纳米光学表征方法

分为三大类：

1. 扫描探针技术

2. 光谱技术

3. 成像技术

分为三大类：

1.扫描探针技术：
- NSOM
- AFM
- STM

2.光谱技术：
- General introduction
- Raman Spectrometry

3.成像技术：
- Confocal imaging
- multi-photon imaging

Atomic Force Microscopy

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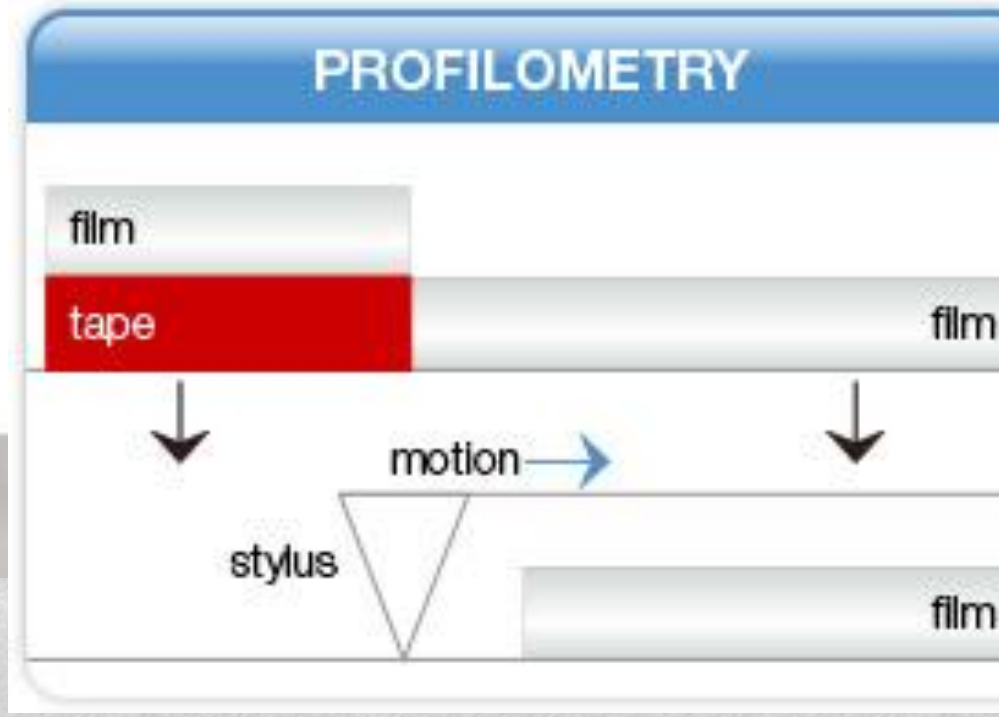
University of Electronic Science and Technology of China

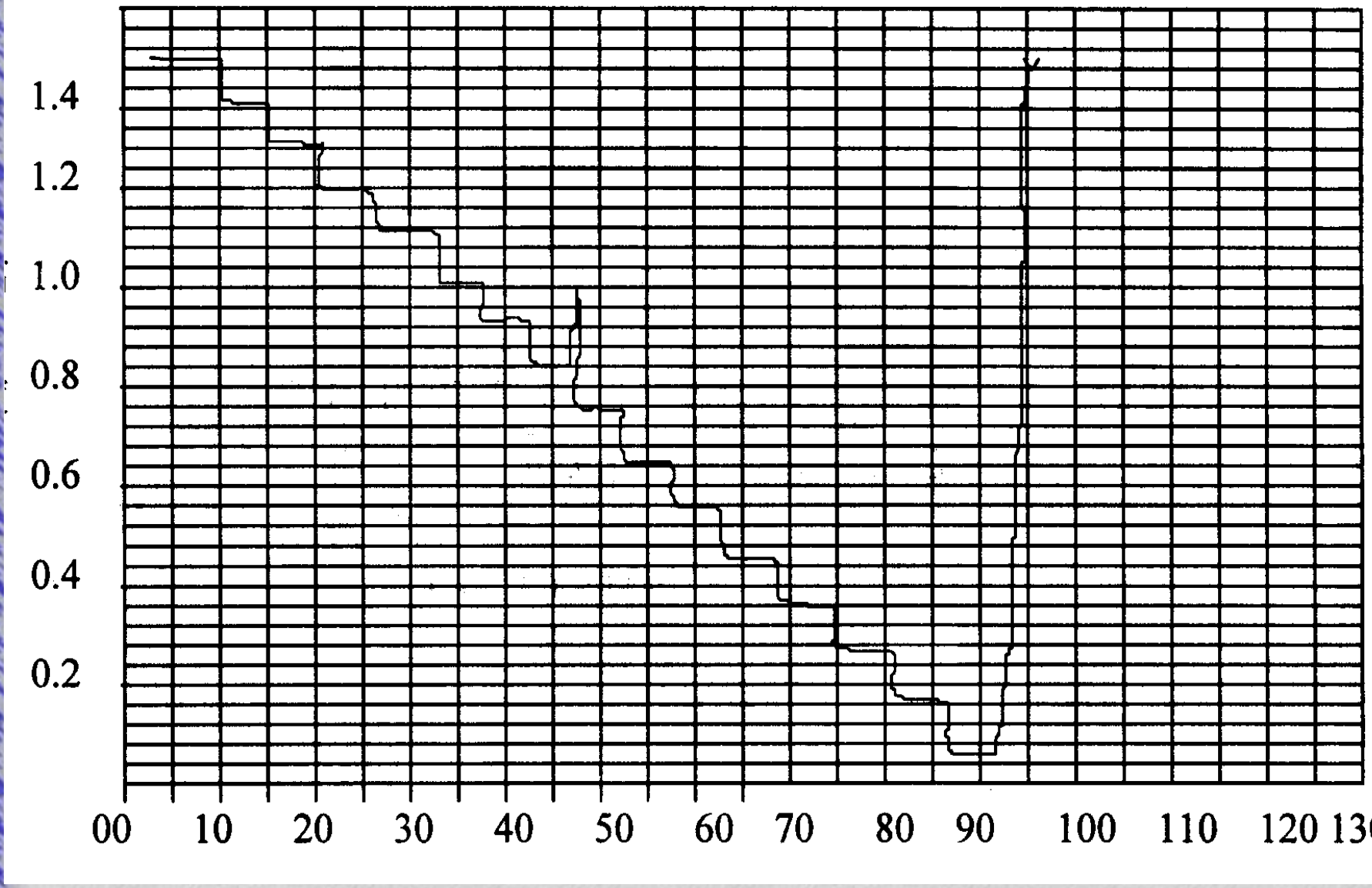
Lecture Plan

- Introduction
- Atomic Force Microscopy
  - Contact
  - Non-contact
  - Intermittent (Tapping Mode)
- Tip
- Other AFM-like SPM methods
- Applications

Origination of AFM

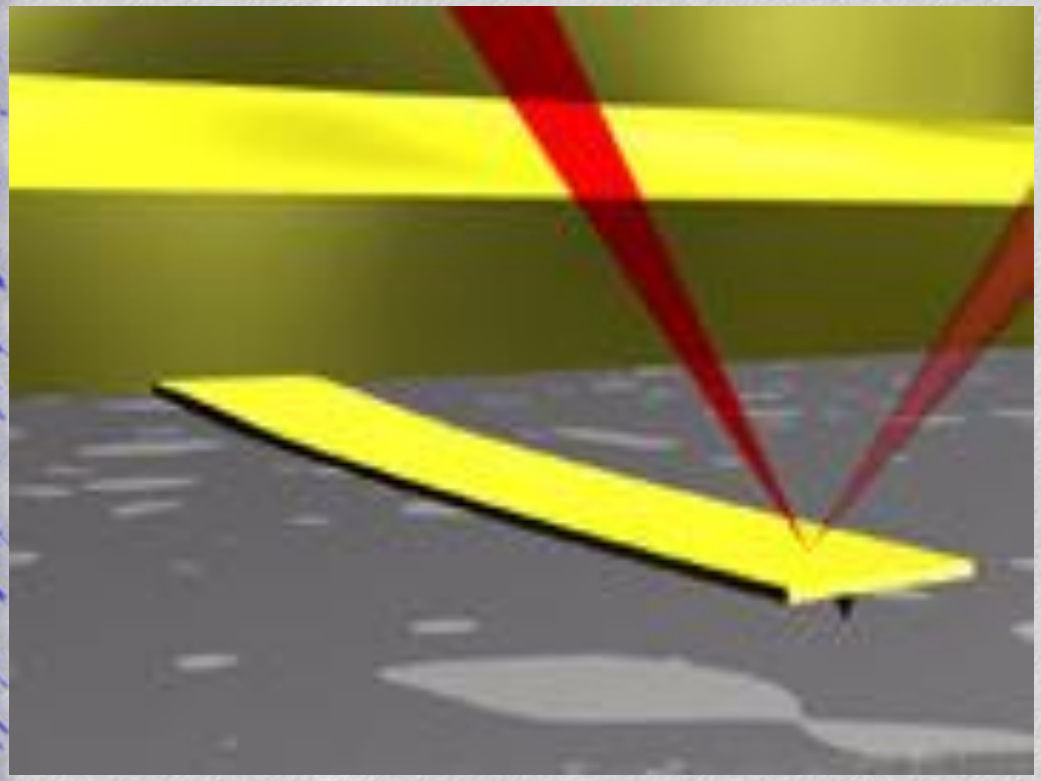
Profilormetry

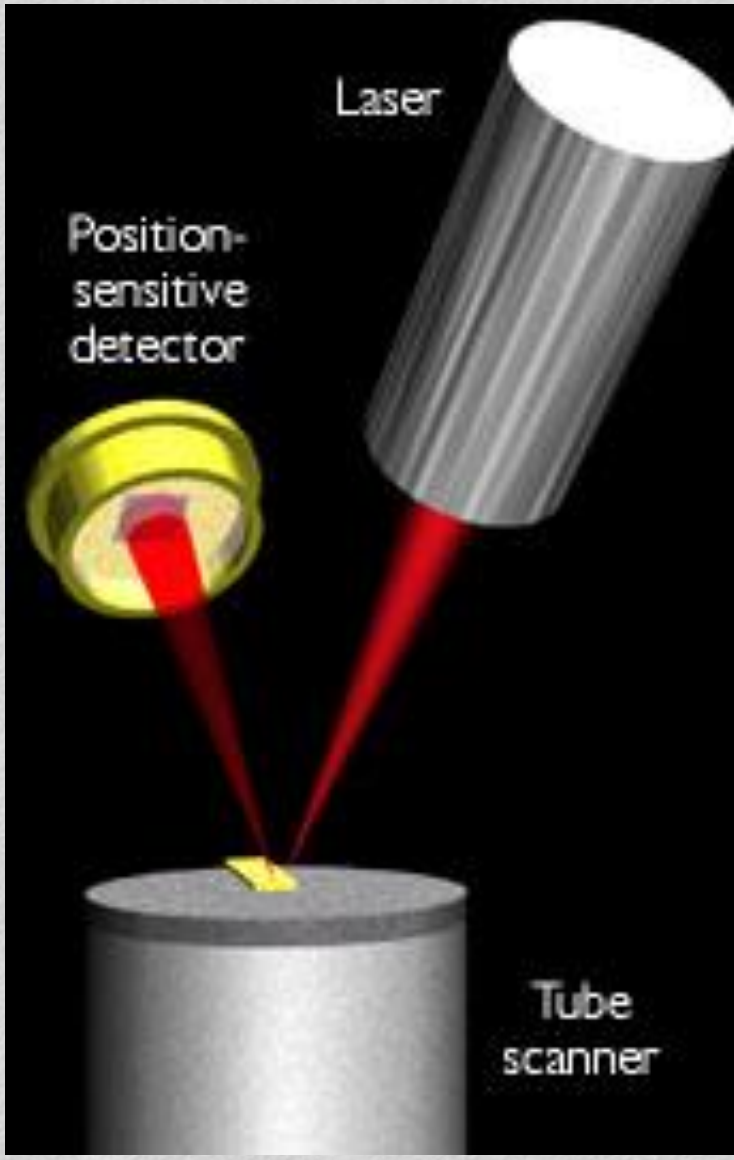




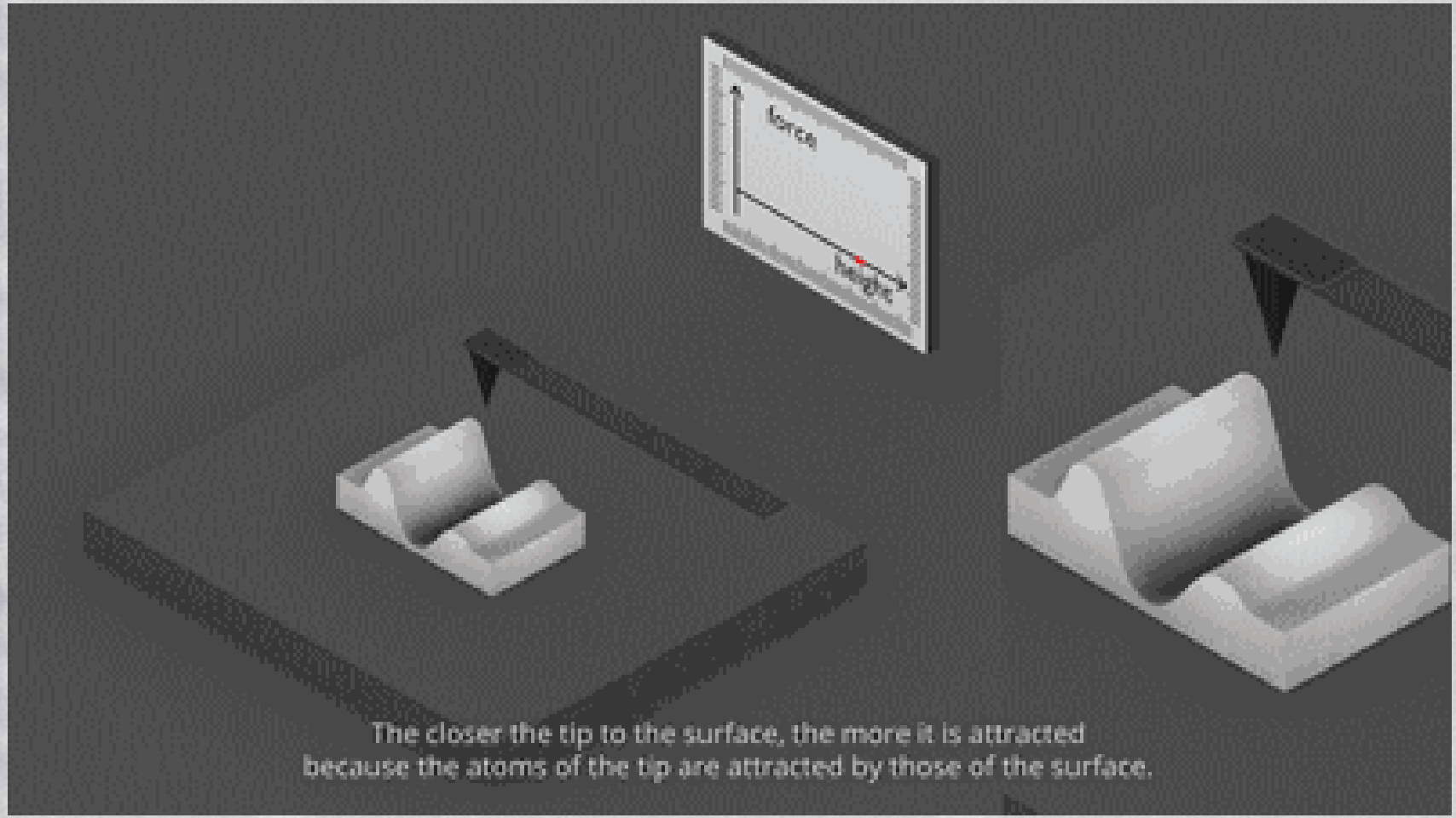
Measured profile of 16 level DOE by profilometer

Atomic Force Microscopy



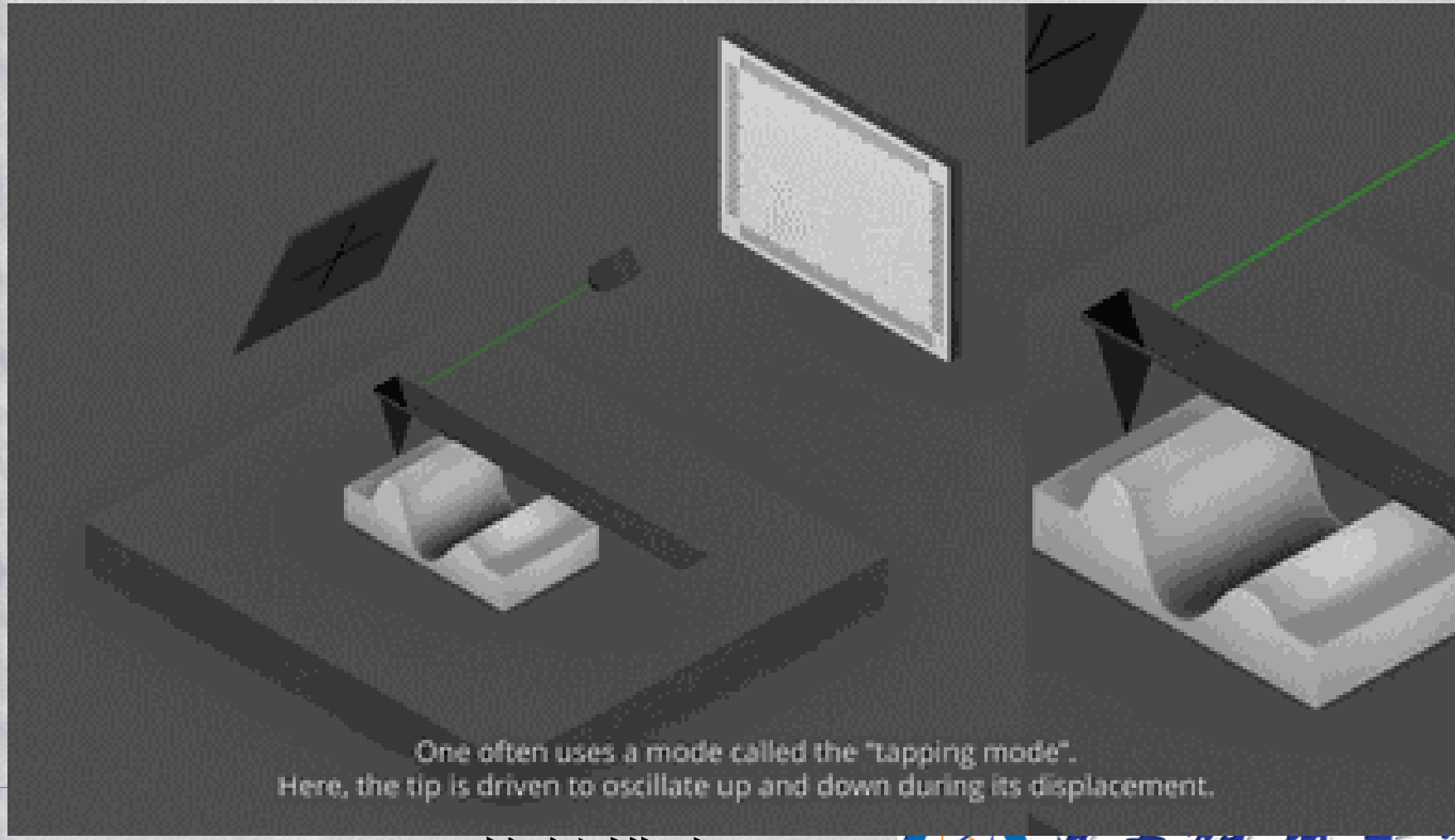


将一个对微弱力极敏感的微悬臂一端固定，另一端有一微小的针尖，由于**针尖尖端原子与样品表面原子间存在极微弱的作用力**，通过在扫描时控制这种力的恒定，带有针尖的微悬臂将在垂直于样品的表面方向起伏运动。测出微悬臂对应于扫描各点的位置变化，从而可以获得样品表面形貌的信息。

