UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION
INTERNATIONAL ATOMIC ENERGY AGENCY
INTERNATIONAL CENTRE FOR THEORETICAL PHYSICS

SMR/917 - 8

SECOND WORKSHOP ON SCIENCE AND TECHNOLOGY OF THIN FILMS

(11 - 29 March 1996)

" Experimental techniques for atomic-scale characterization (focussing on AFM/STM) "

presented by:

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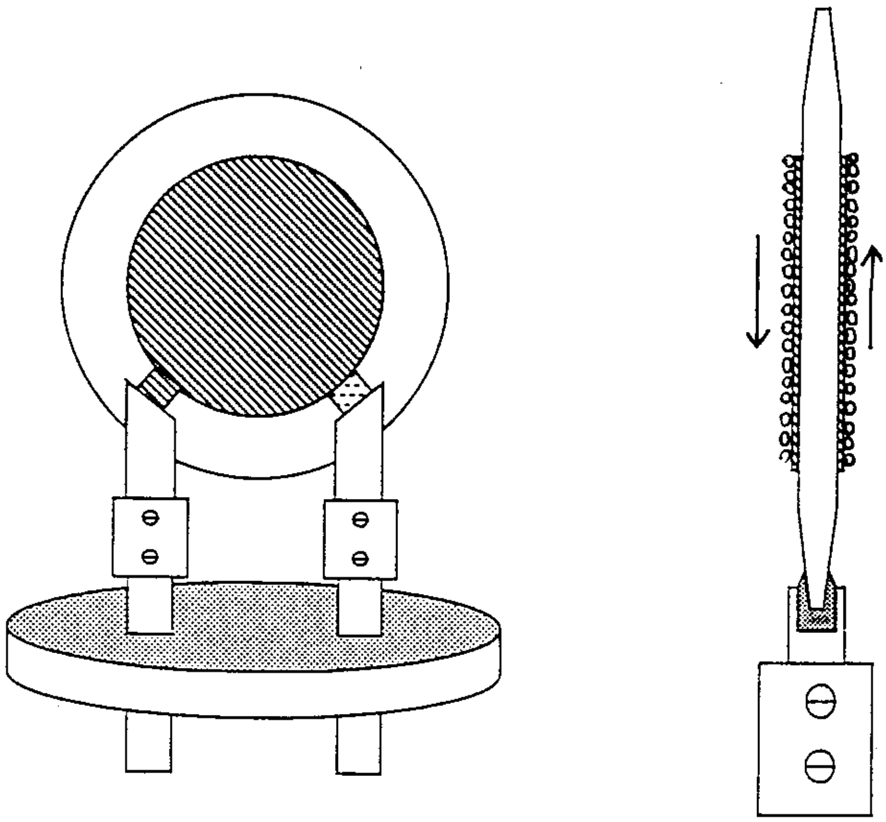
AFM/STM Characterization of Film Growth

I Experimental techniques for atomic-scale characterization (focussing on AFM/STM)

II Fractal/Kinetic Scaling treatment of film growth.

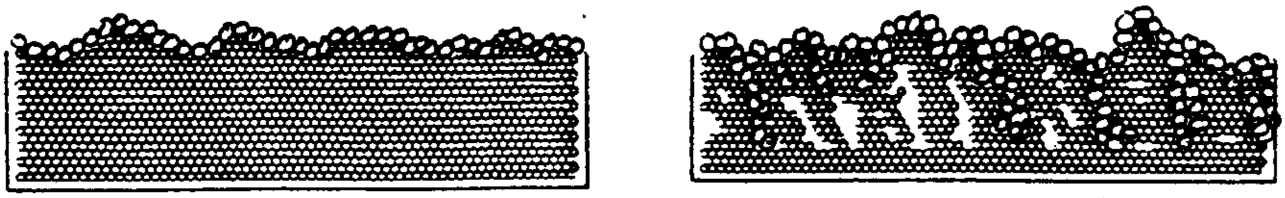
↕

III STM/AFM studies of kinetic scaling



$$\frac{\delta f}{f_0} = \frac{(m/a)}{\rho_q t_q}; \qquad \delta\left(\frac{1}{Q}\right) = \frac{R}{\pi f_0 \rho_q t_q}$$

(m/a): mass per unit area of the adsorbed film
R: proportional to film energy dissipation
ρ_q, t_q: quartz crystal density and thickness



Adsorption:

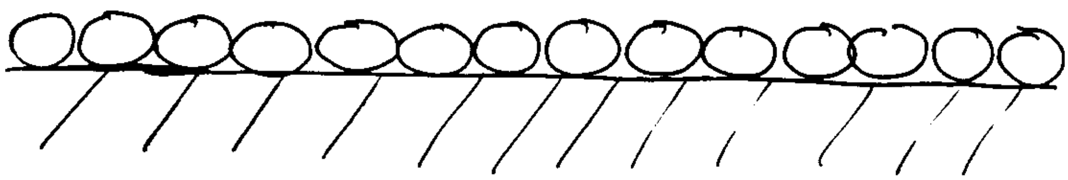
References: - Pfeifer, Wu, Cole & Krim PRL 62, 1997 (1989)
- Kardar & Indekeu Europhys Lett. 12, 161 (1990)
- Pfeifer et al. Proc. Royal Soc Lond, A 423, 169 (1989)
- Panella & Krim (preprints)

Advantages: - Probes all outermost surface topology, including that in pores
- Truly atomic scale probe

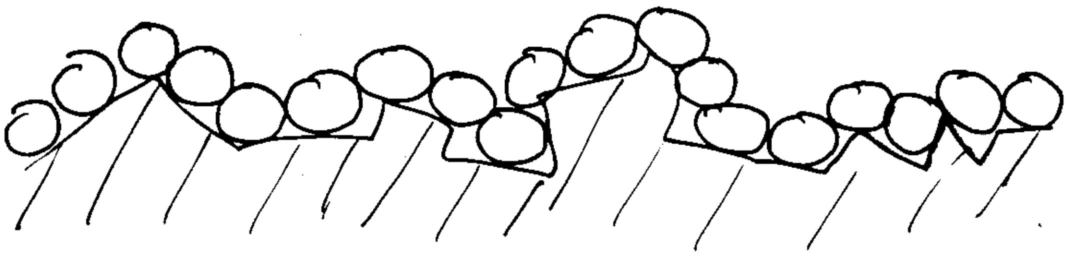
Disadvantages: - Indirect probe of scaling exponents
- Liquid adsorbate surface tension effects as complications.

More particles are required to cover a rough surface than a flat surface.

