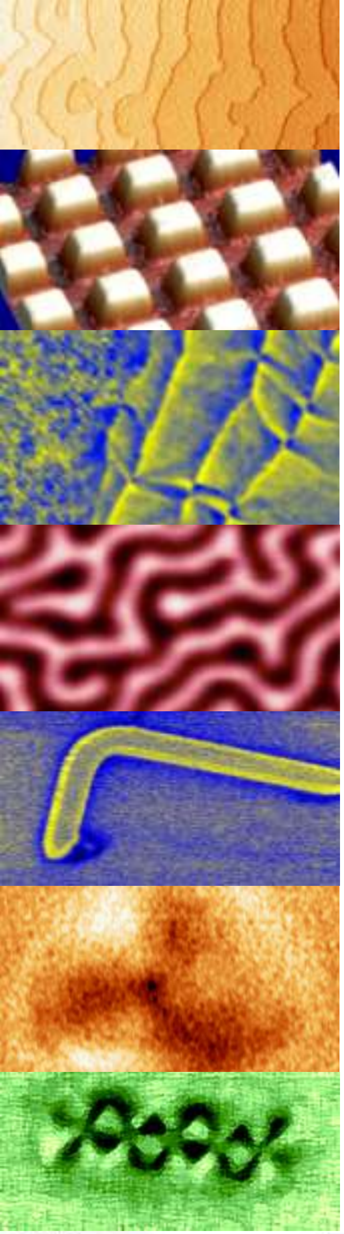


# Novel Frontiers in Magnetism

## SPM-Magnetic Force Microscopy


Agustina Asenjo

*ICMM-CSIC*

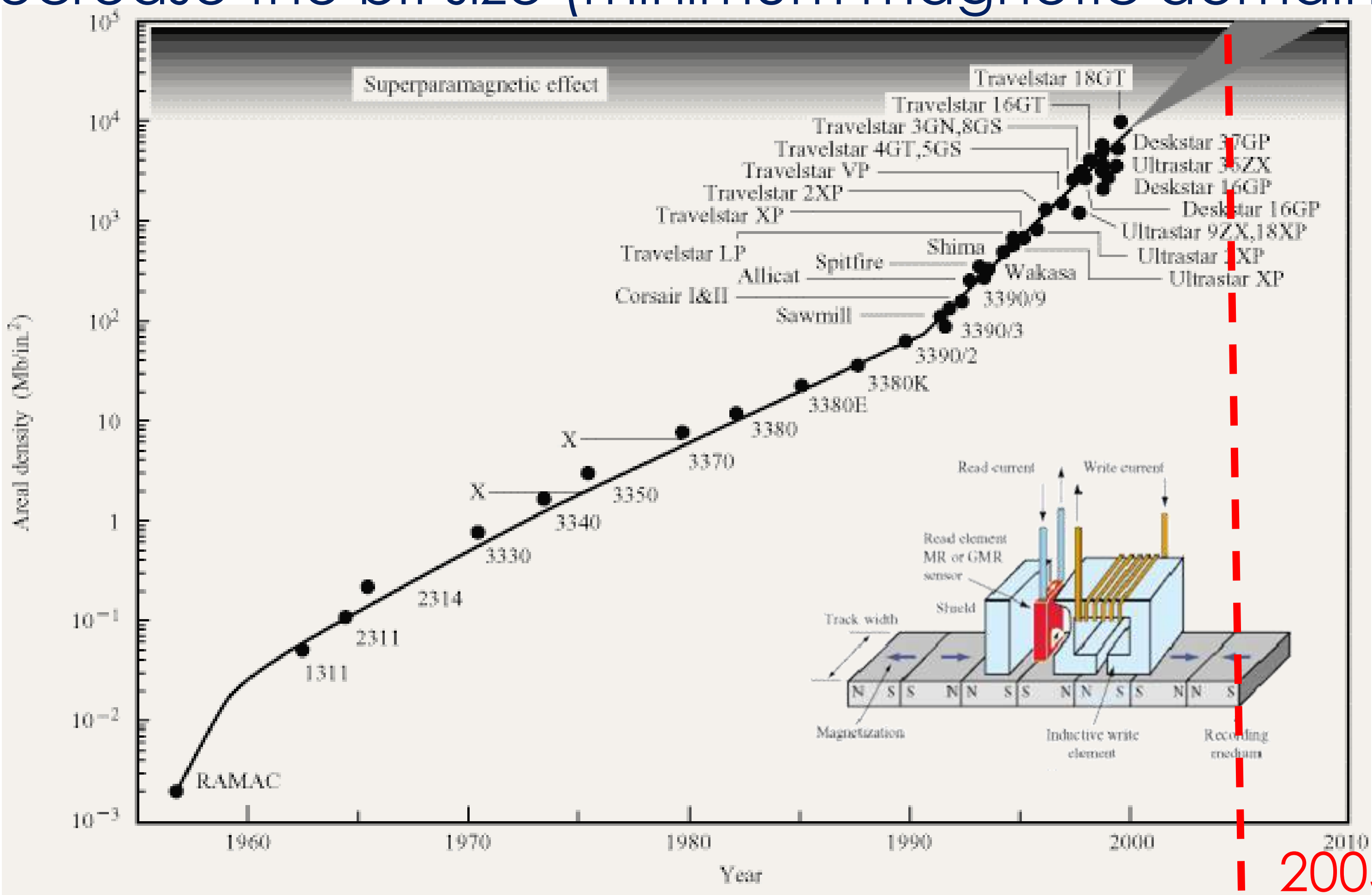
Nanomagnetism and Magnetic Materials Group

*MFM Laboratory*

# Longitudinal magnetic recording media

Increasing the storage density….

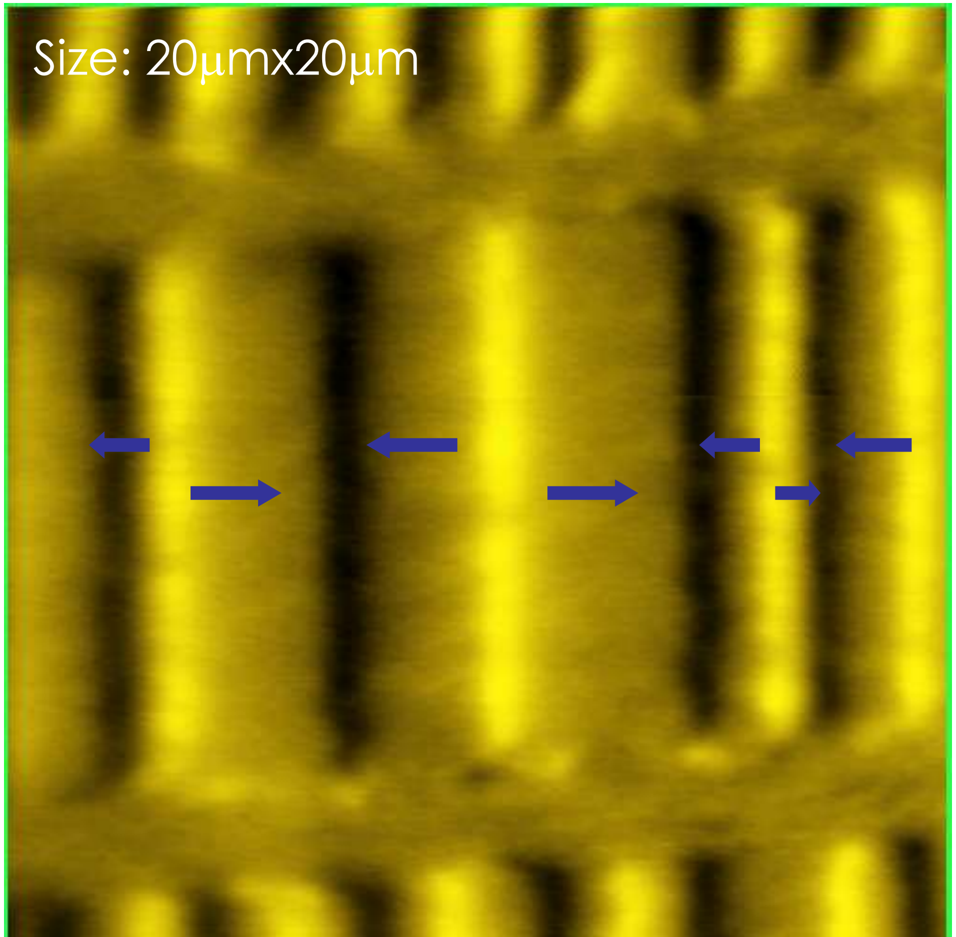
To decrease the bit size (minimum magnetic domain)



