



An overview on ...

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Measurements using magnetic force microscopy

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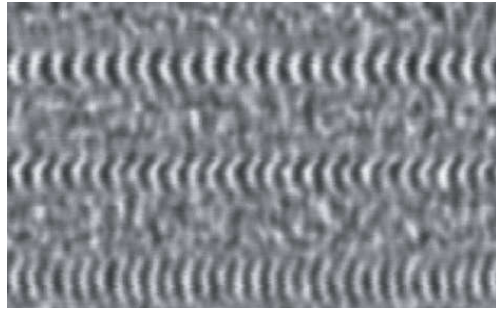
LOCATIONS IN GRENOBLE



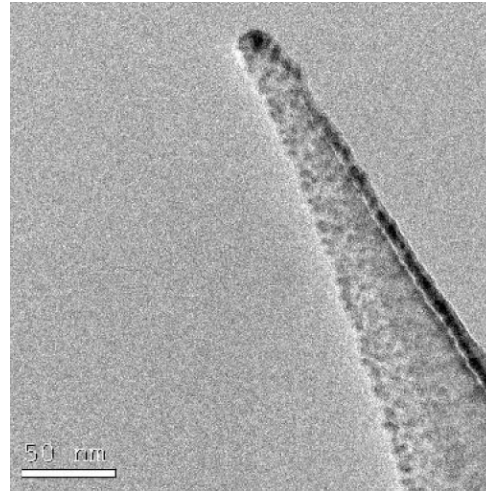


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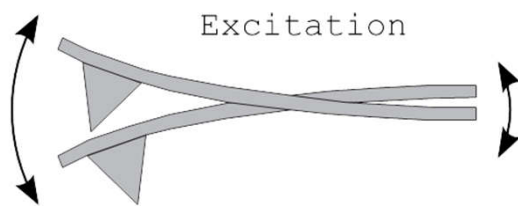
- Motivation and criteria



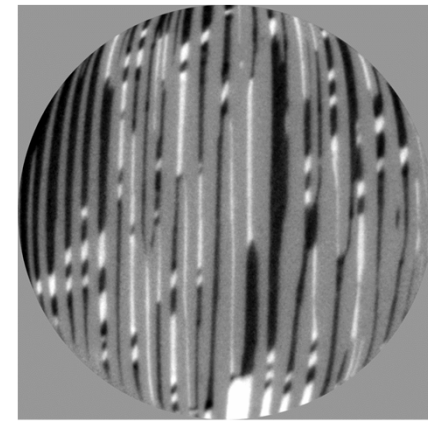
- Choice of tips



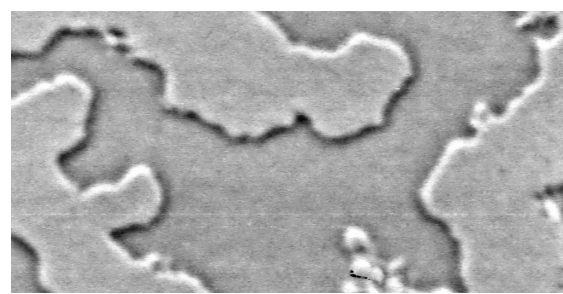
- Working principle



- Panorama of other microscopies



- Image analysis



- Operando imaging

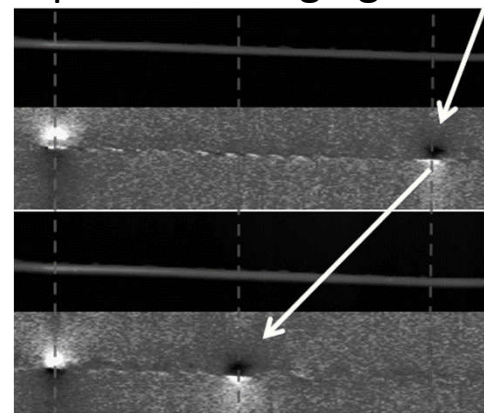
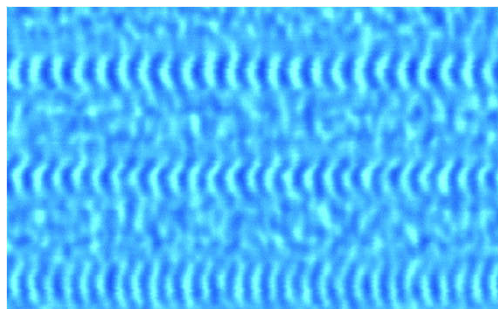


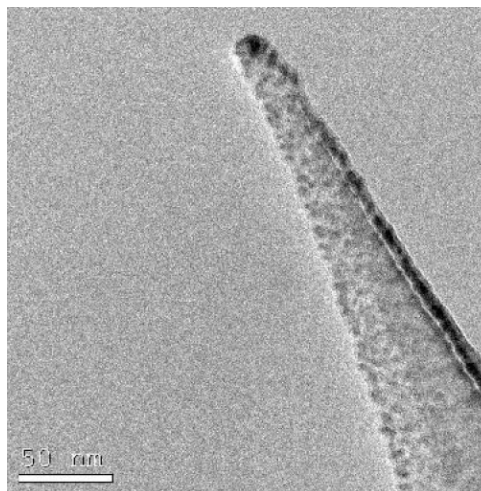


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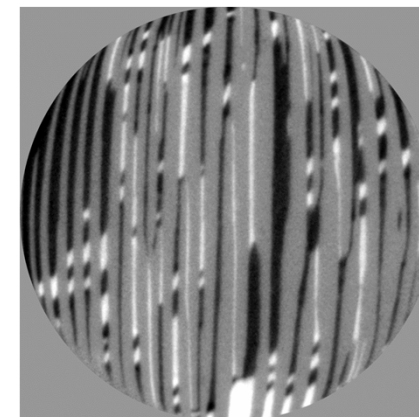
■ Motivation and criteria



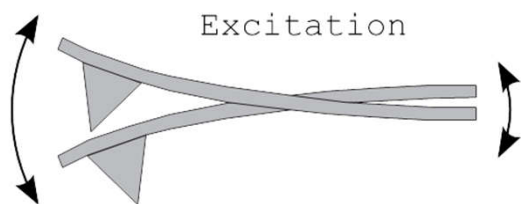
■ Choice of tips



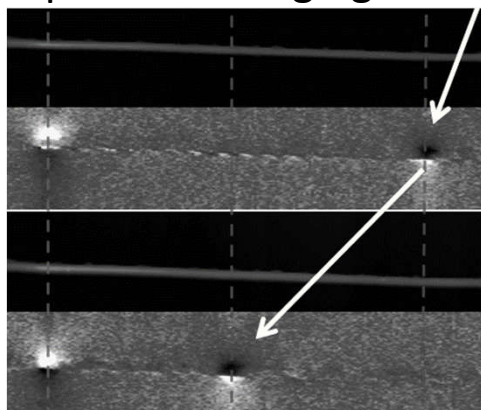
■ Panorama of other microscopies



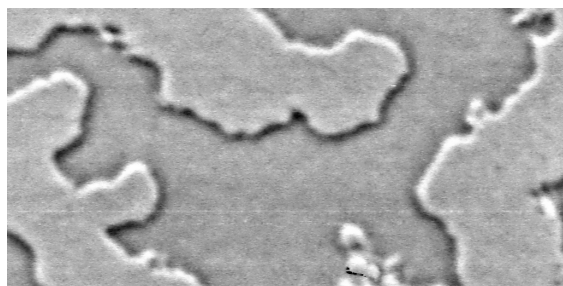
■ Working principle



■ Operando imaging



■ Image analysis



Magnetic domains

Numerous and complex magnetic domains



(History : Weiss domains)

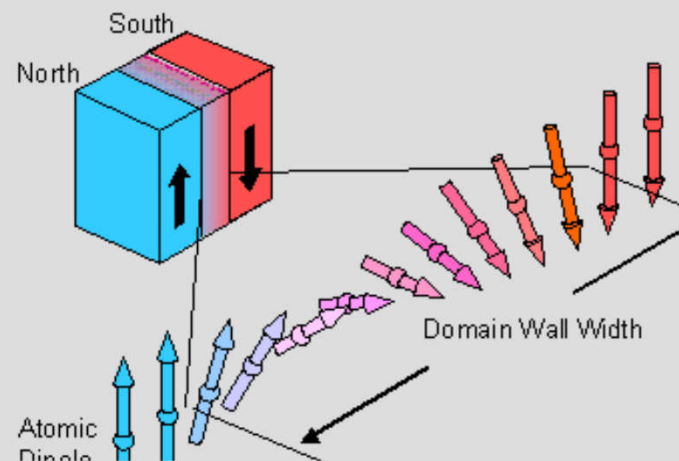
Anisotropy exchange length (domain wall width)

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

Exchange \rightarrow J/m Anisotropy \rightarrow J/m³

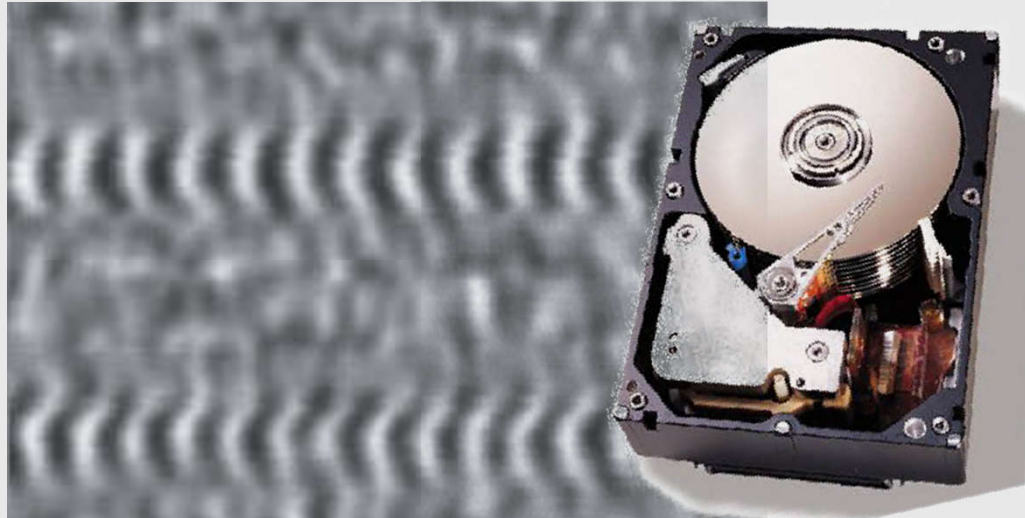
Anisotropy exchange length: $\Delta_u = \sqrt{A/K}$

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



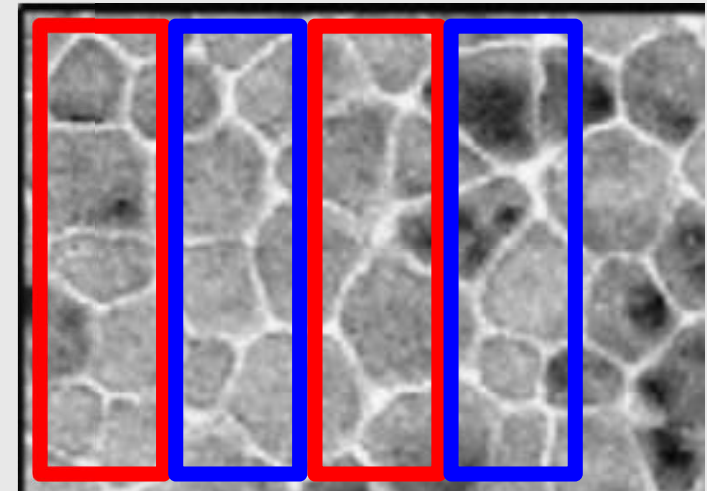
Magnetic bits on Hard Disk Drive

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



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

Underlying microstructure



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

Relevant spatial resolution

↻ 10-100nm

Relevant time resolution

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

Basics of precessional switching

Magnetization dynamics:

Landau-Lifshitz-Gilbert equation:

$$\frac{dM}{dt} = \gamma_0 [M \times H_{\text{eff}}] + \frac{\alpha}{M_s} \left[M \times \frac{dM}{dt} \right]$$

Gyromagnetic factor

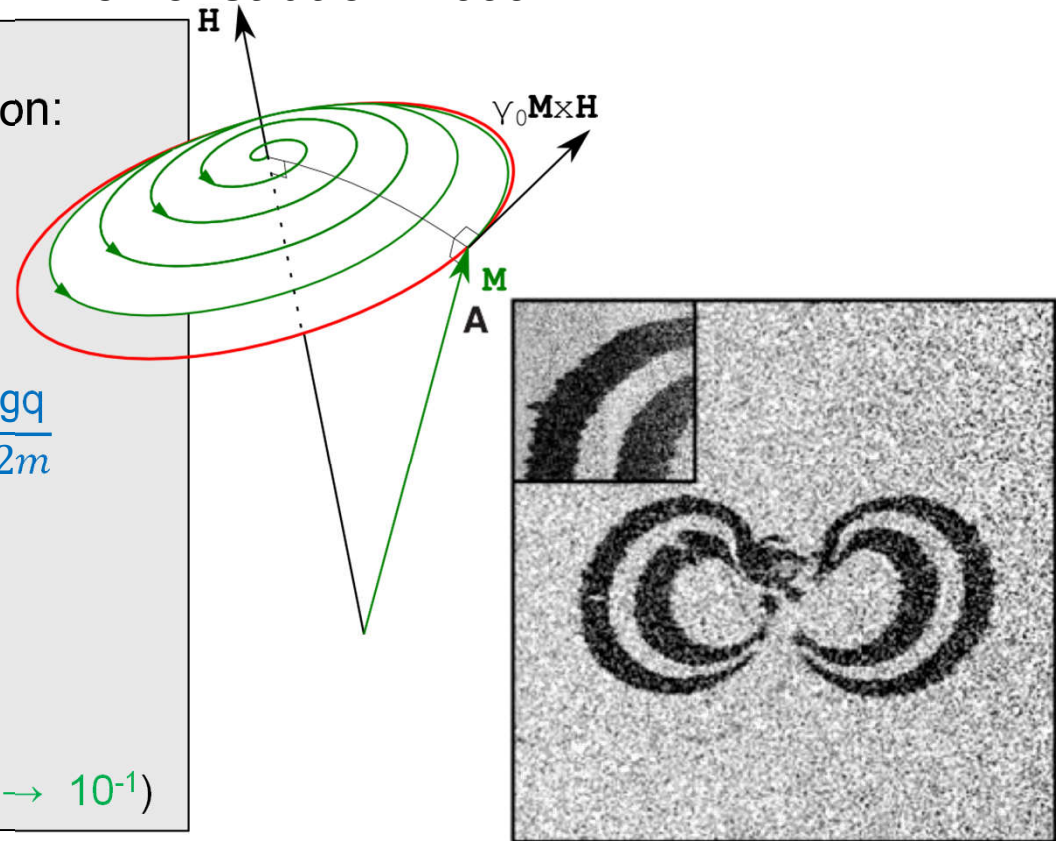
$$\gamma_0 = \mu_0 \gamma \quad \gamma = \frac{gq}{2m}$$

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

H_{eff} Effective field
(including applied)

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

Demonstration: 1999



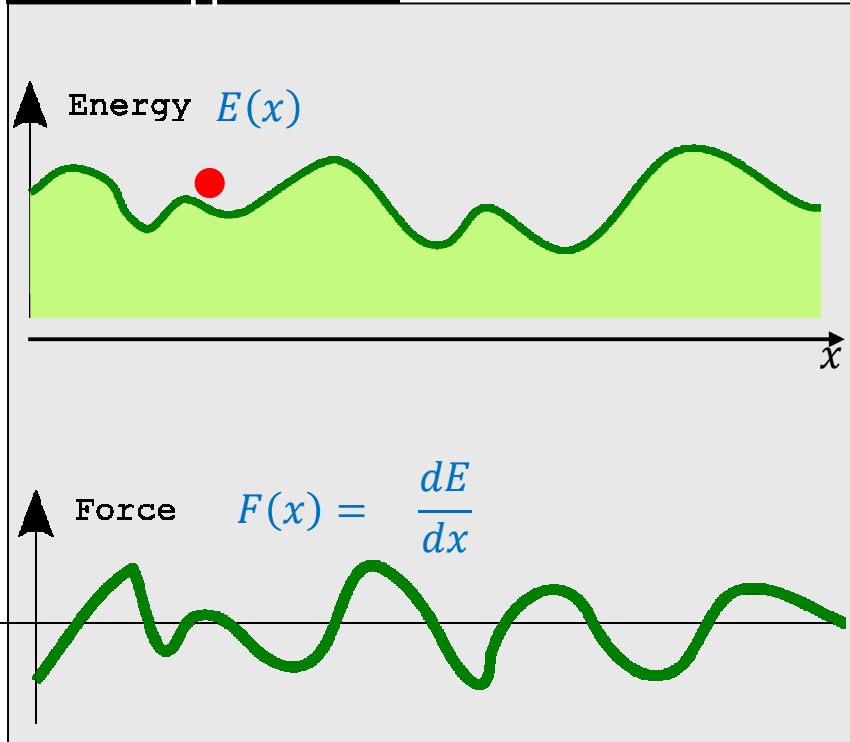
C. Back et al., Science 285, 864 (1999)

150 μm

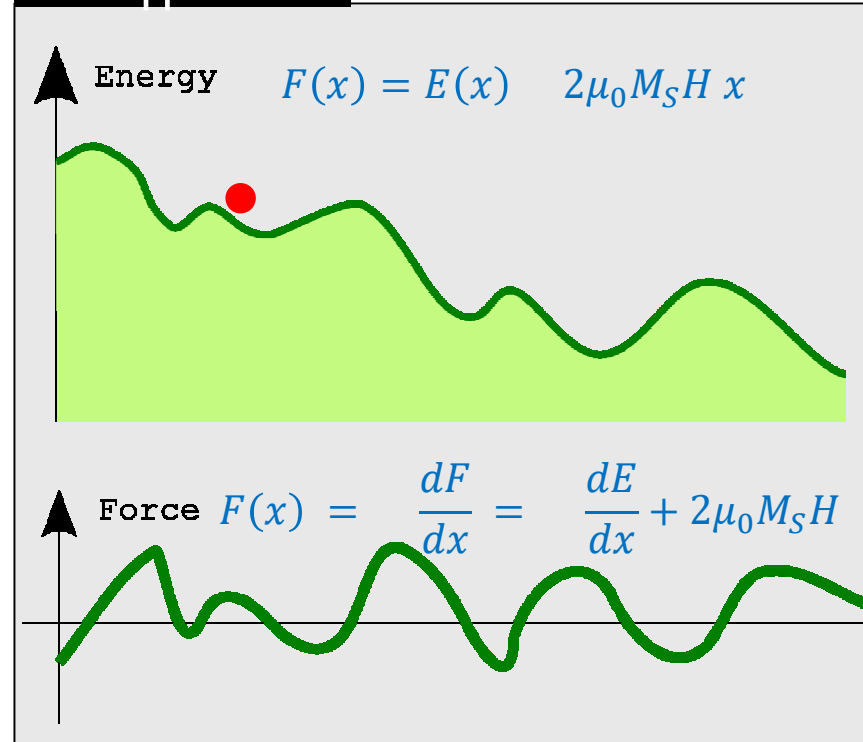
MOTIVATION / Link with structure

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 ?

What is probed

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

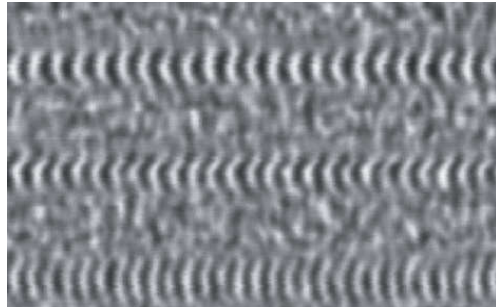
Conclusion

No universal technique

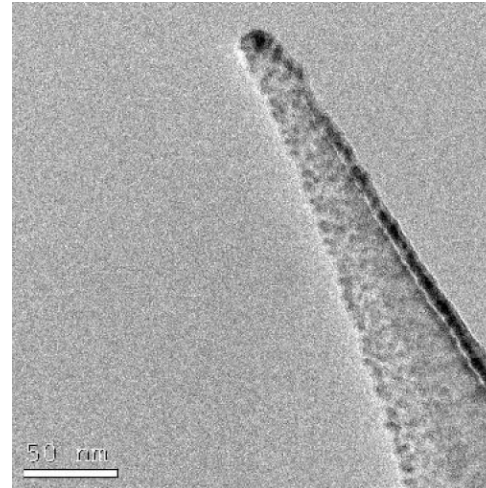
Many criteria to be balanced

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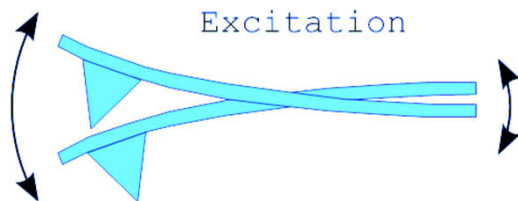
- Motivation and criteria



