Optical, Electron, and Scanning Probe Microscopy

Online Workshop *November 6th, 2024*

nffa-di

CNR ISMN

ISTITUTO PER LO STUDIO DEI MATERIALI NANOSTRUTTURATI

Exploring Nanoworlds by Scanning Probe Microscopy

C. Albonetti

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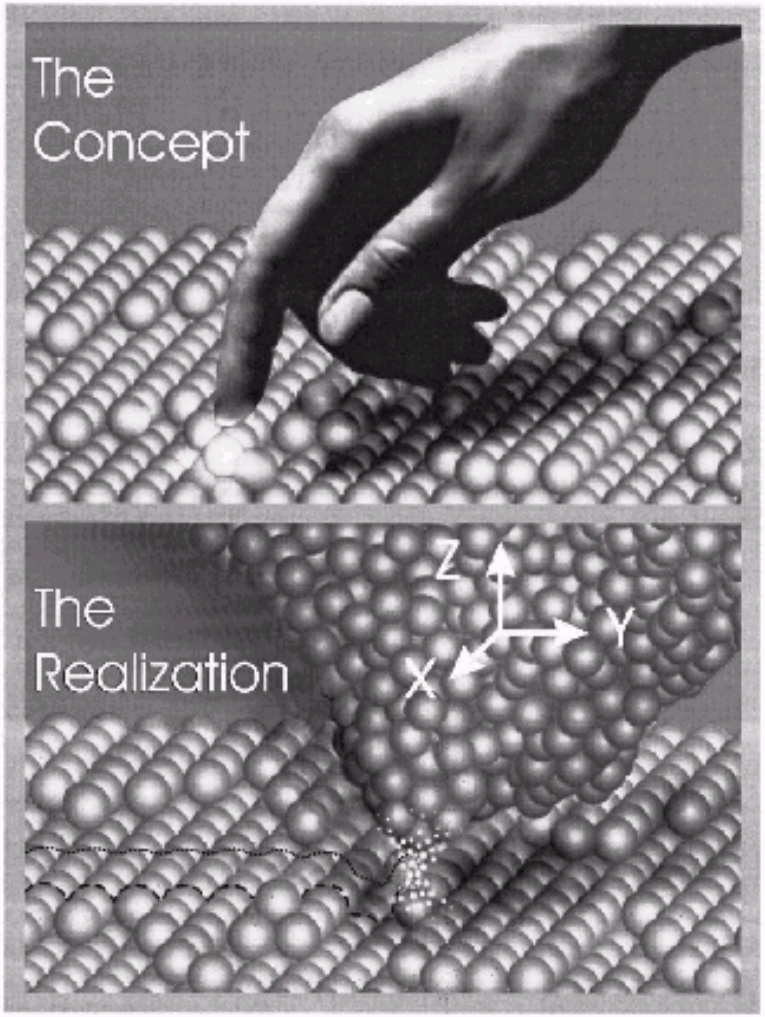
"Seeing is believing, and now we can do that on the atomic scale." - Christoph Gerber

2016 Kavli Prize in Nanoscience

A Discussion with Gerd Binnig and Christoph Gerber



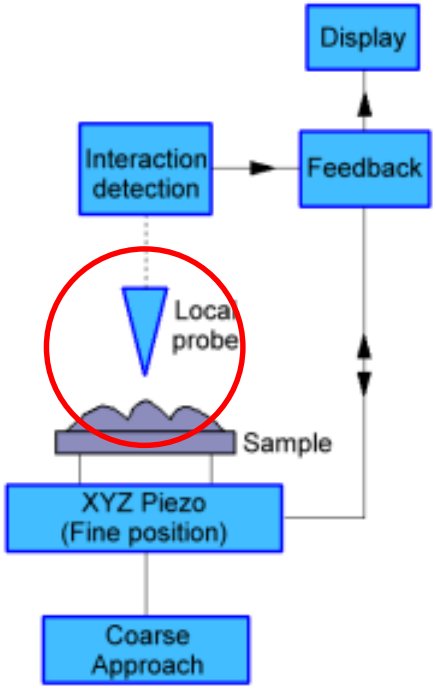
Gentle touch of a nano-finger

"If the interaction between tip and sample decays sufficiently rapidly on the atomic scale, only the two atoms that are closest to each other are able to "feel" each other."

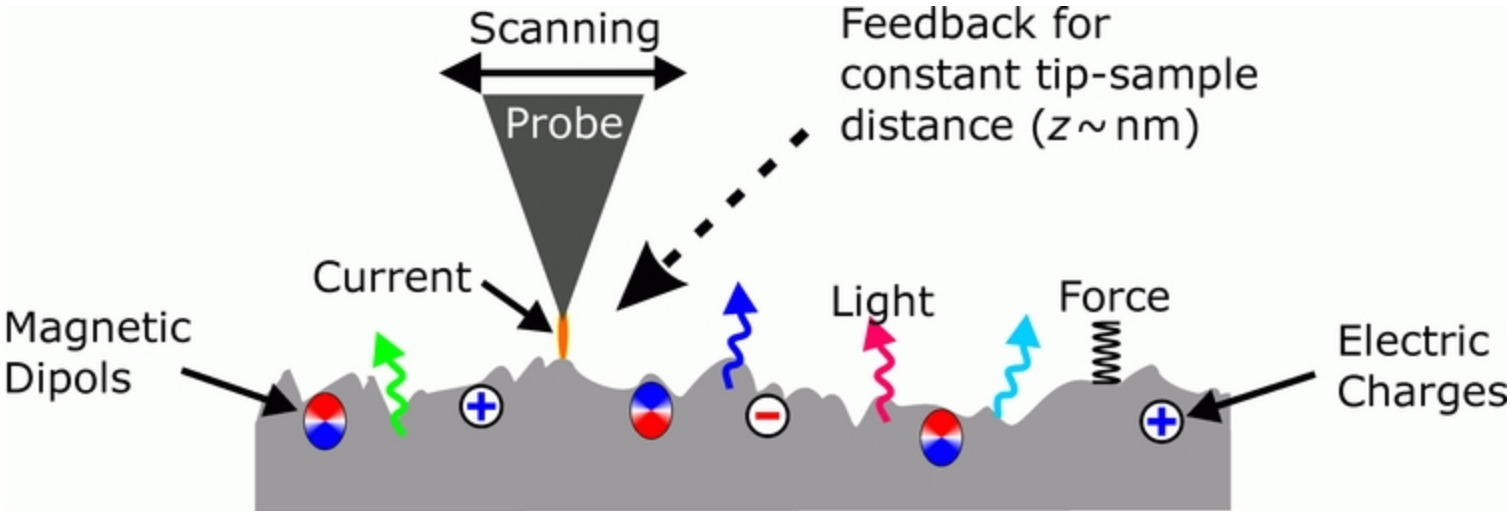
G. Binnig, H. Röhrer, Rev. Mod. Phys. **71**, S324 (1999)

Generic scheme of an SPM

Locality: Surface chemical – physical properties are measured from the spatial region involved in the tip – sample interaction



