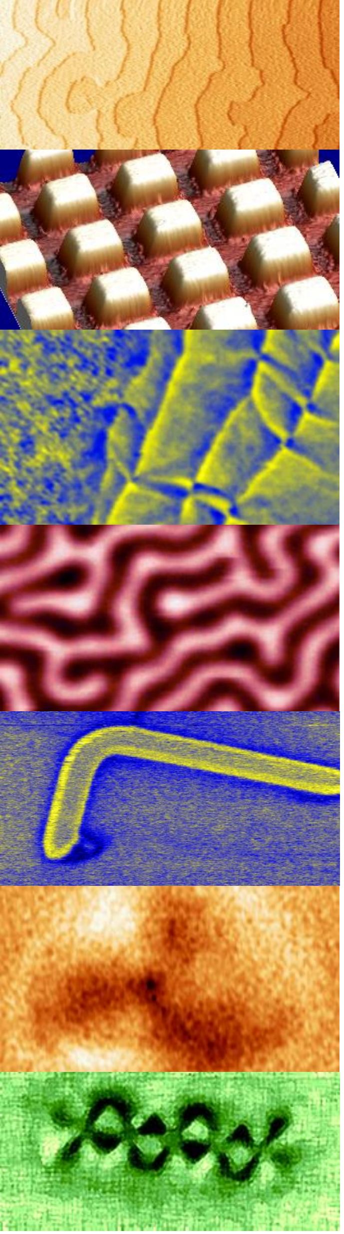


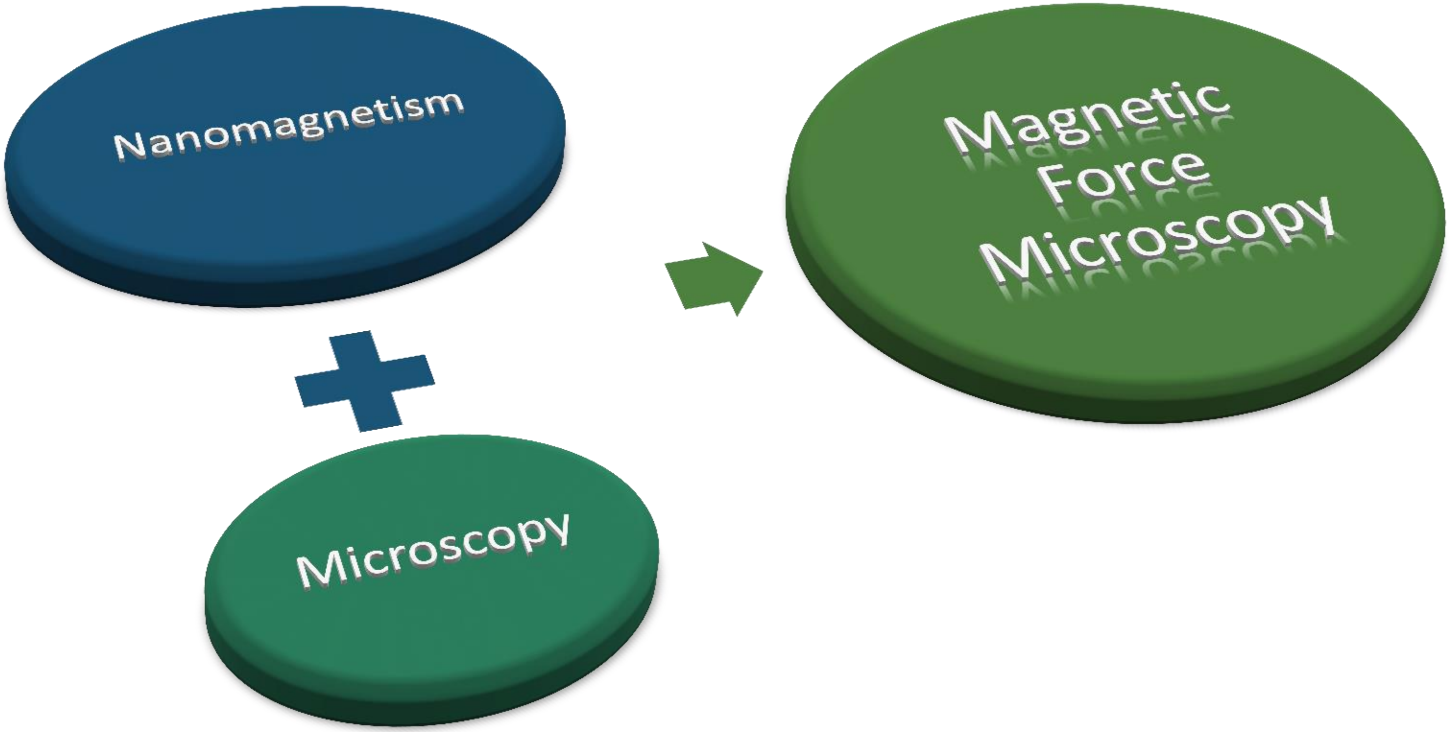
Magnetic Force Microscopy imaging: from permanent magnets to bacteria

Agustina Asenjo
ICMM-CSIC

Nanomagnetism and Magnetic Materials Group
MFM Laboratory

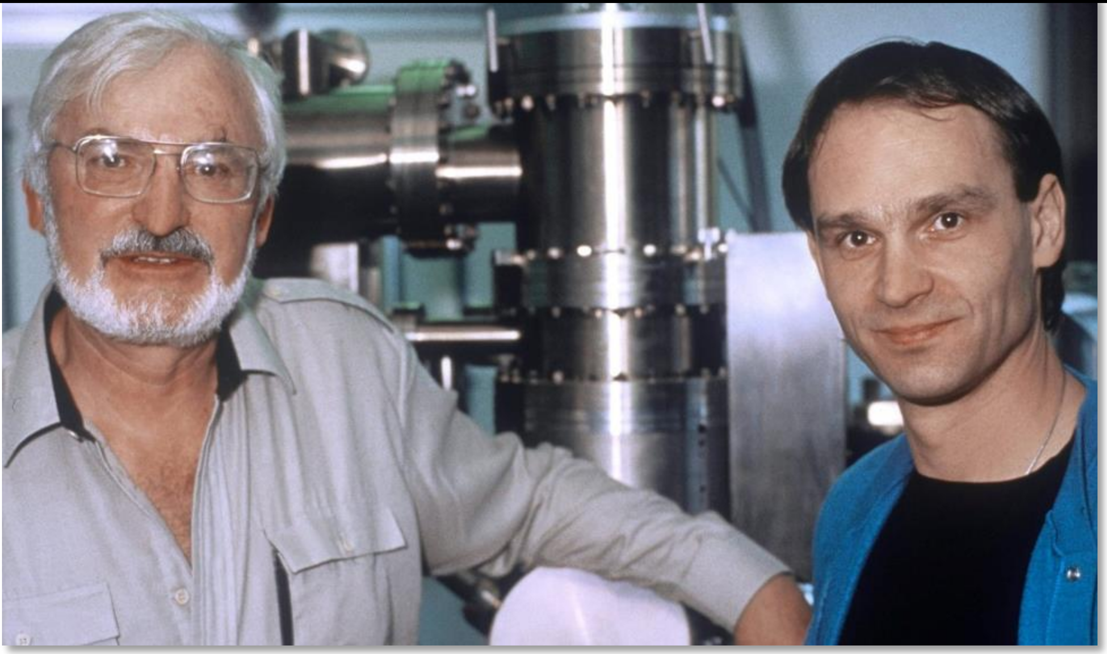
icmm
CSIC
Consejo Superior de Investigaciones Científicas
Gobierno de España
Ministerio de Economía y Competitividad
Programa ingenio 2010
Force for Future
REFREEPERMAG

Magnetic Force Microscopy



SPM. Tools for Future

Magnetic Force Microscopy



Microscopy

1981- **G. Binnig and H. Rohrer**

Scanning Tunneling Microscope (STM) revolutioned surface science and allowed for atomic resolution in conducting samples.

1986- **Nobel Prize in Physics**

1986- **G. Binnig, C.F. Quate and C. Gerber**

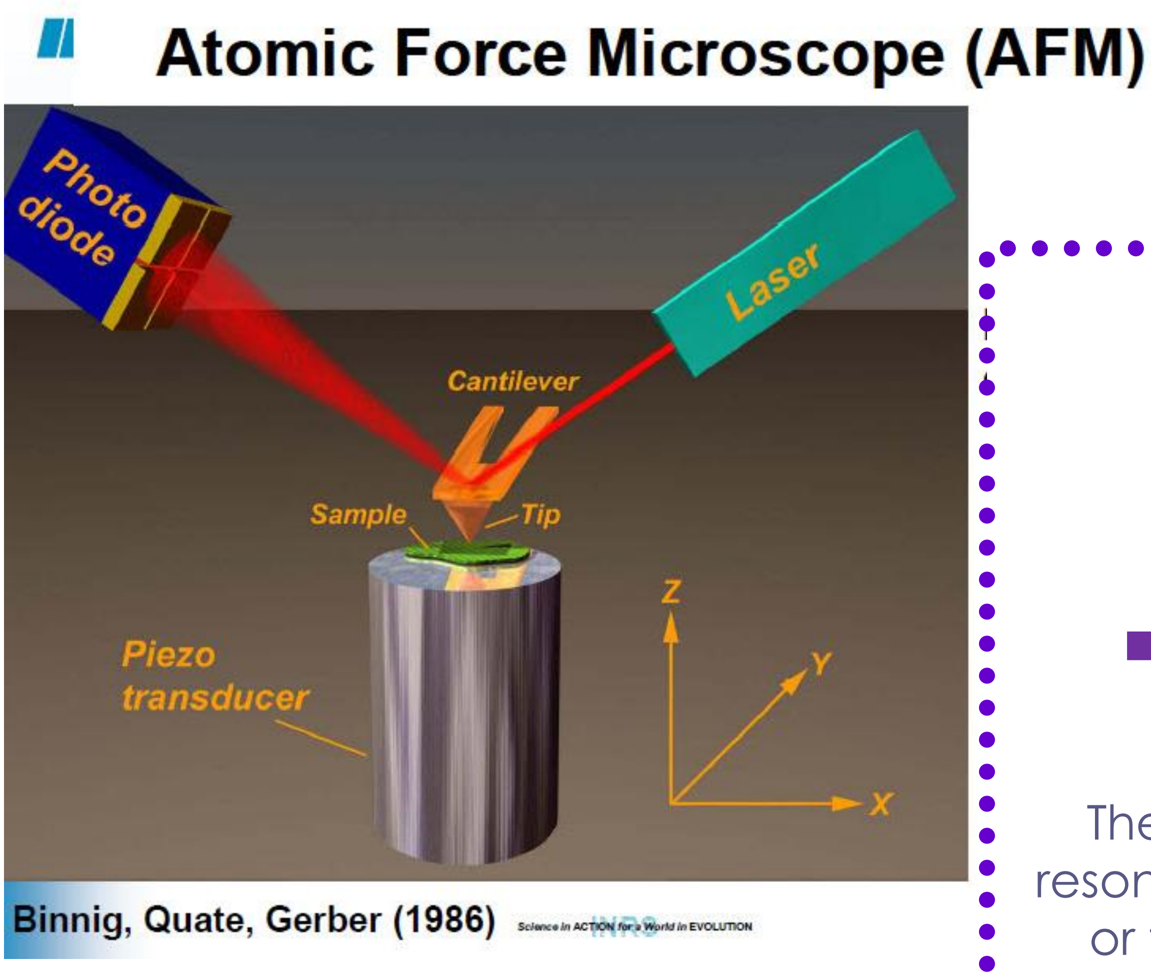
Atomic Force Microscope (AFM) is invented. It allowed for the study of non-conducting samples and a great variety of properties.

1987- **Two different groups**

MFM. The use of *AFM* to measure magnetic samples was first reported

Y. Martin et al., Appl. Phys. Lett. 50 (1987) 1455
J.J. Sáenz et al., J. Appl. Phys. 62 (1987) 4293

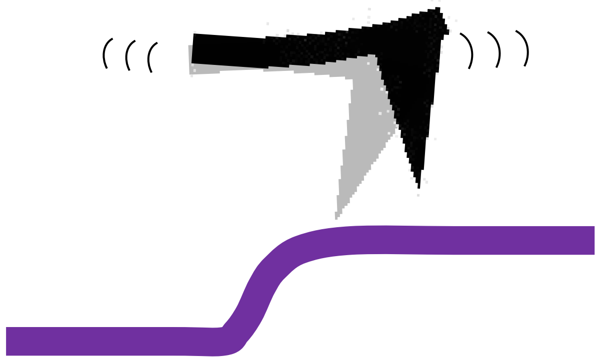
Magnetic Force Microscopy



Atomic Force Microscope (AFM)

Binnig, Quate, Gerber (1986)

Science in ACTION for a World in EVOLUTION



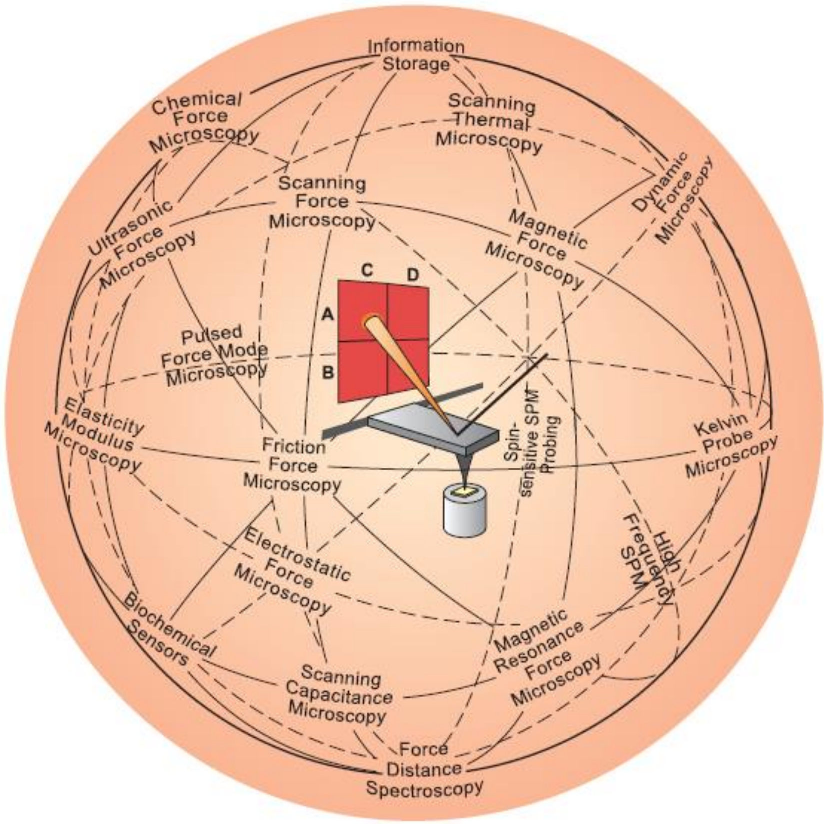
Dynamic mode:

The cantilever is excited at its resonance frequency: **amplitude** or the **frequency shift** can be controlled

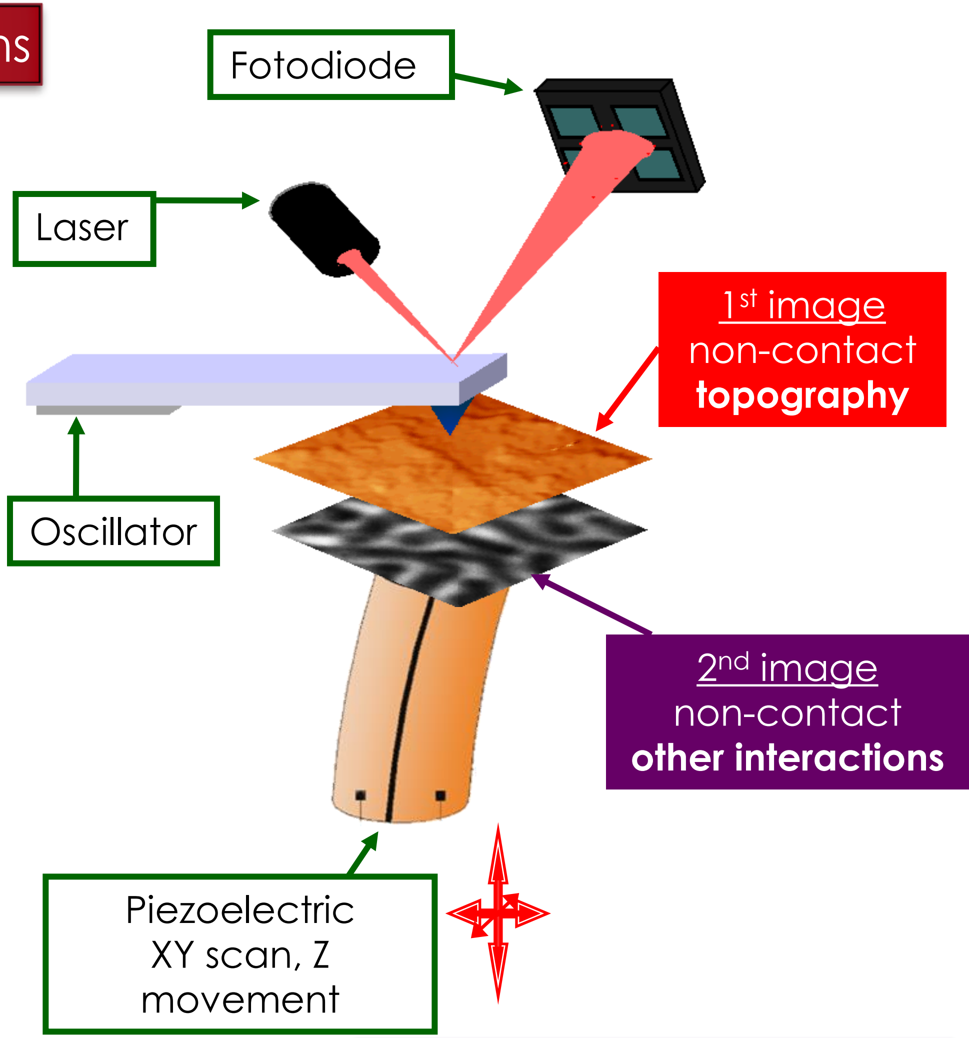
~ constant tip-sample distance

SPM - Magnetic Force Microscopy

A variety of applications and interactions



C. Berger *et al* **Nature Nanotechnology** 1, 3 (2006)



Fotodiode

Laser

Oscillator

1st image
non-contact
topography

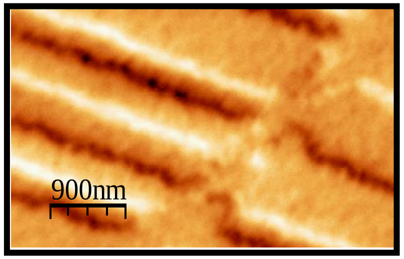
2nd image
non-contact
other interactions

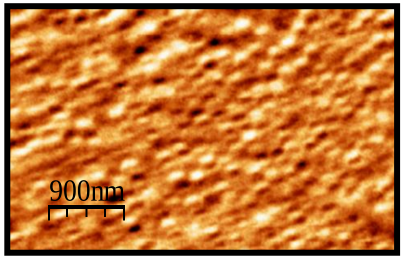
Piezoelectric
XY scan, Z
movement

- **Operation modes**
- **Functionalize probes**

SPM. Tools for Future

Magnetic Force Microscopy





Nanomagnetism

- Magnetic imaging at nanoscale
- Domain configuration
- Domain Wall characterization
- Reversal magnetization processes

Biomagnetic materials

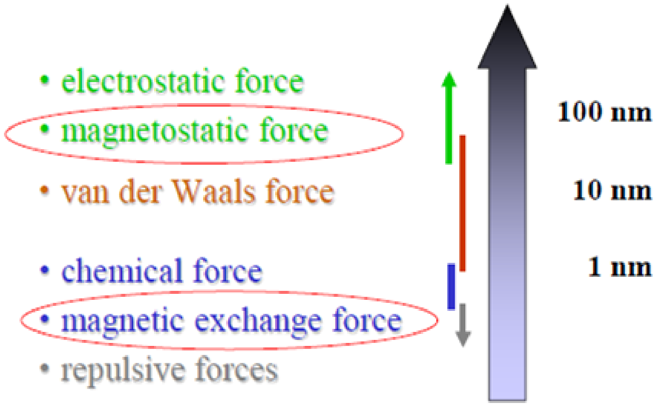
- Fundamental studies
- Quality control in HD industry
- Characterization of thin films, nanostructures…

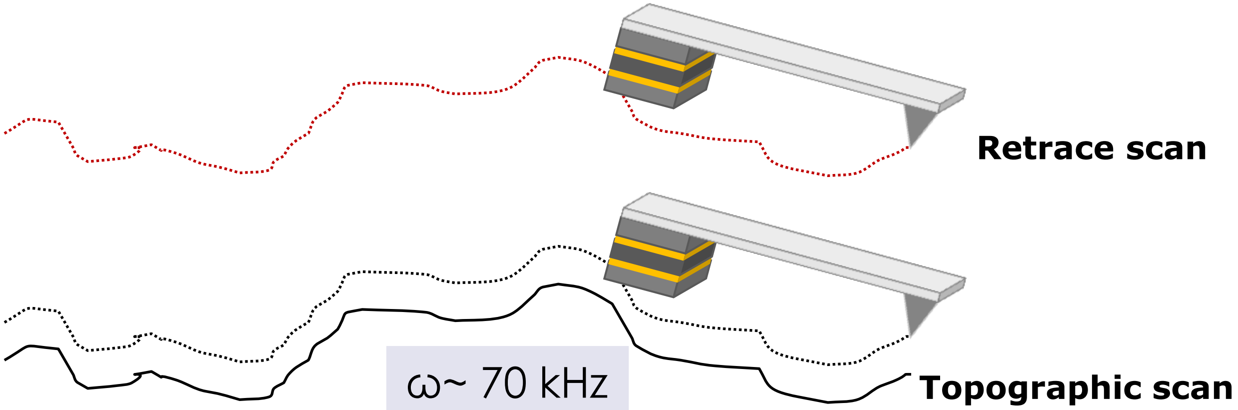
- New strategies in spintronic

Outline:

