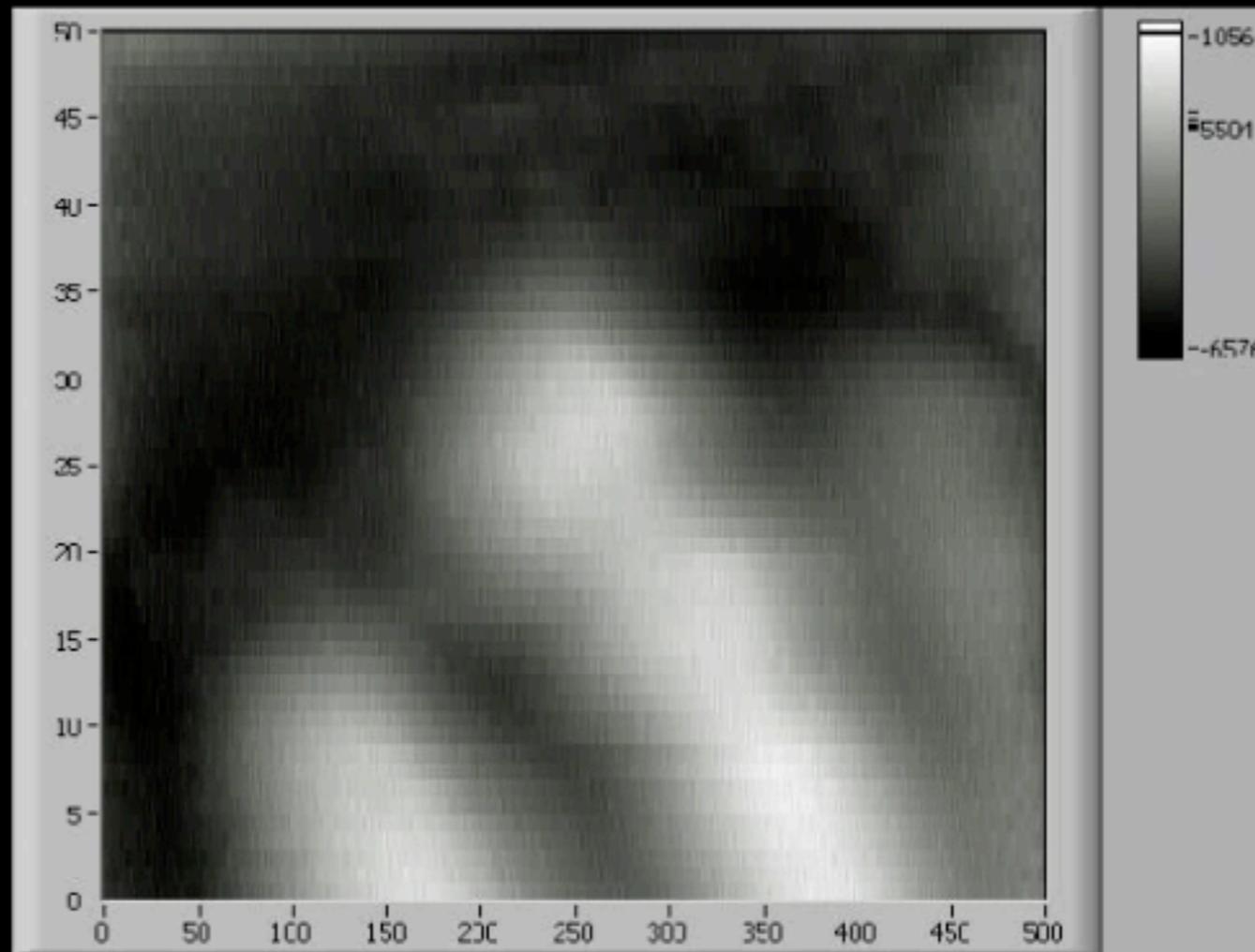
chromosomes: 1, 2, 7(?), 22



~ 1 μm

Human Chromosomes in 2mM NaCl buffer

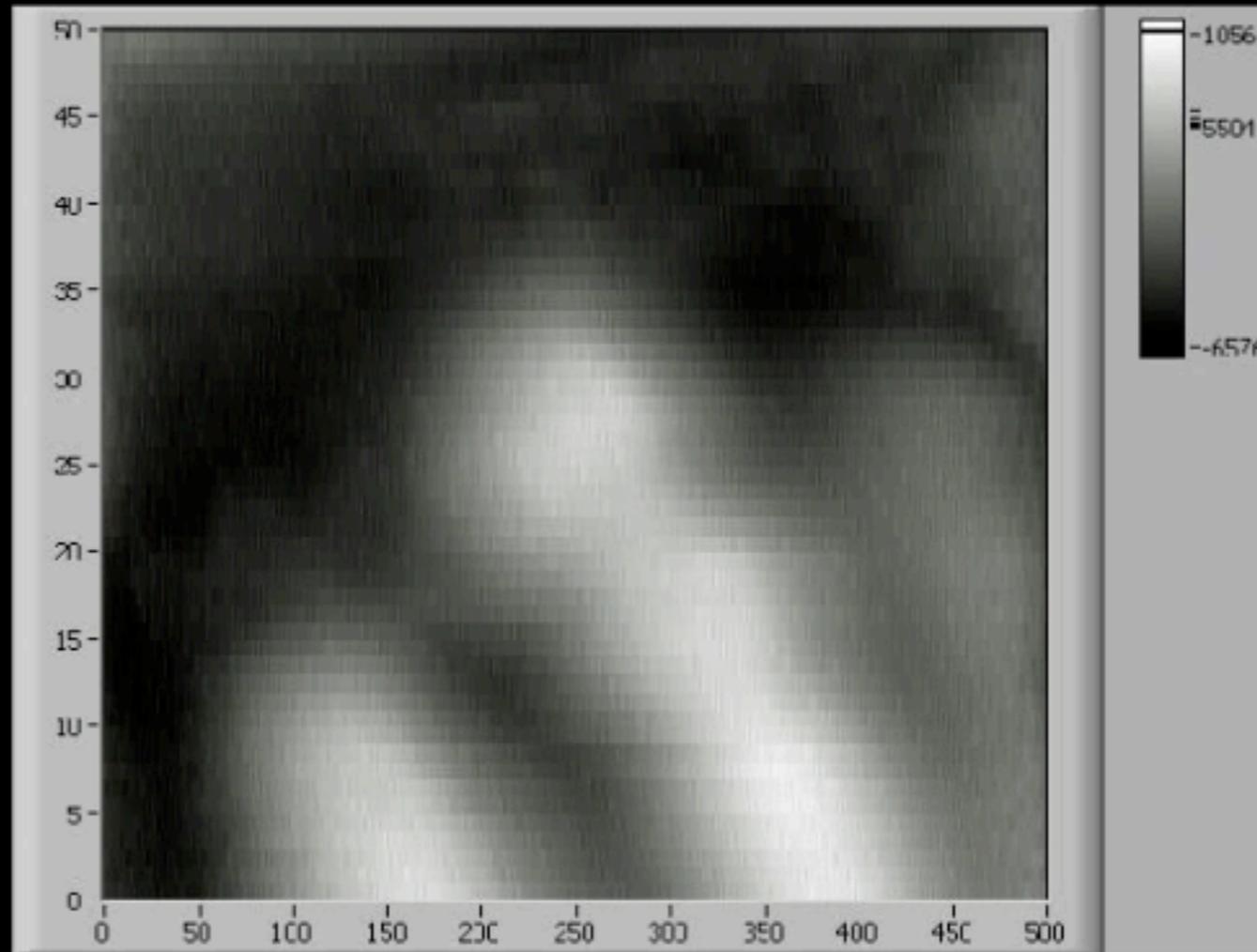
chromosomes: 1, 2, 7(?), 22



~ 1 μm

Human Chromosomes in 2mM NaCl buffer

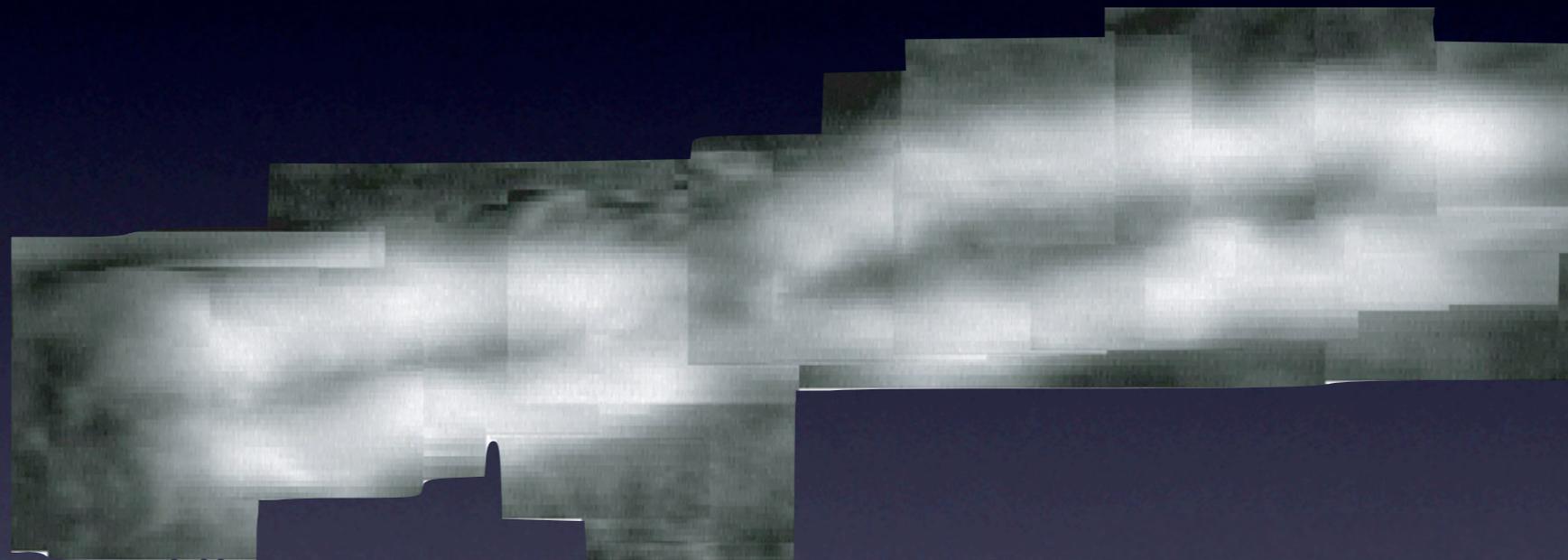
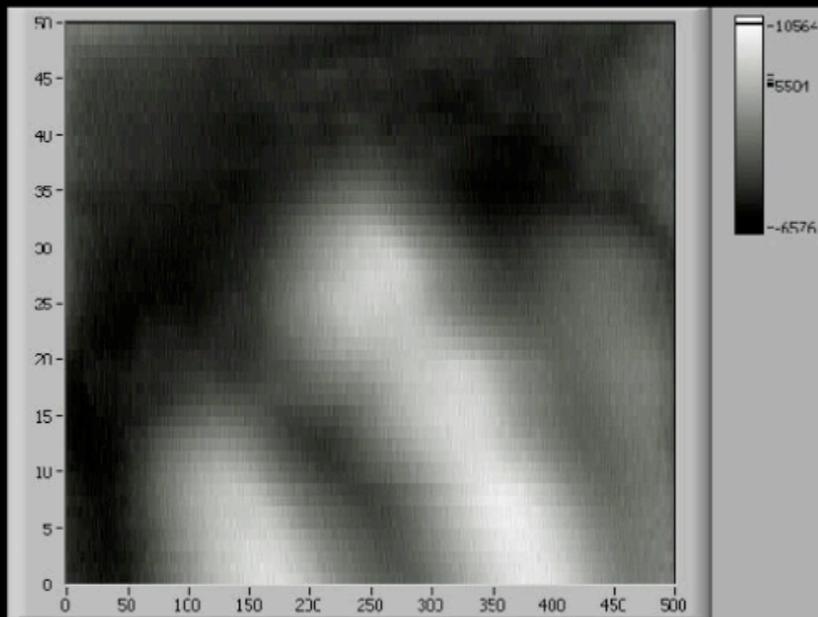
chromosomes: 1, 2, 7(?), 22



~ 1 μm

Human Chromosomes in 2mM NaCl buffer

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

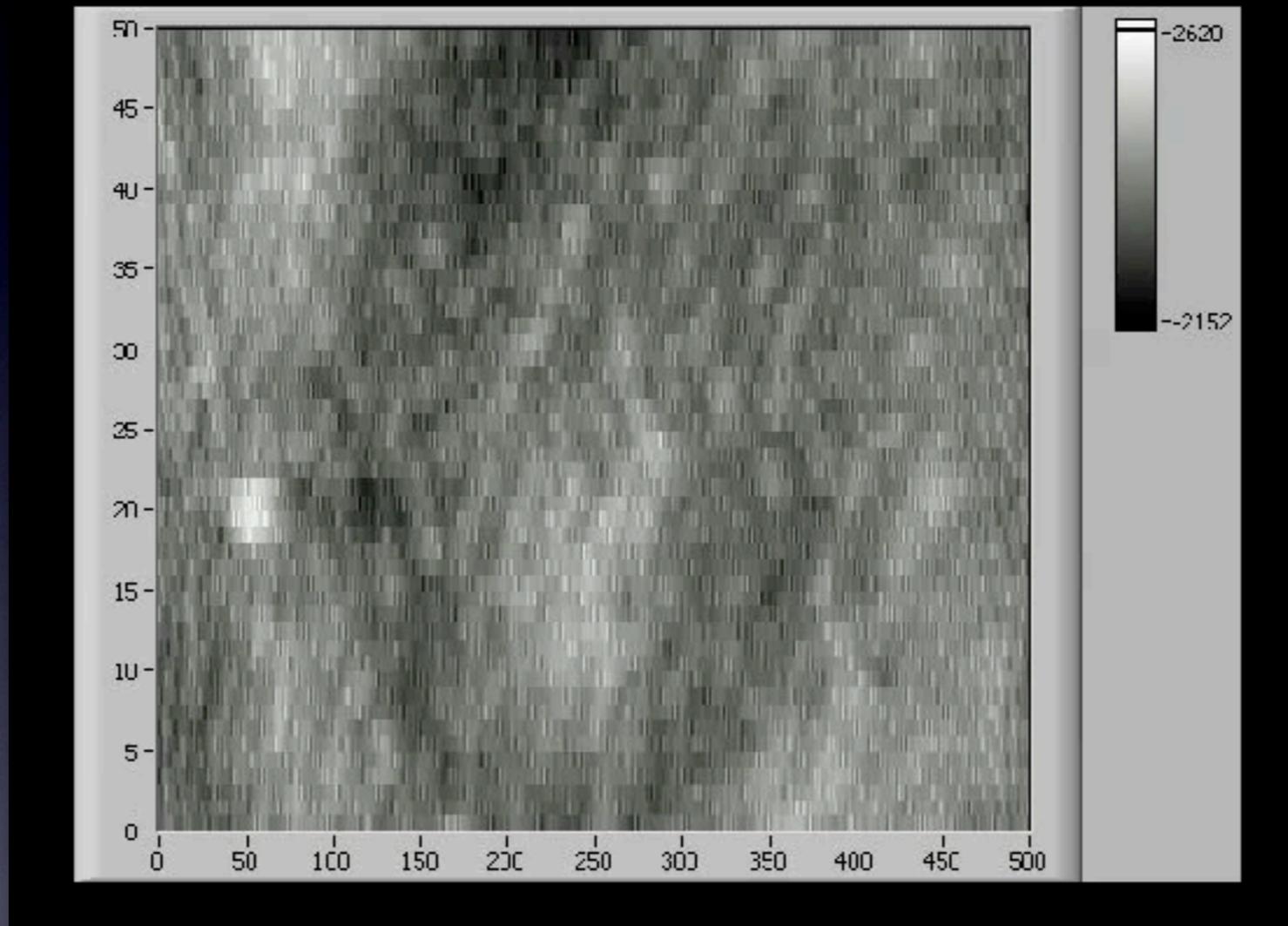


~ 1 μm

Video AFM Human Chromosome - higher mag

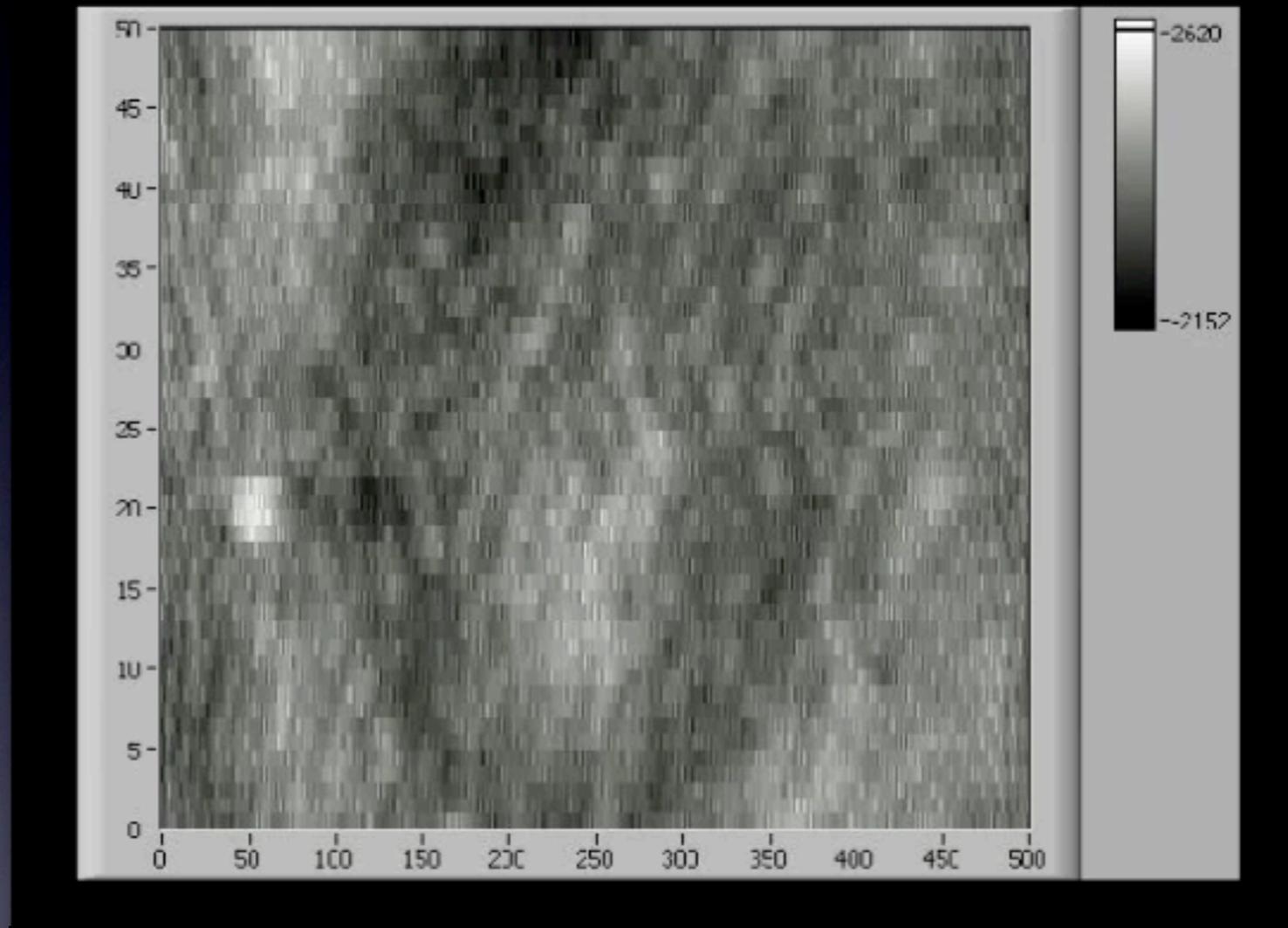
Picco, Dunton, Ulcinas, Engledew, Hoshi Ushiki, Miles, *Nanotechnology* **19** (2008) 384018

Video AFM Human Chromosome - higher mag



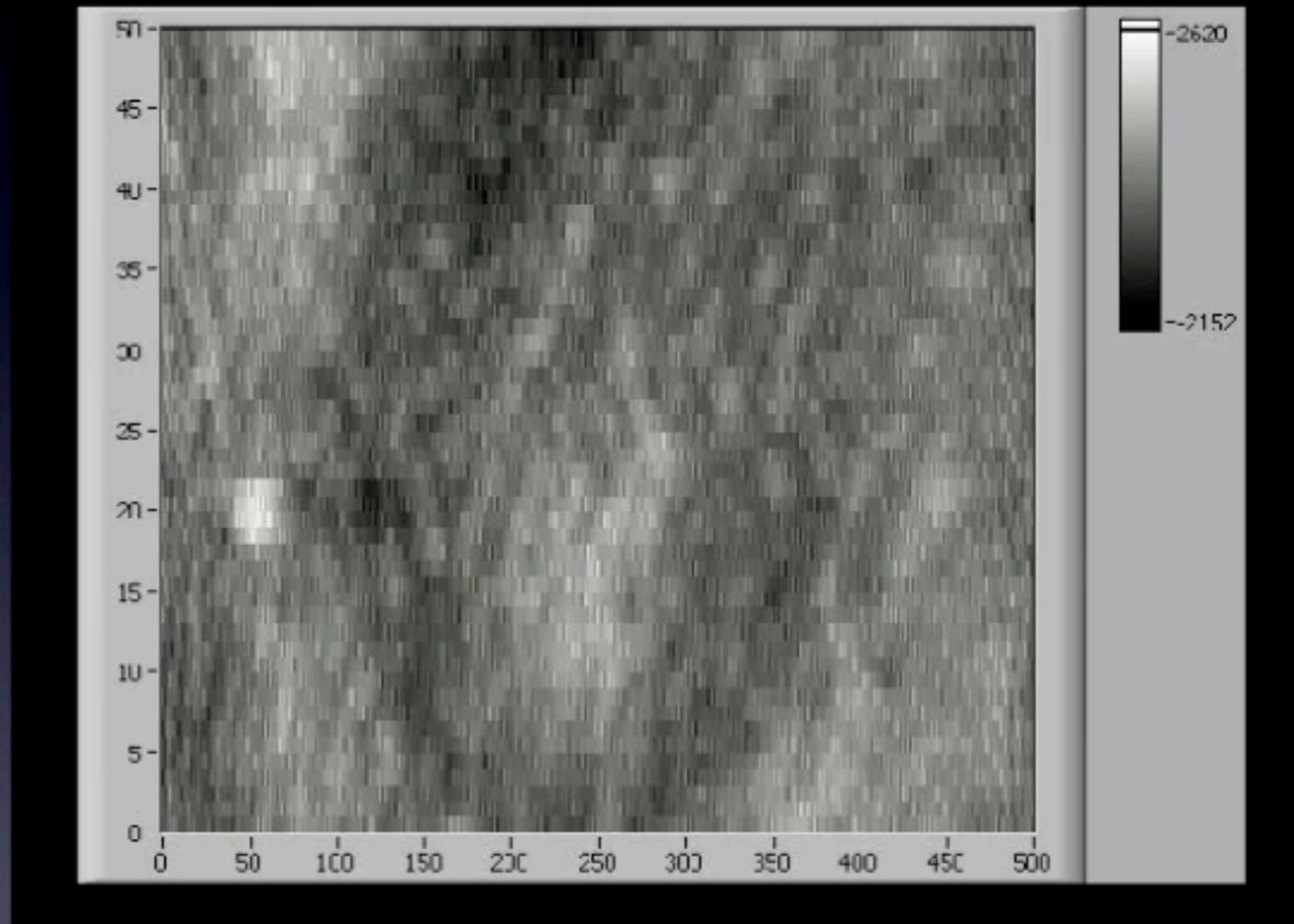
Picco, Dunton, Ulcinas, Engledew, Hoshi Ushiki, Miles, *Nanotechnology* **19** (2008) 384018