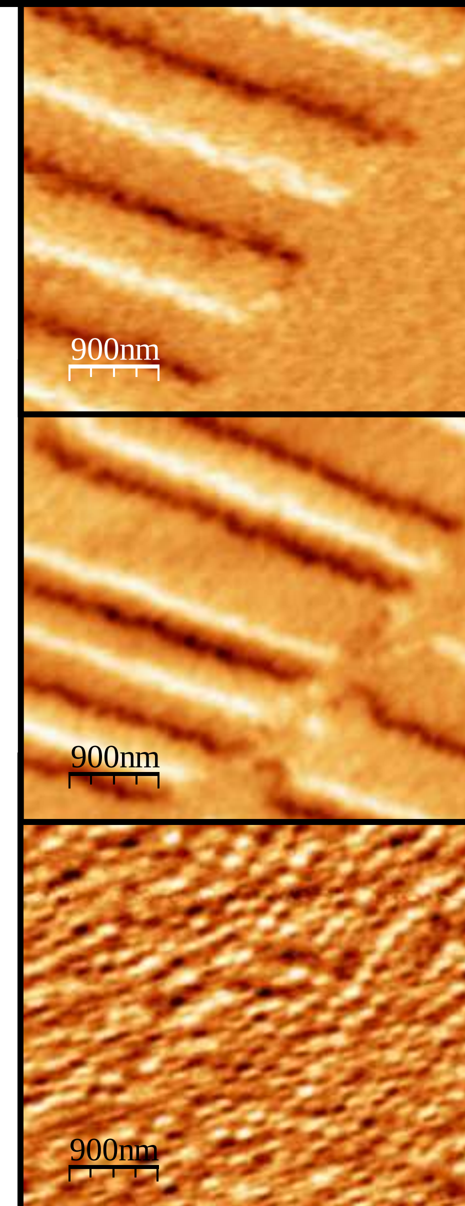

2005 Toshiba
179 Gbit/in²

# Longitudinal magnetic recording media

## Domain in longitudinal hard disk

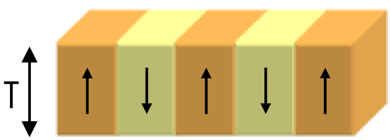



The magnetization direction in every domain is marked by the arrows



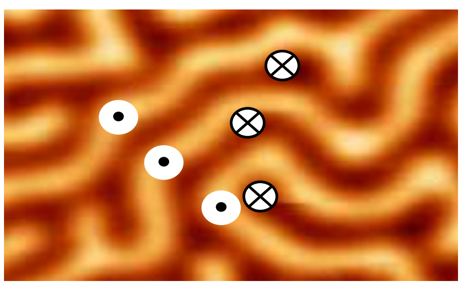
# Domains in ferromagnetic materials

Thin film of perpendicular magnetic material → Magnetization in remanence T< Tc




*Domain size*

$D= (\gamma T/1.6 * M_s^2)^{1/2}$



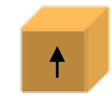
Decreasing the size →

Single domain

$L_D \propto K^{1/2}$

if smaller.... →

Superparamagnetic limit



$\tau=\tau_0 \, e^{KV/k_B T}$

Demagnetized due to the thermal energy.
Critical size depends on the magnetic anisotropy

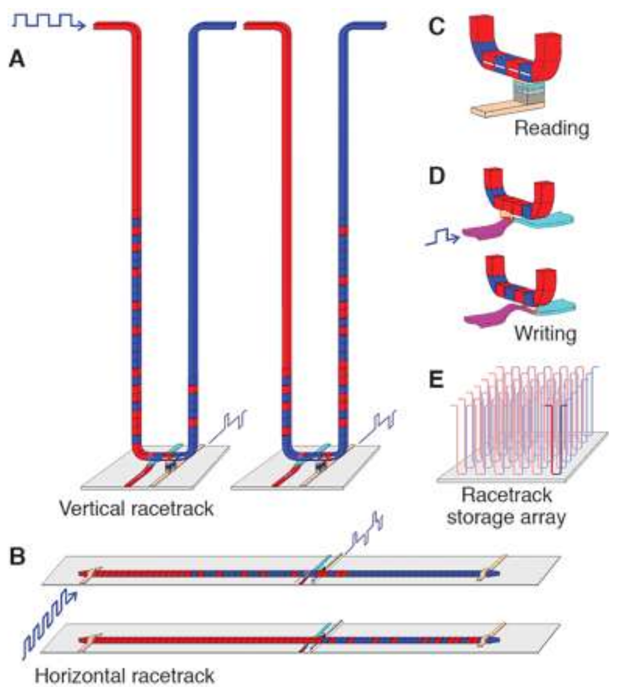
To increase the **density** it is necessary to increase the **anisotropy**.

The development of **high resolution** techniques is **mandatory**.

# Longitudinal magnetic recording media

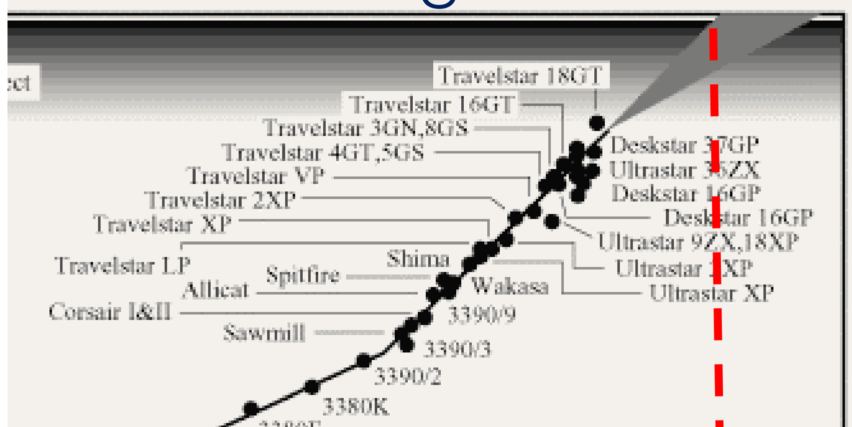
[Magnetic Domain-Wall Racetrack **Memory**]
**S. S. P. Parkin et al.** Science (2008) 320

Pulses of highly spin-polarized current move the entire pattern of DWs coherently along the length of the wire to read and write elements

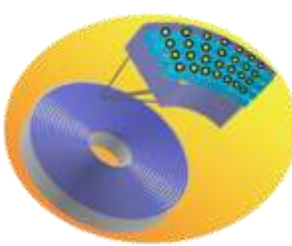


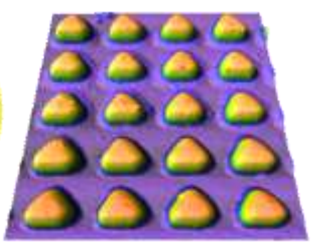

ensity….