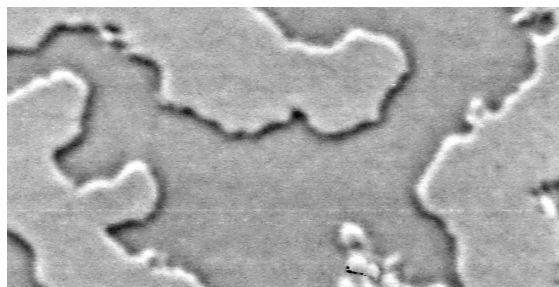
- Choice of tips



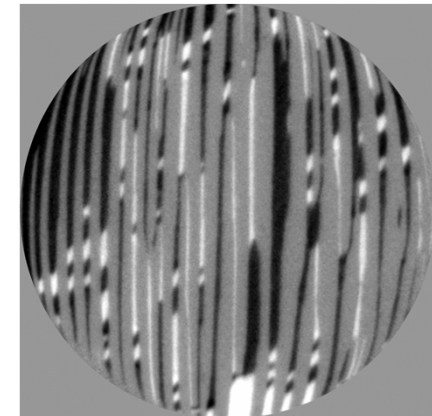
- Working principle



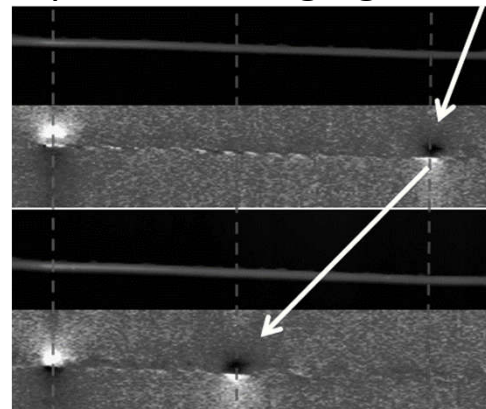
- Image analysis



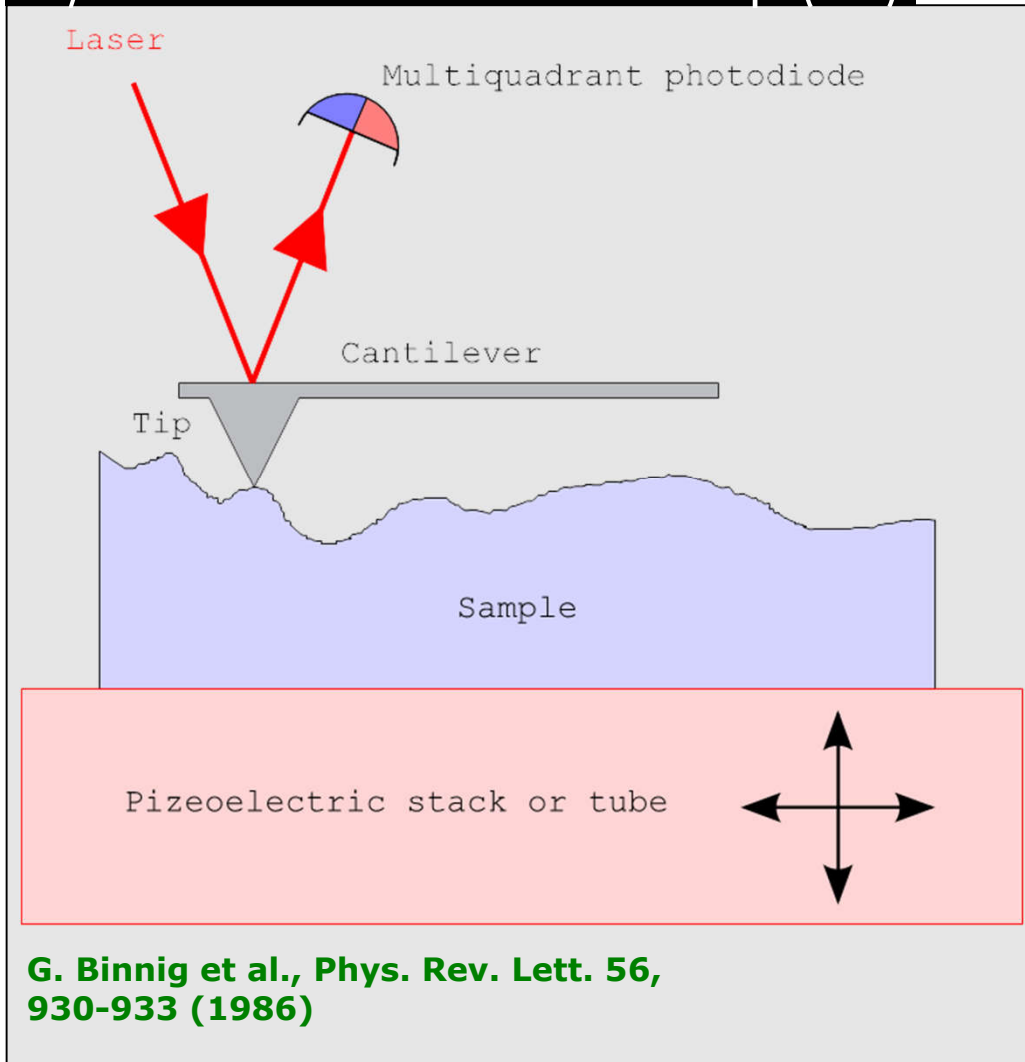
- Panorama of other microscopies



- Operando imaging



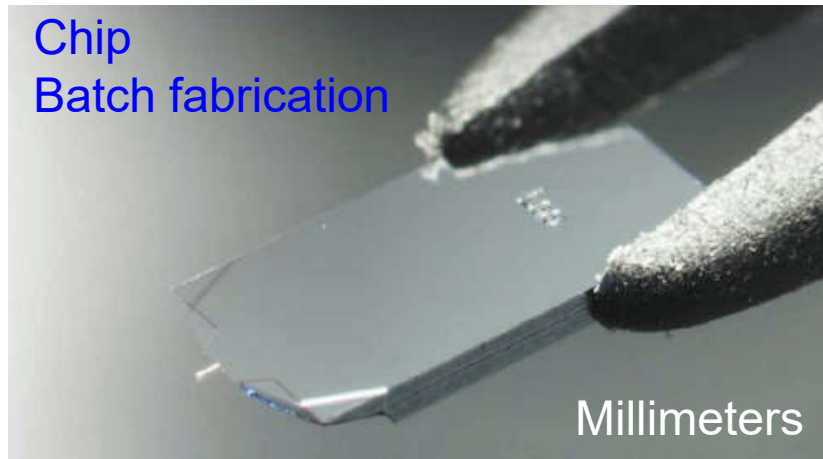
Key elements of an Atomic Force Microscope (AFM)



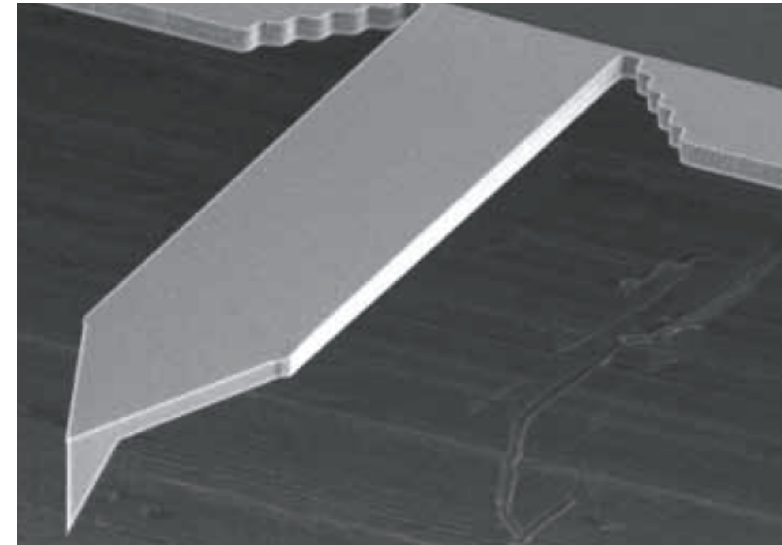
Overview

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

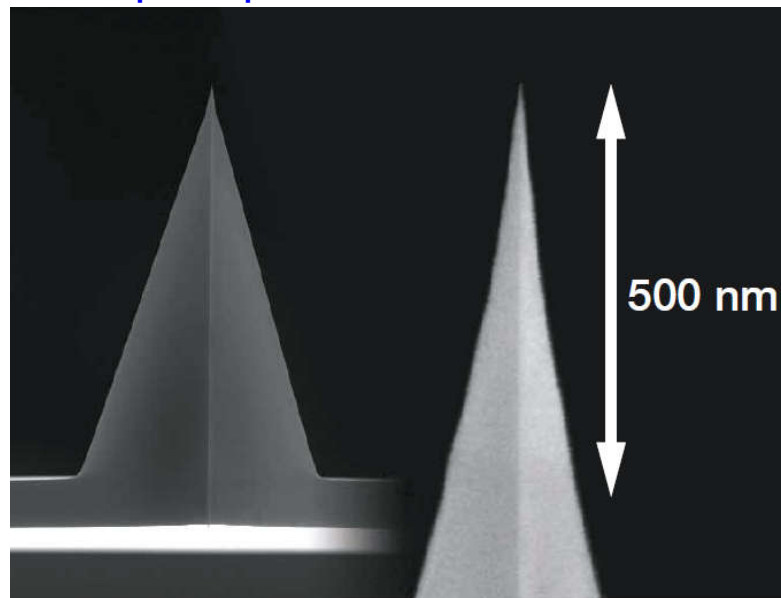
Chip
Batch fabrication



Cantilever



Full tip + apex



Overview

↪ Price 10-200eur/tip

↪ Radius of curvature ≈ 5 nm

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

Mechanical excitation of cantilevers

At rest

$z=0$

$h=0$

Excitation

$z_{0,m} \cos(\omega t)$

$h = h_m \cos(\omega t)$

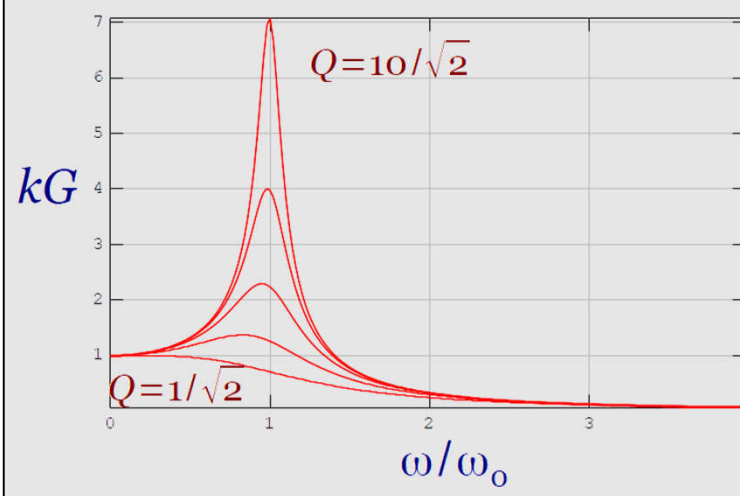
$$m \ddot{z} + \Gamma \dot{z} + k z = F(z, t)$$

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

Notations

Seek solutions for	$F=0$	$z(t) = z_0 e^{j\omega t}$	\Rightarrow Transfert function
Reference angular velocity		$\omega_0 = \sqrt{\frac{k}{m}}$	$H = \frac{z}{F} = \frac{1}{k} \frac{1}{-\left(\frac{\omega}{\omega_0}\right)^2 + \frac{j}{Q} \left(\frac{\omega}{\omega_0}\right) + 1}$
Quality factor		$Q = \frac{\sqrt{k m}}{\Gamma}$	

Gain

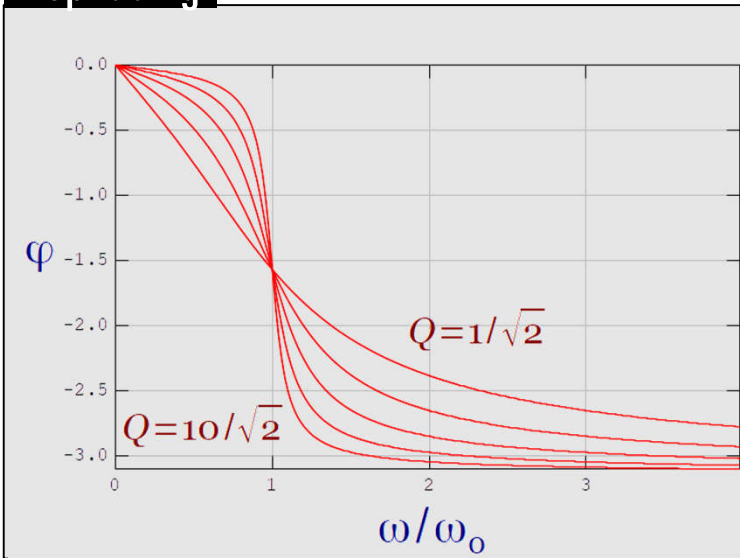


$$G = |H| = \frac{1}{k} \frac{1}{\sqrt{\left[1 - \left(\frac{\omega}{\omega_0}\right)^2\right]^2 + \frac{1}{Q^2} \left(\frac{\omega}{\omega_0}\right)^2}}$$

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



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

$$\begin{cases} \varphi(0) = 0 \\ \varphi(\infty) = -\pi \end{cases}$$

$Q \gg 1$

$$\begin{cases} \omega_r \approx \omega_0 \\ \varphi(\omega_r) \approx -\pi/2 \\ \frac{\Delta \omega_r}{\omega_0} \approx \frac{\sqrt{3}}{Q} \end{cases}$$

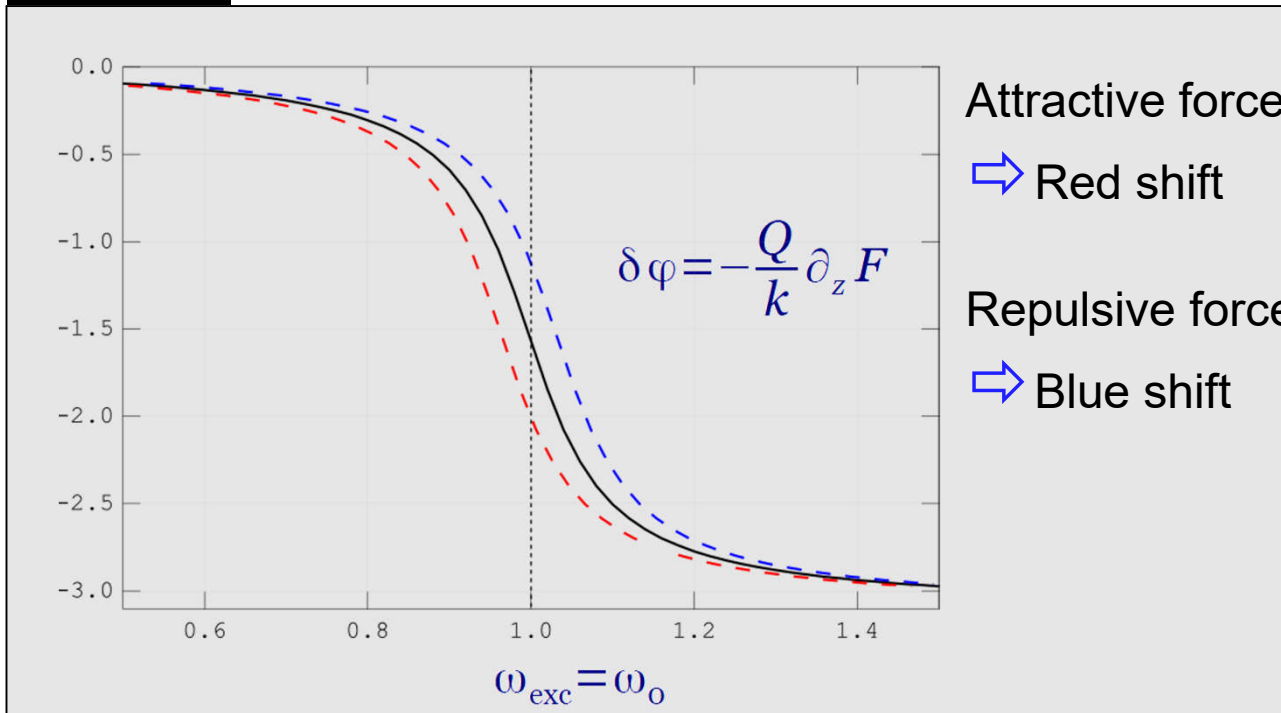
MFM / Detecting forces on the phase shift

Tip-sample interaction treated as perturbation

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

⇒ 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

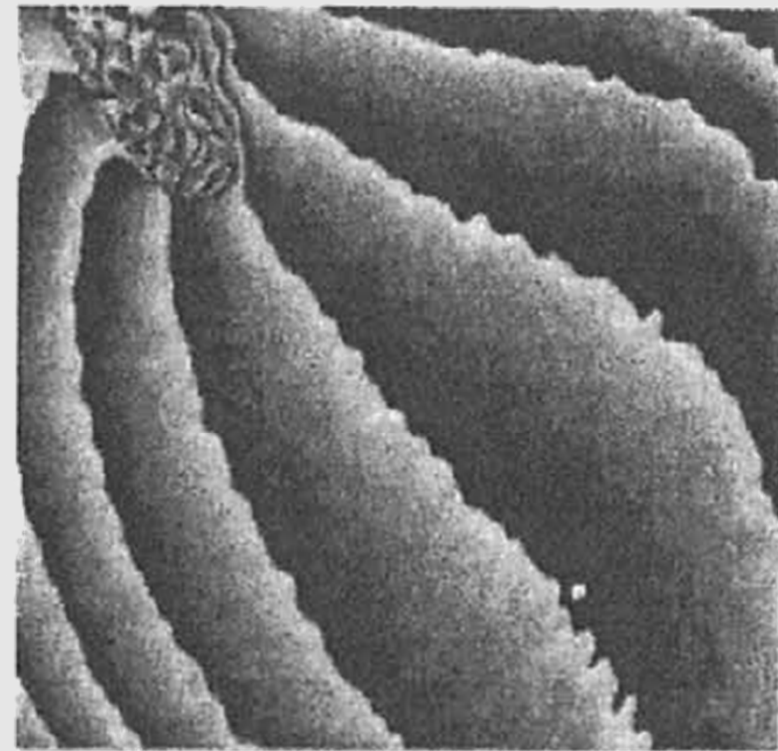
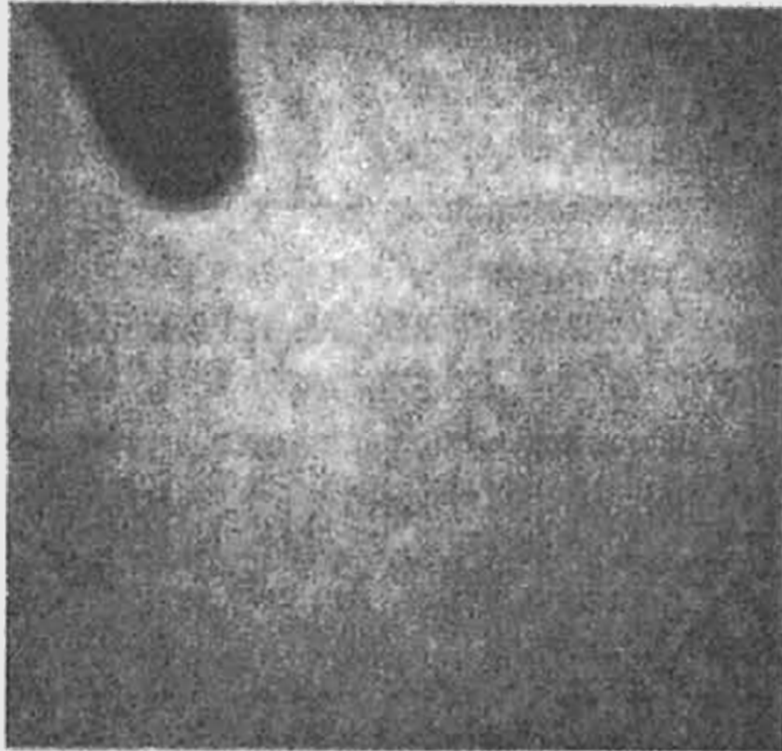
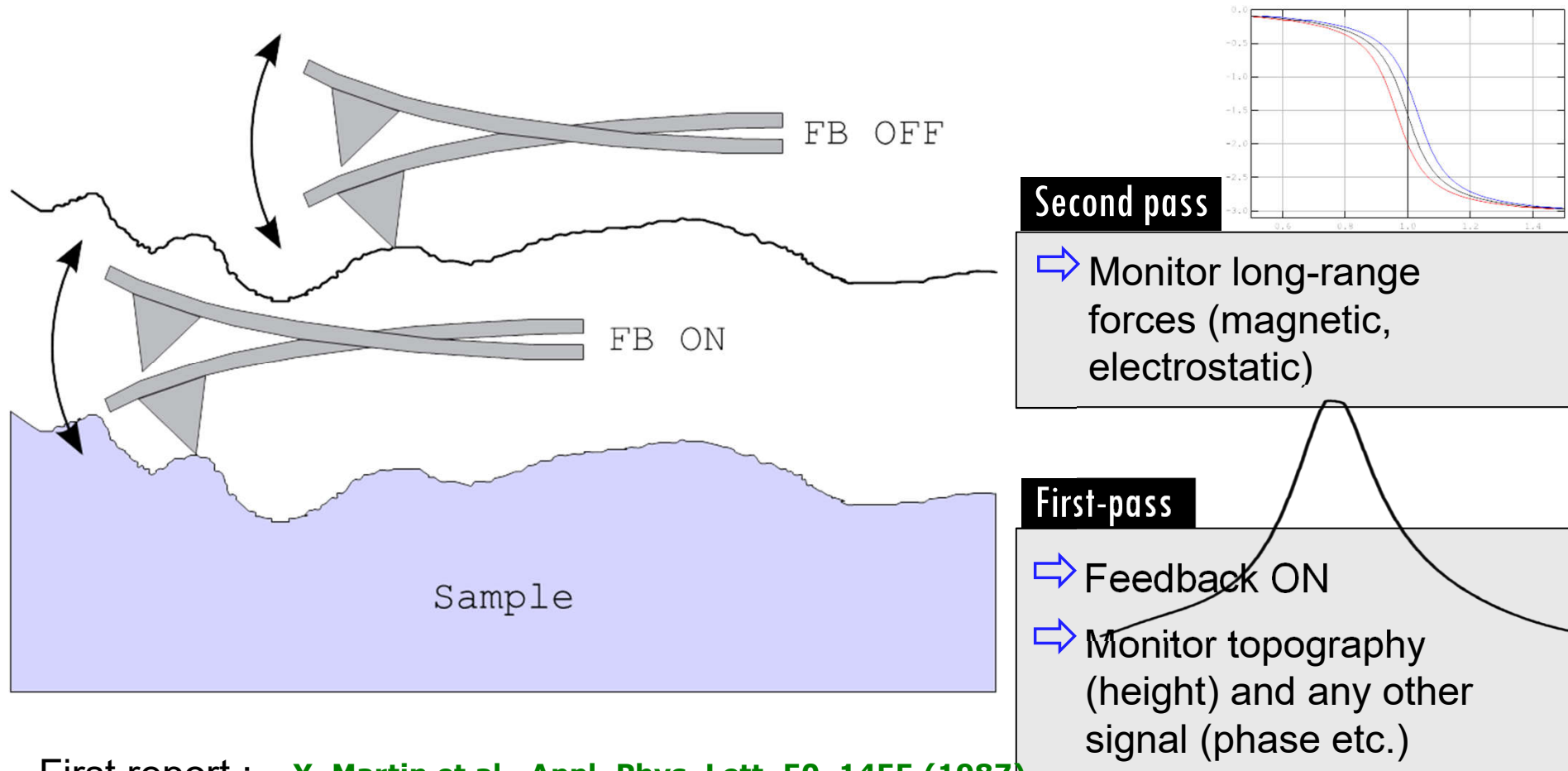


Figure 11-20: The electron amplitude (left) and phase (right) near an MFM tip visible as a dark shadow on the upper left corner of the left image.