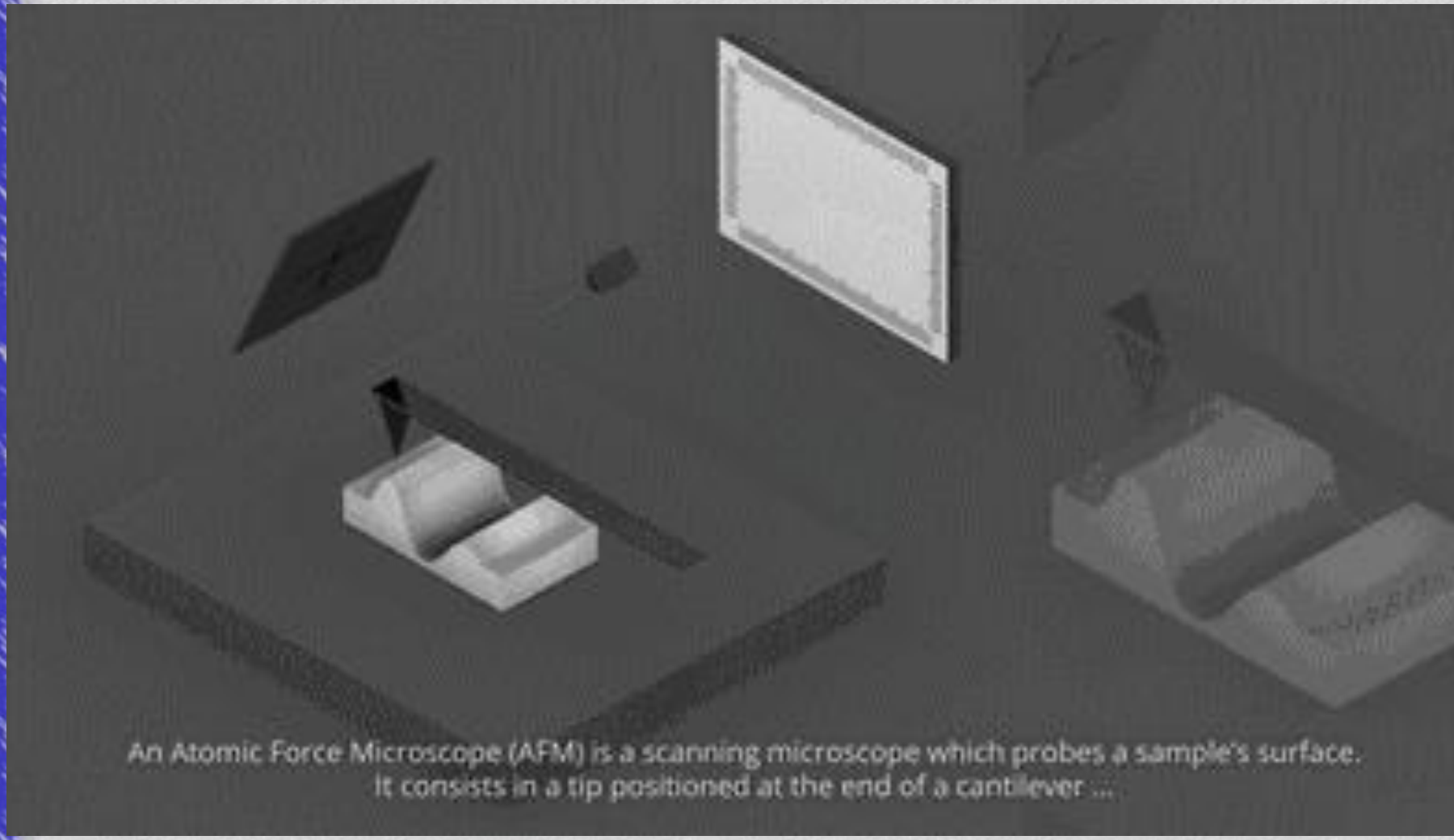


AFM原理：针尖与表面原子相互作用

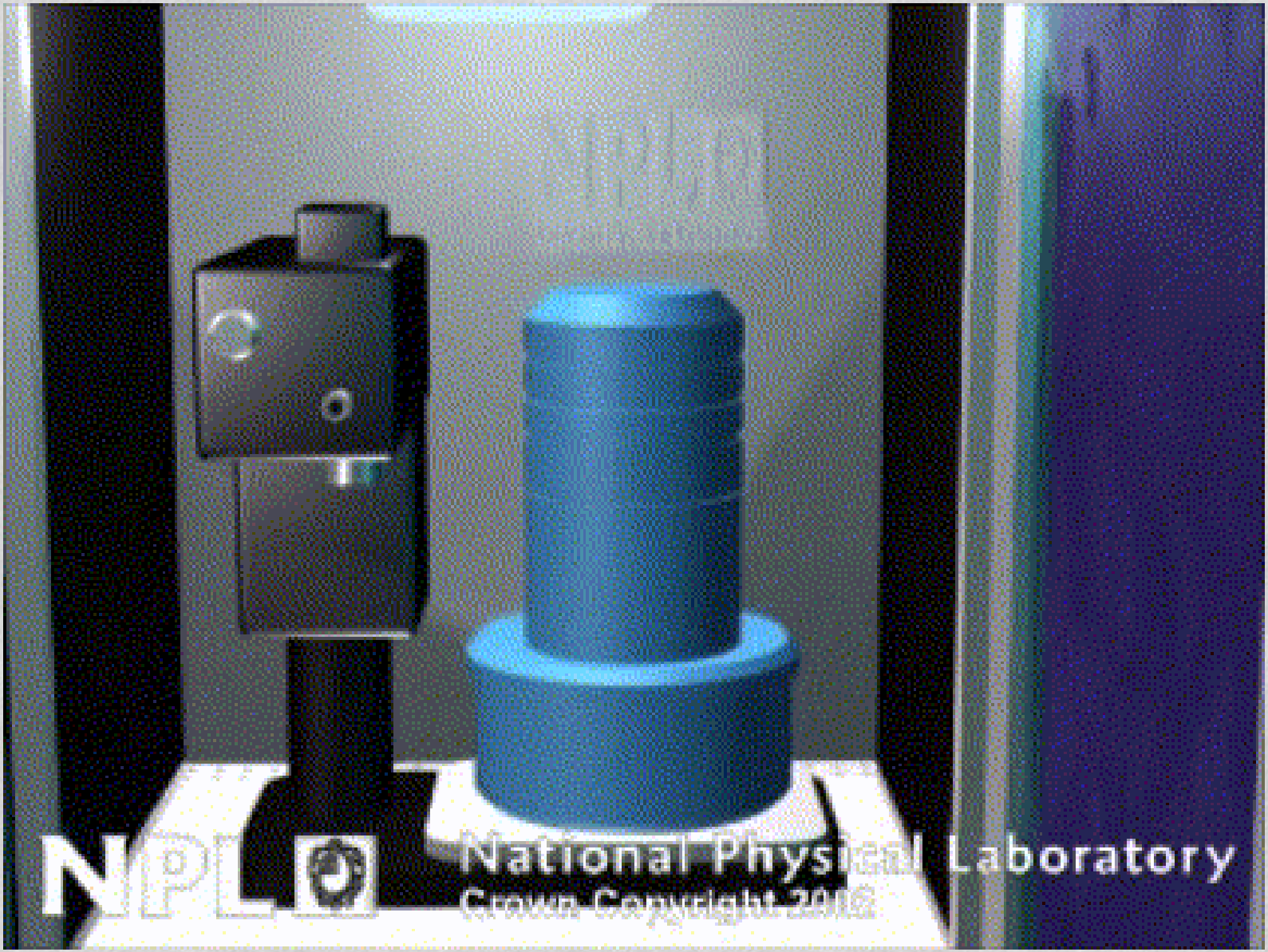
AFM的扫描模式有接触模式和非接触模式，接触式利用原子之间的排斥力的变化而产生样品表面轮廓；非接触式利用原子之间的吸引力的变化而产生样品表面轮廓。



接触模式



动态/轻敲模式（tapping mode）


NPL
National Physical Laboratory
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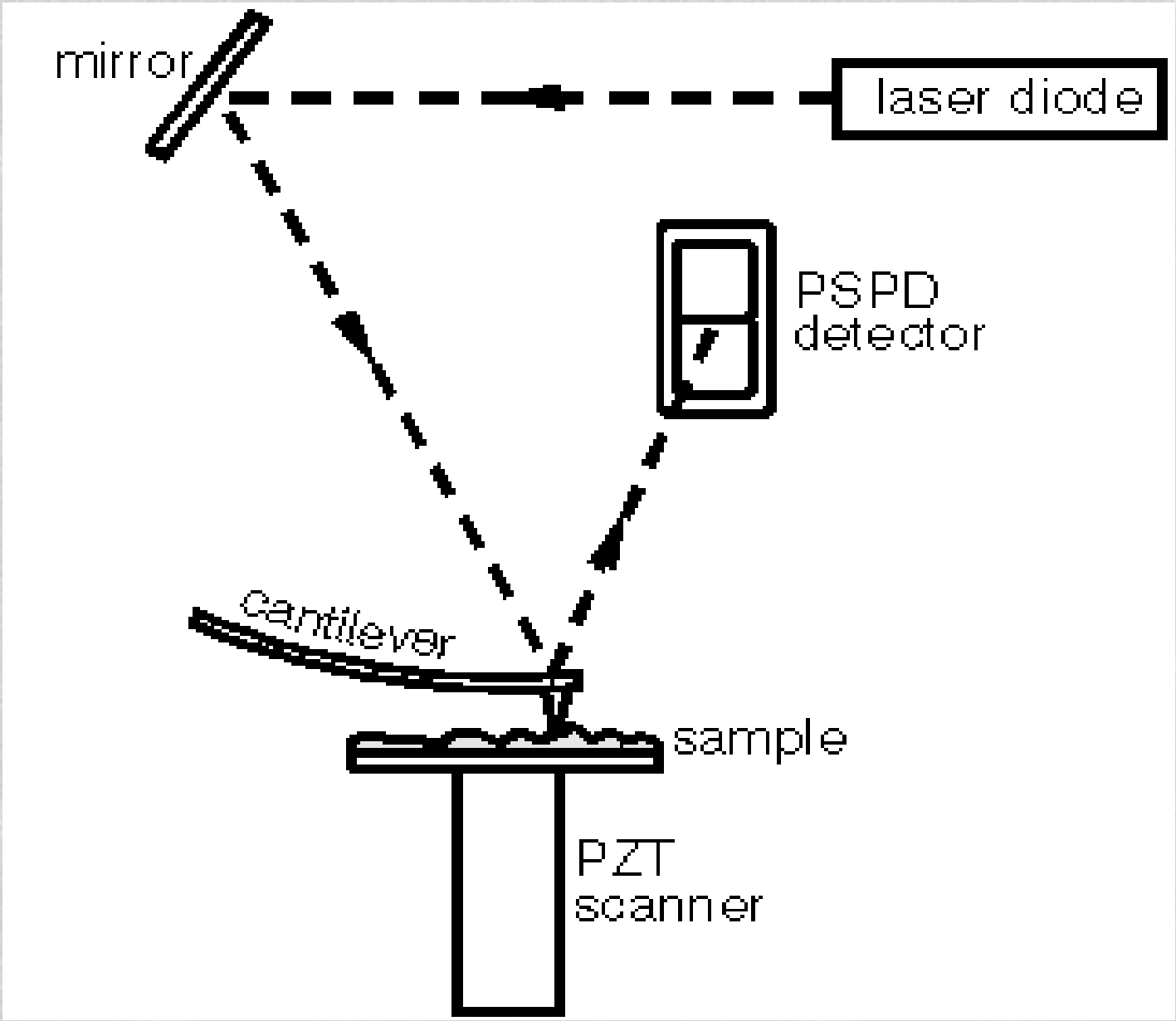
AFM vs. Stylus Profilometry

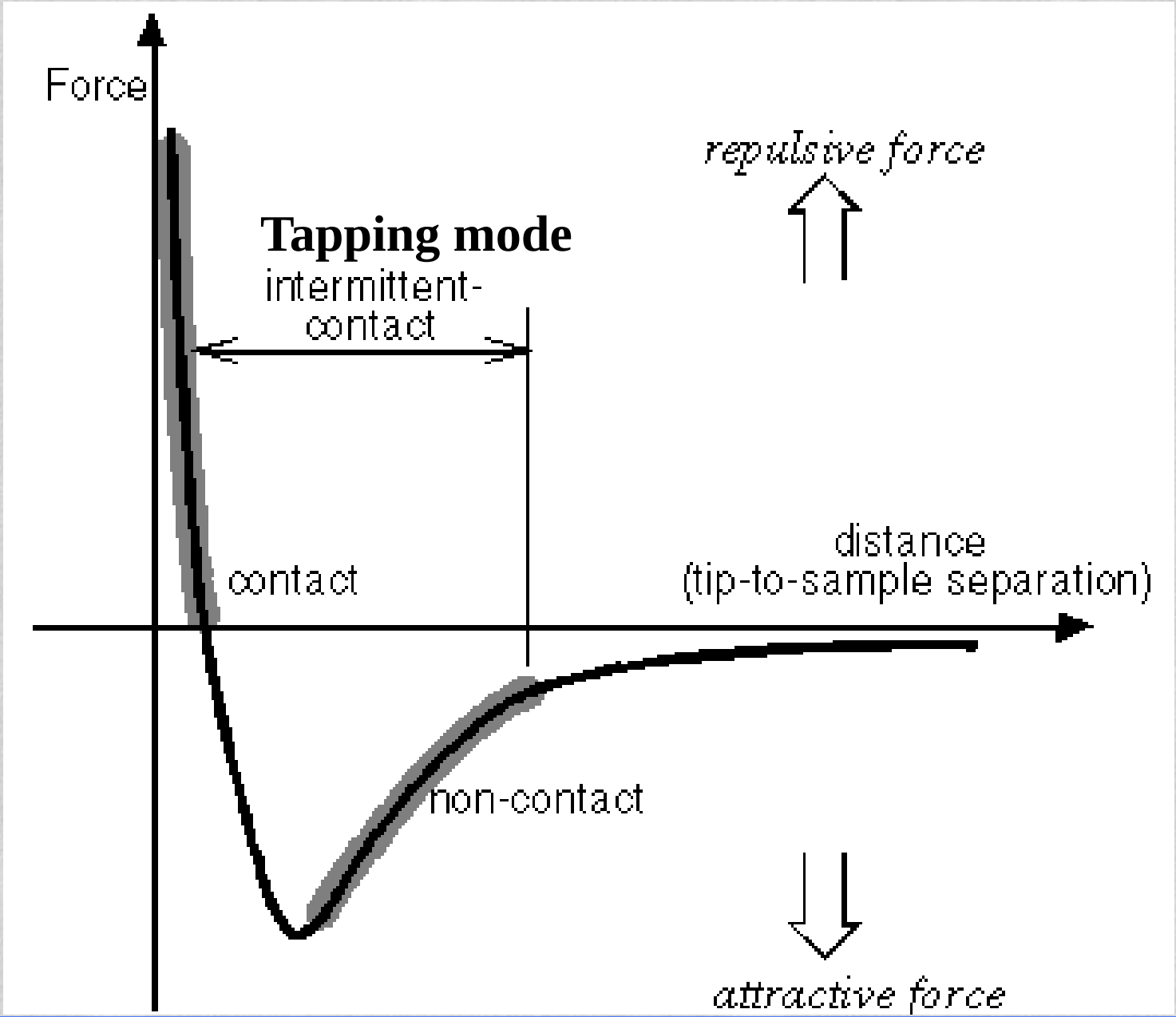
- In comparison with stylus profilometry

- AFMs have a number of refinements
  - sensitive detection
  - flexible cantilevers
  - sharp tips
  - high resolution tip-sample positioning
  - force feedback