1. Fundamentals of MFM
2. MFM based modes
3. Variable Field MFM
4. Special MFM Probes
5. Conclusions

Operation mode in MFM





ω~ 70 kHz
k~ 3 N/m

$$F_{magnetic} = m_{tip} \bullet \nabla H_{sample}$$

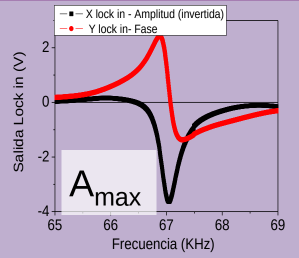
Only for small amplitudes

$$\Delta A = \frac{A_0 Q}{2k} \frac{\partial F_z^{vdW}}{\partial z}$$

$$\Delta \omega \propto \frac{\omega_0}{2k} \frac{\partial F_z^{mag}}{\partial z}$$

Z retrace>10nm → Two scans

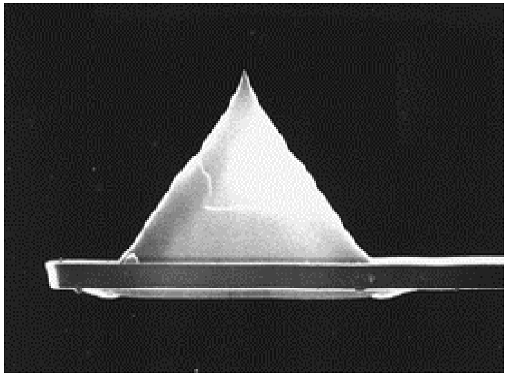
PLL feedback

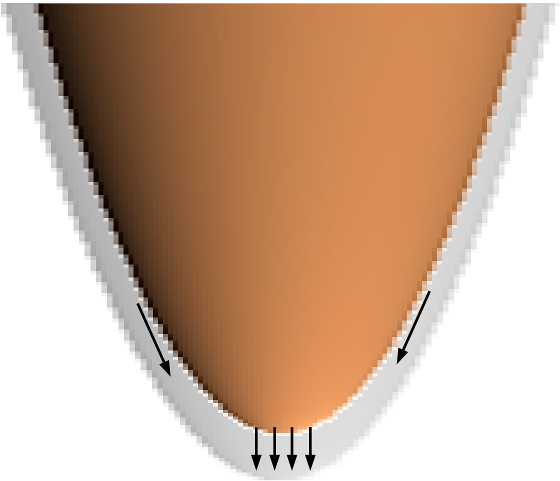


Constant phase

MFM signal = frequency shift

Magnetic tips





MFM Probes:

- Commercial AFM probes coated with a thin film (20-100nm) Co, CoCr, permalloy, ...
- Axial magnetization due to shape anisotropy

Tip Models

Monopole

$$\frac{\partial F}{\partial z} \prec \mu_0 \sigma \frac{\partial H_z}{\partial z}$$

Dipole

$$\frac{\partial F}{\partial z} \prec \mu_0 m \frac{\partial^2 H}{\partial z^2}$$

H, stray field of the sample
σ, surface charge density of the tip
m, dipolar moment of the tip

MFM images interpretation

Assuming the tip-sample influence is negligible:

- The MFM contrast is proportional to the magnetic pole density at the surface.
- **Perpendicular anisotropy:** Poles at the center of the domains.
- **In-plane anisotropy:** Poles at the domain walls

Pole density

MFM Contrast

Domains

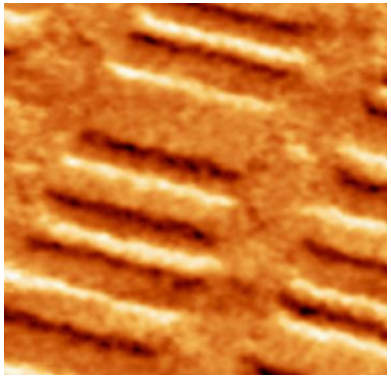
$$\text{MFM contrast} \equiv \nabla M$$

Especially sensitive to the out of plane magnetization

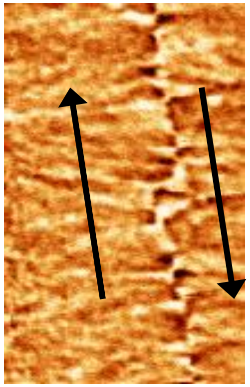
MFM images interpretation

Assuming the tip-sample influence is negligible:

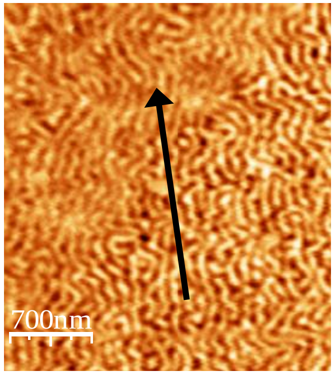
- The MFM contrast is proportional to the magnetic pole density at the surface.
- **Perpendicular anisotropy:** Poles at the center of the domains.
- **In-plane anisotropy:** Poles at the domain walls



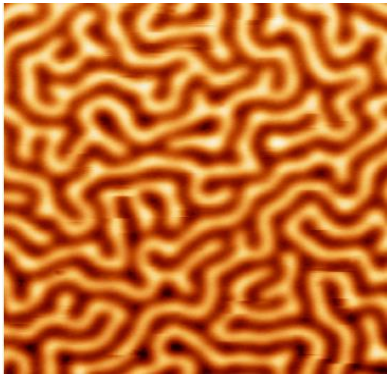
Hard disk image.
10 μm x 10 μm



Cross-tie domain wall in FePt thin film



Dense stripe domains in FePt thin film.

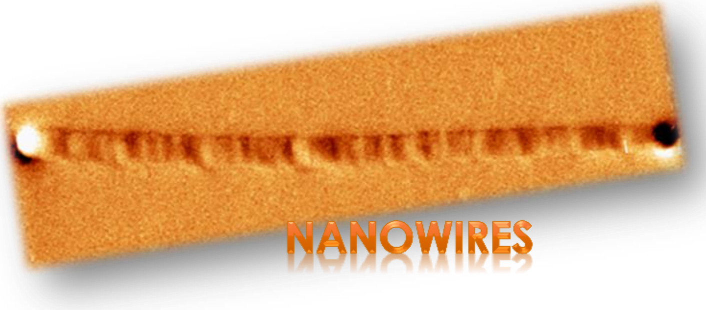


FePd thin film.
3μmx3μm

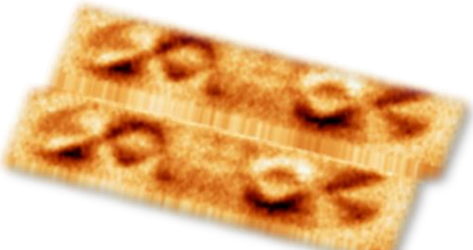
What are the applications? Why MFM?

- Low cost technique. XMCD ~10000 AFM
- Lateral resolution better than 20nm
- Additional information (3D topo,...)
- To study individual elements
- Trouble-free sample preparation

THIN FILMS



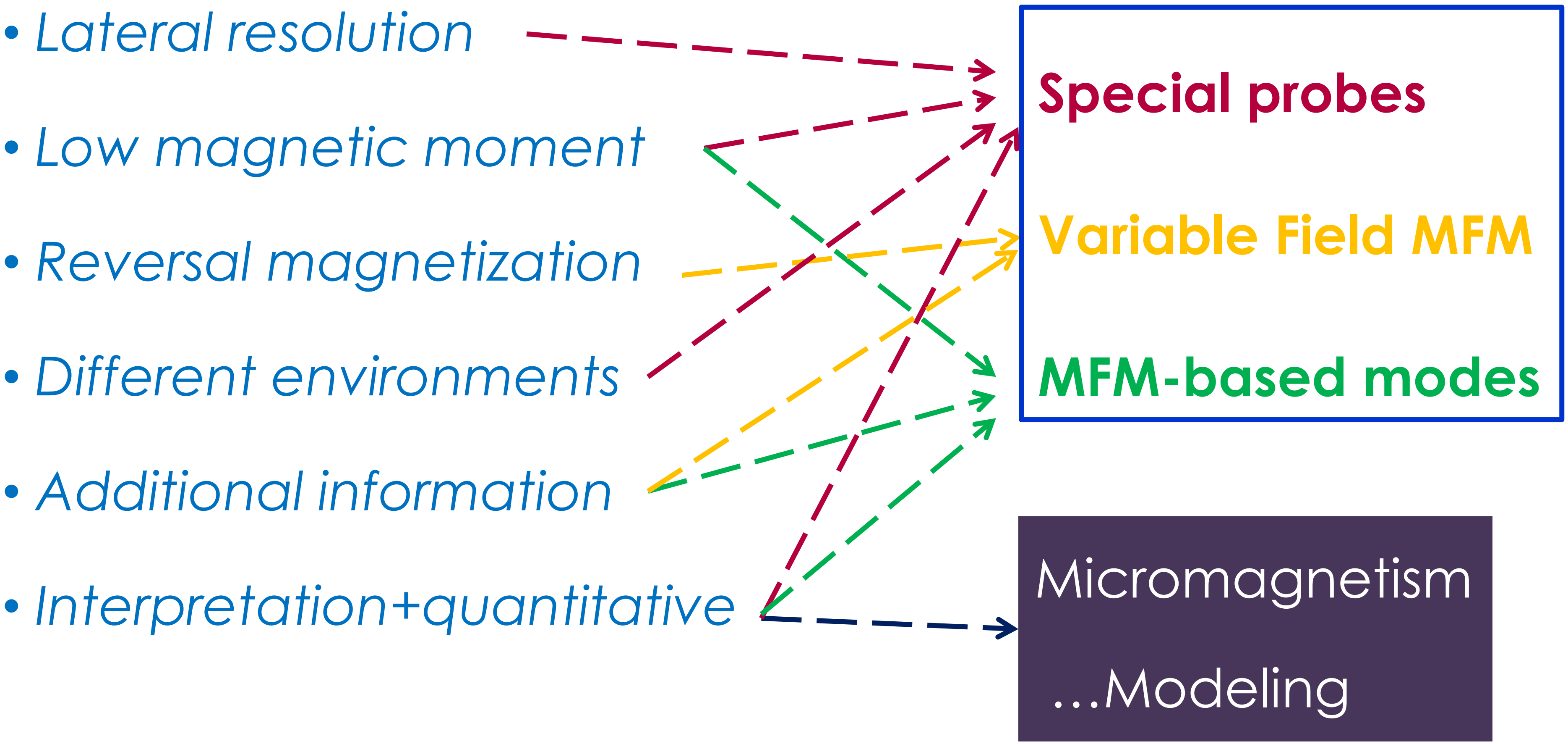
NANOWIRES



NANOSTRUCTURES

- Interpretation of the contrast. Is it magnetic? MFM quantitative?
- Influence of the tip stray field. Could we control it??
- What's the higher lateral resolution?
- Could we work in environment?

Some challenges in MFM



- *Lateral resolution*
- *Low magnetic moment*
- *Reversal magnetization*
- *Different environments*
- *Additional information*
- *Interpretation+quantitative*

Special probes

Variable Field MFM

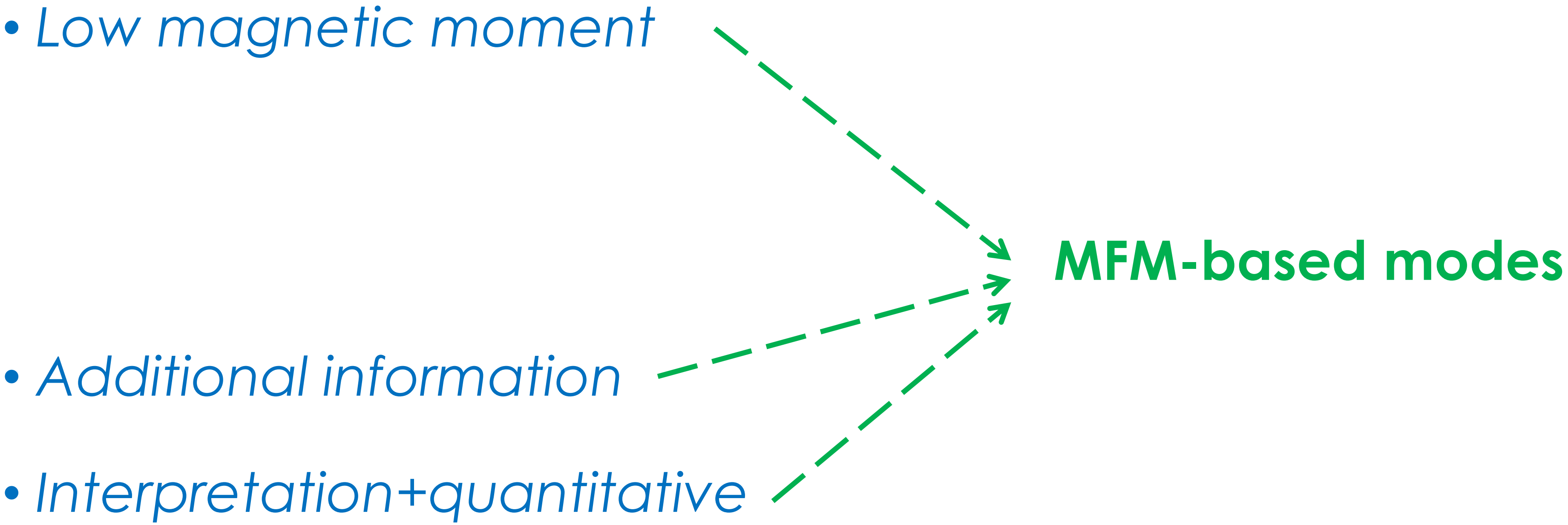
MFM-based modes

Micromagnetism

…Modeling

……..from hard disk to bacteria

Some challenges in MFM



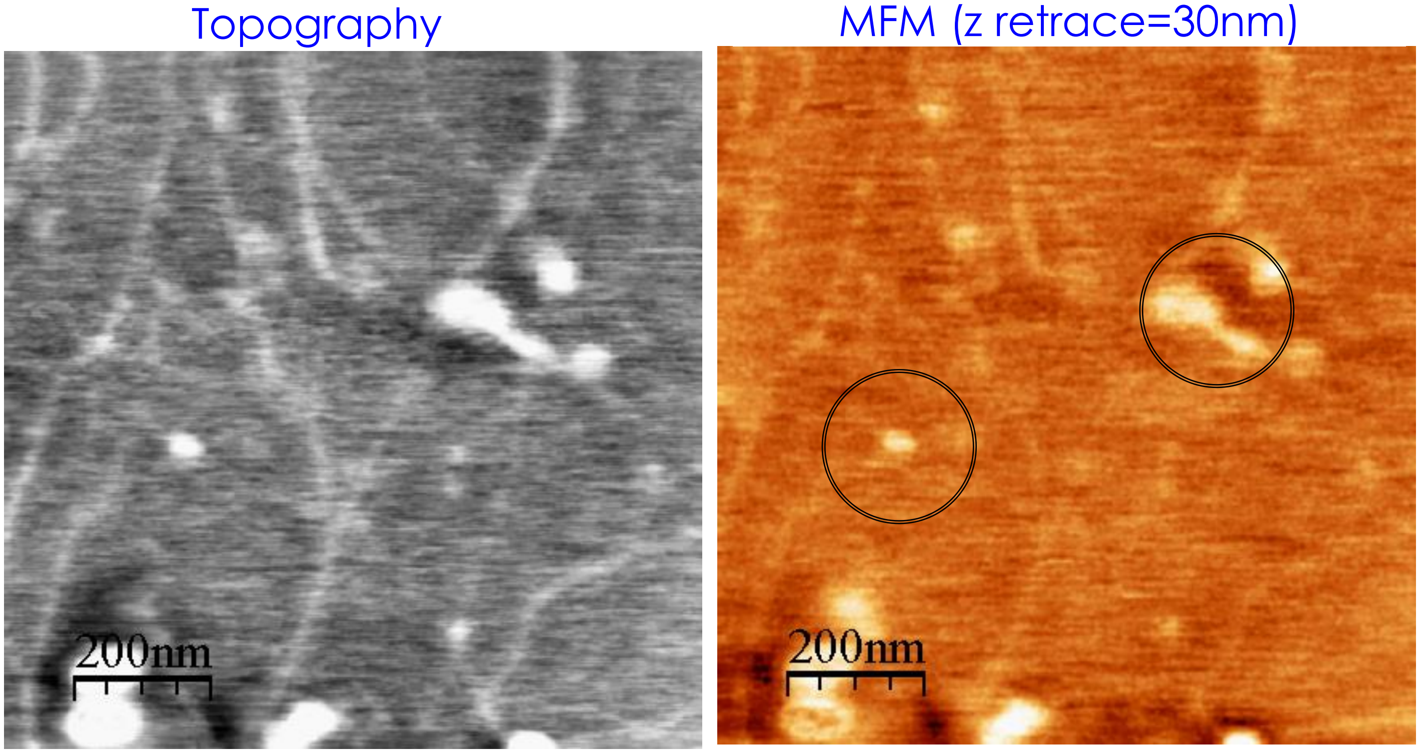
- *Low magnetic moment*
- *Additional information*
- *Interpretation+quantitative*

MFM-based modes

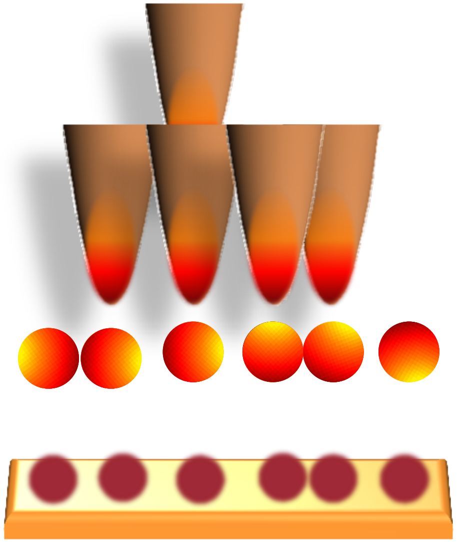
……..from hard disk to bacteria

Some challenges in MFM: measuring low moment-low coercivity nanoparticles

Iron oxide nanoparticles,10nm in diameter, prepared by co-precipitation.
G. Pourroy's group, IPCMS –CNRS



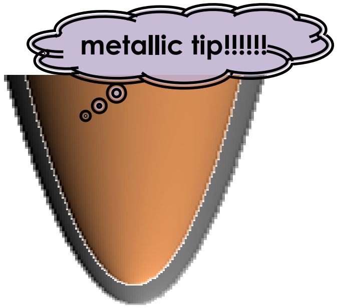
Low coercivity NP



Artifacts in MFM: unexpected repulsive interaction
The origin: topography, electrostatic?

KPFM and MFM combination

Kelvin Probe Force Microscopy



$$V = (V_{dc} - \Delta\Phi/q) + V_{ac}\sin(\omega t)$$

$$\Downarrow$$

$$\boxed{F_{es} = -\frac{1}{2}\frac{\partial C}{\partial z}[(V_{dc} - \Delta\Phi/q) + V_{ac}\sin(\omega t)]^2}$$

$$F_{dc} = -\frac{\partial C}{\partial z}\left(\frac{1}{2}(V_{dc} - \Delta\Phi/q)^2 + \frac{1}{4}V_{ac}^2\right)$$

$$\cancel{F_{\omega}} = -\frac{\partial C}{\partial z}(V_{dc} - \Delta\Phi/q)V_{ac}\sin(\omega t) \rightarrow$$

$$F_{2\omega} = +\frac{\partial C}{\partial z}\frac{1}{4}V_{ac}^2\cos(2\omega t).$$

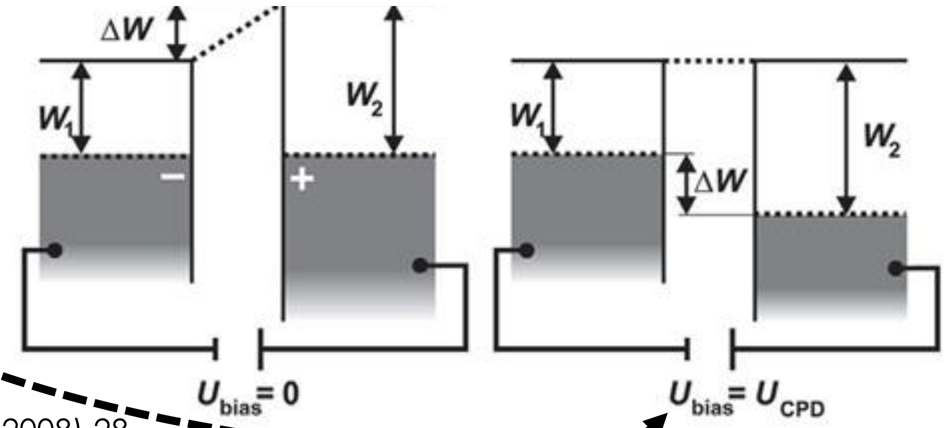
Mode	Frequency
KPFM (air)	7 kHz
Resonant Frequency	70 kHz
KFPM (HV, 2nd mode)	400 kHz

$$V_{dc} = \Delta\phi/q = V_{CPD}$$

Different materials, different <u>work functions</u>, $W_1 \neq W_2$

Impossible to compensate the electrostatic force contrast with a **fixed bias voltage**

We need to employ **<u>Kelvin Probe Force Microscopy.</u>**



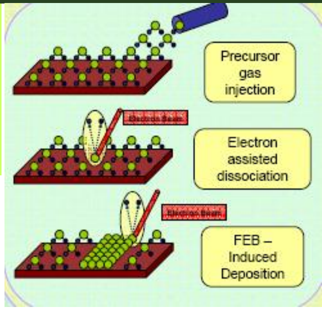
A. Schwarz and R. Wiesendanger, Nanotoday, 3, 1-2 (2008) 28

M. Jaafar, O. Iglesias-Freire, L. Serrano-Ramón, M. R. Ibarra, J. M. De Teresa and A. Asenjo, BJNano., 2, 552-560 (2011)
D. Martínez – Martín, M. Jaafar, J. Gómez – Herrero, R. Pérez and A. Asenjo, Phys. Rev. Lett. 105, 257203 (2010)

KPFM and MFM combination

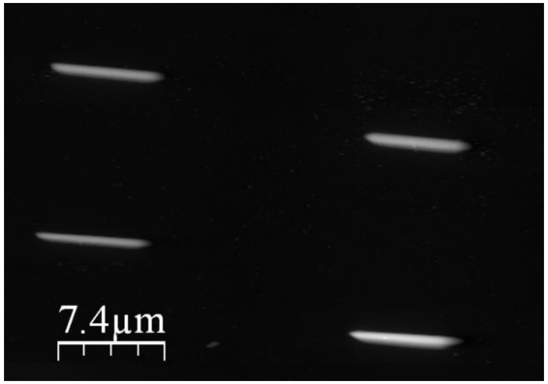
Co nanostripes/SiO_2 prepared by Focused Electron Beam

Local deposition of materials using a focused electron beam in the presence of a gas precursor. The electron beam interacts with the gas molecules adsorbed at the substrate surface and decomposes them. As a consequence, the volatile fragments are evacuated in the vacuum system, while the rest is deposited.

