Other emerging dAFM techniques

Slide Smith ~ 46:00

- Multi-frequency AFM
- Sub-surface imaging

Stan ~50:30

- High-speed/video rate AFM

LL

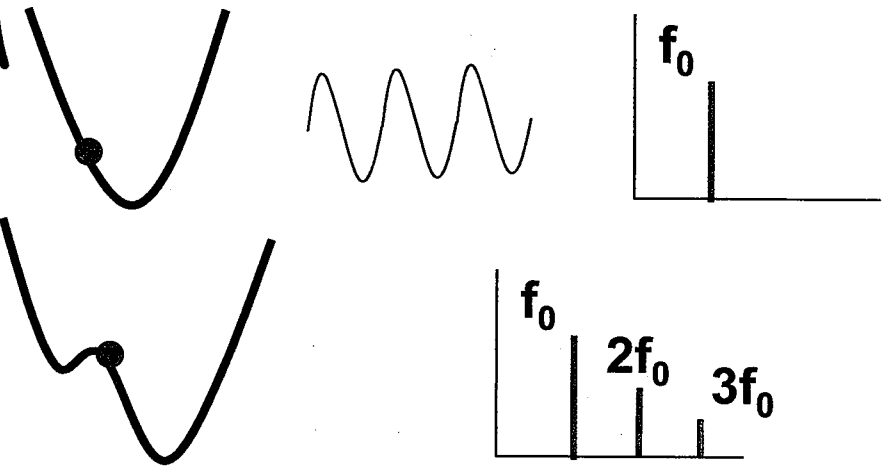
PURDUE
UNIVERSITY

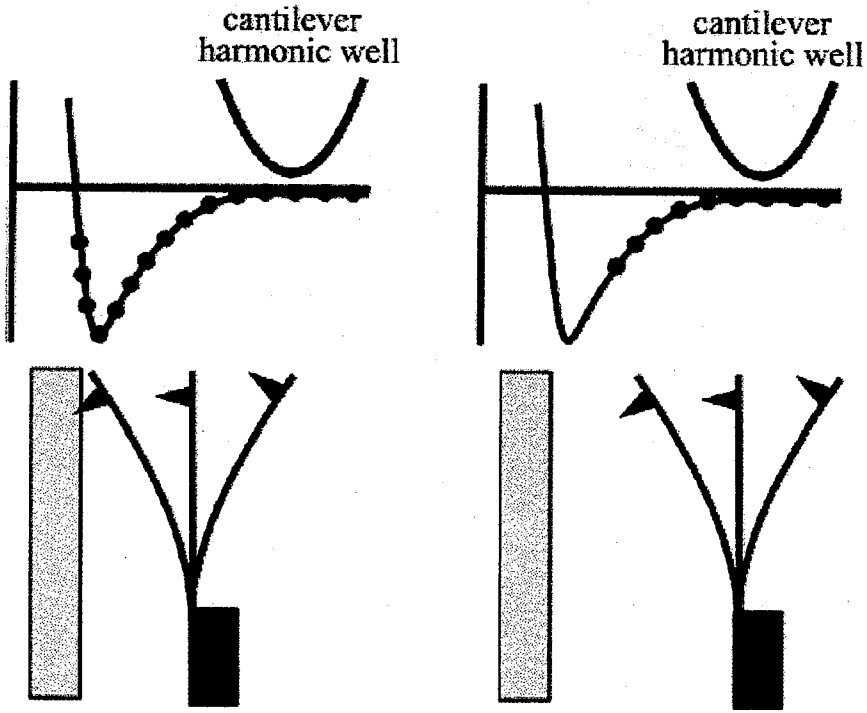
Multi-frequency AFM

- Generic term applied to all methods where ei ther cantilever is excited and or measured at more than one frequency
  - Kelvin force microscopy in tapping mode (discussed in class)
  - Higher harmonic imaging
  - Internally resonant or "harmonic" cantilevers
  - Momentary excitation in liquids
  - Bimodal or dual AC mode
  - Band excitation (Oakridge, S. Jesse, S. Kalinin)

Multi-frequency AFM

- Higher harmonic dAFM
- Insight:





mechanical properties &
loss mechanisms

Hamaker constants,
electro-statics & -dynamics

U. Durig, New J. Phys.,2, 2000
M. Stark et al, PNAS, 99, 2002
Crittenden et al, PRB, 72, 2006

PURDUE
UNIVERSITY

Multi-frequency AFM

Higher harmonic dAFM

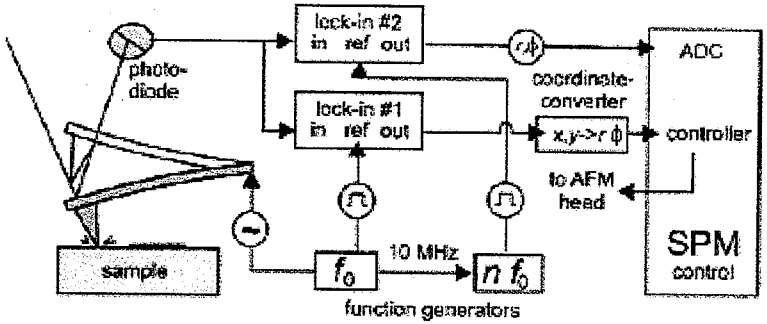


FIG. 1. Experimental setup for the detection of anharmonic signals. A commercial AFM is equipped with a second lock-in amplifier for the detection of anharmonic signals.

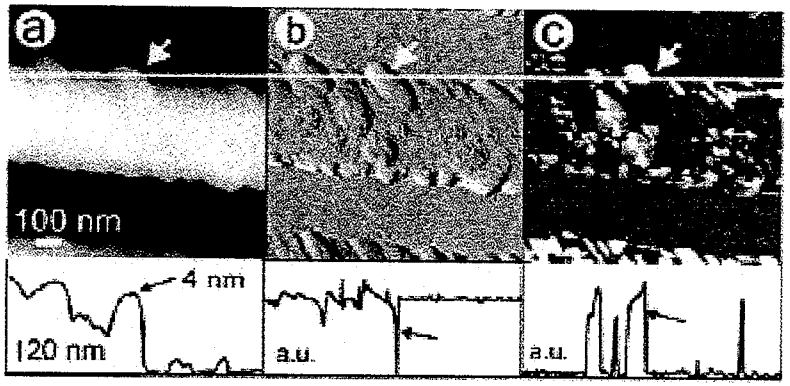


FIG. 4. Detail of a silicon test structure imaged in tapping mode (scan direction right to left). (a) Topography, (b) control error, and (c) eighth harmonic. The instabilities due to the bistable behavior of the system are difficult to be seen in the conventional images (a) and (b). However, in the harmonic image (c) a strong contrast prevails.

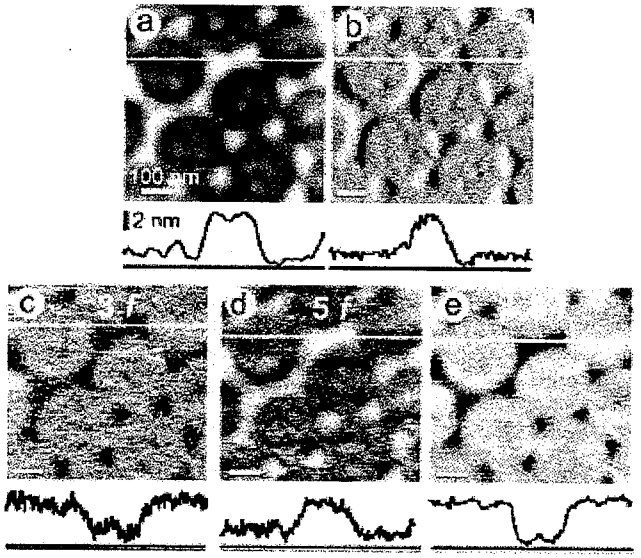


FIG. 2. (a) Topographic (b) control error, and (c)–(e) higher order harmonic images of a 4-nm-thick Pt–C test structure on a fused silica cover slip. The driving frequency was $f=52.2$ kHz, the detection frequencies were (c) $3f=156.6$ kHz, (d) $5f=261.0$ kHz, and (e) $8f=417.6$ kHz.