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

MFM / Two-pass technique



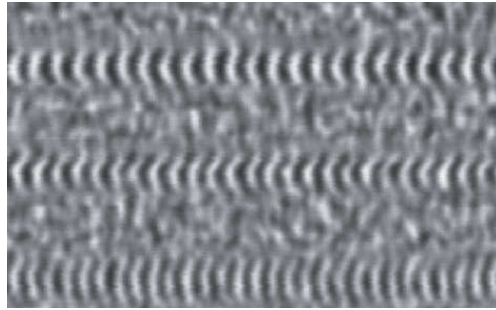
First report : **Y. Martin et al., Appl. Phys. Lett. 50, 1455 (1987)**

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

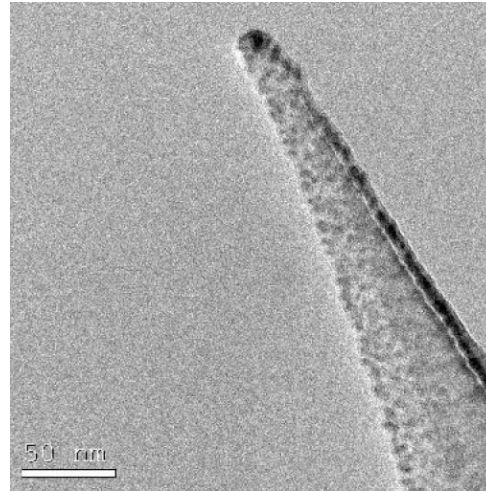


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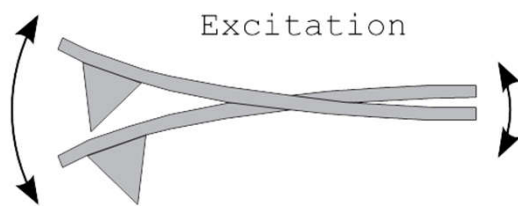
- ❑ Motivation and criteria



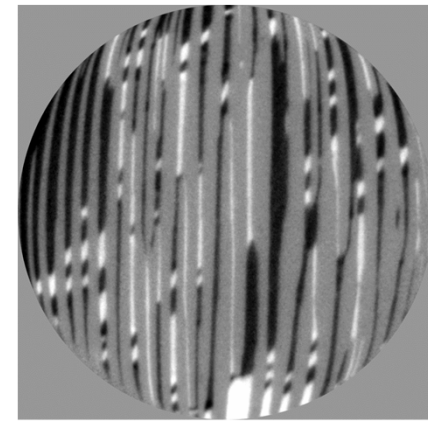
- ❑ Choice of tips



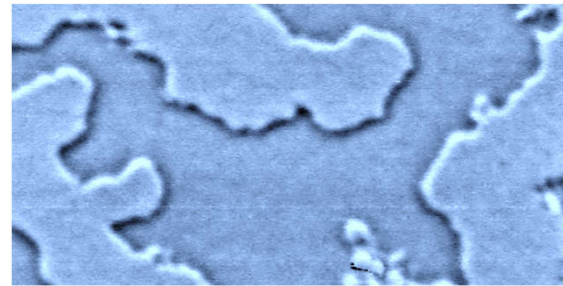
- ❑ Working principle



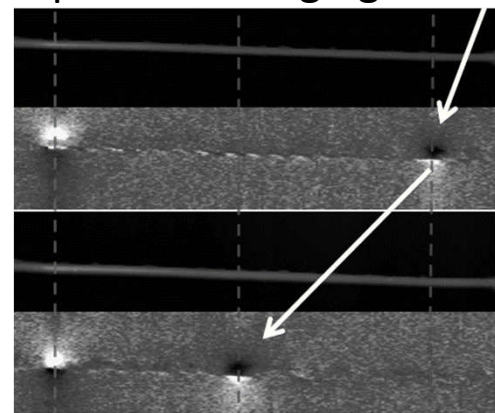
- ❑ Panorama of other microscopies



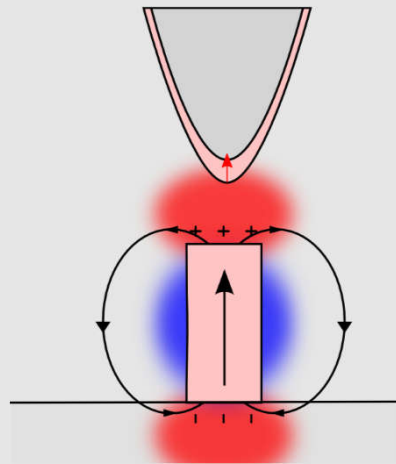
- ❑ Image analysis



- ❑ Operando imaging



Tip is a dipole

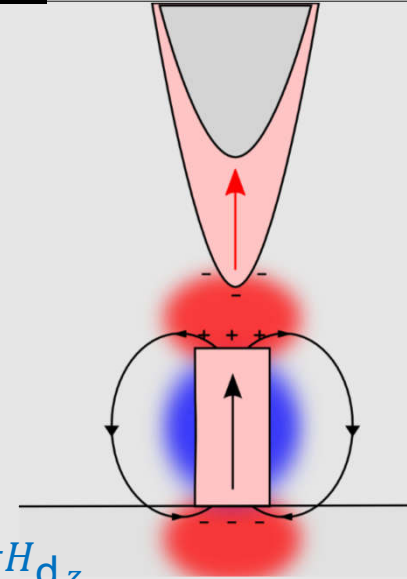


$$E_{1,2} = \mu_0 \mu_2 \cdot H_d$$

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

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

Tip is a monopole



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

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

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

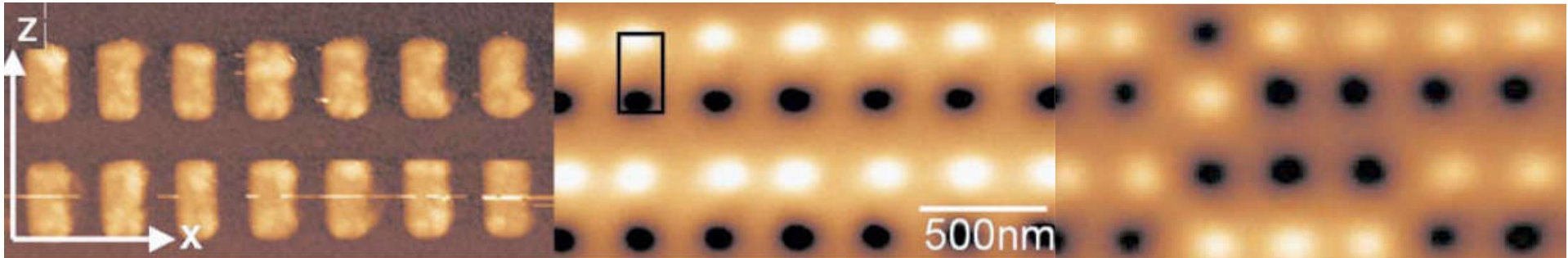
- ↪ In practice, a combination of both models is best suited (dipole is more important)
- ↪ MFM is sensitive to some derivative(s) of the stray field from the sample
- ↪ MFM may be sensitive to in-plane field, depending on the tip magnetic moment

PRACTICE / Single-domain (planar)

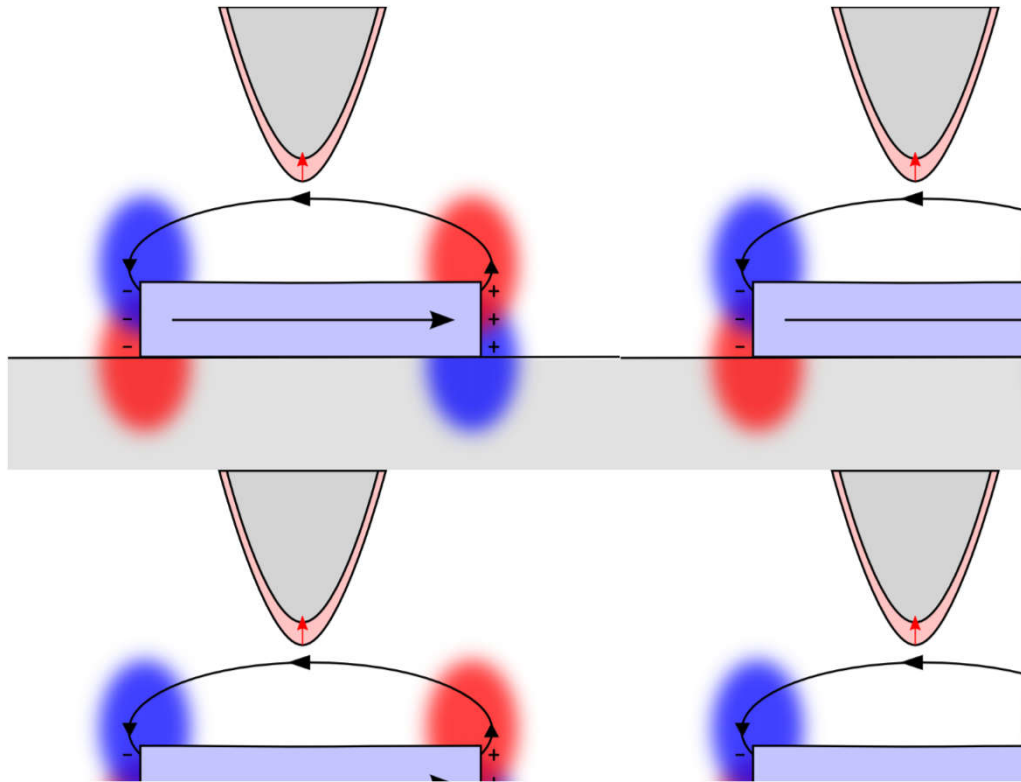
Topography

MFM, saturated

MFM, partly reversed

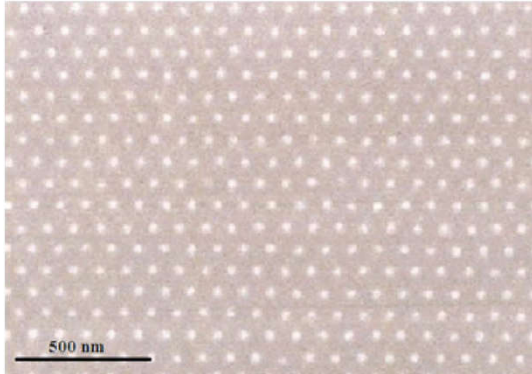


S. Y. Suck et al., APL95, 162503 (2009)

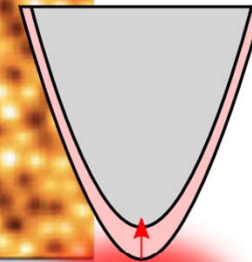
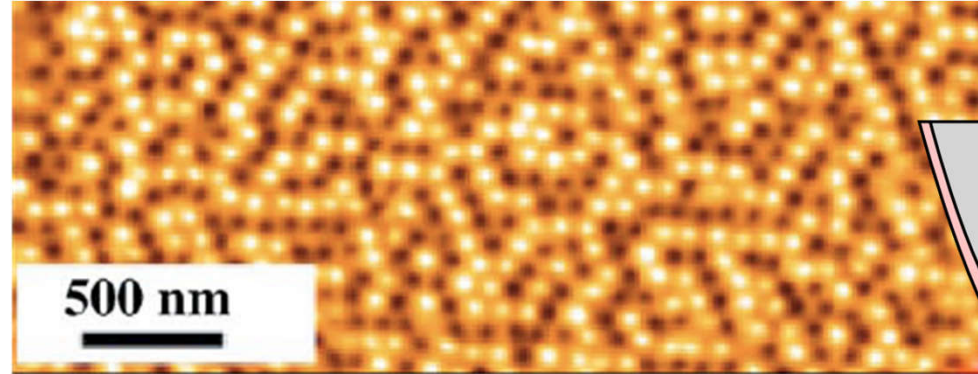


↪ Single-domain in-plane magnetized dots appear as dipoles

Structure (SEM)

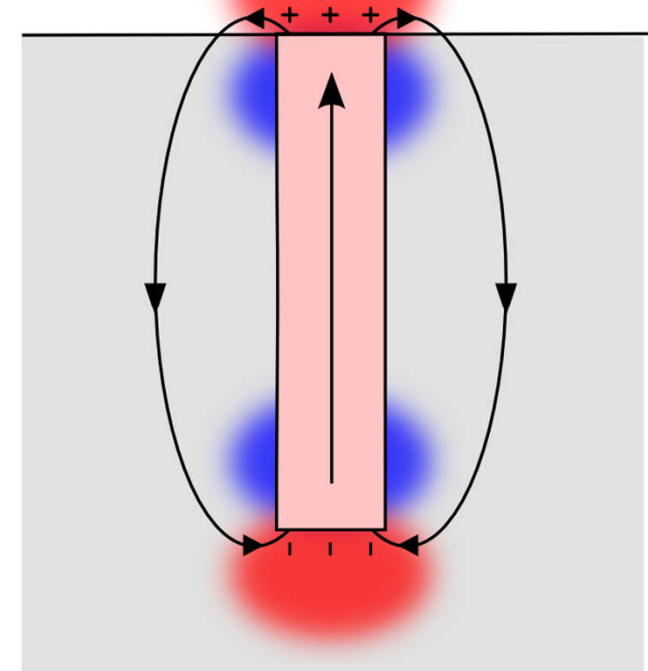


MFM, partly reversed

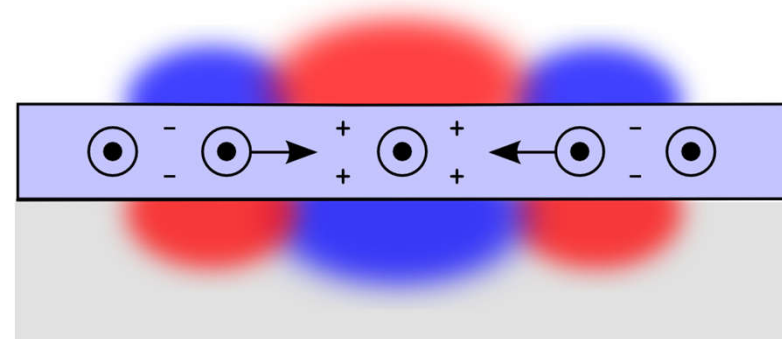
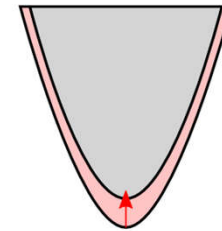
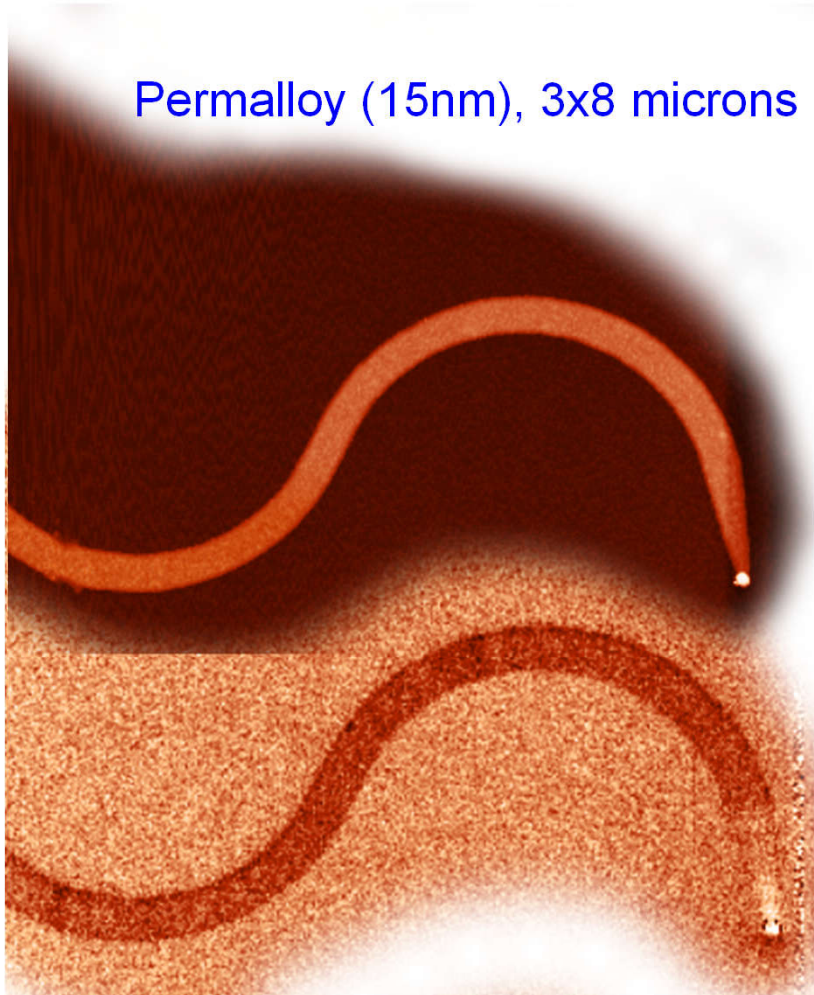


T. Wang et al., APL 92, 192504 (2008)

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



Permalloy (15nm), 3x8 microns



Principle :

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

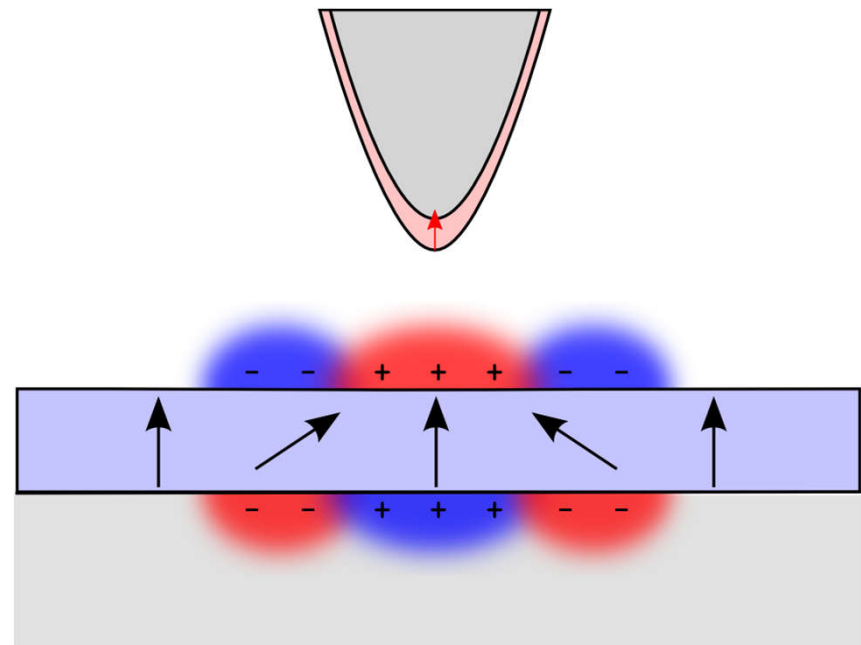
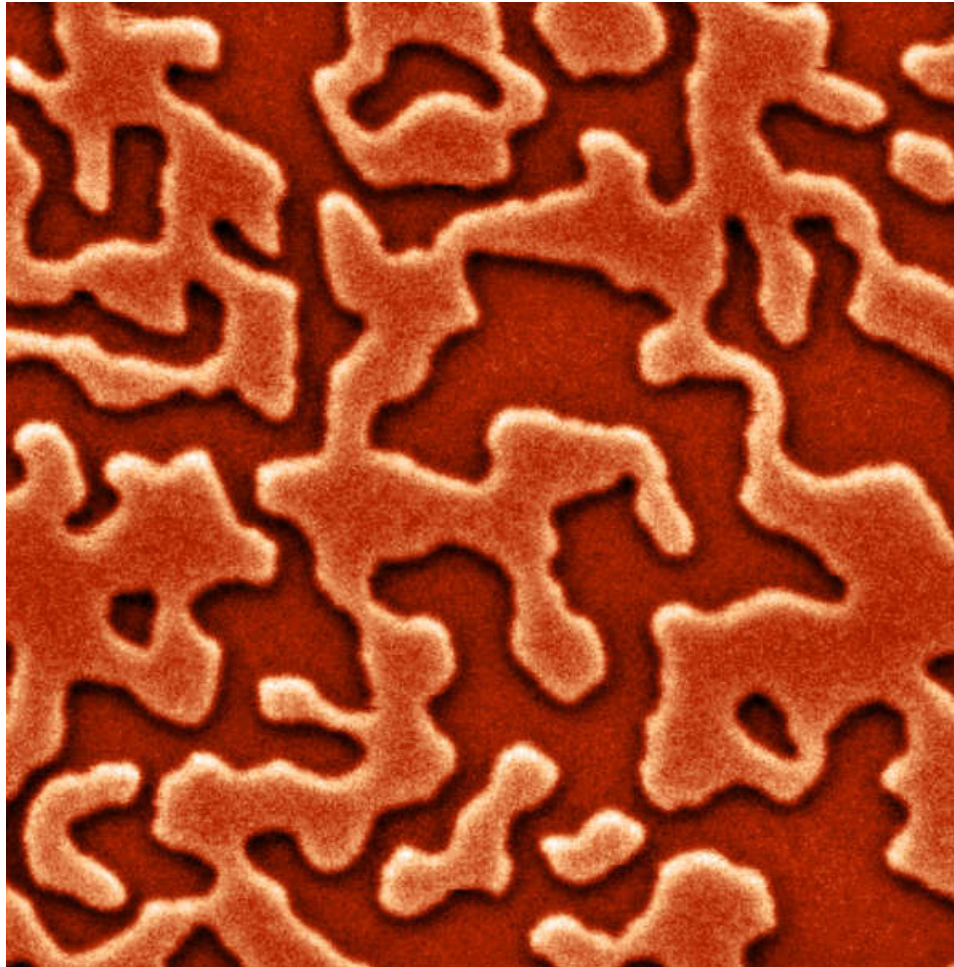
- \curvearrowright It is a DOMAIN contrast
- \curvearrowright Interaction is ALWAYS attractive : red shift
- \curvearrowright Contrast is proportionnal to the square of the tip moment

Contrast : -0.1° , LM tip

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

FePt (4nm)

5x5 μm



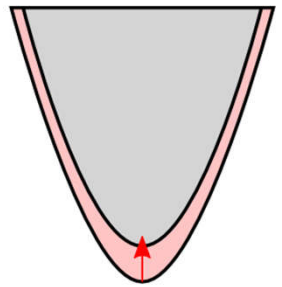
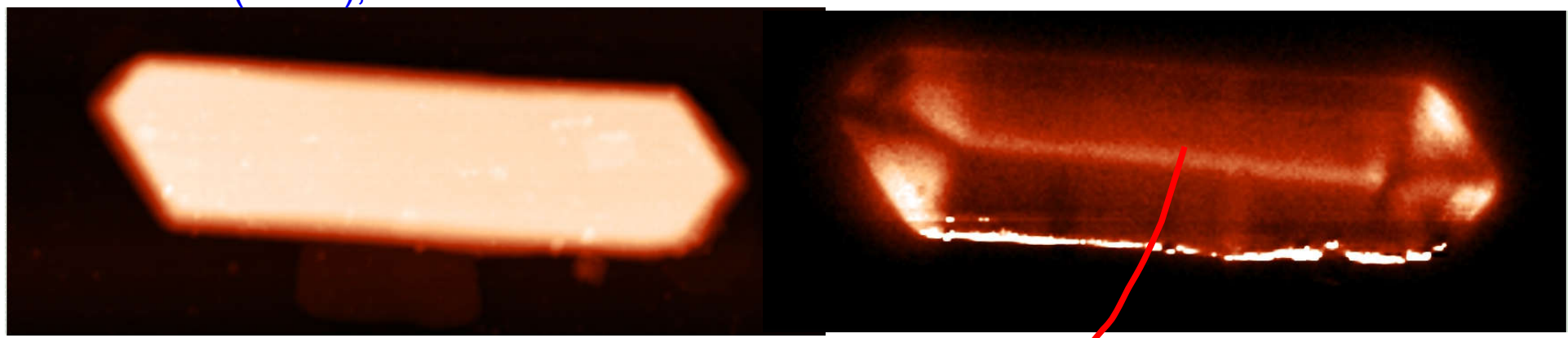
↪ It is a DOMAIN contrast
↪ The direction of magnetization is deduced

