

An overview on ...

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Measurements using magnetic force microscopy

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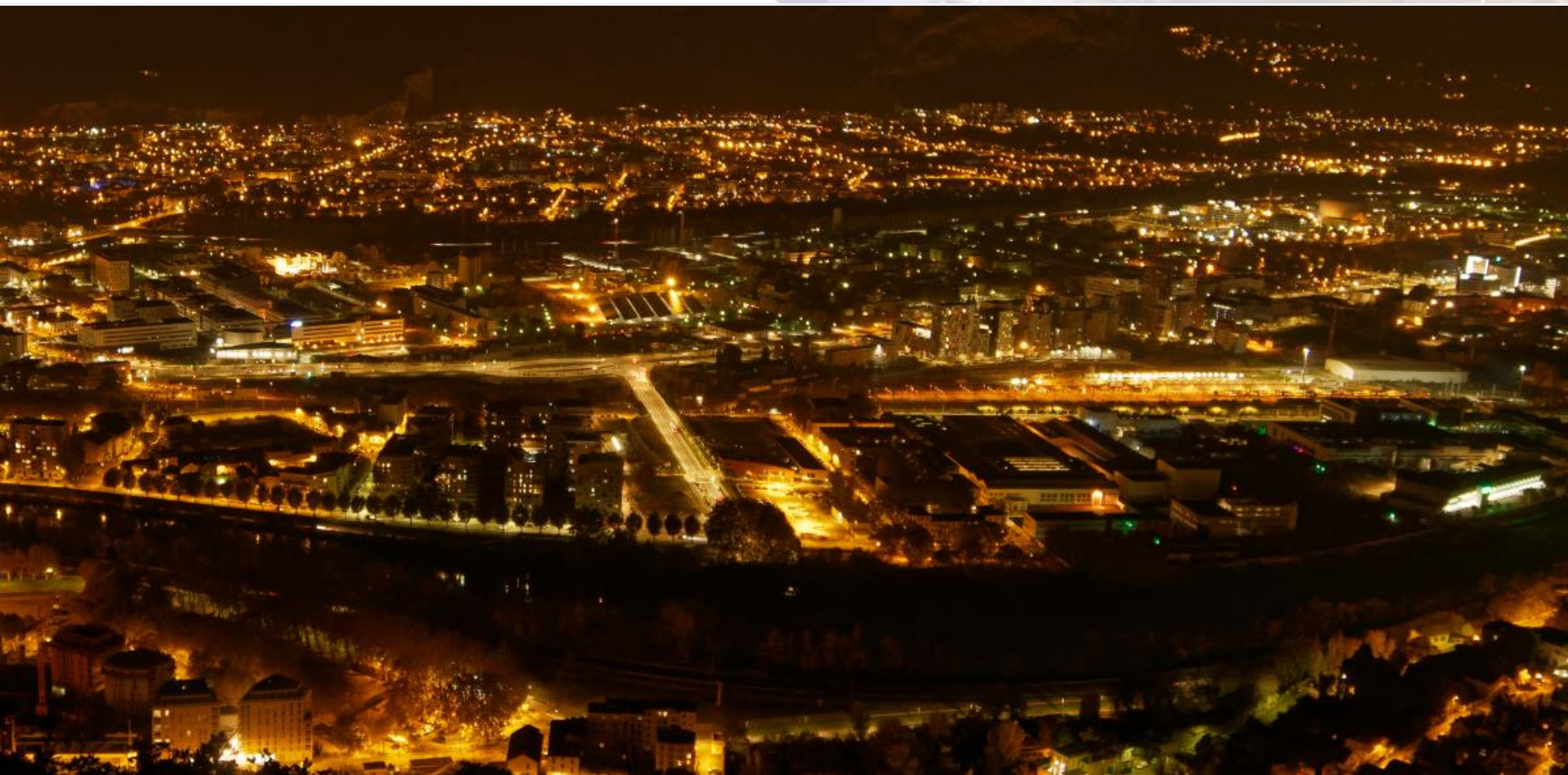
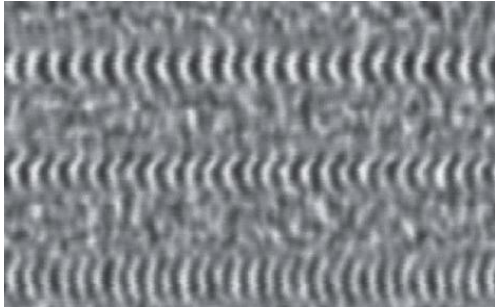


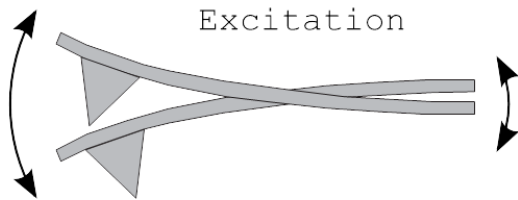


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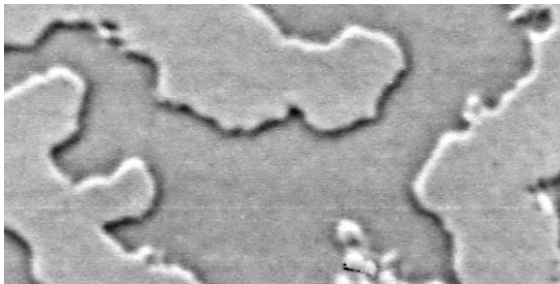
■ Motivation and criteria



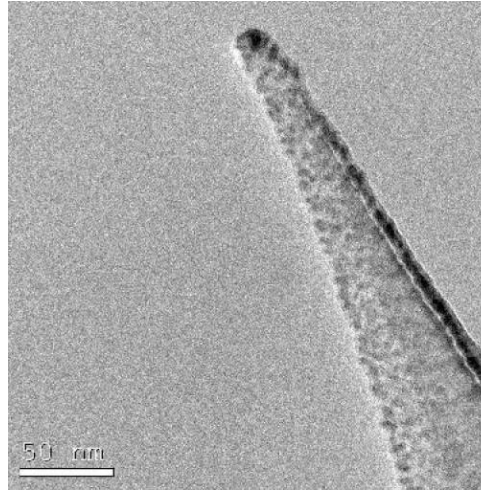
■ Working principle



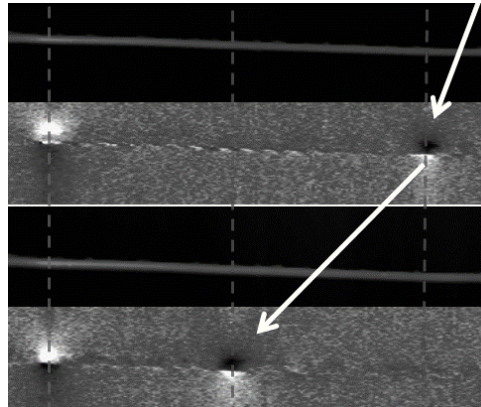
■ Image analysis



■ Choice of tips



■ Operando imaging



■ Panorama of other microscopies

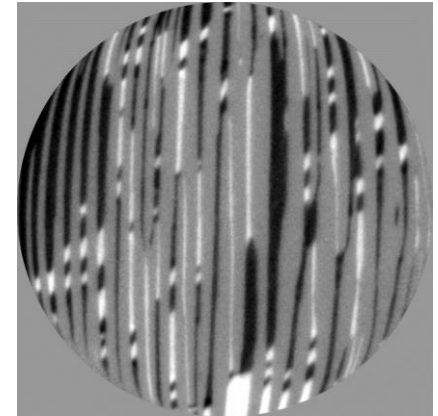
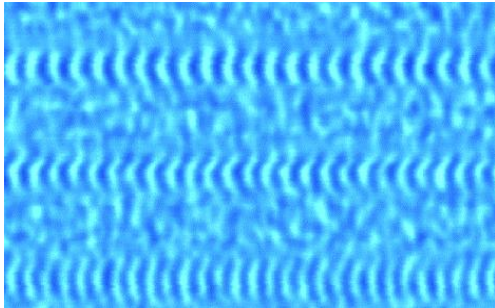




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Motivation and criteria



Working principle

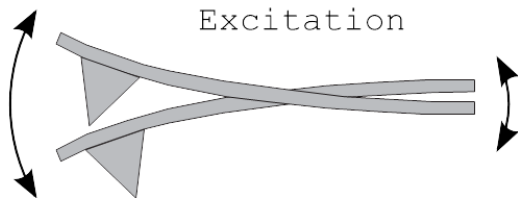
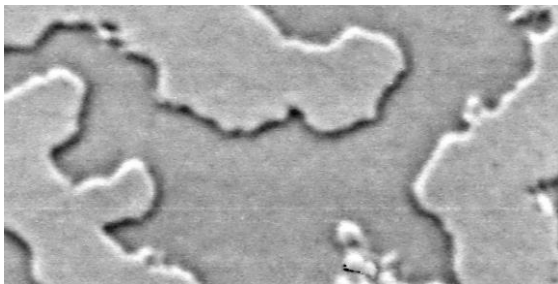
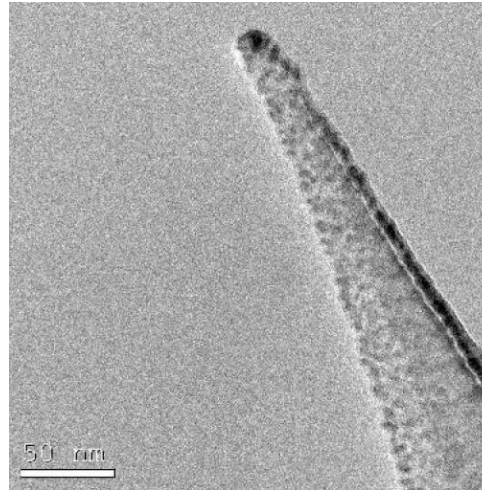


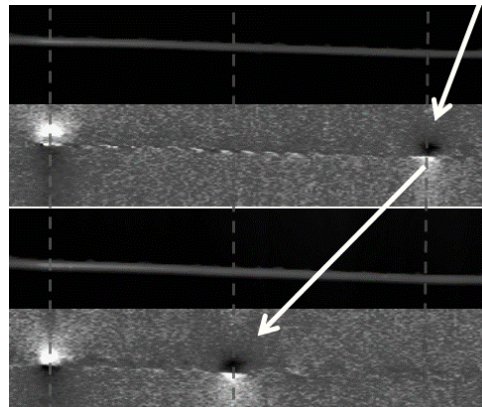
Image analysis



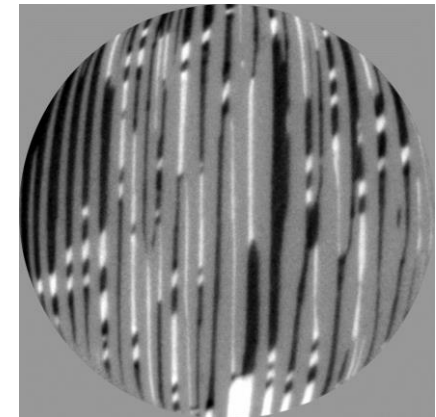
Choice of tips



Operando imaging



Panorama of other microscopies



Magnetic domains

Numerous and complex magnetic domains



(History : Weiss domains)

Anisotropy exchange length

(domain wall width)

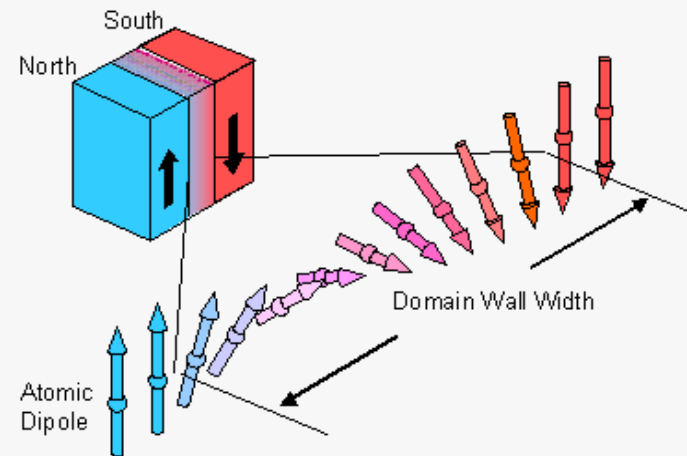
$$E = A(\partial_{x_i} m_j)^2 + K \sin^2 \theta$$

Exchange	Anisotropy
$\rightarrow J/m$	$\rightarrow J/m^3$

Anisotropy exchange length: $\Delta_u = \sqrt{A/K}$

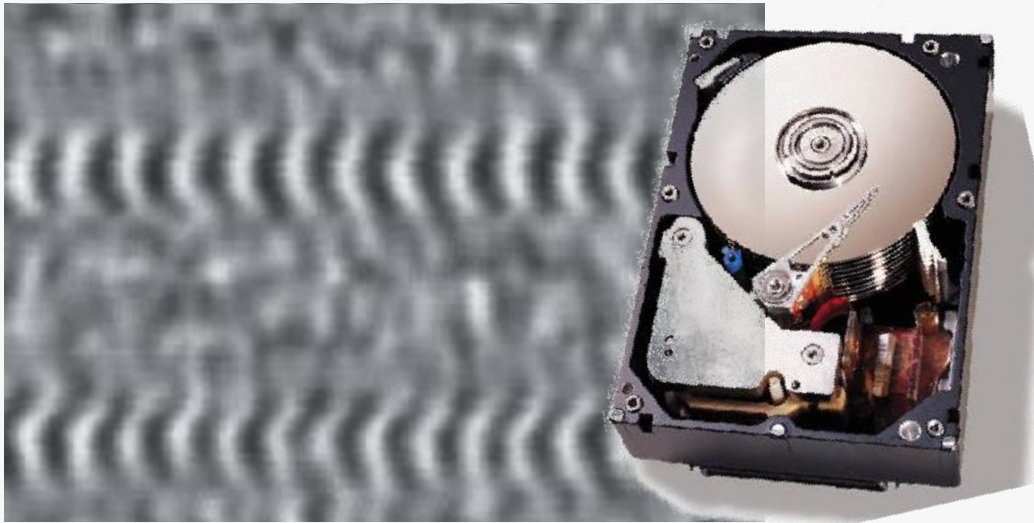
$$\Delta_u \approx 1 \text{ nm} \rightarrow \Delta_u \geq 100 \text{ nm}$$

Hard **Soft**



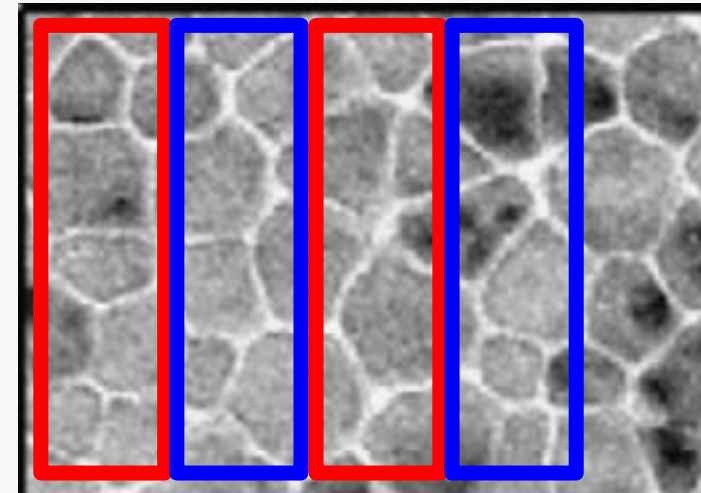
Magnetic bits on hard disk drive

Co-based hard disk media : bits 20nm in length



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

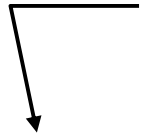
Underlying microstructure



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

Relevant spatial resolution

- 10-100nm



Basics of precessional dynamics

Magnetization dynamics:

Landau-Lifshitz-Gilbert equation:

$$\frac{d\mathbf{M}}{dt} = -\gamma_0 [\mathbf{M} \times \mathbf{H}_{\text{eff}}] + \frac{\alpha}{M_s} \left[\mathbf{M} \times \frac{d\mathbf{M}}{dt} \right]$$

Gyromagnetic factor

$$\gamma_0 = \mu_0 \gamma$$

$$\gamma = \frac{g\mu_B}{\hbar}$$

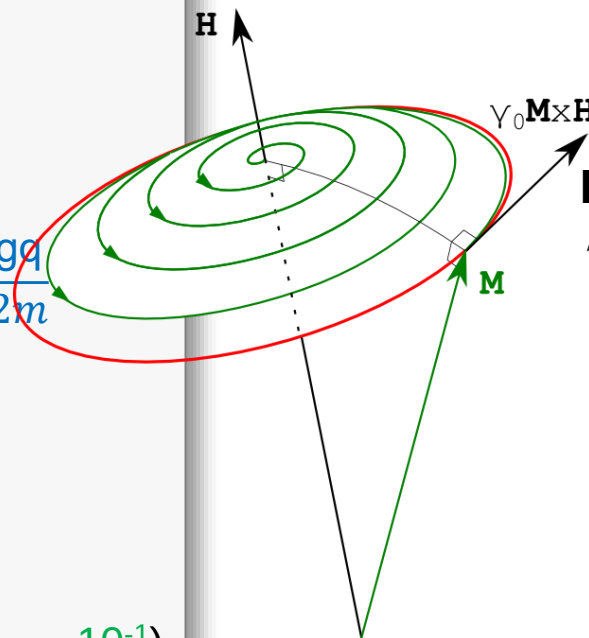
$$\gamma / 2\pi = 28 \text{ GHz/T}$$

$\mathbf{H}_{\text{eff}}$ Effective field
(including applied)

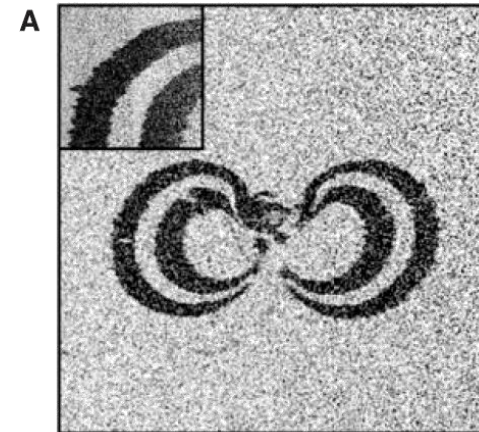
α Damping coefficient ($10^{-3} \rightarrow 10^{-1}$)

Relevant time resolution

- $> 1\mu\text{s}$: thermally-activated magnetization processes
- 1 ns : precession of magnetization
- 1 ps : ultrafast demagnetization



Demonstration: 1999

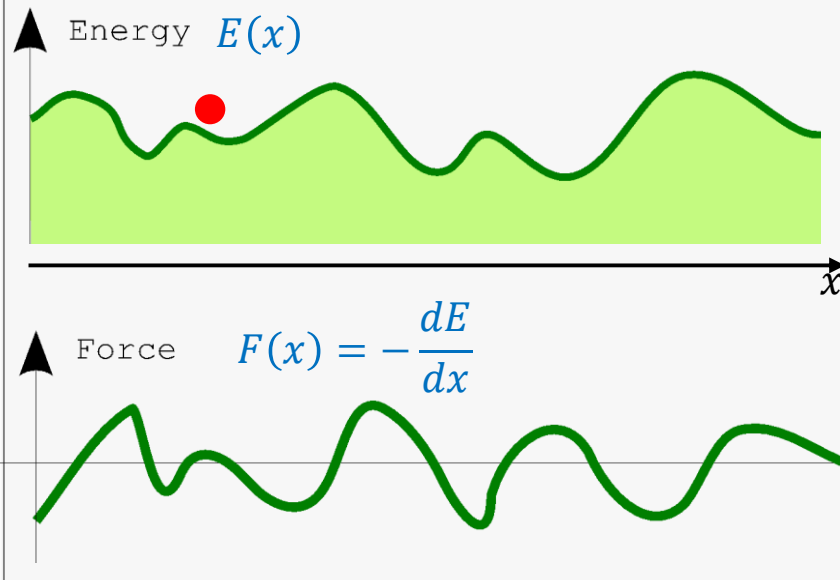


150 μm

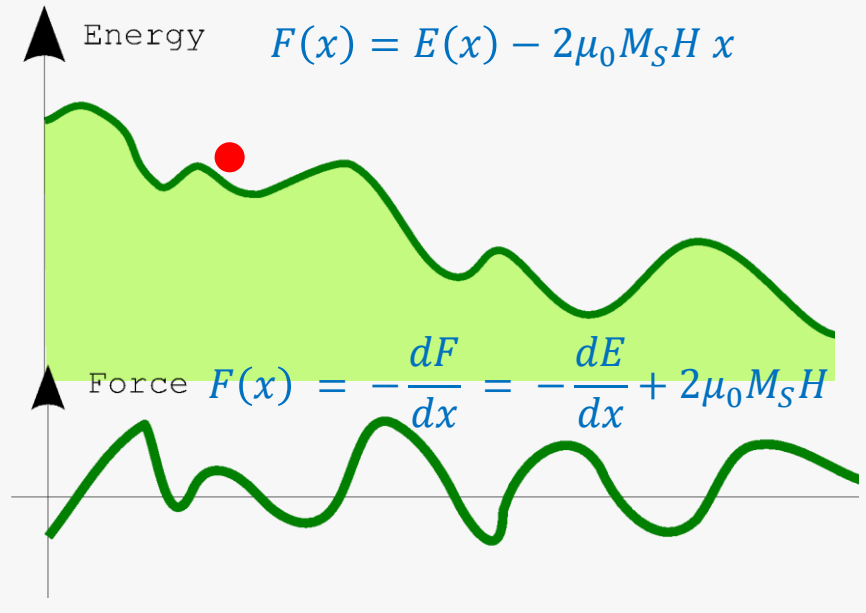
C. Back et al., Science 285, 864 (1999)

Example : domain wall to be moved along a 1d system

Without applied field



With applied field



E. Kondorski, On the nature of coercive force and irreversible changes in magnetisation, Phys. Z. Sowjetunion 11, 597 (1937)

Relevant information

- ❑ Microstructure
- ❑ Chemical composition
- ❑ Crystal structure

Spectroscopy,
diffraction etc.

Versatility

- ❑ Samples made with lithography or ex situ OK ?
- ❑ Need for sample preparation ?
- ❑ Compatible with various environments ? (temperature, field etc.)

Speed of acquisition

- ❑ Sample preparation needed ?
- ❑ How much time for one image ?

Access

- ❑ Large-scale instrument or in-lab ?
- ❑ Expensive or cheap ?

Measured quantity

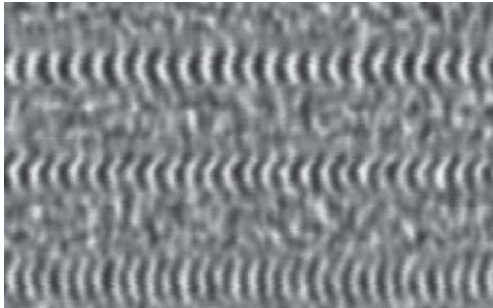
- ❑ Surface or volume technique ?
- ❑ Sensitivity ?
- ❑ Magnetization, stray field, other ?

