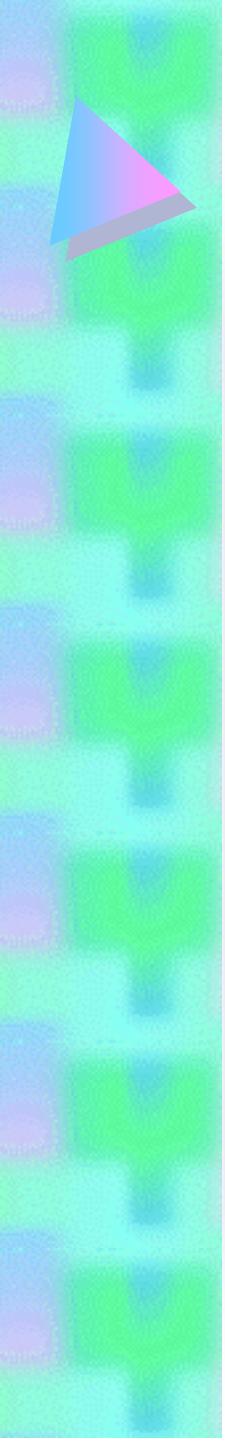




# **Scanning Probe Microscopy: STM, AFM and Co.**

**Eliana Quartarone**  
**Dipartimento di Chimica Fisica “M. Rolla”**  
**Università di Pavia**

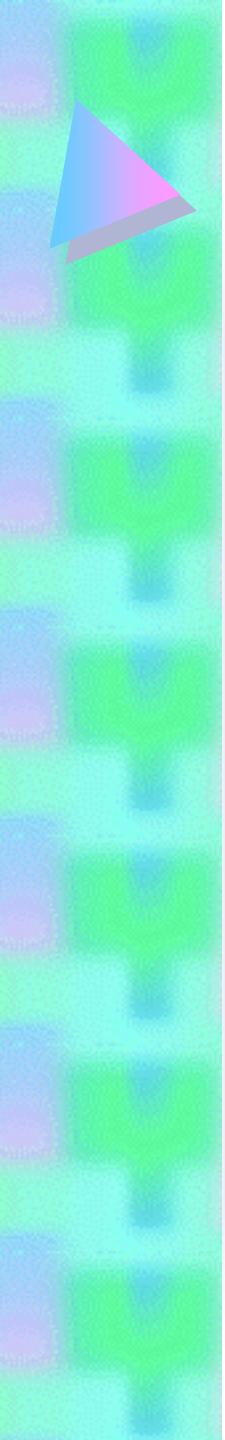


# Introduzione

- **Microscopia ottica ed elettronica**
  - Ingrandimenti:  $\sim 1000\times$  e  $\sim 100000 \times$  rispettivamente
  - Immagini nel piano orizzontale alla superficie
- **SPM**
  - Ingrandimenti: fino a  $1,000,000 \times$
  - Alta risoluzione spaziale
  - Immagini in tre dimensioni: piano orizzontale X-Y e verticale Z
  - Nessun trattamento del campione



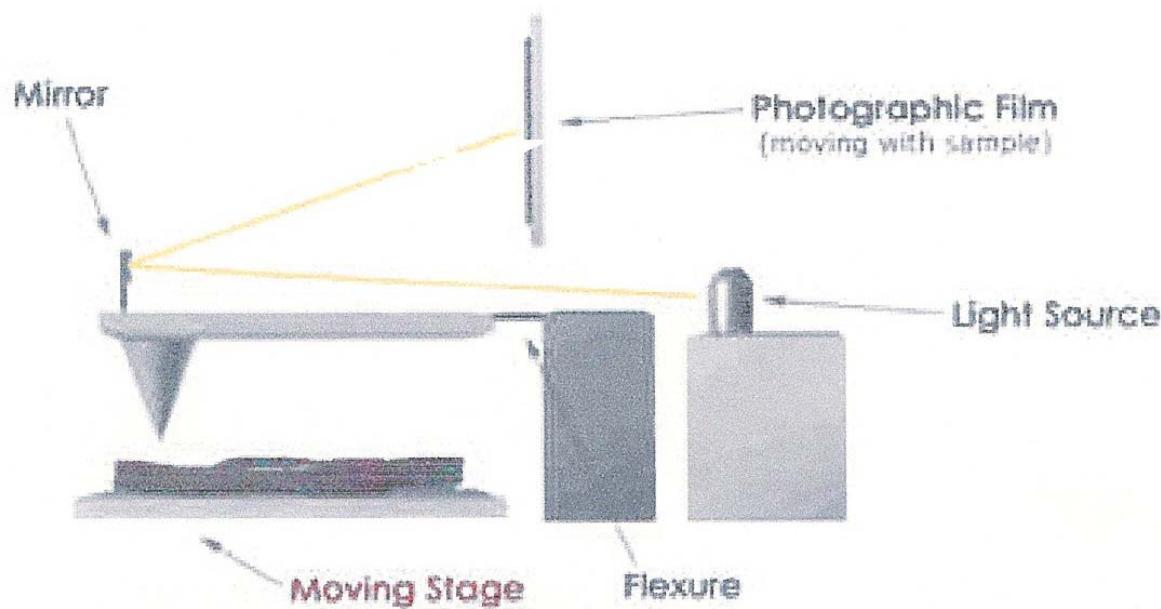
**Misure puntuali di superfici e proprietà**



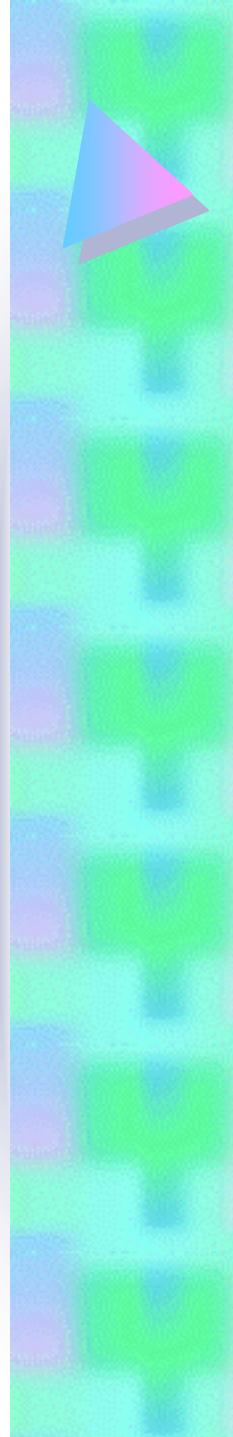
# SPM: Le origini

- **Profilometro a stilo (Schmalz, 1929)**
- **Il c.d. Topographiner (Young, 1971)**
- **Microscopio ad Effetto Tunnel (Benning e Rohrer, 1980)**
- **Microscopio a Forza Atomica (Benning e Quate, 1996)**

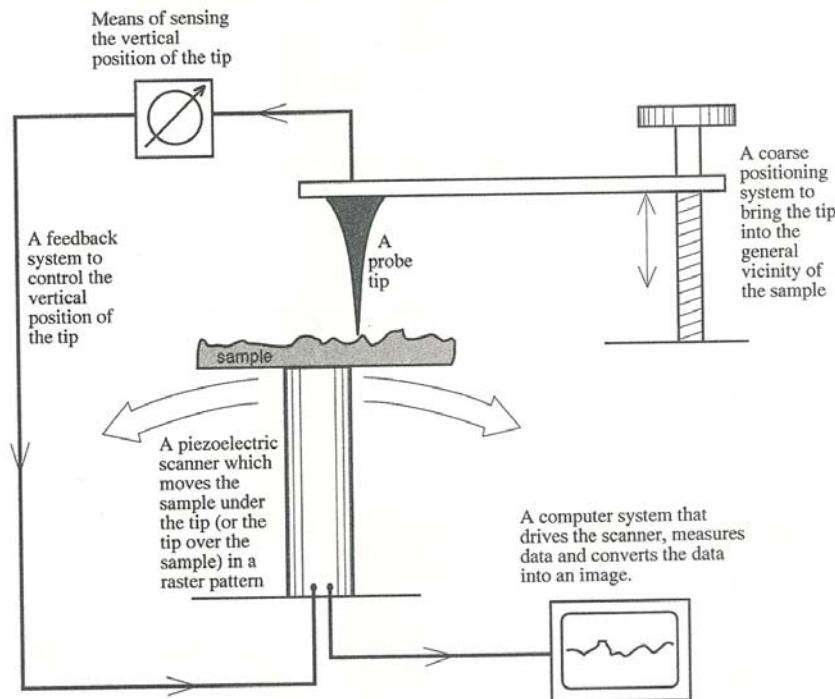
# Profilometro a stilo



*Figure 1: An example of a surface profiler made in 1929.*

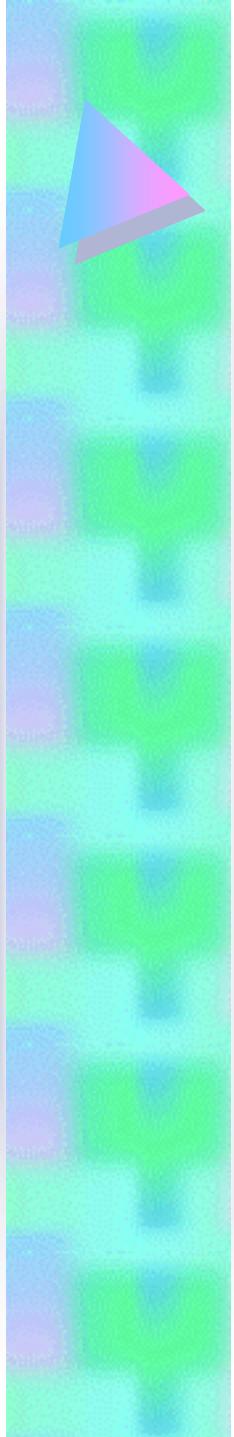


# SPM: il c.d. approccio “*Local probe*”



Sonda “puntuale” in grado di lavorare a ridottissima distanza dalla superficie del campione:

**ALTA RISOLUZIONE SPAZIALE**

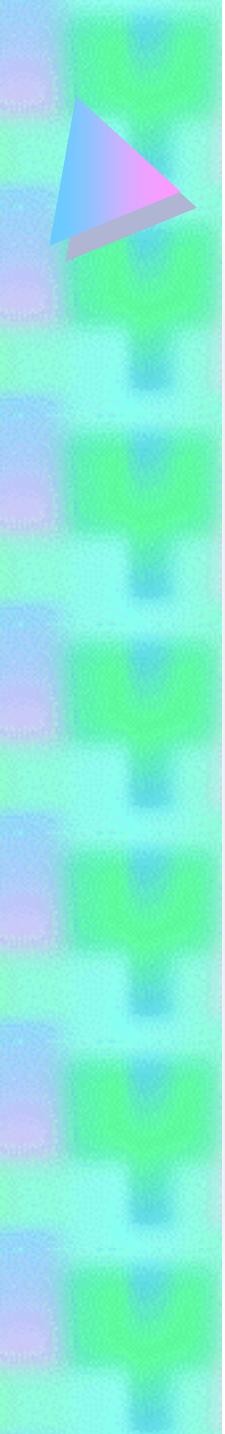


# SPM: il c.d. approccio “*Local probe*”

- **STM: Corrente di tunnelling  
(distanza punta-campione 5÷50 Å)**
- **AFM: Forze di interazione punta-campione  
(contatto e non contatto)**

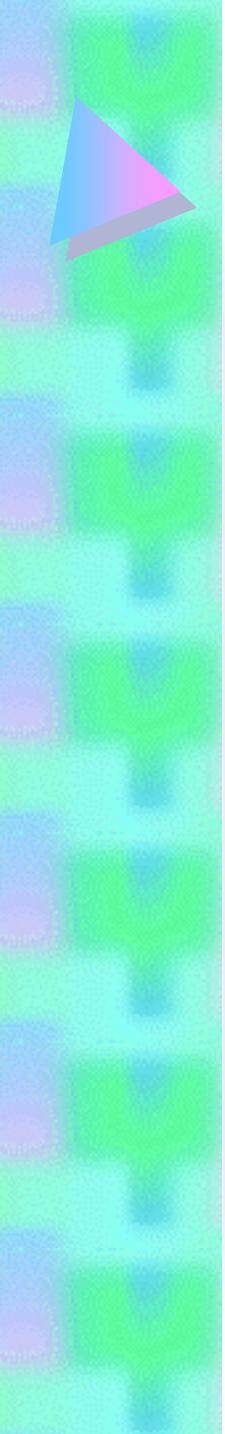


**TOPOGRAFIA**



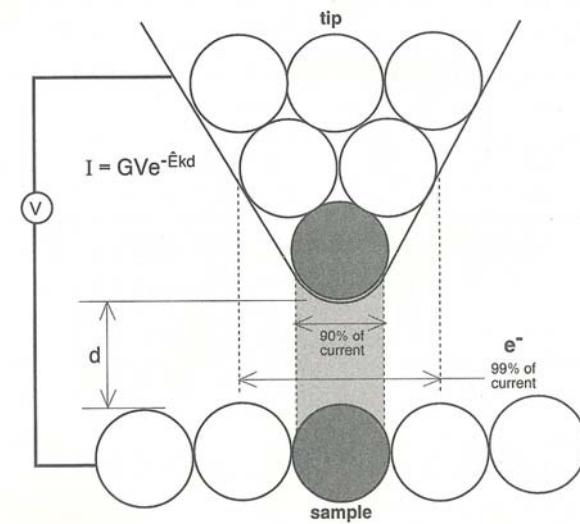
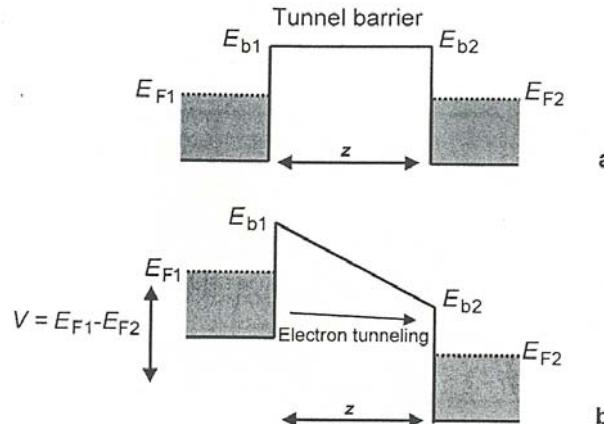
# **Microscopia ad Effetto Tunnel (STM)**

- **Fondamenti teorici**
- **Modalità di scansione**
- **Applicazioni**



# Microscopia ad Effetto Tunnel (STM): fondamenti teorici

- **Principi del tunnelling elettronico**



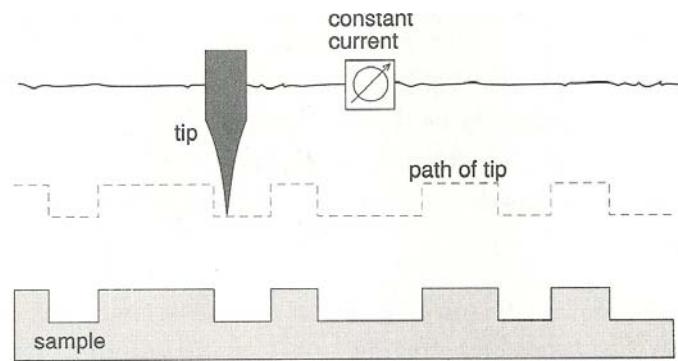
$$\Psi(z) = \Psi(0)e^{-kz}$$

$$k = \frac{\sqrt{2m(V - E)}}{\hbar}$$

$$I \propto e^{-2kz}$$

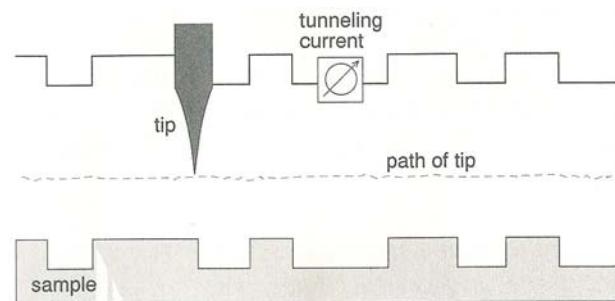
# Microscopia ad Effetto Tunnel (STM): modalità di scansione

- **Corrente costante**

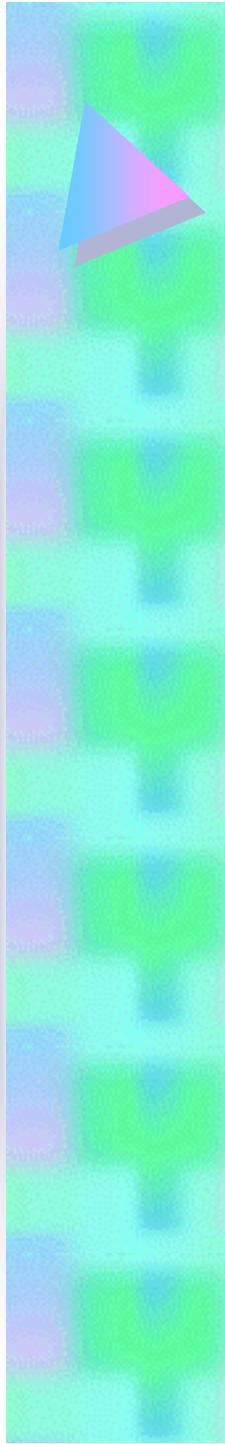


Topografia: misura puntuale di  $I$

- **Altezza costante**

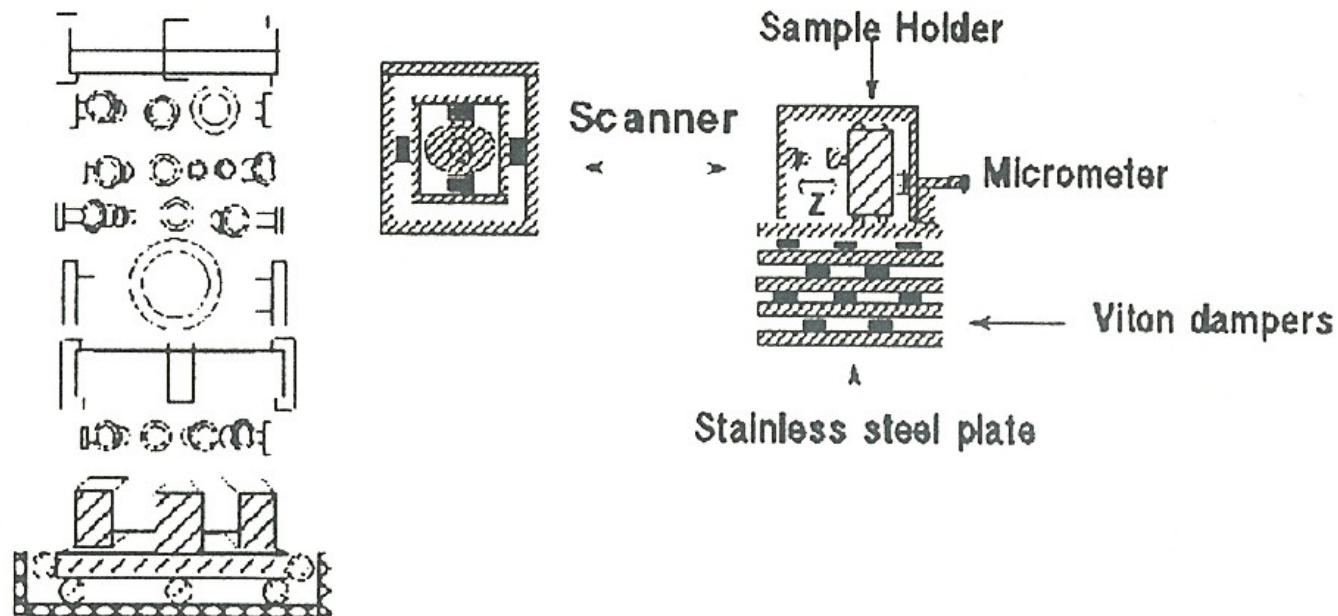


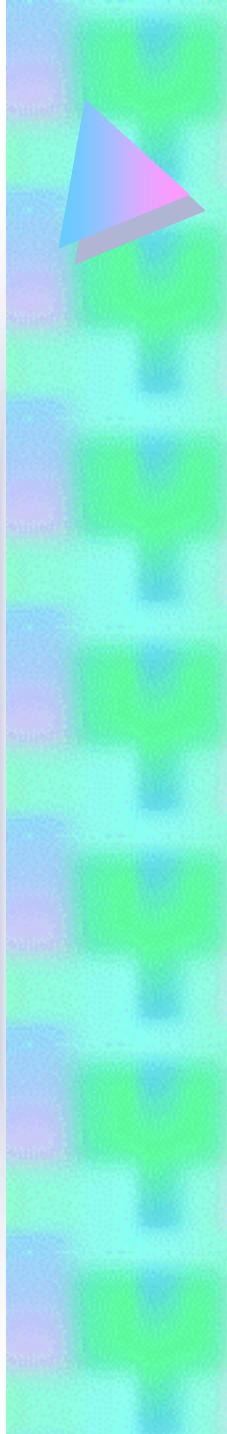
Topografia: misura puntuale del  
movimento dello scanner



# Microscopia ad Effetto Tunnel (STM): strumentazione

UHV STM system



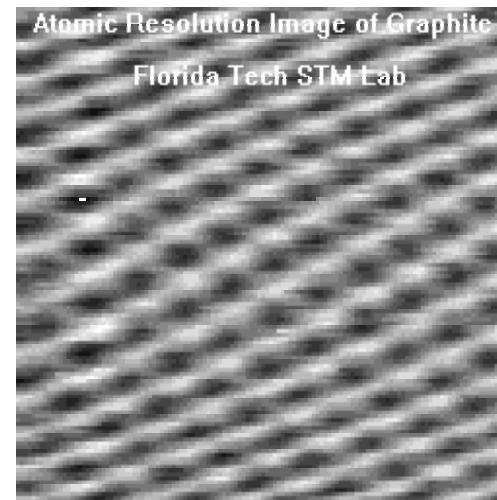


# Microscopia ad Effetto Tunnel (STM): applicazioni

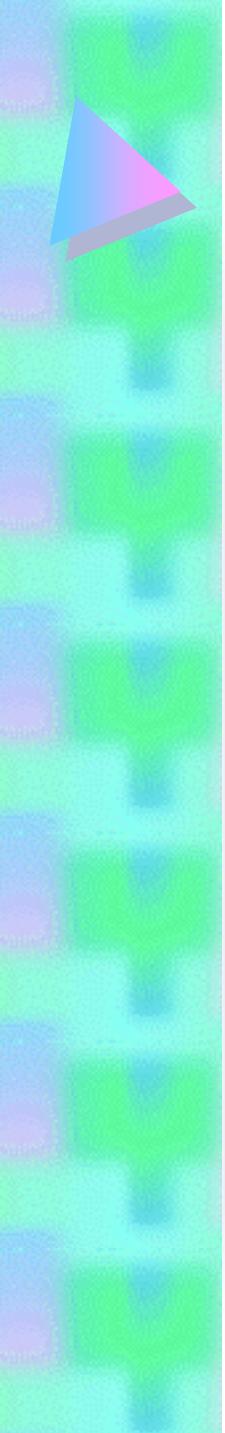
- Metalli
- Semiconduttori (es. Si, GaAs)
- Molecole biologiche (es. DNA)
- Strutture non periodiche

# Microscopia ad Effetto Tunnel (STM): applicazioni

- Vantaggi: risoluzione atomica



- Svantaggi: solo materiali conduttori



# **Microscopia a forza atomica (AFM)**

## **VANTAGGI**

- **Materiali sia isolanti sia conduttori**
- **Alta versatilità**
- **Semplicità di utilizzo**
- **Alta risoluzione (0.1Å verticale, 1.0Å laterale)**
- **Non distruttività**
- **Nessuna preparazione del campione**

# Microscopia a forza atomica (AFM): strumentazione

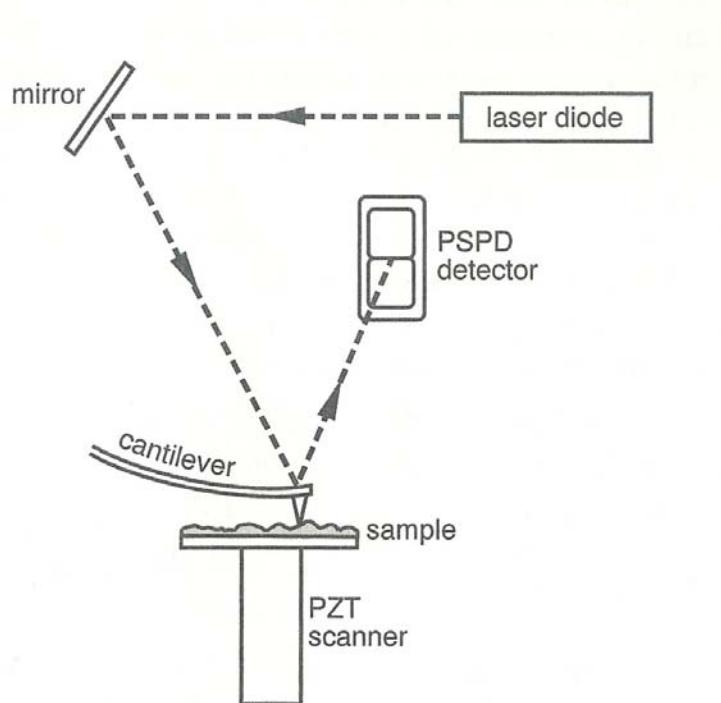
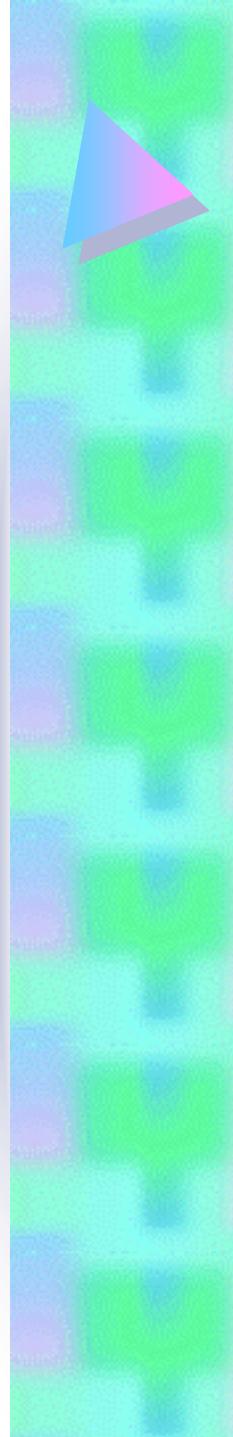
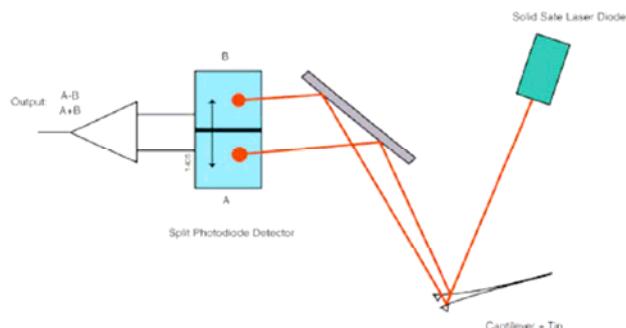
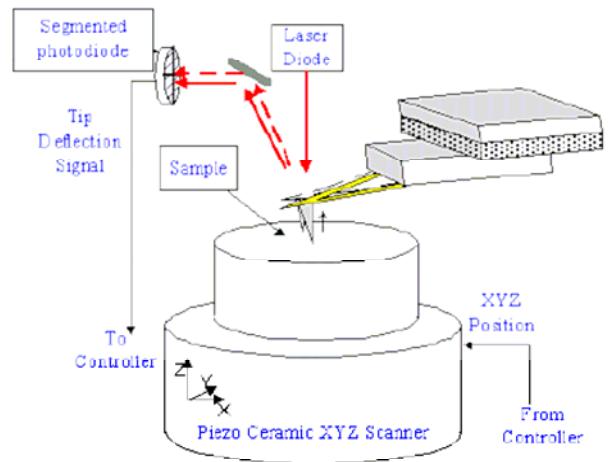


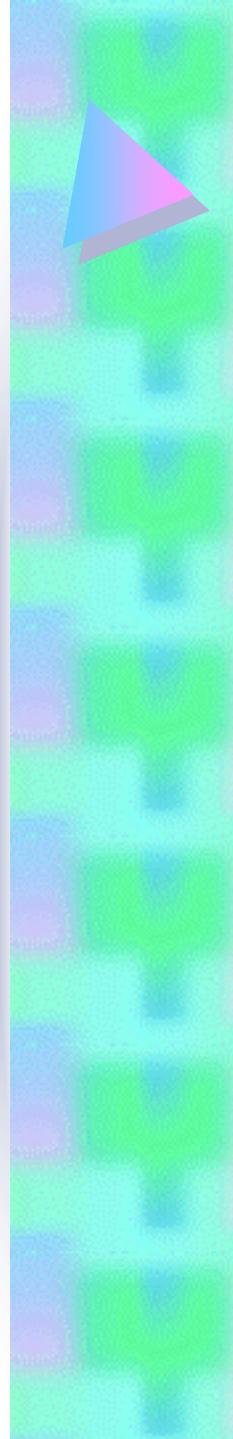
Figure 1-5. The beam-bounce detection scheme.