PURDUE UNIVERSITY

Heckl and Stark, RSI, 74, 2003

Amplifying higher harmonics

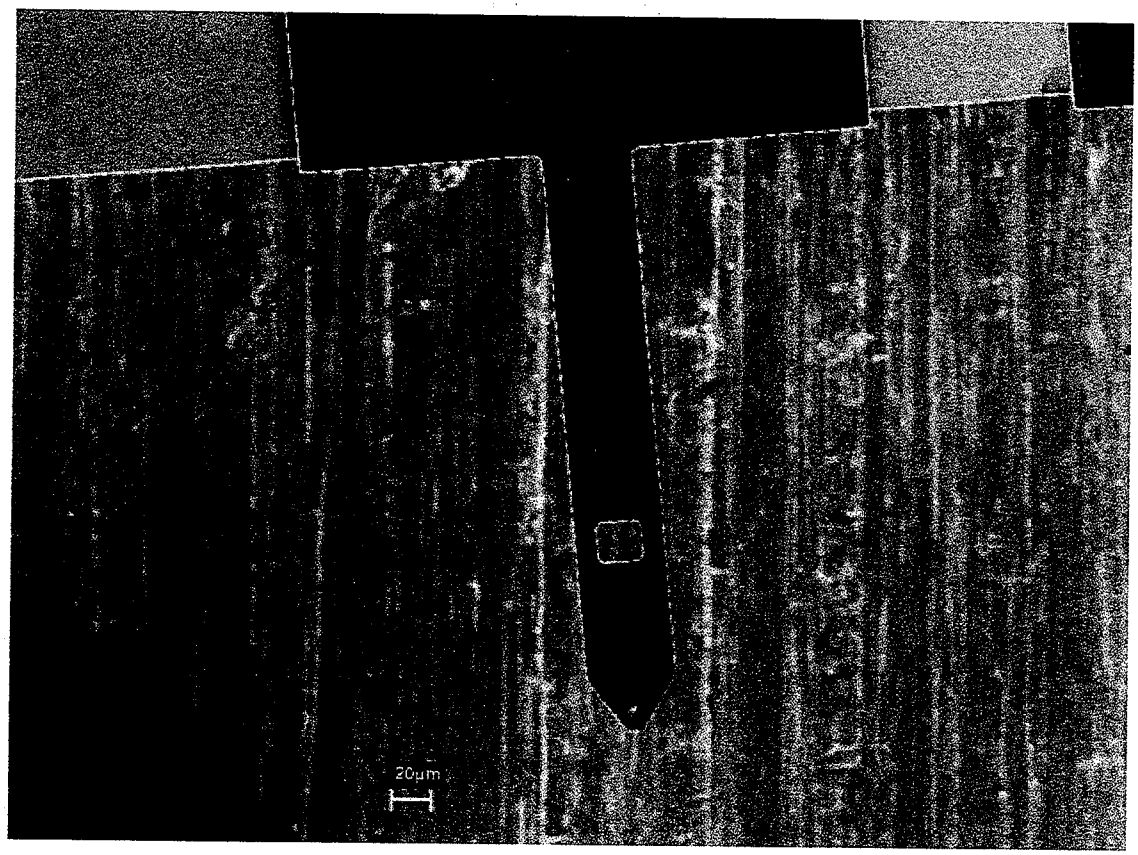


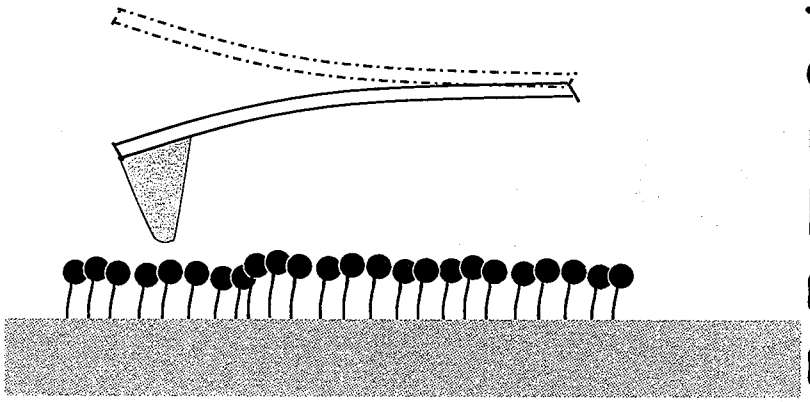
Fig. 6. SEM image of a harmonic cantilever. Width, length and thickness of the cantilever are 50, 300, and 2.2 μm, respectively. The rectangular opening is 22 μm × 18 μm and centered 190 μm away from the cantilever base.

- Tune second eigenmode frequency to an integer multiple of the fundamental

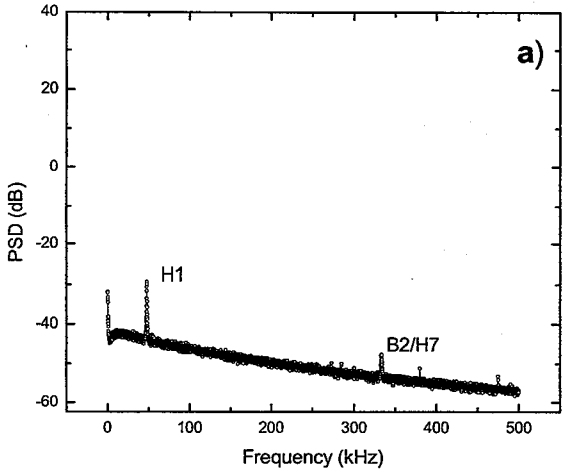
PURDUE UNIVERSITY

Sahin et al, Sensors and Actuators, 114, 2003, also PRB (69), 2004

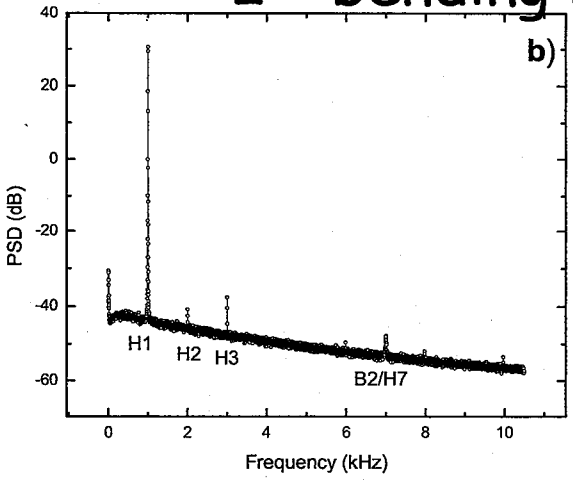
Using tuned cantilevers



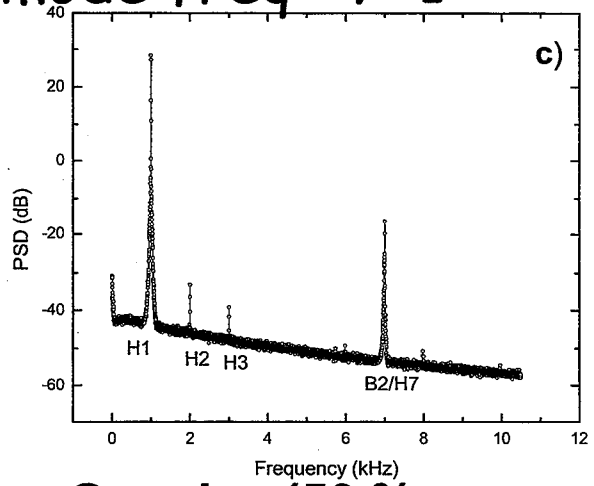
- In attractive regime, vibration spectrum depends on local vdW and electrostatic forces
- Experiments performed using 47 kHz microcantilever on wild and mutant bacteriorhodopsin membrane
- 2nd bending mode freq ~7*1st



Thermal vibration



Driven in air



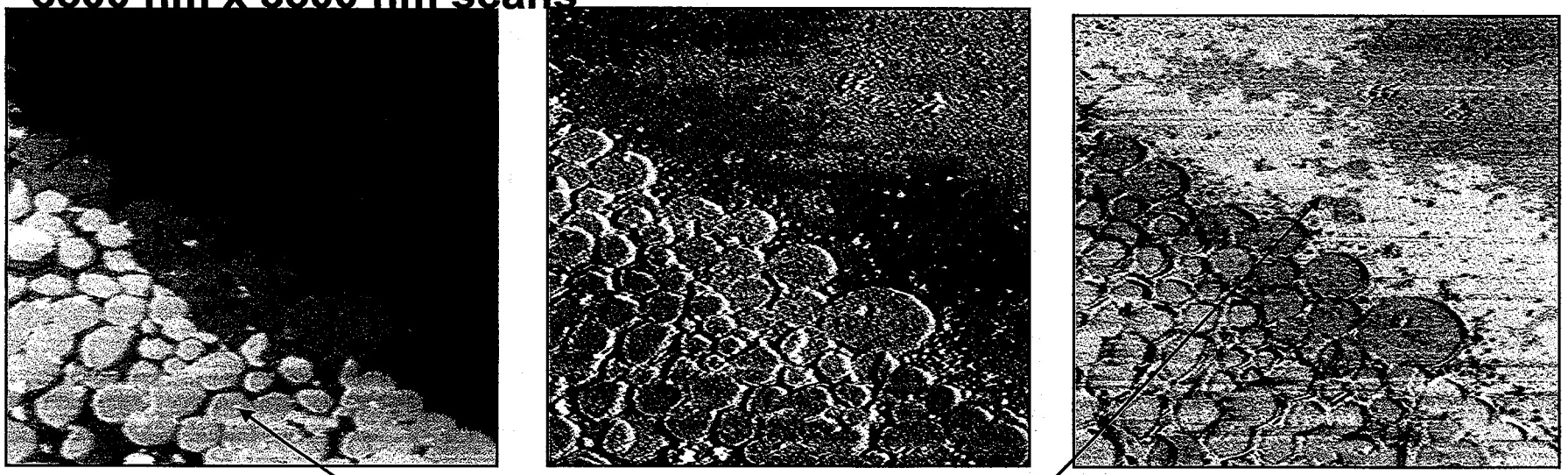
On mica (50 % setpoint)