- ❑ No universal technique
- ❑ Many criteria to be balanced

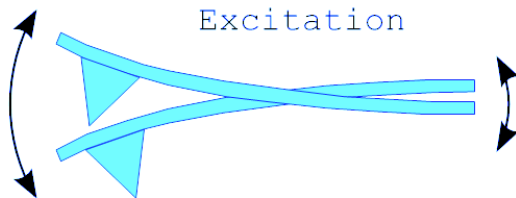


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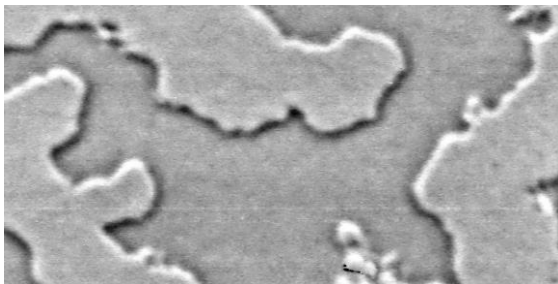
■ Motivation and criteria



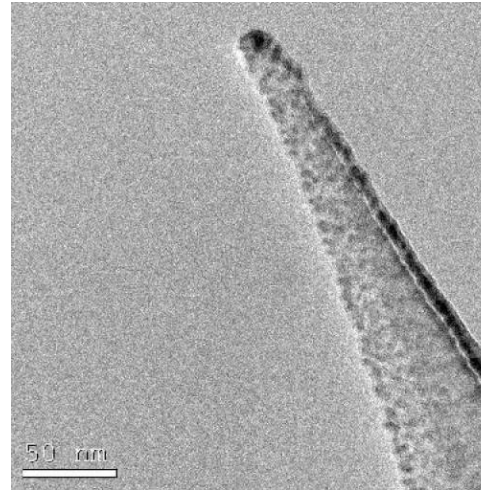
■ Working principle



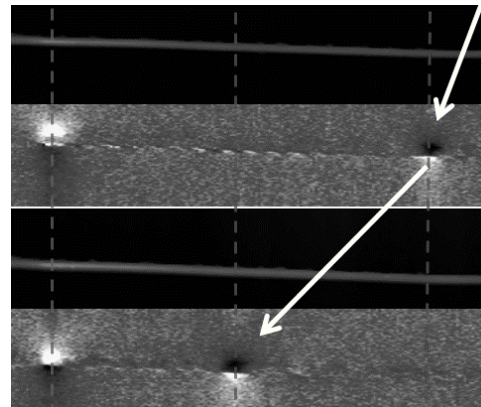
■ Image analysis



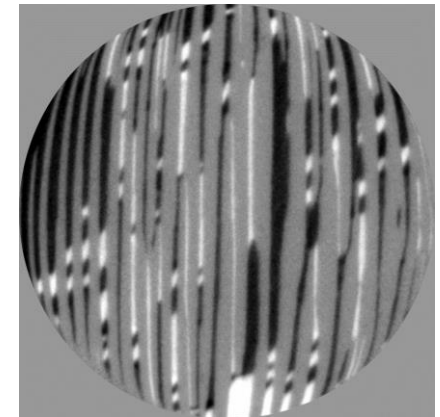
■ Choice of tips



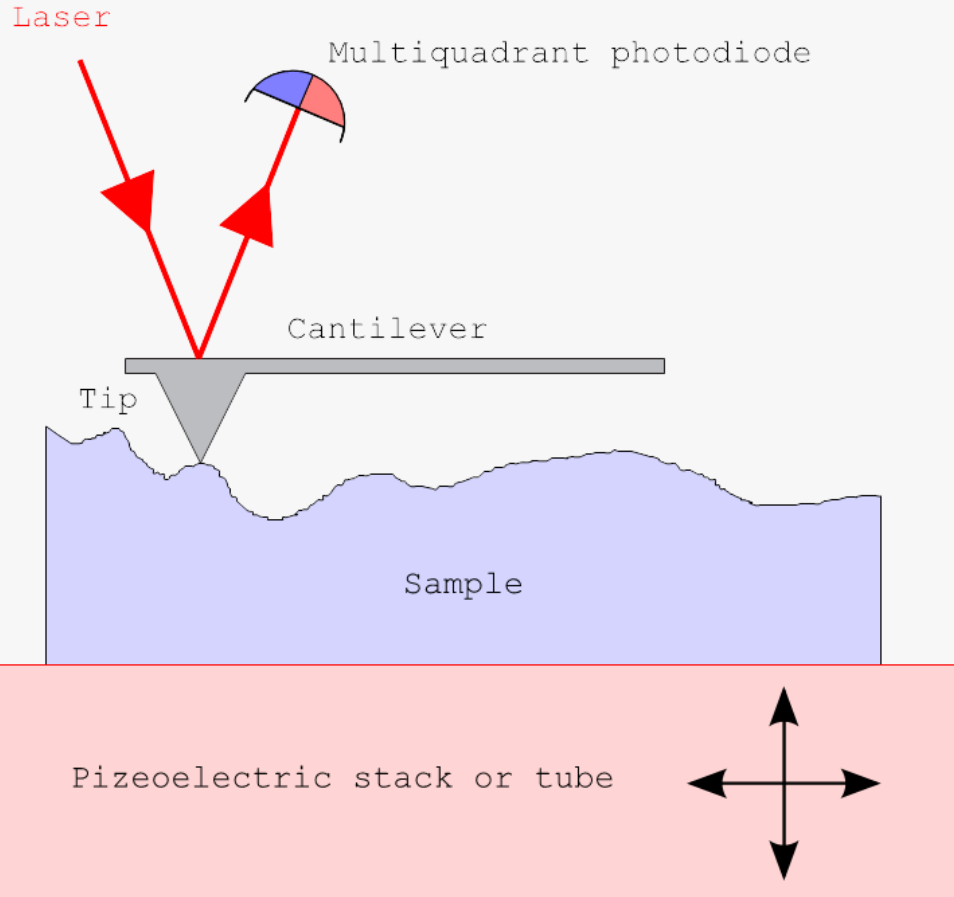
■ Operando imaging



■ Panorama of other microscopies



Key elements of an Atomic Force Microscope (AFM)

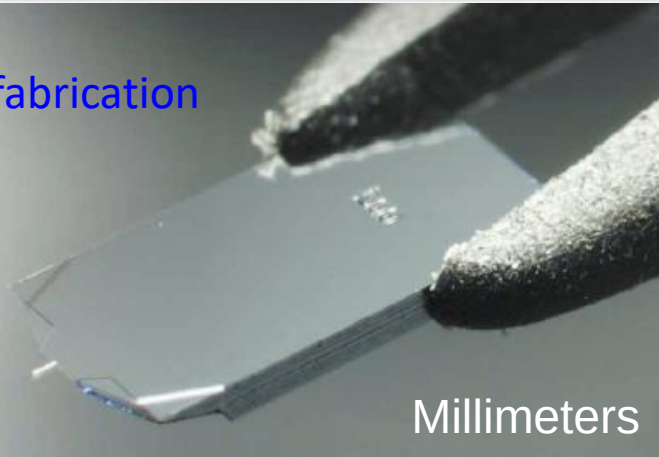


G. Binnig et al., Phys. Rev. Lett. 56, 930-933 (1986)

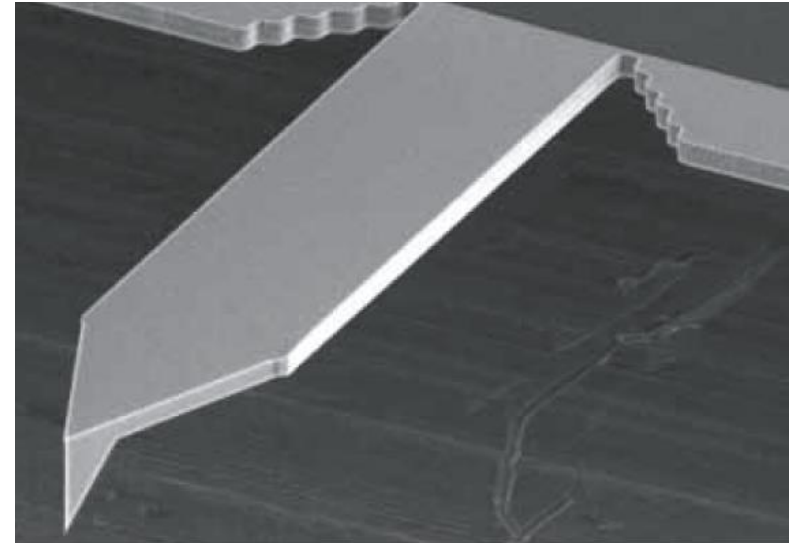
Overview

- Measures forces (vertical and lateral) between sample and tip

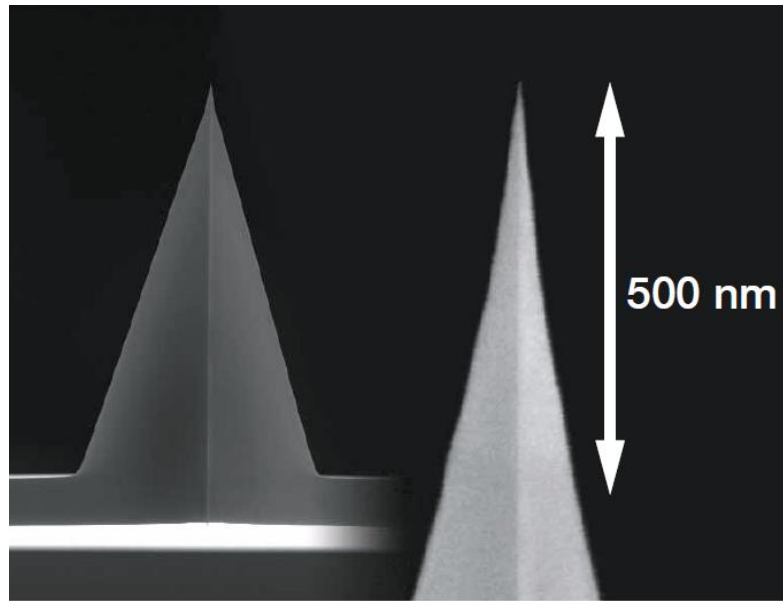
Chip
Batch fabrication



Cantilever



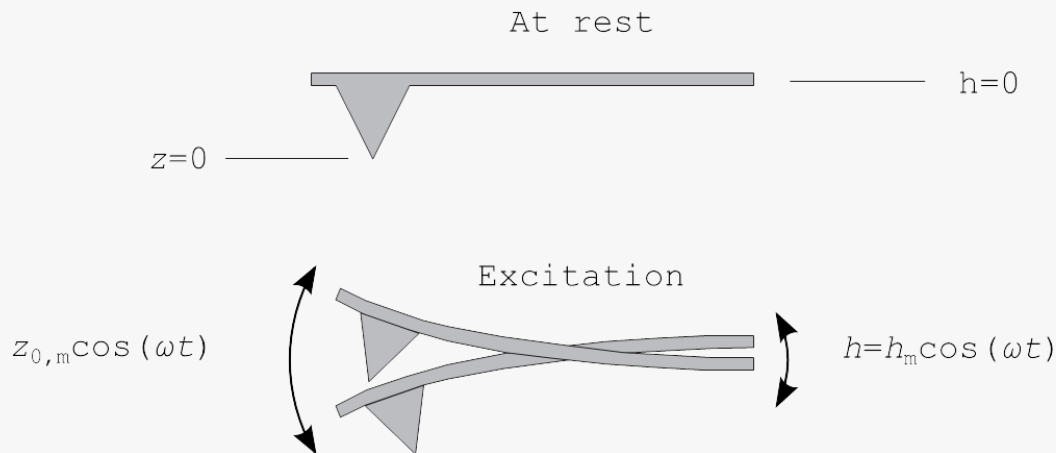
Full tip + apex



- Price 10-200€ / tip
- Radius of curvature $\approx 5\text{nm}$

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

Mechanical excitation of cantilevers



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

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

Notations

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

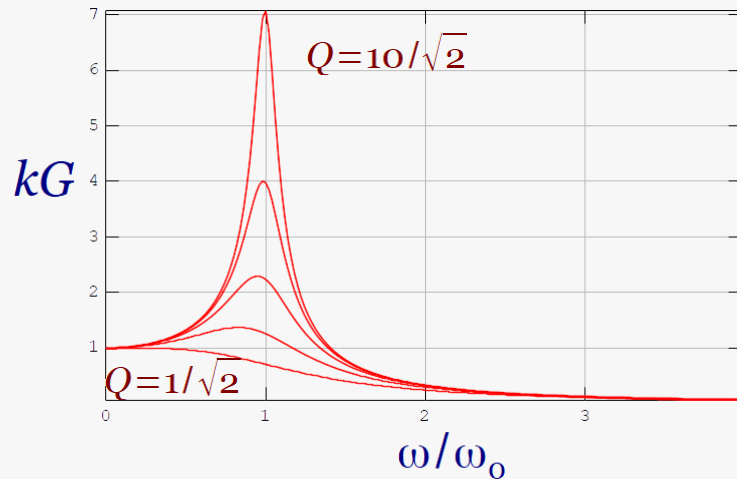
Reference angular velocity $\omega_0 = \sqrt{\frac{k}{m}}$

Quality factor $Q = \frac{\sqrt{k m}}{\Gamma}$

➔ Transfer 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

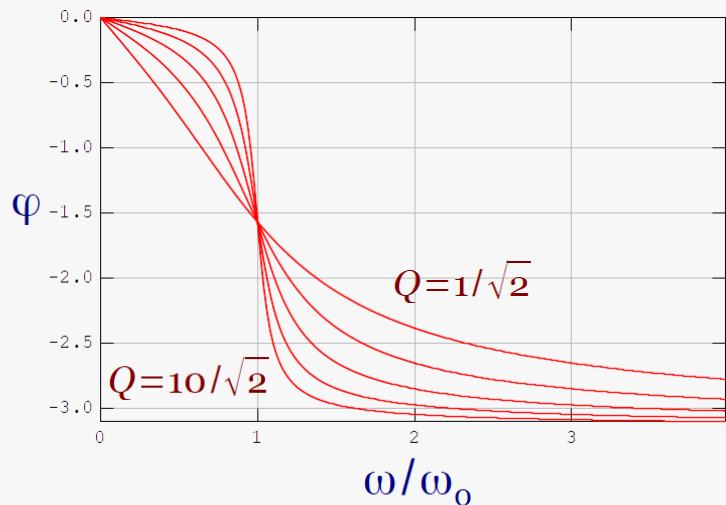


$$G = |H| = \frac{1}{k} \frac{1}{\sqrt{\left[1 - \left(\frac{\omega}{\omega_0}\right)^2\right]^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}}$$

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

$$\begin{cases} kG(0) = 1 \\ kG(\omega_r) = Q \\ kG(\infty) = 0 \end{cases}$$

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

$$\omega_r \approx \omega_0$$

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

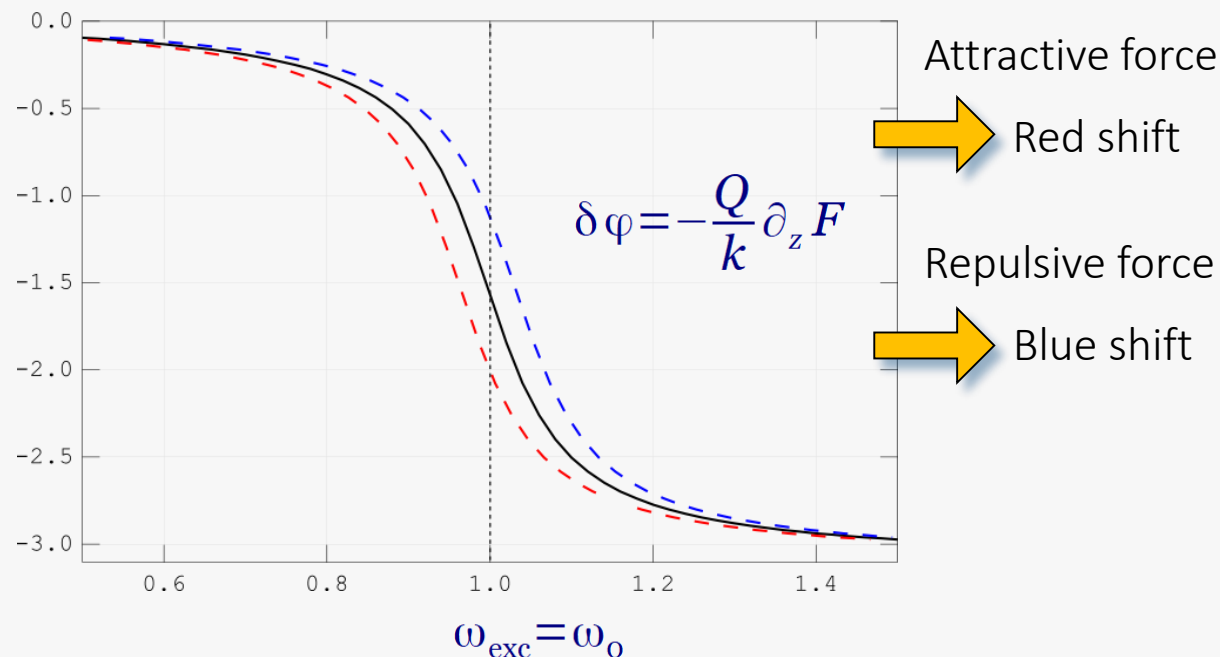
$$\frac{\Delta \omega_r}{\omega_0} \approx \frac{\sqrt{3}}{Q}$$

Tip-sample interaction treated as perturbation

$$m \ddot{z} + \Gamma \dot{z} + k z = F(z) \quad \text{with} \quad F(z) = F(z_0) + (z - z_0) \partial_z F$$

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

Phase shift



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

MFM tips: AFM tip + magnetic coating

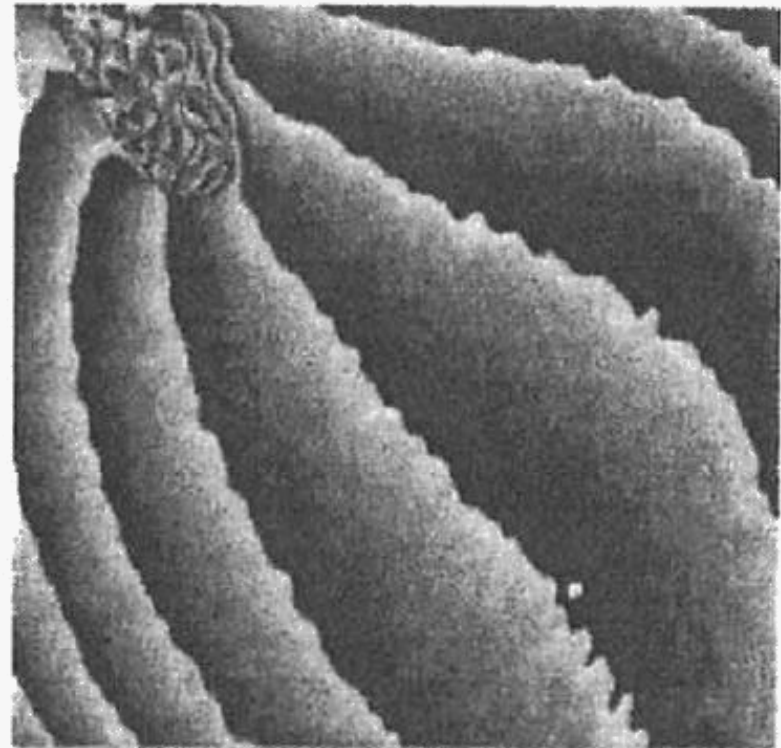
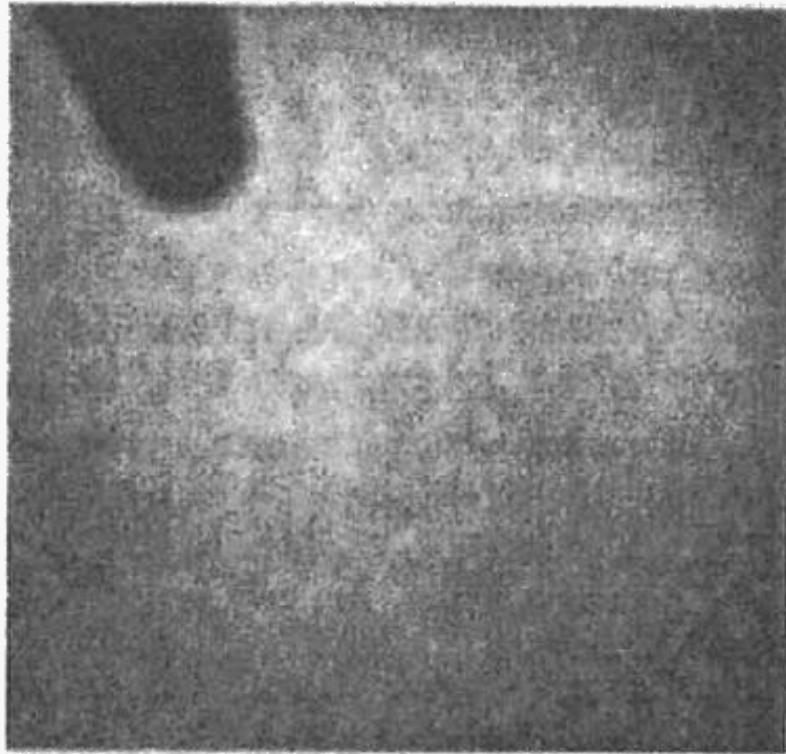
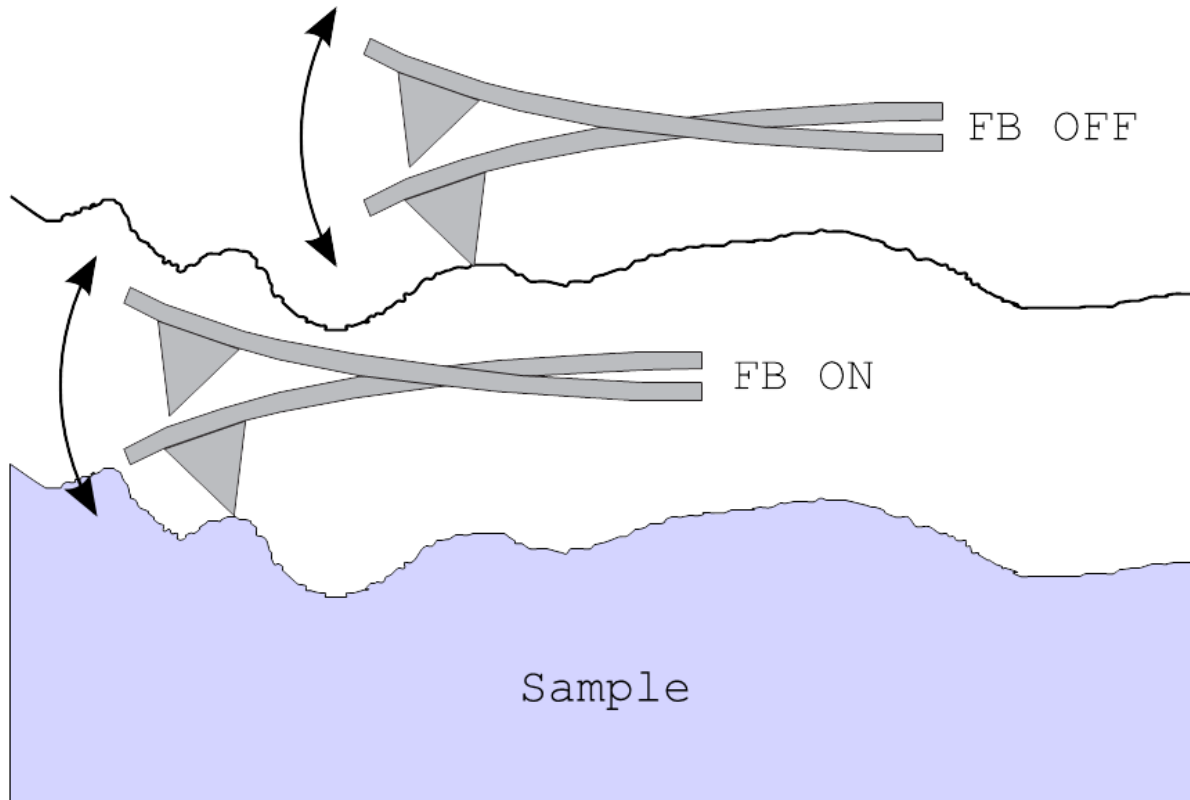


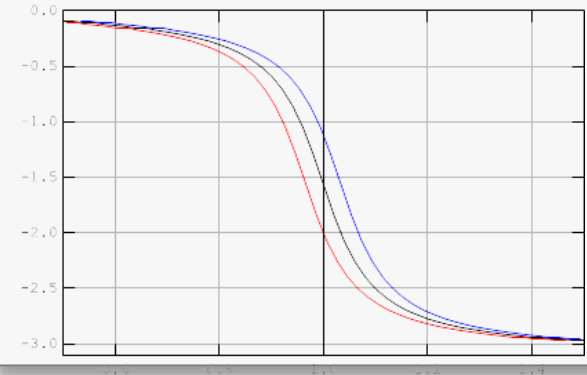
Figure 11-20: The electron amplitude (left) and phase (right) near an MFM tip visible as a dark shadow on the upper left corner of the left image.