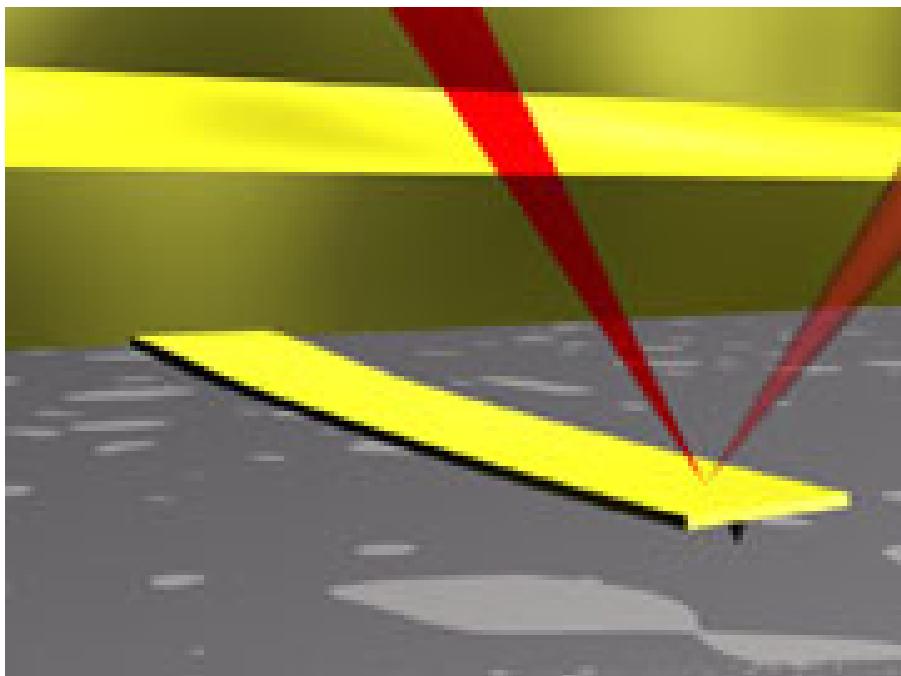
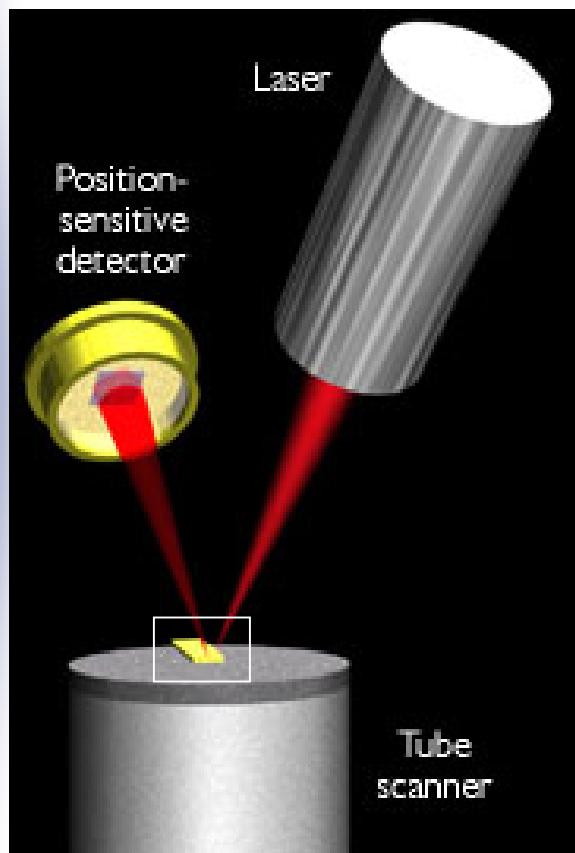
# Atomic Force Microscopy



- Light lever used for contact mode, non-contact mode, and tapping mode.
- Long beam path (several cm) amplifies changes in beam angle.
- Laser light from a solid state diode is reflected off the back of the cantilever and collected by a position sensitive detector (PSD).
- Angular displacement of cantilever results in one photodiode collecting more light than the other photodiode, producing an output signal which is proportional to the deflection of the cantilever.
- Detects cantilever deflections  $< 1\text{\AA}$  (thermal noise limited).



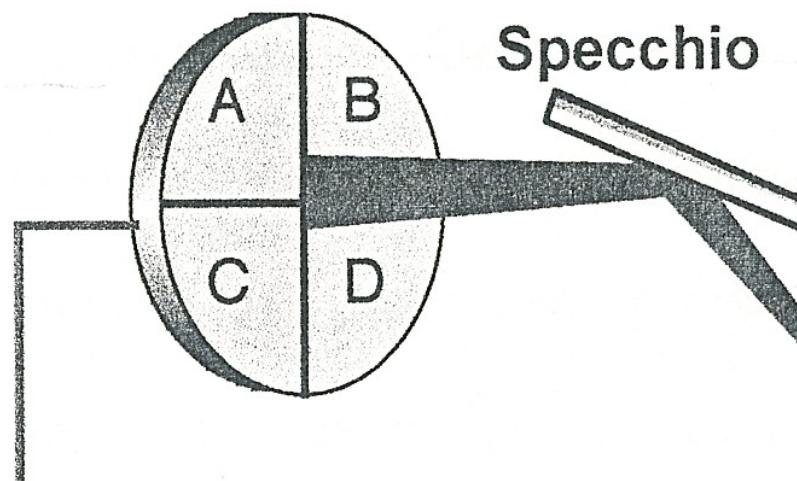
# Microscopia a forza atomica (AFM): strumentazione



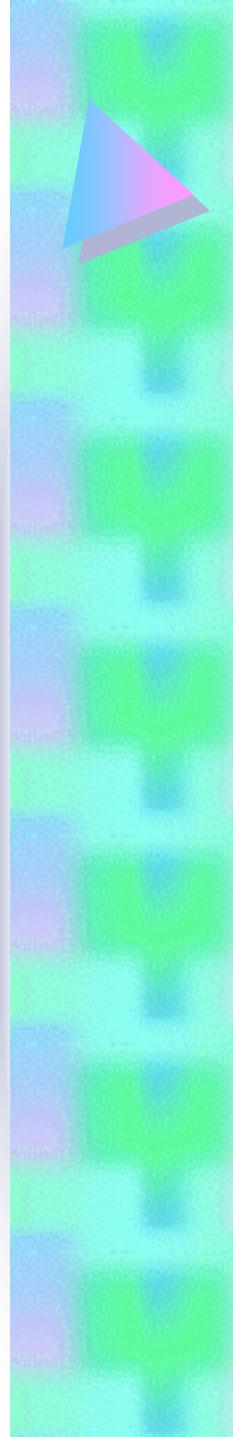
# Microscopia a forza atomica (AFM): strumentazione

## Il detector

Rivelatore sensibile  
alla posizione



- Posiziona il laser sulla punta (A+B)
- Massimizza l'intensità del laser (A-B)



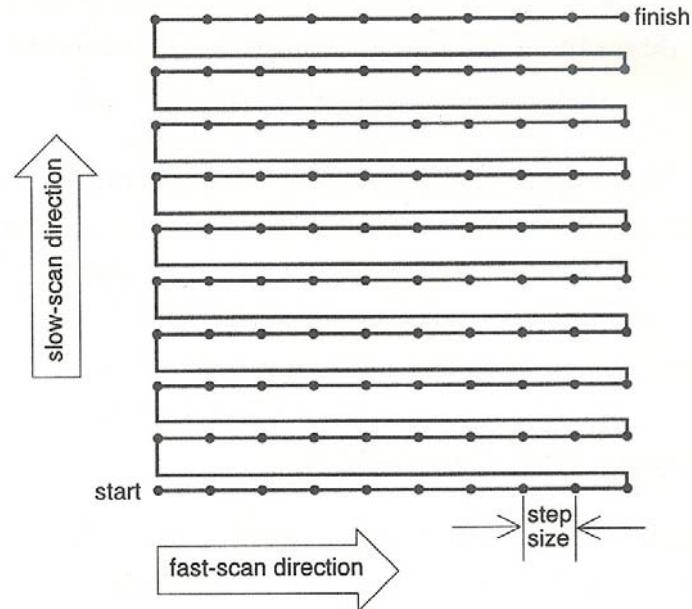
# Microscopia a forza atomica (AFM): strumentazione

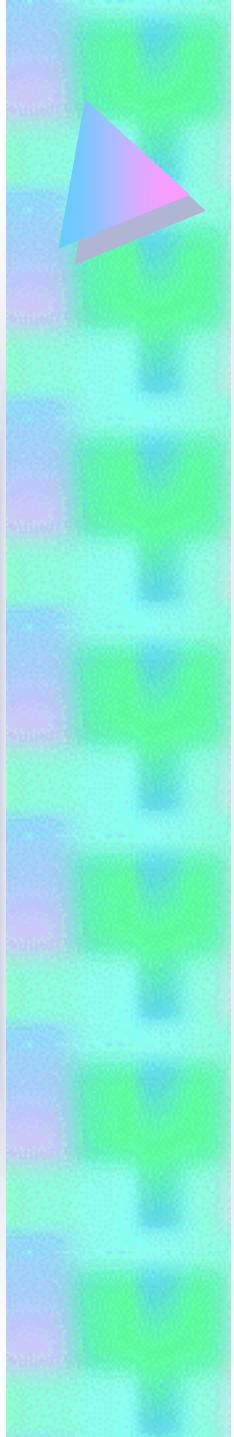
## Lo scanner

- Ceramico Piezoelettrico: posizionamento fine
- Movimento in x, y e z da pochi angstrom fino a 100 micron

### DATA SET

- Altezza dello scanner in z: costant force (AFM) o constant current (STM) mode
- Deflessioni del cantilever o della corrente i tunneling: constant height mode (AFM, STM)





# Microscopia a forza atomica (AFM): strumentazione

## Lo scanner

- Non linearità
  - Non linearità intrinseca
  - Isteresi
- Errori e deformazione dell'immagine
- Correzioni:
  - Software (grate di calibrazione) e hardware (sensori esterni e sistemi di feedback)

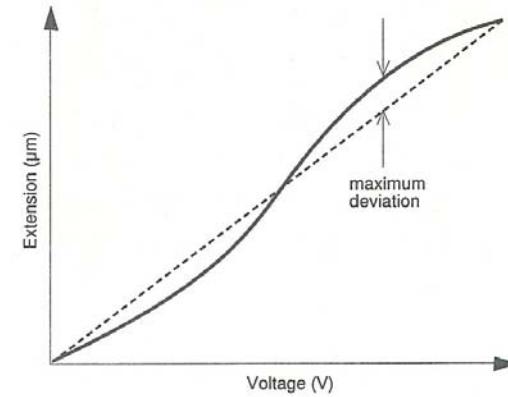


Figure 2-3. Intrinsic nonlinearity of a scanner.

# Microscopia a forza atomica (AFM): strumentazione

## Il cantilever

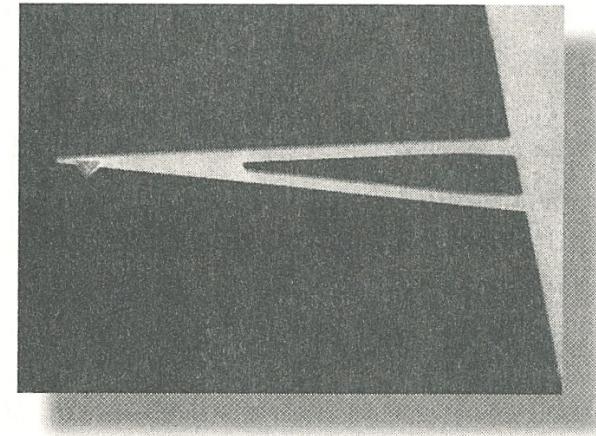
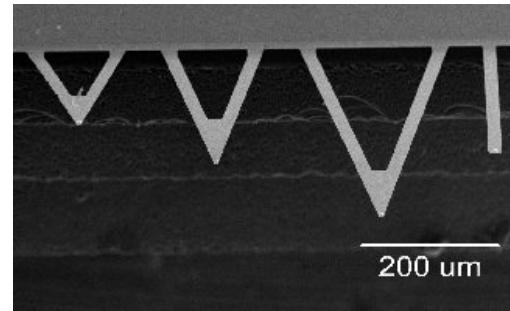
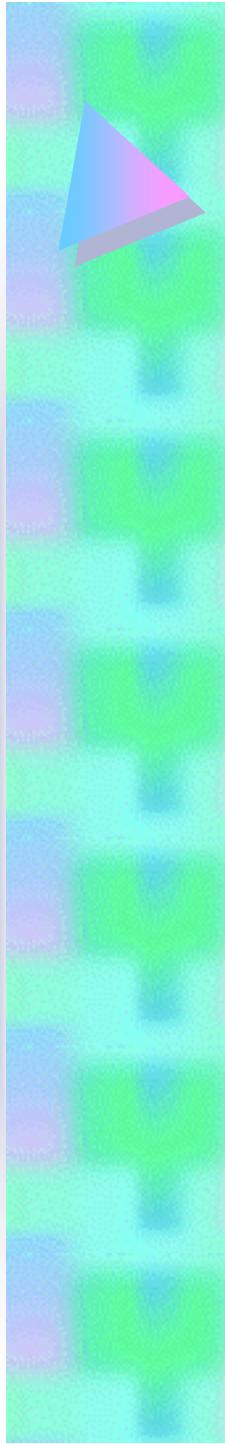
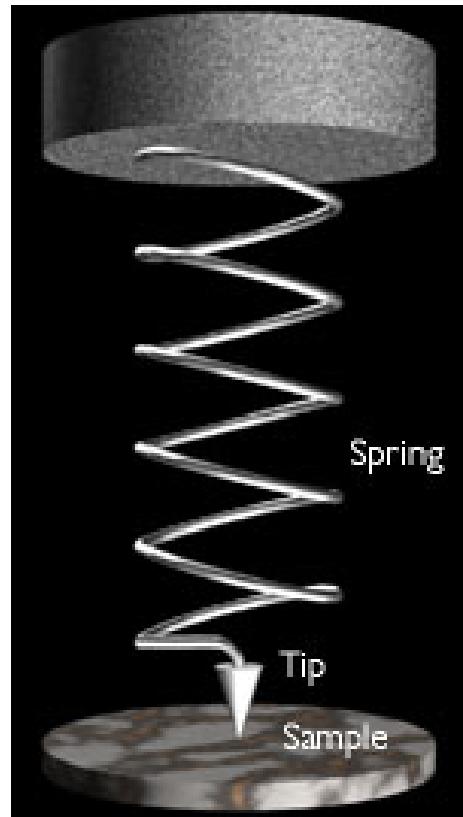


Figure 3-1. SEM image of a cantilever.



# Microscopia a forza atomica (AFM): strumentazione

## Il cantilever



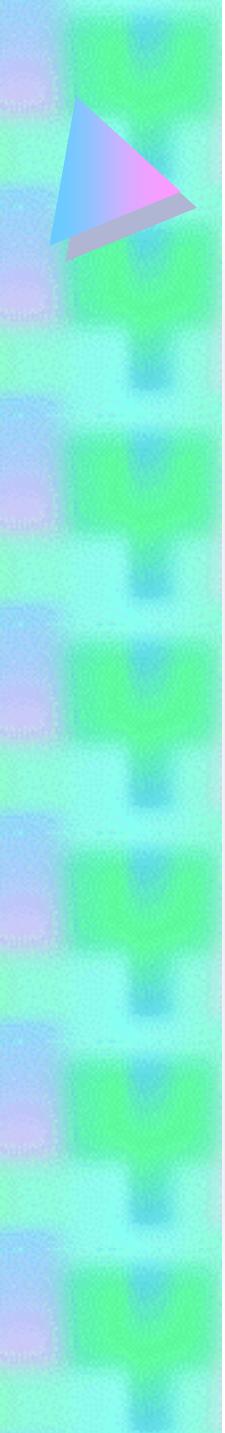
AFM cantilevers have spring constants of ~0.1 N/m and should have a high resonant frequency.

Resonant frequency of a spring:

$$1/2\pi \sqrt{\text{spring constant}/\text{mass}}$$

Therefore AFM cantilevers should have a small mass.

$$f = \frac{1}{2} \pi \times \frac{\sqrt{k}}{m_{eff}}$$

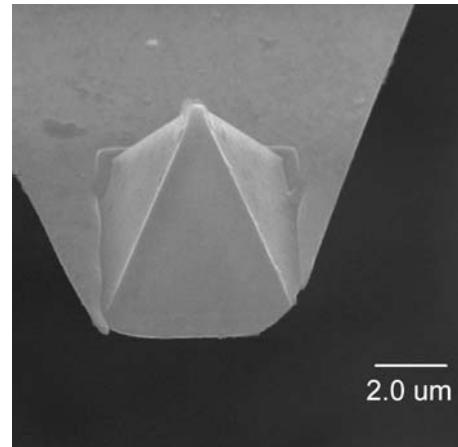


# Microscopia a forza atomica (AFM): strumentazione

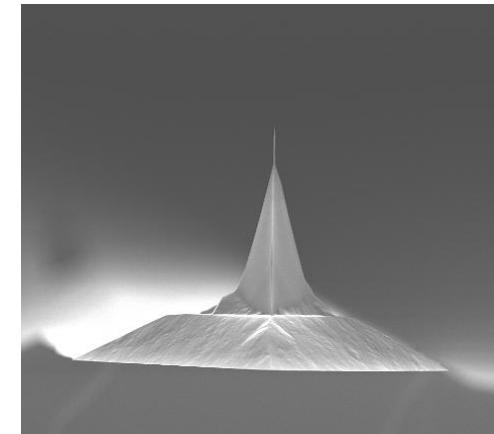
## La punta

- Costruzione: fotolitografia
- Criteri di scelta della punta (Si, SiN<sub>3</sub>)

Punta piramidale



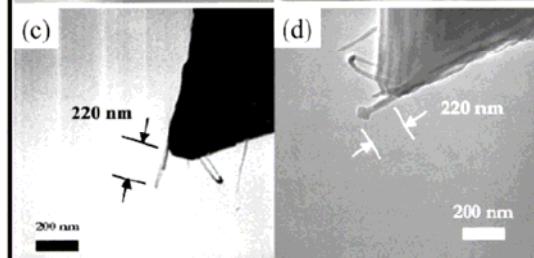
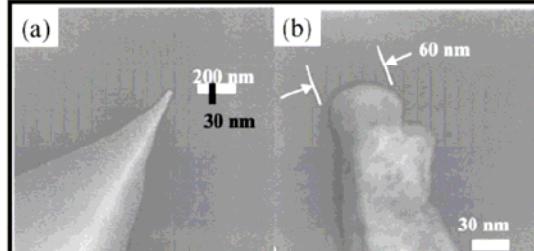
Punta conica



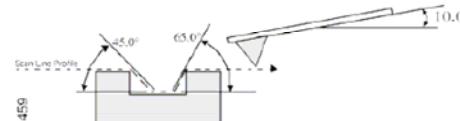
# Microscopia a forza atomica (AFM): strumentazione

## La punta

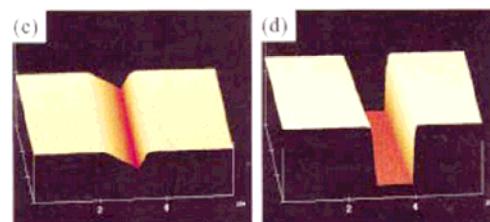
Carbon Nanotube AFM Tips



- A. New Tip
- B. After 180 Scans
- C. New CNT Tip
- D. After 1105 Scans



1 μm Wide / 2 μm Deep Trench

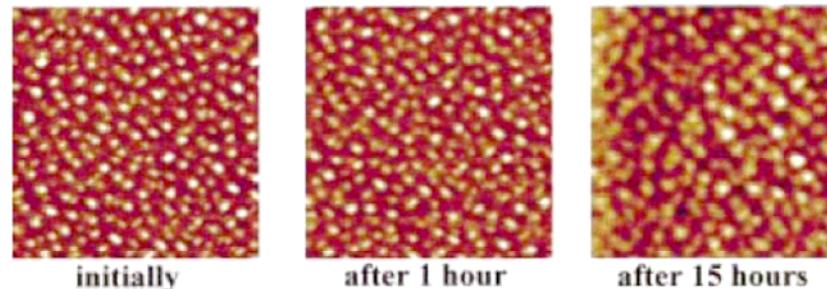


- C. Standard Tip
- D. CNT Tip

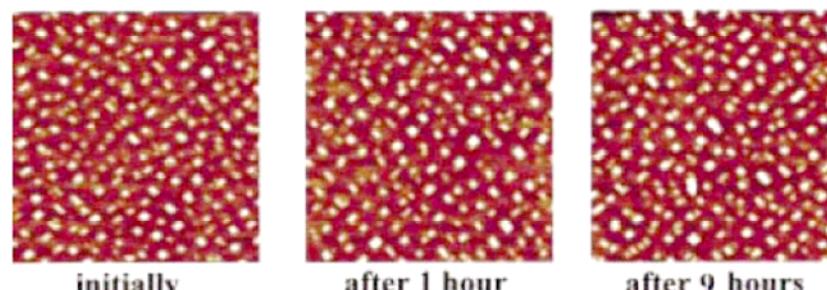
# Microscopia a forza atomica (AFM) · strumentazione

## CNT AFM Tips – Reduced Wear

SiGe 3D Islands / Quantum Dots

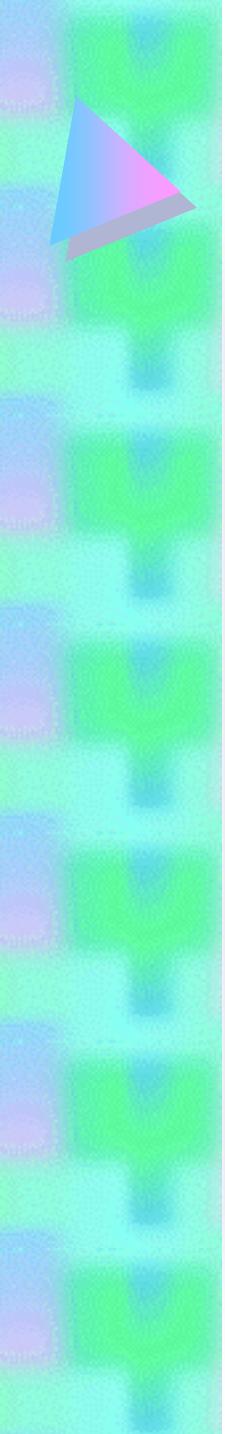


Standard Contact  
Mode Tip



CNT Tip

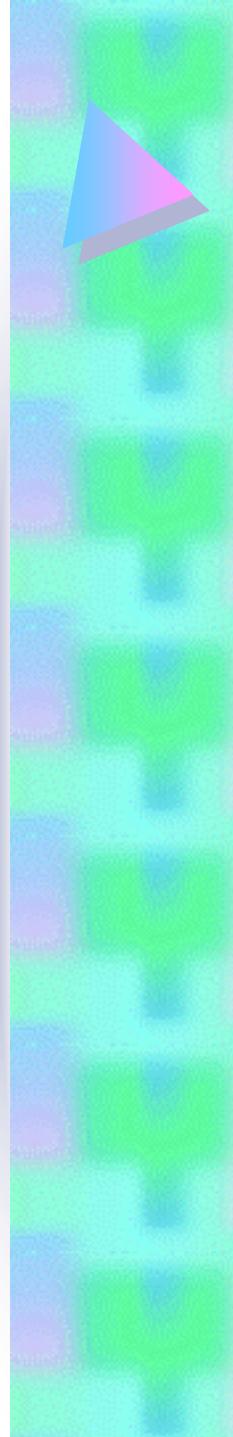
CNT AFM Tip shows no wear or decreased resolution, while standard AFM tip shows wear after 1 hour.



