



**The Abdus Salam
International Centre for Theoretical Physics**



2063-21

ICTP/FANAS Conference on trends in Nanotribology

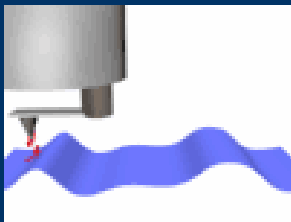
19 - 24 October 2009

Ultrasonic nanolithography on hard substrates

Teresa Cuberes
*University of Castilla La Mancha
Almaden
Spain*

ULTRASONIC NANOLITHOGRAPHY ON HARD SUBSTRATES

Teresa Cuberes

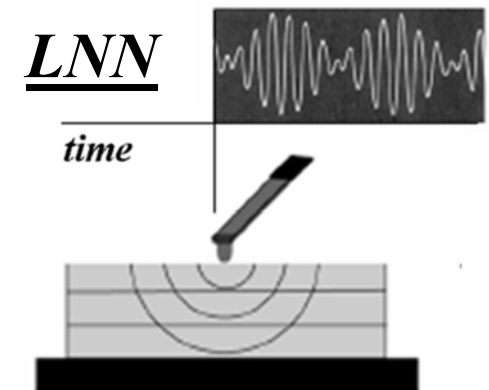


teresa.cuberes@uclm.es



[http://www.uclm.es/organos/
vic_investigacion/gruposweb/
nanotecnologia/](http://www.uclm.es/organos/vic_investigacion/gruposweb/nanotecnologia/)

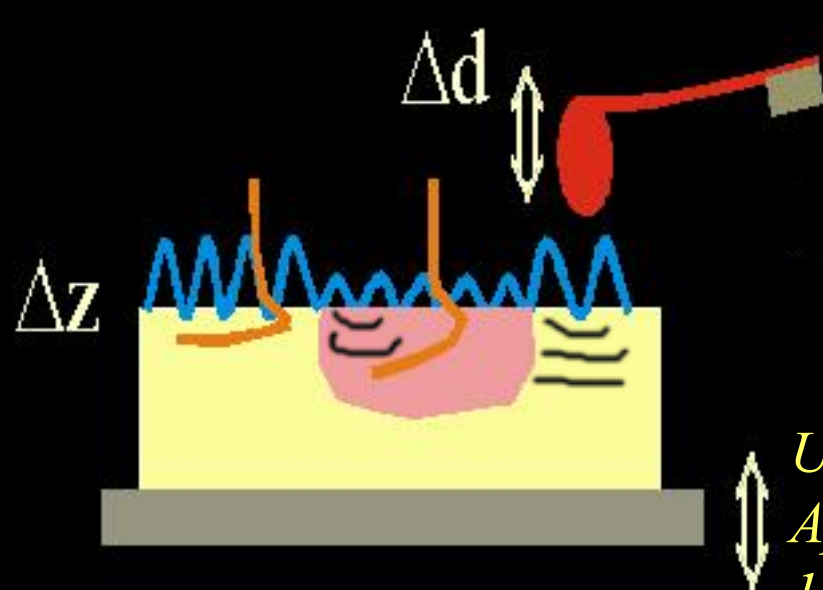
*The Laboratory of Nanotechnology,
Almaden, SPAIN*



Outline

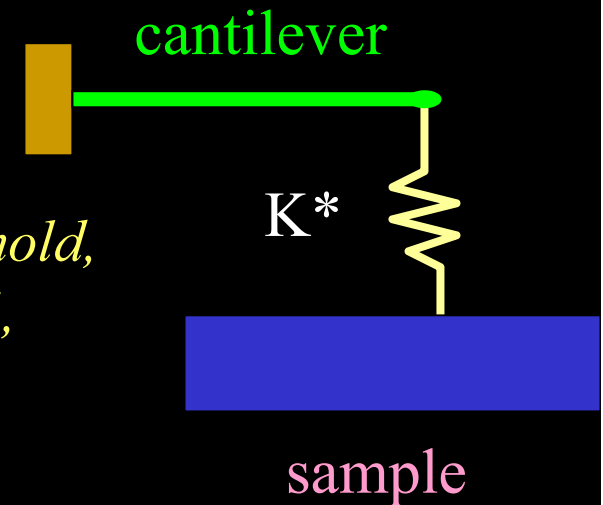
- *Mechanical-diode-mode Ultrasonic AFM*
 - *The Mechanical-Diode (MD) effect: UFM, HFM, IC-HFM*
- *Tribology with ultrasonic-AFM*
 - *Study of adhesion hysteresis and energy dissipation with UFM*
 - *Study of friction and lubrication with MD- UFFM*
 - *Control of friction and generation of wear using ultrasound*
- *Ultrasonic nanolithography on hard substrates*
 - *Results on Si(111)*
- *Summary*

ACOUSTIC ATOMIC FORCE MICROSCOPY (AFAM)



The cantilever can support high-frequency resonant modes

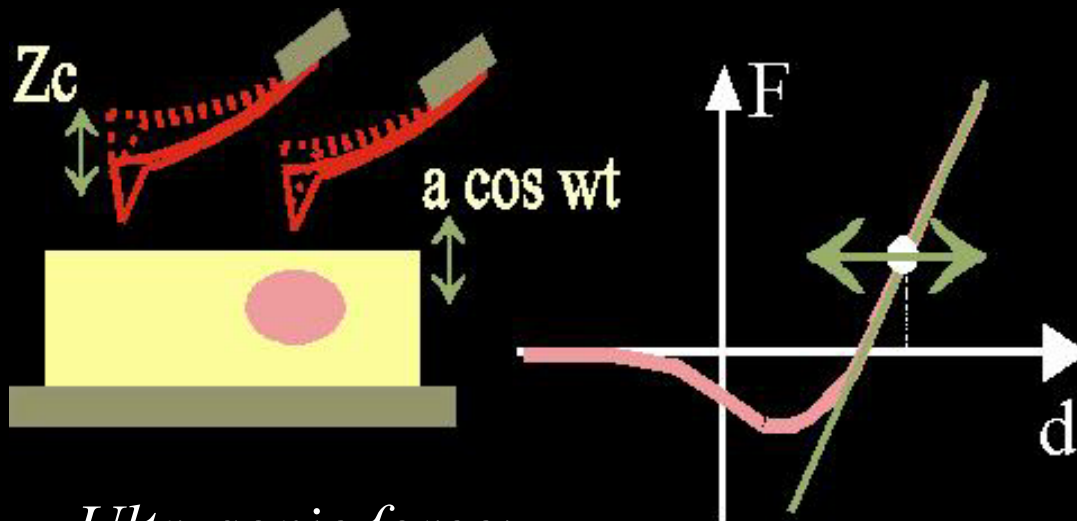
*U. Rabe and W. Arnold,
Appl. Phys. Lett. 64,
1493 (1993)*



The tip-sample interaction is kept in the linear tip-sample force regime

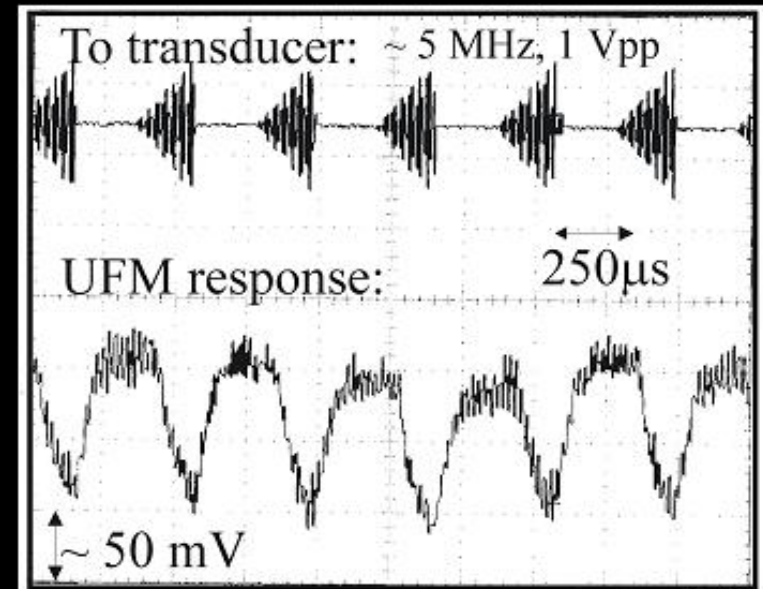
AFAM provides information about **sample elasticity** with nanoscale lateral resolution. Measured magnitude: Resonance frequency of the cantilever high-order modes (the contact stiffness and the Young modulus can be evaluated)

ULTRASONIC FORCE MICROSCOPY



Ultrasonic force:

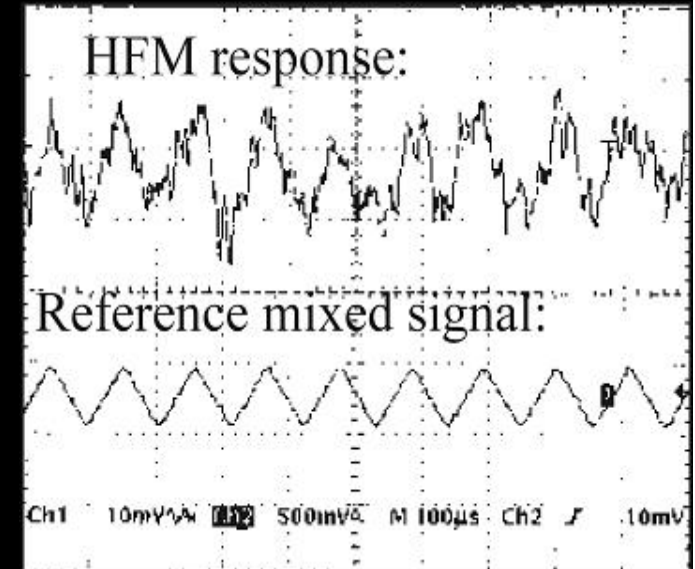
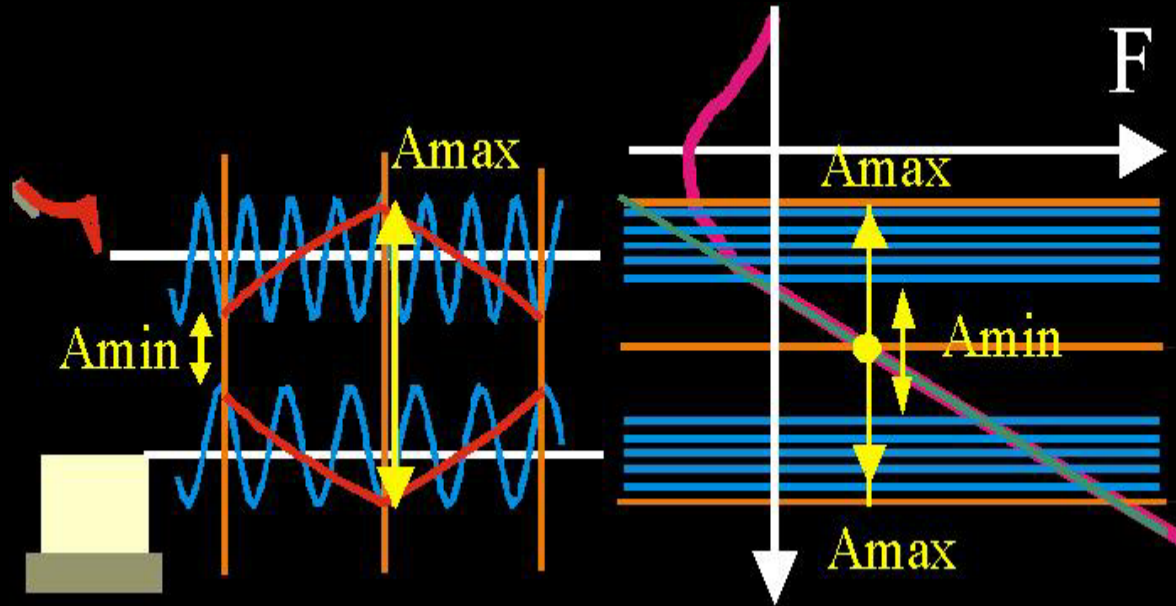
$$kz_c = \frac{1}{T} \int_0^T F(z_c - z_s - A \cos \omega t) dt$$