(14)

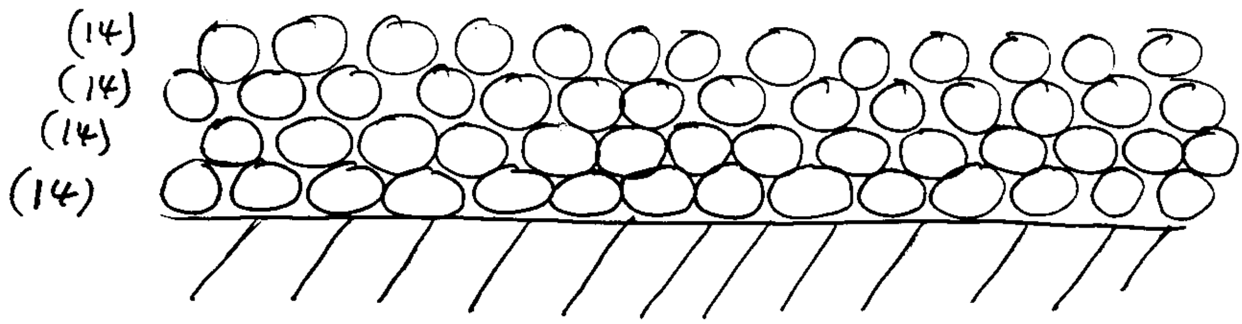


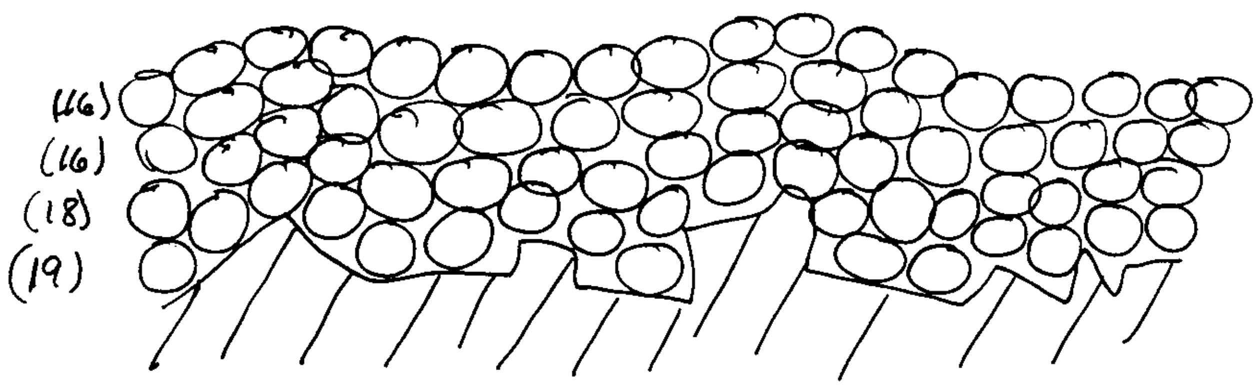
(19)



- But -

As the rough surface is covered, it will be smoothed ..... and will require less particles ..





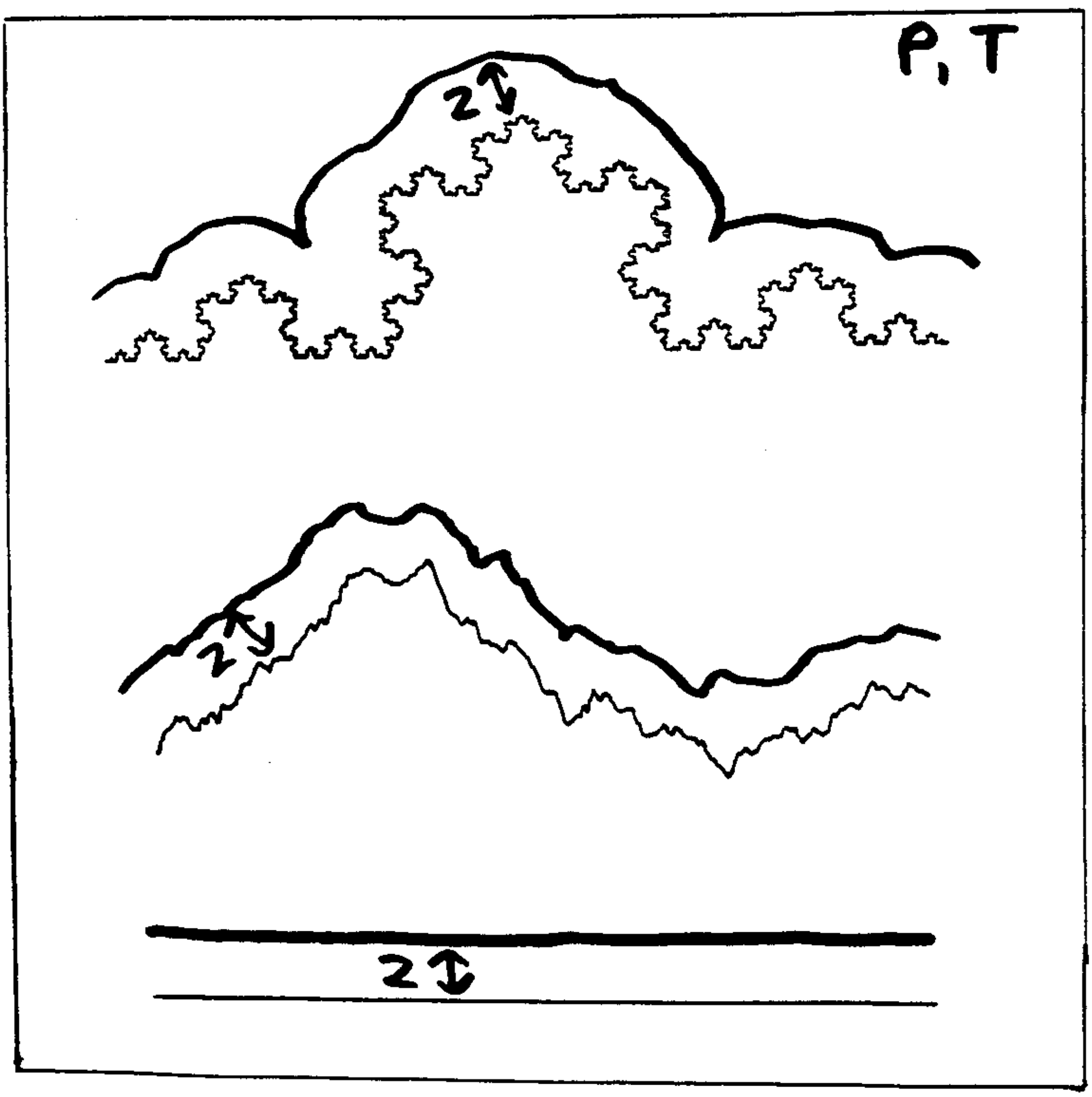
(complete wetting required!!)

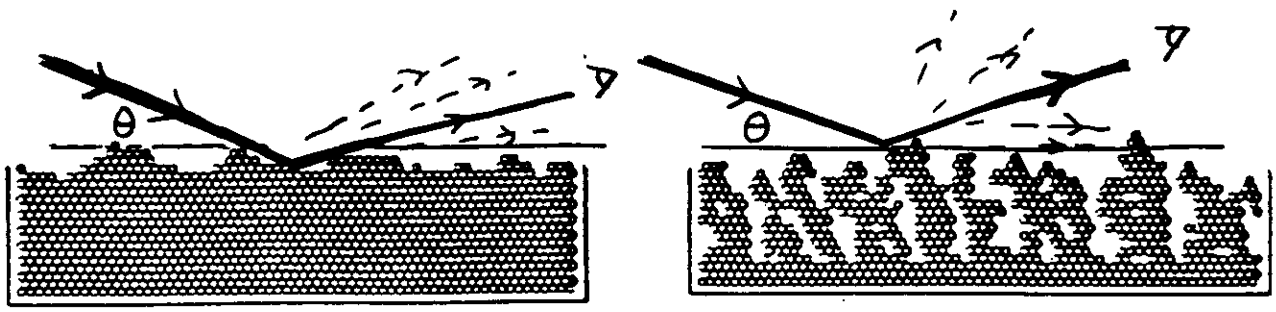
Adsorption on a Flat Surface:

$$ln(P/P_o) = -\frac{\alpha}{k_B T(\text{coverage})^3}$$

Adsorption on a Fractal Surface:

