Scanning Probe Microscopy (Basics, and then magnetism)

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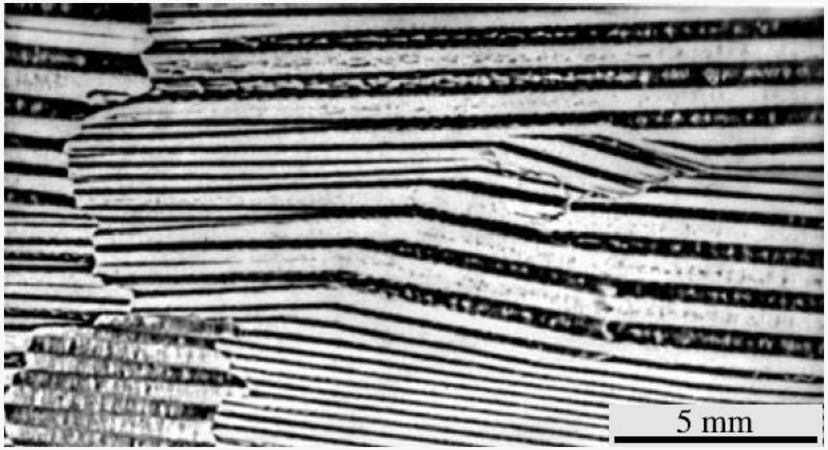
Slides: http://fruchart.eu/slides

OUTLINE

- Motivations (magnetism)
- Scanning Tunneling Microscopy
- Scanning probe microscopy: many fields
- Magnetic Force Microscopy (MFM)

Magnetic domains

- Numerous and complex shape of domains



History: Weiss domains

Magnetic length scales

- Magnetic energy

$$E = A\left(\frac{\partial m_i}{\partial x_j}\right)^2 + K\sin^2\theta$$

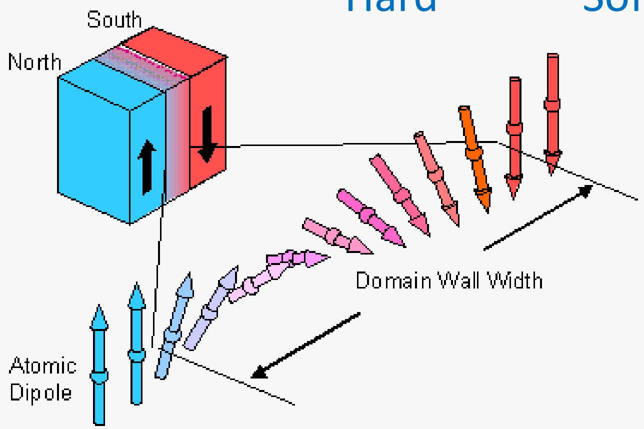
Exchange: J/m

Anisotropy: $\mathrm{J/m^3}$

- Anisotropy exchange length

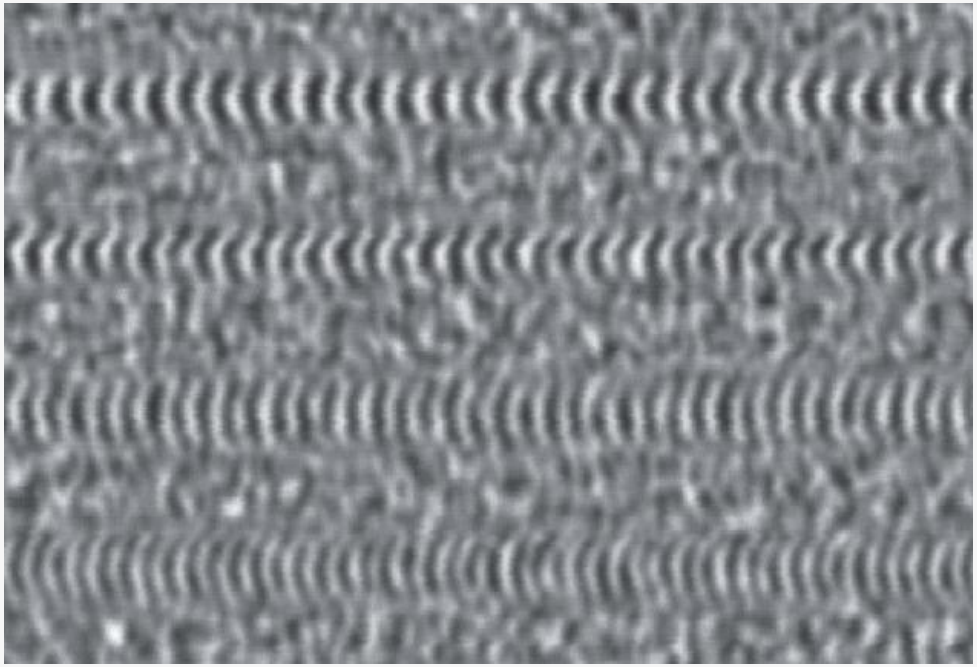
$$\Delta_\mathrm{u} = \sqrt{A/K} \quad 1\ \mathrm{nm} \rightarrow 100\ \mathrm{nm}$$

Hard → Soft



Magnetic bits on hard disk drive

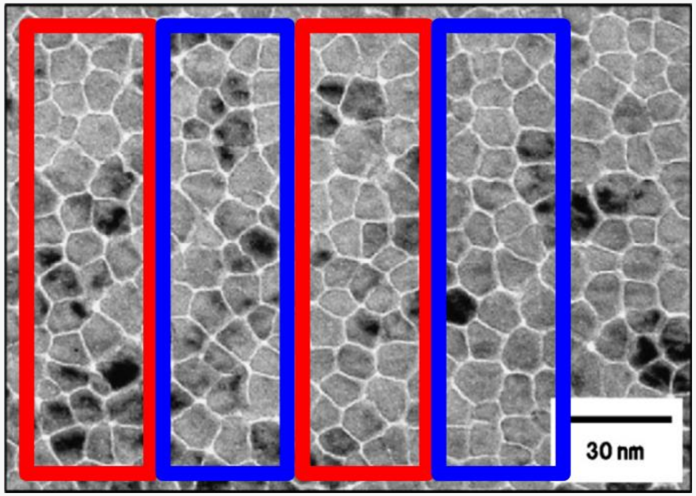
Co-based hard disk media : bits 50nm and below



B. C. Stipe, Nature Photon. 4, 484 (2010)

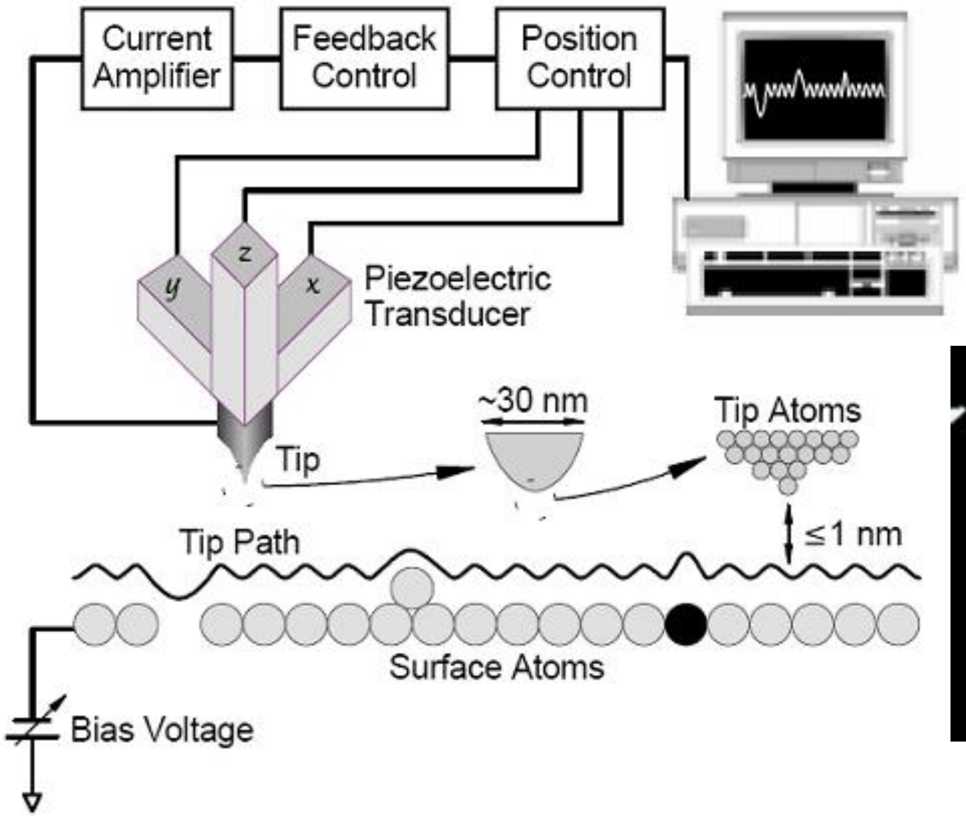
Underlying microstructure

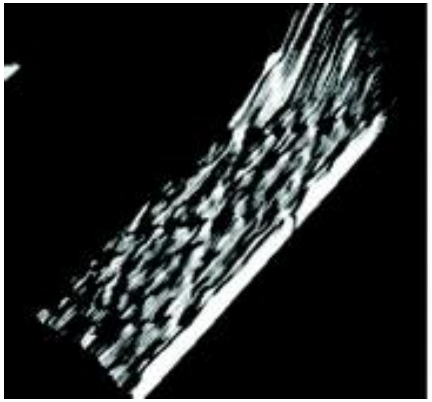
10 nm magnetic grain

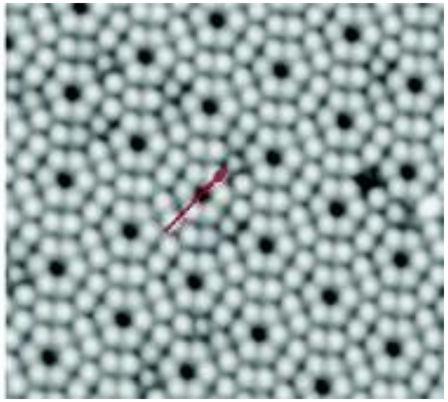


S. Takenoiri, J. Magn. Magn. Mater. 321, 562 (2009)

1982 : inventing the scanning tunneling microscope

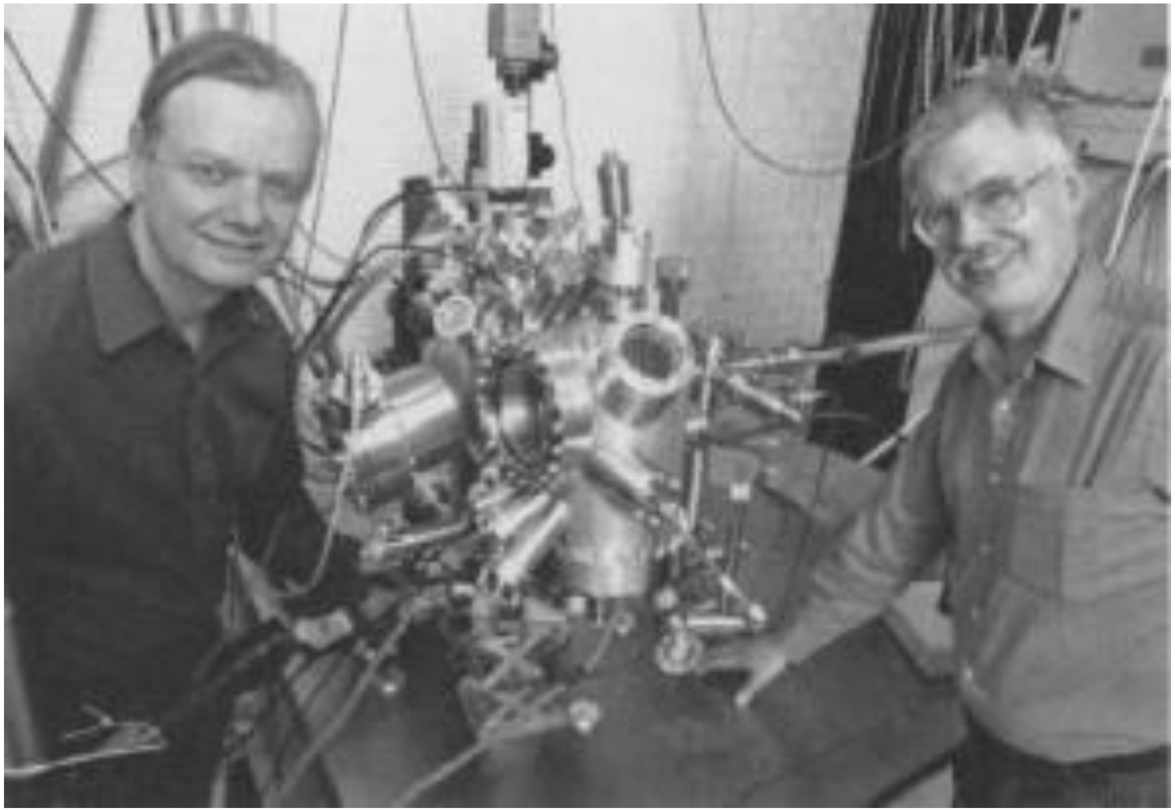






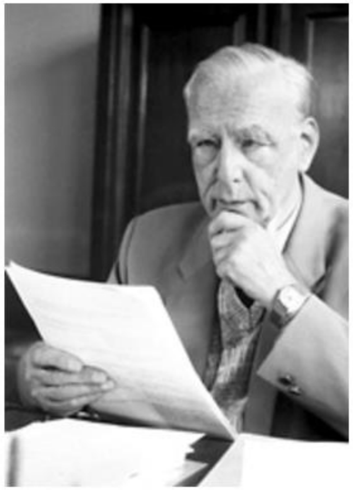
G. Binnig, H. Rohrer, C. Gerber & E. Weibel
Tunneling through a controllable vacuum gap apl 40, 178 (1982)

1982 : inventing the scanning tunneling microscope



G. Binnig, H. Rohrer, C. Gerber & E. Weibel
Tunneling through a controllable vacuum gap apl 40, 178 (1982)

The Nobel Prize in Physics 1986



Ernst Ruska
Prize share: 1/2

Gerd Binnig
Prize share: 1/4

Heinrich Rohrer
Prize share: 1/4

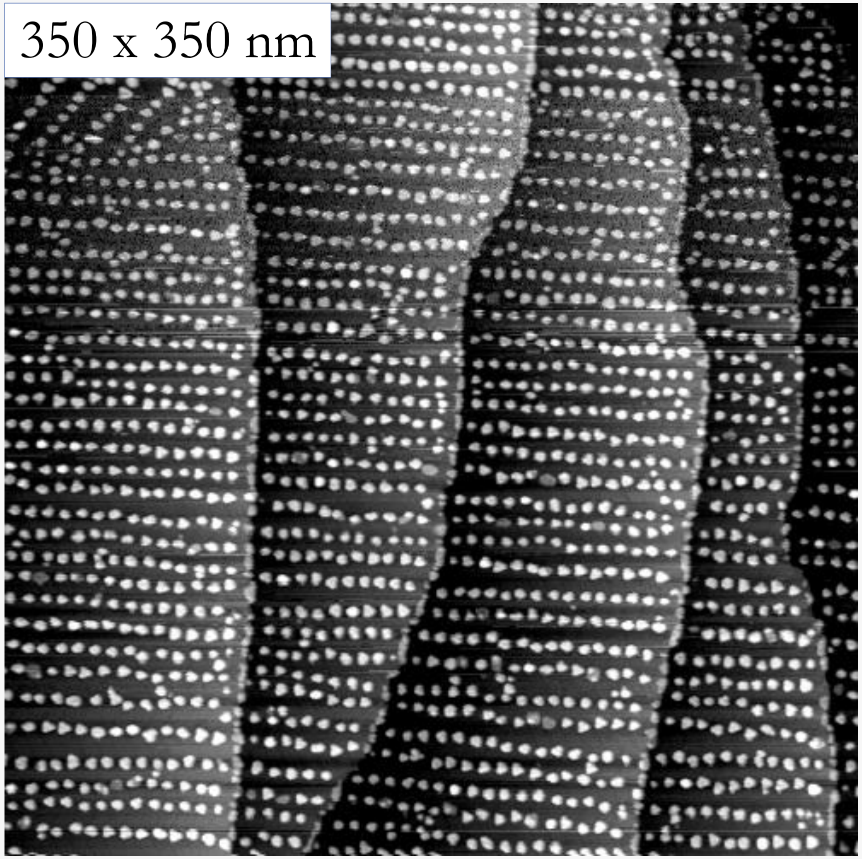
The Nobel Prize in Physics 1986 was divided, one half awarded to Ernst Ruska *"for his fundamental work in electron optics, and for the design of the first electron microscope"*, the other half jointly to Gerd Binnig and Heinrich Rohrer *"for their design of the scanning tunneling microscope"*.

https://www.nobelprize.org

Scanning Tunneling Microscopy

Topography – Large scale

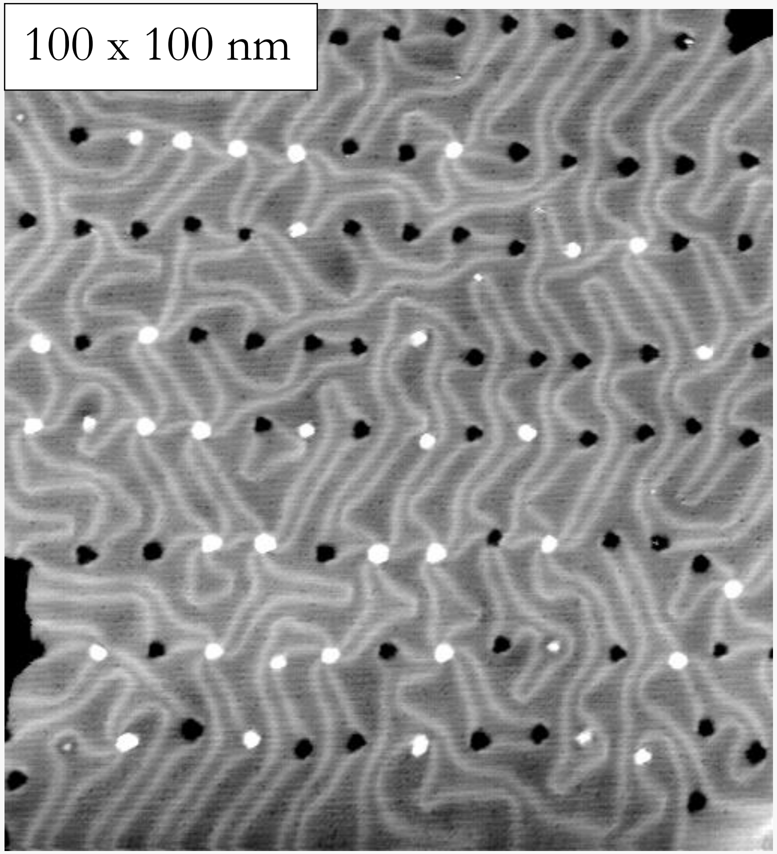
Self-organized growth of magnetic dots (Co)