Sample : A. Marty (CEA-Grenoble)
Imaging : M. Darques (Institut Néel)
Contrast : $\pm 0.4^\circ$, LM tip

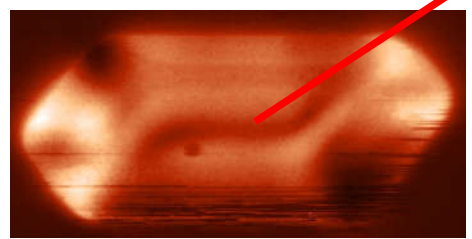
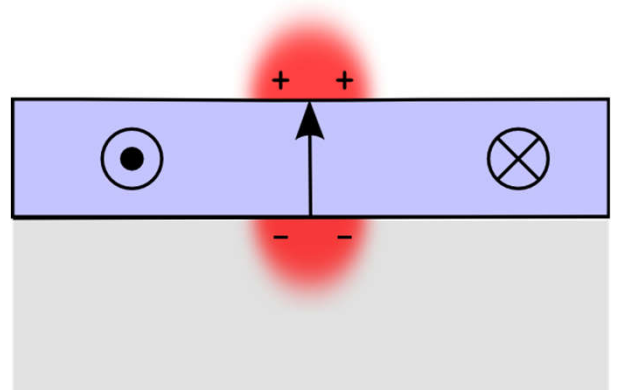
Quantitative analysis :
L. Belliard et al., J. Appl. Phys. 81,
3849 (1997)

PRACTICE / Imaging domain walls (Bloch)

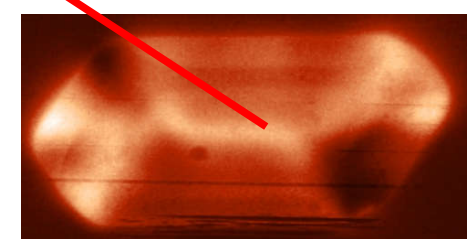
Fe dot (25nm), 2.5x1 microns



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



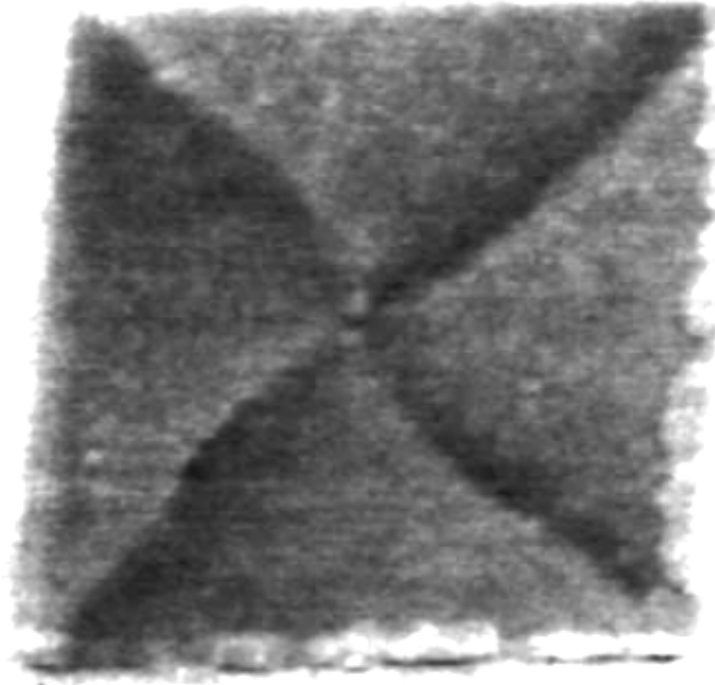
Down core



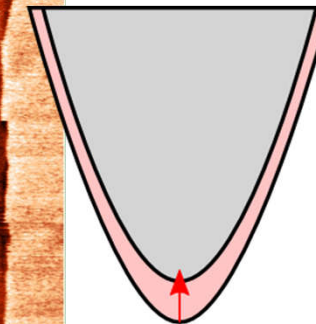
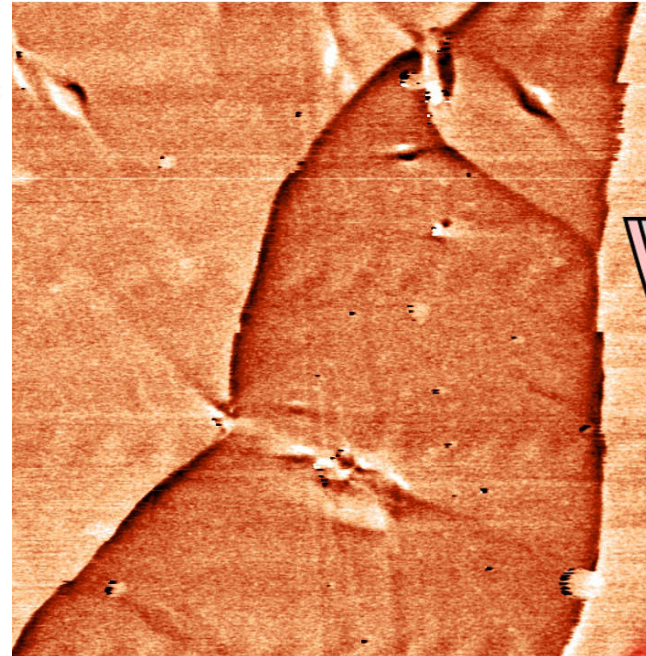
Up core

PRACTICE / Imaging domain walls (Néel)

Permalloy dot (16nm)
2x2 microns

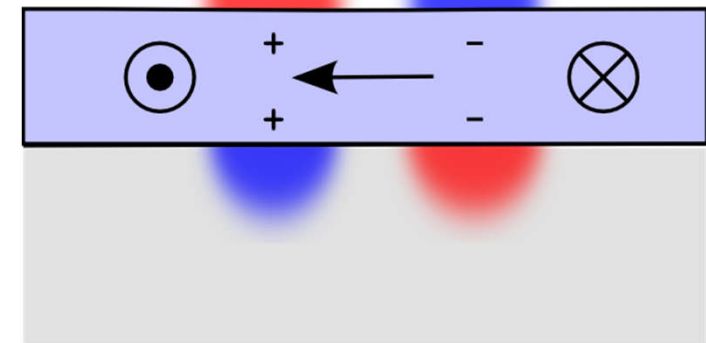


Permalloy film (20nm)
10x10 microns



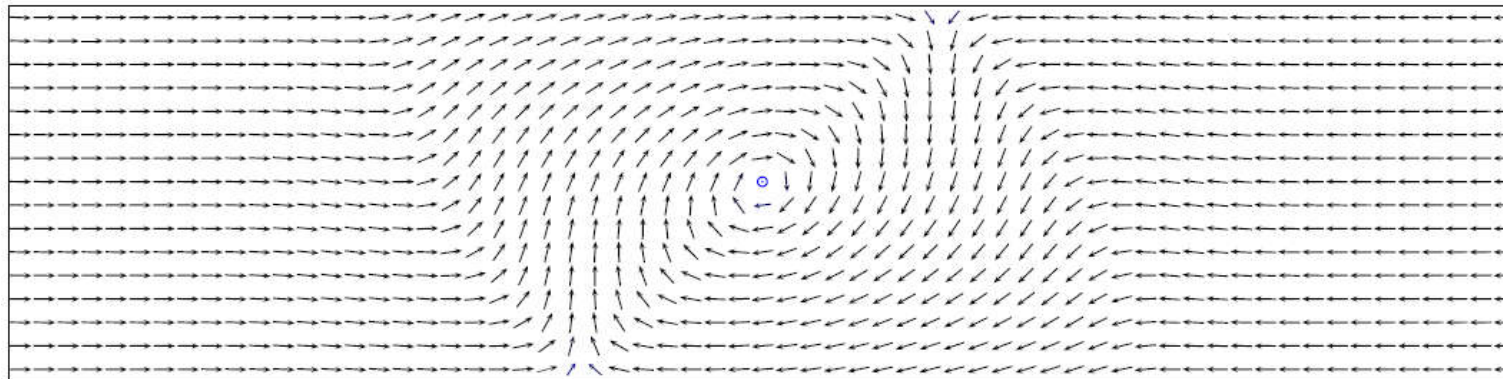
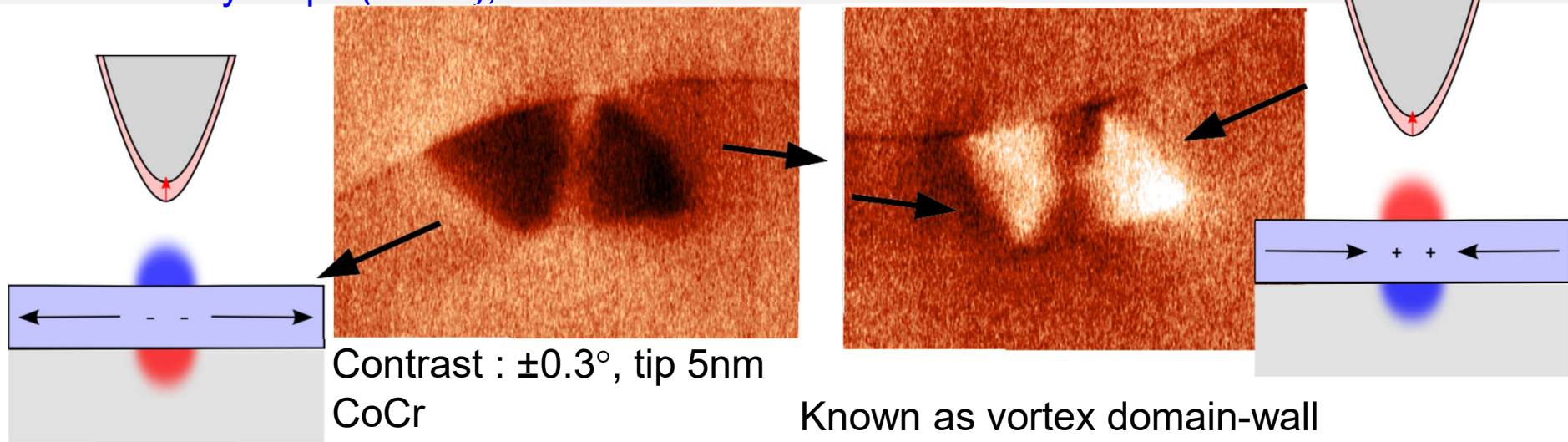
J. M. Garcia et al., APL 79, 656 (2001)

↪ 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

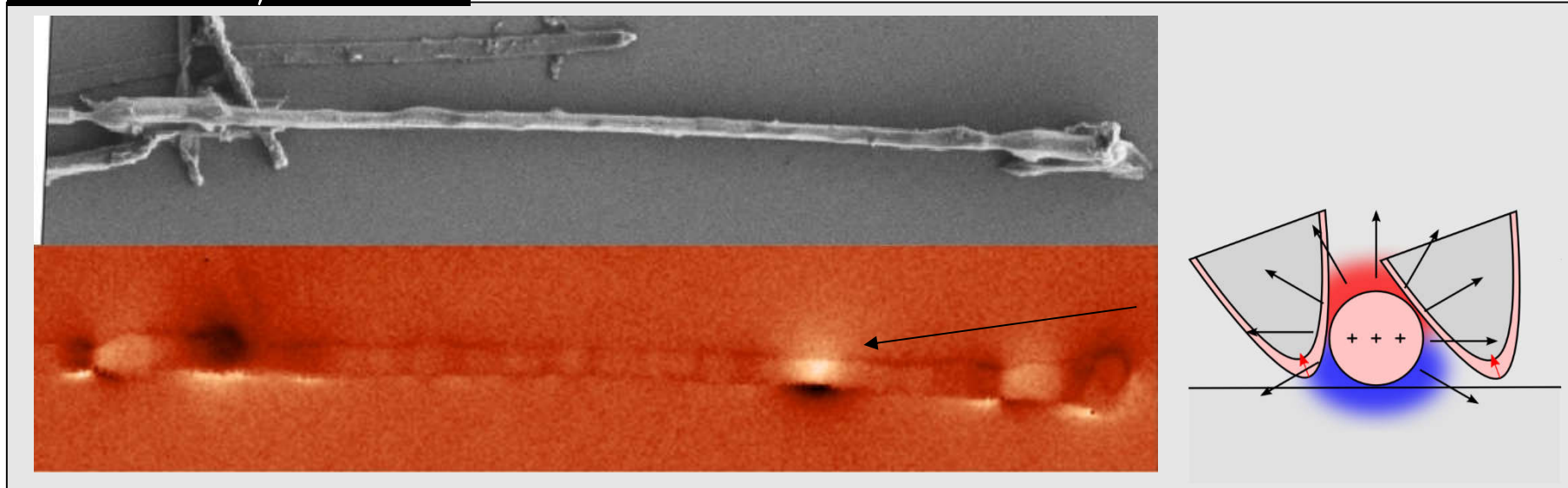


R. McMichael and M. Donahue, IEEE Trans. Magn. 33, 4167 (1997)

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

PRACTICE / Effect of tilted cantilever and tip

Tilted cantilever, across wire



Tilted cantilever, along wire

@NEEL/SPINTEC : S. Da Col et al., APL109, 062406 (2016)

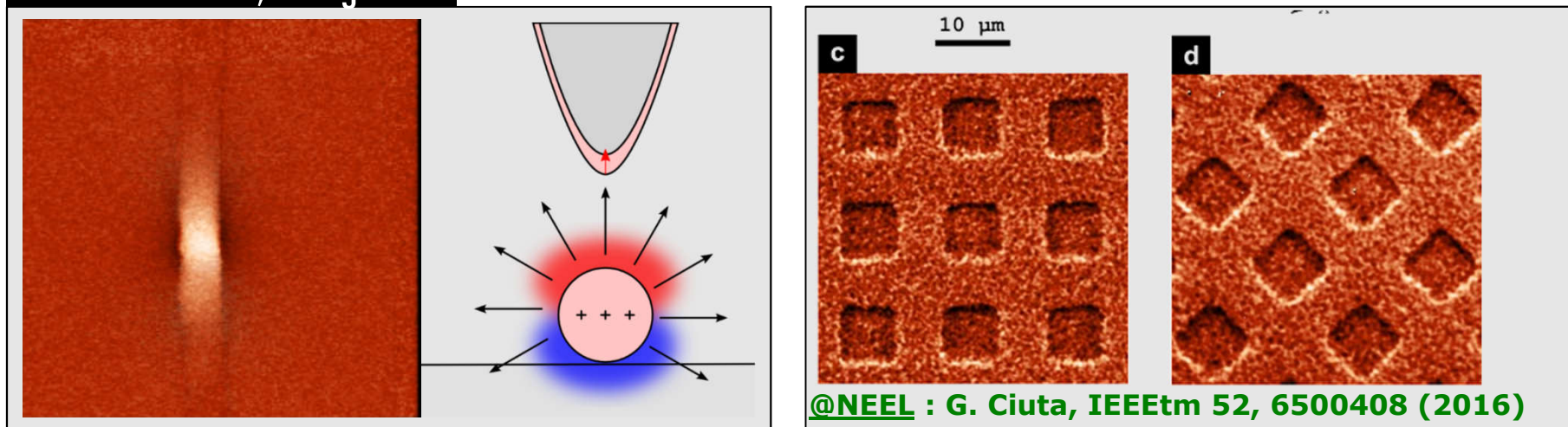
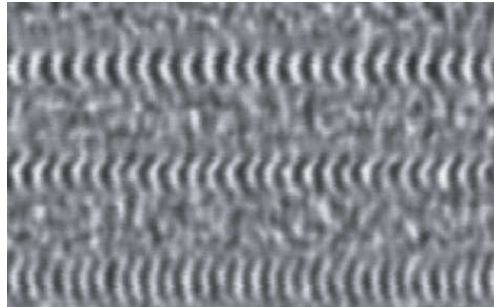
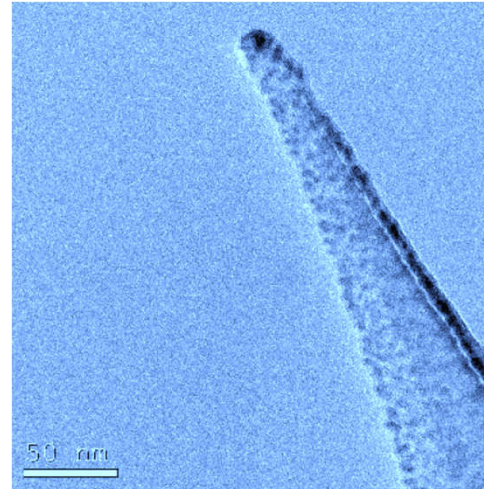


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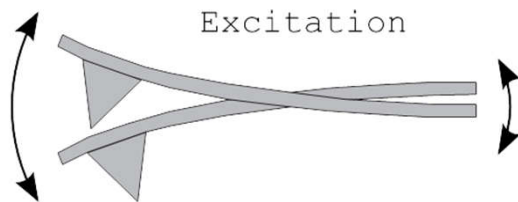
- Motivation and criteria



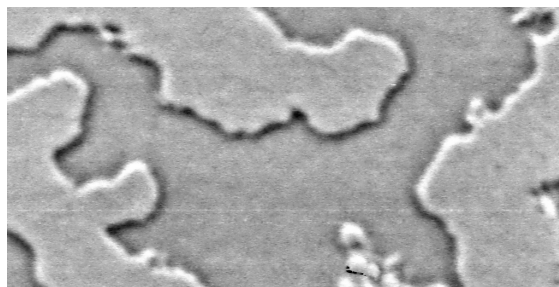
- Choice of tips



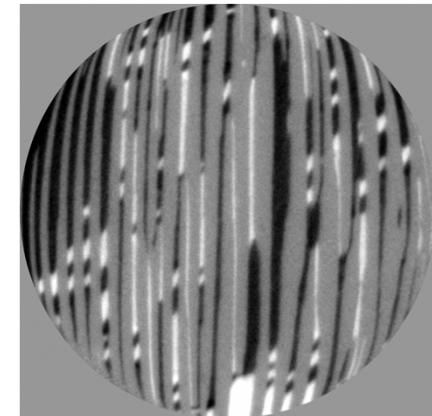
- Working principle



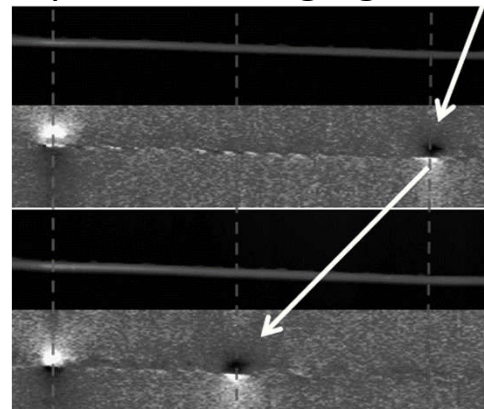
- Image analysis



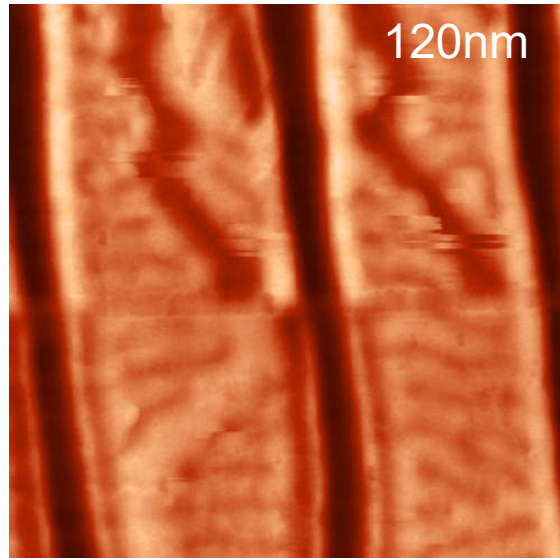
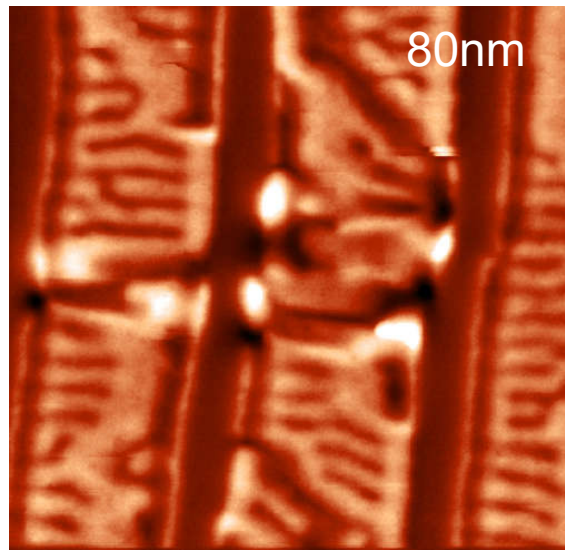
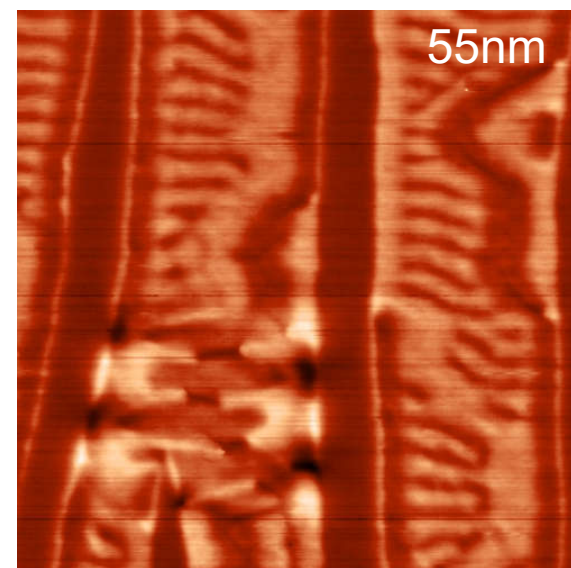
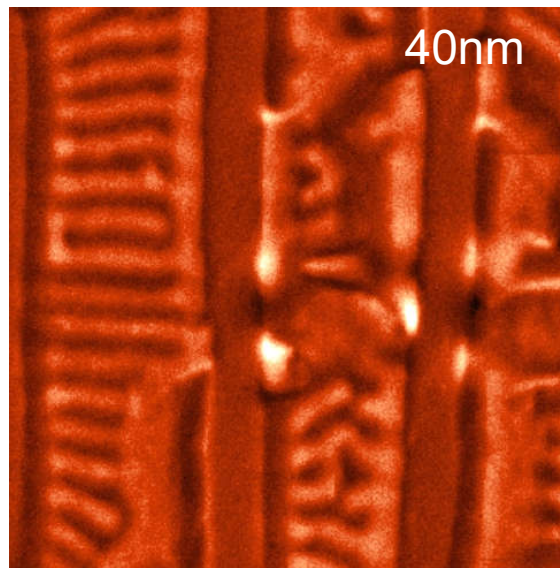
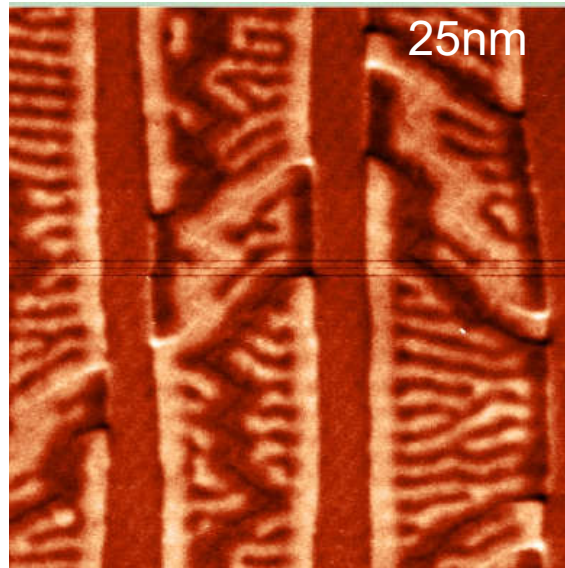
- Panorama of other microscopies