O. Kolosov and K. Yamanaka, Jpn. J. Appl. Phys. 32, L1095 (1993)

*UFM provides information about sample elasticity and adhesion with nanoscale lateral resolution. Measured magnitude: static cantilever displacement induced by the *ultrasonic force*.*

HETERODYDYNE FORCE MICROSCOPY



Phase-HFM makes possible to study **dynamic relaxation processes** in *nanometre volumes* with a *time-sensitivity of nanoseconds*

M. T. Cuberes et al.
J. Phys. D. Appl.
Phys. 33, 2347 (2000)

SCANNING NEAR FIELD ULTRASOUND HOLOGRAPHY

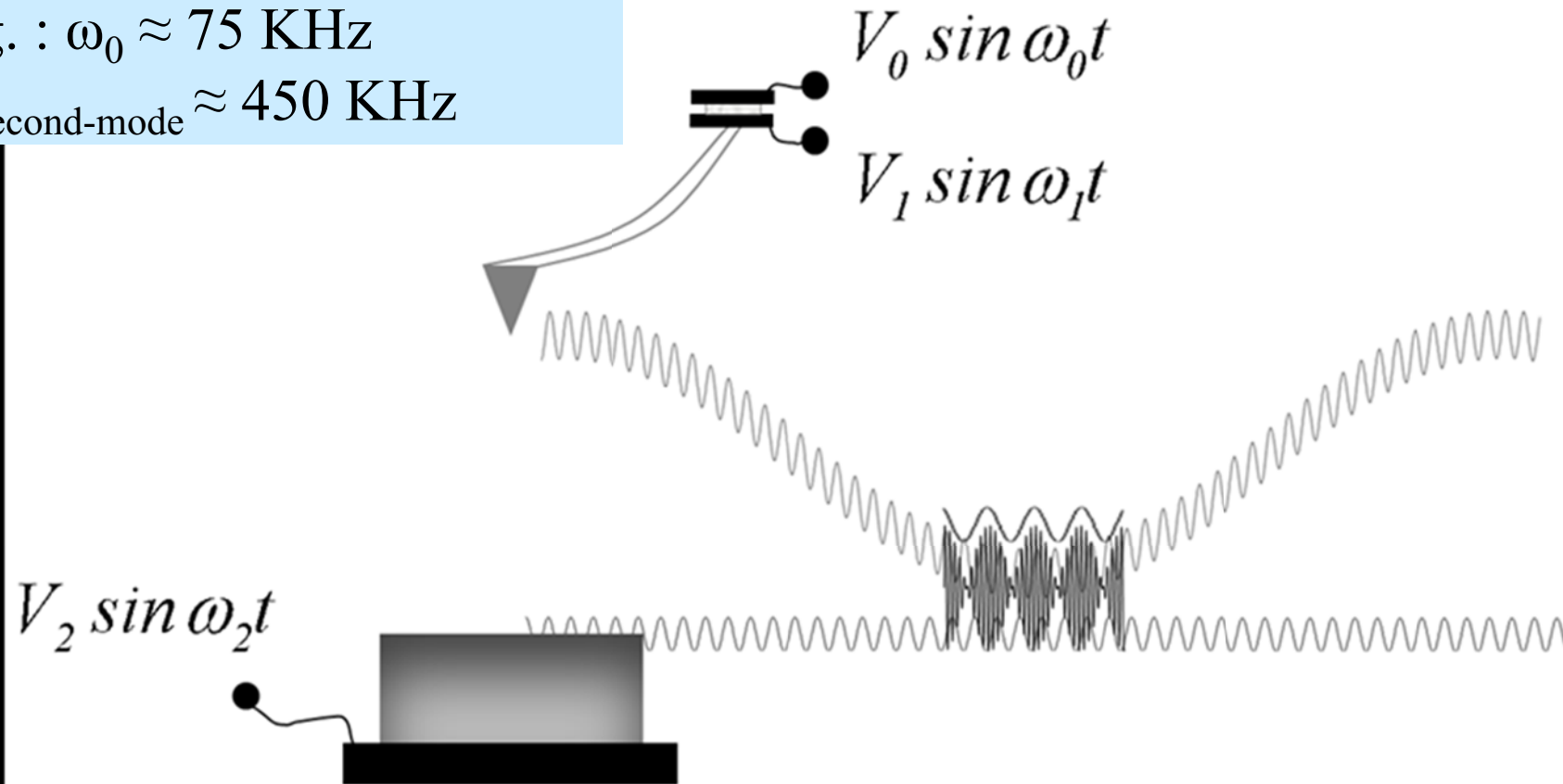
Phase-SNFUH provides **elastic information** of **buried features** with *great sensitivity*.

Shekhawat and Dravid.
Science 310, 90 (2005)

INTERMITTENT-CONTACT HETERODYNE FORCE MICROSCOPY

e. g. : $\omega_0 \approx 75$ KHz

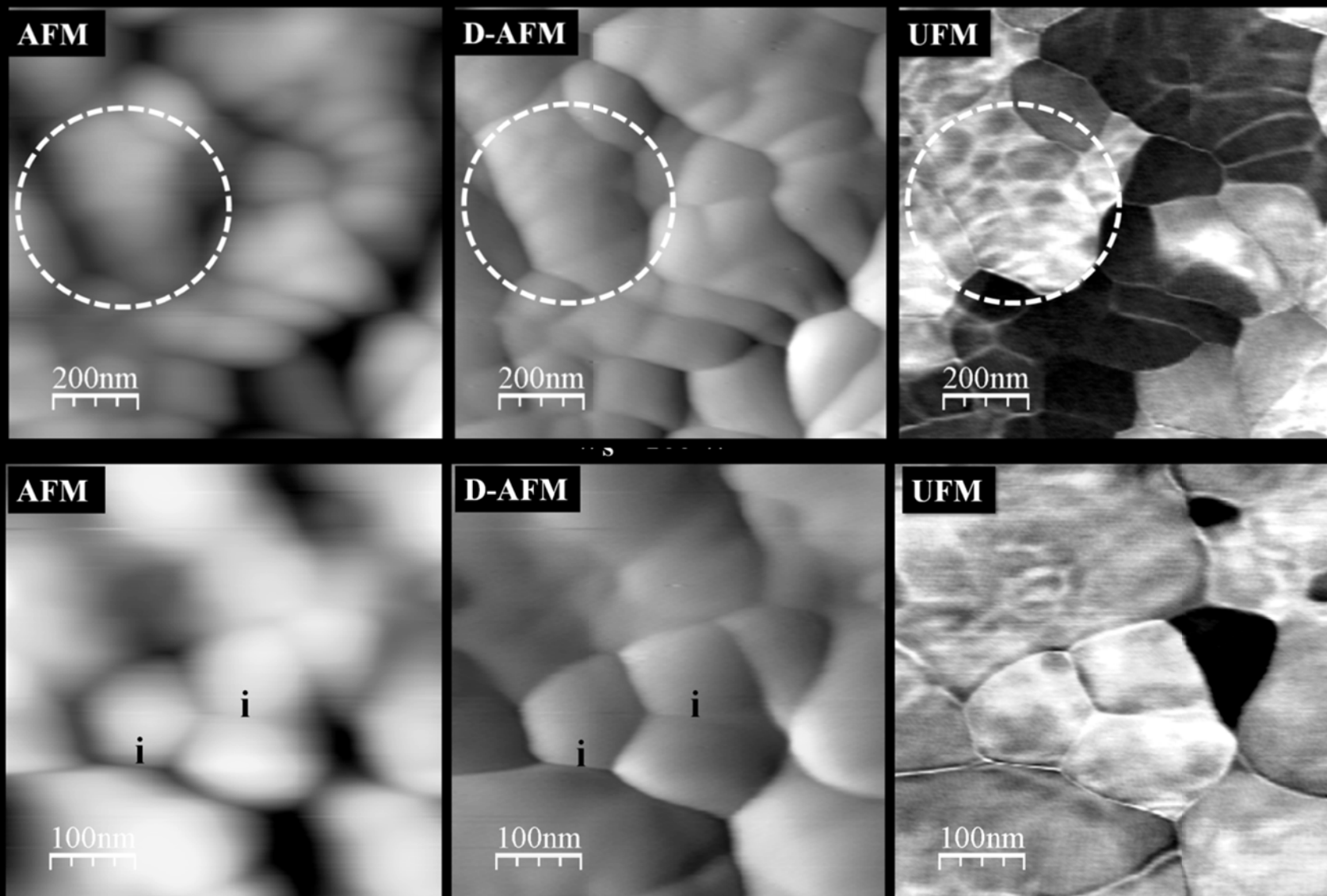
$\omega_{\text{second-mode}} \approx 450$ KHz



Principle: the cantilever is driven at *its fundamental flexural eigenmode*. Ultrasonic vibration in the megahertz range is additionally input at the tip-sample contact *from the cantilever base and from the back of the sample*. The ultrasonic frequencies are such *that their difference is coincident with the second cantilever eigenmode*.

M. T. Cuberes, J. of Nanomaterials (2009)

ULTRASONIC FORCE MICROSCOPY ON TIN COATINGS

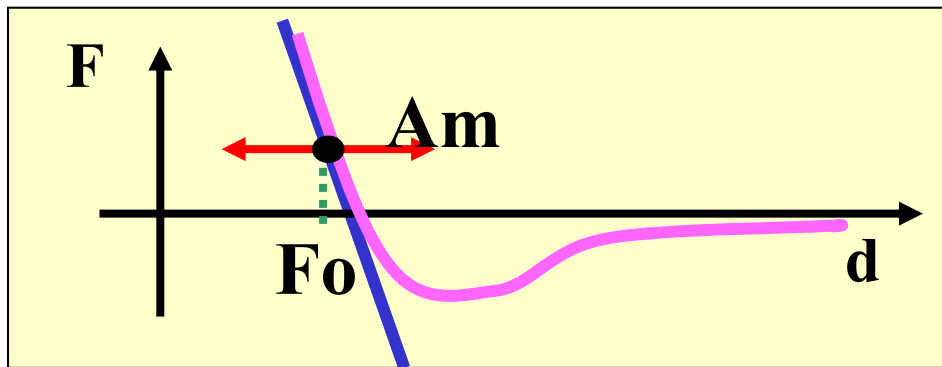


J. A. Hidalgo, C. Montero-Ocampo, and M. T. Cuberes, Nanoscale Res Lett (2009)

Outline

- *Mechanical-diode-mode Ultrasonic AFM*
 - *The Mechanical-Diode (MD) effect: UFM, HFM, IC-HFM*
- *Tribology with ultrasonic-AFM*
 - *Study of adhesion hysteresis and energy dissipation with UFM*
 - *Study of friction and lubrication with MD- UFFM*
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UFM: ADHESION HYSTERESIS AND ENERGY DISSIPATION

