

FOR
DUMMIES®



**Magnetic force microscopy :
basics and tricks**

**Imaging nanodevices and
materials at the frontiers of the
NTEGRA microscope**

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<http://www.dummies.com/>

<http://neel.cnrs.fr>



Institut Néel, Grenoble, France.

<http://perso.neel.cnrs.fr/olivier.fruchart/>

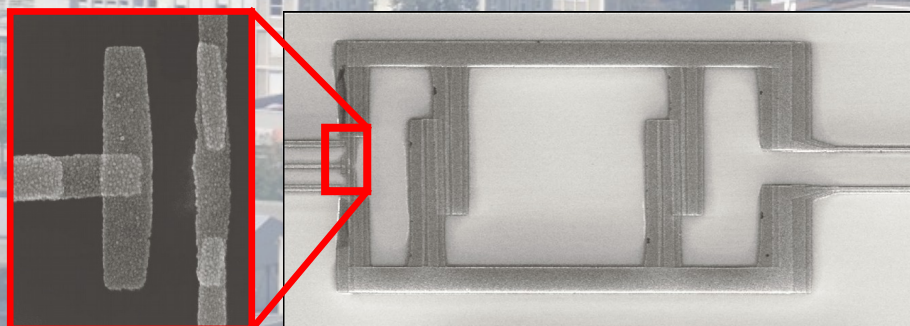


Facts

- ⇒ Core of Condensed Matter Physics
- ⇒ 180 full-staff scientists, 130 technical staff
- ⇒ 40 PhD / year

Internal structure

- ⇒ Research teams + technological support groups
- ⇒ Three scientific departments
 - Condensed Matter – Low Temperatures
 - Condensed Matter – Functional Materials
 - Nanosciences





MAGNETISM.eu

A gateway to the
European community
of magnetism

ESM



The European
School on
Magnetism

Links

Conference,
school, tools,
societies

News

[2014-02-28]

Web site for ESM2015
opens

[2014-02-14]

Tools for magnetism

[2013-08-25]

ESM2015: to be held in Cluj,
Aug-Sep2015

[2013-08-25]

JEMS2013: held 25-30 Aug
2013, Rhodes

JEMS



The Joint European
Magnetic Symposia

JOB MARKET

Open positions related to
Magnetism

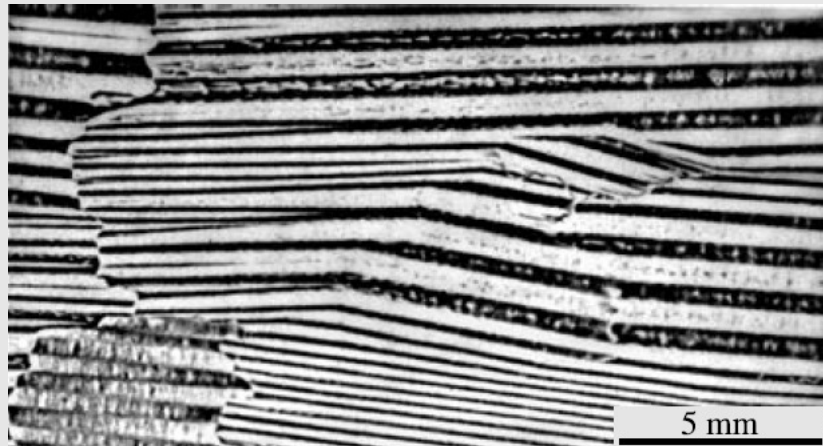


- ➡ **1. Motivation and criteria for magnetic microscopies**
- ➡ **2. Overview of major magnetic microscopies**
- ➡ **3. Theory and working principle**
- ➡ **4. A bit of practice**
- ➡ **5. Some developments at Institut NEEL**



Magnetic domains

Numerous and complex magnetic domains



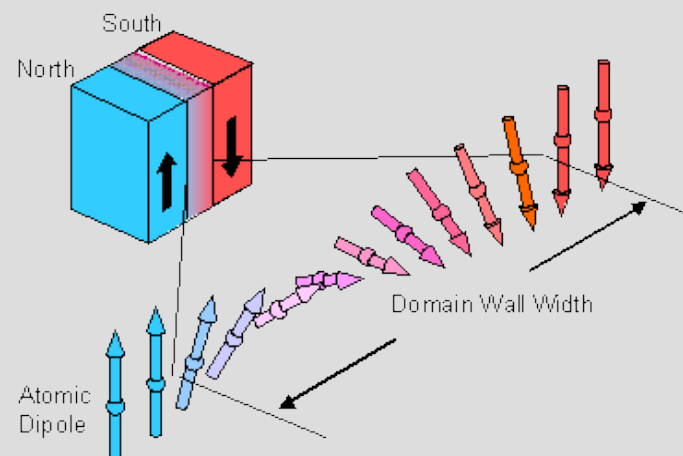
(History : Weiss domains)

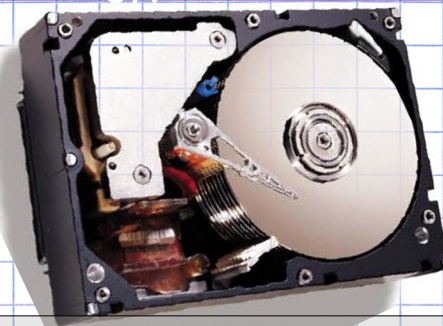
Anisotropy exchange length (domain wall width)

$$E = \underbrace{A \left(\partial_{x_i} m_j \right)^2}_{\text{Exchange } J/m} + \underbrace{K \sin^2 \theta}_{\text{Anisotropy } J/m^3}$$

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

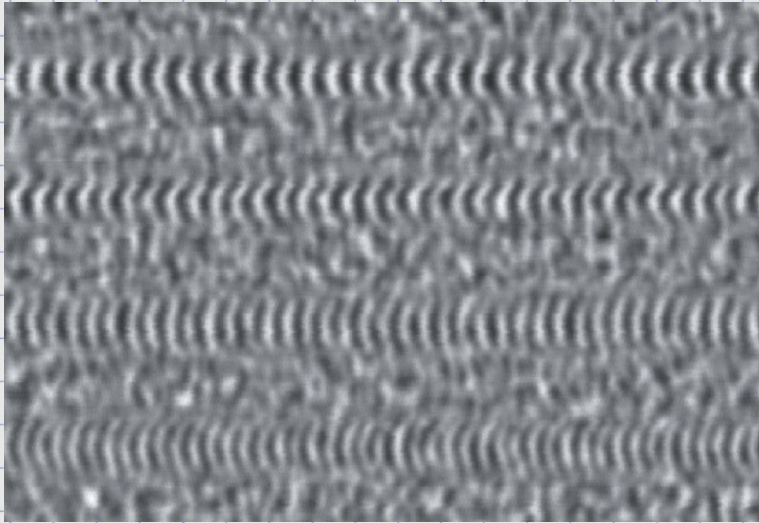
$\Delta_u \approx 1 \text{ nm}$ → $\Delta_u \geq 100 \text{ nm}$
 Hard Soft





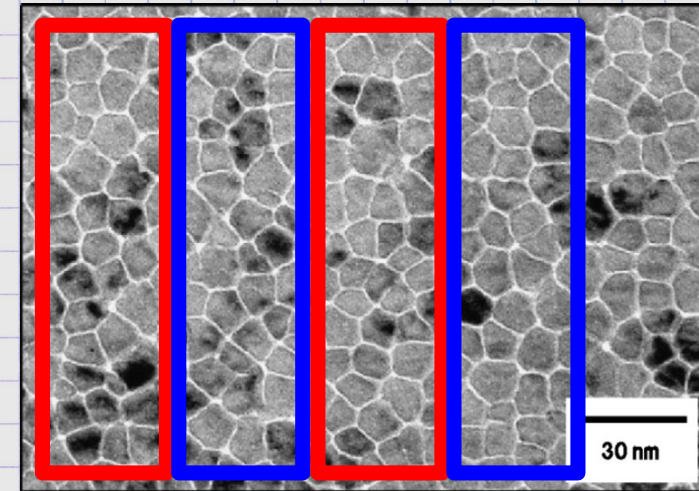
Magnetic bits on Hard Disk Drive

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



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

Underlying microstructure



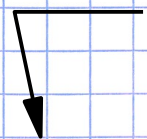
S. Takenori, J. Magn. Magn. Mater. 321, 562 (2009)

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:

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

γ_0 Gyromagnetic factor

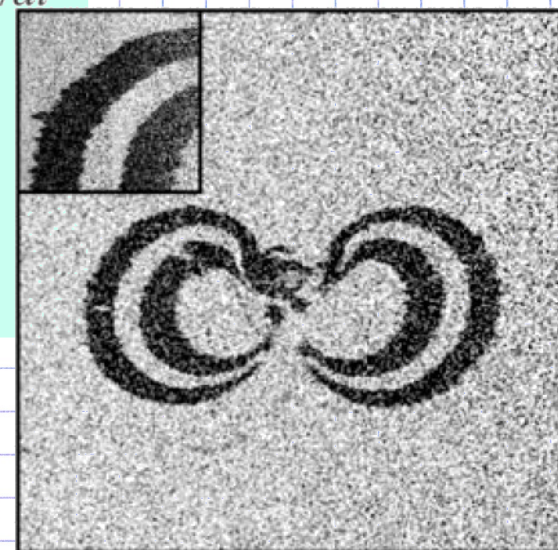
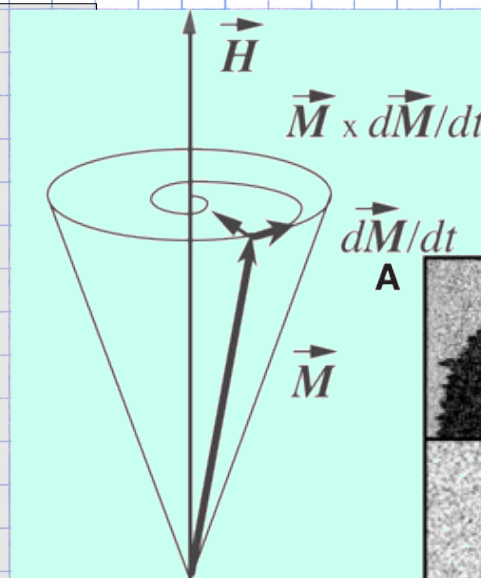
$$\gamma_0 = \mu_0 \gamma \quad \gamma = \frac{gq}{2m}$$

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

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

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

Demonstration: 1999

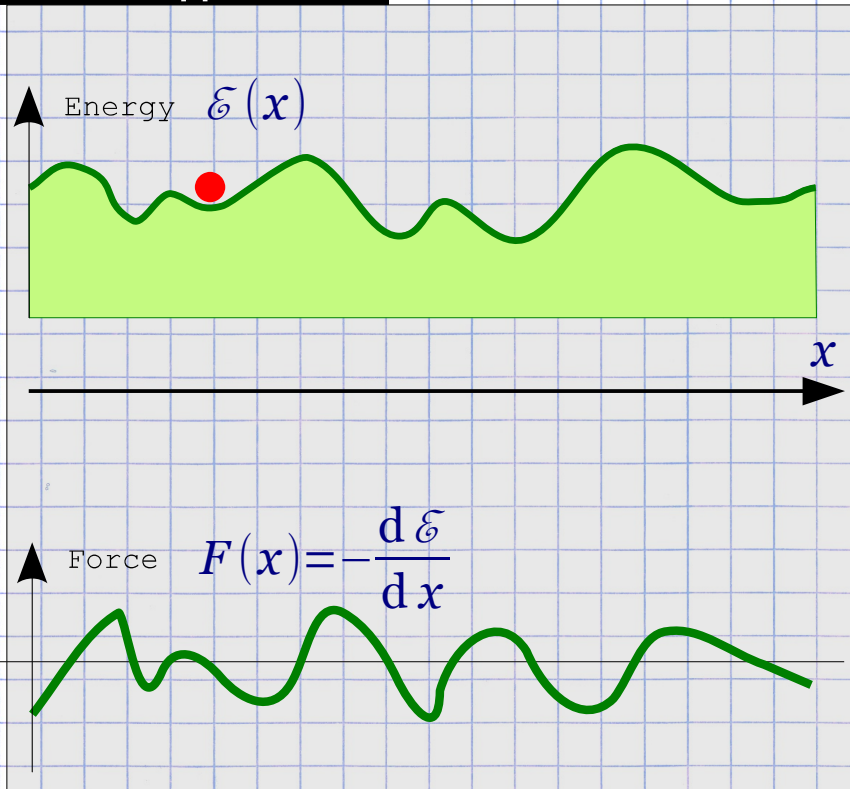


150 μm

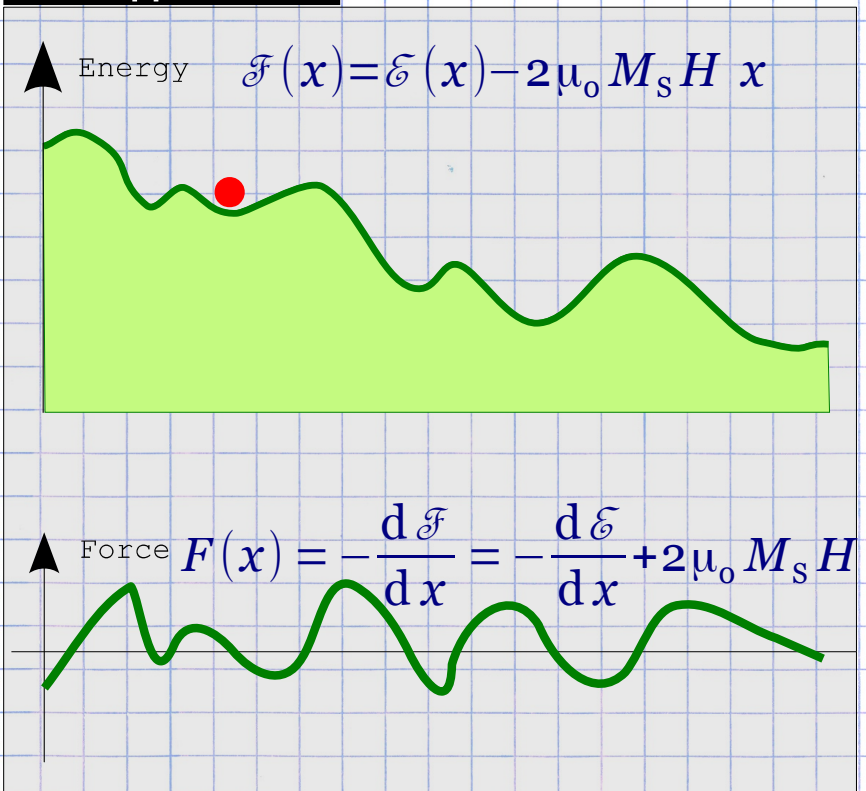
C. Back et al., Science 285, 864 (1999)

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 ?