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



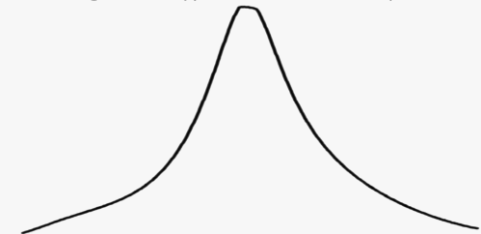
Second pass

- Monitor long-range forces (magnetic, electrostatic)



First pass

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



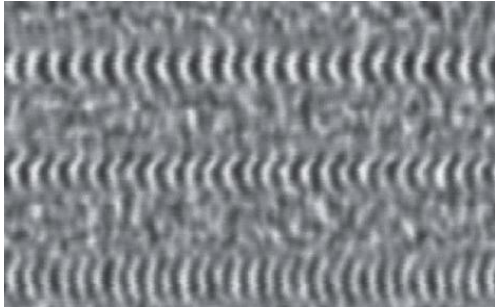
First report : Y. Martin et al., Appl. Phys. Lett. 50, 1455 (1987)

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

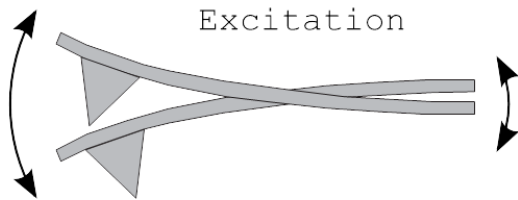


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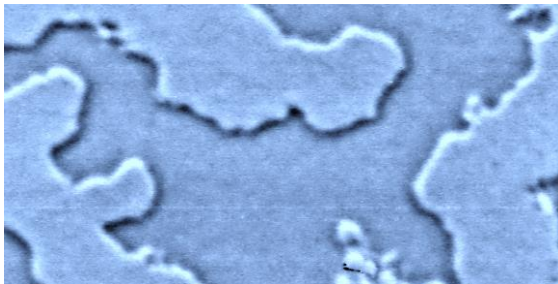
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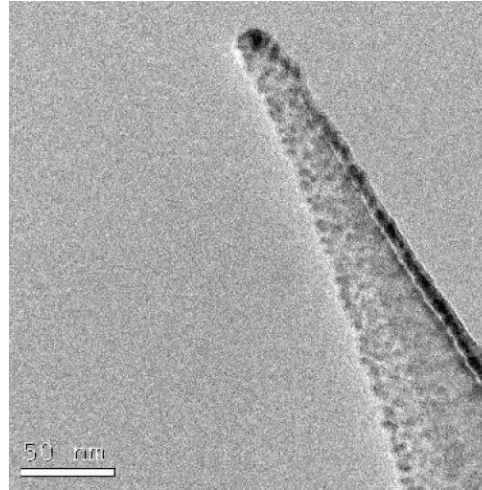
■ Working principle



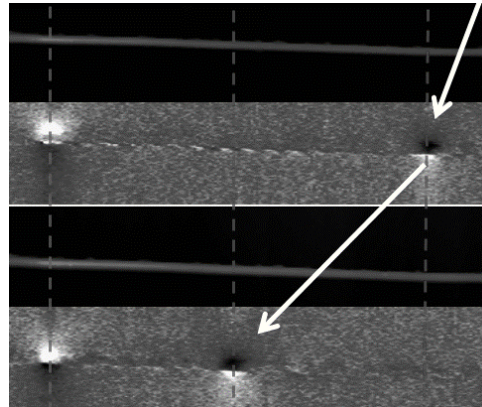
■ Image analysis



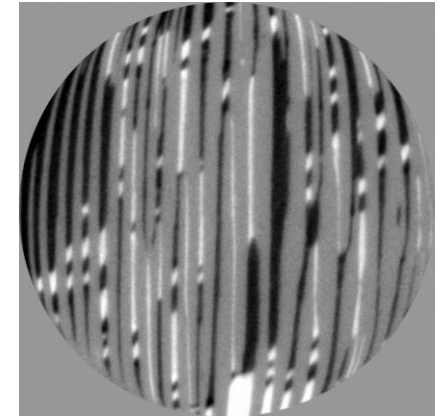
■ Choice of tips



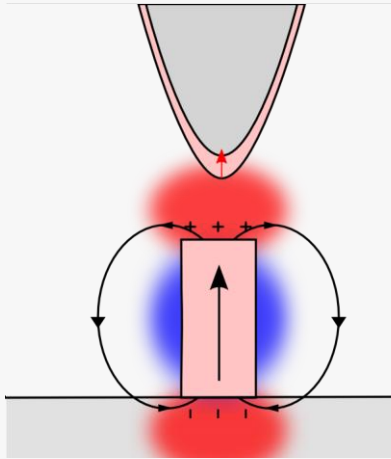
■ Operando imaging



■ Panorama of other microscopies



Tip is a dipole

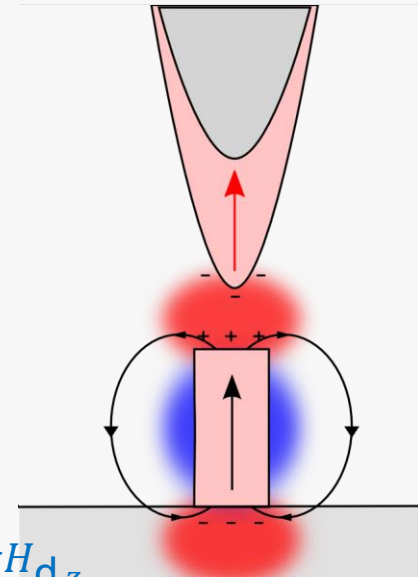


$$E_{1,2} = -\mu_0 \mu_2 \cdot H_d$$

$$E_{1,2} = -\mu_0 (\mu_x \cdot H_{d,x} + \mu_y \cdot H_{d,y} + \mu_z \cdot H_{d,z})$$

➔ $\delta\phi = \frac{Q}{k} \mu_0 \mu_i \partial_z^2 H_{d,i}$

Tip is a monopole



$$E_{1,2} = \mu_0 \sigma \cdot \phi$$

$$F_z = -\mu_0 \sigma H_{d,z}$$

➔ $\delta\phi = \frac{Q}{k} \mu_0 \sigma \partial_z H_{d,z}$

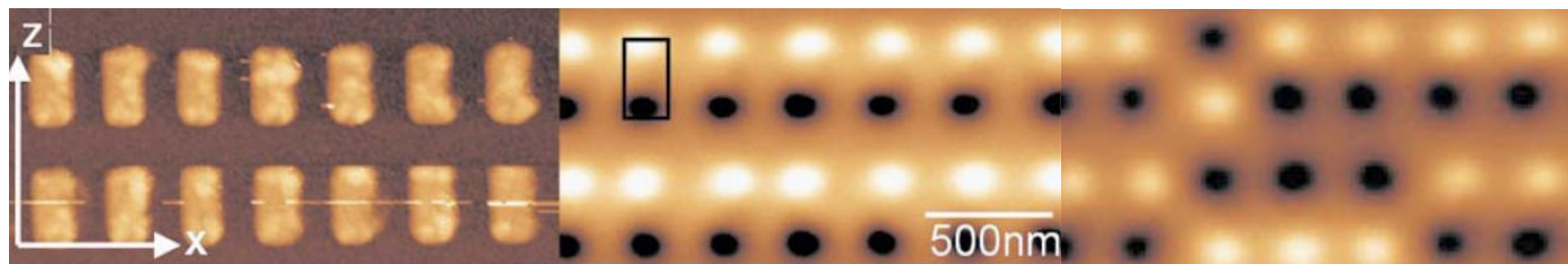
- ❑ In practice, a combination of both models is best suited (dipole is more important)
- ❑ MFM is sensitive to some derivative(s) of the stray field from the sample
- ❑ MFM may be sensitive to in-plane field, depending on the tip magnetic moment

PRACTICE / Single-domain (planar)

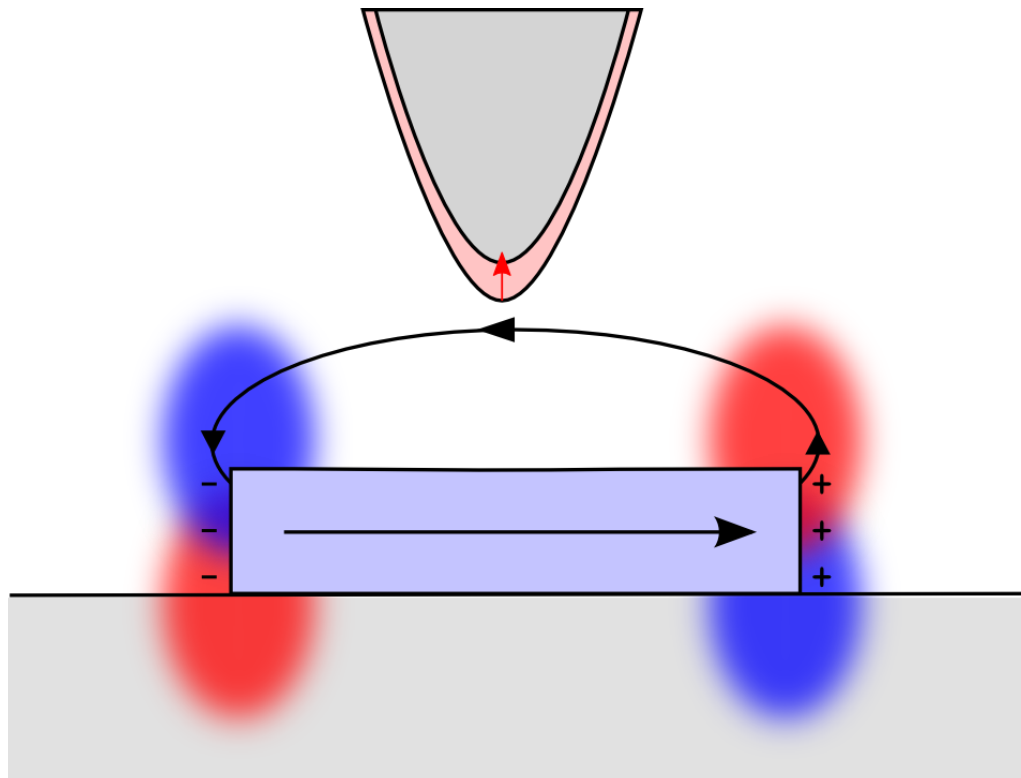
Topography

MFM, saturated

MFM, partly reversed



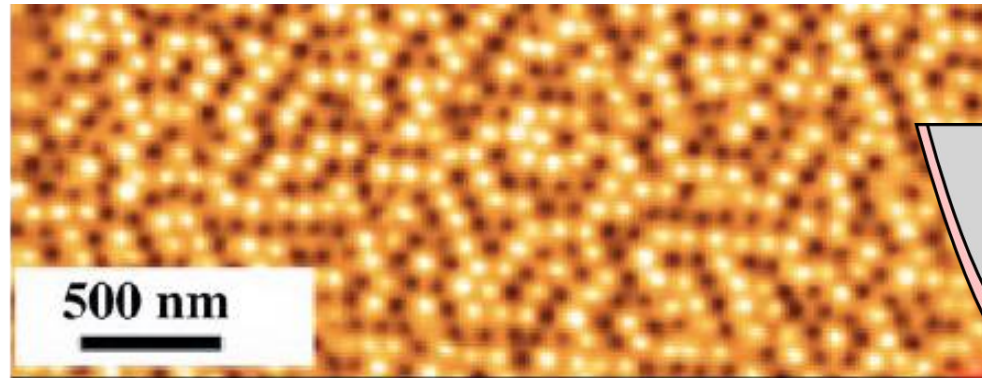
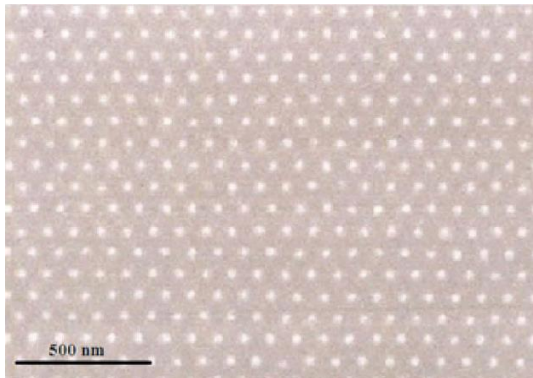
S. Y. Suck et al., APL95, 162503 (2009)



- Single-domain in-plane magnetized dots appear as dipoles

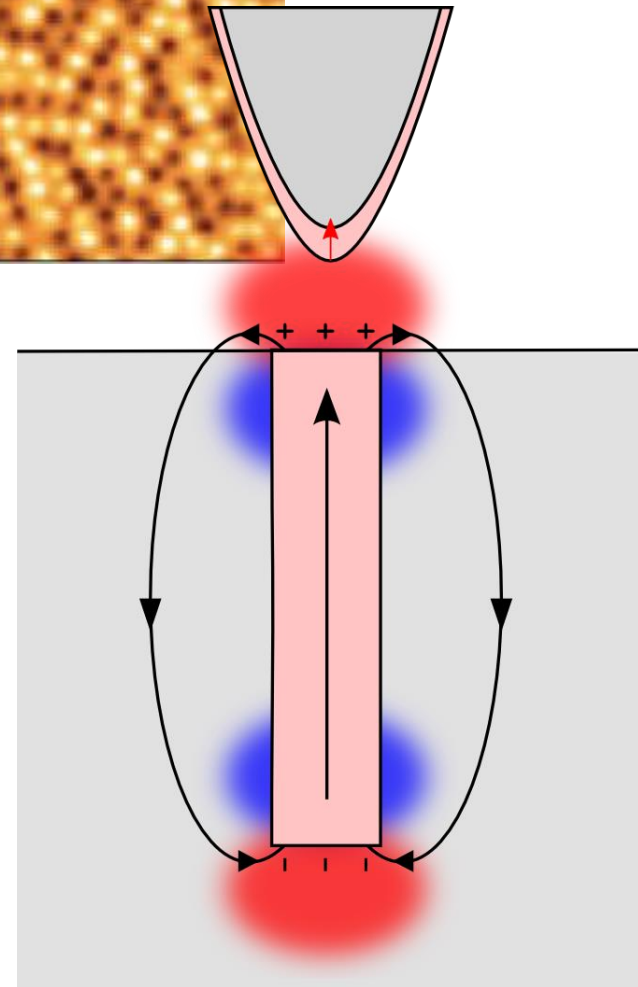
Structure (SEM)

MFM, partly reversed

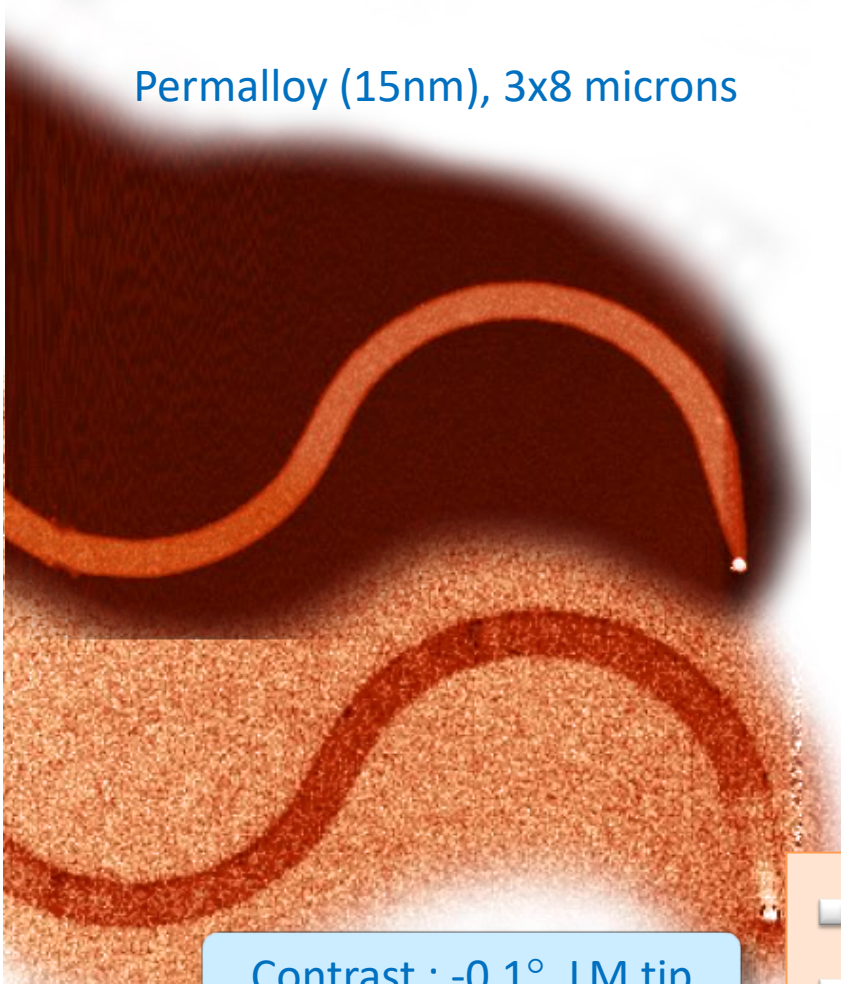


T. Wang et al., APL 92, 192504 (2008)

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

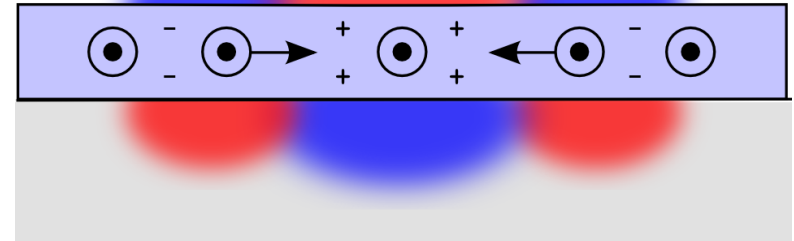
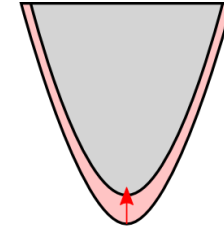


Permalloy (15nm), 3x8 microns



Contrast : -0.1° , LM tip

Lithography : S. Pizzini (Institut Néel)
Imaging : Z. Ishaque (Institut Néel)



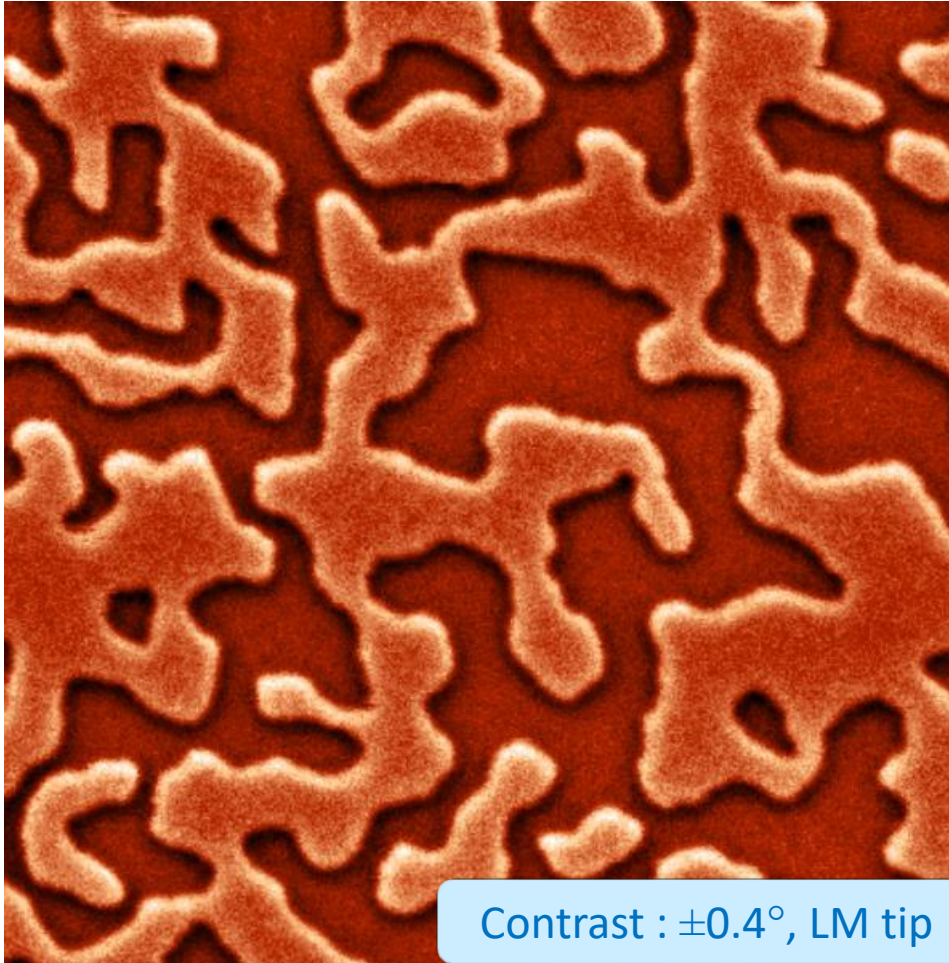
Principle :

1. Stray field magnetizes sample
2. Sample is non-uniform $\rightarrow$ 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

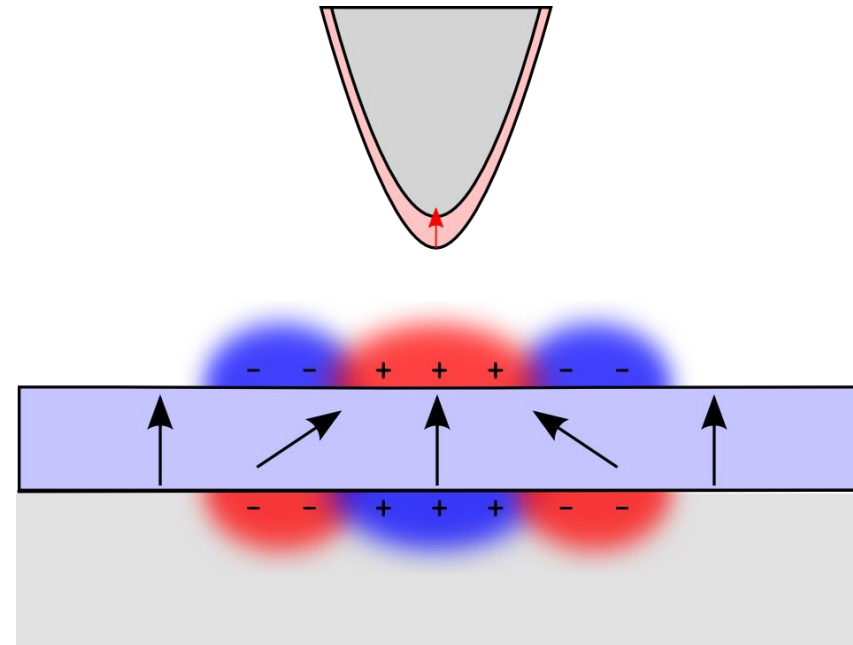
FePt (4nm)

5x5 μm



Contrast : $\pm 0.4^\circ$, LM tip

Sample : A. Marty (CEA-Grenoble)
Imaging : M. Darques (Institut Néel)

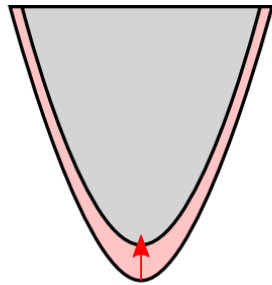
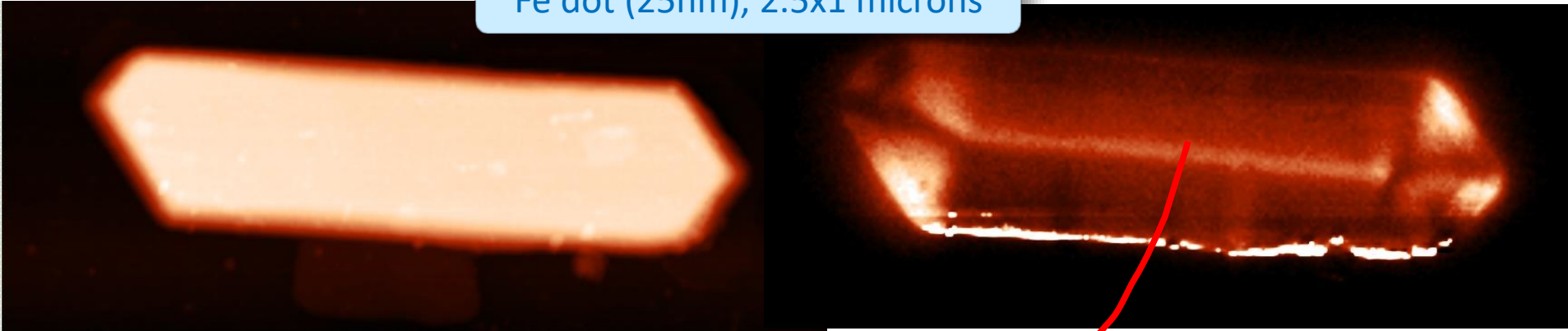