- Operando imaging



TIPS. Calibration | here : commercial



↪ Compare tips !
↪ Use identical sample and imaging parameters

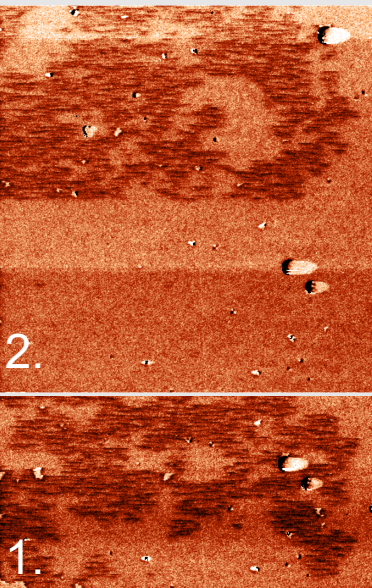
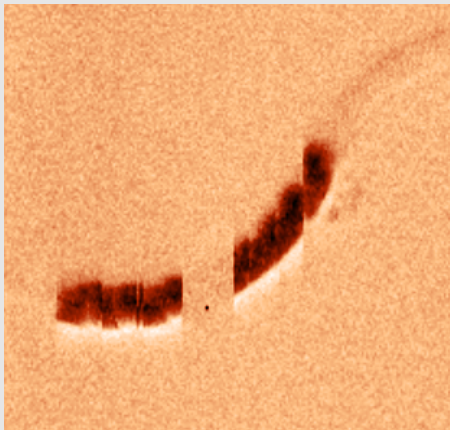
Sample : MnAs(001)
R. Belkhou (Soleil)

TIPS. Influencing samples

High

Scanning ↑

Attraction

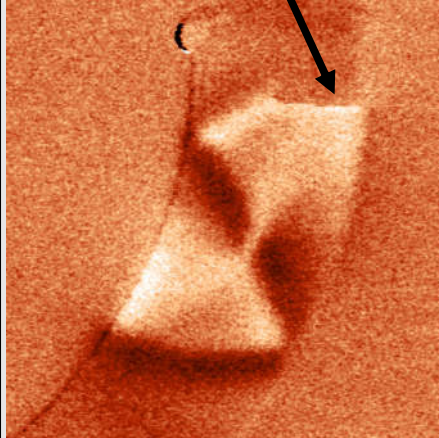


Permalloy (15nm)
500nm strips
Sample : S. Pizzini
Imaging : Z. Ishaque

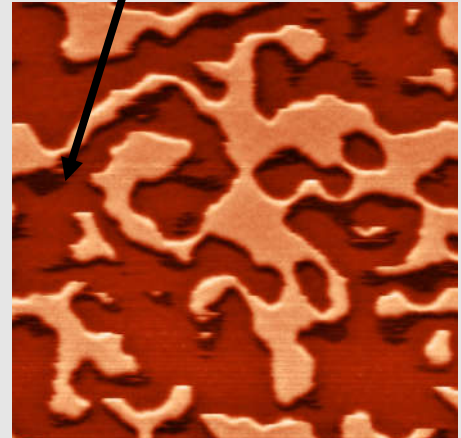
Co (0.6nm)/
graphene (20 mic)
C. Dieudonné

Moderate

Repulsion



Attraction



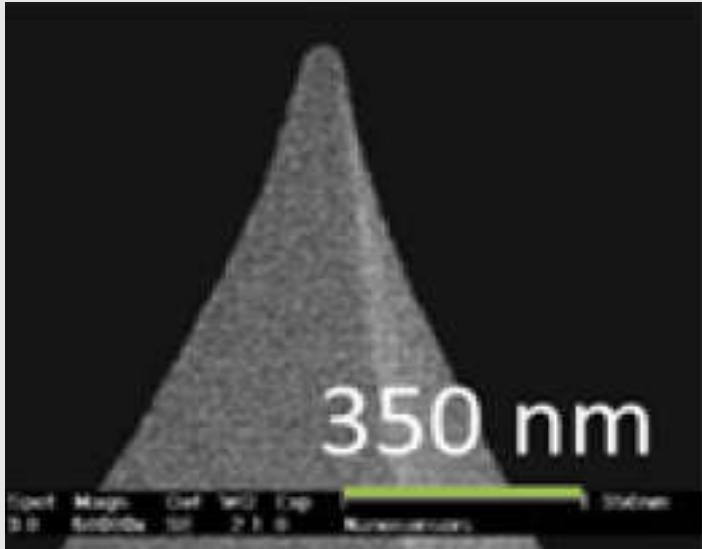
Permalloy (15nm)
500nm wide
Sample : S. Pizzini
Imaging : Z. Ishaque

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

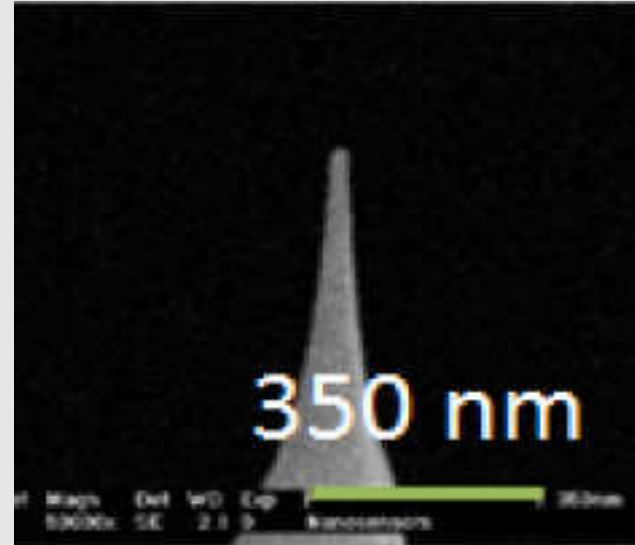
Improve resolution

From sharp to ultrasharp AFM tips



Asylum 240TS

Radius of curvature : 10 nm



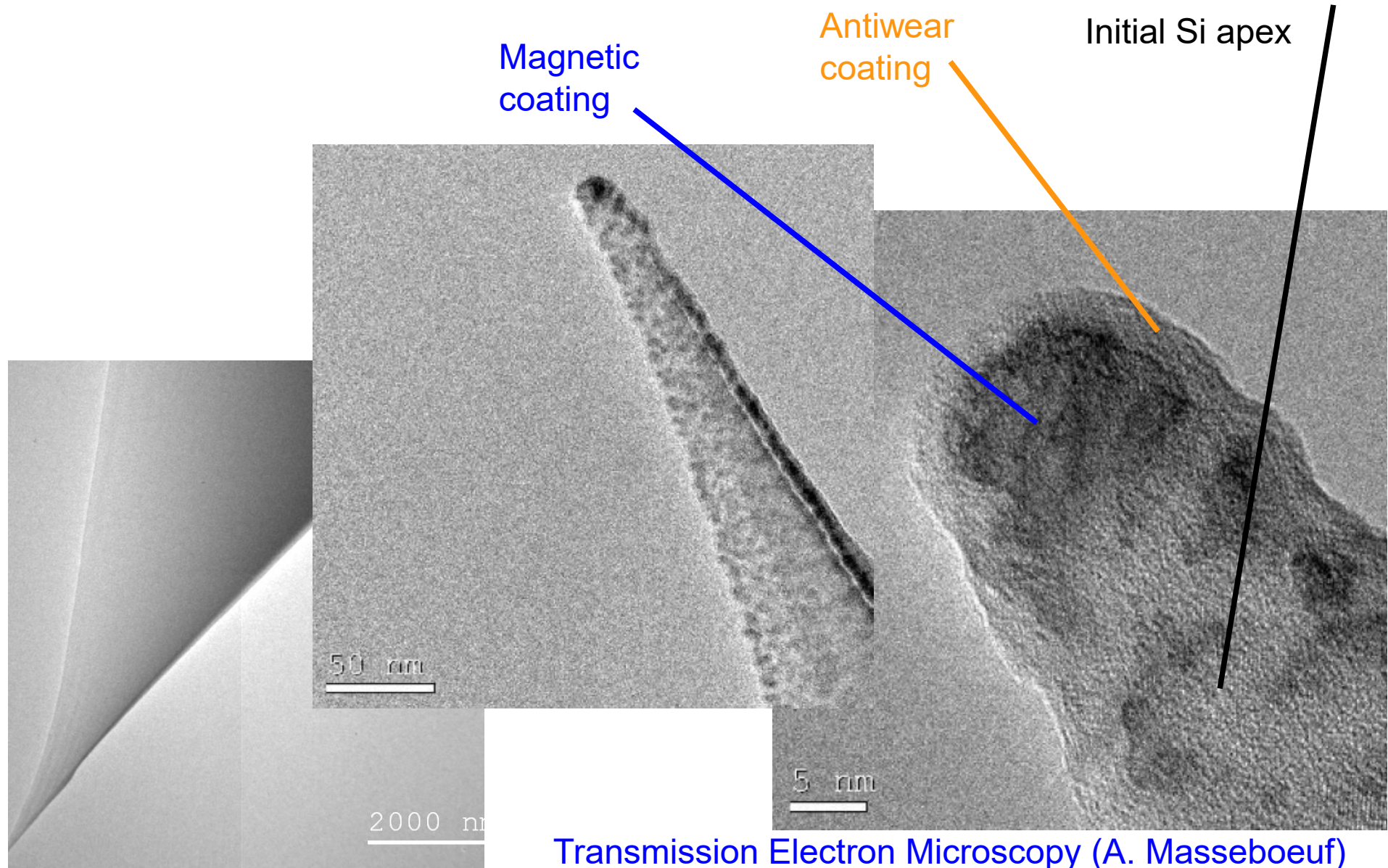
Nanosensors PPP-SSS

Radius of curvature : 2-5 nm

Engineer magnetic coating

- ⇒ Basis : $\text{Co}_{80}\text{Cr}_{20}$ (similar to hard disk media)
- ⇒ Means : sputtering.
- ⇒ Stacking : Based on CoCr [2-50 nm], various buffer and capping layers

TIPS. High-resolution MFM tips



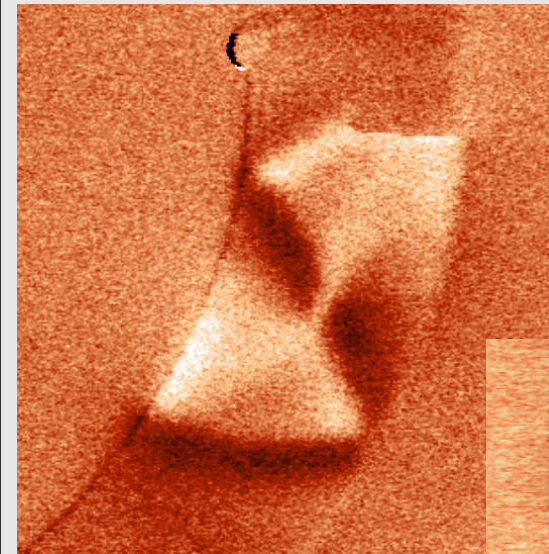
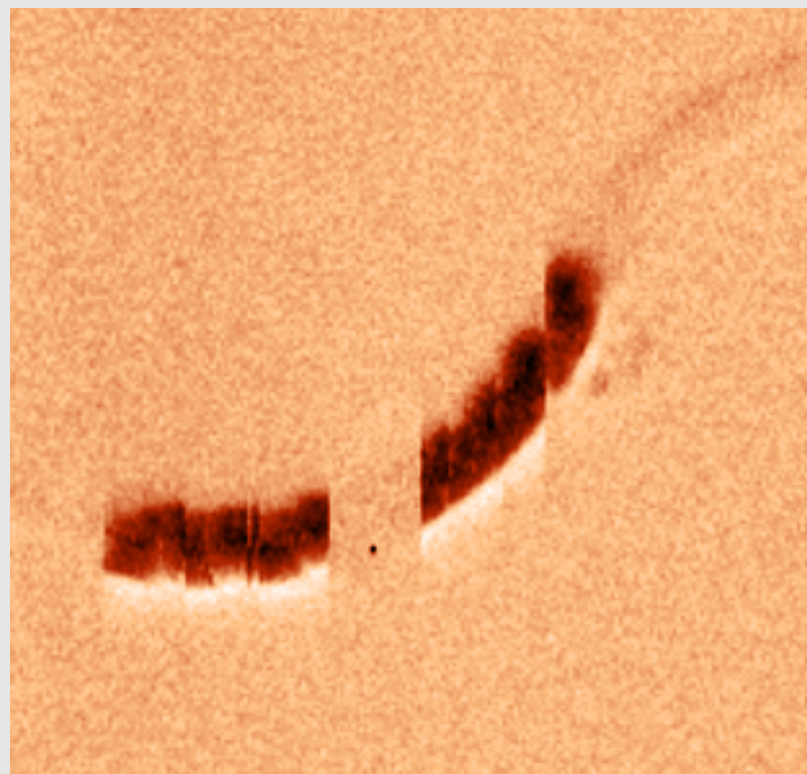
Transmission Electron Microscopy (A. Masseboeuf)

TIPS. Reduce tip-sample interaction

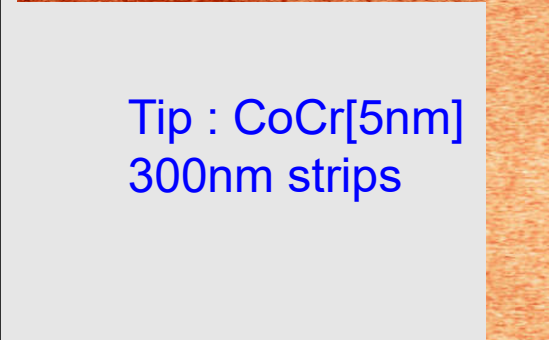
Commercial tips, 'low moment'

Permalloy strips [15nm]

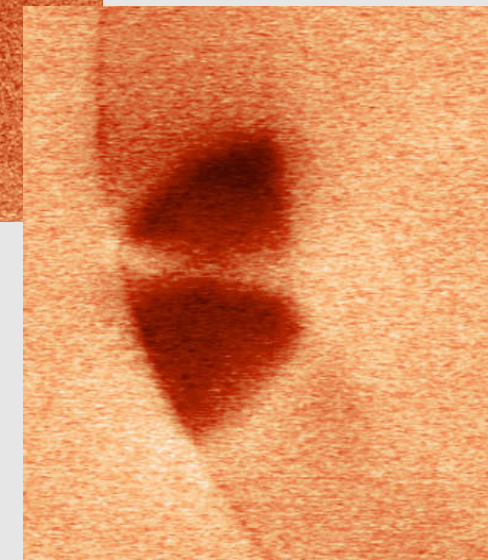
Home-made tips, really low moment



Tip : CoCr[10nm]
500nm strips



Tip : CoCr[5nm]
300nm strips



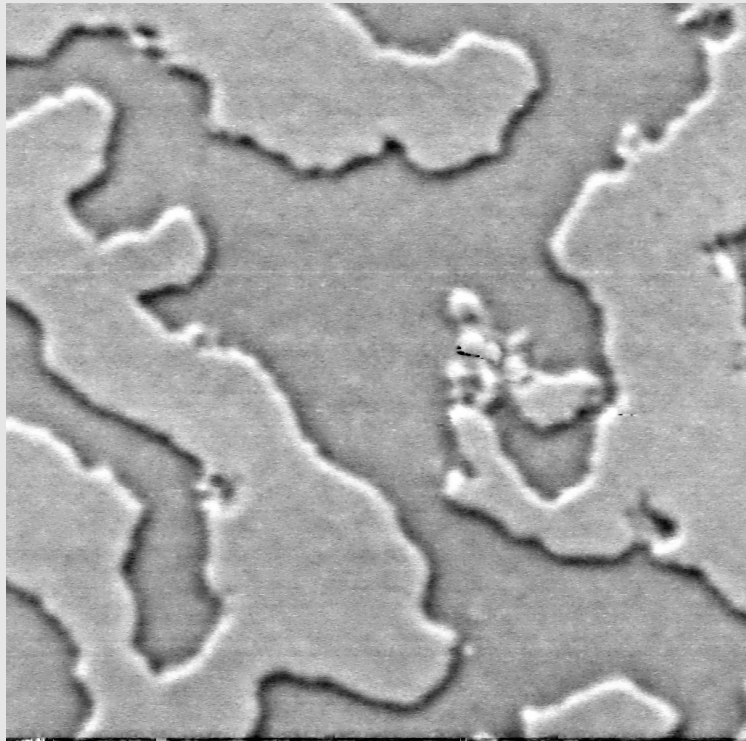
Sample : S. Pizzini
Imaging : Z. Ishaque

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

TIPS. Improved spatial resolution

Spatial resolution 15nm

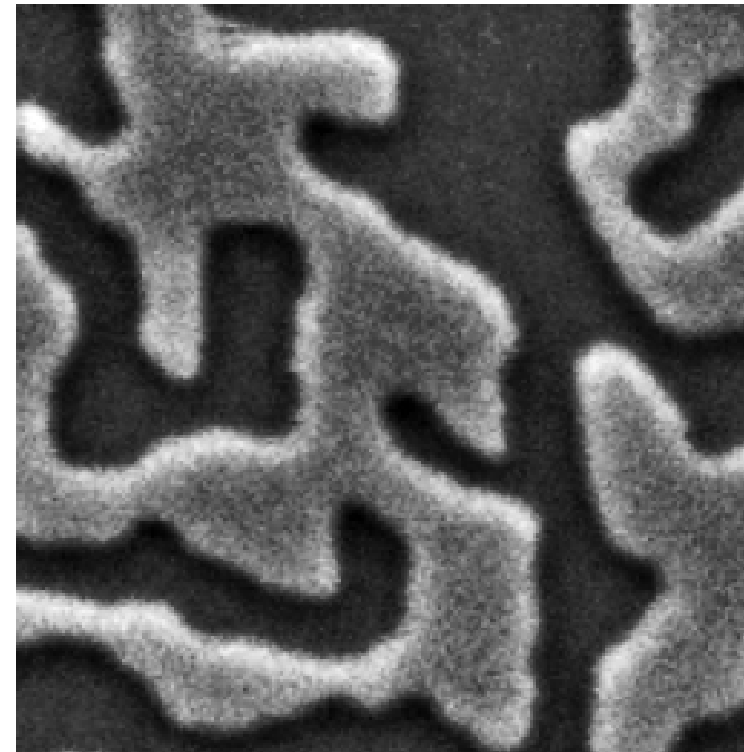
Test sample : FePt[4nm]. Perpendicular magnetization, narrow domain walls