Video AFM Human Chromosome - higher mag



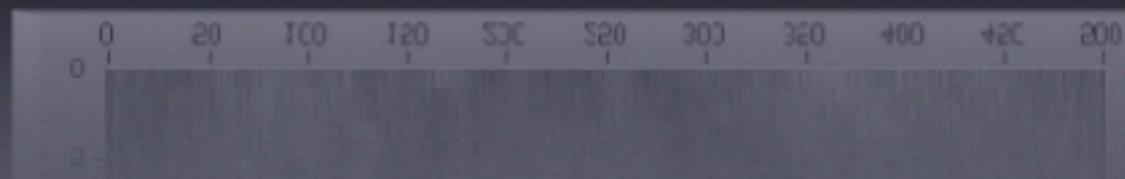
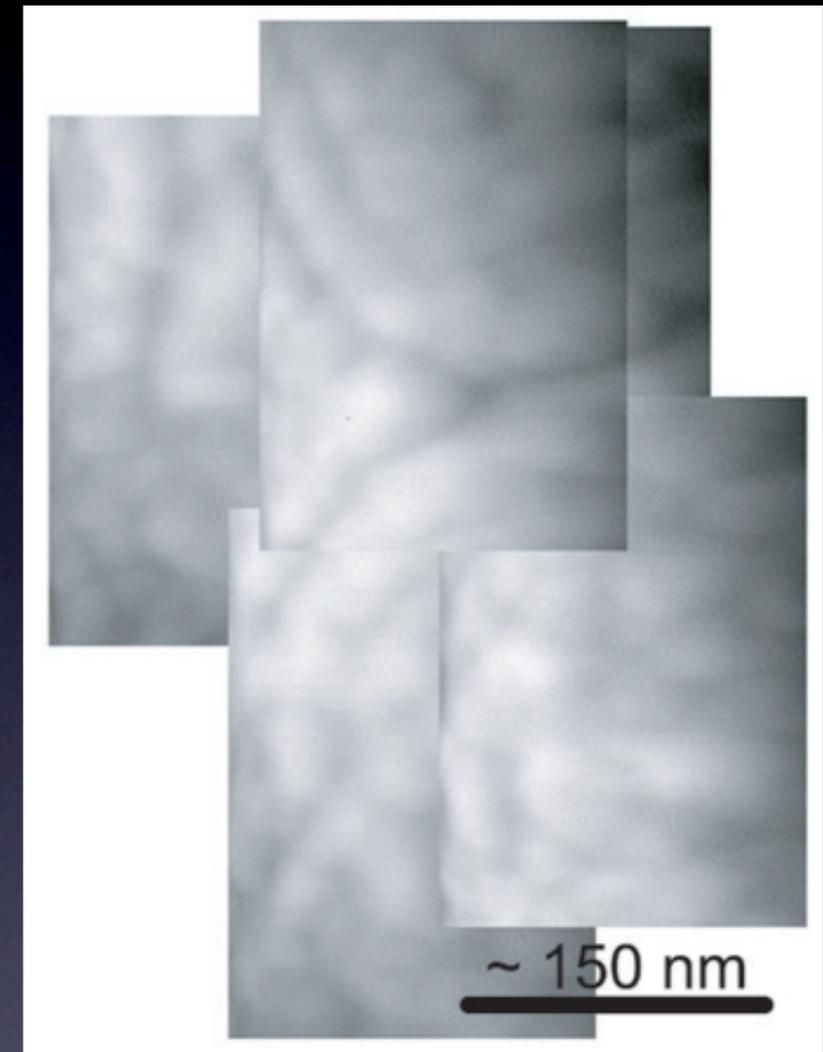
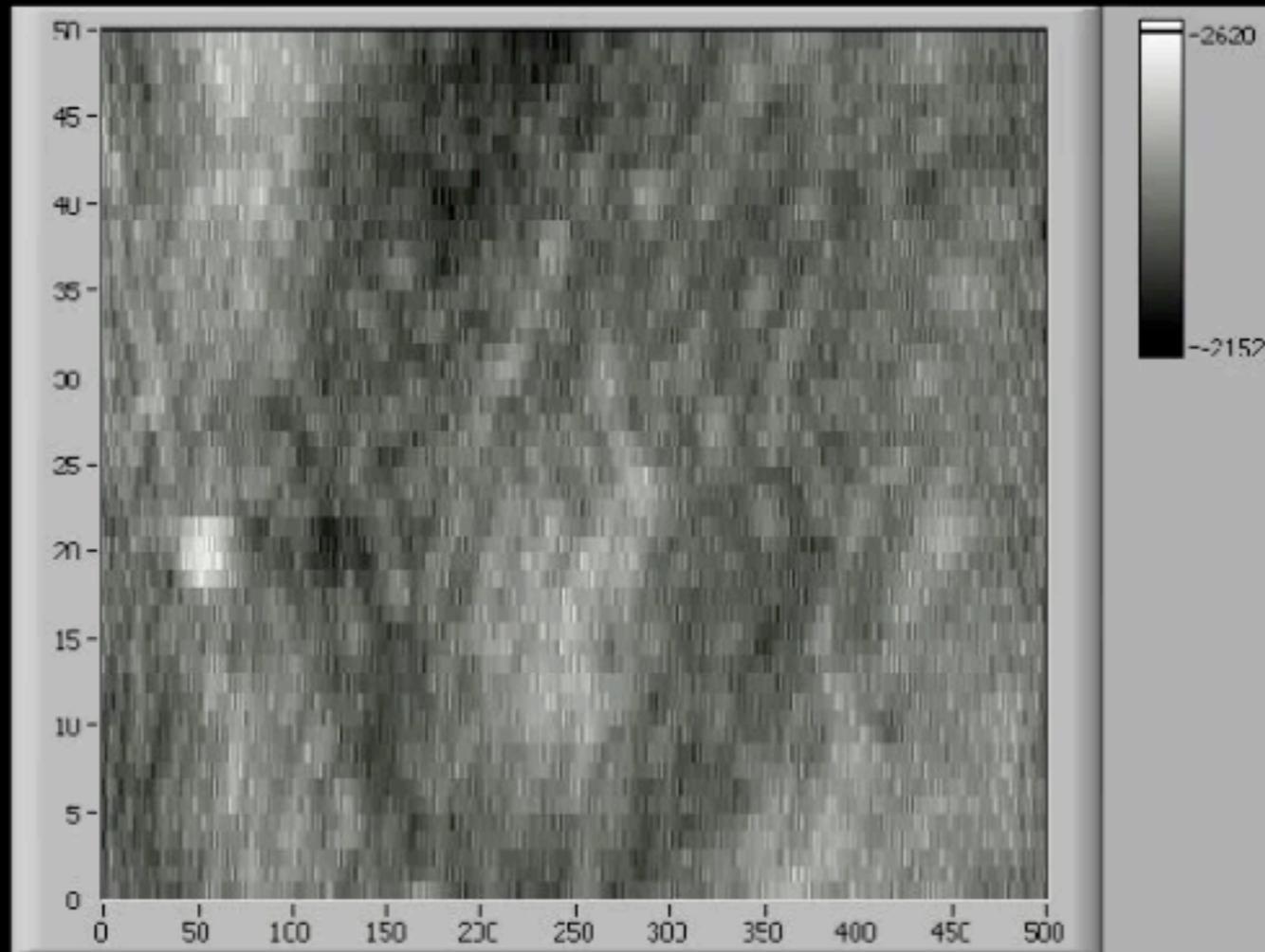
Picco, Dunton, Ulcinas, Engledew, Hoshi Ushiki, Miles, *Nanotechnology* **19** (2008) 384018

Video AFM Human Chromosome - higher mag



Picco, Dunton, Ulcinas, Engledew, Hoshi Ushiki, Miles, *Nanotechnology* **19** (2008) 384018

Video AFM Human Chromosome - higher mag



Dissolution of bovine dental enamel

citric acid

pH 4.0

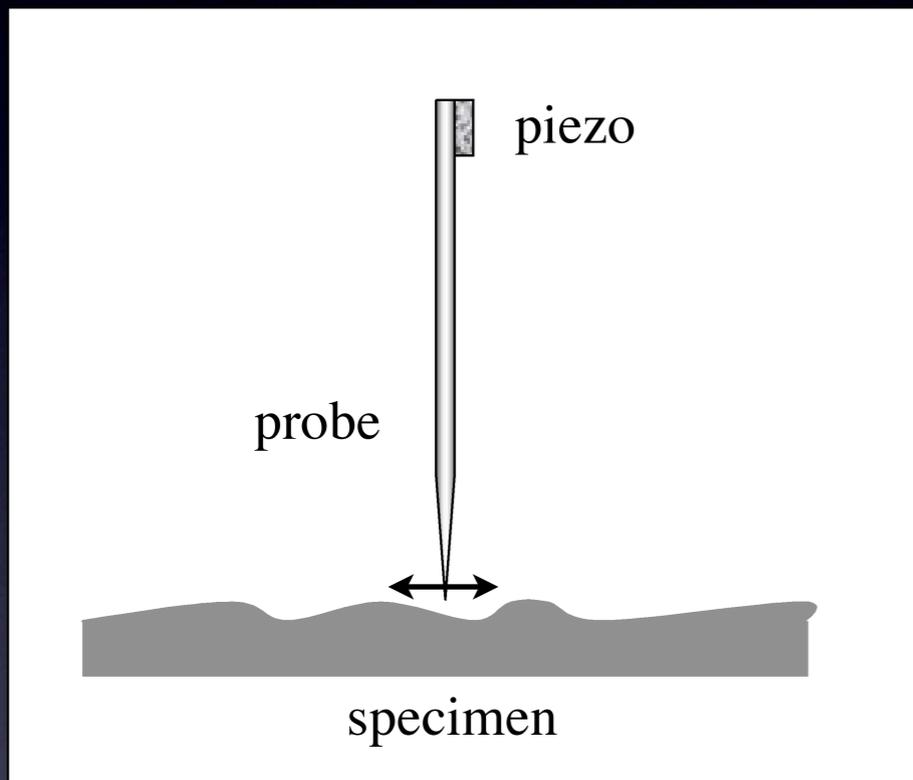
Dissolution of bovine dental enamel

citric acid

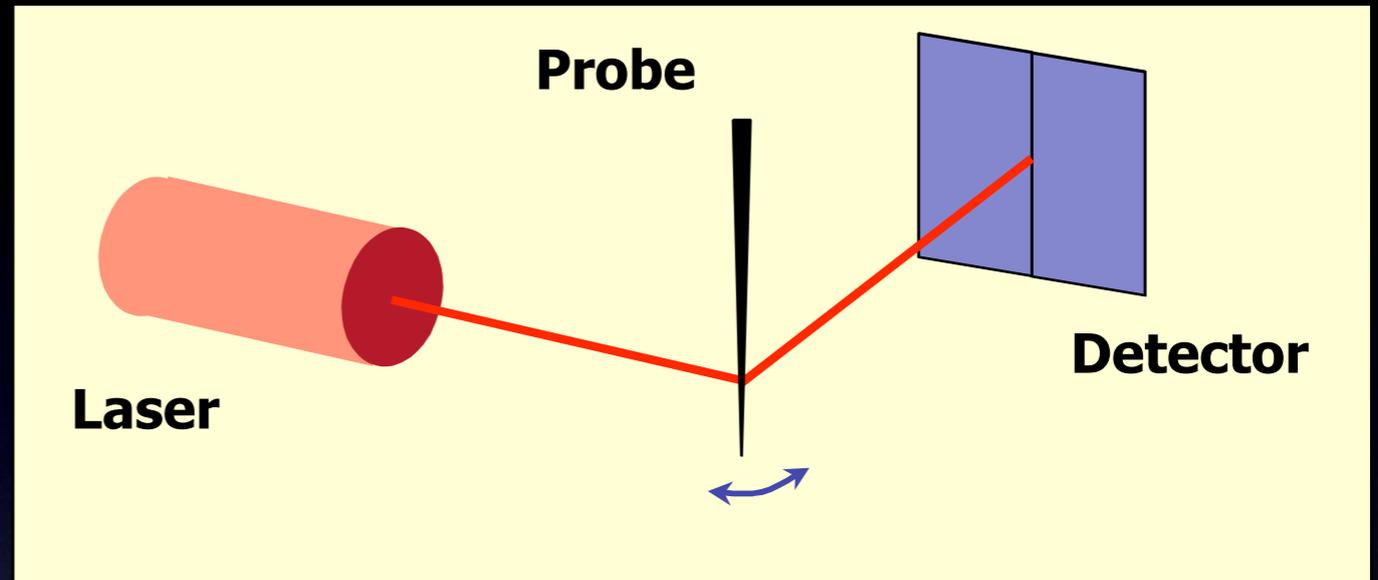
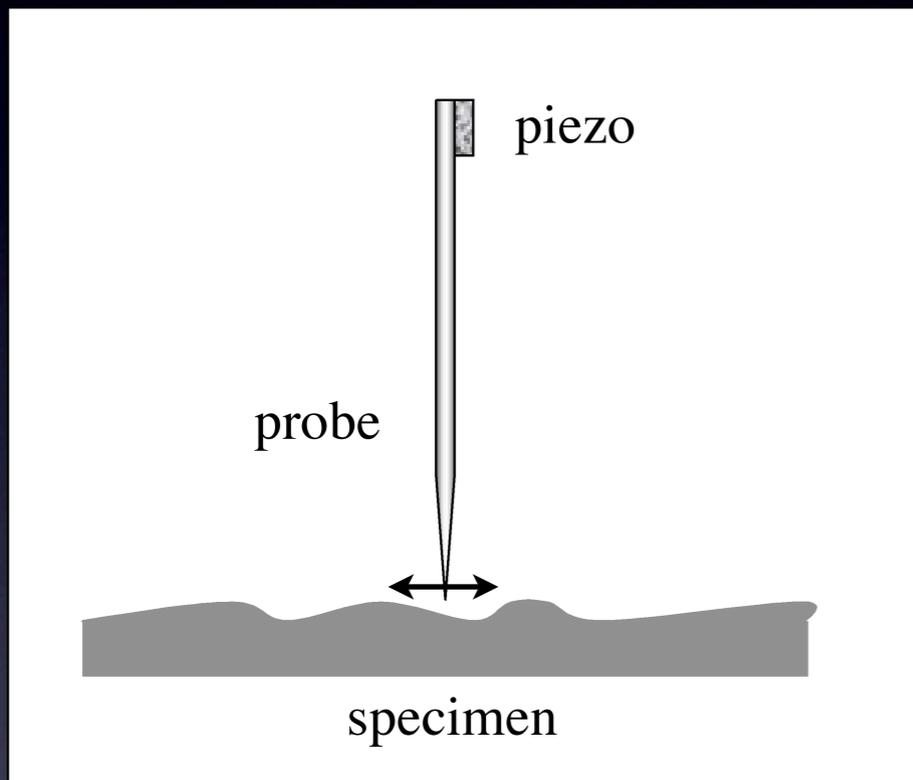
pH 4.0

High-speed Transverse Dynamic Force Microscope (TDFM)

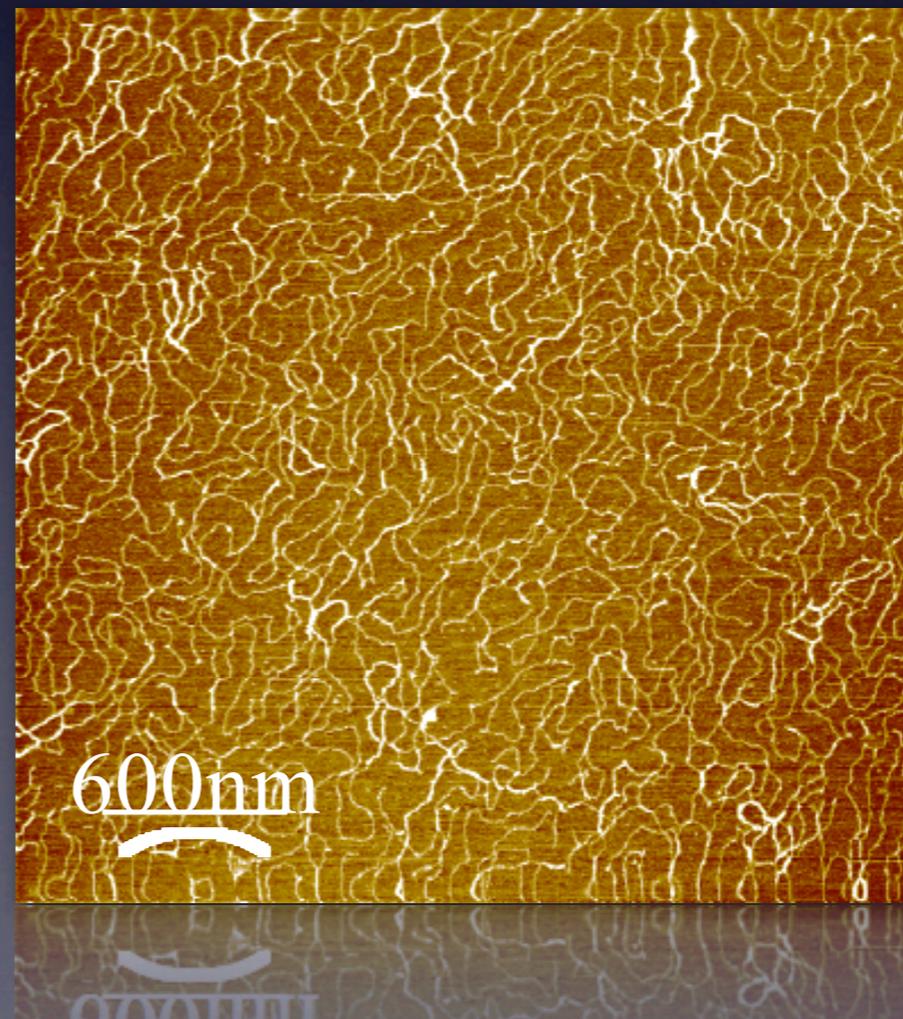
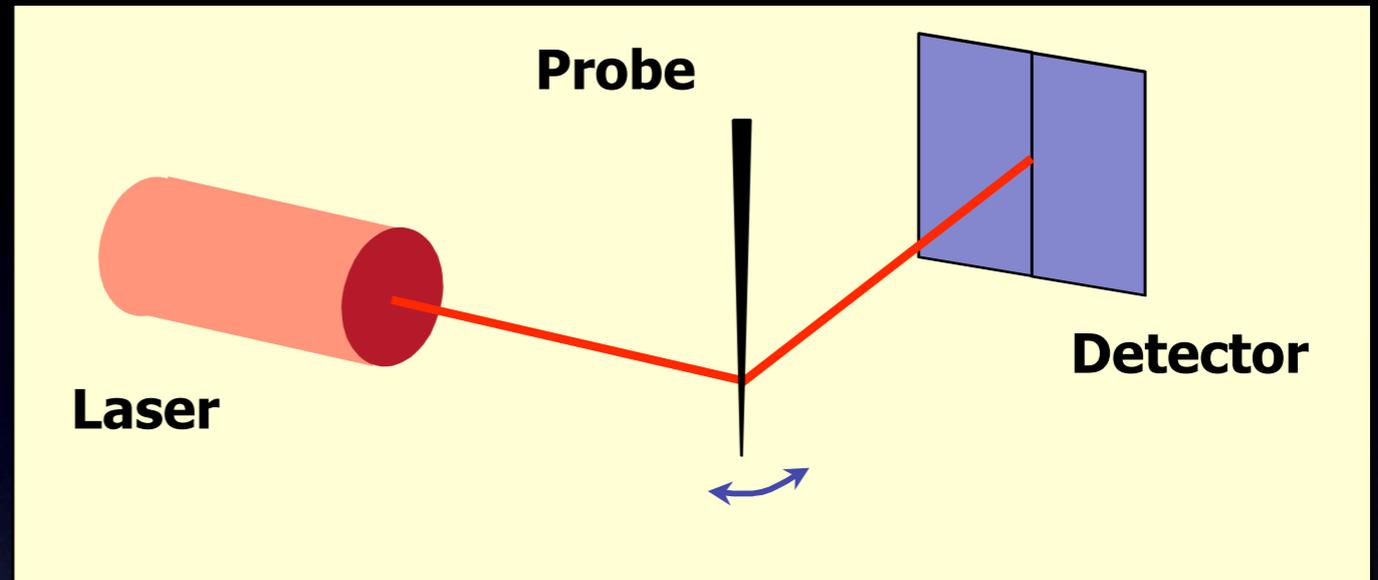
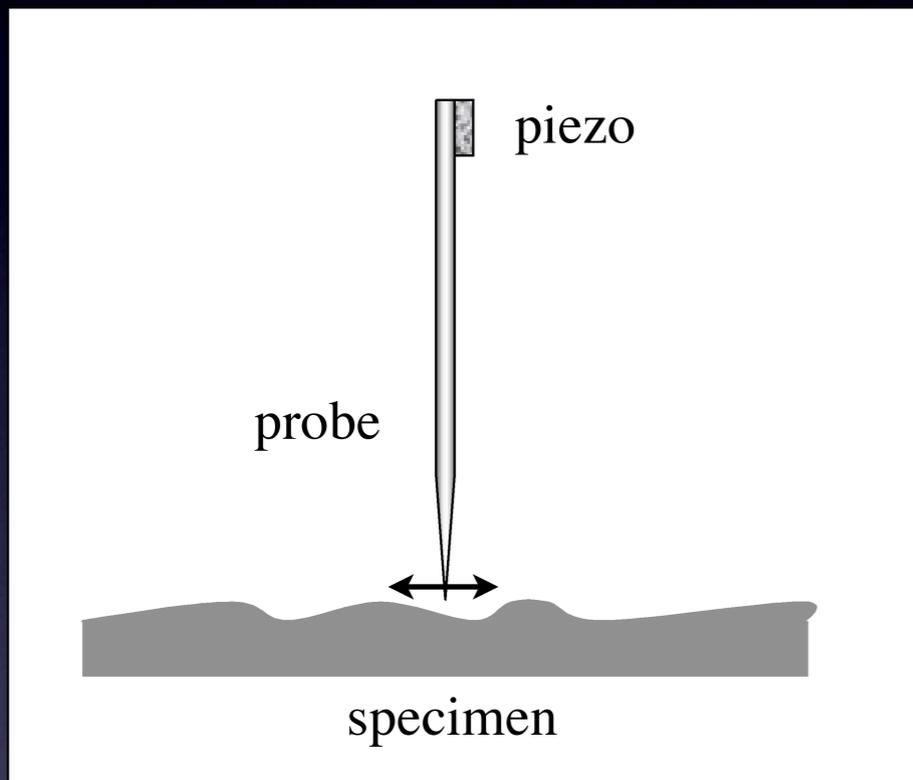
Conventional Transverse Dynamic Force Microscopy



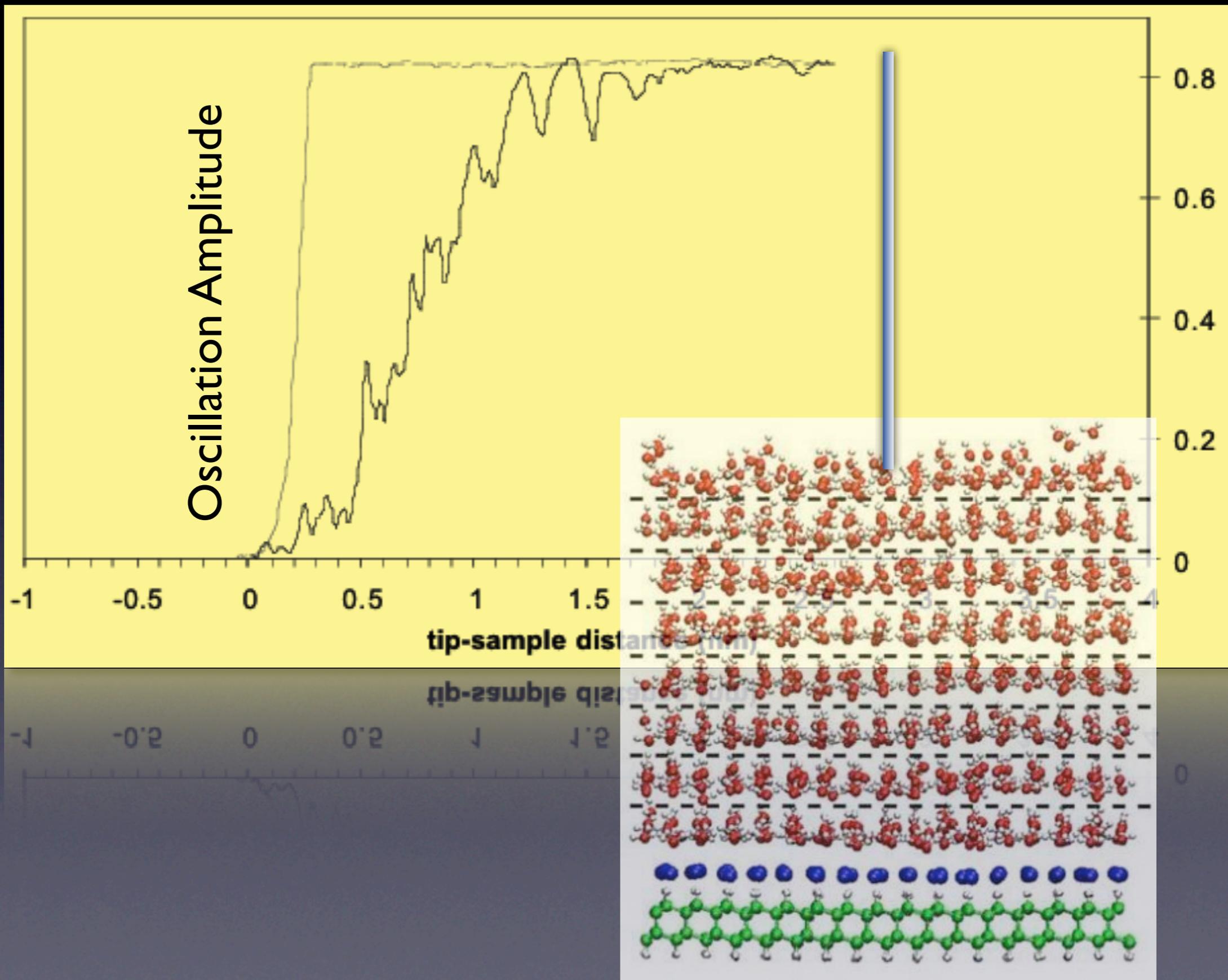
Conventional Transverse Dynamic Force Microscopy