- It is a DOMAIN contrast
- The direction of magnetization is deduced

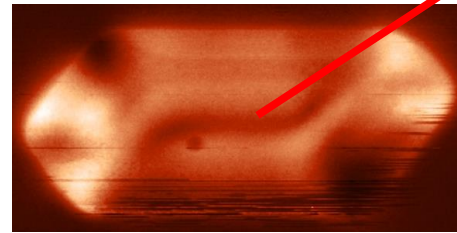
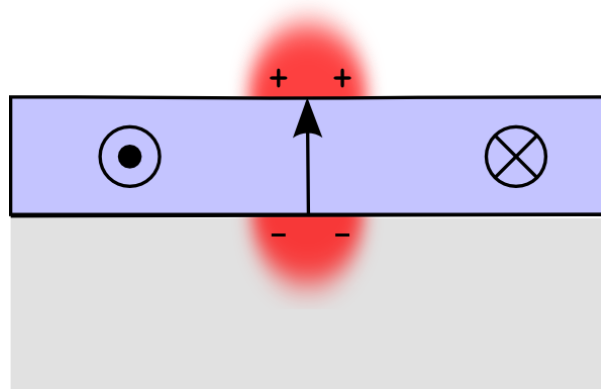
Quantitative analysis:

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

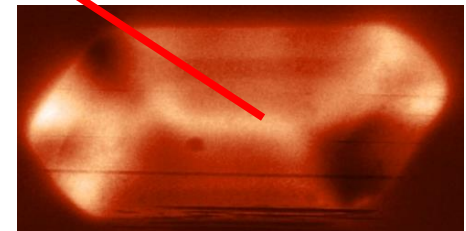
Fe dot (25nm), 2.5x1 microns



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

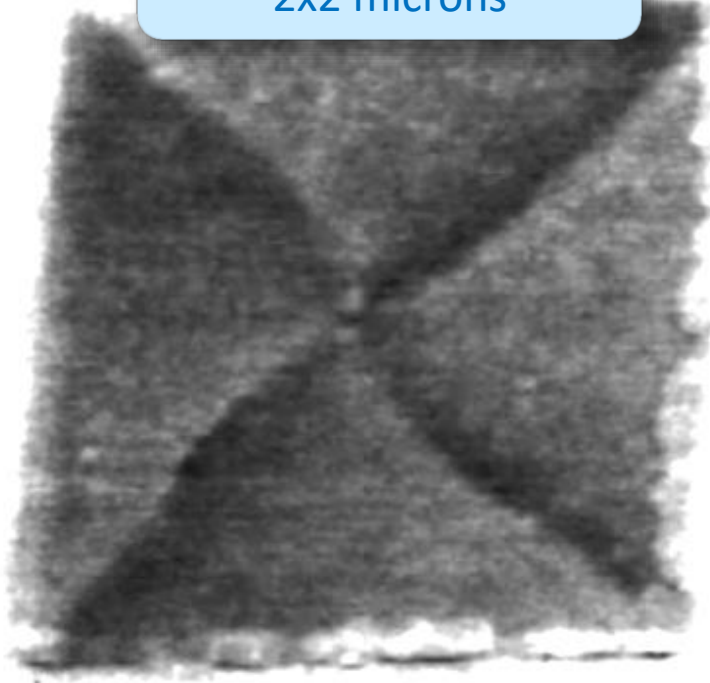


Down core

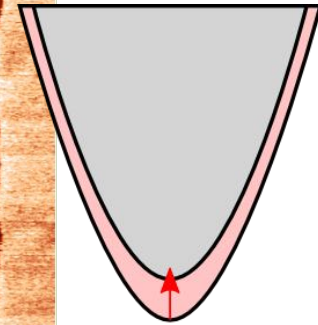
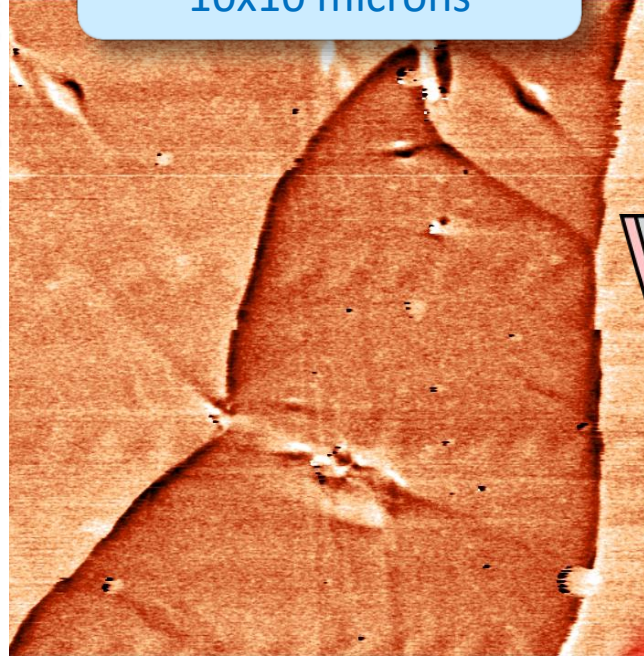


Up core

Permalloy dot (16nm)
2x2 microns

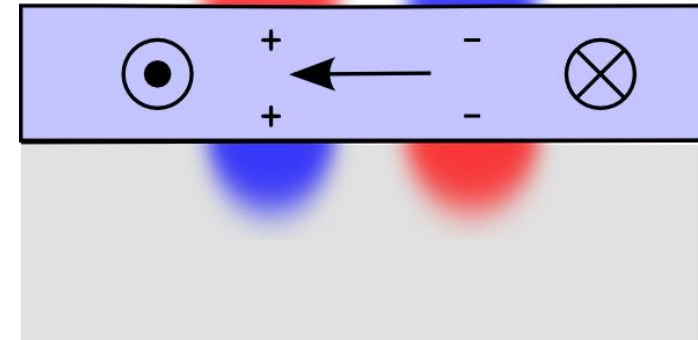


Permalloy film (20nm)
10x10 microns



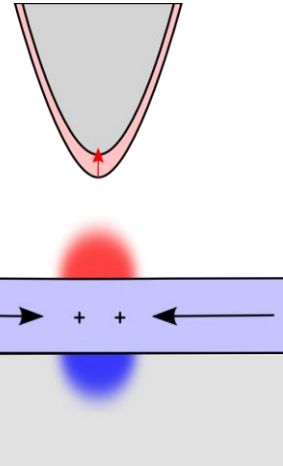
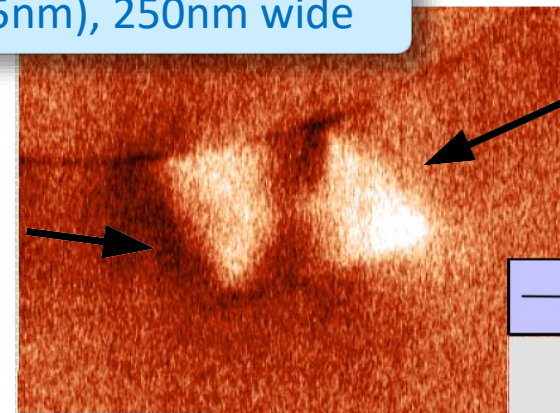
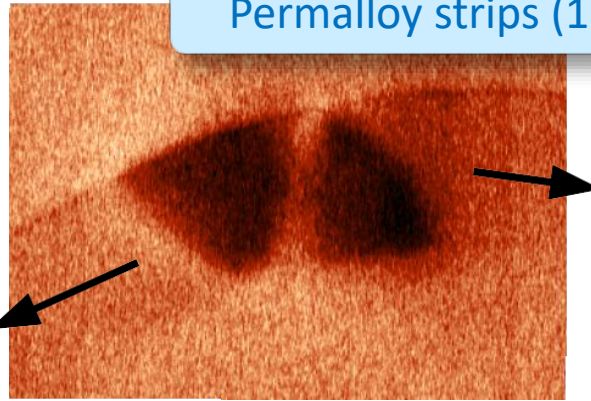
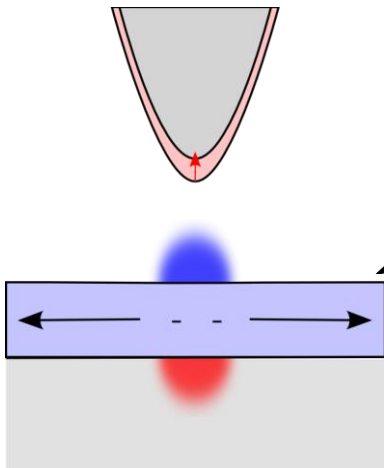
J. M. Garcia et al., APL 79, 656 (2001)

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



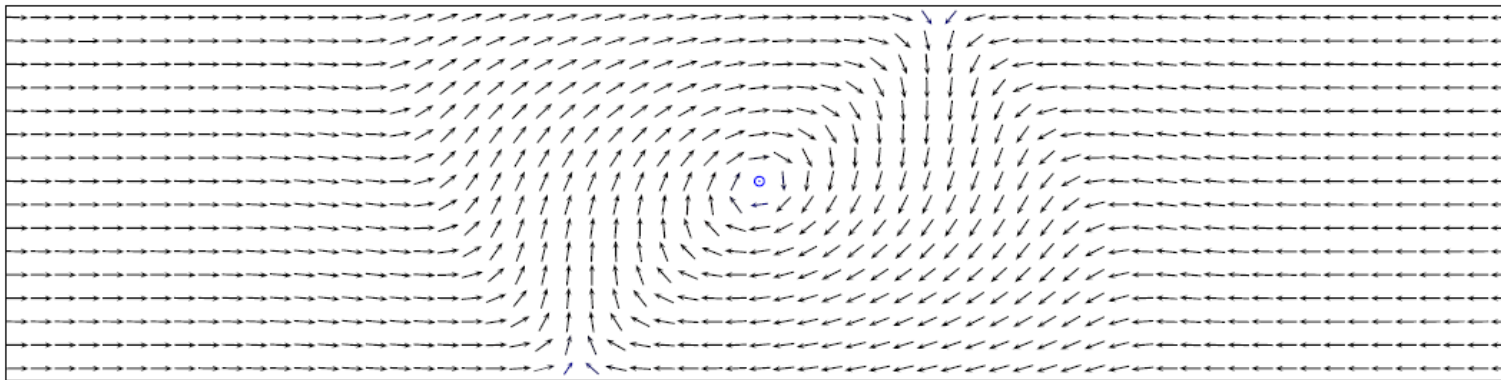
PRACTICE / Head-to-head domain walls

Permalloy strips (15nm), 250nm wide



Contrast : $\pm 0.3^\circ$,
tip 5nm CoCr

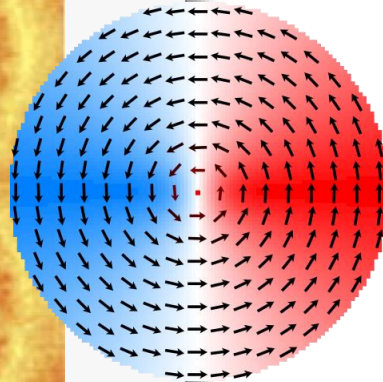
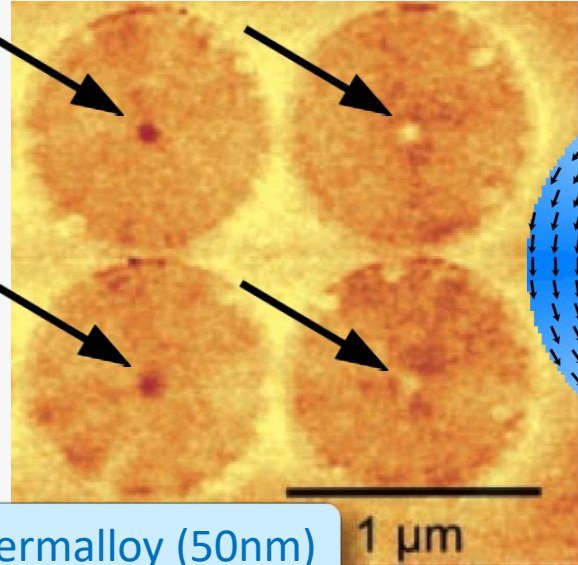
Known as vortex domain wall



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

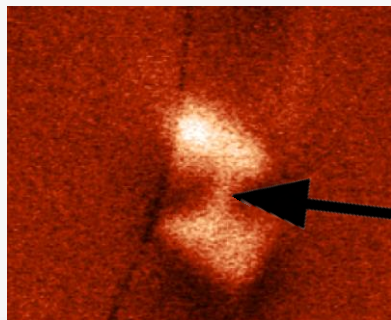
- Walls in in-plane magnetized stripes $\rightarrow$ MONOPOLAR
- Contrast informs about head-to-head or tail-to-tail

Observation Disk, vortex state



Permalloy (50nm)

T. Shinjo et al., Science 289, 930 (2000)

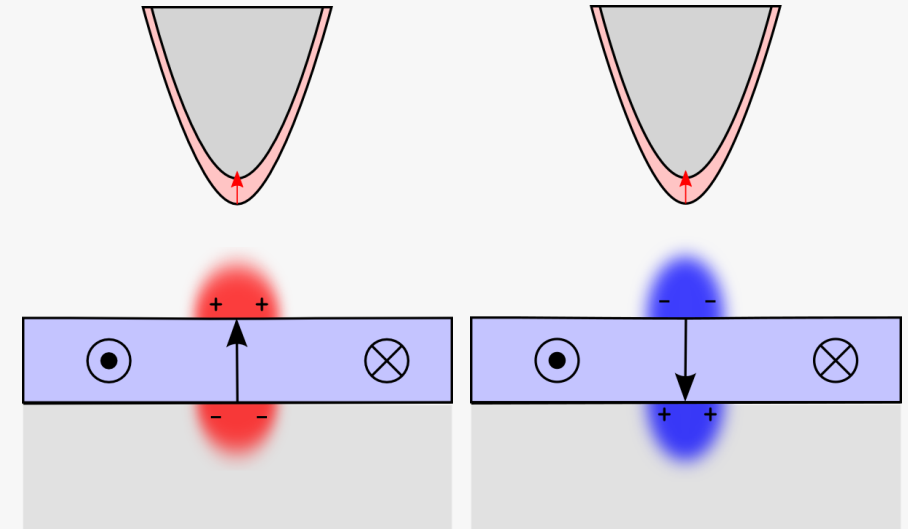


Strip,
vortex wall

Vortex is up

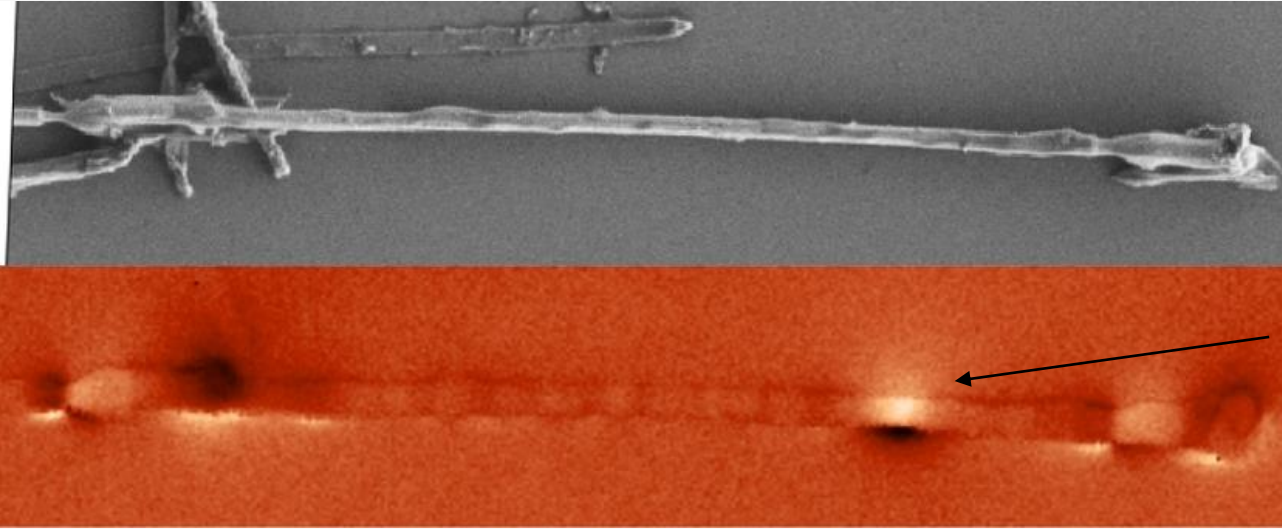
FeNiMo (15nm), strip width 200nm

Contrast

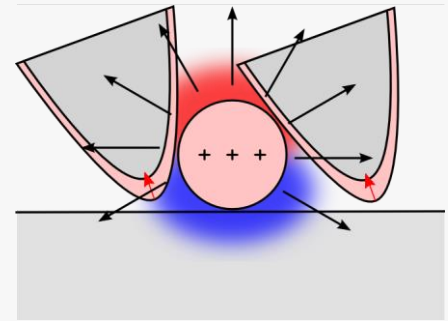


- MONOPOLAR, informs on core polarization
- May be hidden in surrounding wall contrast
- Vortex may be influenced by the tip

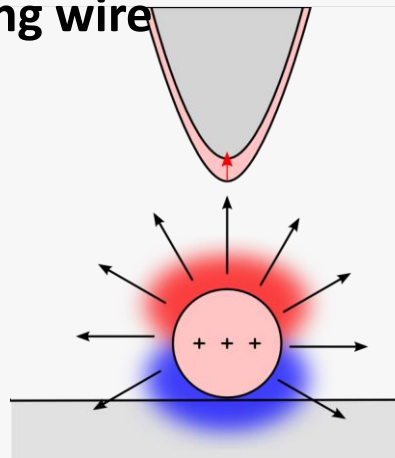
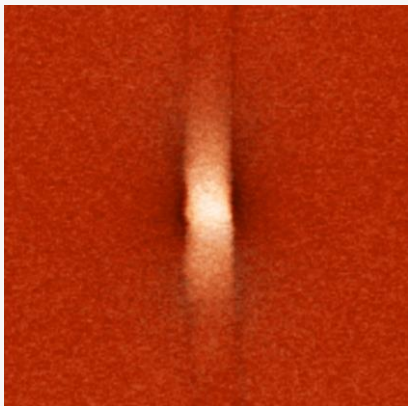
Tilted cantilever, across wire



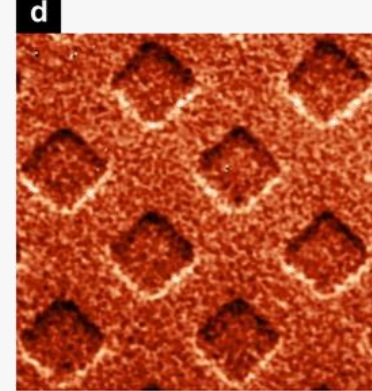
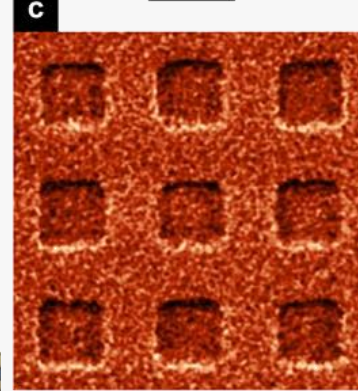
S. Da Col et al., APL109, 062406 (2016)



Tilted cantilever, along wire



10 μm

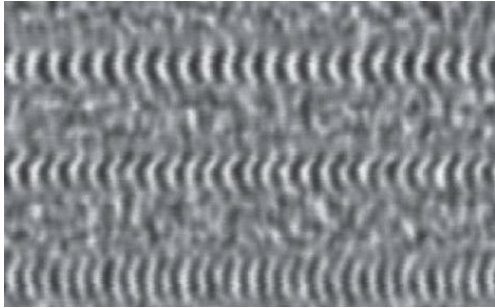


G. Ciuta, IEEETm 52, 6500408 (2016)

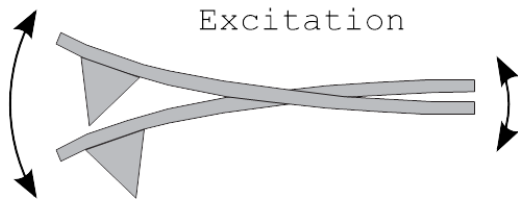


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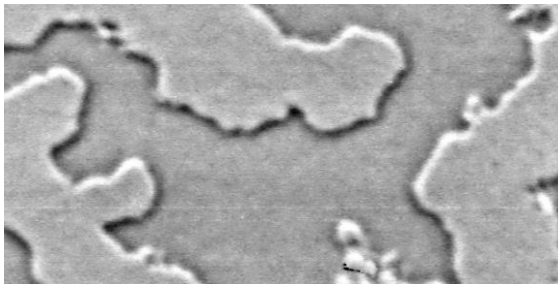
■ Motivation and criteria