O. Fruchart et al., Phys. Rev. Lett. 23 (14), 2769 (1999)

High resolution and sensitivity

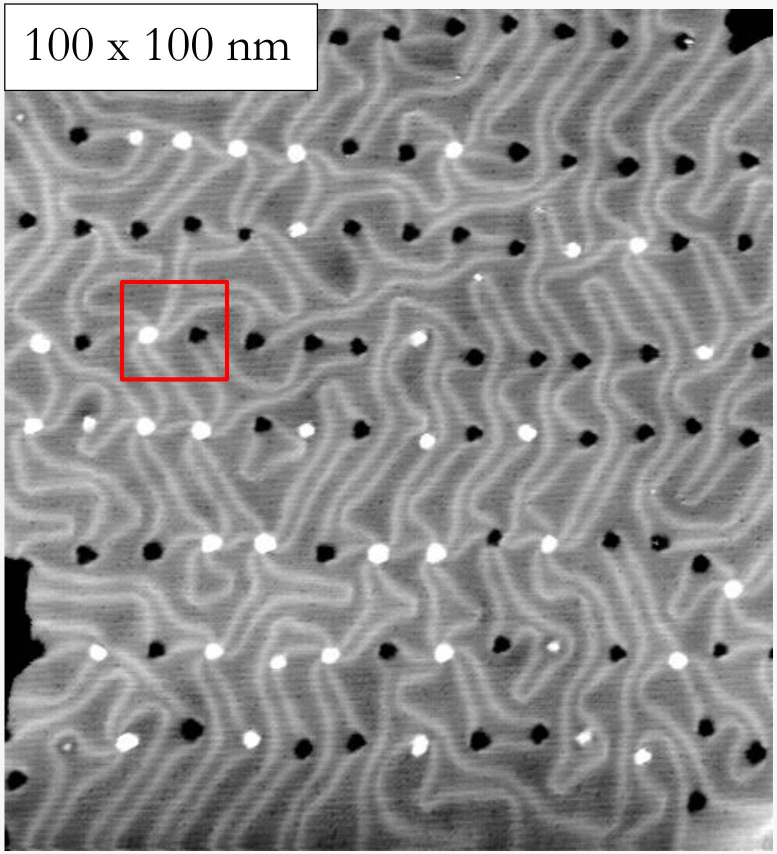
Dots embedded in Au matrix



Scanning Tunneling Microscopy

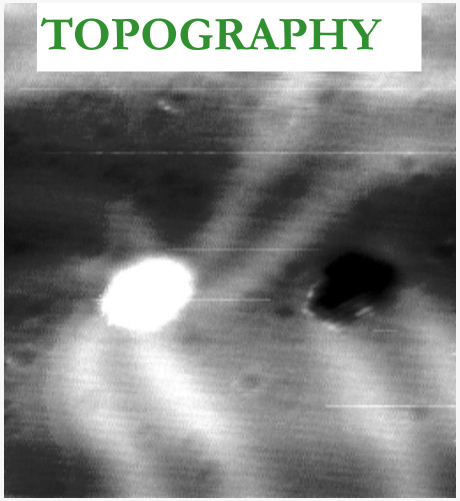
High resolution and sensitivity

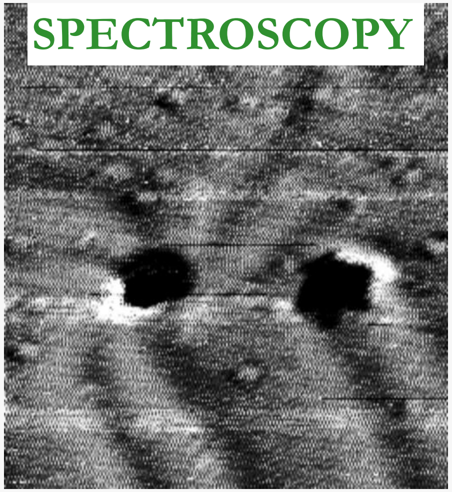
Dots embedded in Au matrix

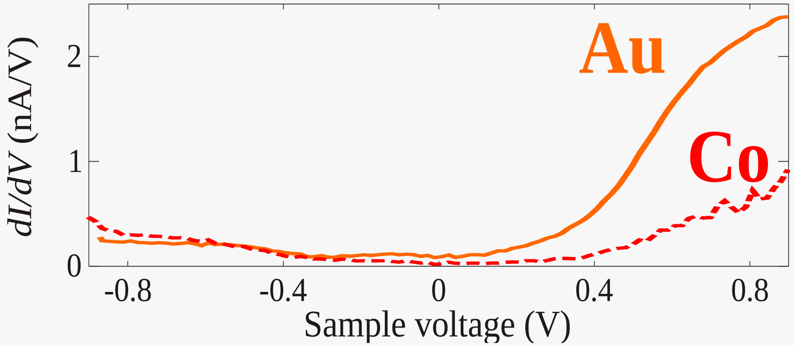


O. Fruchart et al., PRL 23, 2769 (1999)

Spectroscopy → Elemental information

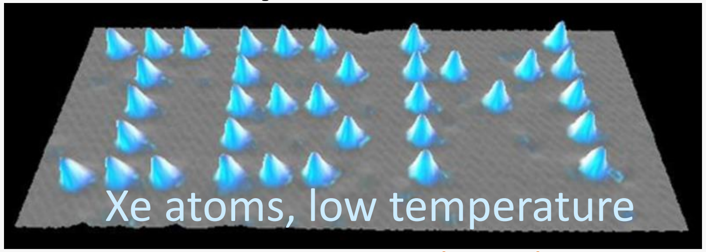




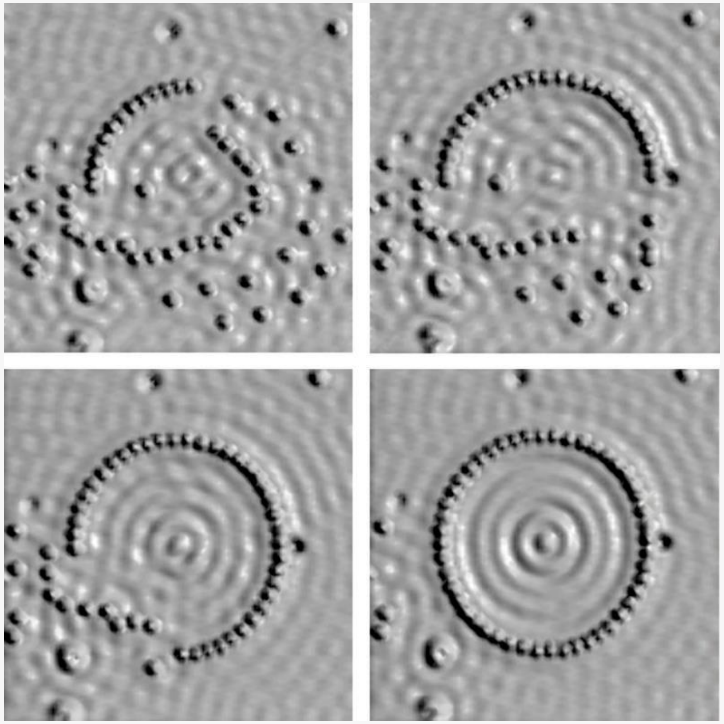


Scanning Tunneling Microscopy

Atom manipulation



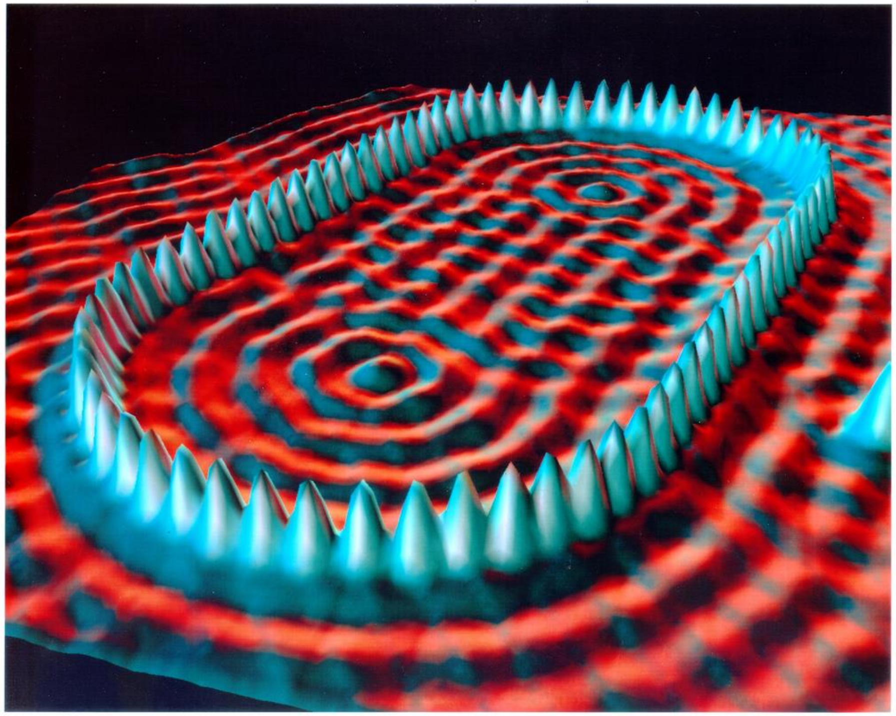
D. Eigler, Nature (1990)



http://research.physics.berkeley.edu/crommie

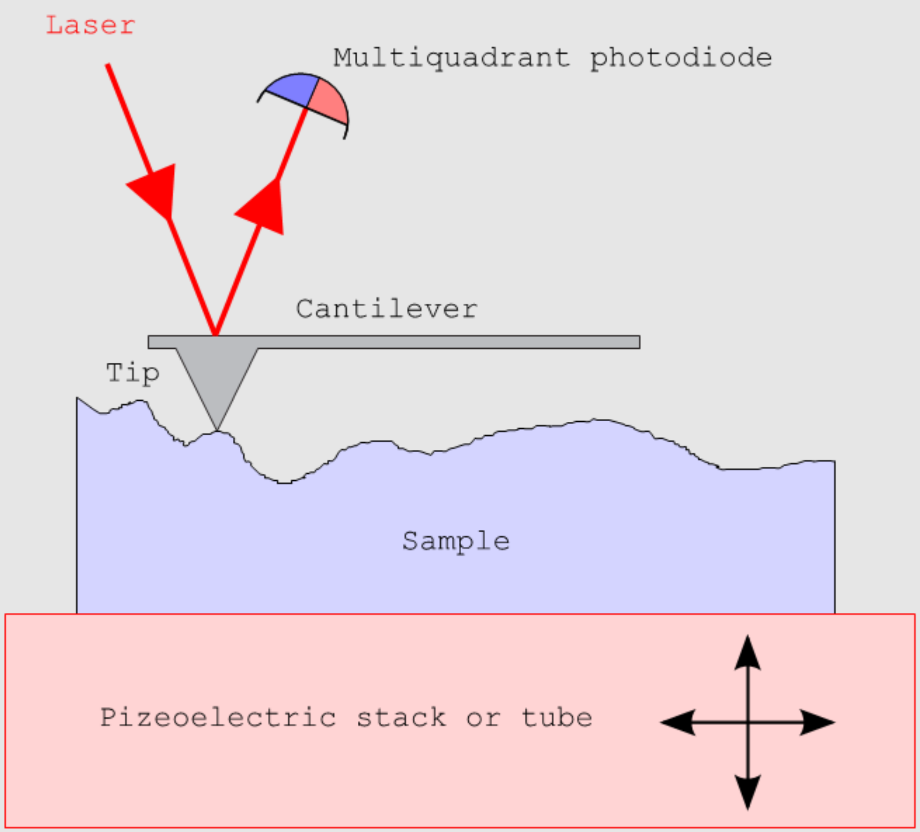
Mapping surface quantum well states

Fe atoms on Copper, low temperature



For magnetism: H. Oka et al., Spin-polarized quantum confinement in nanostructures: Scanning tunneling microscopy, Rev. Mod. Phys. 86, 1127-1168 (2014)

Key elements of an Atomic Force Microscope (AFM)



G. Binnig et al., Phys. Rev. Lett. 56, 930-933 (1986)

Probing

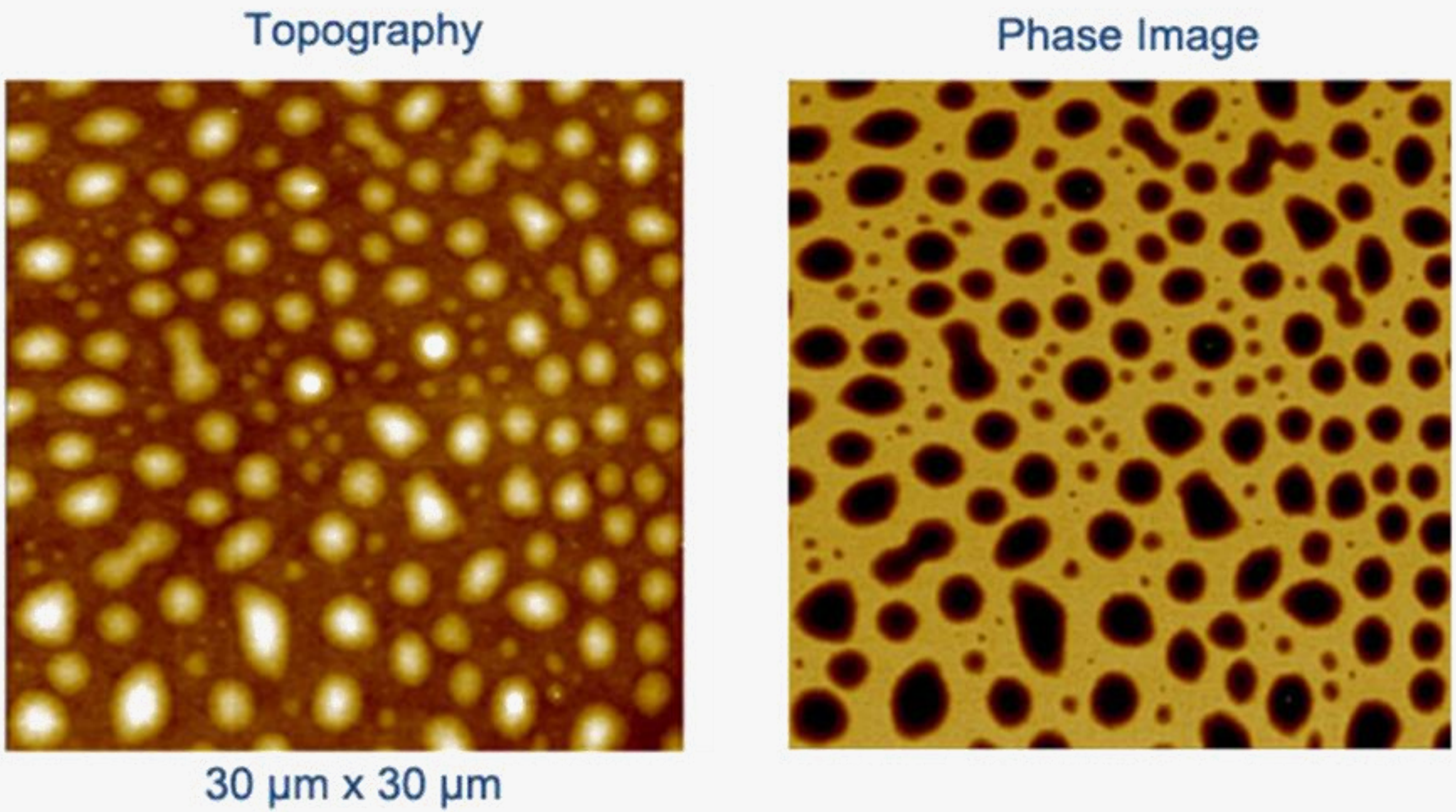
- Mechanical force -> Topography, tribology (adhesion etc.)
- Magnetic force -> magnetic domains
- Electric forces -> ferroelectric domains, semiconductor memory cells etc.

Detecting

- Laser deflection / interference
- Capacitance

Elasticity and losses

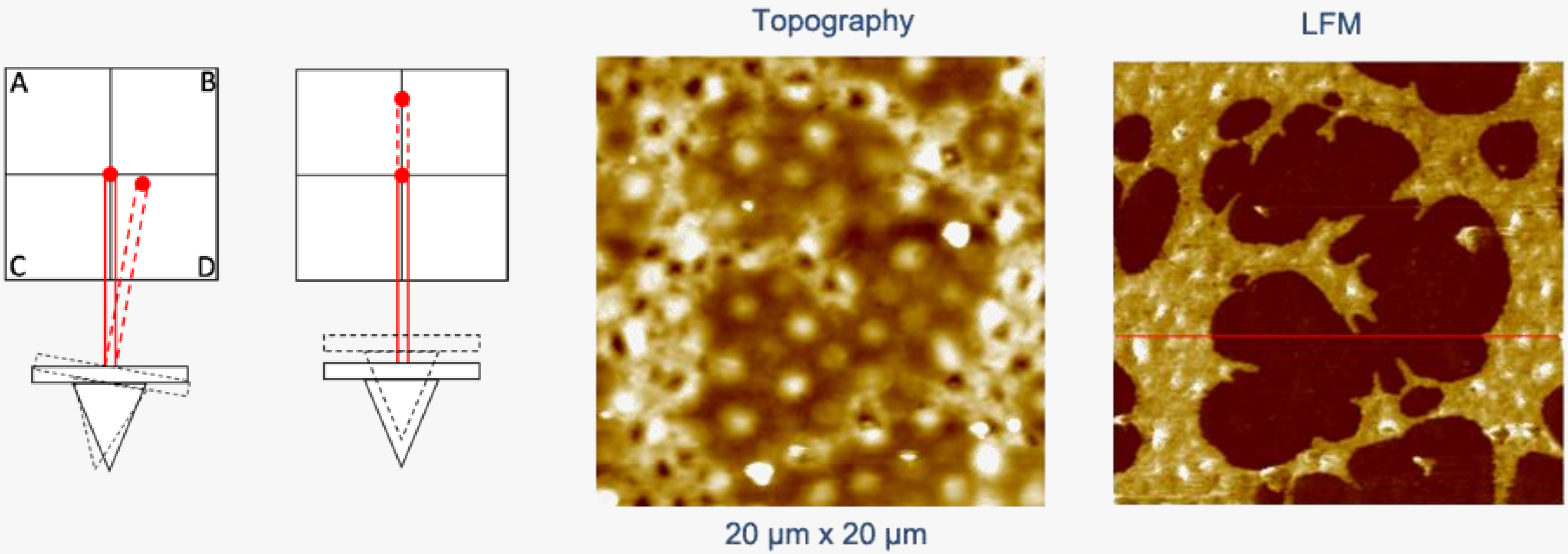
Lubricant on glass. Non-contact (dynamic) mode



Source: Park company

Lateral Force Microscopy (LFM)