$$ln(P/P_o) = -\frac{\alpha}{k_B T(\text{coverage})^n}$$





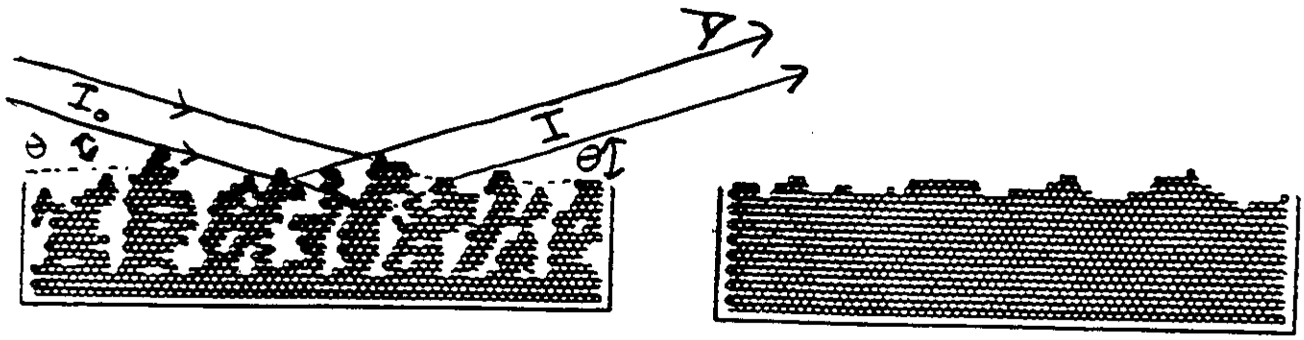
X-ray Reflectivity

References : - Sinha et al. PRB 38, 2297 (1990)
- Braoslav et al. PRA 38, 2457 (1988)
- Chiarello, Panella, Krim & Thompson PRL 67, 3408 (1991)
- Krim et al. (preprint)

Advantages : - Probes entire film density
- Provides accurate measure of σ

Disadvantages : - Lower interface contributes to signal
- Susceptible to fitting parameter assumptions

DIFFUSE X-RAY REFLECTIVITY



$$I(q_z) \propto (L_x L_y) q_z^{-[2+(1/H)]}; \quad q_z = \frac{4\pi}{\lambda}\sin(\theta)$$

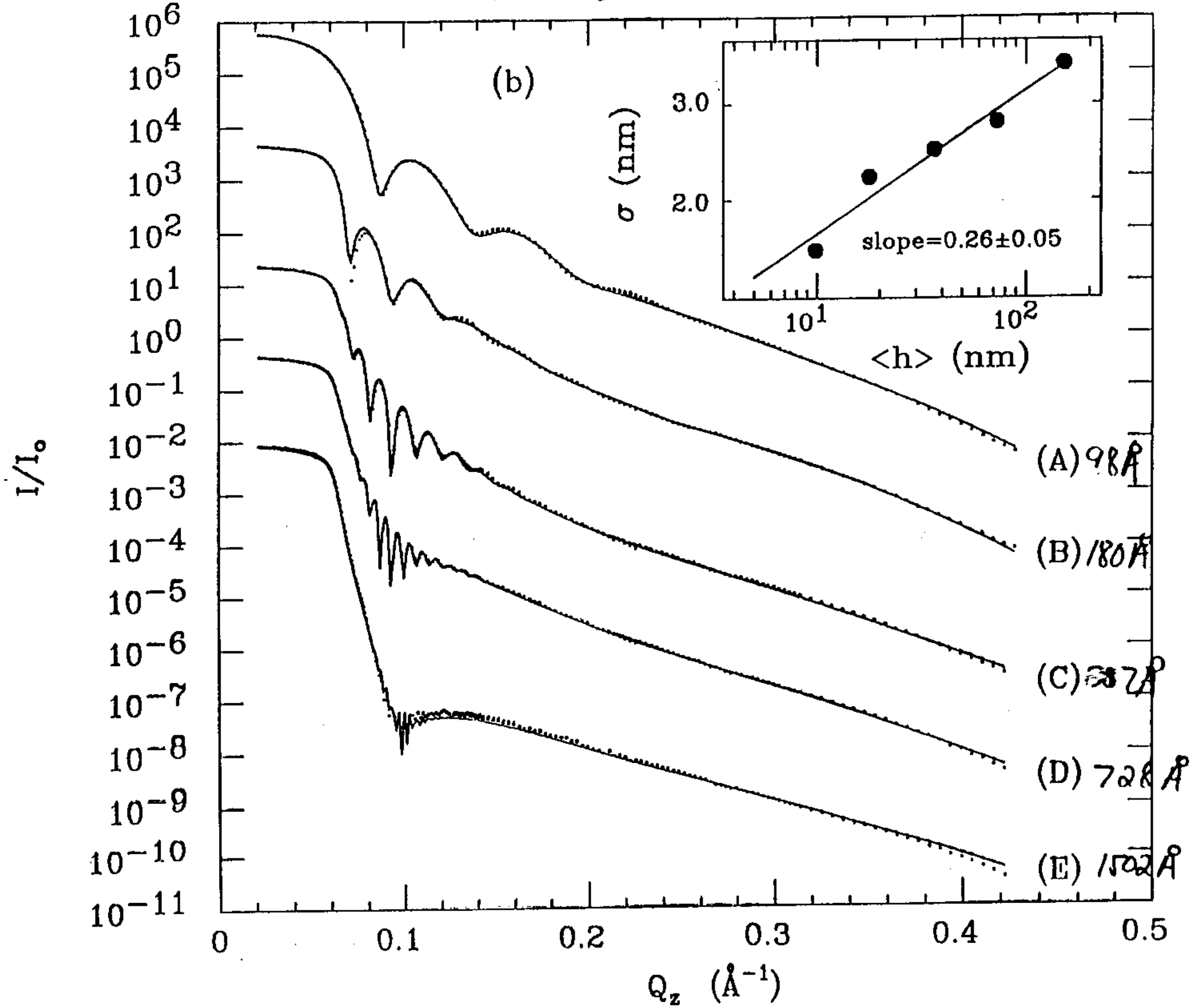
If the area illuminated by the beam changes as the diffuse data are recorded, then $(L_x L_y) \propto q_z^{-1}$ and the experimentally observed power law will have the form

$$I(q_z) \propto q_z^{-(3+1/H)} \tag{3}$$

C. Thompson, Y.P. Feng,
G. Palasantzas, S.K. Sinha & J. Krim, PRB

S.K. Sinha et al. PR? '88

Specular X-ray reflectivity on Ag films prepared at 300 K



C. Thompson et. a.l. PRB

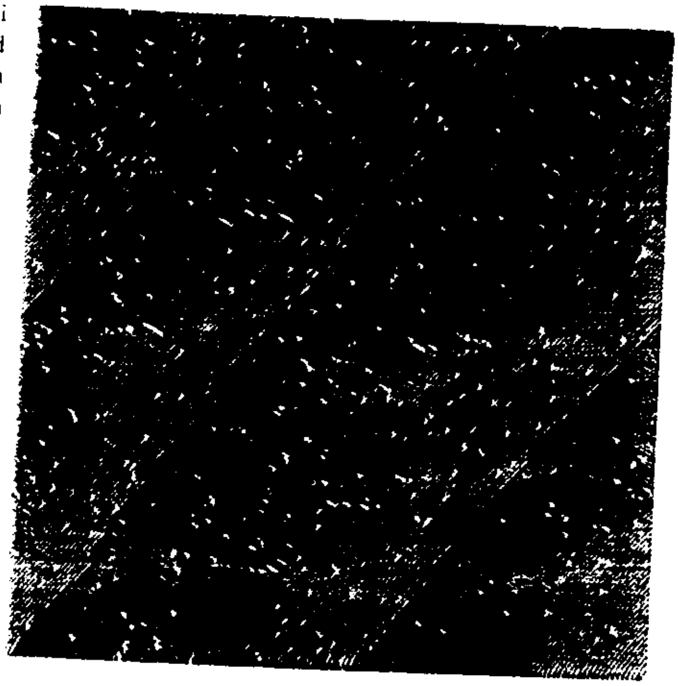


Figure 1.6 A Si surface, as examined by STM. One can distinguish both individual atoms and vacancies. The rugged lines correspond to steps on the surface, where the height of the interface increases by one atom. (Courtesy of M. Lagally).