# Microscopia a forza atomica (AFM): principi di base

**Forze coinvolte ◀▶ separazione punta-campione:  $10^{-13}$  N÷ $10^{-6}$  N**

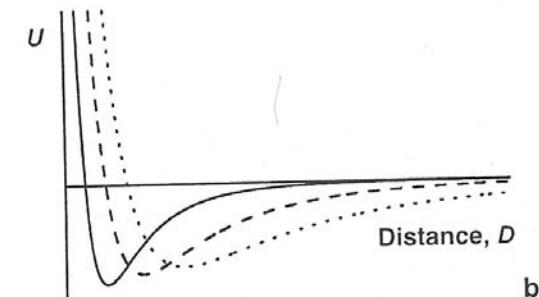
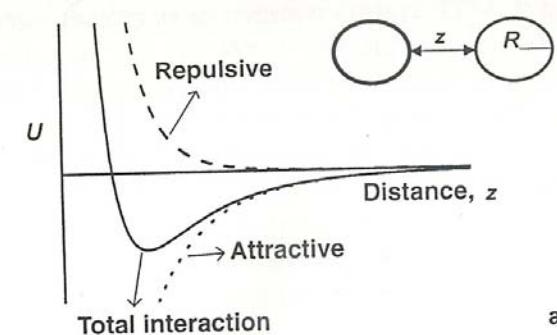
- Forze di Van der Waals (di dispersione)
- Repulsione ionica
- Forze di attrito
- Forze di adesione
- Forze elettrostatiche
- Forze magnetostatiche



# Microscopia a forza atomica (AFM): interazioni a corto raggio

## Interazioni di van der Waals:

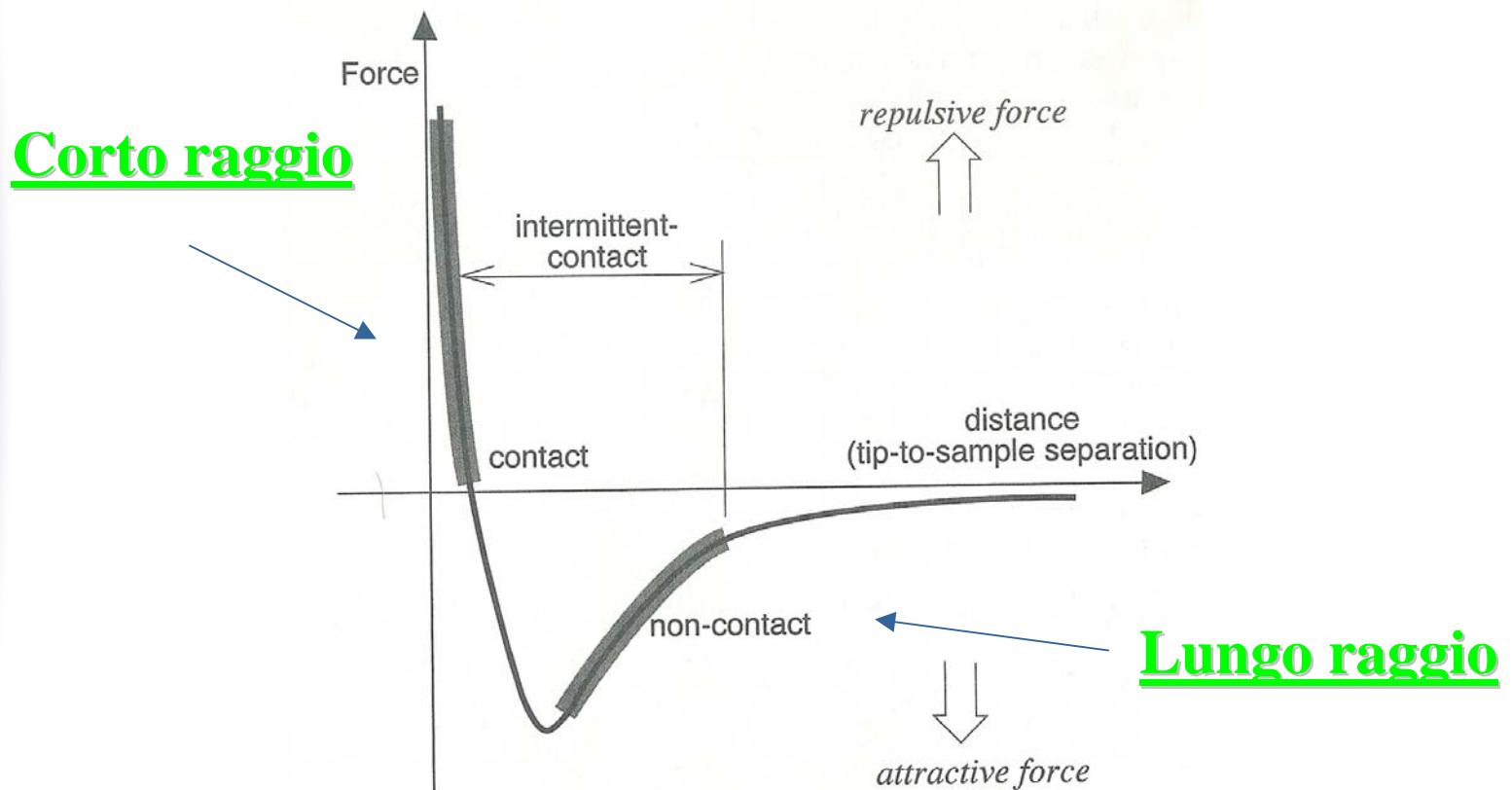
- Attrattive
- Repulsive

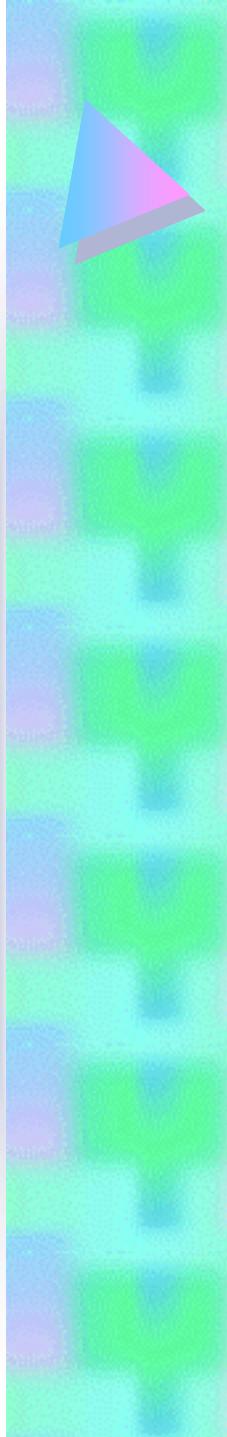


	For a Spring	For a Sphere-plane, $z \ll R$
Potential	$= U$	$= 1/2 kz^2$
Force	$= -dU/dz$	$= -kz$
Force gradient	$= -d^{-2}U/dz^2$	$= -k$

# Microscopia a forza atomica (AFM): interazioni a corto raggio

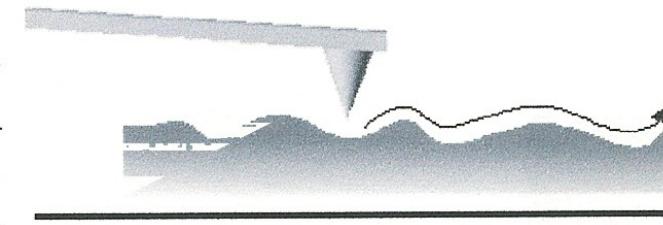
## Interazioni di van der Waals:



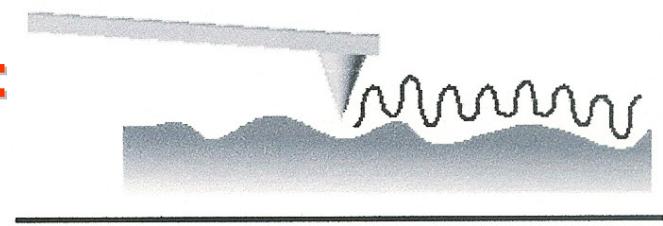


# Microscopia a forza atomica (AFM): le tecniche di scansione

- **Contact AFM (C-AFM):**  
modalità statica



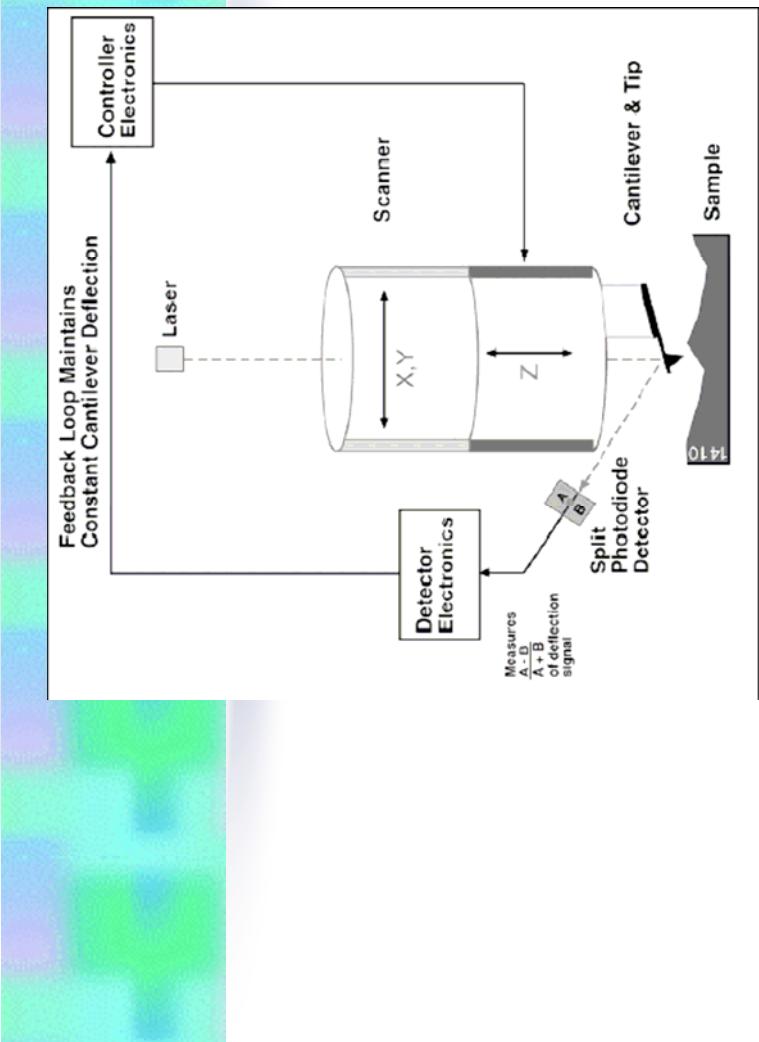
- **Non Contact AFM (NC-AFM):**  
modalità dinamica



- **Intermittent o Tapping AFM (IC-AFM)**

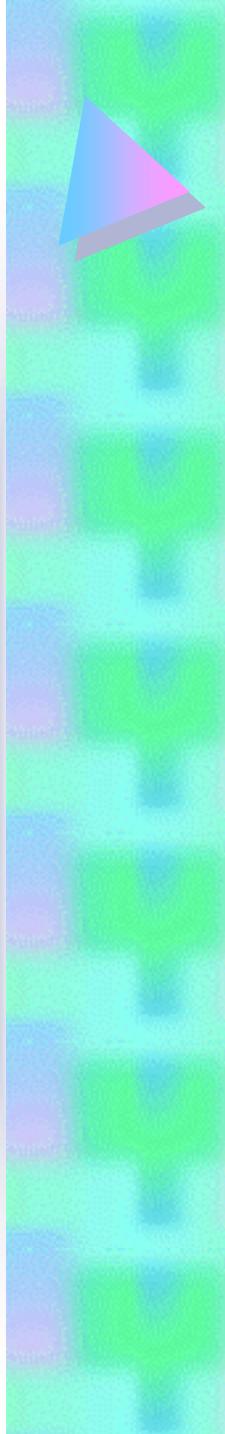


# Microscopia a forza atomica (AFM): contact AFM



## Contact Mode

- Operates by scanning a tip across the sample surface while monitoring the change in cantilever deflection.
- A feedback loop maintains a constant deflection between the cantilever and the sample by vertically moving the scanner at each (x,y) data point.
- Force constants usually range from 0.01 to 1.0 N/m, resulting in forces ranging from nN to  $\mu$ N.
- The distance the scanner moves vertically at each (x,y) data point is stored to form the topographic image of the sample surface.
- Operation can take place in ambient and liquid environments.



# Microscopia a forza atomica (AFM): contact AFM

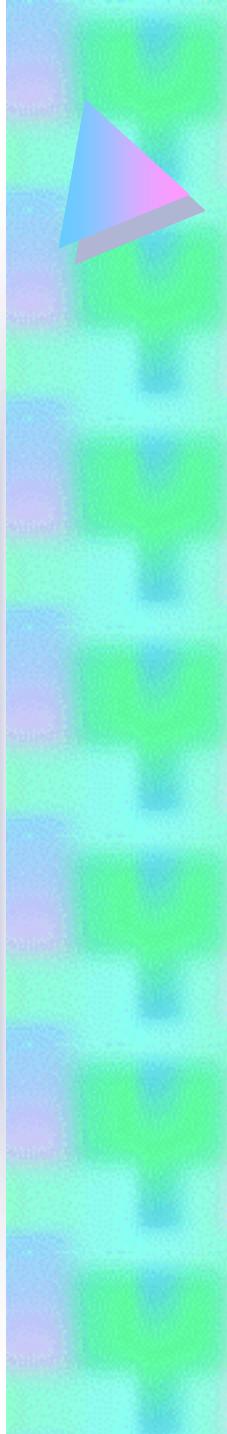
**Modalità repulsiva:** la punta è in contatto fisico ( $z \sim$  pochi Å) con la superficie del campione



Misura della deflessione del cantilever durante la scansione

**Forze coinvolte:**

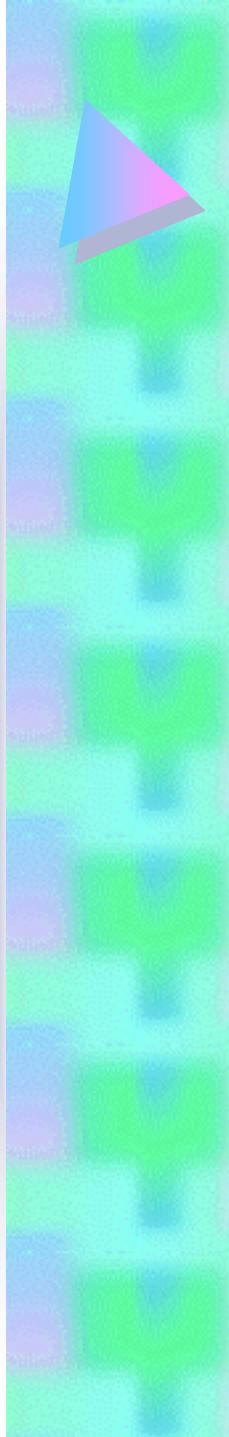
- Van der Waals
- Forze capillari
- Forza esercitata dal cantilever stesso



# Microscopia a forza atomica (AFM): contact AFM

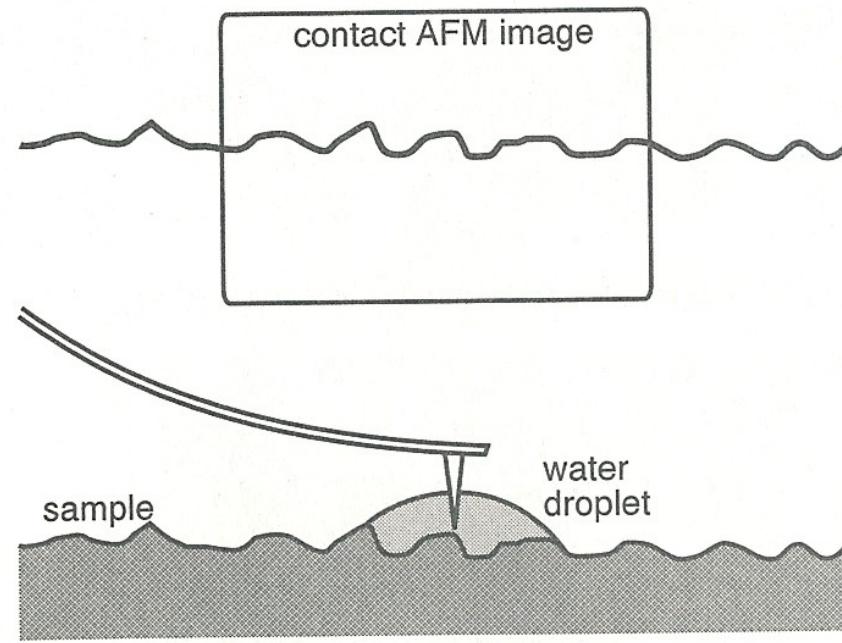
## Modi di operare:

- **Modalità a forza costante**
  - deflessione costante mediante feed-back (o circuito di retroazione)
  - mappatura del campione lungo l'asse z in funzione delle posizioni x-y
- **Modalità a deflessione variabile o altezza costante**
  - disattivazione del feed-back



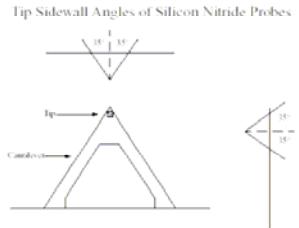
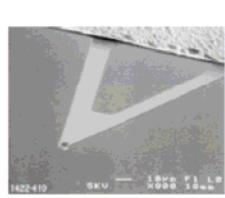
# Microscopia a forza atomica (AFM): contact AFM

- **Frequenze di risonanza: 7-30 kHz**
- **Cantilever: “soffici”  $k=0.01\text{-}0.1 \text{ N/m}$**
- **Forze:  $\sim 10^{-6}\text{N}$**



# Microscopia a forza atomica (AFM): contact AFM

## Contact Mode Tips / Cantilevers



Silicon Nitride Probe Characteristics

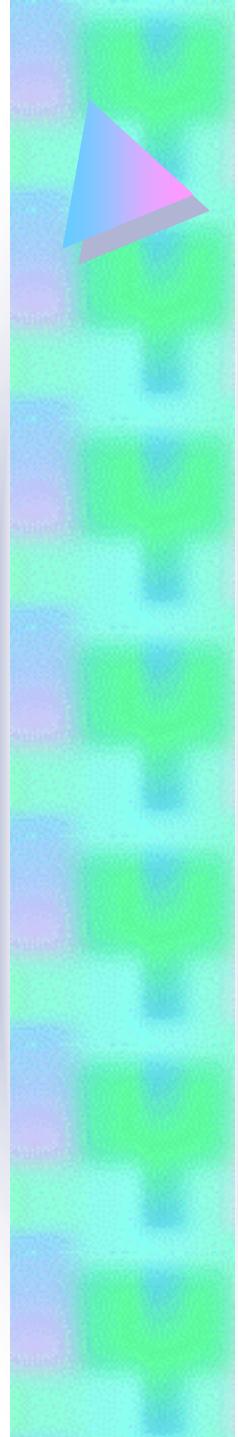
Spring Constant ( $k$ )	0.58, 0.32, 0.12, 0.06 N/m <sup>a</sup>
Nominal Tip Radius of Curvature	20 - 60 nm
Cantilever Lengths	100 & 200 $\mu$ m
Cantilever Configuration	V-shaped
Reflective Coating	Gold
Sidewall angles	35° on all 4 sides

a. Calculated spring constant values are based on the 0.6  $\mu$ m silicon nitride thickness; however, this value can actually vary from 0.4  $\mu$ m to 0.7  $\mu$ m. Thickness is cubed in the spring constant calculation, thus, actual values can vary substantially.

- **Silicon nitride probes consist of a cantilever integrated with a sharp tip on the end.**

- **It is necessary to have a cantilever which is soft enough to be deflected by very small forces and has a high enough resonant frequency to not be susceptible to vibrational instabilities.**

- **This is accomplished by making the cantilever short, to provide a high resonant frequency, and thin, to provide a small force constant.**



## Contact AFM: estensioni

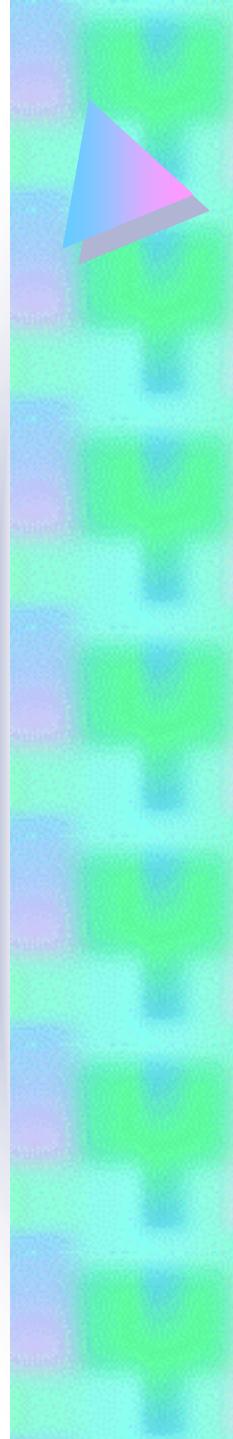
- Lateral Force Microscopy (LFM)
- Force Modulation Microscopy (FMM)
- Curve F vs d
- Nanoindentation
- Chemical Force Microscopy



# Contact AFM: LFM

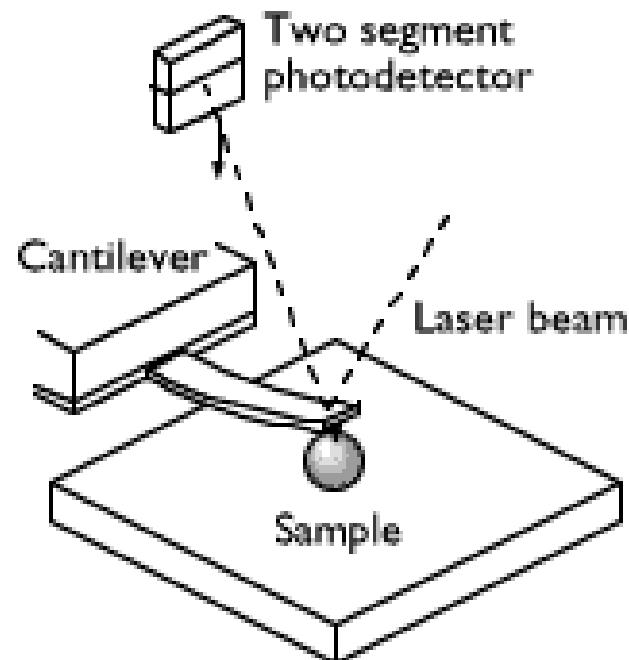
## **Forze laterali o di attrito**

- Misura delle deflessioni laterali del cantilever
- Alta risoluzione laterale
- Individuazione della variazione del coefficiente di attrito lungo la superficie (individuazione di fasi diverse)

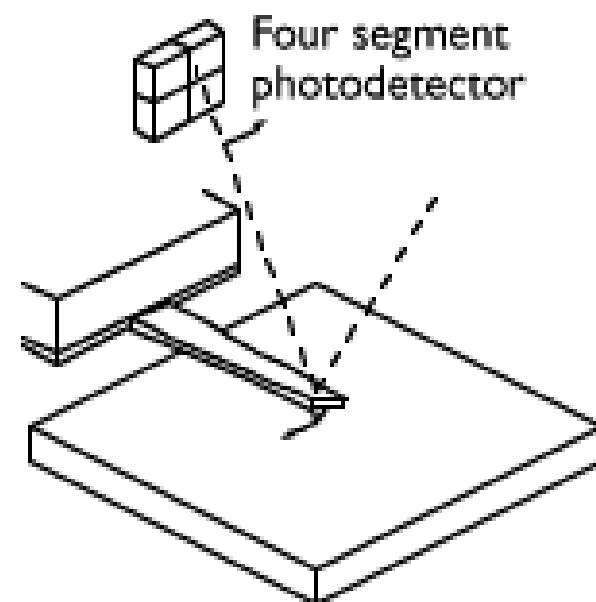


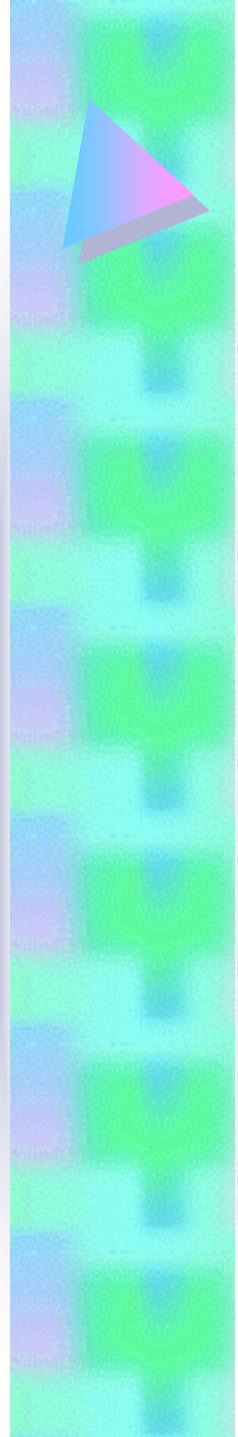
# Contact AFM: LFM

**Atomic force microscopy**



**Frictional force microscopy**





# Contact AFM: LFM

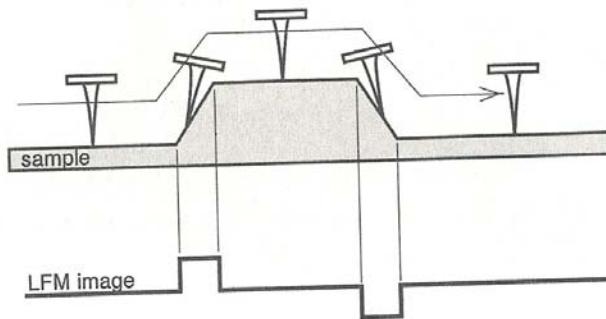
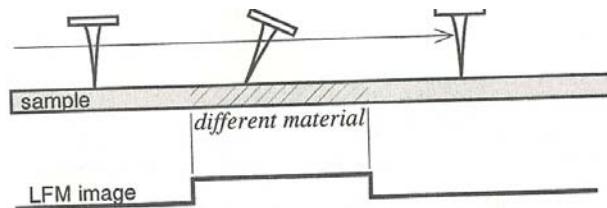
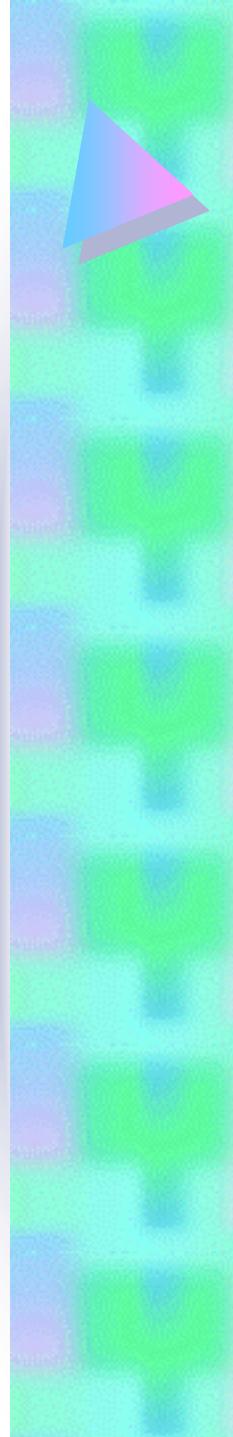


Figure 1-9. Lateral deflection of the cantilever  
from changes in surface friction (top) and from changes in slope (bottom).