Polymer on glass. Contact mode and lateral force

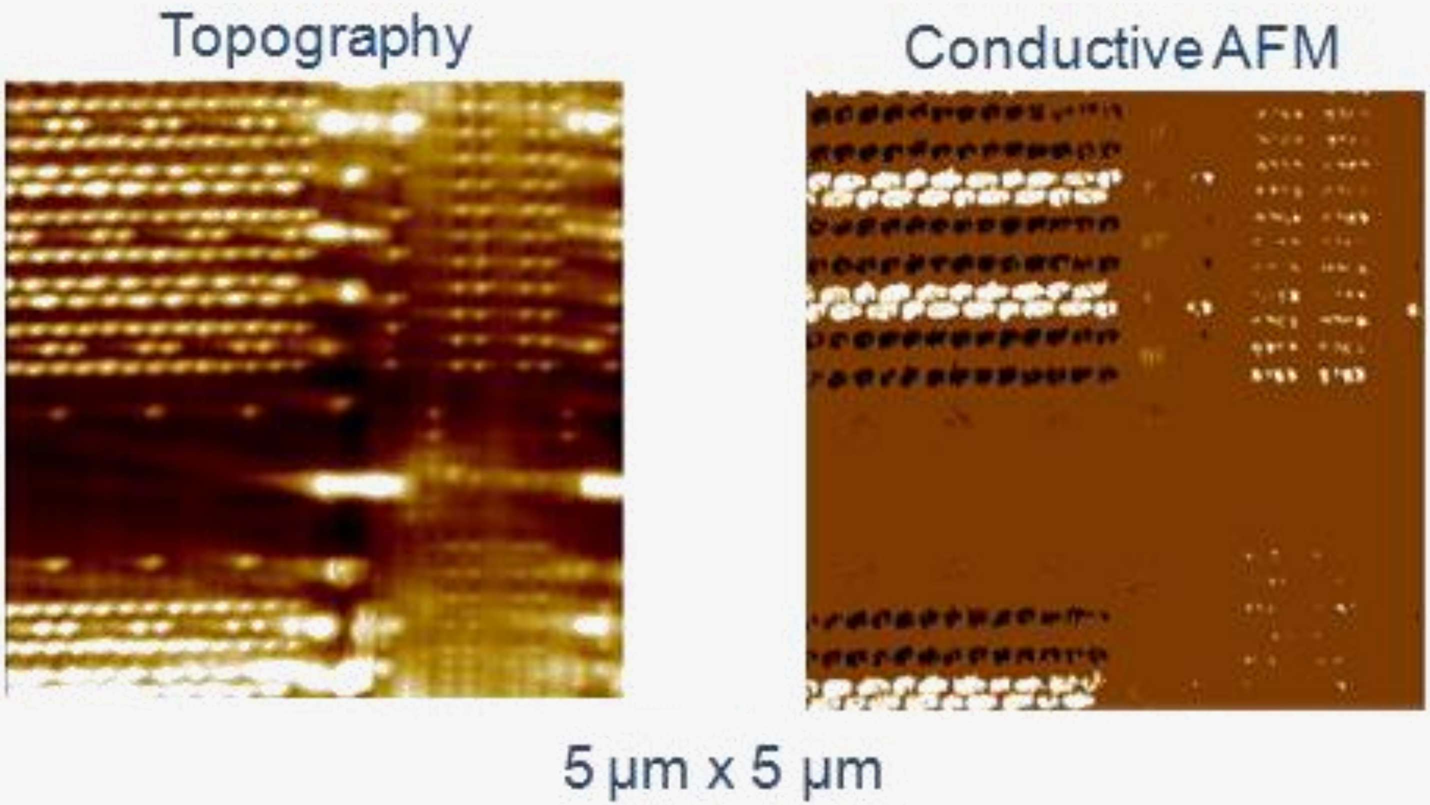


- Information: friction

Source: Park company

Conductive AFM

Top view on an SRAM chip

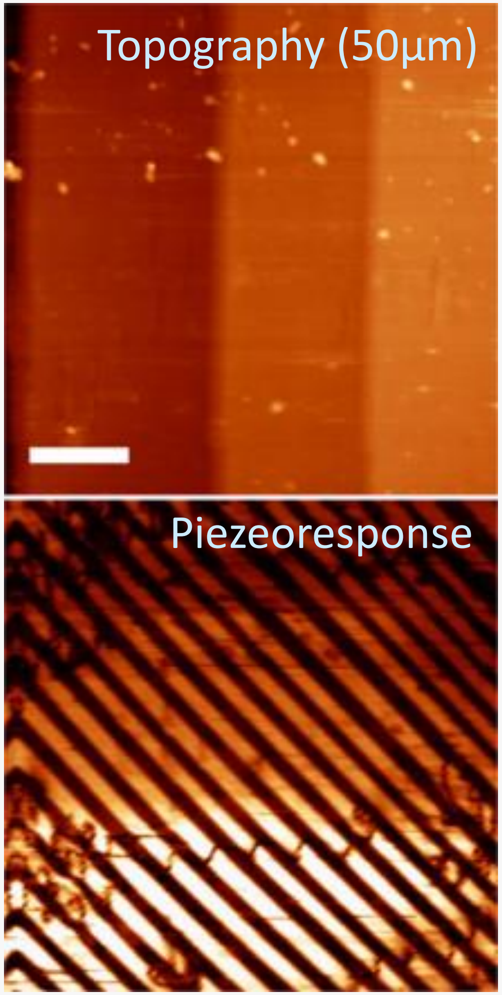


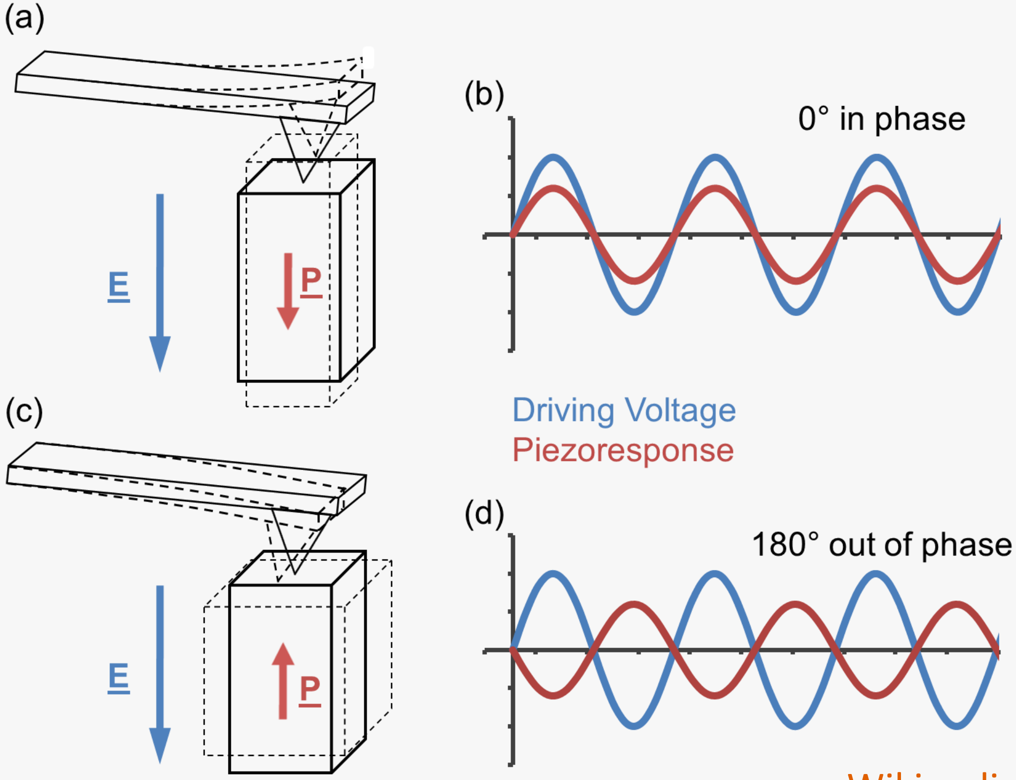
- Information: local conductivity

Source: Park company

Piezoresponse Force Microscopy (PFM)

BaTiO3 (ferroelectric material)





Wikipedia

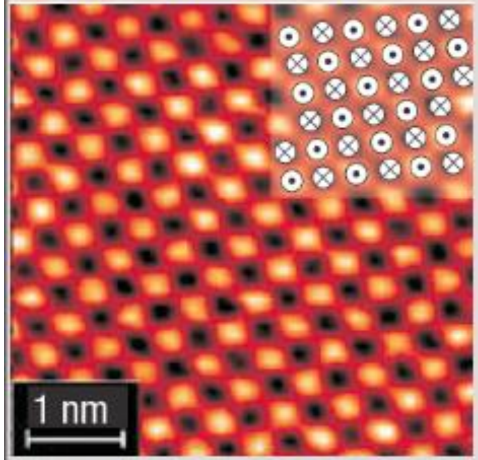
- Ferroelectric polarization and domains

Invention: P. Güthner et al., Appl. Phys. Lett. 61 (9), 1137 (1992)

Scanning Probe Microscopy - Magnetism

Spin-polarized STM

Fe(1ML)/W(001)



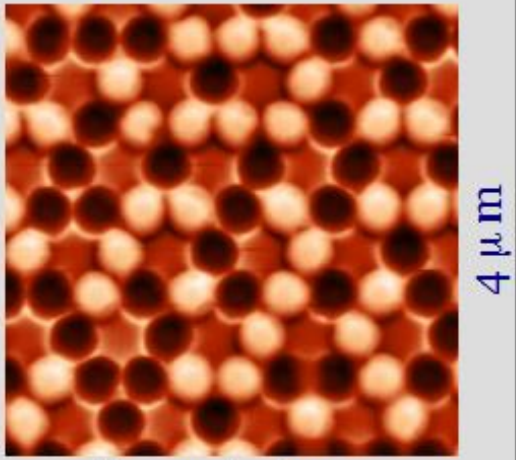
Antiferromagnetic domain

M. Bode et al., Nat. Mater. 5, 477-481 (2006)

REVIEW :
R. Wiesendanger, Rev. Mod. Phys. 81, 1495 (2009)

Magnetic Force Microscopy

Array of dots



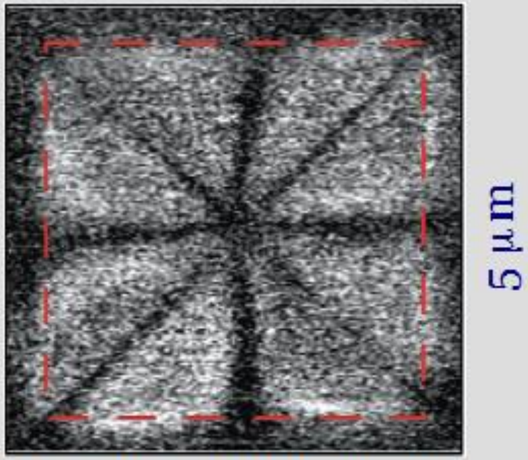
Up-and-down 'single-domains'

NEEL, sample courtesy:
N. Rougemaille, I. Chioar

REVIEW :
R. Proksch et al., Modern techniques for characterizing magnetic materials, Springer, p.411 (2005)

NV center microscopy

Square Fe20Ni80 dot



Signature of flux-closure

L. Rondin et al., arXiv:1302.7307

Others : scanning Hall probe, near-field optical etc.

Overview
- Large variety
- Rather slow

End of the general presentation

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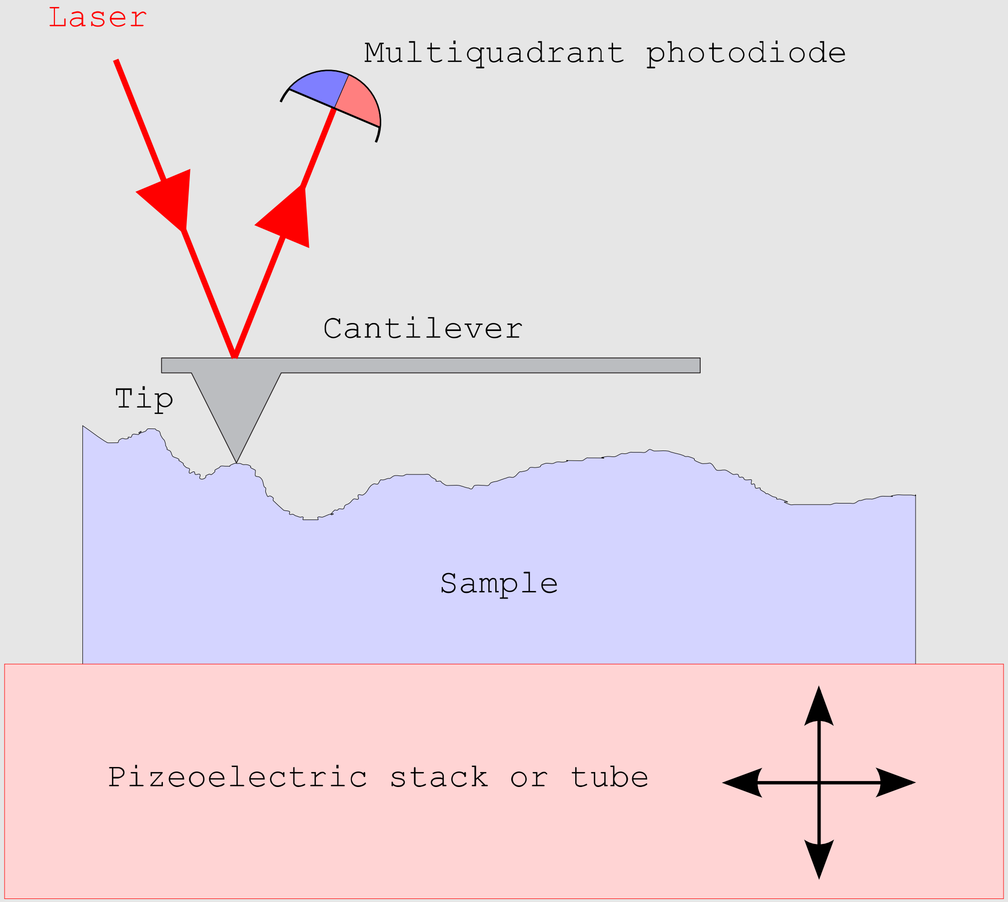
Suggested reading: lectures of Hans Hug (EMPA) at ESONN school: https://www.esonn.fr/2016-lectures

Key elements of an Atomic Force Microscope (AFM)

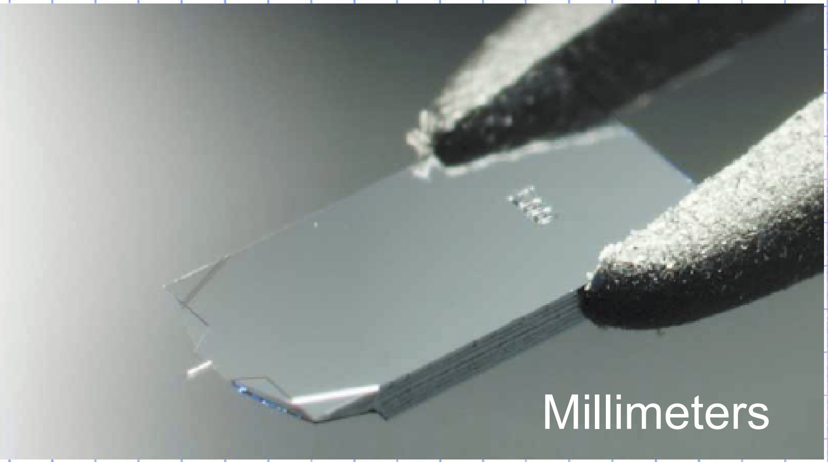


G. Binnig et al., Phys. Rev. Lett. 56, 930-933 (1986)

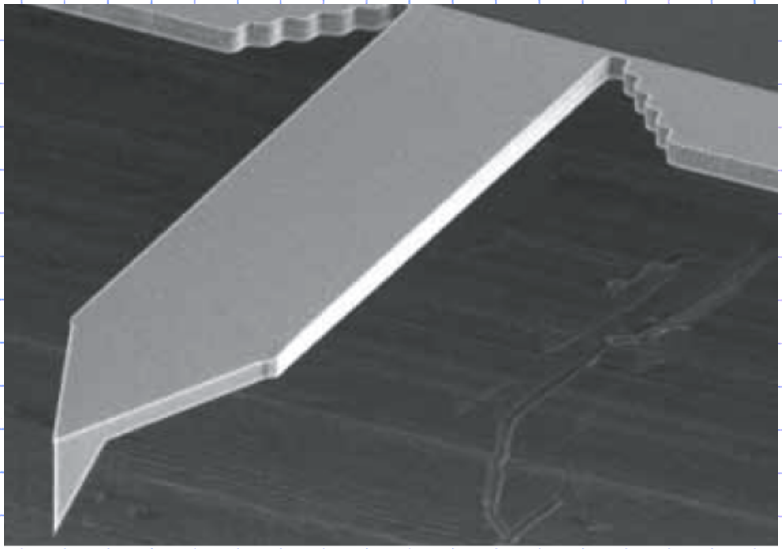
Overview

Measures forces (vertical and lateral) between sample and tip

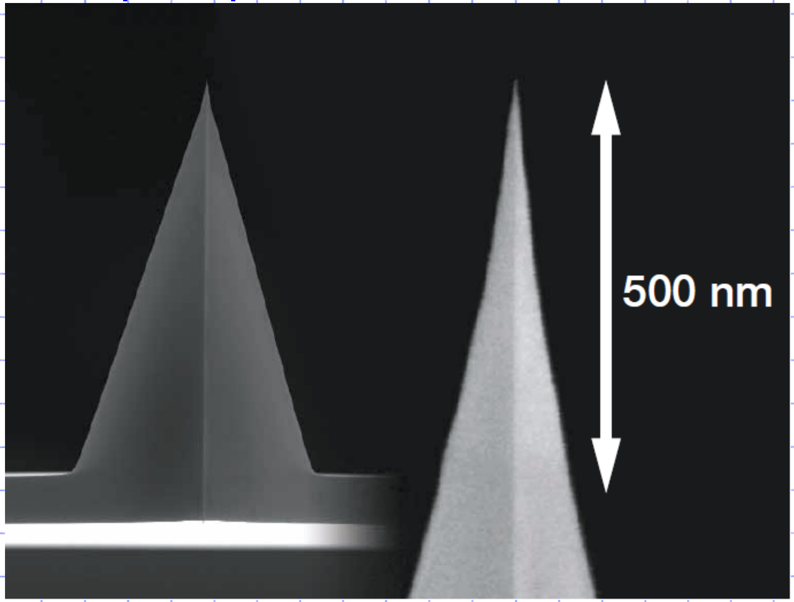
Chip – Batch fabrication



Cantilever



Full tip + apex

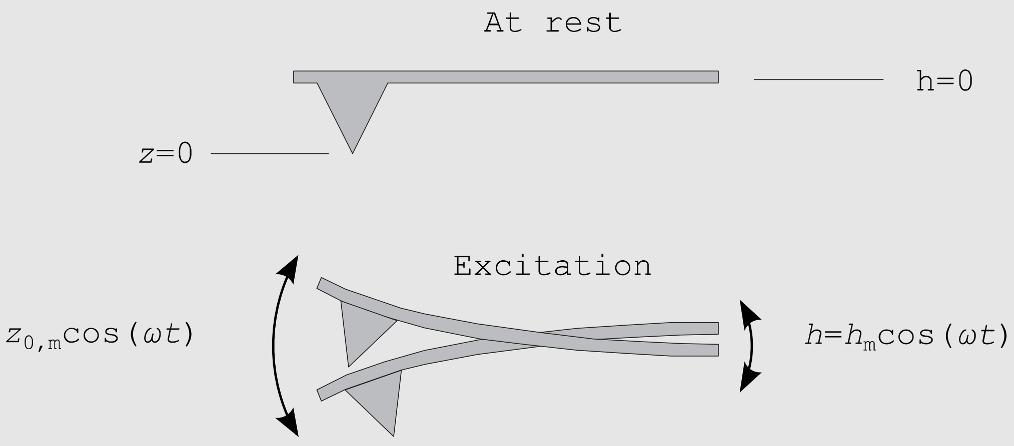


Overview

- Price 10-200eur/tip
- Radius of curvature ≈ 5nm

Images : Olympus catalog (http://www.olympus.co.jp/probe)

Mechanical excitation of cantilevers



$$m\ddot{z}+\Gamma\dot{z}+kz=F(z,t)$$

m	Inertia
Γ	Damping
k	Spring
$F(z,t)$	External force

Notations

Seek solutions for $F=0$ $\quad z(t)=z_0 e^{j\omega t}$

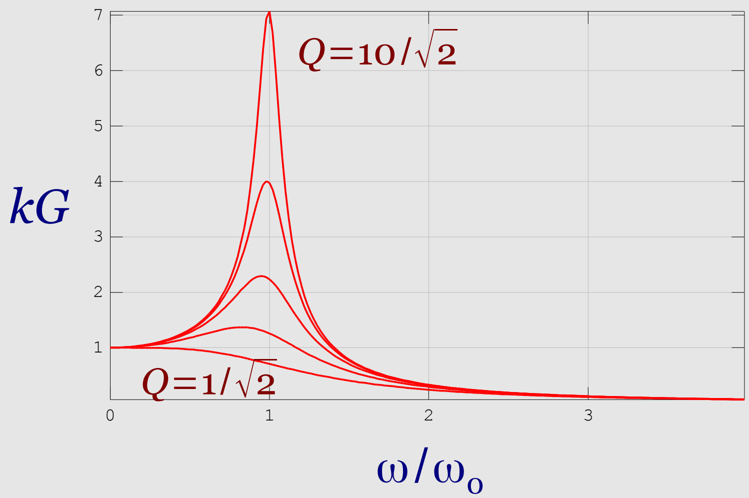
Reference angular velocity $\quad \omega_0=\sqrt{\dfrac{k}{m}}$