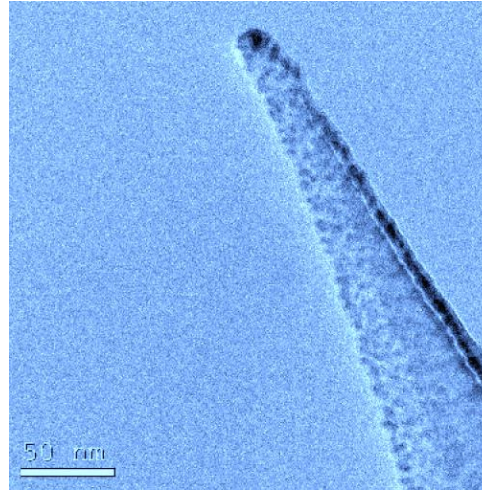
■ Working principle



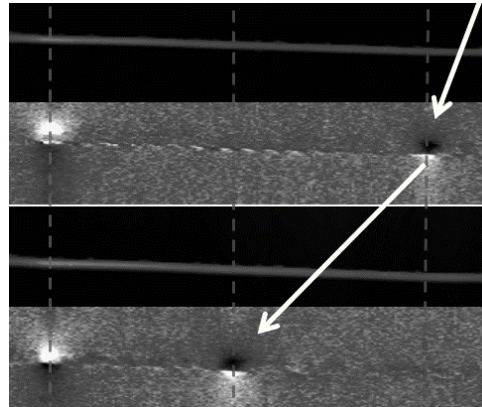
■ Image analysis



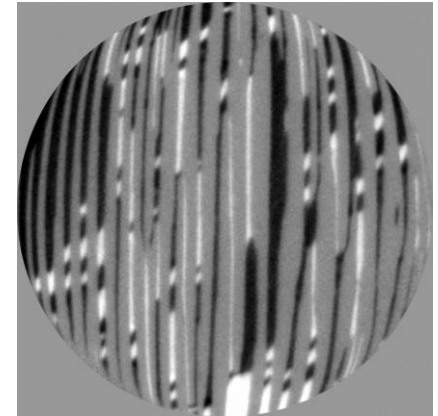
■ Choice of tips

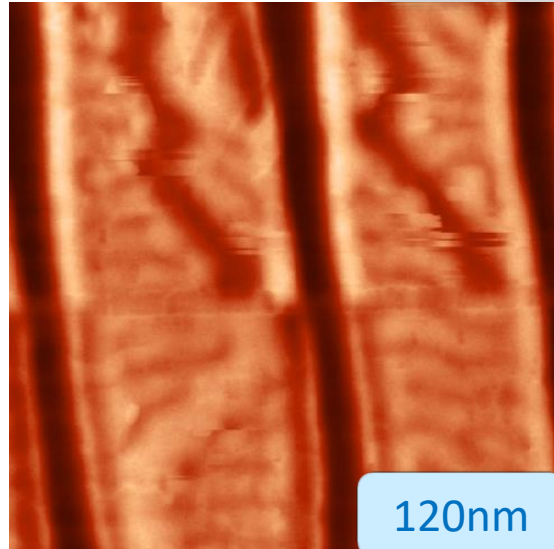
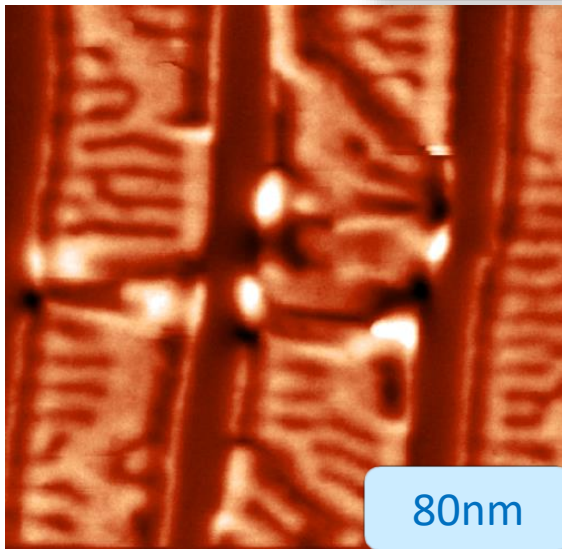
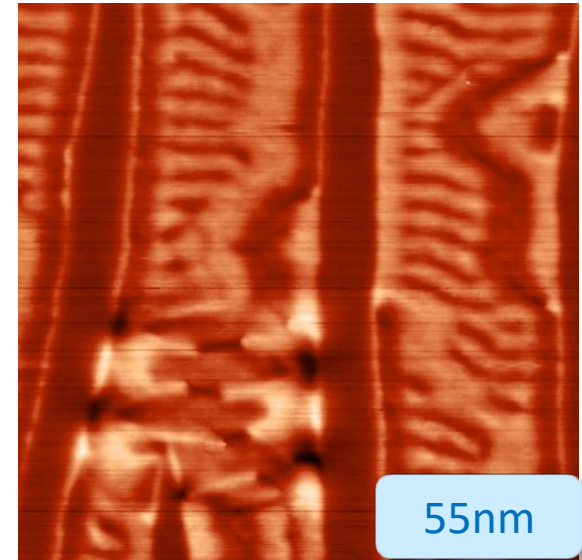
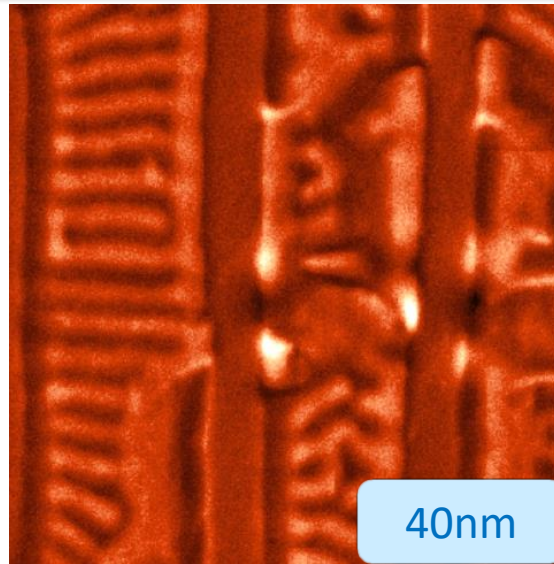
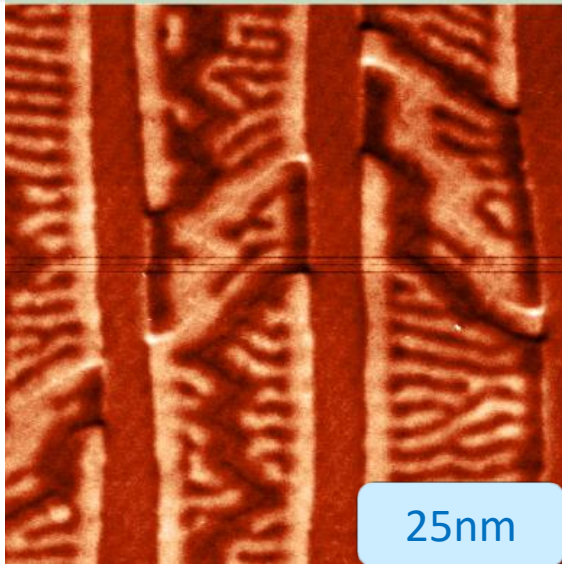


■ Operando imaging



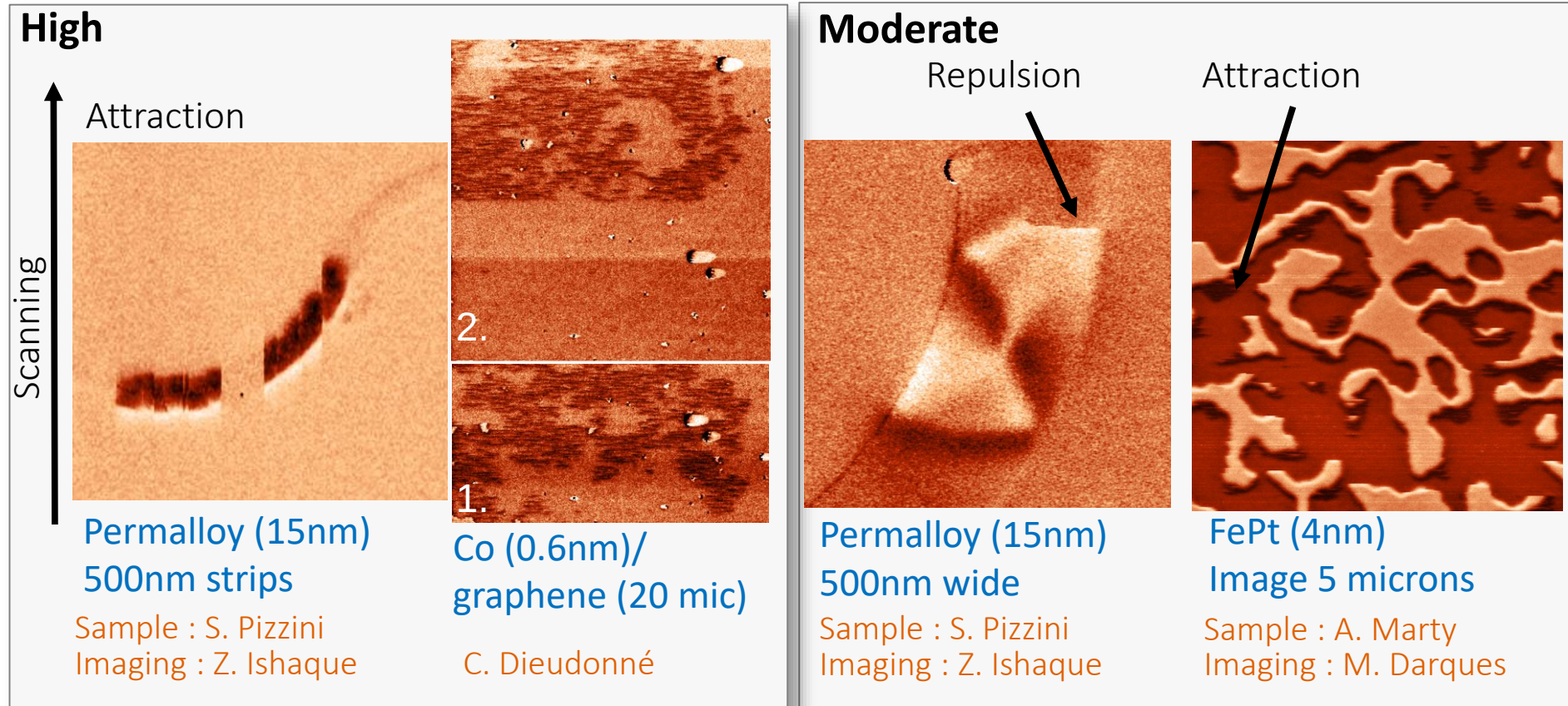
■ Panorama of other microscopies





- Compare tips !
- Use identical sample and imaging parameters

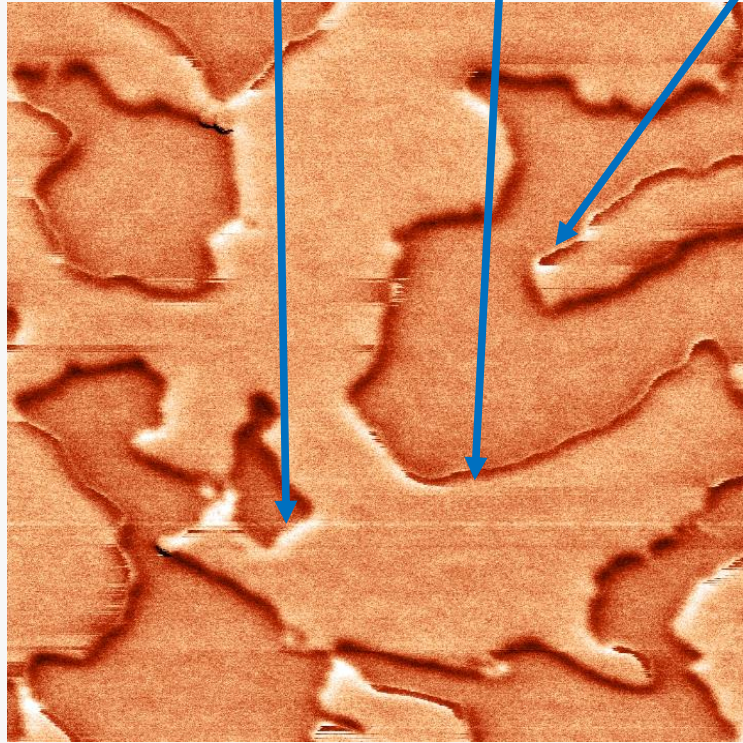
Sample : MnAs(001)
R. Belkhou (Soleil)



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

TIPS. Sample influencing tips

High Normal Attractive Switch

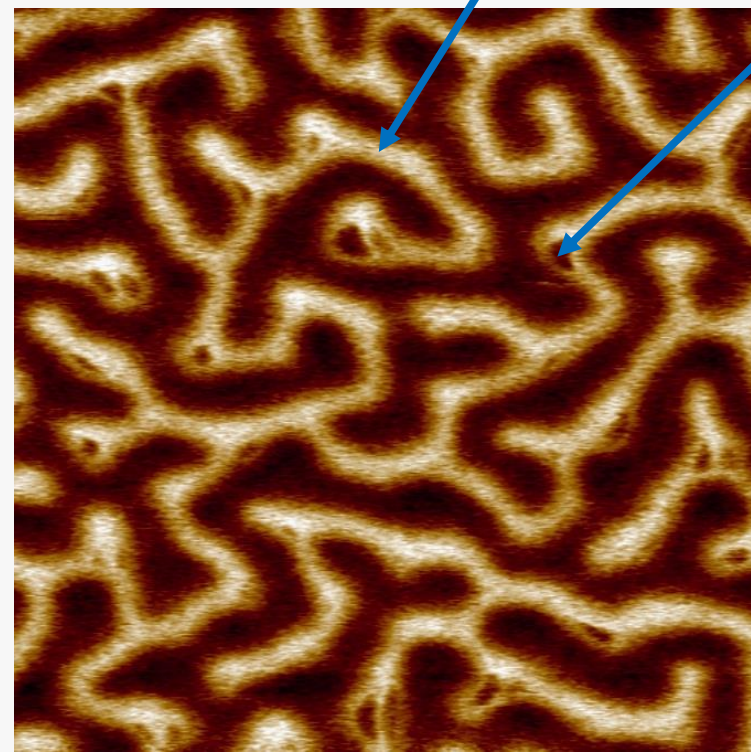


FePt(001) 4nm

Sample: Alain Marty

2x2 μm

Moderate Repulsion Attraction



Pd/Co/Au multilayers

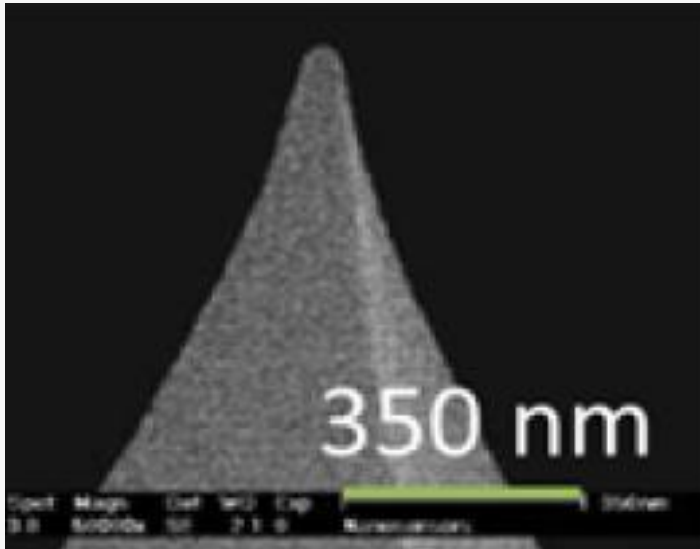
Sample: Chloé Bouard

2x2 μm

- Sharp reversal features, and/or streaks parallel to fast direction
- Occurs at the highest repulsive fields

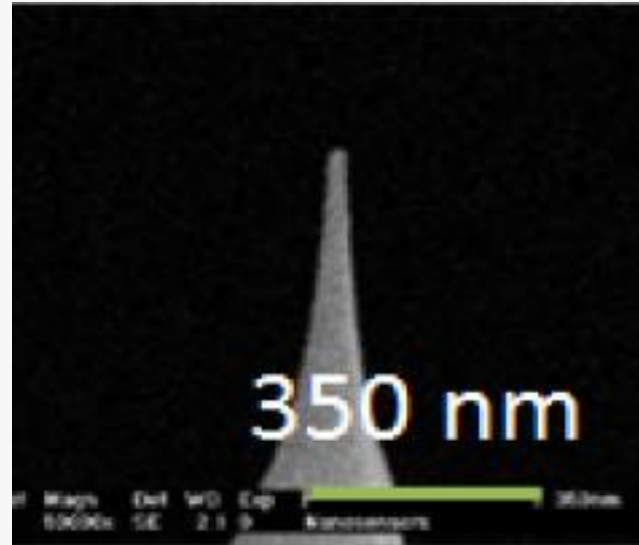
Improve resolution

From sharp to ultrasharp AFM tips



Asylum 240TS

Radius of curvature : 10 nm



Nanosensors PPP-SSS

Radius of curvature : 2-5 nm

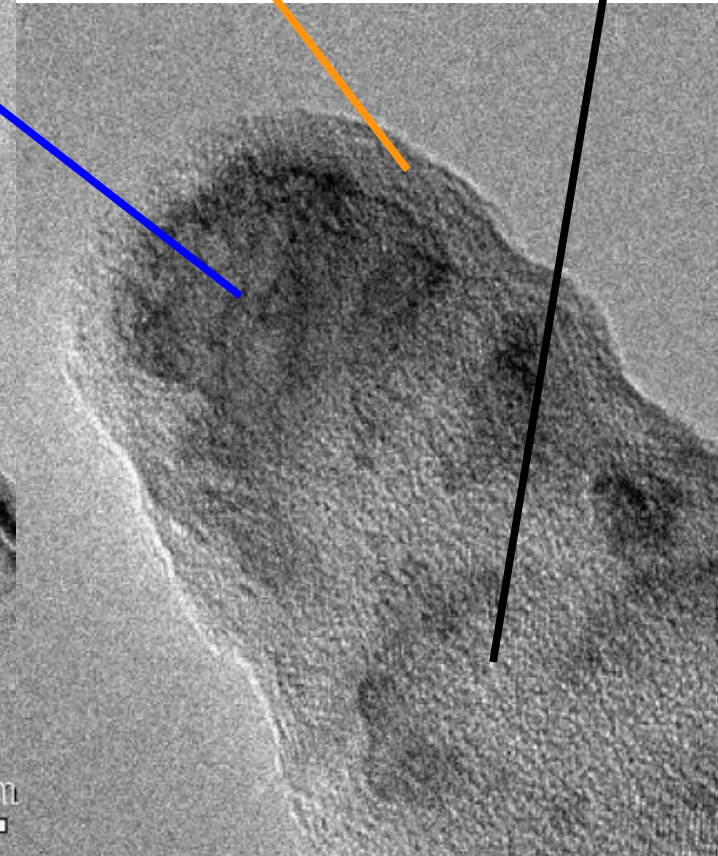
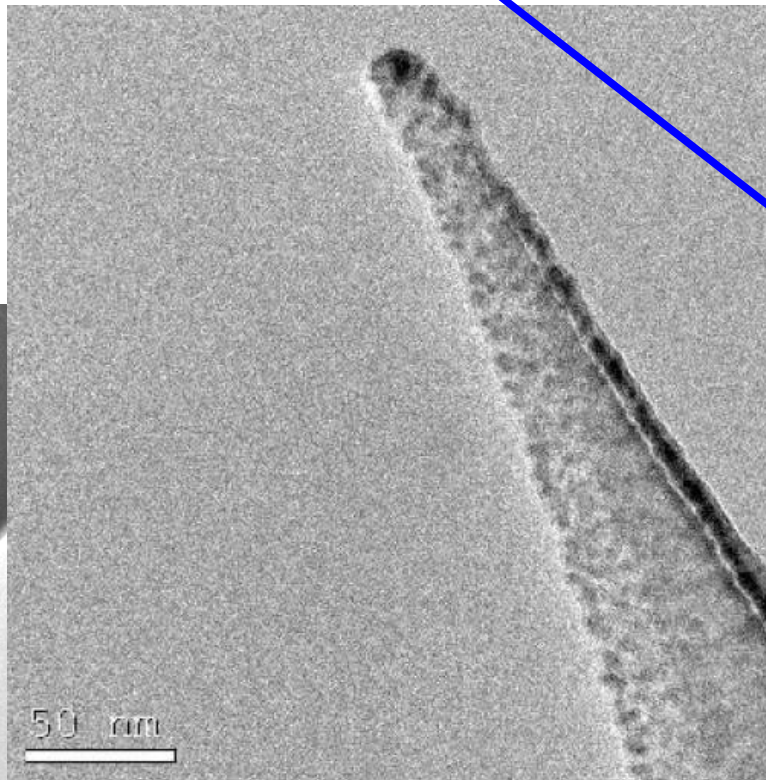
Engineer magnetic coating

- ❑ Basis : Co₈₀Cr₂₀ (similar to hard disk media)
- ❑ Means : sputtering.
- ❑ Stacking : Based on CoCr [2-50 nm], various buffer and capping layers

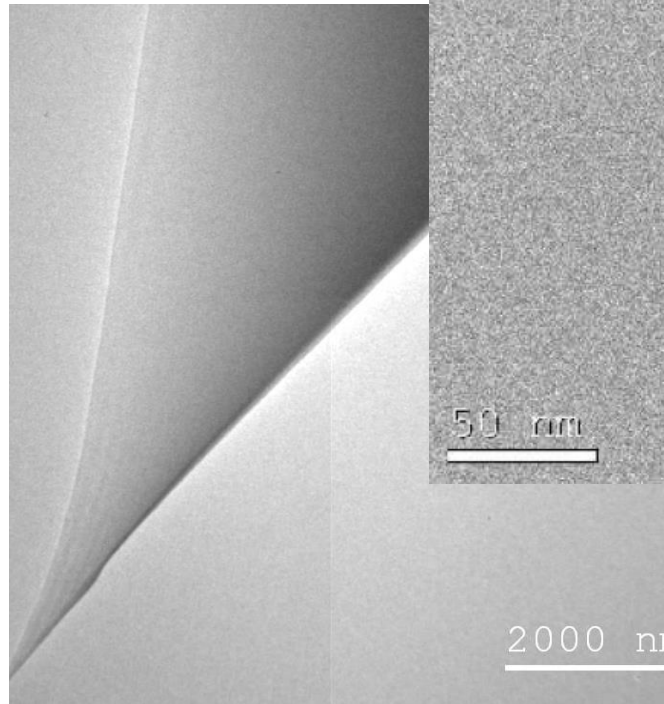
Magnetic
coating

Antiwear
coating

Initial Si apex



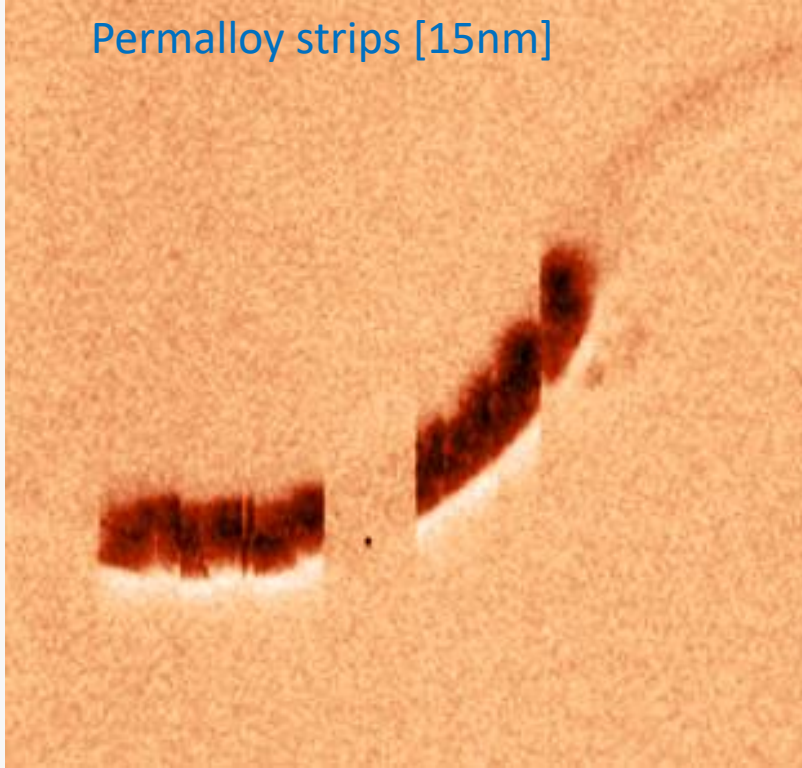
Transmission Electron Microscopy (A. Masseboeuf)



TIPS. Reduce tip-sample interaction

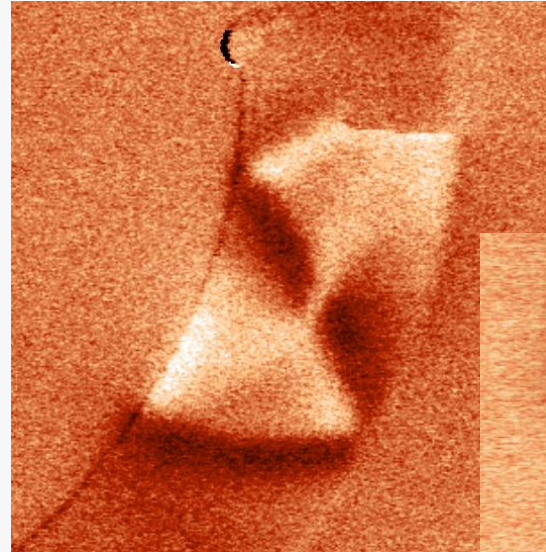
Commercial tips, “low moment”

Permalloy strips [15nm]