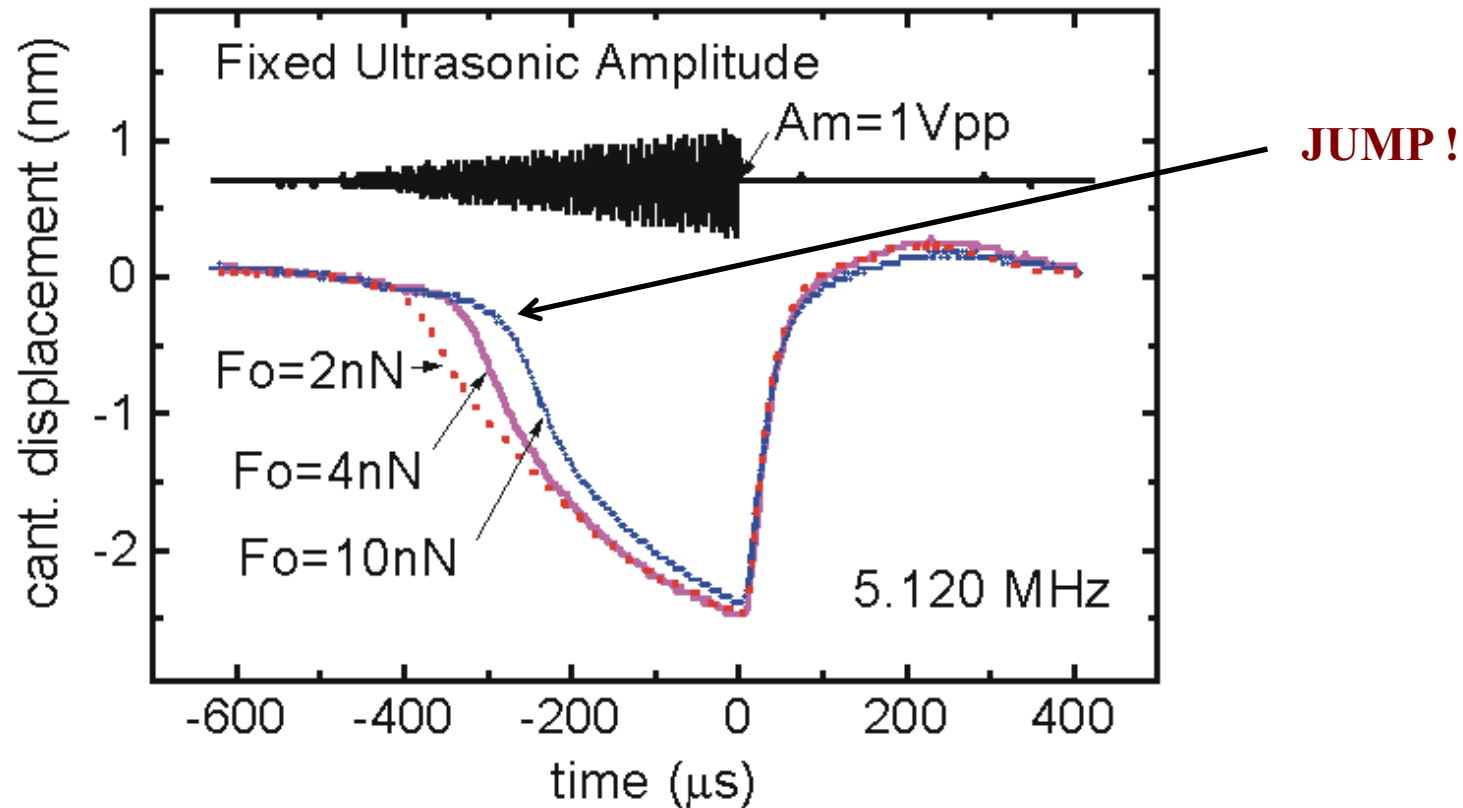


ULTRASONIC CURVES

*O. Kolosov and K. Yamanaka,
Jpn. J. Appl. Phys. (1993)*

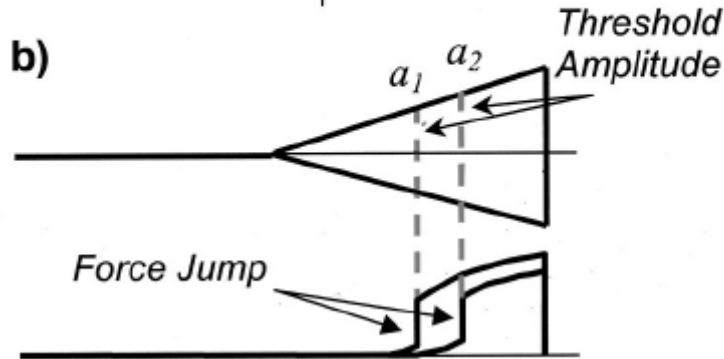
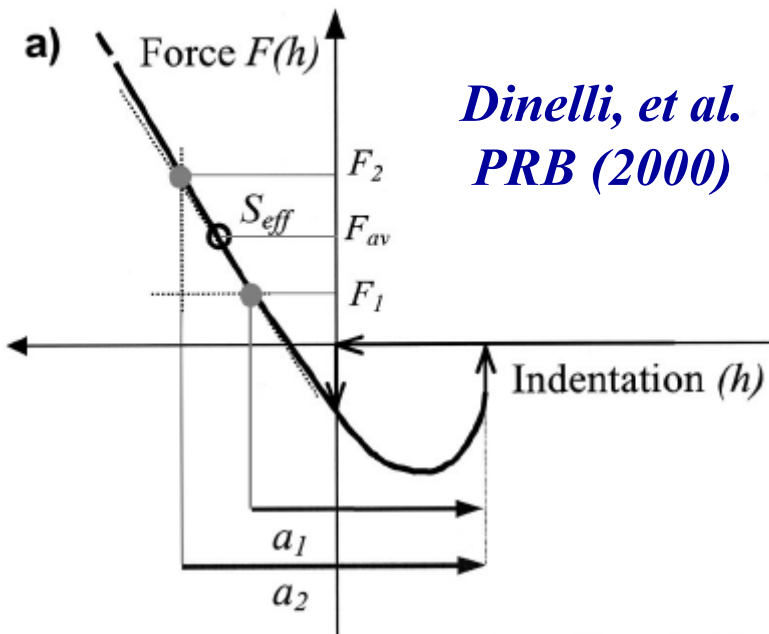
*M. T. Cuberes et al.
Nanotechnology (2001)*

W-UFM on PMMA

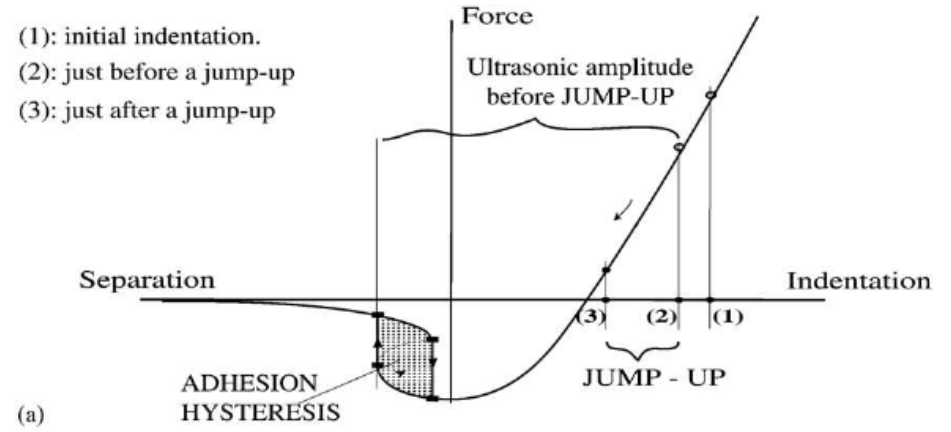


ELASTIC MODULUS AND ADHESION

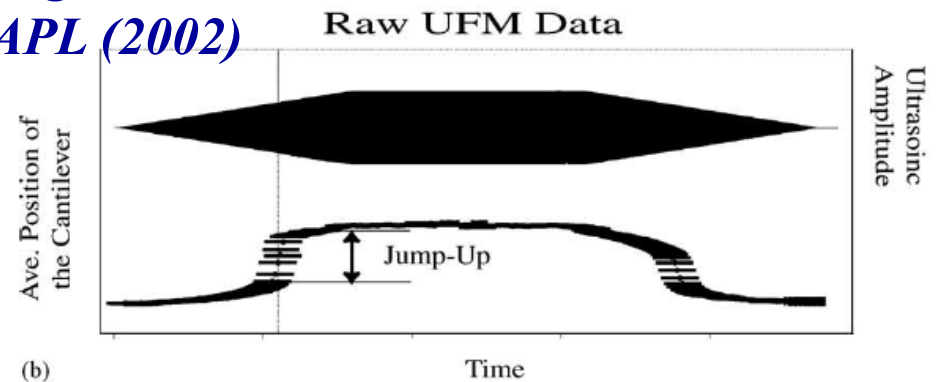
Moving along the Force - Distance Curve



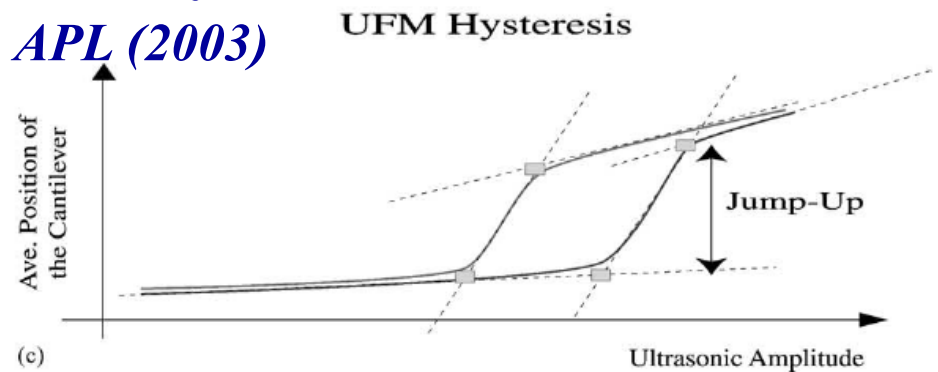
$$S_{eff}(F_{av}) = \frac{F_2 - F_1}{a_2 - a_1}$$



Inagaki et al. APL (2002)