Quality factor $\quad Q=\dfrac{\sqrt{k\,m}}{\Gamma}$

⇨ Transfert function

$$H=\frac{z}{F}=\frac{1}{k}\frac{1}{-\left(\frac{\omega}{\omega_0}\right)^2+\frac{j}{Q}\left(\frac{\omega}{\omega_0}\right)+1}$$

Gain



$$G=|H|=\frac{1}{k}\frac{1}{\sqrt{\left[1-\left(\frac{\omega}{\omega_0}\right)^2\right]^2+\frac{1}{Q^2}\left(\frac{\omega}{\omega_0}\right)^2}}$$

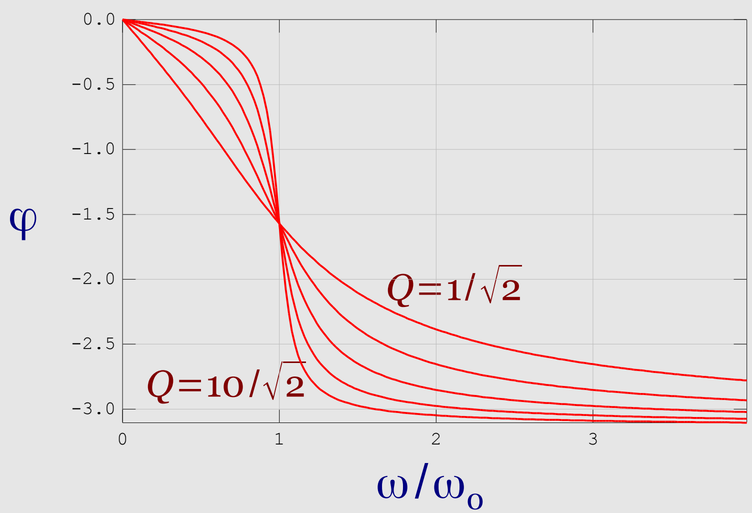
Peak at : $\omega_r=\omega_0\sqrt{1-\frac{1}{2Q^2}}$

$$kG(0)=1$$

$$kG(\omega_r)=Q$$

$$kG(\infty)=0$$

Dephasing



$$\cos\varphi=\frac{1-\left(\frac{\omega}{\omega_0}\right)^2}{\sqrt{\left[1-\left(\frac{\omega}{\omega_0}\right)^2\right]^2+\frac{1}{Q^2}\left(\frac{\omega}{\omega_0}\right)^2}}$$

$$\varphi(0)=0$$

$$\varphi(\infty)=-\pi$$

Q>>1

$$\omega_r\approx\omega_0$$

$$\varphi(\omega_r)\approx-\pi/2$$

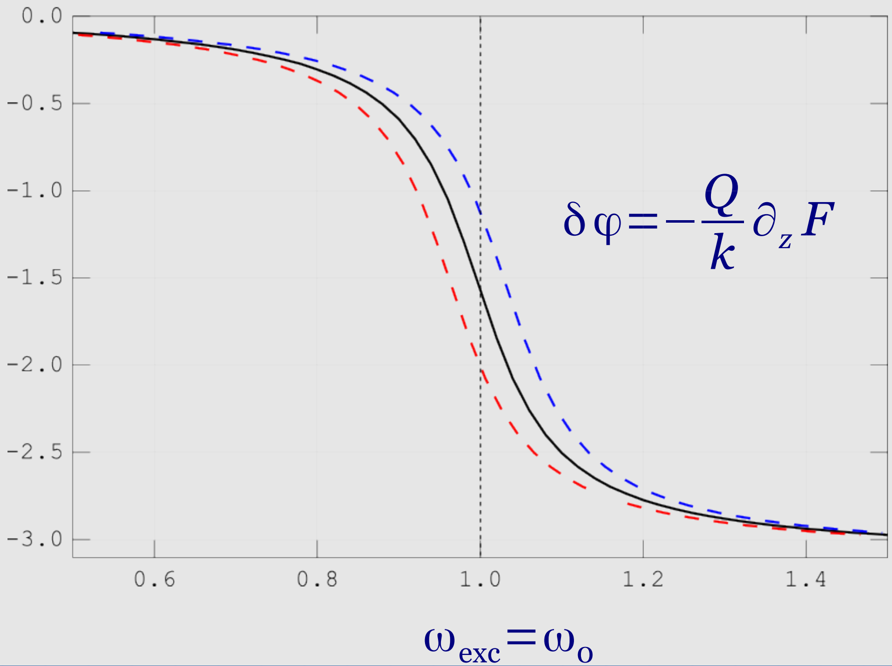
$$\frac{\Delta\omega_r}{\omega_0}\approx\frac{\sqrt{3}}{Q}$$

Tip-sample interaction treated as perturbation

$$m\ddot{z}+\Gamma\dot{z}+kz=F(z) \quad \text{with} \quad F(z)=F(z_o)+(z-z_o)\partial_z F$$

⇨ Mere renormalization : $\omega_{o,eff}=\omega_o\left(1-\frac{1}{2k}\partial_z F\right)$

Phase shift



Attractive force

⇨ Red shift

Repulsive force

⇨ Blue shift

- Forces monitored through phase shift
- Notice my convention : decreasing phase (may be set in the software)

MFM tips : AFM tip + magnetic coating

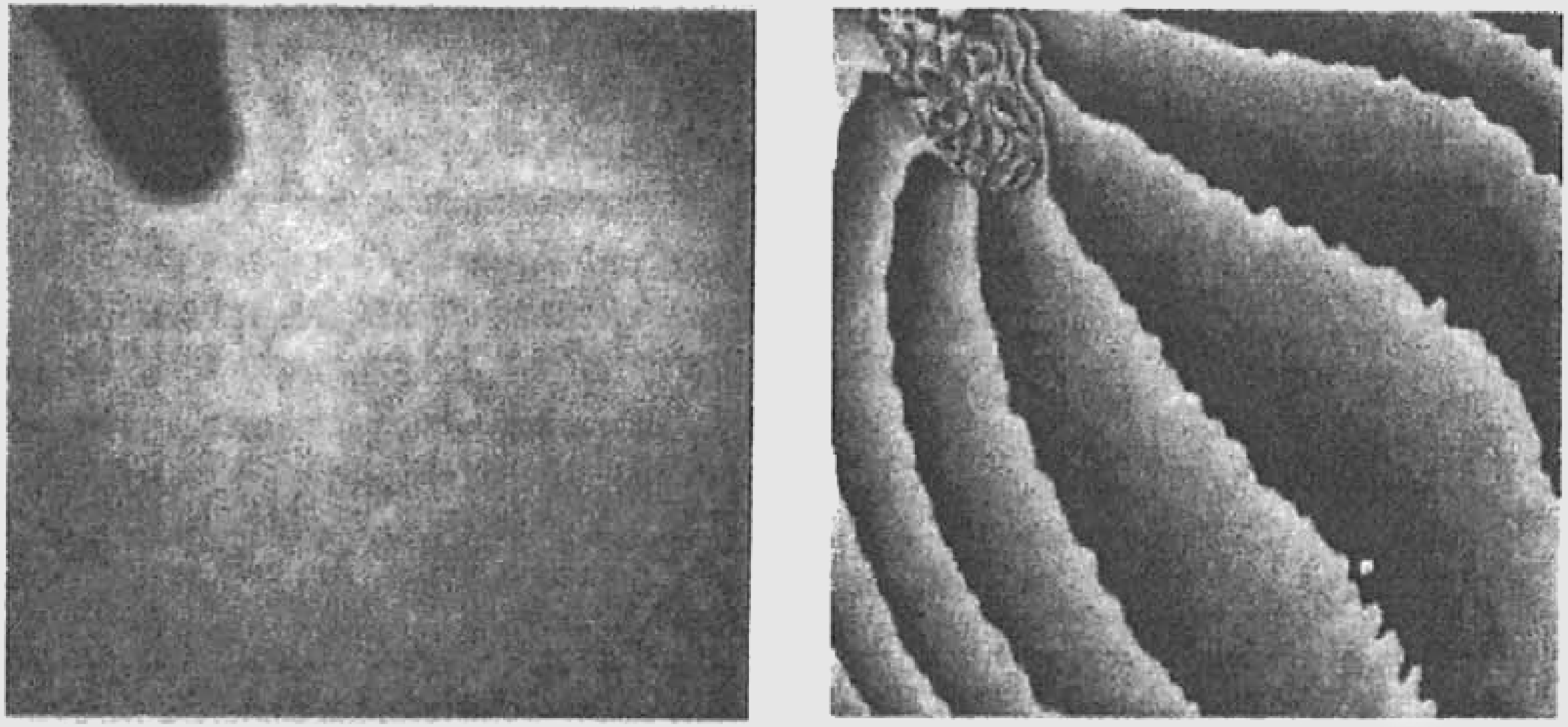
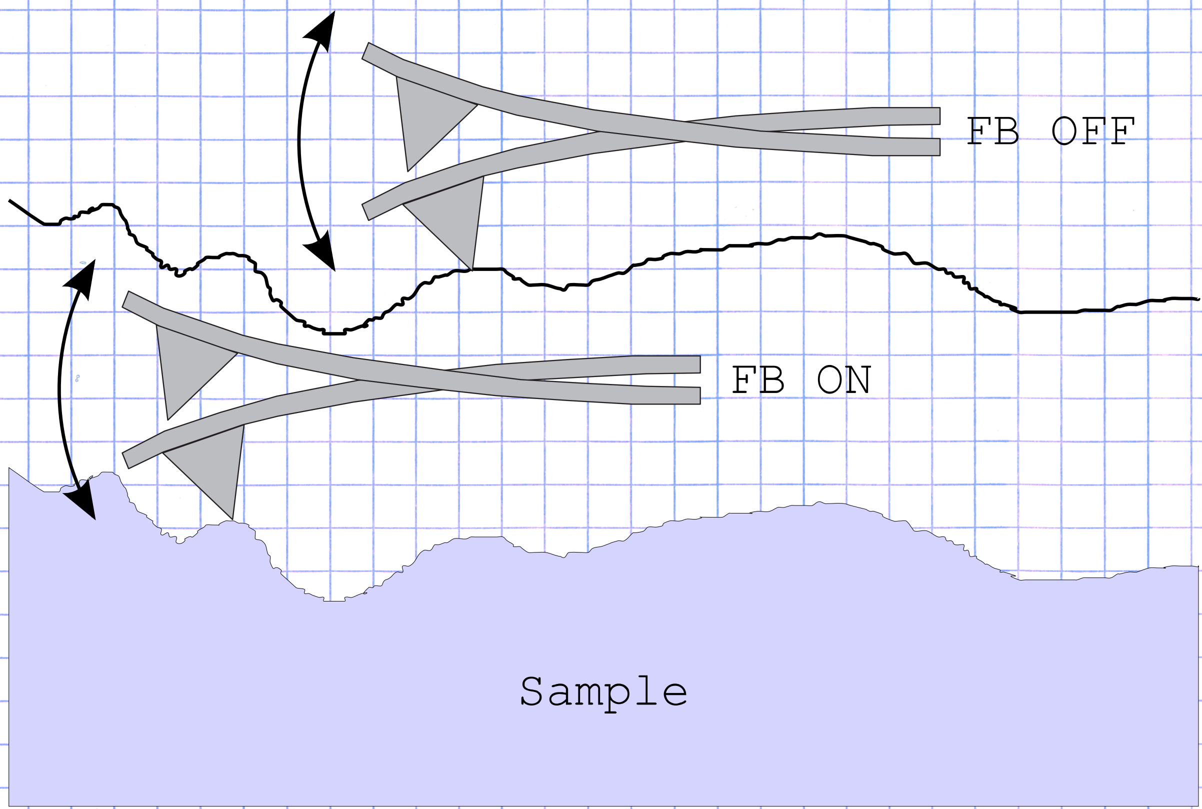
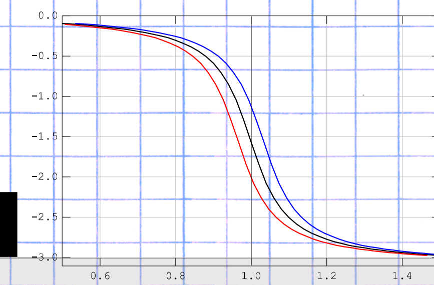


Figure 11-20: The electron amplitude (left) and phase (right) near an MFM tip visible as a dark shadow on the upper left corner of the left image.

R. Proksch et al., Modern techniques for characterizing magnetic materials, Springer, p.411 (2005)





Second pass

- Monitor long-range forces (magnetic, electrostatic)

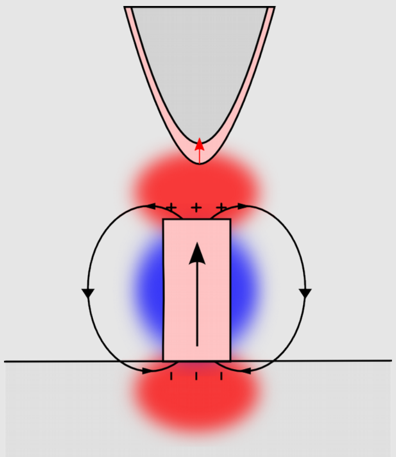
First-pass

- Feedback ON
- Monitor topography (height) and any other signal (phase etc.)

First report : **Y. Martin et al., Appl. Phys. Lett. 50, 1455 (1987)**

Review : **R. Proksch et al., Modern techniques for characterizing magnetic materials, Springer, p.411 (2005)**

Tip is a dipole

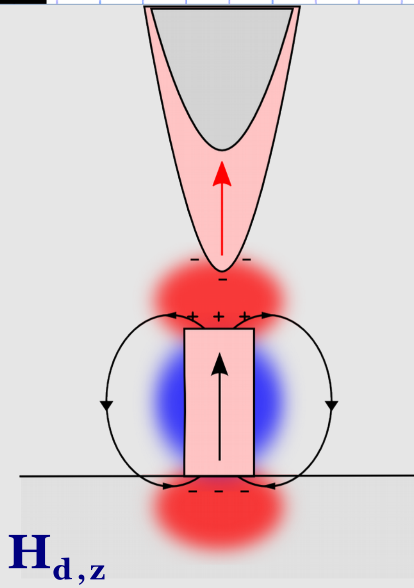


$$\mathcal{E}_{1,2} = -\mu_0 \boldsymbol{\mu}_2 . \mathbf{H}_\mathrm{d}$$

$$\mathcal{E}_{1,2} = -\mu_0 \left(\mu_x . H_{\mathrm{d},x} + \mu_y . H_{\mathrm{d},y} + \mu_z . H_{\mathrm{d},z}\right)$$

$$\Rightarrow \quad \delta\varphi = \frac{Q}{k}\mu_0 \mu_i \partial_z^2 H_{\mathrm{d},i}$$

Tip is a monopole



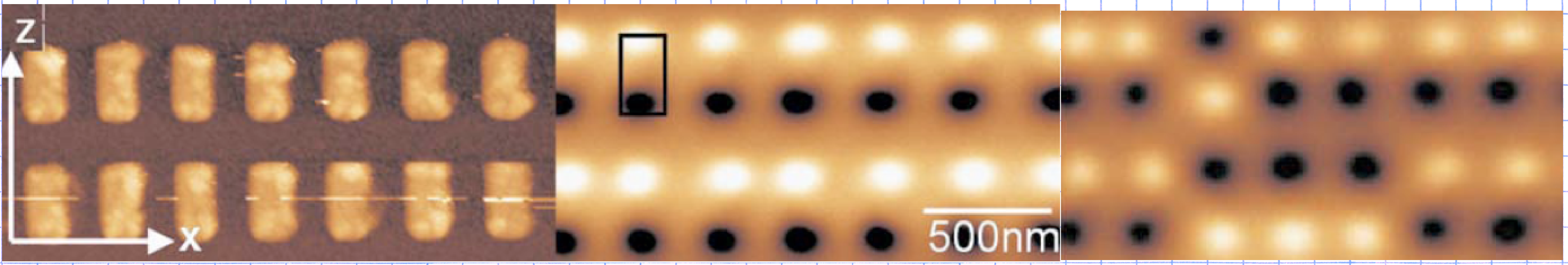
$$\mathcal{E}_{1,2} = \mu_0 \sigma . \phi$$

$$\mathbf{F}_\mathbf{z} = -\mu_0 \sigma \mathbf{H}_{\mathbf{d},\mathbf{z}}$$

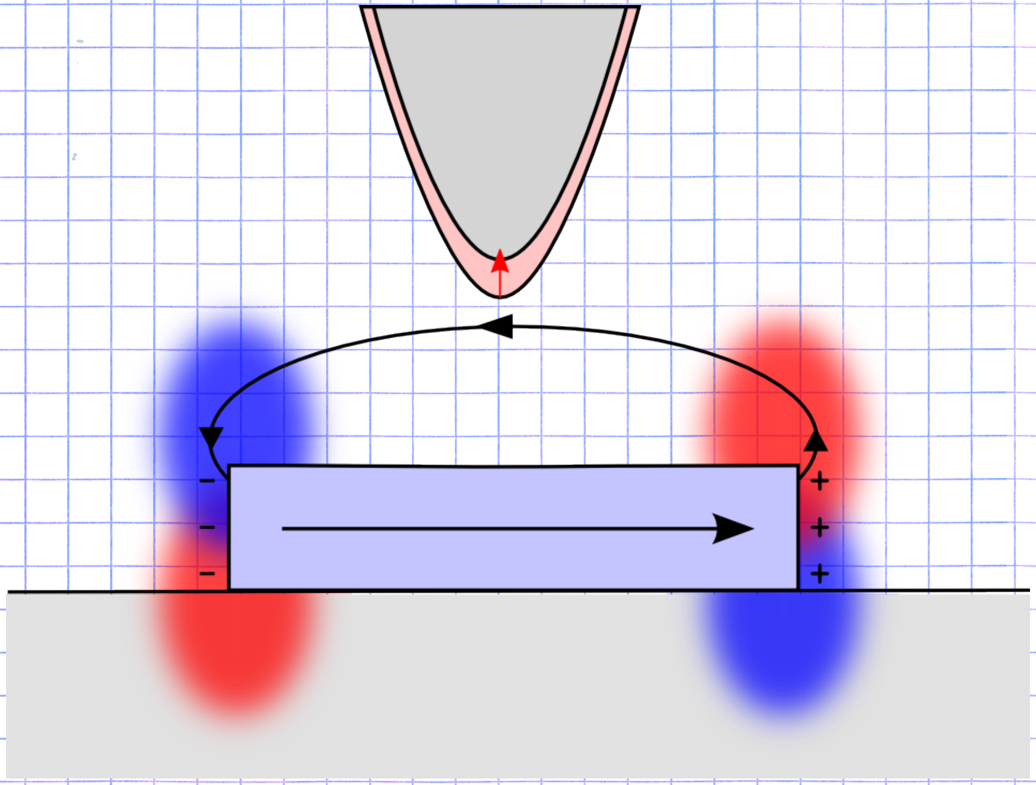
$$\Rightarrow \quad \delta\varphi = \frac{Q}{k}\mu_0 \sigma \partial_z H_{\mathrm{d},z}$$