AFM General Arrangement

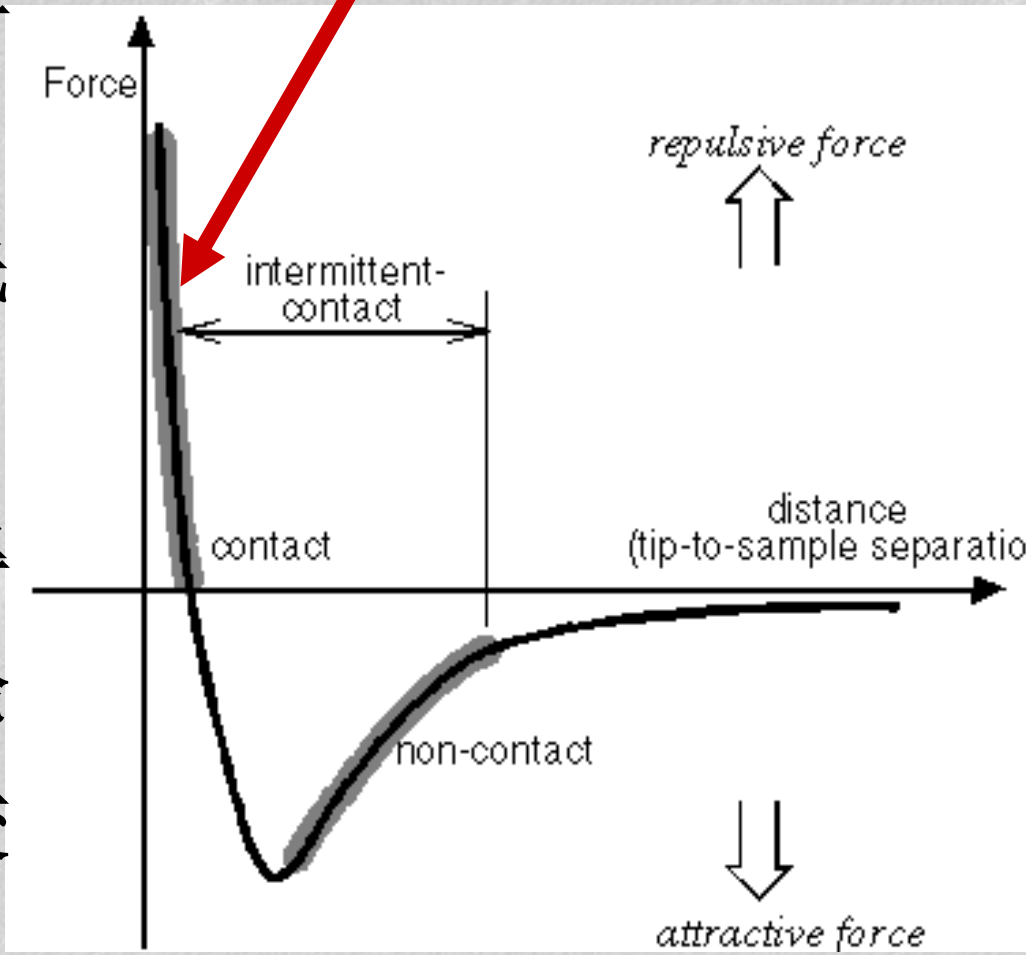


Force vs. Distance



Force vs Distance : Contact mode

- 探针与样品软“物理接触”
- 探针的弹性系数很低。
- 扫描时，接触力使得悬臂梁随样品表面轮廓起伏的变化而改变。



Contact Mode

——Constant Height or Constant Force

- **Constant-force mode**
  - 扫描头随样品表面轮廓上下移动时悬臂梁的变形作为恒定原子间力反馈控制电路的输入。
- **Constant-height mode**
  - 扫描头高度方向的变化设为恒定值，悬臂梁的空间变化可直接用于产生样品形貌轮廓的数据。

Deflection of cantilever

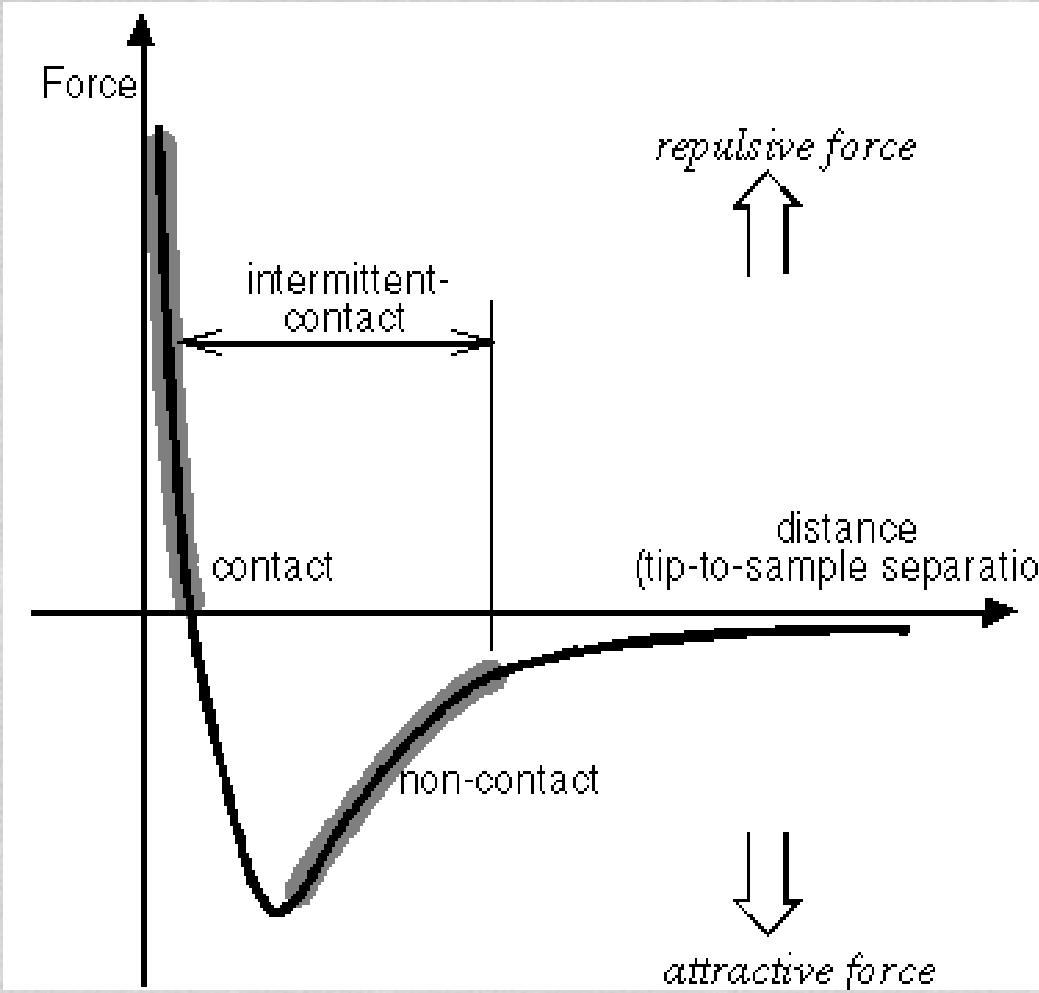
Contact Mode

—Constant Height or Constant Force

- **Constant-force mode**
  - 扫描速度取决于反馈电路的响应时间，但通过探针施加于样品表面的力可得到很好地控制。该模式已逐渐成为主要的工作模式。
- **Constant-height mode**
  - 用于平坦表面原子量级的成像，因为悬臂梁的变形量很小。对于扫描速度很重要的场合，该模式可记录表面的实时变化。

Non Contact Mode

由于探针施加于样品表面的力很低，约 10^{-12} N，该模式更适合于软的或者弹性表面的测量。另一优点是样品不易被探针弄脏。

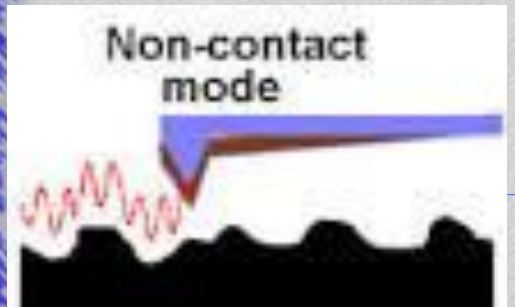


Non Contact Mode

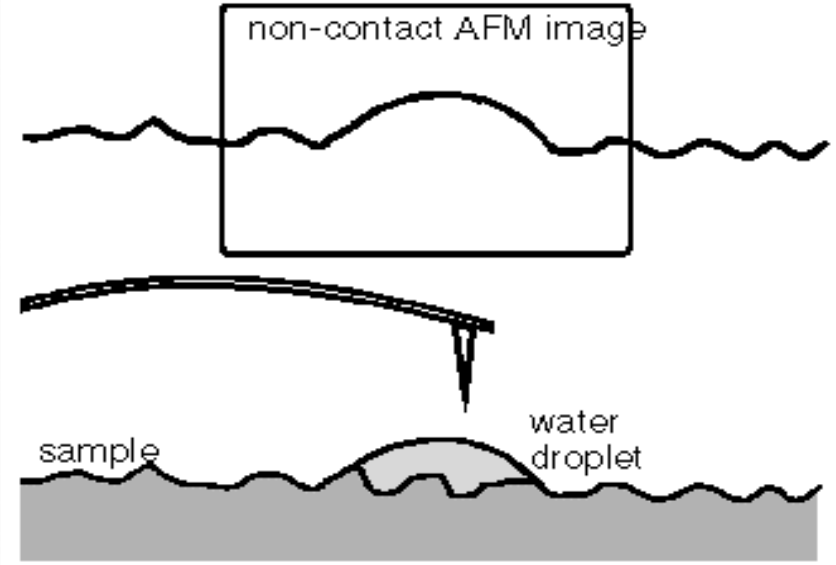
- 非接触模式中，刚性悬臂梁的振动频率(**100~400 kHz**) 以及振幅为几十至几百埃。它能探测出探针接近表面时共振频率或振幅的微小变化。探测灵敏度在垂直方向可达到小于1埃的水平。
- 非接触模式中，如果系统探测出悬臂梁共振频率或振幅的变化并通过反馈系统保持为恒定不变，这样也可使扫描探针上下移动。

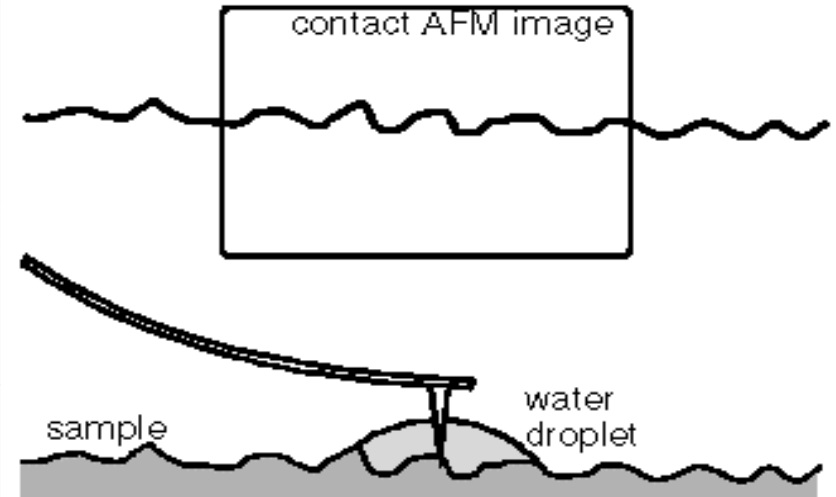
Non-contact Mode

- **Advantage**
  - 探针保持与样品很小的间隙，避免了划伤样品表面。
- **Drawbacks**
  - van de Waals 力很弱以致于需要靠振荡来探测样品与探针之间的微小力的变化。
  - 因此，非接触模式测量分辨率很低。