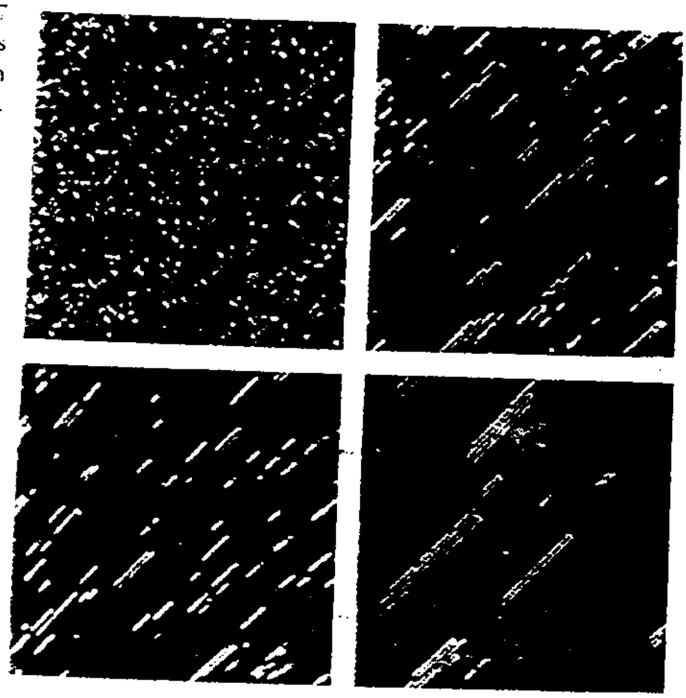
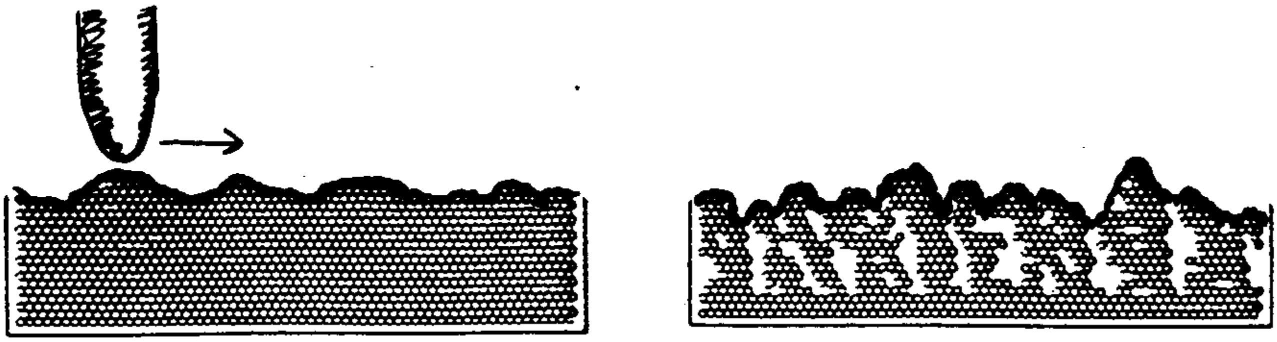


Figure 1.7 Formation of islands by atoms deposition on a Si surface. (Courtesy of M. Lagally).

Barabasi & Stanley, "Fractal Concepts in Surface Growth"



Scanning Tunneling Microscopy, of Fractal Surfaces

References: Mitchell & Bonnell, J. Mat. Res. 5, 2244 (1991)

Gomez-Rodriguez et al. JVSTB9, 495 (1991)

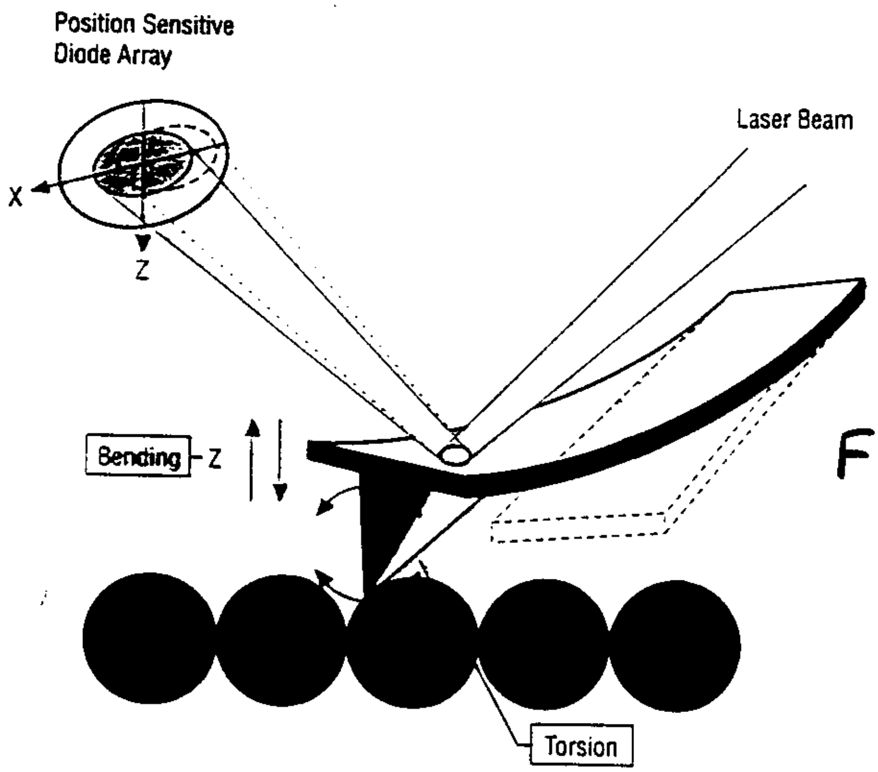
Eklund et al. PRL 67, 1759 (1991)

Krim et al. (~~pre~~print) PRL Jan '93 70, 57 (1993)

Advantages: - Direct topographic information
- Wide spread availability

Disadvantages: - Misses all pores and overhangs
- Sensitive to tip radius and chenical inhomogeneities
- operates only on a conducting surface

ATOMIC FORCE MICROSCOPY



$F \propto K \Delta z$

$F_f/A \approx 10^9$ N/m^2 at 1nm/s

C. Lieber et al., Harvard Univ., C.M. Mate, G. McClelland et al., IBM Almaden, E. Meyer et al., Basel, M.B. Salmeron et al., Lawrence Berkeley Lav.