- In practice, a combination of both models is best suited (dipole is more important)
- MFM is sensitive to some derivative(s) of the stray field from the sample
- MFM may be sensitive to in-plane field, depending on the tip magnetic moment

Topography MFM, saturated MFM, partly reversed



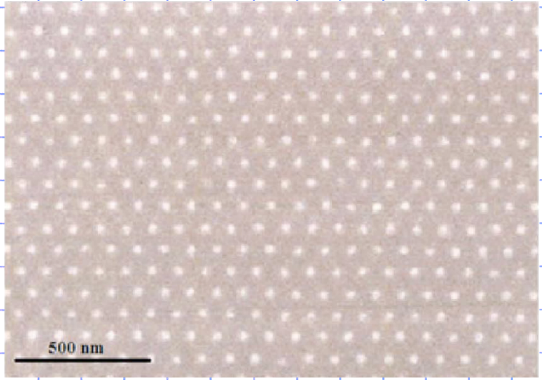
S. Y. Suck et al., APL95, 162503 (2009)

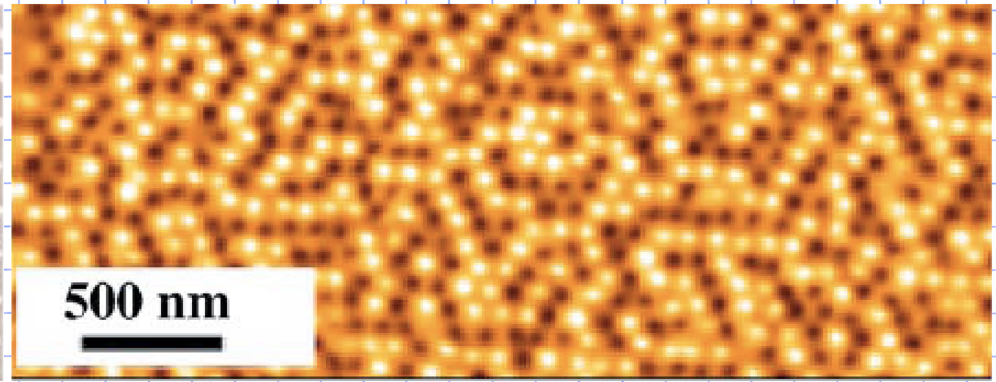


Single-domain in-plane magnetized dots appear as dipoles

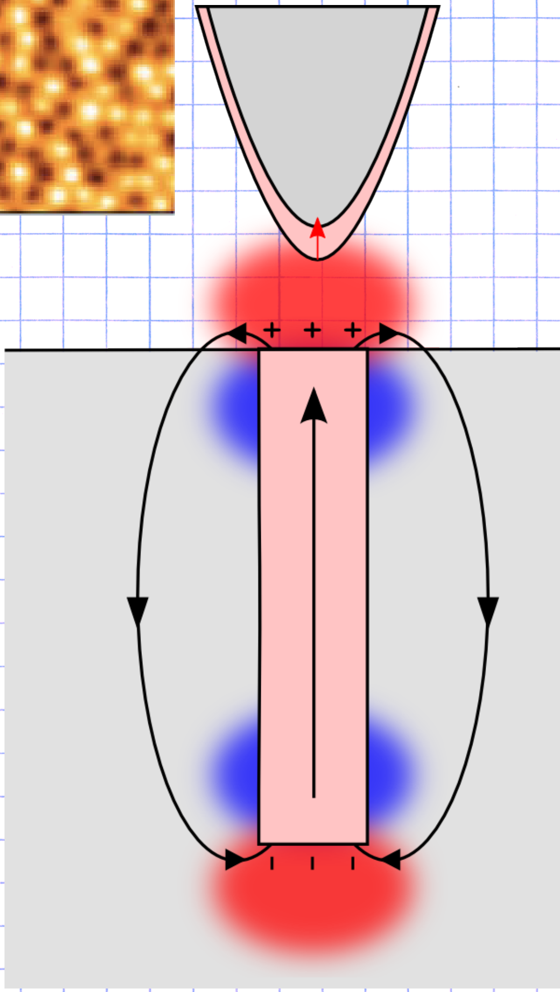
Structure (SEM)

MFM, partly reversed

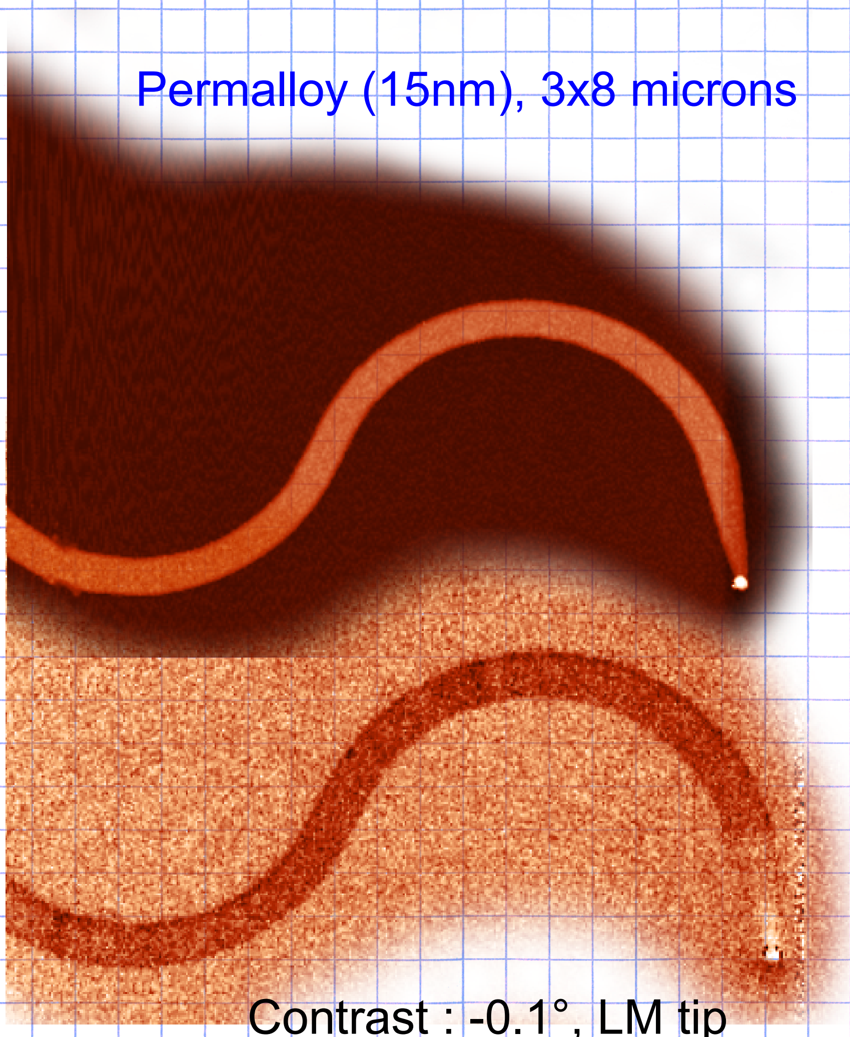




T. Wang et al., APL 92, 192504 (2008)

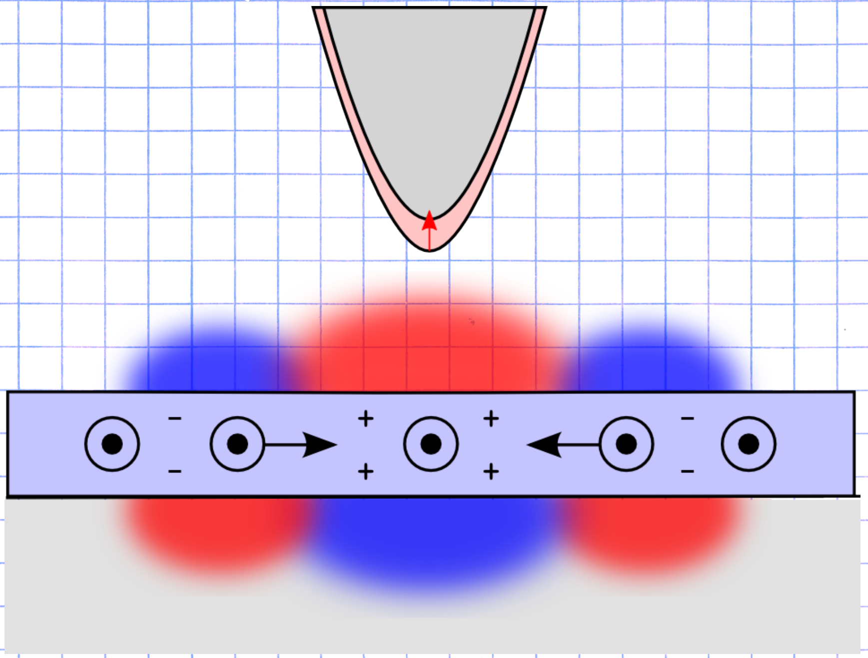


↳ Single-domain out-of-plane magnetized dots appear as monopoles



Lithography : S. Pizzini (Institut Néel)

Imaging : Z. Ishaque (Institut Néel)



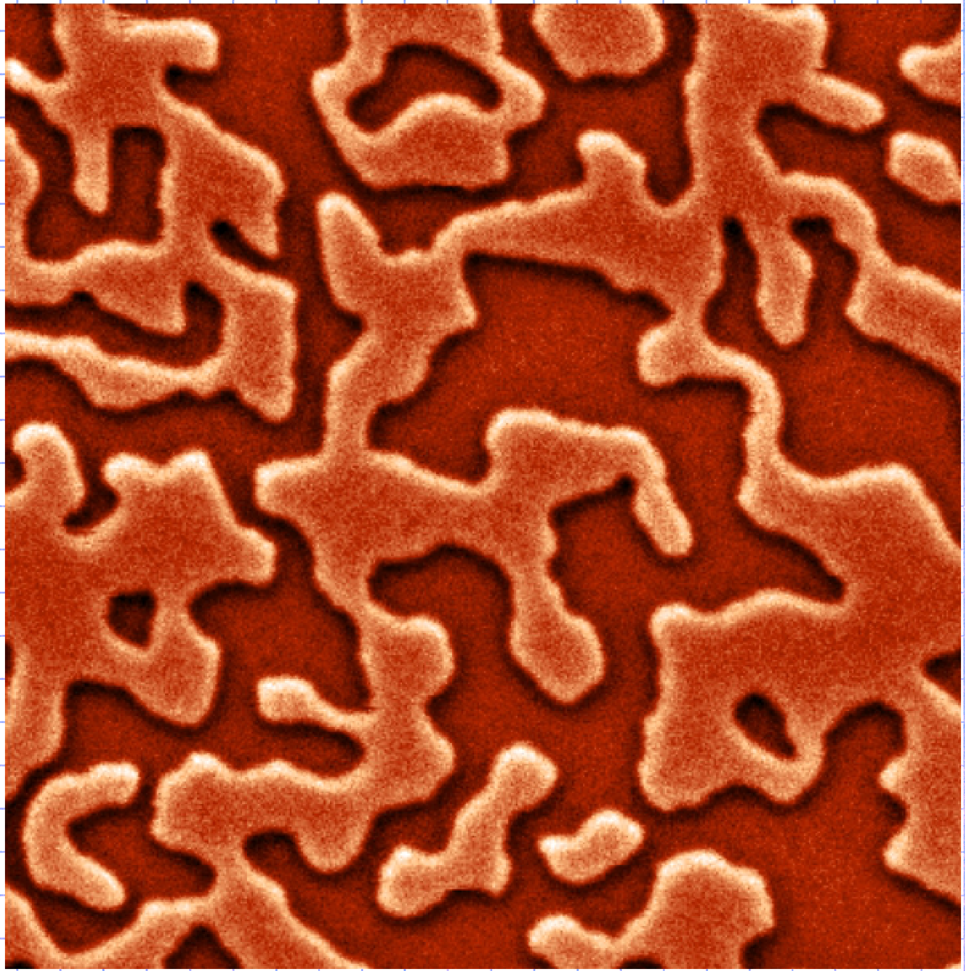
Principle :

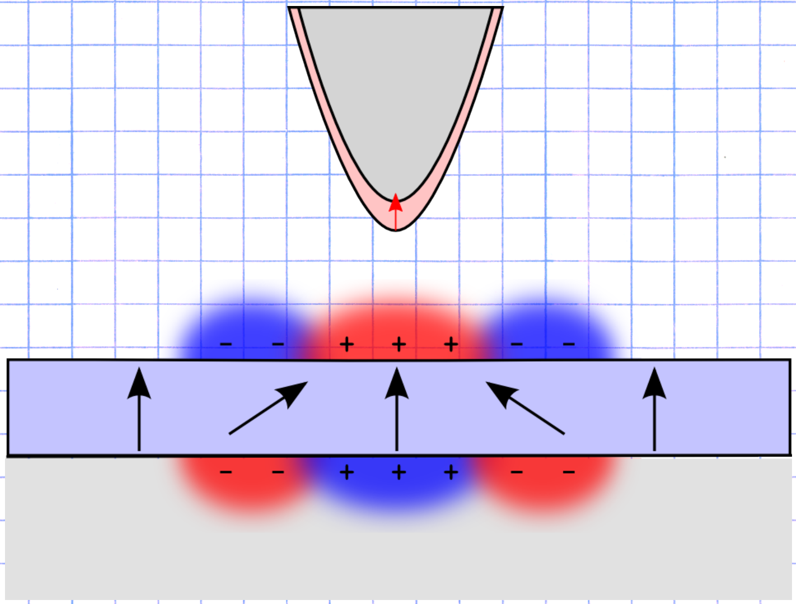
1. Stray field magnetizes sample
2. Sample is non -uniform → stray field
3. Tip measures sample's stray field

- It is a DOMAIN contrast
- Interaction is ALWAYS attractive : red shift
- Contrast is proportionnal to the square of the tip moment

FePt (4nm)

5x5 μm





It is a DOMAIN contrast

The direction of magnetization is deduced

Sample : A. Marty (CEA-Grenoble)

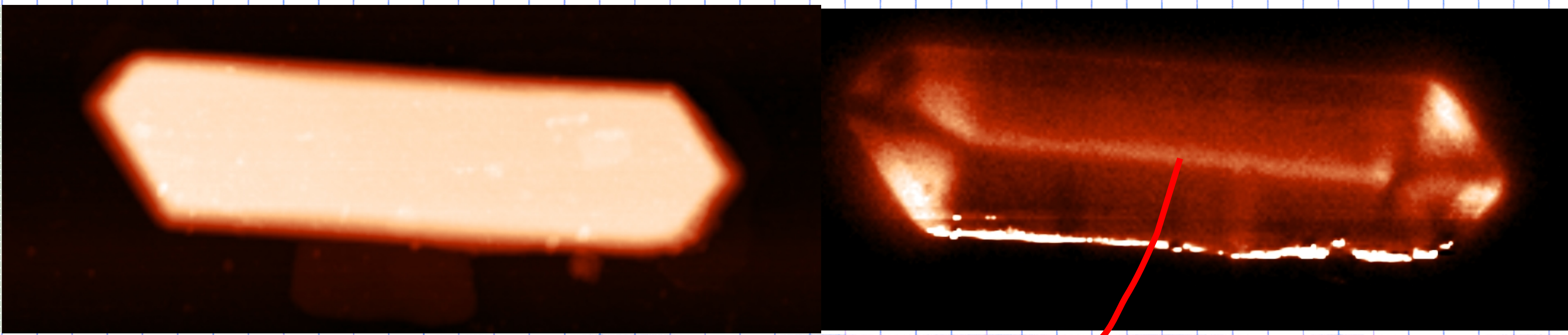
Imaging : M. Darques (Institut Néel)

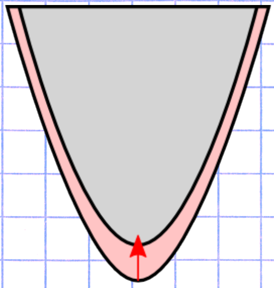
Contrast : ±0.4°, LM tip

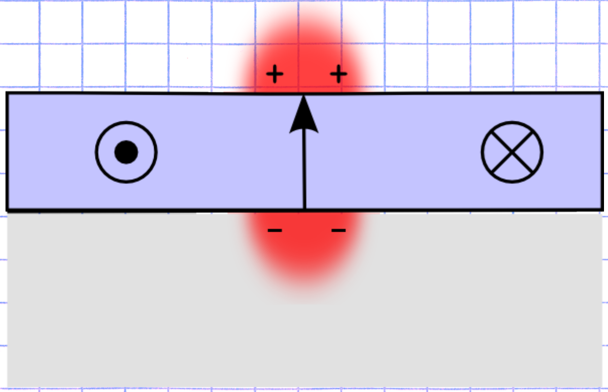
Quantitative analysis :