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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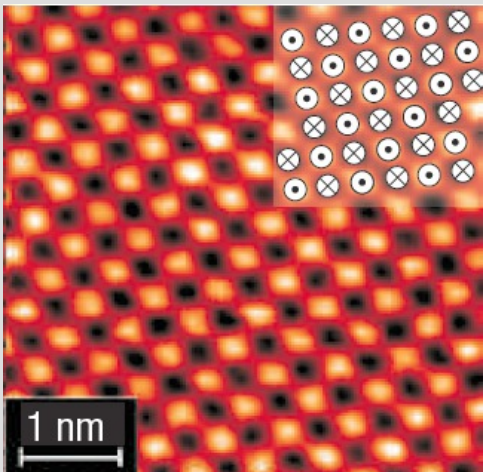
See : slides on techniques for characterizing magnetic nano-objects :

<http://perso.neel.cnrs.fr/olivier.fruchart/slides/tech/tech-fruchart-gdr2009.pdf>



Spin-polarized STM

Fe(1ML)/W(001)



Antiferromagnetic domain

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

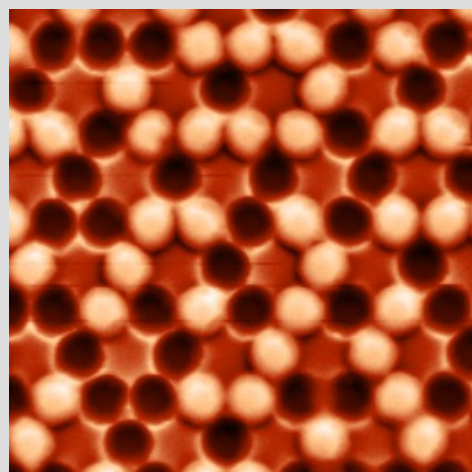
REVIEW :

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

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

Magnetic Force Microscopy

Array of dots



Up-and-down 'single-domains'

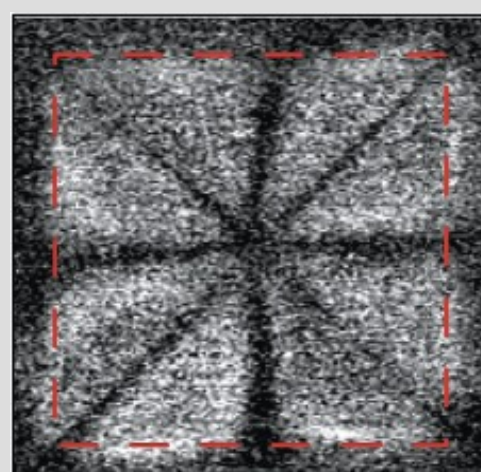
NEEL, sample courtesy:
N. Rougemaille, I. Chioar

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

Overview

↪ Large variety
↪ Rather slow

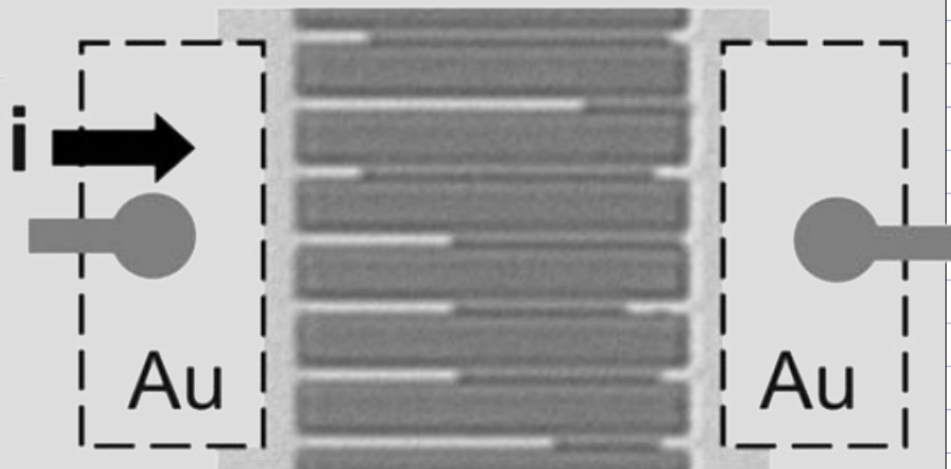


Principle

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

Example

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



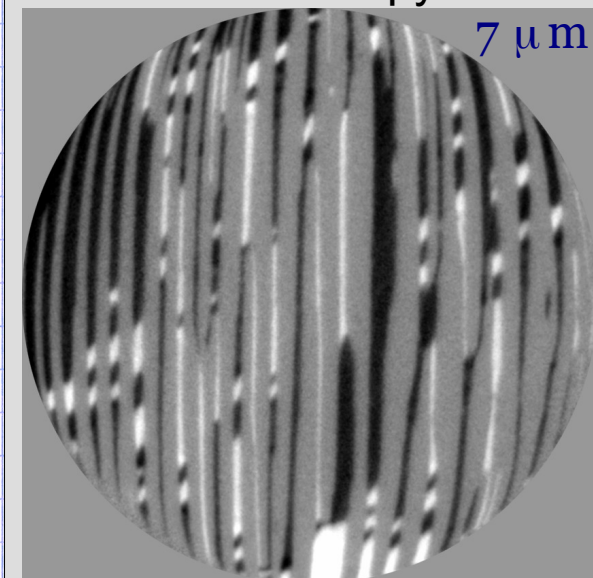
@NEEL : T. A. Moore et al., Appl. Phys. Lett 93, 262504 (2008)

Overview

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

SPLEEM

Spin-Polarized Low Energy Electron Microscopy

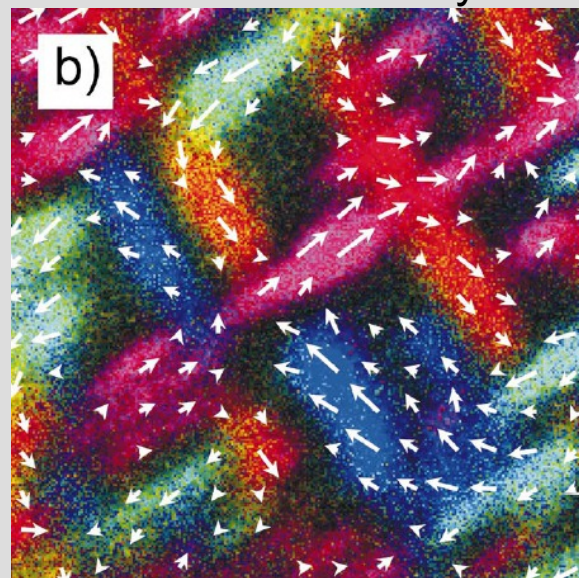


Stripes of Fe/W(110)

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

SEMPA

Scanning Electron Microsc.
with Polarization Analysis

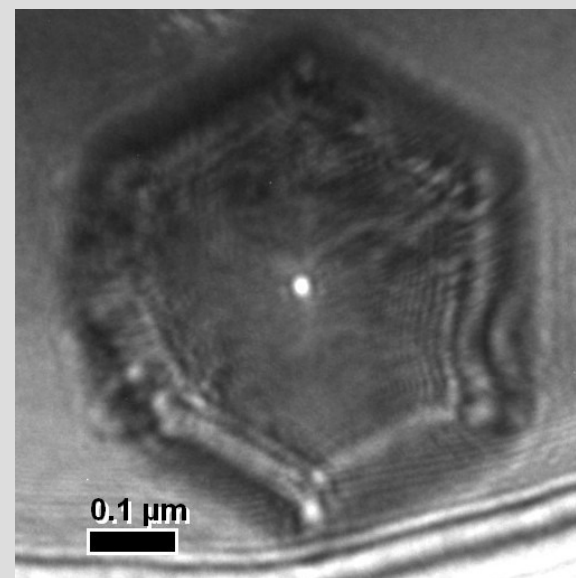


Maze of Fe/W(001) 1.5 μm

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., in preparation

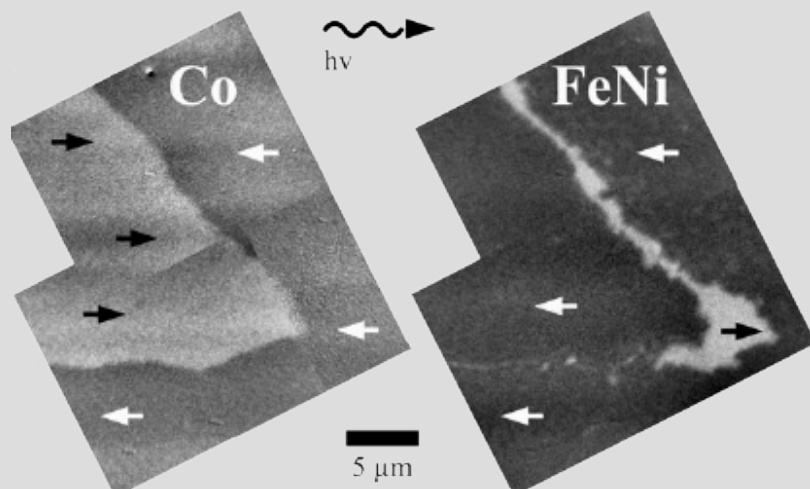
Overview

- ↕ 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

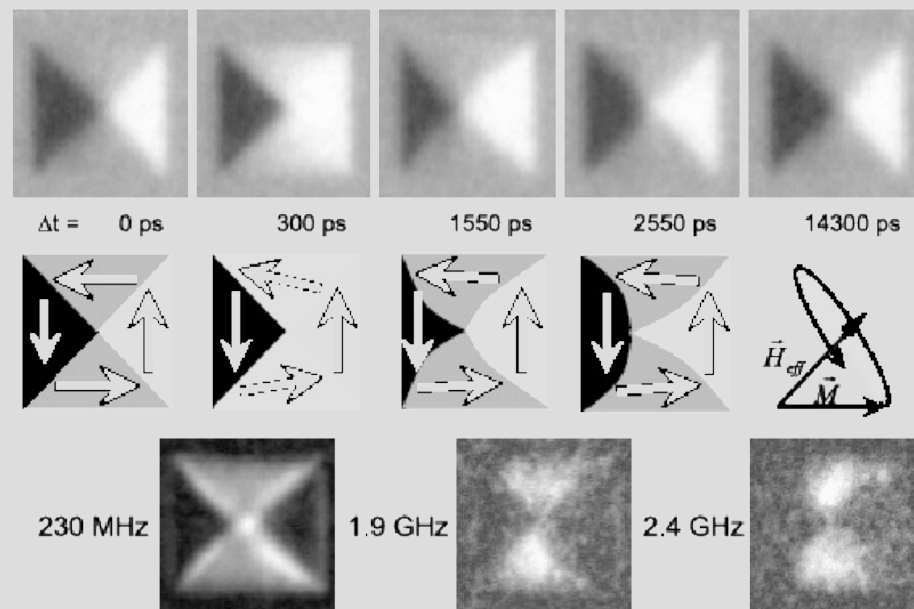
→ elemental resolution

@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)

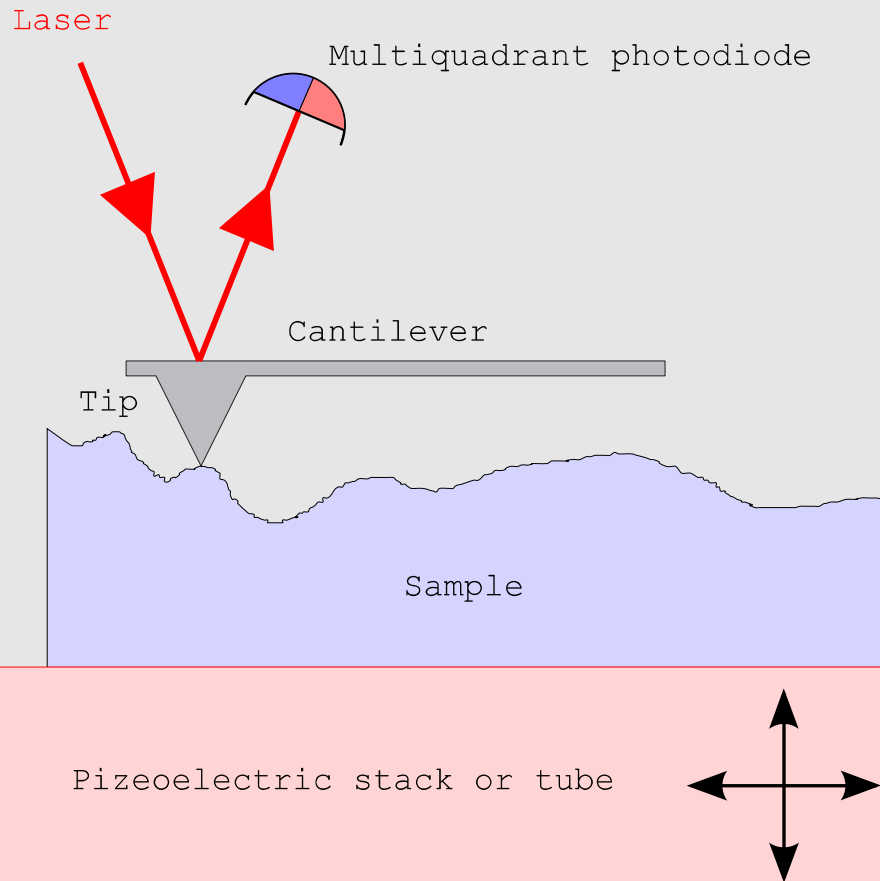
Others : holography, sca

Overview

↻ Elemental sensitivity
↻ Compatible with time resolution
↻ Rather versatile

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Key elements of an Atomic Force Microscope (AFM)