(minimum magnetic domain)




**New hard disks:** materials with perpendicular anisotropy, nanowires, nanostructures ……





2005 Toshiba
179 Gbit/in²

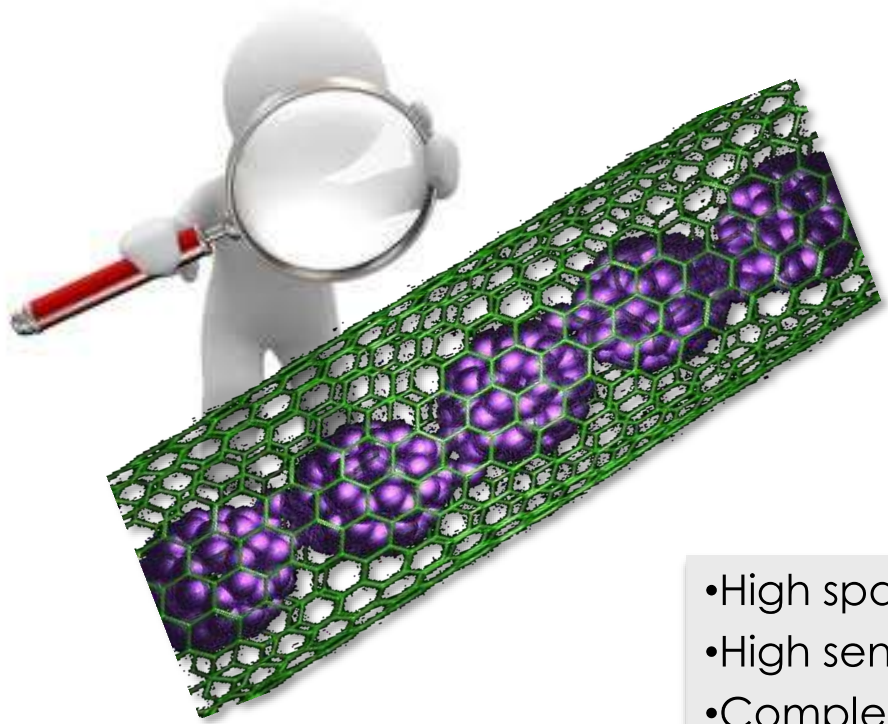
# Magnetic Imaging

| Technique | Signal | Sample preparation | Spatial resolution | Contrast | Materials | External field | Drawback |
|---|---|---|---|---|---|---|---|
| **Magneto-Optical** | Light polarization | Flat and Smooth surfaces | 200nm | Walls and domains | $K_{per}$ and $K_{in\text{-}plane}$ | Available | Resolution |
| **Bitter** | Magnetic colloids distribution | None | 100nm | Walls | High stray fields | Available (low speed) | Dirty Sample |
| **SEMPA** | Polarized S.E | HV cleaning | 50nm | Domains | $K_{per}$ and $K_{in\text{-}plane}$ | Difficult | Cleanness |
| **MFM** | Charge density | None | 20nm | Walls and domains | $K_{per}$ and $K_{in\text{-}plane}$ | Available | Tip-sample interaction |
| **Lorentz ME** | $F_{lorentz}$ | UHV cleaning | ~nm | Domains | $K_{in\text{-}plane}$ | Difficult | Sample preparation |
| **X-Ray Microscopy** | Dicroism | Synchrotron | 15 nm | Domains | $K_{per}$ and $K_{in\text{-}plane}$ | Available | Special Facility |

# Importance of the characterization.......

Significant advances in Science require the development of new theories, new fabrication techniques, but also accurate characterization methods

It is important to use the appropriate methods!



- High spatial resolution
- High sensitivity
- Complementary information

# Outline:

1. Scanning Probe Microscopies.
2. Fundaments of MFM. Measuring under standard conditions
3. Improving the hardware:
   a. Variable Field MFM
   b. HV-Low Temperature MFM
   c. MFM probes
4. Developing new operation modes:
   a. 3D modes
   b. KPFM-MFM combination: Co nanostripes, Graphite, Graphene based hall sensor
   c. Dissipation
5. Conclusions

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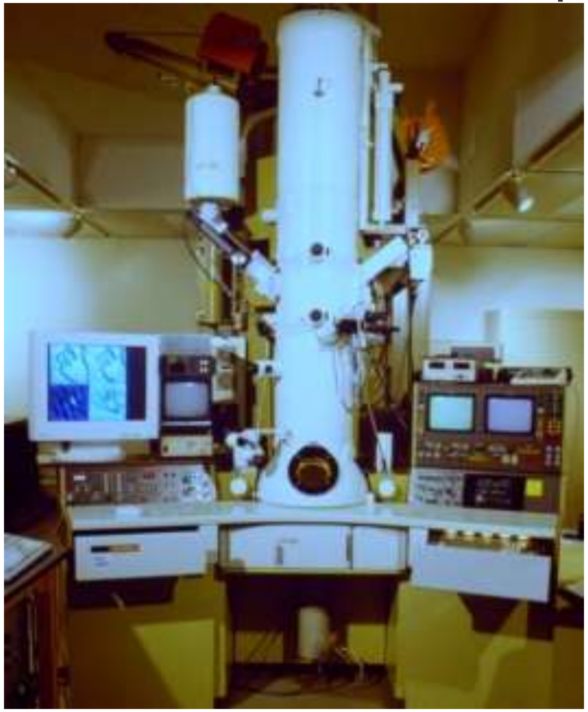
   c. Dissipation
5. Conclusions

# Comparing with other microscopes….

Optical microscope

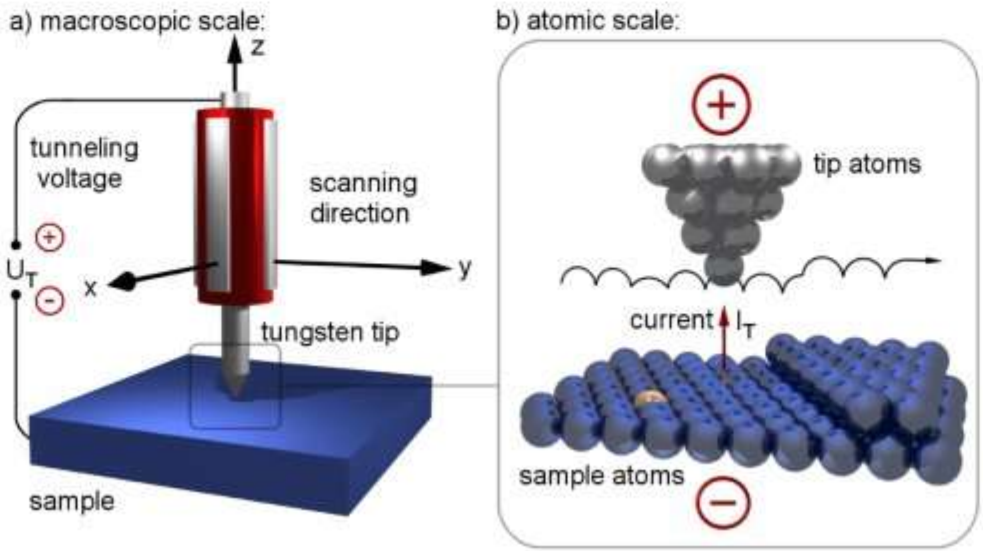
SPM

Electron microscope



| Microscopy | Researcher | Date | Probe | Range |
|---|---|---|---|---|
| **Optical microscopy:** | Galileo | 1611 | light | 1mm-200nm |
| **Electron microscopy** | E. Runska | ~1930 | electrons | 1mm-1nm |
| **Scanning Probe Microscopy** | G.Binnig, H.Rohrer | 1981 | Sharp tip | 100µm- 0.1nm |