L. Belliard et al., J. Appl. Phys. 81, 3849 (1997)

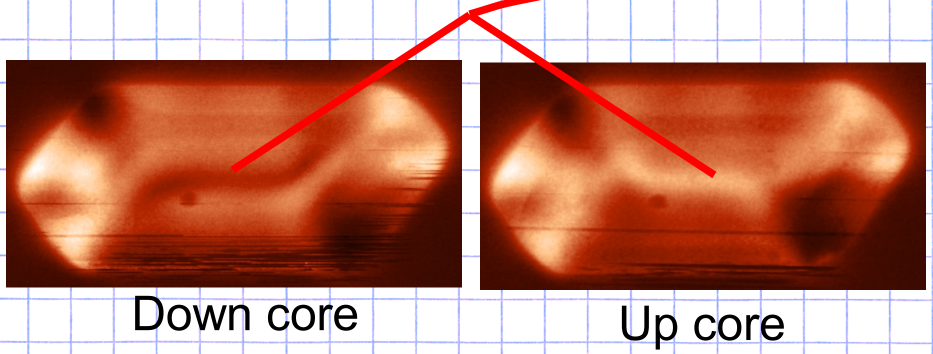
Fe dot (25nm), 2.5x1 microns





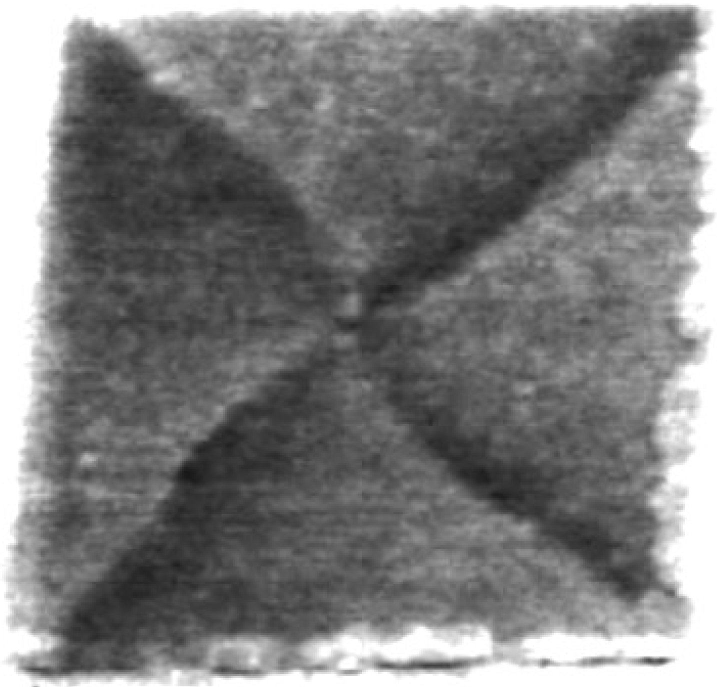


- Contrast is MONOPOLAR
- Informs about the polarity of the wall core

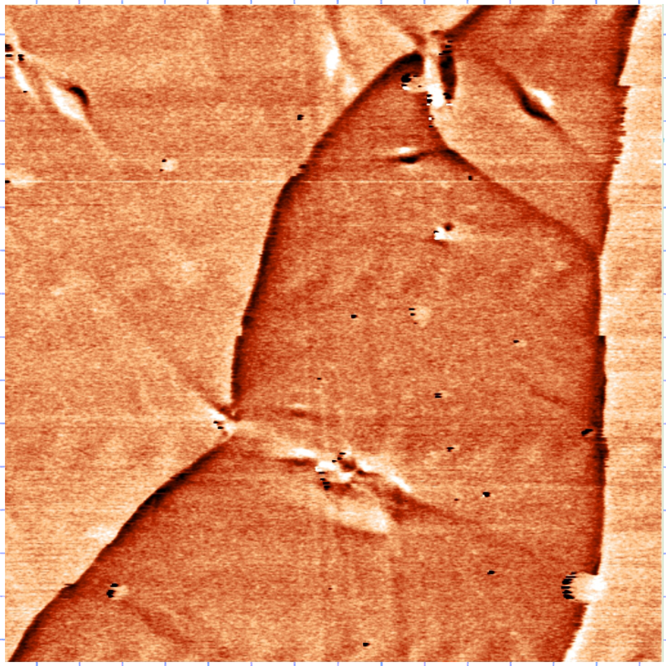


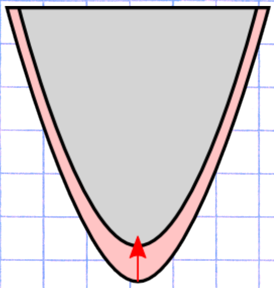
Permalloy dot (16nm)
2x2 microns

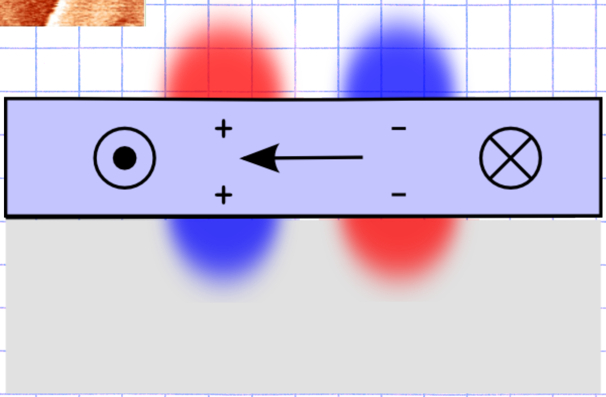
Permalloy film (20nm)
10x10 microns



J. M. Garcia et al., APL 79, 656 (2001)

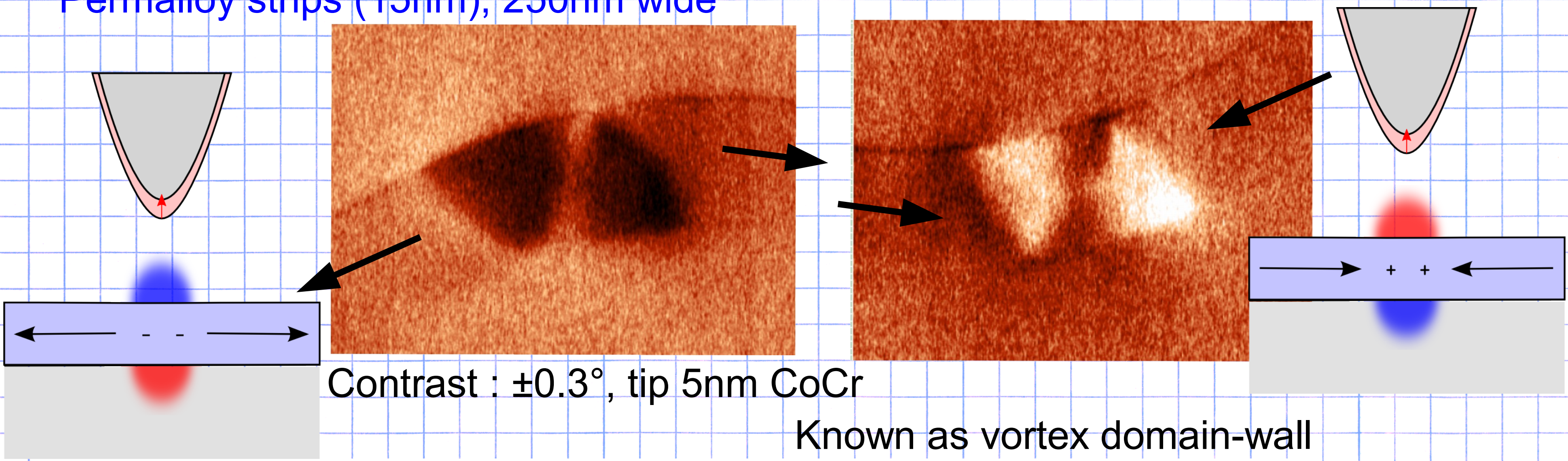






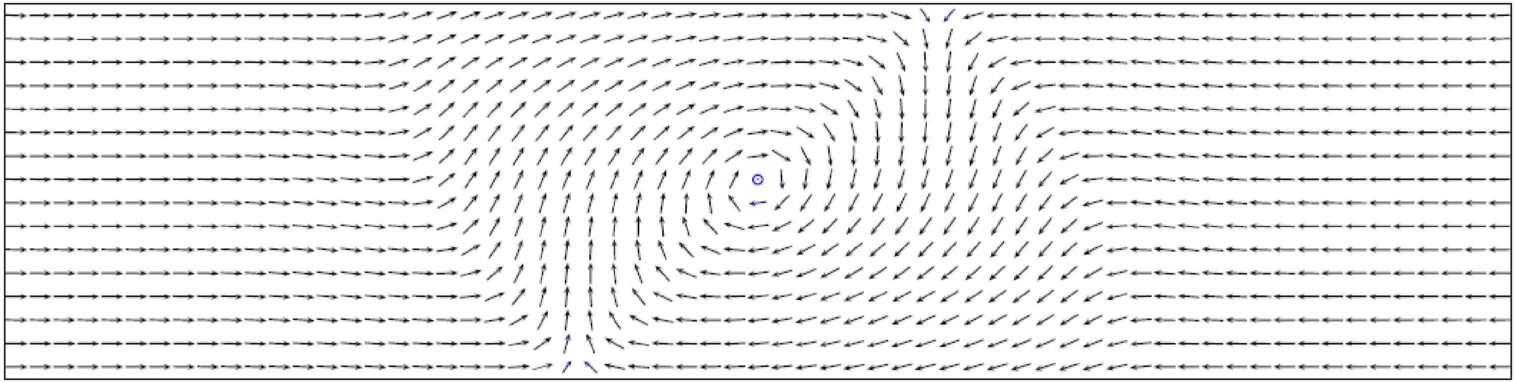
- Néel wall give rise to DIPOLAR contrast
- Informs about the chirality of the wall core

Permalloy strips (15nm), 250nm wide



Contrast : ±0.3°, tip 5nm CoCr

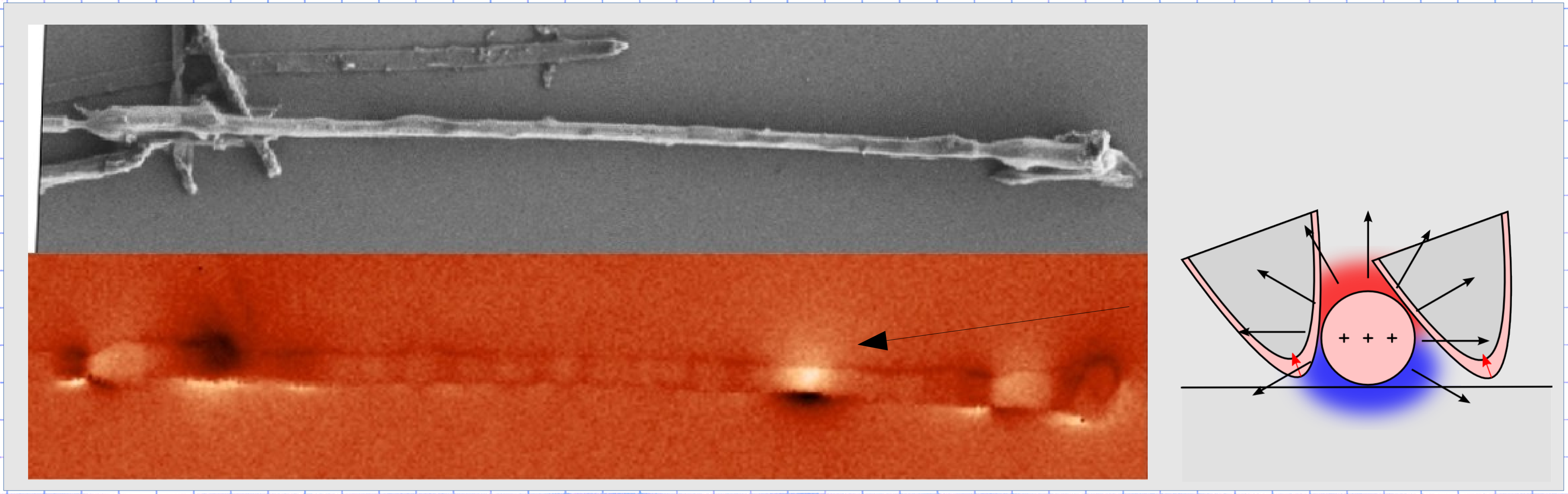
Known as vortex domain-wall



R. McMichael and M. Donahue, IEEE Trans. Magn. 33, 4167 (1997)

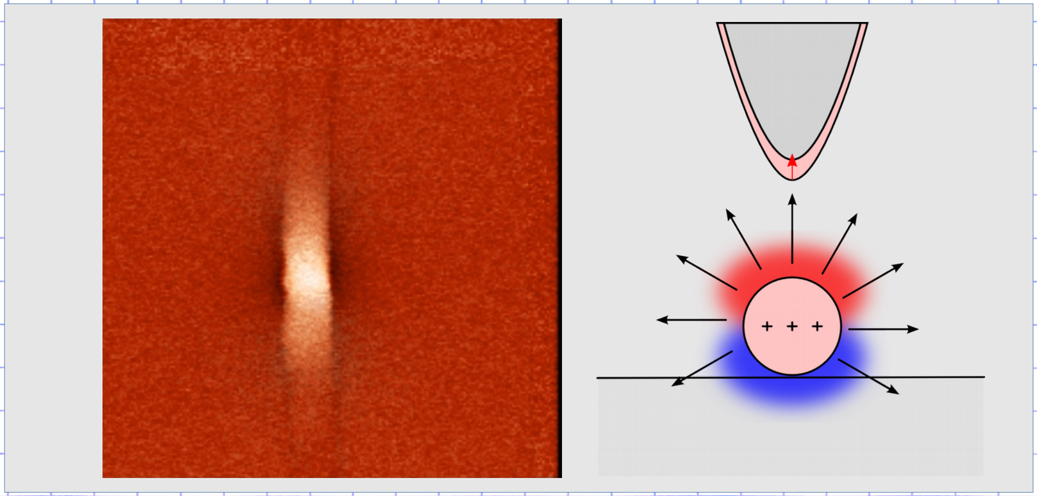
- Walls in in-plane magnetized stripes → MONOPOLAR
- Contrast informs about head-to-head ot tail-to-tail

Tilted cantilever, across wire



Tilted cantilever, along wire

@NEEL : S. Da Col et al. (in preparation)



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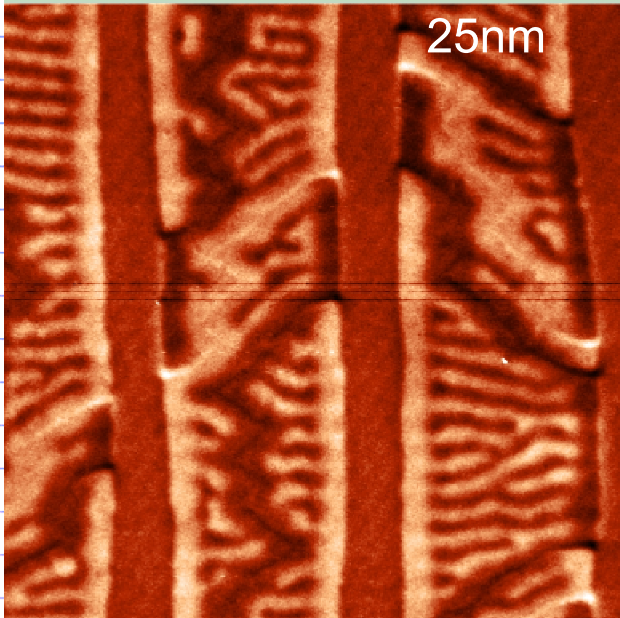
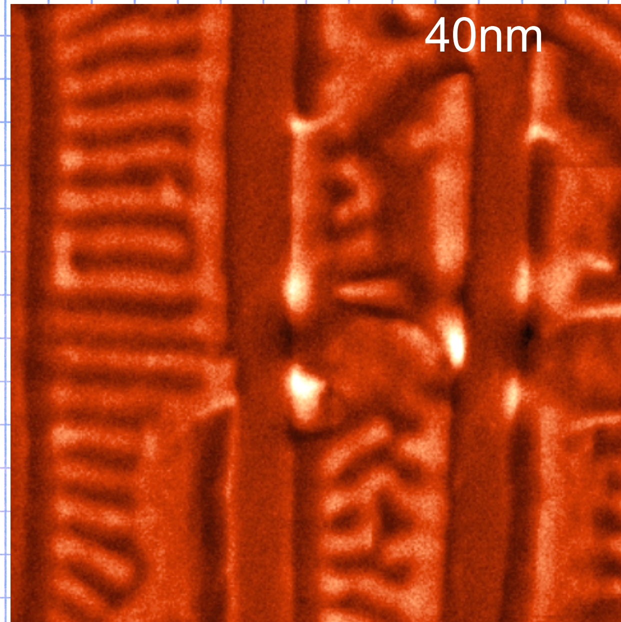
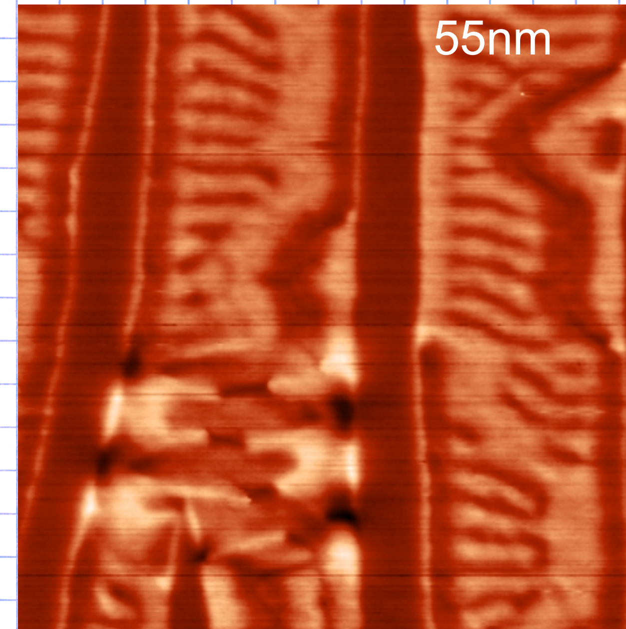
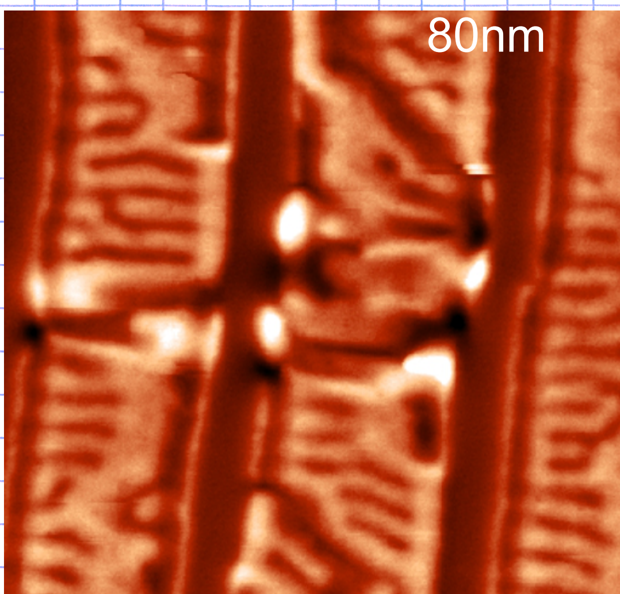
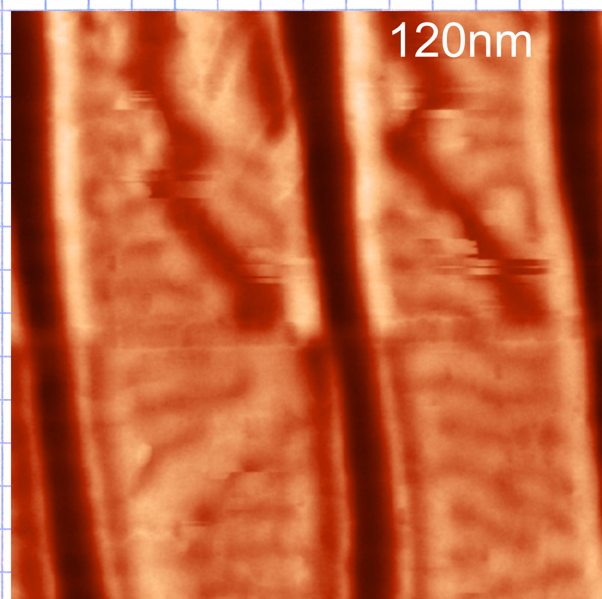
3. SOME DEVELOPMENTS / Calibration of tips and cantilevers (here : commercial)







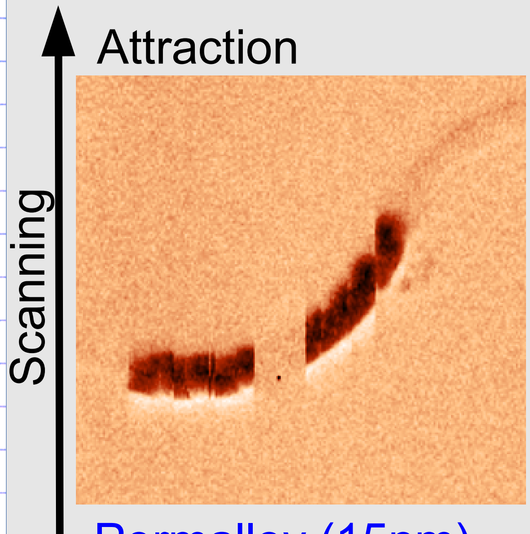




- Compare tips !
- Use identical sample and imaging parameters

Sample : MnAs(001)
R. Belkhou (Soleil)