# Contact AFM: LFM

## Lateral Force Microscopy (Friction)

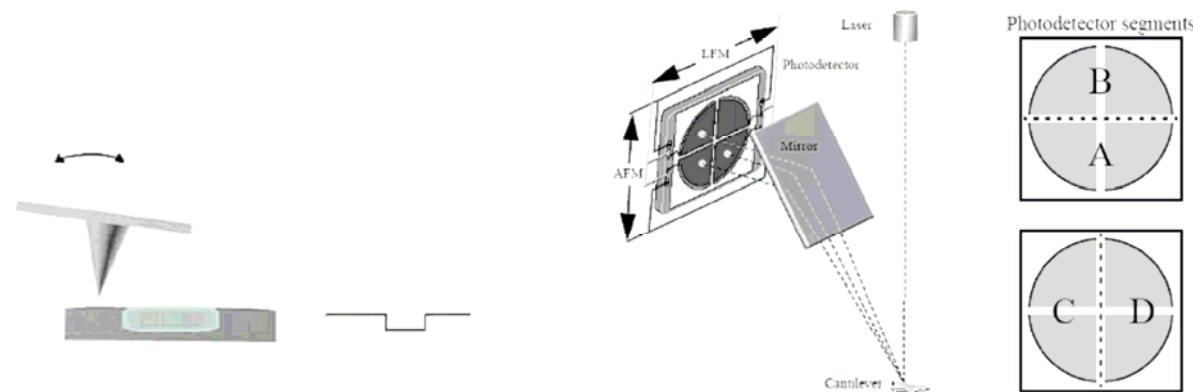
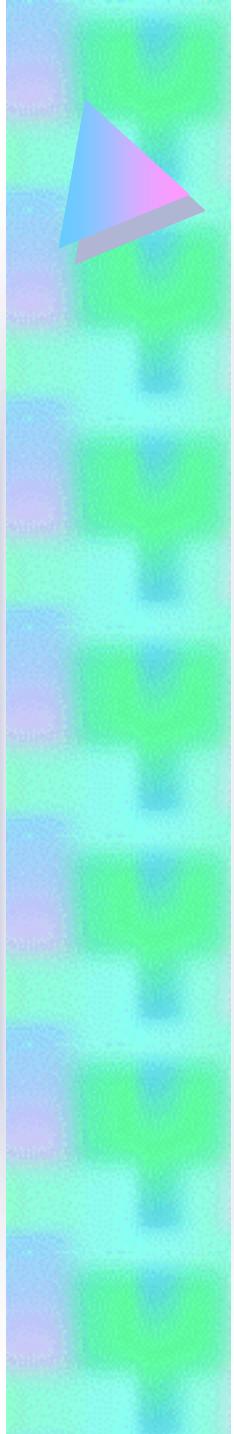


Figure 2.4. Quad photodetector arrangement. Different segments of the photodetector are used for generating AFM and LFM signals.

- The amount of torsion of the cantilever is controlled by changes in topography as well as changes in surface chemical properties.
- Possible to detect / image changes in material properties.



# Contact AFM: FMM

## Modulazione delle forze

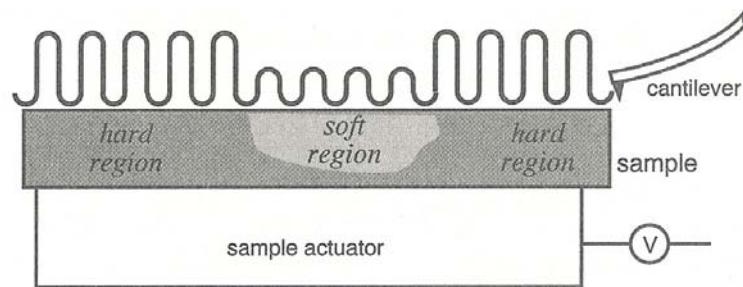


Figure 1-11. The amplitude of cantilever oscillation varies according to the mechanical properties of the sample surface.

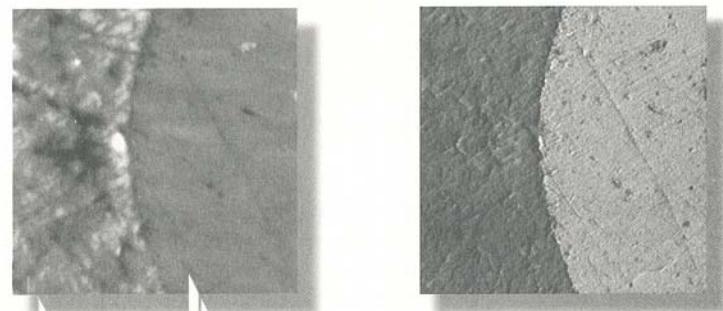
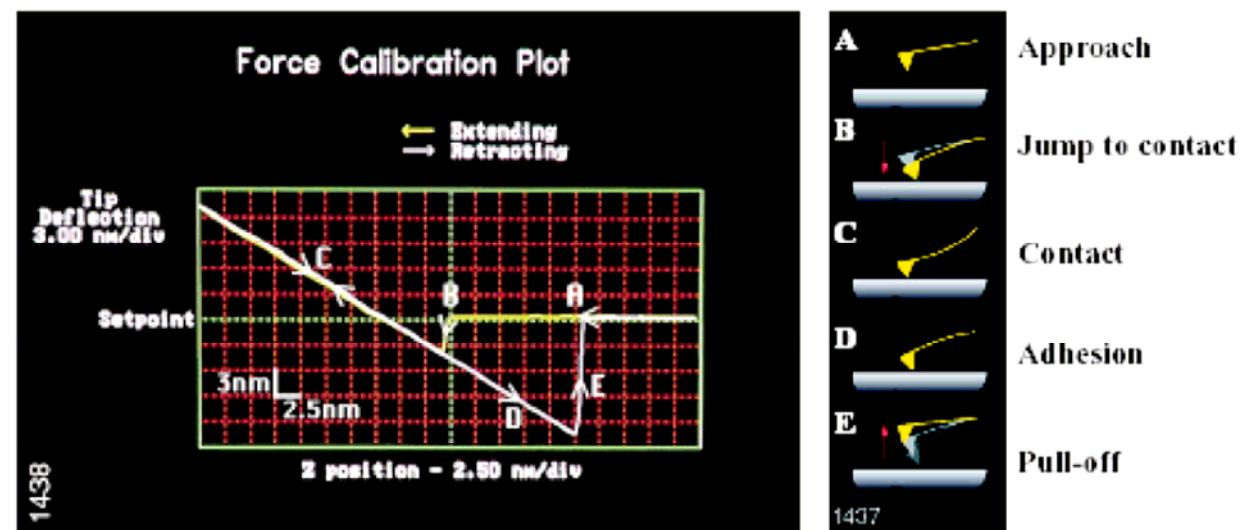


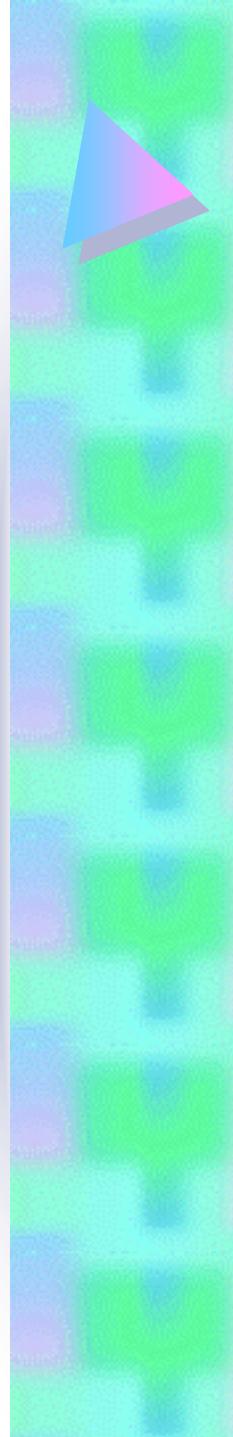
Figure 1-12. Contact-AFM (left) and FMM (right) images of a carbon fiber/polymer composite collected simultaneously.  
Field of view 5 $\mu$ m.

# Contact AFM: F vs d

## Force Curves



Force curves are commonly used to set the imaging force in contact mode and to study attractive, repulsive, and adhesive interactions between the tip and the sample.



# Contact AFM: F vs d

## Curve forza vs distanza

- proprietà elastiche della superficie
- viscosità dei contaminanti della superficie
- spessore dei lubrificanti

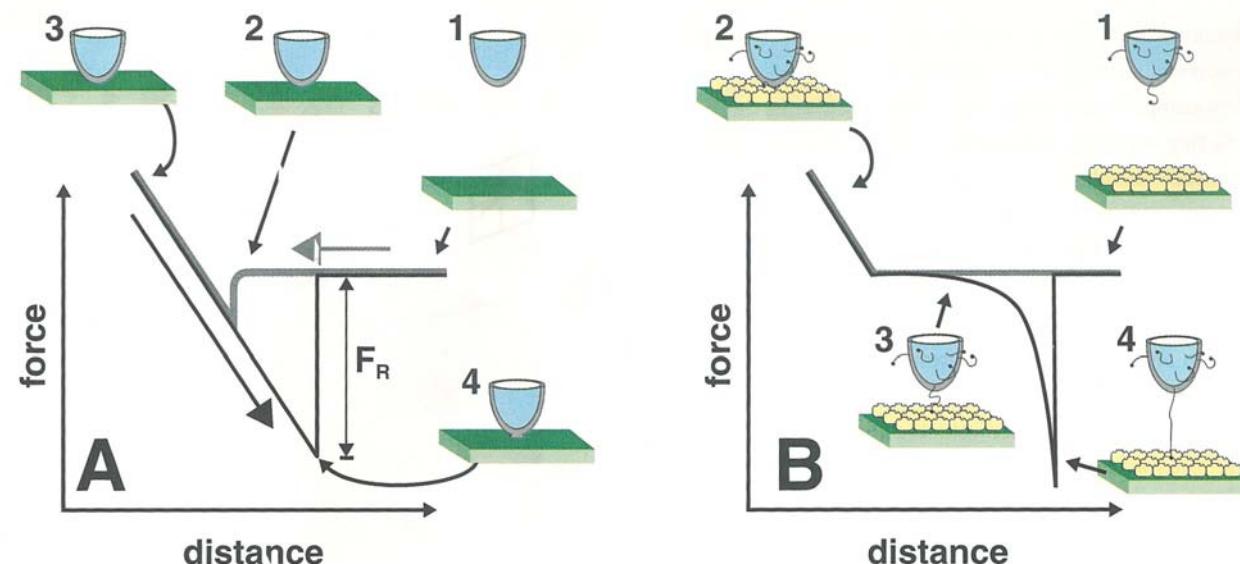
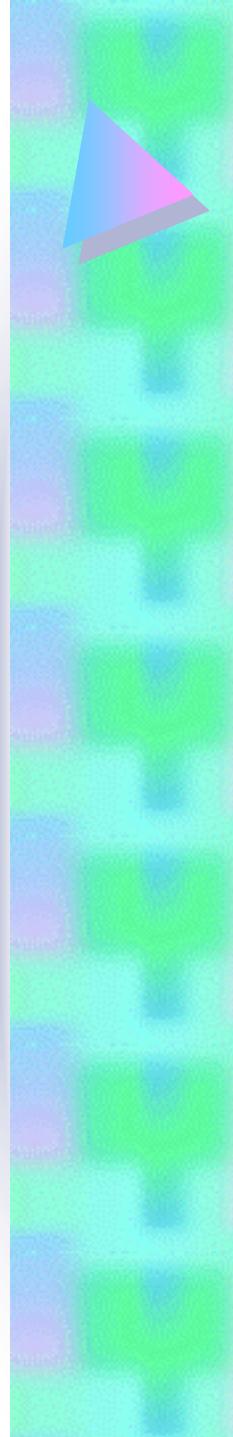
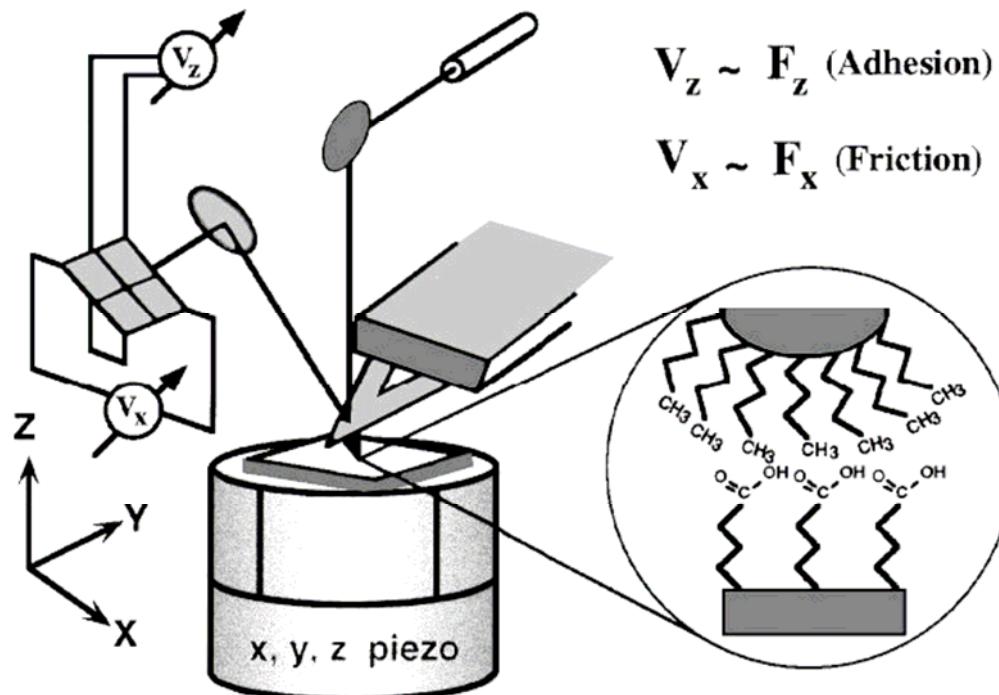


Fig. 2 Force curves. (a) Contact mechanics: a simulated force-distance curve where the rupture force  $F_R$  is measured when the tip and sample rupture. The tip and sample are far from each other (1), as the sample is moved toward the tip they begin to interact (2), and then they are put into repulsive contact (3), when the scan direction is reversed, eventually the cantilever restoring force is balanced by the adhesive force (4), and the tip-sample contact is ruptured. (b) Single molecule: a simulated force-distance curve for a single molecule pulling experiment where  $F_R$  is measured when the tip and sample are distant from each other.

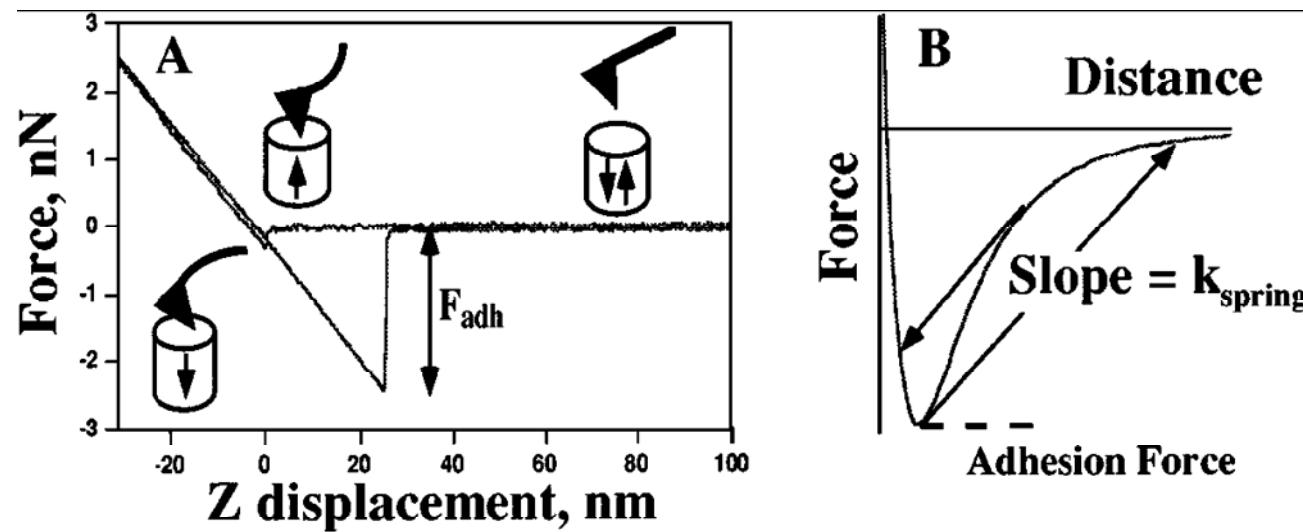


# Contact AFM: CFM

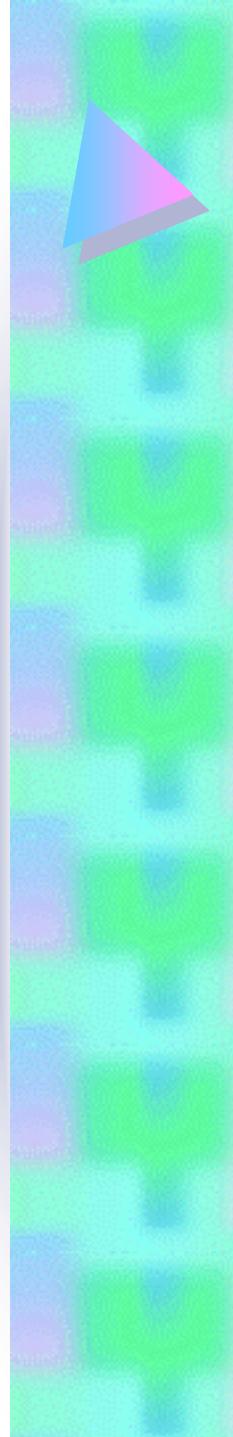


*Figure 1* Schematic drawing of the CFM setup. The sample rests on a piezoelectric x, y, z translator. A laser beam is reflected from the backside of the tip onto a photodiode to measure two types of tip-surface interactions. When the sample approaches, touches, and is withdrawn from the tip, the tip will move up and down in response to surface normal forces  $F_z$ , resulting in the vertical deflection signal  $V_z$ . The cantilever will also twist in response to friction forces  $F_x$ , yielding the lateral deflection signal  $V_x$ . The inset illustrates the chemically specific interactions between a Au-coated,  $\text{CH}_3$ -terminated tip contacting a  $\text{COOH}$ -terminated region of a sample.

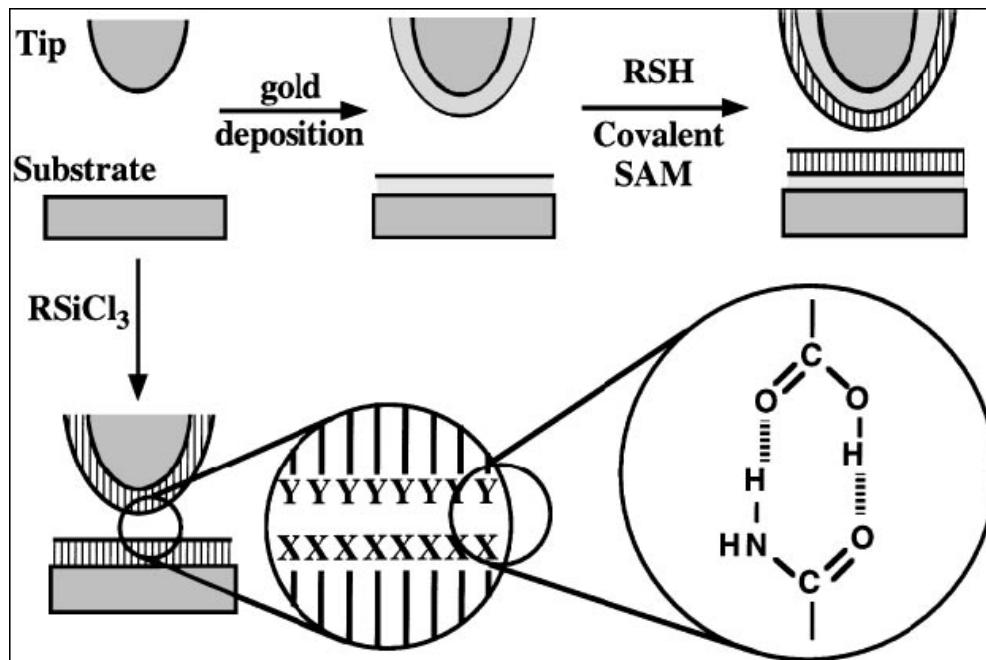
# Contact AFM: CFM



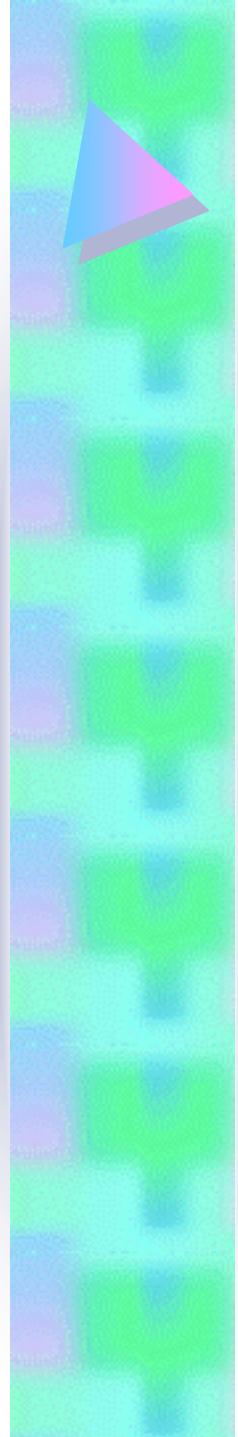
*Figure 2* (A) Typical force-vs-sample z displacement curve. At large separations, no force is observed between tip and sample. At short distances, the van der Waals attraction will pull the tip abruptly into contact with the sample (jump into contact point). After that, the deflection of the soft cantilever tracks the movement of the sample (linear compliance regime). Hysteresis in the force between tip and sample is observed when the tip is withdrawn from the sample. The finite force necessary to pull the tip off the sample surface corresponds to the adhesive force between functional groups on the surfaces of the tip and sample. (B) The cycle in (A) is shown in terms of a schematic intermolecular potential between the tip-sample functional groups. Whenever the force gradient exceeds the cantilever spring constant, the system becomes mechanically unstable and cantilever jumps occur.



# Contact AFM: CFM

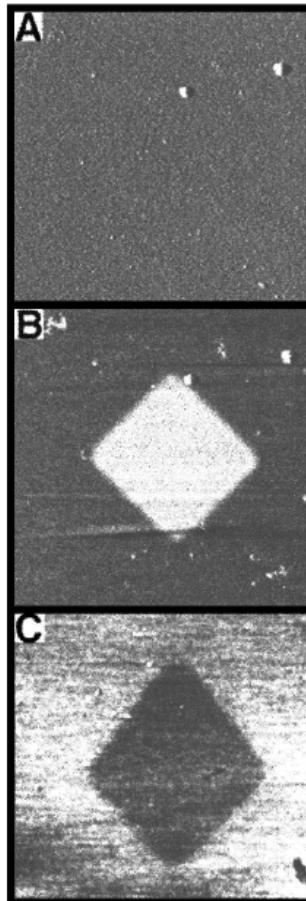


*Figure 3* Scheme for chemical modification of tips and sample substrates. Tips and substrates are first coated with a thin layer of Au (50–100 nm) and then, upon immersion in a solution of organic thiol, a dense SAM is formed on the Au surface. Similarly, cleaned Si or  $\text{Si}_3\text{N}_4$  tips can be derivatized with reactive silanes. The functional groups comprise the outermost surface of the crystalline SAM, and the tip-sample interaction can be fine-tuned by varying the chemistry at the free SAM surfaces. The R in  $\text{RSH}$  and  $\text{RSiCl}_3$  represents an organic alkyl chain that ends with a functional group X (X D  $\text{CH}_3$ ,  $\text{COOH}$ ,  $\text{CH}_2\text{OH}$ ,  $\text{NH}_2$ , etc).

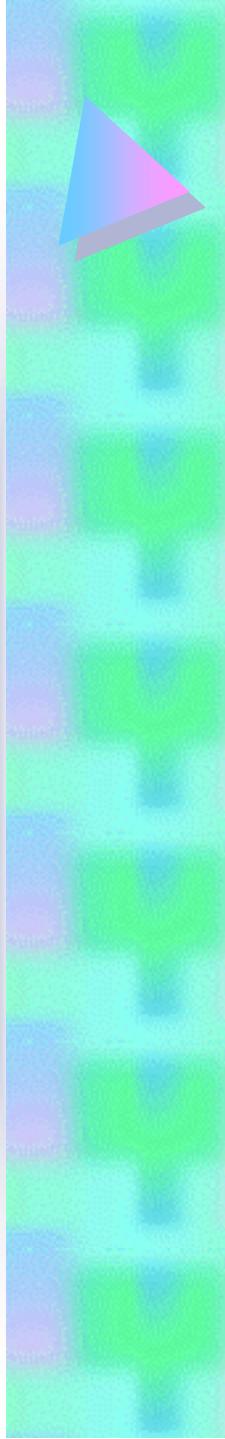


# Contact AFM: CFM

CHEMICAL FORCE MICROSCOPY 411

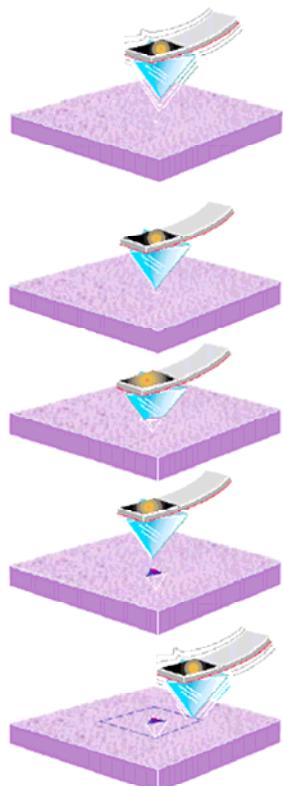


*Figure 16* Force microscopy images of a photopatterned SAM sample. The  $10 \times 10 \mu\text{m}$  square region terminates in COOH, and the surrounding region terminates in CH<sub>3</sub>. The images are of (A) topography, (B) friction force using a tip modified with a COOH-terminated SAM, and (C) friction force using a tip modified with a CH<sub>3</sub>-terminated SAM. Light regions in (B) and (C) indicate high friction; dark regions indicate low friction (reproduced from Reference 33).