2x2 μm

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

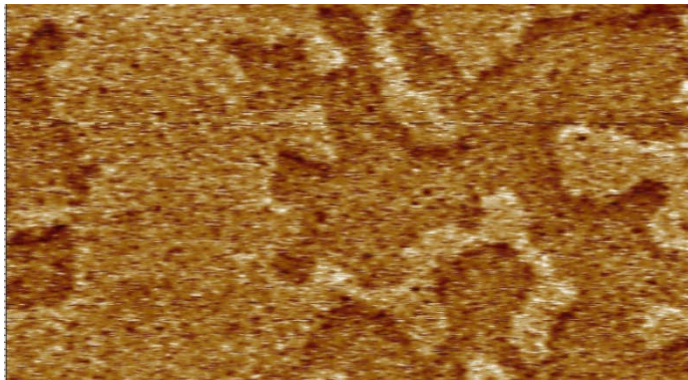
Commercial 'low moment' tip



 Spatial resolution : 20nm

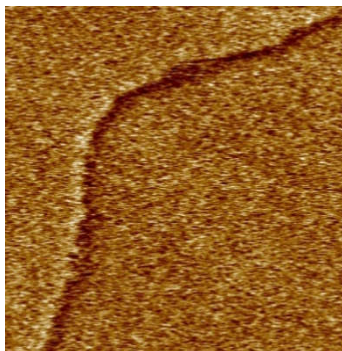
Specific aspects

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



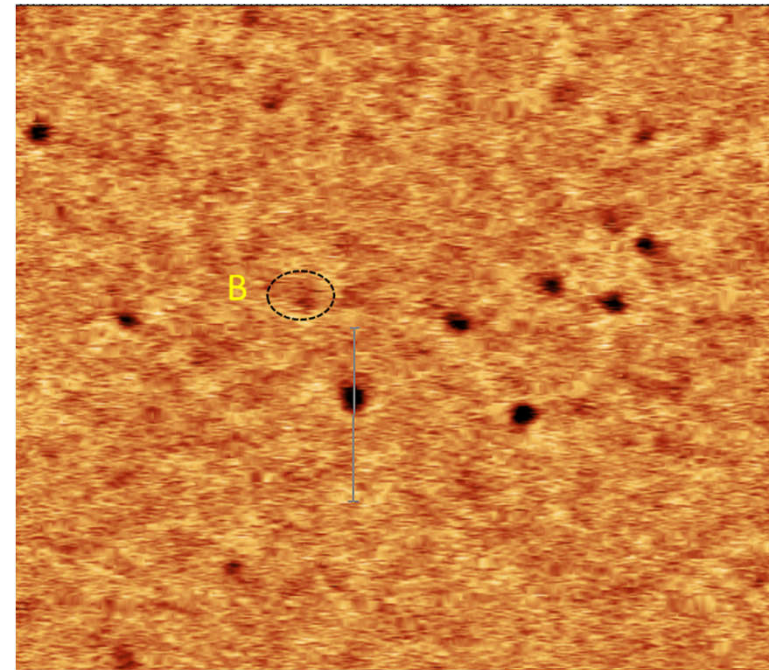
Sample: R. Juge

5x2.8 μm



Imaging: K. Richter

1.2x1.2 μm



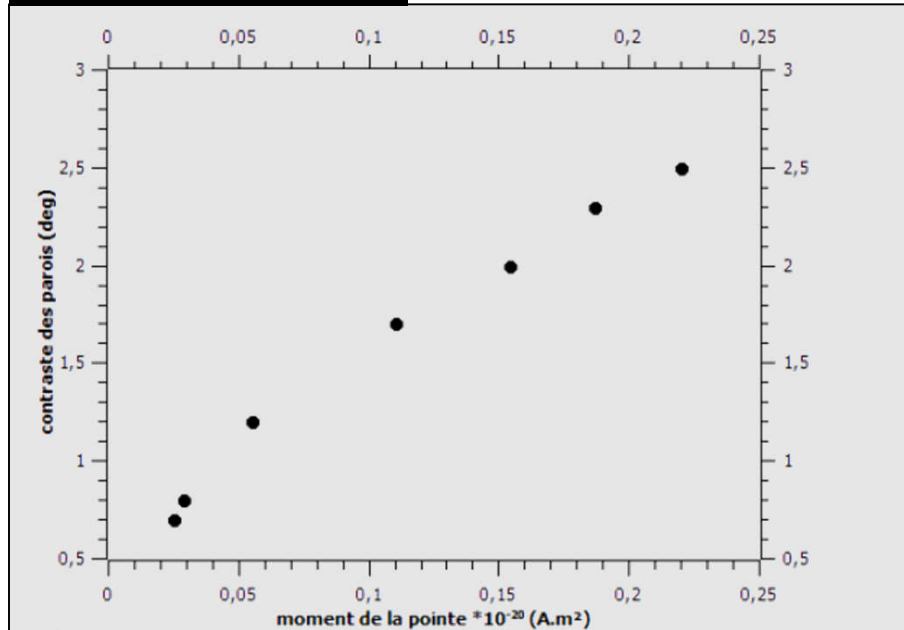
Imaging: G. Rana

3x2.5 μm

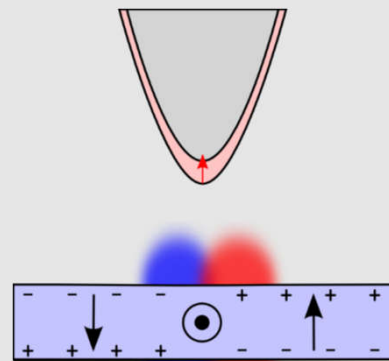
Conclusion

- Remains measurable
- Mutual contrast nearly absent

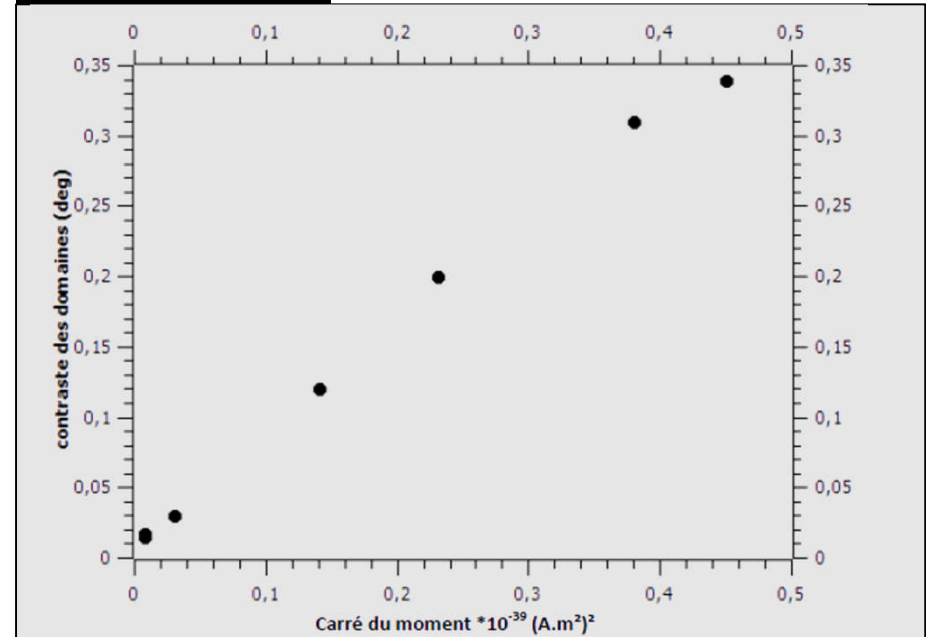
Domain wall contrast



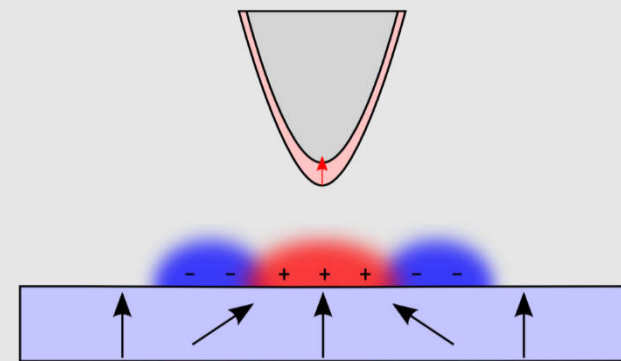
➔ Linear with tip moment



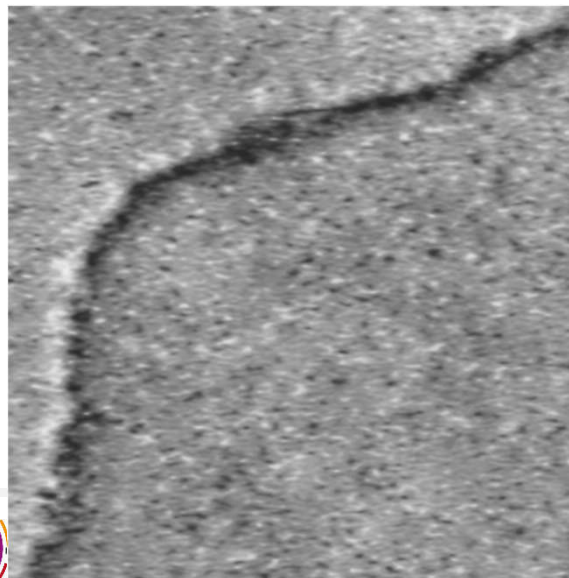
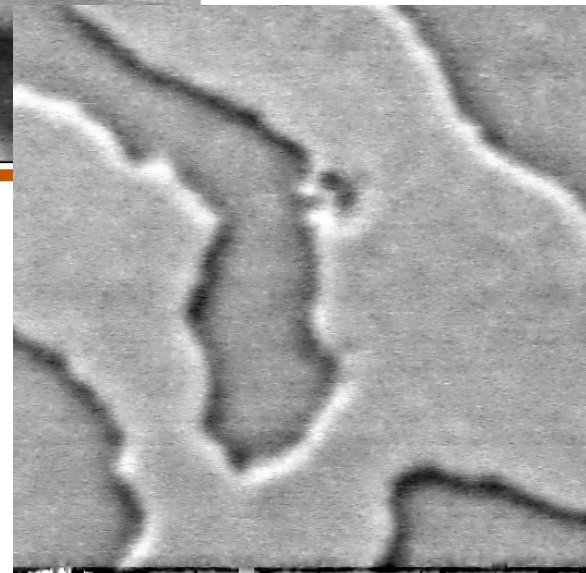
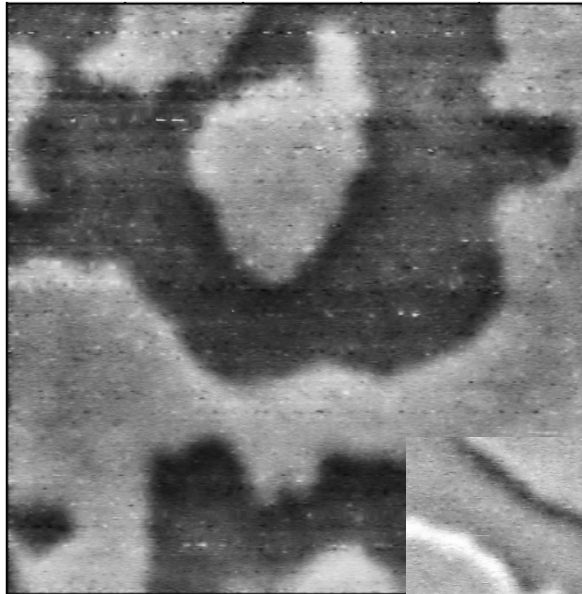
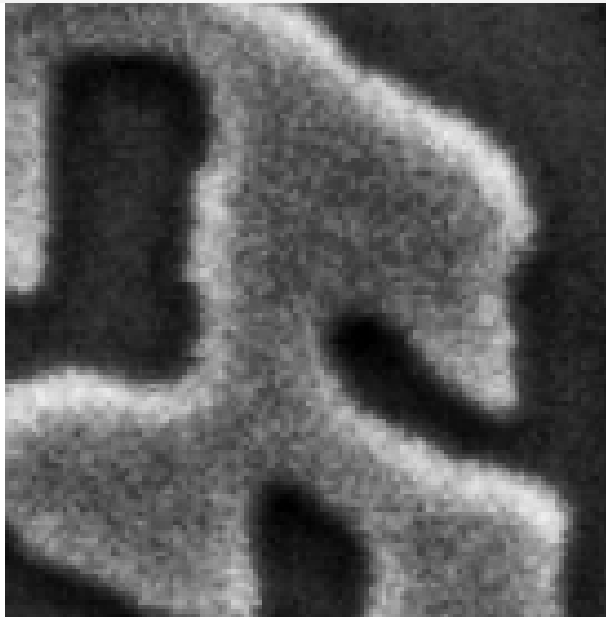
Domain contrast



➔ Linear with square of tip moment



TIPS. Both resolution and sensitivity matter



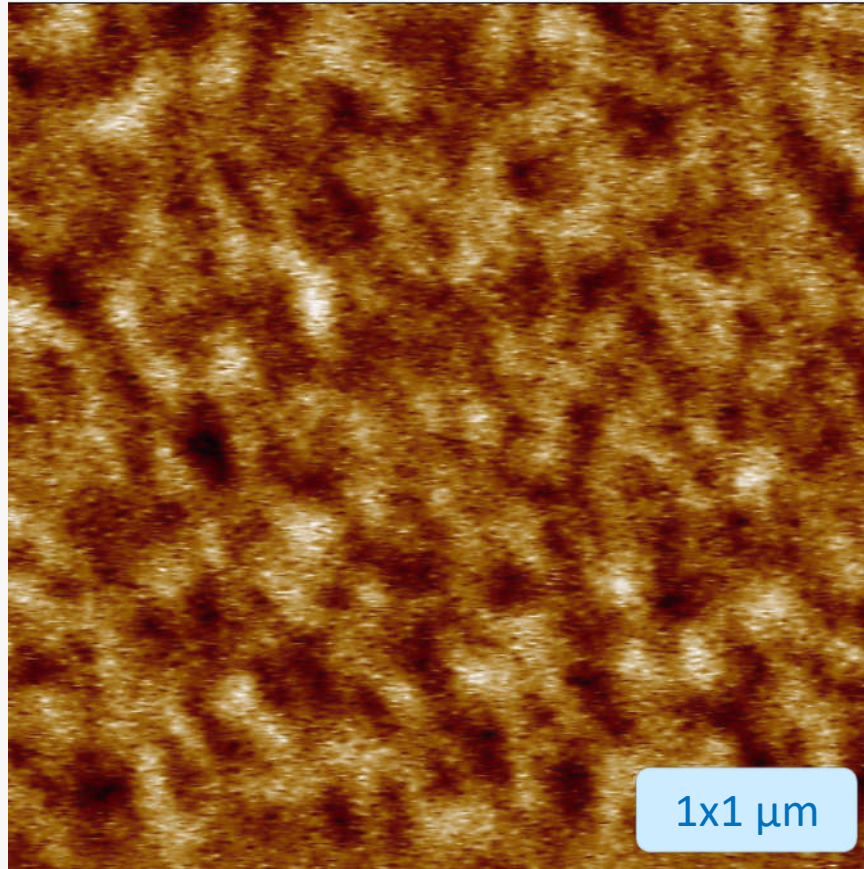
1x1 μm

Sensitivity

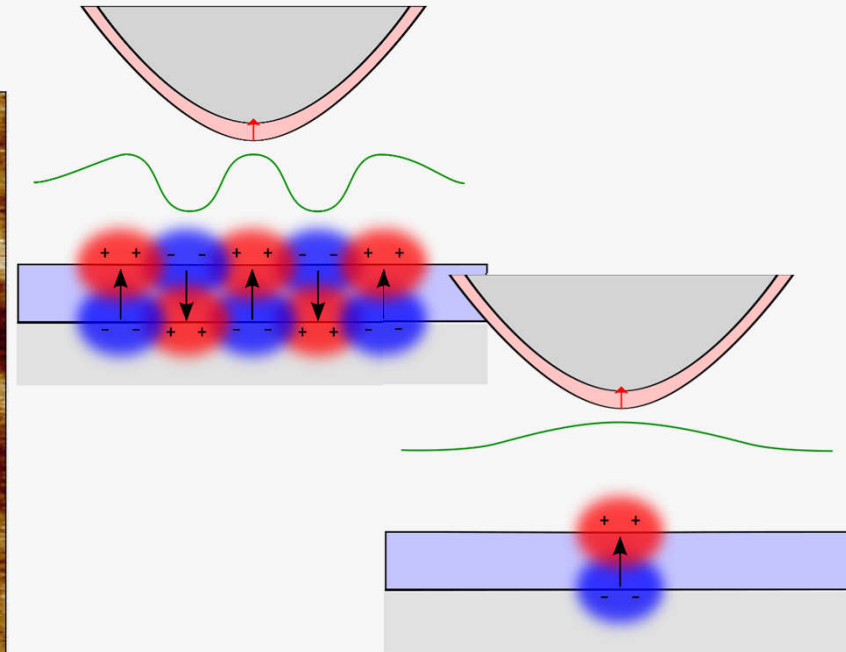
Spatial resolution

Non-invasive

NOT the smallest feature on an image



FePt-C recording media

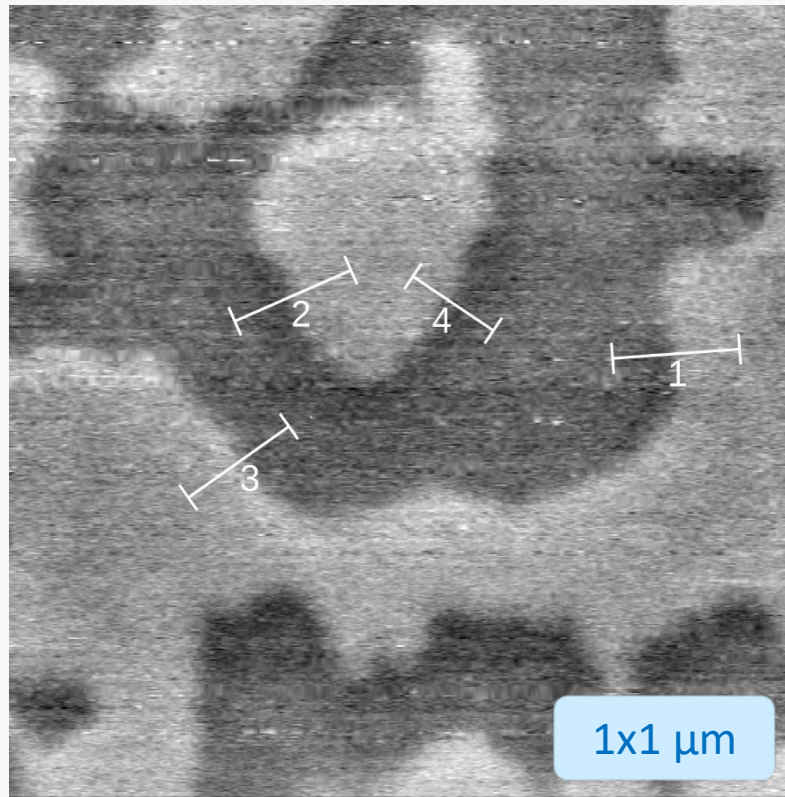


- Small scale modulation
- Reduced magnitude
- A single object would appear larger

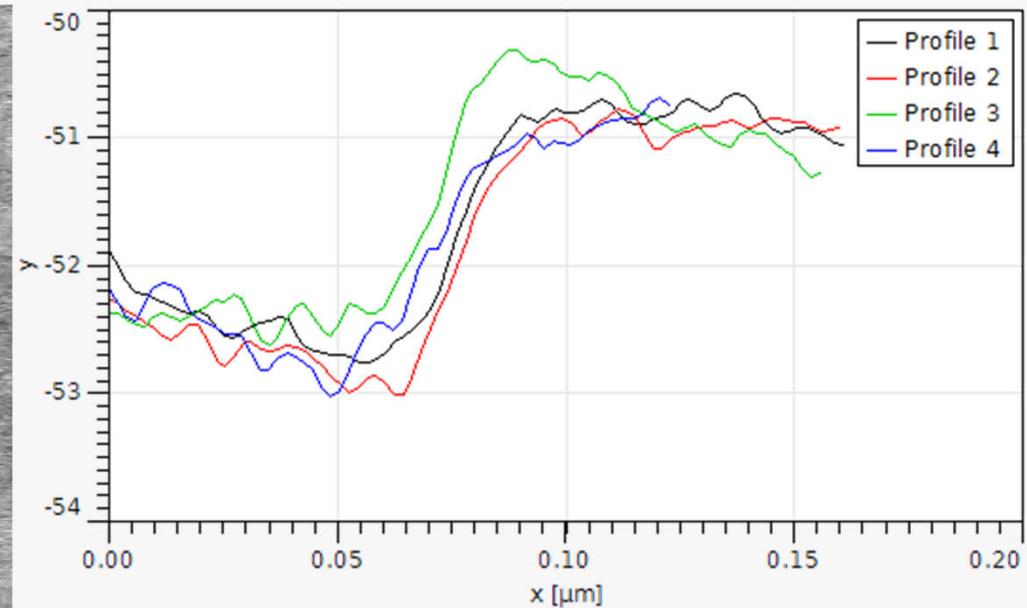


D. Diény et al., IEEE Trans. Mag., in print (2018)

Use isolated well-defined magnetic pattern



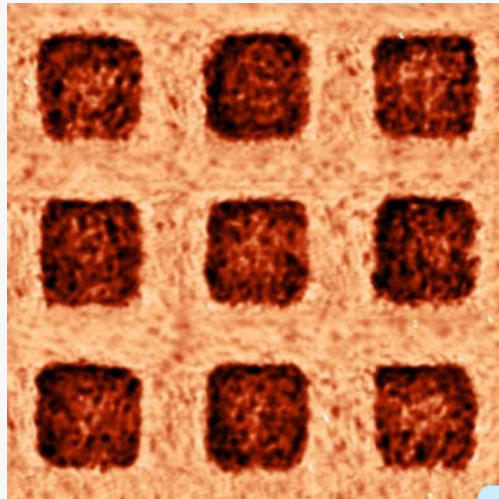
FePt, epitaxial (4nm)



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

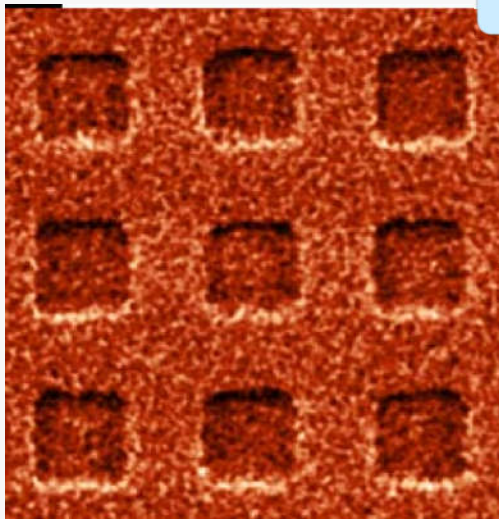
Quantitative analysis, see e.g.: H. Hug, J. Appl. Phys. 83, 5609 (1998) and followers

May require hard tip coating



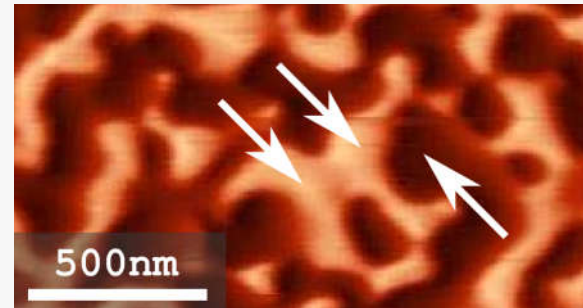
Soft coating

50x50 μm

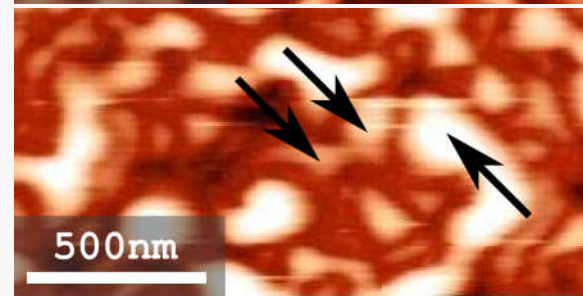


Hard coating

May require stiff cantilevers



MFM



Topography (artefacts)

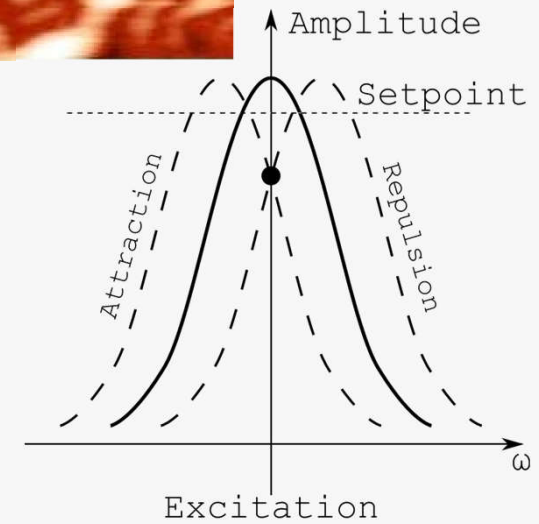
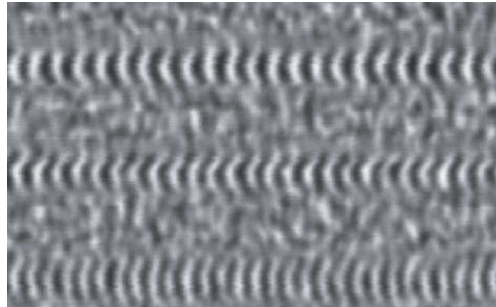
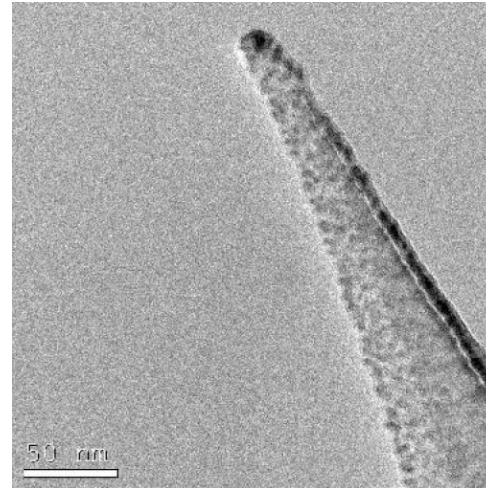