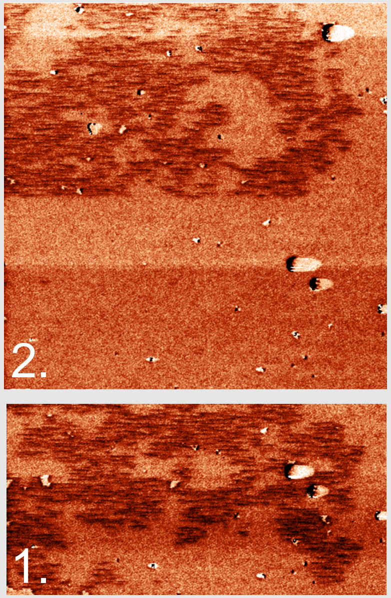
High



Permalloy (15nm)
500nm strips

Sample : S. Pizzini

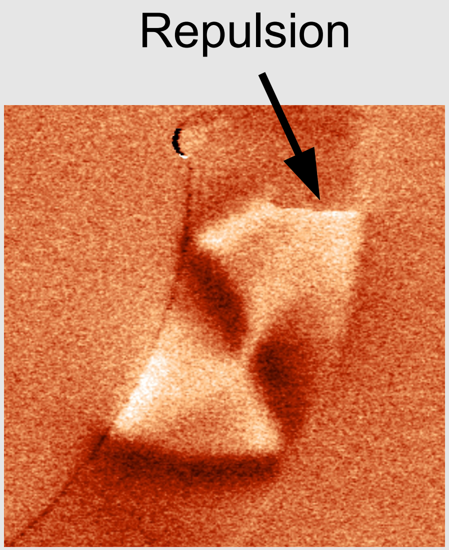
Imaging : Z. Ishaque



Co (0.6nm)/
graphene (20 mic)

C. Dieudonné

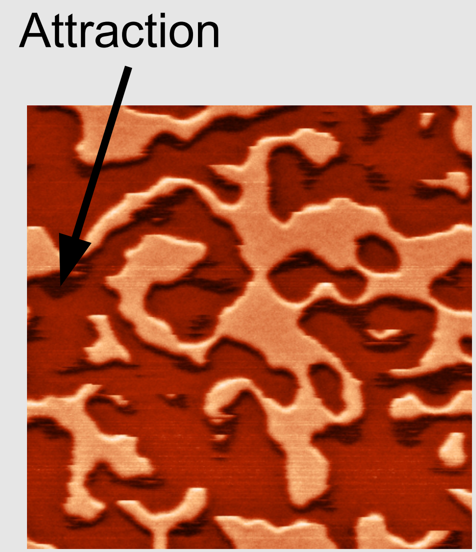
Moderate



Permalloy (15nm)
500nm wide

Sample : S. Pizzini

Imaging : Z. Ishaque



FePt (4nm)
Image 5 microns

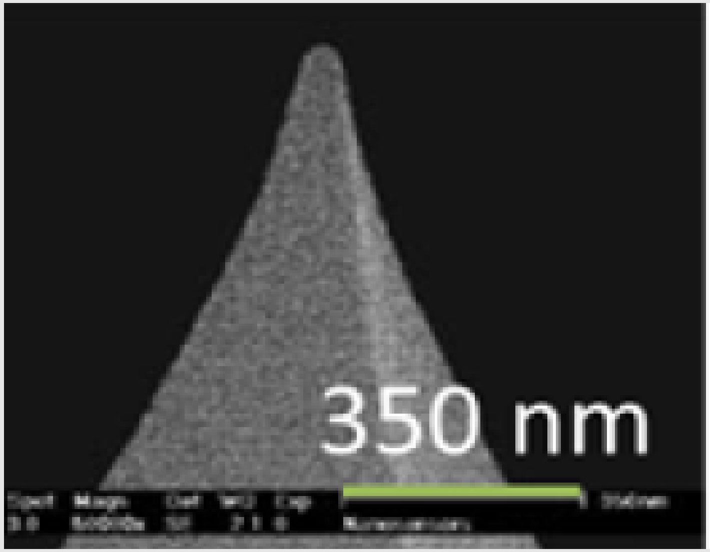
Sample : A. Marty

Imaging : M. Darques

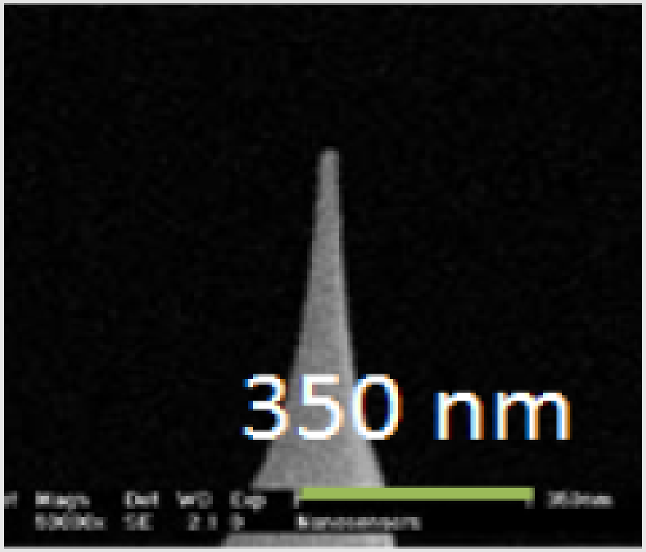
- Repeat measurement and/or change scanning direction
- Low-coercive samples require low-moment tips
- Commercial 'low-moment' may not be low enough

Improve resolution

From sharp to ultrasharp AFM tips



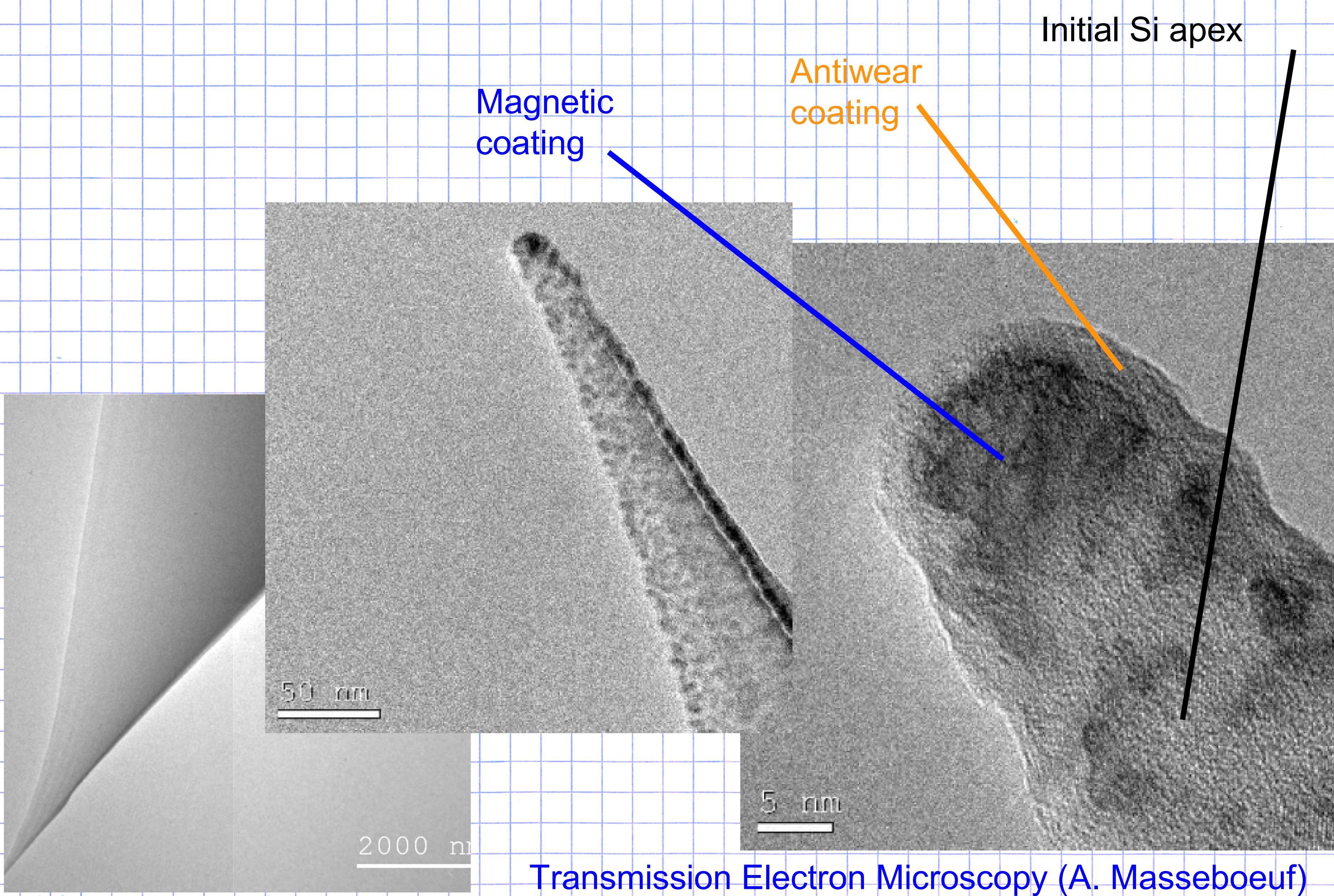
Asylum 240TS
Radius of curvature : 10 nm



Nanosensors PPP-SSS
Radius of curvature : 2-5 nm

Engineer magnetic coating

- Basis : $Co_{80}Cr_{20}$ (similar to hard disk media)
- Means : sputtering, DP850.
- Stacking : Based on CoCr [2-50 nm], various buffer and capping layers

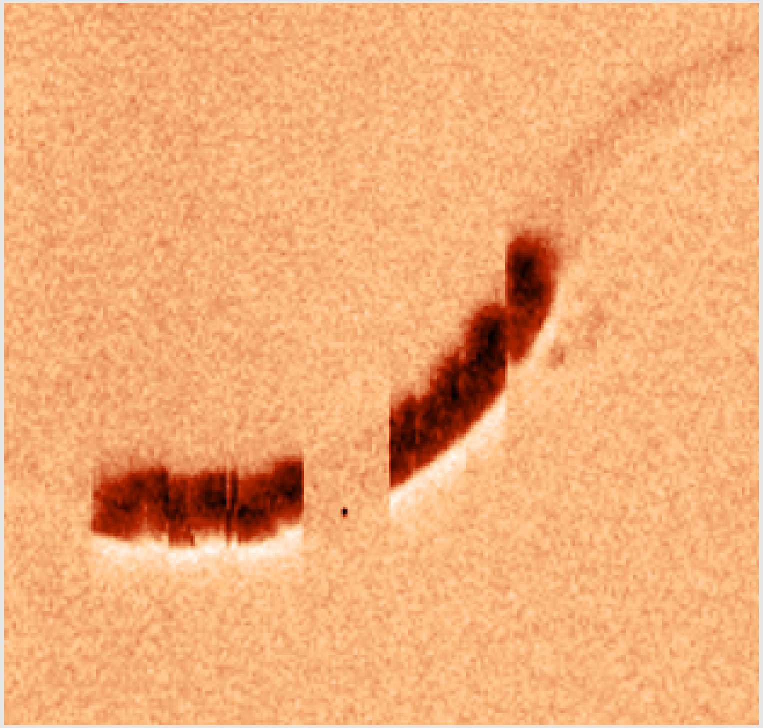


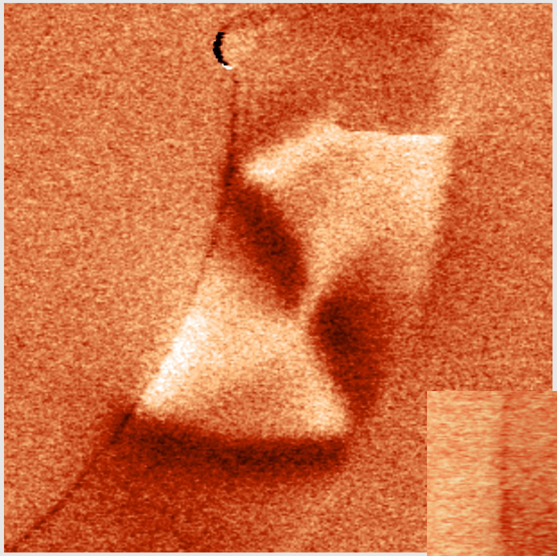
Transmission Electron Microscopy (A. Masseboeuf)

Commercial tips, 'low moment'

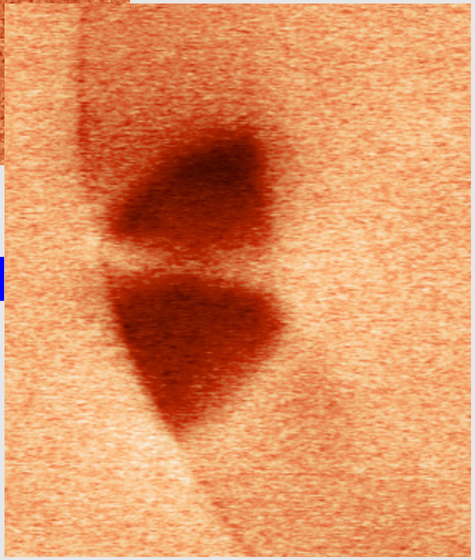
Permalloy strips [15nm]

Home-made tips, really low moment





Tip : CoCr[10nm]
500nm strips



Tip : CoCr[5nm]
300nm strips

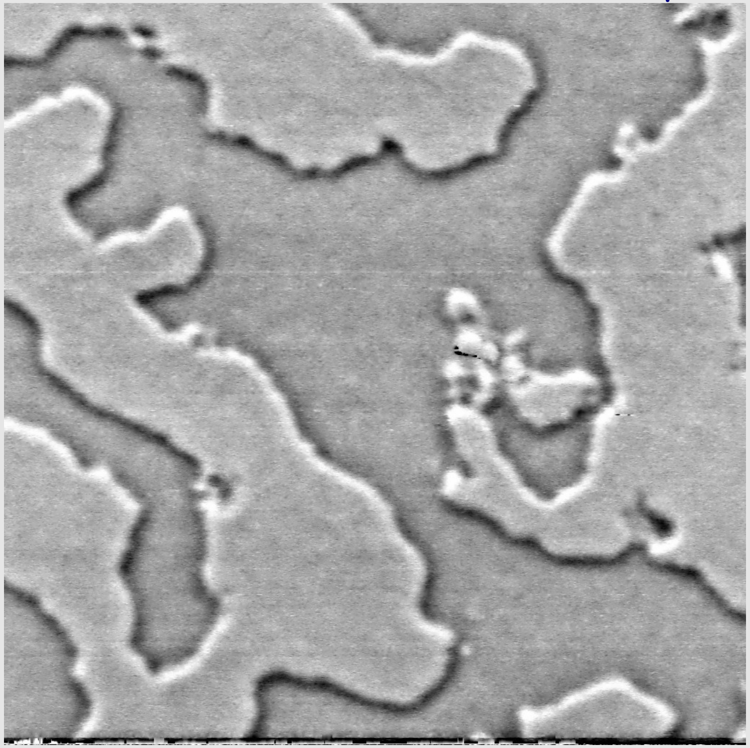
Sample : S. Pizzini
Imaging : Z. Ishaque

- Domain-wall motion under field or current
- Optimized tips for all topics

Spatial resolution 15nm

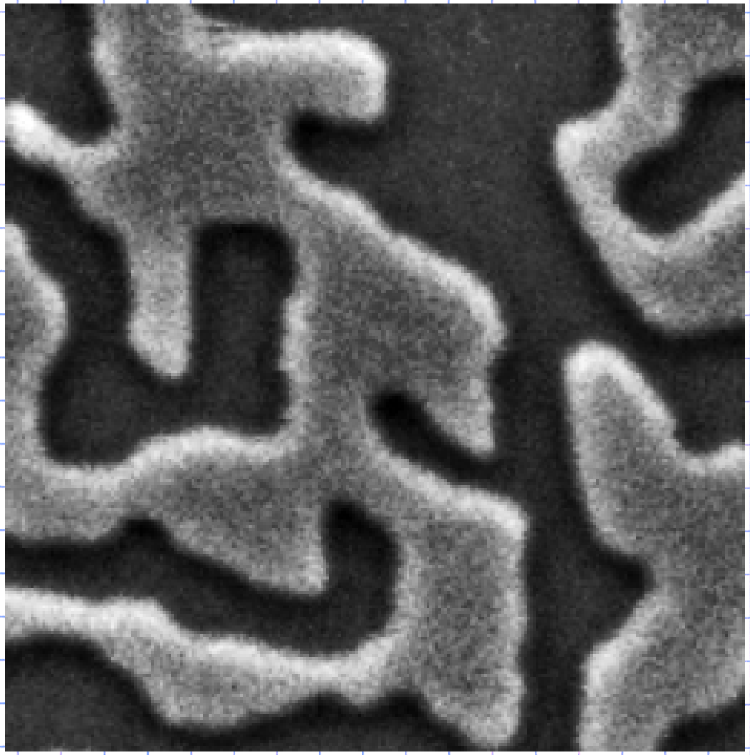
Test sample : FePt[4nm]. Perpendicular magnetization, narrow domain walls

2x2 µm



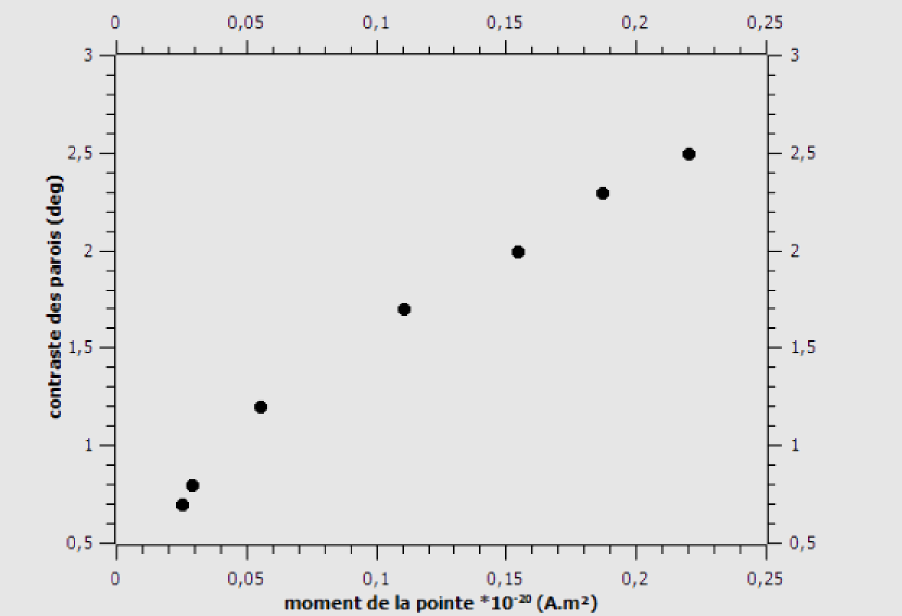
Tip : Nanosensors SSS \ 5nm CoCr
Fly height 0nm, amplitude 10nm