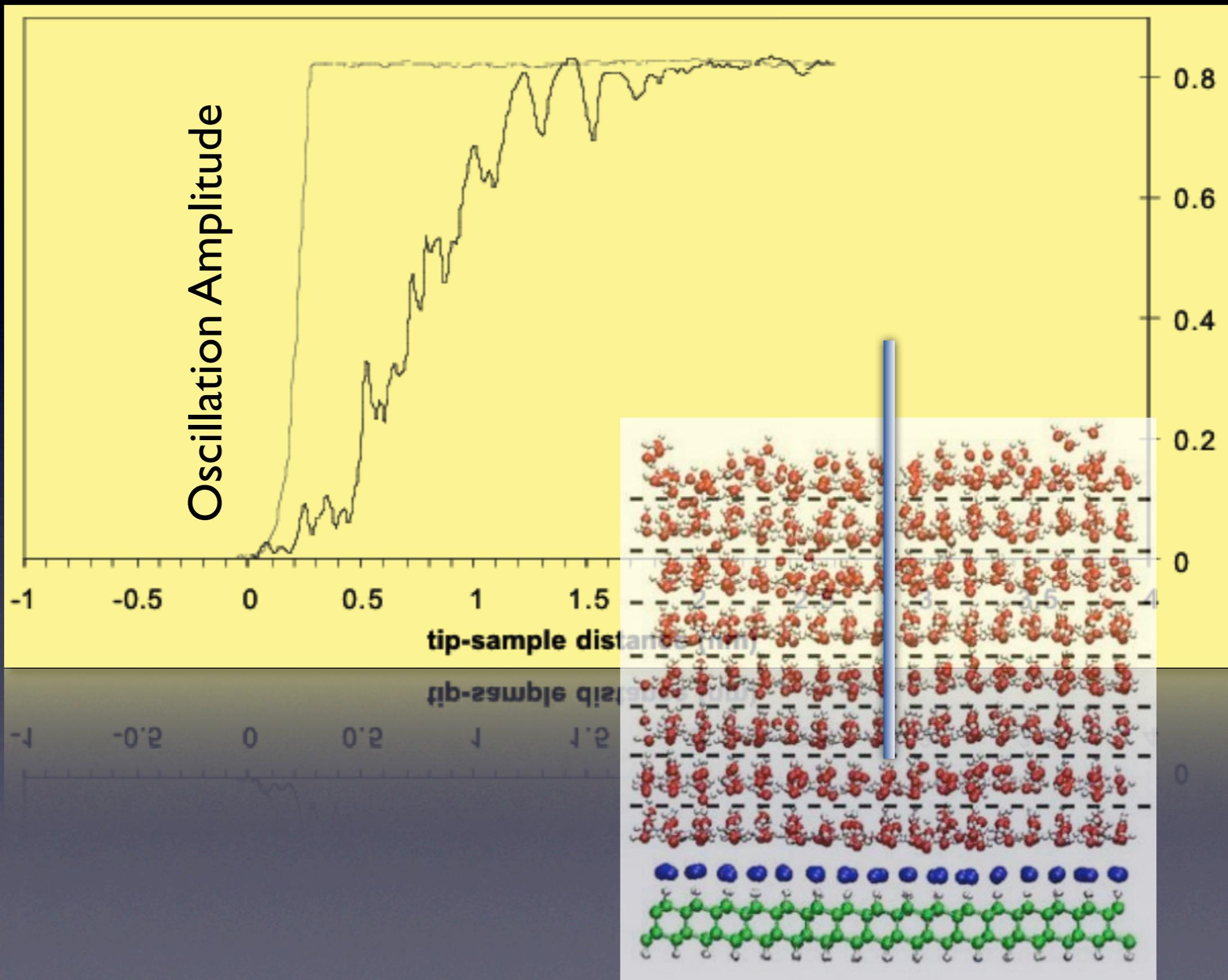
Conventional Transverse Dynamic Force Microscopy



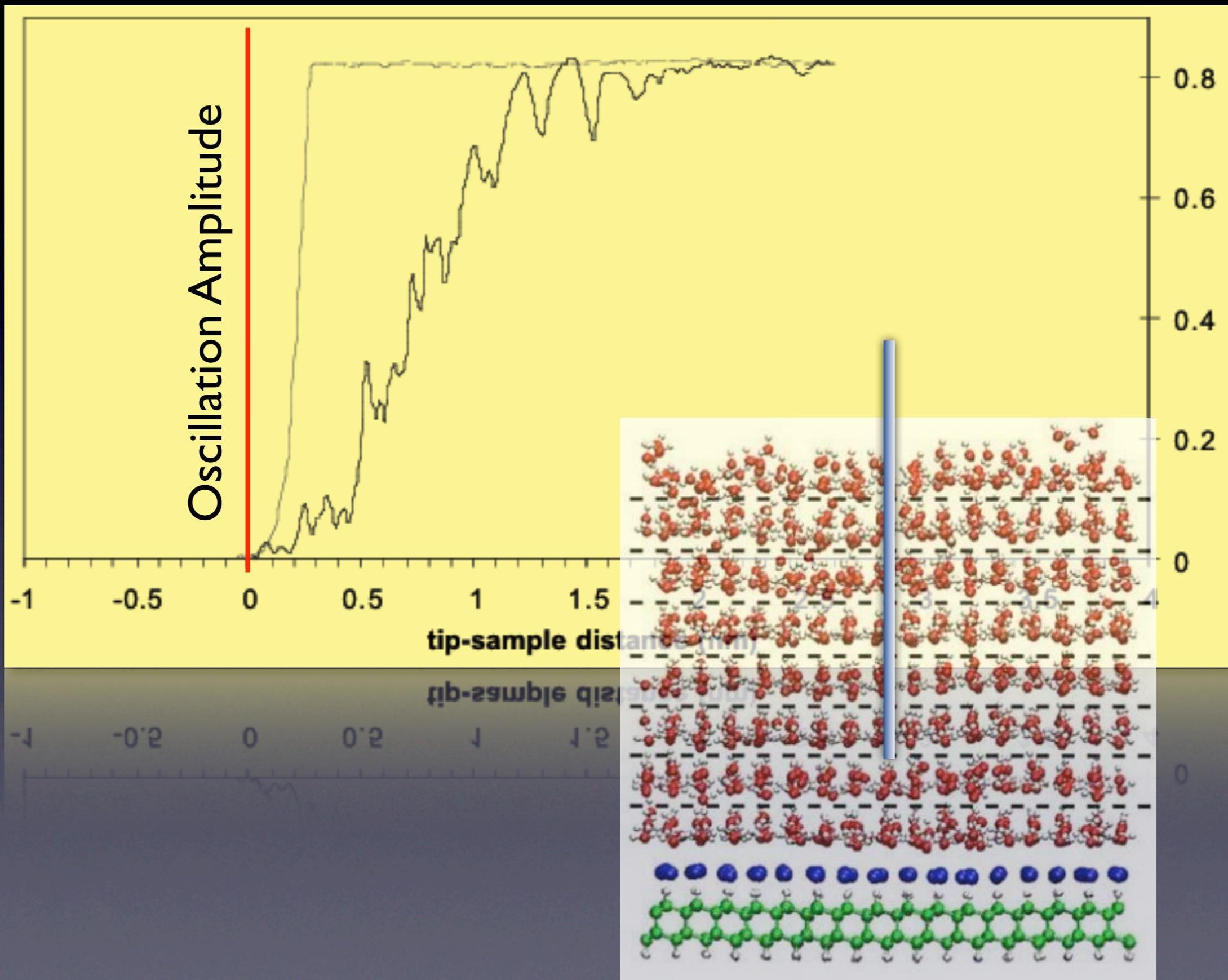
TDFM approach curve - water layers



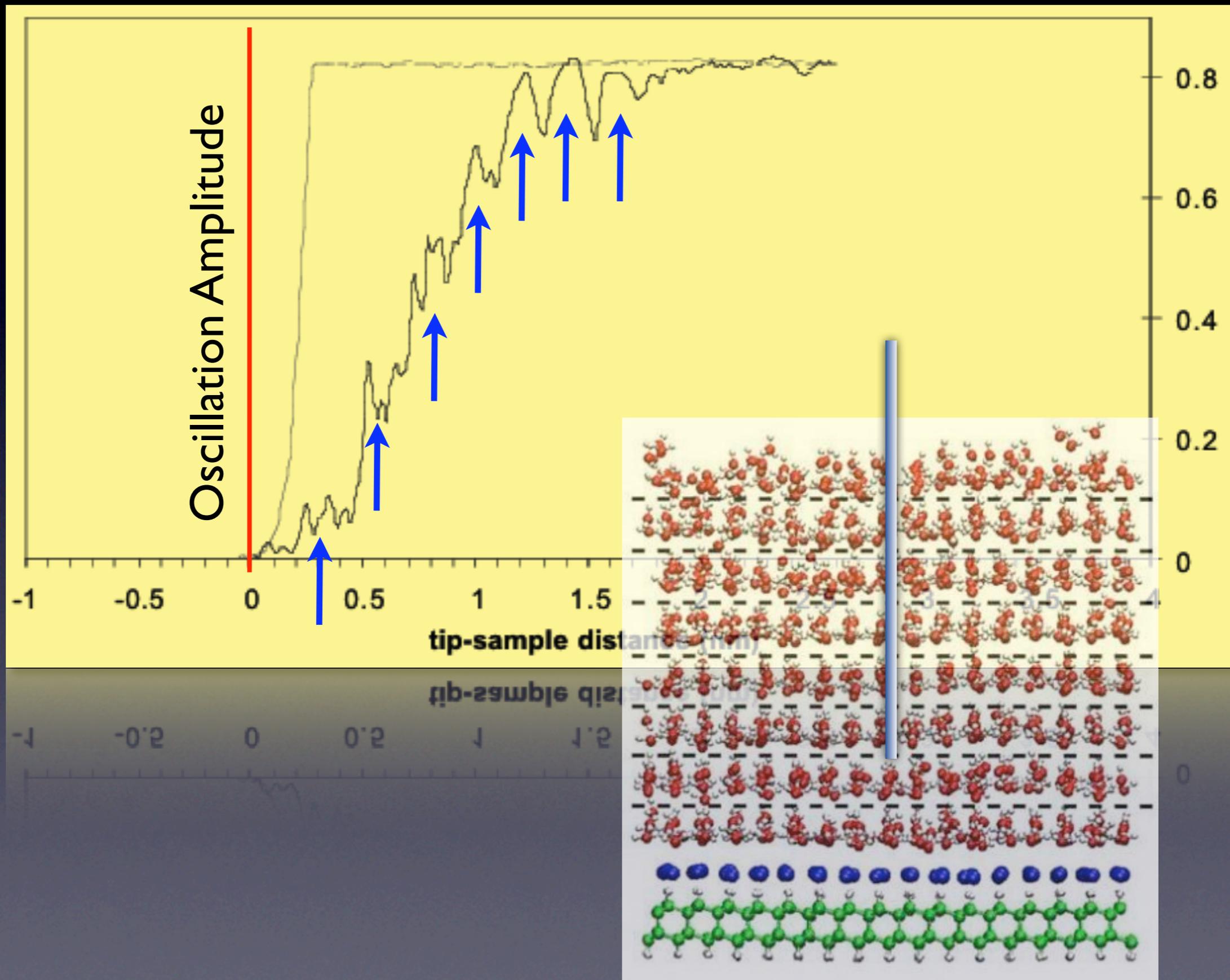
TDFM approach curve - water layers



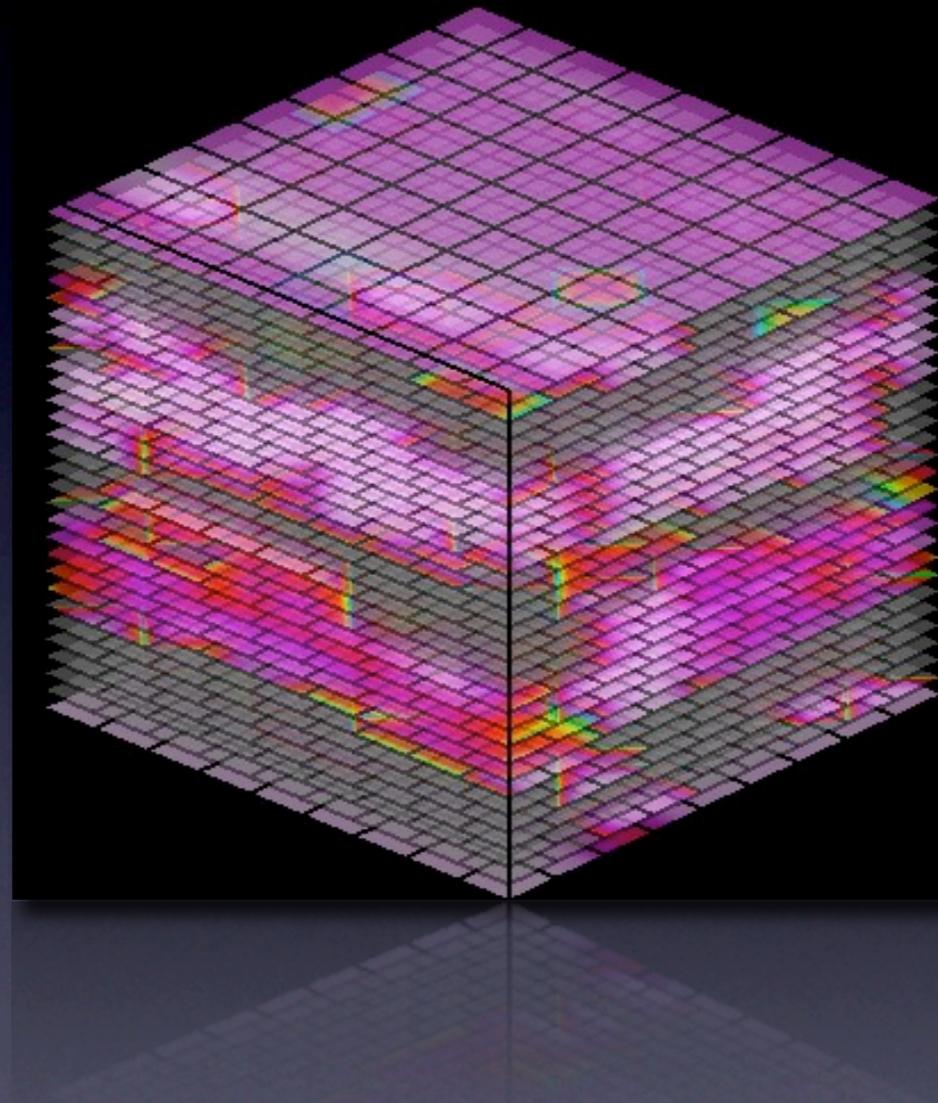
TDFM approach curve - water layers



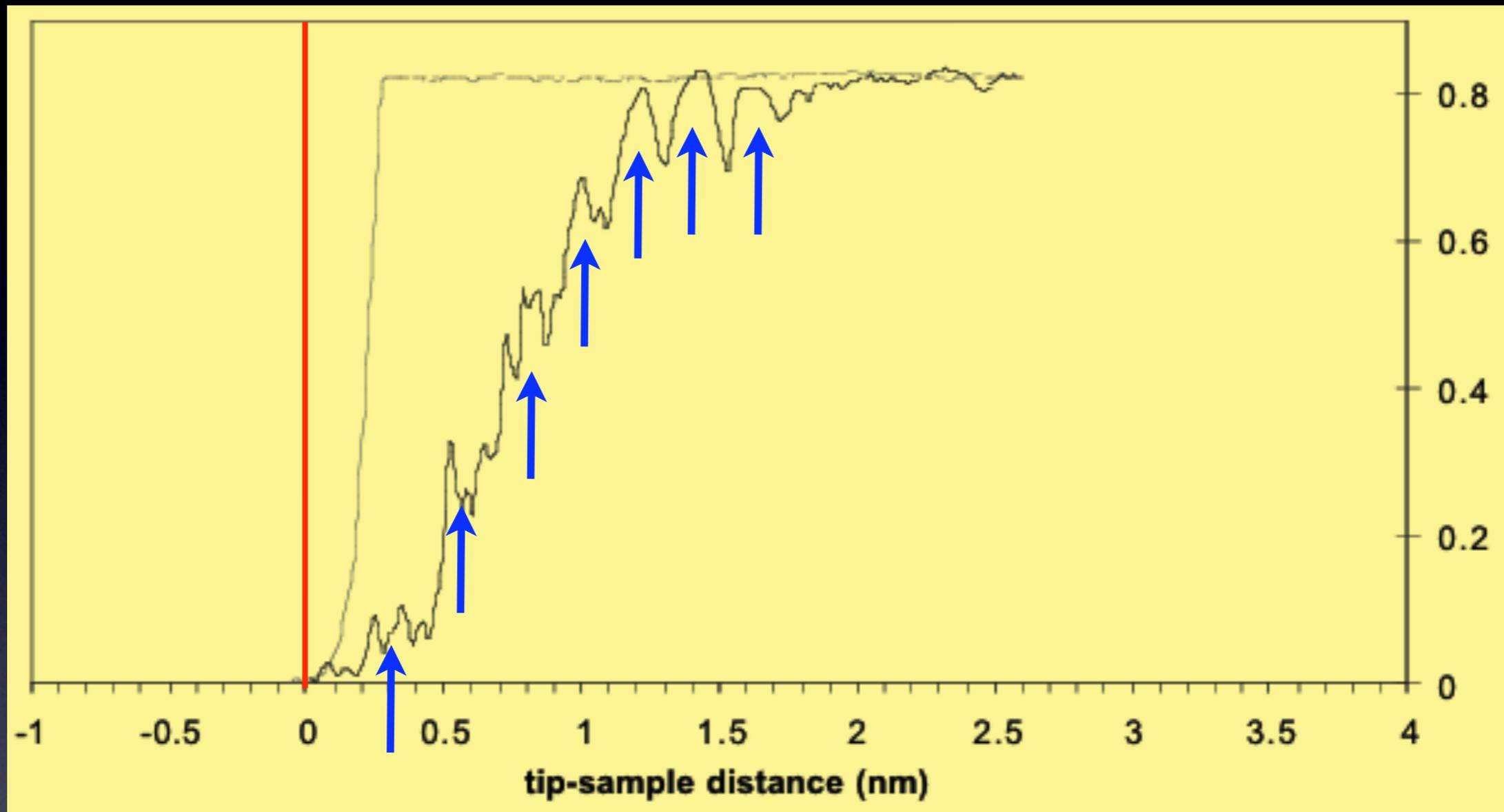
TDFM approach curve - water layers



3D Mapping of Water Layers above a Surface



TDFM approach curve - water layers

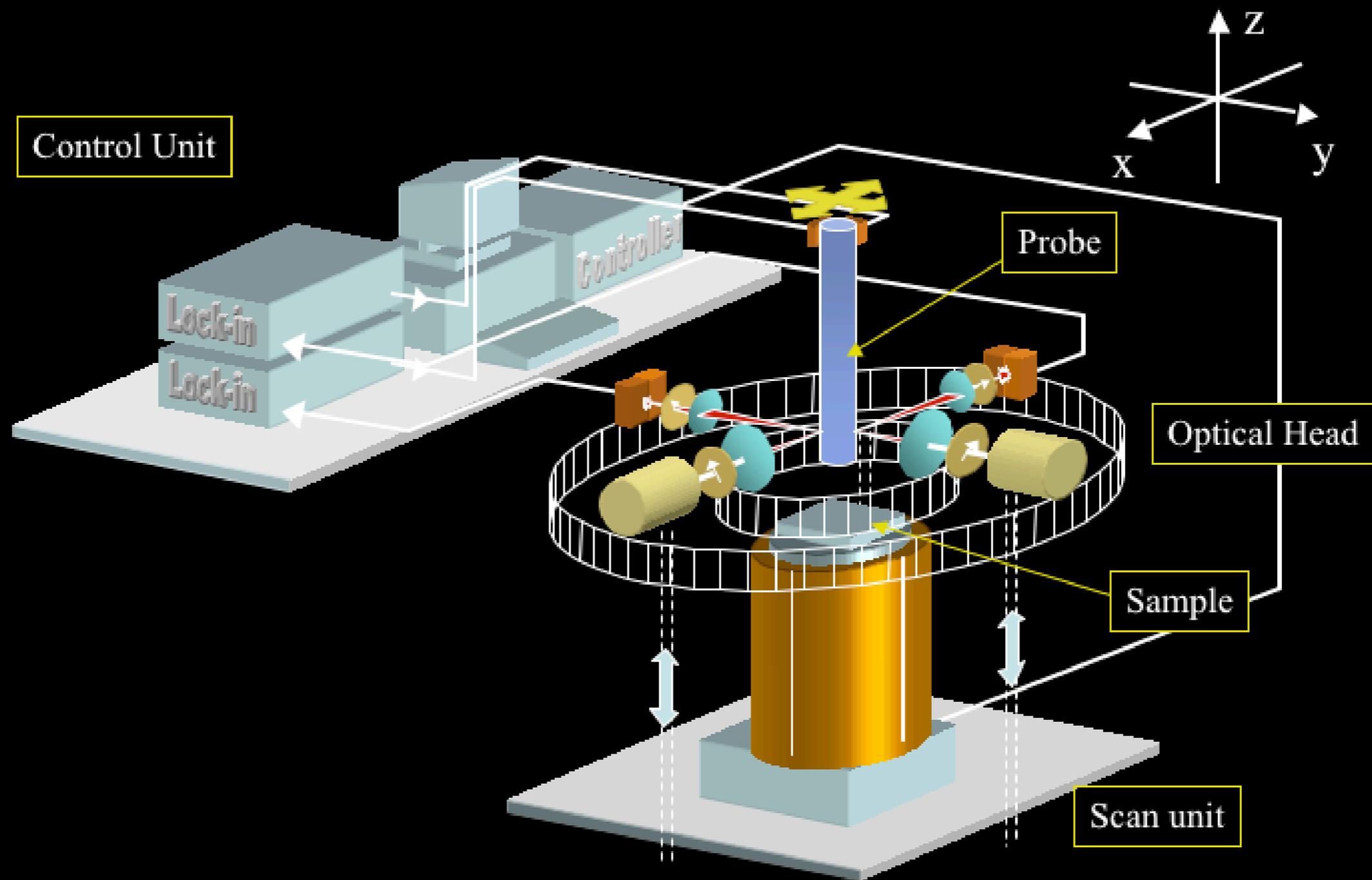


tip-sample distance (nm)

-1 -0.5 0 0.5 1 1.5 2 2.5 3 3.5 4

0

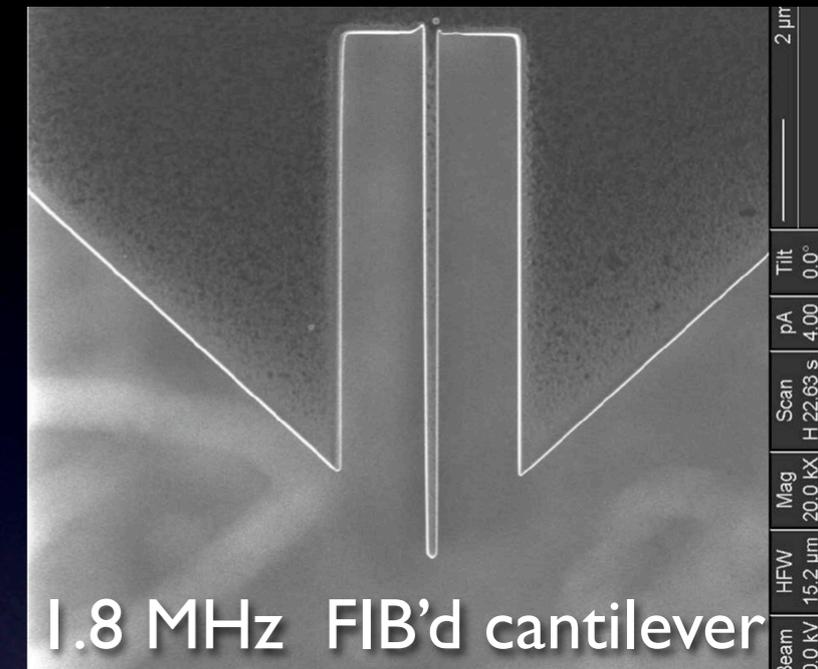
Conventional TDFM



High-speed Non-contact TDFM

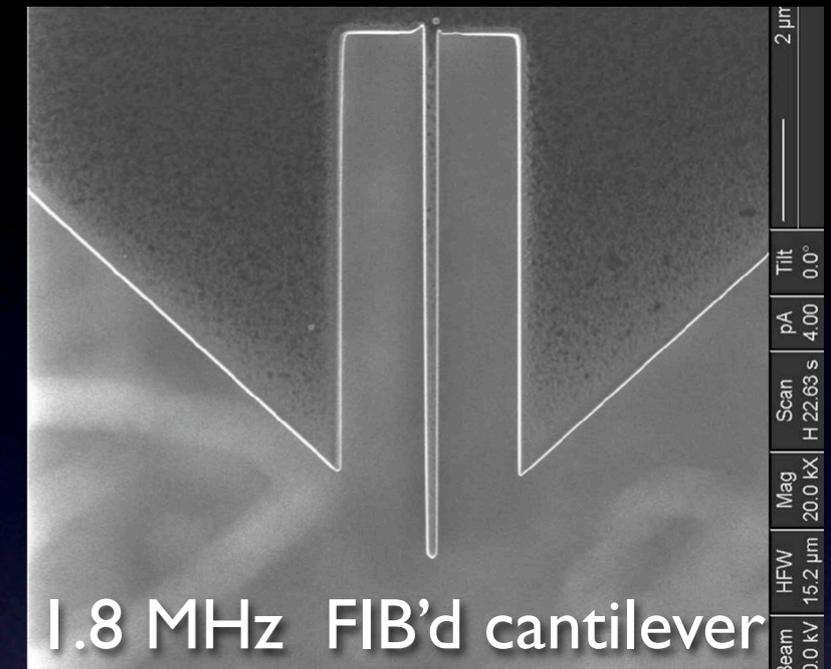
Why is TDFM a good candidate for High-speed?