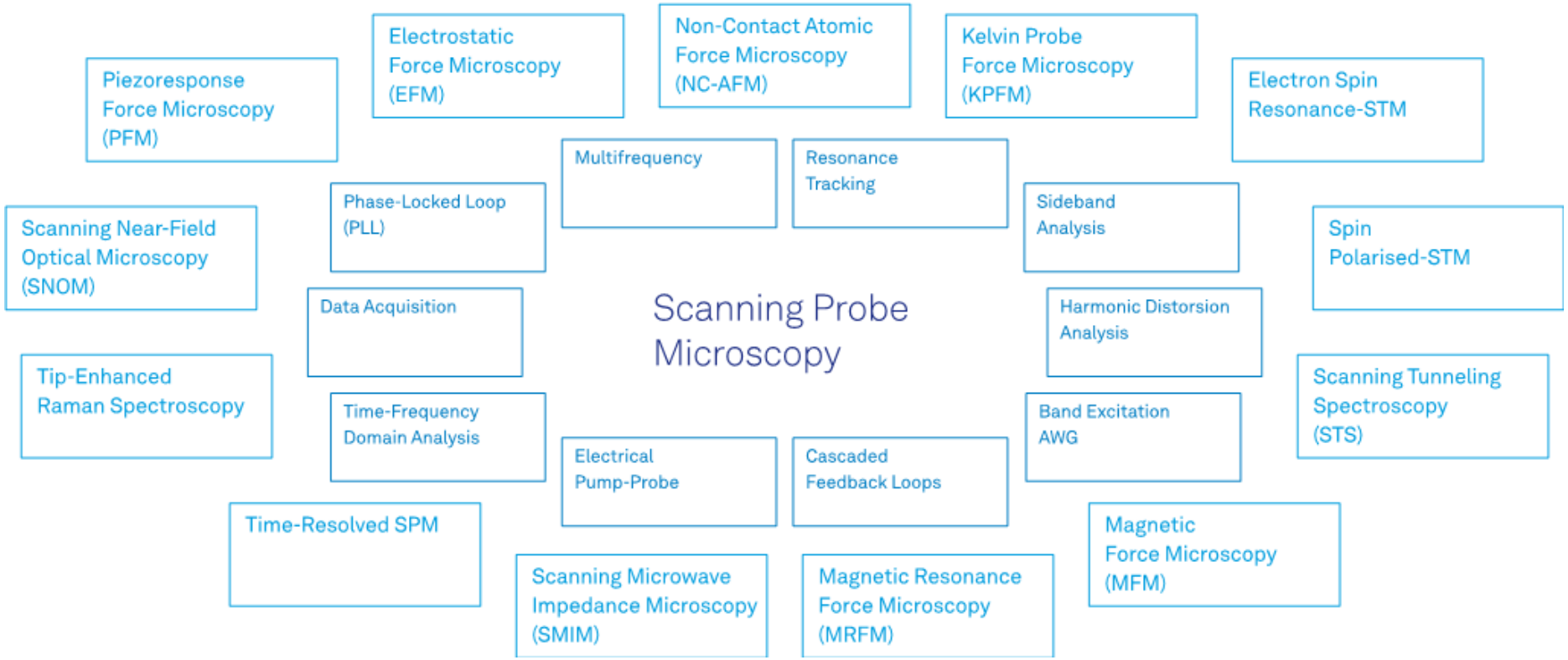
Basics of a local probe



Loos, Adv. Mat. **17**, 1821 (2005)

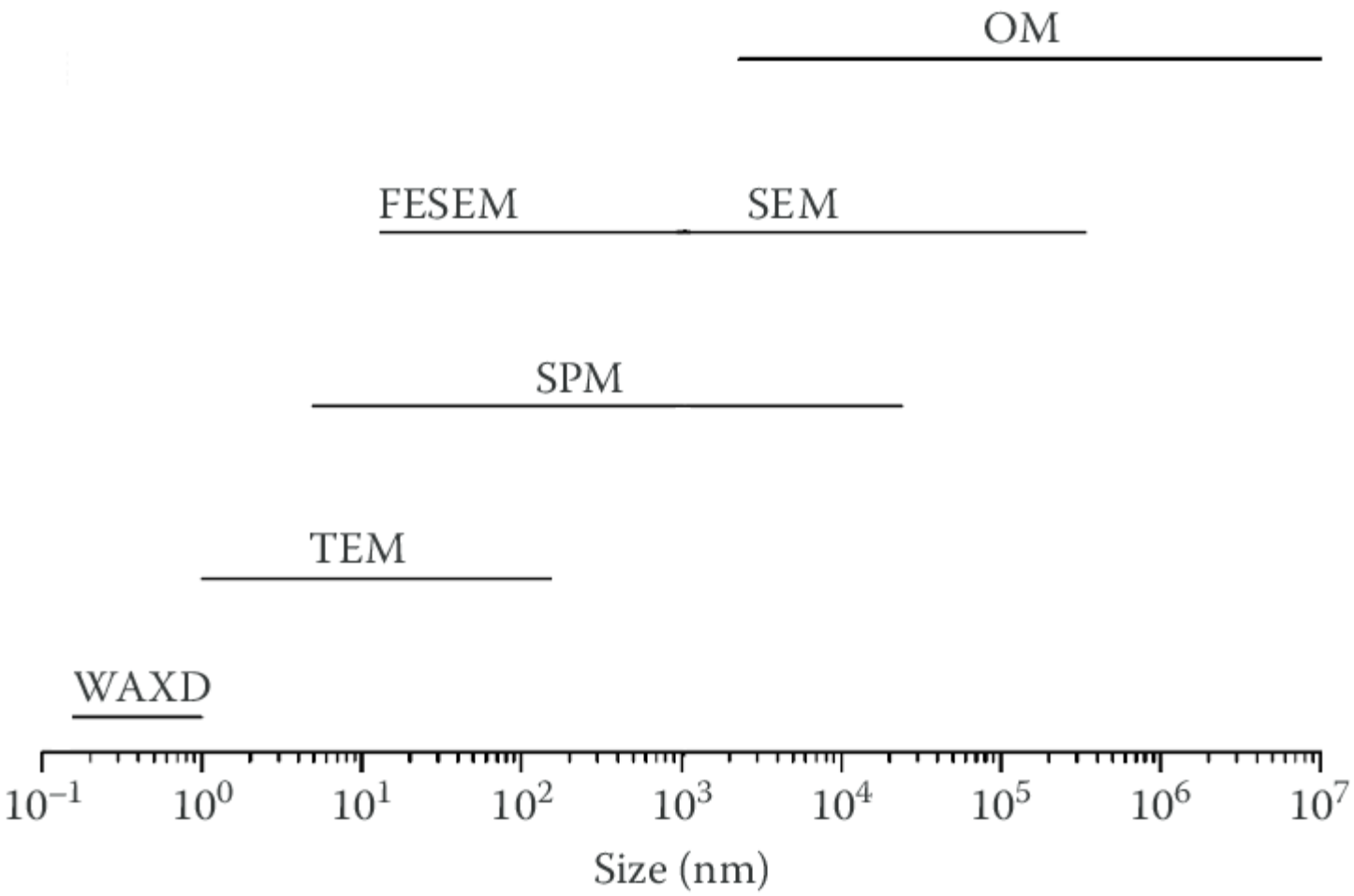
"We present here the historic development of Scanning Tunneling Microscopy; ***the physical and technical aspects*** have already been covered in a few recent reviews and two conference proceedings [l] and many others are expected to follow in the near future."

From the Nobel Lecture by Gerd Binning and Heinrich Rohrer



https://www.zhinst.com/ch/en/applications/scanning-probe-microscopy/scanning-probe-microscopy-spm

Basics of a local probe



Advantages and disadvantages of SPM

- The resolution of the microscopes is limited by the size of the probe-sample interaction volume (as small as a few pm). Laterally the probe-sample interaction extends only across the tip atom or atoms involved in the interaction;
- The interaction can be used to modify the sample to create small structures (nanolithography);
- Unlike electron microscope methods, specimens do not require a partial vacuum but can be observed in air at standard temperature and pressure or even in liquid;
- Real topogaphy

- The shape of the scanning tip is, sometimes, difficult to determine;
- Has problem if the specimen varies greatly in height;
- The scanning techniques are generally slower in acquiring images. Nowadays, efforts are being made to greatly improve the scanning rate (video rate);
- The embedding of spatial information into a time sequence generate uncertainties in metrology which arise due to time-domain effects like specimen drift, feedback loop oscillation, and mechanical vibration;
- The maximum image size is generally small (compensate combining micrometric table);