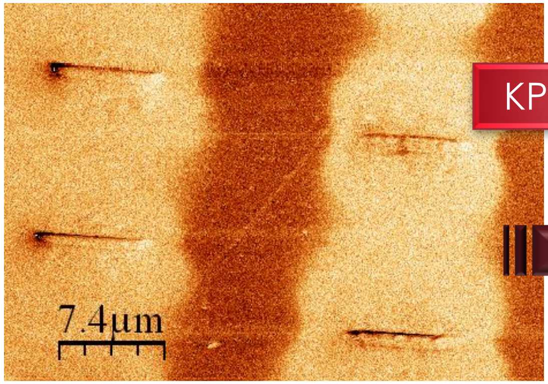


Heterogeneous electrostatic interaction between tip and sample that can be interpreted as magnetic interaction

Magnetic nanoelements and substrate present different **surface potential**



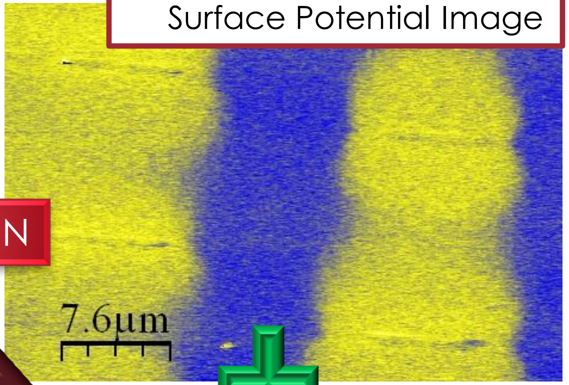
Topography

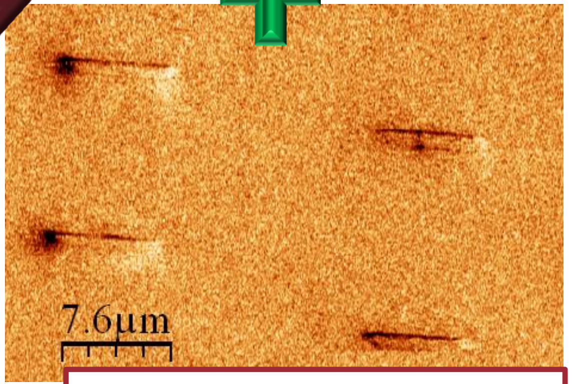


Frequency Shift (MFM probe)

KPFM ON

Surface Potential Image

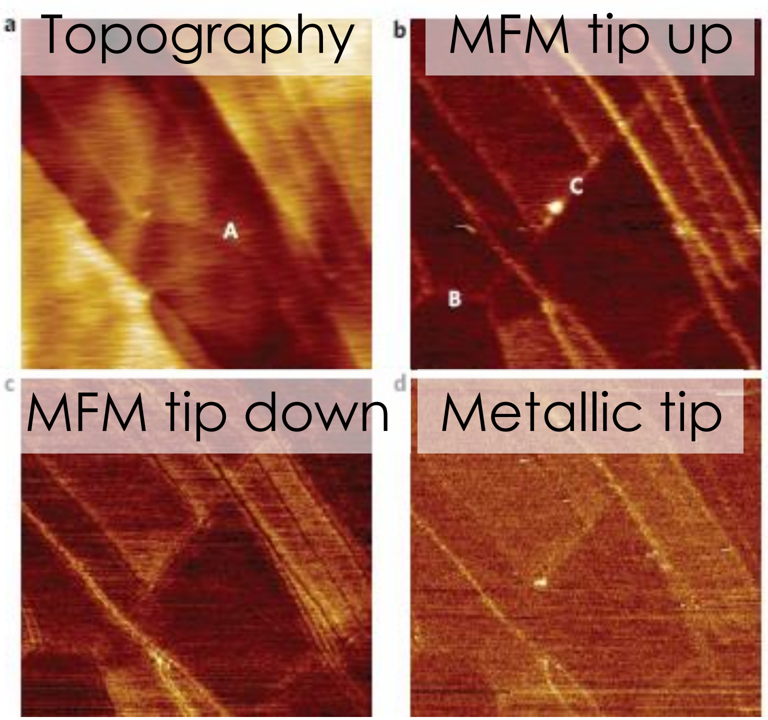




Frequency Shift (MFM image)

M. Jaafar, O. Iglesias-Freire, L. Serrano-Ramón, M. R. Ibarra, J. M. De Teresa and A. Asenjo, BJNano., 2011, 2, 552-560

MFM in Graphite



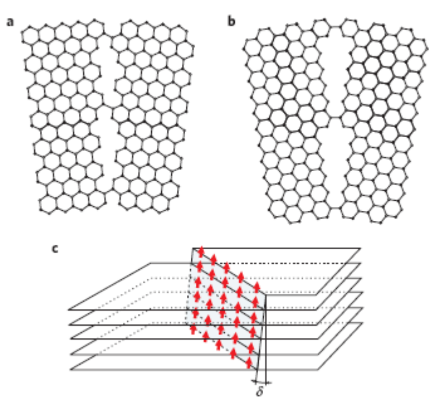
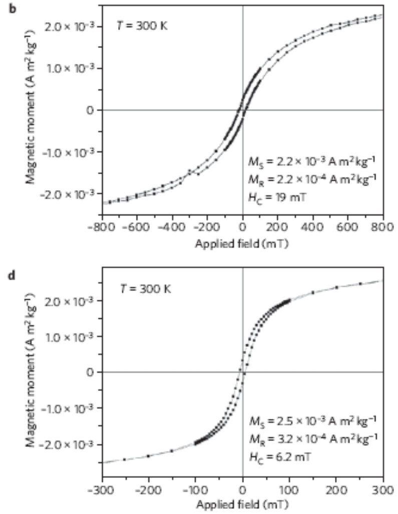


Figure 4 | Schematic models of two basics shapes of grain boundaries in graphite. **a**, Armchair direction with periodicity *D*. **b**, Zigzag direction with periodicity $\sqrt{3}D$. **c**, 2D in-plane magnetized grain boundary propagating through bulk HOPG.



- **According to** Cervenka et *al. [Nat. Phys.* **5**, 840,(2009)] grain boundaries in graphite can be visualized as a 2D plane defects propagating to the volume.

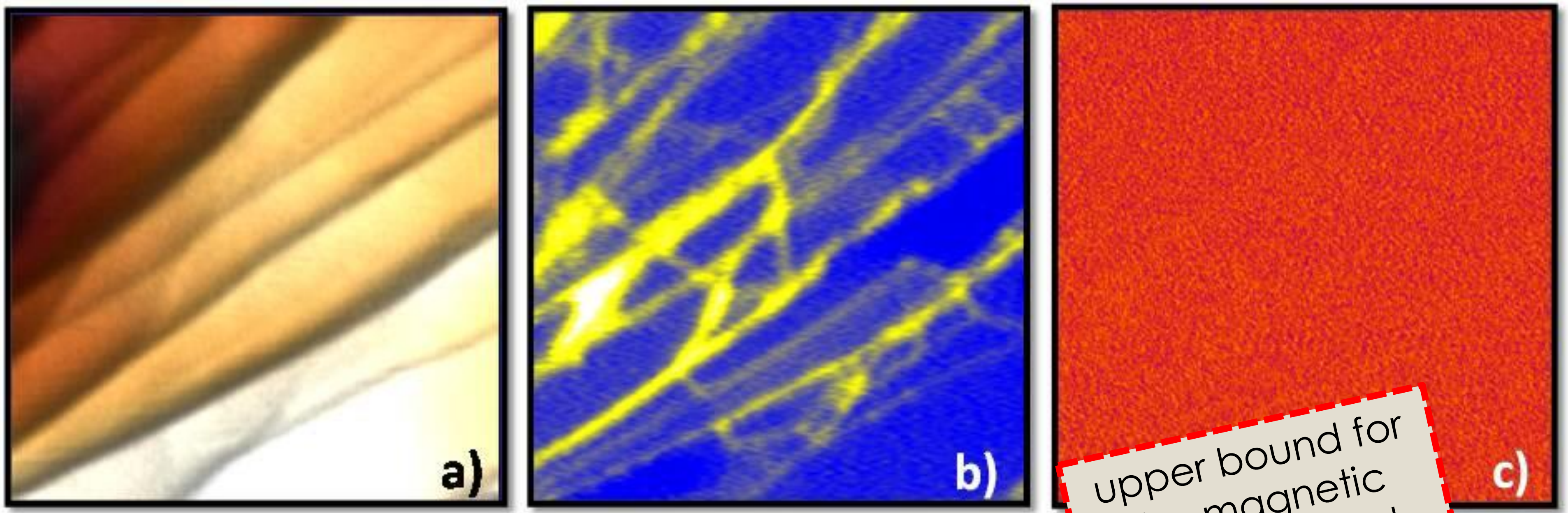
"Room-temperature ferromagnetism in **graphite** driven by two-dimensional networks of point defects"
Cervenka et al. *Nature Physics* **5**, 840 (2009)

- The implication is that grain boundaries should present a magnetic field gradient of ~**0.1-1 mN/m at 50 nm** from de surface that **should be possible to detect with magnetic force microscopy (MFM).**

Ferromagnetic domains located in the grain boundaries

KPFM and MFM in Graphite

KPFM ON



Topography

KPFM image, 1st scan

Frequency shift at 50nm

Amplitude, 4 nm

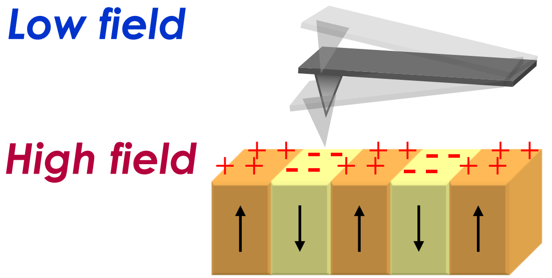
upper bound for the magnetic force gradient

The magnetic signal, if present, is lower than **16 μN/m** predicted theoretically

D. Martínez – Martín, M. Jaafar, J. Gómez – Herrero, R. Pérez and A. Asenjo, *Phys. Rev. Lett*. 105, 257203 (2010)

Dissipation in MFM

Cantilever oscillating

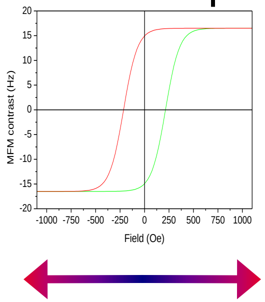


+

“High” stray field of the sample/tip

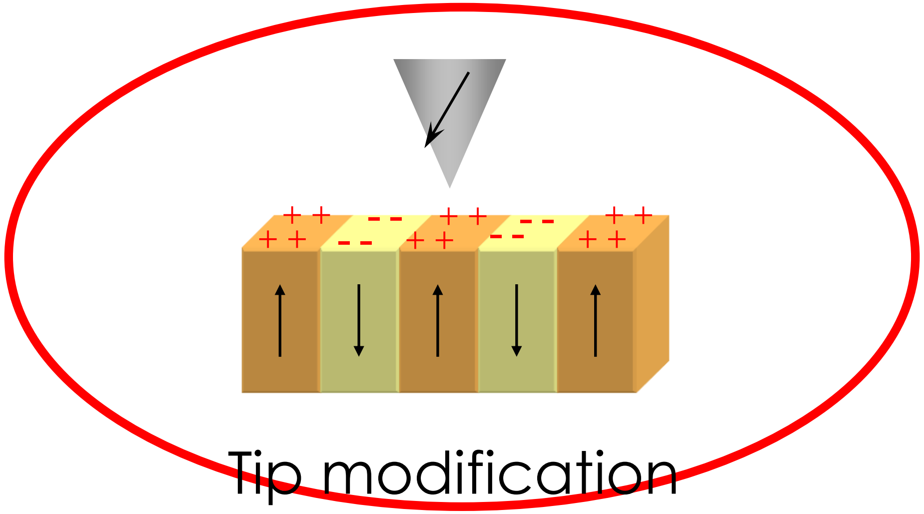
+

Hysteresis minor loops

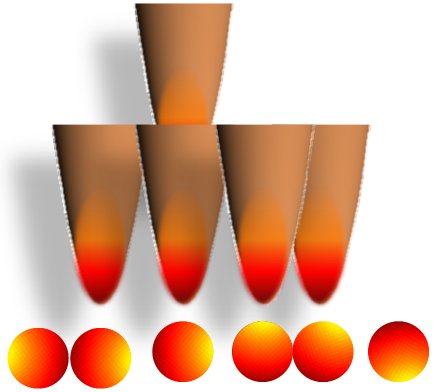


=

Dissipation of energy!!!!!



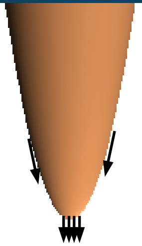
Tip modification



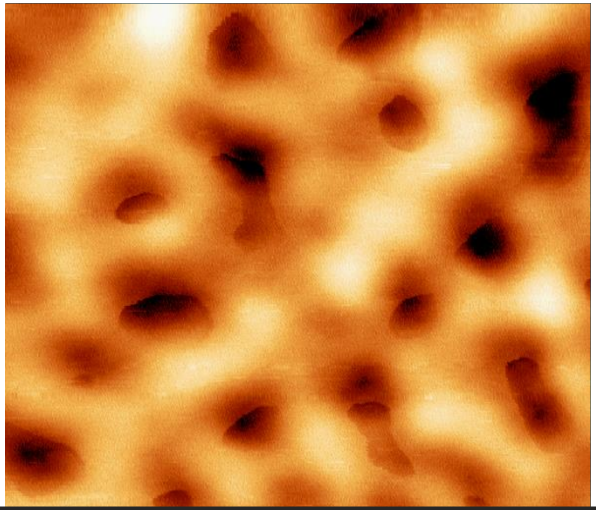
Sample modification

Two experiments in different conditions….

Air, Amplitude modulation
Two scan technique

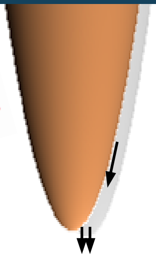


Standard MFM probe



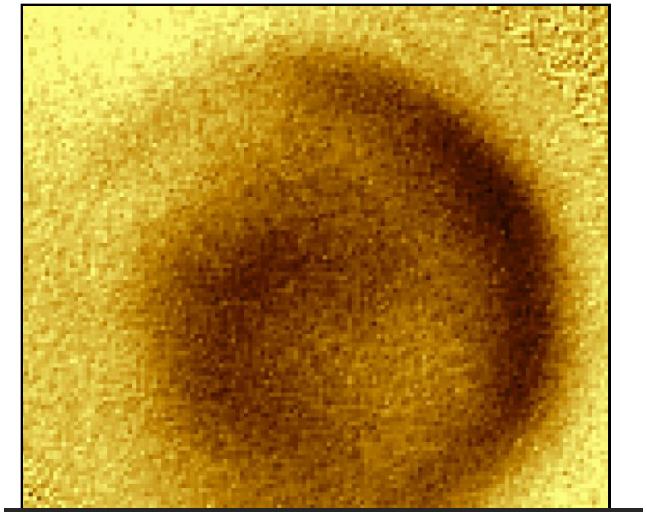
Co/Ni multilayer. Stripe domains

HV, Frequency modulation
+ Amplitude constant



One face-coated probe

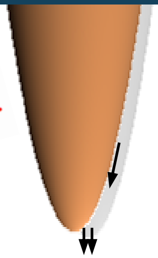
In plane magnetic field



Py dots. Vortex state

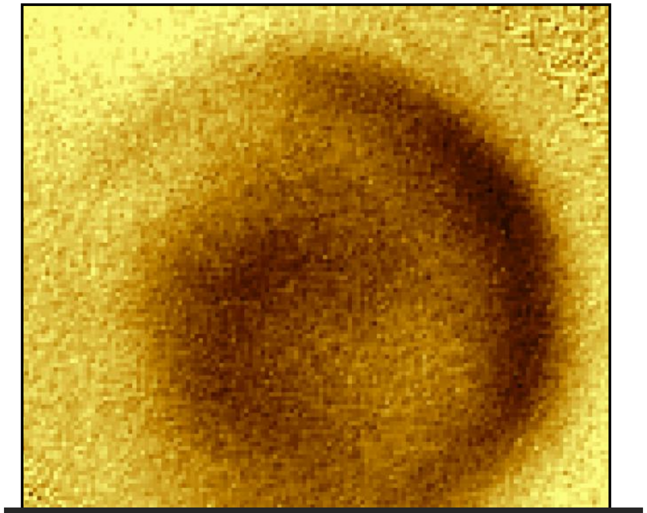
Two experiments in different conditions….

HV, Frequency modulation
+ Amplitude constant



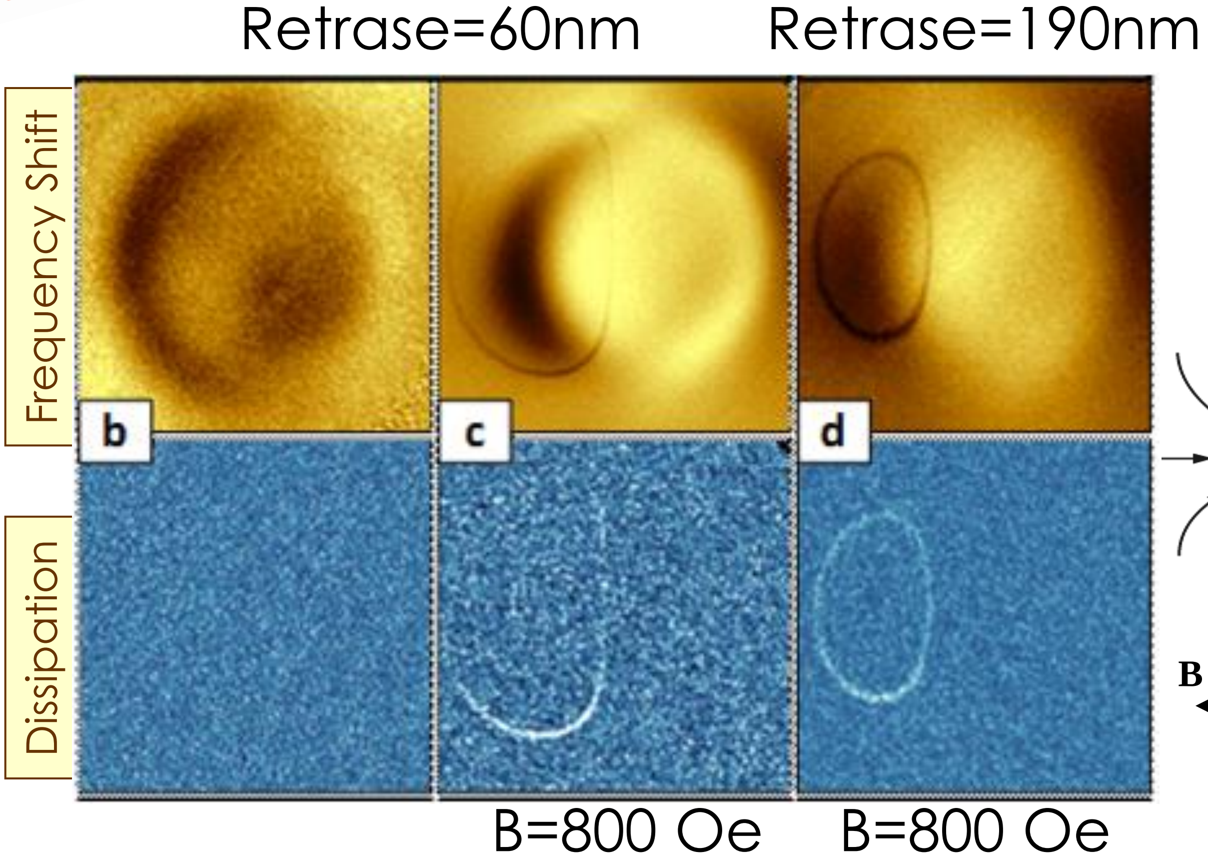
One face-coated probe

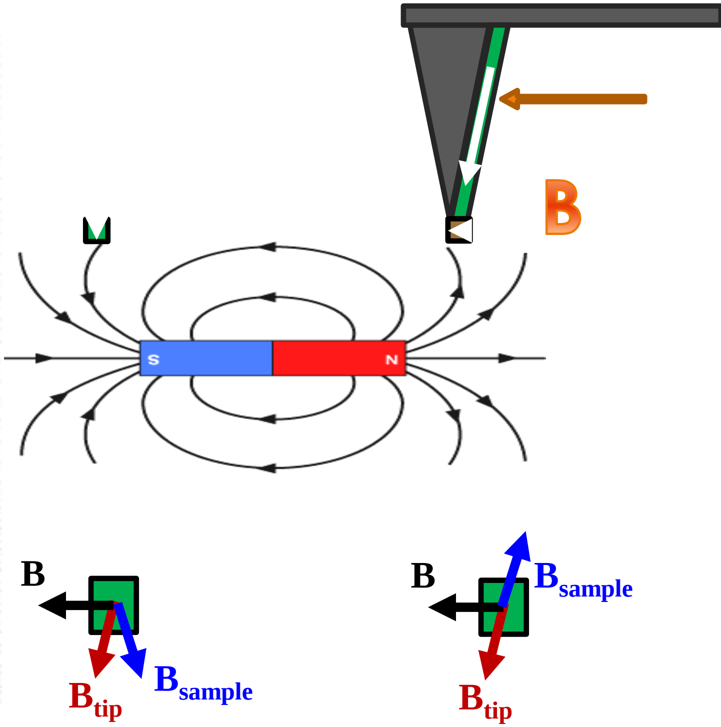
In plane magnetic field



Py dots. Vortex state

Tips for mapping the magnetic field

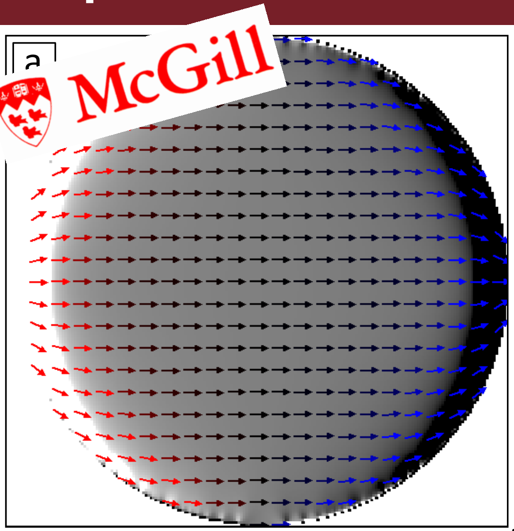


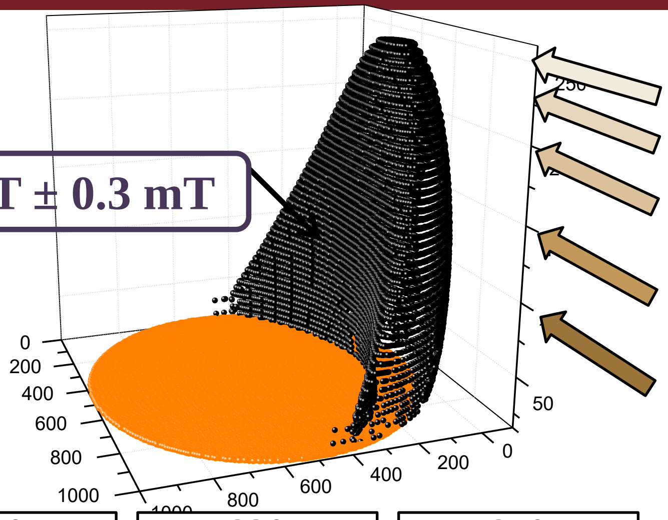


A ring appears in one side when the **Py dot is saturated** under in-plane magnetic field.