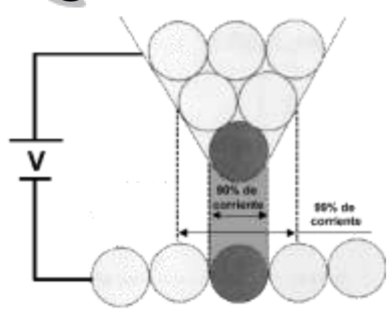
# Scanning Tunneling Microscopy





## STM advantages

$$I = f(V) \cdot e^{-\sqrt{\varphi} \cdot s}$$

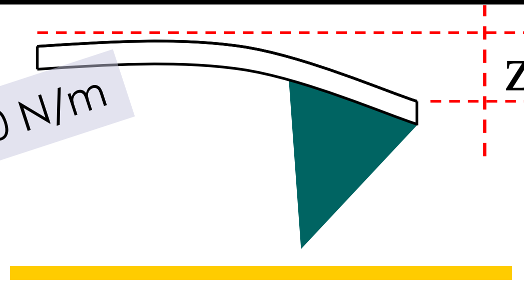



High vertical resolution
High lateral resolution
Spectroscopy

G. Binnig y H. Rohrer (1981)
(Nobel Prize in 1986)

# The cantilever and the Hook law

$$F= -k\ z$$

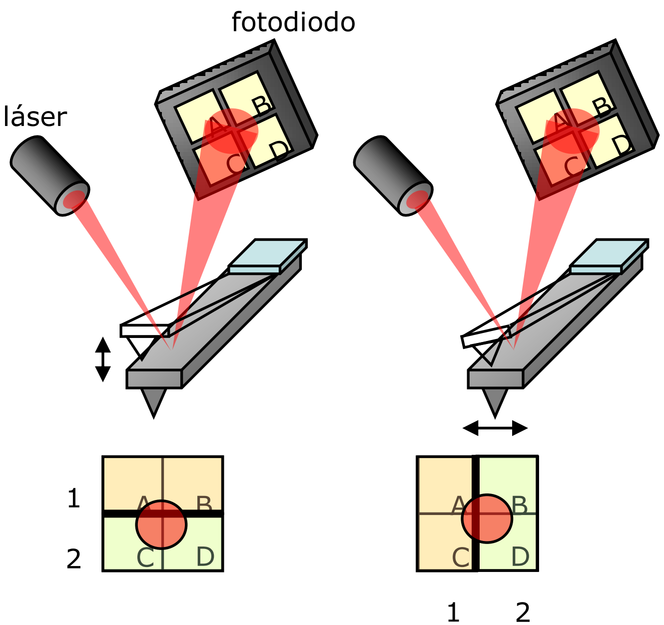



k ranges between 0.01 and 100 N/m

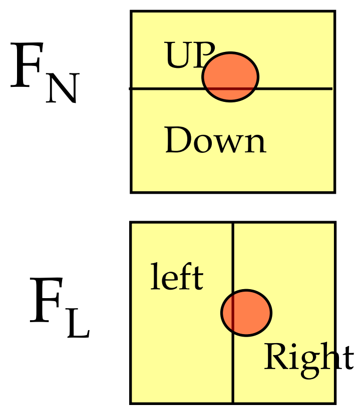
$k= E/4 \cdot W \cdot (T/L)^3$

k dependes on the geometry and composition

## Cantilever deflection. Detection system ⟶ Laser beam reflexion



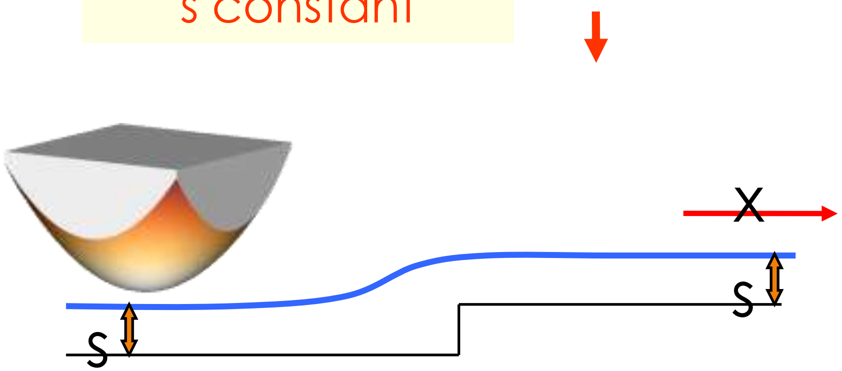

$F_N$ o $F_L$ detection

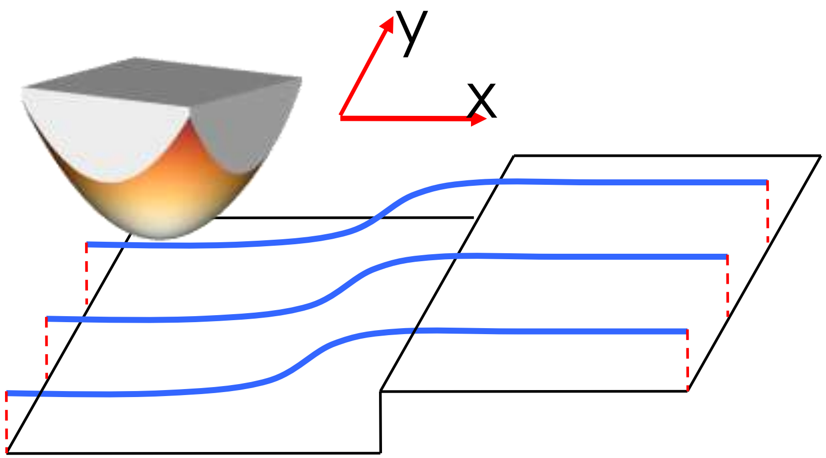


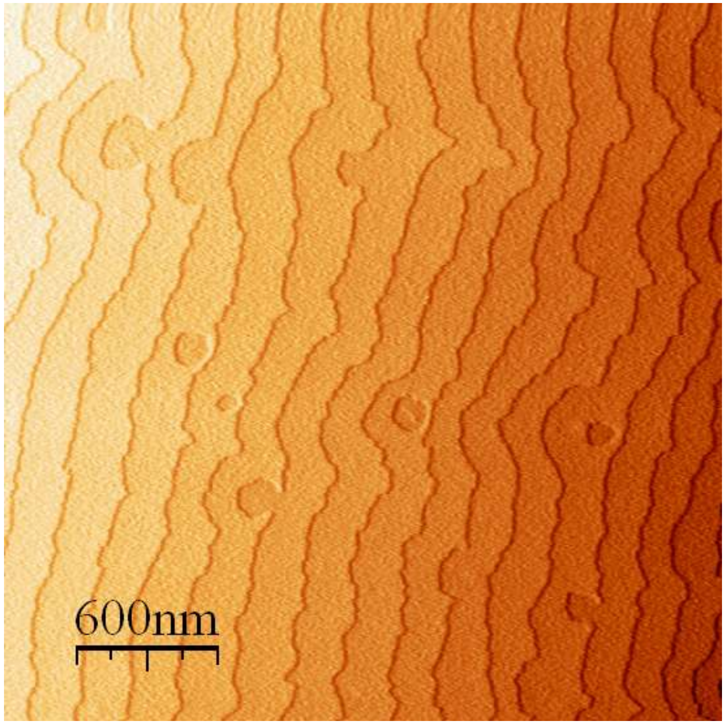
# Contact mode. Building maps...