- ★ 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



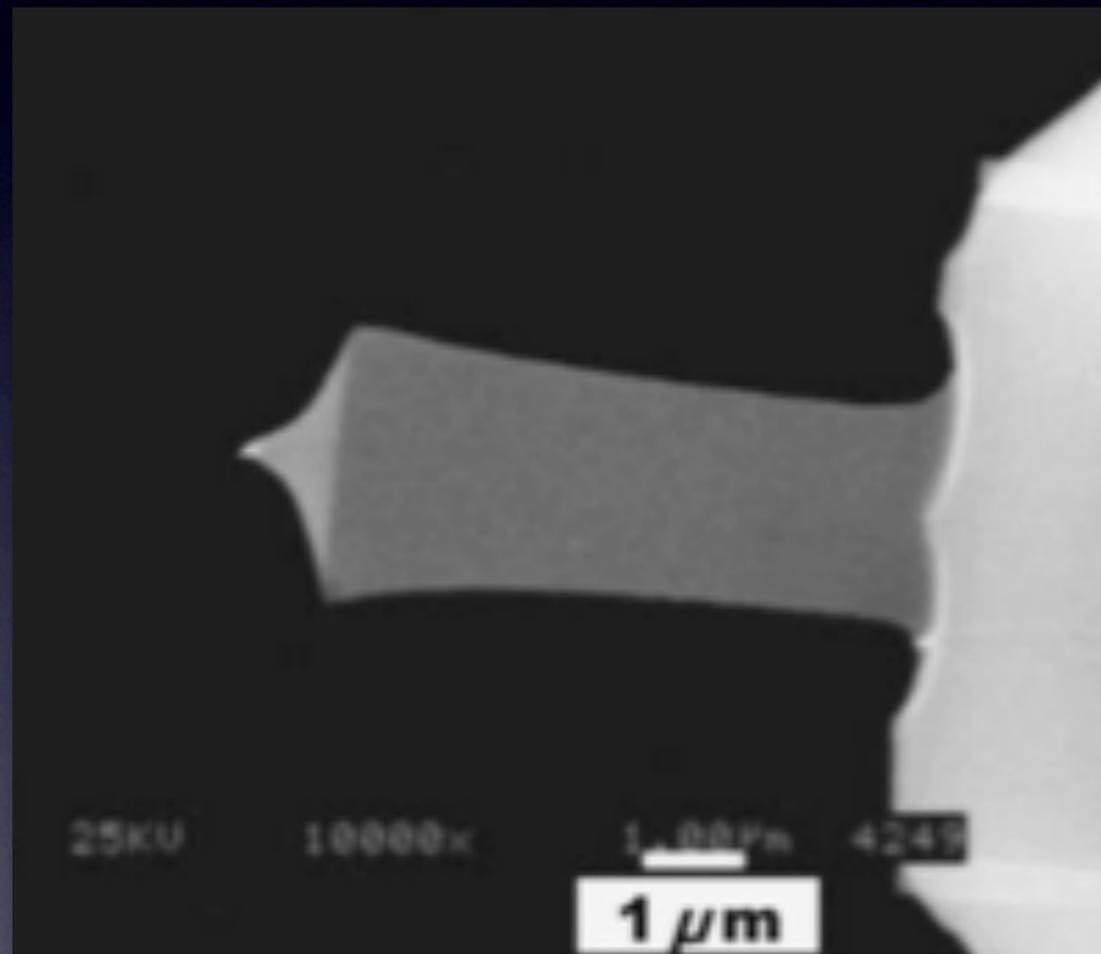
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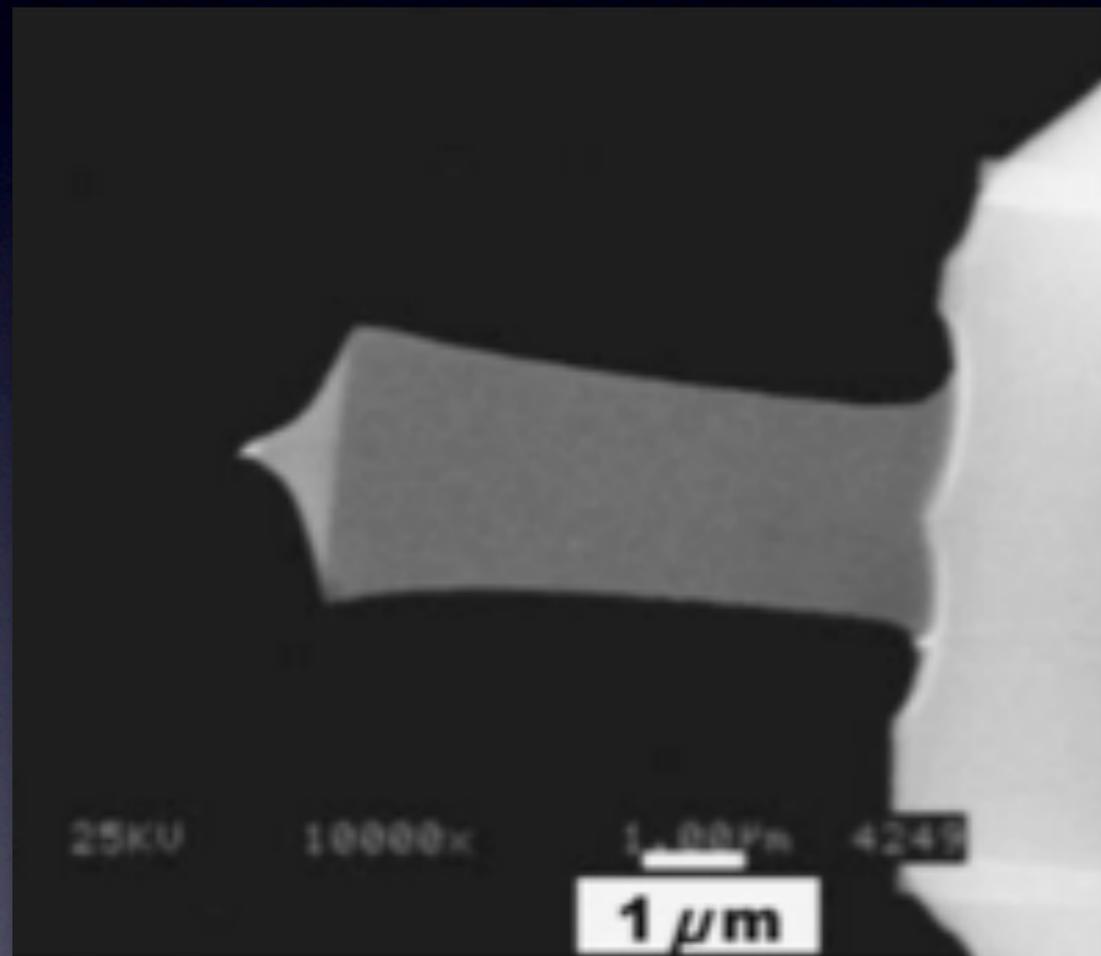


Tips: etched; cut; FIB; CNT; etc. from silicon, glass, metal, polymer, nanowires, ...

Compare with problems making and using small cantilevers

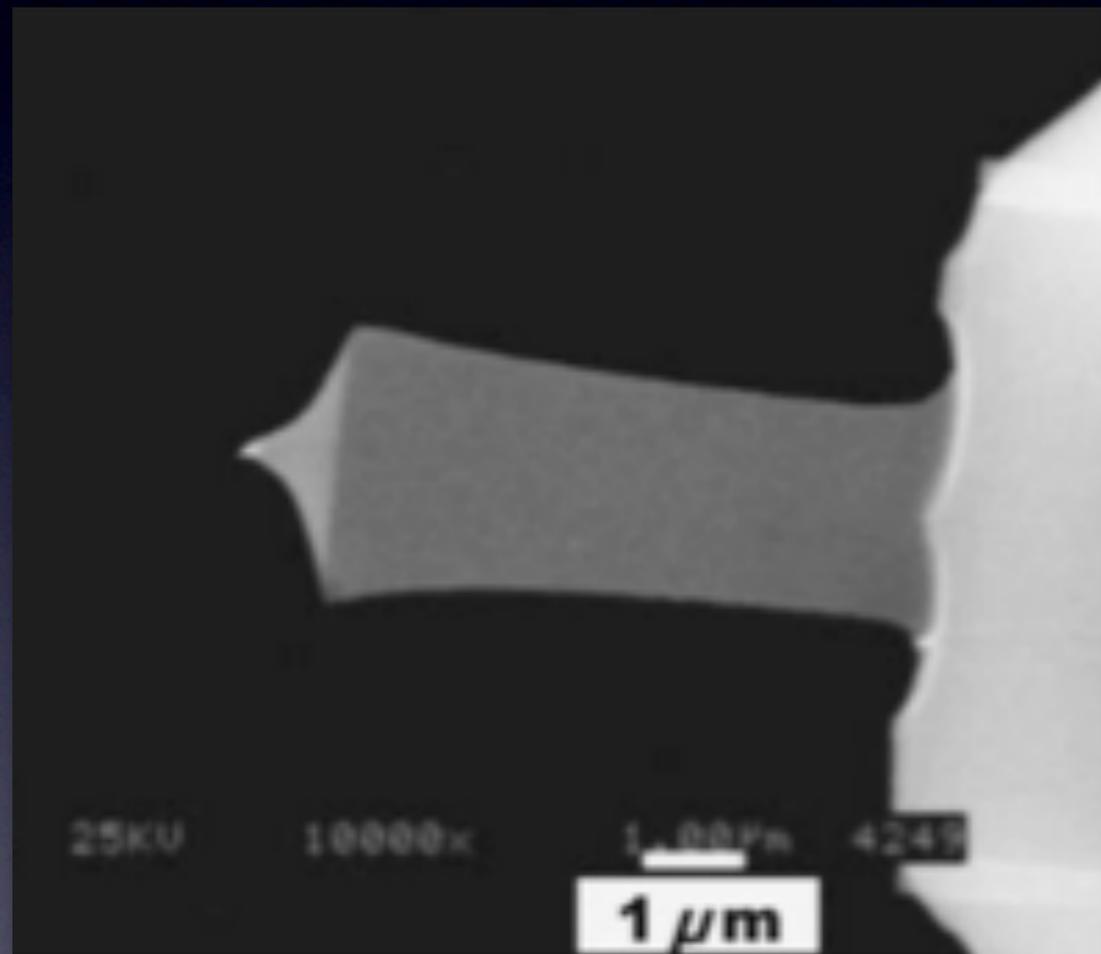


Compare with problems making and using small cantilevers



Difficult to fabricate these small cantilevers reproducibly

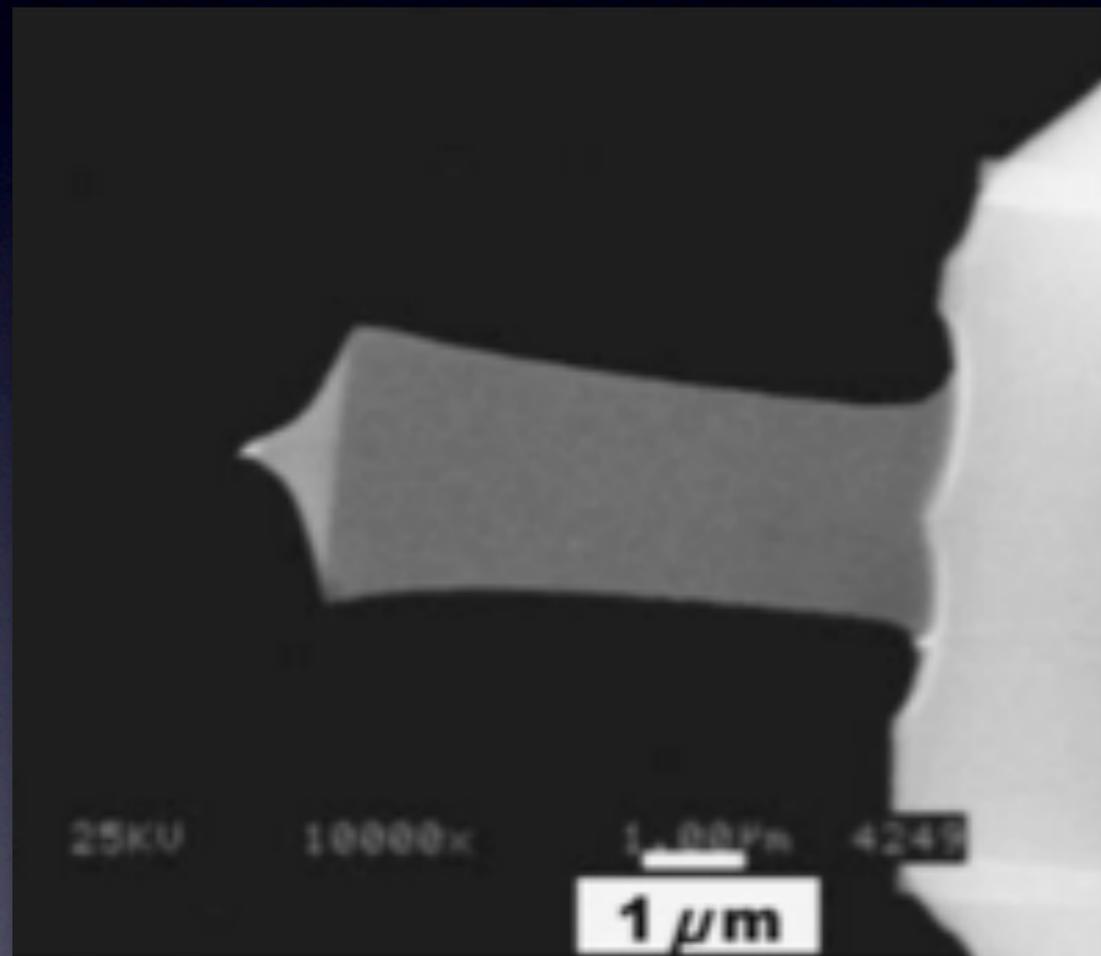
Compare with problems making and using small cantilevers



Difficult to fabricate these small cantilevers reproducibly

Difficult to avoid cantilever substrate contacting sample

Compare with problems making and using small cantilevers

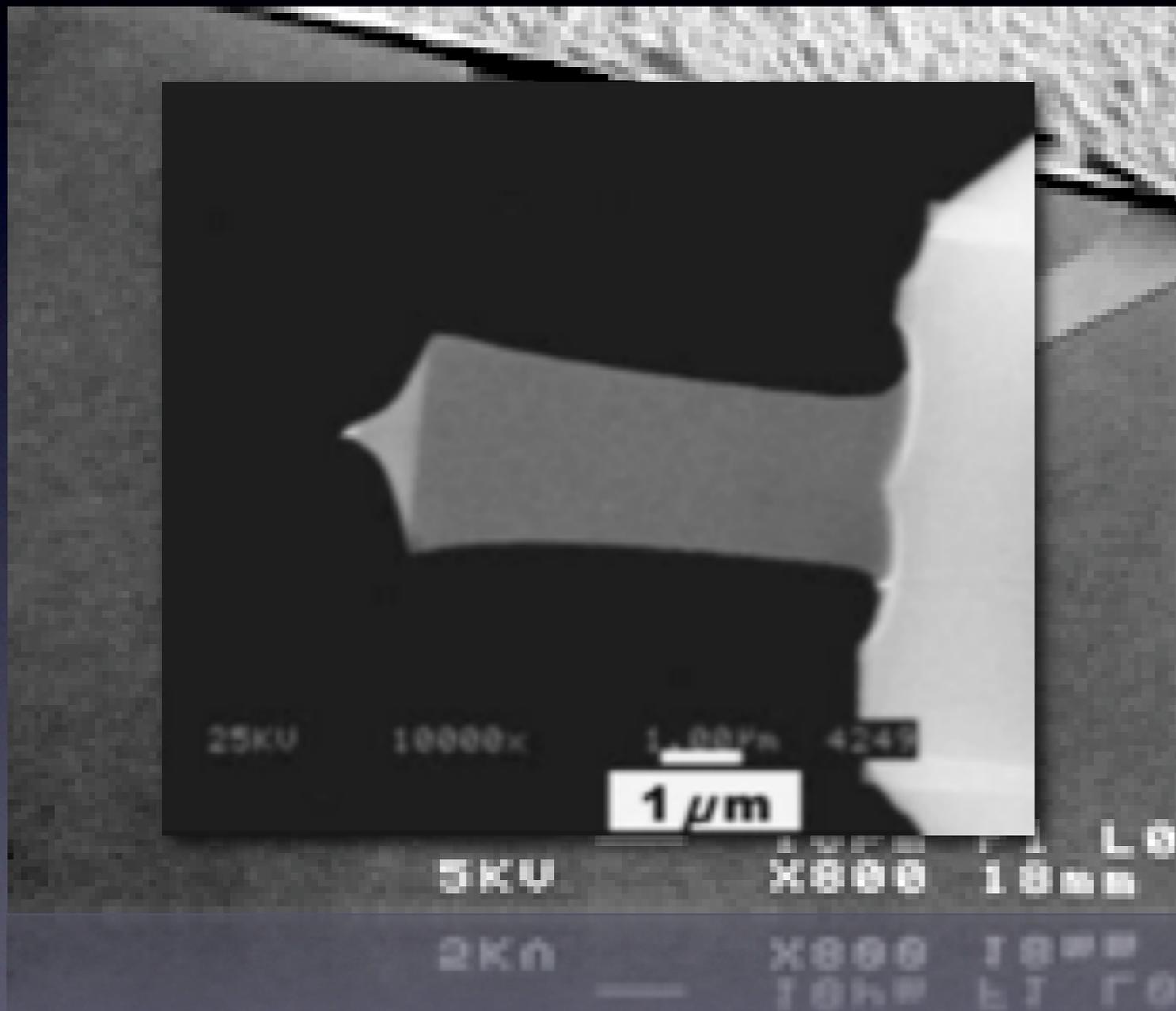


Difficult to fabricate these small cantilevers reproducibly

Difficult to avoid cantilever substrate contacting sample

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Compare with problems making and using small cantilevers



Difficult to fabricate these small cantilevers reproducibly

Difficult to avoid cantilever substrate contacting sample

Difficult to avoid cantilever substrate contacting sample

Compare with problems making and using small cantilevers

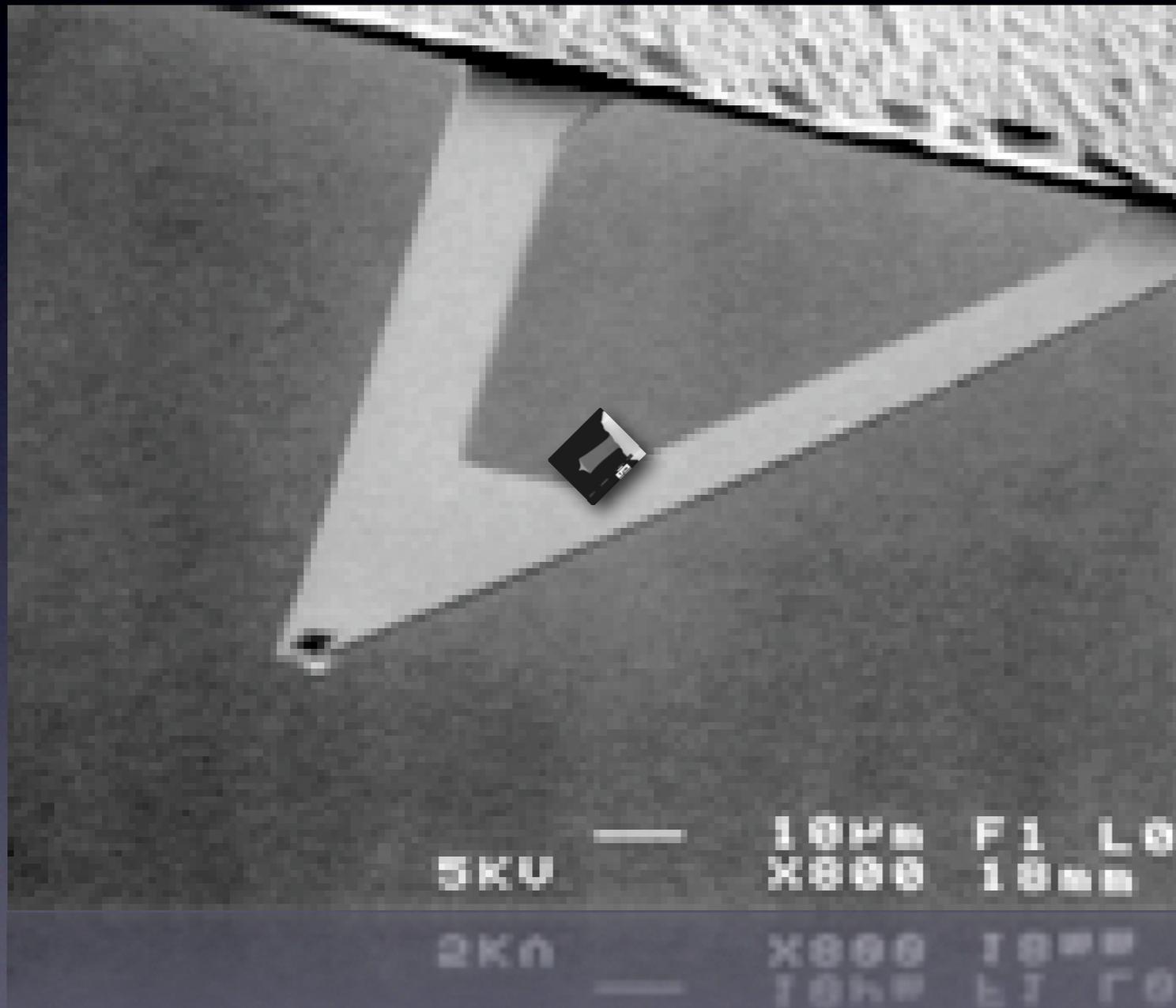


Difficult to fabricate these small cantilevers reproducibly

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Difficult to avoid cantilever substrate contacting sample

Compare with problems making and using small cantilevers



Difficult to fabricate these small cantilevers reproducibly

Difficult to avoid cantilever substrate contacting sample

Difficult to avoid cantilever substrate contacting sample

A New Detection Arrangement

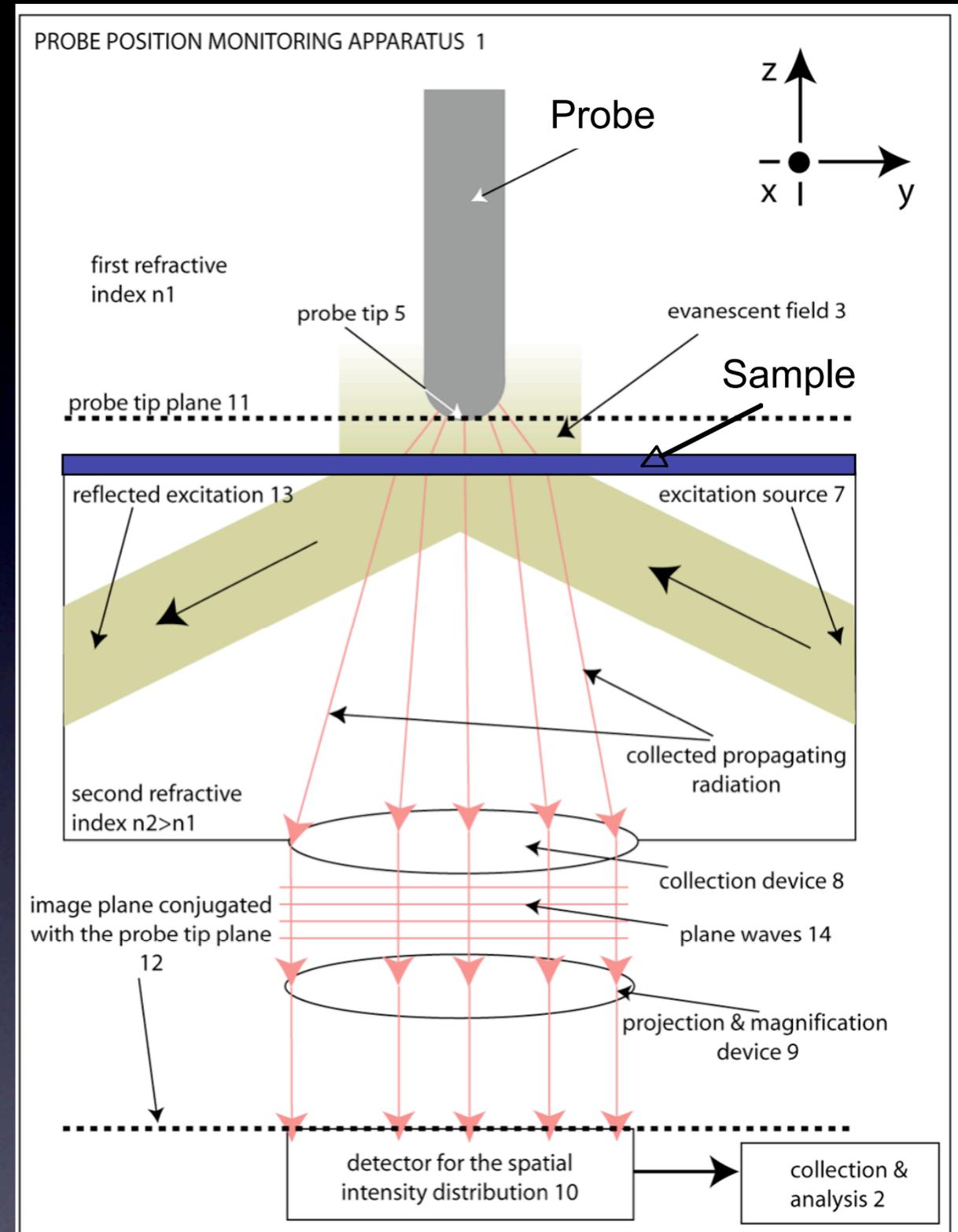
An evanescent wave is generated above the sample surface