Measurement of liquids





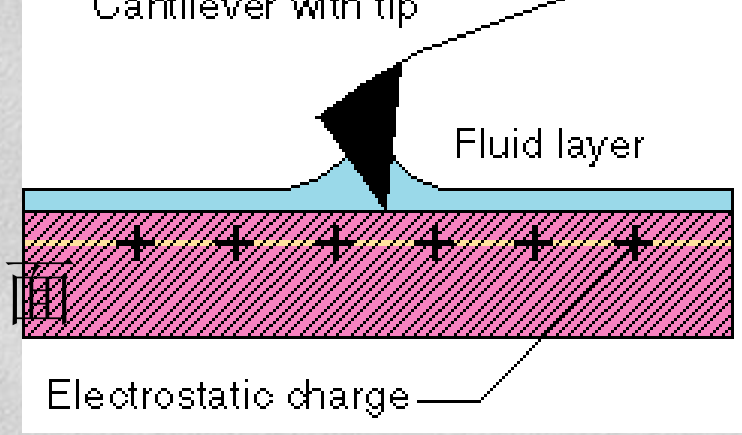
Unique feature of

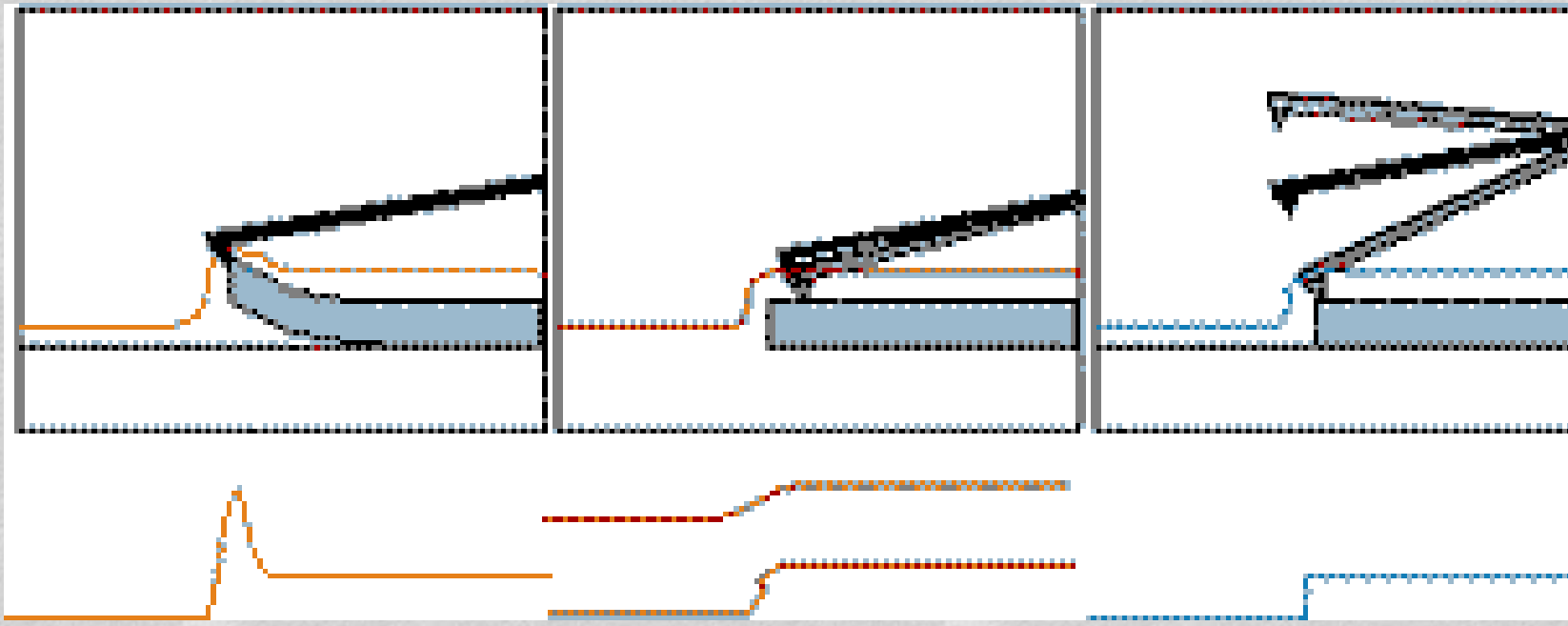
non-contact mode

icroscopy.
ESTC

Contact Mode

- 对于**接触模式**
  - 探针被拖拽着扫过样品表面
- 缺点
  - 容易造成样品表面以及探针的损伤；
  - 来自样品表面的静电荷可形成额外的粘附力。



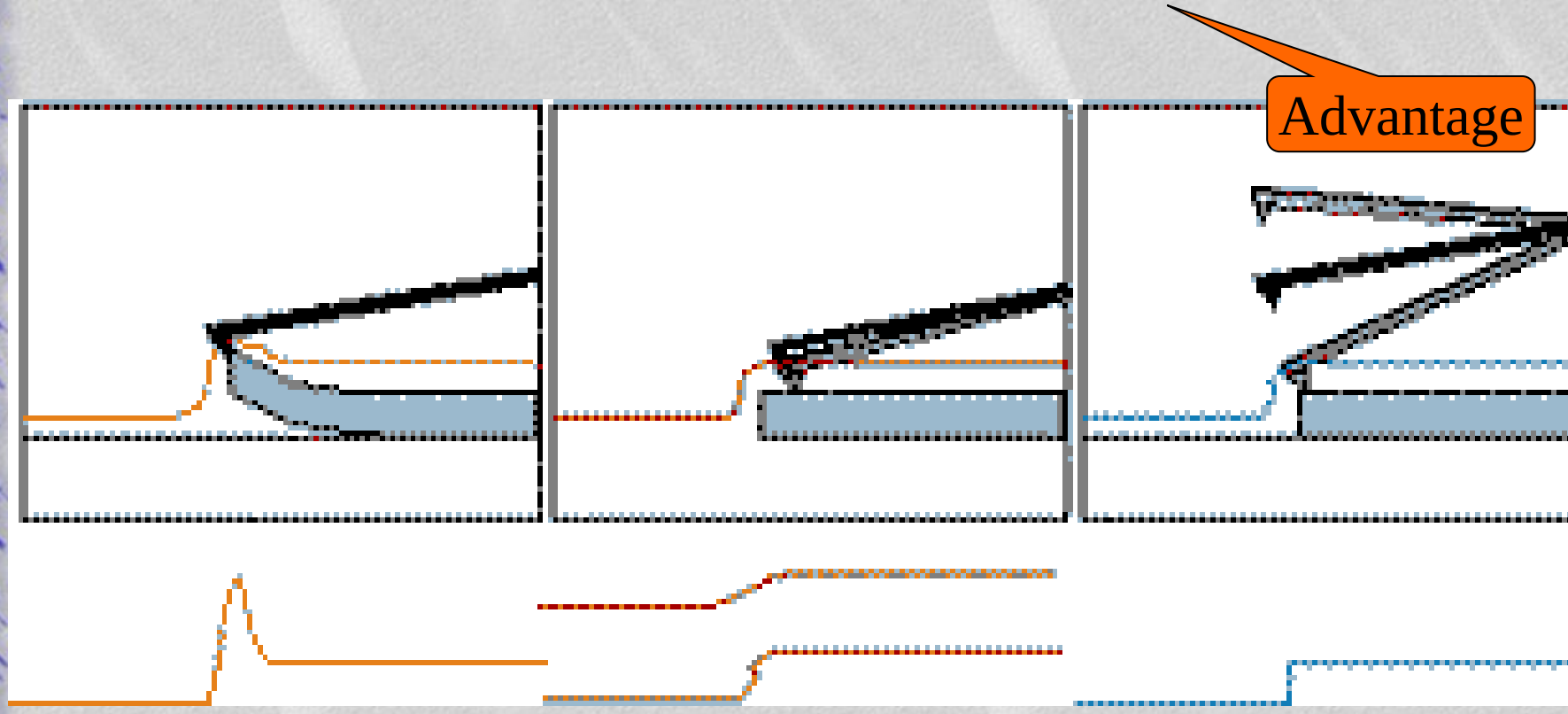


Tapping Mode

轻敲模式克服了前面两者的限制：**探针与样品接触可**

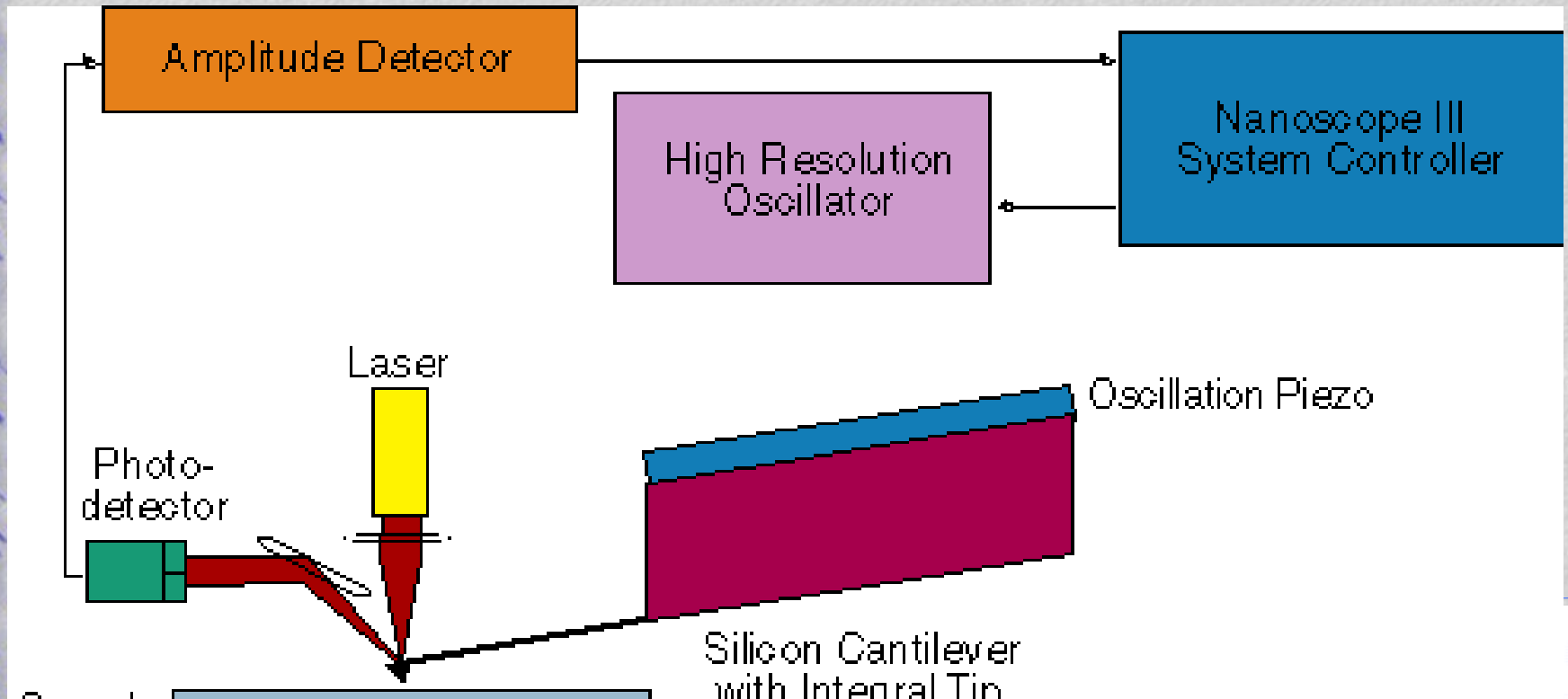
提供高测量分辨率； 扫描过程中将探针抬起有助于避

免探针被拖拽划过样品表面凸起时损伤。



优点：

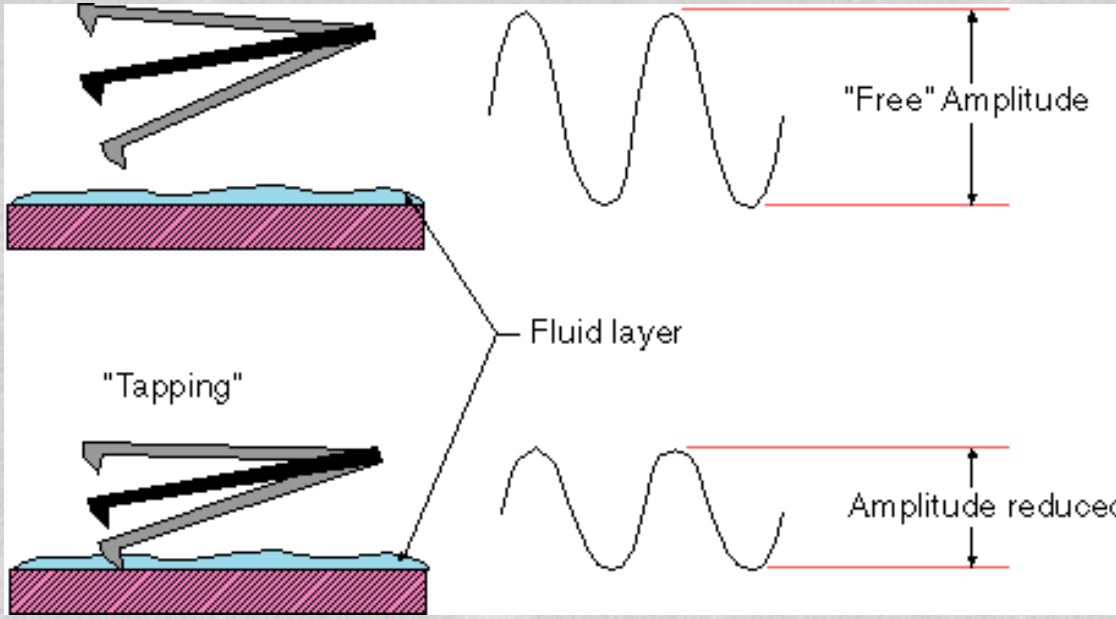
1. 施加的是垂直方向的力，无横向剪切力，故对样品表面无损伤。
2. 避免了探针与样品表面的粘连使探针损坏。
3. 足够大的震荡幅度克服了样品与探针间的粘附力。



Principle of Tapping Mode

- 扫描过程中，探针垂直振荡交替地接触并抬离表面，频率为 **50~500 KHz.**
- 振荡悬臂梁不断地接触表面，由于接触表面带来的能量损失造成震荡幅度减小。幅度减小量用来测算出表面的形貌变化量。

探测的是样品表面力引起的探针共振振幅的改变量。



Cantilevers

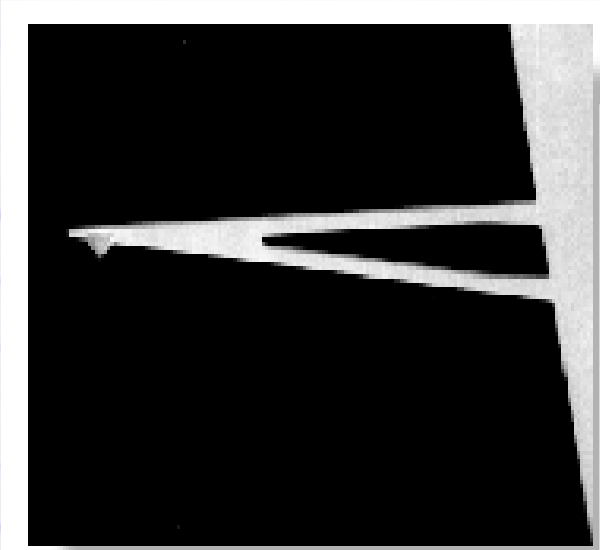
- 高度灵活的探针对样品施加一个向下的力。
- 弹性系数通常为 0.1 N/m
- 具有较高的共振频率

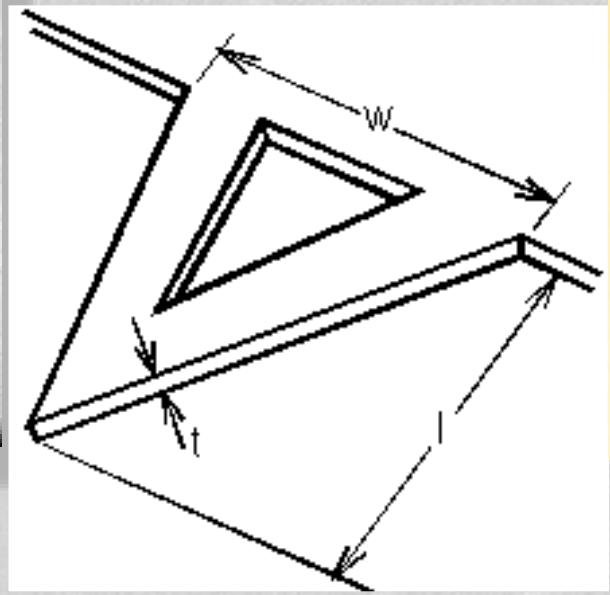
$$\text{resonant frequency} = \frac{1}{2\pi}\sqrt{\frac{\text{spring constant}}{\text{mass}}}$$

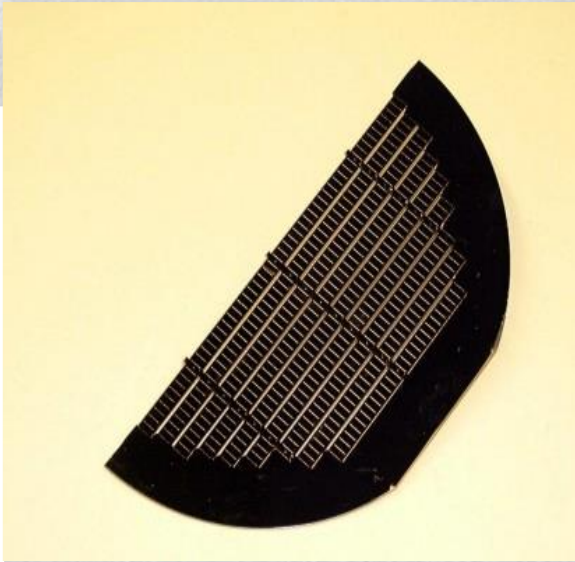
- 当悬臂梁质量较小时可兼具低弹性系数和高共振频率的特点。

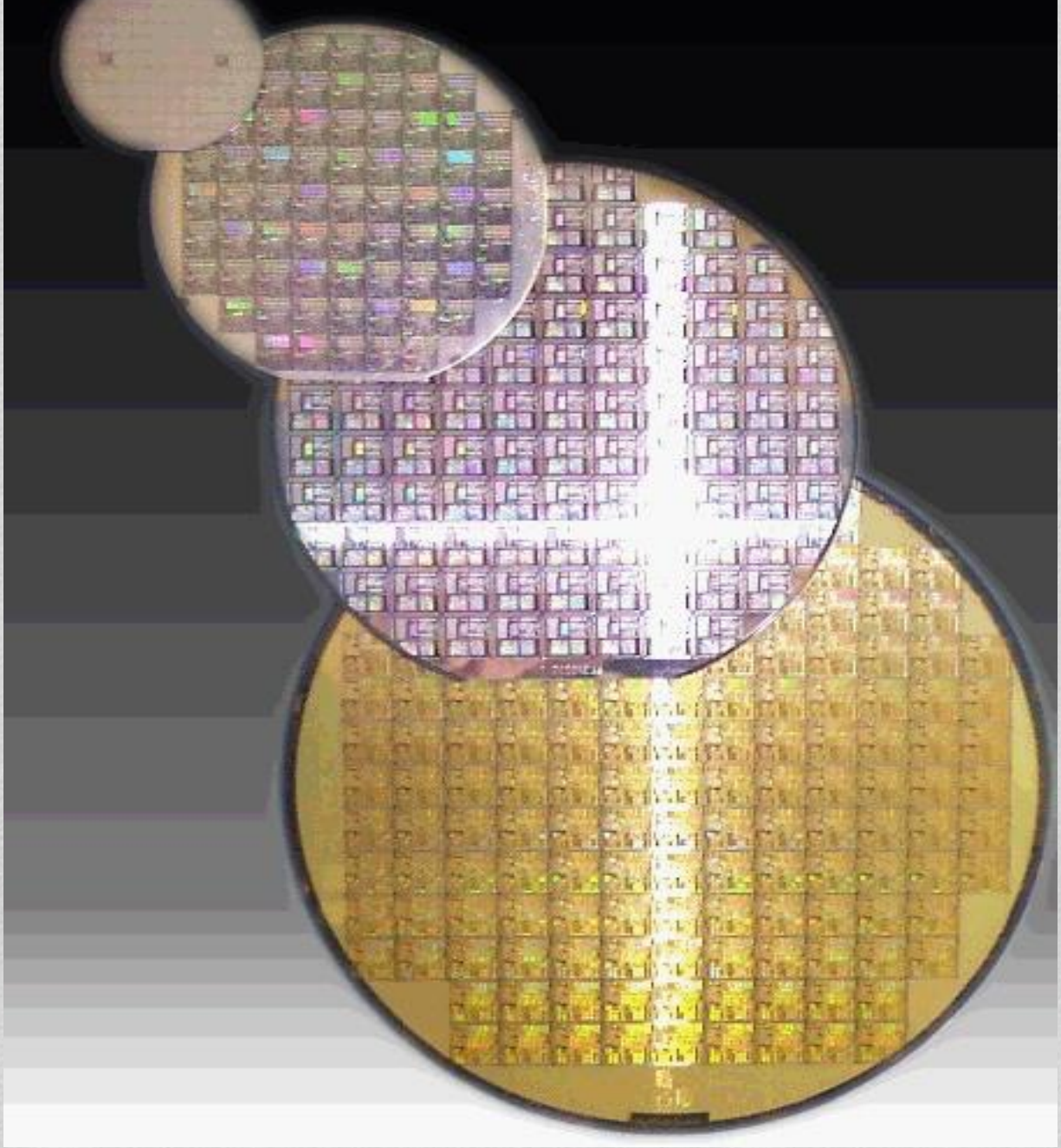
Cantilevers

- Typically measure 100 μm in length
- Silicon nitride (Si_3N_4)
- thin film of gold for reflectivity



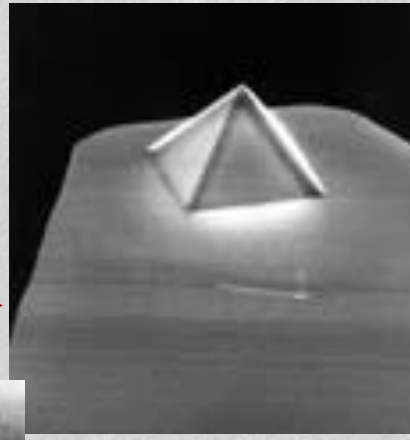




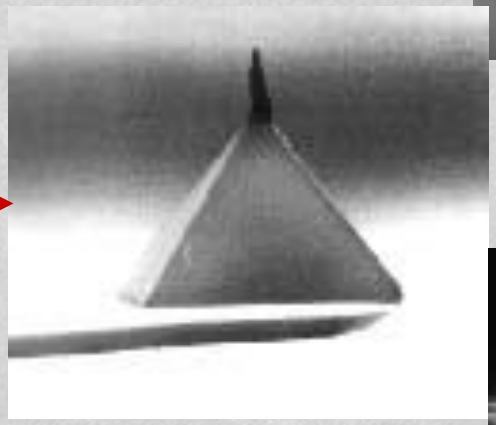


Tip types

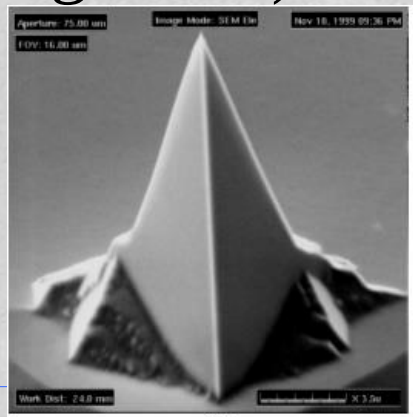
- normal tip (3 μm in tall) for contact mode



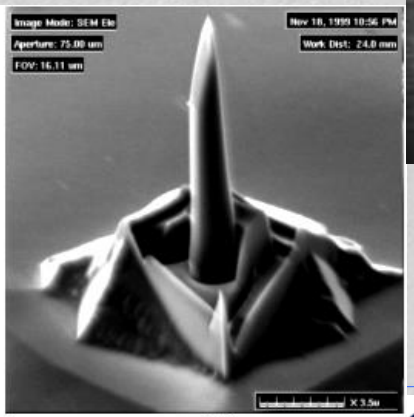
- super tip for tapping mode



- Ultralever (tapping mode)
- FIB trimmed tip



(a)



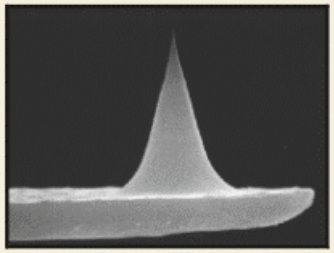
(b)

MAXIMUM RESOLUTION CANTILEVERS

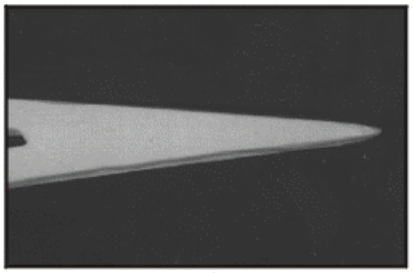
Tips

FEATURES:

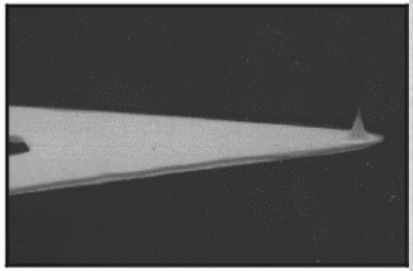
- Silicon cantilever with silicon conical tip.
- Tip height nominally 5 - 7 µm.
- Sharp, high aspect ratio tip.
- Typical radius of curvature 10 nm (60 nm for MFM Ultralevers).
- Recommended for non-contact mode.
- Gold coated for high reflectivity.
- Boron doped silicon, 0.001Ω/cm.
- Wide range of spring constants.
- Four different cantilevers on every chip.
- Compatible with any major brand AFM.
- Also available are MFM Ultralevers coated with cobalt for Magnetic Force Microscopy, and Tipless Ultralevers for special applications.



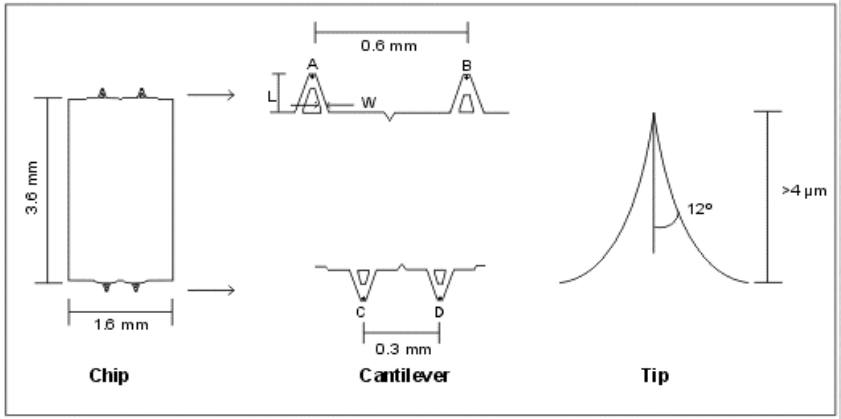
ThermoMicroscopes Ultralevers are high quality cantilevers ideal for most topographic applications including, metrology, high resolution, non-contact or intermittant contact imaging, and IC measurements. MFM Ultralevers are ideal for high resolution MFM imaging on "hard" magnetic surfaces. Tipless cantilevers are a perfect platform for the reasercher who wants to make a tip out of a custom material or other chemical applications.