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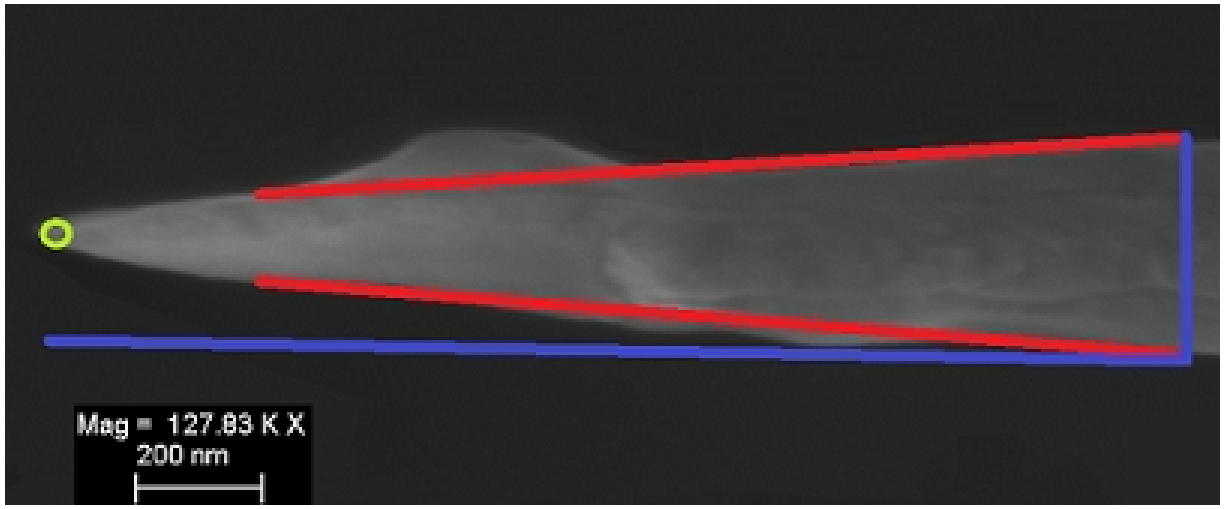
High quality STM tips



RADIUS OF CURVATURE

OPENING ANGLE

ASPECT RATIO

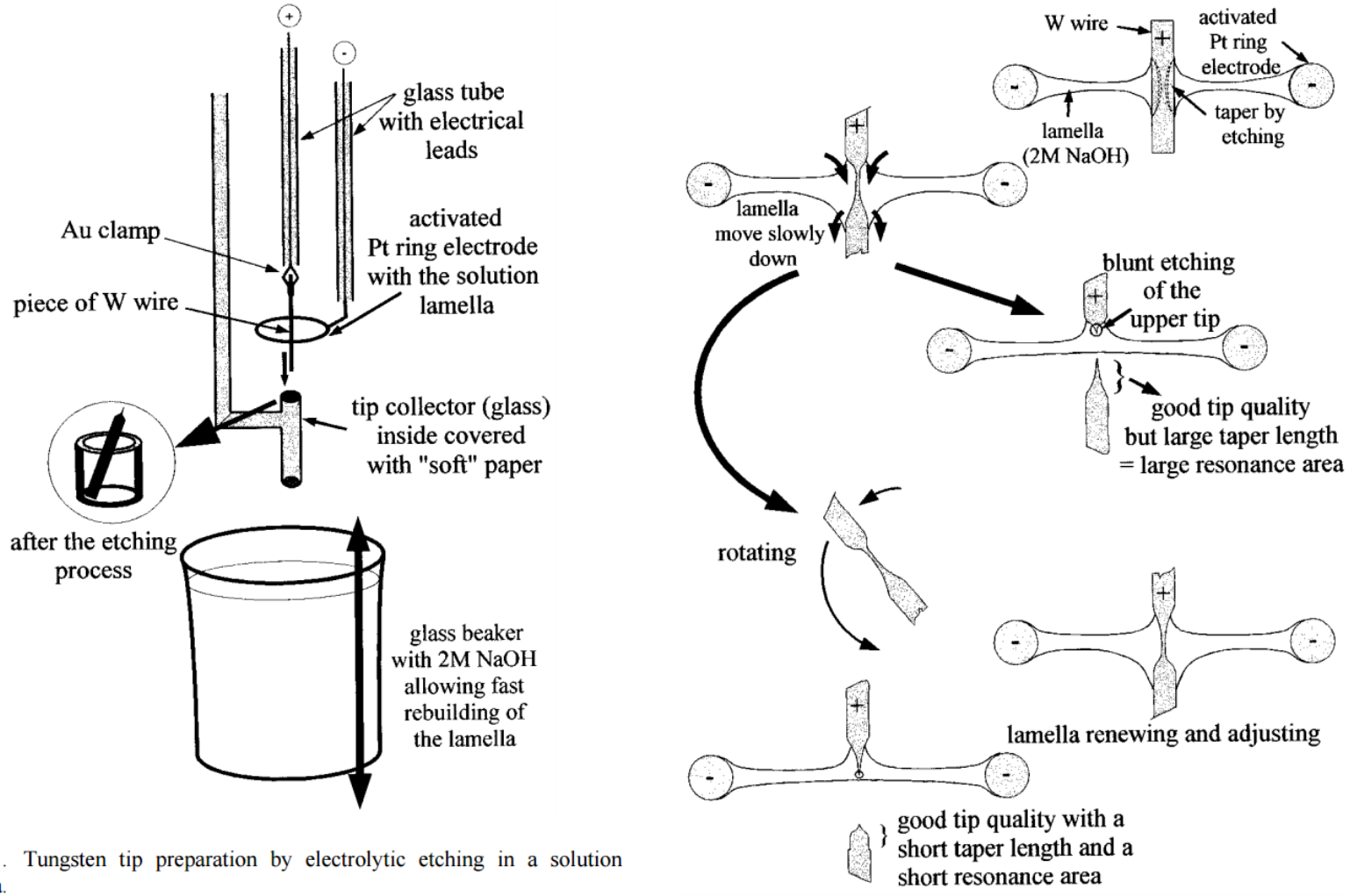
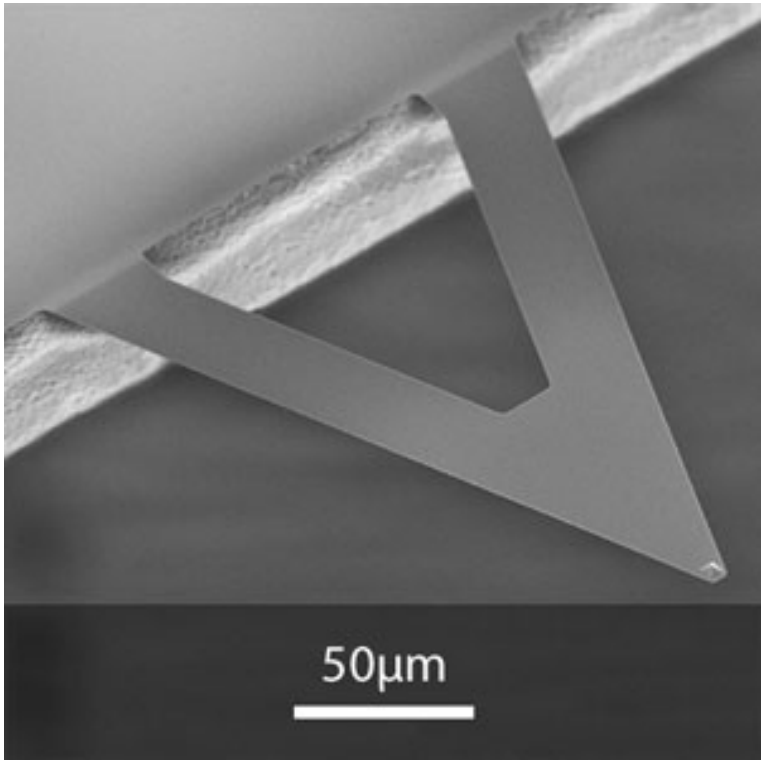
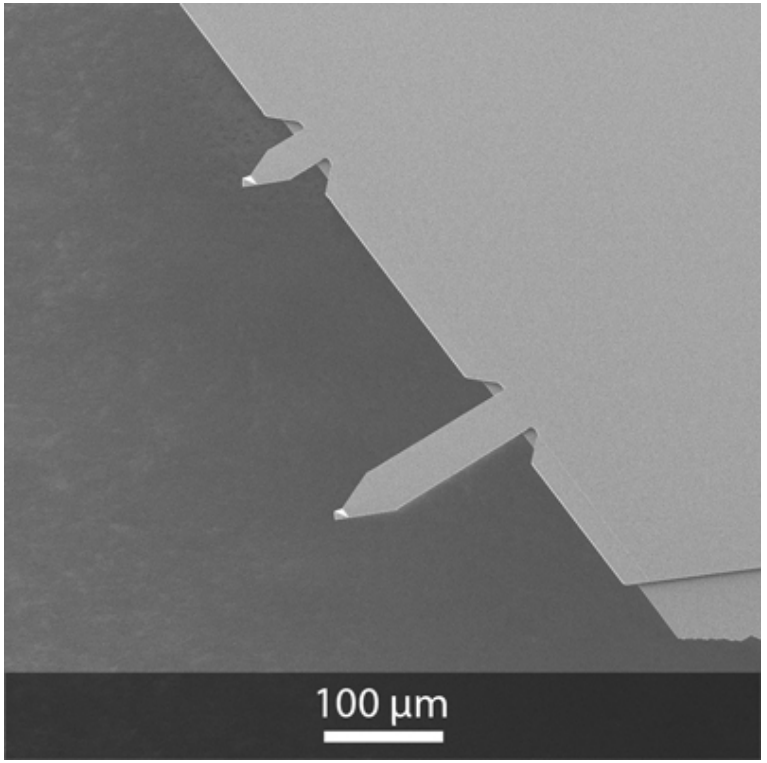


FIG. 1. Tungsten tip preparation by electrolytic etching in a solution lamella.