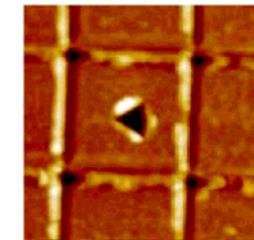


# Contact AFM: nanoindentation

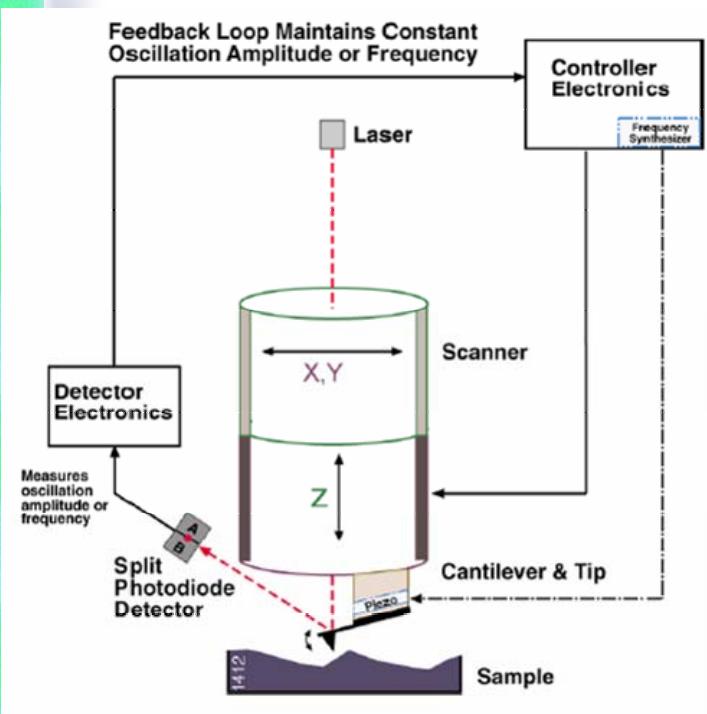
## Nanoindentation Techniques



- Image surface in tapping mode
- Stop scan and force tip into surface
- Withdraw tip and image indentation
  
- Collect force plot during indentation
- force range available 1-100  $\mu\text{N}$
- resolution better than 0.5  $\mu\text{N}$
  
- Measurements of material hardness / wear characteristics

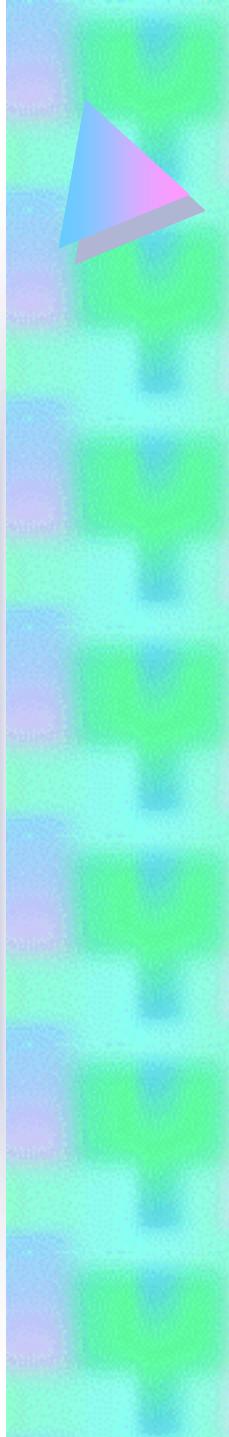


# Microscopia a forza atomica (AFM): non-contact AFM



## Non-Contact Mode

- The cantilever is oscillated slightly above the cantilever's resonance frequency (amplitude of ~10 nm).
- The tip does not contact the sample surface, but oscillates above the adsorbed surface fluid layer during scanning.
- The cantilever's resonant frequency is decreased by the van der Waals forces, which extend from 1 nm to 10nm above the adsorbed fluid layer, and by other long range forces.
- The feedback loop maintains a constant oscillation amplitude or frequency by vertically moving the scanner at each (x,y) data point.

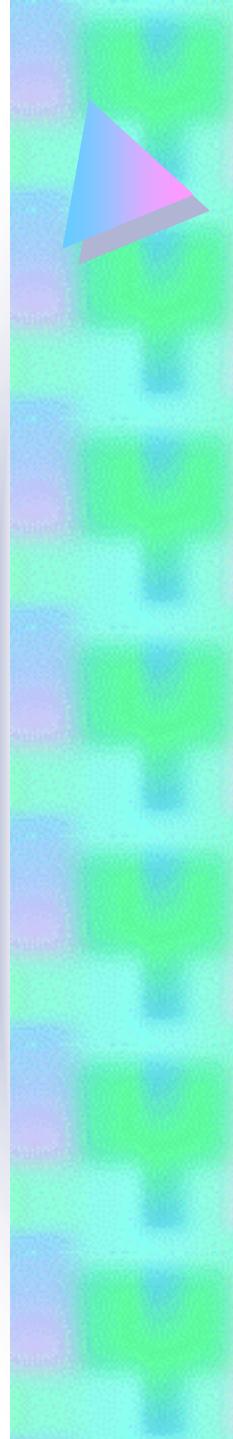


# Microscopia a forza atomica (AFM): non-contact AFM

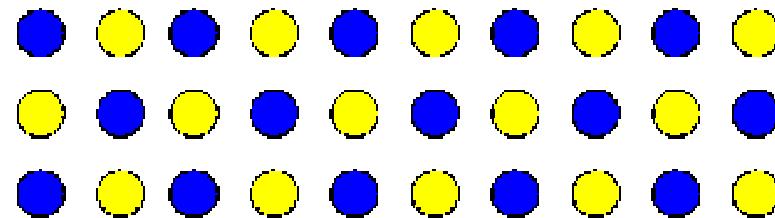
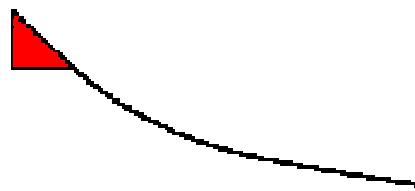
**Modalità attrattiva:** la punta oscilla intorno alla sua frequenza di risonanza (f) con un'ampiezza variabile fra le decine e centinaia di Å

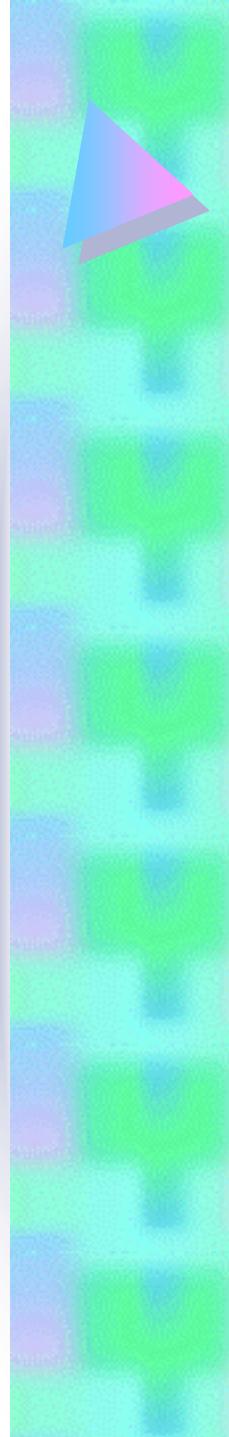


**Misura delle variazioni di f e Ampl in funzione della distanza punta-campione (topografia) durante la scansione**



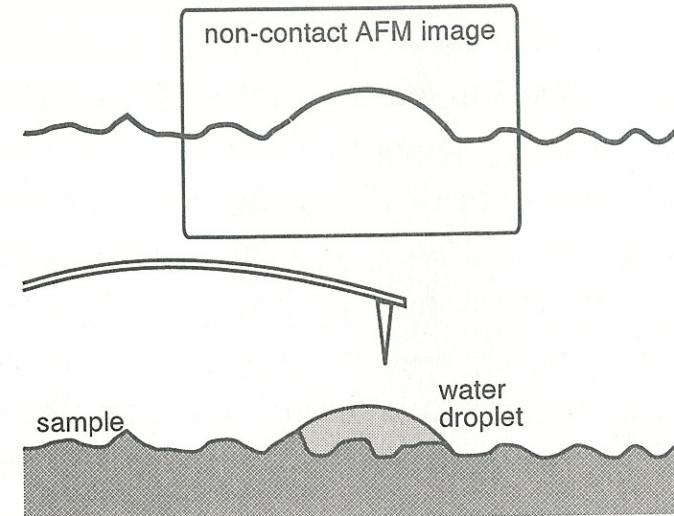
# Microscopia a forza atomica (AFM): non-contact AFM



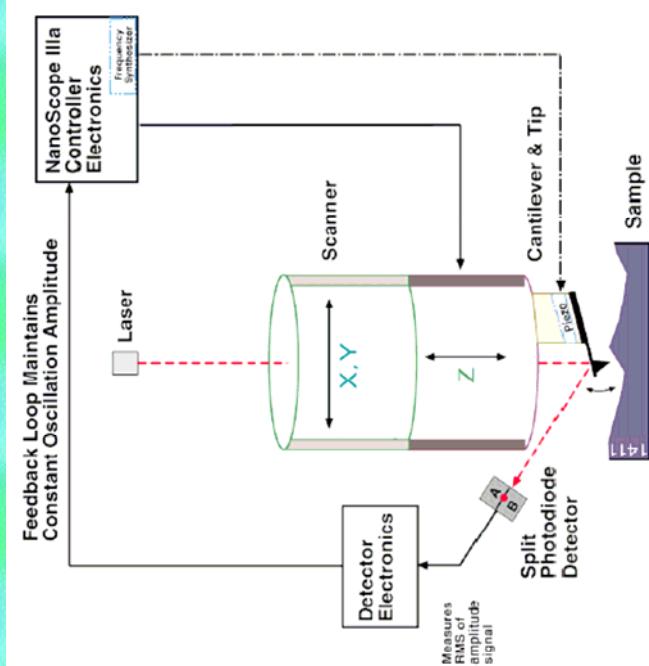
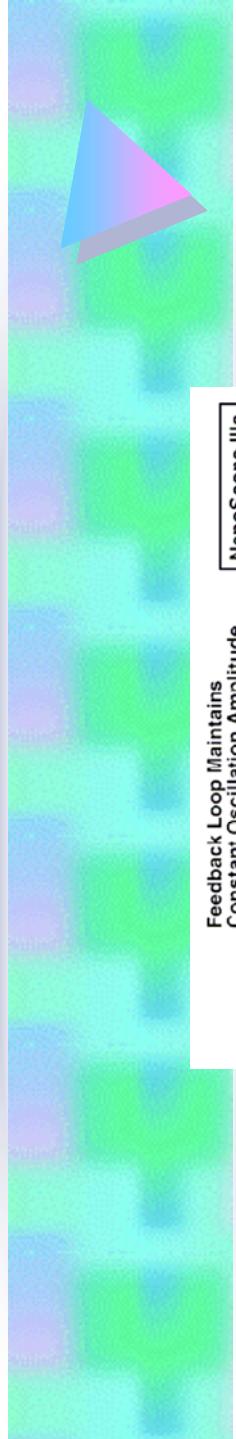


# Microscopia a forza atomica (AFM): non-contact AFM

- **Frequenze di risonanza: 100-400 kHz**
  - **Cantilever: molto rigidi  $k=5-50 \text{ N/m}$**
  - **Forze:  $\sim 10^{-12} \text{ N}$**
- 
- Ideale per campioni soffici
  - Nessuna degradazione della superficie
  - Nessuna contaminazione da punta

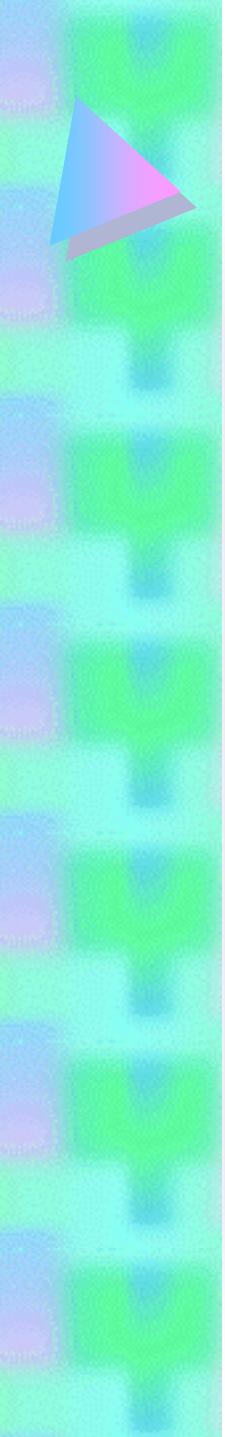


# Microscopia a forza atomica (AFM): IC-AFM



## Tapping Mode

- Tapping Mode AFM operates by scanning a tip on an oscillating cantilever across the sample surface.
- The cantilever is oscillated at or near its resonance frequency (amplitude typically from 20 to 100 nm).
- The tip lightly “taps” on the sample surface during scanning.
- The feedback loop maintains a constant oscillation amplitude by maintaining a constant RMS of the oscillation signal acquired by the split photodiode detector.
- Operation can take place in ambient and liquid environments.

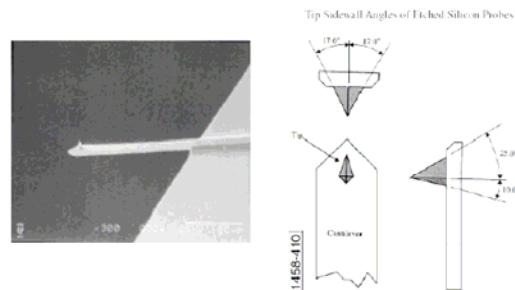


# **Microscopia a forza atomica (AFM): IC-AFM**

- Modalità complementare fra C-AFM e NC-AFM**
- La punta oscilla entrando anche in contatto con la superficie del campione**
- Superamento dei limiti di C-AFM e NC-AFM**

# Microscopia a forza atomica (AFM): IC-AFM

## Tapping Mode Tips / Cantilevers



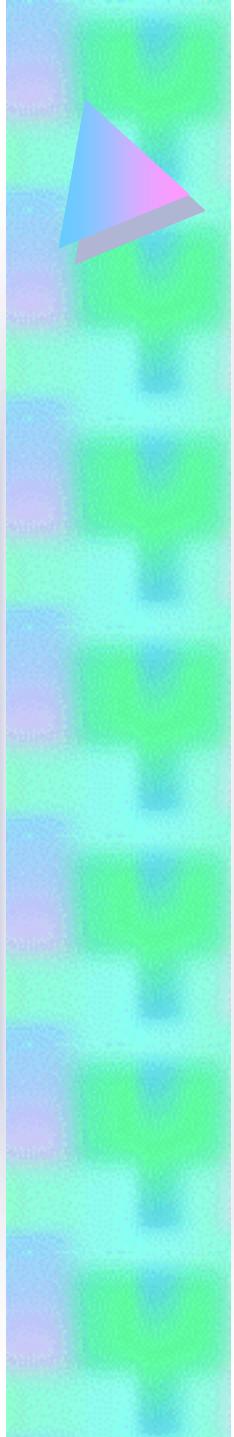
TappingMode Etched Silicon Probe (TESP) Characteristics

Spring Constant (k)	20 - 100 N/m
Resonant Frequency	200 - 400 kHz
Nominal Tip Radius of Curvature	5 - 10 nm
Cantilever Length	125 $\mu$ m
Cantilever Configuration	Single Beam
Reflective Coating	Uncoated, Optional Al Coating

- Silicon probes are used primarily for Tapping Mode applications.

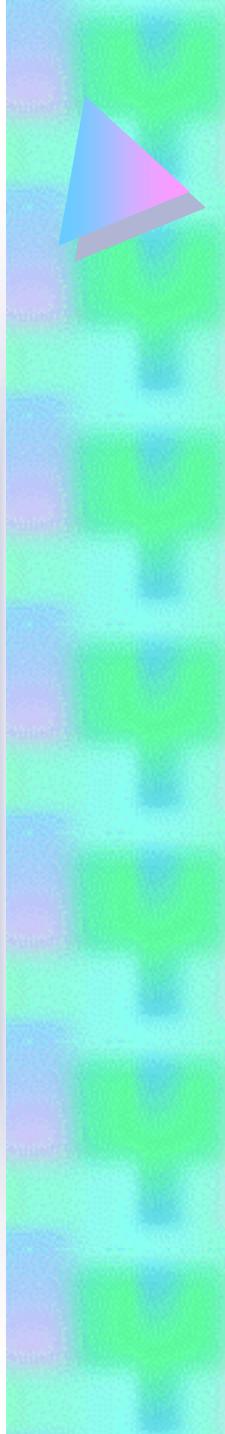
- The tip and cantilever are an integrated assembly of single crystal silicon, produced by etching techniques.

- These probes can be much stiffer than the silicon nitride probes, resulting in larger force constants and resonant frequencies.



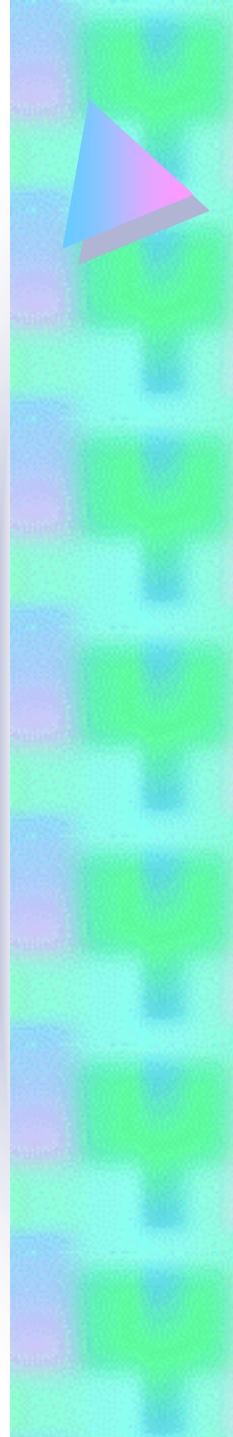
## AFM Imaging Mode Comparison

	Contact Mode	Tapping Mode	Non-Contact Mode
Advantages	<ul style="list-style-type: none"><li>• High scan speeds (throughput)</li><li>• Rough samples with extreme changes in vertical topography can sometimes be scanned more easily in contact mode.</li></ul>	<ul style="list-style-type: none"><li>• Higher lateral resolution on most samples</li><li>• Lower forces and less damage to soft samples</li><li>• Lateral forces are virtually eliminated, so there is no scraping.</li></ul>	<ul style="list-style-type: none"><li>• No force exerted on the sample surface.</li></ul>
Disadvantages	<ul style="list-style-type: none"><li>• Lateral (shear) forces can distort features in the image.</li><li>• The combination of lateral forces and high normal forces can result in reduced spatial resolution and may damage soft samples due to scraping between the tip and sample.</li></ul>	<ul style="list-style-type: none"><li>• Slightly slower scan speed than contact mode AFM.</li></ul>	<ul style="list-style-type: none"><li>• Lower lateral resolution, limited by the tip-sample separation</li><li>• Slower scan speed than Tapping Mode and Contact Mode</li><li>• Non-contact usually only works on extremely hydrophobic samples, where the adsorbed fluid layer is at a minimum.</li></ul>



# **Microscopia a forza atomica NC-(AFM): estensioni**

- **Magnetic Force Microscopy (MFM)**
- **Phase Detection Microscopy (PDM)**
- **Electrostatic Force Microscopy (EFM)**
- **Scanning Capacitance Microscopy (SCM)**
- **Thermal Scanning Microscopy (TSM)**
- **Near-field Scanning Optical Microscopy (NSOM)**
- **Nanolitografia**



# Non Contact AFM: MFM

## Topografia e proprietà magnetiche

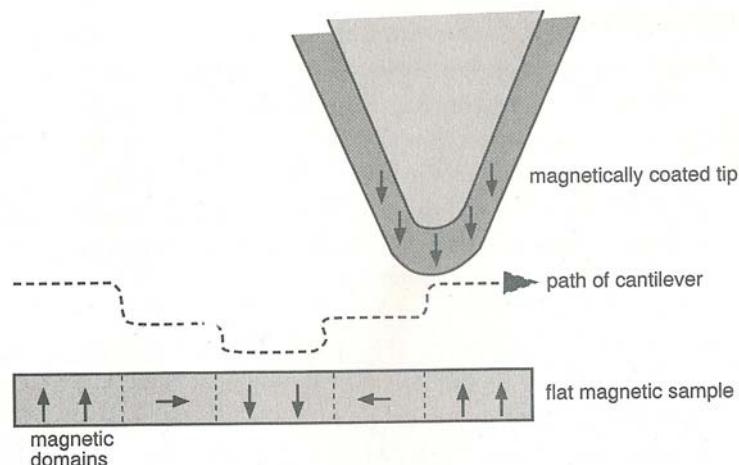


Figure 1-7. MFM maps the magnetic domains of the sample surface

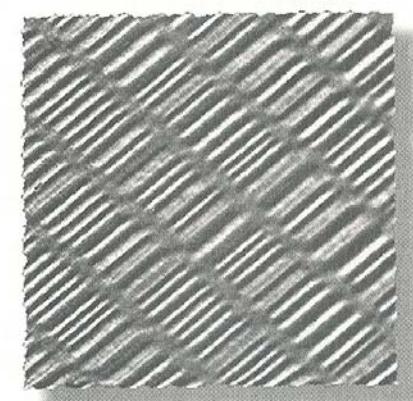
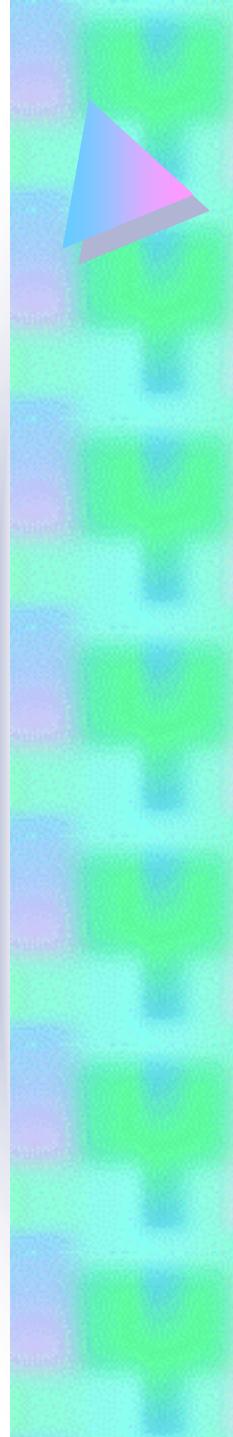
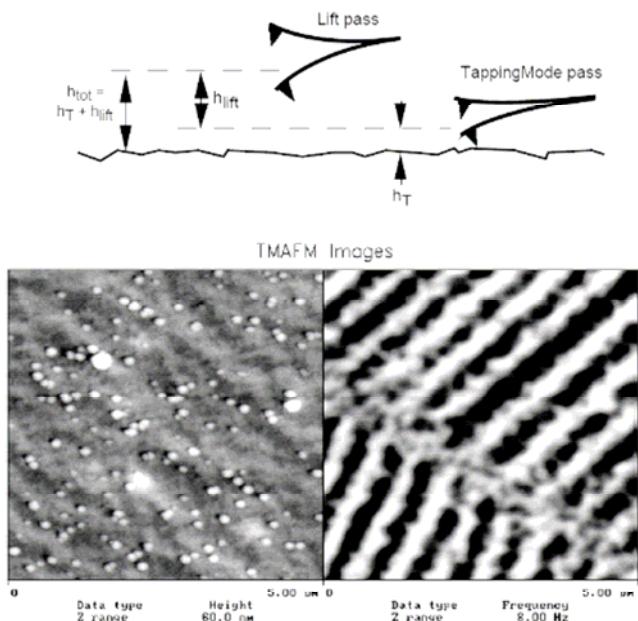


Figure 1-8. MFM image showing the bits of a hard disk.  
Field of view 30 μm.



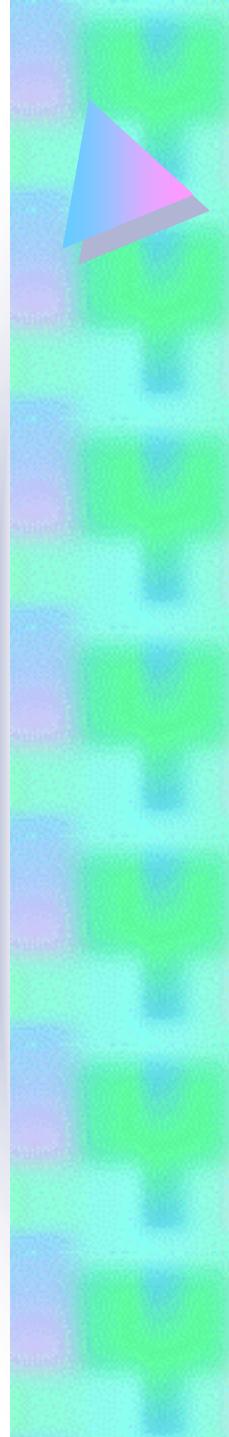
# Non Contact AFM: MFM

## Magnetic Force Microscopy



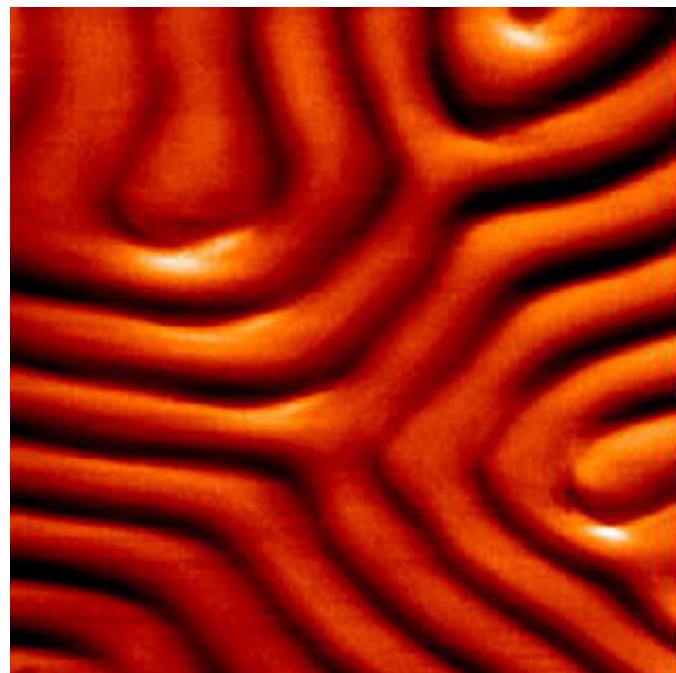
- Uses a tip coated with a magnetic material.
- Scan 1 → Topography
- Scan 2 → Lift + Measure Mag.
- Magnetic features are often hidden in the topographic image

Figure 229-7 Topographic (left) and Magnetic Force Gradient Image (right) of Metal Evaporated Tape at 100 nm Lift Height



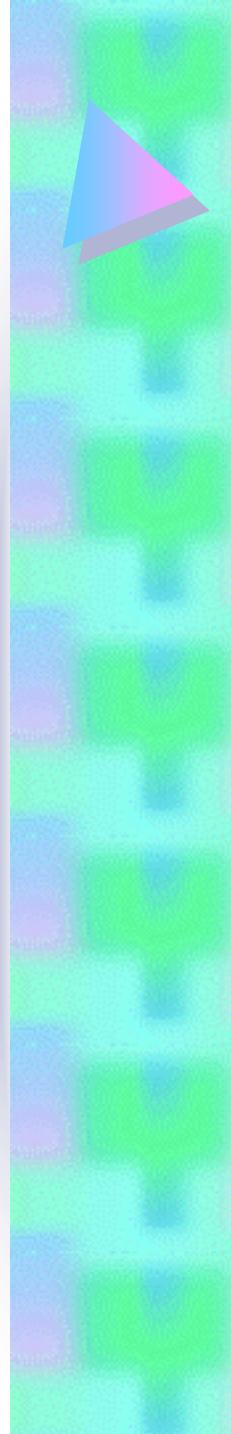
# Non Contact AFM: MFM

Imaging of ferromagnetic surfaces



Magnetic domain walls on  
 $\text{BaFe}_{12}\text{O}_{19}$ , measured with  
iron coated AFM tip.