Tipless Ultralever



MFM Ultralever



Typical mechanical characteristics

Cantilever type	A	B	C	D	A	B	C	D
Standard mode of operation	Contact				Intermittent and non-contact			
Cantilever length	180 µm	180 µm	85 µm	85 µm	180 µm	180 µm	85 µm	85 µm
Cantilever width	25 µm	38 µm	18 µm	28 µm	25 µm	38 µm	18 µm	28 µm
Cantilever thickness	1 µm	1 µm	1 µm	1 µm	2 µm	2 µm	2 µm	2 µm
Force Constant	0.26 N/m	0.40N/m	1.6 N/m	2.1 N/m	2.1 N/m	3.2 N/m	13 N/m	17 N/m

Tips

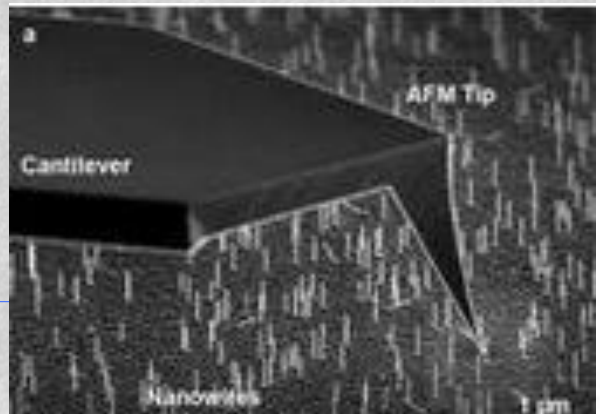
Resolution issues

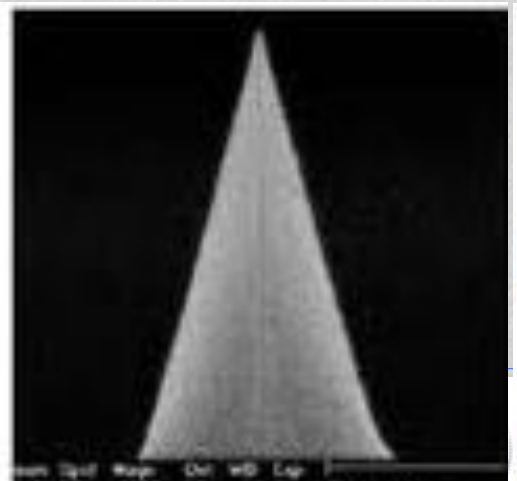
测量结果的横向分辨率取决于下面两个因素:

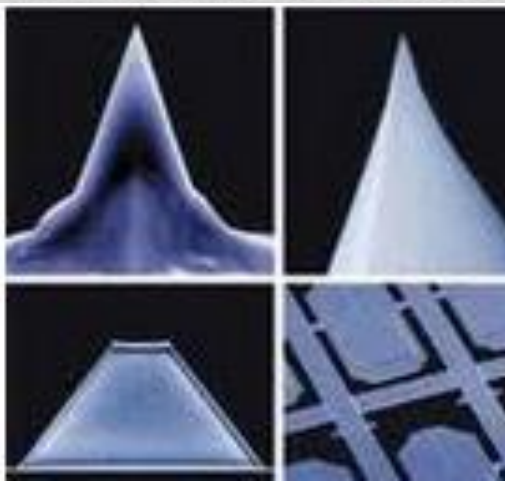
- 扫描步距;
- 针尖的最小曲率半径

Tips Manufacture : Silicon

- 锥形硅探针**制作：湿法化学腐蚀Si**
- 具有较高深宽比的锥形探针可测量较深的沟槽底部，但比金字塔或四面体形探针更易折断。
- 硅探针可掺杂其它元素形成导电回路，适用于控制探针与样品之间的偏置电压，防止不需要的电荷在探针表面堆积。

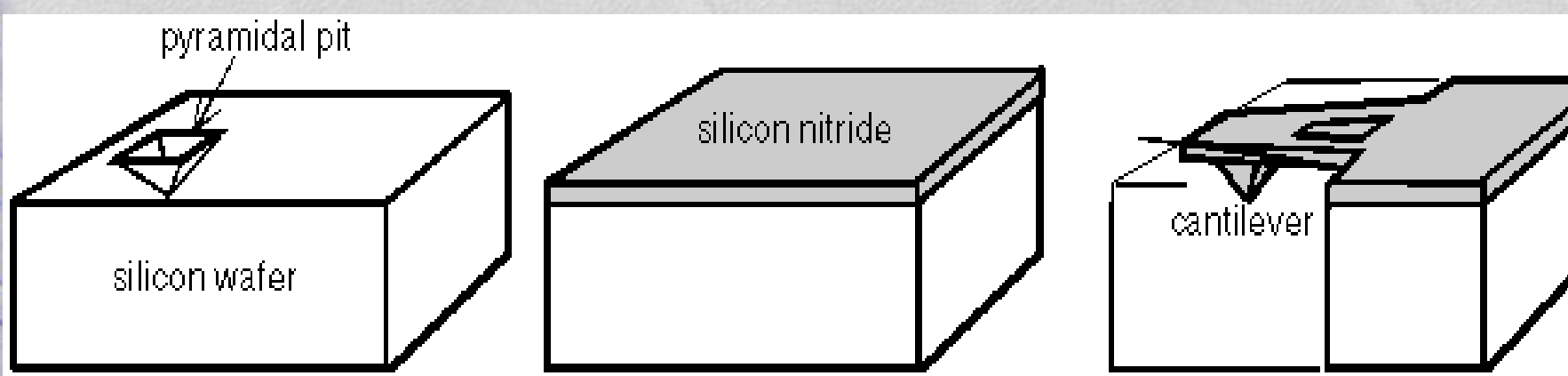






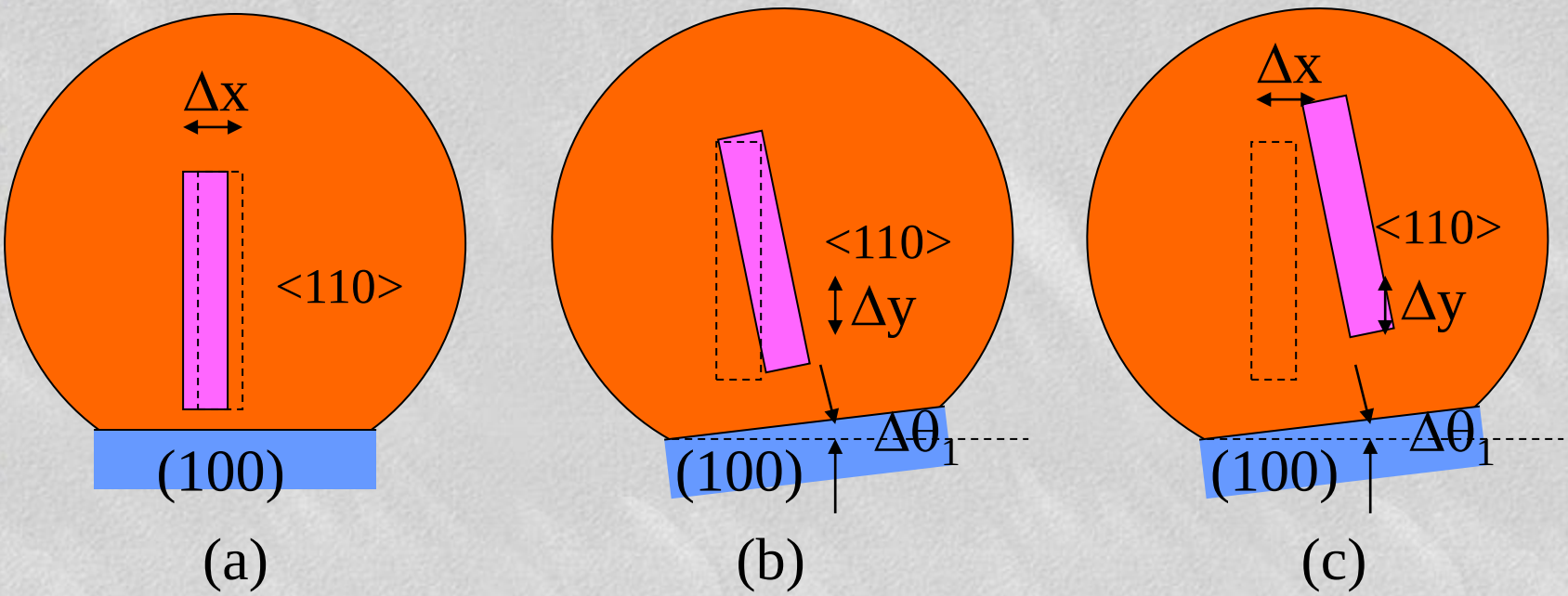
Tips Manufacture : Silicon Nitride

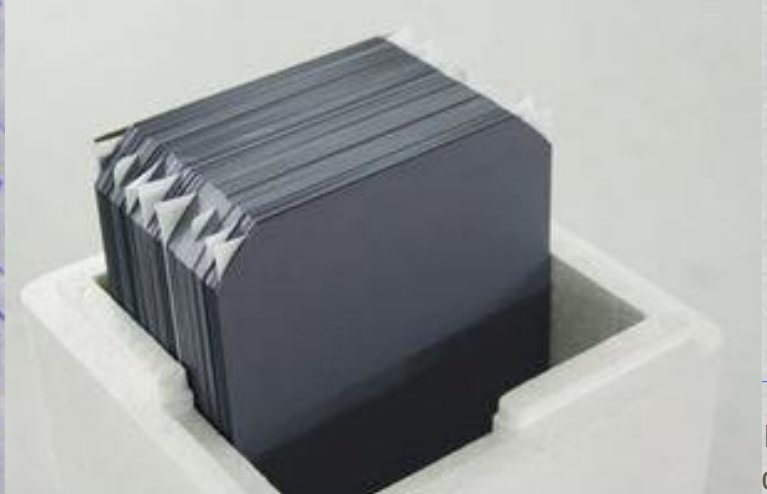
- 氮化硅探针由沉积在多晶硅表面的Si_3N_4经化学腐蚀而成。
- 该方法可制作金字塔或四面体形探针。
- 氮化硅探针的深宽比由被腐蚀材料硅的晶体学结构决定。



Mask aligner accuracy: x±0.5μm, y±0.5μm,

$\Delta\theta_1 = \pm 0.477$ (for 2” wafer)





Probe Microscopy.
qi Fu, UESTC

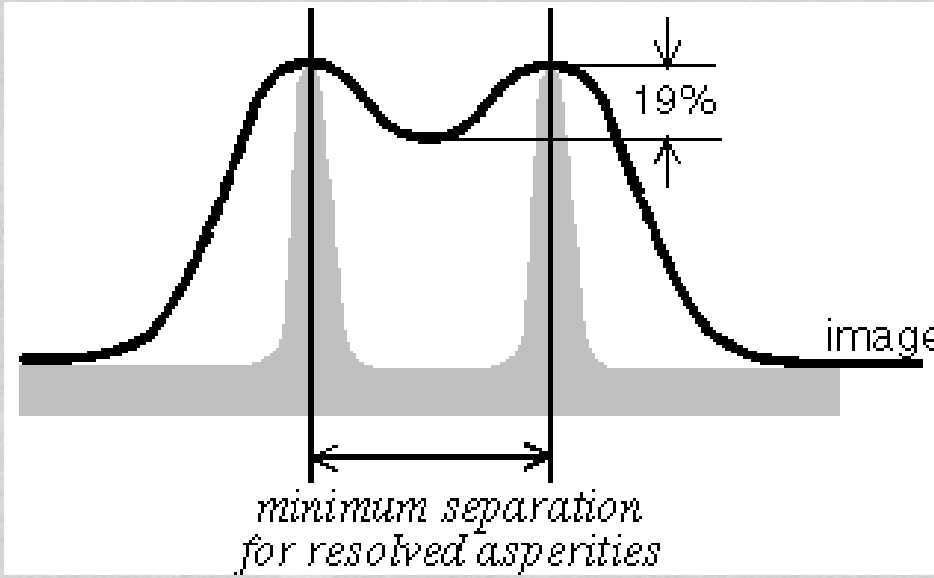
Si anisotropic etching Ratio

	KOH	TMAH
R_{110}/R_{100}	2	
R_{100}/R_{111}	400	12.5~50
R_{110}/R_{111}	600	

Data is cited from “P.Rai-Choudhury Edited, Handbook of Microlithograpgy, Micromachining, and Microfabrication. SPIE Optical Engineering Press, 1997.”

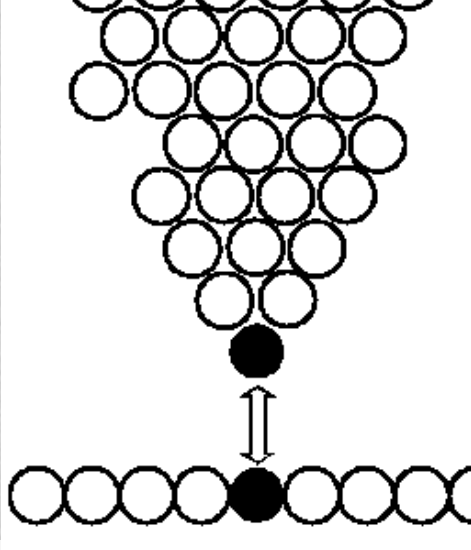
Tips: Resolution

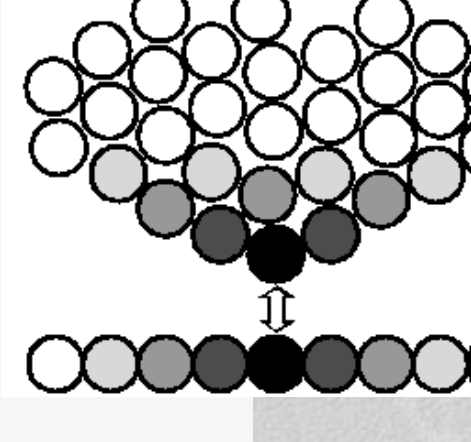
- 最尖的商业化探针其尖部半径只有5纳米。由于探针与样品之间相互作用的区域属于探针半径的一部分，这类探针可提供1~2纳米的分辨率。因此，AFM扫描成像≥1μm×1μm的分辨率往往不是由针尖半径决定，而是由扫描步距决定。

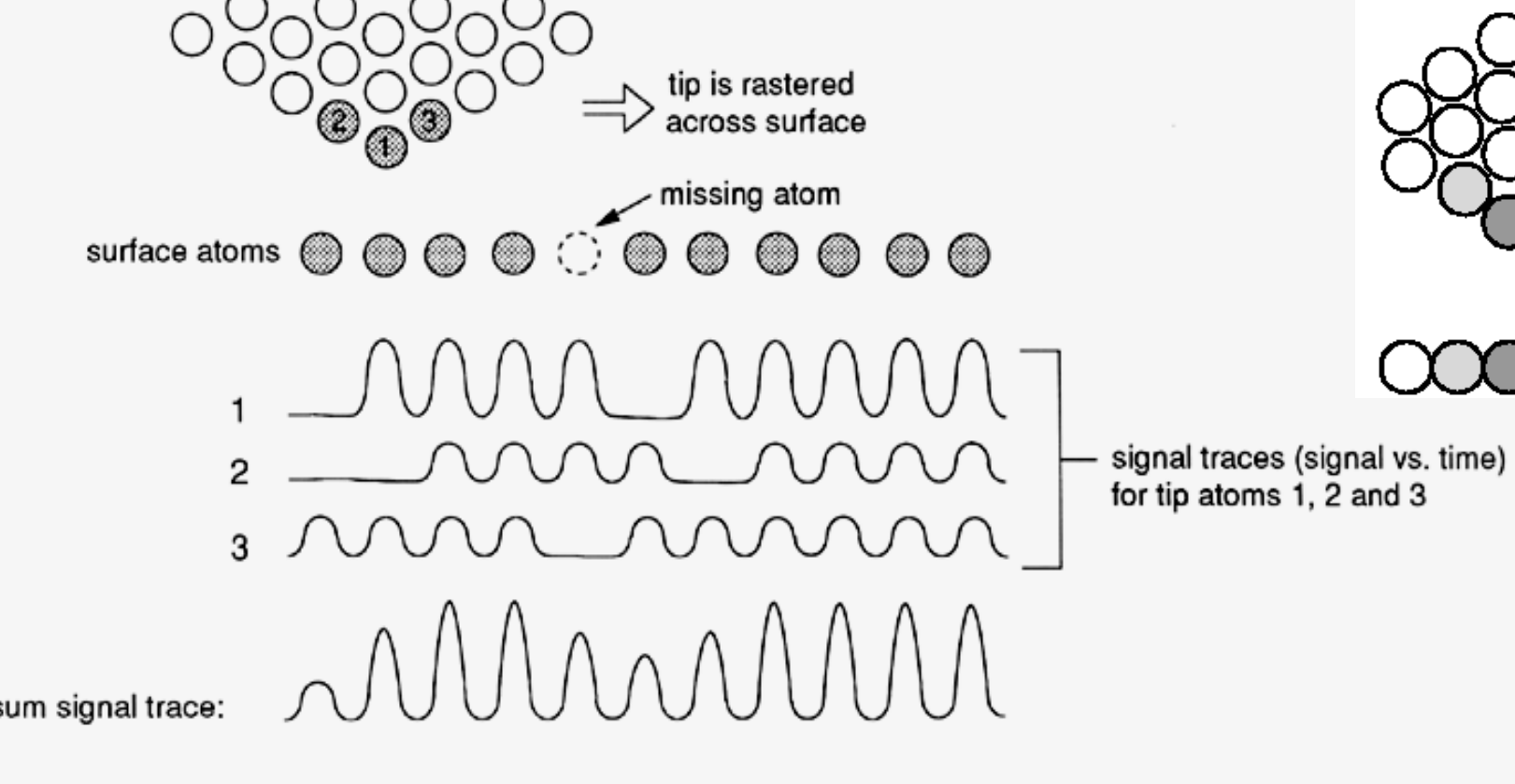


Tips

Atomic Resolution?