Its geometrical and mechanical characteristics and the importance for imaging





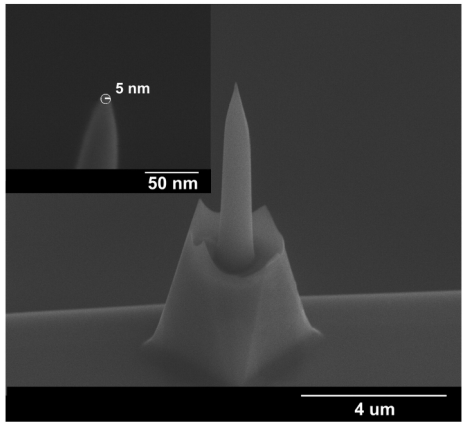
A few requisites for tips

1. Must be sharp

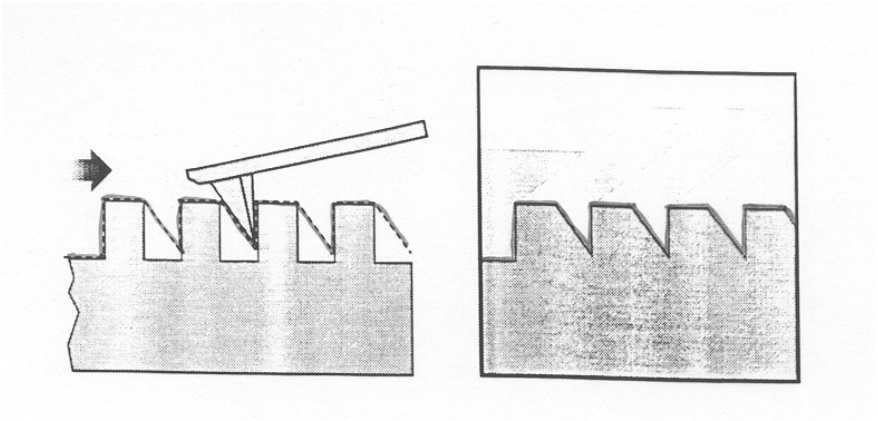
Small curvature at tip apex

Commercial tips: 10 – 20 nm

+ FIB milling or nanotubes: a few nm



High aspect ratio



2. Must be robust

It should resists to damage when in contact with the surface

3. Must be soft

$$F = k\,\Delta z$$

force — F

k: cantilever spring constant

Δz: vertical deflection

⇨ Minimize *k*

For rectangular cantilevers

$$k \sim E\, w\, t^3\, l^{-3}$$

E: Young's modulus (160 Gpa)

w: width

t: thickness

l: length

Example: Al l = 4mm, w = 1mm, t = 10μm $k \sim 1\ Nm^{-1}$

k (C-C stretch.) ~ 500 Nm^{-1} k (C-C-H bend.) ~ 50 Nm^{-1}

4. Must be insensitive to external vibrations

⇨ Maximize eigenfrequencies

For rectangular cantilevers $\omega_0 \sim (k/m)^{1/2}$

⇨ Minimize m

Example: Si or Si_3N_4 (microlithography)

l = 140μm, w = 40μm, t = 1.5μm $k \sim 0.7$ Nm^{-1} $\omega_0 \sim 60$ kHz

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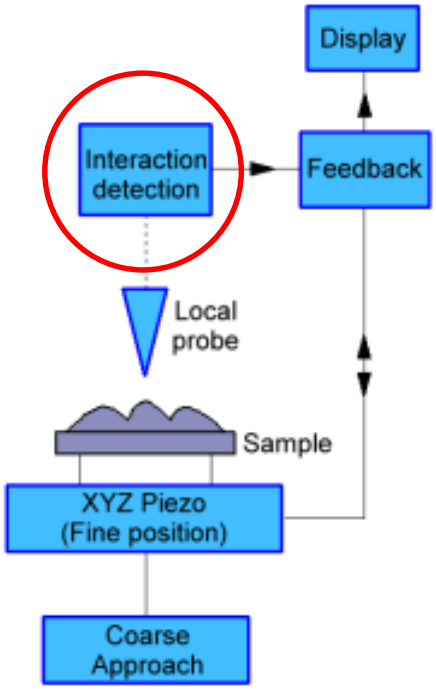
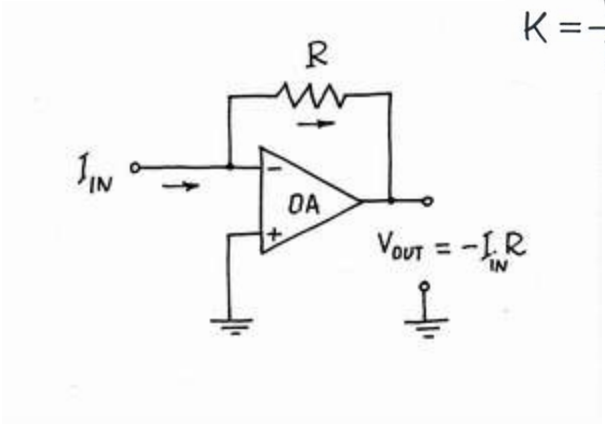


How to measure electric current?

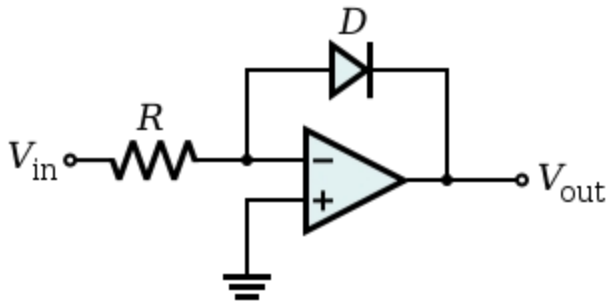
Current to Voltage converter

$$K = \frac{V_{OUT}}{I_{IN}} \left[\frac{V}{mA}\right]$$

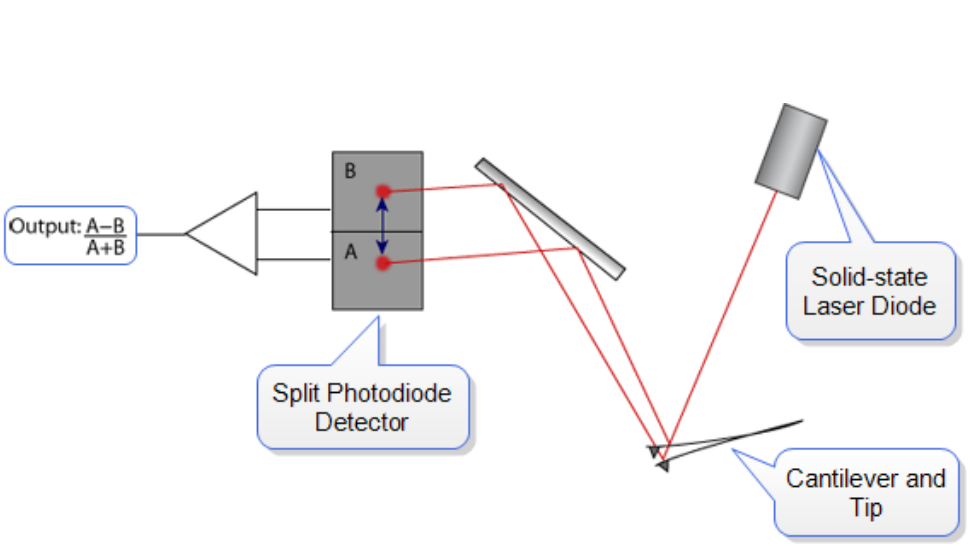


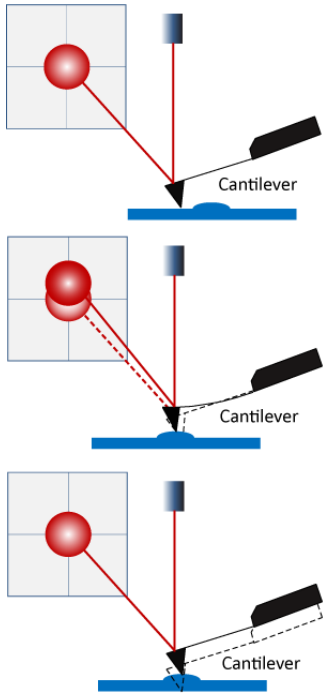
Logarithmic amplifier

$$v_{out} = -V_\gamma \ln\left(\frac{v_{in}}{I_S \cdot R}\right)$$



How to measure forces?