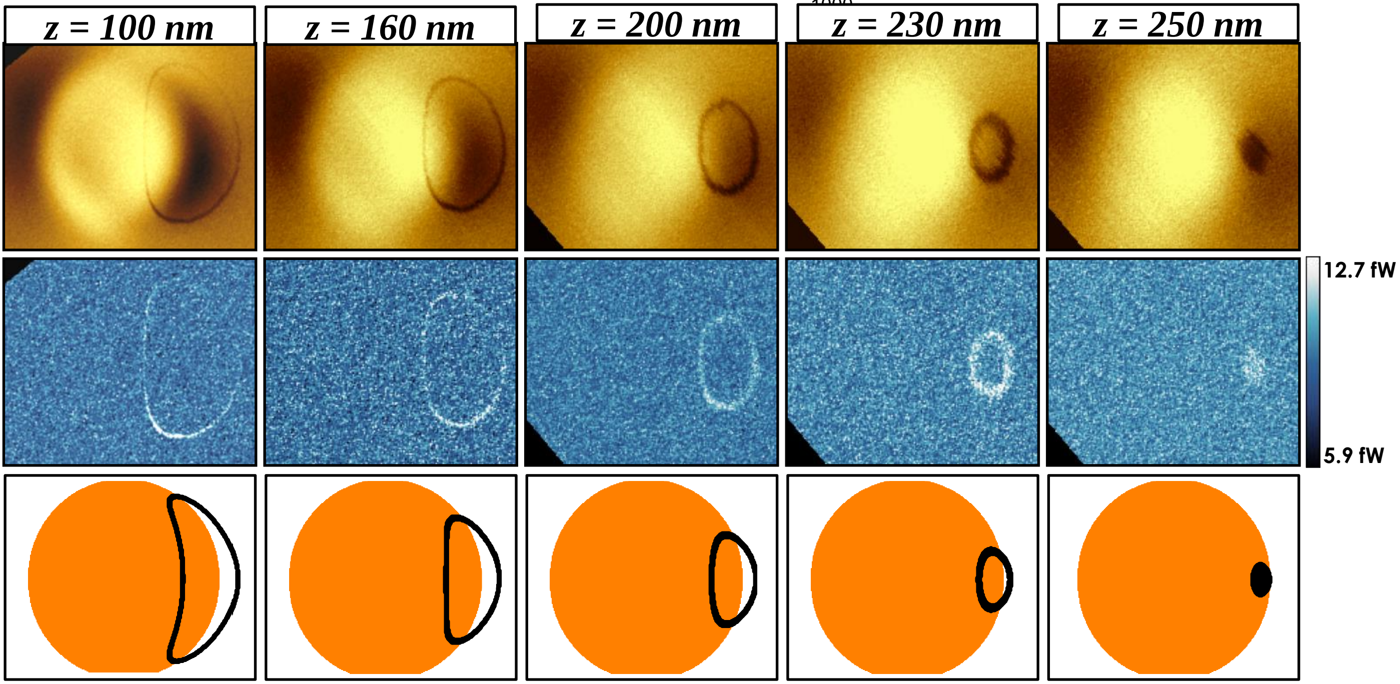
Ó. Iglesias-Freire , J. Bates, Y. Miyahara, A. Asenjo and P. Grütter , Appl. Phys. Lett. 102, 022417 (2013)

Tips for mapping the magnetic field





B_z = =-14.0 mT ± 0.3 mT



z = 100 nm | *z = 160 nm* | *z = 200 nm* | *z = 230 nm* | *z = 250 nm*

Frequency Shift

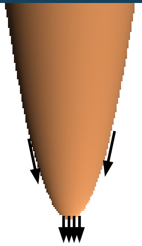
Dissipation

Simulation

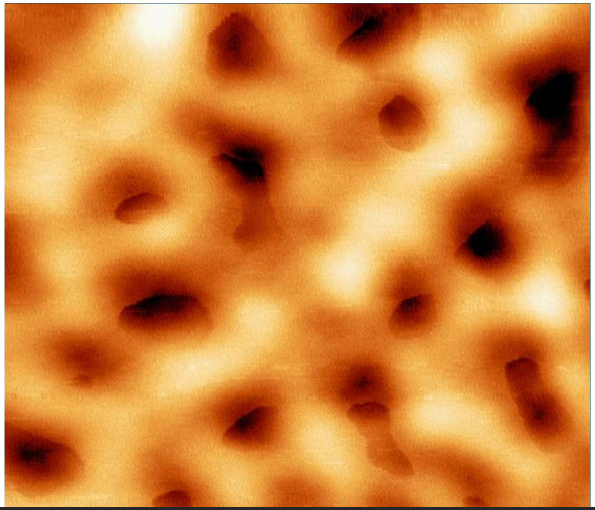
Ó. Iglesias-Freire , J. Bates, Y. Miyahara, A. Asenjo and P. Grütter , Appl. Phys. Lett. 102, 022417 (2013)

Two experiments in different conditions….

Air, Amplitude modulation
Two scan technique



Standard MFM probe



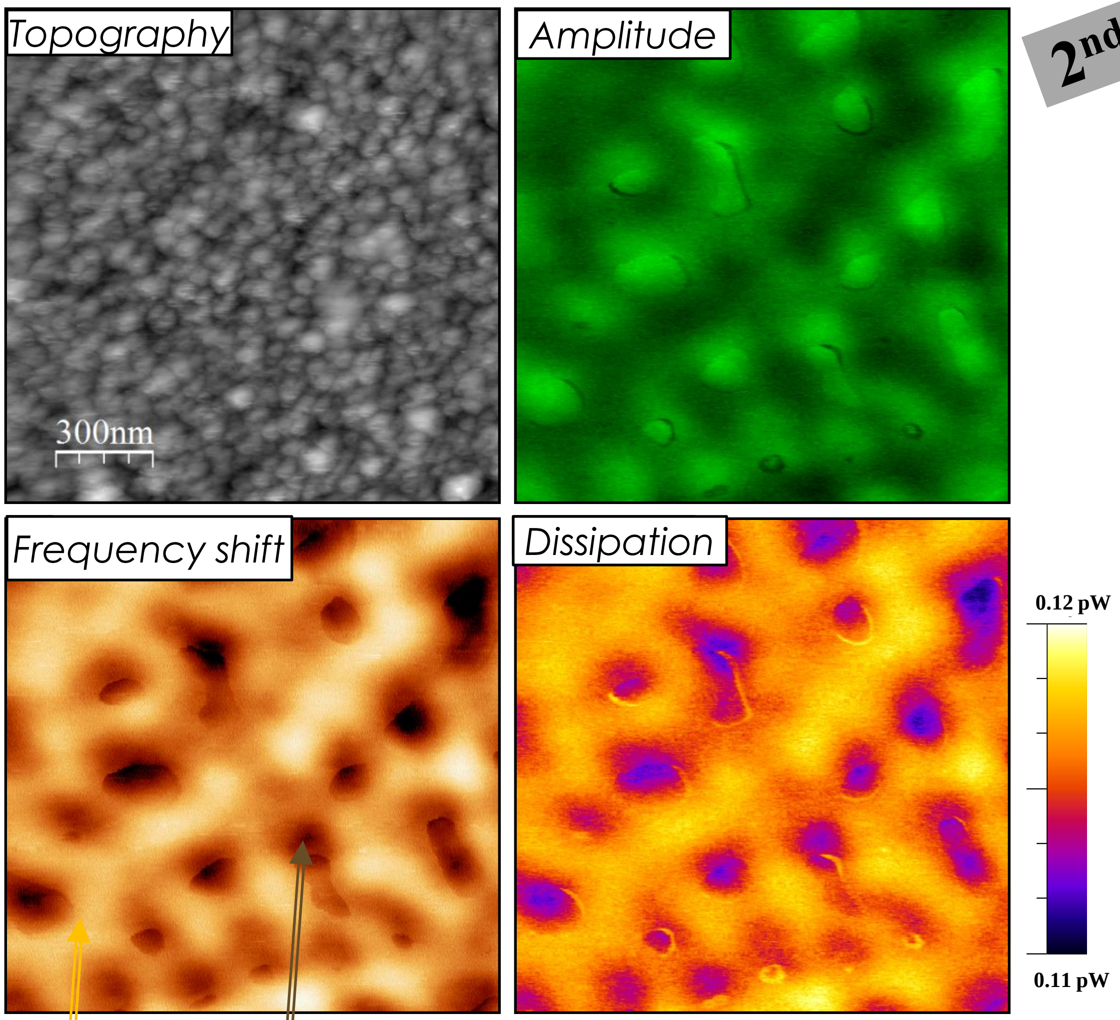
Co/Ni multilayer. Stripe domains

Experimentally accessible

$$\langle P_{tip}\rangle = \frac{1}{2}\cdot\frac{k\cdot A^2\cdot\omega}{Q}\cdot\left[\frac{A_0}{\boxed{A}} - \frac{\boxed{\omega}}{\omega_0}\right]$$

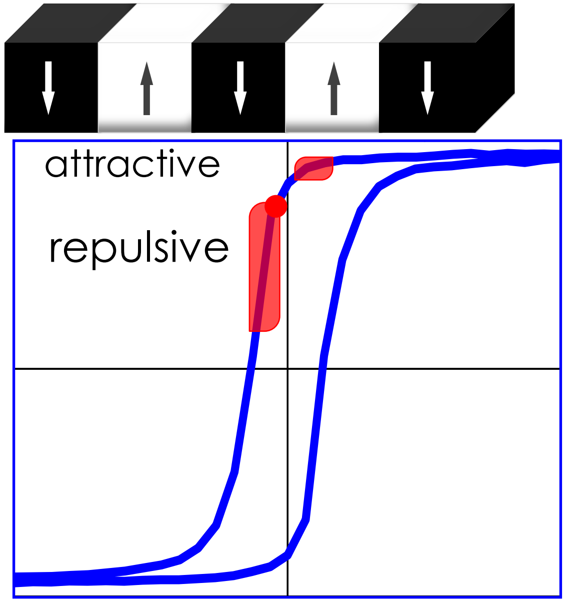
Dissipation in MFM. Co / Ni multilayer

2nd scan



Attractive=paralell

Repulsive=antiparalell



H_C = 165 mT

$M_{rem} = 0.87\cdot M_S$

Domain size ≈140 nm

Nanosensors MFM

Low humidity

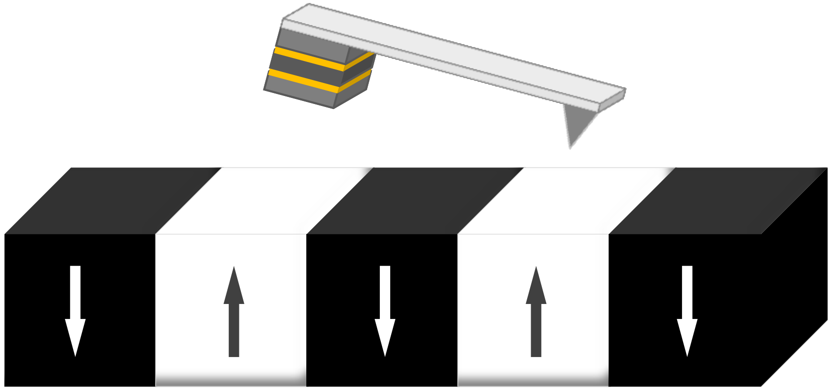
M. Jaafar et al. Nanoscale, 8, 16989-16994 (2016))

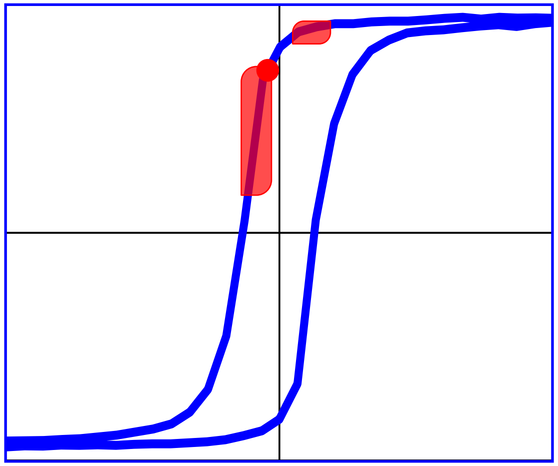
Dissipation in MFM. Co / Ni multilayer

$$P_{input} = P_{cantilever} + P_{tip}$$

Experimentally accessible

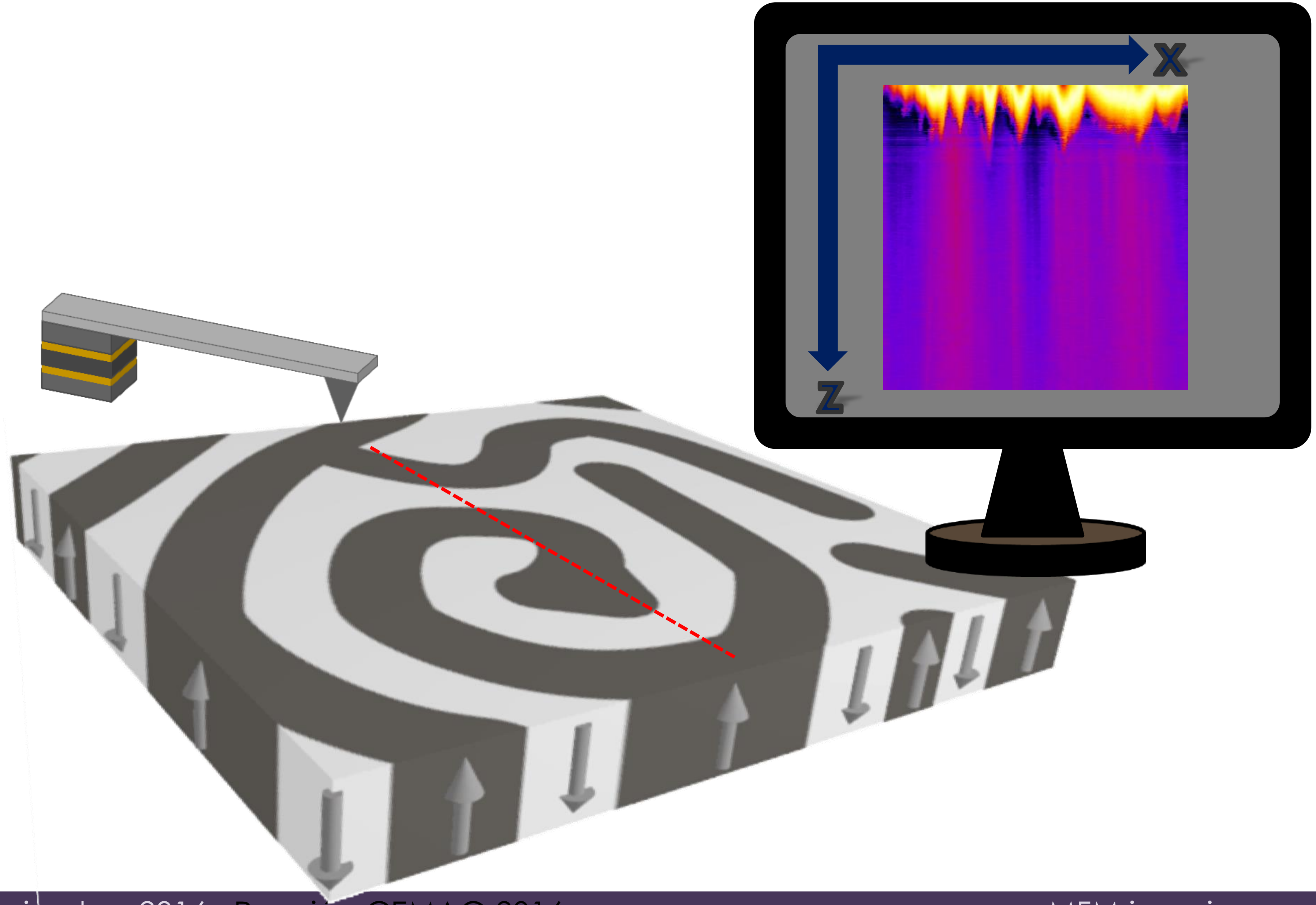
$$\boxed{\langle P_{tip}\rangle = \frac{1}{2}\cdot\frac{k\cdot A^{2}\cdot\omega}{Q}\cdot\left[\frac{A_0}{\boxed{A}} - \frac{\boxed{\omega}}{\omega_0}\right]}$$





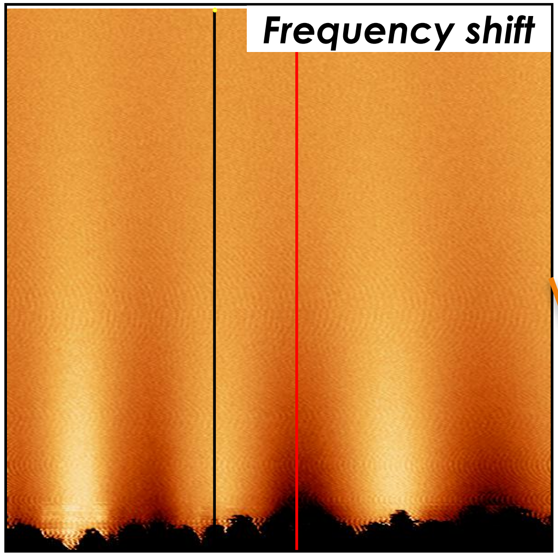
J. P. Cleveland et al., Appl. Phys. Lett. 72 (1998) 2613

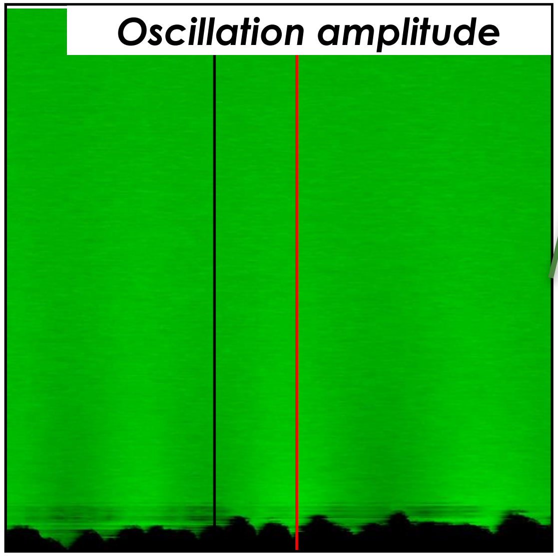
Dissipation in MFM. Co / Ni multilayer



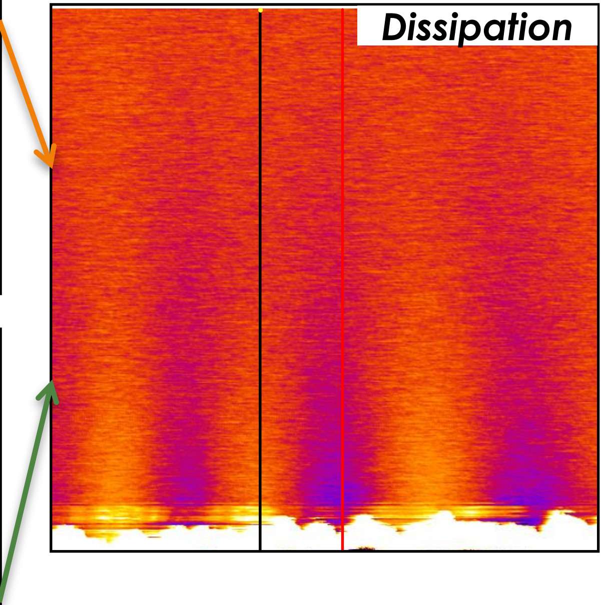
Dissipation in MFM. Co / Ni multilayer

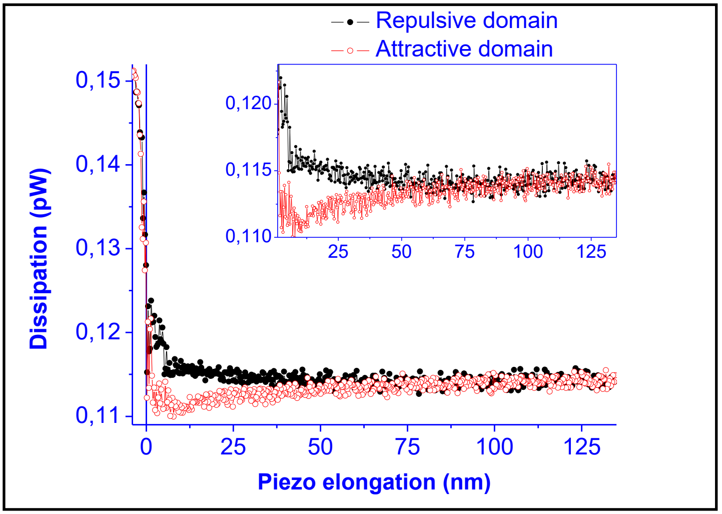
Tip-sample distance *(Δz = 150 nm)*

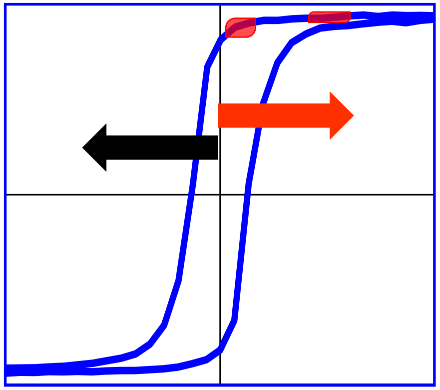




$$\langle P_{tip}\rangle = \frac{1}{2}\cdot\frac{k\cdot A^2\cdot\omega}{Q}\cdot\left[\frac{A_0}{A}-\frac{\omega}{\omega_0}\right]$$







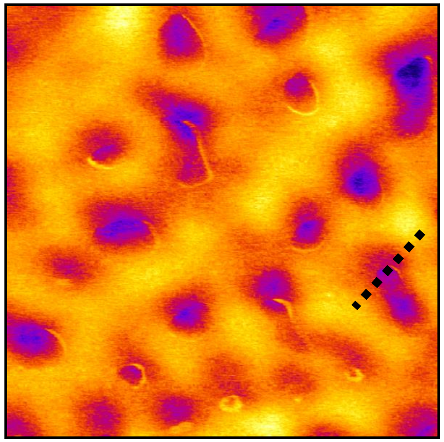
•Dissipated power of the order of 10^2 fW is partially attributed to rotations of large amounts of spins at **the tip apex**.