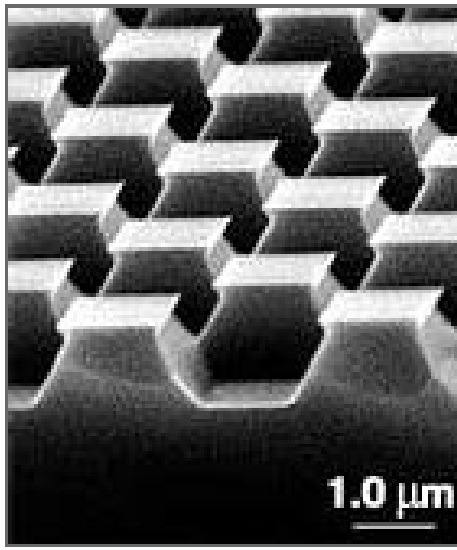
Calibration of AFM

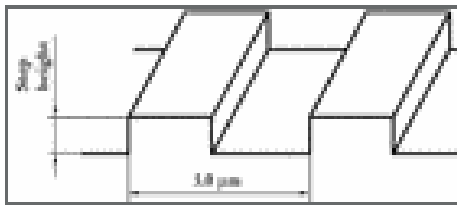
Method I

Calibration gratings

AFM calibration gratings

- Porous Aluminium
- TGX (lateral calibration)
- TGG (triangular steps for lateral calibration and tip characterization)
- TGZ (steps 20 nm up to 1500 nm)
- TGF (trapezoidal steps, edge angle calibration)
- HOPG (graphite)





Application

Calibration of AFM and STM

Verification of tip sharpness and geometry

Calibration of AFM

Method II

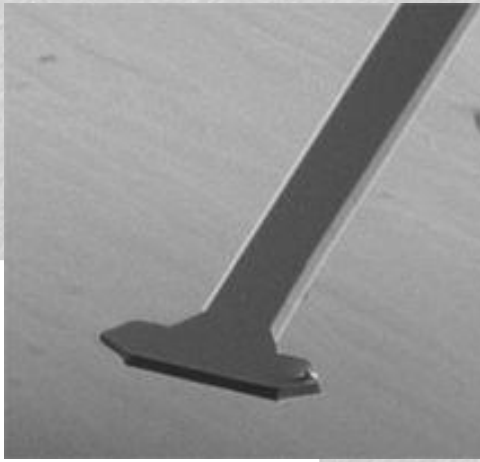
Other types SPM

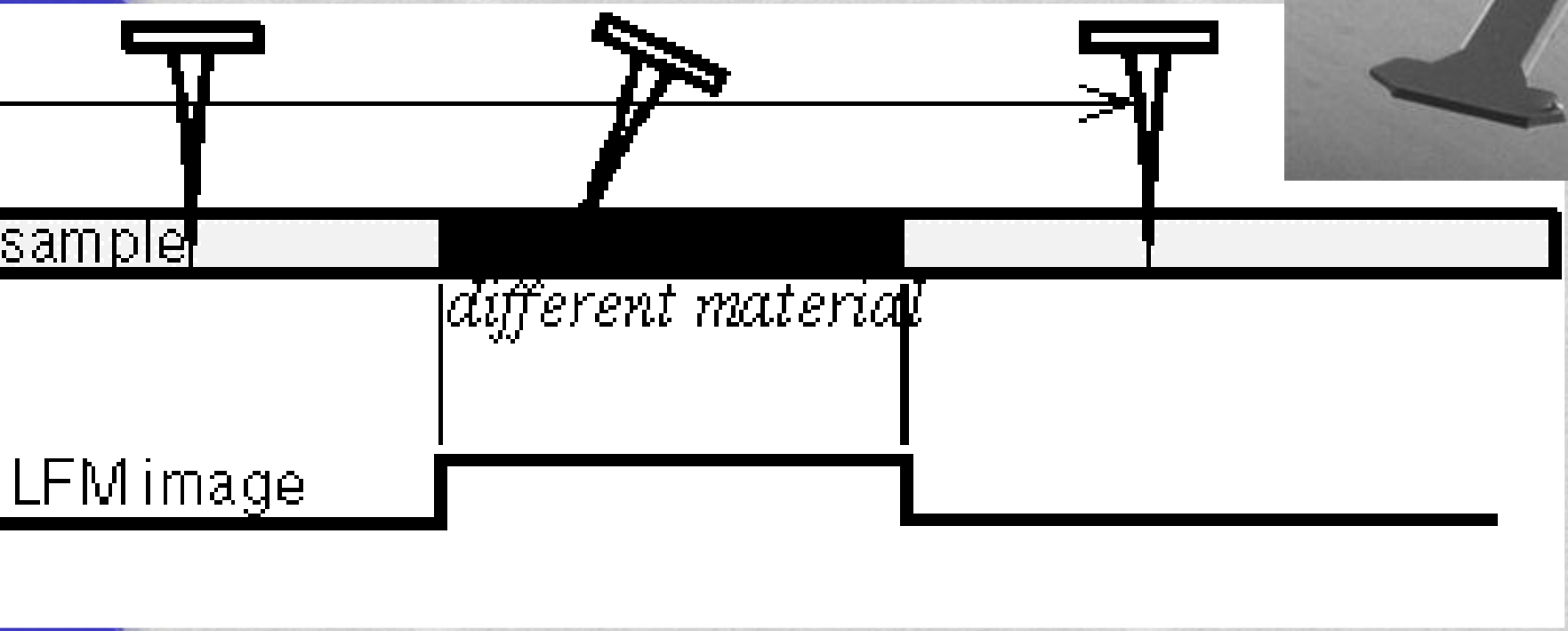
- Lateral force microscopy
- Magnetic force microscopy
- Electrostatic force microscopy
- Phase detection microscopy

SPM Types

Lateral Force Microscopy (LFM)

- 测量侧向变形(扭曲变形)
- 适用于
  - 测量材料表面摩擦的改变

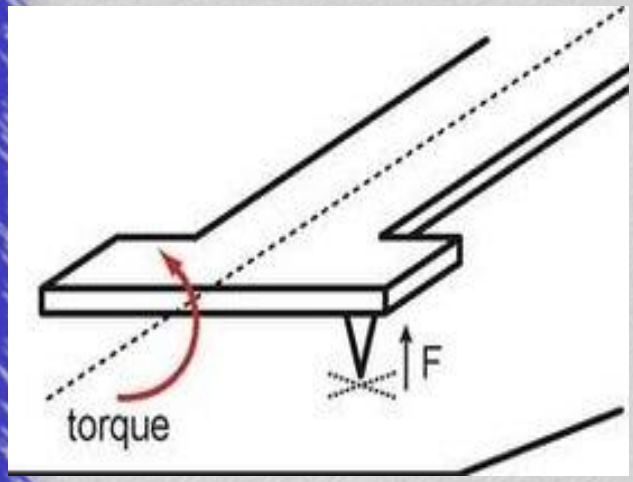


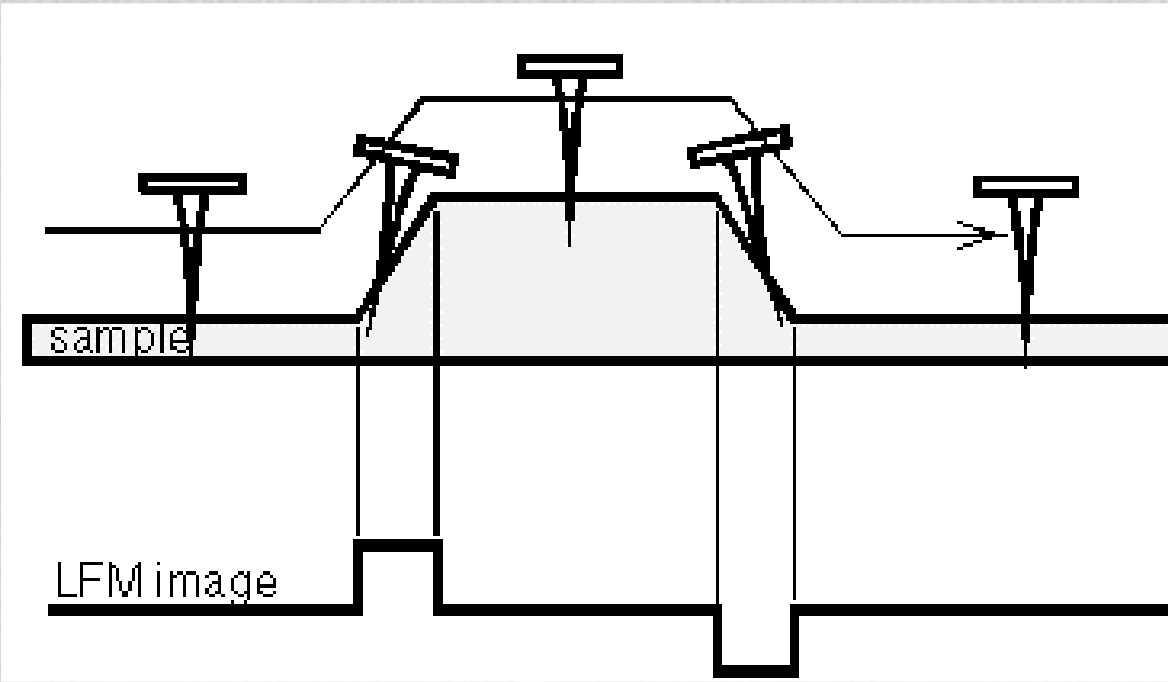


SPM Types

侧向力显微镜 (LFM)

- 样品具有坡度变化的情况；
- 为了确定表面摩擦和坡度变化，LFM和AFM结果图都需要。

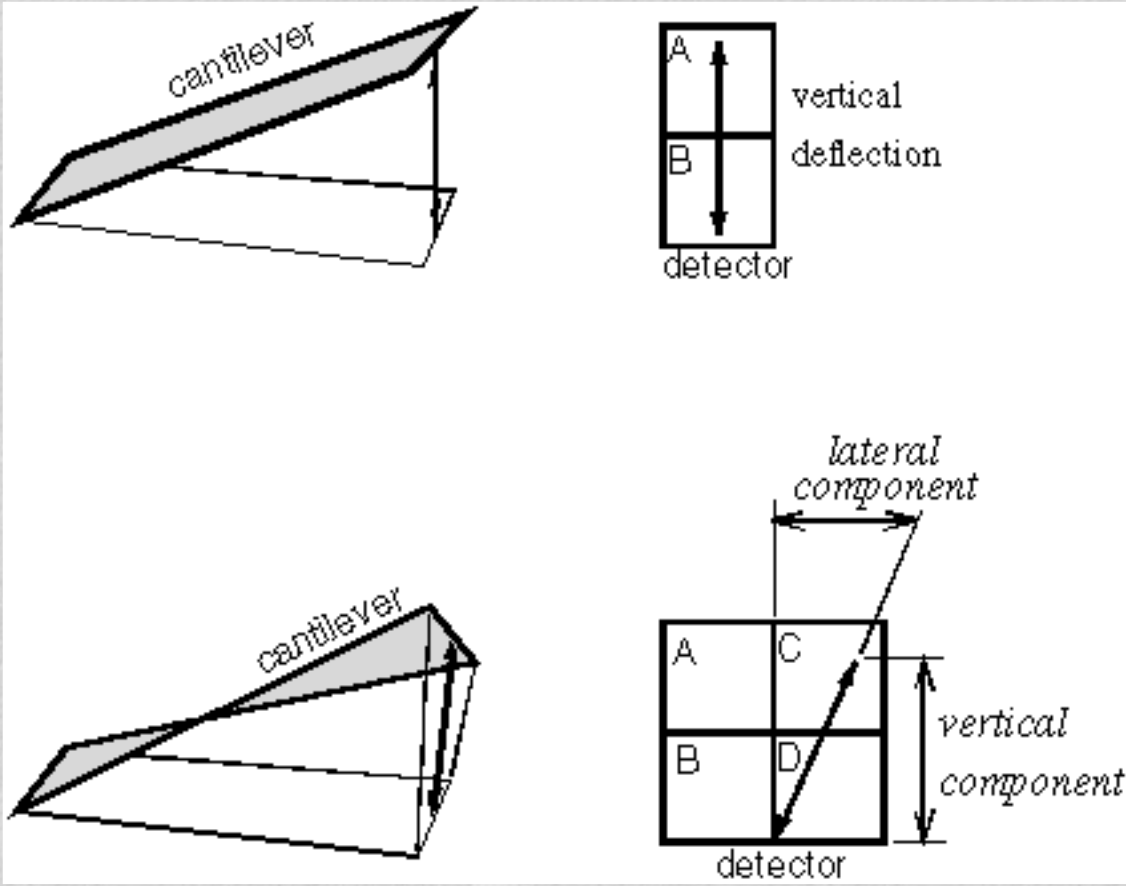




SPM Types

侧向力显微镜 **(LFM)**

- 采用位置敏感的四象限光探测器 (PSPD)
  - 分四个象限
  - 可确定
    - 侧向
    - 垂直方向

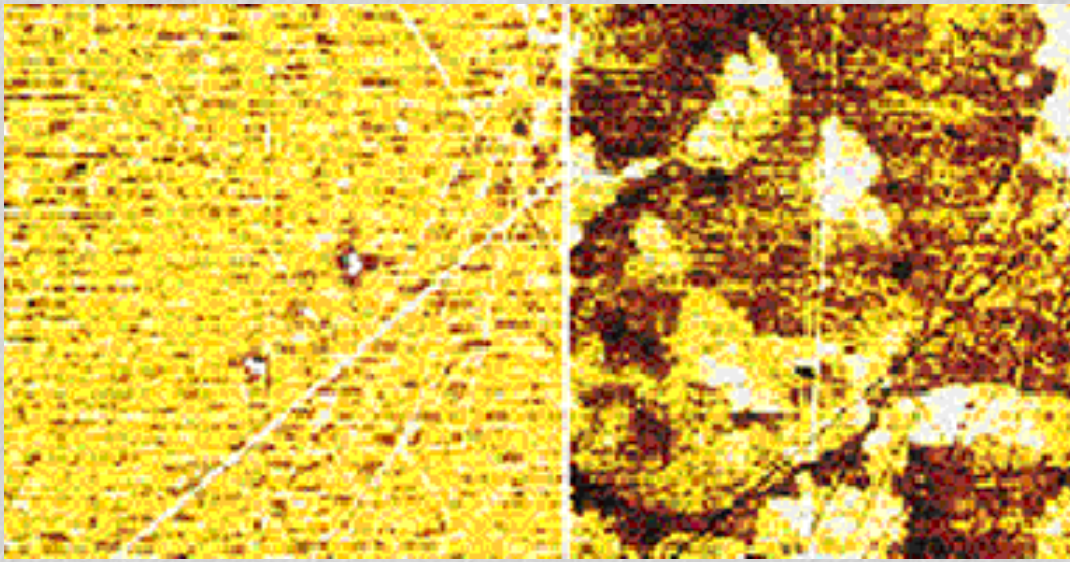


SPM Types

侧向力显微镜 (LFM)

- 适用于识别表面材料组成的差别，样品具有相同的形貌轮廓、不同的摩擦特性。
- 影响结果的因素：
  - 粗糙的表面轮廓
  - 表面污染

AFM LFM



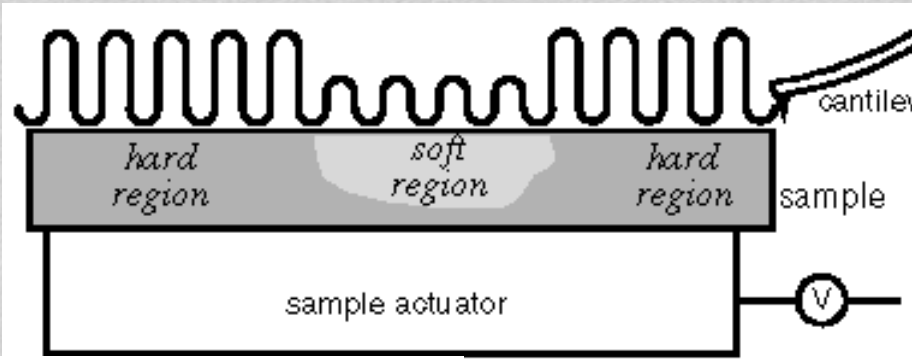
抛光的多晶碳化硅表面

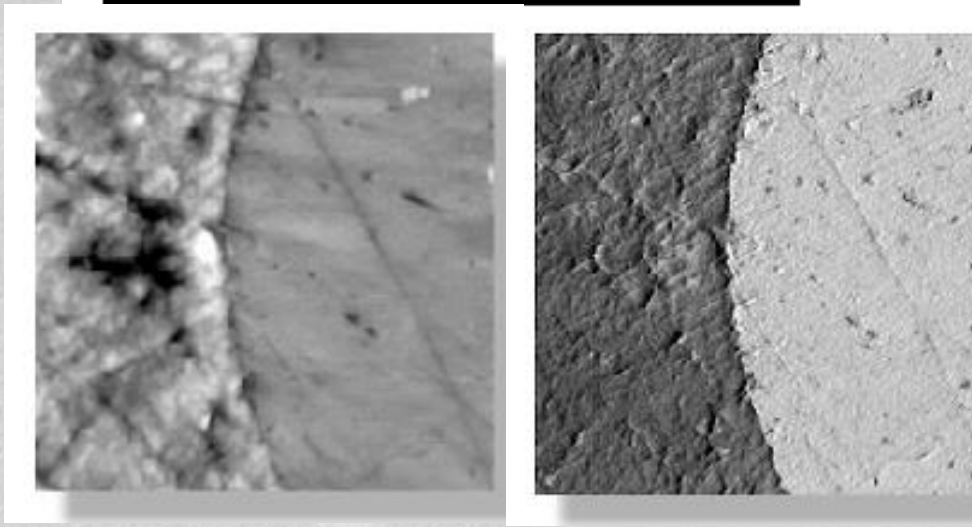
SPM Types

Force Modulation Microscopy (FMM)

力调制显微镜

- 测量表面刚度和弹性差别



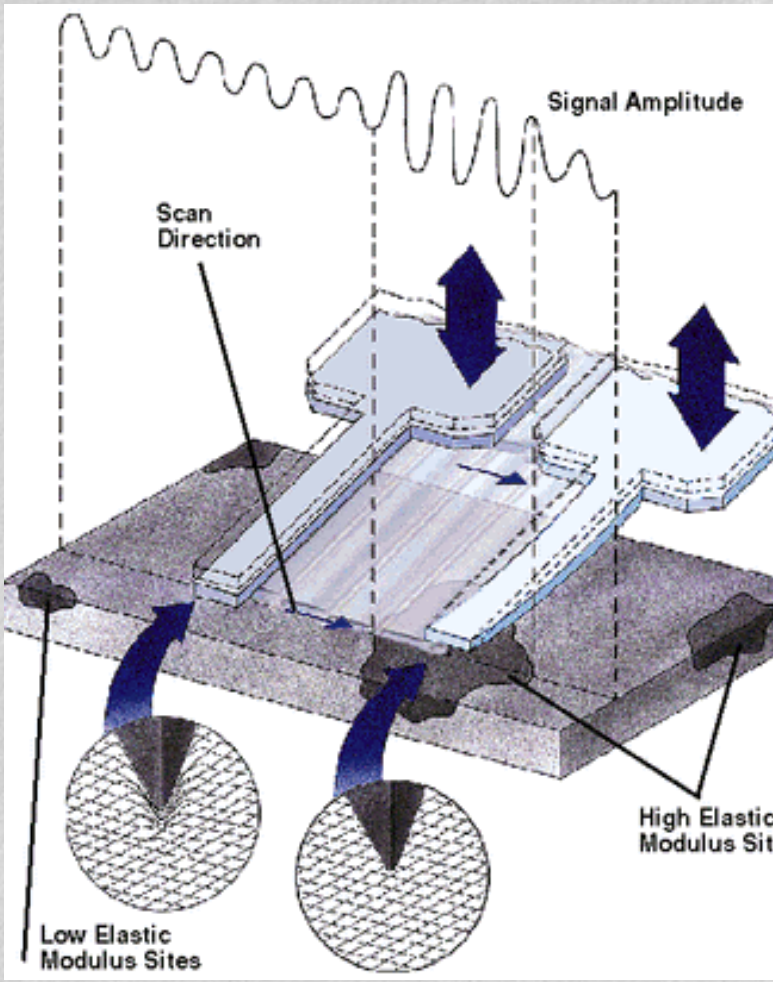


SPM Types

Force Modulation Microscopy (FMM)