1. Cantilever is engaged on sample. Vertical deflection is at setpoint.

2. Tip encounters a particle. Cantilever is pushed up and vertical deflection increases.

3. Z peizo lifts up the cantilever and brings vertical deflection back to setpoint.

Generic scheme of an SPM

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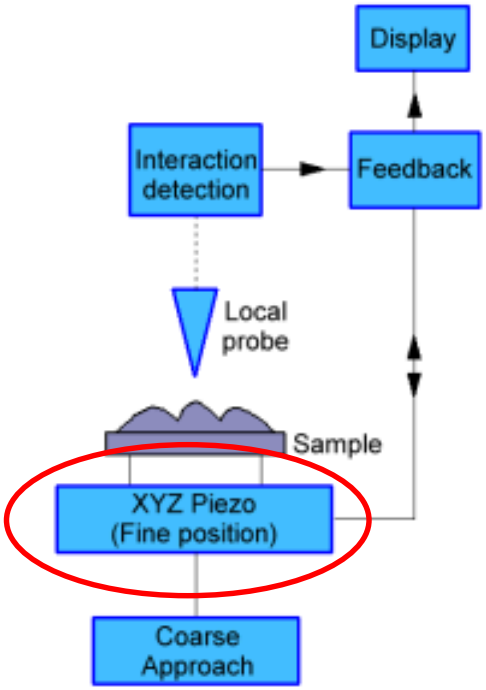
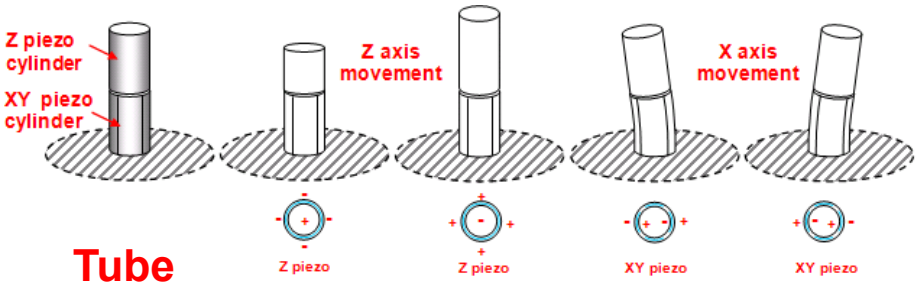
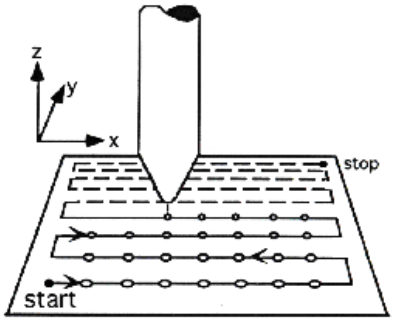


Piezoelectric elements

Sample movements

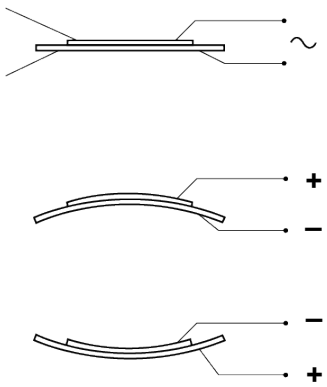


Tube



Raster

Tip oscillations (through cantilever)



Plate

Generic scheme of an SPM

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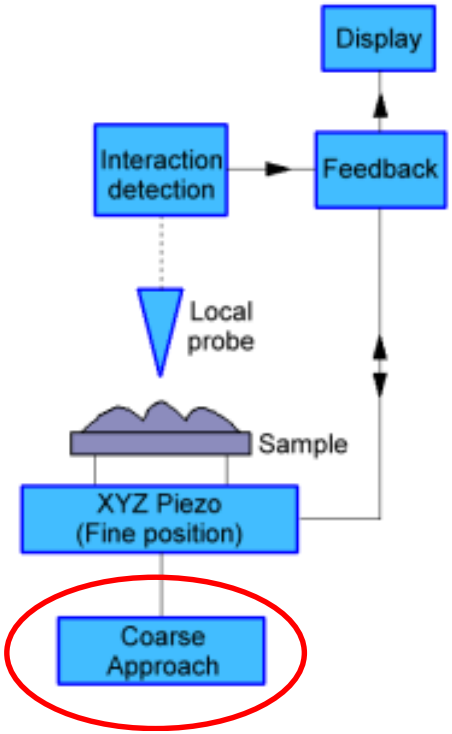

Stepper Motor (steps of 1µm)

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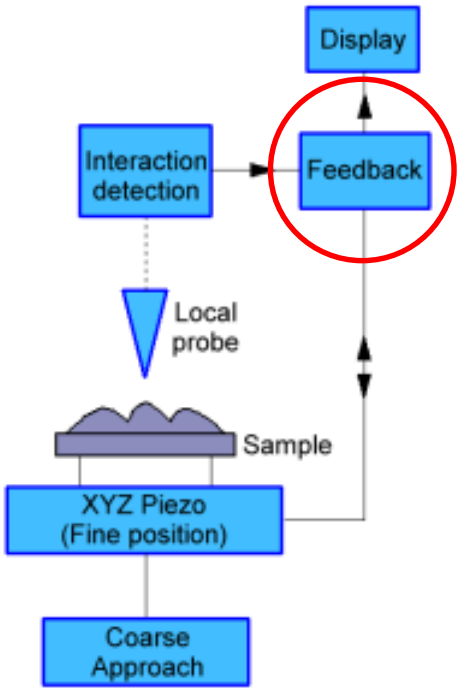
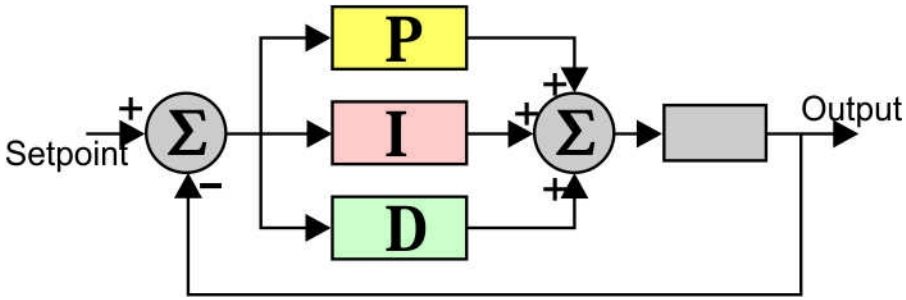


Feedback system

How to control the approaching tip



P (***Proportional***) **0 → 1** (***rise time***)

I (***Integral***) **0 → 1** (***steady-state error***)

D (***Differential***) **0 → 1** (***settling time***)

The set-point has been chosen by you: $\mathbf{V_{SP}}$

The interaction is measured in Volt: $\mathbf{V_{Output}}$

The tip-surface distance is reduced by moving the piezoelectric tube

The error ***e*** is calculated as difference: $\boldsymbol{e = V_{SP} - V_{Output}}$