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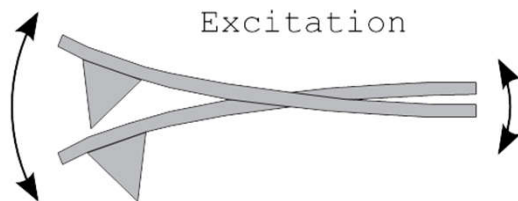
- Motivation and criteria



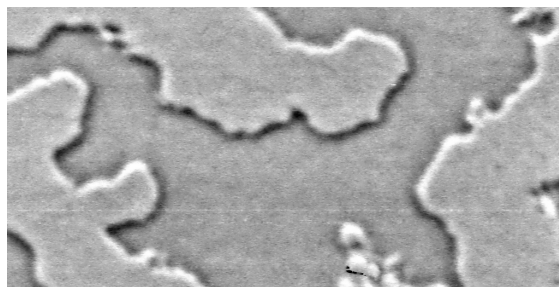
- Choice of tips



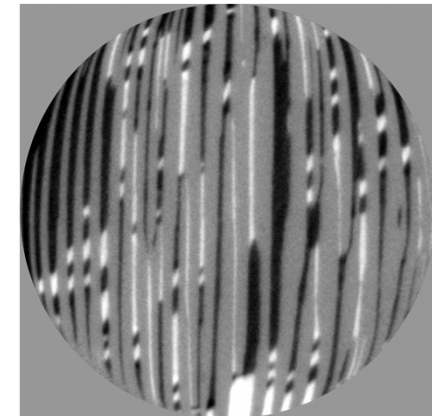
- Working principle



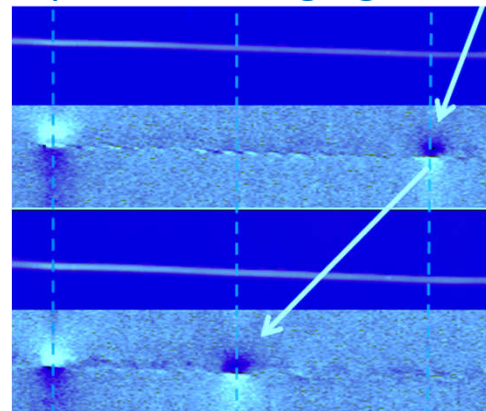
- Image analysis

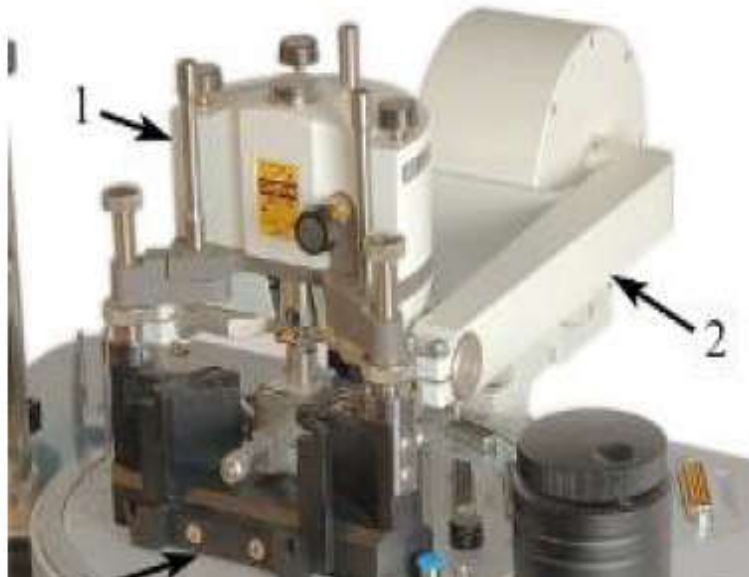


- Panorama of other microscopies

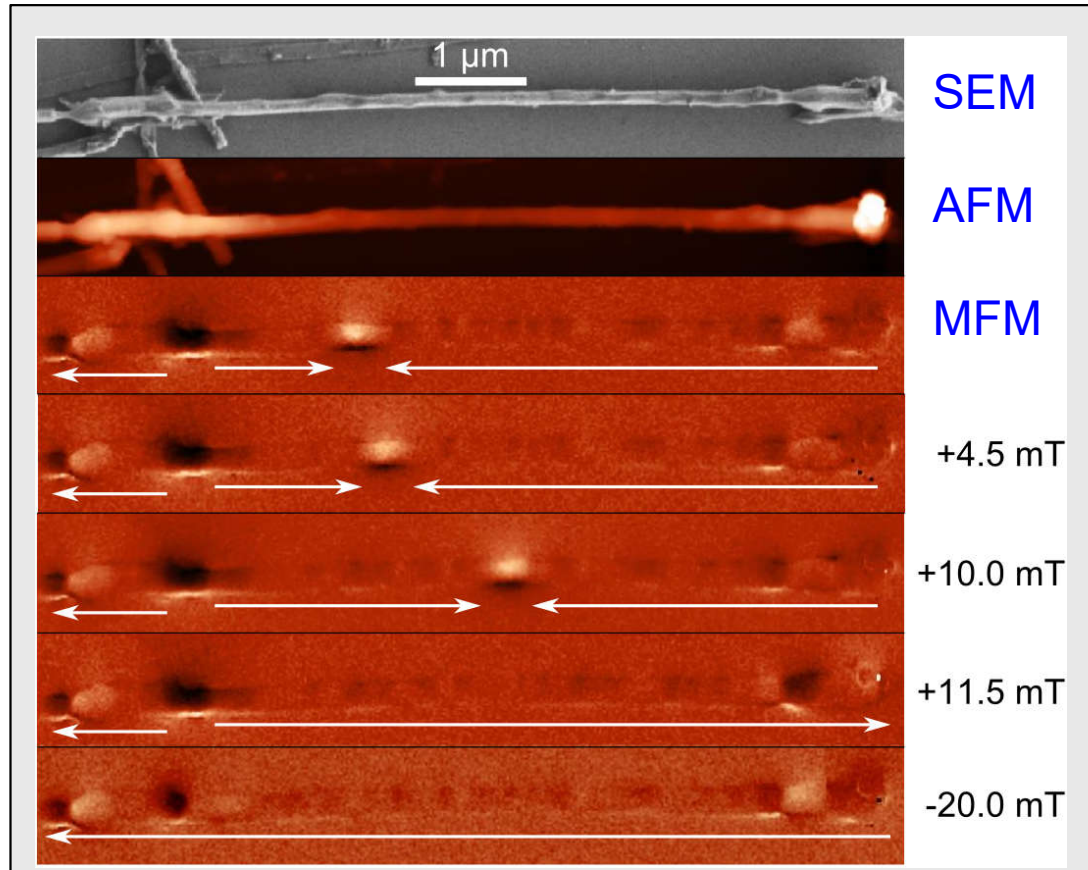


- Operando imaging



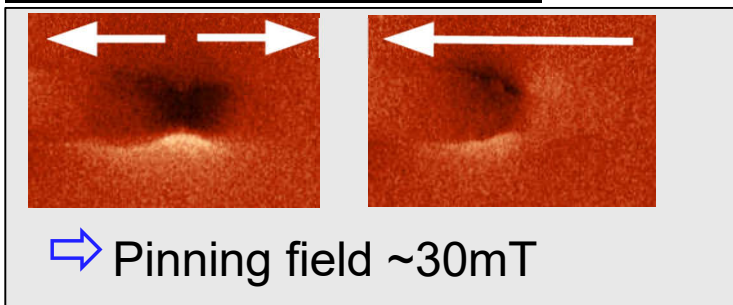


Domain wall motion



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

Protrusions and constrictions

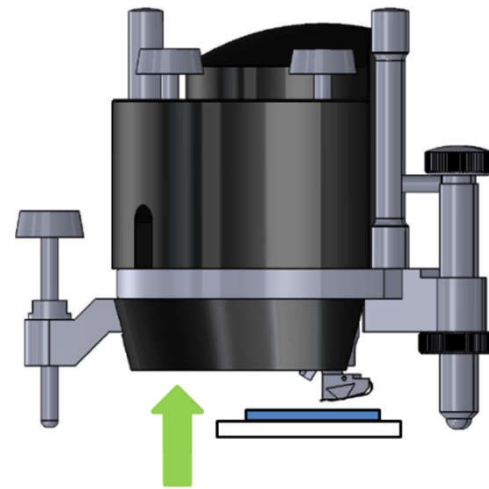


↻ Imaging under field
↻ Magnetization processes

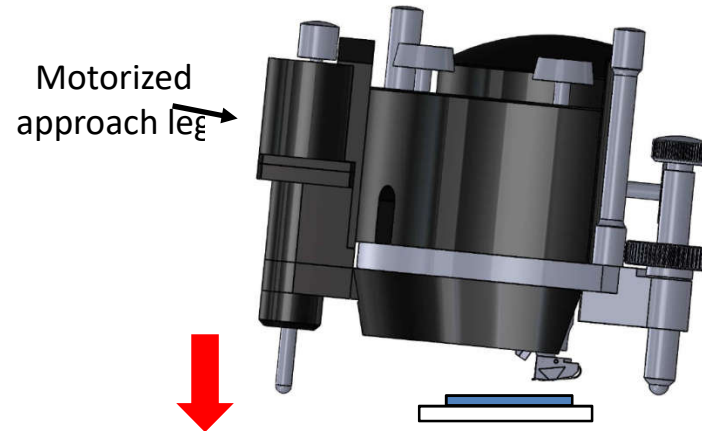
OPERANDO. Out-of-plane field



Field >1T (custom-made)
Optimized cooling



Microscope approach
by moving the sample up
(standard configuration)

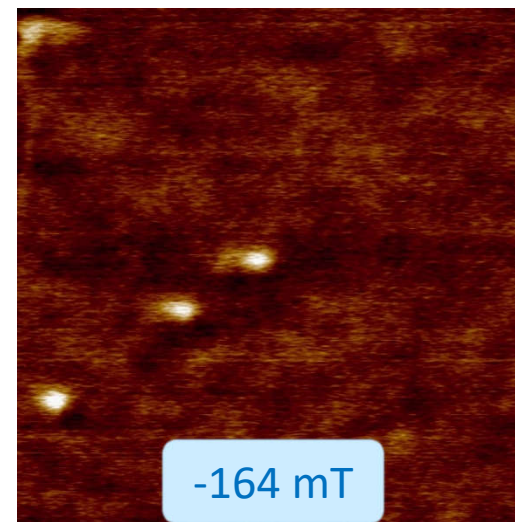
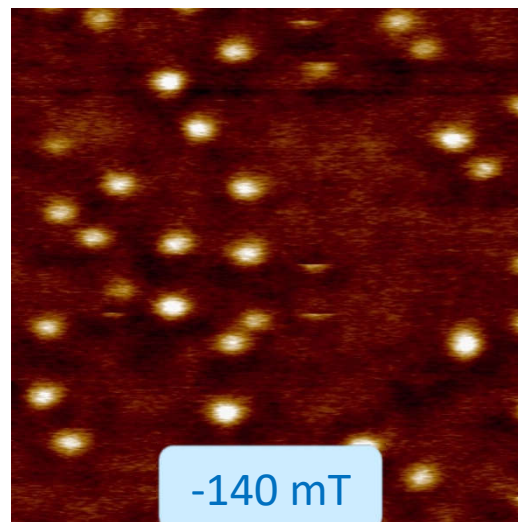
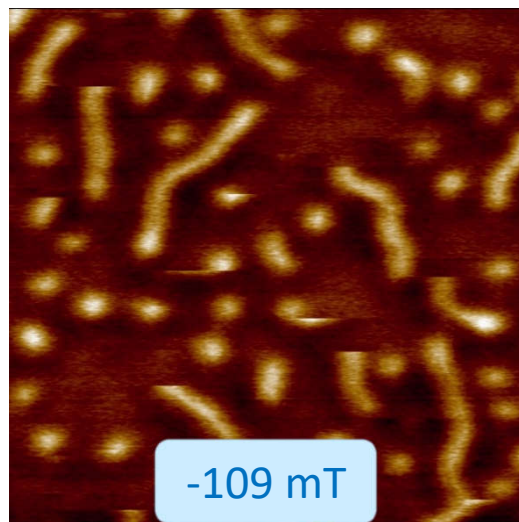
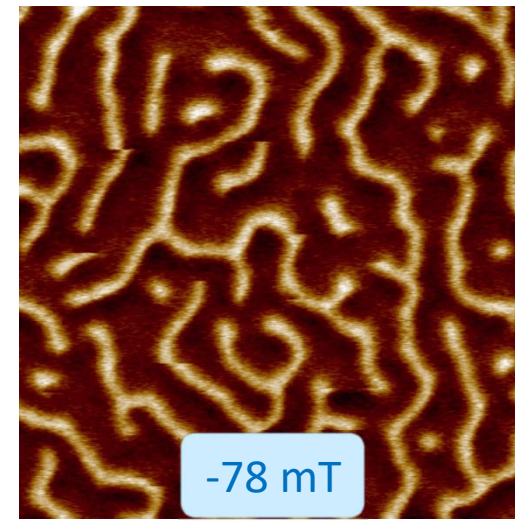
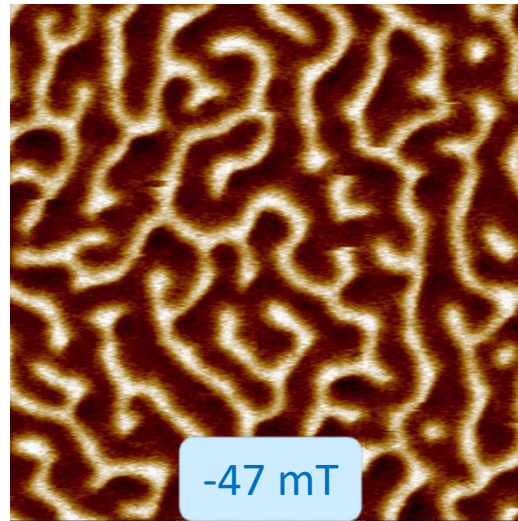
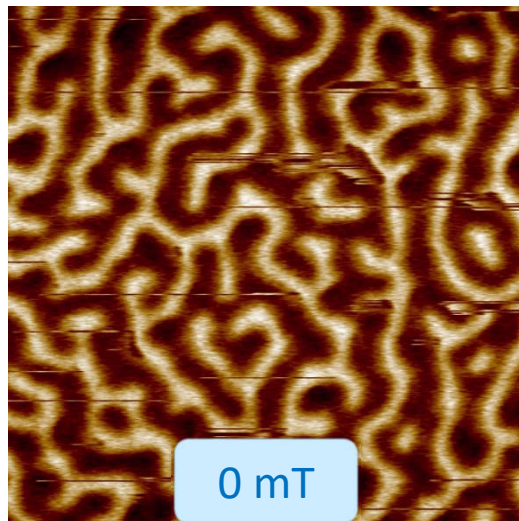


Microscope approach
by tilting the head
thanks to the motorized leg
(coil coupled configuration)

↪ Approach by head (motorized leg)

OPERANDO. Out-of-plane field

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

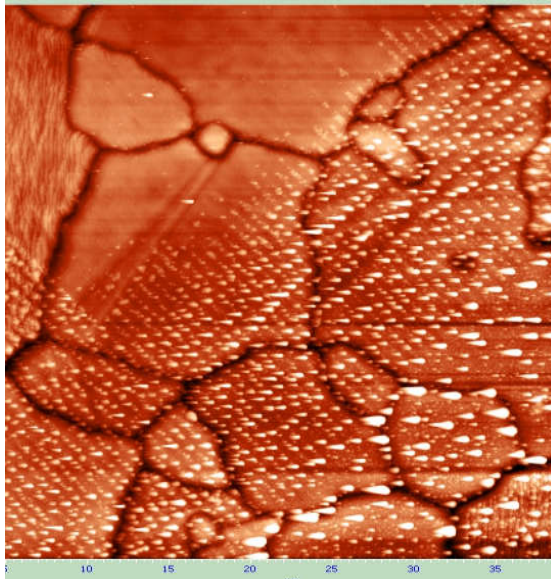


Sample: Chloé Bouard

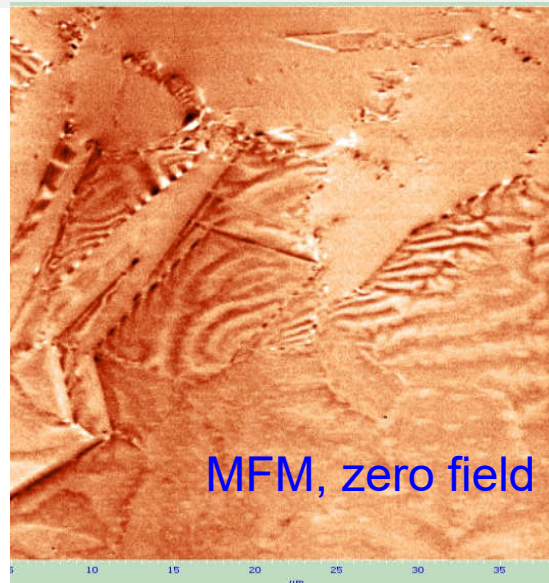
OPERANDO. Out-of-plane field

Magnetic shape-memory alloy

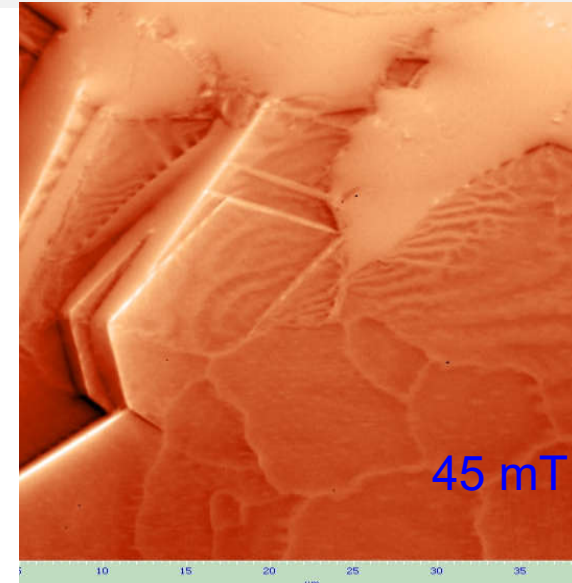
G. Crougneau et al.



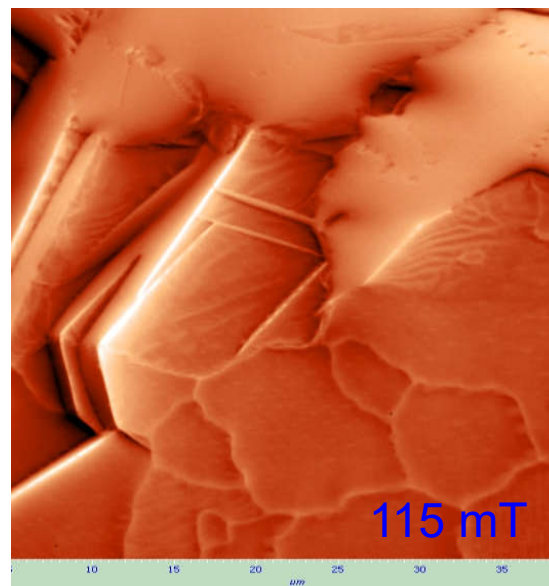
Topography (grains)