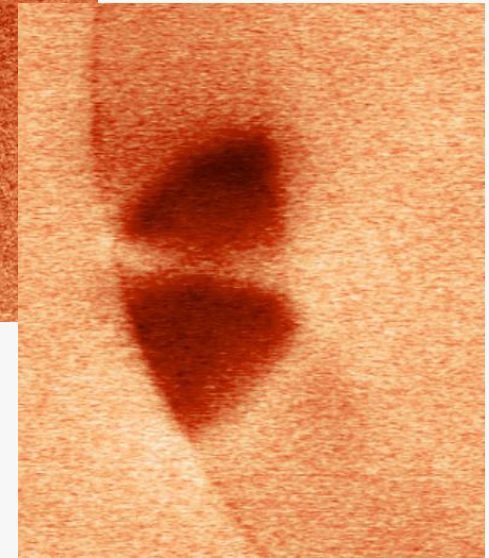


Home-made tips, truly low moment

Tip : CoCr[10nm]
500nm strips
15nm permalloy



Tip : CoCr[5nm]
300nm strips
15nm Permalloy

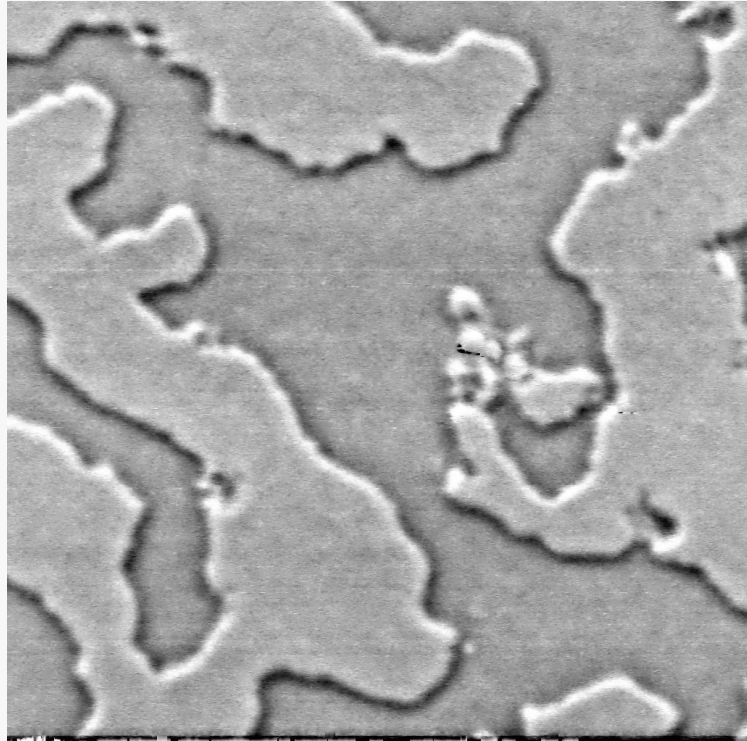


Sample: S. Pizzini
Imaging: Z. Ishaque

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

Spatial resolution 15nm

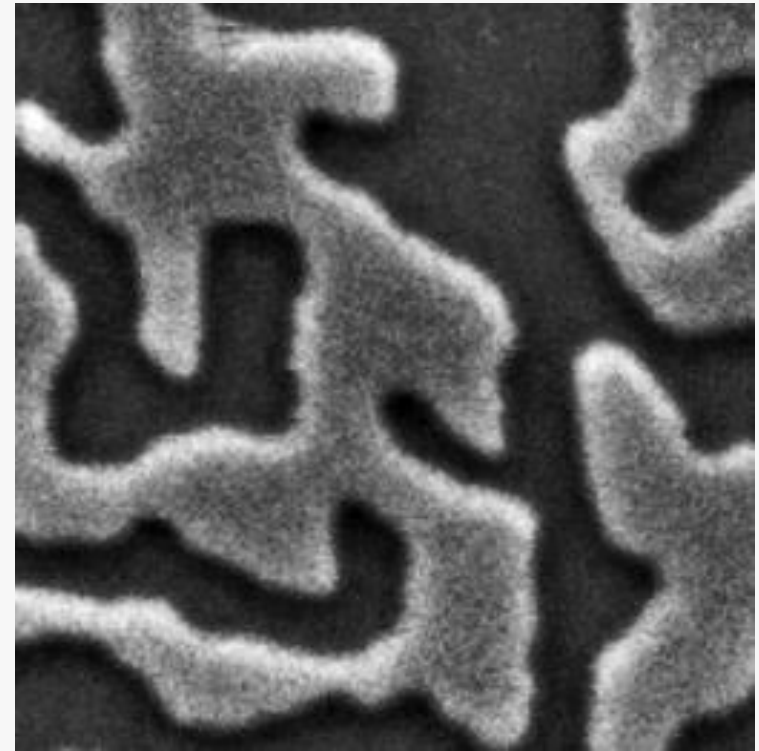
Test sample : FePt[4nm]. Perpendicular magnetization, narrow domain walls



2x2 μm

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

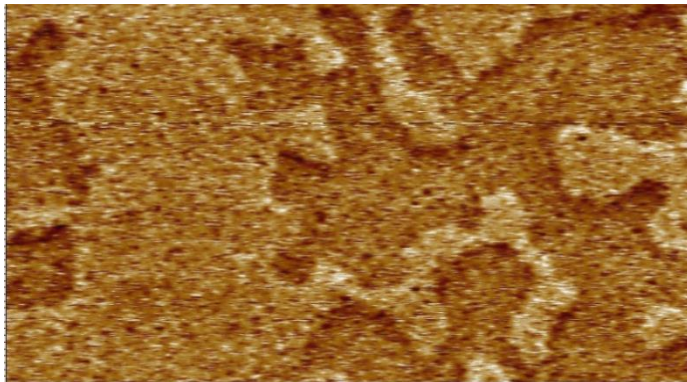
Commercial “low-moment” tip



■ Spatial resolution: 15-20nm

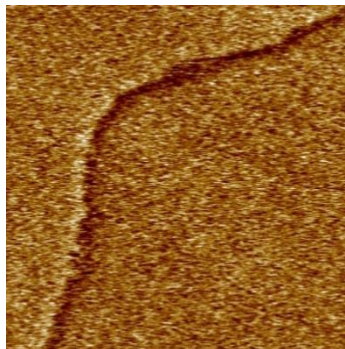
Specific aspects

- Require low stray field tips
- Low stray fields and low film thickness -> Sensitivity?



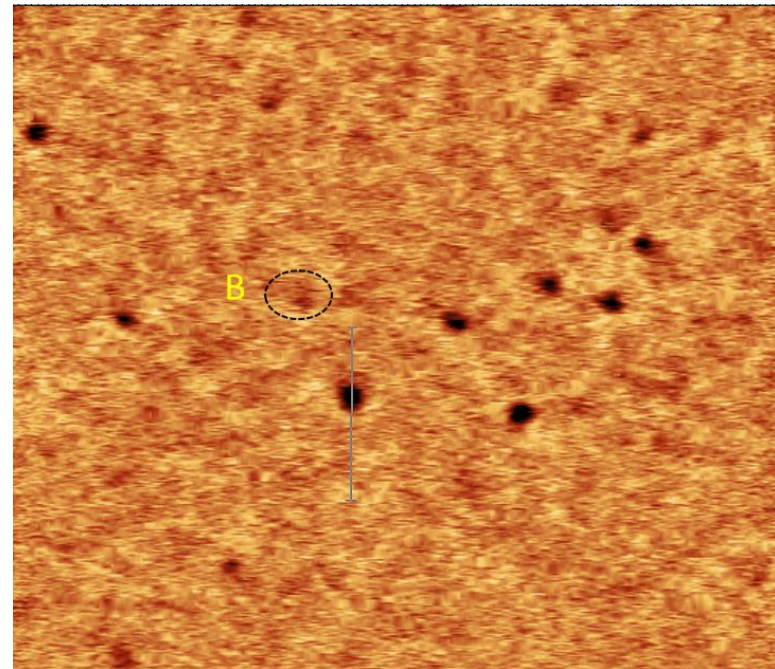
Sample: R. Juge

5x2.8 μm



Imaging: K. Richter

1.2x1.2 μm



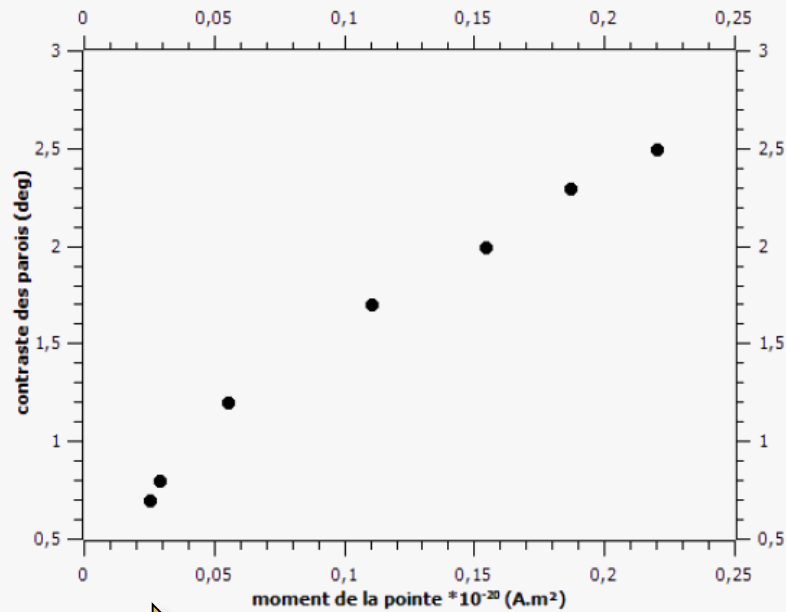
Imaging: G. Rana

3x2.5 μm

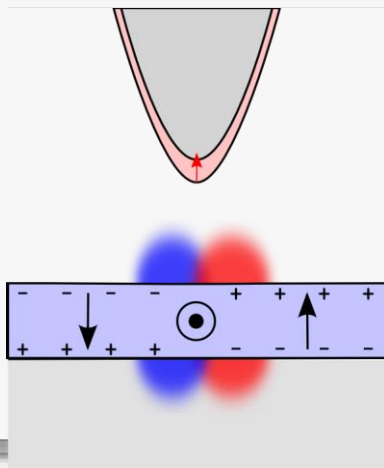
Conclusion

- Remains measurable
- Mutual contrast nearly absent

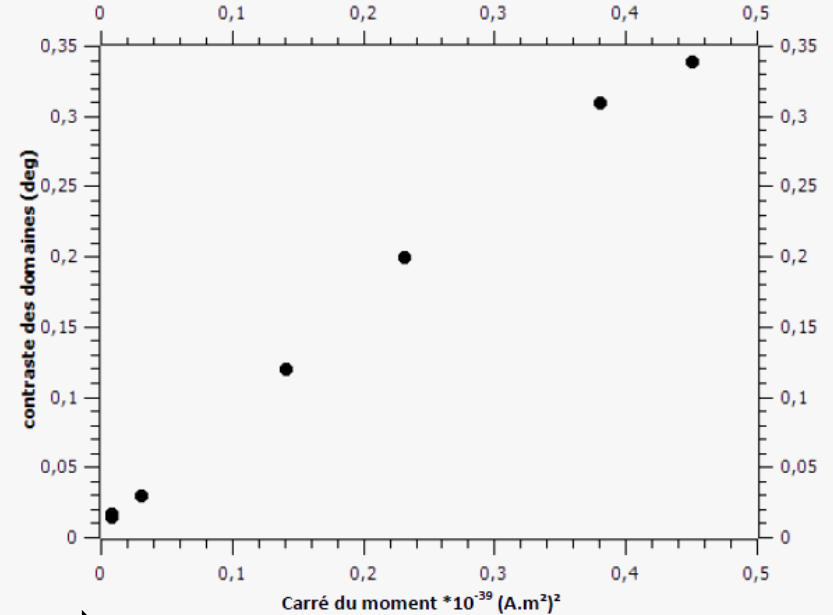
Domain-wall contrast



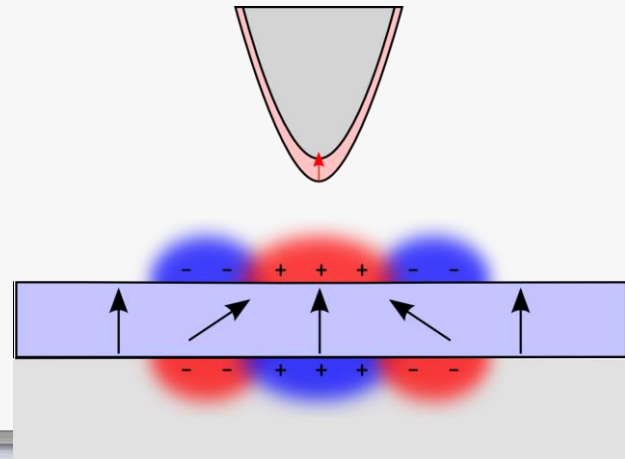
Linear with tip moment



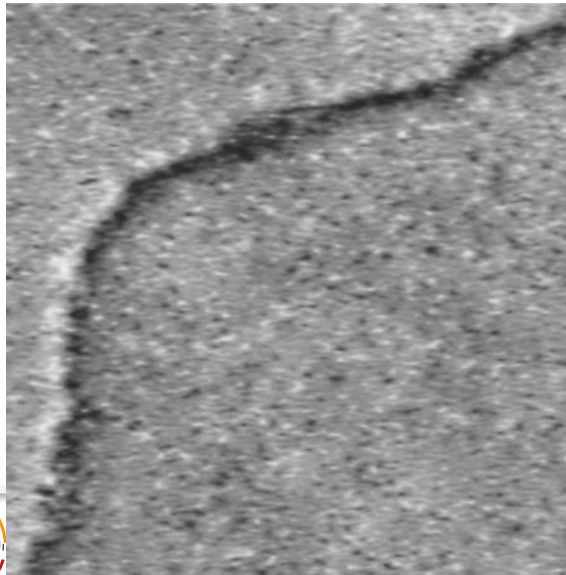
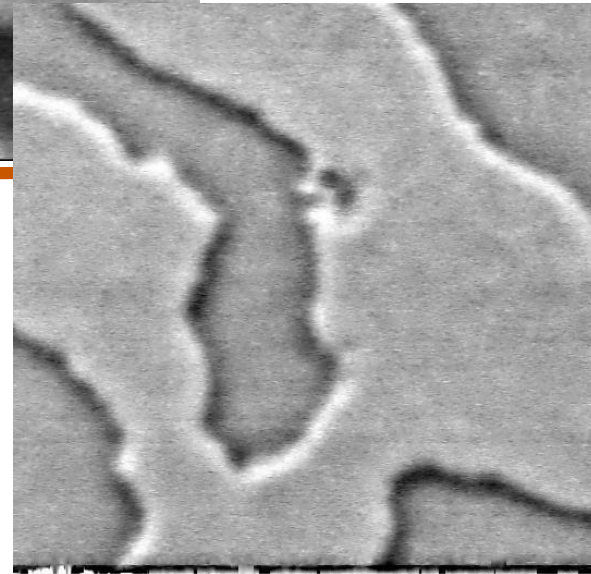
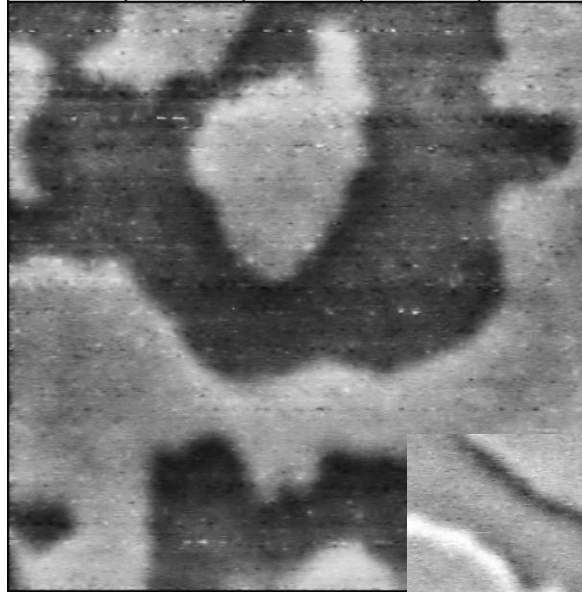
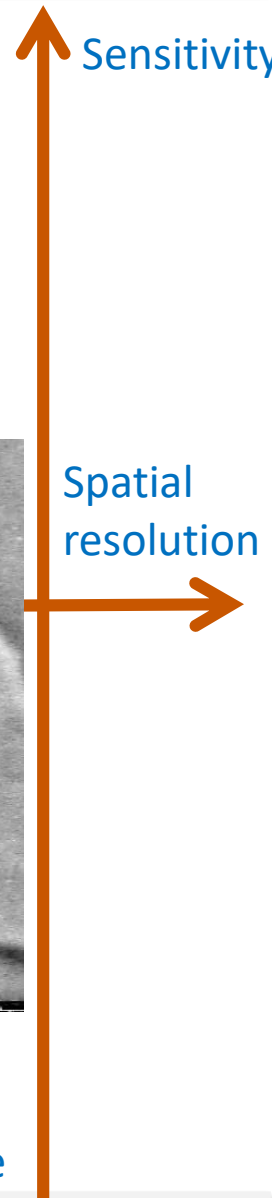
Domain contrast



Linear with square of tip moment



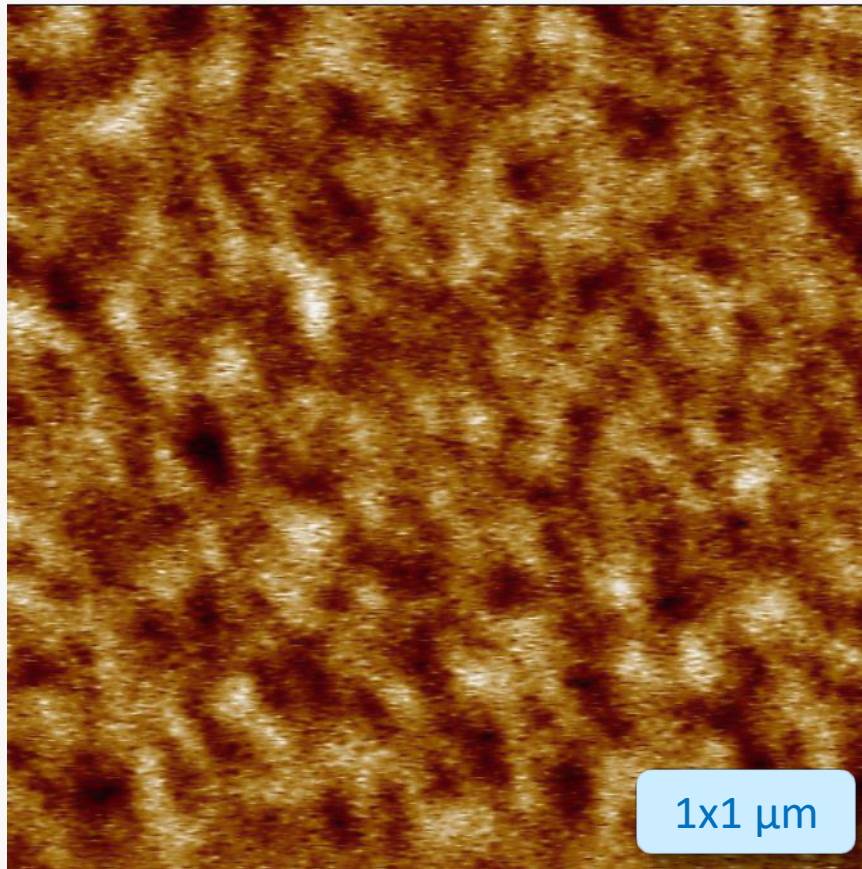
TIPS. Both resolution and sensitivity matter



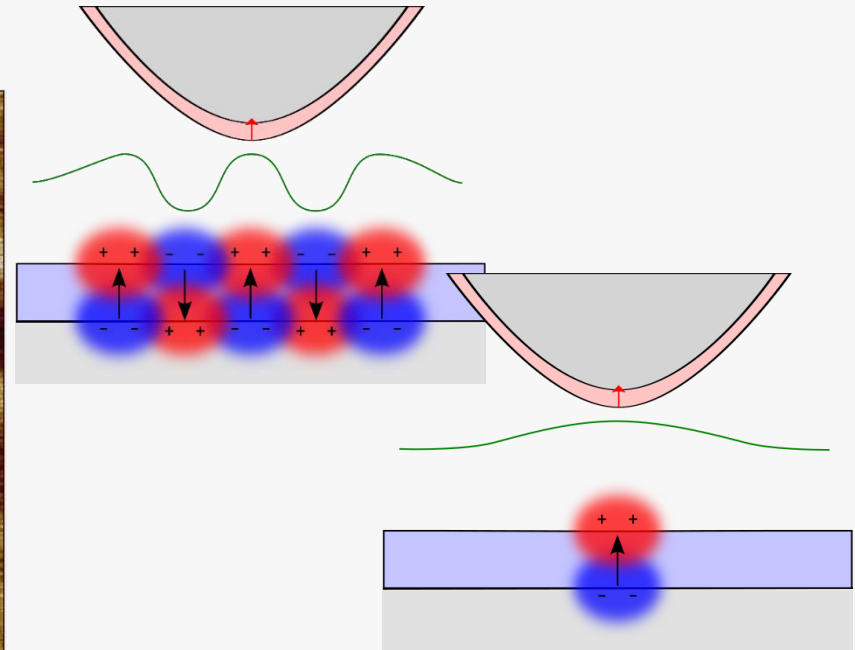
1x1 μm

Non-invasive

NOT the smallest feature on an image



FePt-C recording media

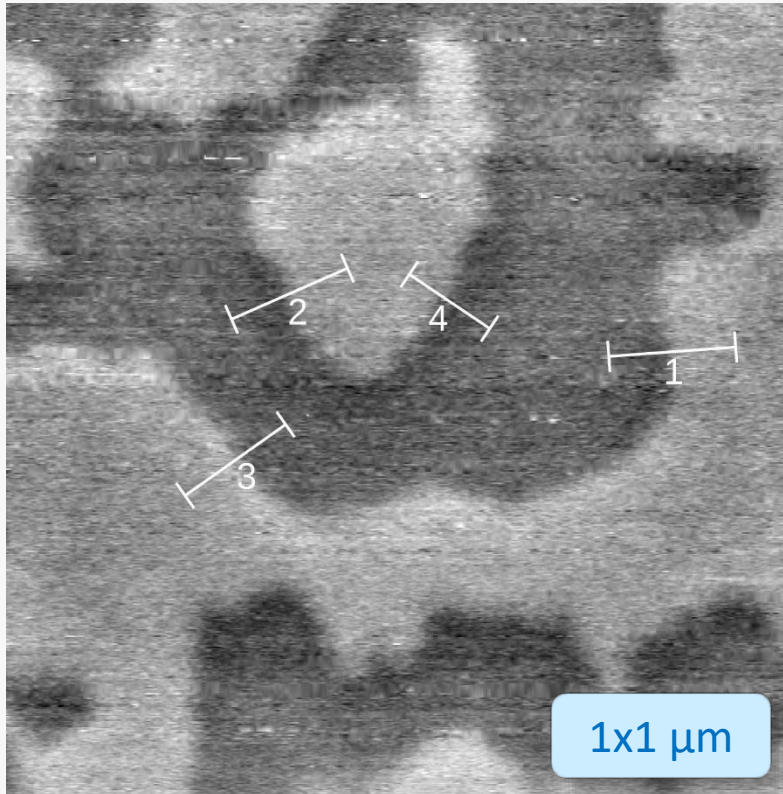


- Small scale modulation
- Reduced magnitude
- A single object would appear larger

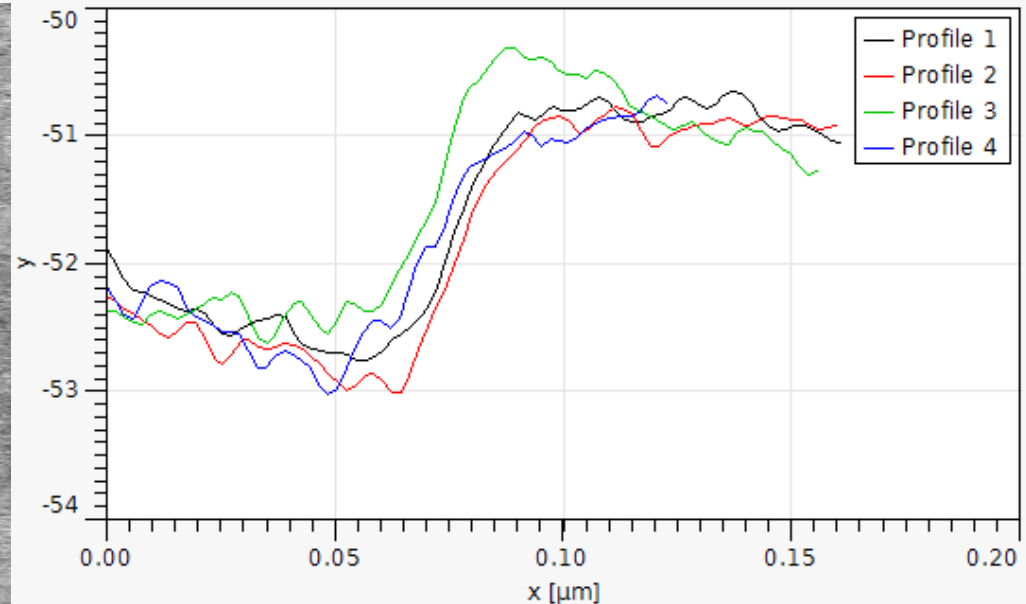
D. Diény et al., IEEE Trans. Mag., in print (2018)