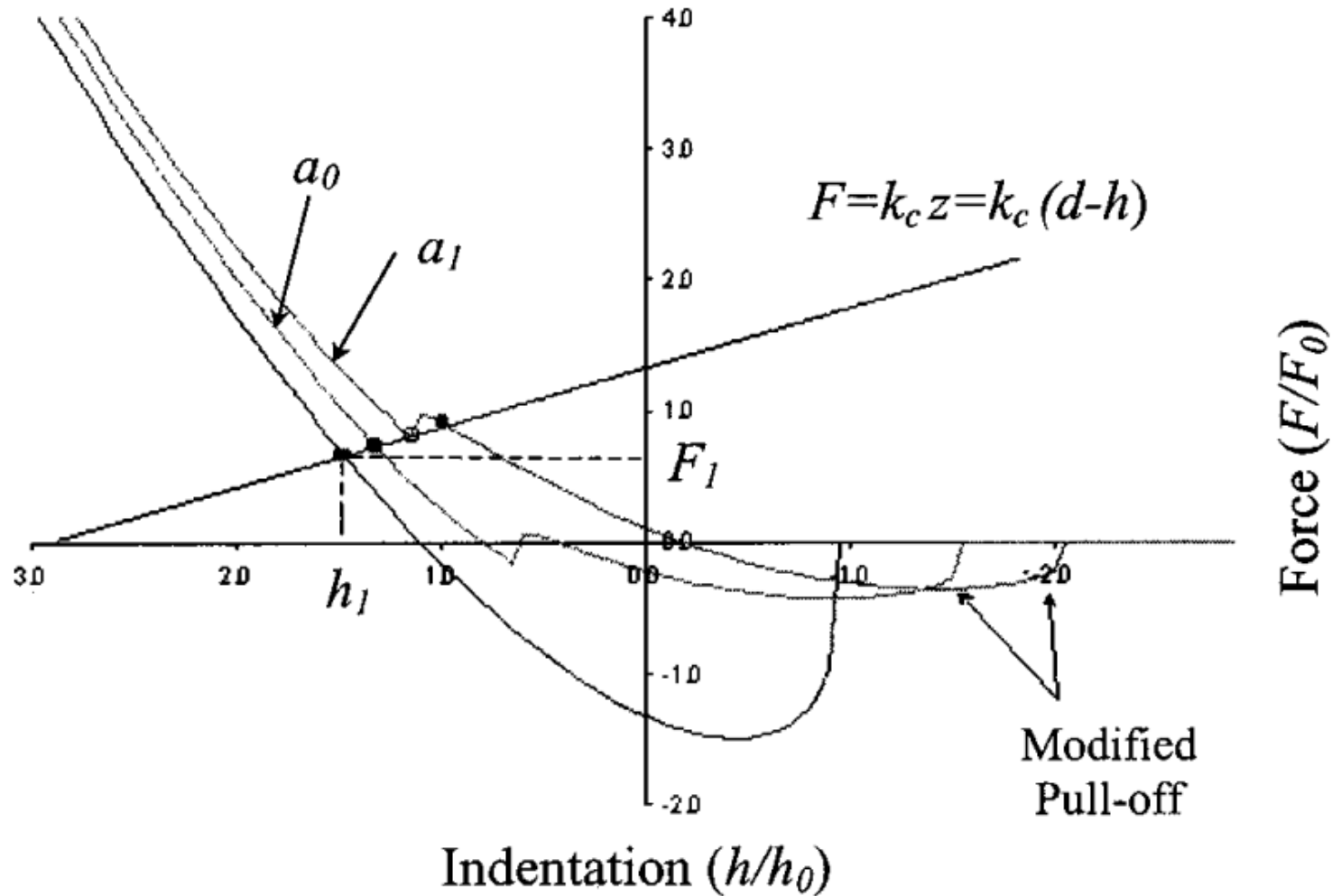


Szoskiewicz et al. APL (2003)

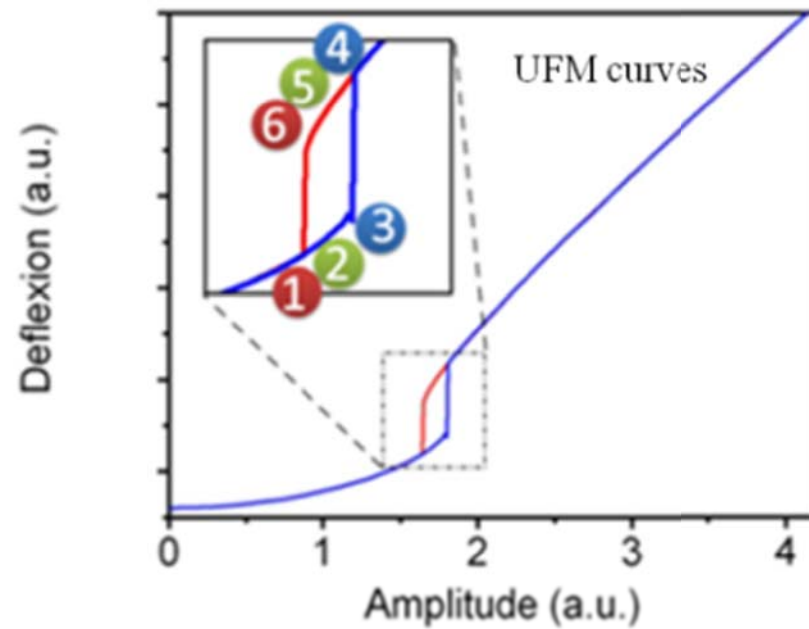
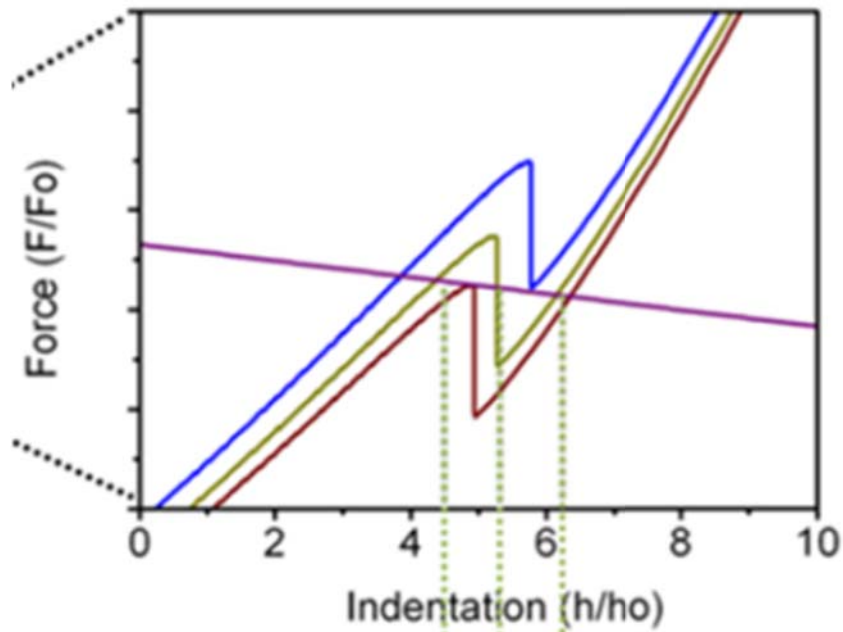


Simulations of the *ultrasonic force* versus *ultrasonic amplitude* curves:

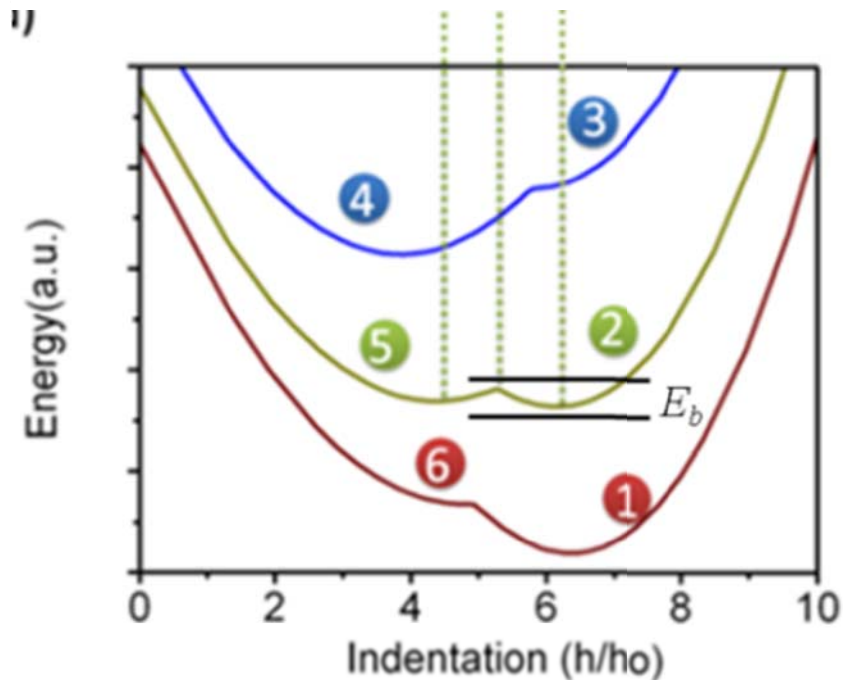
ULTRASONIC-AMPLITUDE DEPENDENT TIP-SAMPLE FORCE CURVES



Dinelli, Biswas, Briggs, and Kolosov, Phys. Rev. B (2000), 13995



ULTRASONIC-AMPLITUDE DEPENDENT POTENTIAL ENERGY CURVES

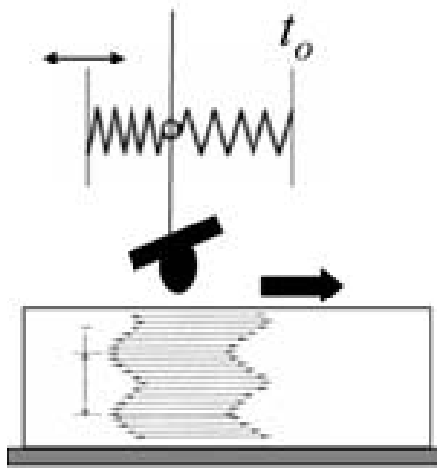


For certain ultrasonic amplitudes, the modified tip-sample forces lead to two quasi-static equilibrium states separated by an energy barrier.

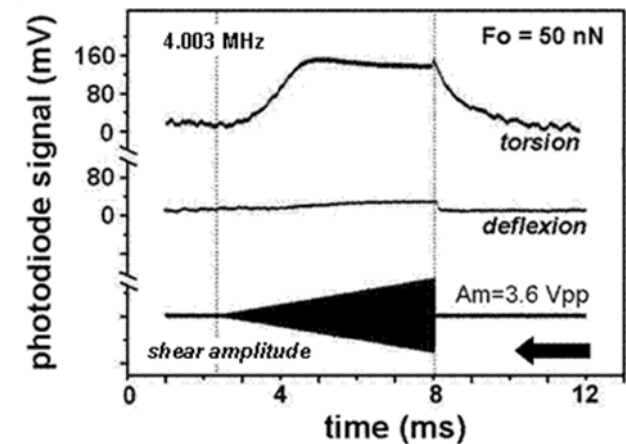
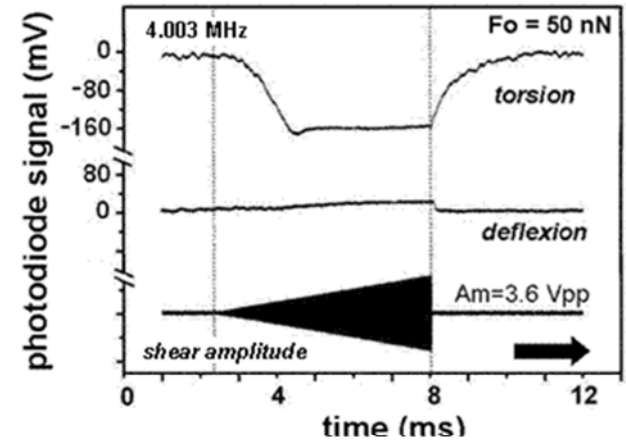
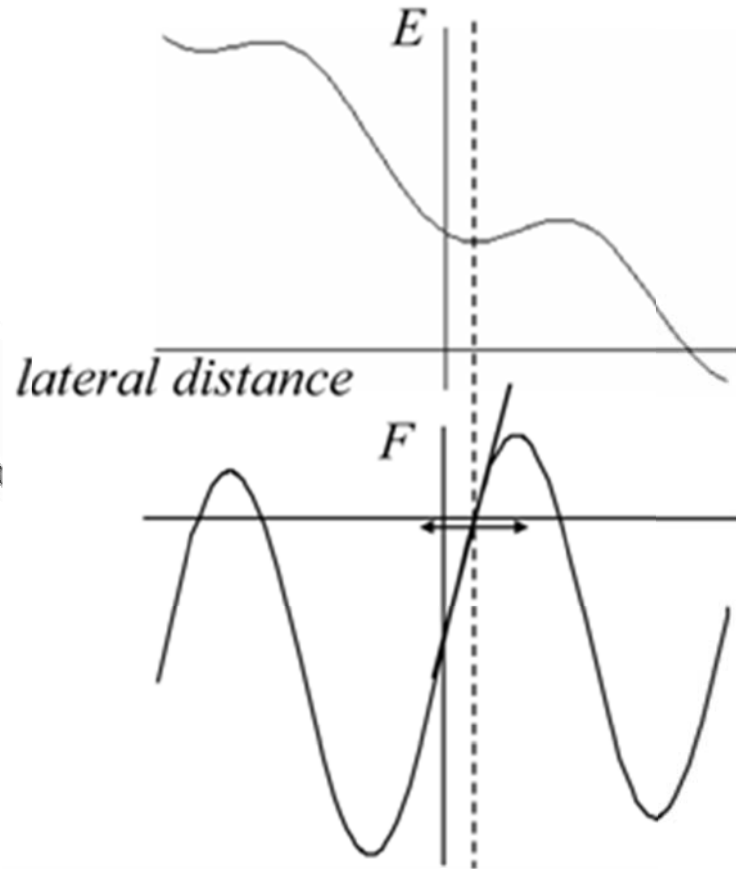
J. J. Martínez and M. T. Cuberes, Mater. Res. Soc. Symp. Proc. Vol. 1085 (2008)