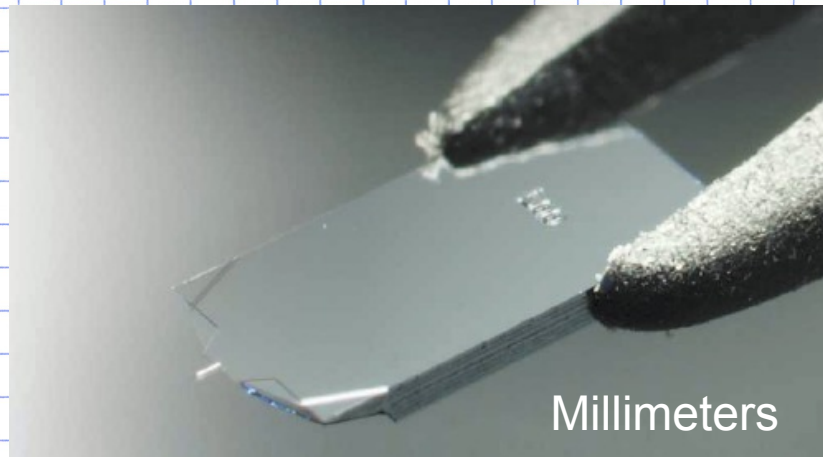


G. Binnig et al., Phys. Rev. Lett. 56, 930-933 (1986)

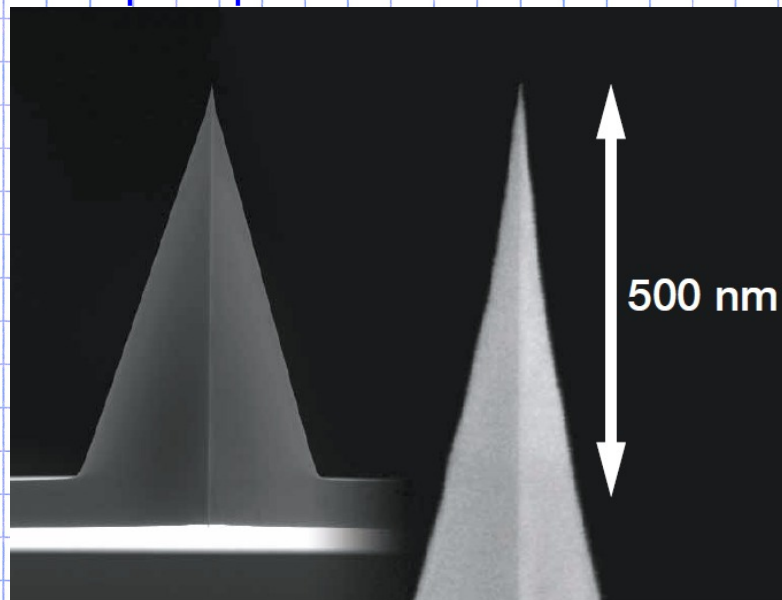
Overview

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

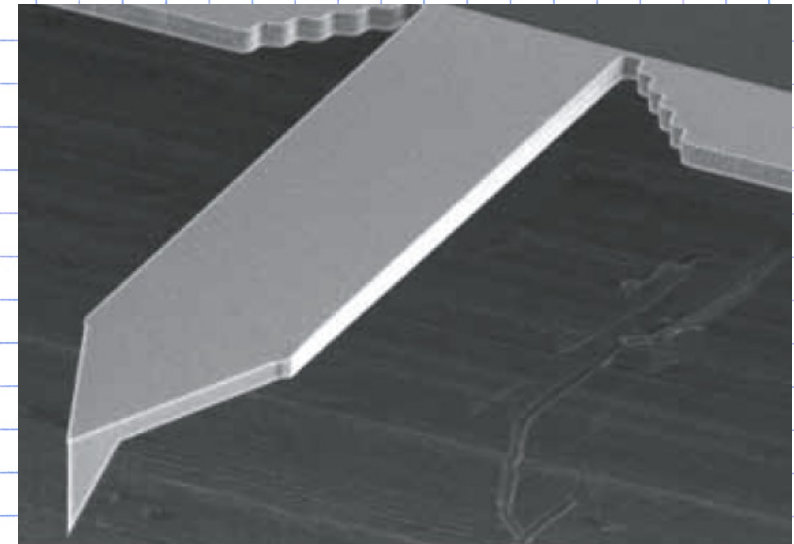
Chip – Batch fabrication



Full tip + apex



Cantilever

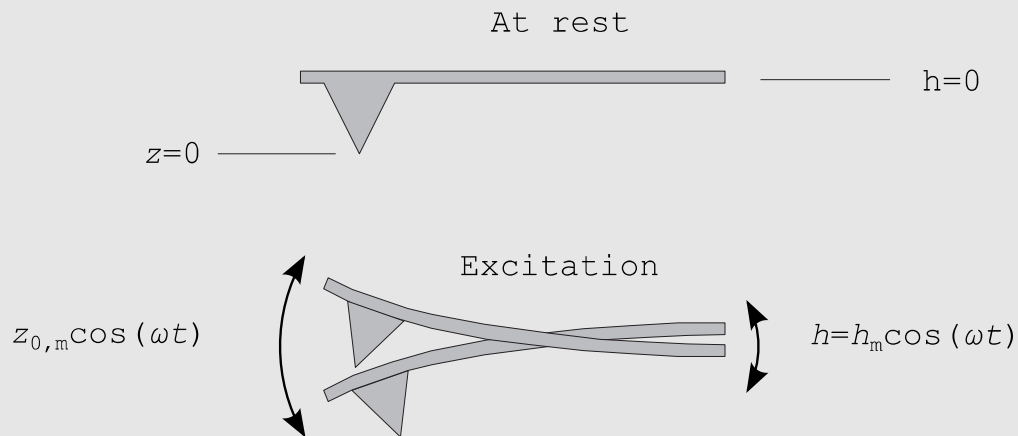


Overview

- ↻ Price 10-200eur/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

$$\omega_0 = \sqrt{\frac{k}{m}}$$

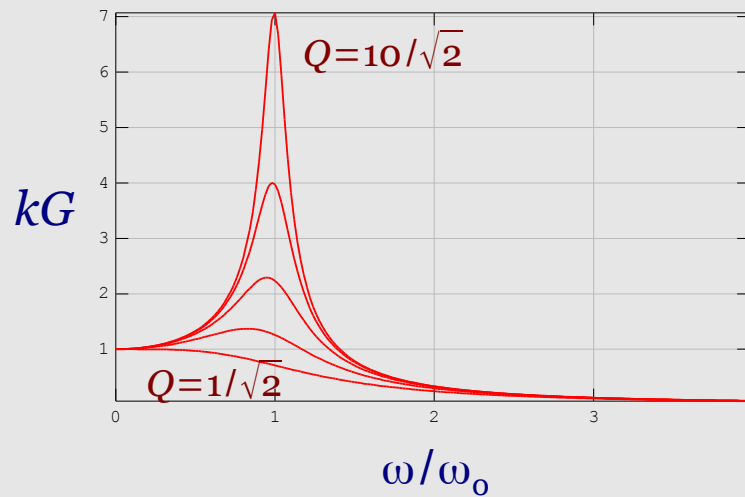
Quality factor

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

⇒ 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

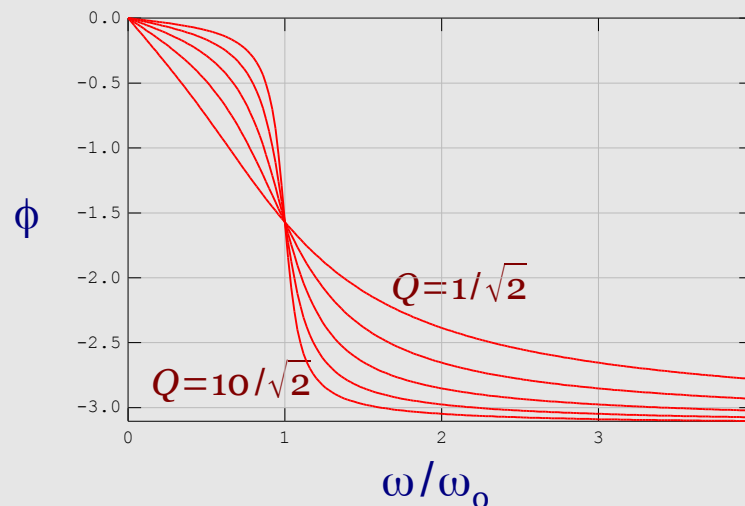


$$G=|H|=\frac{1}{k} \frac{1}{\sqrt{\left[1-\left(\frac{\omega}{\omega_0}\right)^2\right]^2 + \frac{1}{Q^2}\left(\frac{\omega}{\omega_0}\right)^2}}$$

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

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

Dephasing



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

$$\phi(0)=0$$

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

$Q \gg 1$

$$\omega_r \approx \omega_0$$

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

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

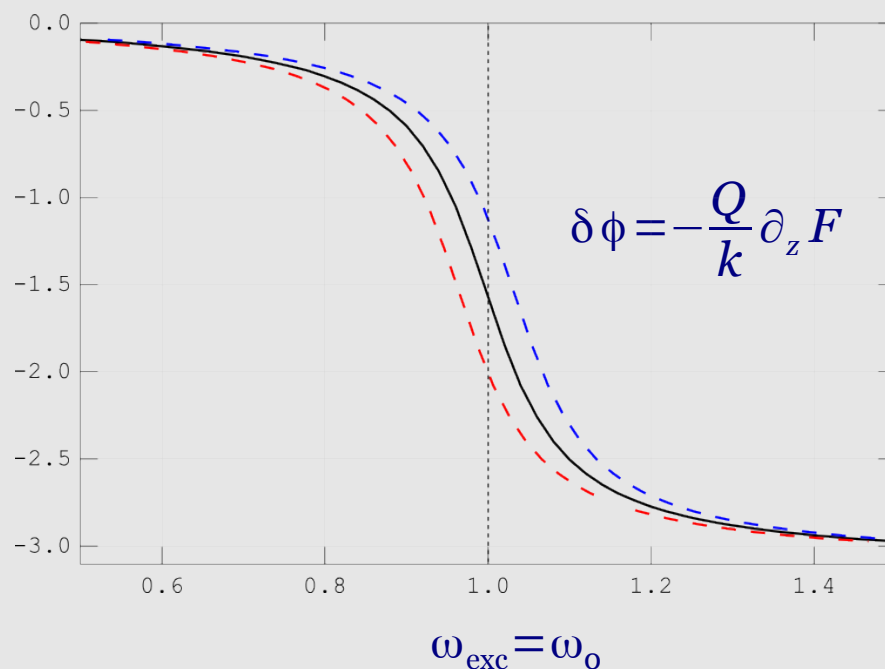


Tip-sample interaction treated as perturbation

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

⇒ Mere renormalization : $\omega_{\text{o,eff}} = \omega_0 \left(1 - \frac{1}{2k} \partial_z F \right)$

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

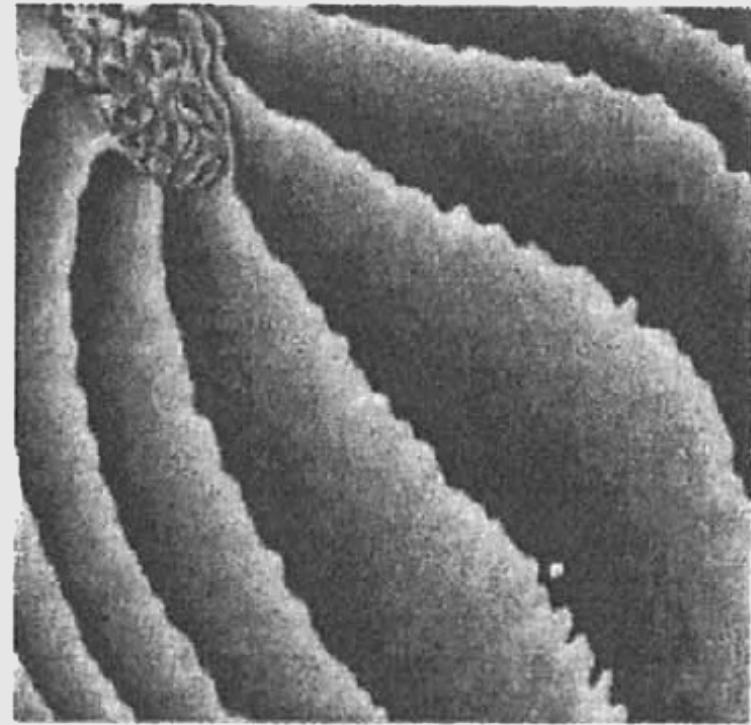
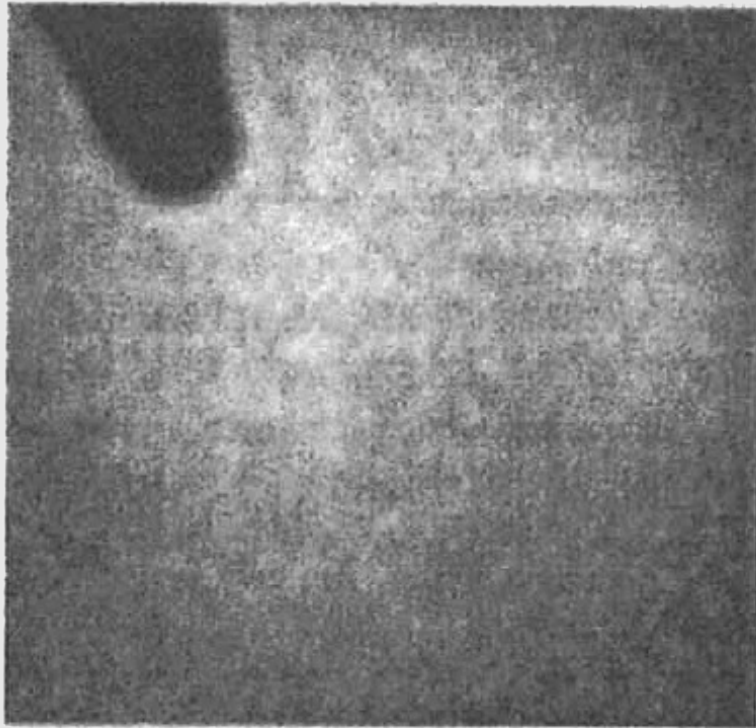
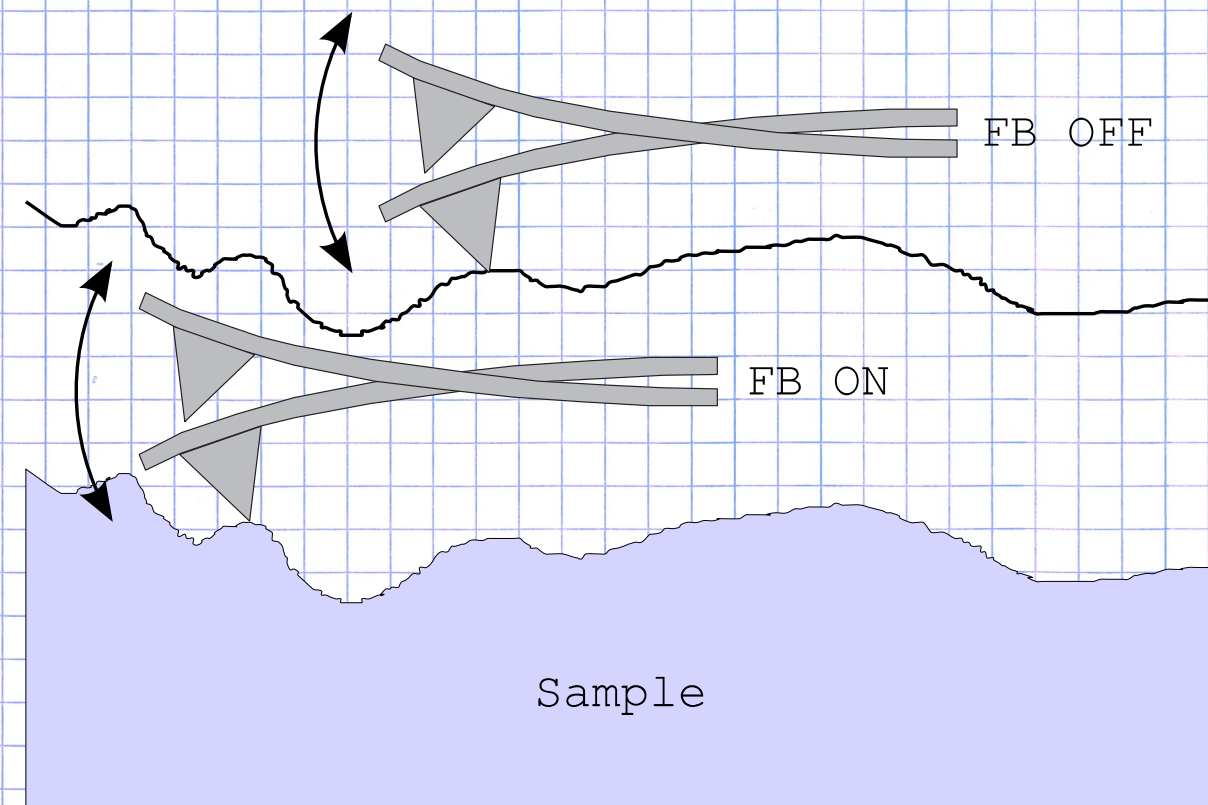


Figure 11-20: The electron amplitude (left) and phase (right) near an MFM tip visible as a dark shadow on the upper left corner of the left image.

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



Second pass

⇒ Monitor long-range forces (magnetic, electrostatic)



First-pass

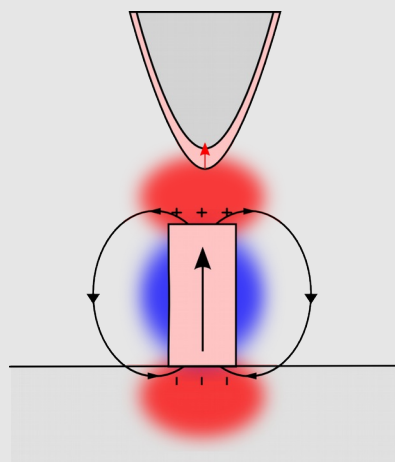
⇒ Feedback ON
⇒ Monitor topography (height) and any other signal (phase etc.)

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)**

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Tip is a dipole

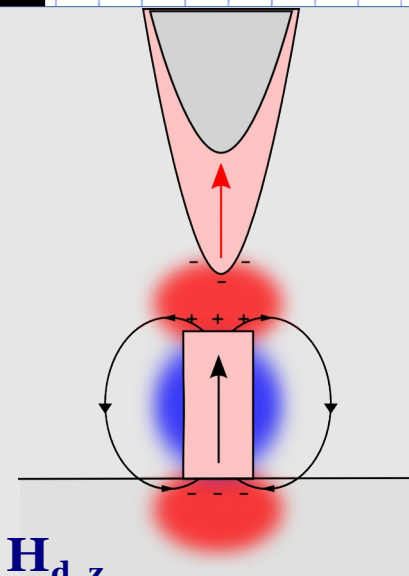


$$\mathcal{E}_{1,2} = -\mu_0 \mu_2 \cdot \mathbf{H}_d$$

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

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

Tip is a monopole



$$\mathcal{E}_{1,2} = \mu_0 \sigma \cdot \varphi$$

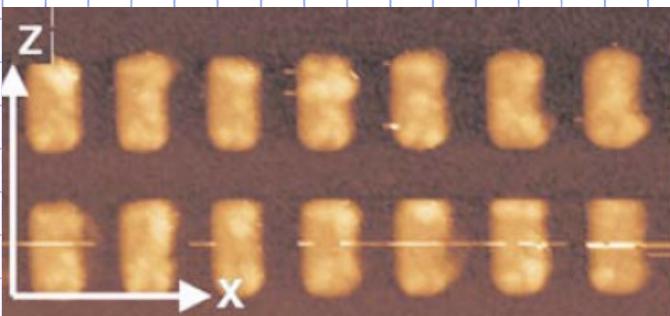
$$\mathbf{F}_z = -\mu_0 \sigma \mathbf{H}_{d,z}$$

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

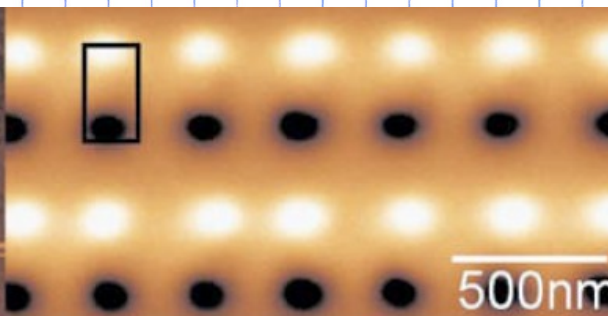
- ↪ In practice, a combination of both models is best suited (dipole is more important)
- ↪ MFM is sensitive to some derivative(s) of the stray field from the sample
- ↪ MFM may be sensitive to in-plane field, depending on the tip magnetic moment