A. Wadas et al., University of  
Hamburg, Germany



# Non Contact AFM: PDM

## Topografia e proprietà di superficie come adesione, elasticità frizione

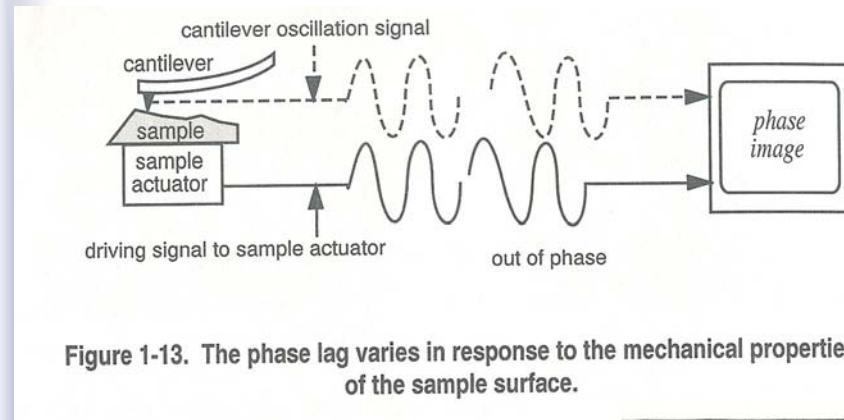


Figure 1-13. The phase lag varies in response to the mechanical properties of the sample surface.

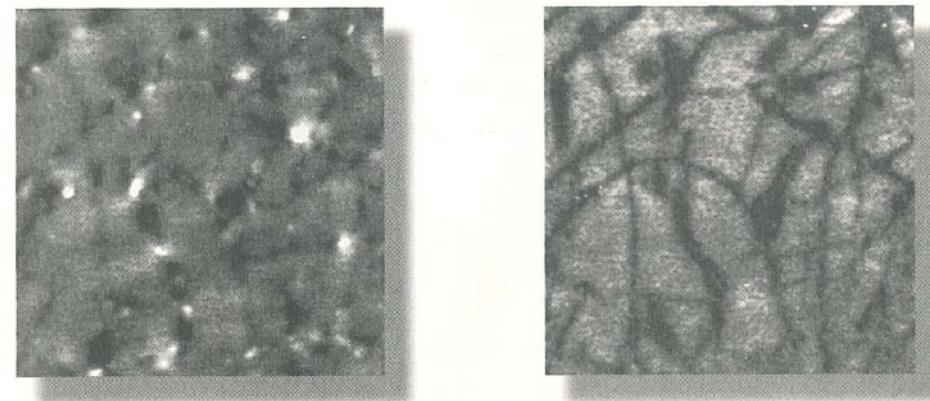
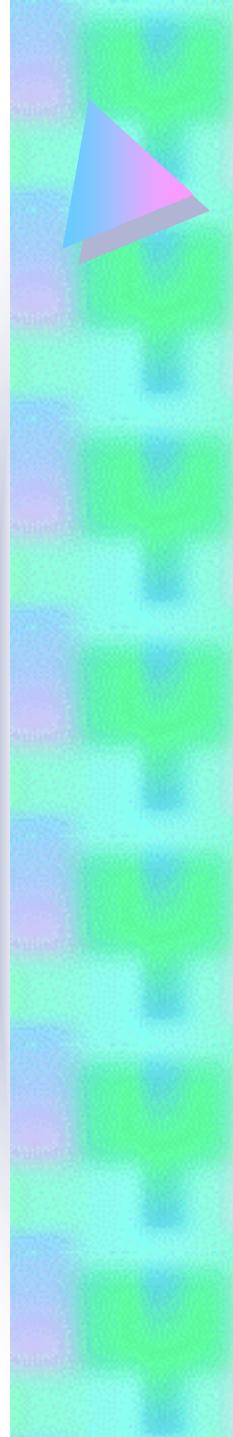


Figure 1-14. Non-contact AFM image (left) and PDM image (right) of an adhesive label, collected simultaneously. Field of view 3 $\mu$ m.



# Non Contact AFM: EFM

## Topografia e variazione della densità di carica lungo la superficie

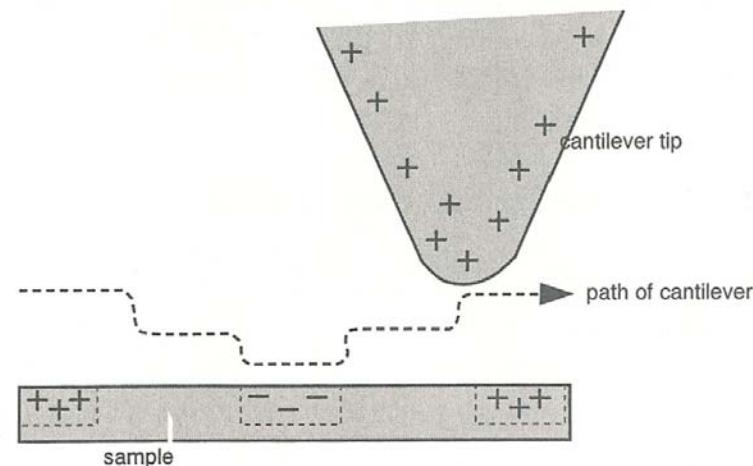
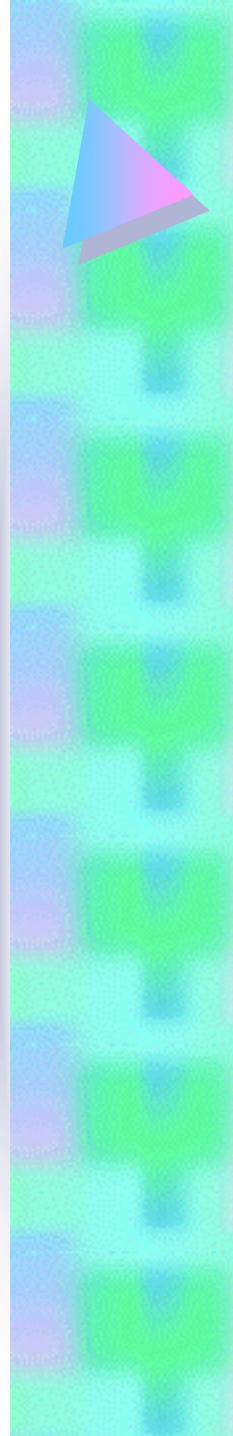


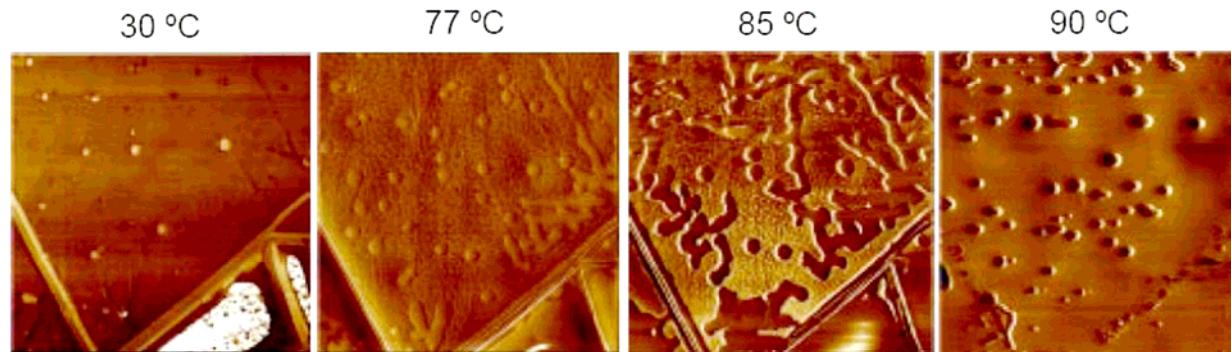
Figure 1-15. EFM maps locally charged domains on the sample surface.



# Non Contact AFM: TSM

## Studies of Thermal Phase Transitions

*Phase images of poly(hexacyclodimethyl)siloxane*

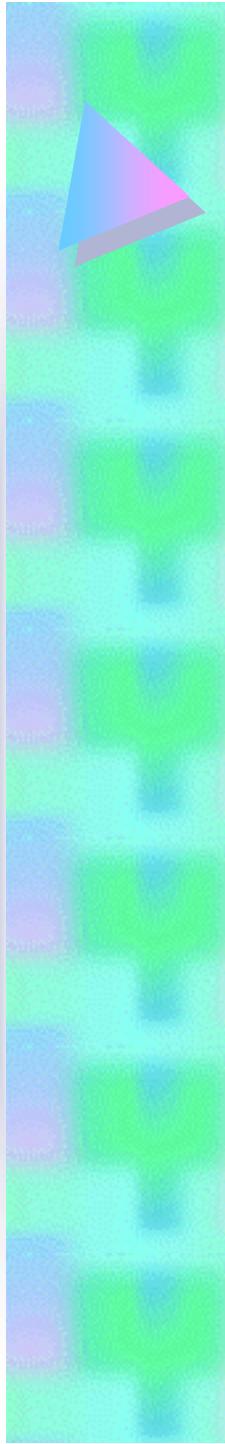


30 °C → Mixture of amorphous and mesophase (straight lamellea) structures

77 – 85 °C → Heating induces formation of liquid islands

90 °C → Melting complete – arrays of dots

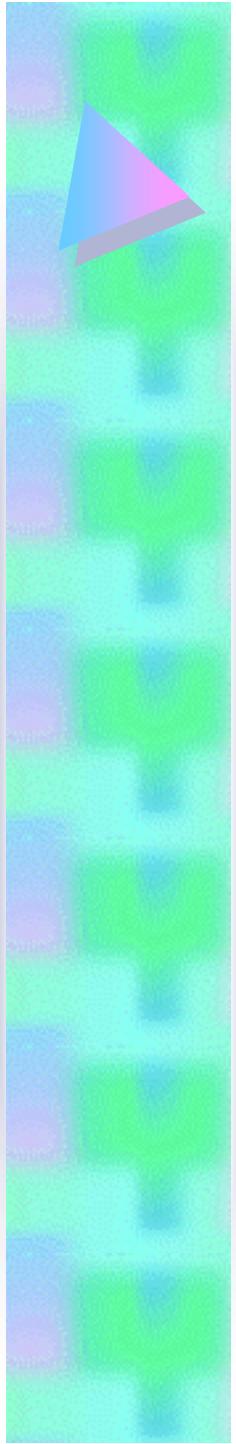
-50 to ~300 °C on Sample Stage



# Microscopia a forza atomica: analisi dei dati

## Immagine

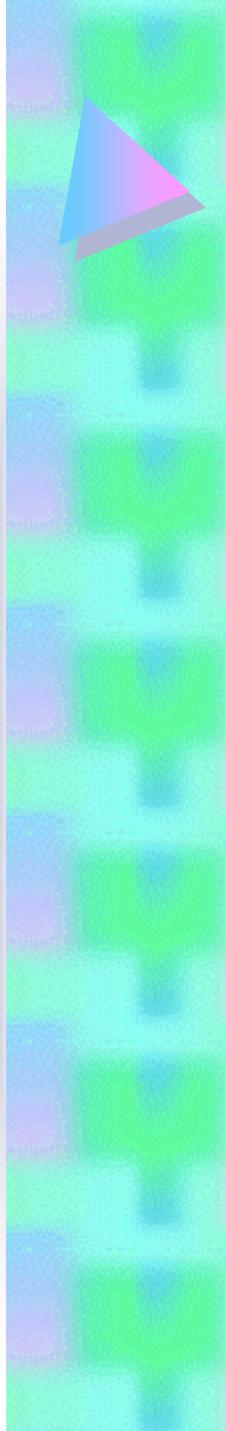
- matrice quadrata di numeri in cui ogni posizione nella matrice corrisponde alle coordinate x, y e il numero rappresenta l'intensità del segnale in quella posizione.
- Matrici tipiche:  $128 \times 128$ ,  $256 \times 256$ ,  $1024 \times 1024$
- Densità di informazioni laterali: scan size/n.ro di pixel  
(es. matrice  $124 \times 124$  di  $10\mu\text{m} \times 10 \mu\text{m}$ : risoluzione di 100 nm)
- Densità di informazioni verticali: scanner da  $5 \mu\text{m}$   
risoluzione di  $0.75\text{\AA}$



# Microscopia a forza atomica: analisi dei dati

## Routine:

- Compensazione dei difetti strumentali
- Quantificare le informazioni di superficie



# Microscopia a forza atomica: analisi dei dati

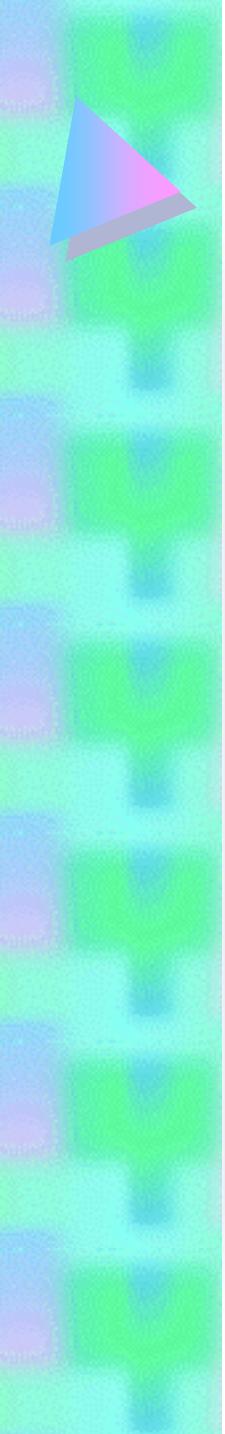
## **Compensazione dei difetti strumentali**

- Scelta di punta adeguata
- Non-planarità dell'immagine

☞ **Sottrazioni o flattening**

Salti della punta e rumori ad alte frequenze

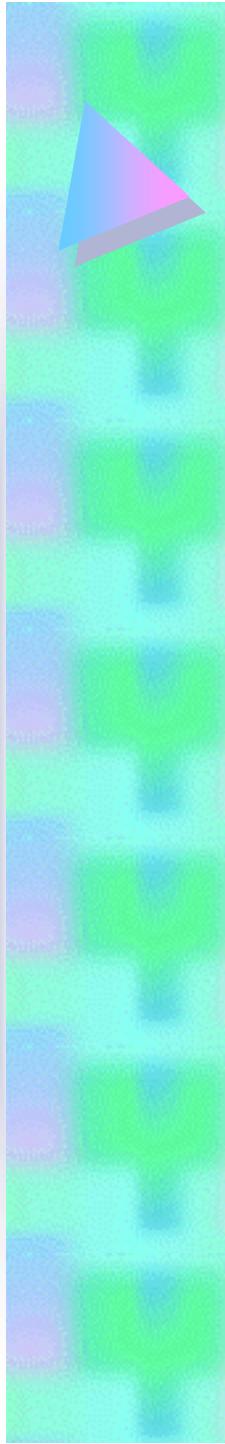
☞ **Uso di filtri e di trasformate di Fourier 2D**



# Microscopia a forza atomica: analisi dei dati

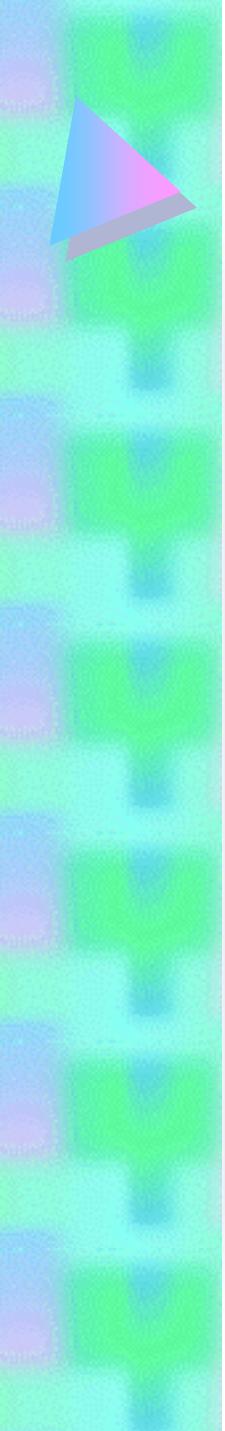
## Quantificare le informazioni morfologiche di superficie

- Rugosità superficiale: diverse definizioni
  1. Media delle variazioni dell'altezza picco-picco, scarto quadratico medio o auto covarianza
  2. Dipendenza dalla scala e non può essere rappresentato come un numero assoluto



# Microscopia a forza atomica: analisi dei dati

- Analisi dei grani
- Analisi della porosità superficiale
- Meccanismi di crescita (diffusione, desorbimento, shadowing)
- Dimensioni frattali



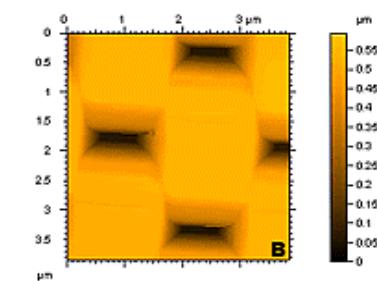
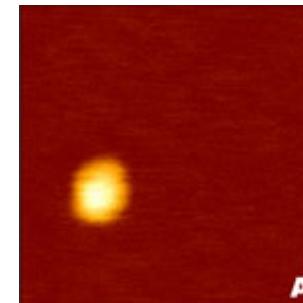
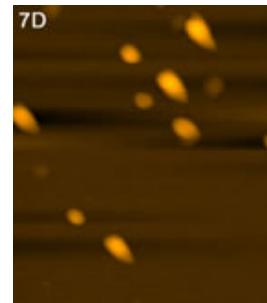
# Microscopia a forza atomica: analisi dei grani

quartarone.EXE

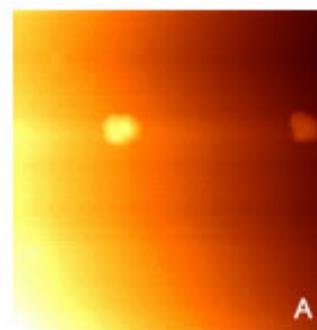
# Microscopia a forza atomica: analisi dei dati

## Artifatti dell'immagine

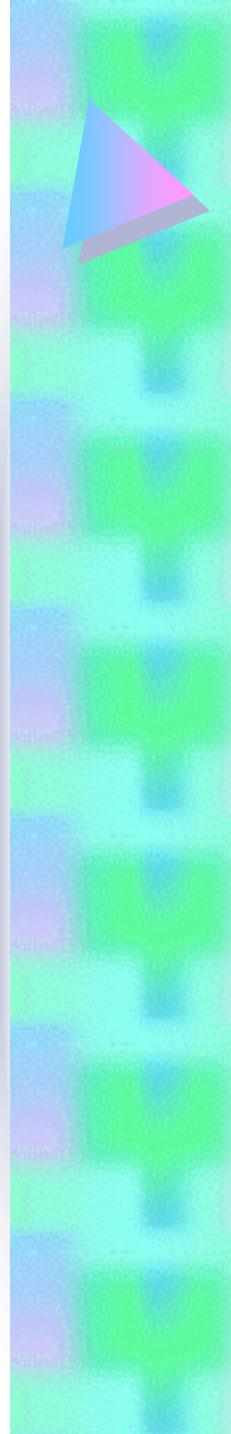
- Punta



- scanner



- Rumore elettronico

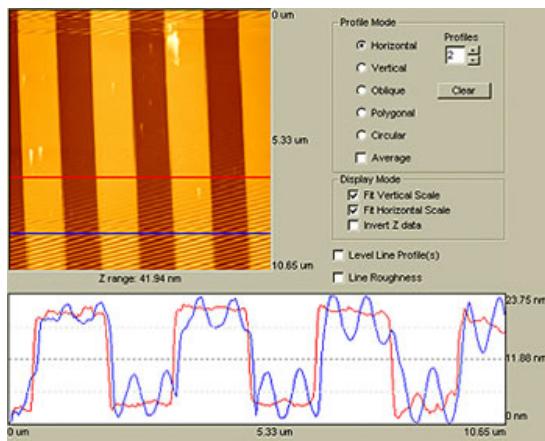


# Microscopia a forza atomica:

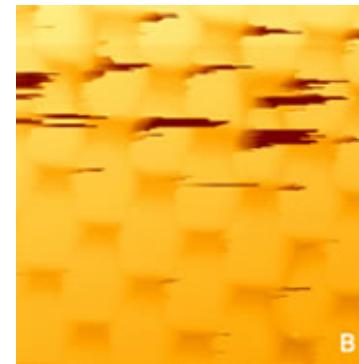
## analisi dei dati

### Artifatti dell'immagine

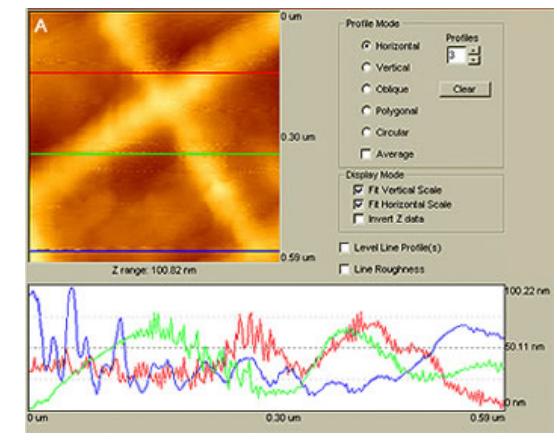
- Rumore elettronico



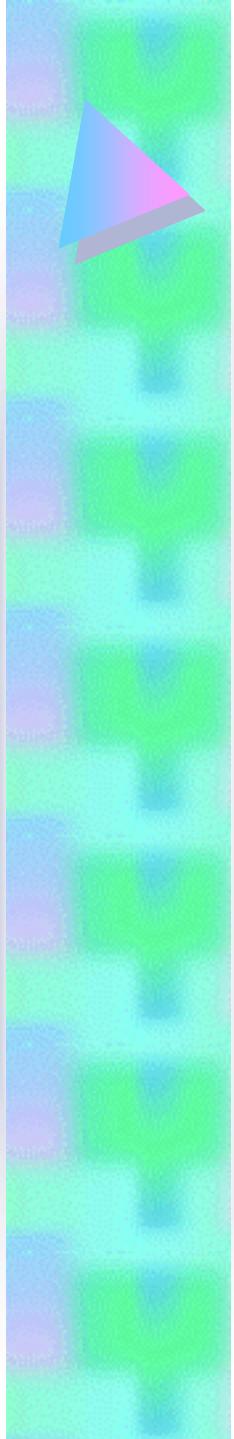
Disturbo elettronico



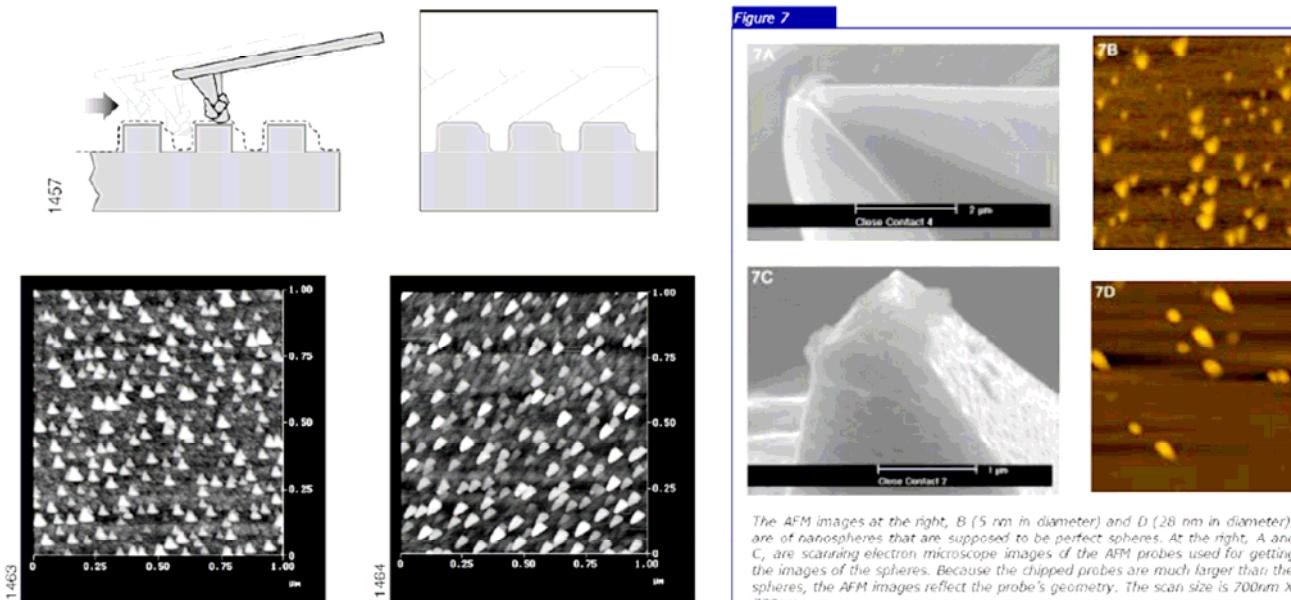
Punta sporca



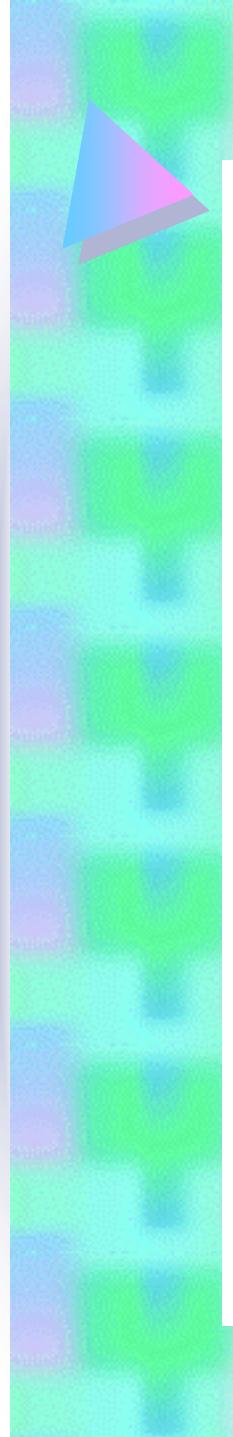
Vibrazioni



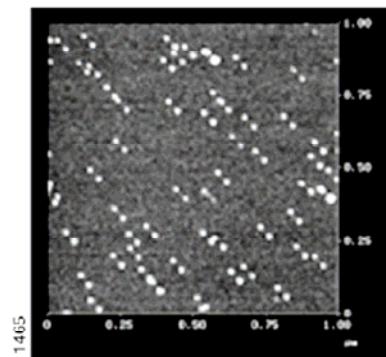
## Imaging Artifacts – Dirty / Dull Tip



If a tip becomes dirty or worn – the features in the image may all seem to have the same shape → really just imaging the damaged or dirty tip.