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

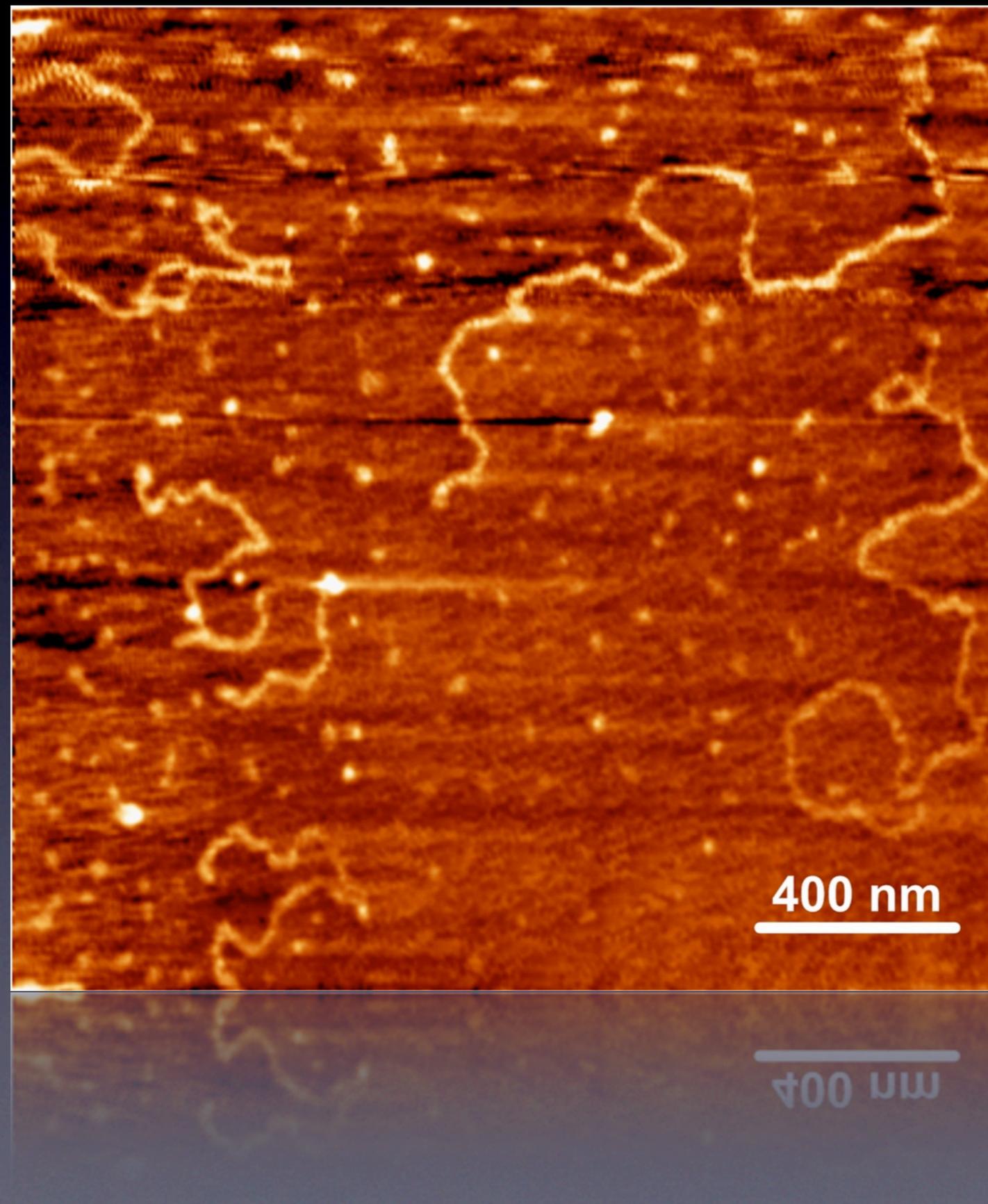
The x and y position of the tip is then monitored directly

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



Antognozzi, Ulcinas, Picco, Simpson, Heard, Szczelkun, Brenner, Miles,
Nanotechnology **19** (2008) 384002

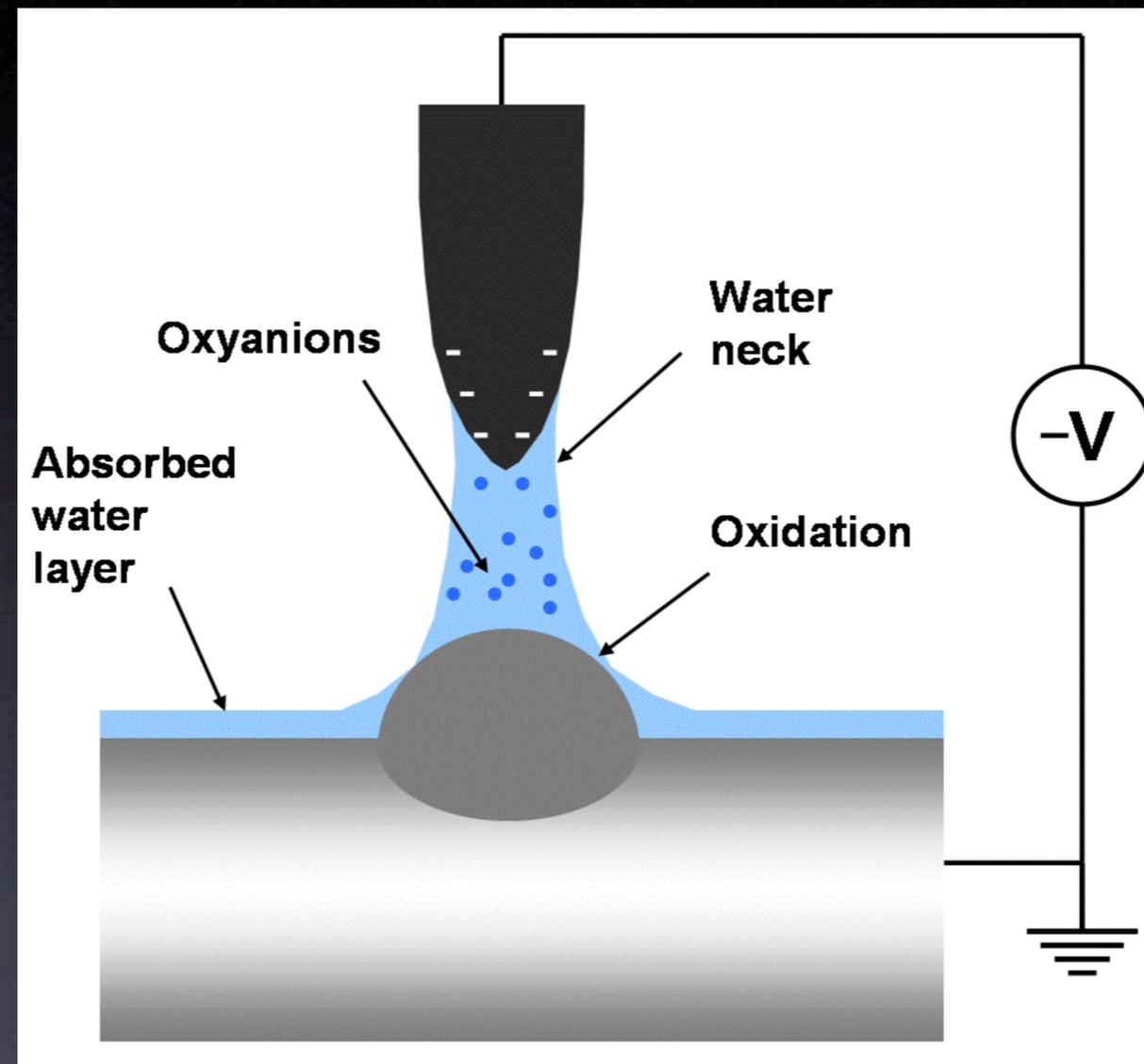
Image of -DNA molecules deposited onto mica and observed in a 5 mM NiCl₂ solution.



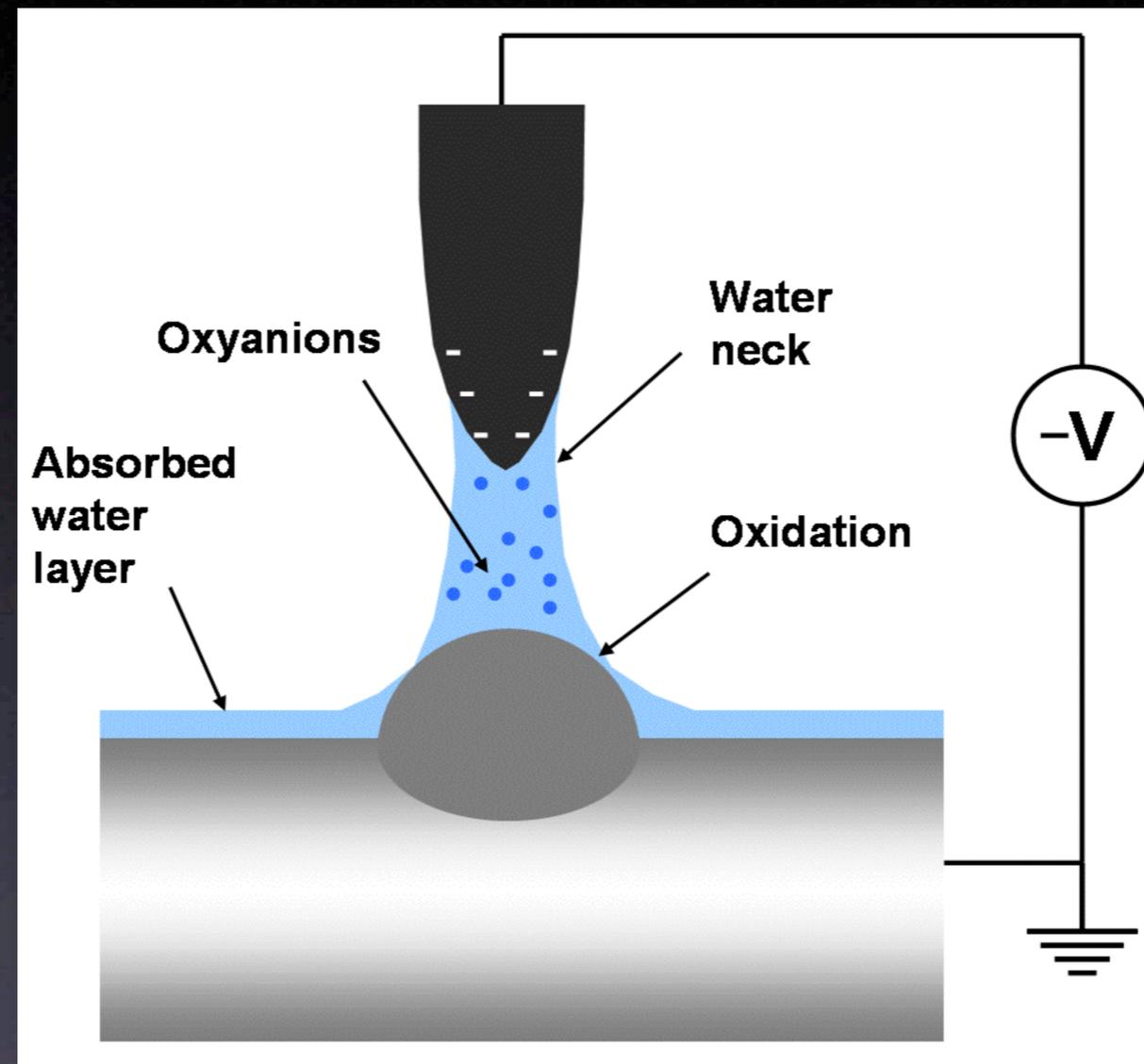
Non-imaging

Electrochemical oxidation of passivated silicon

Local AFM electrochemical oxidation

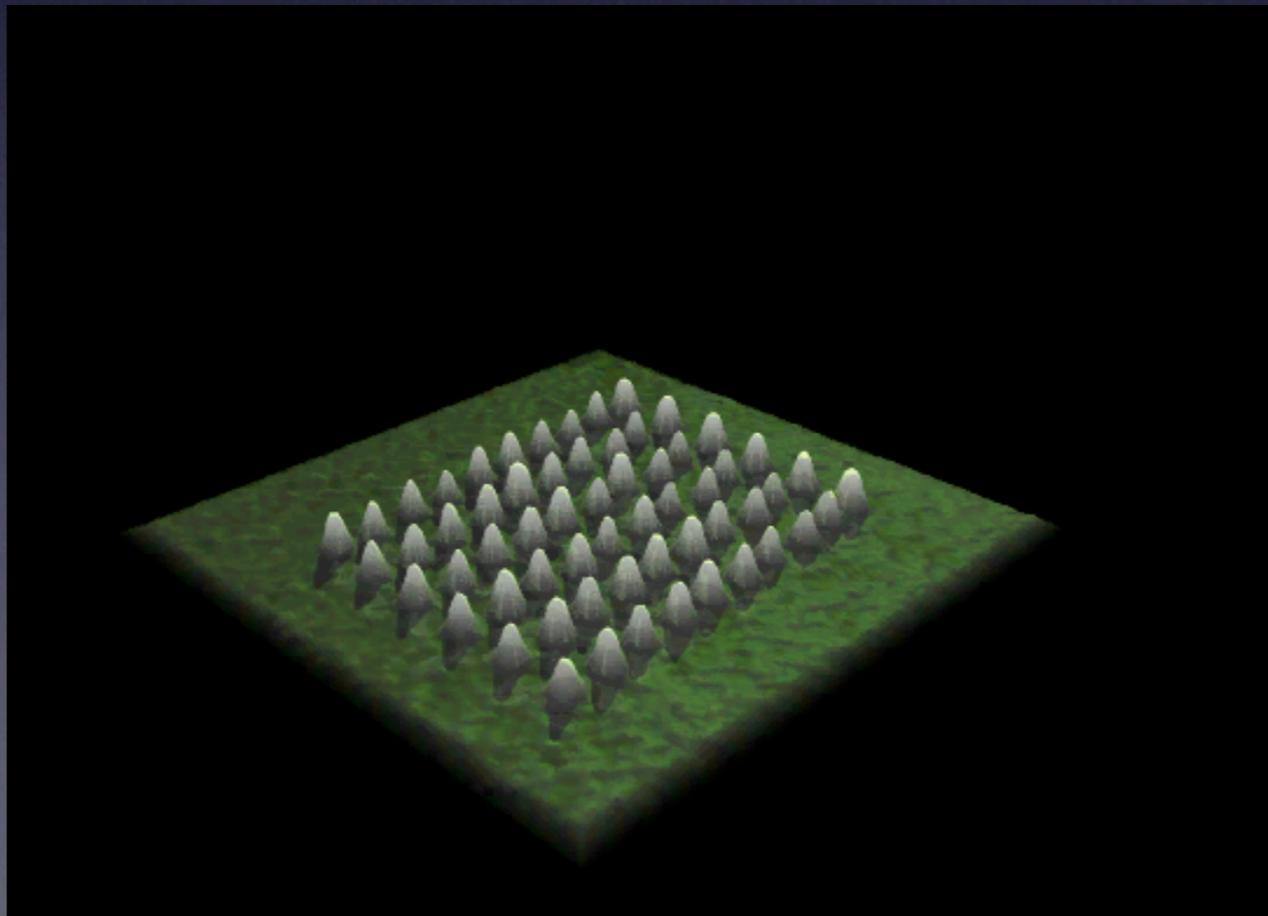


Local AFM electrochemical oxidation

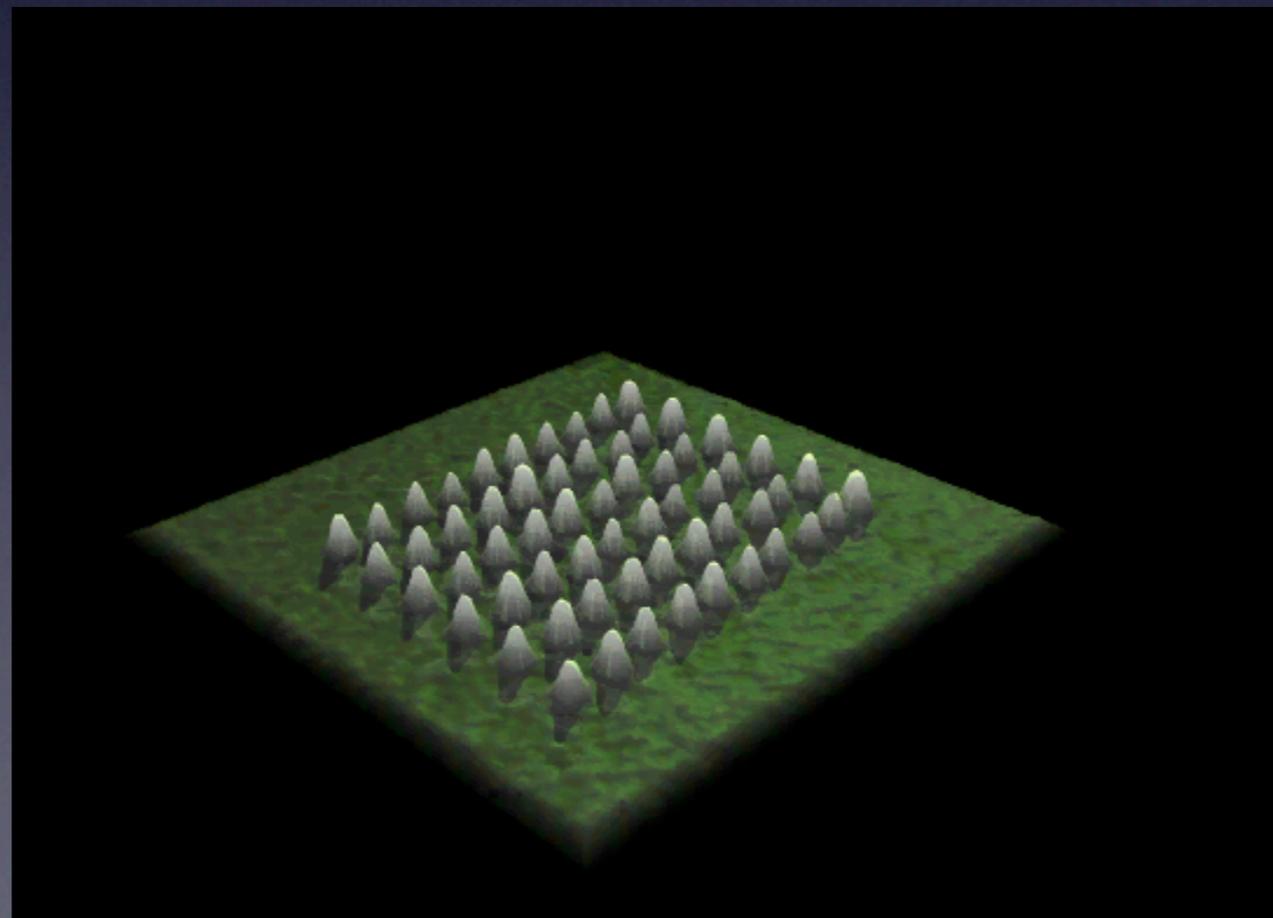
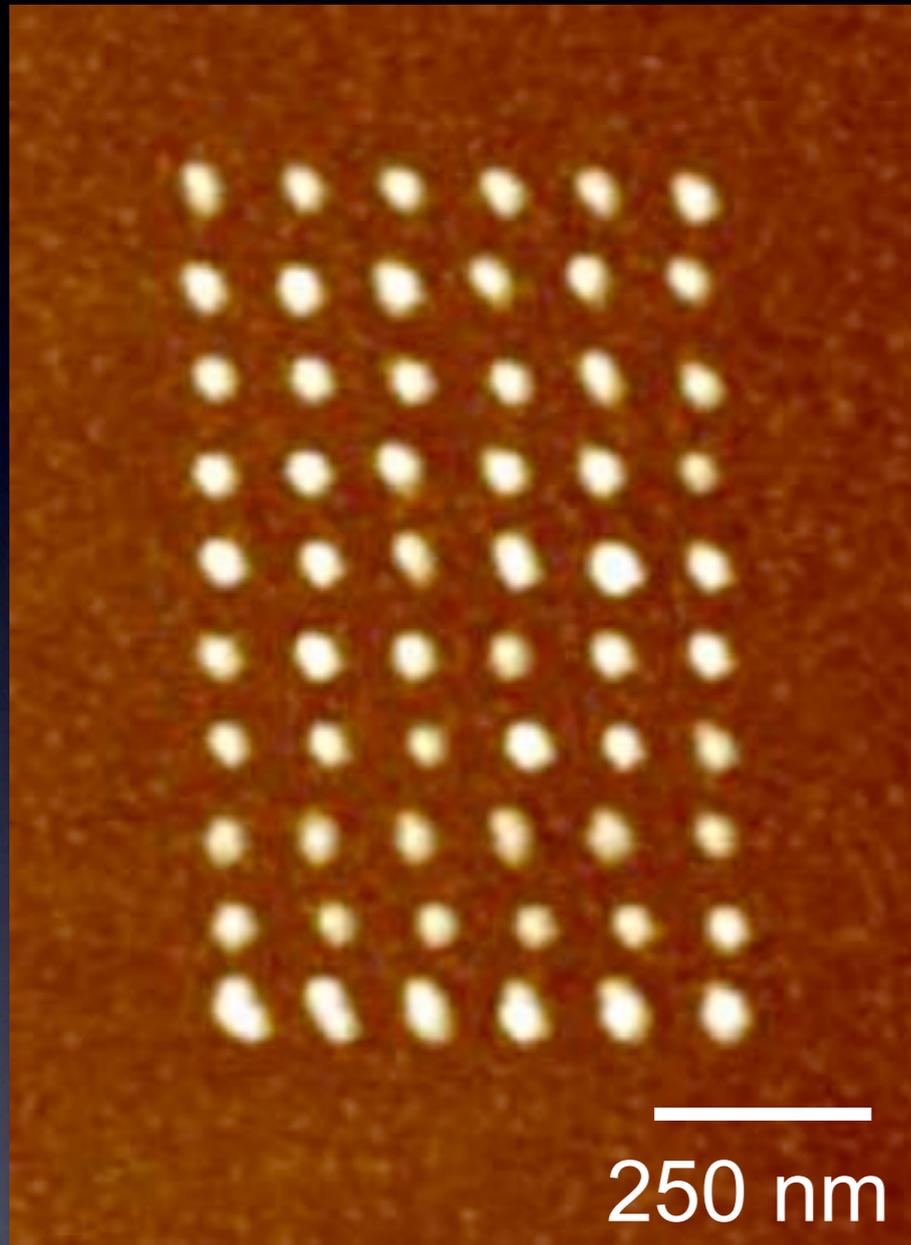


Can be used to pattern surface -
conventional AFM too slow to pattern large areas