When the tip is close to the sample, it start to scan the surface

Constant current
s constant



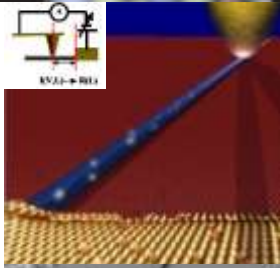
# Applications: contact/non contact

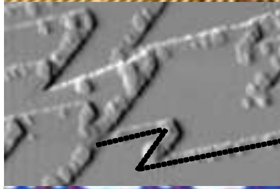
## Contact mode

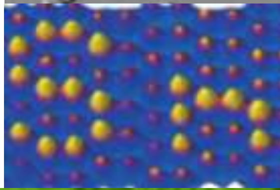
- Topography. Thin films, nanostructures,…
- Conductivity
- Friction
- Chemical process and growth in real time
- Mechanical properties (friction, adhesion, elastic and plastic properties).

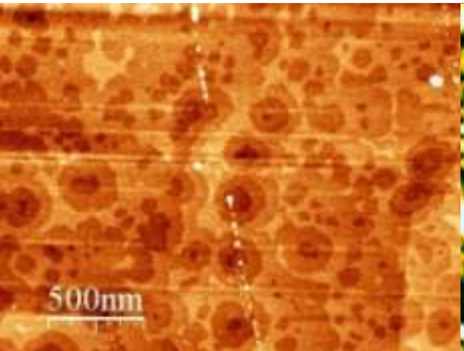
## Non-Contact mode

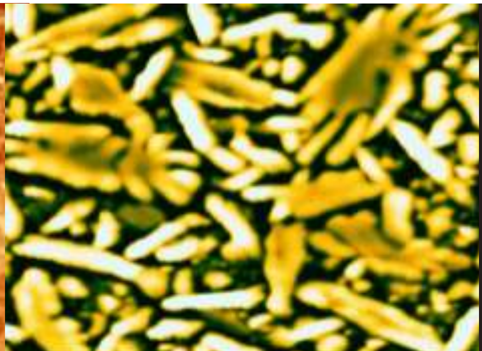
- Magnetic properties, electrostatic forces
- Force spectroscopy

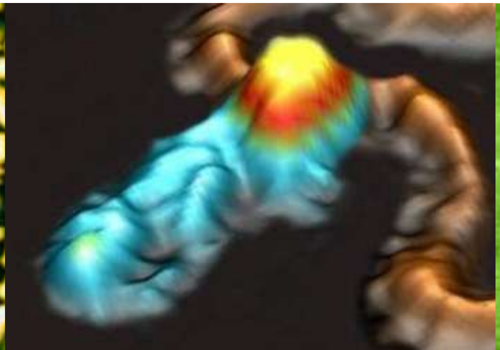



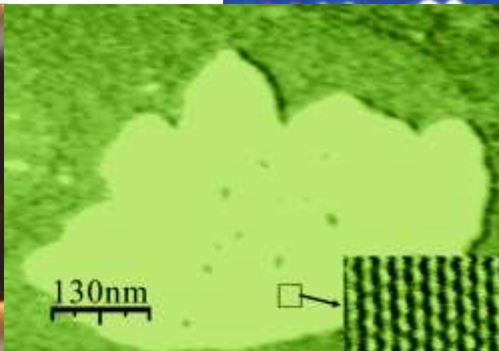










# Applications: contact/non contact

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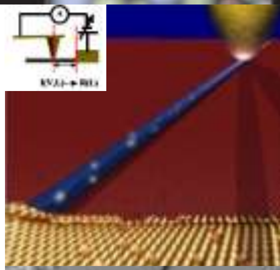
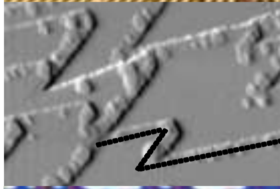
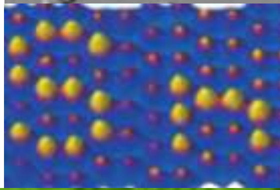
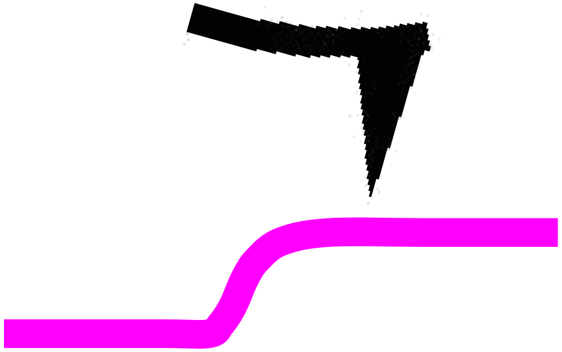
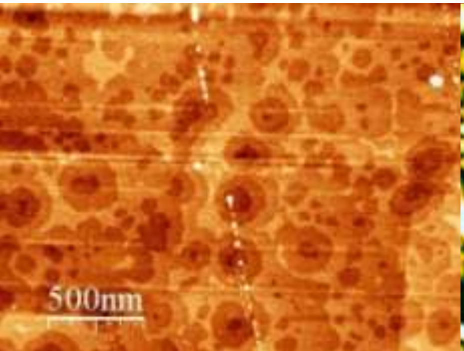
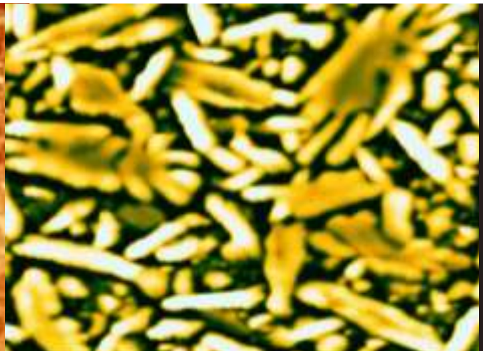