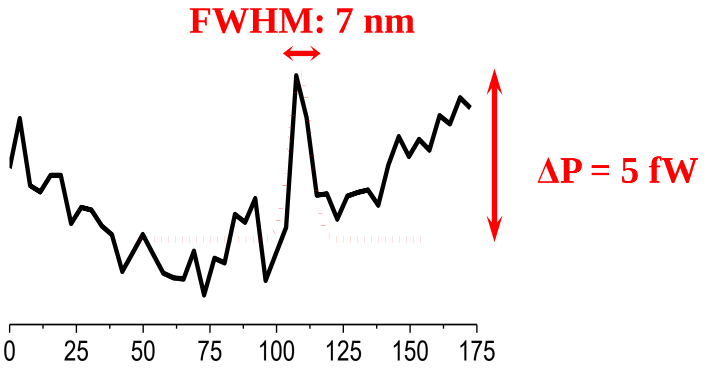
M. Jaafar et al. Nanoscale, 8, 16989-16994 (2016))

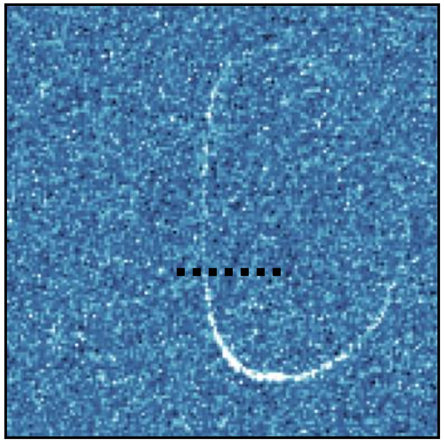
Dissipation in MFM

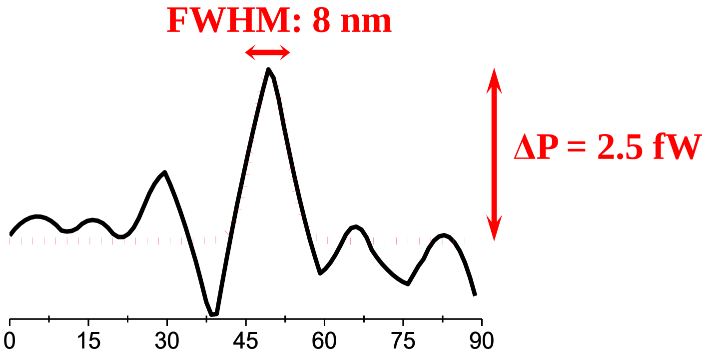
- Power losses of **few fW** → sudden rotations of spins at the apex
- Lateral resolution **below 10 nm** is achieved

M. Jaafar et al. Nanoscale, 8, 16989-16994 (2016))





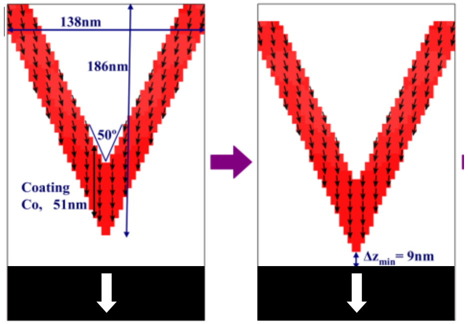


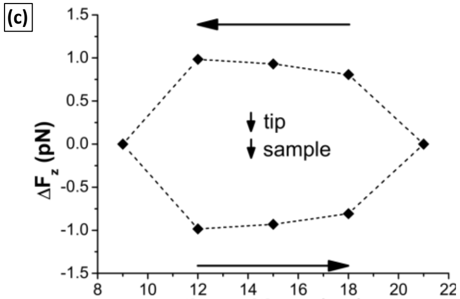


Micromagnetic simulations:

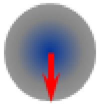
P(attractive) ≈ 1.2 fW

P(repulsive) ≈ 1.4 fW





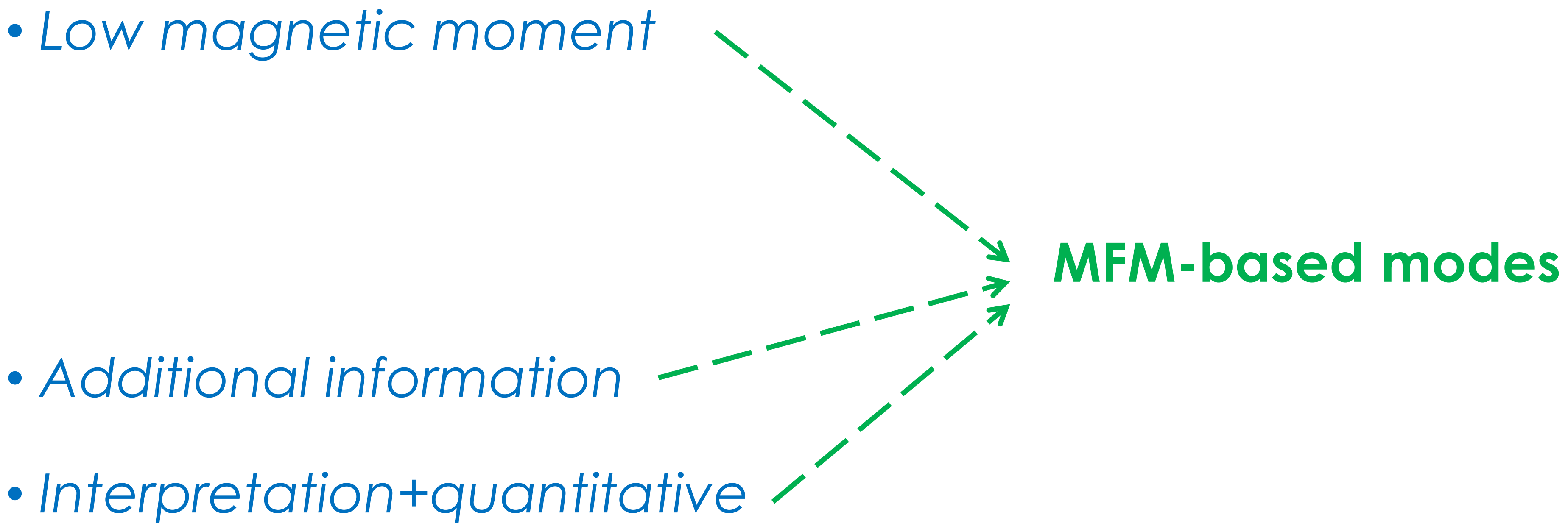
$V \approx 2 \cdot 10^{-24}$ m^3



8 nm

Some challenges in MFM

- **KPFM-MFM** combination: distinguishing signals, necessary for low moment samples
- **Dissipation:** magnetization & resolution

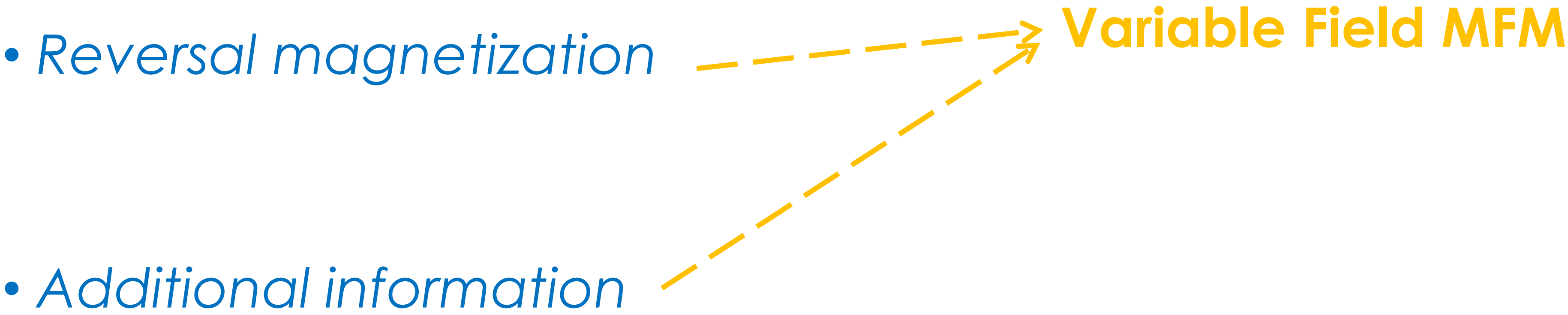


- *Low magnetic moment*
- *Additional information*
- *Interpretation+quantitative*

MFM-based modes

……..from hard disk to bacteria

Some challenges in MFM



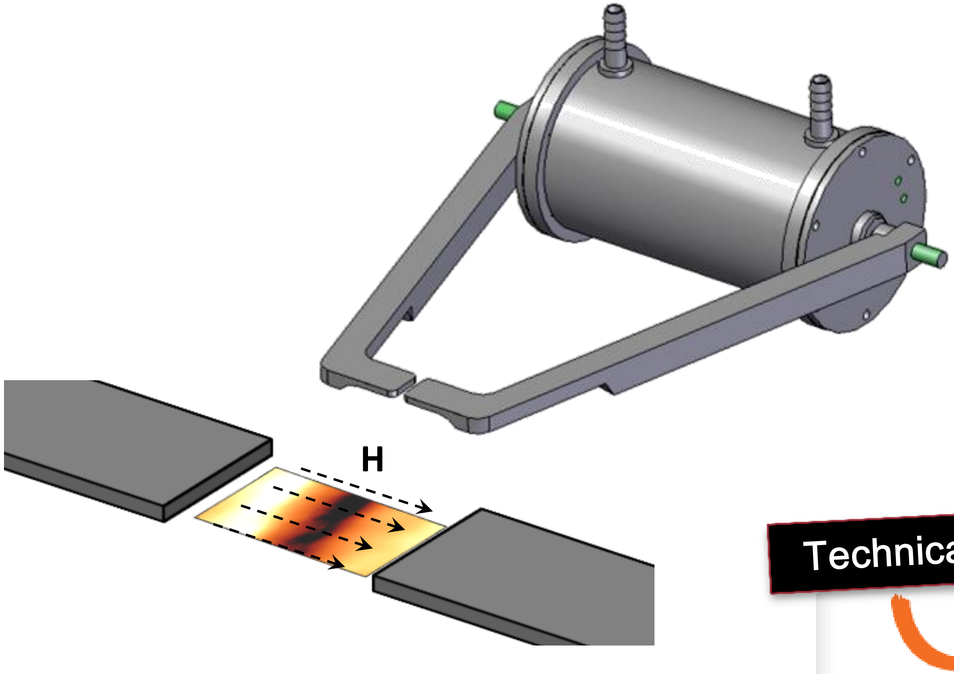
- *Reversal magnetization*
- *Additional information*

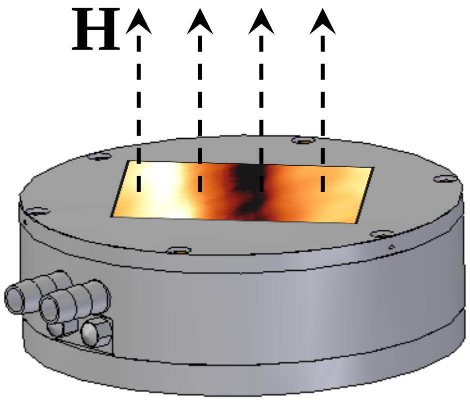
Variable Field MFM

……..from hard disk to bacteria

Operation mode in MFM. Variable field

	In Plane	*Out of Plane*
Maximum Field	**150 mT**	**100 mT**
Thermal Stability *(after 3 hours under $B=B_{max}/2$)*	**+ 2 °C**	**+ 4 °C**
Mechanical Stability	**0.1 nm/mT**	**1 nm/mT**



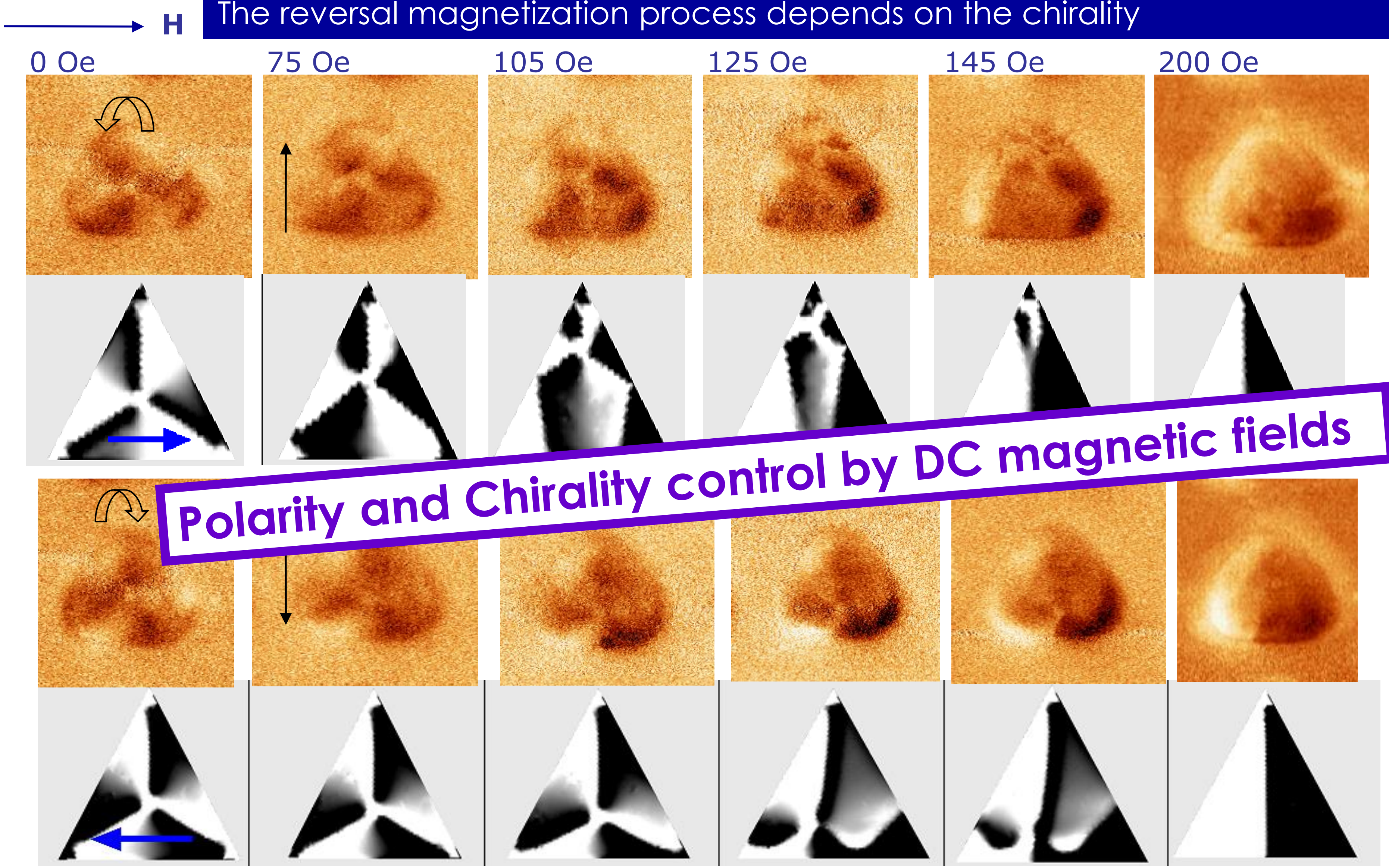


Technical support

NANOTEC
ELECTRONICA

VFMFM. Ni (111) Nanostructures

The reversal magnetization process depends on the chirality

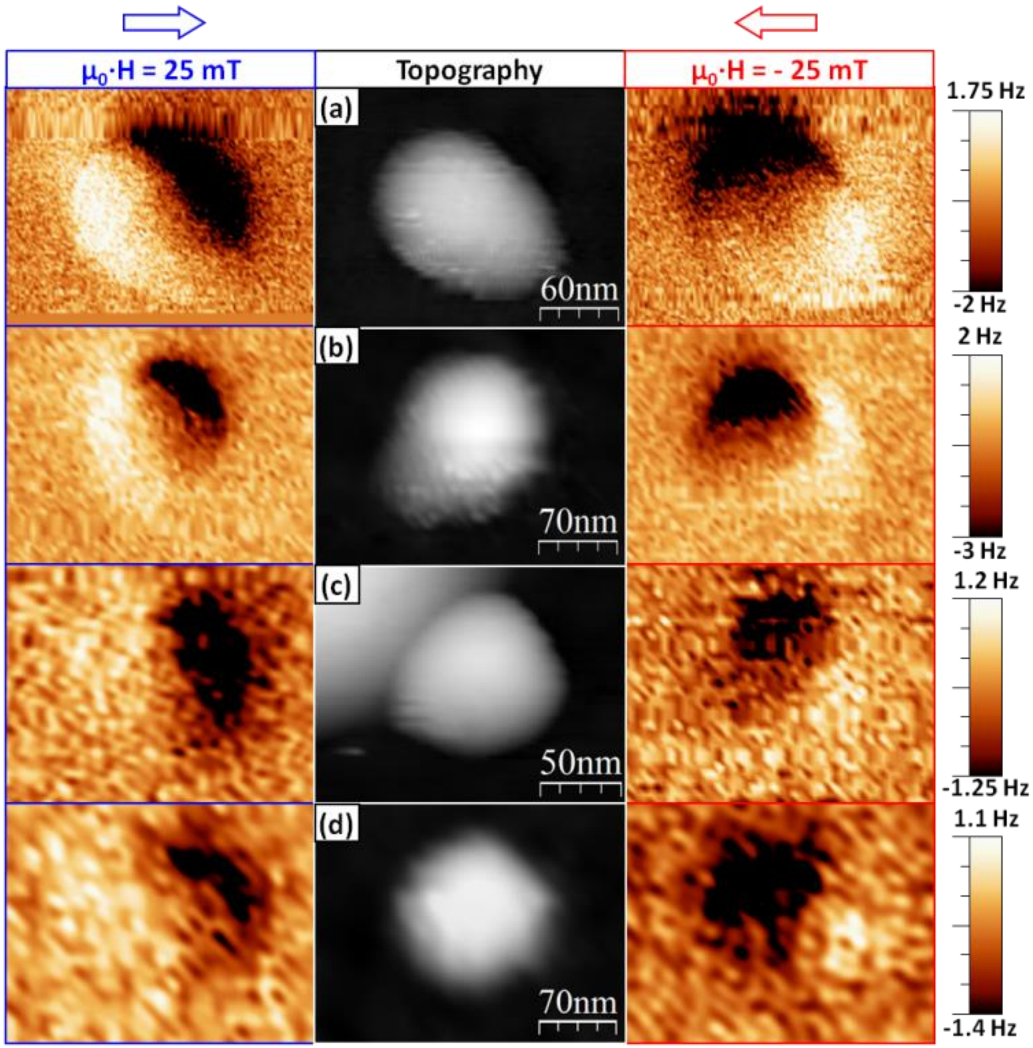


Polarity and Chirality control by DC magnetic fields

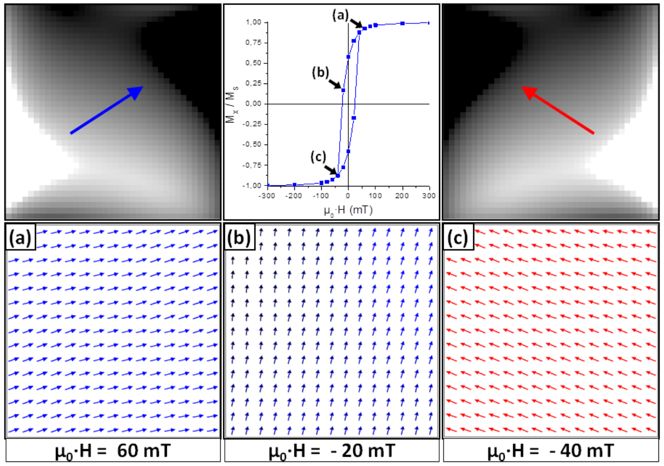
M Jaafar et al. Nanotechnology 19 (2008) 285717

VFMFM. Isolated cubic $Fe_{3-x}O_4$ NP (25nm)

MFM images under in-plane magnetic fields.