4. PRACTICE / Single-domain nanostructures (planar)

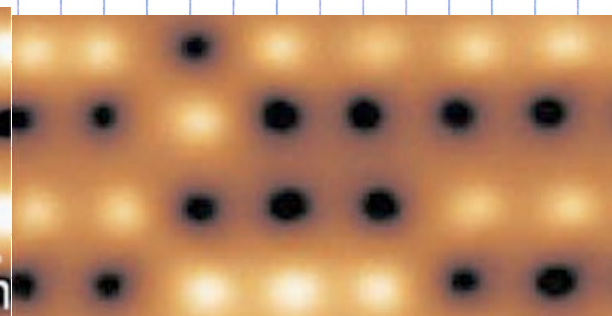
Topography



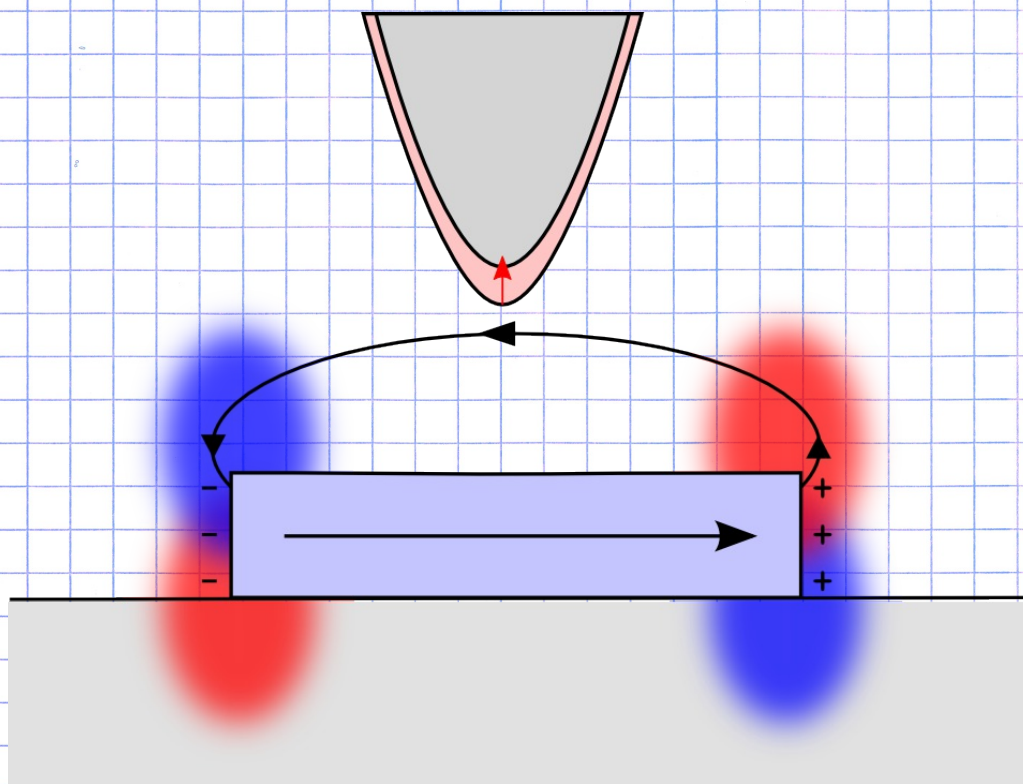
MFM, saturated



MFM, partly reversed



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



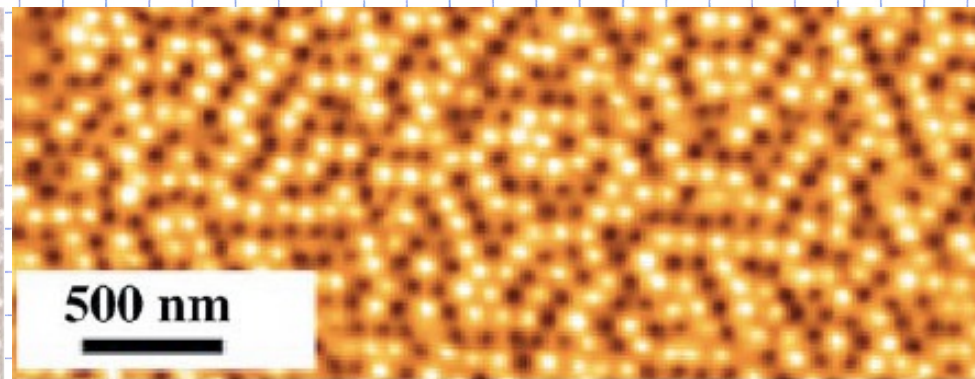
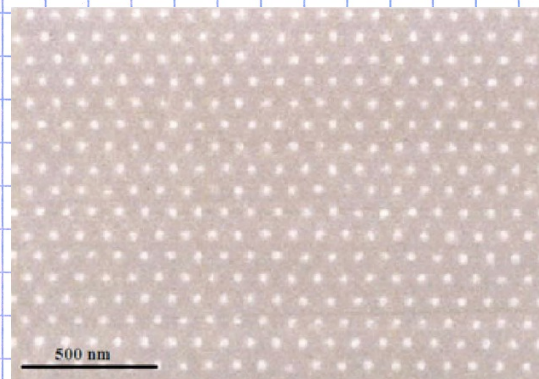
Single-domain in-plane magnetized dots appear as dipoles

4. PRACTICE / Single-domain nanostructures (perpendicular)



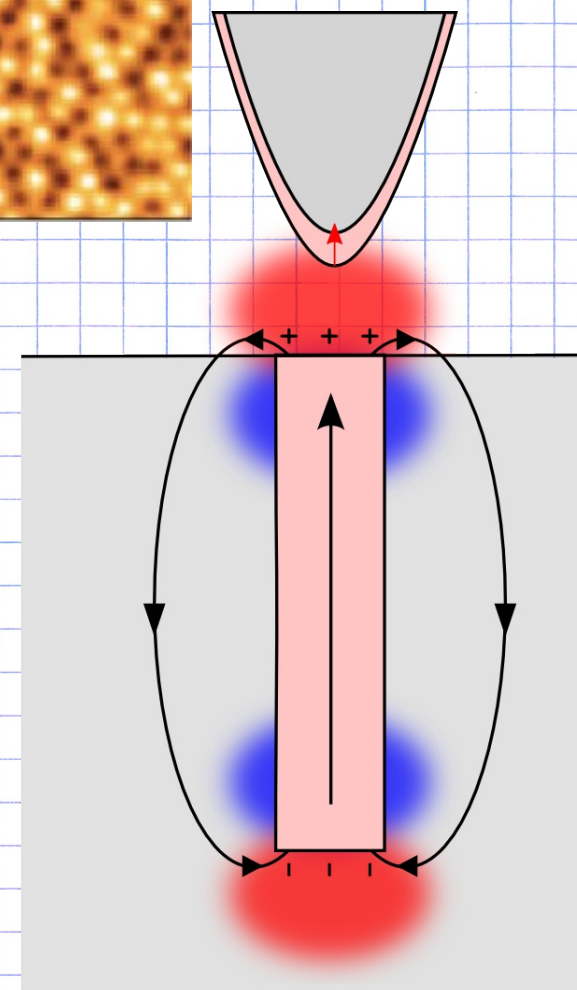
Structure (SEM)

MFM, partly reversed

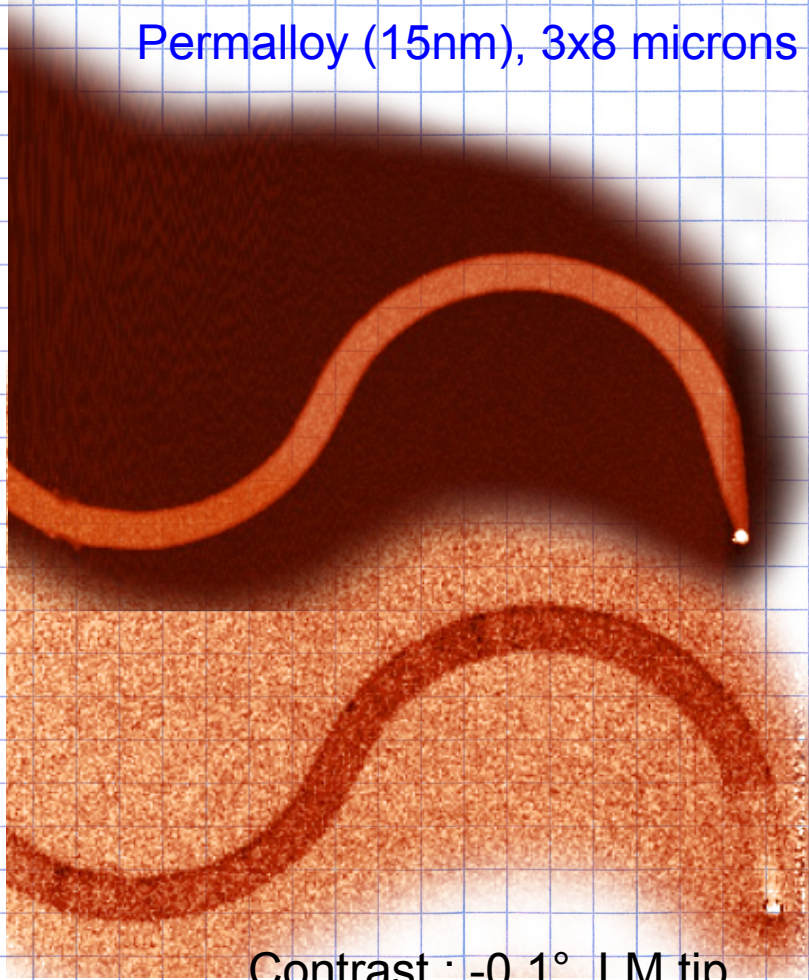


T. Wang et al., APL 92, 192504 (2008)

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



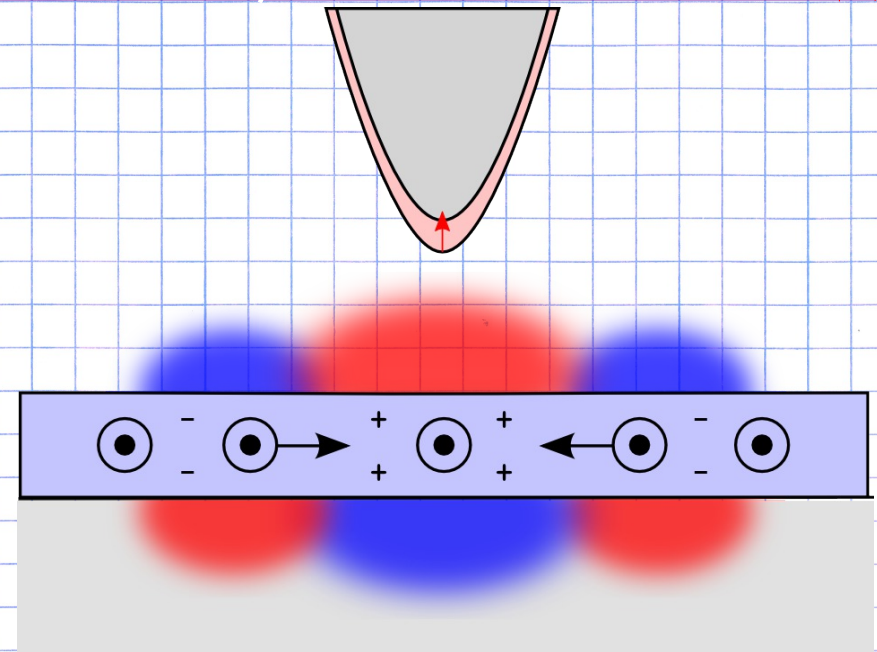
Permalloy (15nm), 3x8 microns



Contrast : -0.1° , LM tip

Lithography : S. Pizzini (Institut Néel)

Imaging : Z. Ishaque (Institut Néel)



Principle :

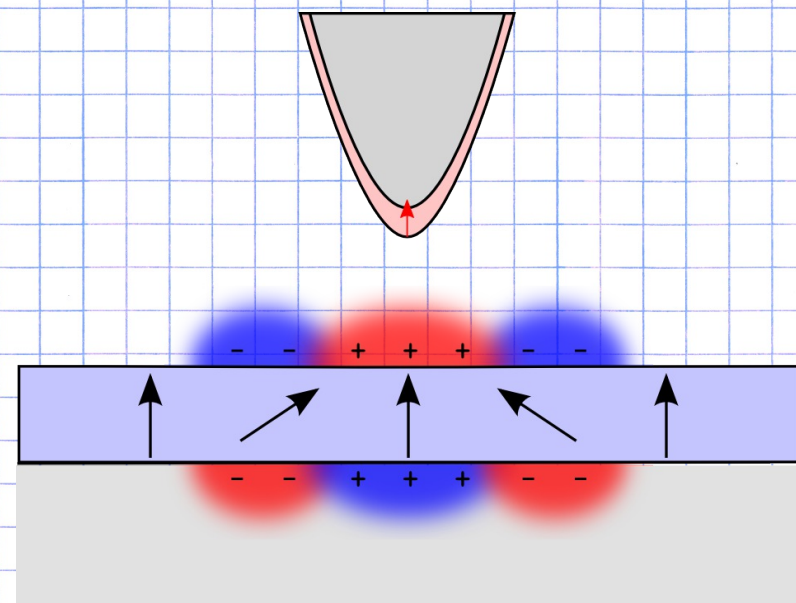
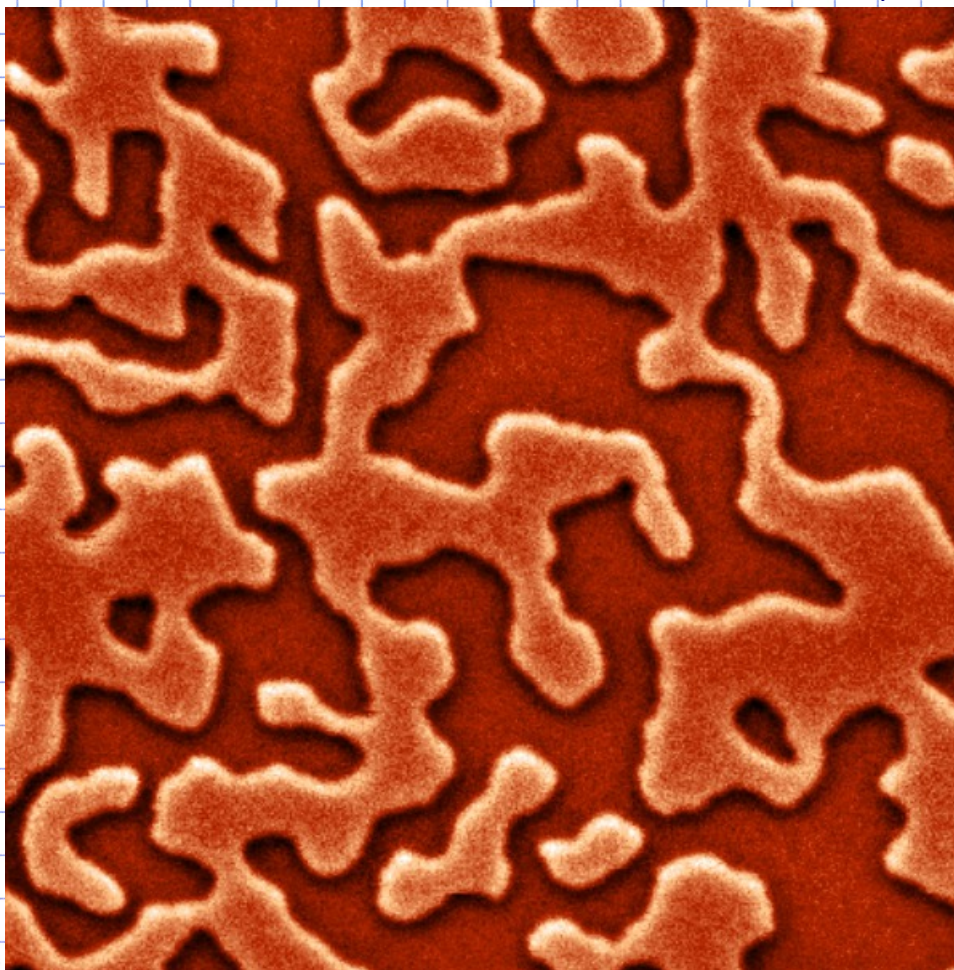
1. Stray field magnetizes sample
2. Sample is non-uniform $\rightarrow$ stray field
3. Tip measures sample's stray field

 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



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

Sample : A. Marty (CEA-Grenoble)