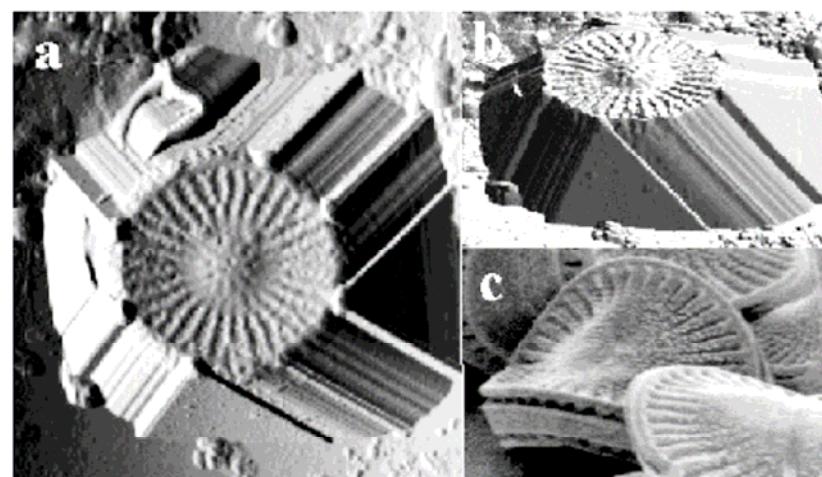
## Imaging Artifacts – Tip Shape



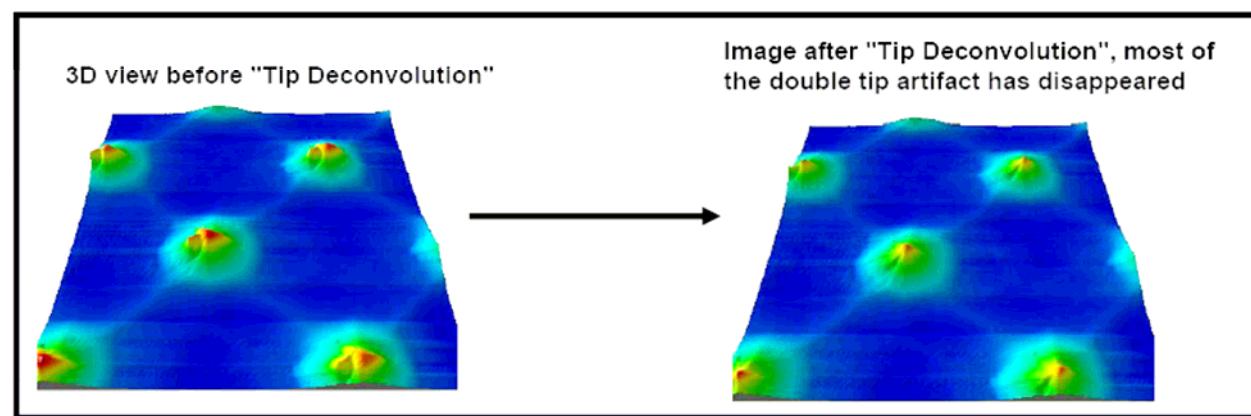
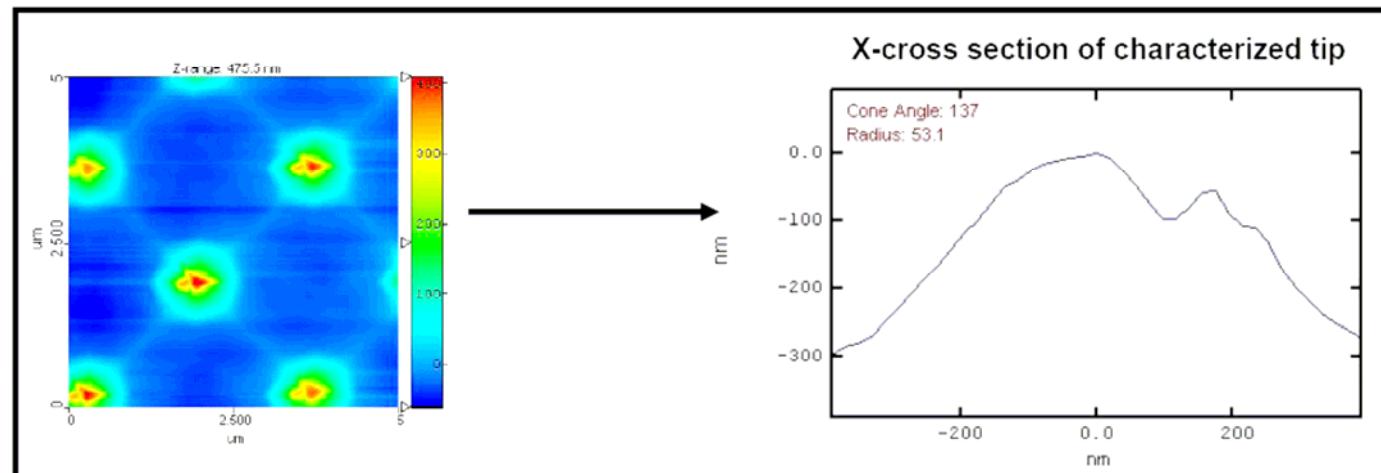
Some damaged tips will show double or multiple tip images → shows up as repeating patterns.

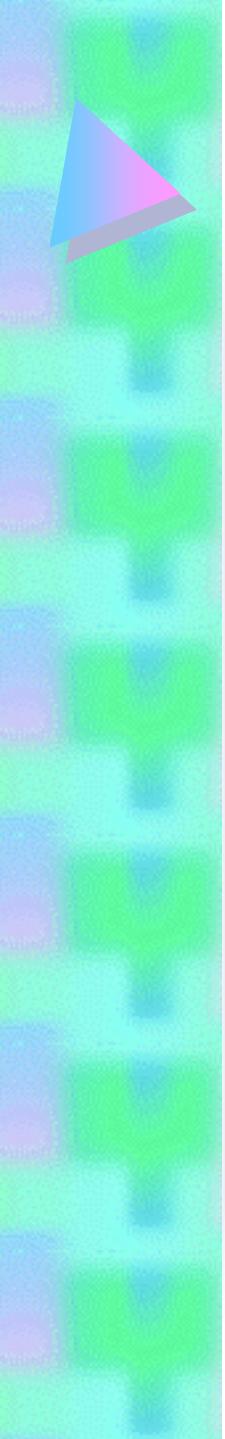
Topographic features too large → image sides of tip (a + b)

(c) SEM image of same sample

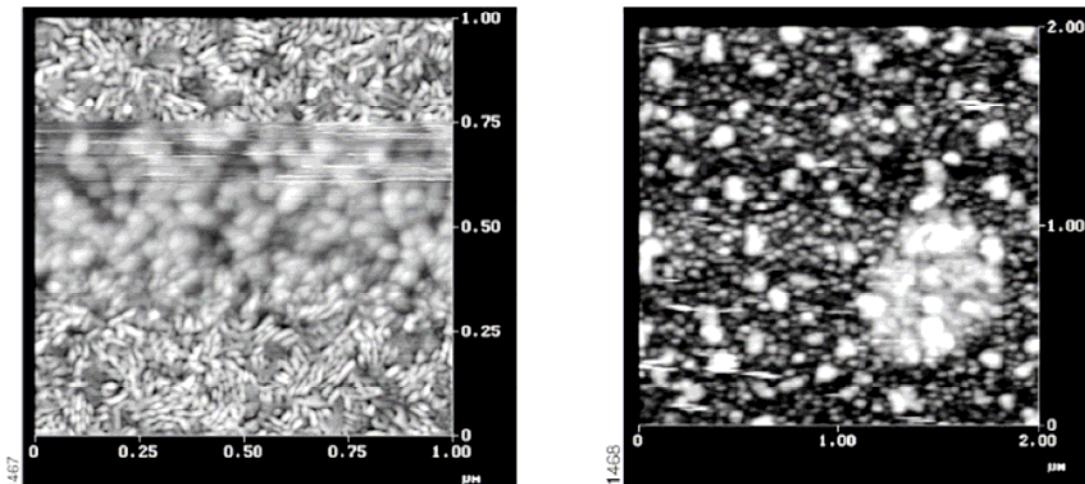


## Tip Characterization - Example

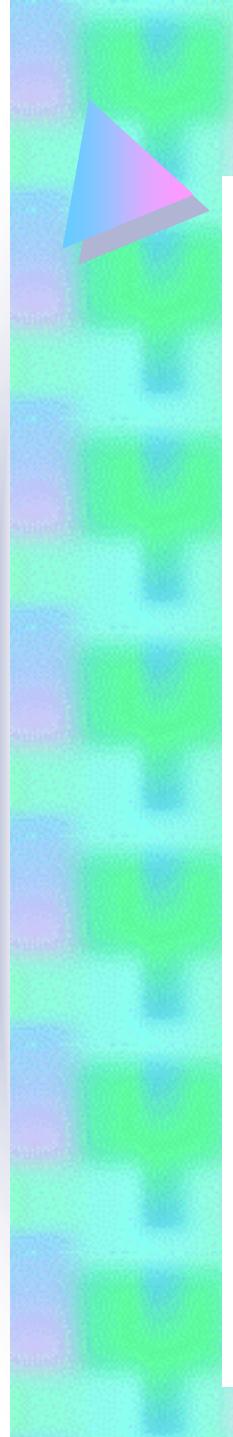




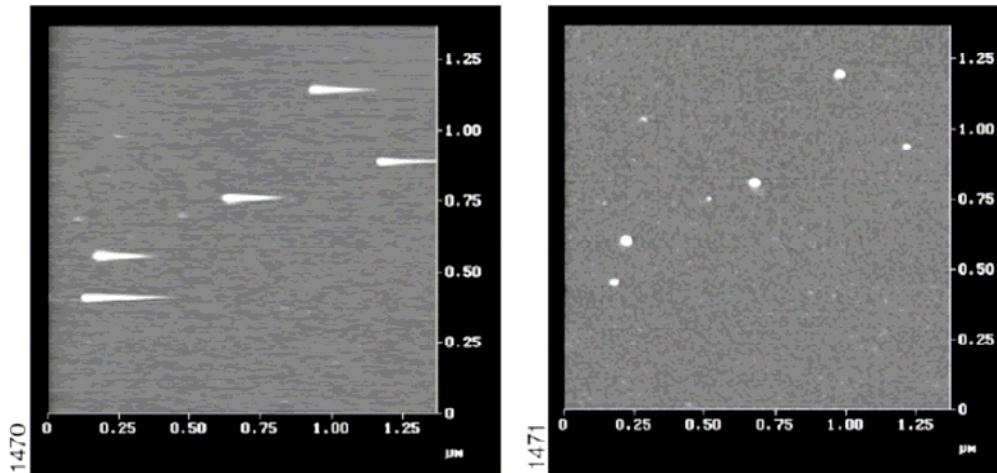
## Imaging Artifacts – Dirty Samples



*Contamination from Sample Surface → tip may pick up contamination from the surface (a common problem with soft materials or nanoparticles).*



## Imaging Artifacts - Other

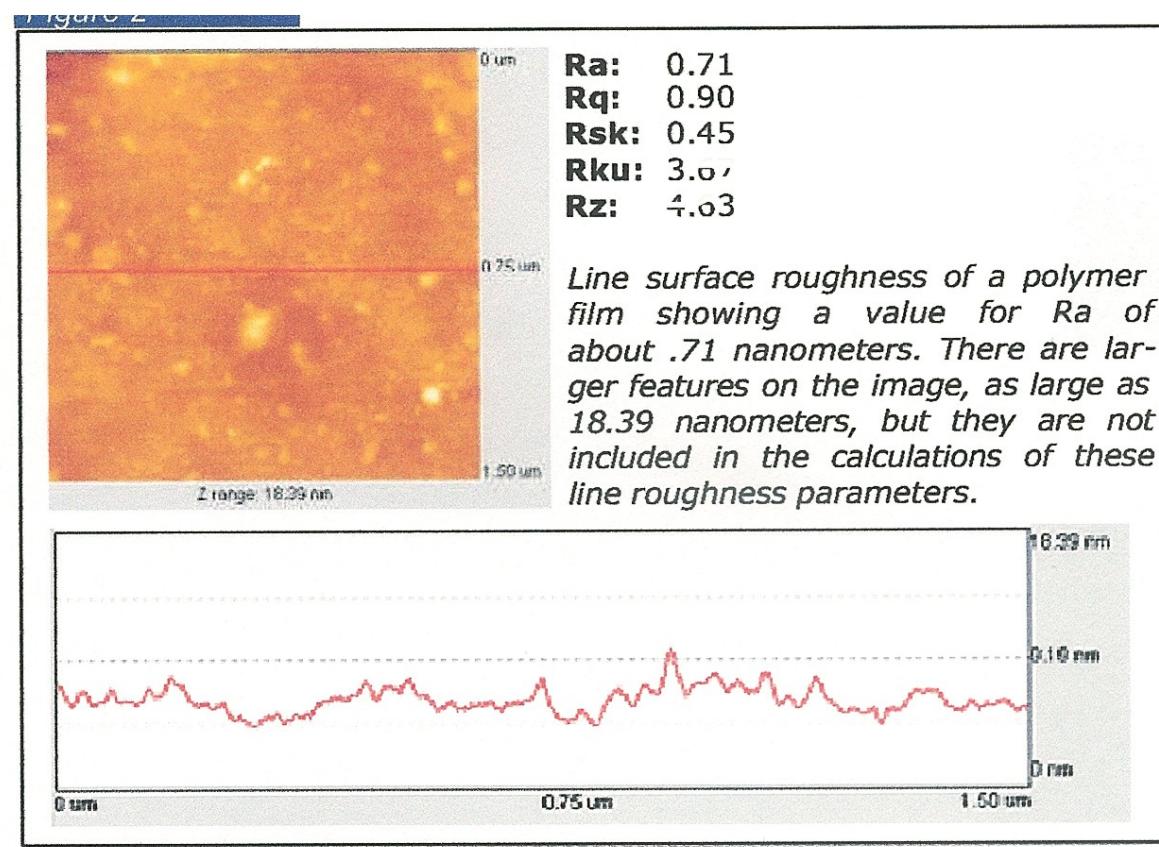


*Tip not tracking surface → adjust feedback parameters, scan rate, etc.*

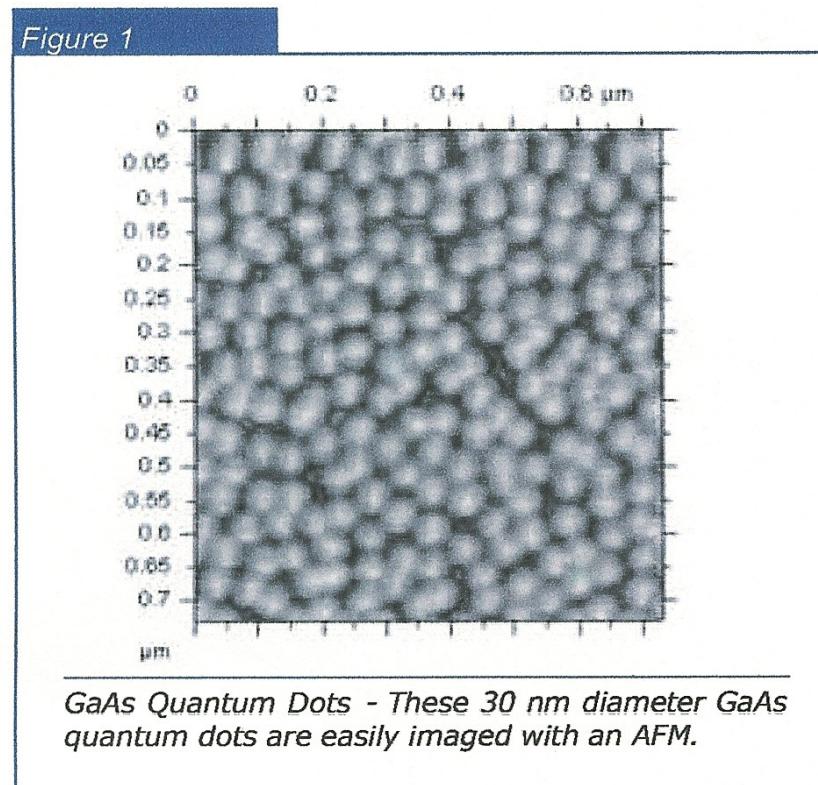
*Other things to watch for:*

- *Vibrational Noise (Sound)*
- *Electronic Noise*
- *Feedback / ringing*

# Microscopia a forza atomica: analisi dei dati



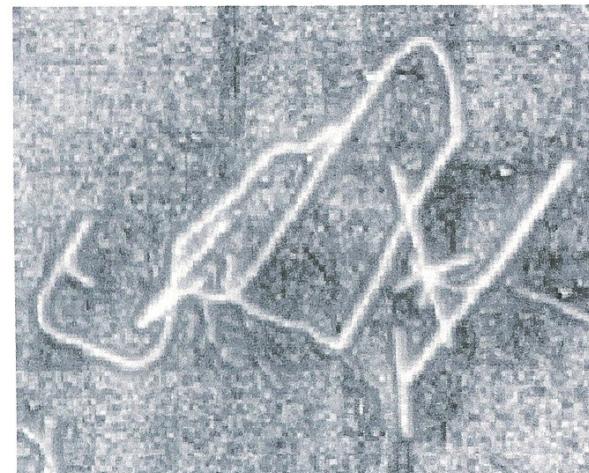
# Microscopia a forza atomica: nanostrutture



# Microscopia a forza atomica: nanostrutture

Figure 3

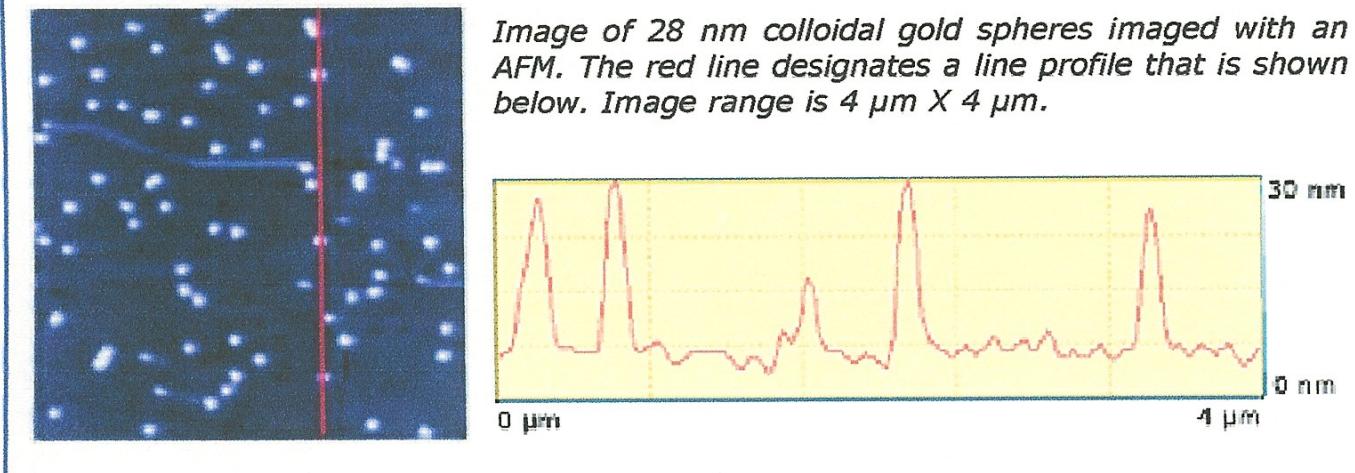
*of the sphere;*



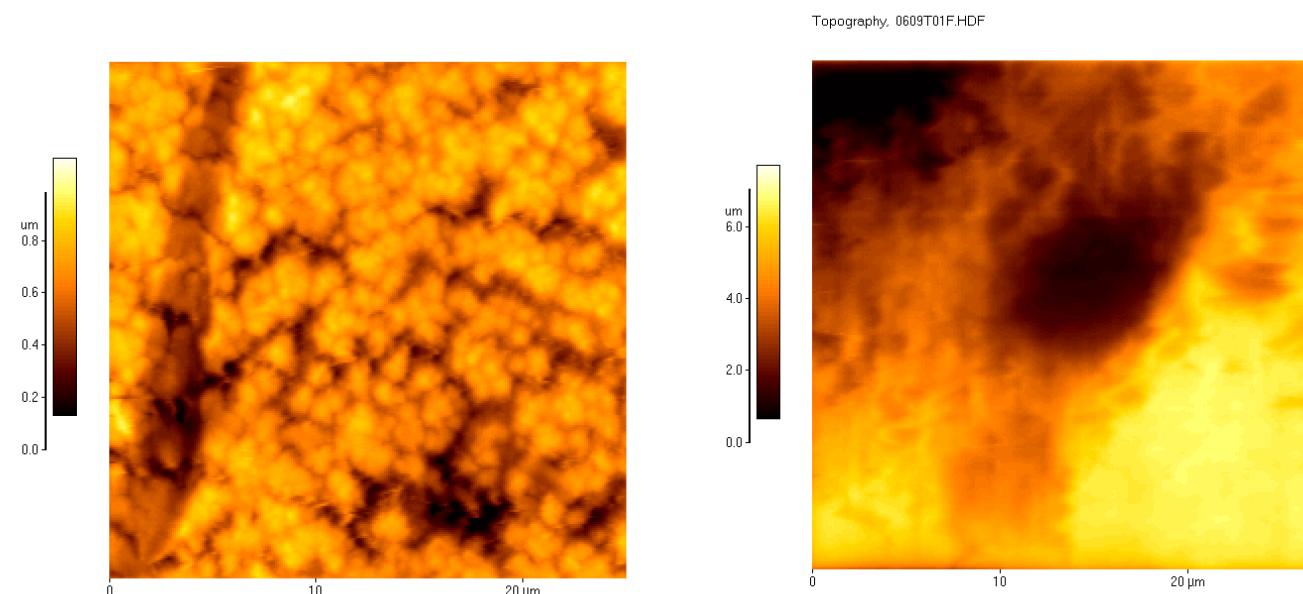
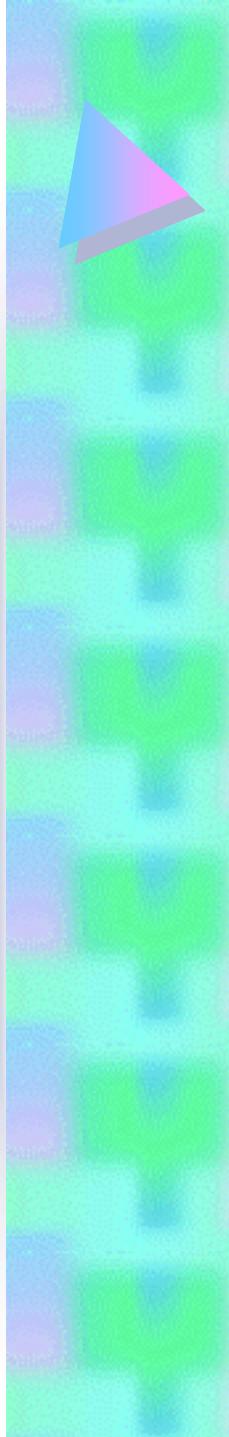
*Single walled Carbon Nanotubes produced with a CVD process and an iron catalyst.*