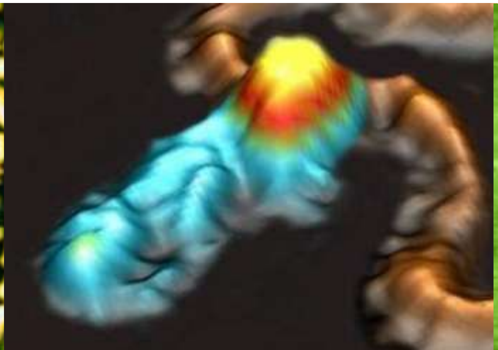


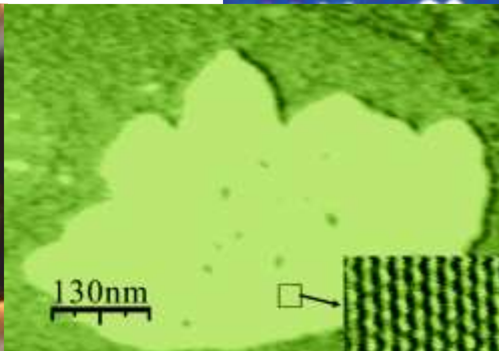




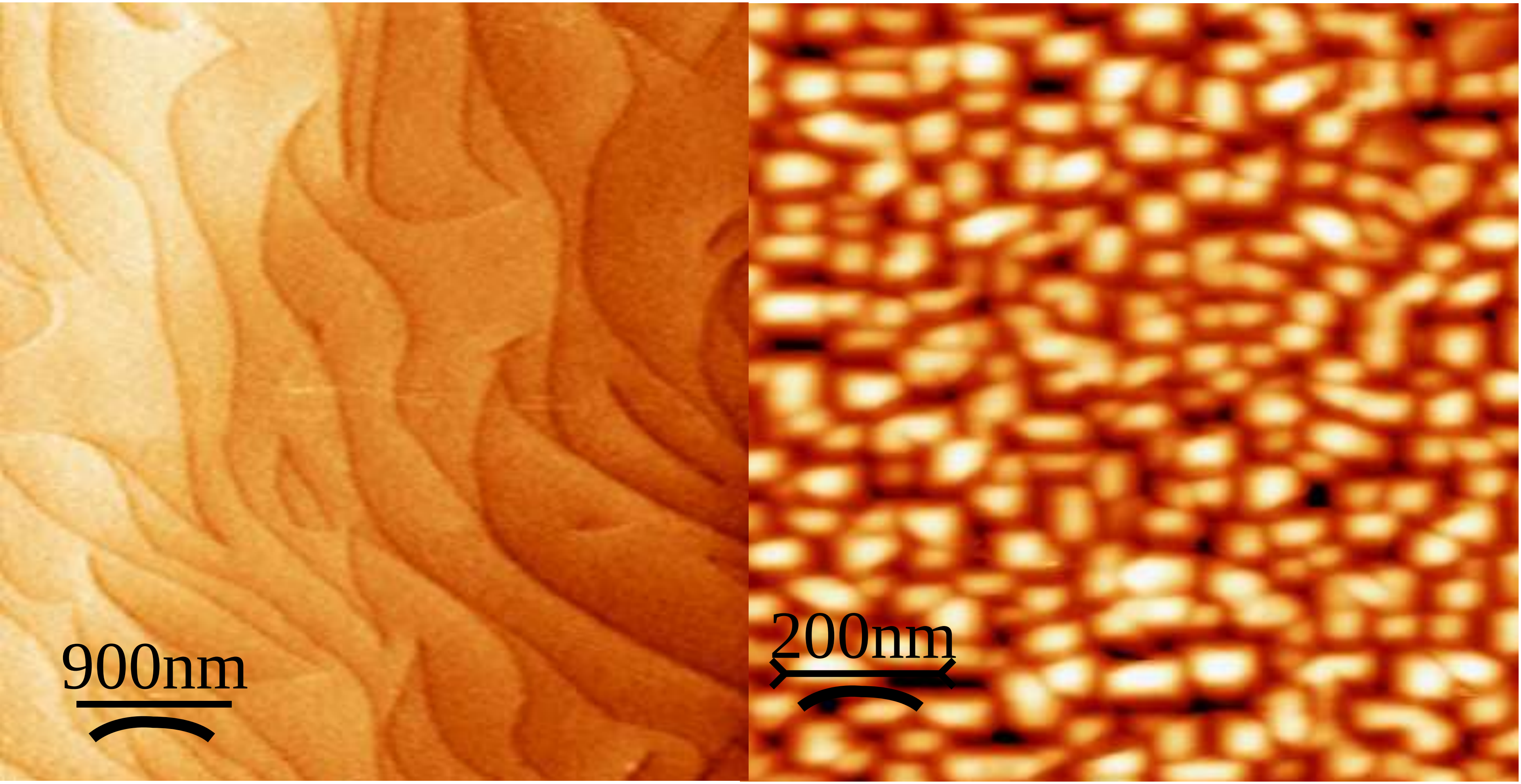


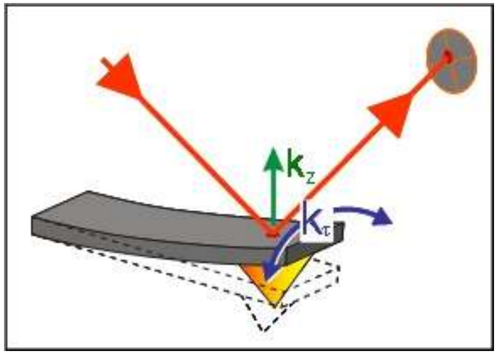




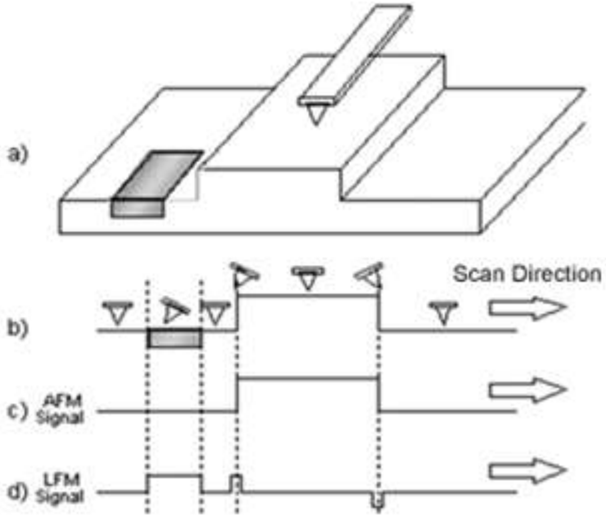
# Surface Morphology and Nanostructures



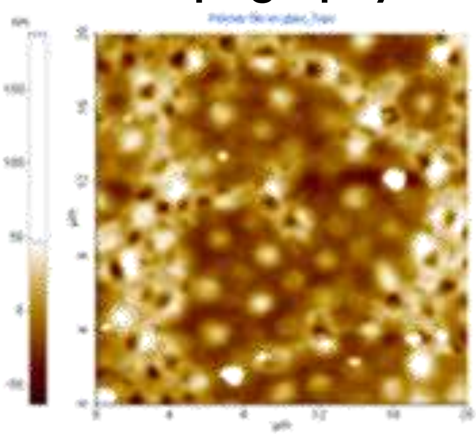
# Applications: Friction



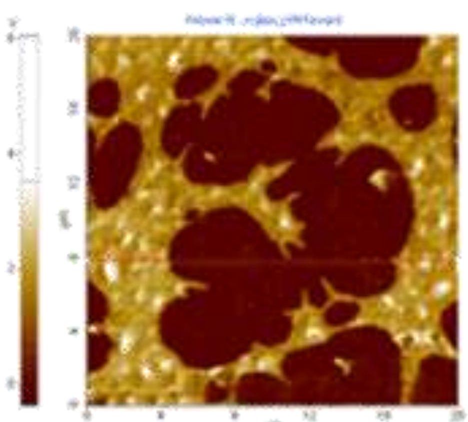

**Higher constant force**



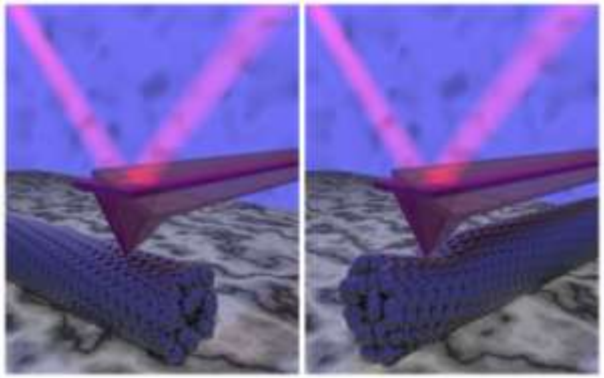