MD-UFFM: NANOSCALE FRICTION AND LUBRICATION

MD-ULTRASONIC FRICTION FORCE MICROSCOPY



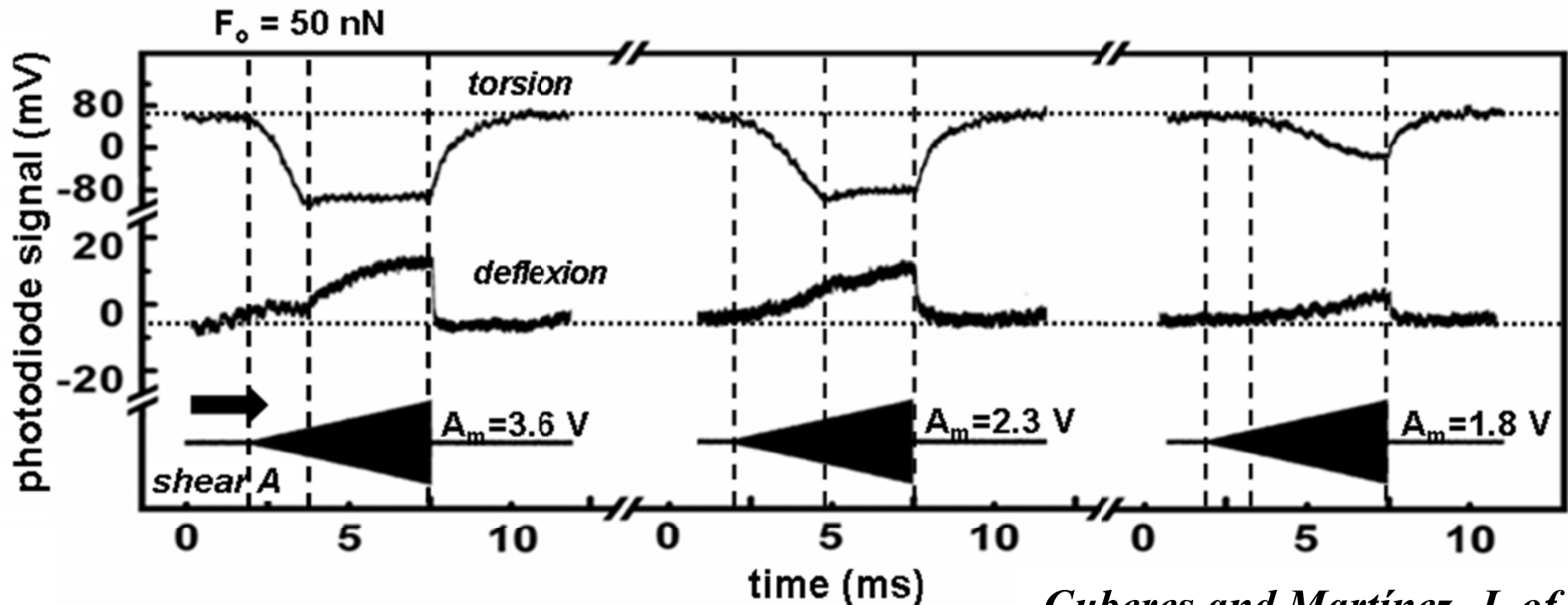
**Lateral
Mechanical
Diode Effect!**



M. T. Cuberes and J. J. Martínez, J. of Phys.: Conf. Ser. (2007)

MD-UFFM provides information about sample shear elasticity and friction at the nanoscale. Measured magnitude: static cantilever torsion induced by the *lateral ultrasonic force*.

Dependence on the ultrasonic amplitude: MD-UFFM on Si(111)



➔ Physical origin of the lift-off?

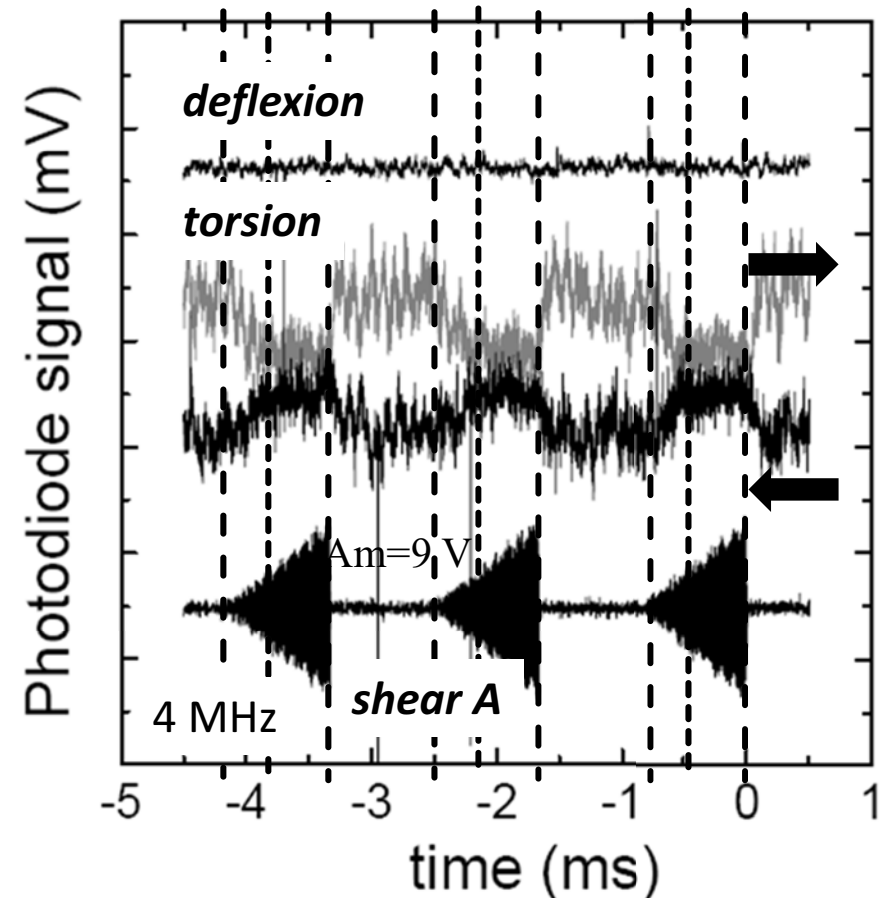
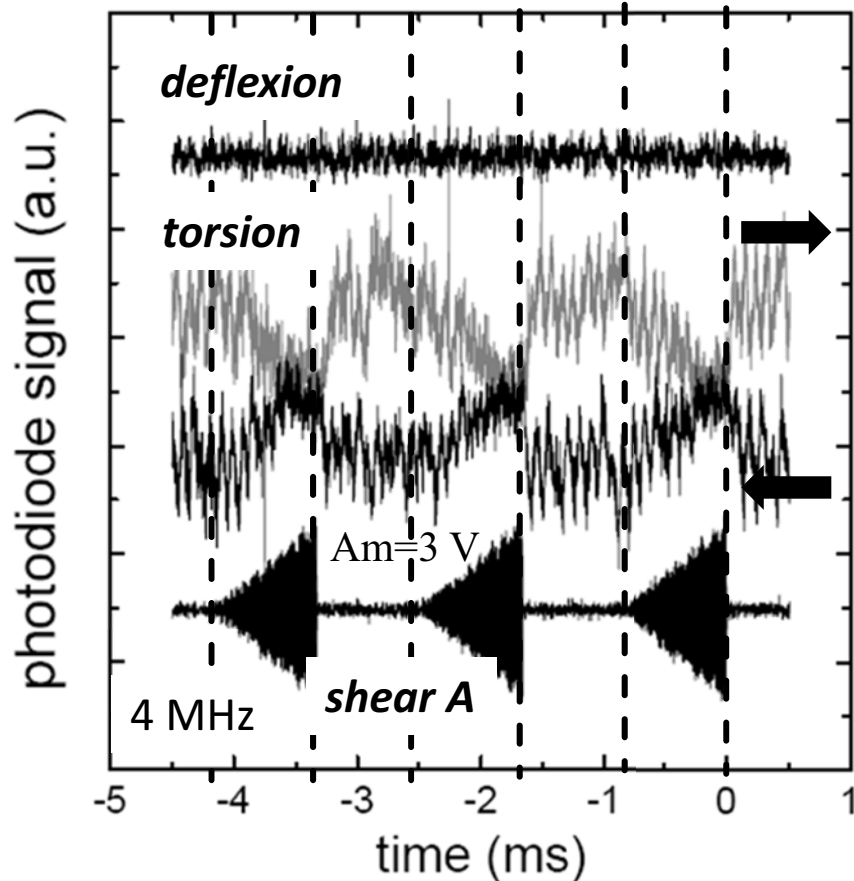
When **increasing the sliding velocity**, the minimum thickness of a viscous squeezed liquid layer increases according to EHD theory, and the *hydrodynamic pressure may support the tip* and reduce friction.