PURDUE UNIVERSITY

Crittenden et al, PRB, 72, 2006

Using tuned cantilevers

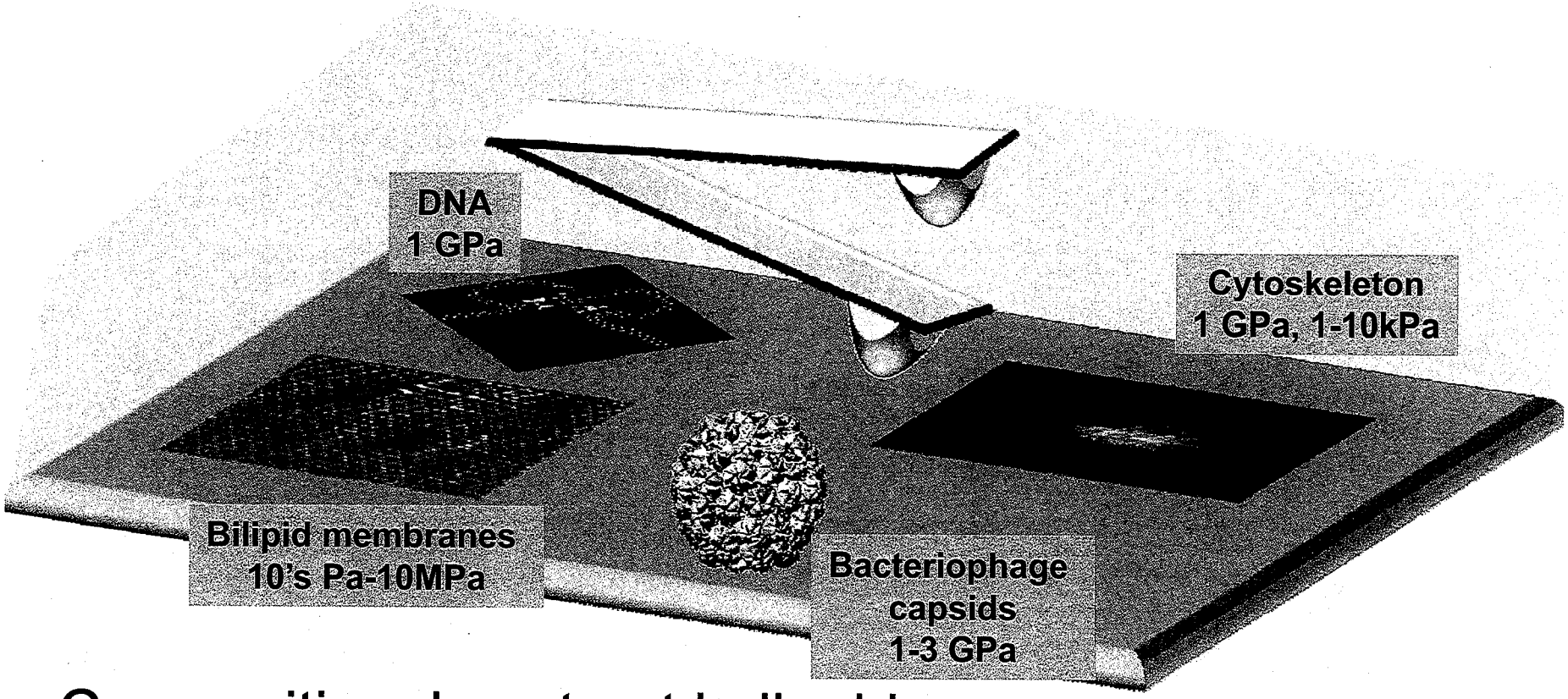
3500 nm x 3500 nm scans



- Clear distinction between lipids and proteins
- Presence of internal resonance critical in the method
- The method shows promise for the measurement of local Hamaker constants of soft biomolecules
- Can be extended to electrostatic force microscopy

PURDUE
UNIVERSITY

Momentary excitation in liquids



Compositional contrast in liquids

- Van Noort et al, (Langmuir, 1999)
- Preiner, Hinterdorfer et al (PRL, 2007) Second harmonic
- Xu, Melcher, Raman, Reifenberger (PRL, 2009) Momentary Excitation

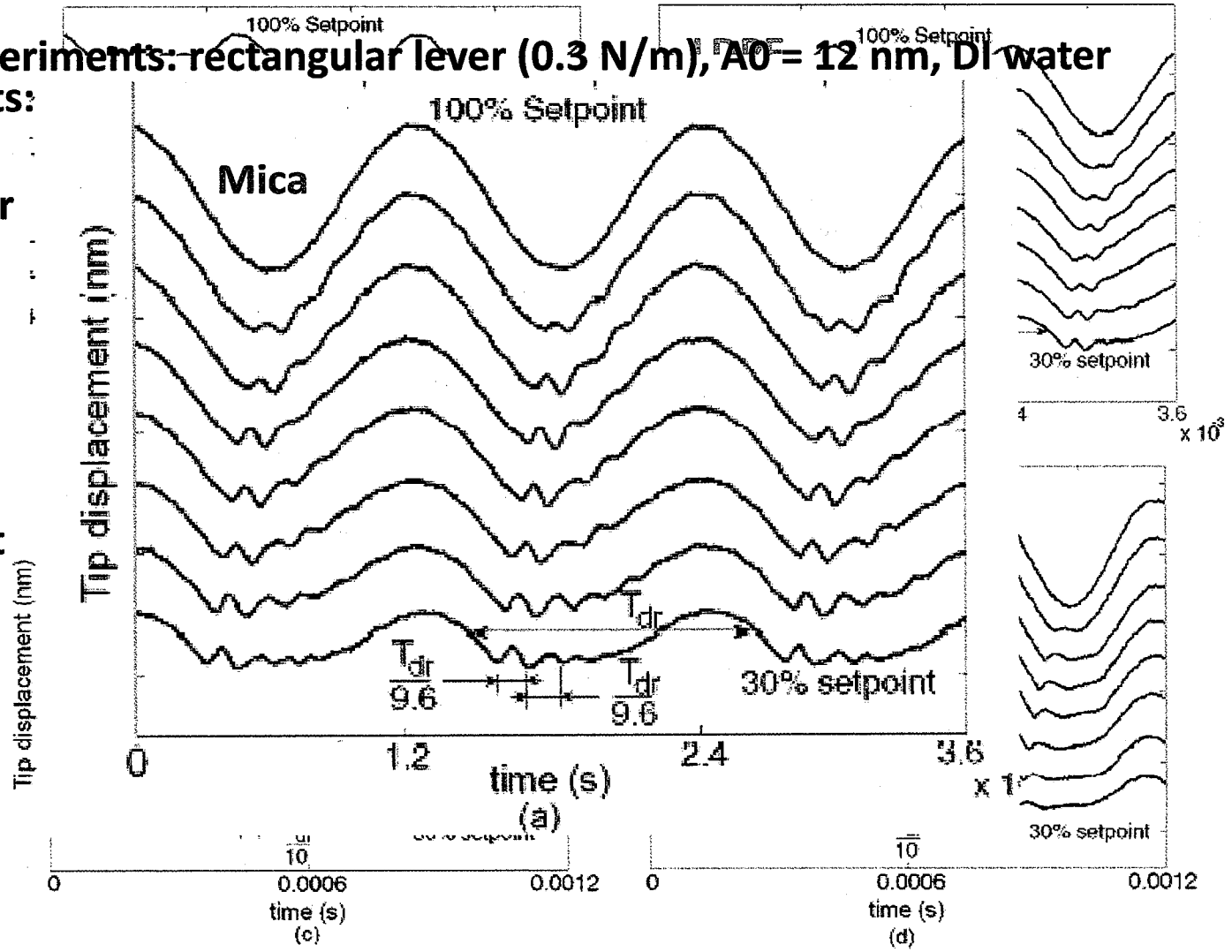
Momentary excitation - experiments

(Basak and Raman, App. Phys. Lett., 2007)