Use isolated well-defined magnetic pattern



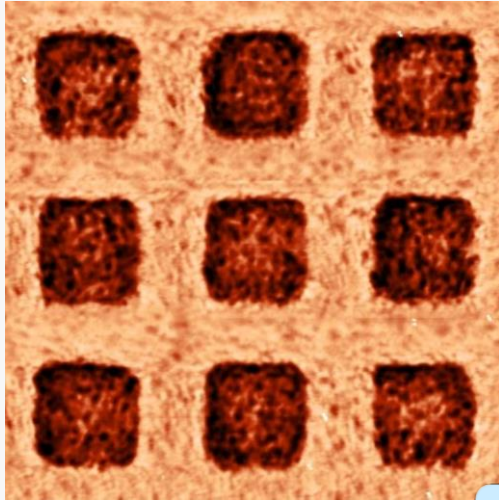
FePt, epitaxial (4nm)



- Definition-dependent: FWHM, variance, 85% etc.
- Make statistics: object, orientation etc.
- Here: around 20nm
- Advanced: modeling, deconvolution

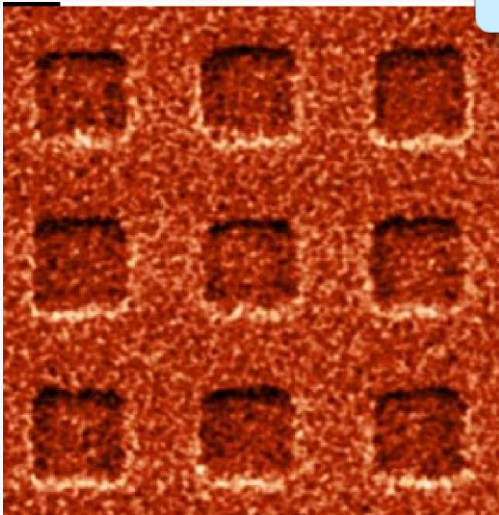
Quantitative analysis, see e.g.: H. Hug, J. Appl. Phys. 83, 5609 (1998) and followers

May require hard tip coating



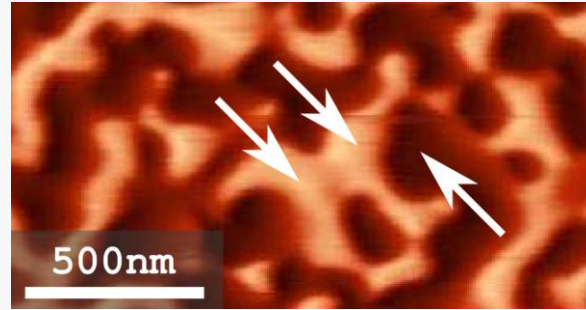
Soft
coating

50x50 μm

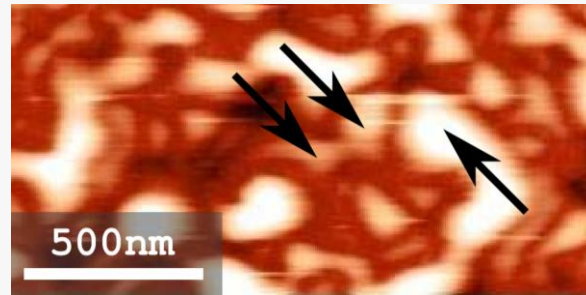


Hard
coating

May require stiff cantilevers



MFM



Topography
(artefacts)

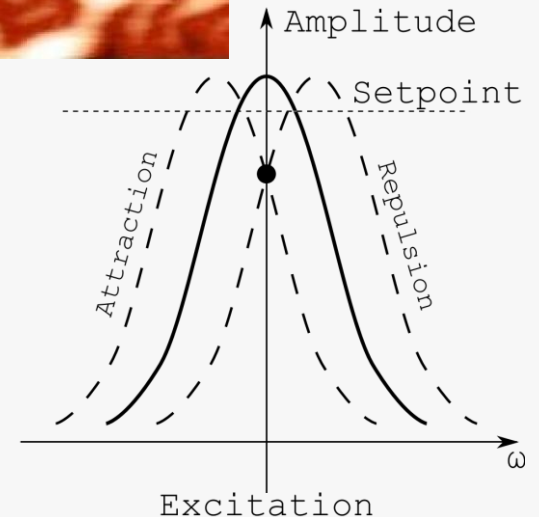
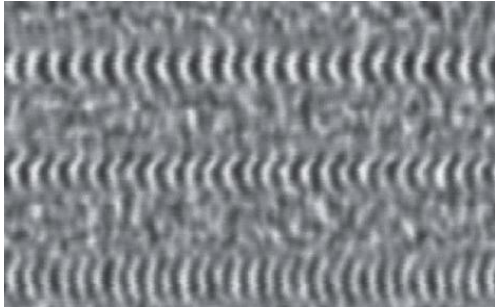


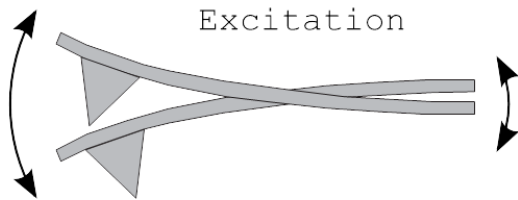


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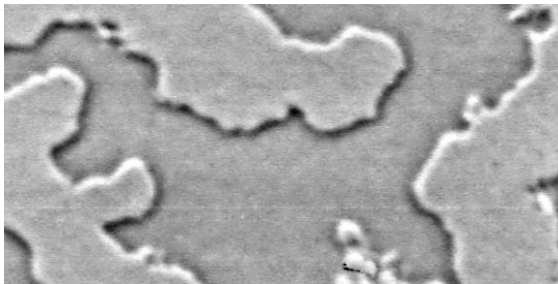
■ Motivation and criteria



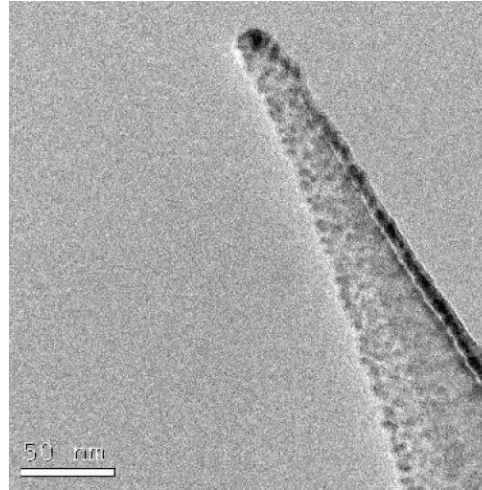
■ Working principle



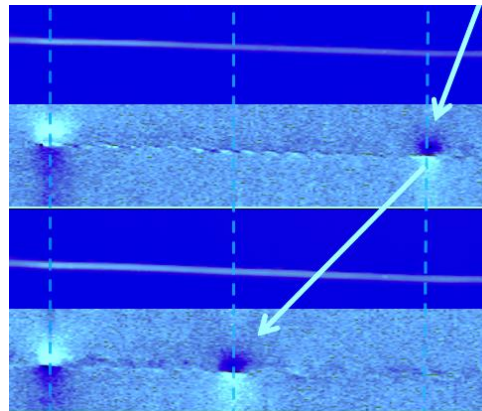
■ Image analysis



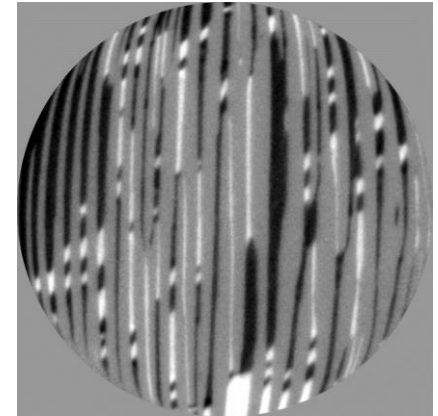
■ Choice of tips



■ Operando imaging

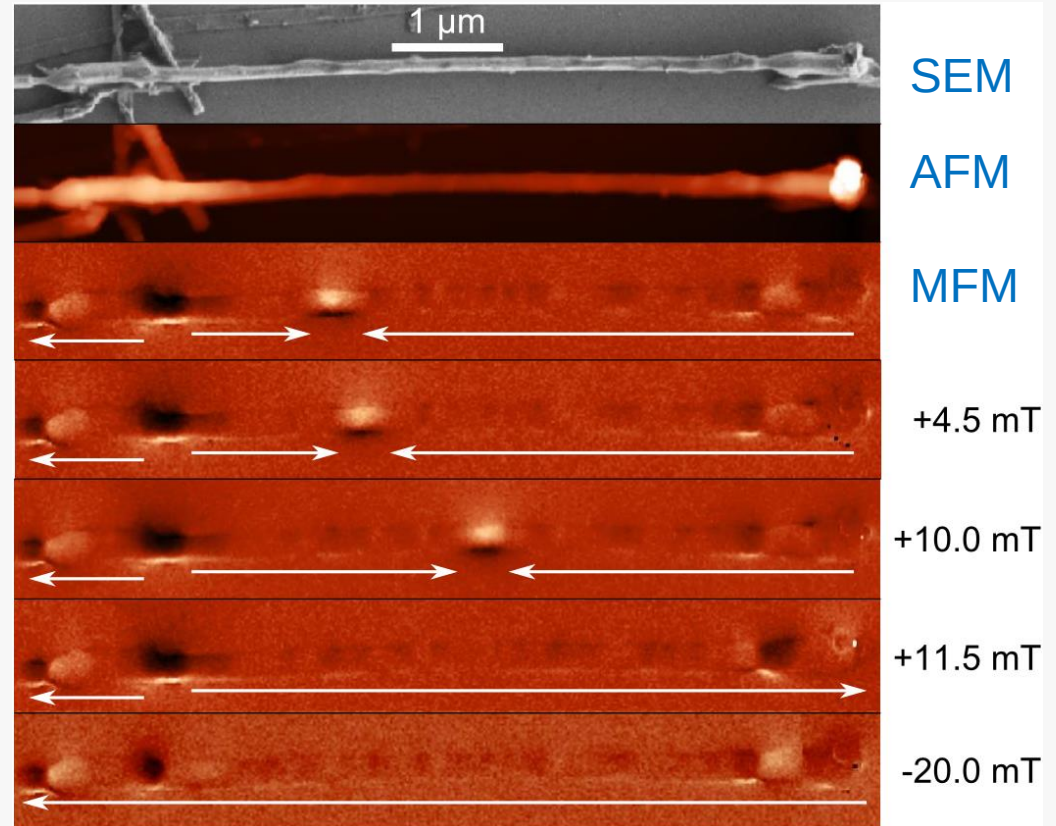


■ Panorama of other microscopies



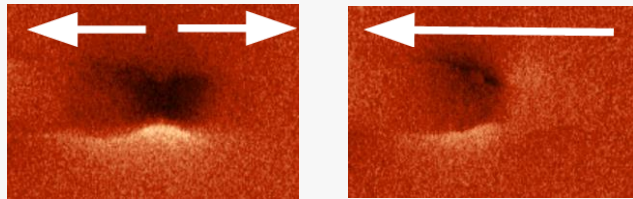


Domain wall motion



S. Da-Col et al., APL109, 062406 (2016)

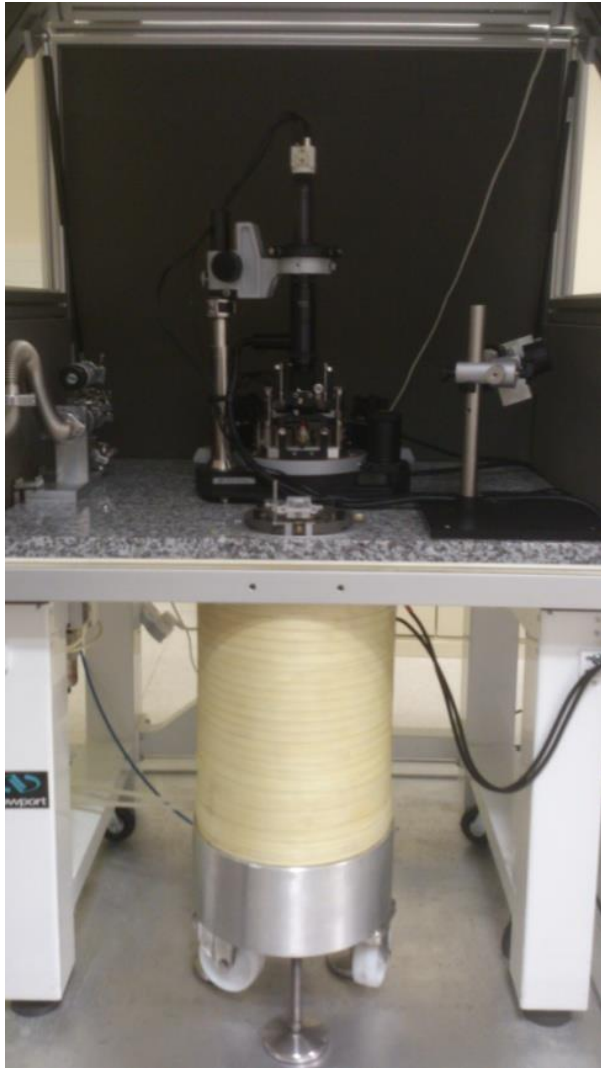
Protrusions and constrictions



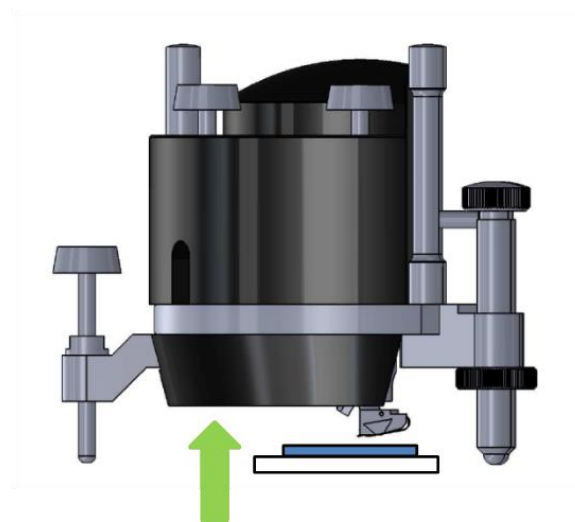
➔ Pinning field ~ 30 mT

- Imaging under field
- Magnetization processes

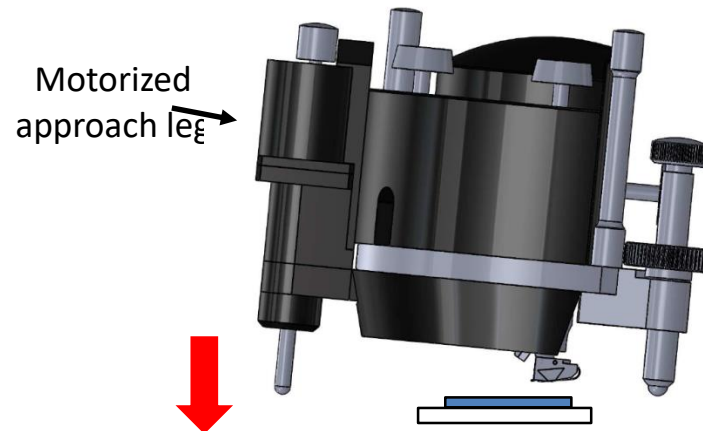
OPERANDO. Out-of-plane field



Field $>1\text{T}$ (custom-made)
Optimized cooling



Microscope approach
by moving the sample up
(standard configuration)

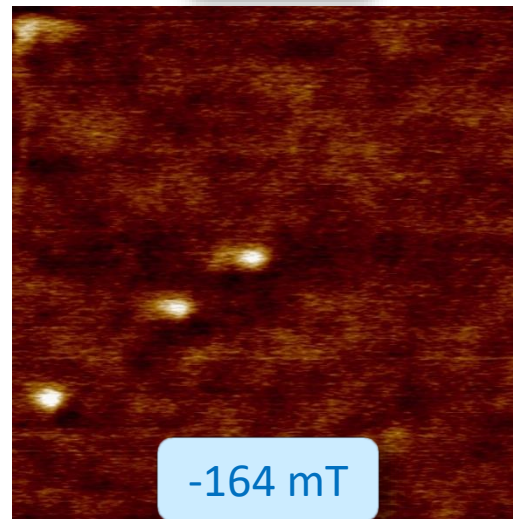
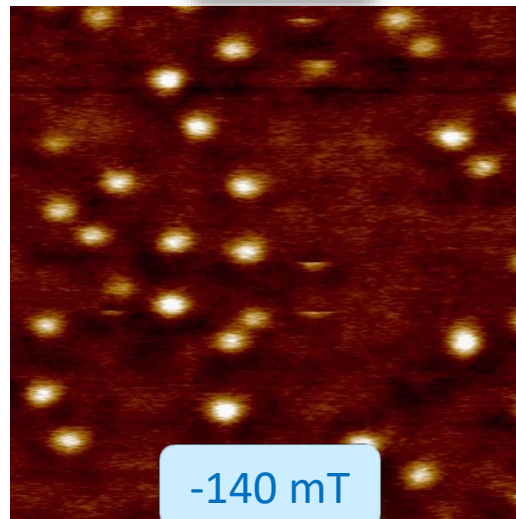
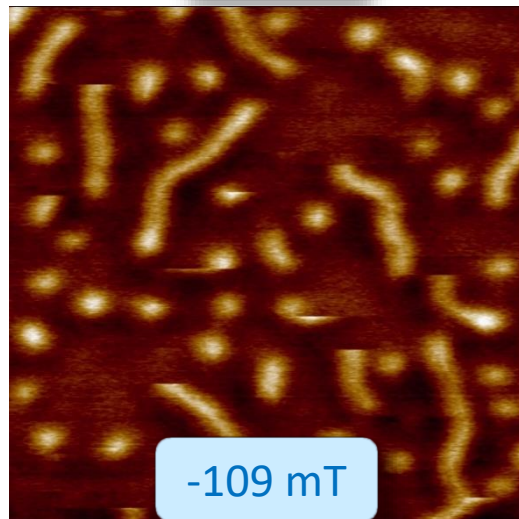
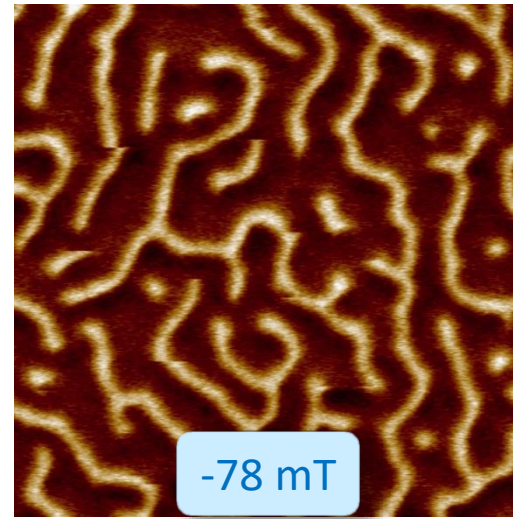
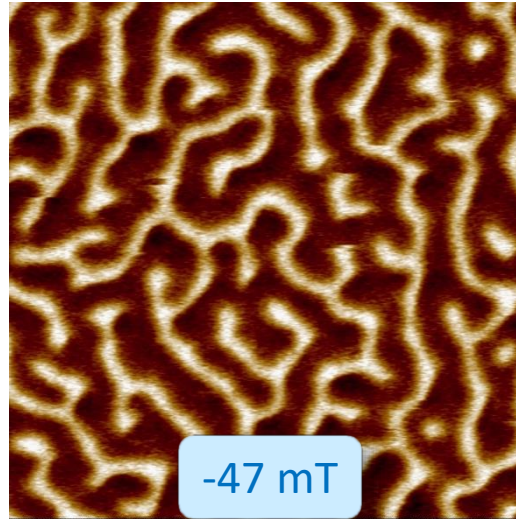
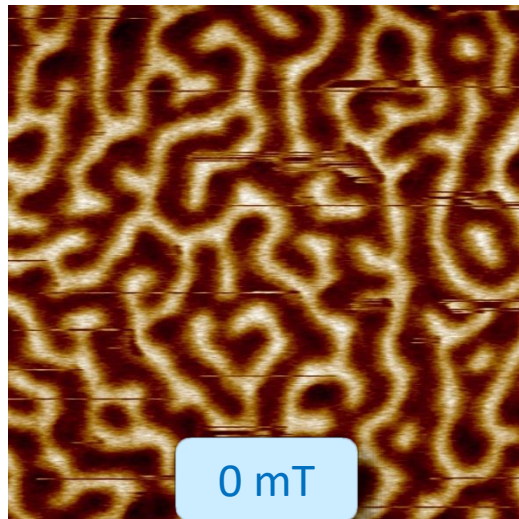


Microscope approach
by tilting the head
thanks to the motorized leg
(coil coupled configuration)

■ Approach by head (motorized leg)

OPERANDO. Out-of-plane field

Pd/Co/Au multilayers, $2 \times 2 \mu\text{m}$



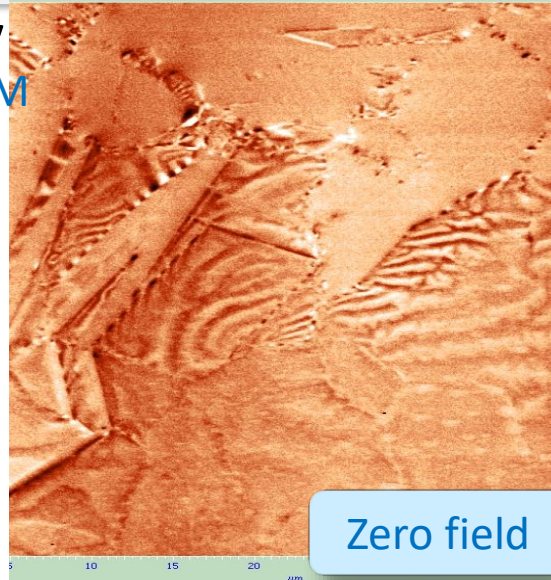
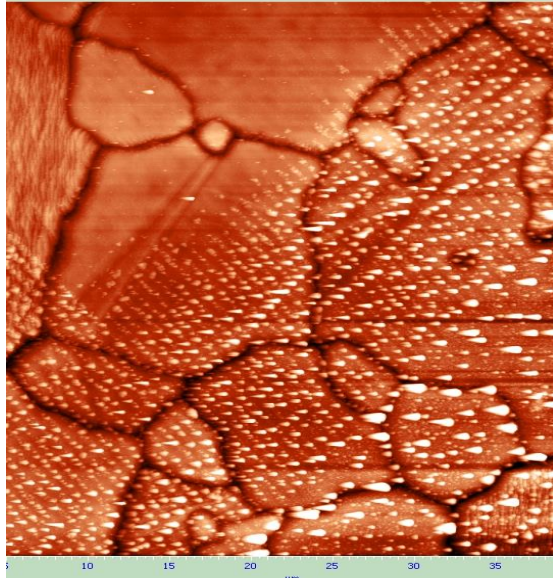
Sample: Chloé Bouard