$$h_{\min} = 1.79R^{0.47}\alpha^{0.49}\eta_0^{0.68}U^{0.68}E^{-0.12}W^{-0.07}$$

V. Scherer, W. Arnold, and B. Bhushan. Surf. Interface Anal. (1999)

CONTROL OF FRICTION

MD-UFFM on HOPG



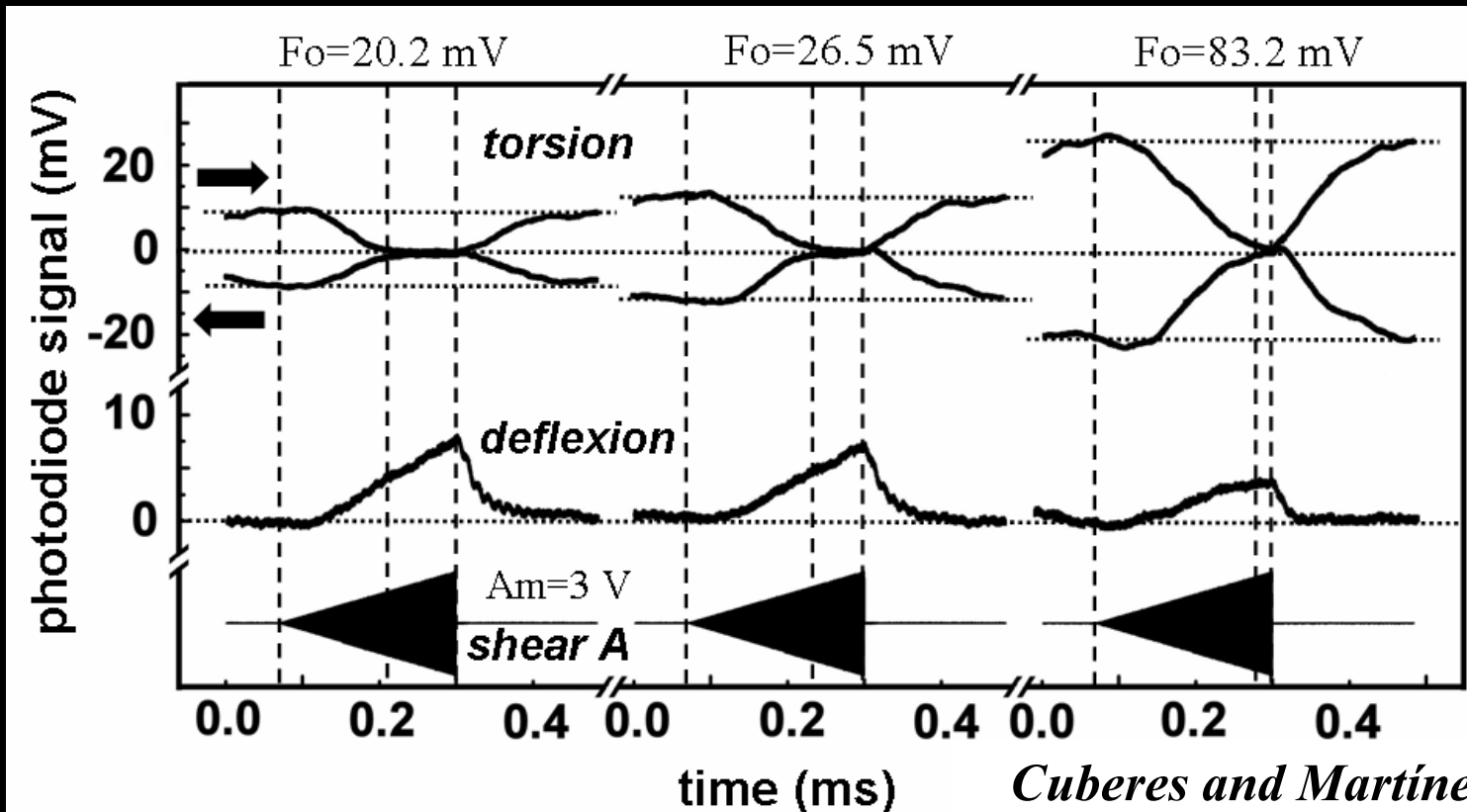
No lift-off is observed on HOPG!

M. T. Cuberes (in preparation for issue in Tribology Letters)

Friction reduces for increasing shear ultrasonic amplitudes; above a critical value, the lateral ultrasonic force remains constant.

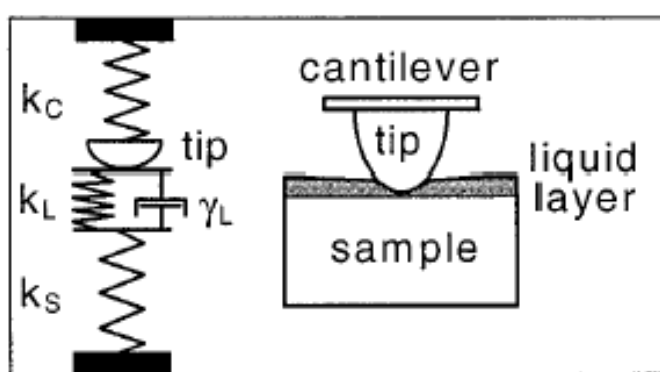
CONTROL OF FRICTION

MD-UFFM on Si(111)



Cuberes and Martínez (2007)

Reduction of friction by out-of-plane vibrations and lubricant layers



Supresion of solvation force and development of liquid-like dynamics

Dinelli, Biswas et al. APL (1997)

Gao et al. J. Phys. Chem. B (1998)

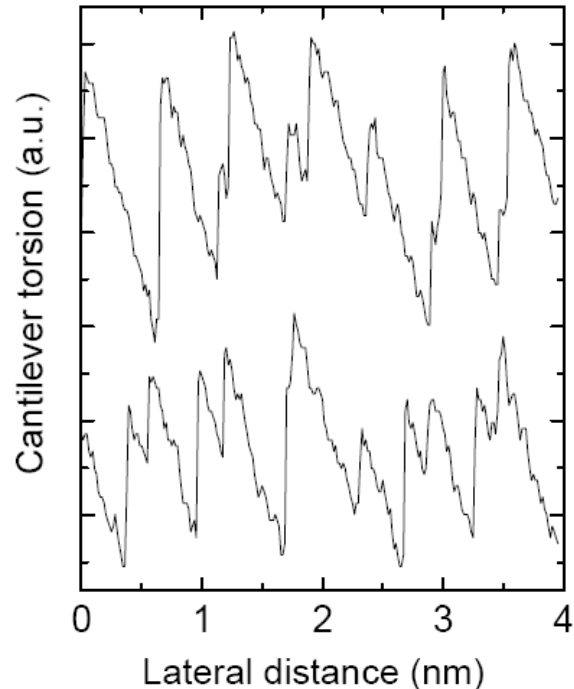
Heuberger et al. J. Phys. Chem. (1998)

CONTROL OF FRICTION

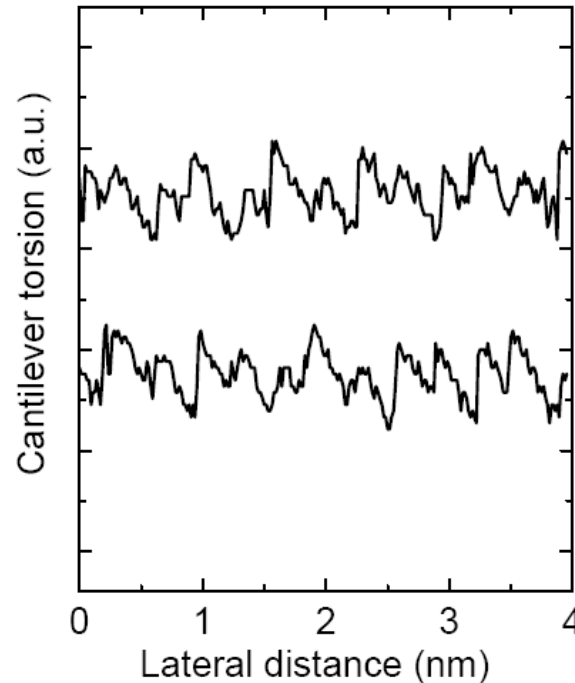
Results on HOPG

REDUCTION OF FRICTION BY LATERAL ULTRASONIC VIBRATION

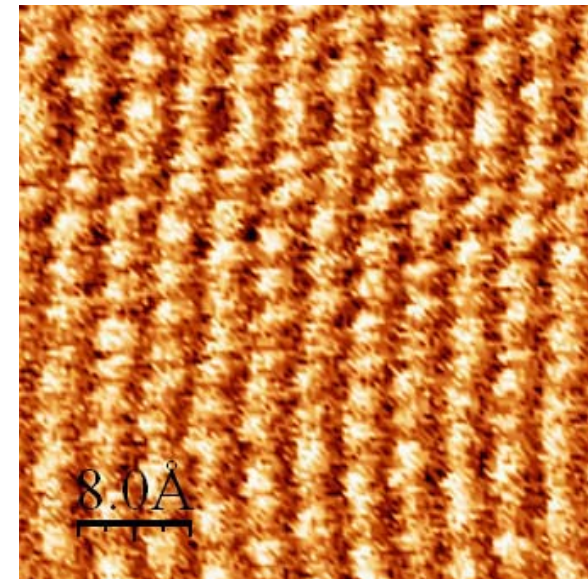
Without ultrasound



Shear ultrasonic A = 1.7 V

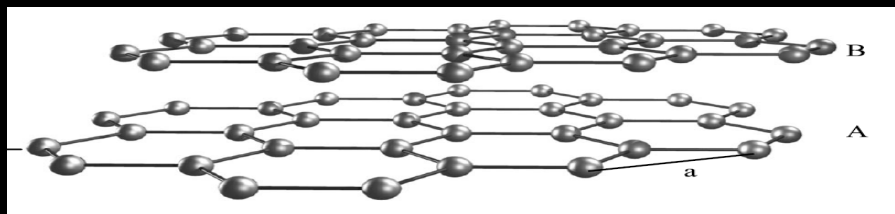


Shear ultrasonic A = 9 V

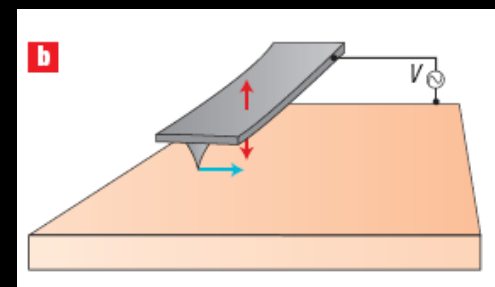


M. T. Cuberes (in preparation for issue in Tribology Letters)

SUPERLUBRICITY: Registry between the sliding surfaces Normal mechanical resonances



Dienwiebel et al. PRL (2004)



Socoliuc et al. Science (2006)



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2063-21

ICTP/FANAS Conference on trends in Nanotribology

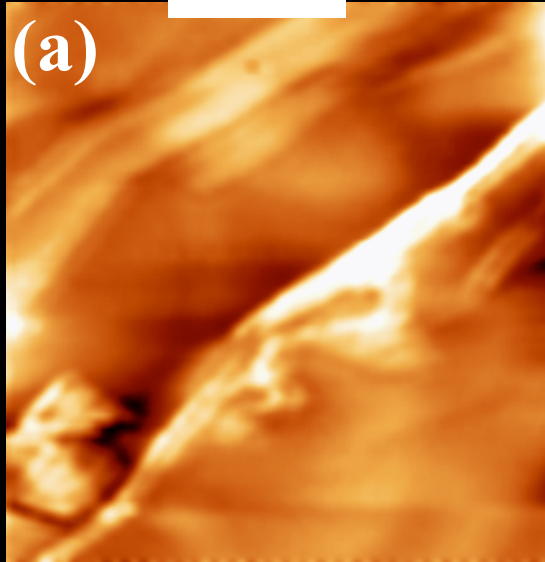
19 - 24 October 2009

Ultrasonic nanolithography on hard substrates

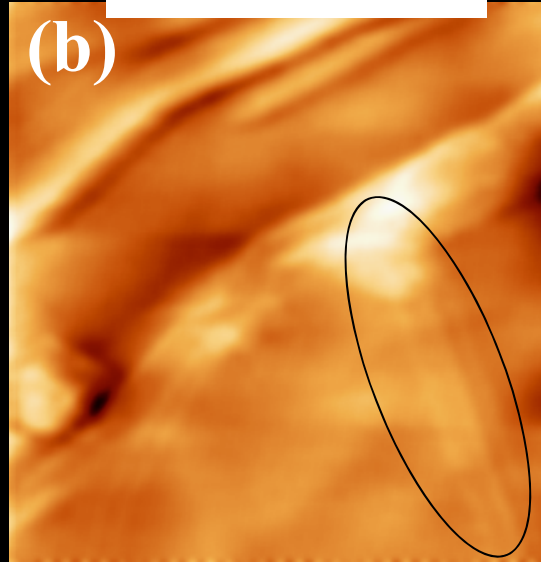
Teresa Cuberes
*University of Castilla La Mancha
Almaden
Spain*