Experiments: rectangular lever (0.3 N/m), A0 = 12 nm, DI water

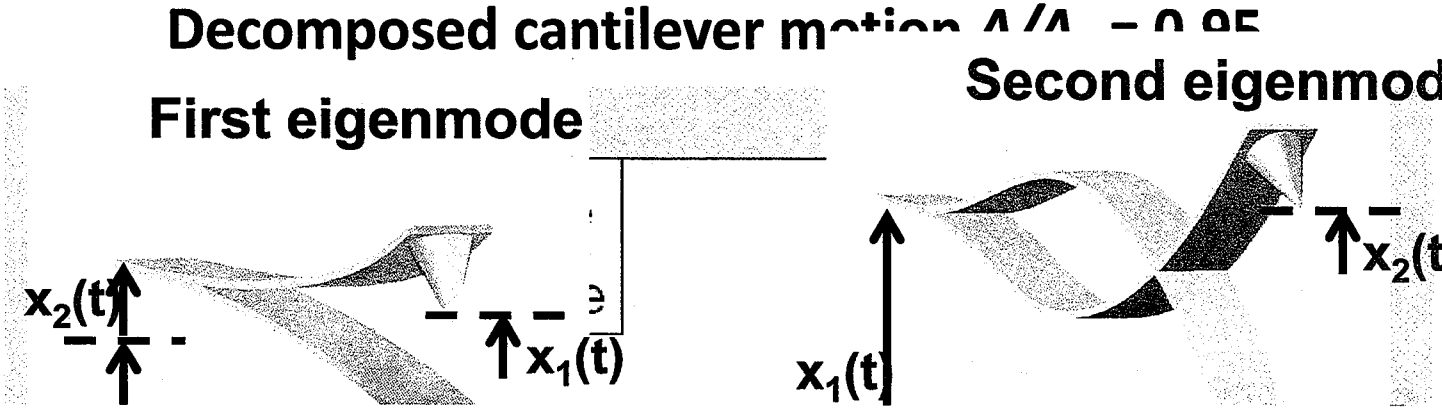
Experiments: 0.3 N/m rectangular lever

Experiments: 0.1 N/m triangular lever

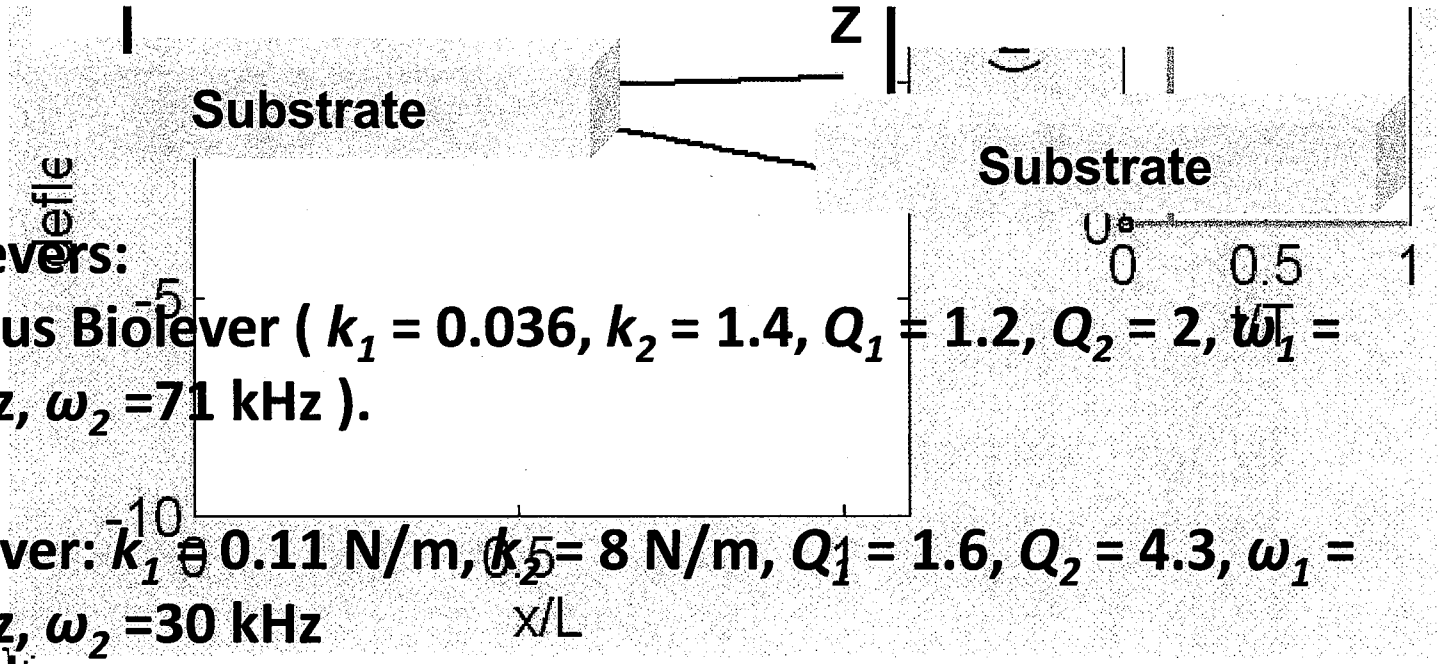


PURDUE UNIVERSITY

Momentary excitation- theory



- **Momentary excitation is greater on stiffer samples**



Cantilevers:

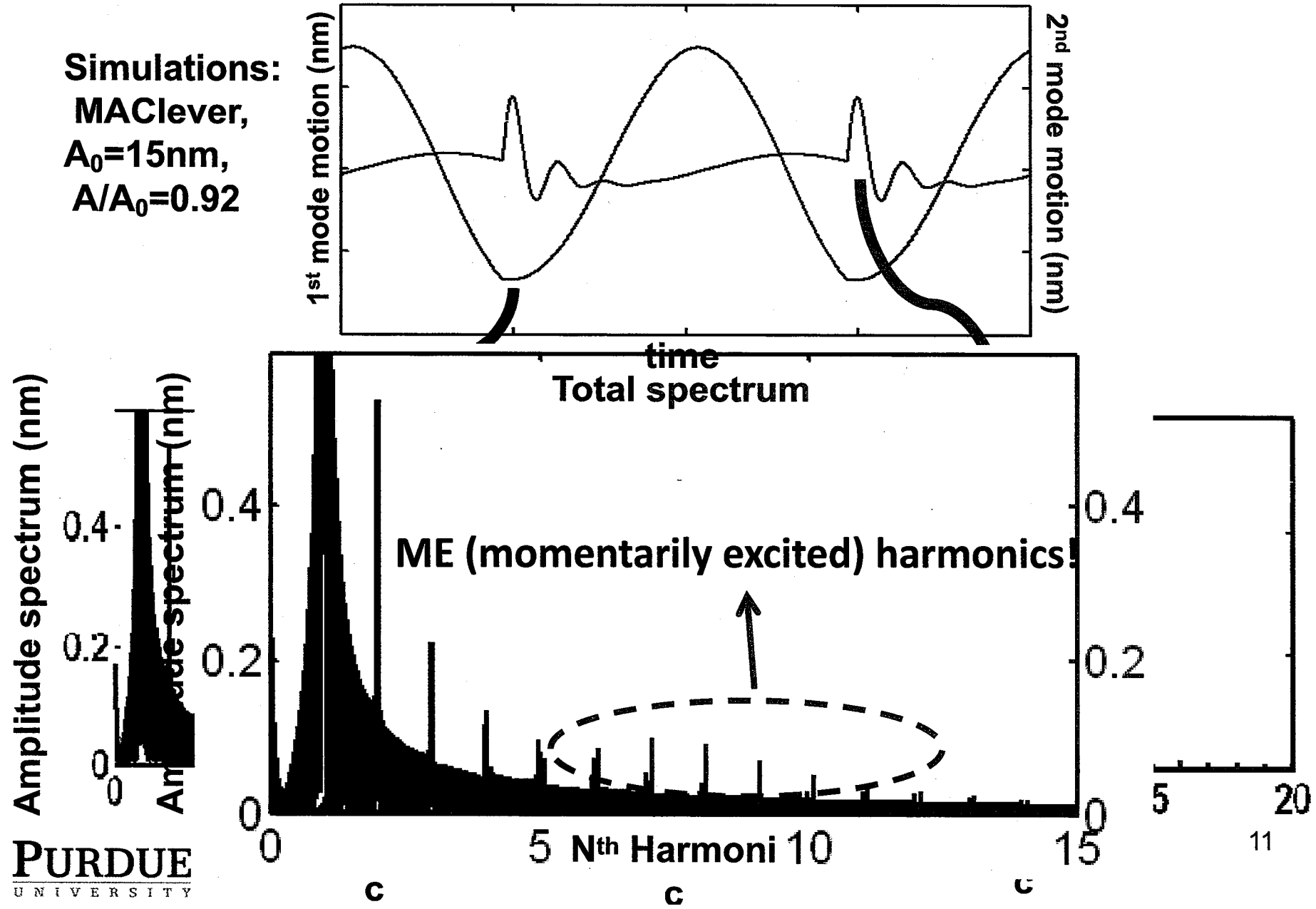
Olympus Biolever (k_1 = 0.036, k_2 = 1.4, Q_1 = 1.2, Q_2 = 2, ω_1 = 9.3 kHz, ω_2 =71 kHz).

MAClever: k_1 = 0.11 N/m, k_2 = 8 N/m, Q_1 = 1.6, Q_2 = 4.3, ω_1 = 3.5 kHz, ω_2 =30 kHz

PURDUE UNIVERSITY

Momentarily Excited (ME) Harmonics

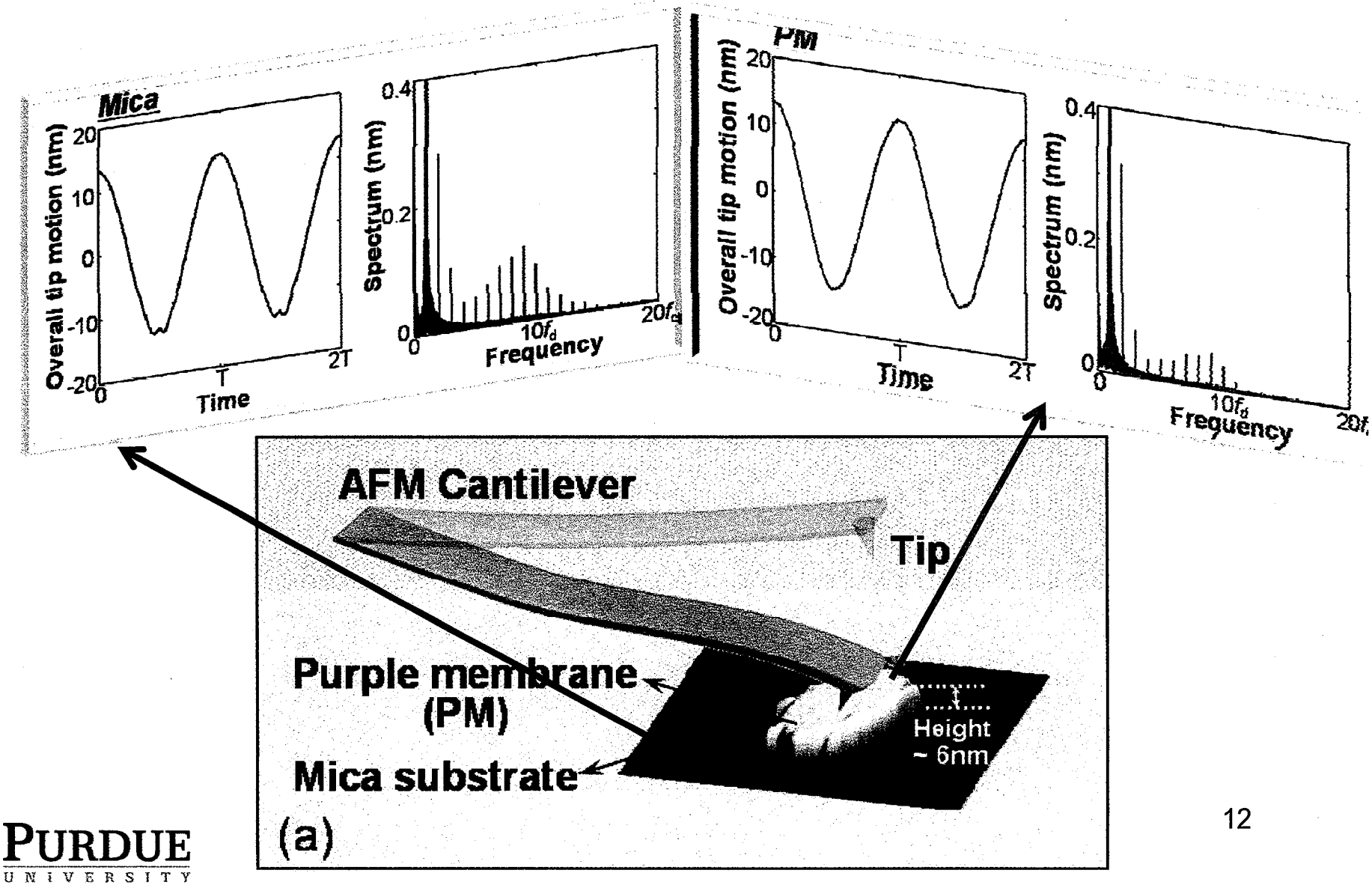
Simulations:
MAClever,
A_0=15nm,
A/A_0=0.92