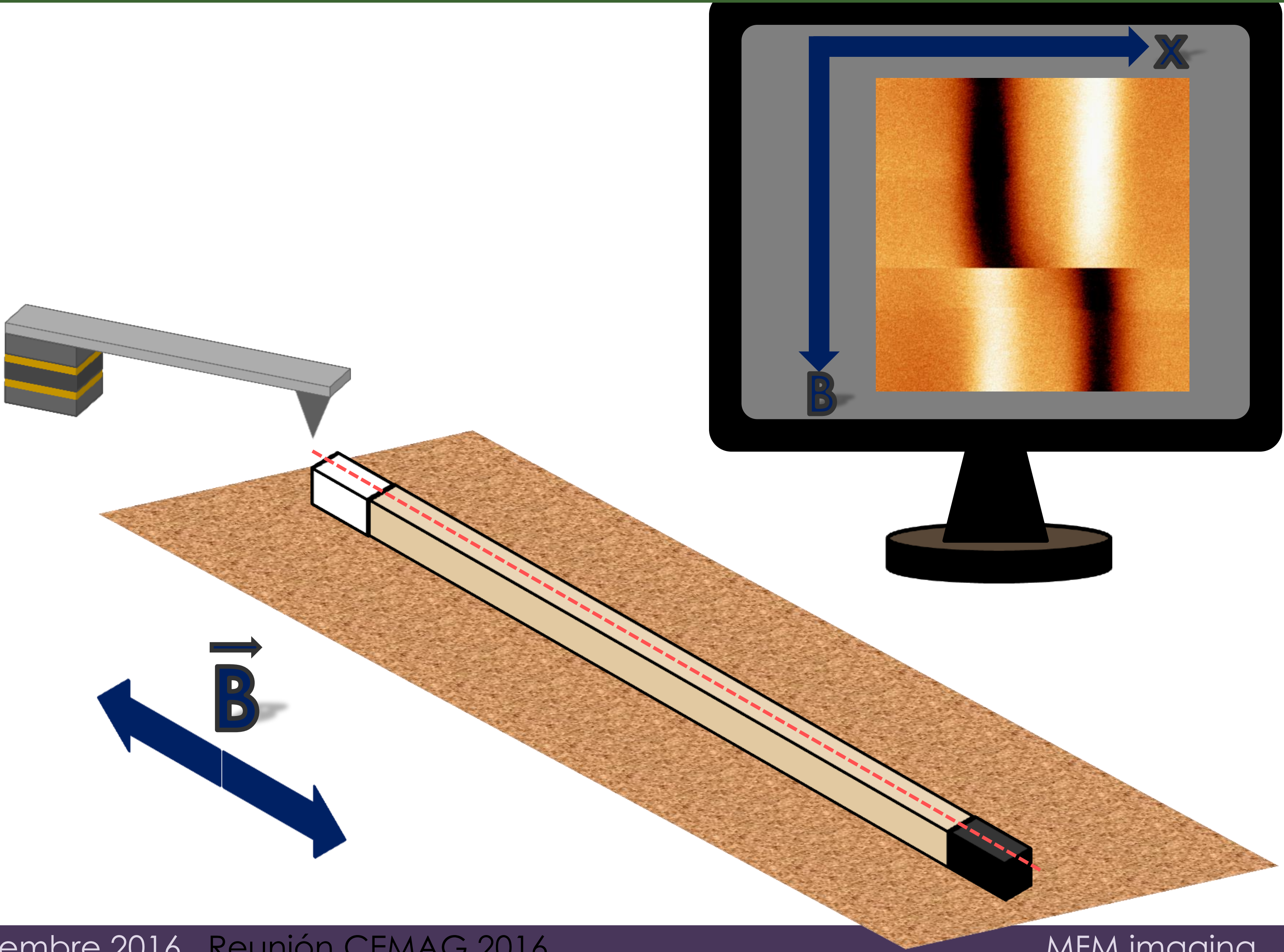


Orientation and polarity are determined by both their magnetocrystalline easy axes and previous magnetic history.



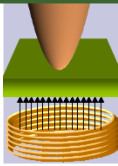
C. Moya, O. Iglesias-Freire, N. Pérez, X. Batlle, A. Labarta, A. Asenjo, Nanoscale, 7, 8110-8114 (2015)

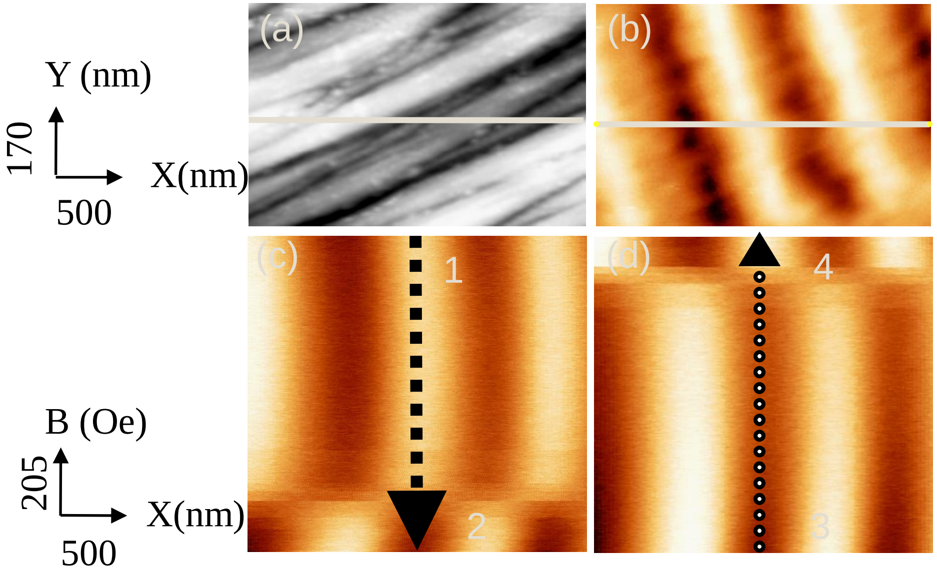
VFMFM: 3D mode images.



In situ hysteresis loops of MFM probes

New method to characterize the tips, faster and more precise

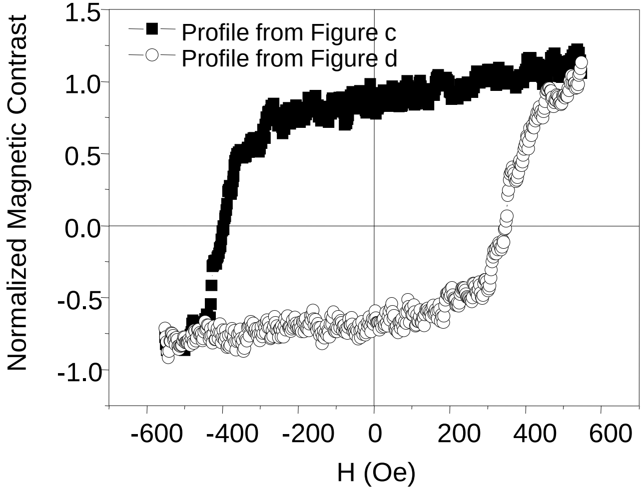


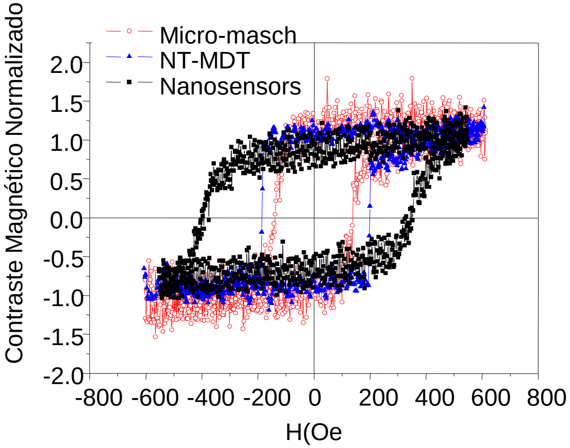


$$F_{magnetic} = m_{tip} \bullet \nabla H_{sample}$$

If m_{sample} does not change, the force gradient is proportional to the m_{tip} .

From images at different magnetic fields we obtain m_{tip} versus H



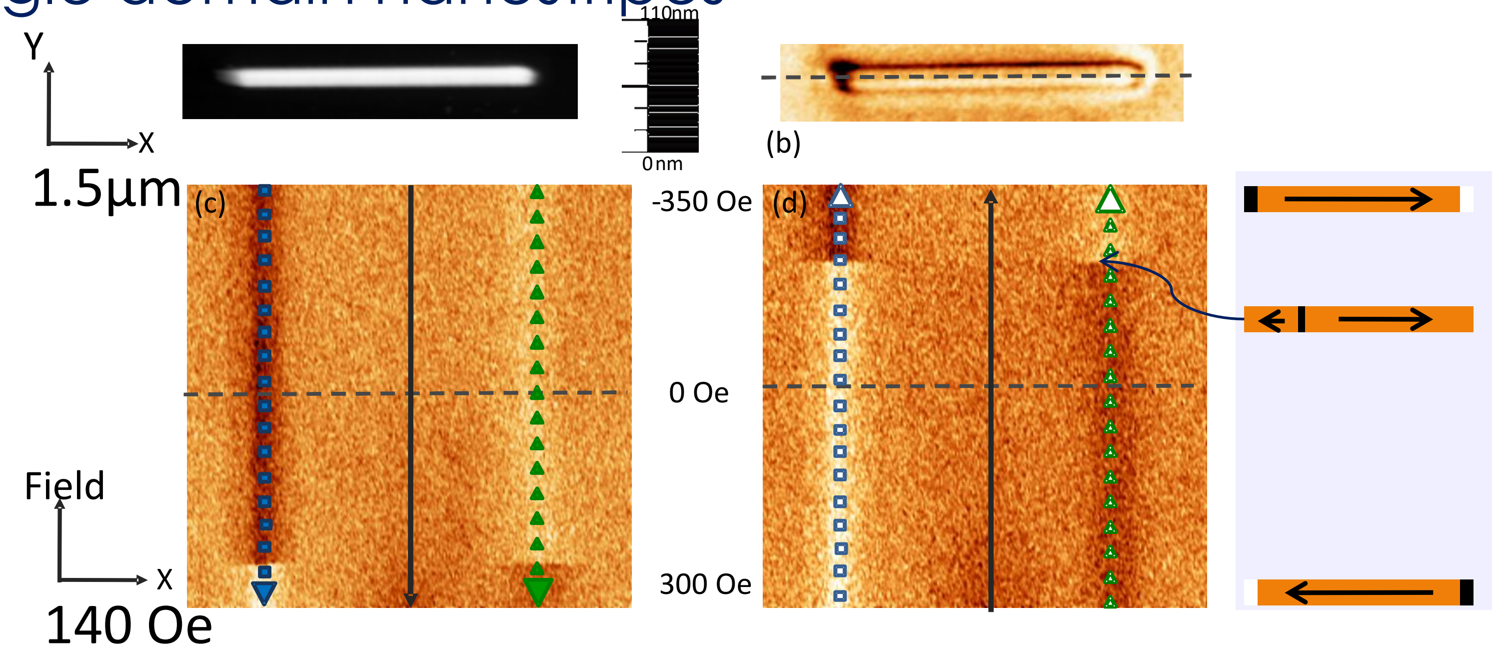


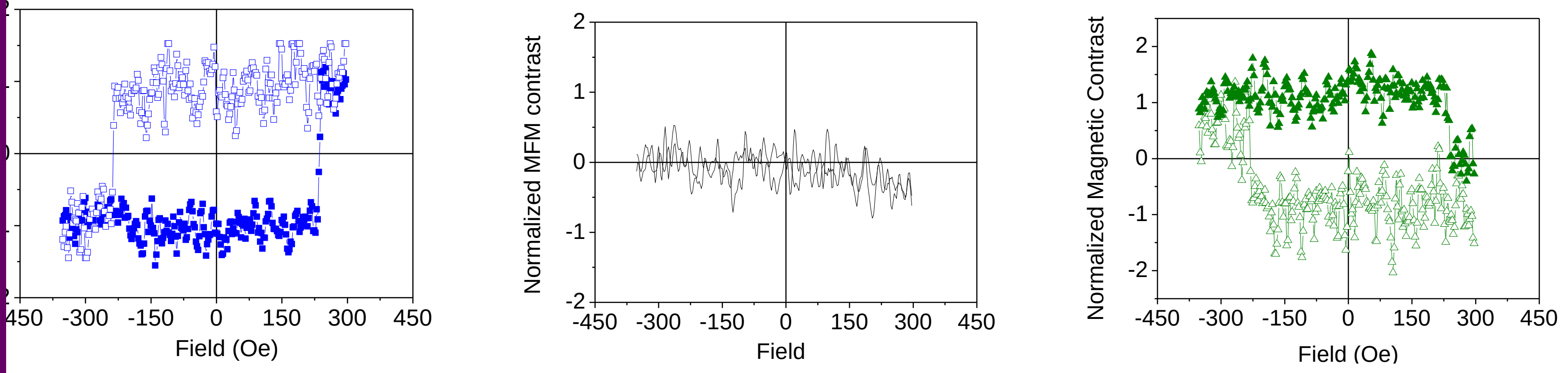
M. Jaafar, J. Gómez-Herrero, A. Gil, P. Ares, M. Vázquez and A. Asenjo, Ultramicroscopy 109 (2009) 693

VFMFM: *In situ* hysteresis loops of Co nanostripes

In the case of single domain configuration, we can obtain the Hc from the 3D mode images

Single domain nanostripes

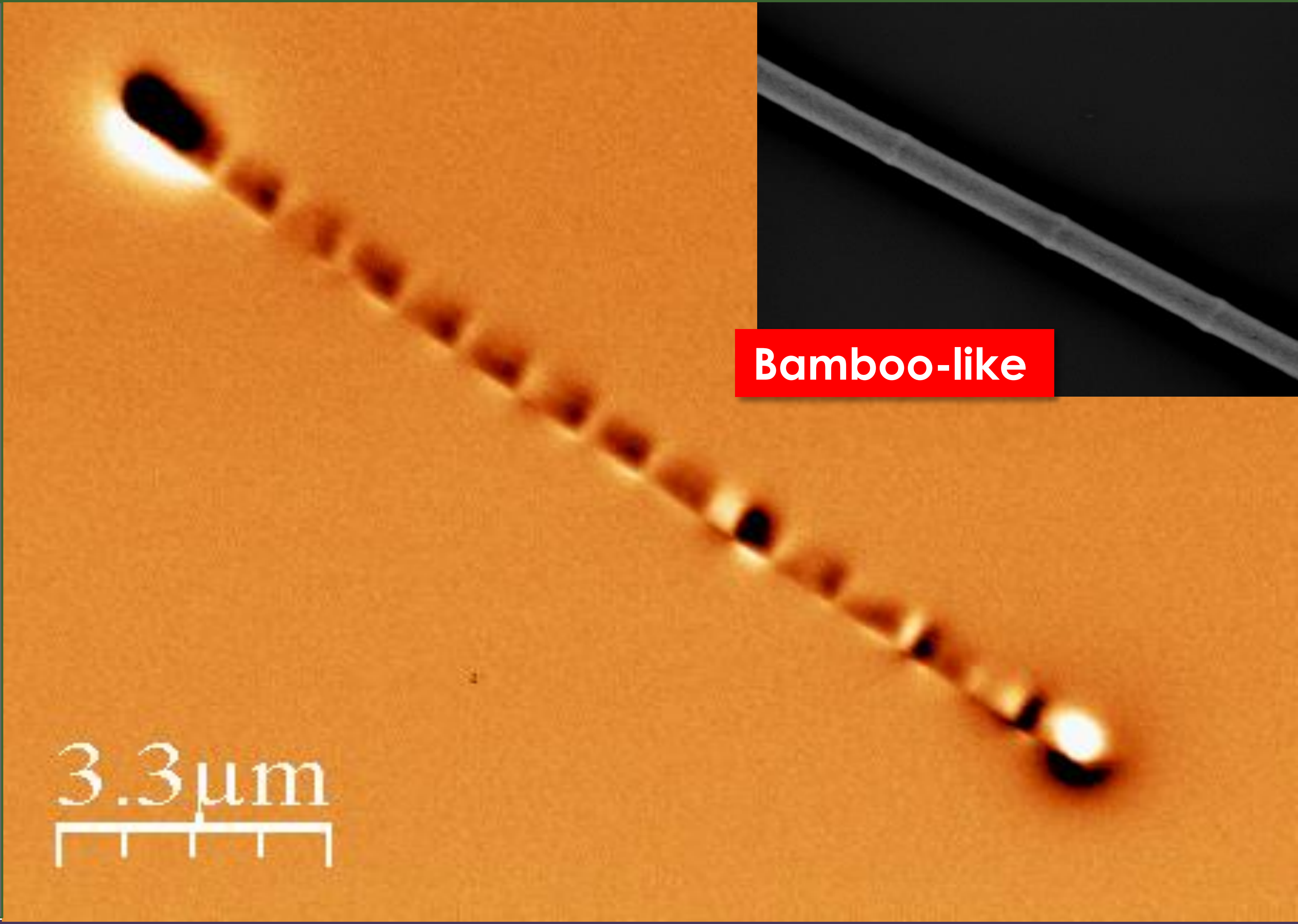




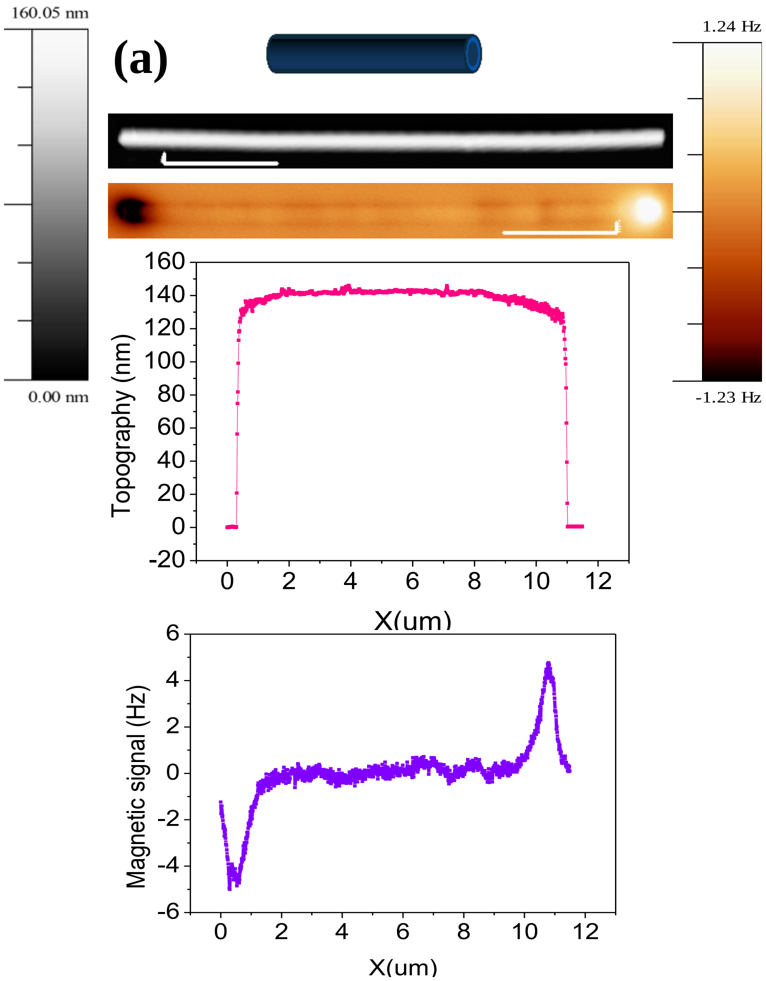
M. Jaafar, O. Iglesias-Freire, L. Serrano-Ramón, M. R. Ibarra, J. M. de Teresa and A. Asenjo, BJ Nano 2 (2011) 552

FeCoCu bamboo-like nanowires

Nanowires with modulated diameter (D~130-140nm)