When ***e* = 0**, the tip interacts with the surface

$$\Delta V = P \cdot e + I \cdot \int_0^t e \cdot dt + D \cdot \frac{de}{dt}$$

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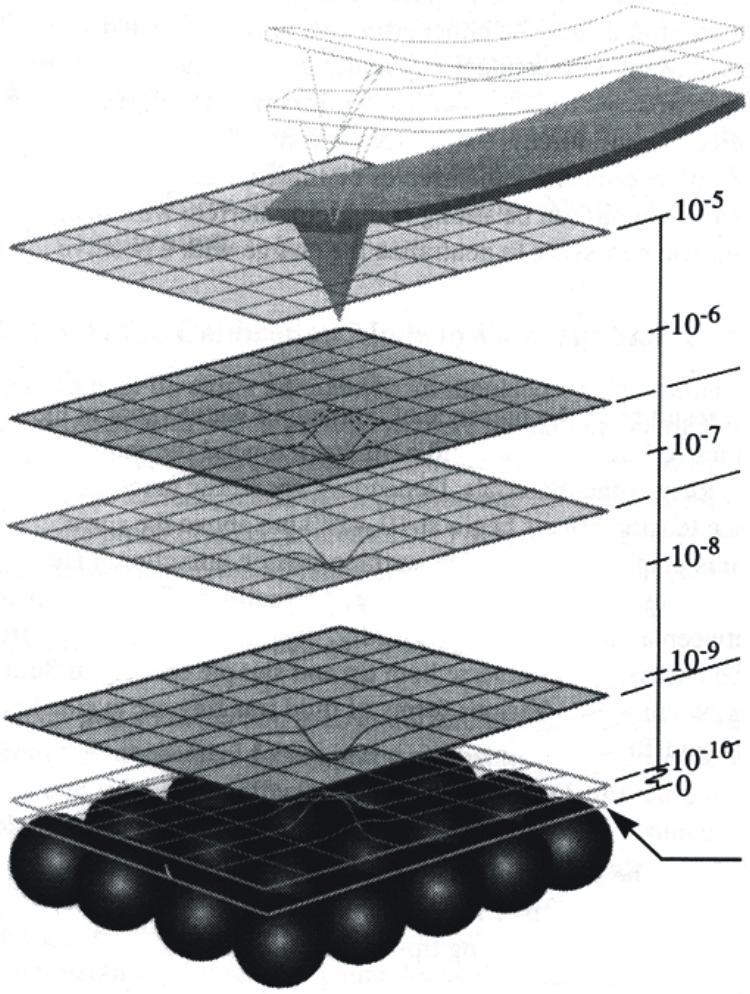
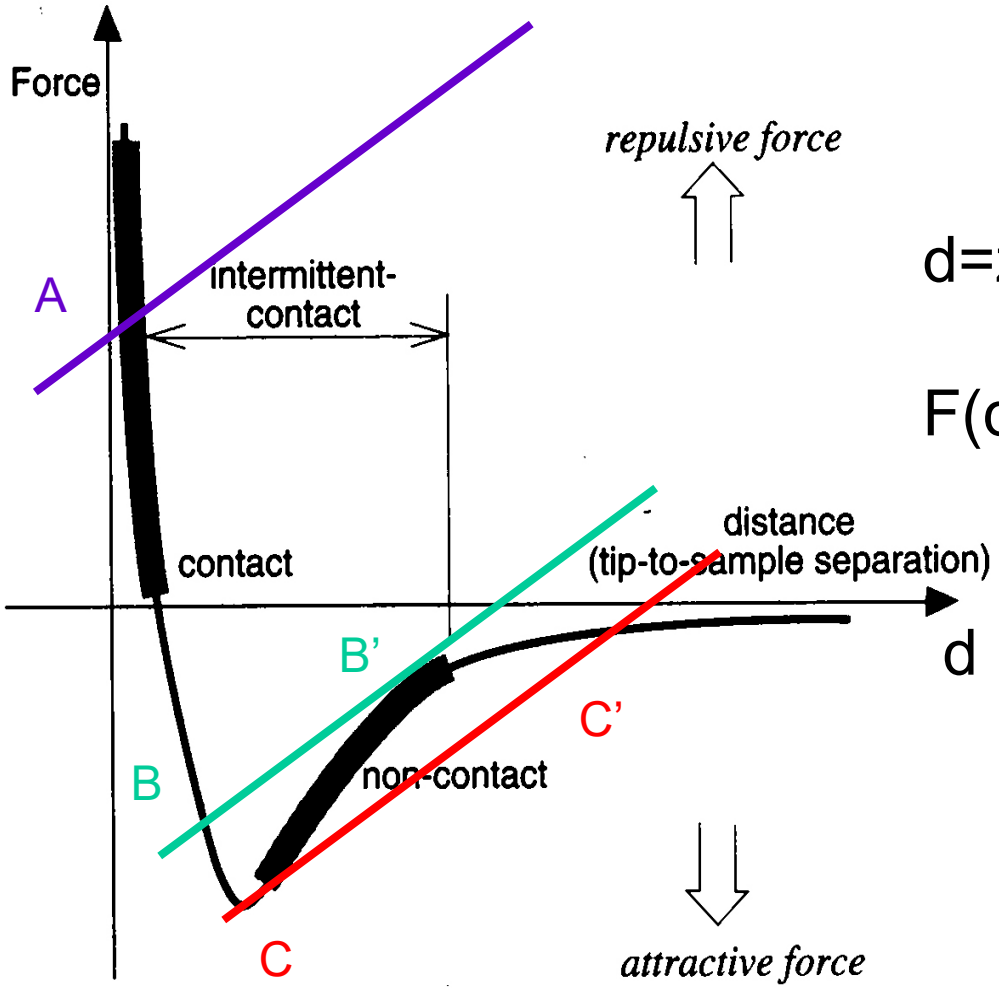
Forces and height above the surface



Fluid Film Damping	10 μ m
Electrostatic Forces (actractive or repulsive)	0,1 - 1,0 μ m
Fluid Surface Tension Forces (attractive)	10 - 200 nm
van der Waals Forces (attractive)	Ångström level
Coulombic Forces (Repulsive)	Fractions of an Ångström
Surface Plane	