PURDUE UNIVERSITY

Application to elasticity mapping

Xu et al. Phys. Rev. Lett. 2009

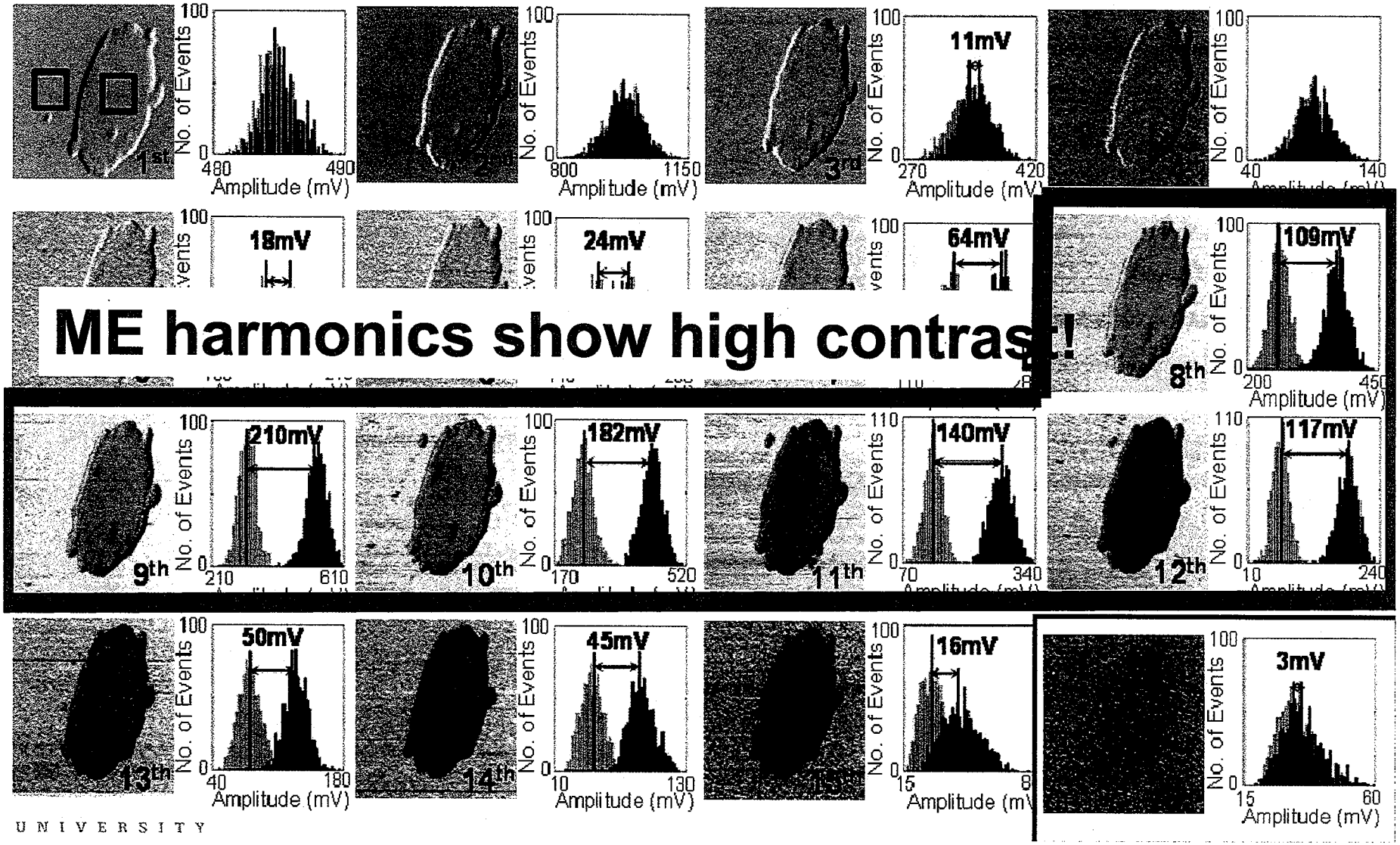


PURDUE UNIVERSITY

Higher Harmonic Imaging

Xu et al Phys. Rev. Lett. 2009

Experiment: purple membrane on mica, k_1=0.11 N/m, A_0=15nm, A_{set}

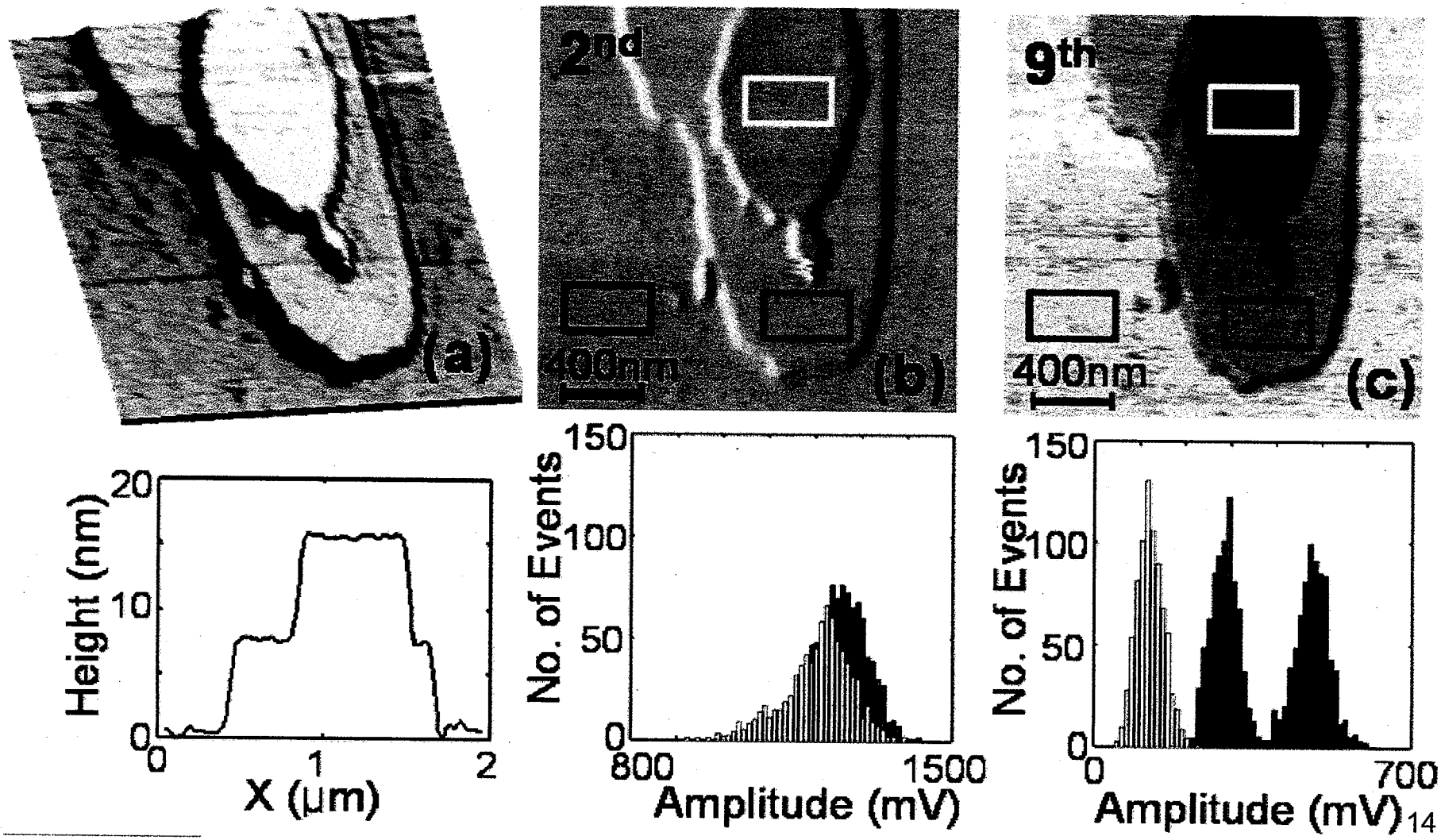


ME harmonics show high contrast!