# Microscopia a forza atomica: nanostrutture

Figure 5



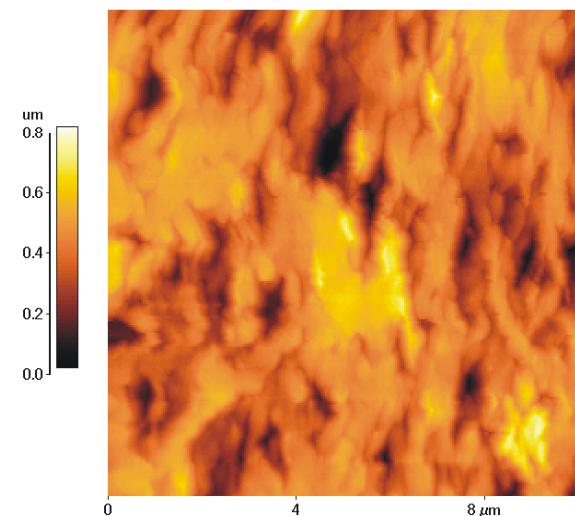
# Microscopia a forza atomica: polimeri



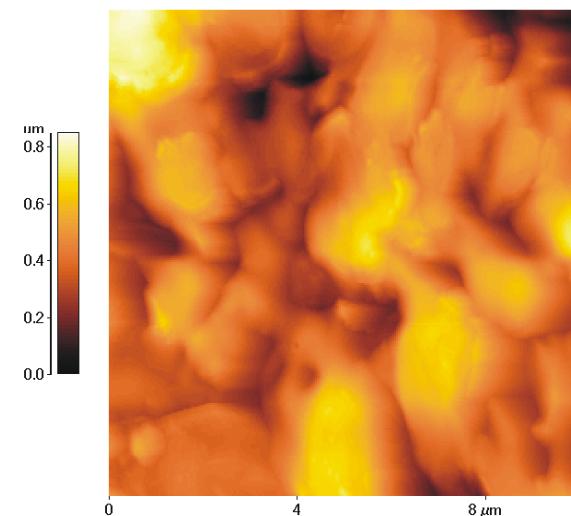
Membrane asimmetriche di PVDF

# Microscopia a forza atomica: polimeri

Topography, 1119S03C.HDF

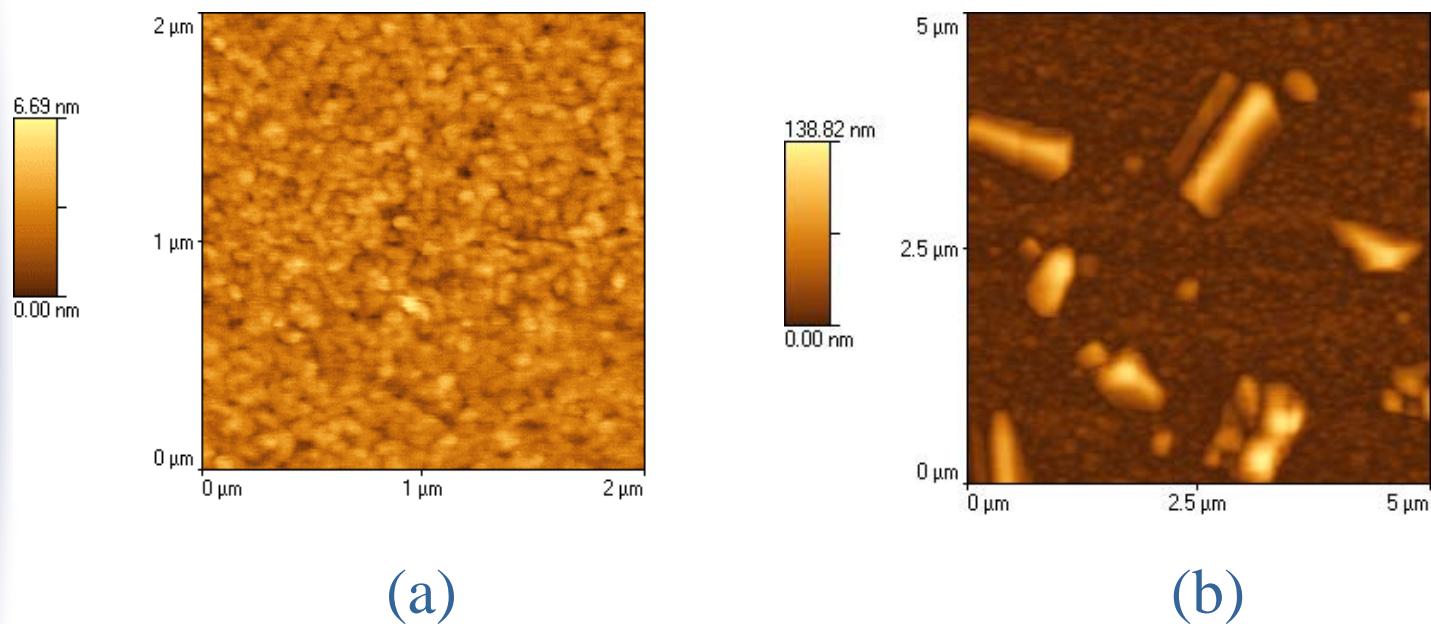
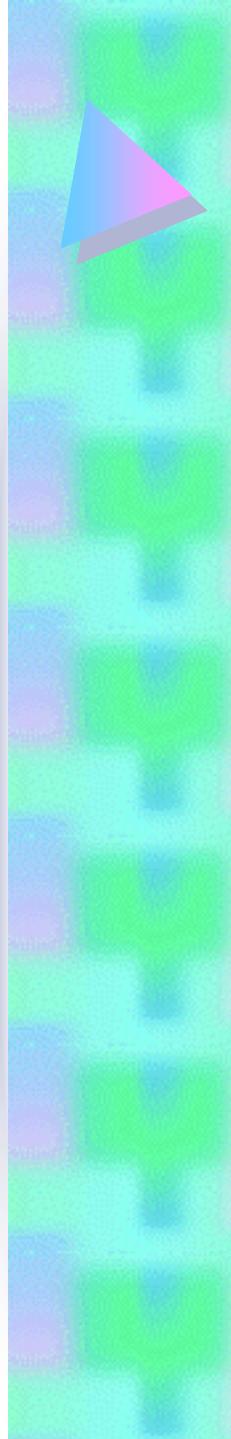


Topography, 1119G003.HDF

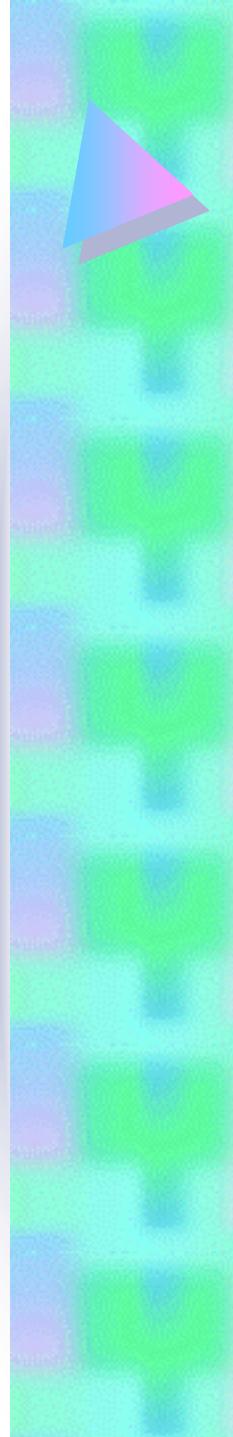


Funzionalizzazione di membrane di PVDF  
(fuel cells)

# Microscopia a forza atomica: film sottili



Film sottili di NdSrCoO preparati per RF sputtering (a) e successivo annealing (b)



# Microscopia a forza atomica: film sottili

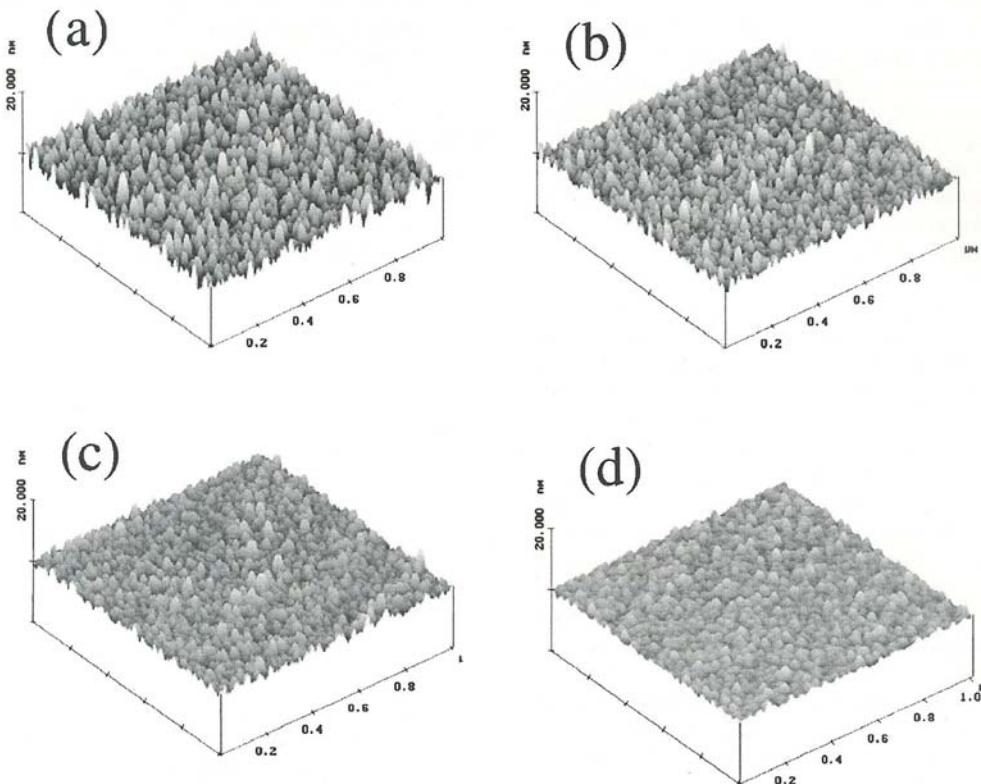
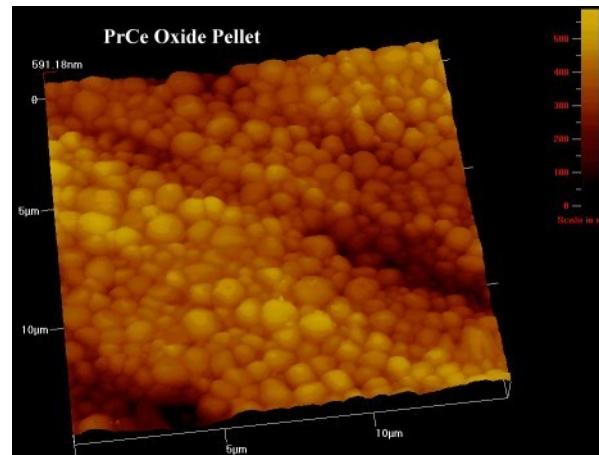


FIG. 3. Surface morphology vs the substrate temperature. The four samples were deposited with the same conditions: rf power of 300 W and Ar flow rate of 15 sccm (about 0.24 Pa), at four different substrate temperatures: room temperature (a), 473 (b), 623 (c), and 763 K (d).

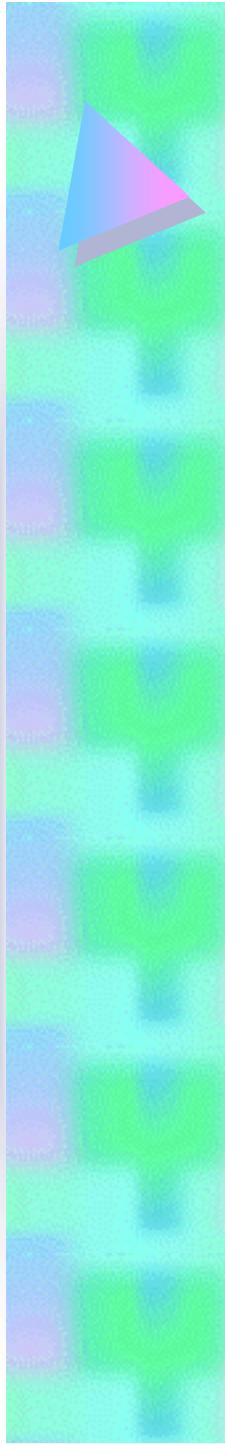
Film sottili di  $\text{SiO}_2$  preparati per rf-sputtering

# Microscopia a forza atomica: film sottili

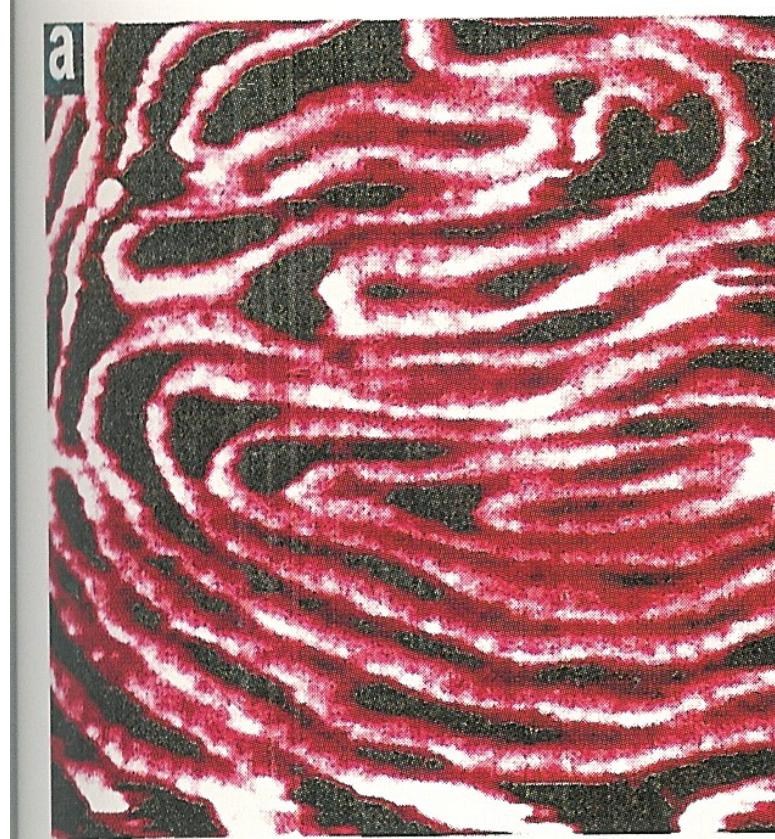


**PrCe Pellet, 3-D view, 15 $\mu$ m scan**

A ceramic pellet of praseodymium and cerium was scanned. The annealed grain structure is resolved easily, as well as the crystal facets on individual grains. A simulated light shading routine enhances the grain structure in 3-D.

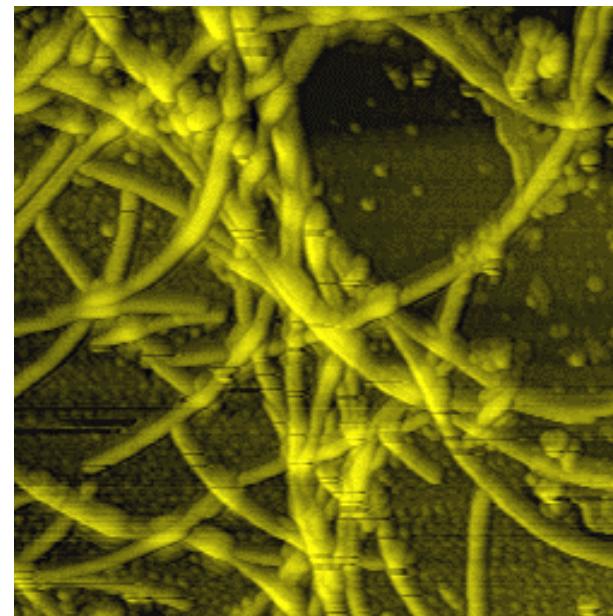


# Microscopia a forza atomica: biologia



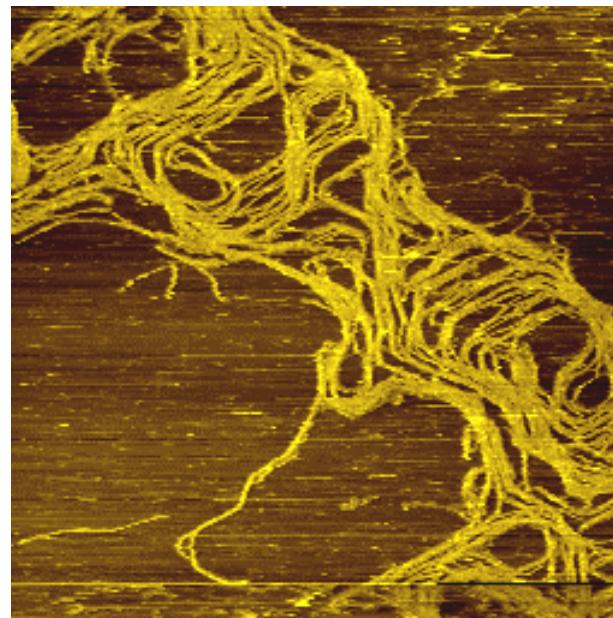
DNA

# Microscopia a forza atomica: biologia

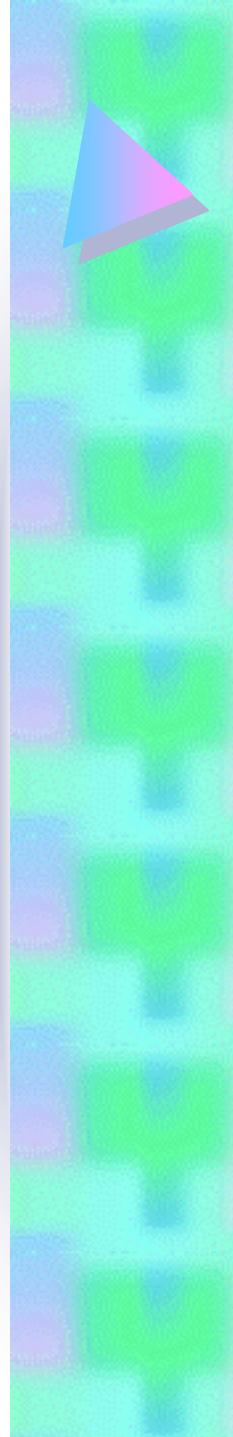


True non-contact AFM image of a tangle of *Pseudomonas putida* bacterial flagellae.

# Microscopia a forza atomica: biologia



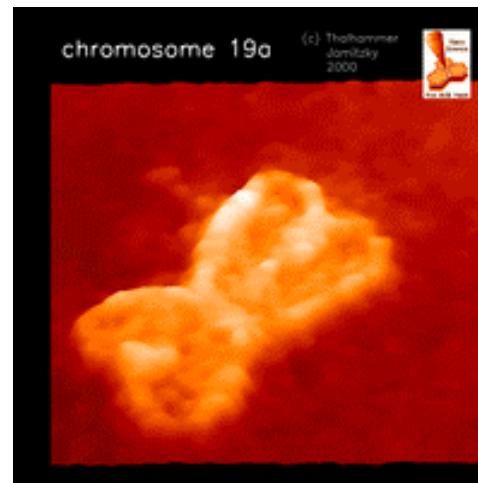
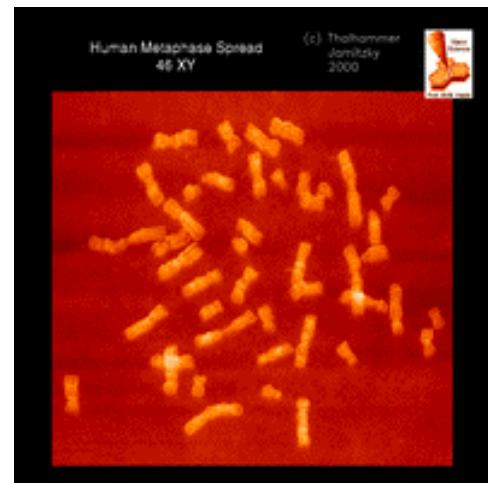
AFM image of aggregated acetan polysaccharide. Image size: 1.2micron x 1.2micron



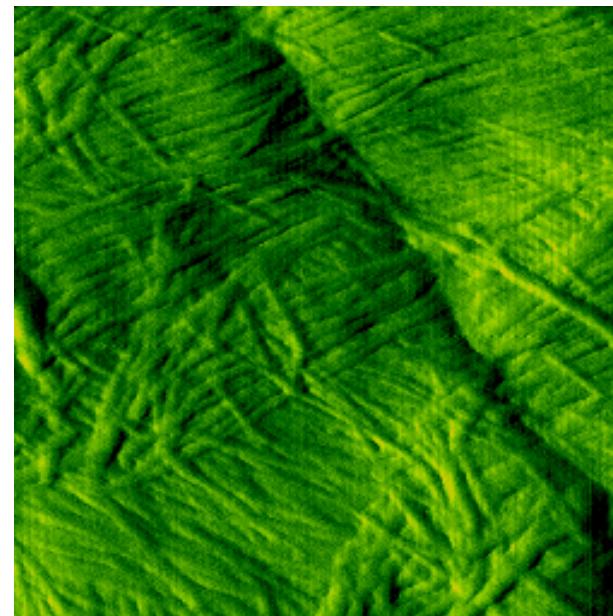
# Microscopia a forza atomica: biologia

## Human Metaphase Chromosomes

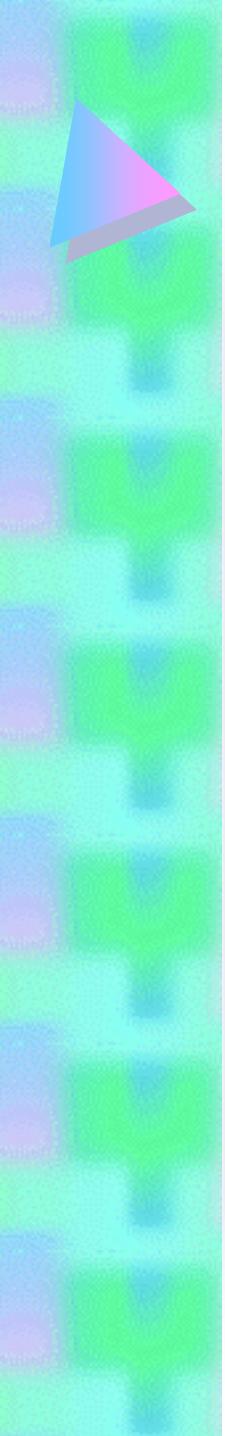
imaged with the atomic force microscope (AFM) in contact mode



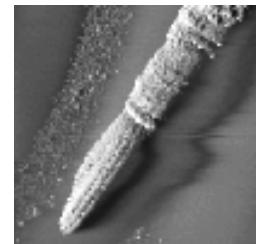
# Microscopia a forza atomica: biologia



AFM error signal image of wet apple cell wall on mica, scanned in air.  
The strands in the image are cellulose microfibrils. Image size: 1 x 1 micron.

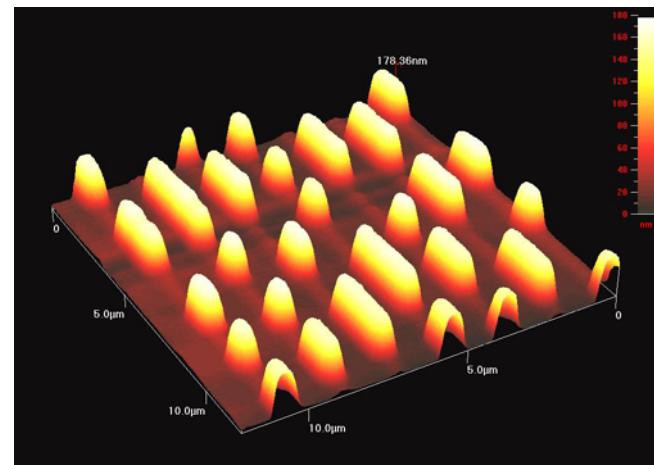


# Microscopia a forza atomica: biologia

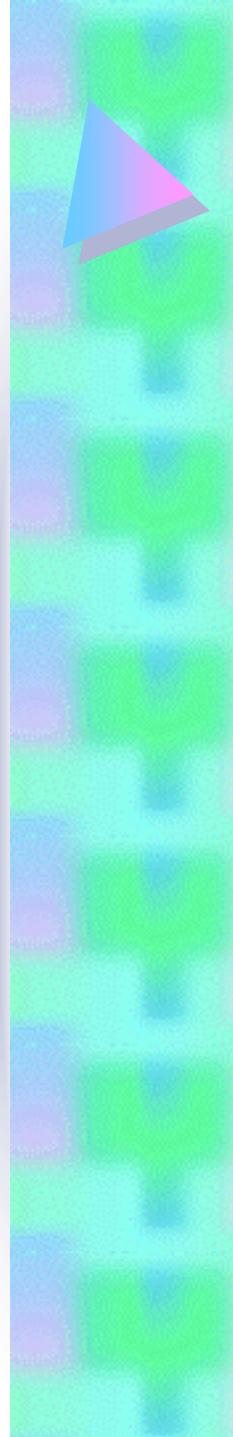


Muscle myofibrils

# Microscopia a forza atomica: data storage media

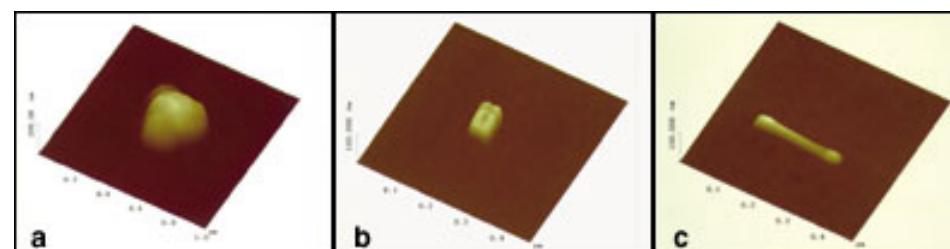
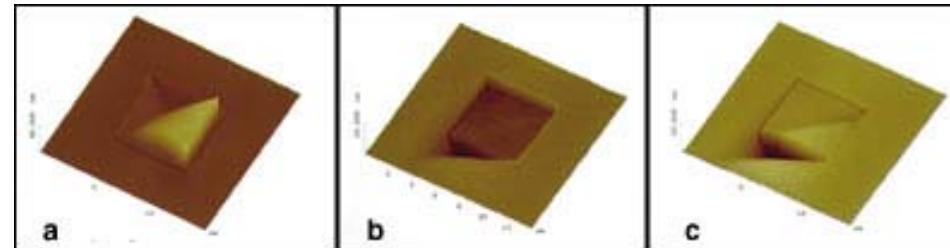


CD Stamper

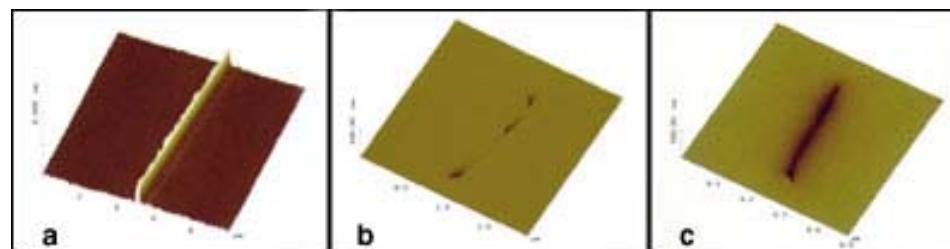


# Microscopia a forza atomica: silicon wafers

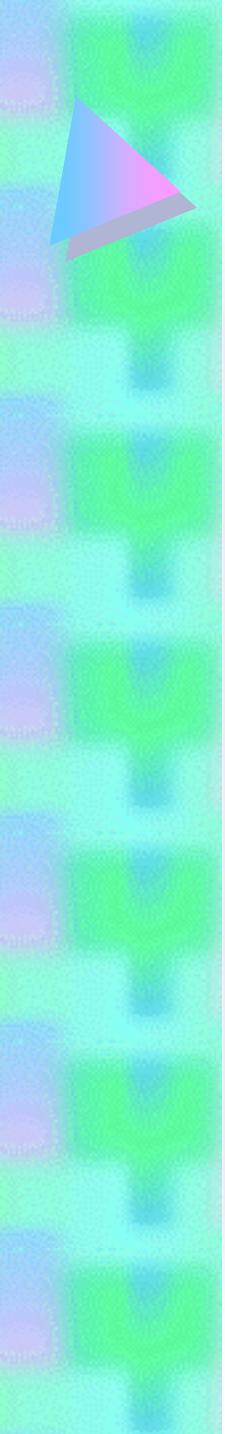
*AFM images of stacking faults on epitaxial Si(100) wafers flagged by laser scattering inspection tools as 0.20-, 0.15-, and 0.13- $\mu\text{m}$  LPDs, respectively.*



*Three AFM images showing (a) a slip line, (b) a microscratch, and (c) a longer microscratch-type defect.*



*Three 0.5 x 0.5- $\mu\text{m}$  AFM images of particle defects flagged by laser scattering inspection tools as 0.10-, 0.067-, and 0.082- $\mu\text{m}$  LPDs, respectively.*



# Microscopia a forza atomica: Bibliografia

1. Scanning Probe Microscopy and Spectroscopy: Theory, Techniques and Applications, Dawn Bonnell, 2nd Edition, Wiley-VCH, 2001
2. Binning G., Quate C.F., Gerber Ch., Phys. Rev. Lett., 1986, 56, 930
3. Amato I. "Atomic Imaging: Candid Cameras for the Nanoworld", Science 276 (5321), 1997