Commercial 'low moment' tip

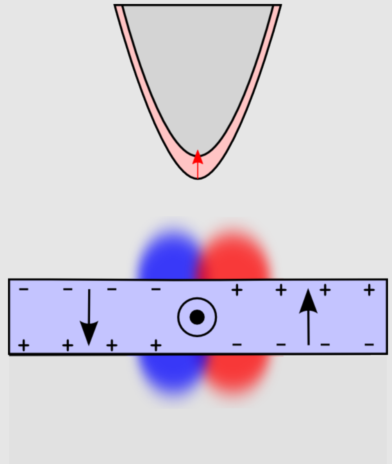


Spatial resolution : 20nm

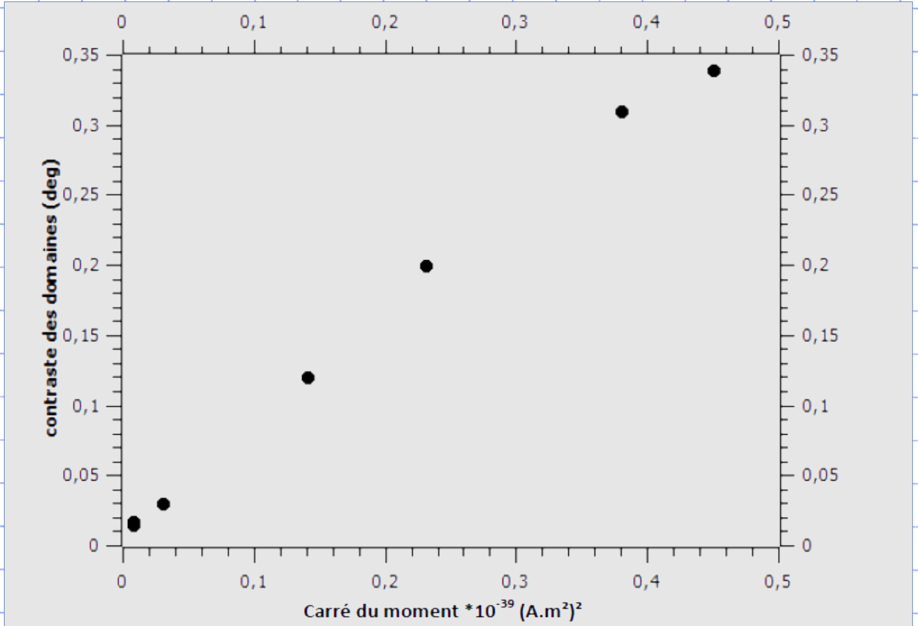
Domain wall contrast



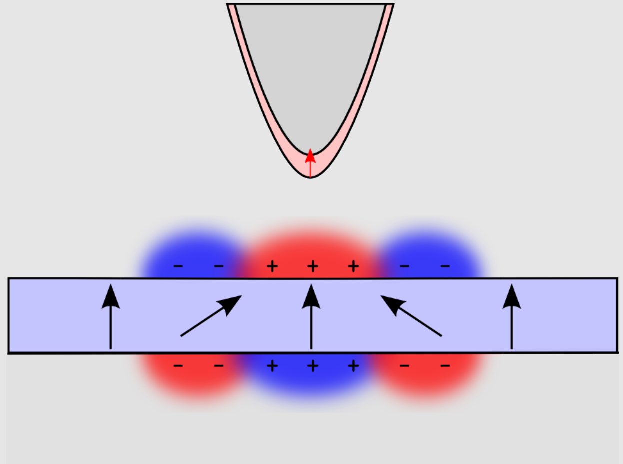
Linear with tip moment



Domain contrast

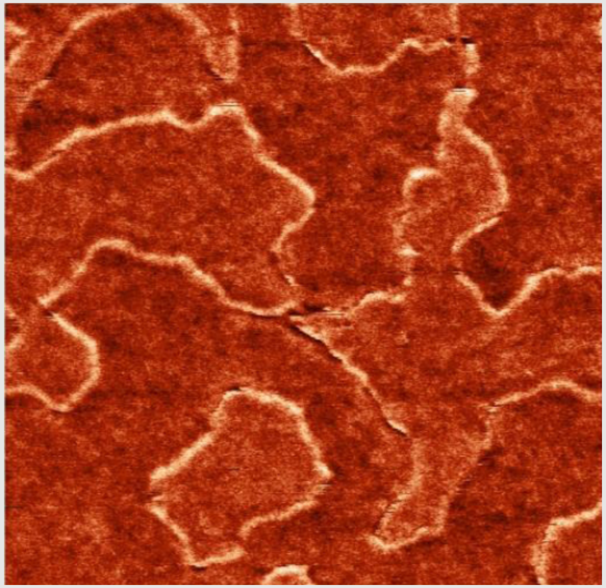


Linear with square of tip moment



Keep developing tips

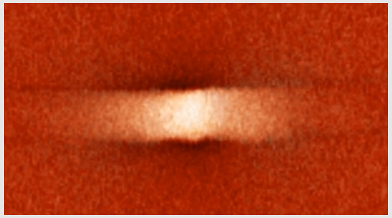
⇨ Superparamagnetic tips (reduced effect on samples : ultrasoft, skyrmions etc)

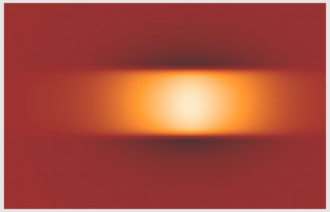


⇨ Hard magnetic tips (imaging permanent magnets)

Towards quantitative analysis

⇨ Simulations



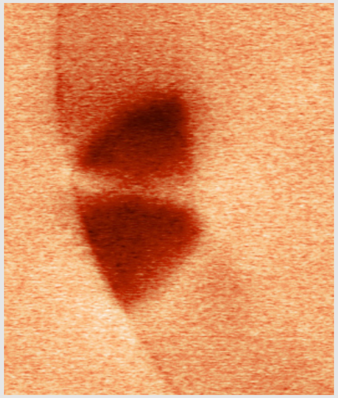


S. Jamet, JC Toussaint

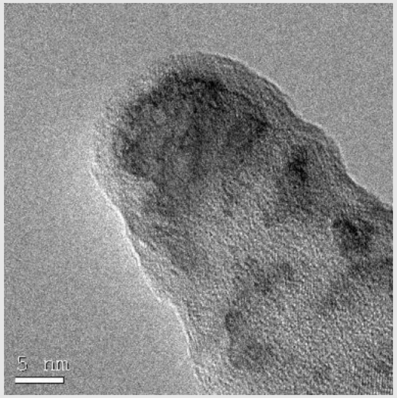
⇨ Special tips (eg filled carbon nanotube)

Pushing further sensitivity

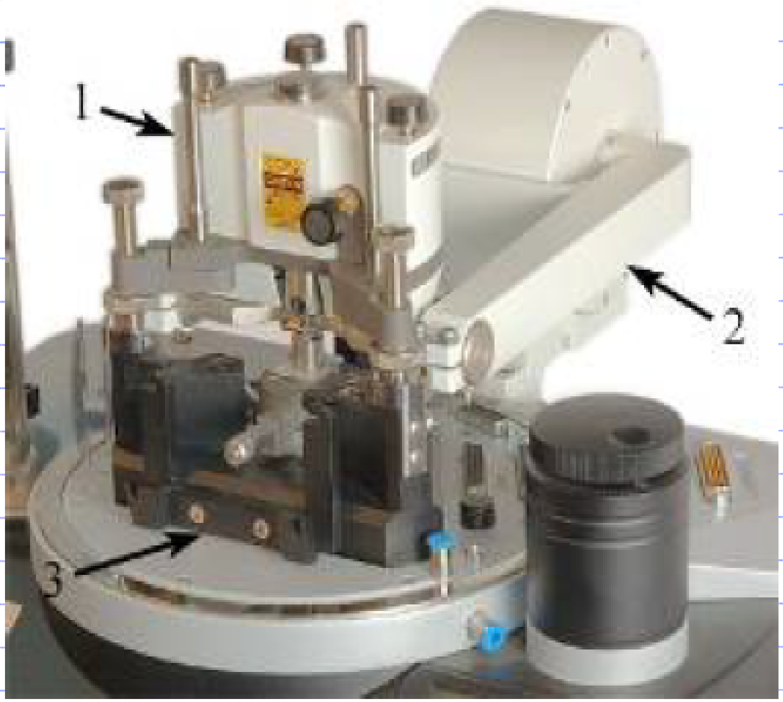
⇨ Work under vacuum → High Q



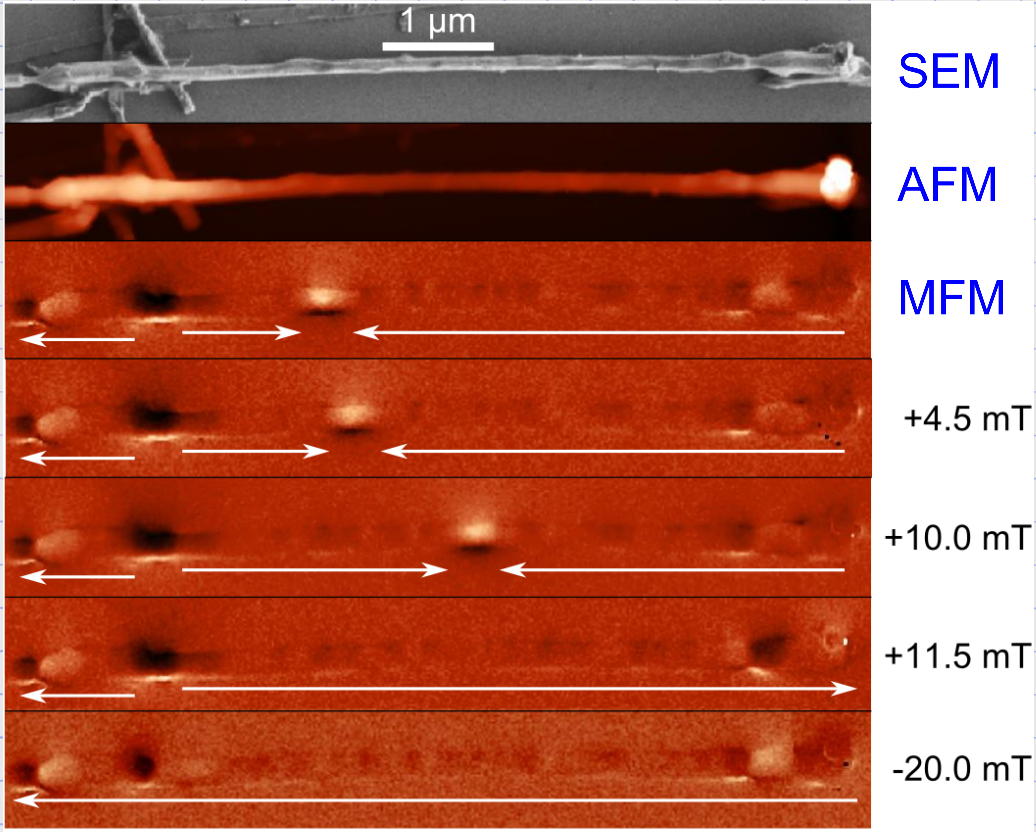
<10nm film



<5nm tip

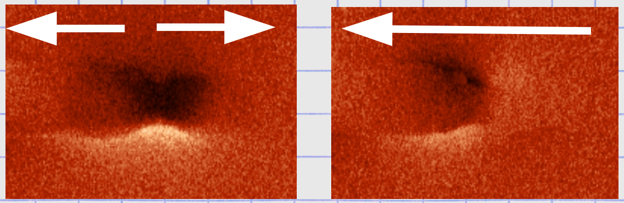


Domain wall motion



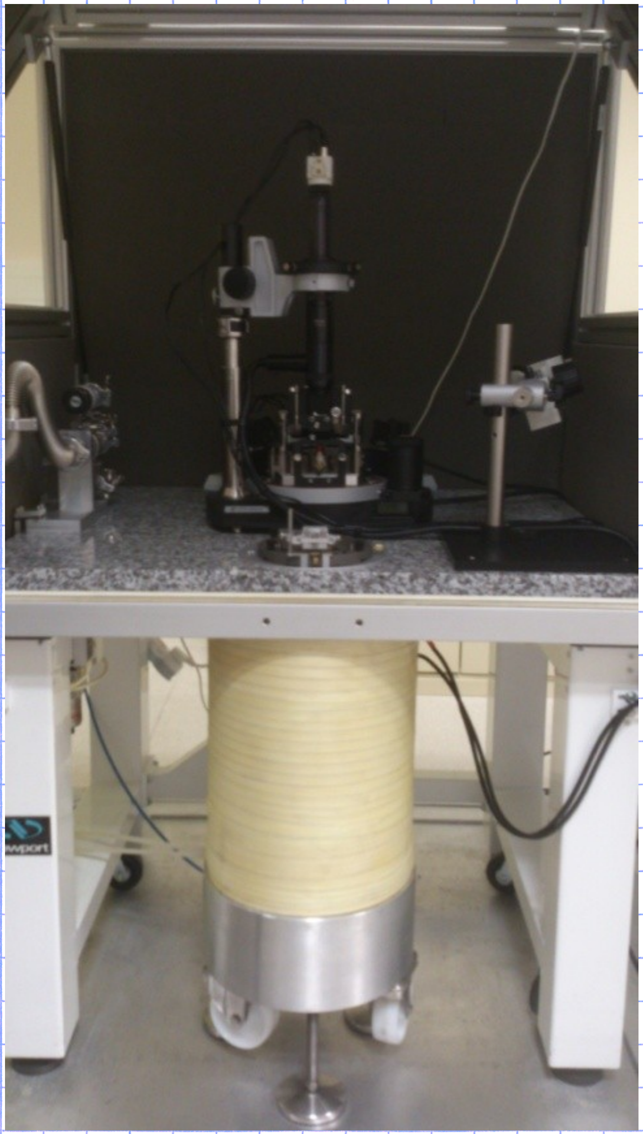
S. Da-Col et al., Appl. Phys. Lett. 109 (2016)

Protrusions and constrictions

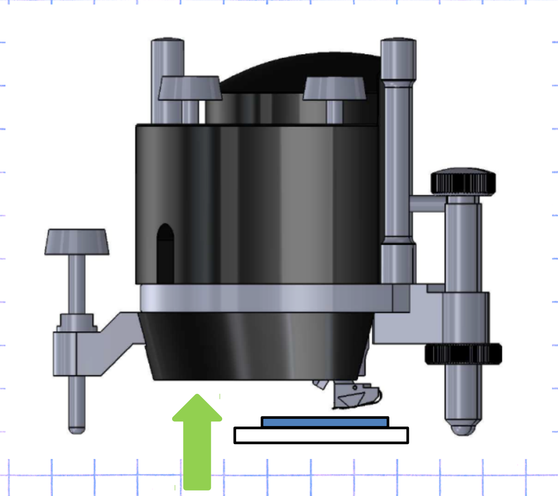


⇨ Pinning field ~30mT

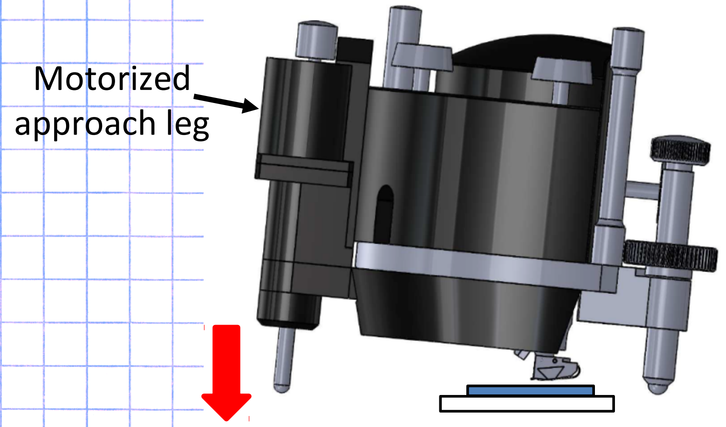
- Imaging under field
- Magnetization processes



Field >1T (custom-made)
Optimized cooling

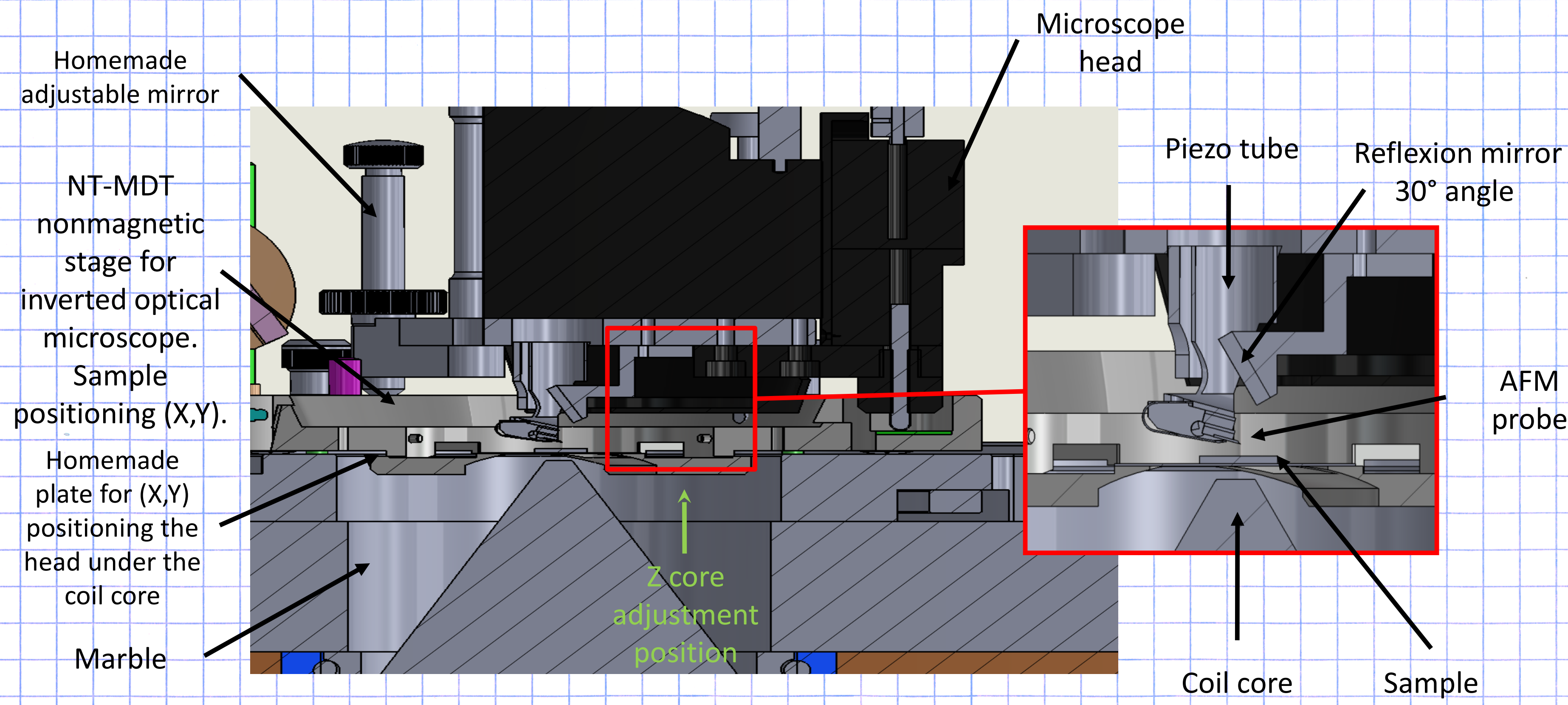


Microscope approach
by moving the sample up
(standard configuration)



Microscope approach
by tilting the head
thanks to the motorized leg
(coil coupled configuration)

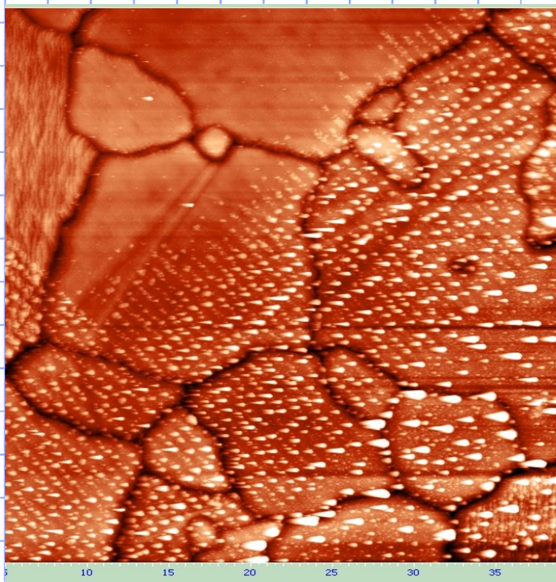
Approach by head (motorized leg)



- Combines NT-MDT and custom-made designs
- High stability thanks to decoupling head and sample from the coil

Magnetic shape-memory alloy

G. Crouigneau et al.



Topography (grains)

- Perturbation-free measurements (non-magnetic head)
- High stability (sample and coil decoupled)