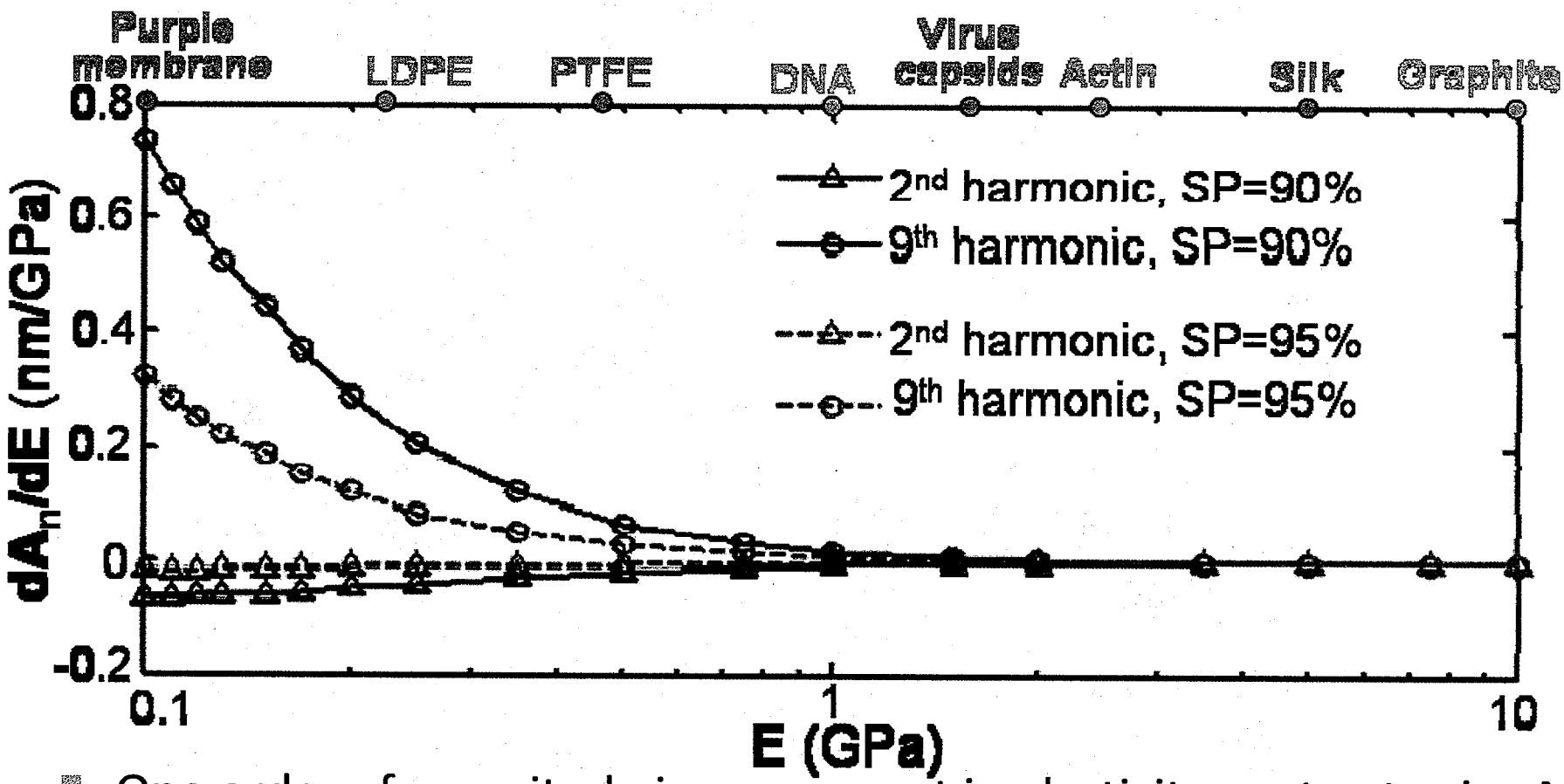
Elasticity contrast for soft samples

Xu, Melcher, Basak, Reifenberger Raman, in Phys. Rev. Lett. 2009

k_1=0.11 N/m, A_0=12.5nm, $A_{setpoint}$=92%, buffer: 300 mM KCl, 20 mM Tris-H



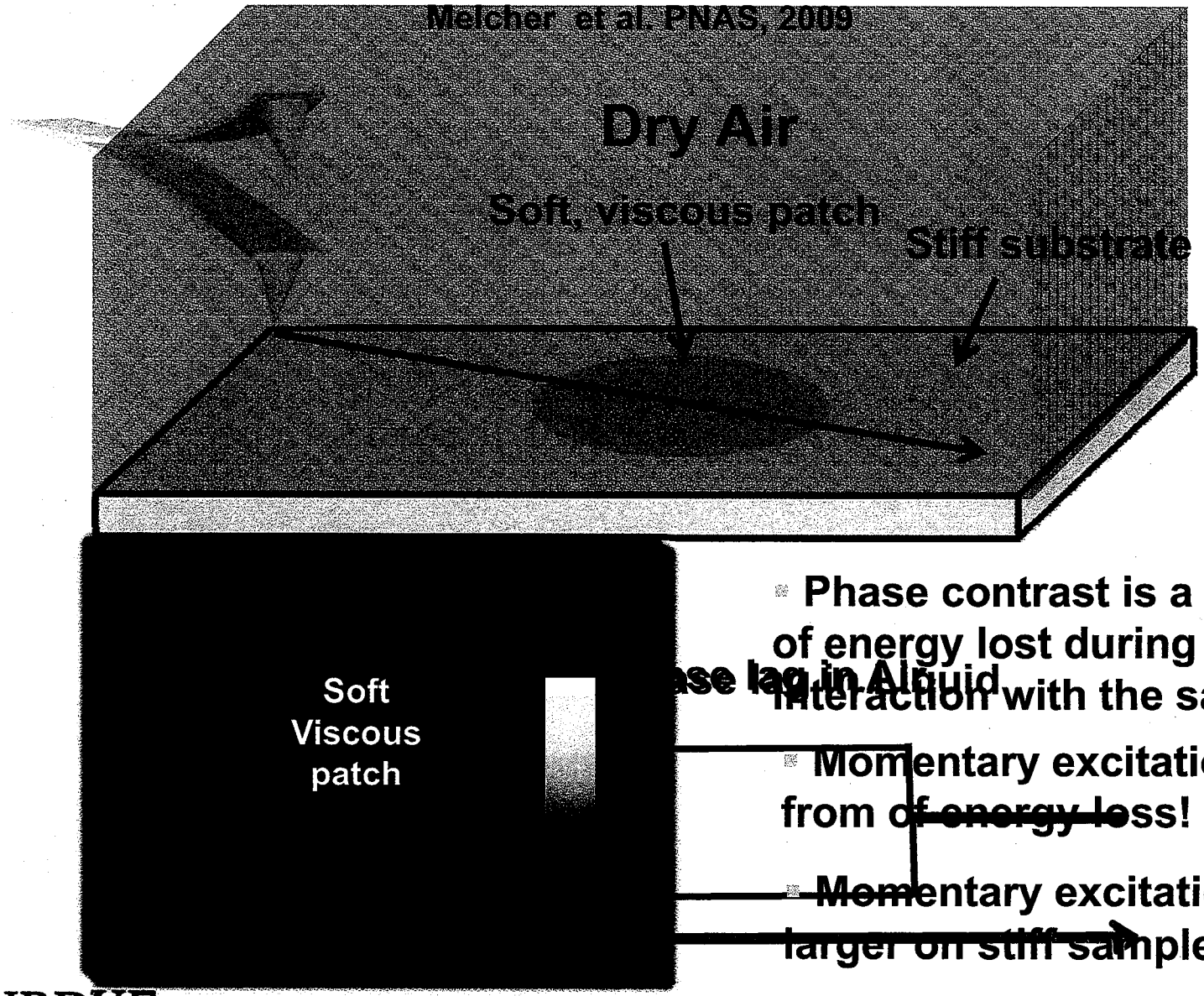
Origin of contrast in ME harmonic images



- One order of magnitude improvement in elasticity contrast using M E harmonics compared to 2nd harmonic for soft materials
- ME harmonics are closely correlated to contact time (which varies i nversely with sample elasticity)
- Image contrast seen is entirely local elasticity contrast

PURDUE UNIVERSITY

Phase contrast imaging in liquids



- Phase contrast is a measure of energy lost during interaction with the sample.
- Momentary excitation is a from of energy loss!
- Momentary excitation is larger on stiff samples

For soft levers in liquids: Phase contrast images = local elasticity maps

PURDUE UNIVERSITY

Experimental data

Melcher et al. (PNAS 2009)