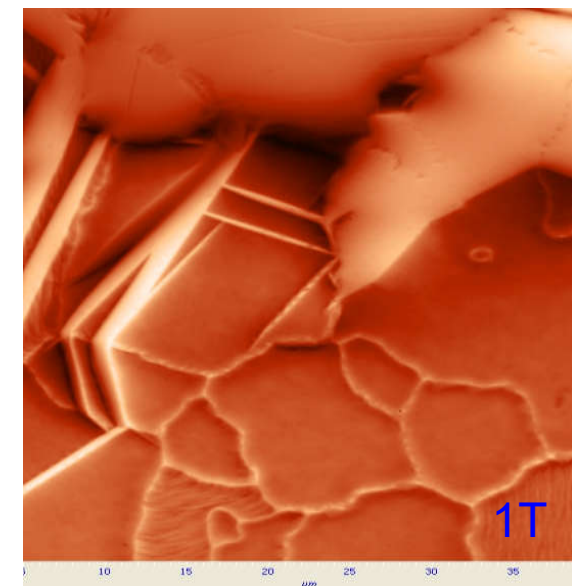
MFM, zero field



45 mT

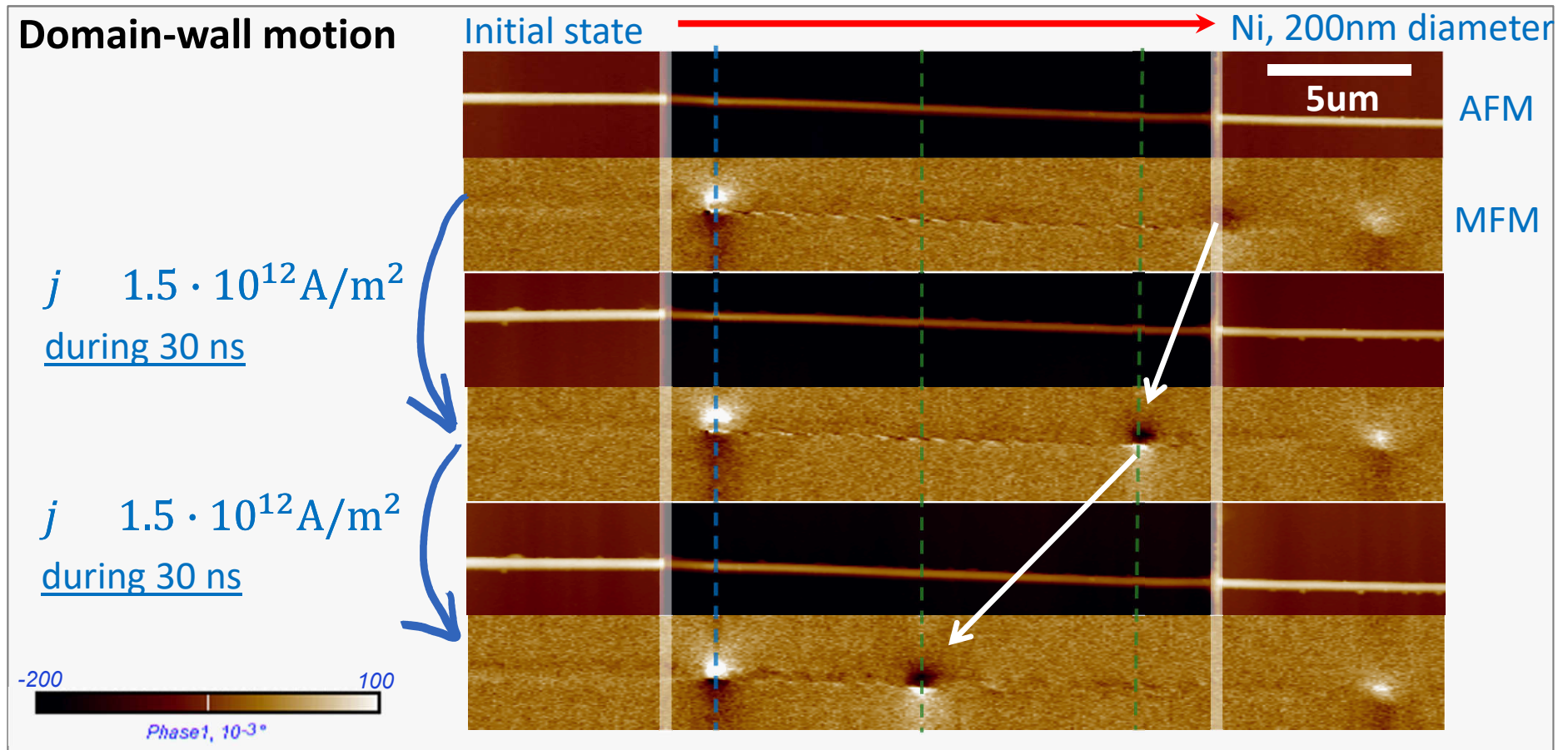


115 mT



1 T

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



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

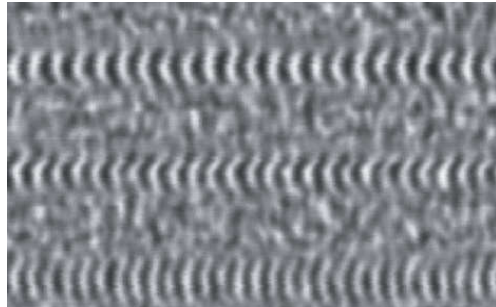
S. Jamet, JC Toussaint

Instrumental development and maintenance

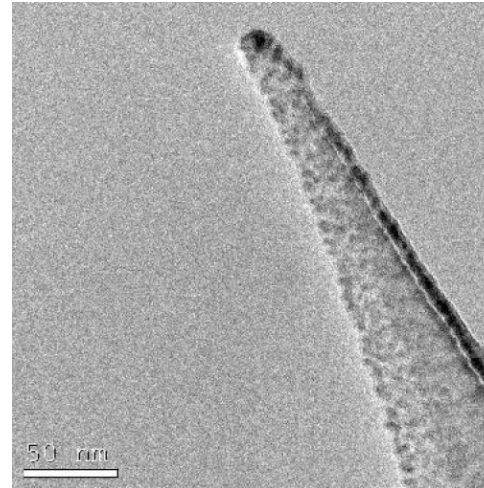
S. Le Denmat, C. Thirion, E. Wagner

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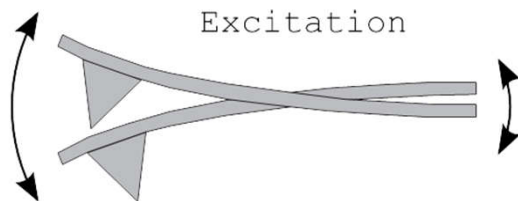
- Motivation and criteria



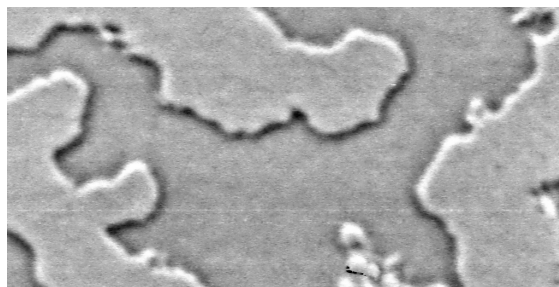
- Choice of tips



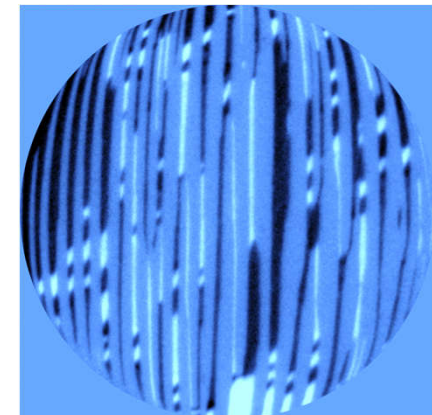
- Working principle



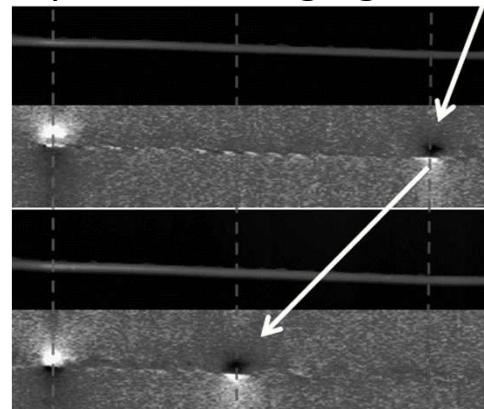
- Image analysis



- Panorama of other microscopies



- Operando imaging



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 ?

What is probed

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

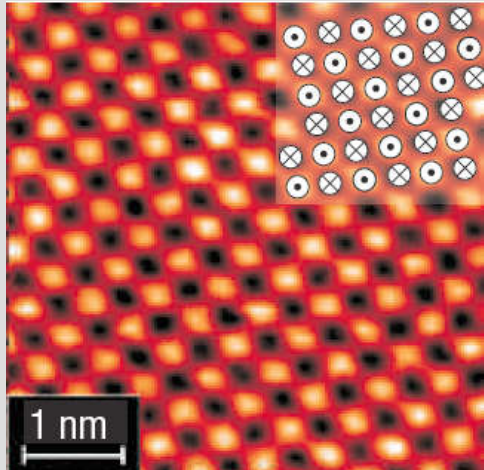
Conclusion

No universal technique

Many criteria to be balanced

Spin-polarized STM

Fe(1ML)/W(001)



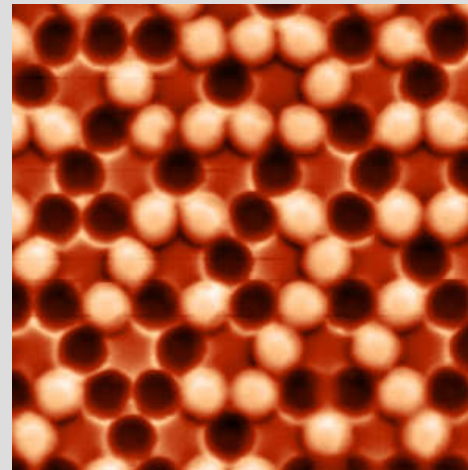
Antiferromagnetic domain

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

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

Magnetic Force Microscopy

Array of dots



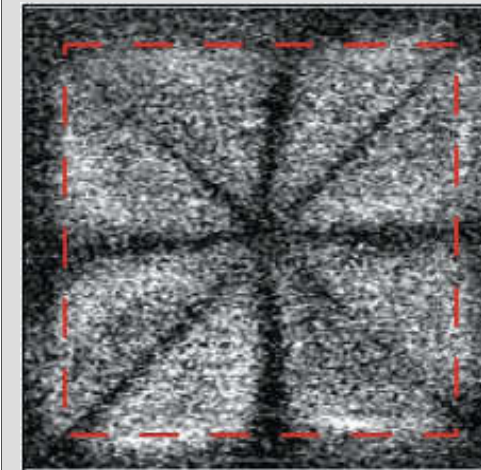
Up-and-down 'single-domains'

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

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

Overview

↪ Large variety

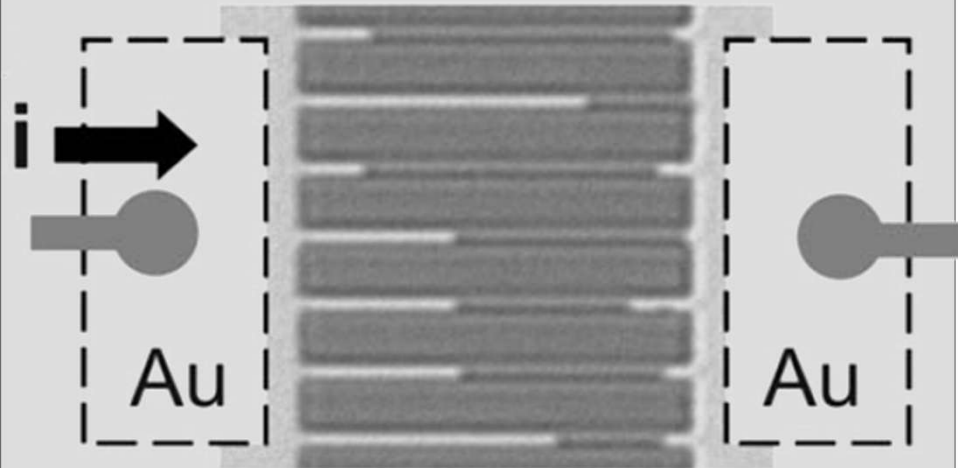
↪ Rather slow

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



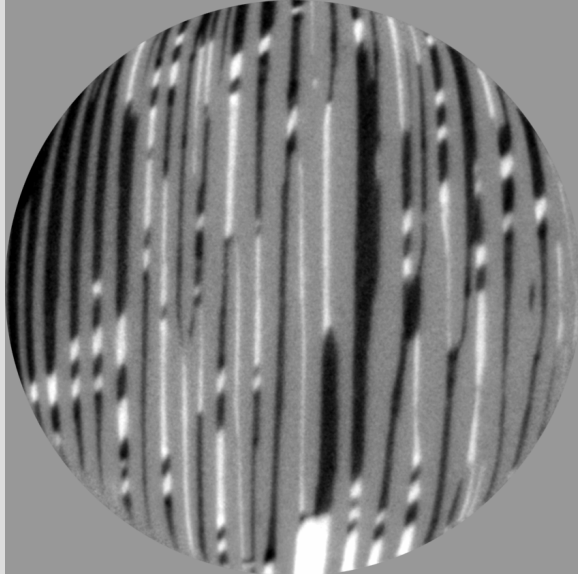
@NEEL : T. A. Moore et al., Appl. Phys. Lett
93, 262504 (2008)

Overview

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

SPLEEM

Spin-Polarized Low Energy Electron Microscopy

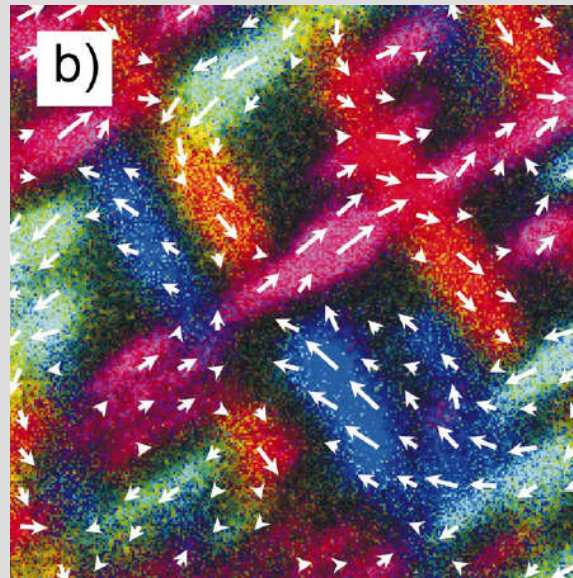


Stripes of Fe/W(110)

@NEEL, REVIEW:
N. Rougemaille et al., Eur. Phys. J. Appl. Phys. 50, 20101 (2010)

SEMPA

Scanning Electron Microsc. with Polarization Analysis

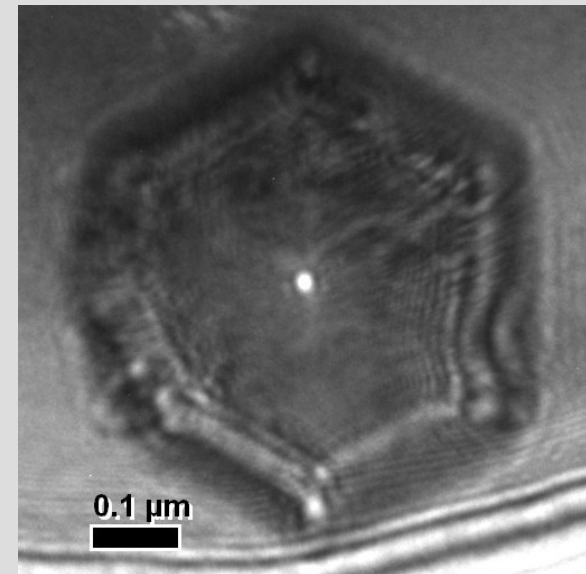


Maze of Fe/W(001) 1.5 μm

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

Lorentz, holography etc.

TEM - based



Self-assembled Co/W(110)

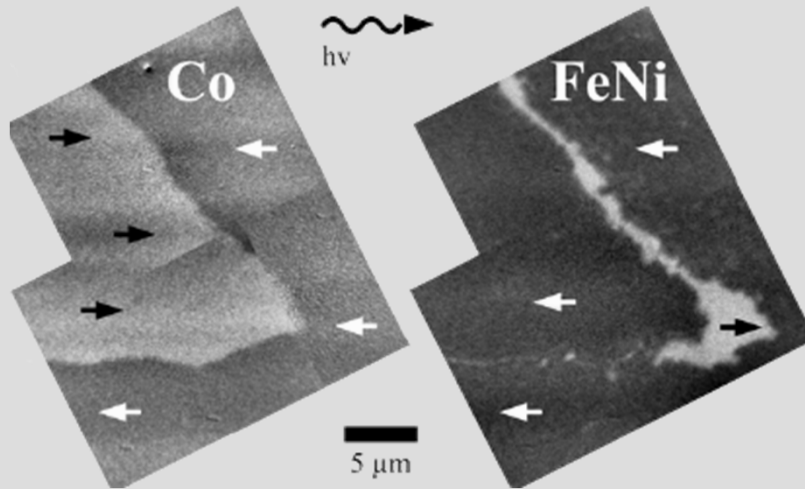
@NEEL :
O. Fruchart et al., J. Phys. Condens. Matter 25, 496002 (2013)

Overview

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

XMCD-PEEM

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



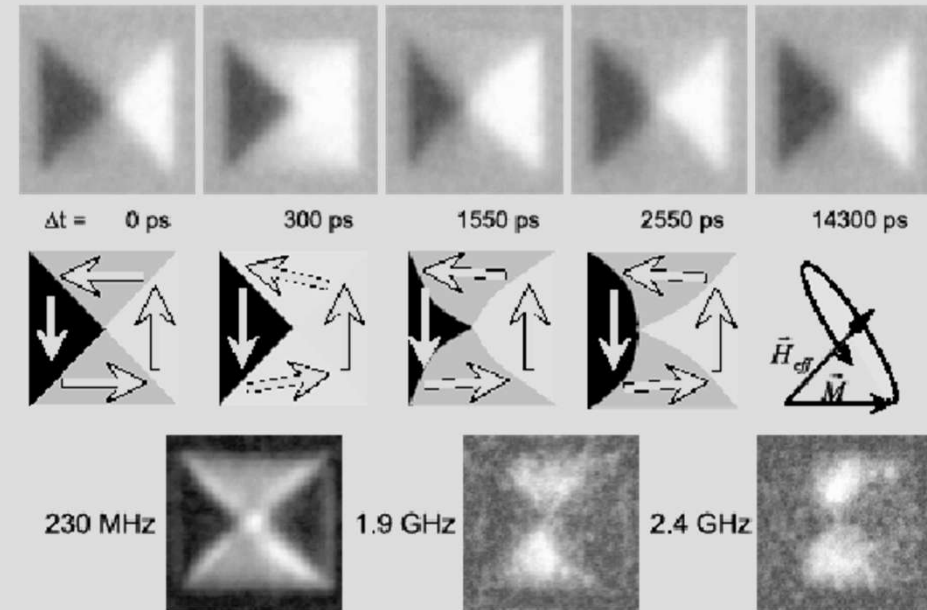
Co\Cu\FeNi trilayer
→ elemental resolution

@NEEL :

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

TXM

Transmission X-ray Microscopy



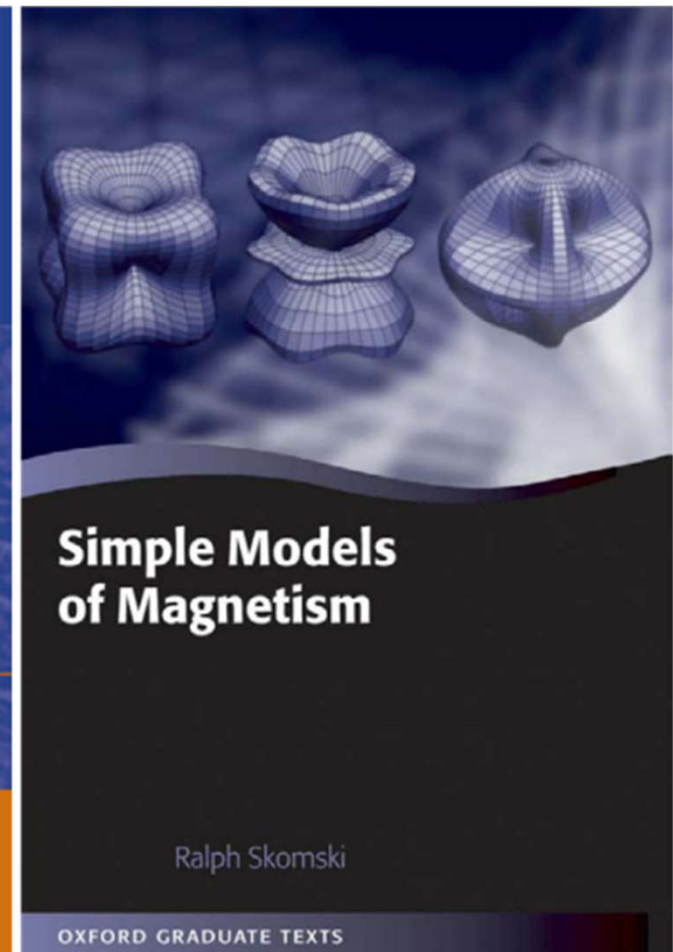
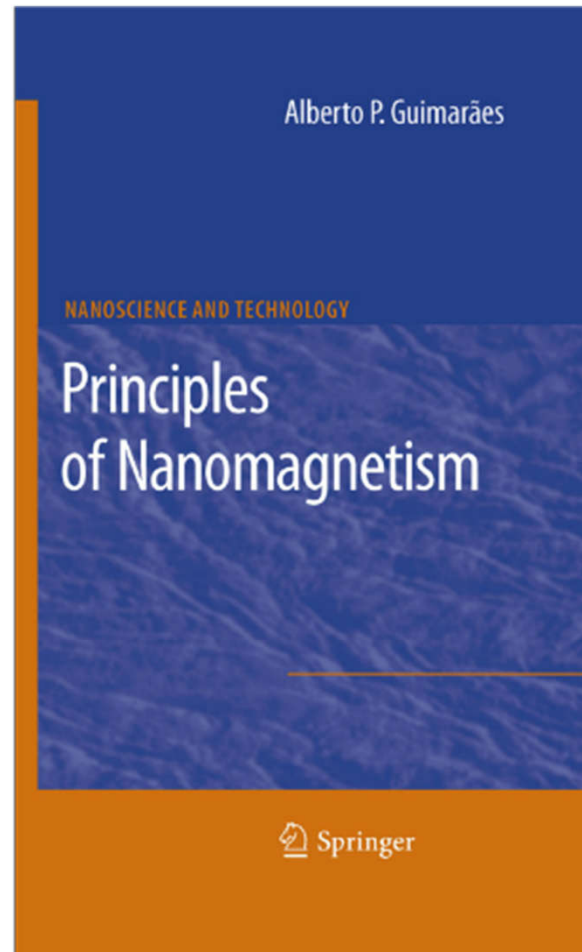
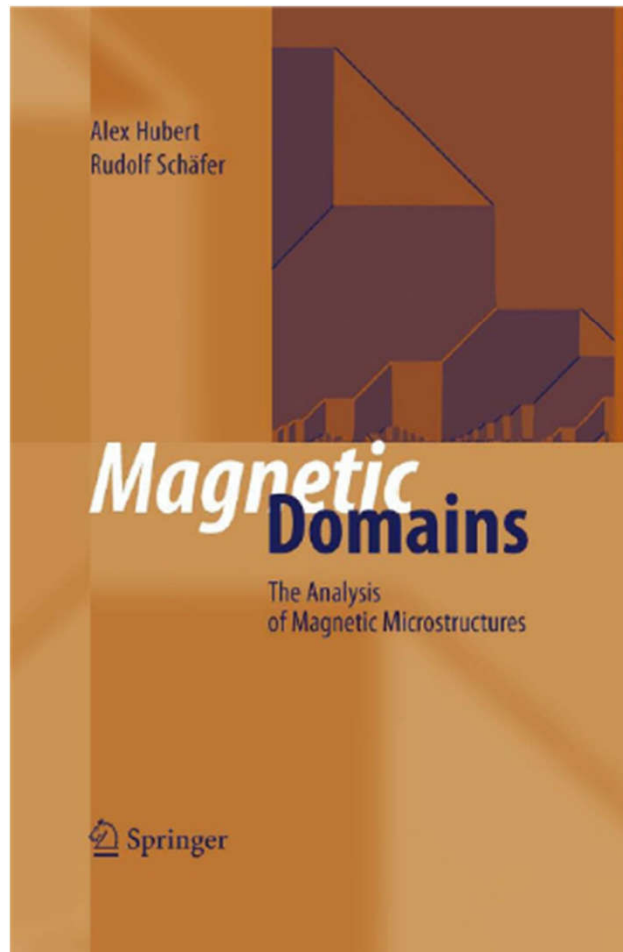
FeNi 6μm square dot → time resolution

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

Others : holography, scattering

Overview

- ↪ Elemental sensitivity
- ↪ Compatible with time resolution
- ↪ Rather versatile





Thank you for your attention !

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<http://fruchart.eu/slides>