**Topography**



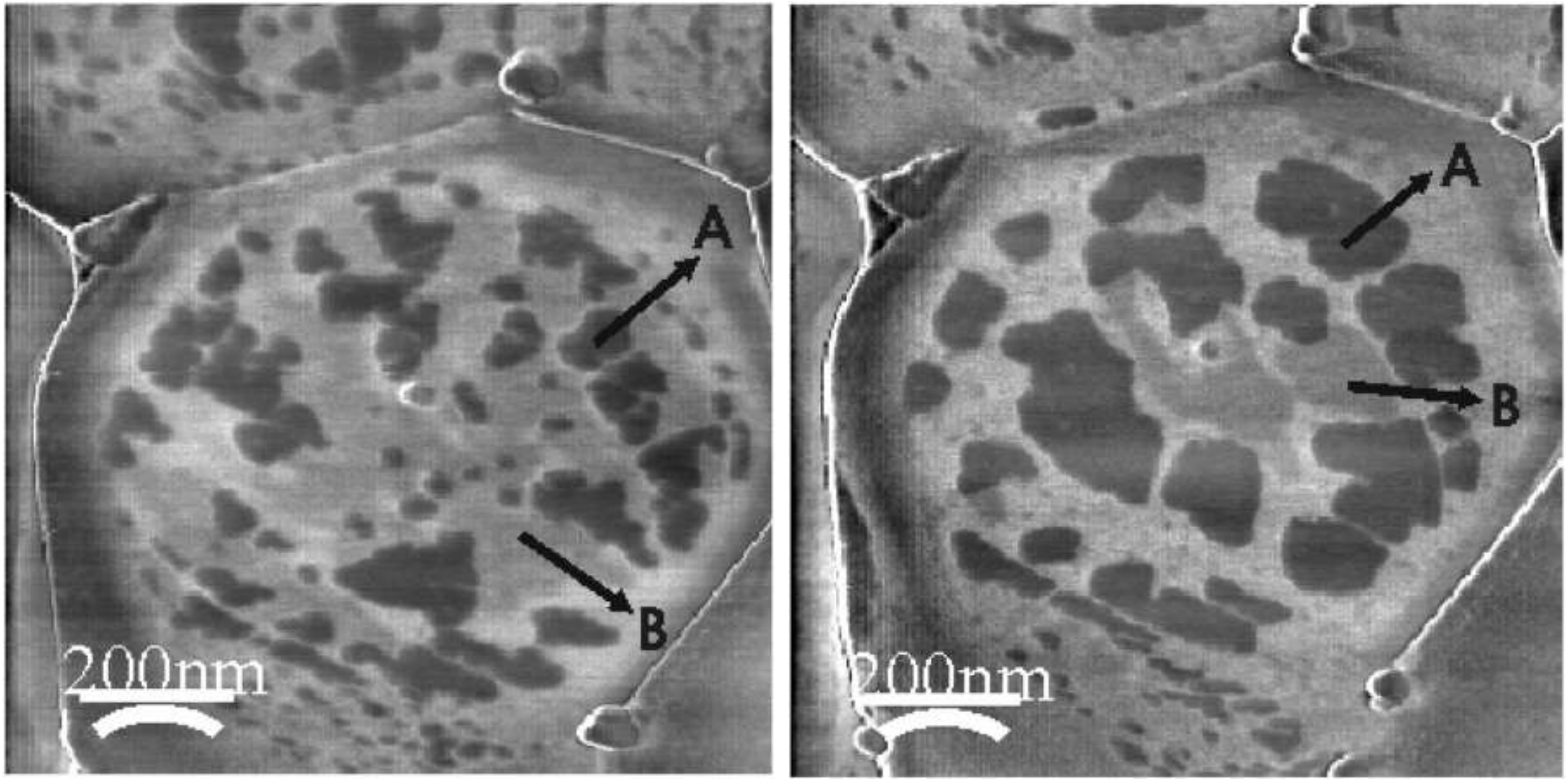
**Lateral force**



**Anisotropy in nanowires**



# Applications: Friction in thiol islands



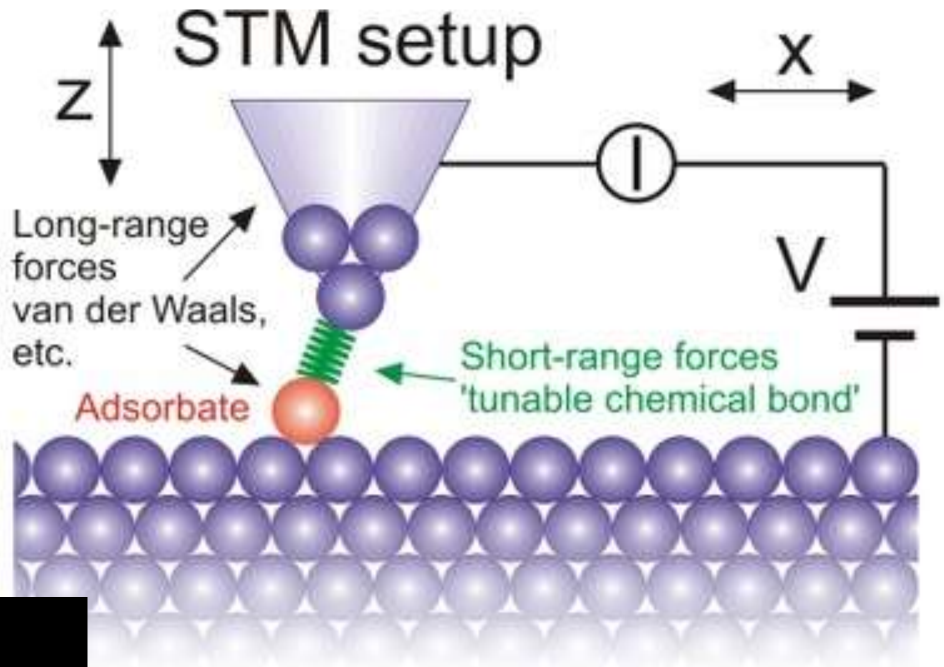

Lateral force images of a typical freshly prepared C12 sample (left) and same area 244 h after preparation (right). The area covered by the upright configurations increases by about 12% from top to bottom images.