Forces involved in AFM: < 10 nm



$d=z_t-z_s$

$F(d)=k(z_t-z_{t0})=k(d+z_s-z_{t0})$

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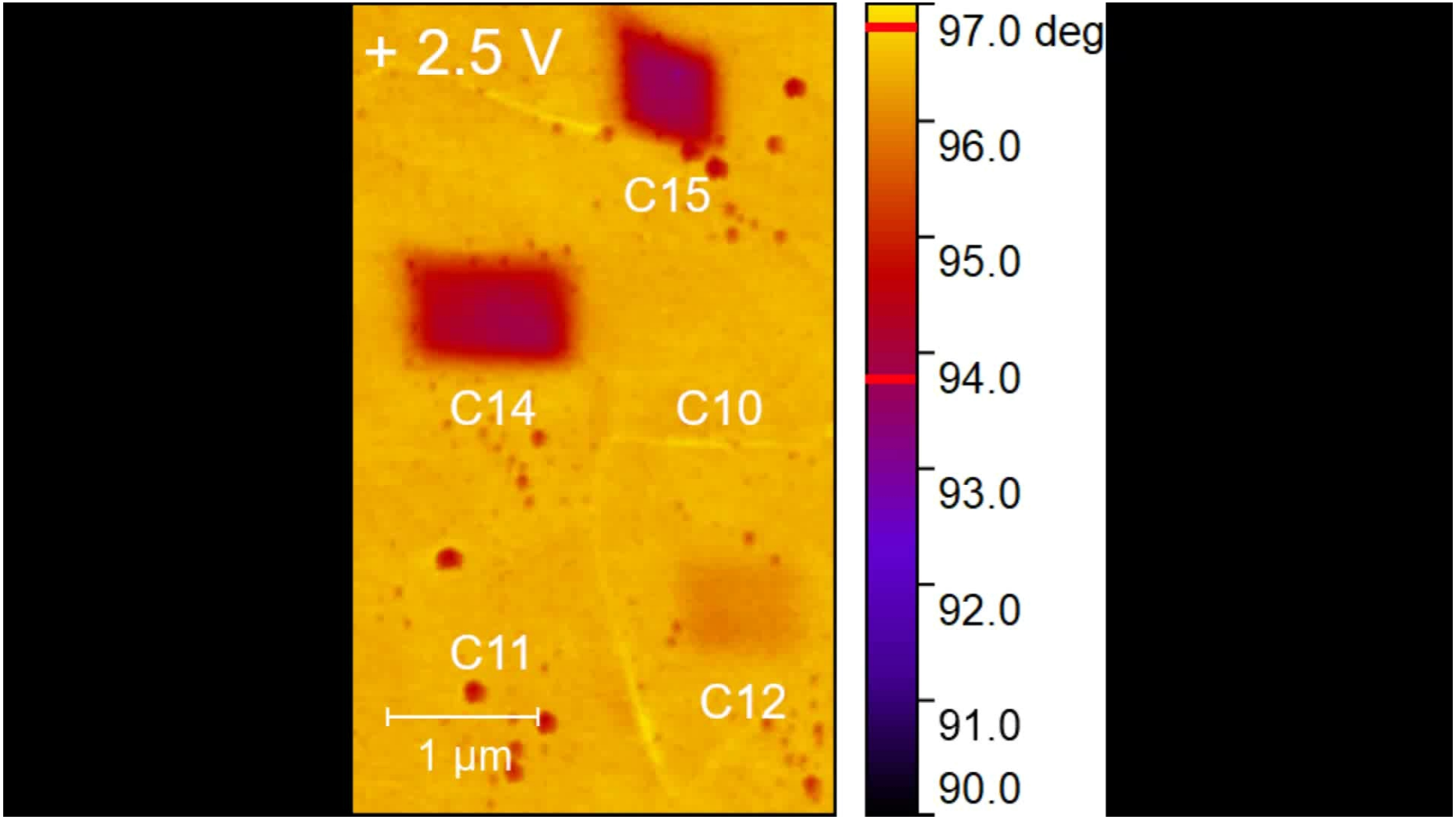
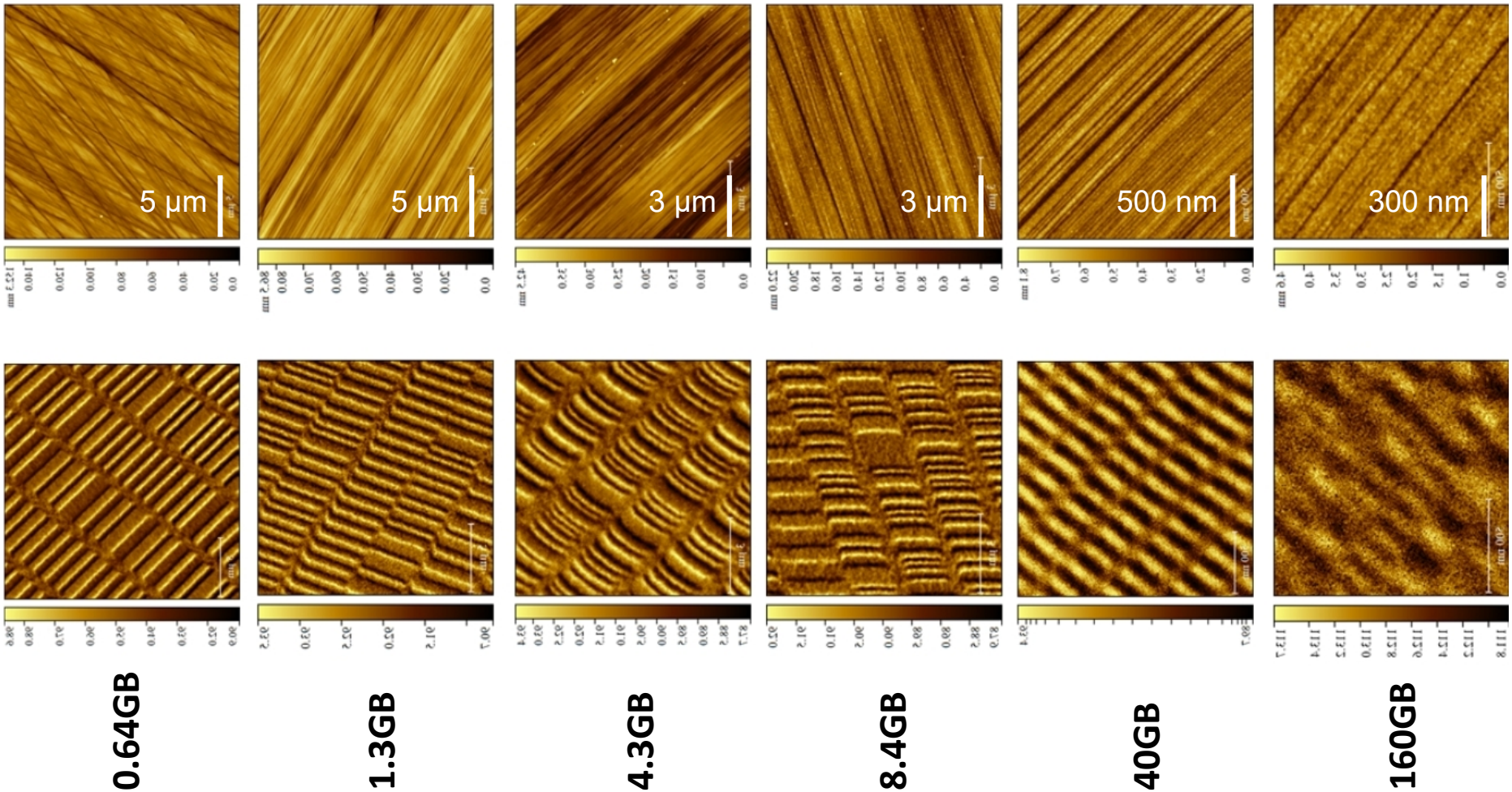
Electrostatic forces



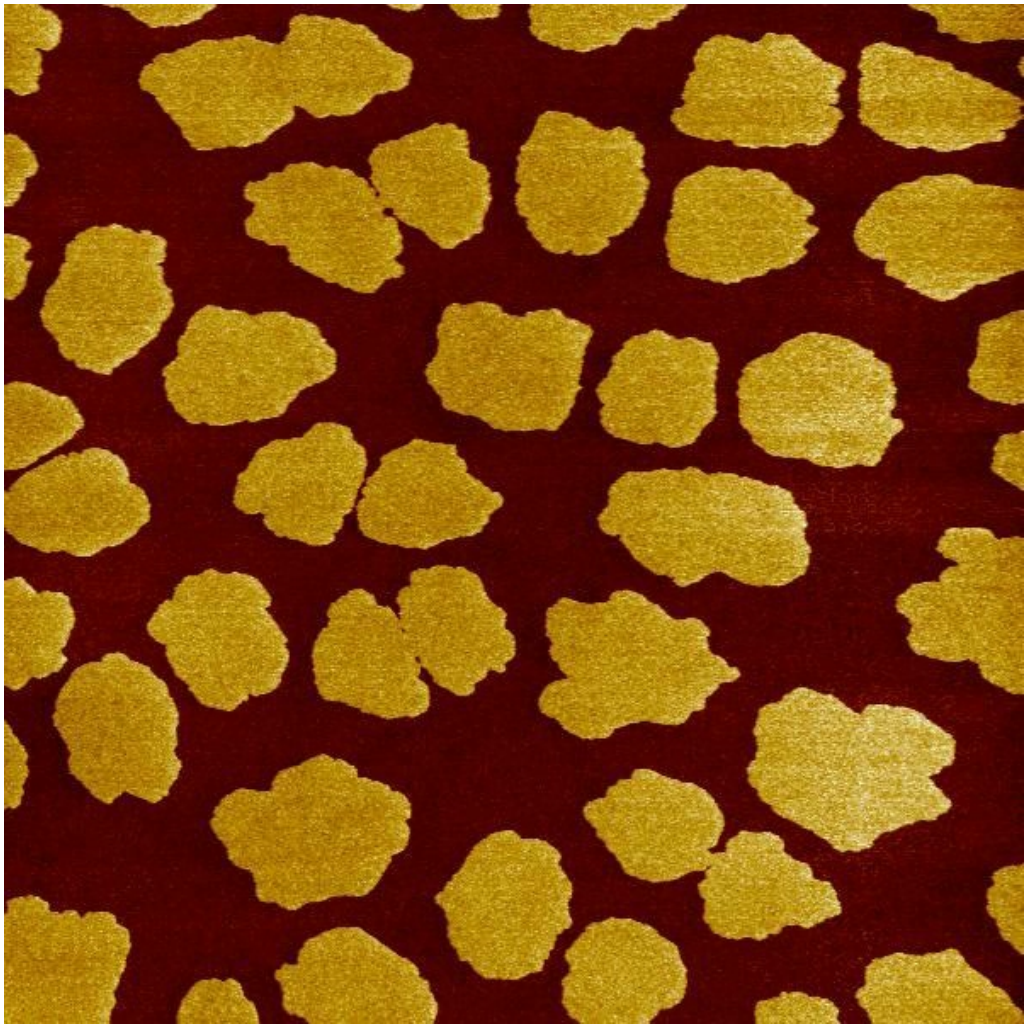
Scaini *et al.*, Chem. Phys. Lett. **803**, 139819 (2022)

Magnetic forces



Albonetti *et al.*, in preparation for Maesurements

Morphological evolution in real-time and *in situ* of an organic film



Chiodini *et al.*, J. Phys Chem. C **124**, 14030 (2020)

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"...gran parte della ricerca di Valdrè e gran parte della nostra ricerca era utilizzare il microscopio elettronico non solo come un banco elettro-ottico, ma come un laboratorio di ricerca."