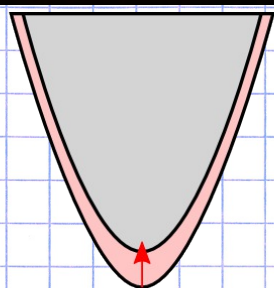
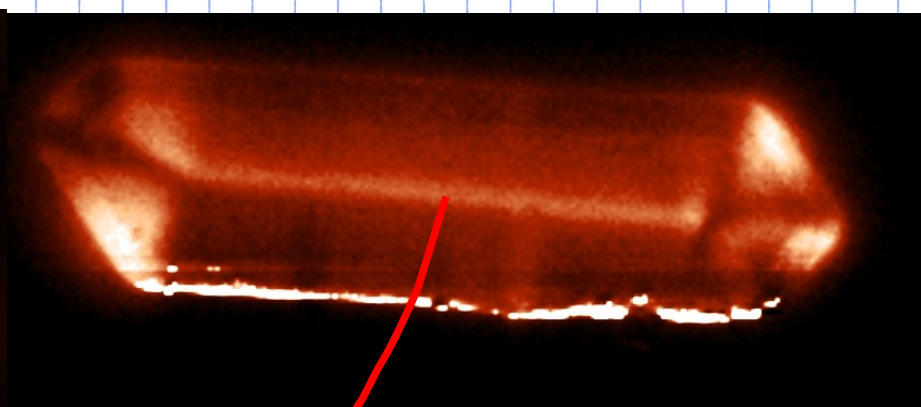
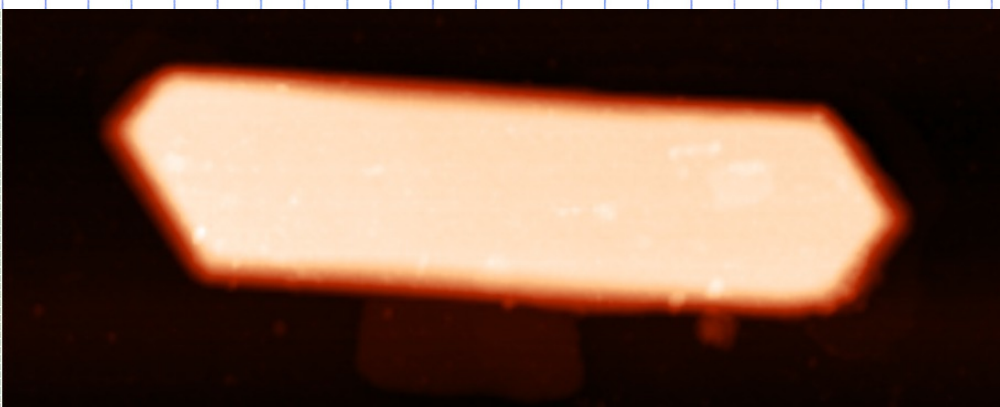
Imaging : M. Darques (Institut Néel)

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

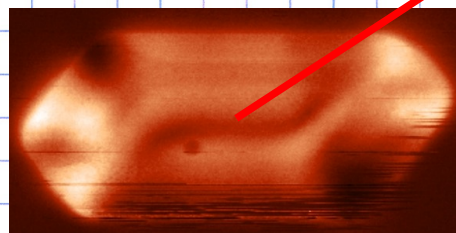
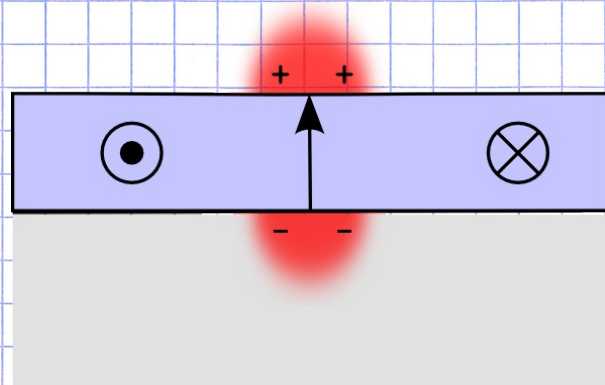
Quantitative analysis :

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

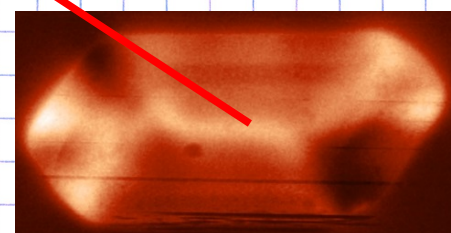
Fe dot (25nm), 2.5x1 microns



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



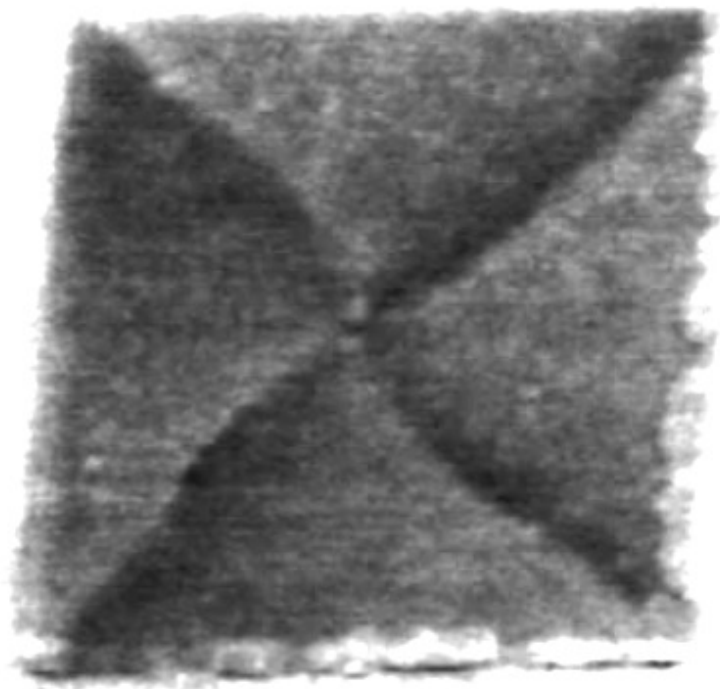
Down core



Up core

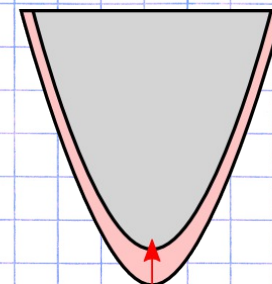
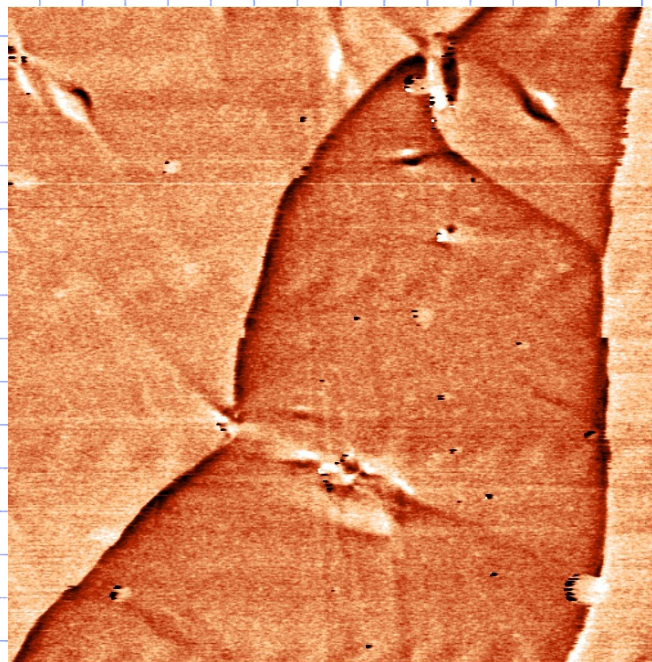


Permalloy dot (16nm)
2x2 microns

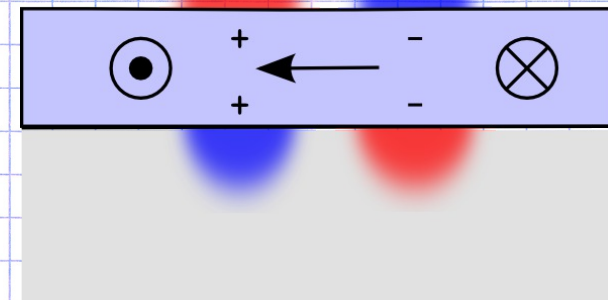


J. M. Garcia et al., APL 79, 656 (2001)

Permalloy film (20nm)
10x10 microns

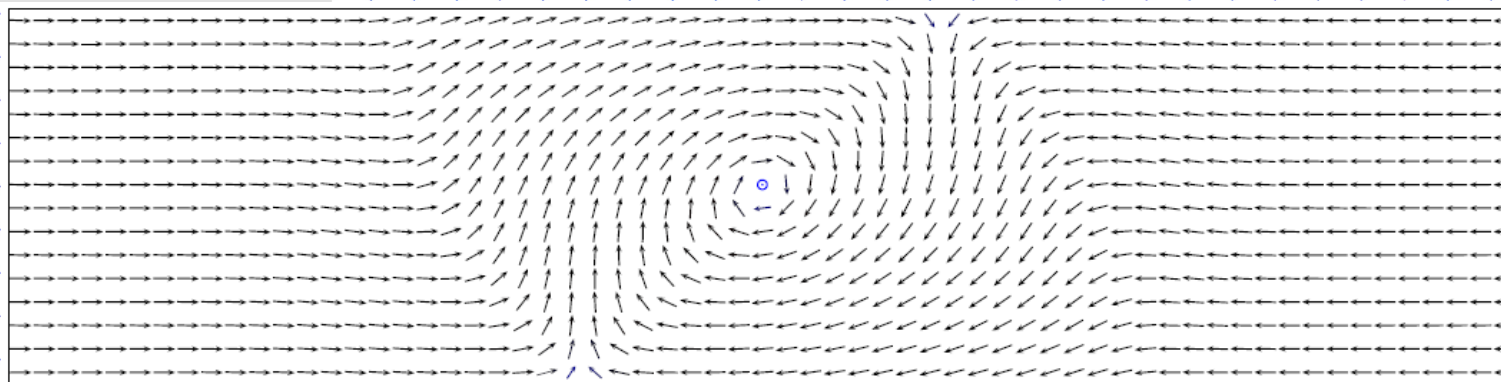
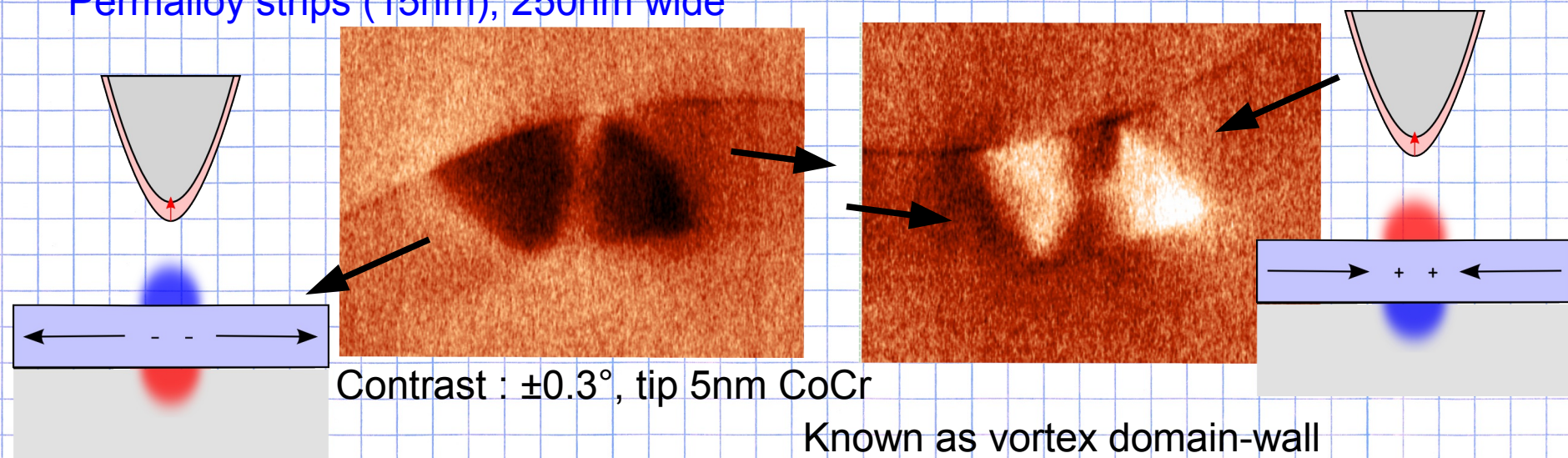


➤ Néel wall give rise to DIPOLAR contrast
➤ Informs about the chirality of the wall core



4. PRACTICE / Head-to-head domain walls

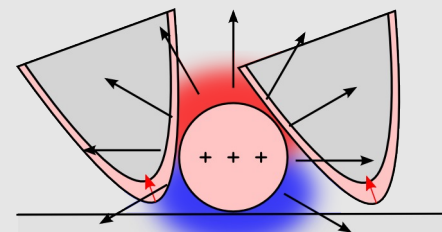
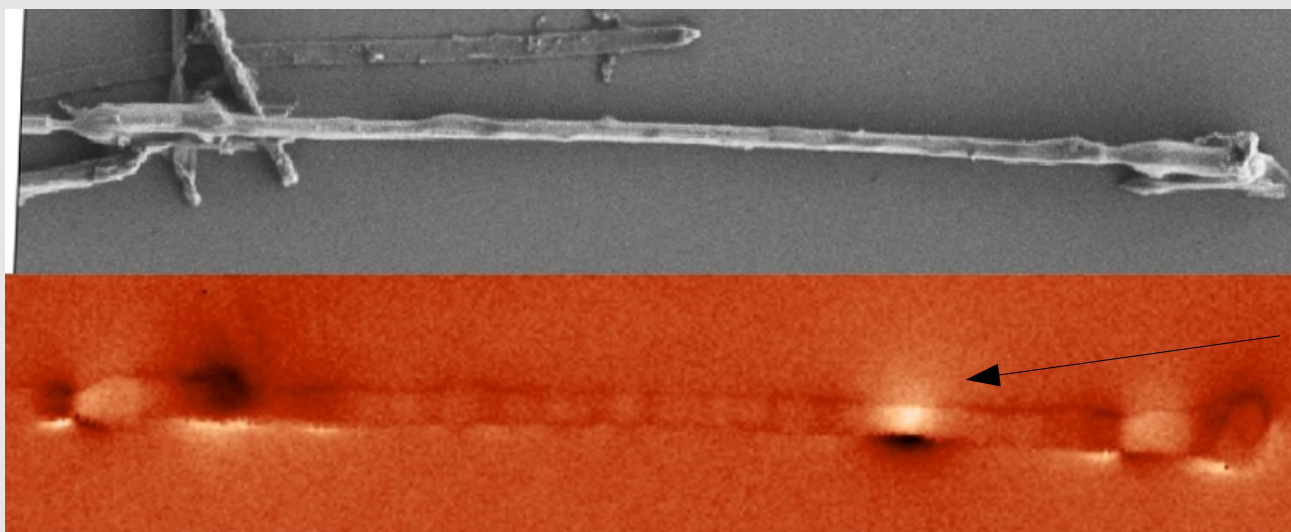
Permalloy strips (15nm), 250nm wide



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

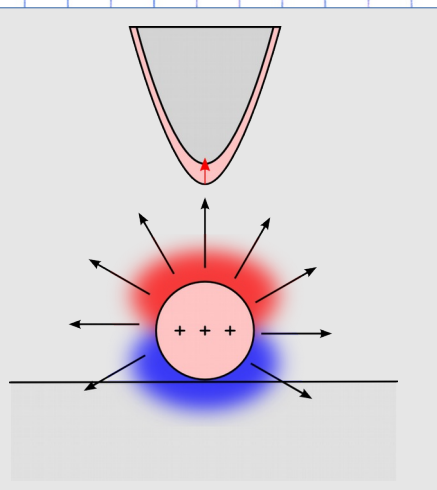
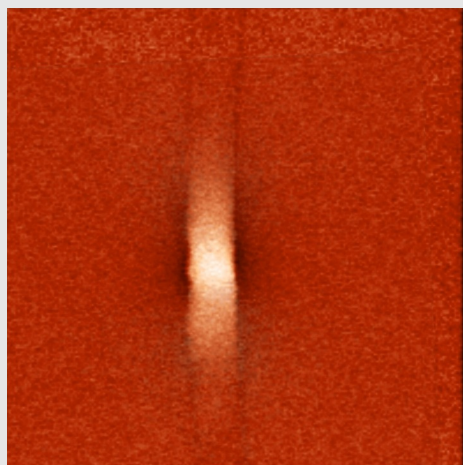
↻ Walls in in-plane magnetized stripes → MONOPOLAR
↻ Contrast informs about head-to-head or tail-to-tail