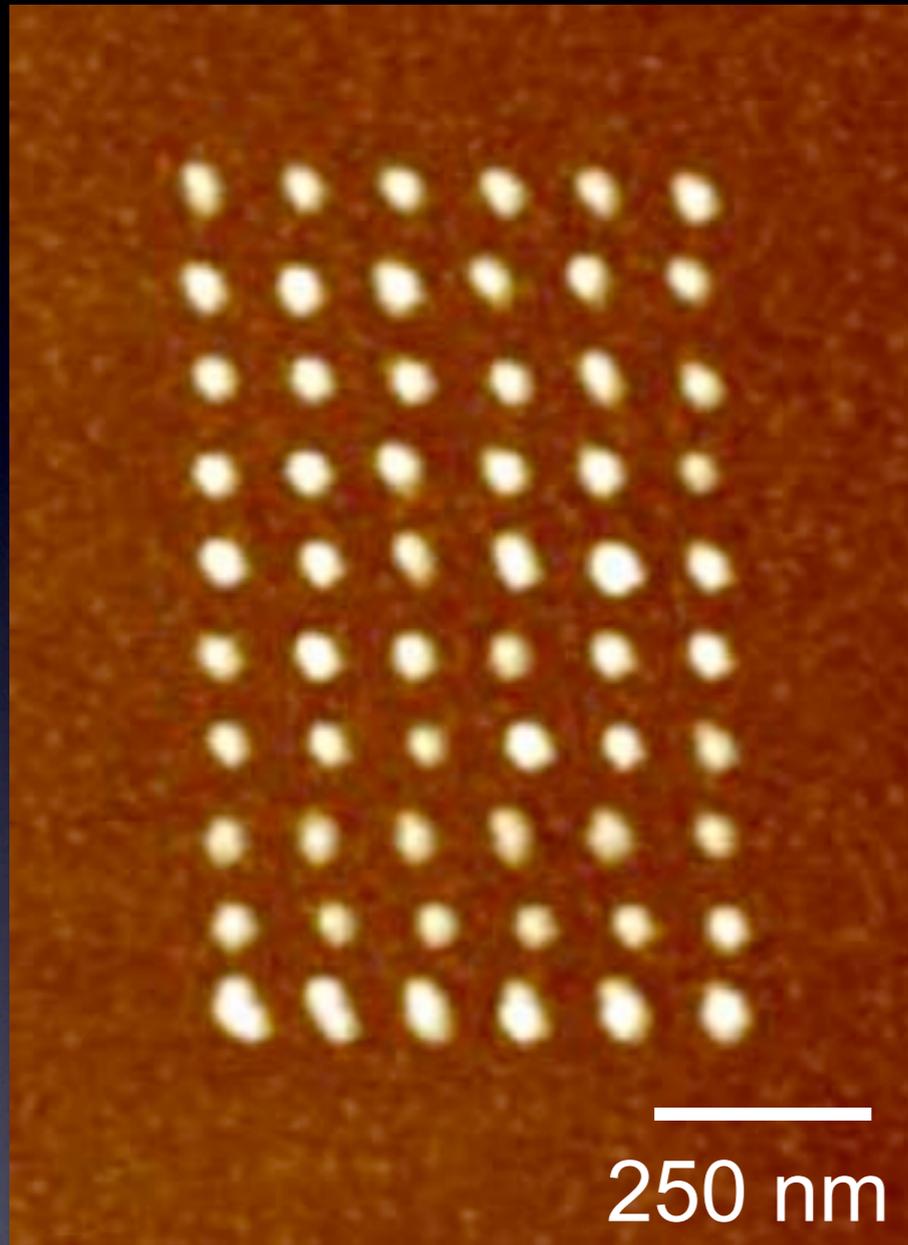
All oxidation experiments performed in an environmental chamber
- typically at 50 % RH



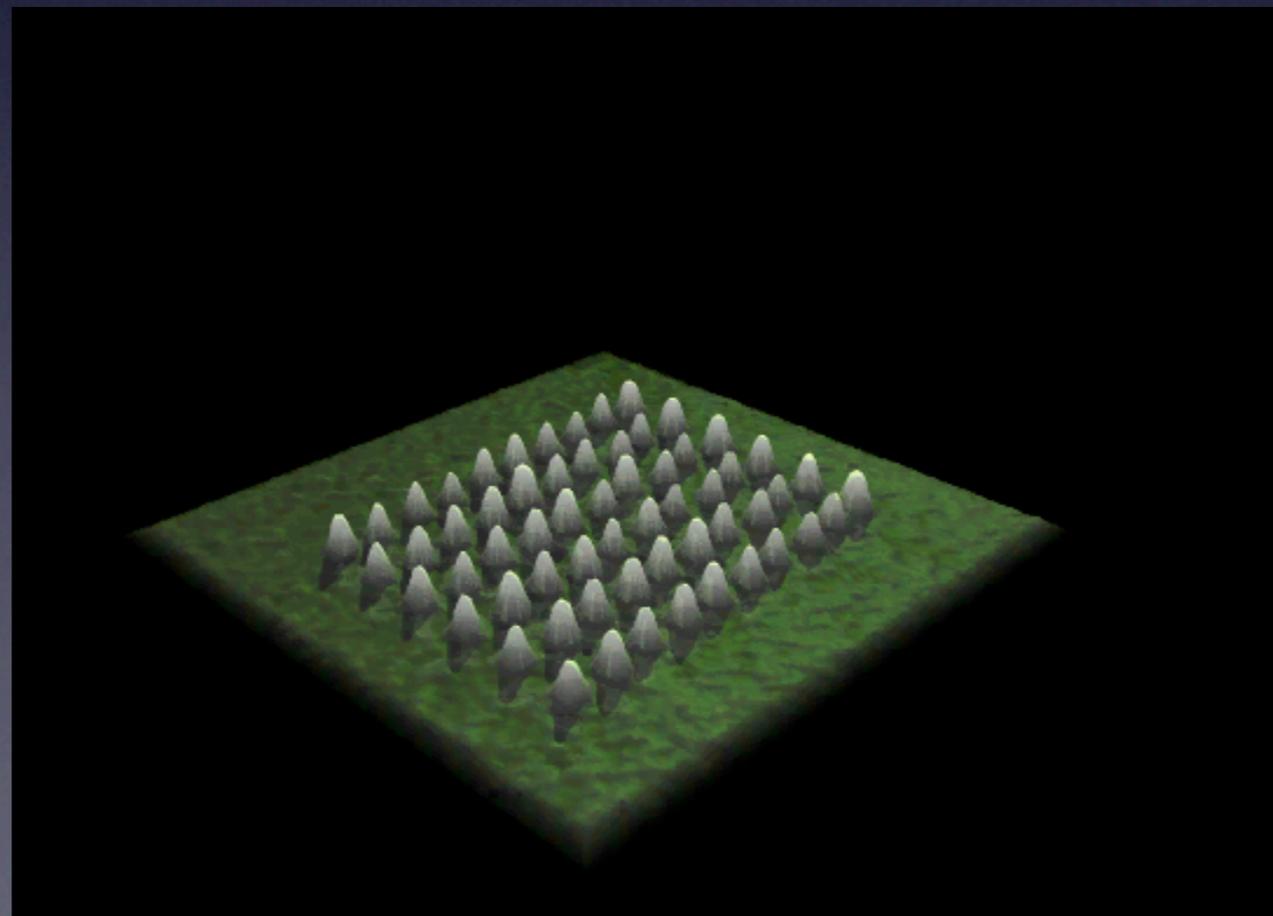
All oxidation experiments performed in an environmental chamber
- typically at 50 % RH



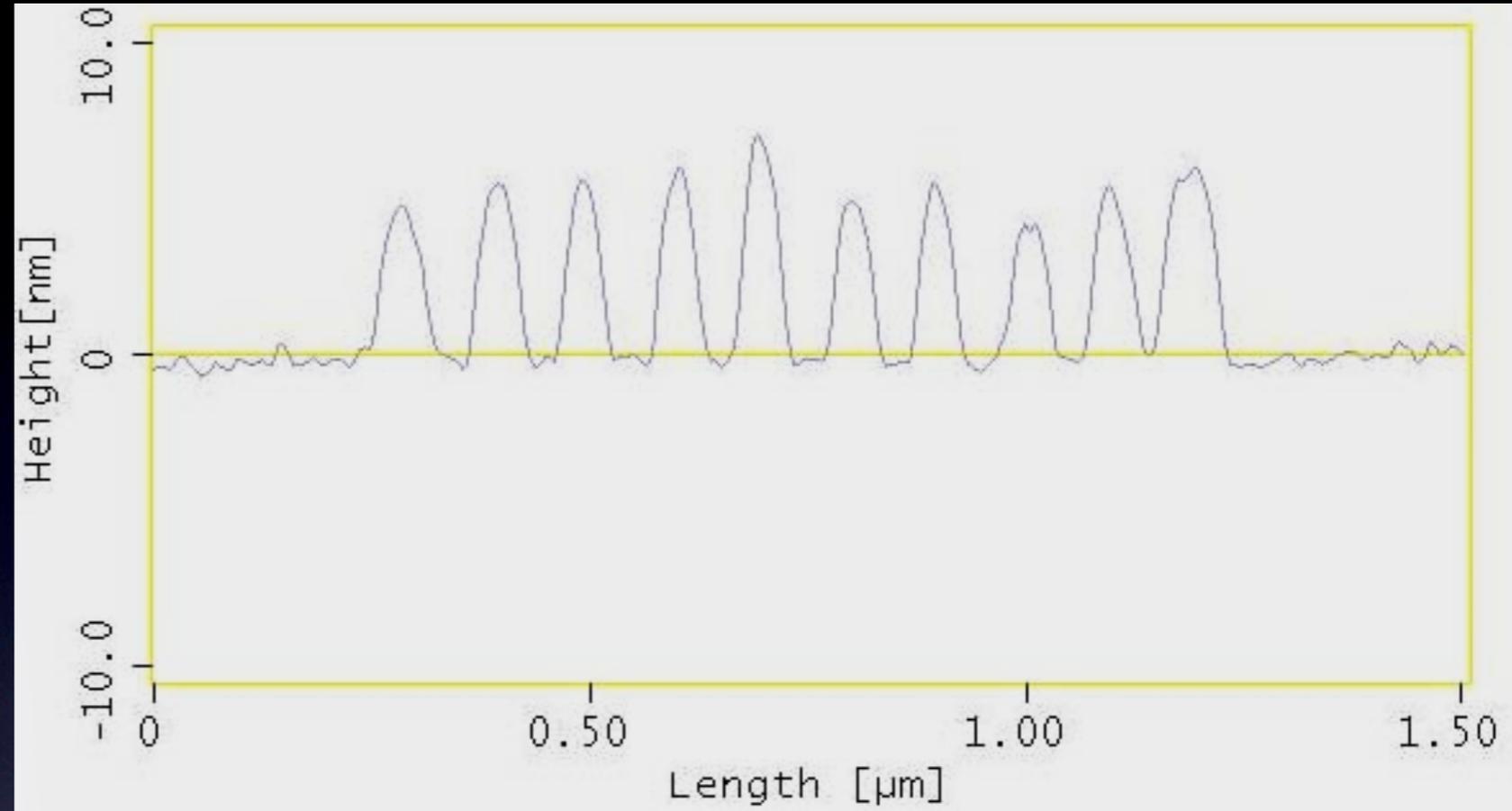
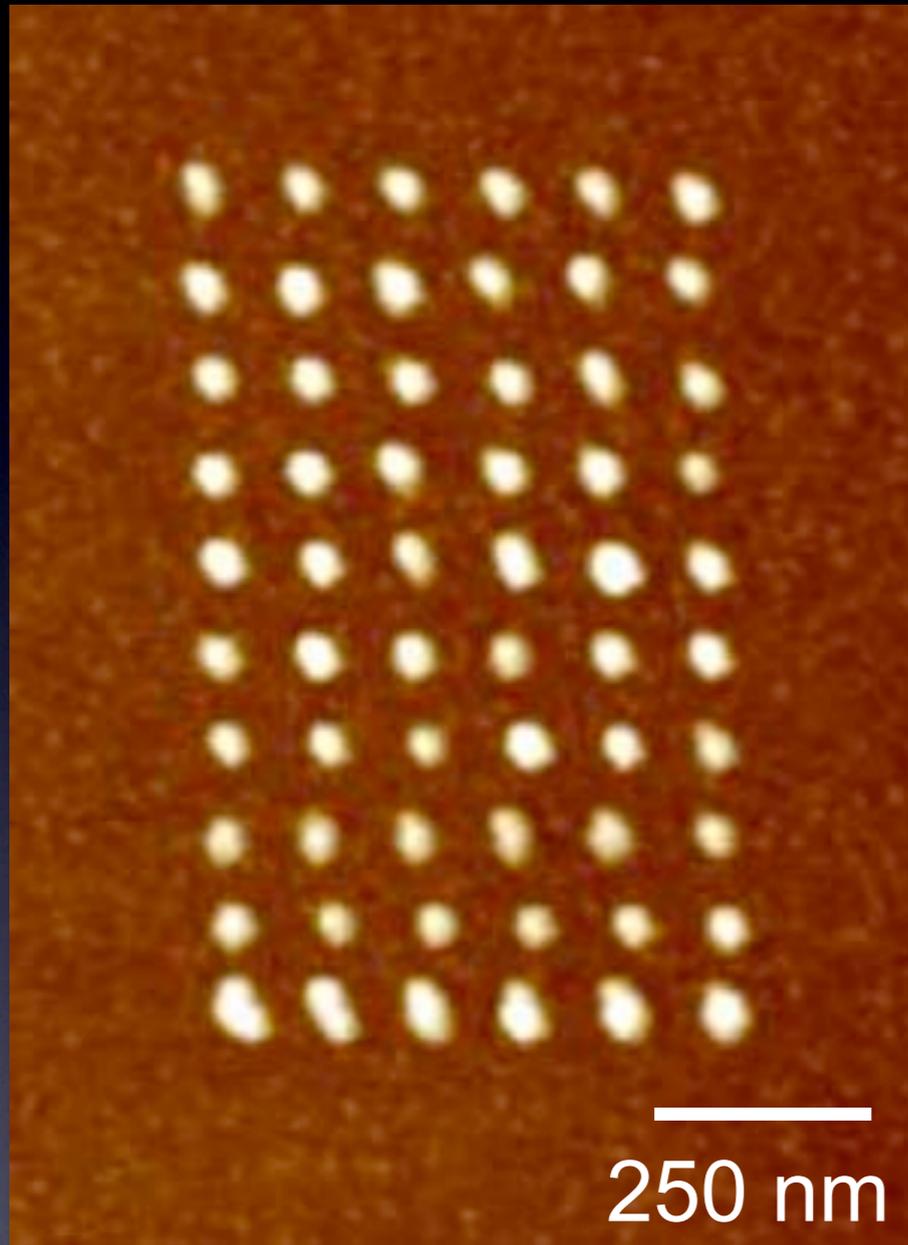
All oxidation experiments performed in an environmental chamber
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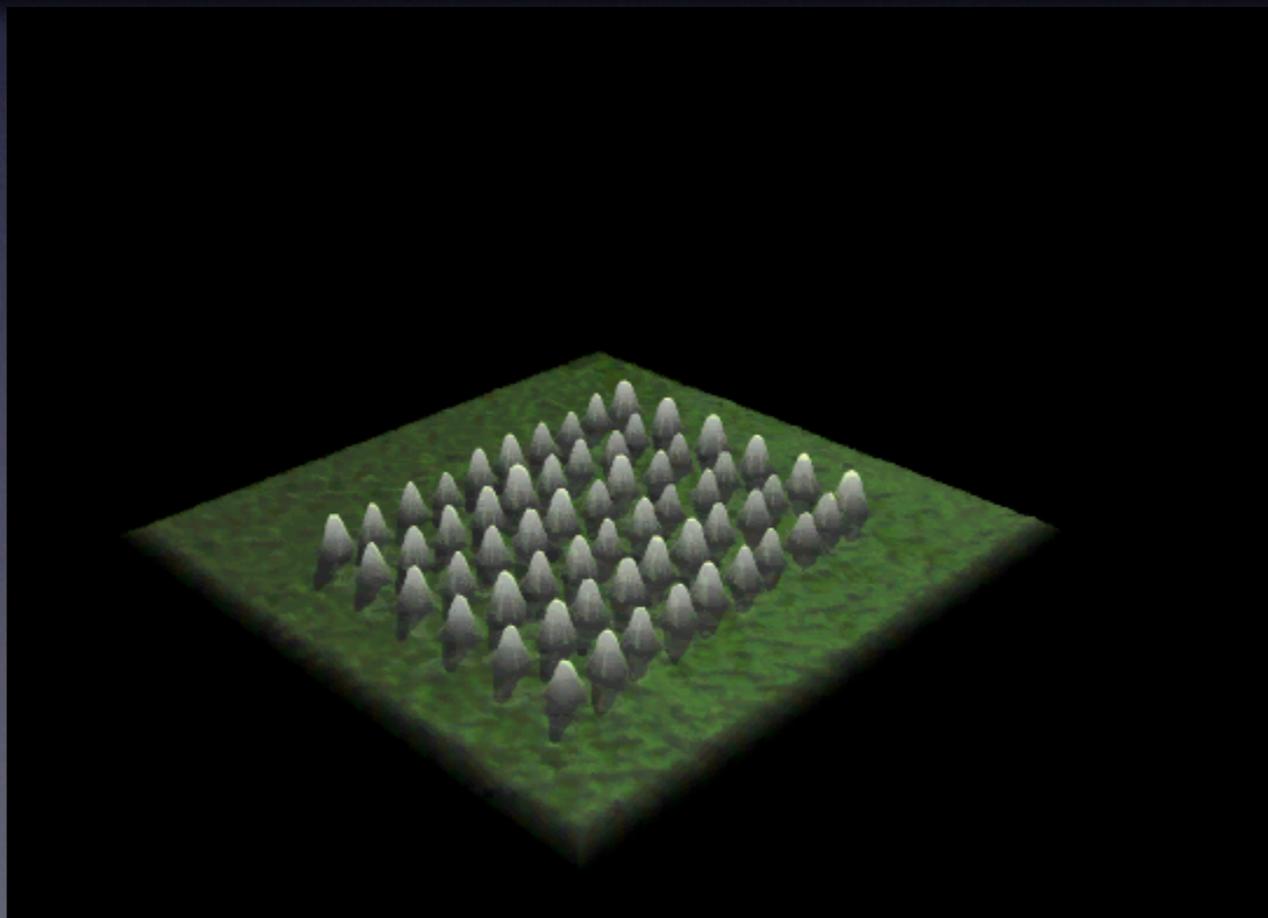
10 ms; -12 V Si tip;
conventional AFM
non-contact



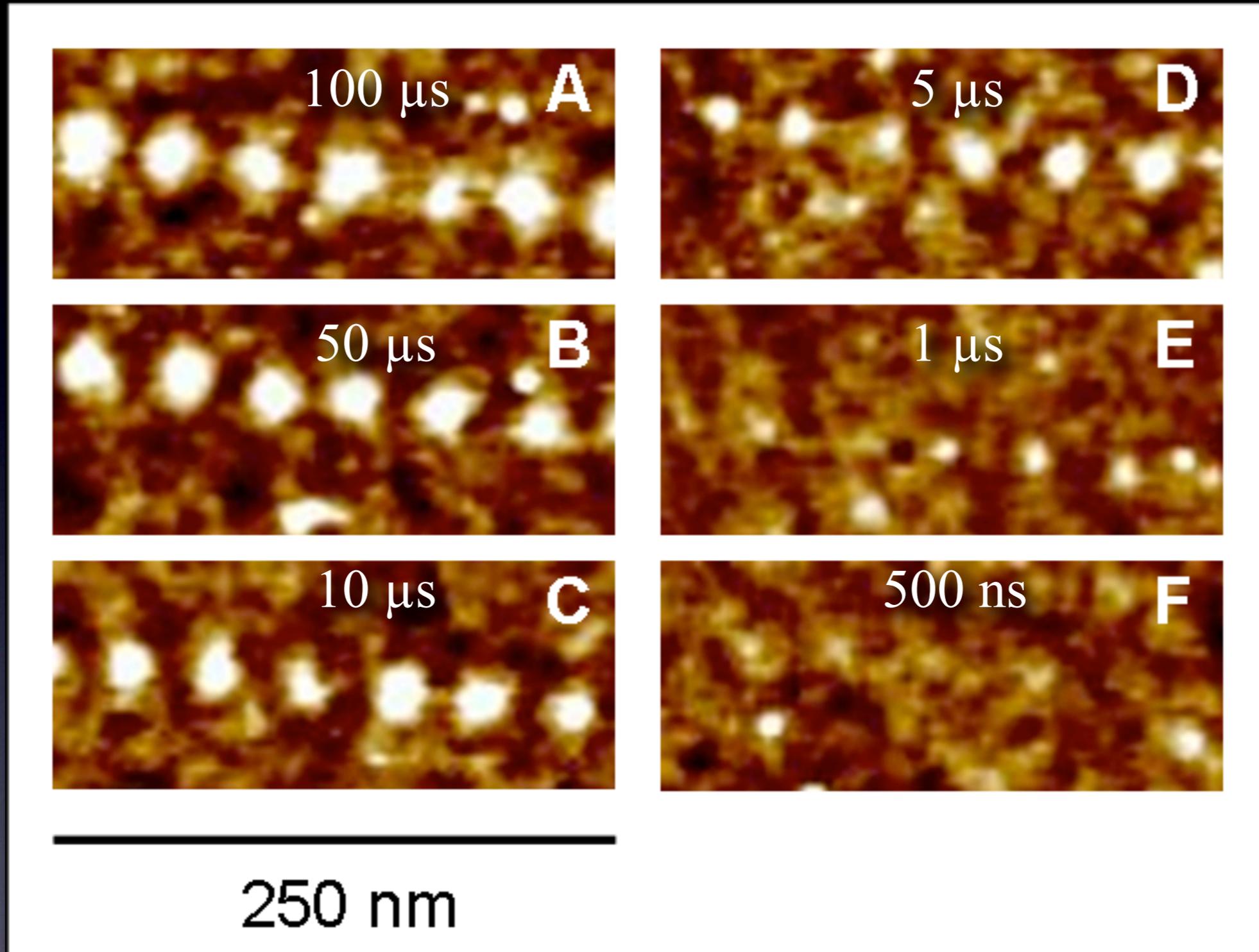
All oxidation experiments performed in an environmental chamber - typically at 50 % RH



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



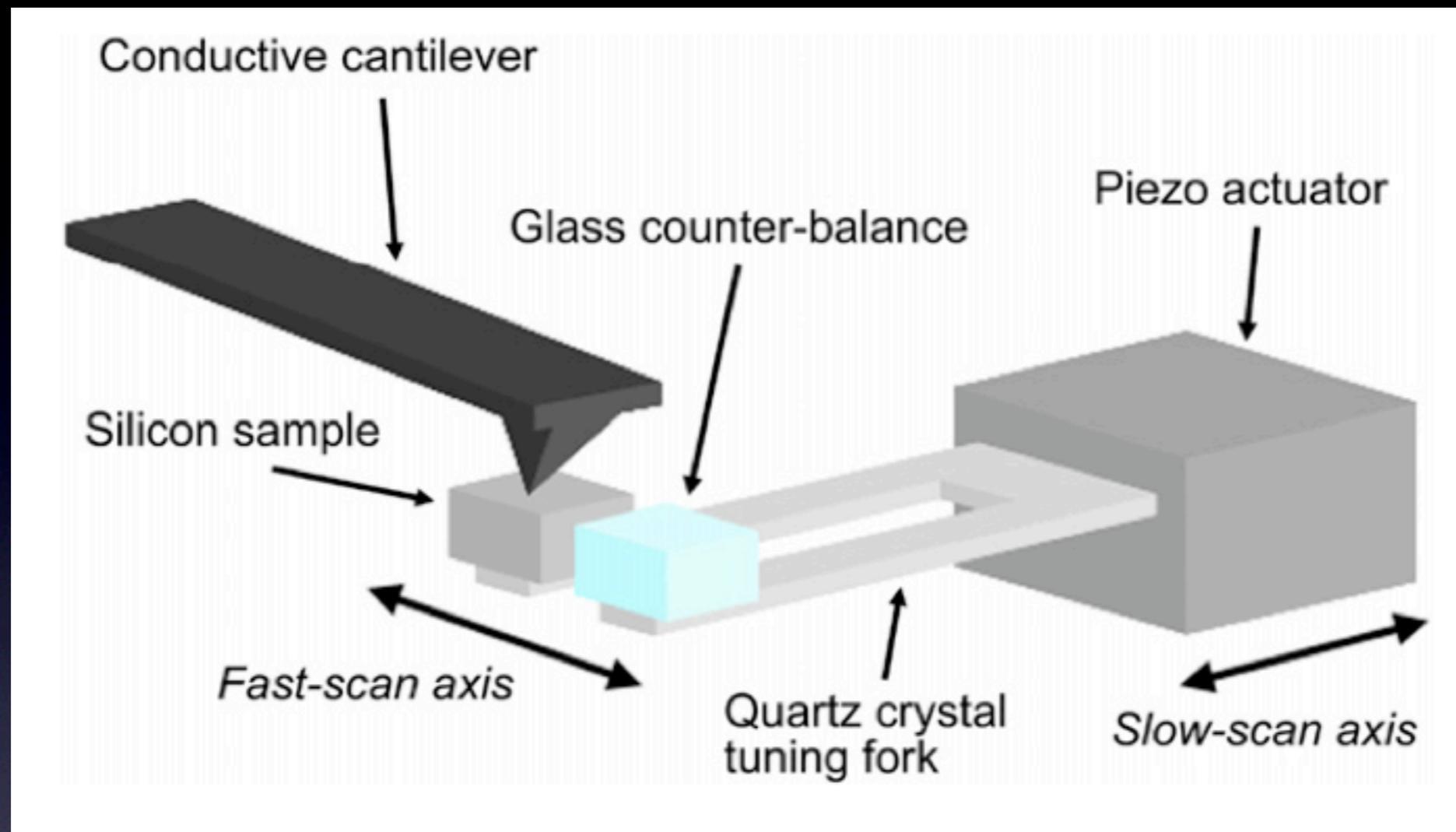
Oxidation of Silicon with high-speed AFM



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

Picco, Vicary

High-speed resonant scan stage

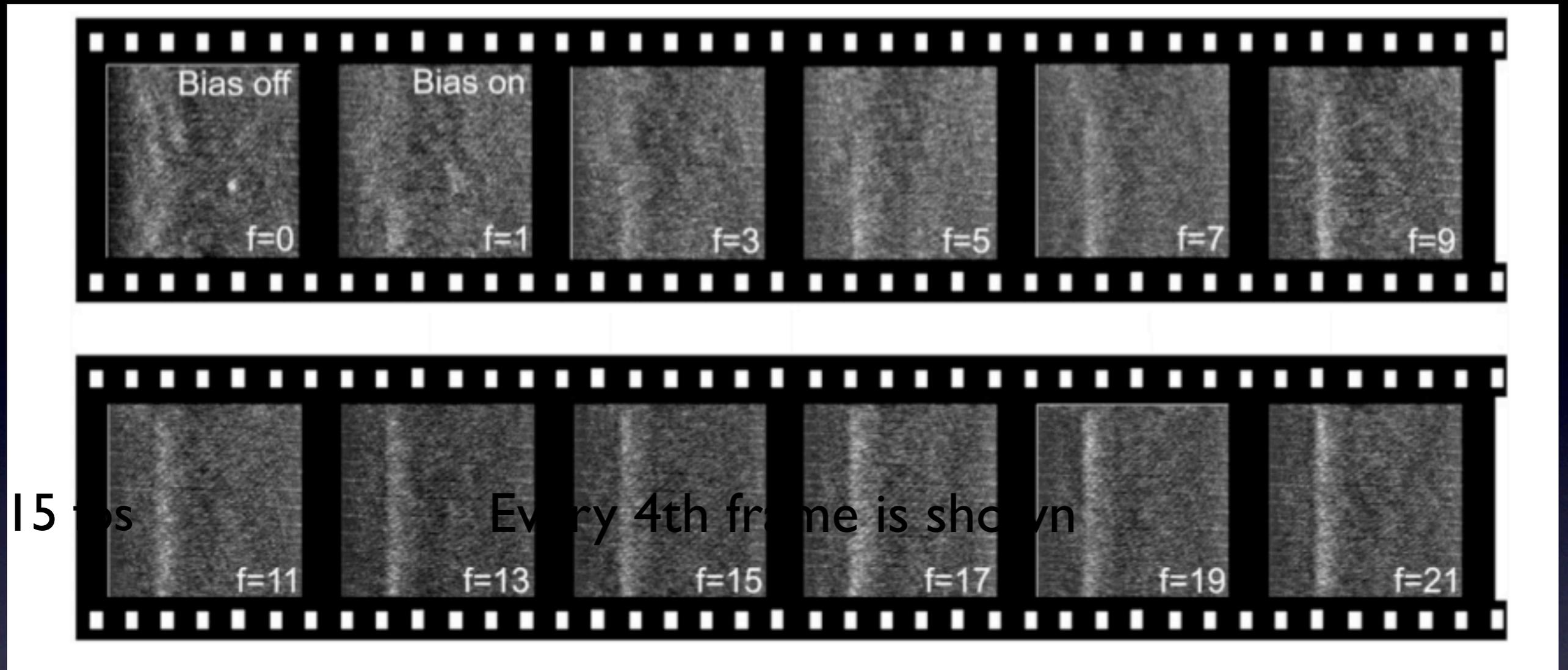


The fast scan axis is provided by a quartz crystal resonator with perpendicular slow scan direction by a piezoactuator.