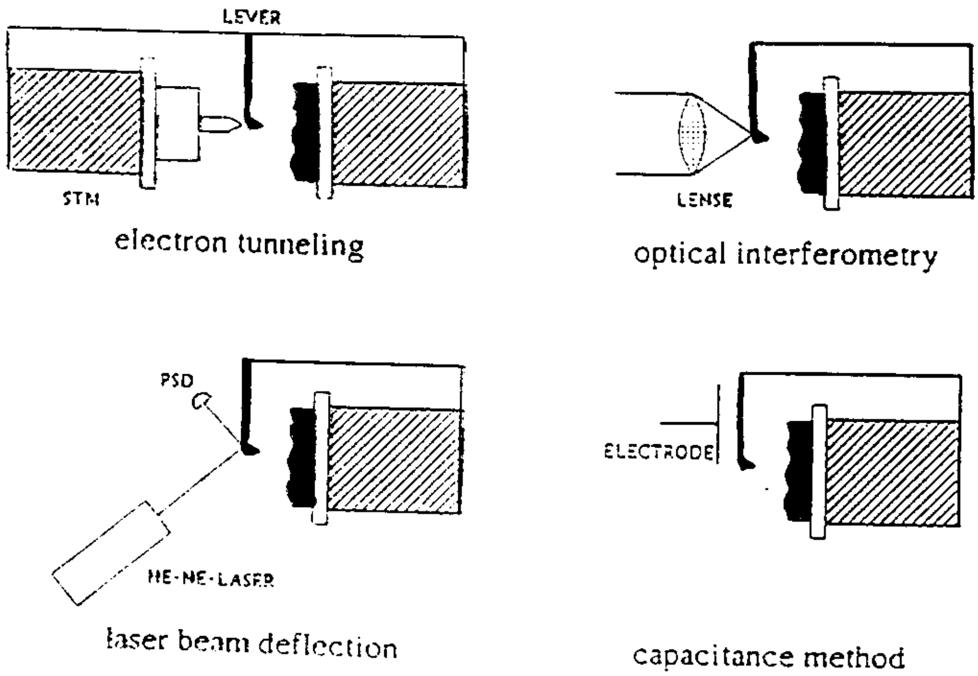
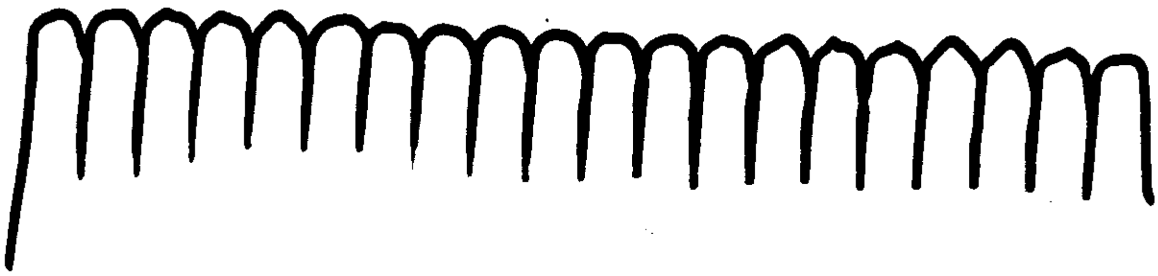


FIGURE 12 Geometries of the four more commonly used detection systems for measurement of cantilever deflection. In each setup, the sample mounted on piezoelectric body is shown on the right, the cantilever in the middle, and the corresponding deflection sensor on the left. (From Meyer, E. (1992), *Surf. Sci.*, Vol. 41, pp. 3-49. With permission.) Sarid, "Scanning Force Microscopy"

$$F \propto k \Delta z$$

- Δz can be accurately measured
- k may only be known within a factor of 2
- F arises from <u>all</u> forces (atomic, electrical, magnetic, capillary, etc.)

Sharp tip AND blunt tip on periodic substrate:



Blunt tip over sharp asperity on surface: (images the tip!)

Sharp tip over sharp asperity on surface:

Atomic Periodicity

$\neq$

Atomic Resolution!

(But atomic resolution has been achieved regularly)

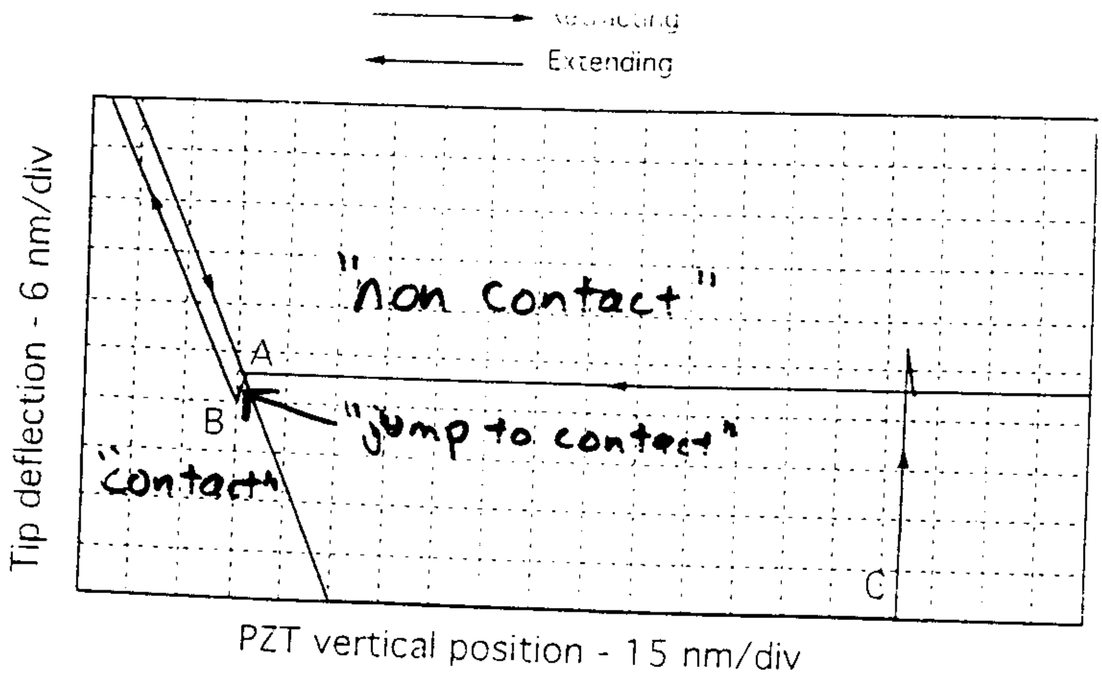


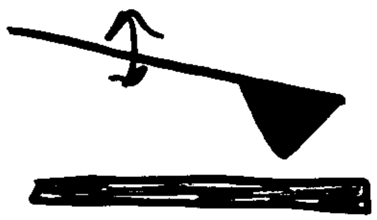
FIGURE 37 Displacement curve of the cantilever tip as it is pushed toward (extending) and pulled away fr((retracting) a silicon sample in measurements made in the ambient environment. The large separation betwe point B where the tip is touching the sample and point C where the tip is pulled off the sample is due a lar pull-off (adhesive) force between the tip and the sample. (From Ruan, J. and Bhushan, B. (1994a), *ASME Tribol.*, Vol. 116, pp. 378–388. With permission.)

"noncontact"

– can be insensitive

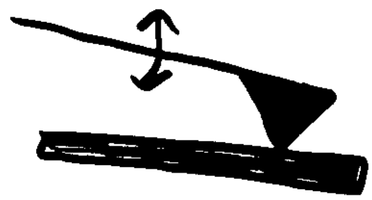
"contact"

– can be destructi



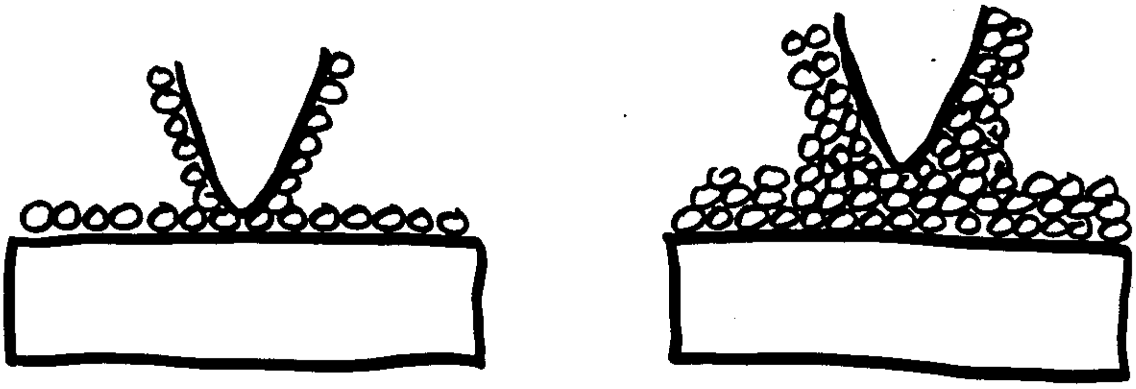
noncontact vibration

- Is sensitive to force, via resonant frequency of cantilever



contact "tapping"