Tilted cantilever, across wire

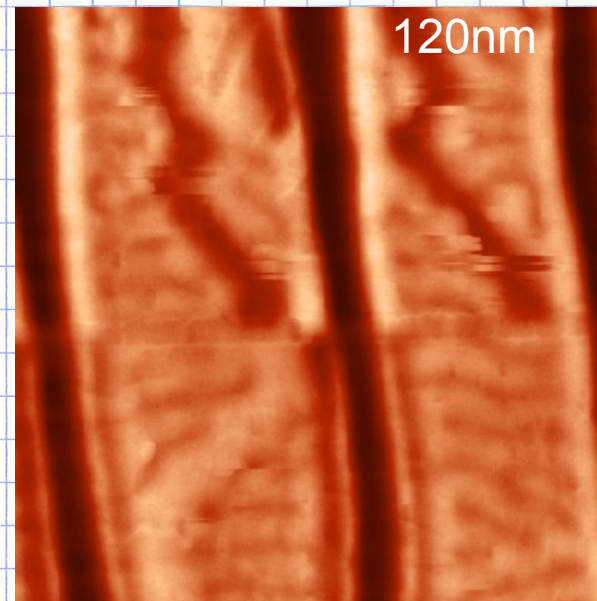
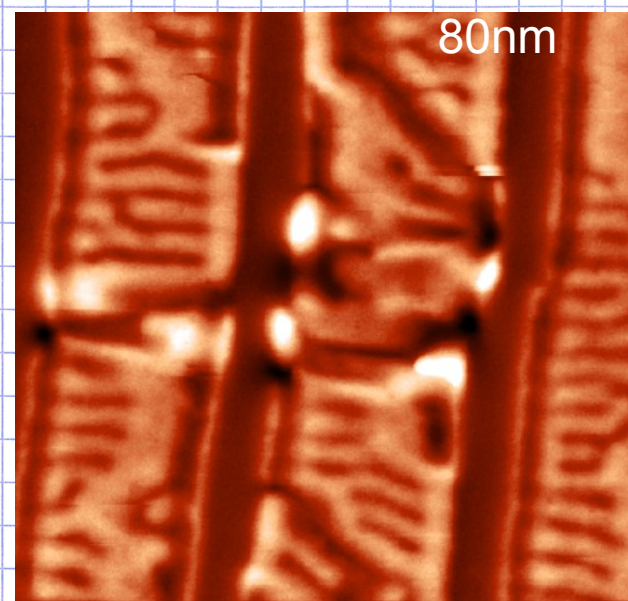
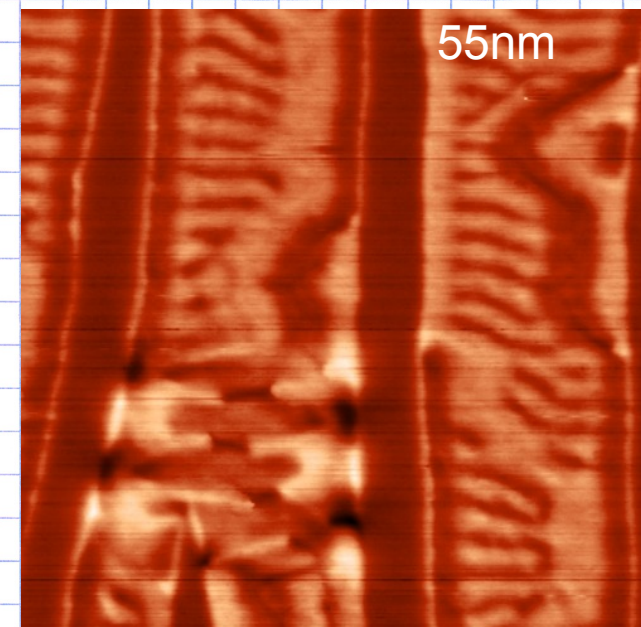
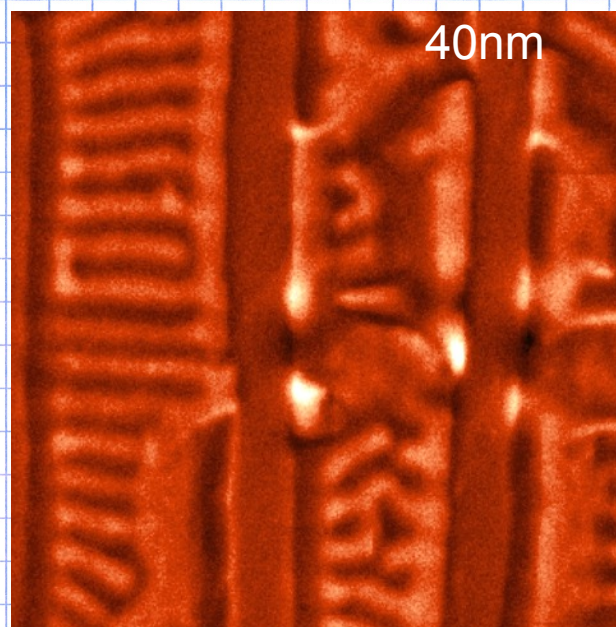
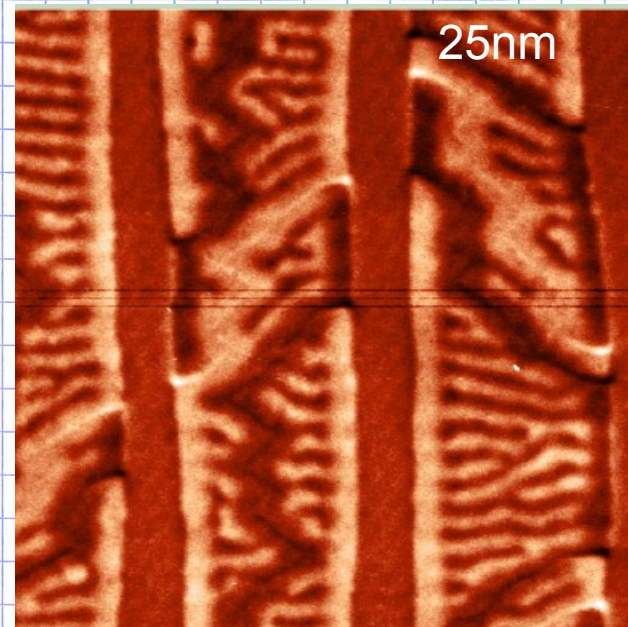


Tilted cantilever, along wire

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



- ➔ **1. Motivation and criteria for magnetic microscopies**
- ➔ **2. Overview of major magnetic microscopies**
- ➔ **3. Theory and working principle**
- ➔ **4. A bit of practice**
- ➔ **5. Some developments at Institut NEEL**



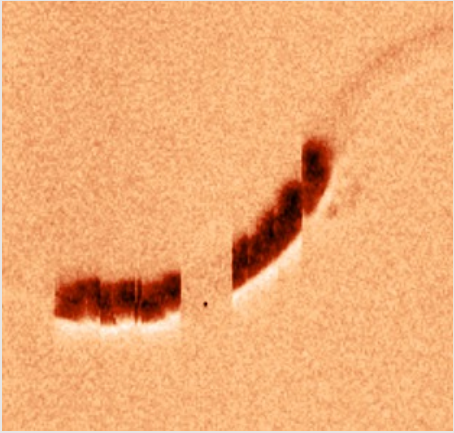
↪ Compare tips !
↪ Use identical sample
and imaging parameters

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

High

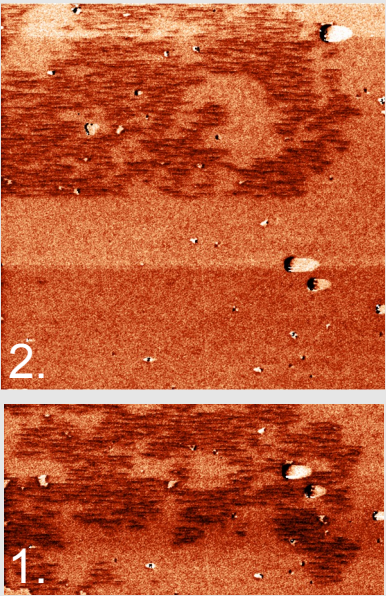
Scanning ↑

Attraction



Permalloy (15nm)
500nm strips

Sample : S. Pizzini
Imaging : Z. Ishaque



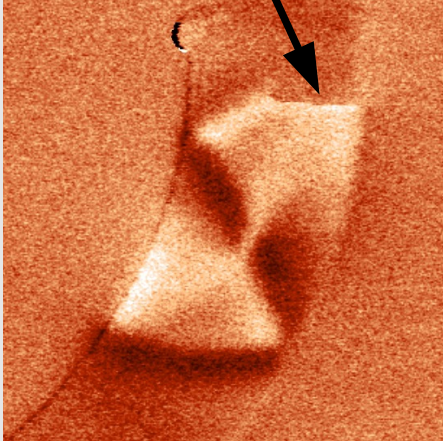
2.
1.

Co (0.6nm)/
graphene (20 mic)

C. Dieudonné

Moderate

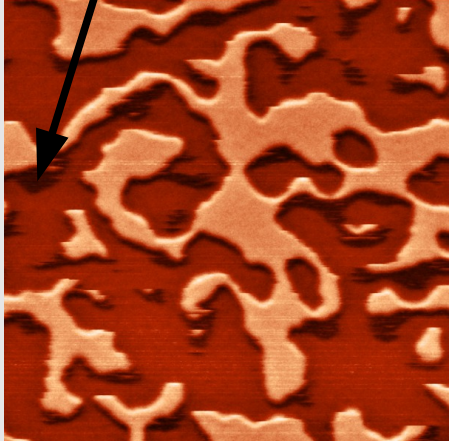
Repulsion



Permalloy (15nm)
500nm wide

Sample : S. Pizzini
Imaging : Z. Ishaque

Attraction



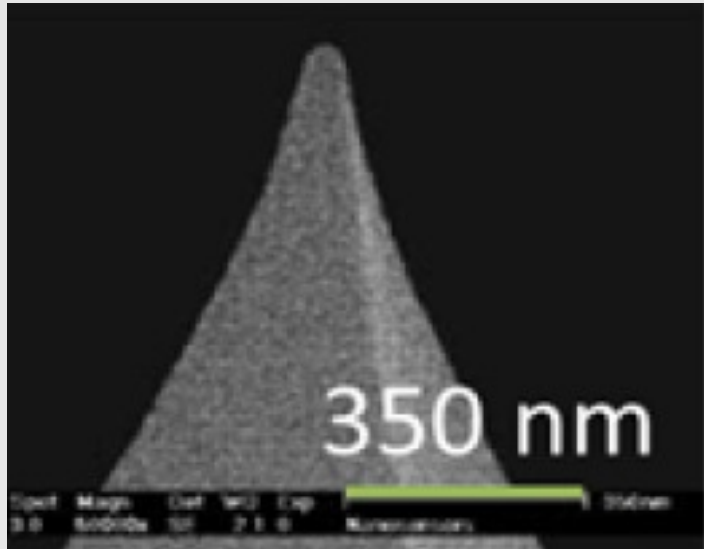
FePt (4nm)
Image 5 microns

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

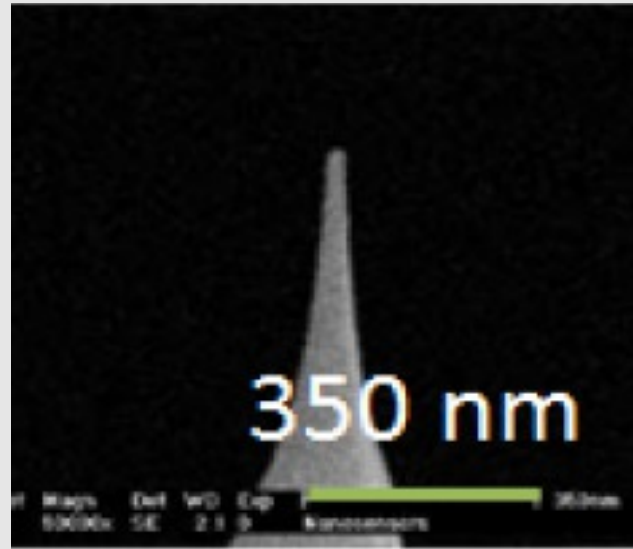
Improve resolution

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

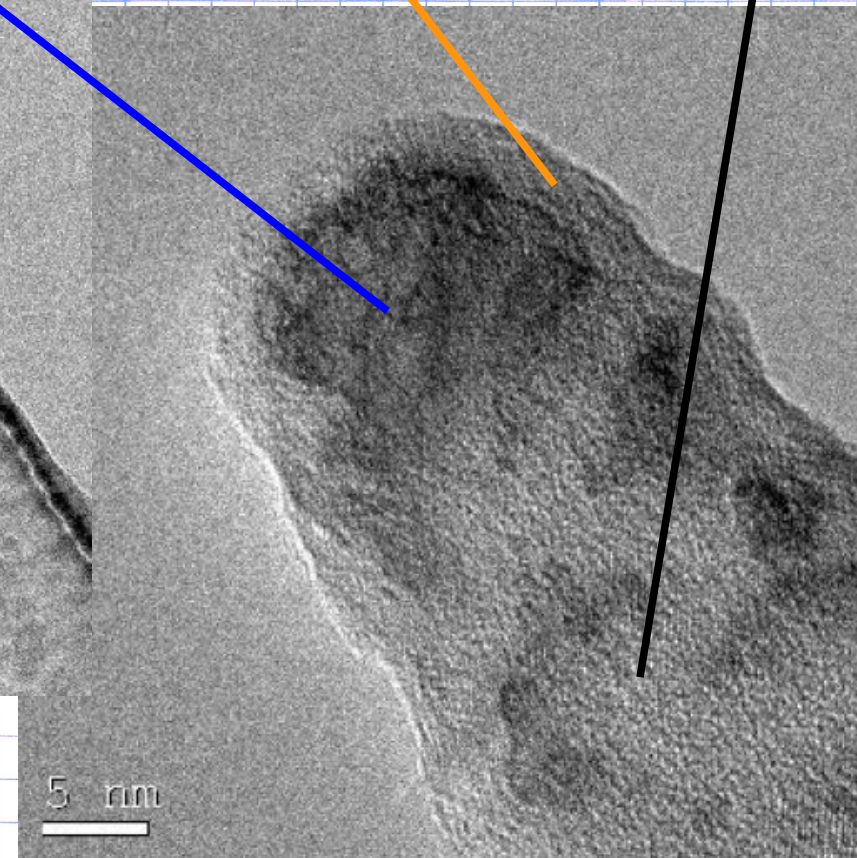
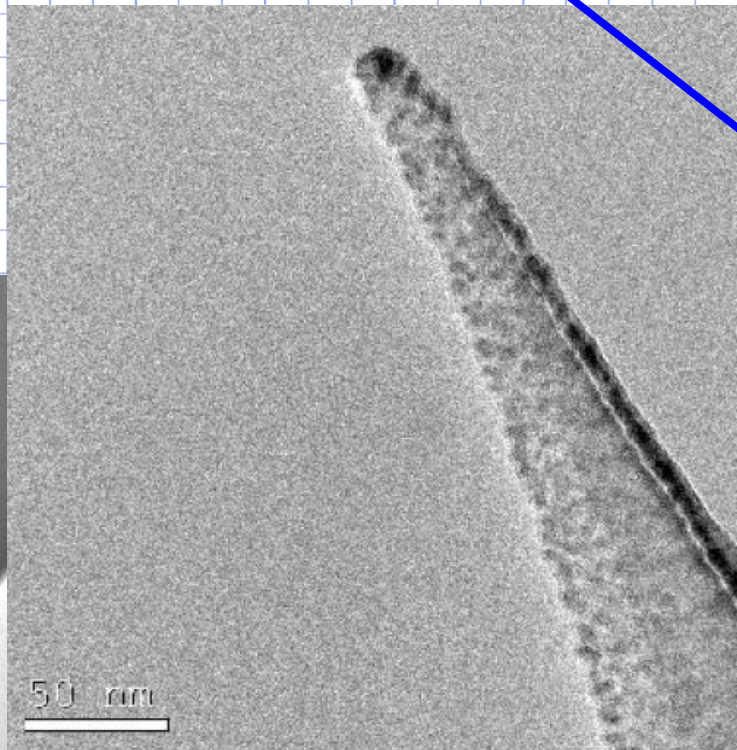
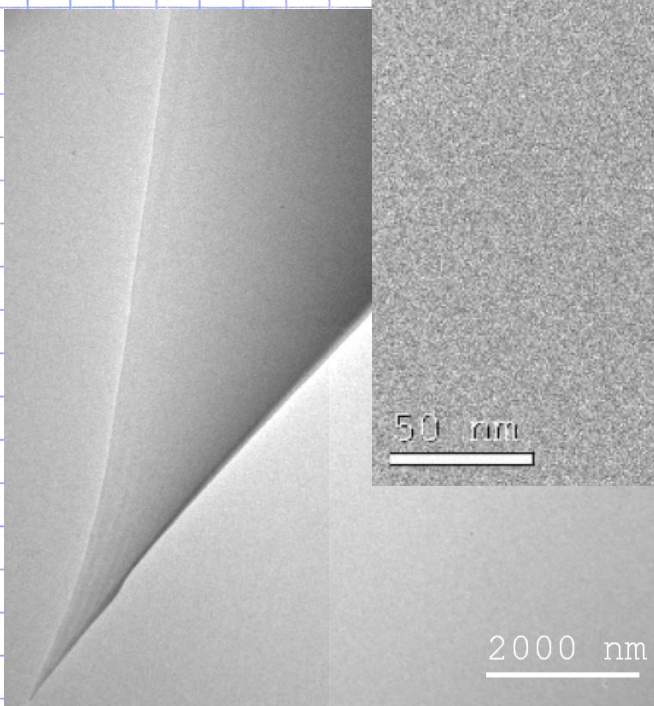
- ⇒ Basis : $\text{Co}_{80}\text{Cr}_{20}$ (similar to hard disk media)
- ⇒ Means : sputtering, DP850.
- ⇒ Stacking : Based on CoCr [2-50 nm], various buffer and capping layers