OPERANDO. Out-of-plane field

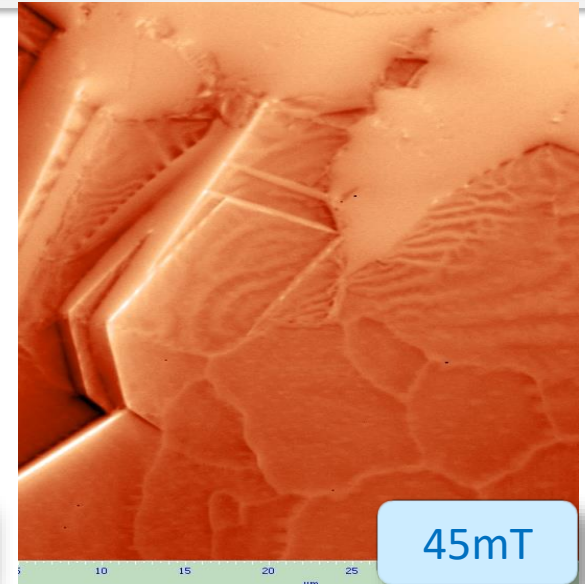
Magnetic shape-memory alloy

Topography (grains)

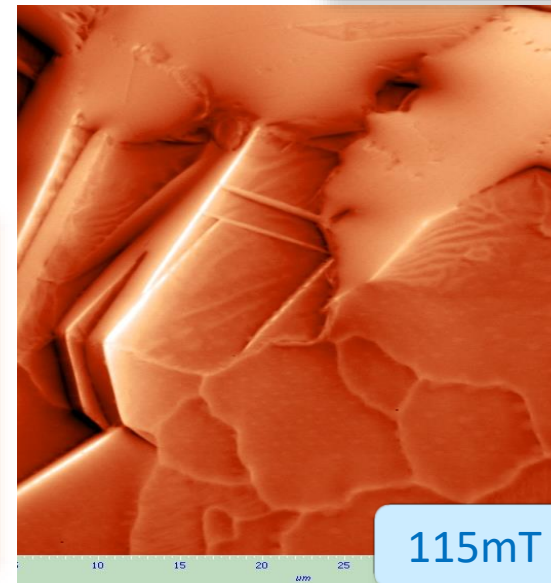
MFM



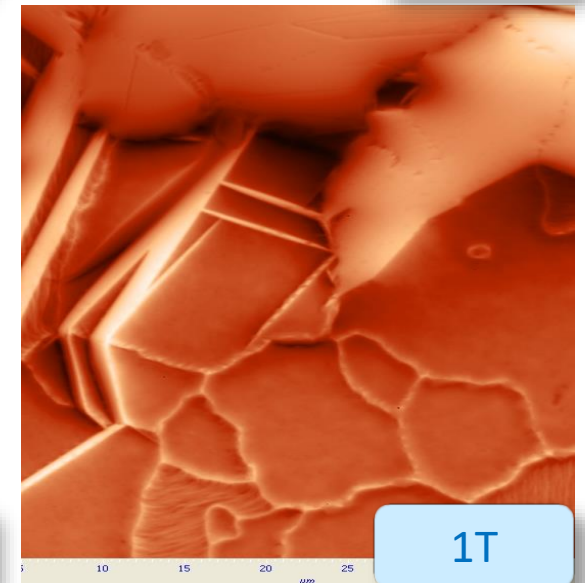
Zero field



45mT



115mT

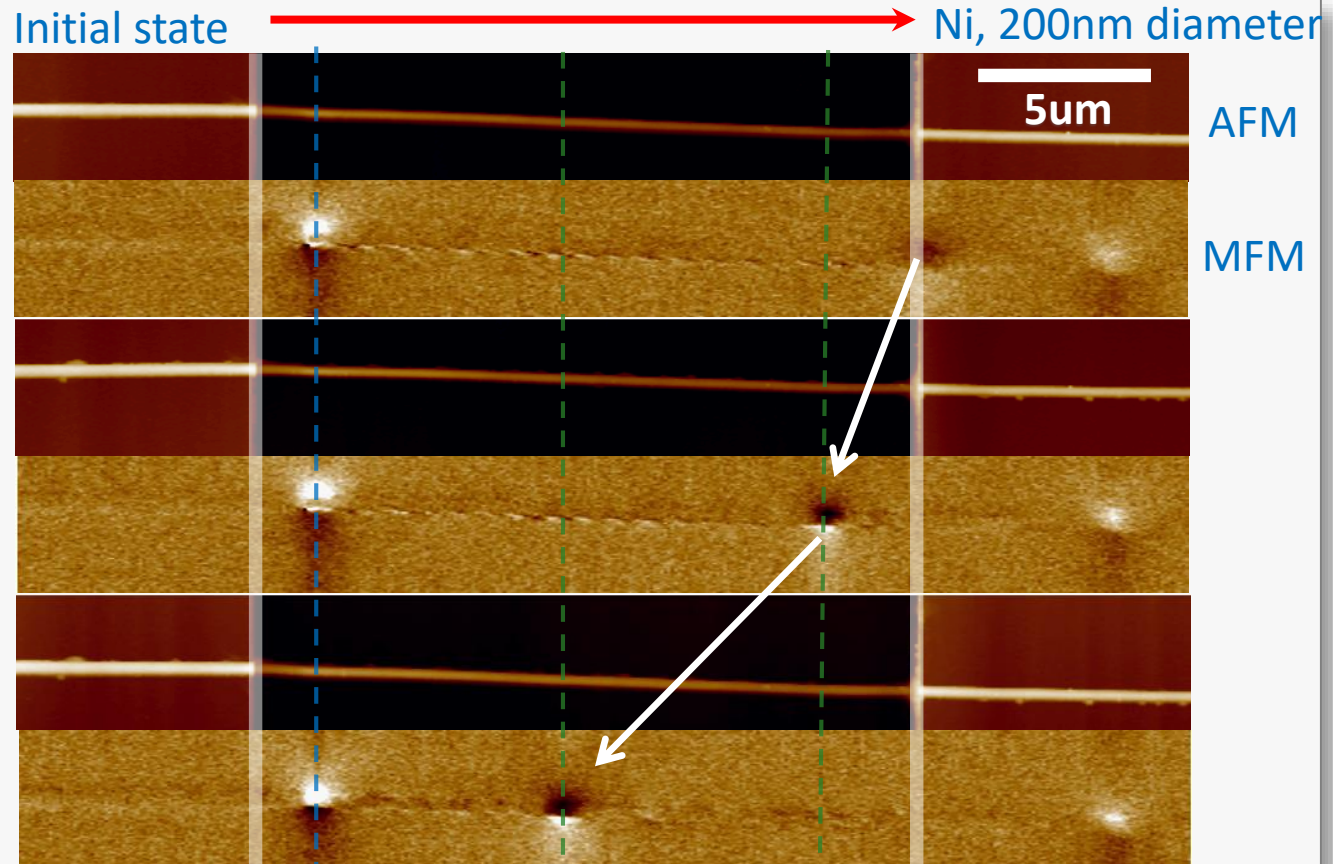


1T

Sample: G. Crougneau

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

Domain-wall motion



$$j \simeq 1.5 \cdot 10^{12} \text{ A/m}^2$$

during 30 ns

$$j \simeq 1.5 \cdot 10^{12} \text{ A/m}^2$$

during 30 ns

Sample, imaging: Sylvain Martin



M. Schöbitz et al, in preparation

Measurements

Fadhel Abedi, Ioan Chioar, Geta Ciuta, Guillaume Crouigneau, Sandrine Da Col, Michael Darques, Christophe Dieudonné, Alexander Grimm, Zahid Ishaque, Keita Ito, Simon Le Denmat, Svenja Perl, Gaurav Rana, Kornel Richter, Jérémy Tillier

Tip development

S. Le Denmat, Ph. David, A. Masseboeuf (CEMES Toulouse)

Simulations

S. Jamet, JC Toussaint

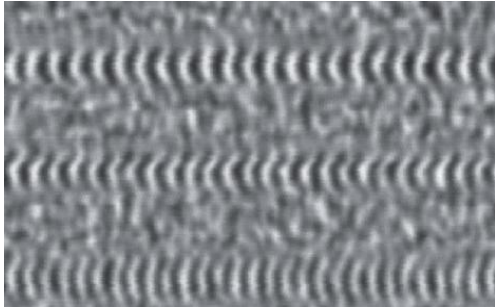
Instrumental development and maintenance

S. Le Denmat, C. Thirion, E. Wagner

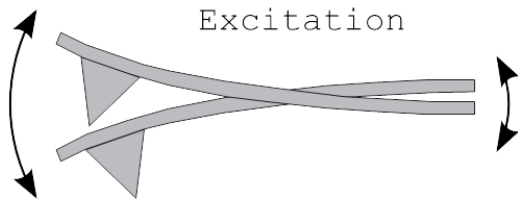


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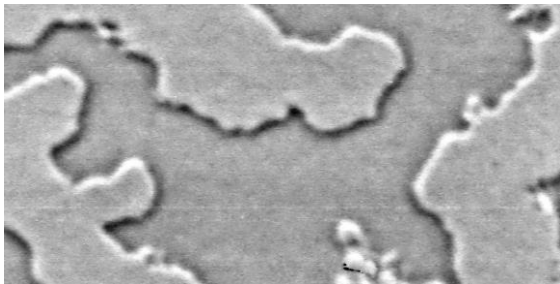
■ Motivation and criteria



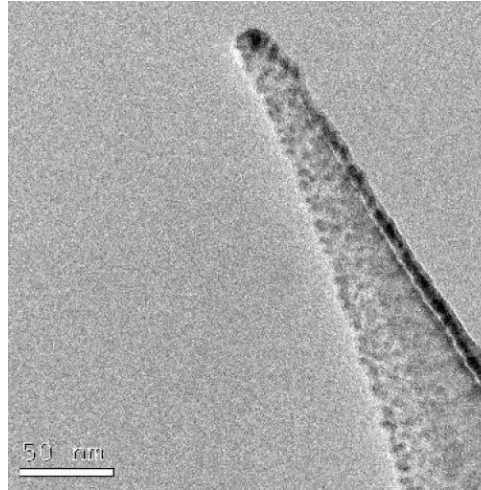
■ Working principle



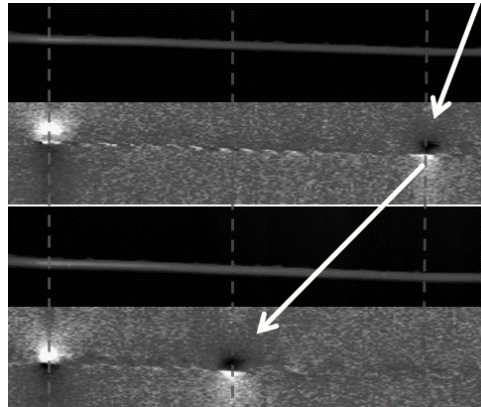
■ Image analysis



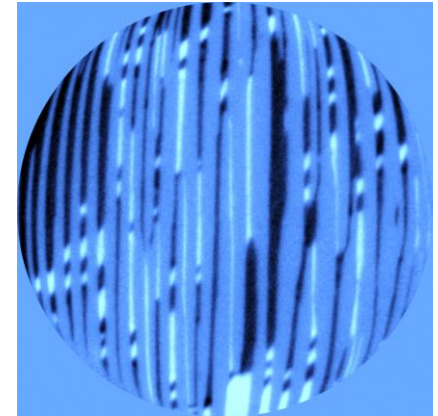
■ Choice of tips



■ Operando imaging



■ Panorama of other microscopies



Versatility

- ❑ Samples made with lithography or ex situ OK ?
- ❑ Need for sample preparation ?
- ❑ Compatible with various environments ? (temperature, field etc.)

Speed of acquisition

- ❑ Sample preparation needed ?
- ❑ How much time for one image ?

Access

- ❑ Large-scale instrument or in-lab ?
- ❑ Expensive or cheap ?

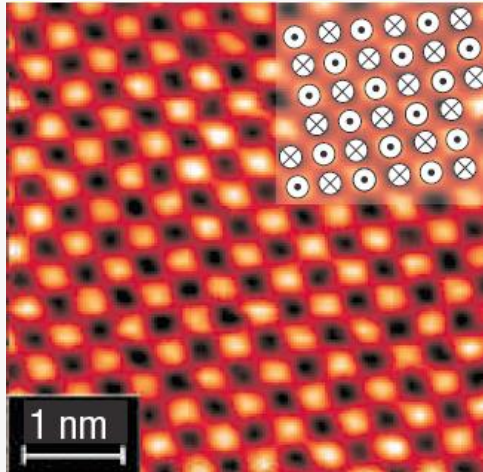
Measured quantity

- ❑ Surface or volume technique ?
- ❑ Sensitivity ?
- ❑ Magnetization, stray field, other ?

- ❑ No universal technique
- ❑ Many criteria to be balanced

Spin-polarized STM

Fe(1ML)/W(001)



Antiferromagnetic
domain

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

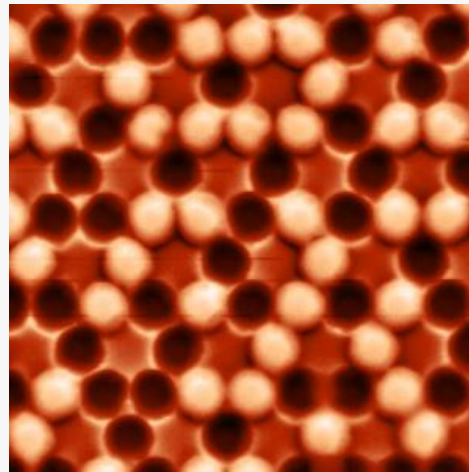
REVIEW :

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

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

Magn. Force Microscopy

Array of dots



Up-and-down
'single-domains'

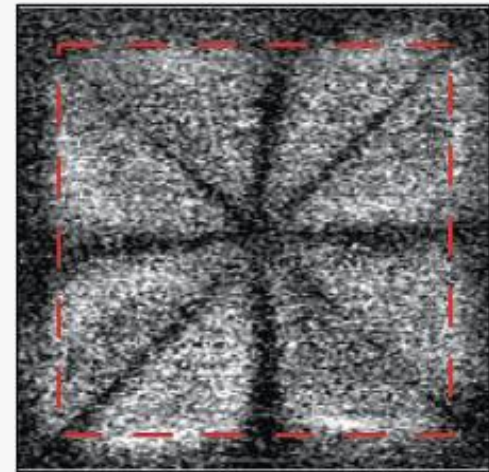
NEEL, sample courtesy:
N. Rougemaille, I. Chioar

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., Nat. Comm. 4,
2279 (2013)

Overview

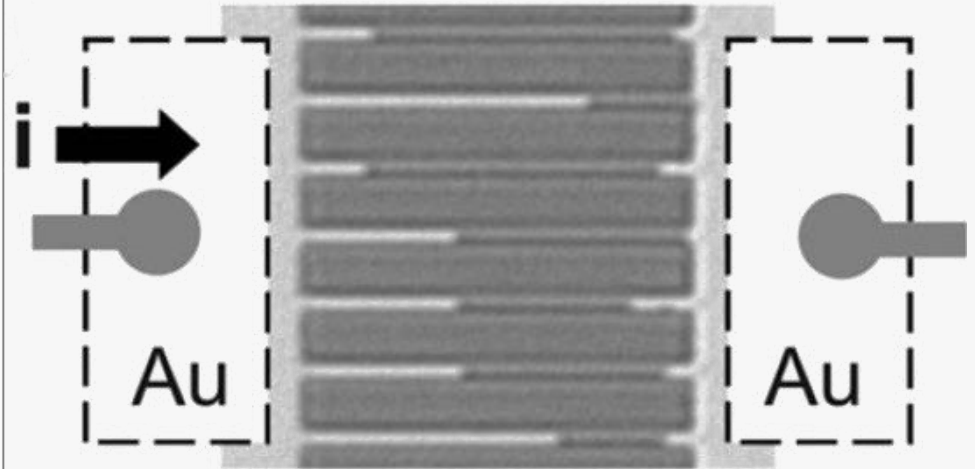
- Large variety
- Rather slow

Principle

- ❑ Polarization of light versus magnetic body
- ❑ Kerr : reflection geometry
- ❑ Faraday : transmission geometry

Example

Kerr microscopy of patterned Pt/Co/AlOx film with perpendicular magnetization



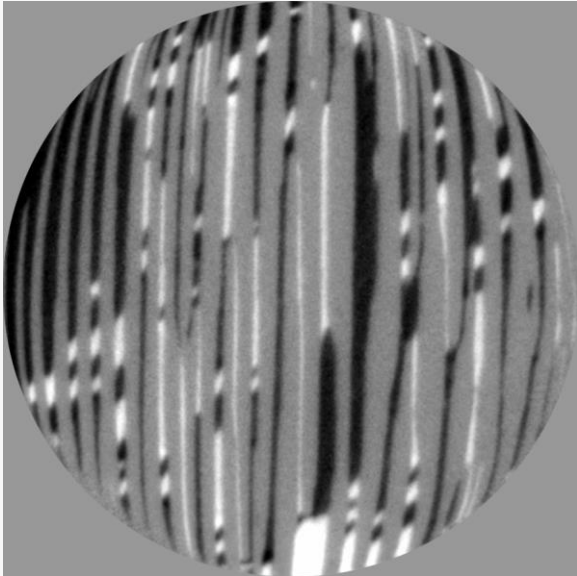
T. A. Moore et al., Appl. Phys. Lett 93, 262504 (2008)

Overview

- ❑ Quick (full field)
- ❑ Compatible with time resolution
- ❑ Limited spatial resolution

SPLEEM

Spin-Polarized Low Energy
Electron Microscopy



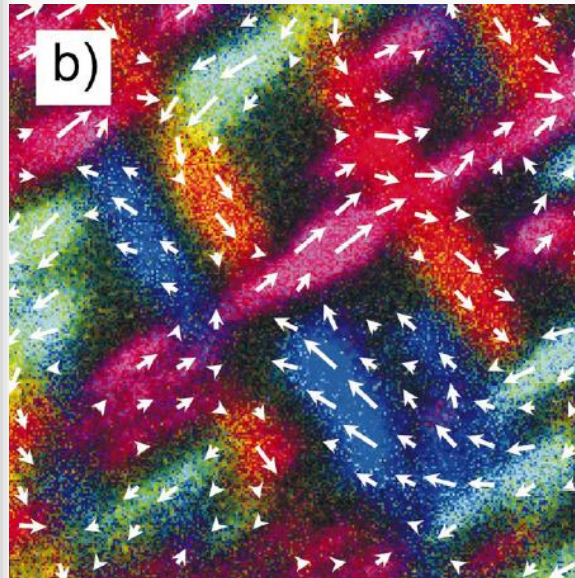
Stripes of Fe/W(110)

REVIEW:

N. Rougemaille et al., Eur.
Phys. J. Appl. Phys. 50, 20101
(2010)

SEMPA

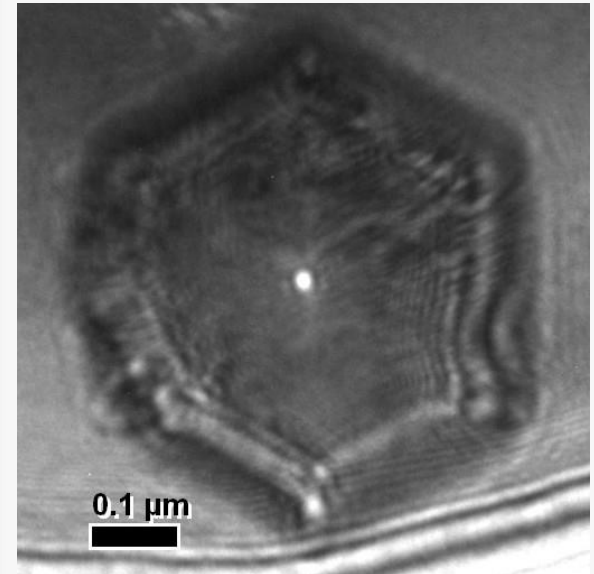
Scanning Electron Microsc.
with Polarization Analysis



Maze of Fe/W(001) 1.5 μm

W. Wulfhekel et al.,
Phys. Rev. B 68,
144416/1-9 (2003)

Lorentz, holography etc.
TEM - based



Self-assembled Co/W(110)

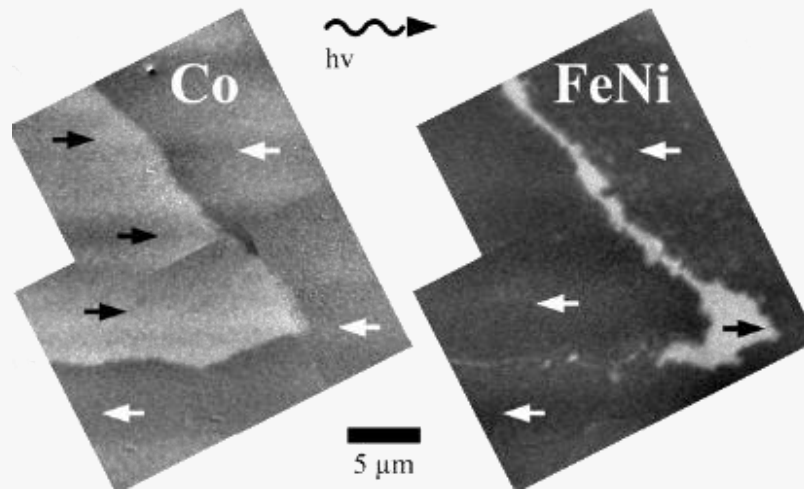


O. Fruchart et al., J. Phys.
Condens. Matter 25, 496002
(2013)

- ❑ Requires sample preparation
- ❑ Good spatial resolution
- ❑ Some information about structure

XMCD-PEEM

X-ray Magnetic Circular Dichroism
Photo-Emission Electron Microsc.



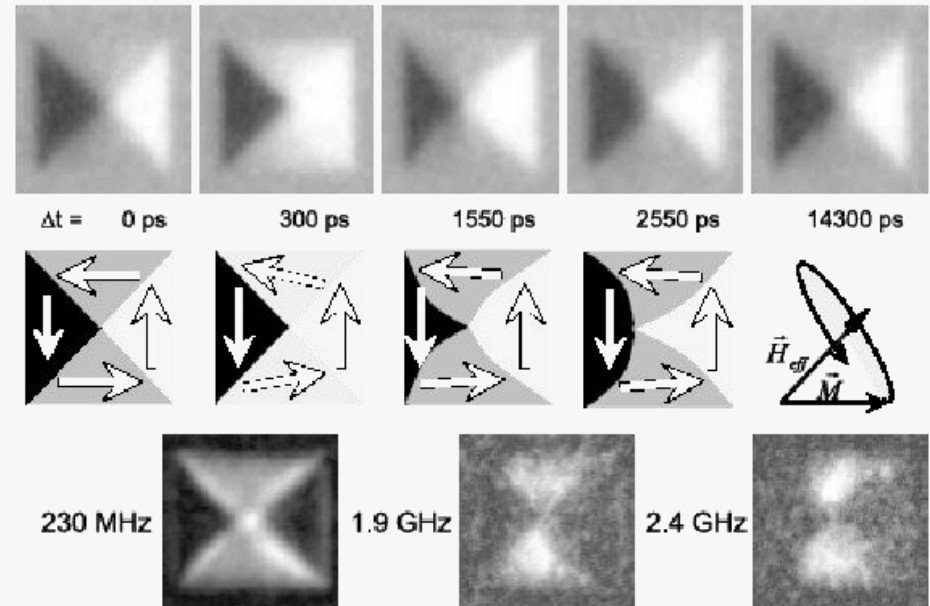
Co\Cu\FeNi trilayer
→ elemental resolution

J. Vogel et al., J. Phys. : Condens. Matter 19, 476204 (2007)

Others : holography, scattering

TXM

Transmission X-ray Microscopy



FeNi 6μm square dot → time resolution

J. Raabe et al., Phys. Rev. Lett. 94, 217204 (2005)

- Elemental sensitivity
- Compatible with time resolution
- Rather versatile

Alex Hubert
Rudolf Schäfer

Magnetic Domains

The Analysis
of Magnetic Microstructures

 Springer

Alberto P. Guimarães

NANOSCIENCE AND TECHNOLOGY

Principles of Nanomagnetism

 Springer

Simple Models of Magnetism

Ralph Skomski

OXFORD GRADUATE TEXTS

Thank you for your attention !

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<http://fruchart.eu/slides>