Cantilever is mounted on the scan tube of a conventional AFM.

James Vicary & Mervyn Miles, *Nanotechnology* **20** (2009) 095302

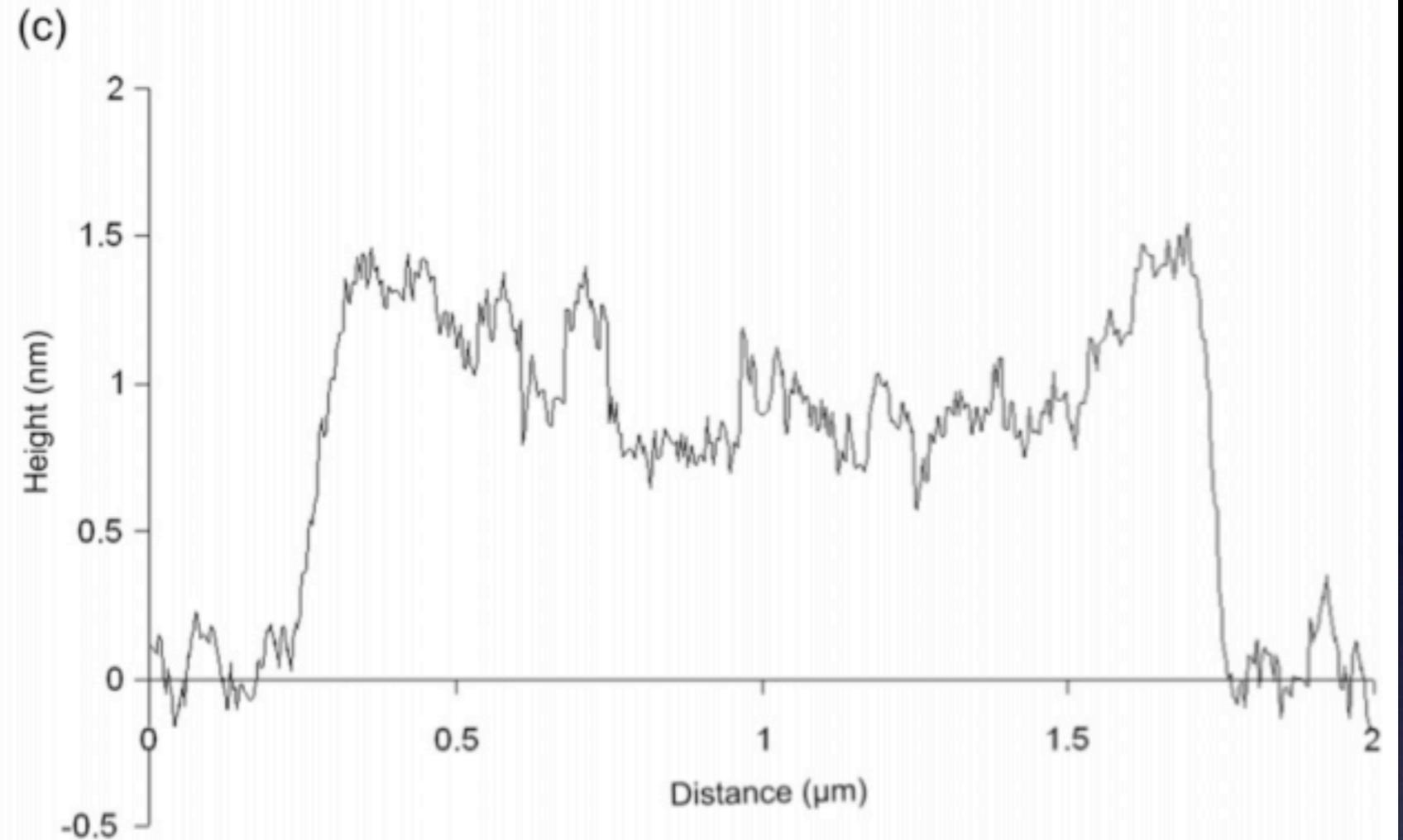
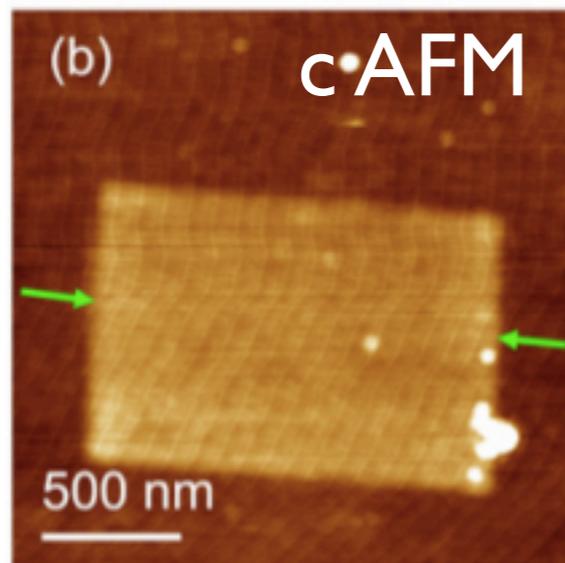
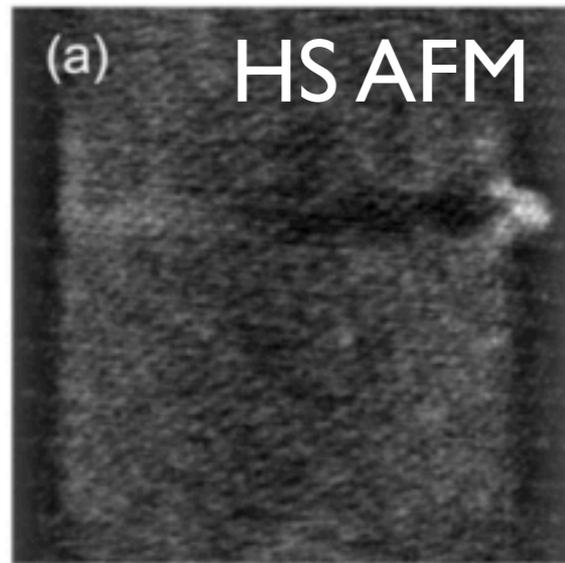
Resonant scanner mounted on Veeco Dimension 3100



Whole scan area oxidized - bias time set to half a frame (33 ms) and repeated at 1 Hz, triggered from scan drive. Scan area is also panned in x 1 Hz; these images are the 8th frame of the pan so that the edge of the oxidation can be seen.

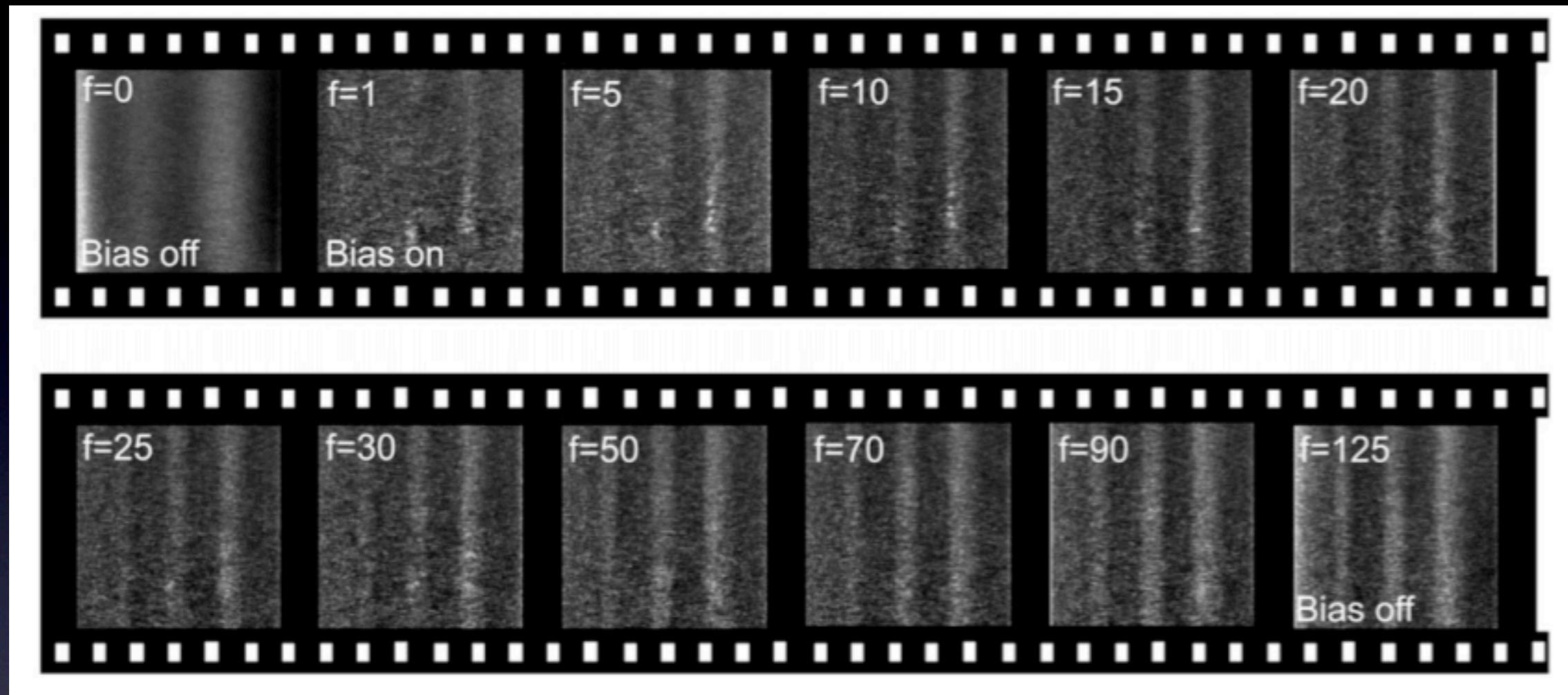
James Vicary & Mervyn Miles, *Nanotechnology* **20** (2009) 095302

After oxidation

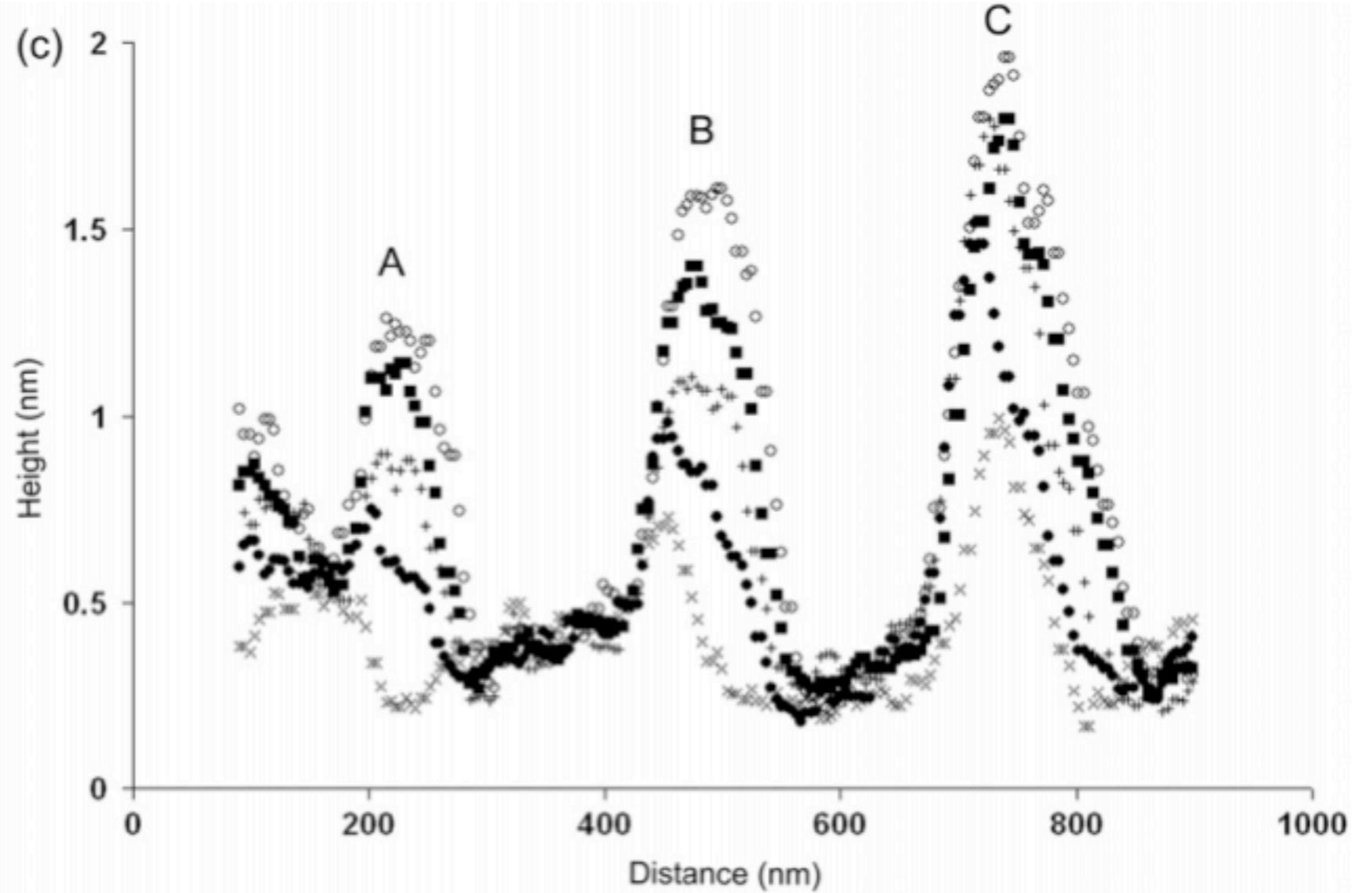
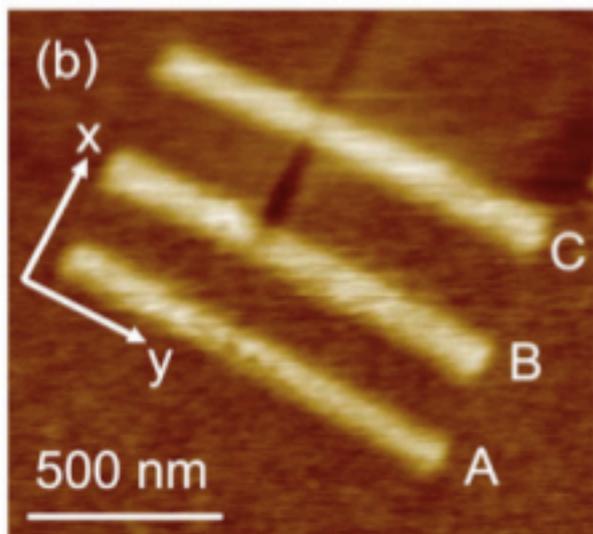
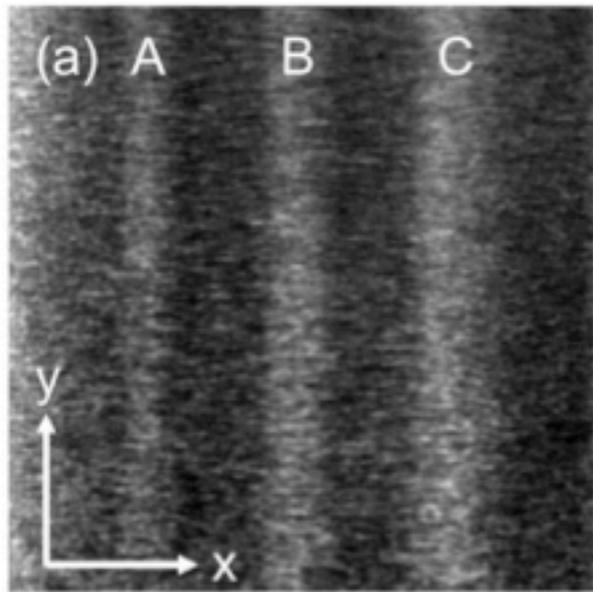


The profile shows the oxide to be thicker at the ends - this is due to the sinusoidal scan speed of the resonant scanner.

Three oxidation bias pulses triggered from scan drive



Simultaneous oxidation and HS AFM imaging;
oxidation growth rate affected by sample tilt and
sinusoidal scan velocity