Initial Si apex

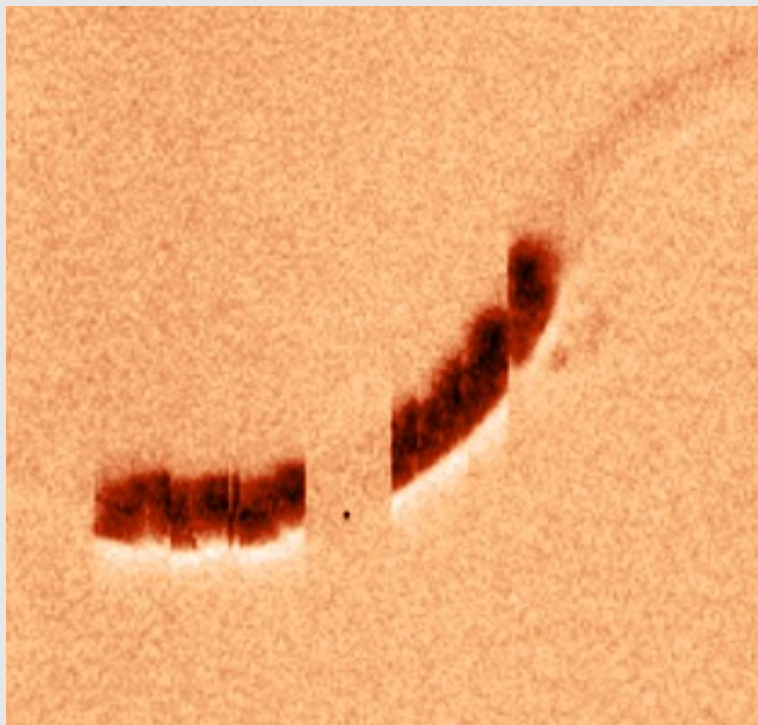
Magnetic coating

Antiwear coating



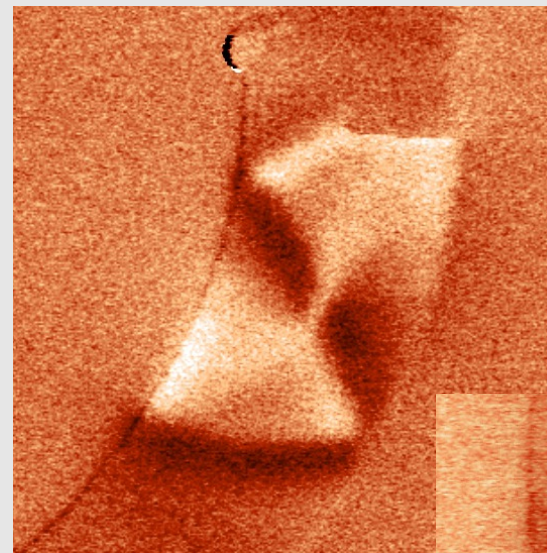
Transmission Electron Microscopy (A. Masseboeuf)

Commercial tips, 'low moment'

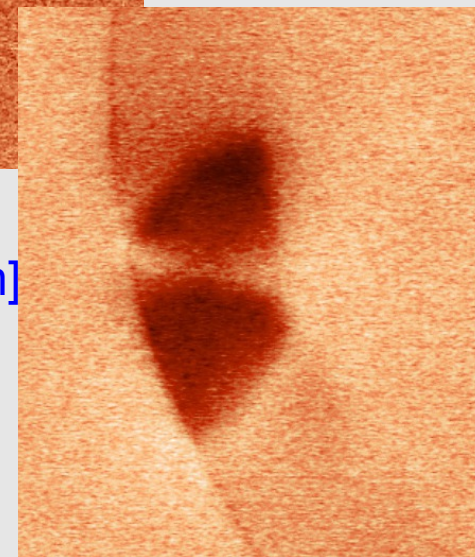


Permalloy strips [15nm]

Home-made tips, really low moment



Tip : CoCr[10nm]
500nm strips



Tip : CoCr[5nm]
300nm strips

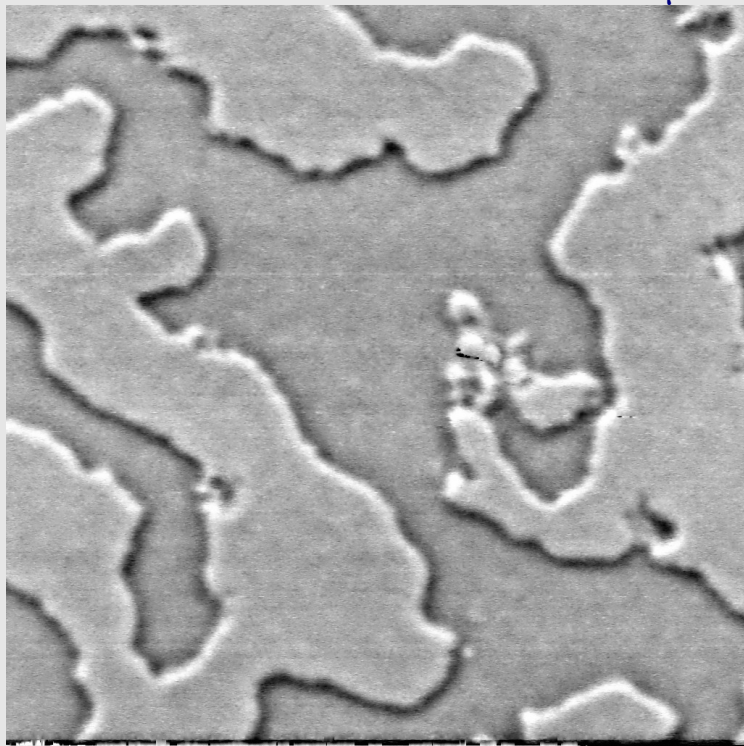
Sample : S. Pizzini
Imaging : Z. Ishaque

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

Spatial resolution 15nm

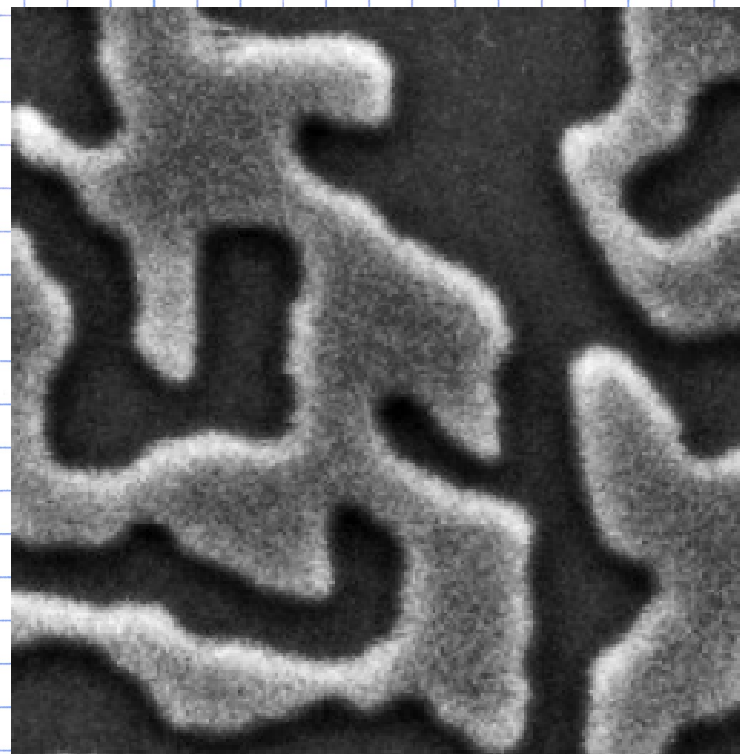
Test sample : FePt[4nm]. Perpendicular magnetization, narrow domain walls

2x2 μm



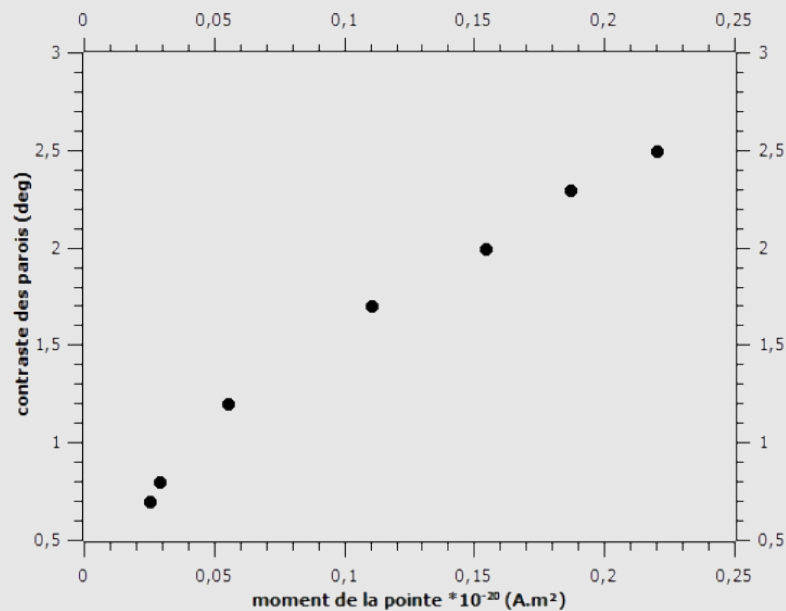
Tip : Nanosensors SSS \ 5nm CoCr
Fly height 0nm, amplitude 10nm