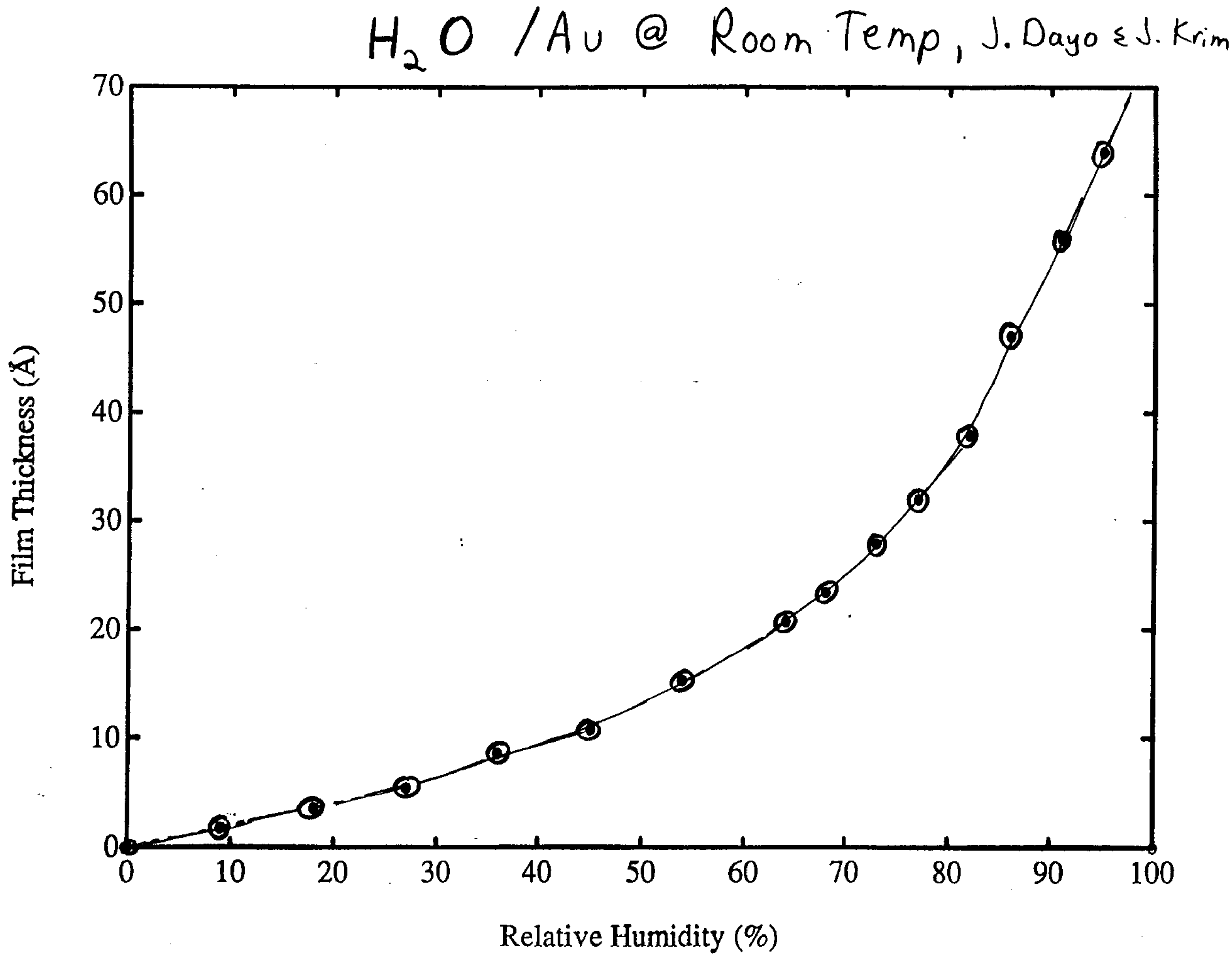
- reverse direction before any damage occurs.



More complications!

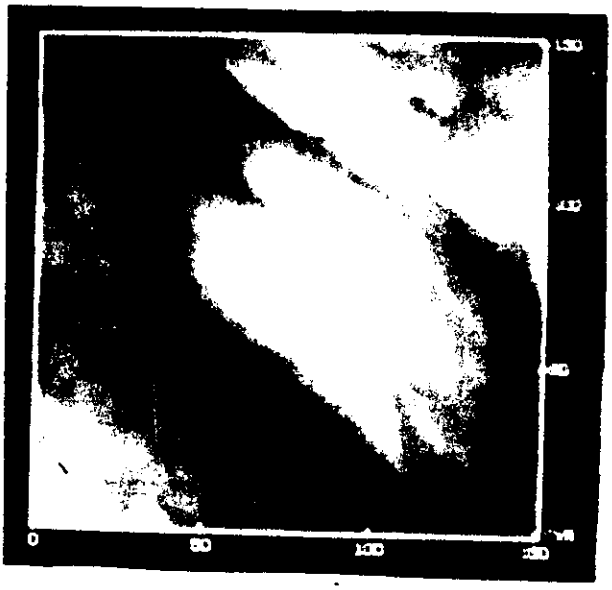
Adsorbed Films /

Contaminant Layers.

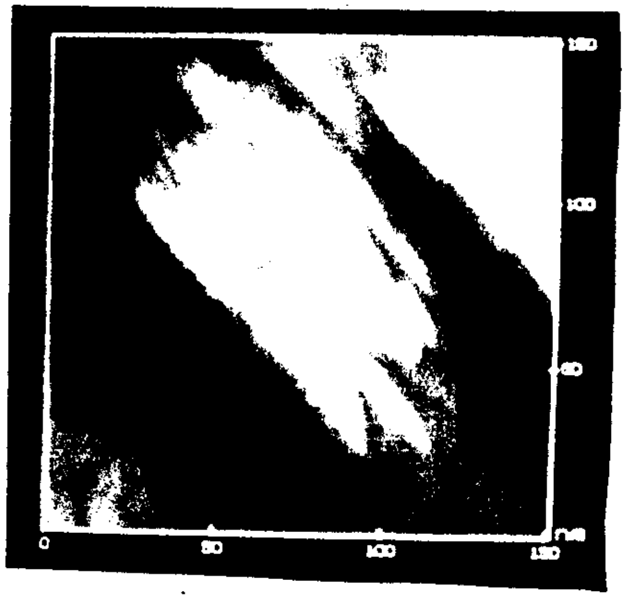


H2O /Au @ Room Temp, J. Dayo & J. Krim
70
60
50
40
30
20
10
0
Film Thickness (Å)
0
10
20
30
40
50
60
70
80
90
100
Relative Humidity (%)