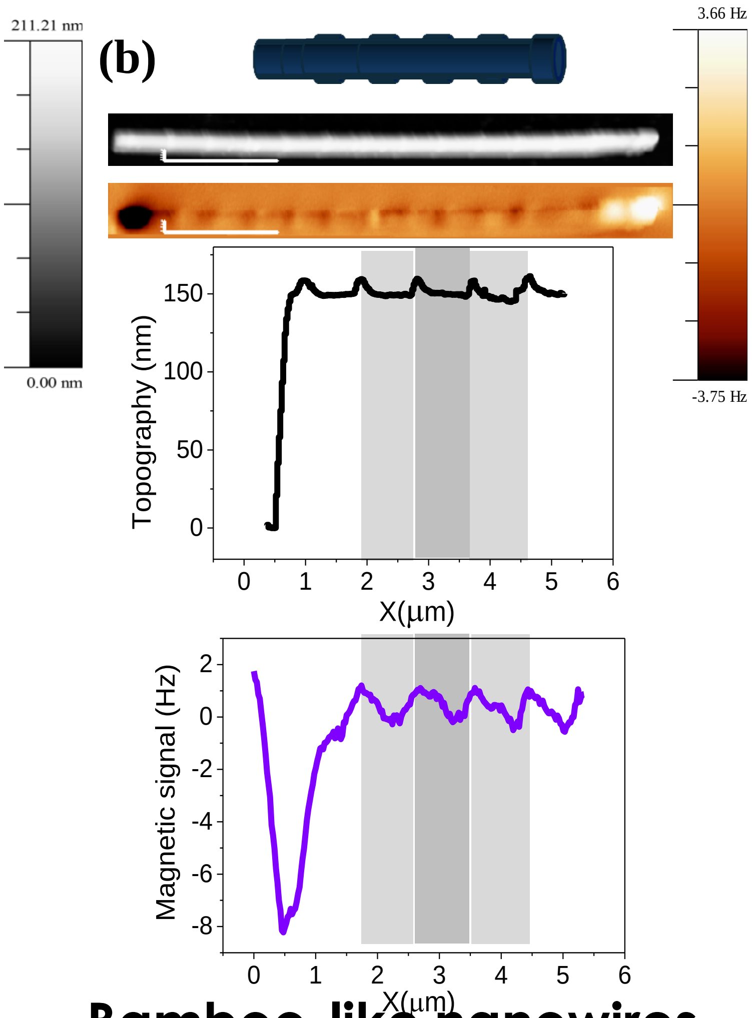


FeCoCu bamboo-like nanowires



Straigh nanowires

Dipolar contrast

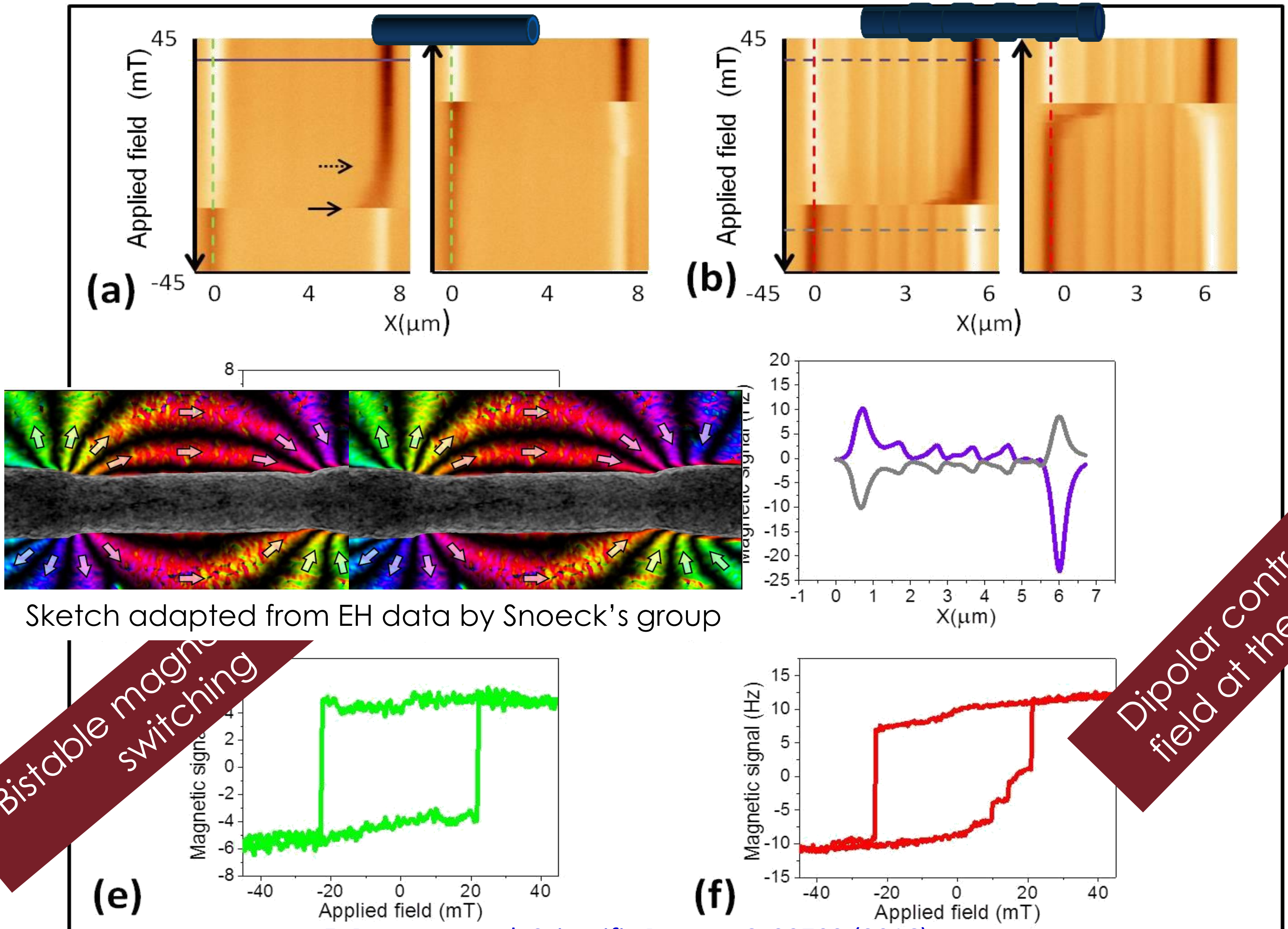


Bamboo-like nanowires

Dipolar +periodic contrast

E. Berganza et al. Scientific Reports 6, 29702 (2016)

FeCoCu bamboo-like nanowires



Sketch adapted from EH data by Snoeck's group

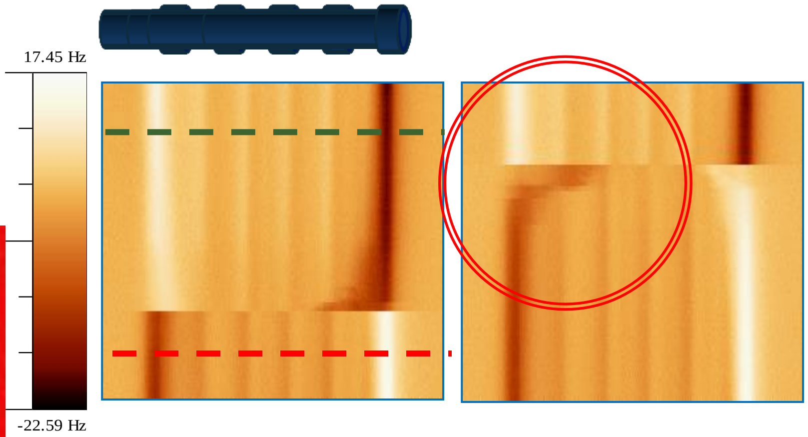
Bistable magne
switching

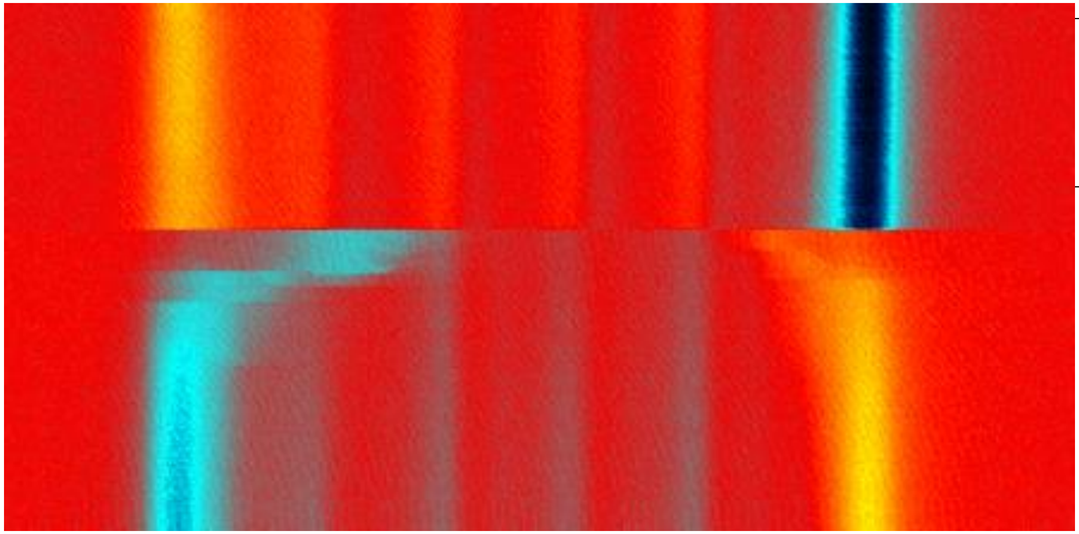
Dipolar contrast + stray
field at the modulation

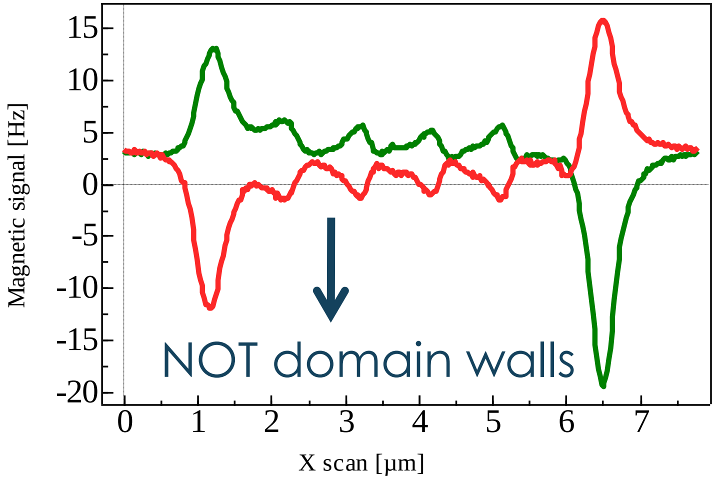
E. Berganza et al. Scientific Reports 6, 29702 (2016)

FeCoCu bamboo-like nanowires

Pinning is observed in several NW at the modulations **close to the edges**







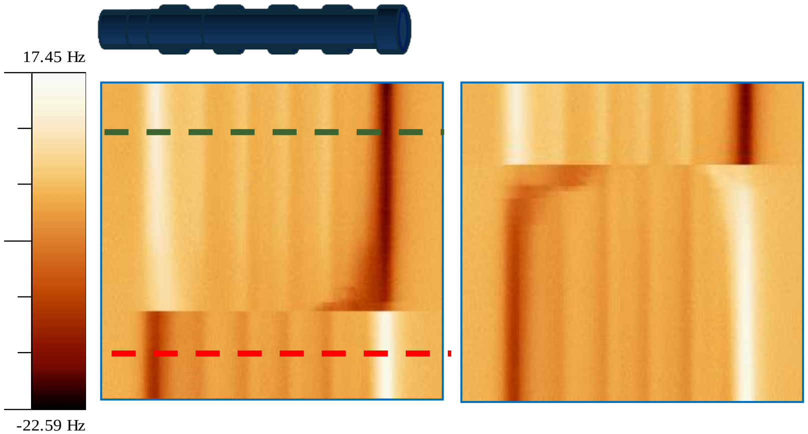
NOT domain walls

Dipolar contrast + stray field at the modulation

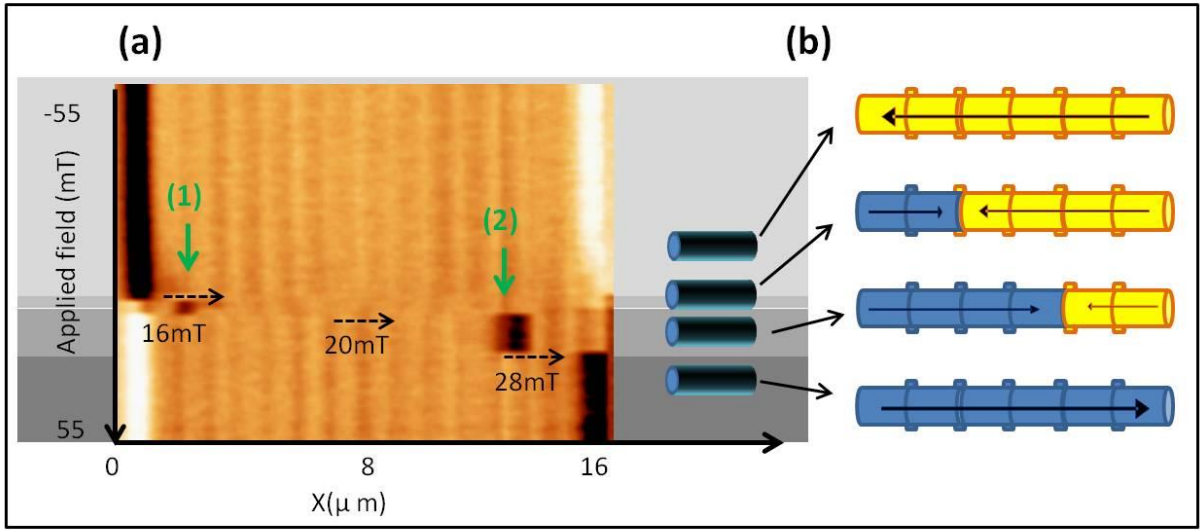
E. Berganza et al. Scientific Reports 6, 29702 (2016)

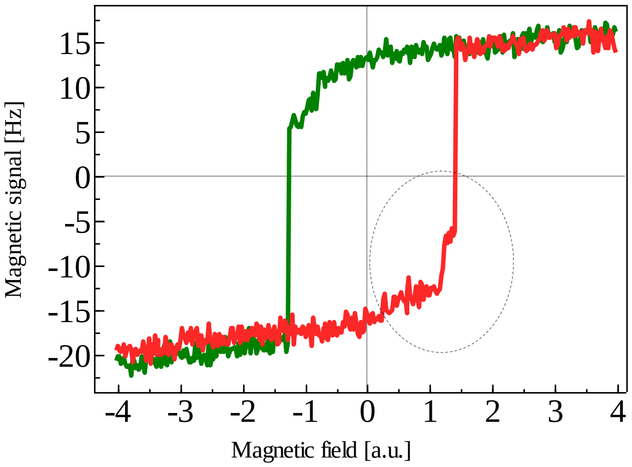
FeCoCu bamboo-like nanowires

Pinning is observed in several NW at the modulations **close to the edges.**



In some cases, the pinning of the domain is observed





This curve is not the standard hysteresis loop

E. Berganza et al. Scientific Reports 6, 29702 (2016)

Some challenges in MFM

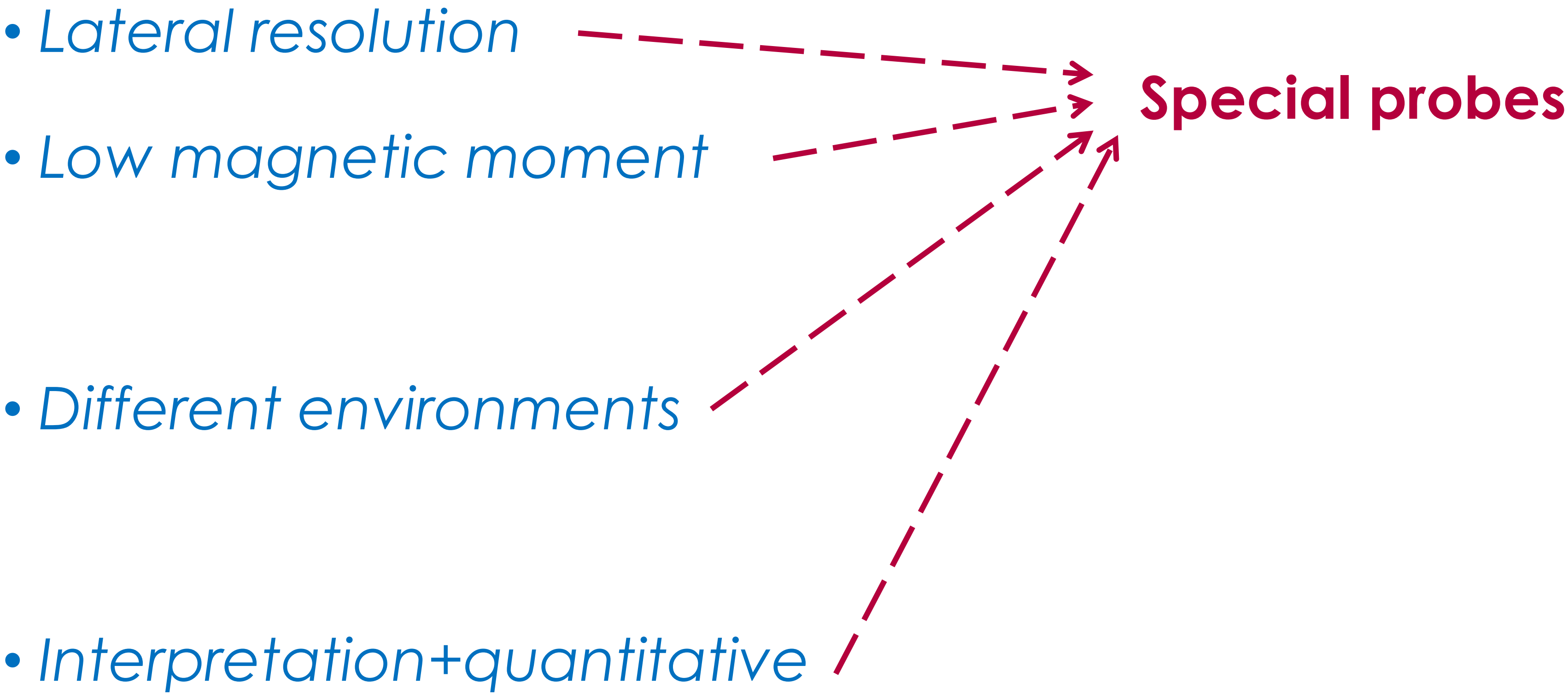
- *Reversal magnetization*
- *Additional information*

Variable Field MFM

Determine the domain configuration
A sort of hysteresis loop
Quantify critical fields

……..*from hard disk to bacteria*

Some challenges in MFM



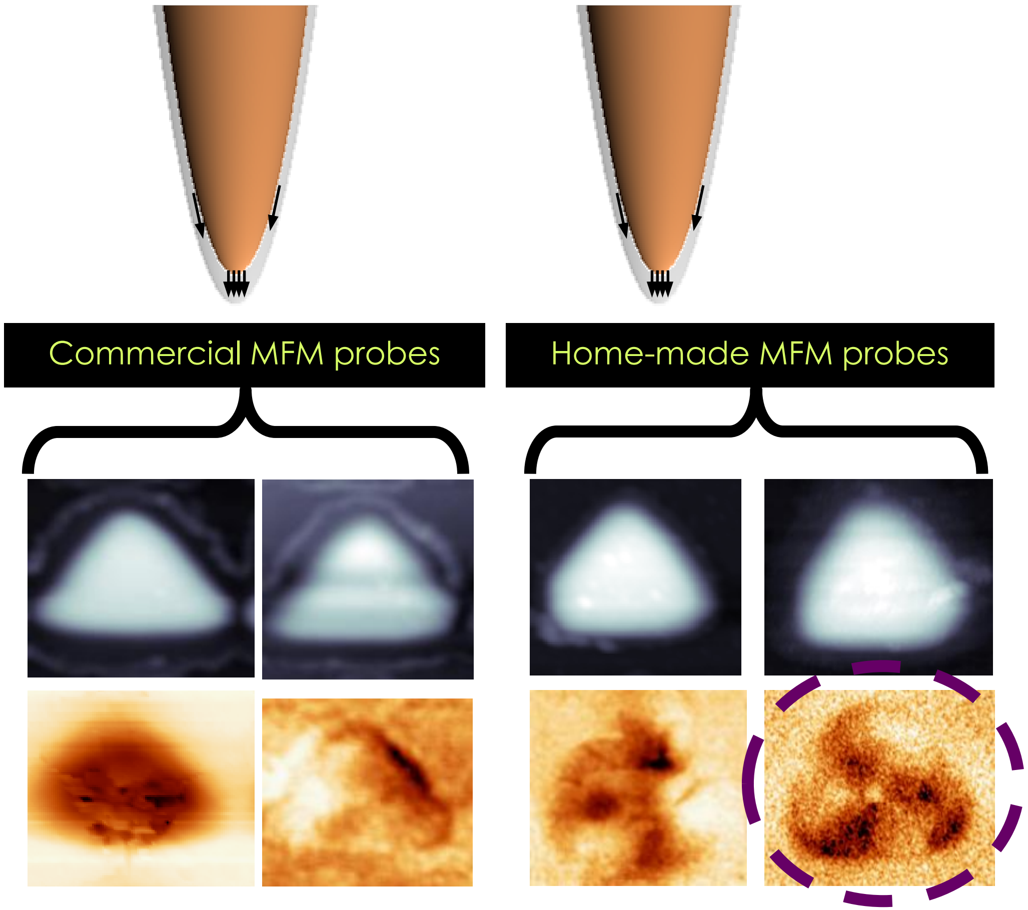
- *Lateral resolution*
- *Low magnetic moment*
- *Different environments*
- *Interpretation+quantitative*

Special probes

……..from hard disk to bacteria

Importance of the tips.

Home-made MFM probes by coating the commercial tips with a magnetic layer.



Commercial MFM probes

Home-made MFM probes

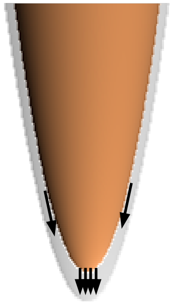
50 nm CoCr 15 nm CoCr 35 nm CoCr 20 nm CoCr

Ni triangules, side 500nm

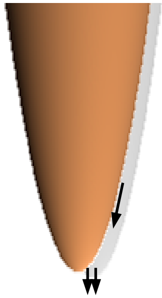
M. Jaafar , A. Asenjo, M. Vázquez, *IEEE Nano* **7** (2008) 245

Special MFM probes

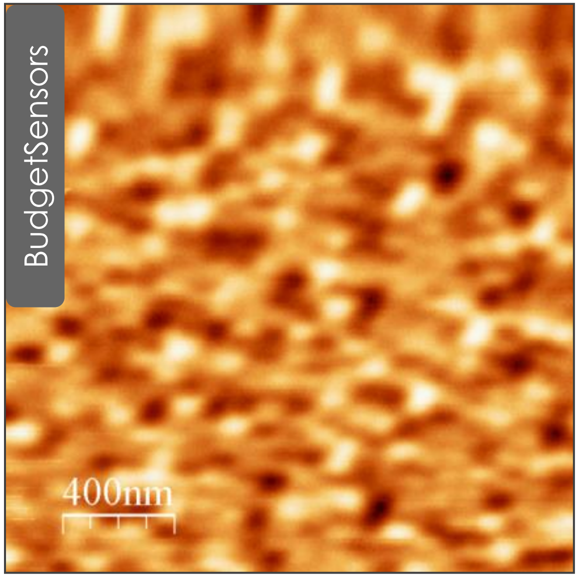
Home-made MFM probes by coating the commercial tips with a magnetic layer.
Standard and One face-coated. Sample, **high density hard disk.**

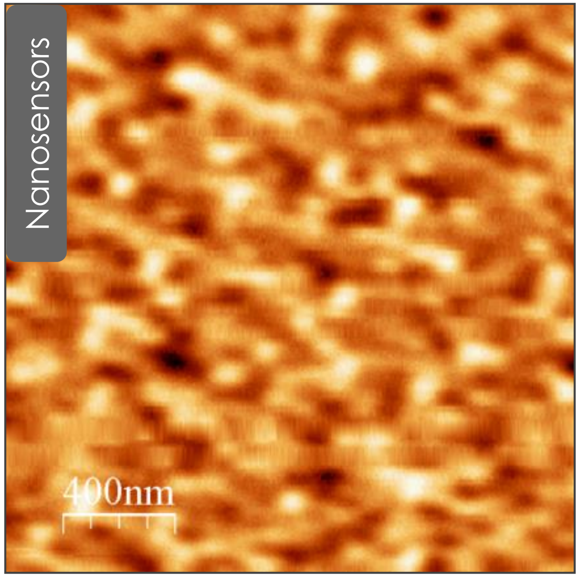


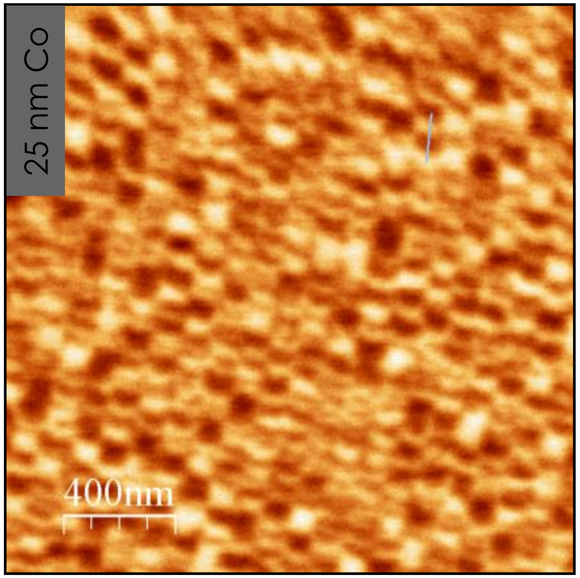
Standard MFM probe



One face-coated probe







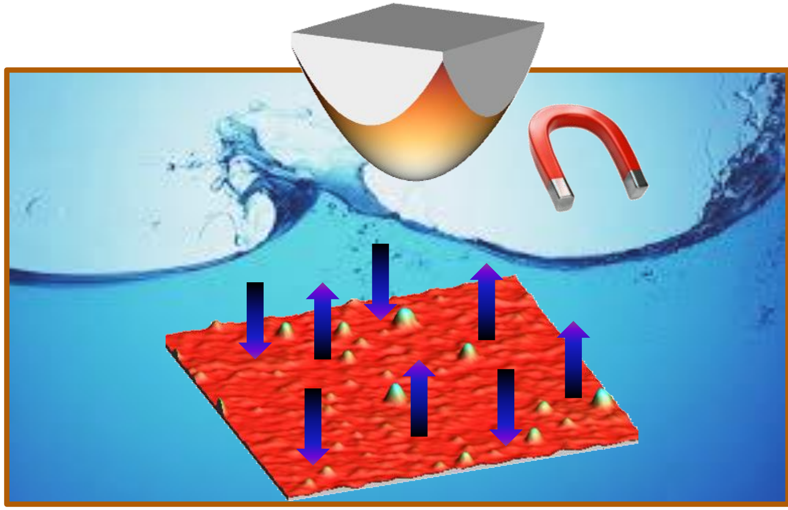
Ó. Iglesias-Freire, M. Jaafar, E. Berganza, A. Asenjo,*Beilstein J. Nanotechnol*. 2016, *7*, 1068–1074 (2016)

MFM and liquids

MFM technique can operate in different **environments**:

- AIR, the most useful and common
- HV, improvement signal/noise ratio
- UHV, useful for fundamentals studies more than for applications
- LIQUIDS, interest for biomedicine

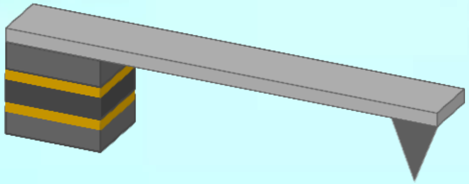
Can we use standard probes?