*D*a "L'esperimento più bello della fisica" (http://l-esperimento-piu-bello-della-fisica.bo.imm.cnr.it/storia/cosa.html)
Prof. Gian Franco Missiroli, ricercatore "anziano" del gruppo

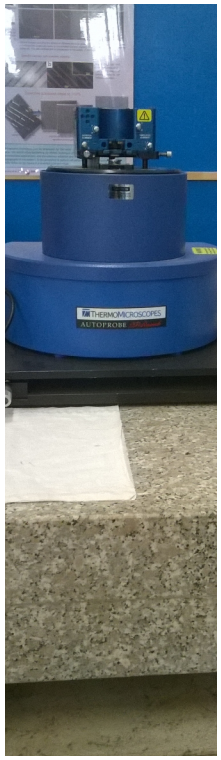
Professional @ CNR

Educational @ Golinelli, DIFA

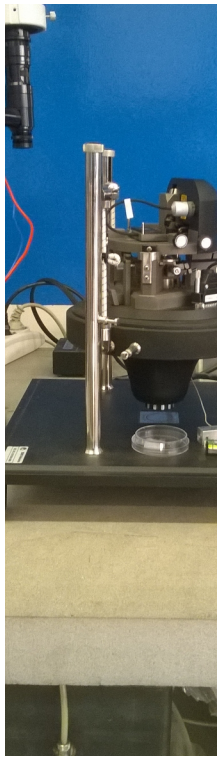
Omicron *UHV*
$2 \cdot 10^{-10}$ mBar
(AFM, STM,
HR-NCAFM)

Smena *HV*
$4 \cdot 10^{-7}$ mBar
(AFM, Electric,
Bimodal AFM,
UFM)

Smena *Air*
(AFM, Electric,
Magnetic)



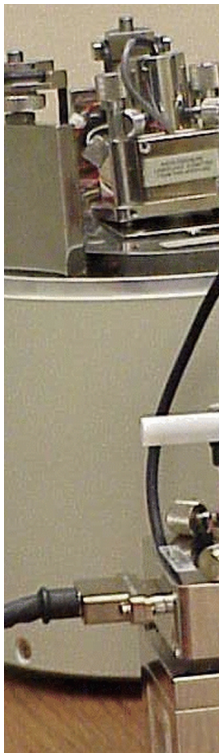
Thermo CPII
Air
(NC AFM, STM)



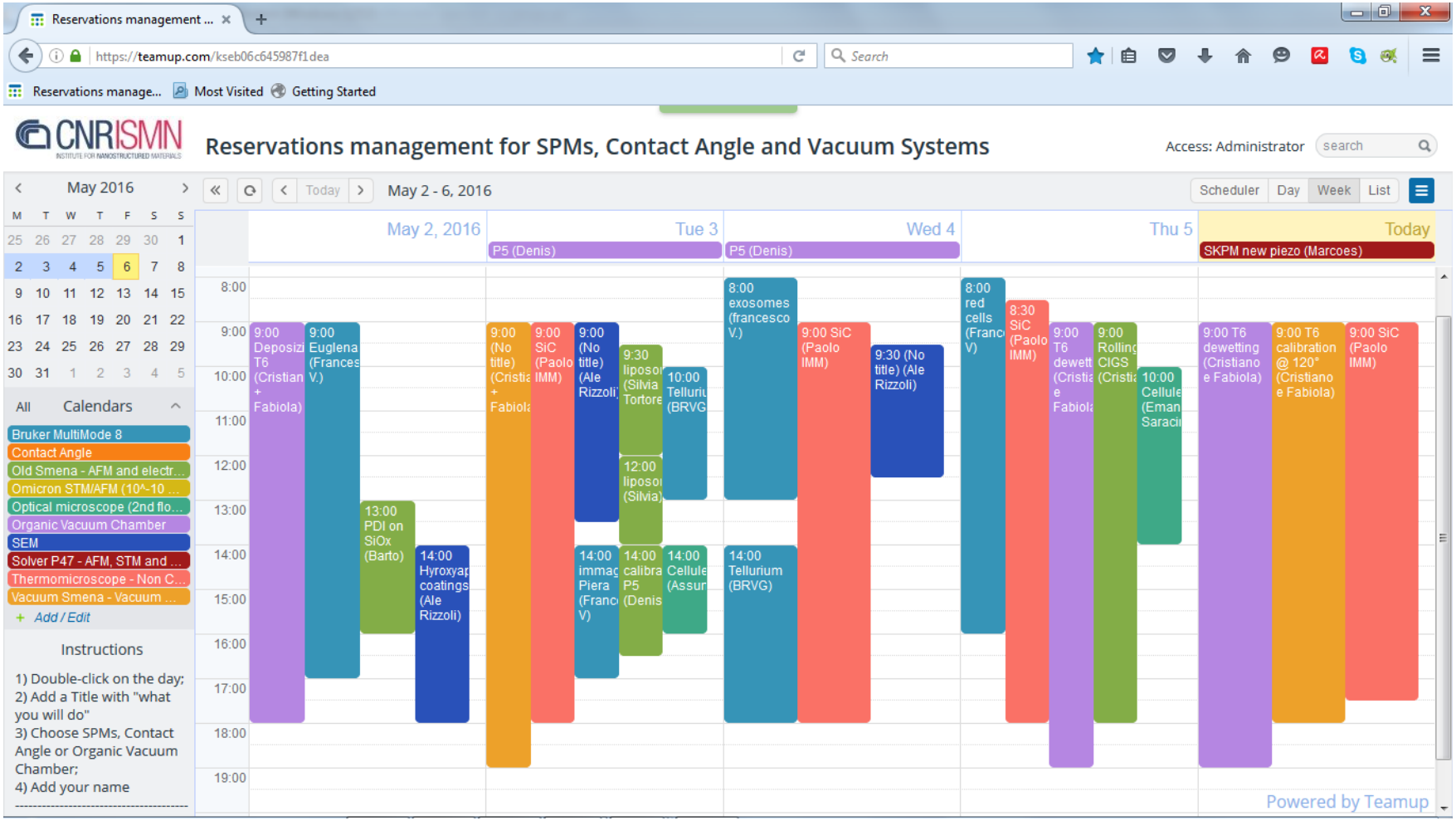
P47H
Air/Liquid
(AFM, STM)

Multimode 8
Air/Liquid
(AFM, STM)

Nanoeducator



Topometrix



2 servers for data exchange and instruments backups

1 workstation for data analysis

micro

MDPI

Review

Probing Italy: A Scanning Probe Microscopy Storyline

Franco Dinelli [1], Marco Brucale [2], Francesco Valle [2], Cesare Ascoli [1,†], Bruno Samorì [3,†], Marco Sartore [4], Manuela Adami [4], Riccardo Galletti [4], Stefano Prato [5], Barbara Troian [5] and Cristiano Albonetti [2,*]

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2 Consiglio Nazionale delle Ricerche-Istituto per lo Studio dei Materiali Nanostrutturati (CNR-ISMN), Via P. Gobetti 101, 40129 Bologna, Italy; marco.brucale@cnr.it (M.B.); francesco.valle@cnr.it (F.V.)
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Thank you for your attention

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