Purple Membrane on mica substrate

ϕ29 virus capsid on a glass substrate

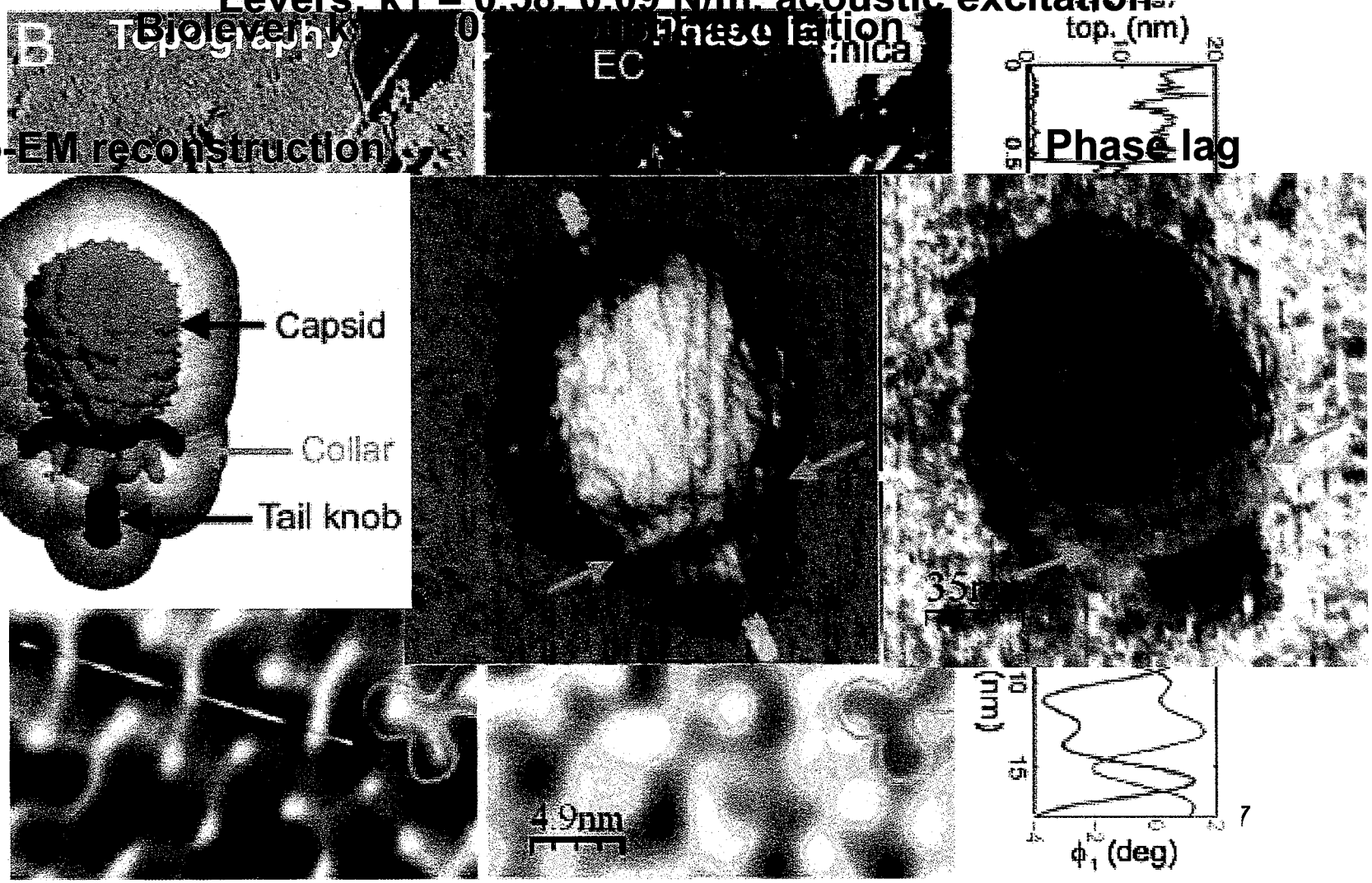
buffer solution: 300 mM KCl, 20 mM Tris-HCl, pH 7.8

buffer: TMS pH 7.8

Levers: k1 = 0.58, 0.09 N/m, acoustic excitation

Bioleverh

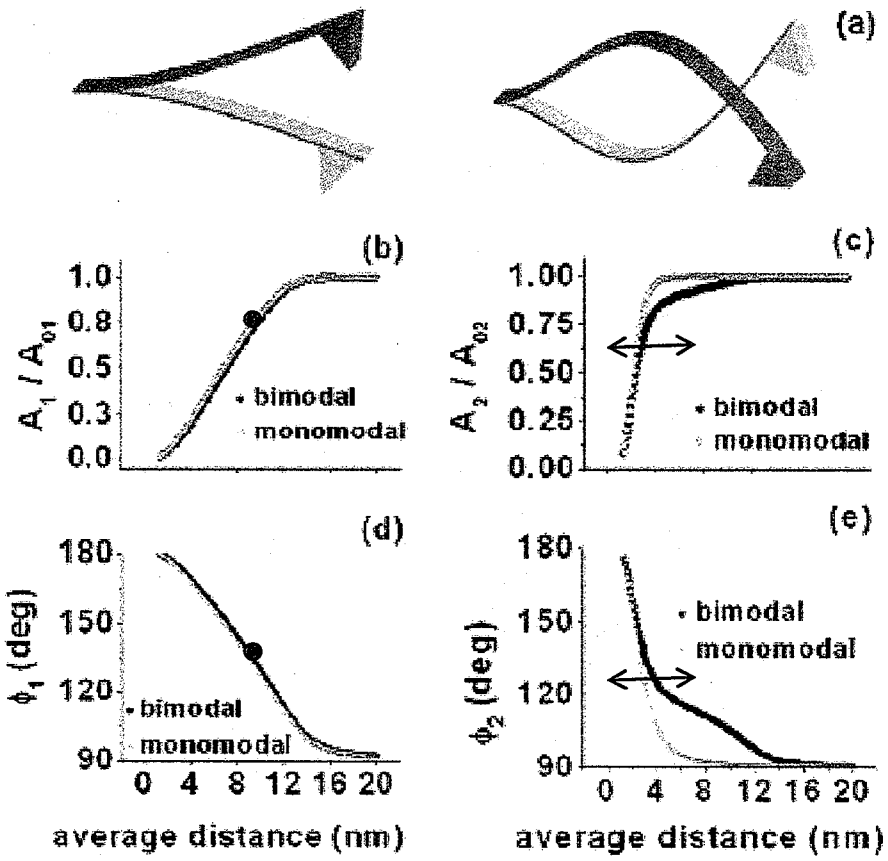
Cryo-EM reconstruction



PURI
UNIVER

Multi-frequency AFM

- Bimodal or dual *AC*
  - Key insight is that the second mode A_2, ϕ_2 varies in time
  - Thus ϕ_2 not only meas ures dissipation but also conservative tip-sample interactions!
  - It becomes possible to see material contrast in the attrac tive regime!



Rodriguez and Garcia, APL,84(3), 2004
Lozano and Garcia, PRL, 100(7), 2008
Lozano, Garcia, PRB, 79(1), 2009
R. Proksch, APL, 89(11), 2006

Multi-frequency AFM

- Bimodal or dual *AC*

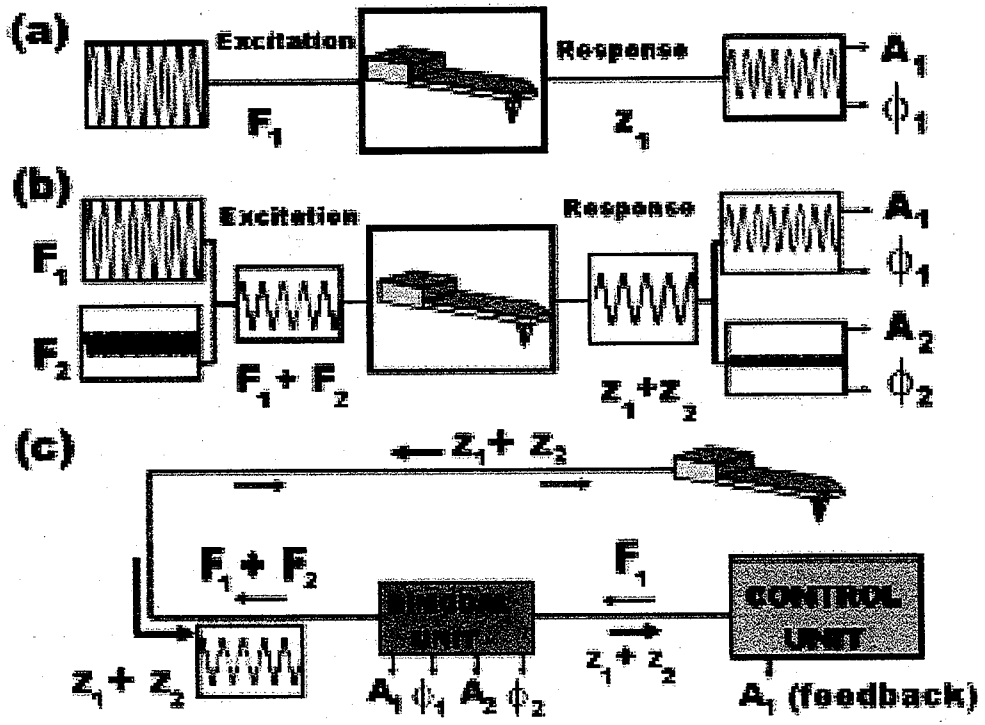


Figure 1. Comparison between amplitude modulation and bimodal AFM. (a) AM-AFM (monomodal excitation). (b) Bimodal AFM. (c) Schematics of the bimodal AFM instrument. The bimodal excitation/detection unit performs the multifrequency excitation and the multicomponent signal processing while the control unit runs the feedback.