MANIPULATION OF SUBSURFACE DISLOCATIONS IN HOPG

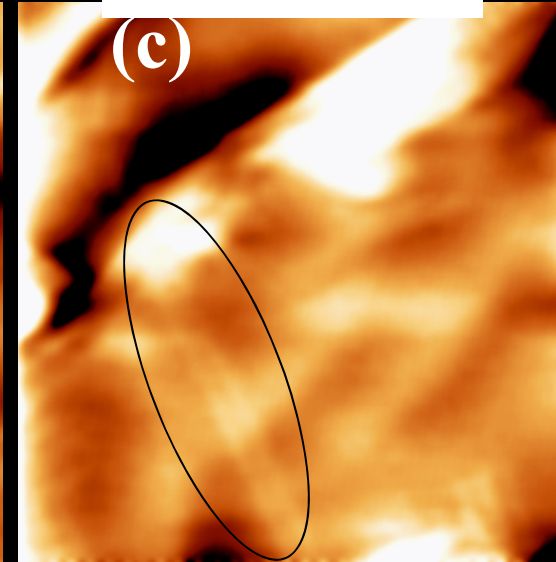
AFM



AFM with us



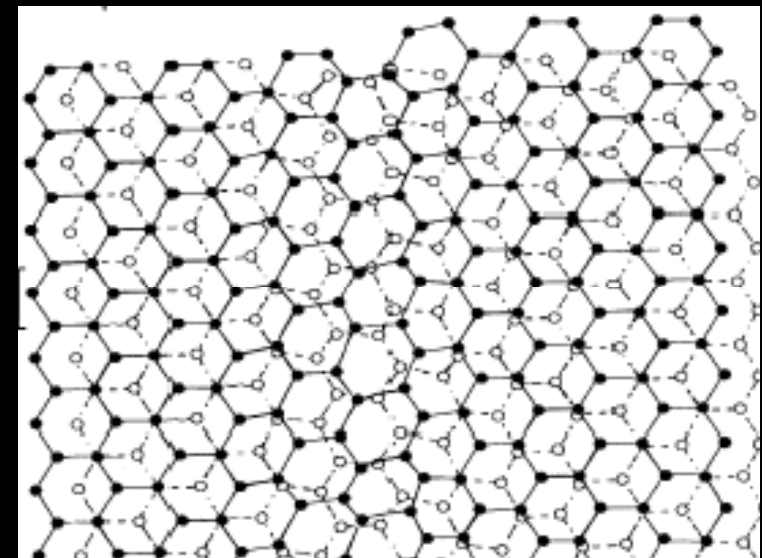
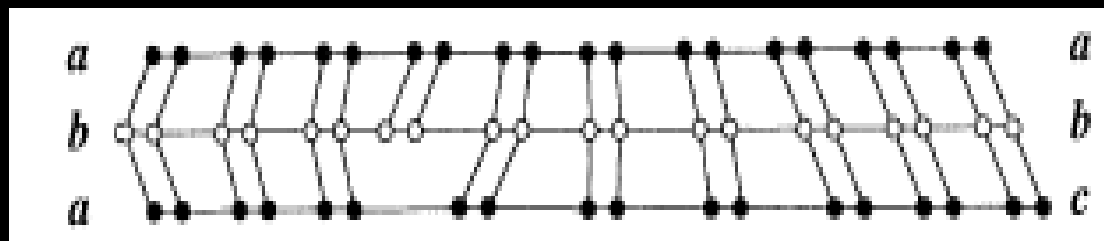
AFM with us



700 nm x 700 nm

F₀: 105 nN

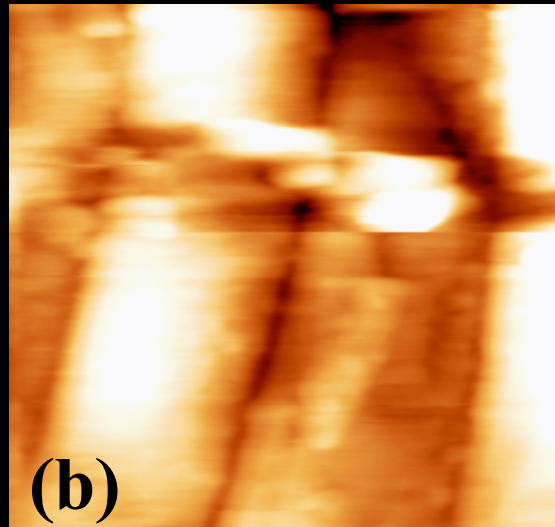
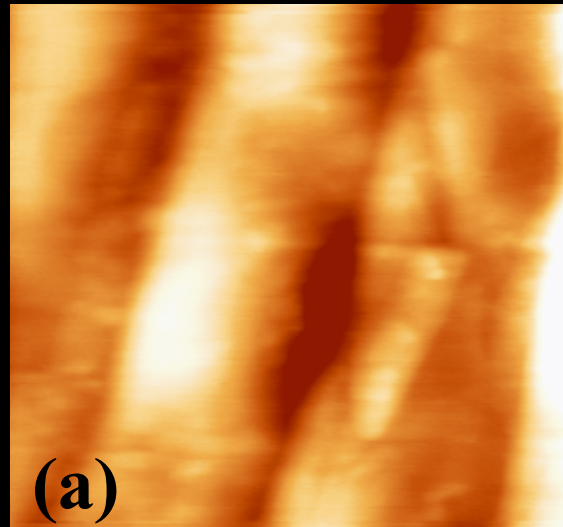
Cantilever stiffness: 0.35 Nm⁻¹



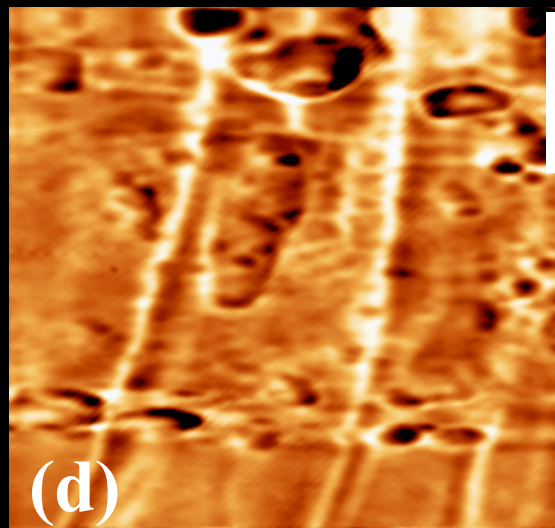
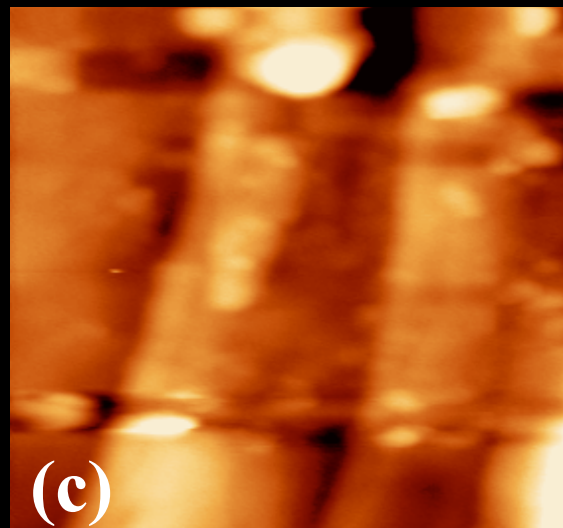
Cuberes J. of Phys. : Conf. Ser. (2007)

ULTRASONIC GENERATION OF WEAR ON HOPG

Wear of HOPG is observed after repeatedly scanning over the same surface region in the presence of us excitation of up to 4.2 nm in A.



(a) and (b) AFM with
us $A \approx 1.7$ nm
recorded in sequence



(c) AFM and (d) UFM
simultaneously recorded

*M. T. Cuberes (in
preparation for issue in
Tribology Letters)*

$2.5 \mu\text{m} \times 2.5 \mu\text{m}$; $F_o: 160$ nN

Outline

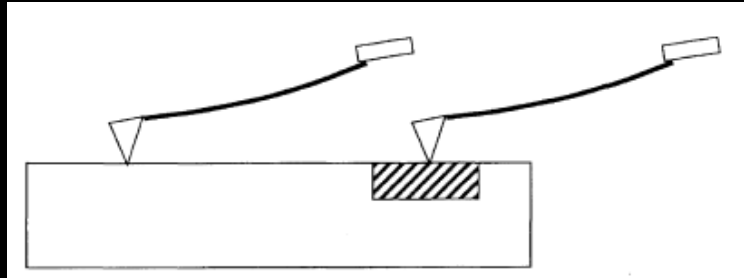
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Advantages for nanofabrication

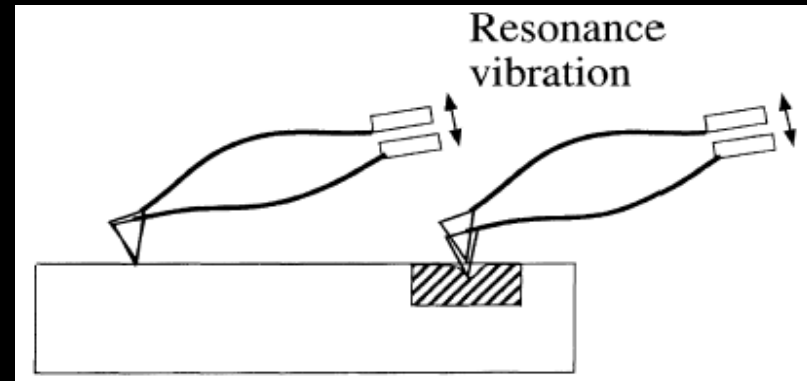
Ultrasonic-AFM

M.T. Cuberes, J. of Phys.: Conf. Ser. 61 (2007) 219

➔ It is possible to *indent hard materials* with a *soft cantilever*

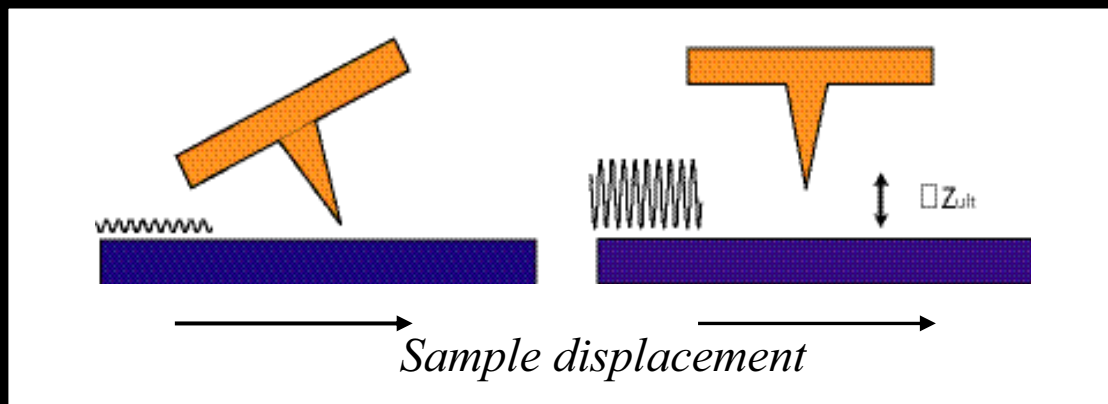


A soft cantilever can be dynamically stiffened!