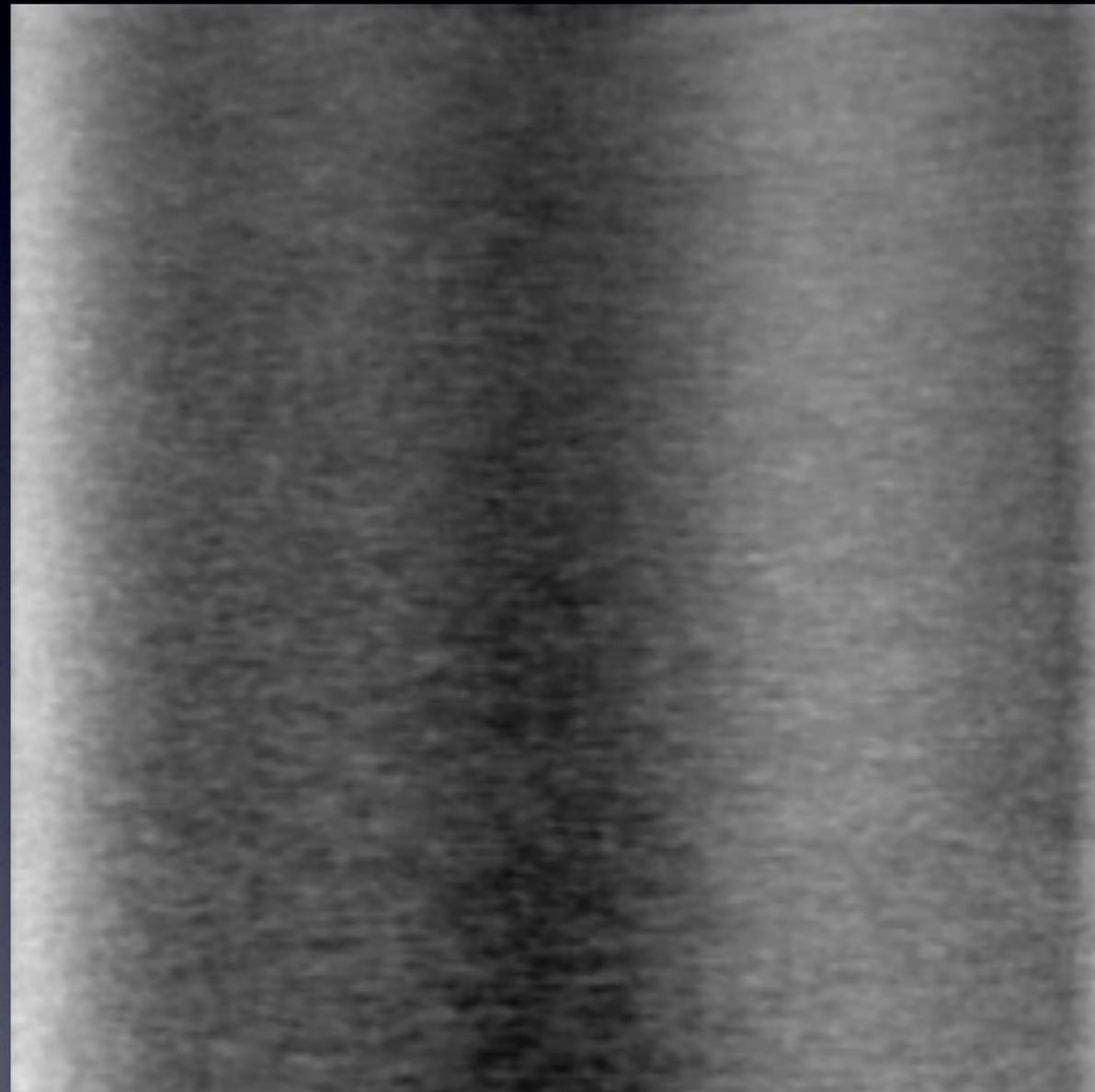


High-speed AFM

Simultaneous writing (oxidation) and imaging

30 fps

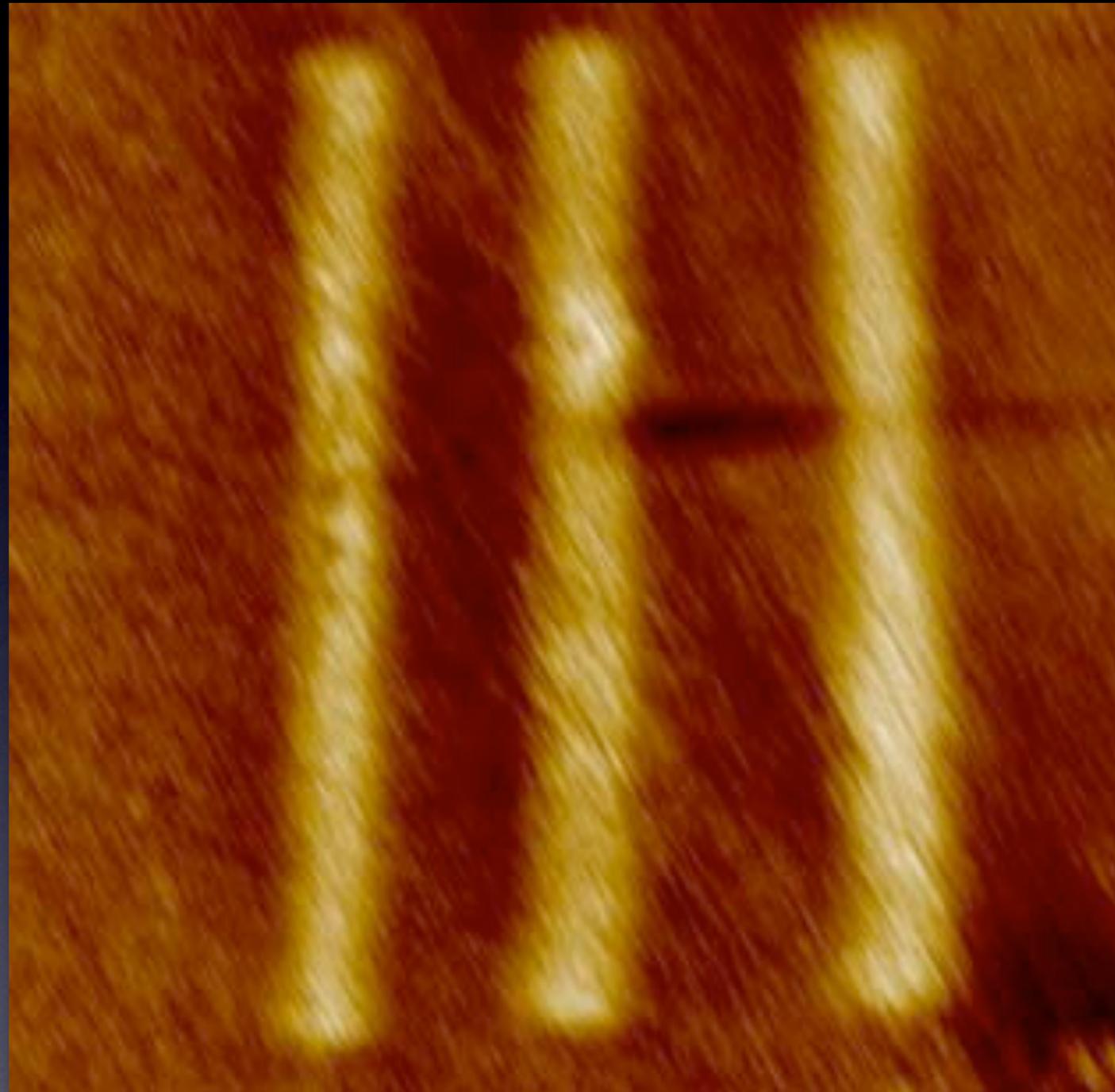
Read-Write



3 voltage pulses synchronized to fast line scan

Picco, Vicary

Conventional AFM of oxidized lines on Si



James Vicary & Mervyn Miles, *Nanotechnology* **20** (2009) 095302

Summary

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



Dr. Loren Picco



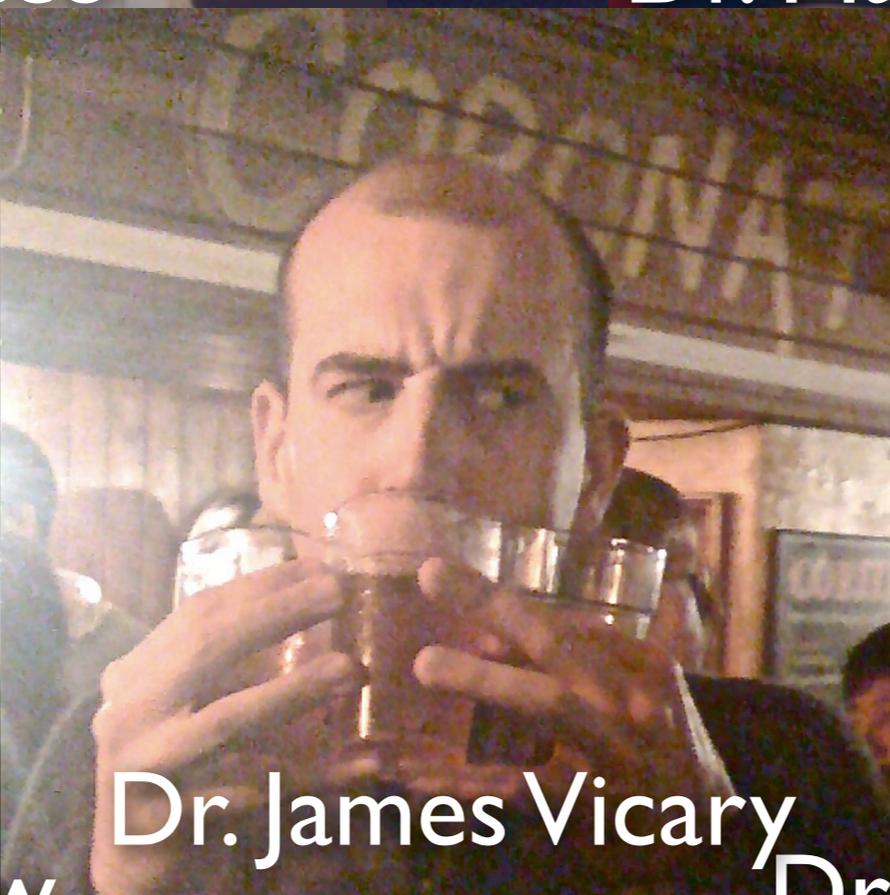
Dr. Arturas Ulcinas



Dr. Massimo Antognozzi



Dave Engledew



Dr. James Vicary



Dr. Peter Dunton

and all members of the Nanophysics Group



Dr. Loren Picco



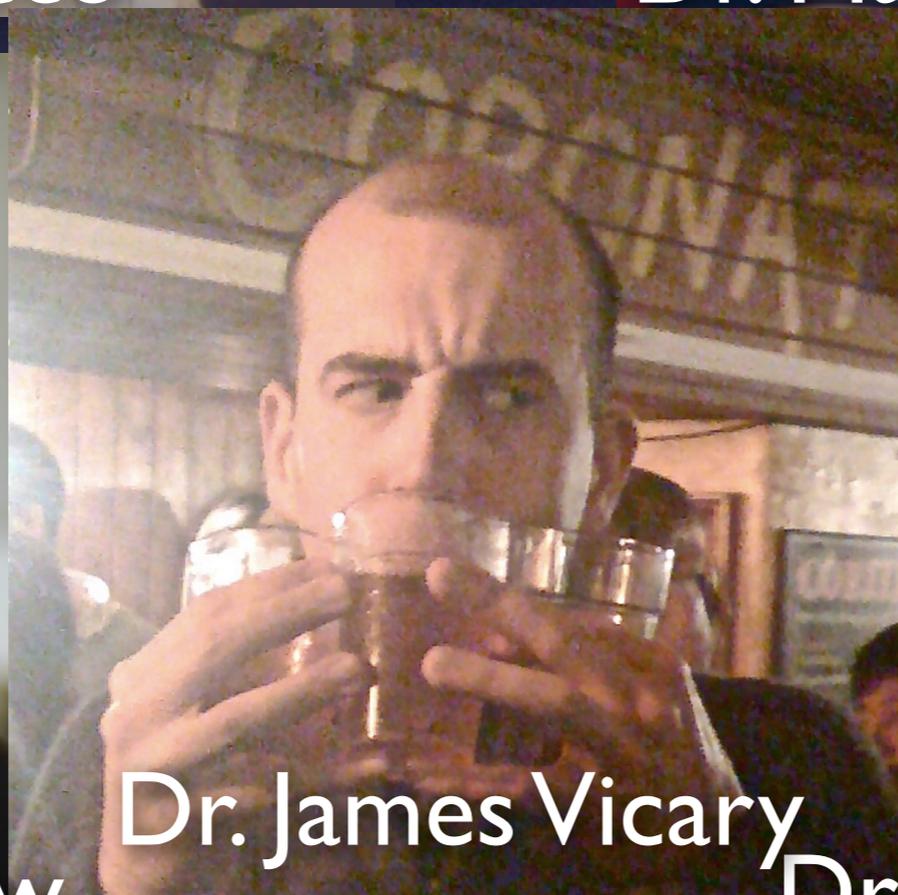
Dr. Arturas Ulcinas



Dr. Massimo Antognozzi



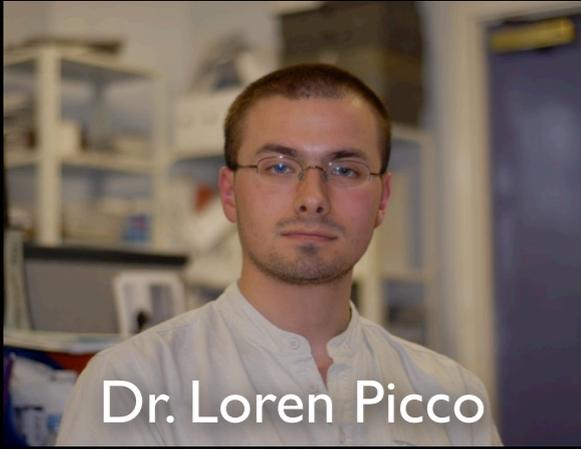
Dave Engledew



Dr. James Vicary



Dr. Peter Dunton



Dr. Loren Picco



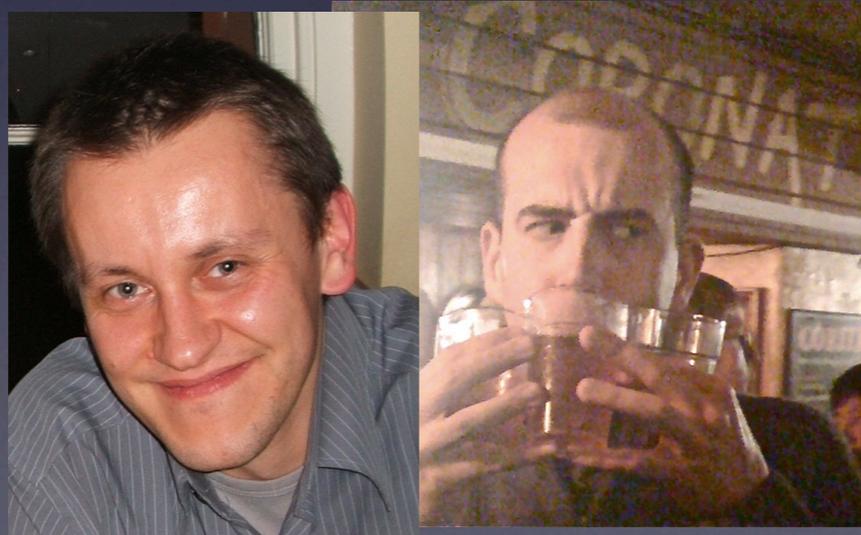
Dr. Peter Dunton



Dave Engledew



Dr. Massimo
Antognozzi



Dr. Arturas
Ulcinas

Dr. James
Vicary



Dr. Loren Picco



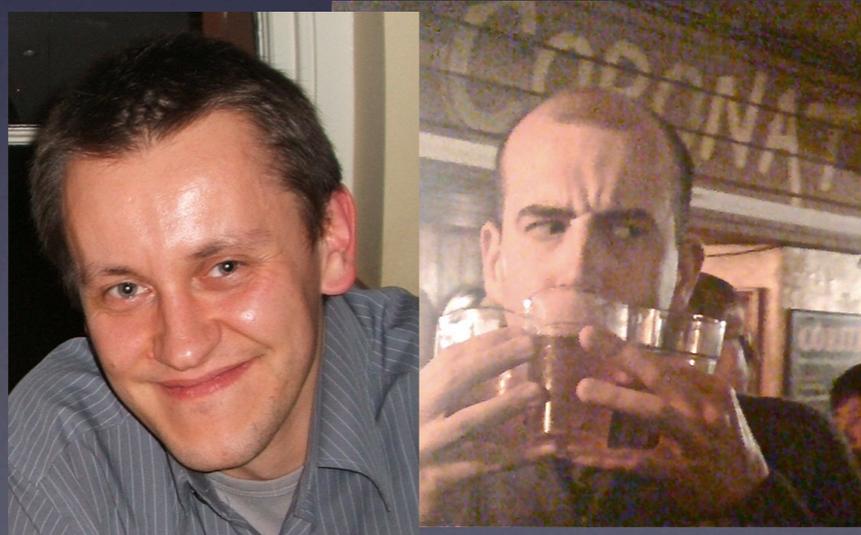
Dr. Peter Dunton



Dave Engledew



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Antognozzi

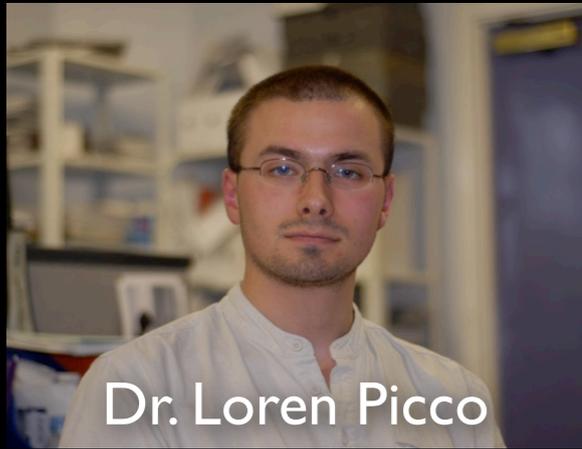


Dr. Arturas
Ulcinas



Dr. James
Vicary





Dr. Loren Picco



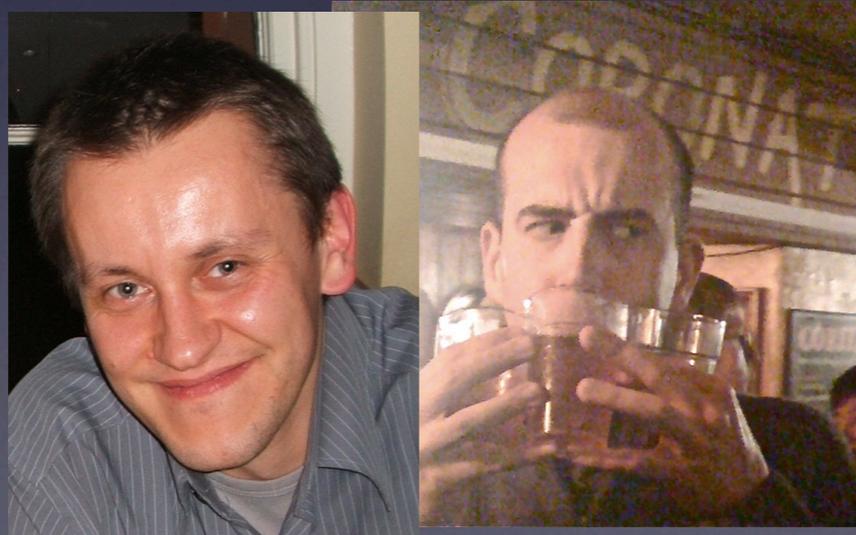
Dr. Peter Dunton



Dave Engledew



Dr. Massimo
Antognozzi



Dr. Arturas
Ulcinas



Dr. James
Vicary



Prof Tatsuo Ushiki



Dr. Loren Picco



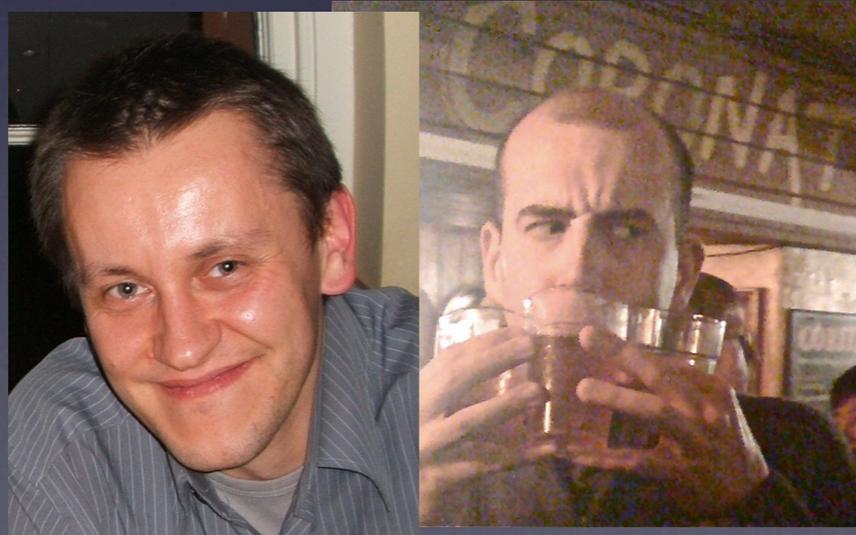
Dr. Peter Dunton



Dave Engledew



Dr. Massimo
Antognozzi



Dr. Arturas
Ulcinas



Dr. James
Vicary



Prof Tatsuo Ushiki Prof Osamu Hoshi



Dr. Loren Picco



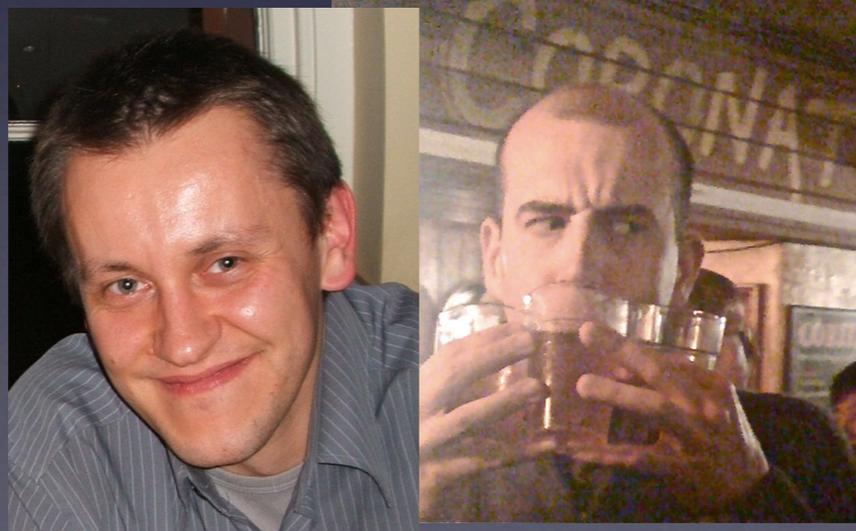
Dr. Peter Dunton



Dave Engledew



Dr. Massimo
Antognozzi



Dr. Arturas
Ulcinas



Dr. James
Vicary



Prof Tatsuo Ushiki Prof Osamu Hoshi





Dr. Loren Picco



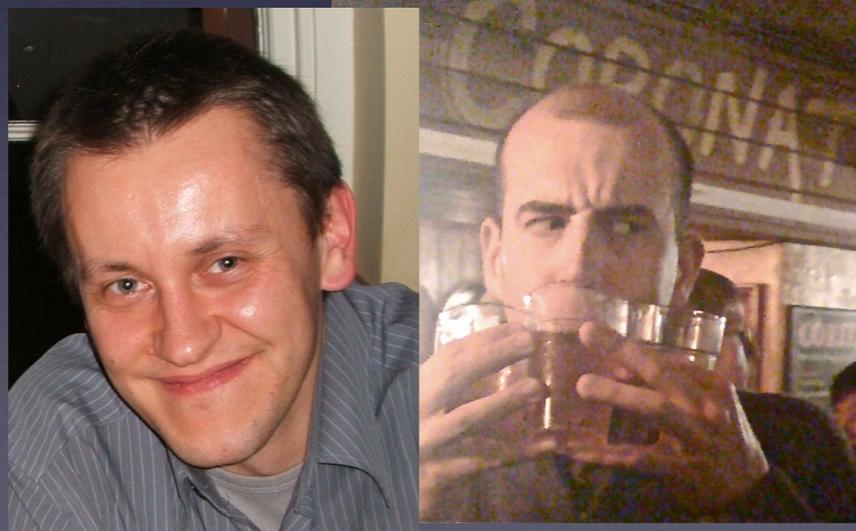
Dr. Peter Dunton



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Dr. James
Vicary



Dr Laurent
Bozec



Prof Tatsuo Ushiki Prof Osamu Hoshi



Dr. Loren Picco



Dr. Peter Dunton



Dave Engledew



Dr. Massimo Antognozzi



Dr. Arturas Ulcinas



Dr. James Vicary



Dr Laurent Bozec

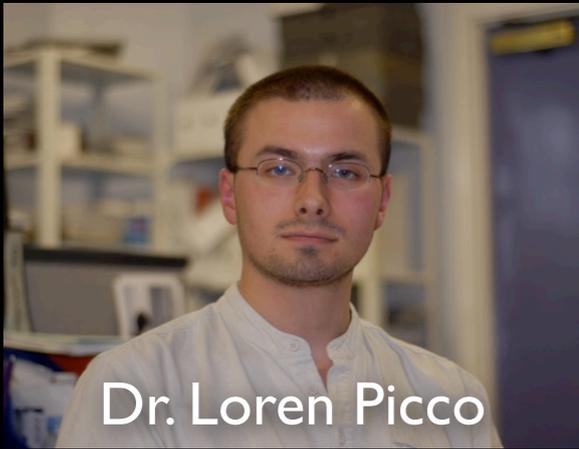


Prof Tatsuo Ushiki

Prof Osamu Hoshi



Prof Mike Horton



Dr. Loren Picco



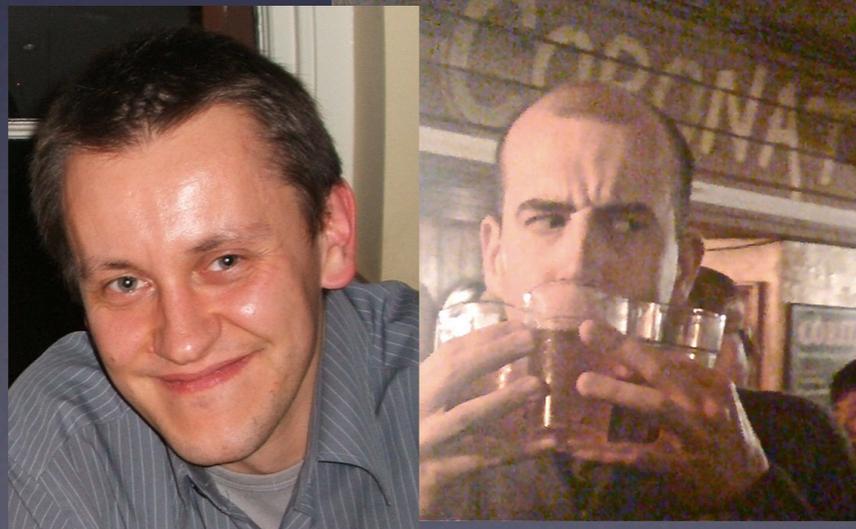
Dr. Peter Dunton



Dave Engledew



Dr. Massimo Antognozzi



Dr. Arturas Ulcinas



Dr. James Vicary



Dr Laurent Bozec



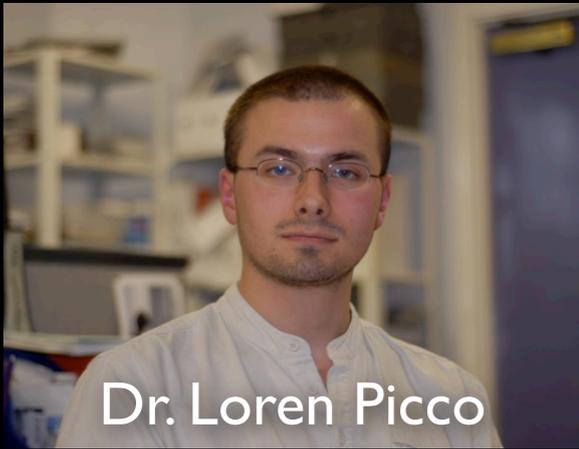
Prof Mike Horton

BBSRC
EPSRC
FP6
Tips4Cells
RCUK
Unilever



Prof Tatsuo Ushiki

Prof Osamu Hoshi



Dr. Loren Picco



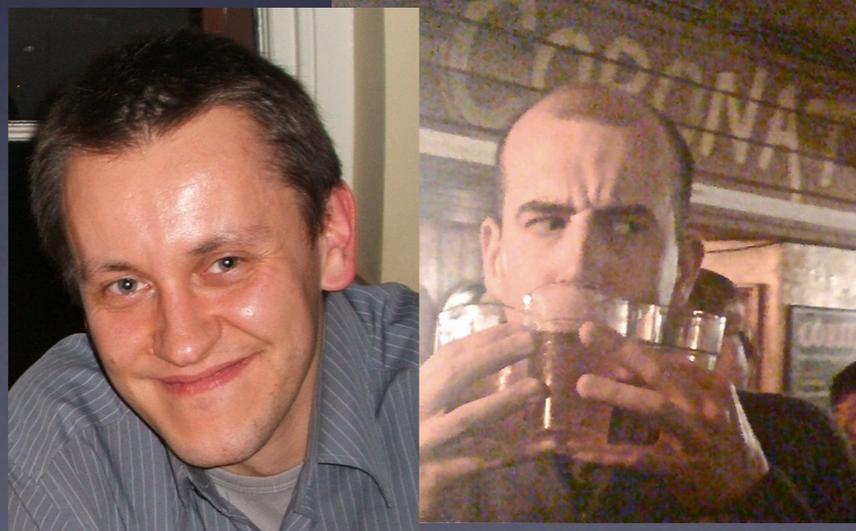
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Dave Engledew



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