21

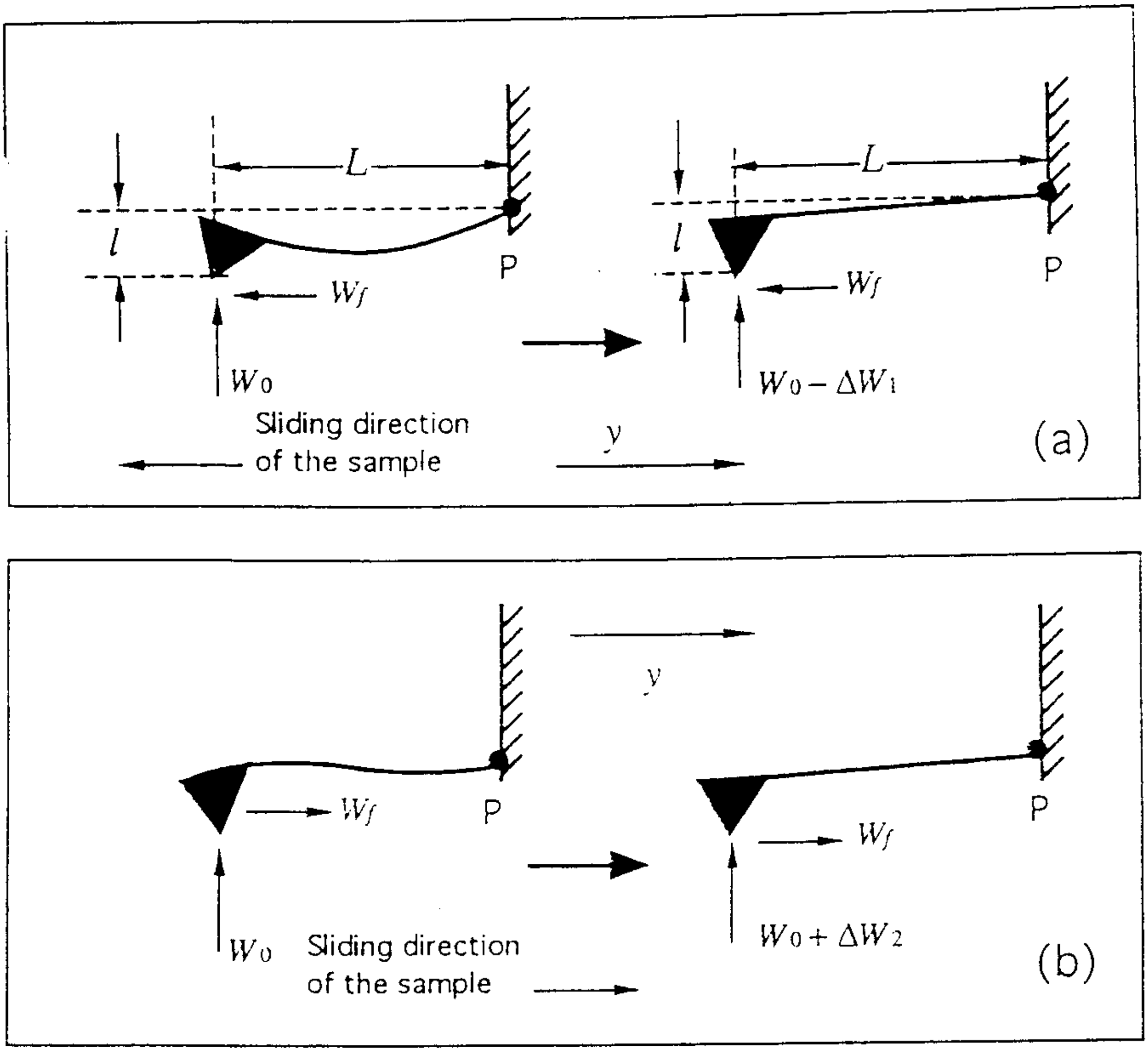


9 Å H_2O/Au



43 Å H_2O/Au

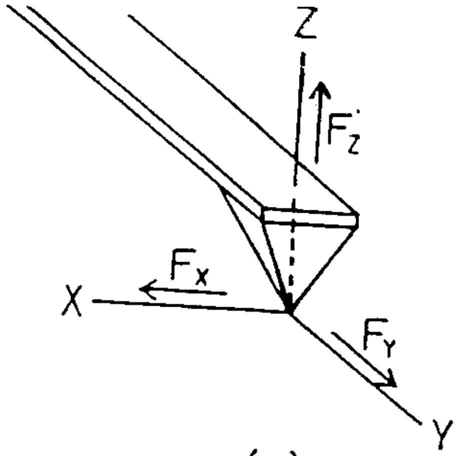
STM images of a gold film with varying thicknesses of water on top.



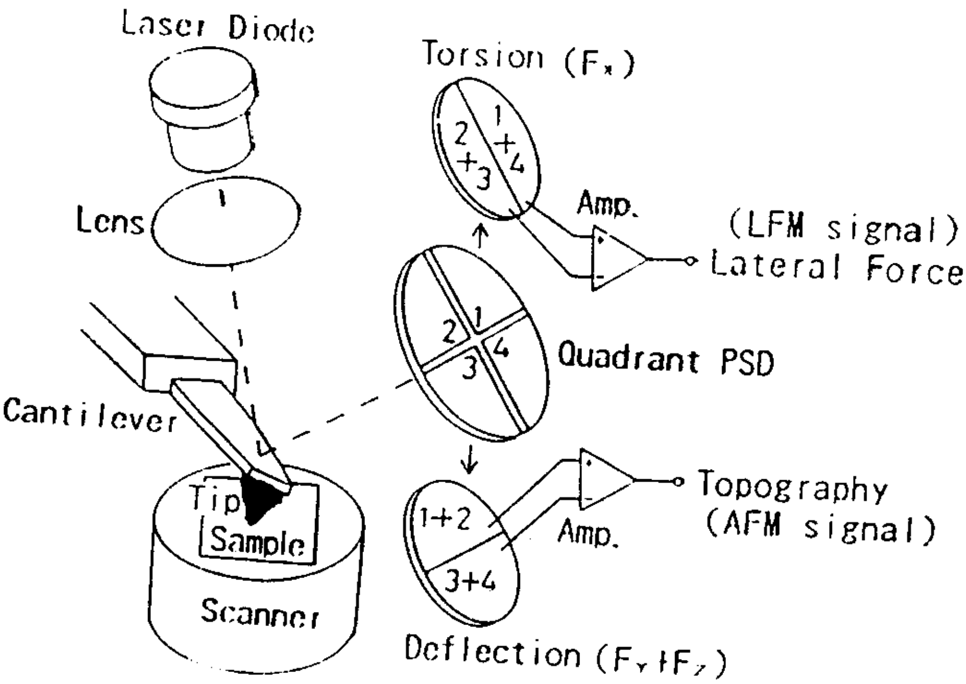
Bhushan, "Nanotribology"

GURE 33 Schematic showing an additional bending of the cantilever due to friction force when the sample
canned in y- or −y-direction (left). This effect will be canceled by adjusting the piezo height by a feedback

Friction!! (Which has high sensitivity to surface chemistry.) (Or lack thereof!!)



(a)



(b)

Bhushan "Nanotribology"

Simultaneous detection of lateral and normal forces.