Martinez et al, Nanotechnology, 19, 2008

Multi-frequency AFM

- Bimodal or dual *AC*

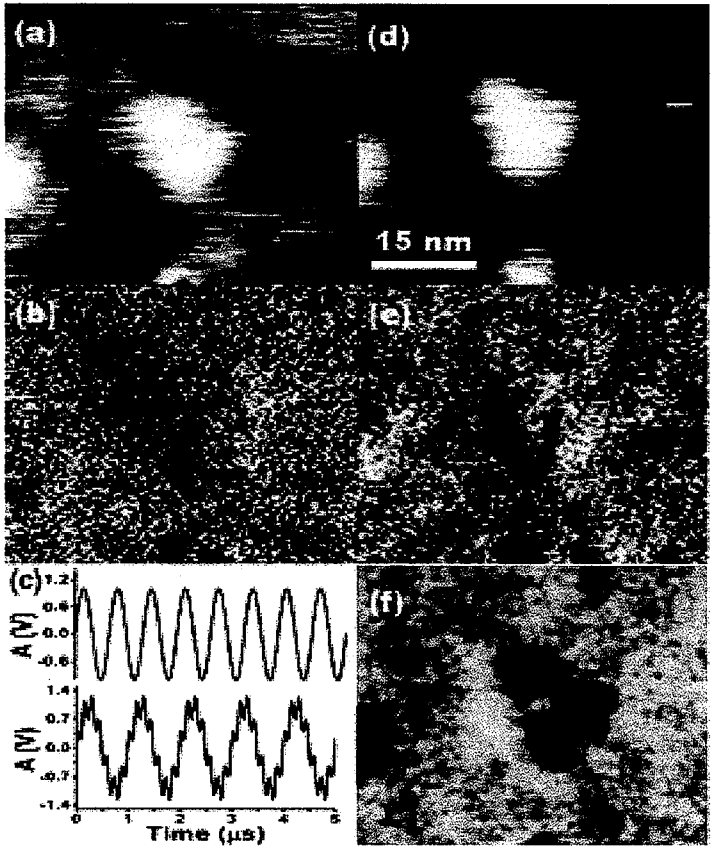


Figure 3. Comparison between AM-AFM and bimodal AFM images of IgG antibodies. (a) Topography and (b) phase images of an IgG obtained in AM-AFM. (c) Tip oscillation in AM-AFM (top) and bimodal AFM (bottom). (d) Topography in bimodal AFM. (e) Phase shift image of the first mode in bimodal AFM. (f) Bimodal AFM phase image (second mode) of the same antibody. The image shows a Y-shaped object.

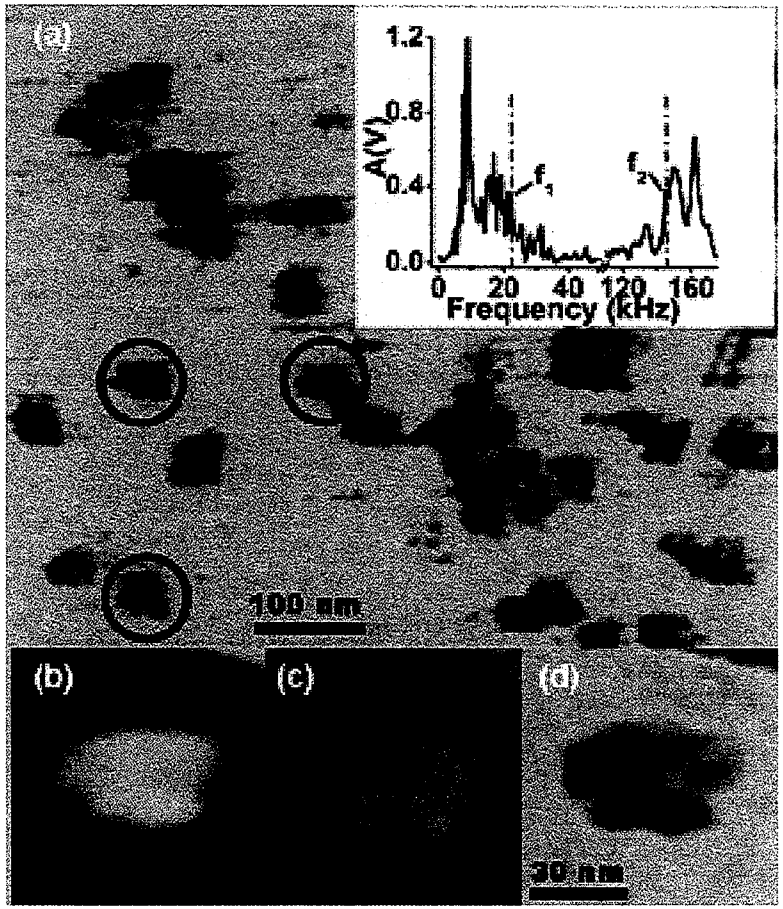


Figure 7. (a) Bimodal AFM phase images (second mode) of IgM antibodies in water. The objects that show a pentagonal shape are marked by circles. The inset shows the frequency spectrum of a commercial cantilever in water. The dashed lines indicate the frequencies of the first and second flexural modes of the cantilever. They were determined by measuring the thermal noise spectrum. (b) Topography of an isolated antibody. (c) First mode phase image and (d) bimodal AFM phase image (second mode) of the same antibody.

PURDUE UNIVERSITY

Martinez et al, Nanotechnology, 19, 2008

Multi-frequency AFM

- Bimodal or dual *AC*

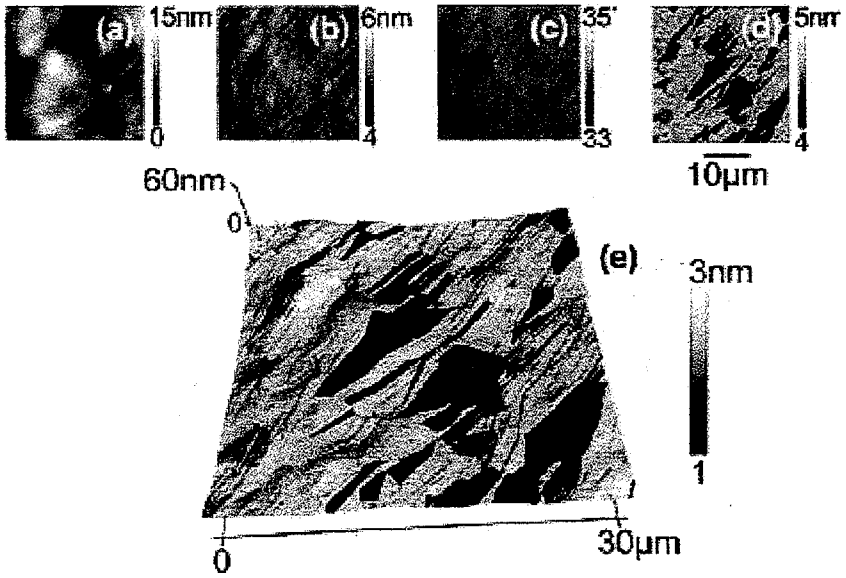


FIG. 2. (Color online) HOPG graphite surface, 30 μm scan. The cantilever was driven at its fundamental (~69.5 kHz) and second eigenfrequency (~405 kHz). (a) shows the topography and (b) is the fundamental amplitude channel, used for the feedback error signal. The fundamental phase image (c) shows an average phase lag of ~34° indicating that the cantilever was in repulsive mode for the entire image. The second mode amplitude is shown in (d). The three dimensional rendered topography colored with the second mode amplitude is shown in (e). This method of display allows easy spatial correlation of the two channels.

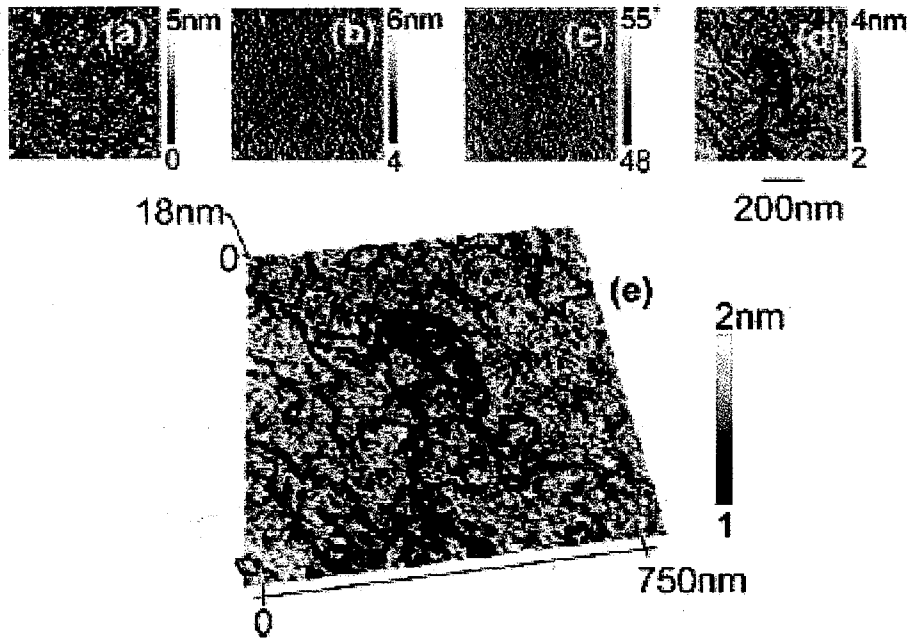


FIG. 3. (Color online) Dense mat of DNA imaged in buffer, 750 nm scan. The 60 μm Bio-Lever was driven at its fundamental resonance (~8.5 kHz) and at its second mode (~55 kHz). The topography (a), fundamental amplitude (b), and fundamental phase (c) all show very little differentiated contrast. The second mode amplitude (d) shows clear, high contrast images of what appear to be strands of DNA molecules. The second mode amplitude was painted onto the three dimensional rendered topography (e) to allow spatial correlation of the two data channels.

R. Proksch, APL, 89, 2006

27 RAMAN

Other emerging dAFM techniques

- Multi-frequency AFM
- Sub-surface imaging
- High-speed/video rate AFM

Slide switch at ~46:02
Start ~50:30

PURDUE
UNIVERSITY