力调制显微镜

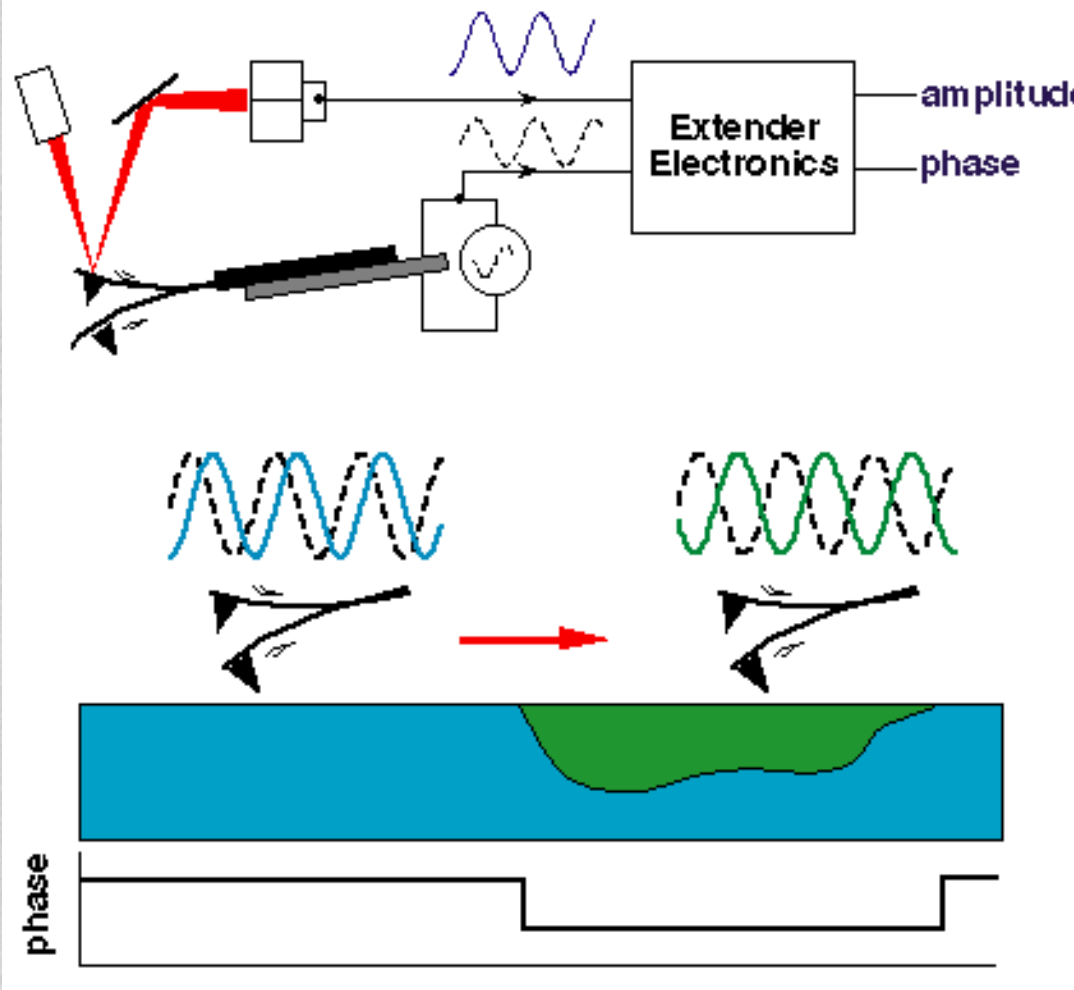
- 缺点
  - 高频调制可激发许多不希望出现的扫描头机械共振
  - 会降低成像质量



SPM Types

Phase Detection Microscopy

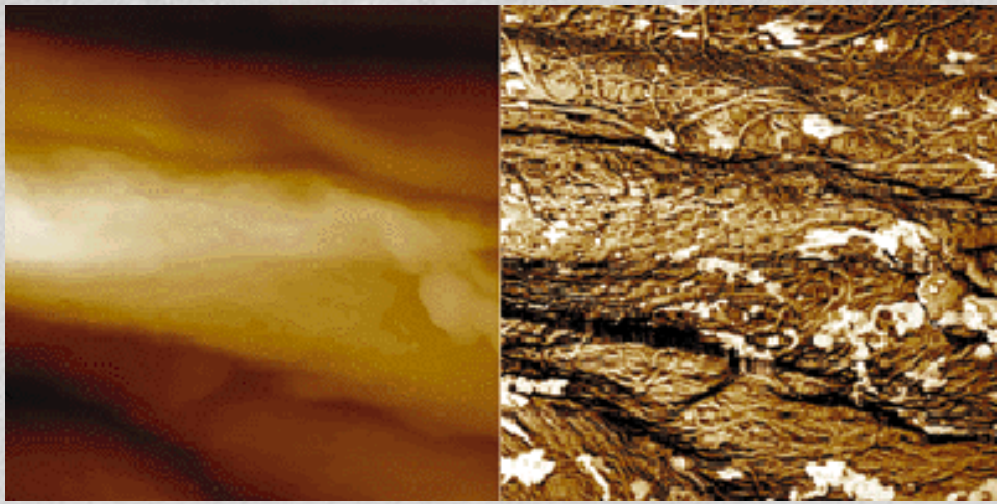
- Map variations
  - elasticity
  - adhesion
  - Friction
- Known as Phase Imaging

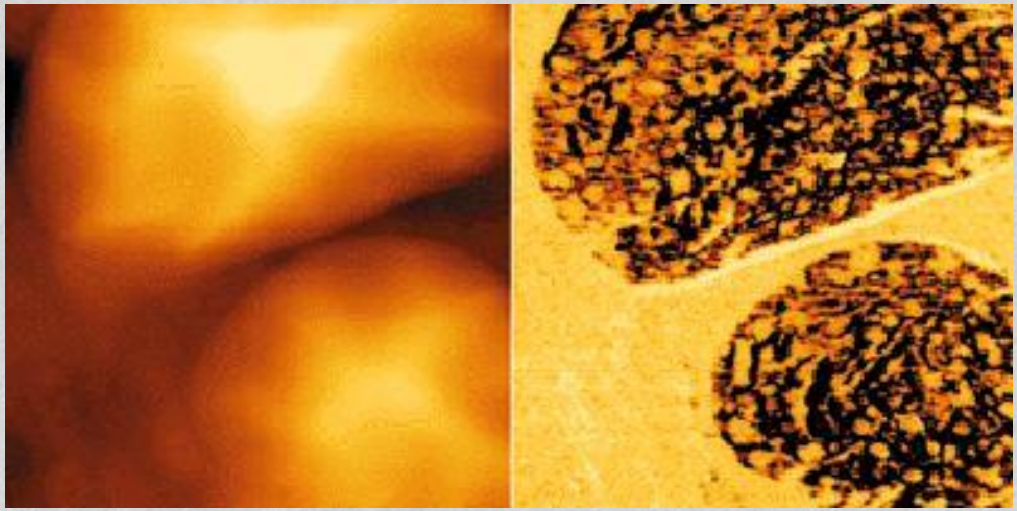


SPM Types

Phase Detection Microscopy

- 位相图适用于识别复合材料各组分的位相分布





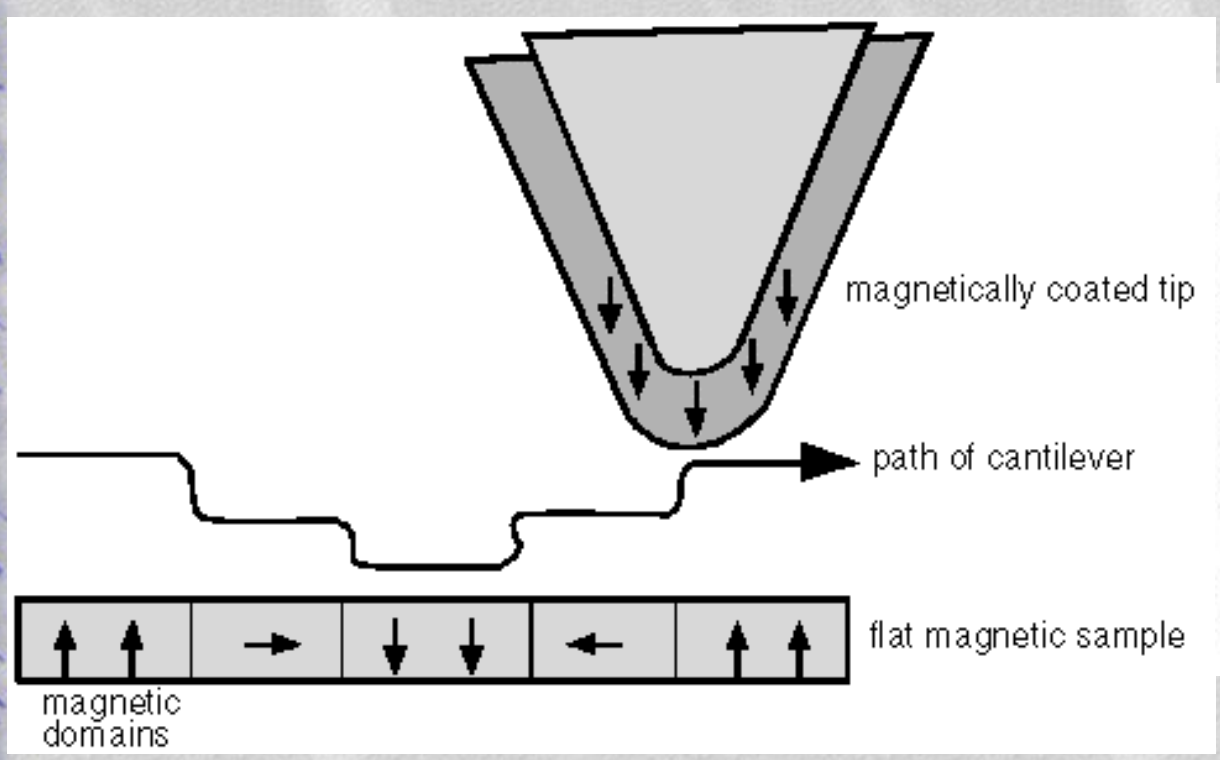
- 图中两聚合物团簇的位相结构由位相图清晰地显示出来。

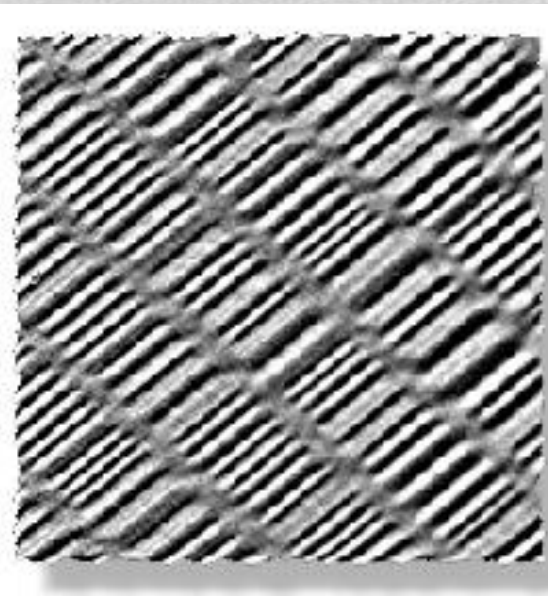
SPM Types

Magnetic Force Microscopy

磁力显微镜

- Magnetic force microscopy (MFM) 可显示样品表面磁力的空间变化。

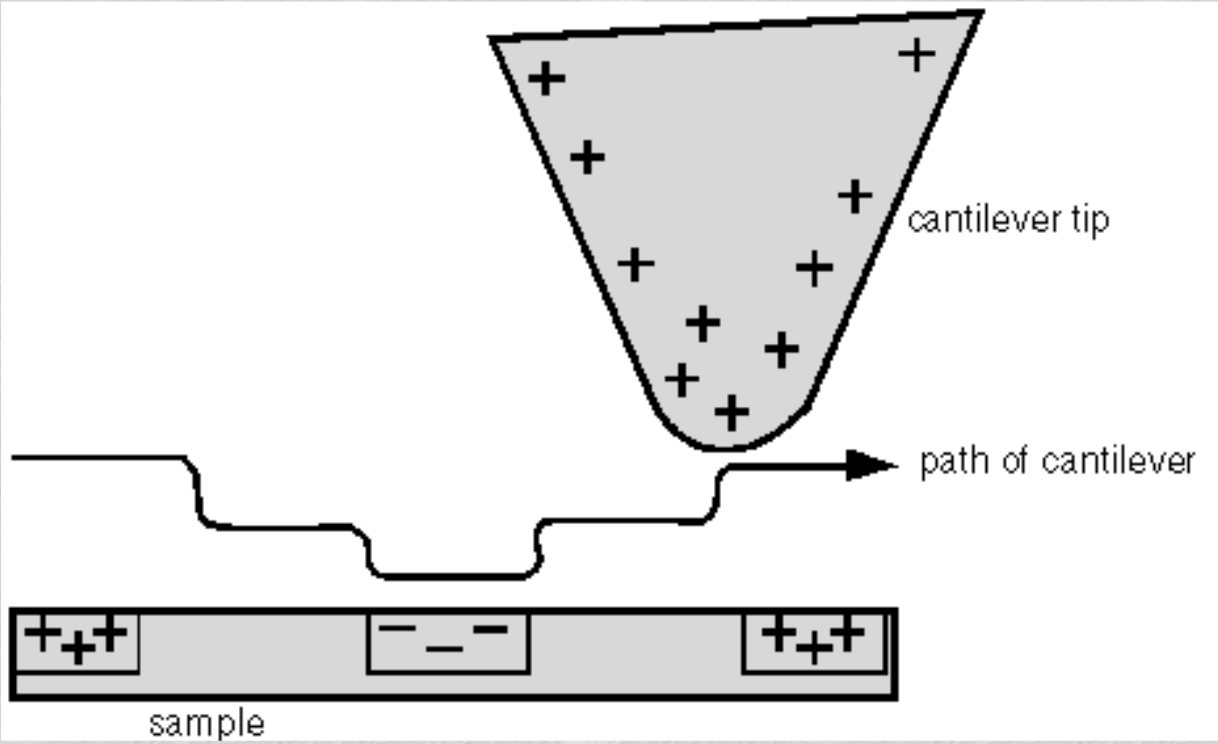




SPM Types

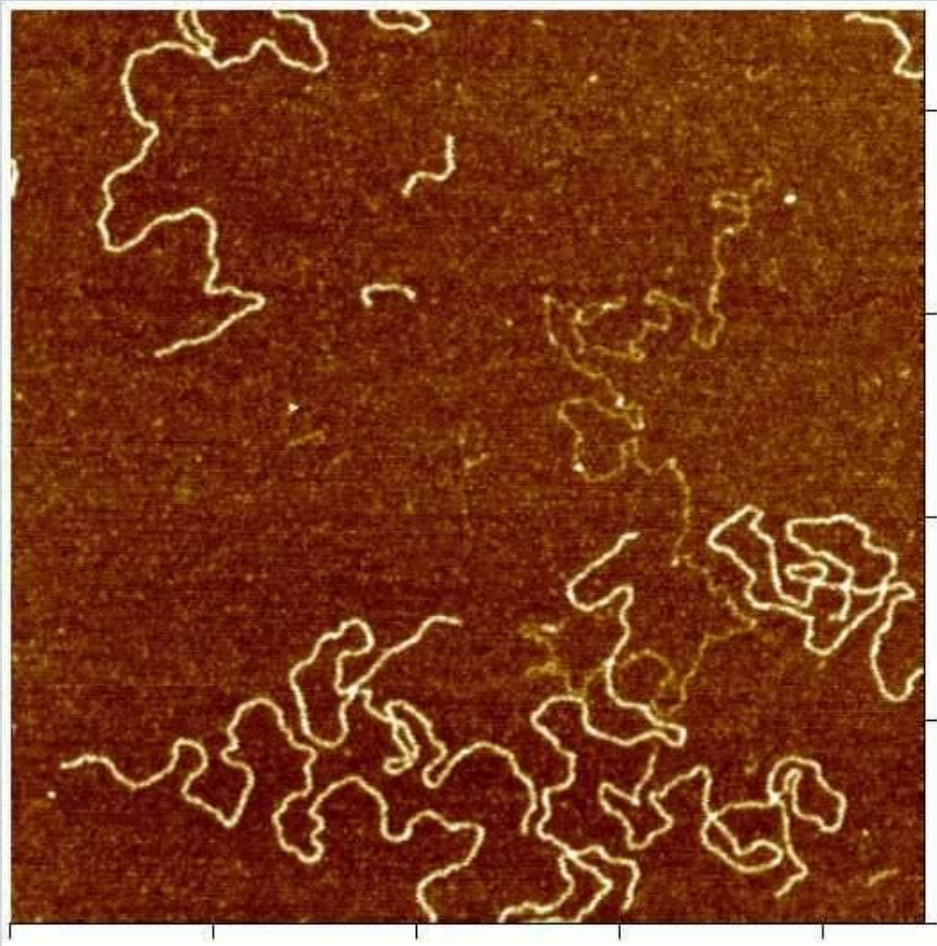
Electrostatic Force Microscopy

- 静电力显微镜结果显示的是样品表面局部电荷分布范围，类似于MFM显示结果。
- EFM用于研究表面电荷密度的空间变化，例如：器件开关时电路中的静电场分布。



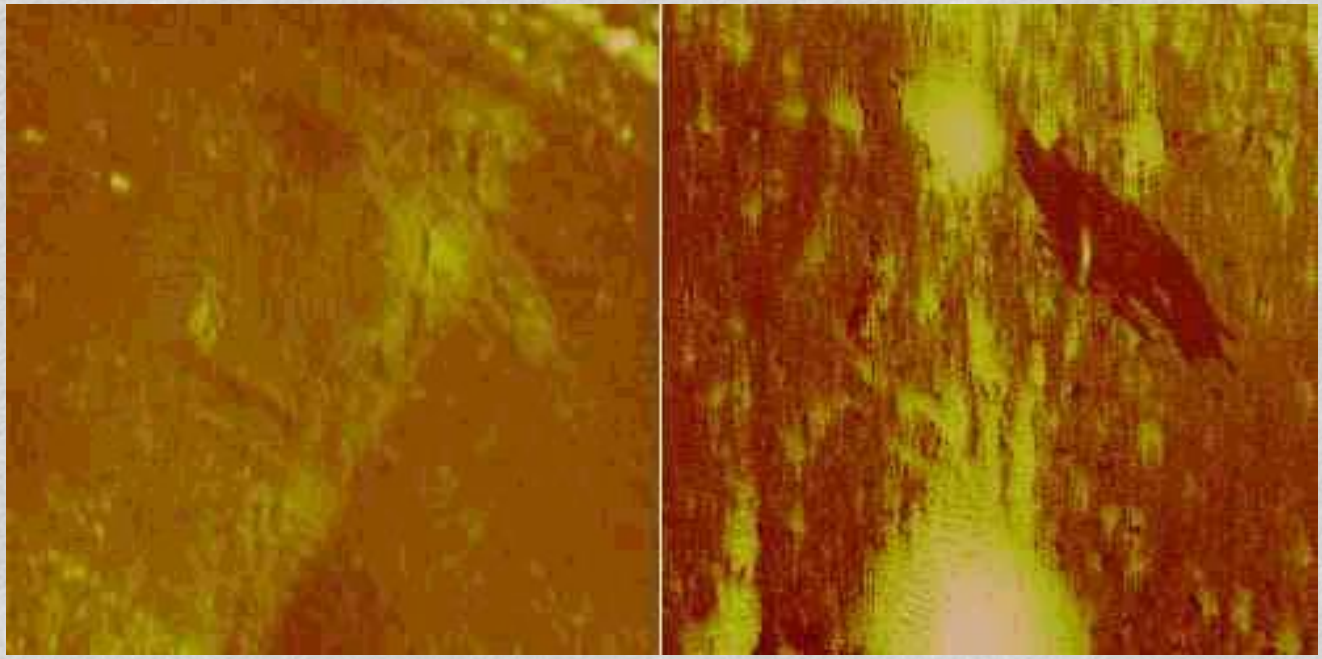
Applications

- *Single-stranded DNA seen as the 'ghostly' strand surrounded by duplex DNA. Sample pRH3 (6.16kb) plasmid DNA, heated to melt duples then adsorbed to poly-l-lysine mice* ***Scan size 900 x 900 nm.***