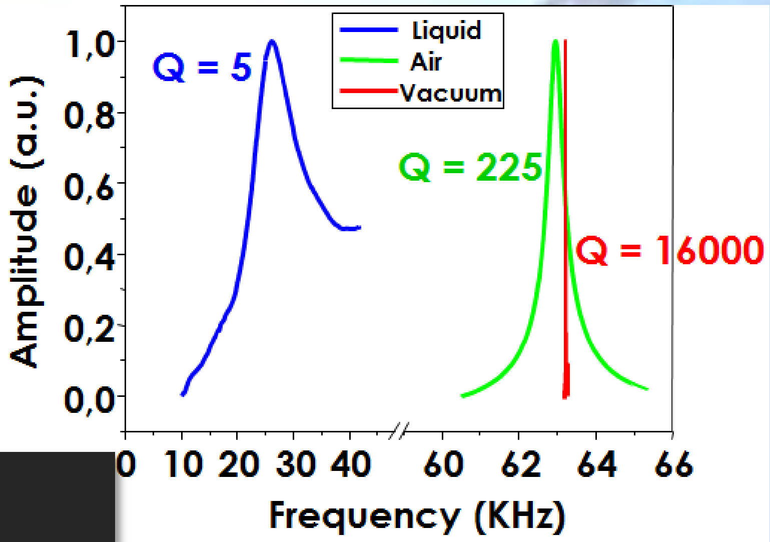


Problems of AFM/MFM in liquids

1.- Tip holder for dynamic mode in liquids. The mechanical excitation of the cantilever excites additional and spurious resonances (**forest peaks**).

2.- Cantilever dynamics under fluids. Special modes and **probes** to solve the problem of the very **low quality factor**.



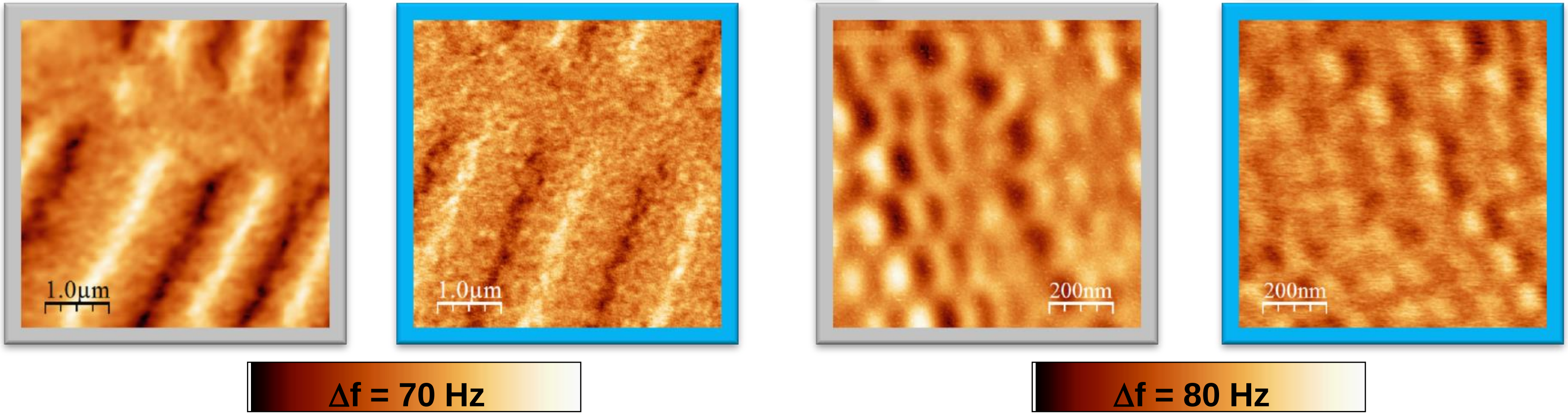


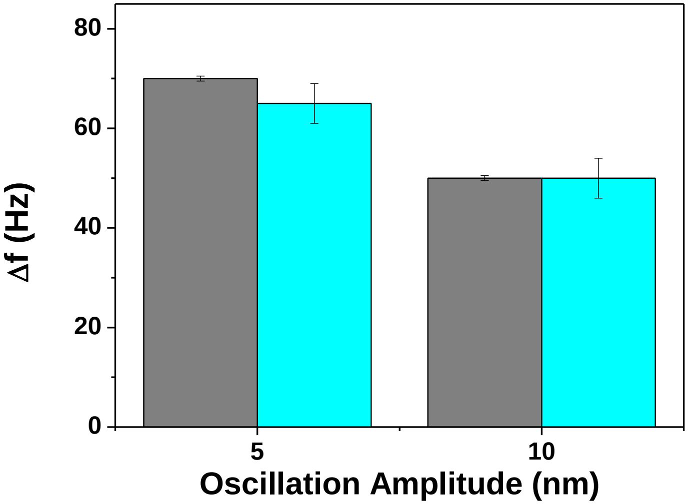
What are the problems?
How to measure in liquids?
What do we need to improve?

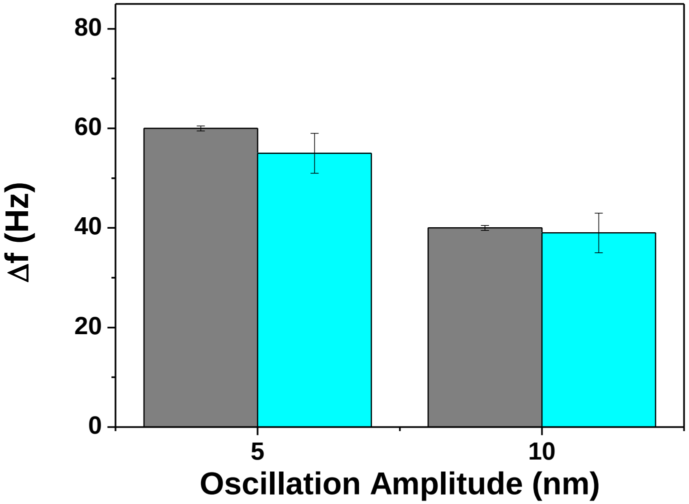
MFM and liquids. HD sample.DAM mode

M. Jaafar et al. Beilstein J. of Nano., 3 (2012)

◼ AIR ◼ LIQUID

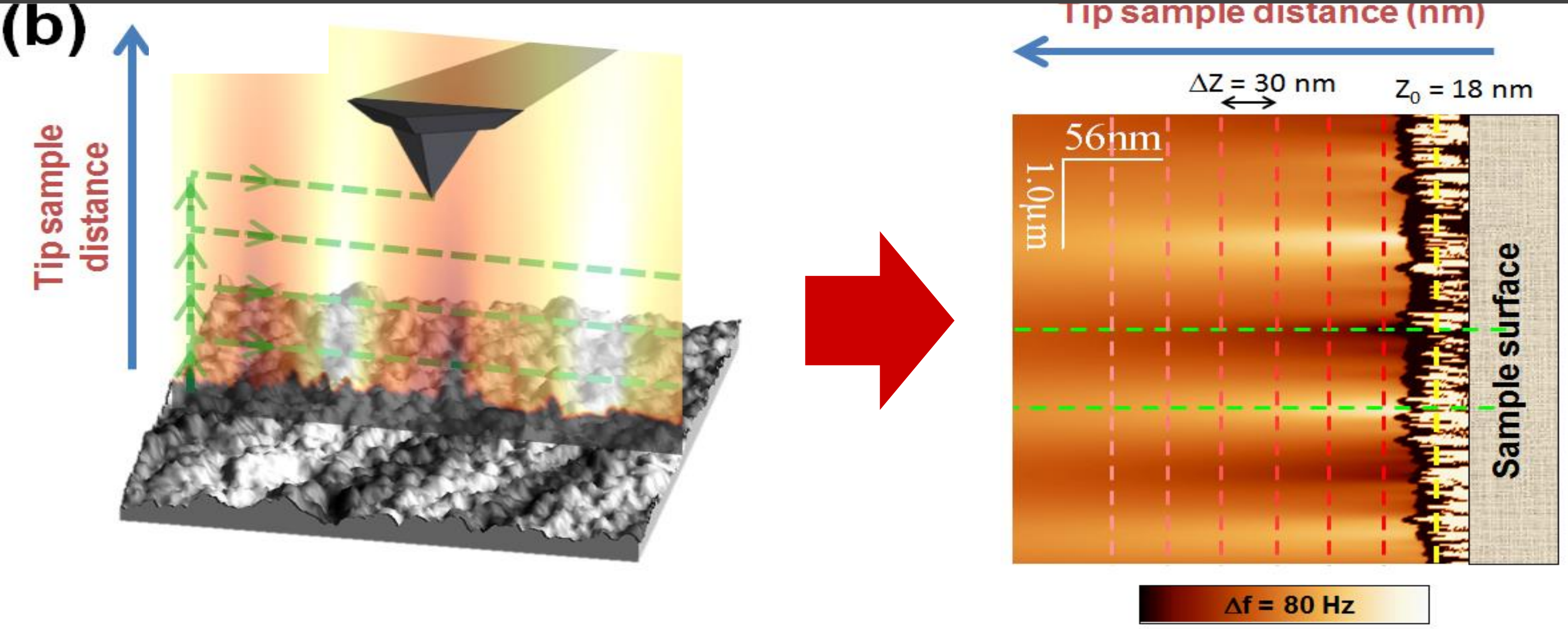


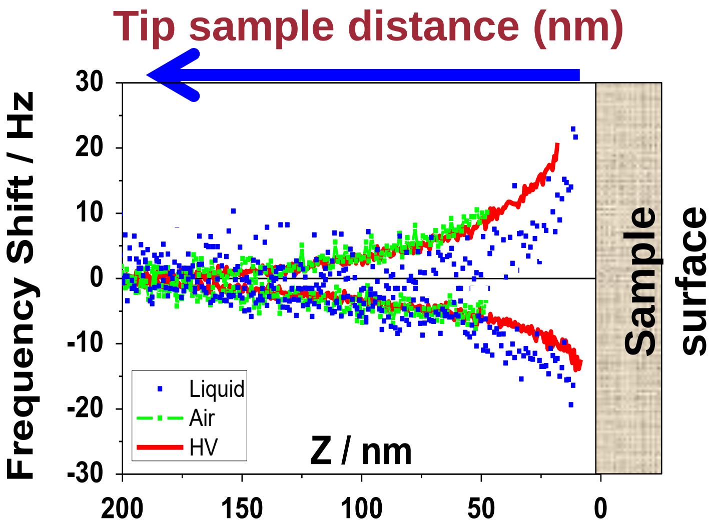




P. Ares, M. Jaafar, A. Gil, J. Gómez-Herrero and A. Asenjo, **Small** 11 (36) 4731-6 (2015)

MFM and liquids. HD sample.DAM mode



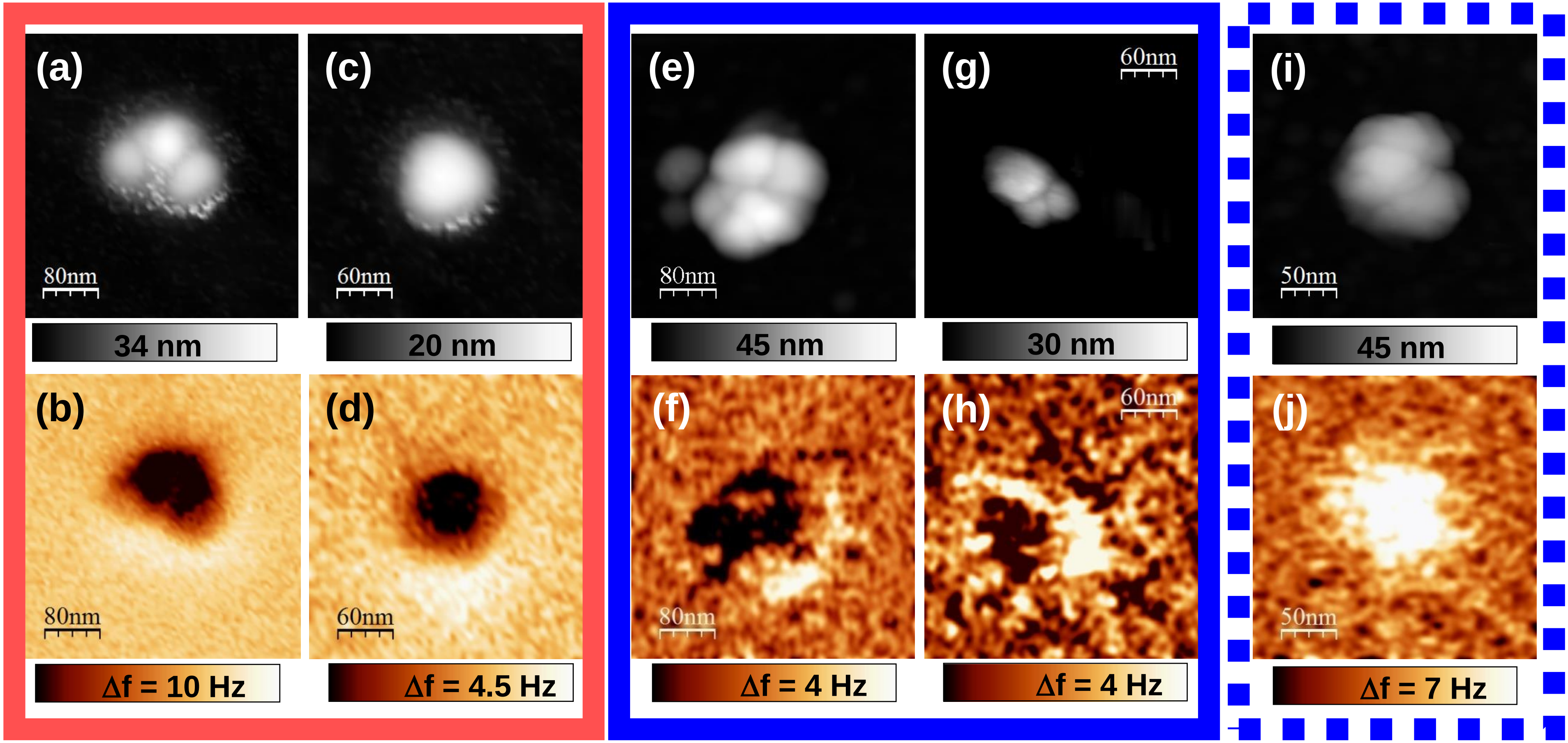


Optimizing the imaging parameters, it is possible to obtain similar contrast in ambient conditions and liquids

MFM in liquids. Fe_3O_4 nanoparticles

Cubic NPs, 30 nm side, courtesy of P. Morales, ICMM- CSIC

🟥 AIR 🟦 LIQUID



NON-MAGNETIC PROBE

P. Ares, M. Jaafar,A. Gil, J. Gómez-Herrero and A. Asenjo, Small, 2015

Further improvements. The MFM probes

To improve the sensitivity we need specific cantilevers, NOT OFFERED by the companies

$$\delta\omega = -\frac{1}{2}\frac{\omega_0}{k}\frac{\partial F}{\partial z}$$

R. García and R. Pérez, Surface Science Reports (2002)

Higher sensitivity
requires ω_0/k as high
as possible

The noise increases for softer cantilevers

$$\sqrt{{}^{1}/_{kQ}}$$

The adhesion of the magnetic coating to the AFM probe is and additional handicap

P. Ares *et al.*, Small (2015)

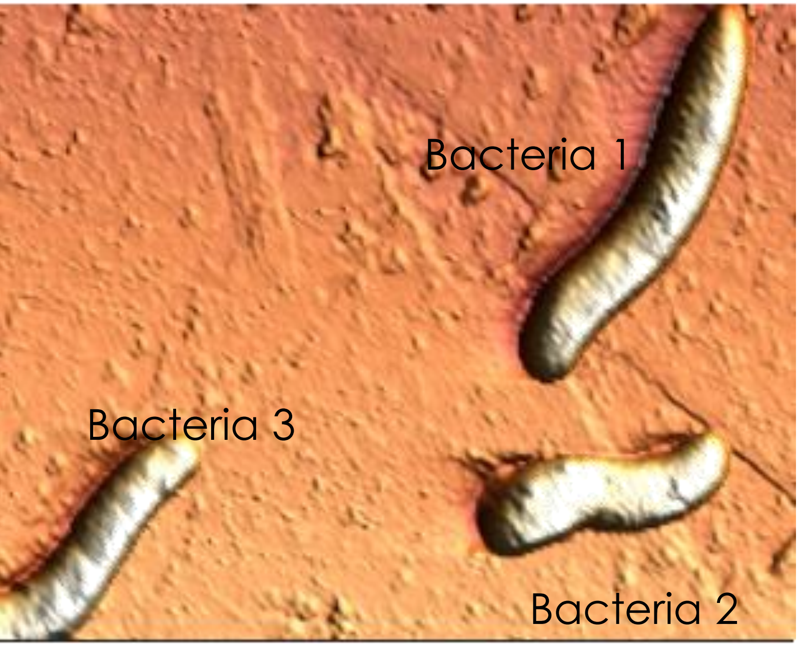
Individual magnetotactic bacterias

Magnetotactic bacteria biosynthesize magnetite nanoparticles of high structural and chemical purity. Nanoparticles size around few tens of nm.

Our goal: to obtain MFM signal in liquids from NP into the bacteria.

Bacterias deposited onto a mica substrate.

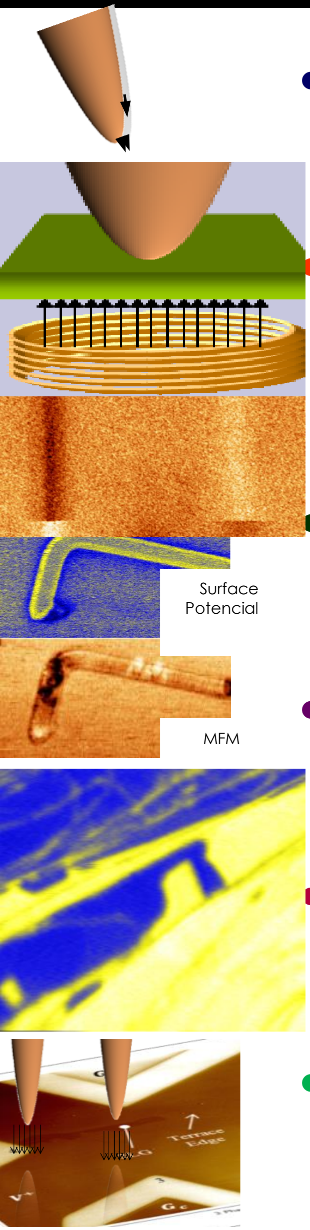
MFM probe: MFM budget Sensors



AFM image

Courtesy of M. L. Fdez-Gubieda’s group

Conclusions



- **Special tips** are presented which allow to improve lateral resolution and to study soft magnetic samples.
- The MFM is compatible con different environments: under external magnetic field, low temperature, high vacuum, UHV, liquid
- MFM is a well established technique for magnetic characterization at the nanoscale.
- **KPFM/MFM** combination mode is useful to separate electrostatic and magnetic contrasts.
- Handicaps, the **interpretation**, cross-talk between different interactions.
- MFM technique is under continuous **development**, new applications and operation modes appear.

In collaboration with....

TEM and Holography

L.A. Rodríguez-Gonz.
E. Snoeck

CEMES

Dissipation

P.G. Mochales
J.J Sáenz

UAM UNIVERSIDAD AUTONOMA DE MADRID

Dissipation in MFM

J. Bates
Y. Miyahara
P. Grütter

McGill

KPFM-MFM in Co nanostripes

L. E. Serrano
J. M. de Teresa
R. M. Ibarra

INA, ICMA-CSIC, Uni. Zaragoza

MFM Laboratory

Miriam Jaafar
Óscar Iglesias-Freire
Eider Berganza

Nanomagnetism and Magnetic Materials Group

Manuel Vázquez
Rafael P. del Real
Cristina Bran

icmm

Oksana Fesenko

Puerto Morales

Technical support

NANOTEC ELECTRONICA

KPFM-MFM in Graphite

P. Ares
D. Martín Martínez
J. Gómez-Herrero

R. Pérez

UAM UNIVERSIDAD AUTONOMA DE MADRID

Fe_3O_4 nanoparticles

C. Moya
X. Batlle
A. Labarta

UNIVERSITAT DE BARCELONA

IN2UB INSTITUT DE NANOCIÈNCIA I NANOTECNOLOGIA

Magnetotactic bacteria

M. L. Fdez-Gubieda
A. Muela

UPV EHU