Yamanaka and Nakano., Jpn. J. Appl. Phys. 35, 3787 (1996)

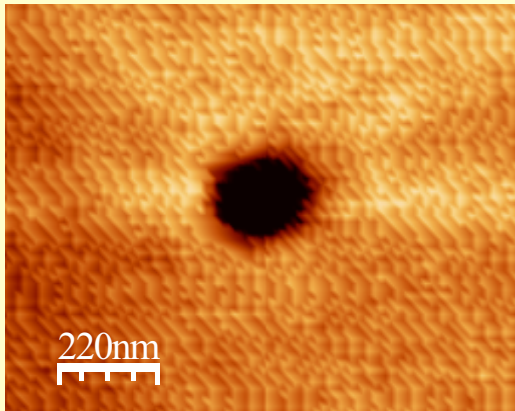
➔ *Friction at the nanometer scale vanishes* in the presence of ultrasonic vibration of sufficiently high amplitude



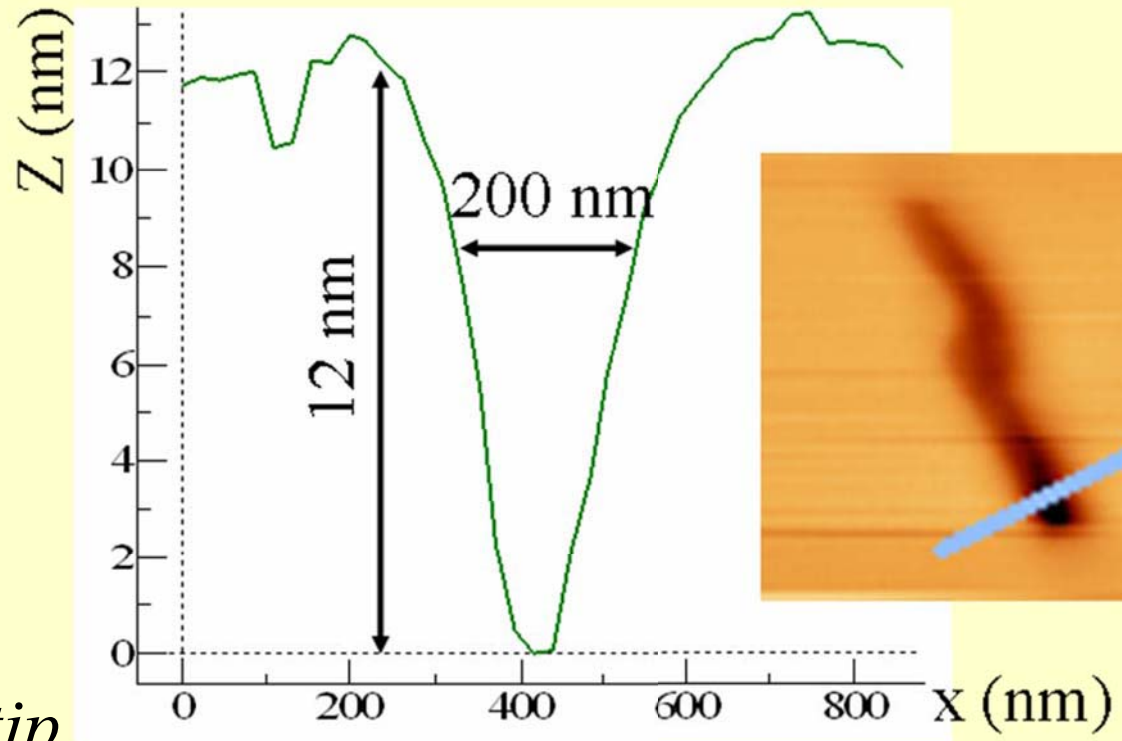
Sonolubrication at the nanoscale!

Dinelli et al. Appl. Phys. Lett. 71 1177 (1997)

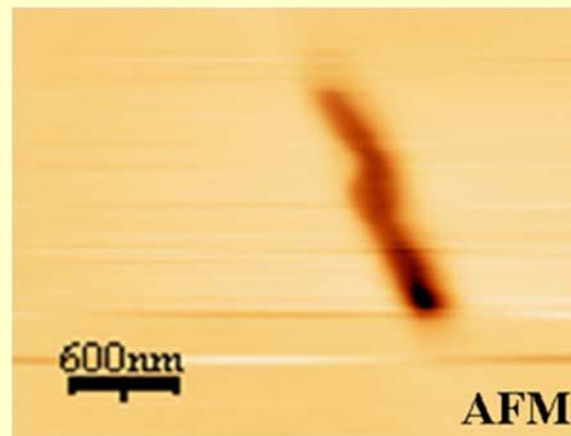
NANO ULTRASONIC MACHINING



Hole and scratch on Si(111) by ultrasonic action with an AFM tip



Diamond-coated tip
DCP20, NT-NDT
Rc:35 nm; L:10-15
 μm ; diamond t: 70 nm;
Kc:28-91 N m^{-1}

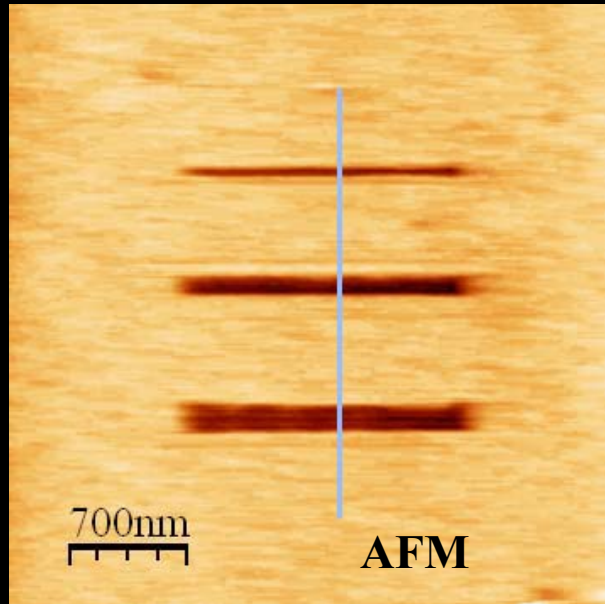


*M.T. Cuberes,
G. I. T. Imaging
& Microscopy 4
(2007) 36*

SCRATCHES on Si(111): 50, 75 and 100 lines with $F_n=37$ nN, $f=5$ MHz, $A_m=0.5$ V

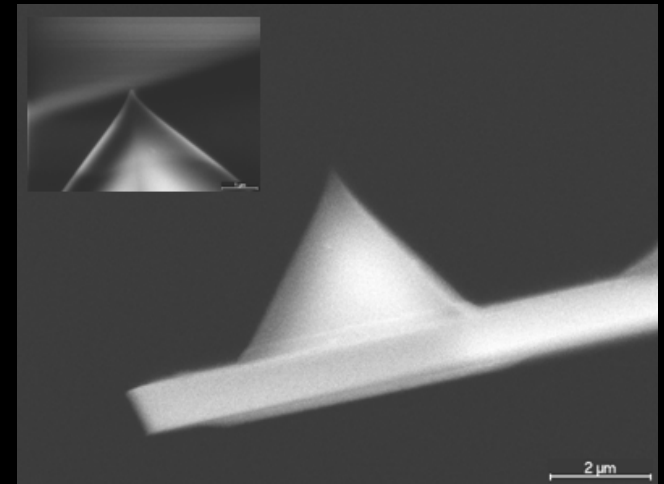
AFM : $F_o:13$ nN

OLYMPUS Si_3N_4
 $K_c:0.11$ N m $^{-1}$ $\omega: 22$ KHz

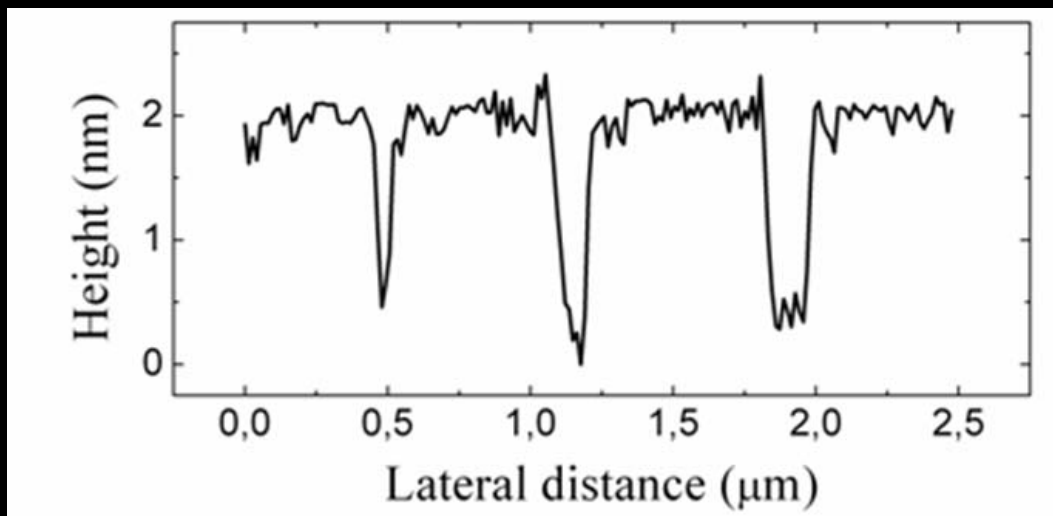
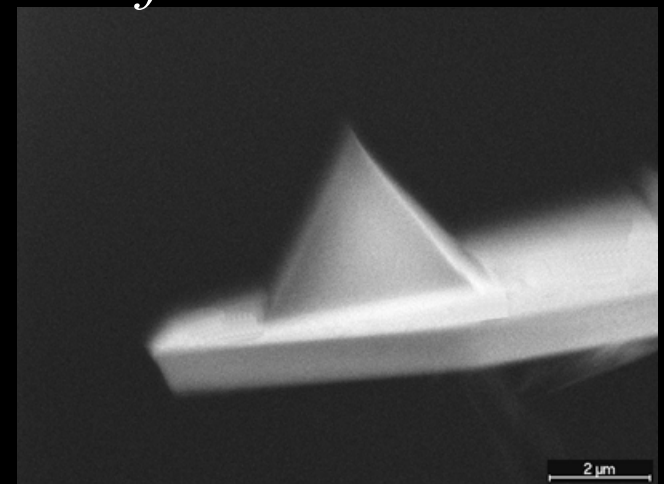


*M.T. Cuberes,
G. I. T. Imaging
& Microscopy 4
(2007) 36*

after



before



- **Mechanical-diode-mode Ultrasonic AFM**

- **The Mechanical-Diode (MD) effect: UFM, HFM, IC-HFM:**
MD mode valuable; novel techniques proposed.

- **Tribology with ultrasonic-AFM**

- **Study of adhesion hysteresis and energy dissipation with UFM:**
ultrasonic-amplitude-dependent quasistatic energy states

- **Study of friction and lubrication with MD- UFFM:**
novel technique proposed with results on Si(111) and HOPG.

- **Control of friction and generation of wear using ultrasound:**
lateral ultrasonic vibration reduces friction; ultrasound facilitates generation of wear

- **Ultrasonic nanolithography on hard substrates**

- **Results on Si(111):** Ultrasound facilitates nanoscratching of Si(111)

ACKNOWLEDGEMENTS

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Carmen Iniesta

Salvatore Marino

Alejandro Rodríguez

**[http://www.uclm.es/organos/
vic_investigacion/gruposweb/nanotecnologia/](http://www.uclm.es/organos/vic_investigacion/gruposweb/nanotecnologia/)**



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(*projectPBI-08-092*) is gratefully acknowledged