Applications

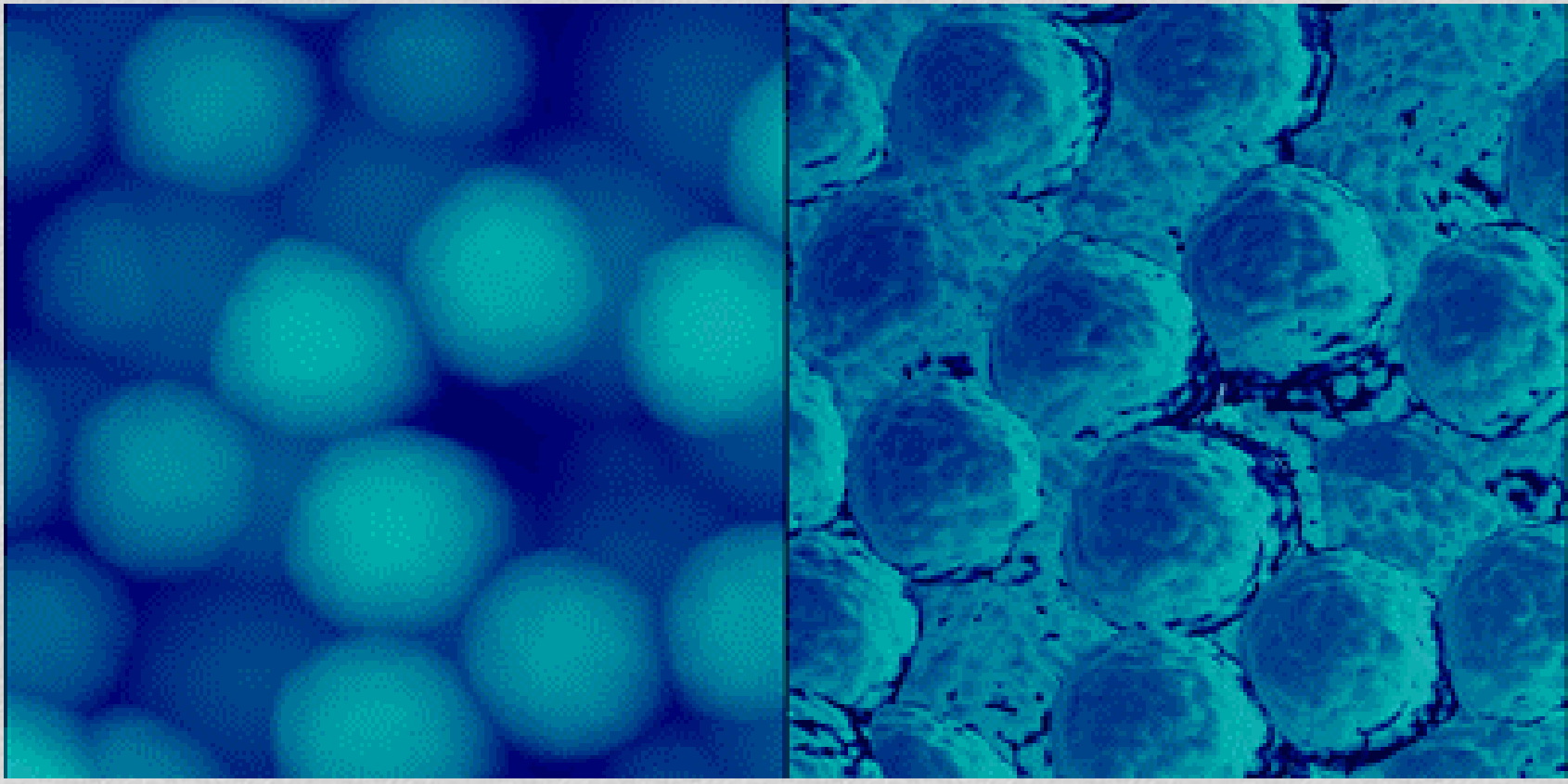
- *Contact mode height and friction image of oil droplets (fingerprint residue) on glass. Contact mode height and friction image of oil droplets (fingerprint residue) on glass.*



Height and friction image of oil droplets

Applications

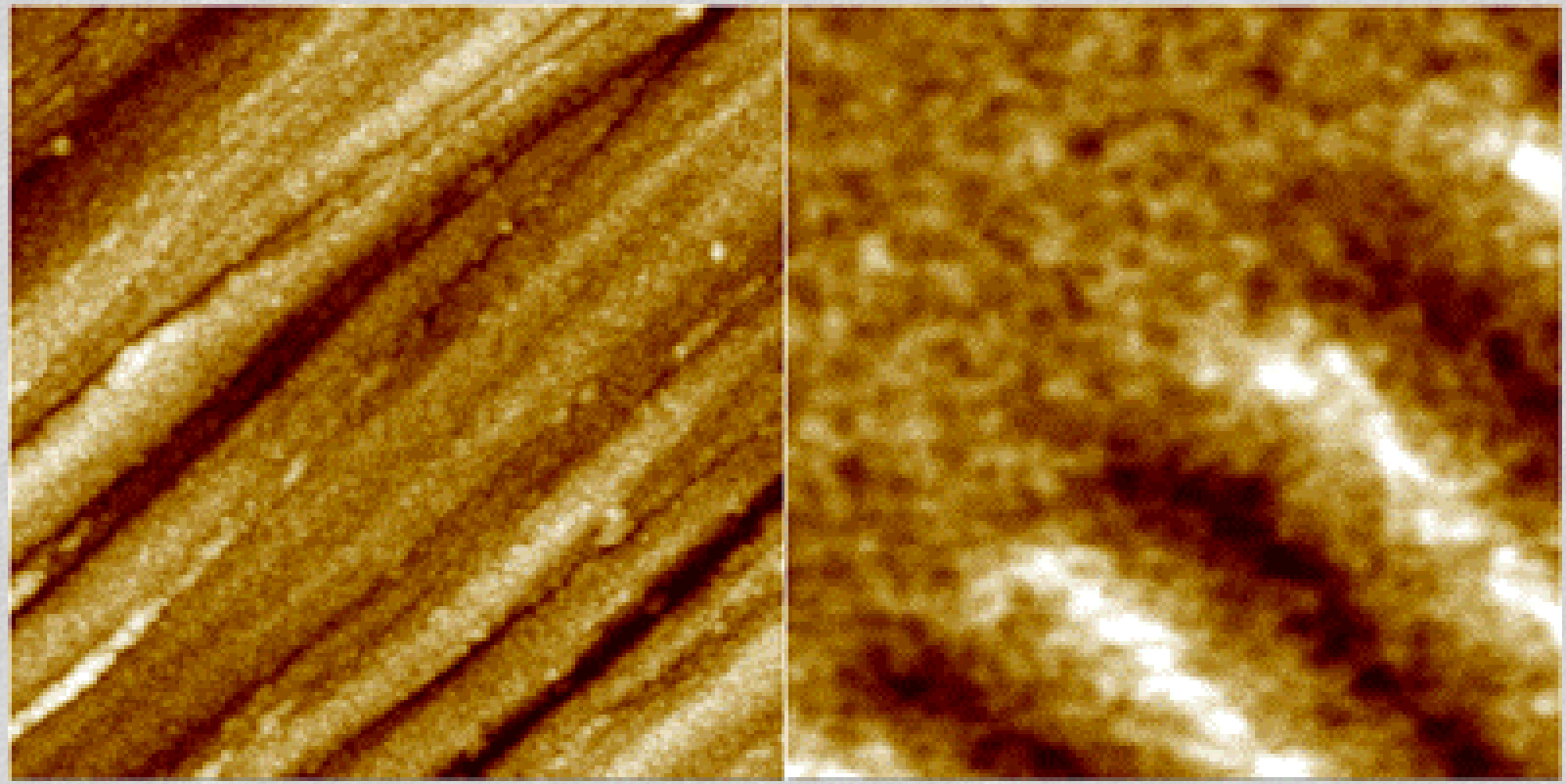
- *AFM height (left) and phase (right) images of poly (methylmethacrylate)*



Topography Phase

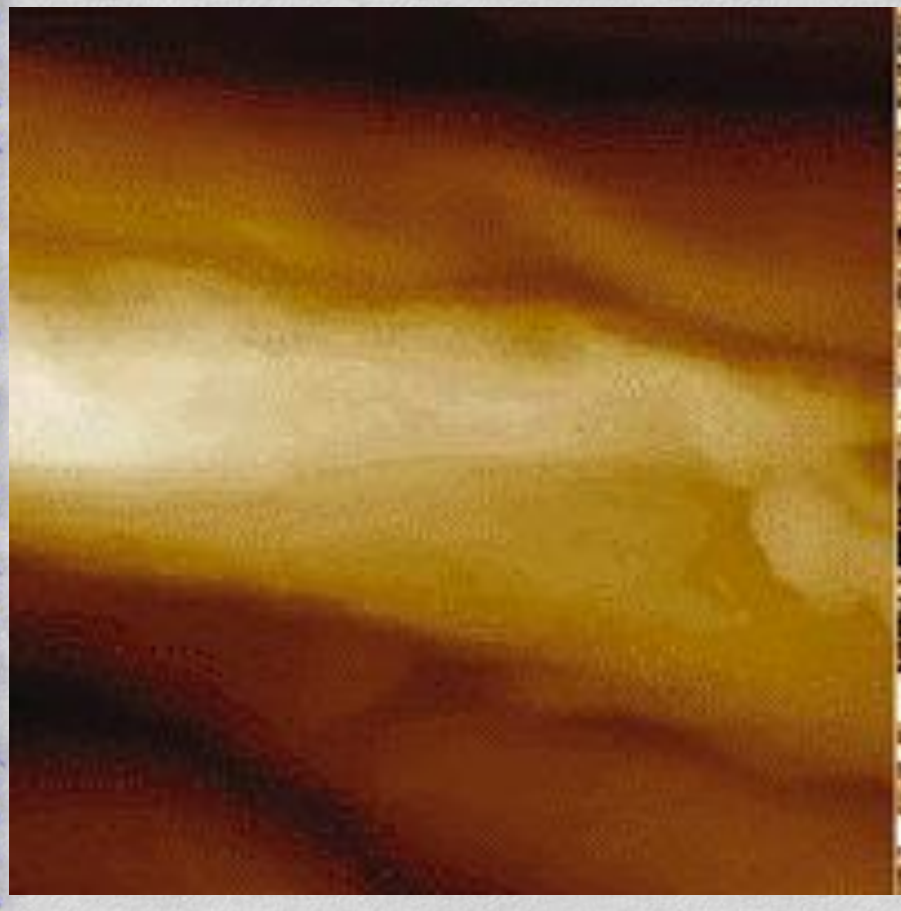
Applications

- *Track edge on a commercial hard disk. The **surface topography (left)** shows texture and 80nm-scale film morphology. The **magnetic force gradient image (right)**,. 5μm scan.*

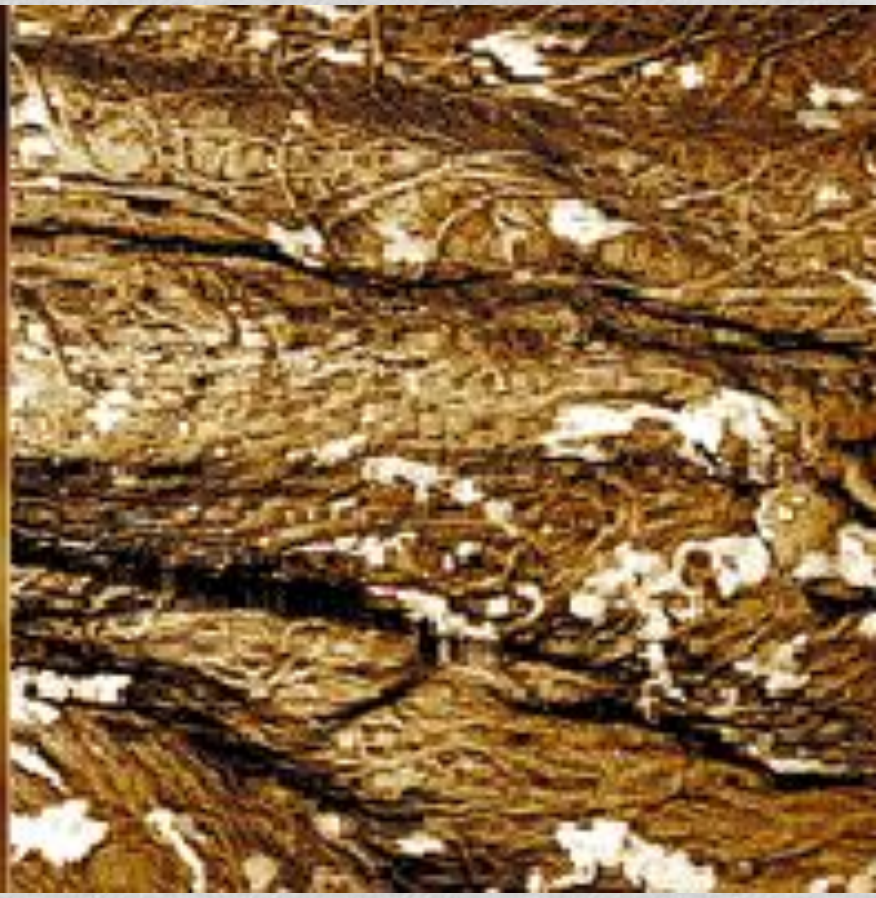


Applications

Phase and Tapping Mode images of wood pulp fiber



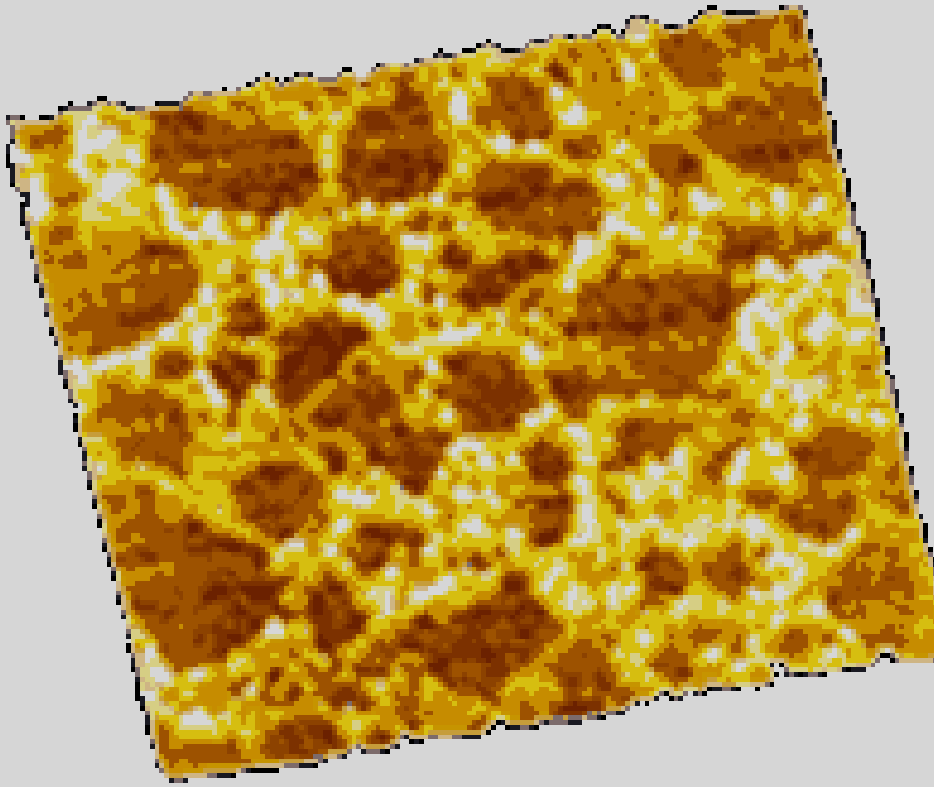
Topography



Phase

Applications

Silicon wafer after an RCA-type clean. This particular clean increased the surface roughness of the silicon from its as received value, typically 0.1nm~ 0.73nm RMS.



Summary

- Introduce AFMs
- Different Modes
- Construction
- Types
- Applications