Commercial 'low moment' tip

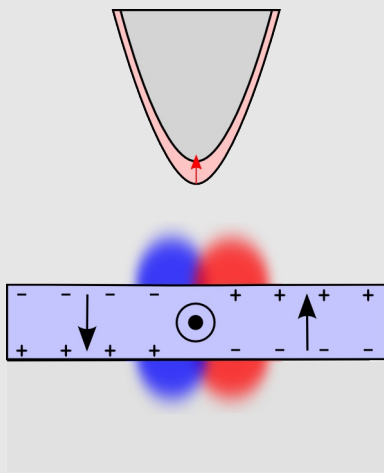


➡ Spatial resolution : 20nm

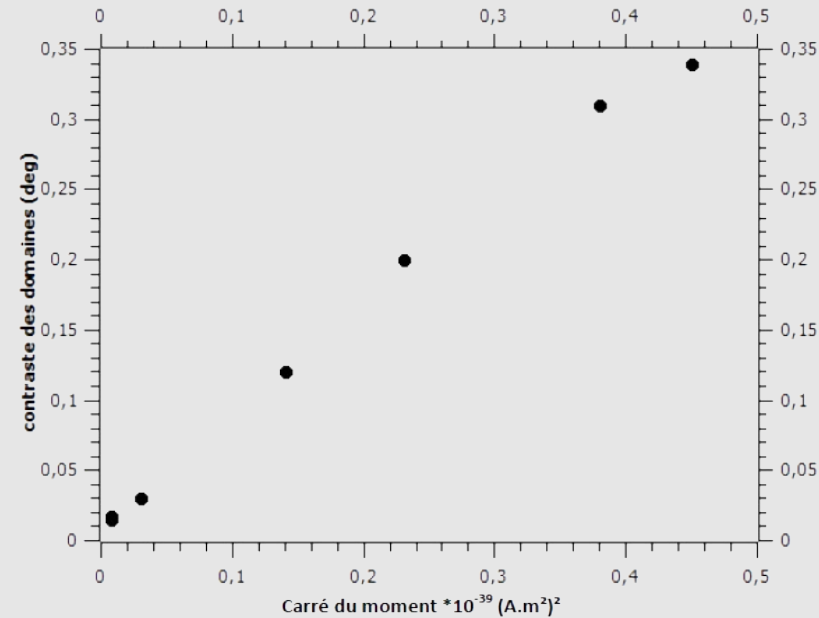
Domain wall contrast



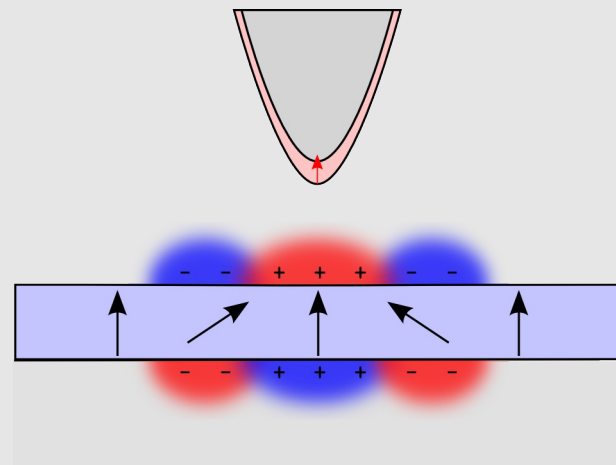
⇒ Linear with tip moment

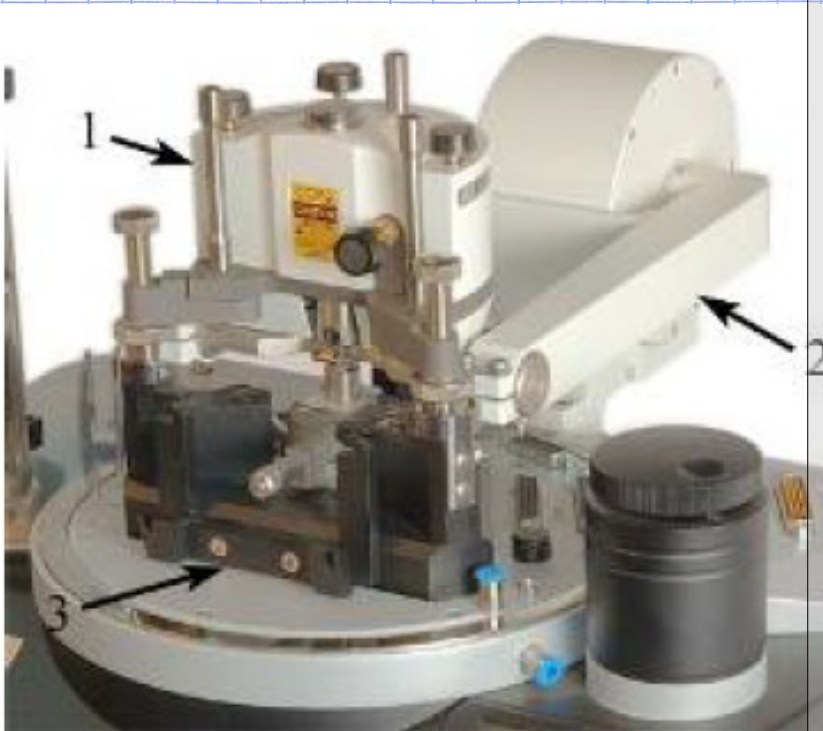


Domain contrast

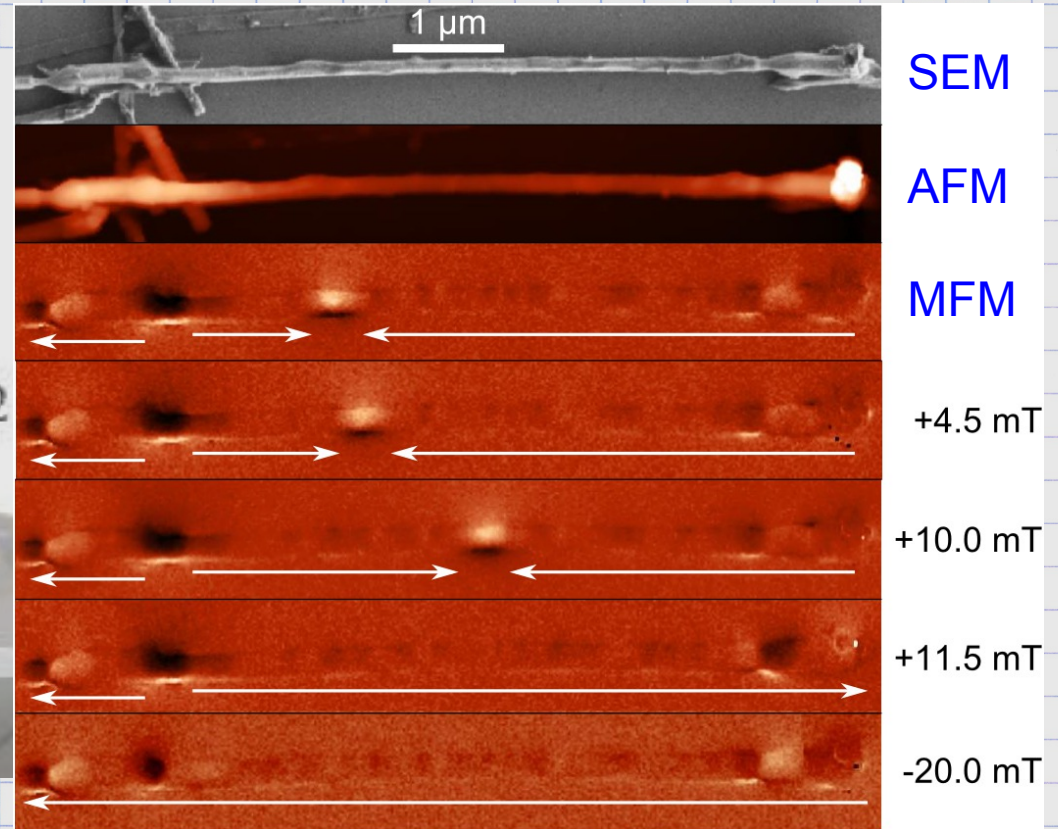


⇒ Linear with square of tip moment



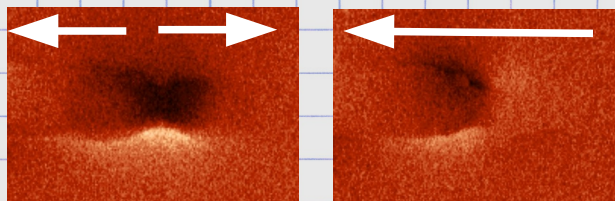


Domain wall motion



S. Da-Col et al., unpublished

Protrusions and constrictions

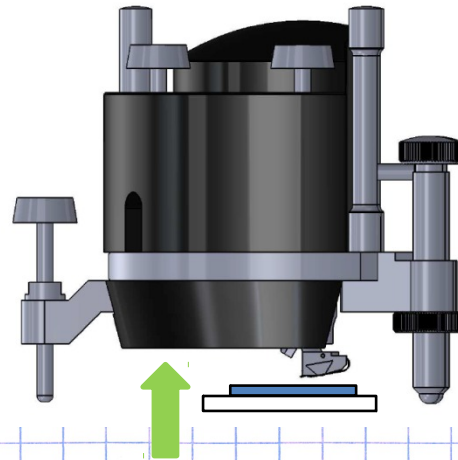


⇒ Pinning field ~30mT

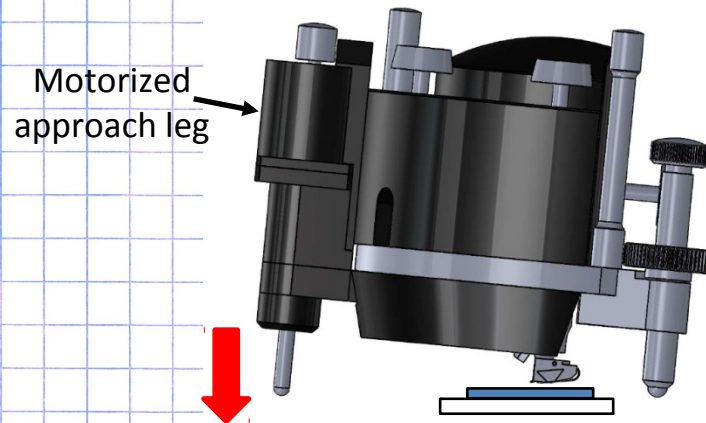
↻ Imaging under field
↻ Magnetization processes



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

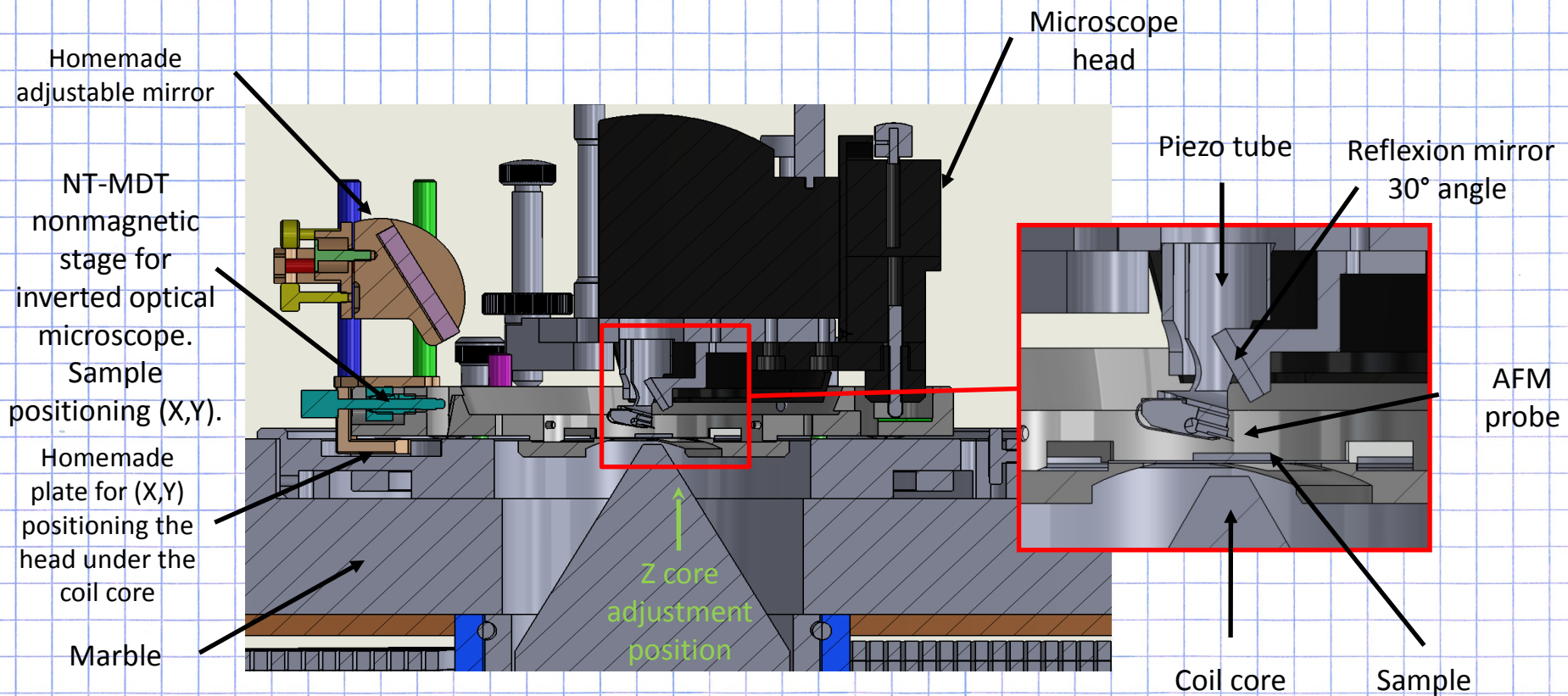


Microscope approach
by moving the sample up
(standard configuration)



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

↪ Approach by head (motorized leg)

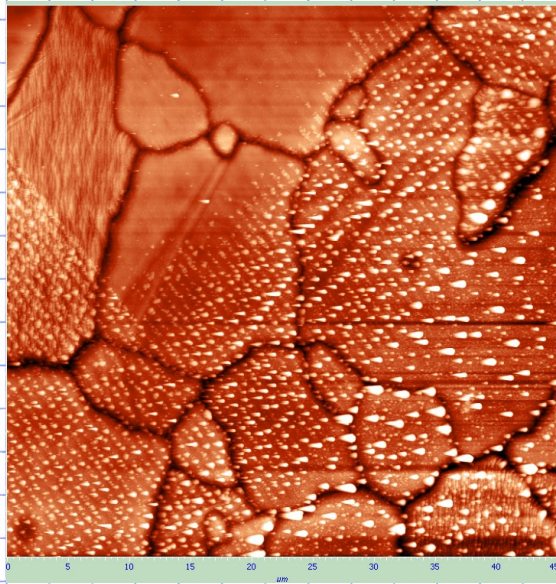


↪ Combines NT-MDT and custom-made designs
 ↪ High stability thanks to decoupling head and sample from the coil

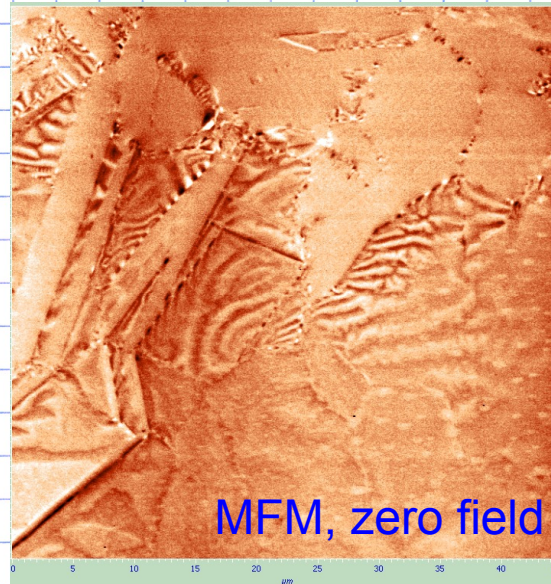


Magnetic shape-memory alloy

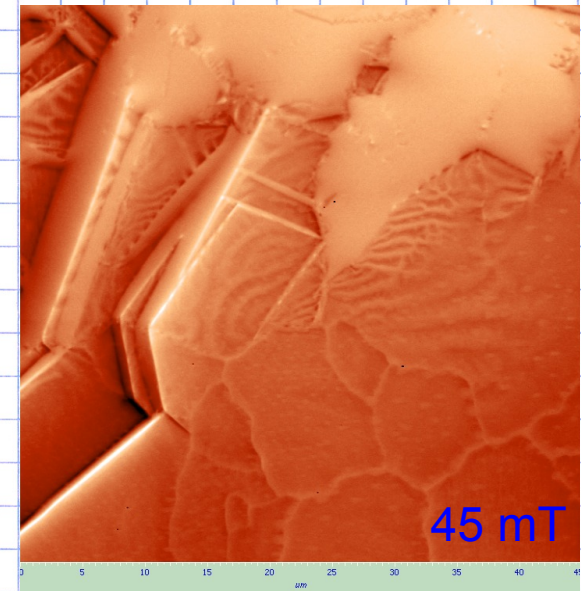
G. Crouigneau et al.



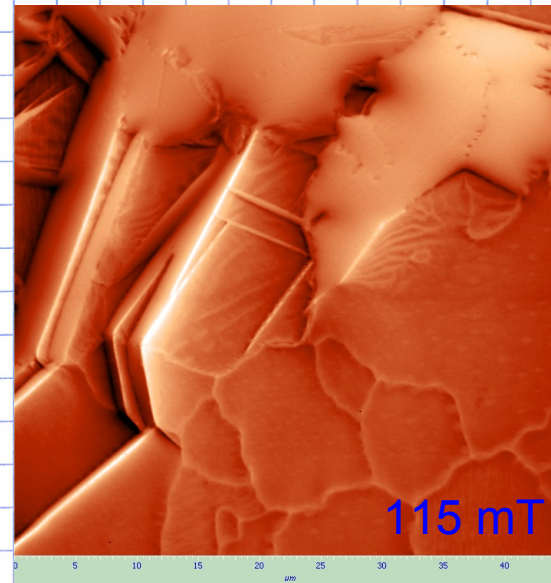
Topography (grains)



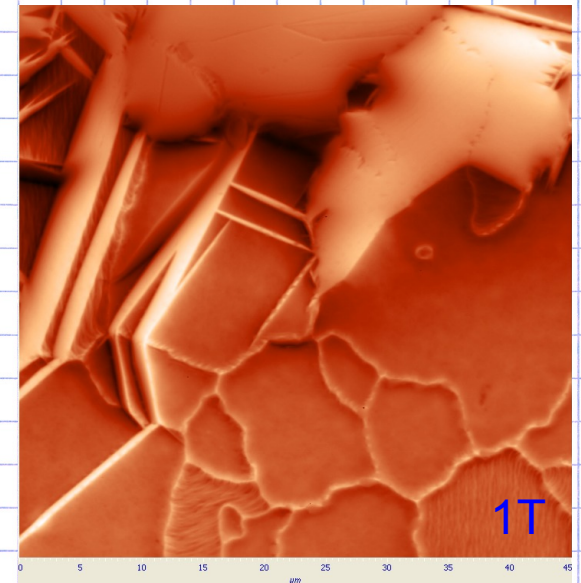
MFM, zero field



45 mT



115 mT

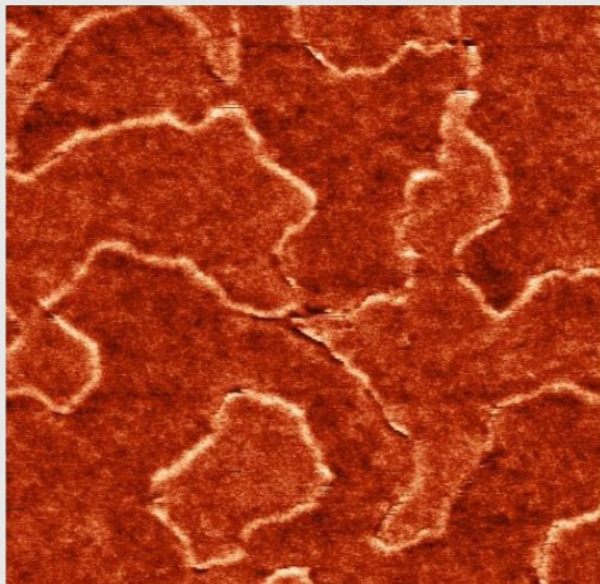


1T

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

Keep developing tips

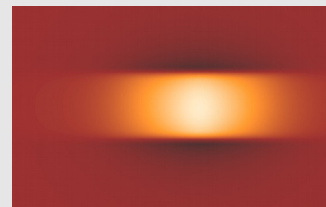
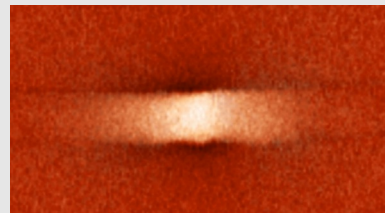
- ⇒ Superparamagnetic tips
(reduced effect on samples :
ultrasoft, skyrmions etc)



- ⇒ Hard magnetic tips
(imaging permanent magnets)

Towards quantitative analysis

- ⇒ Simulations

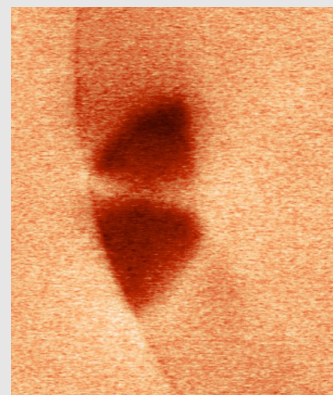


S. Jamet, JC Toussaint

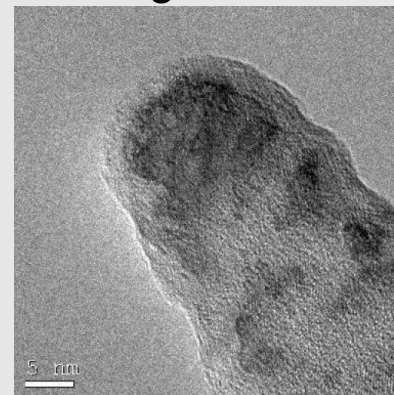
- ⇒ Special tips (eg filled carbon nanotube)

Pushing further sensitivity

- ⇒ Work under vacuum → High Q



<10nm film



<5nm tip



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, 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, E. Wagner



- ⇒ [Magnetic Force Microscopy of hard magnetic films](#), G. Ciuta, F. Dumas-Bouchiat, N. M. Dempsey, O. Fruchart, **submitted**
- ⇒ [Third type of magnetic domain wall in permalloy nanostrips](#), D. N'Guyen, O. Fruchart, S. Pizzini, J. Vogel, J. C. Toussaint and N. Rougemaille, **submitted**
- ⇒ [Manipulating domain wall chirality by current pulses in Permalloy/Ir nanostrips](#), M. Z. Ishaque, V. D. Nguyen, O. Fruchart, S. Pizzini, N. Rougemaille, S. Perl, J. C. Toussaint, J. Vogel, **submitted**
- ⇒ [Non-universality of artificial frustrated spin systems](#), I. A. Chioar, N. Rougemaille, A. Grimm, O. Fruchart, E. Wagner, M. Hehn, D. Lacour, F. Montaigne, B. Canals, **Phys. Rev. B** 90, 064411 (2014) (<http://fr.arxiv.org/abs/1407.7416>)
- ⇒ [Growth and micromagnetism of self-assembled epitaxial fcc\(111\) cobalt dots](#), O. Fruchart, A. Masseboeuf, J. C. Toussaint, P. Bayle-Guillevaud, **J. Phys. Condens. Matter** 25, 496002 (2013). (<http://fr.arxiv.org/abs/1309.0793>)
- ⇒ [Tuning micro-twinned domain sizes and the b-variants content of the adaptive 14-modulated martensite in epitaxial Ni-Mn-Ga films by co-sputtering](#), J. Tillier, D. Bourgault, Ph. Odier, L. Ortega, S. Pairis, O. Fruchart, N. Caillault, L. Carbone, **Acta Mater.** 59 (1), 75 (2011) (<http://fr.arxiv.org/abs/1011.2303>)



Alex Hubert
Rudolf Schäfer

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