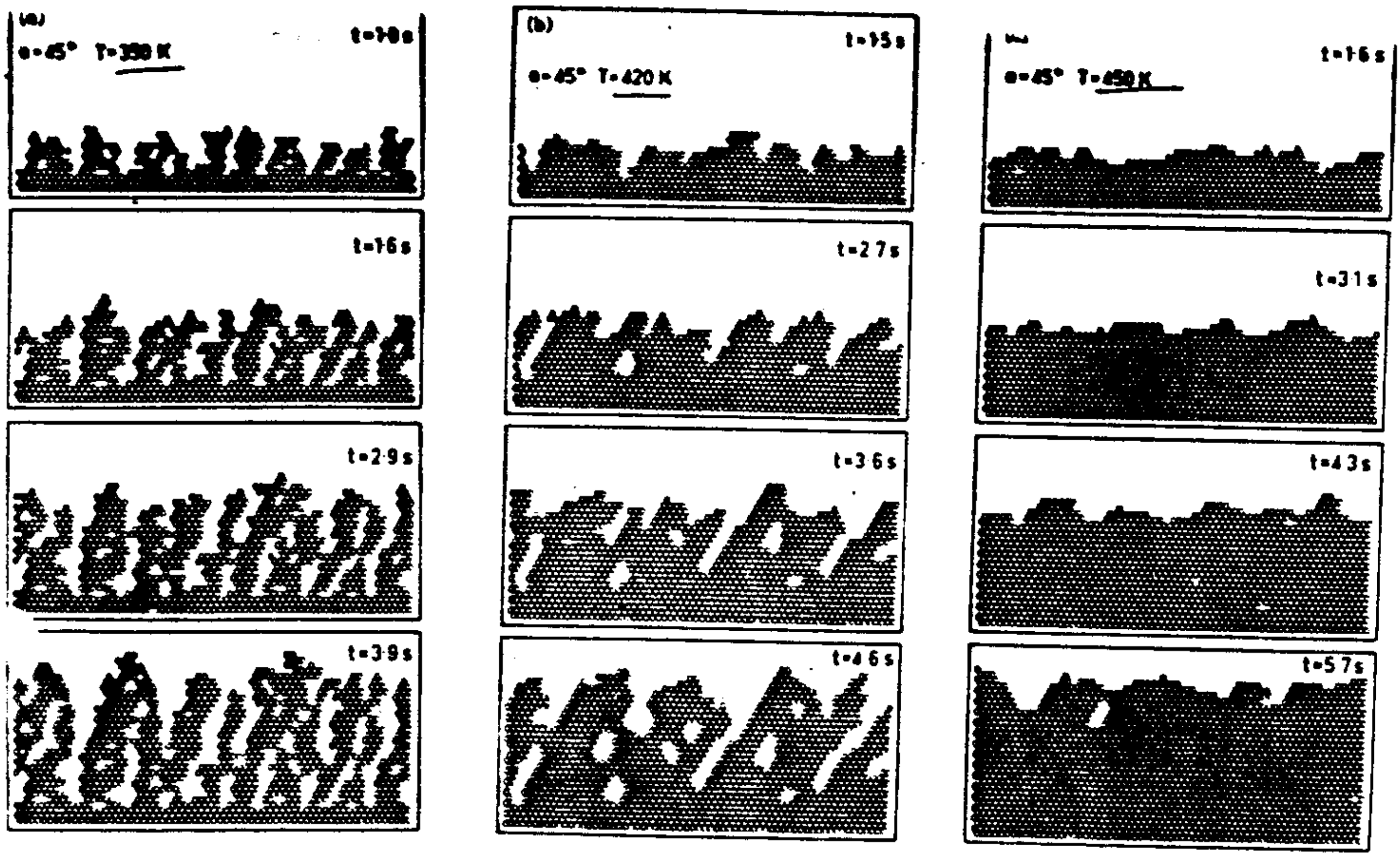
Problem	Problem Resolution
porosity	adsorption, x-rays, depth profiling
tip radius	screen tips via single features
surface films	work in vacuum or completely submerged in liquid, or in dry N_2
friction	monitor lateral and normal forces; be careful of large contact forces.

"Where there's a will, there's a way"

Lecture I Conclusion:

- Scanning Probe Microscopes can provide powerful and quantitative characterizations of atomic-scale film topologies.

- <u>But</u>, they can easily yield artifacts which are difficult to detect by SPM alone. Ideally they should compliment, not replace, existing techiques.



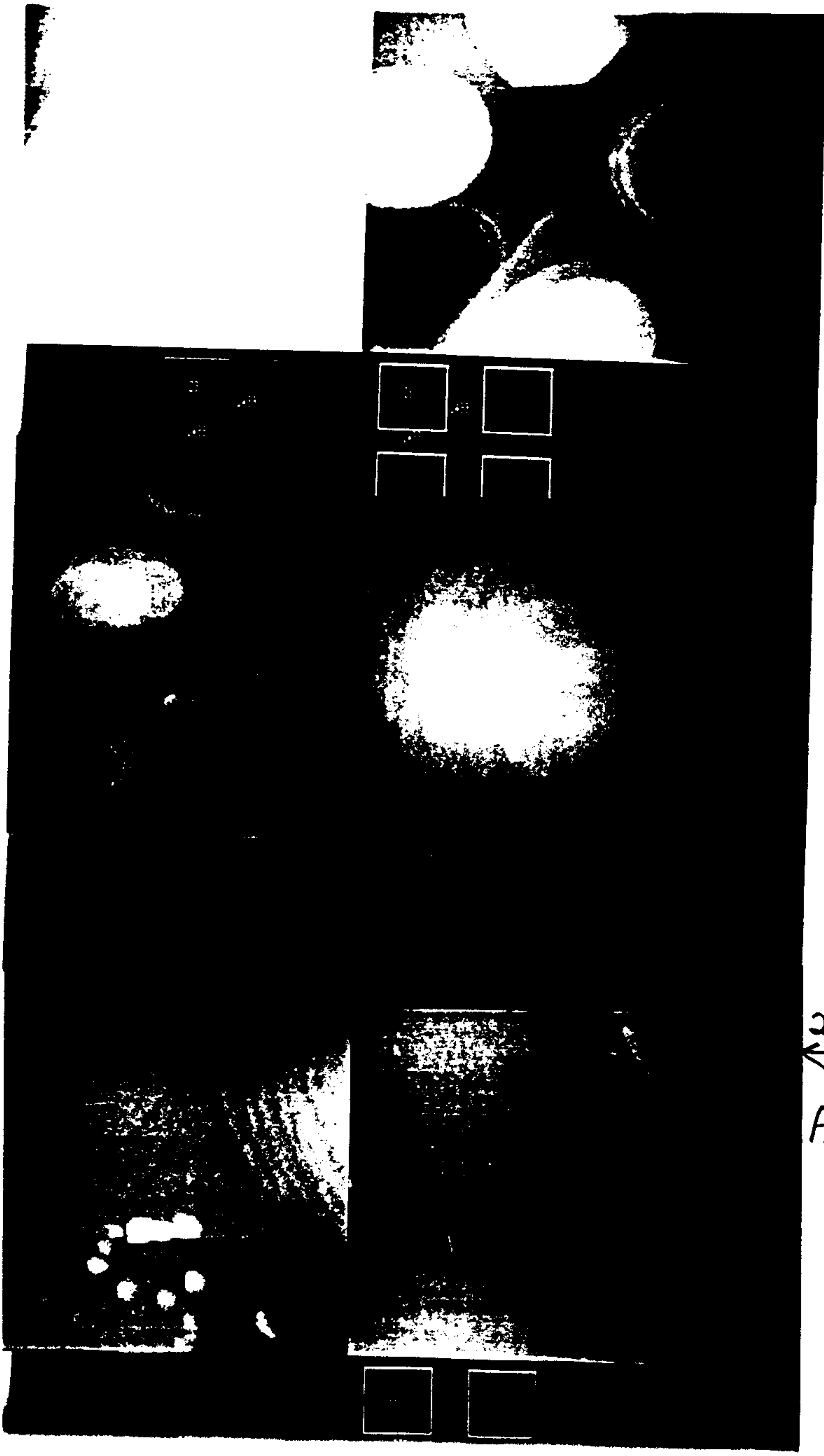
Müller, JVSTA

150 nm

Ag(111)

150nm

3 nm



200 nm

3 nm

100 nm

3 nm

.00 nm

Ag(111)

200 nm

Ag(111)