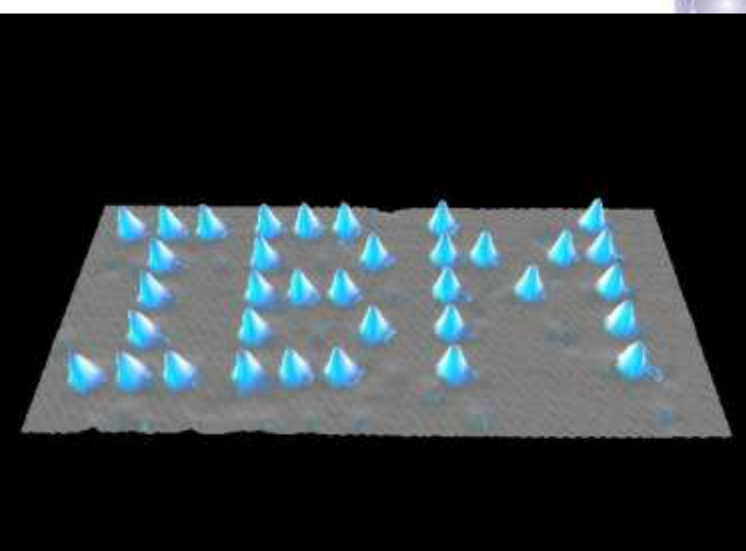
Color code: **dark is lower lateral force, and bright is higher lateral force.**

It is possible to distinguish two different thiol packing

C. Munuera, E. Barrena, and C. Ocal *Langmuir, Vol. 21, No. 18, 2005*

# Applications: Manipulación individual de átomos y moléculas



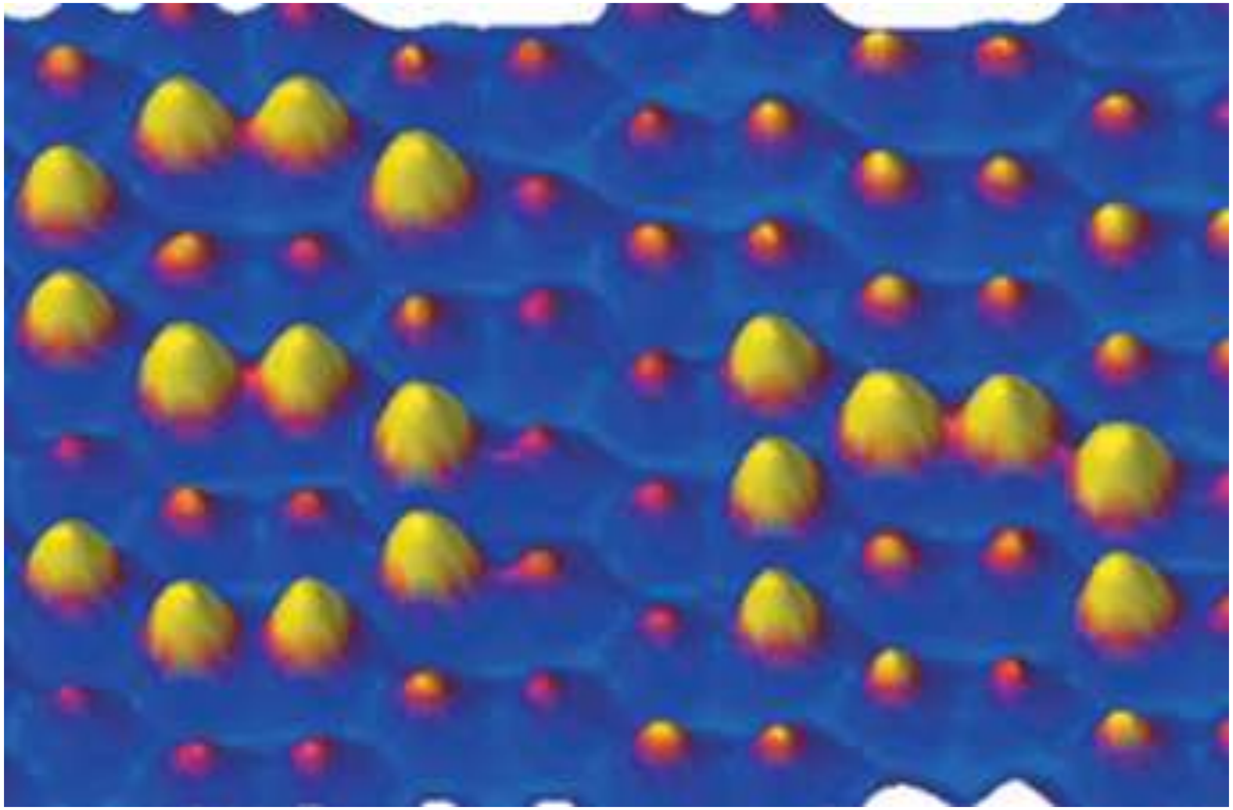



1989. Don Eigler from IBM was the first to manipulate atoms using the STM's tip.

Xenon atoms onto Ni surface at 8K.

D.M. Eigler, E.K. Schweizer, Nature 344, 524-526 (1990).

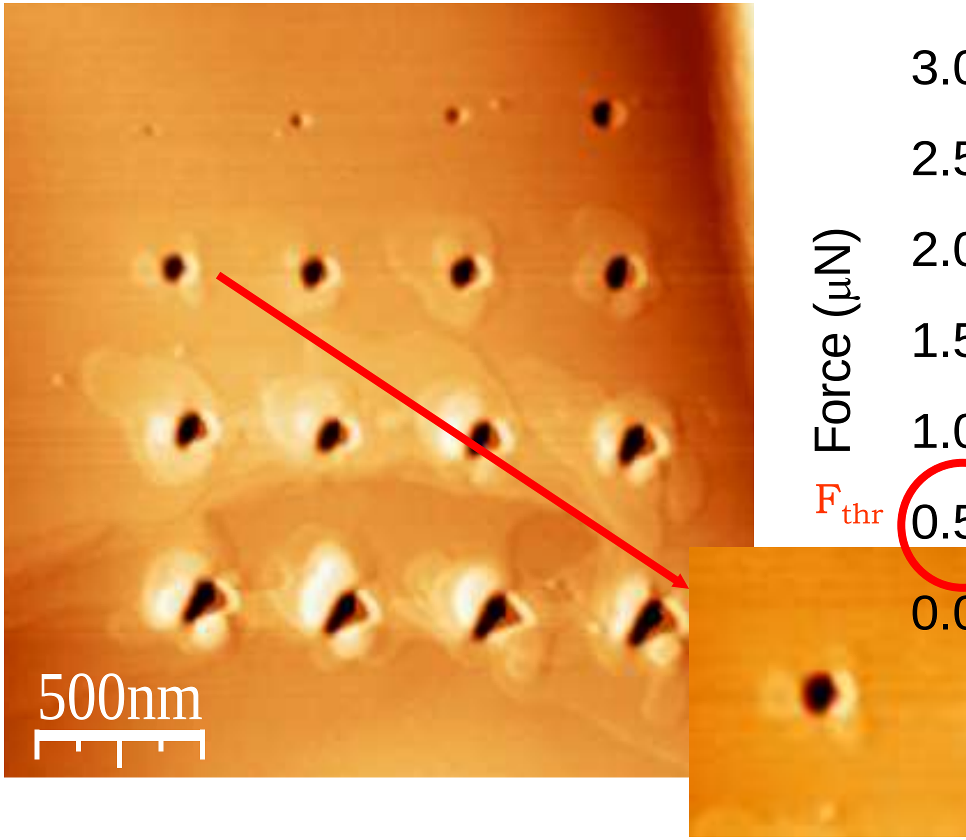
# Applications: Manipulación individual de átomos y moléculas

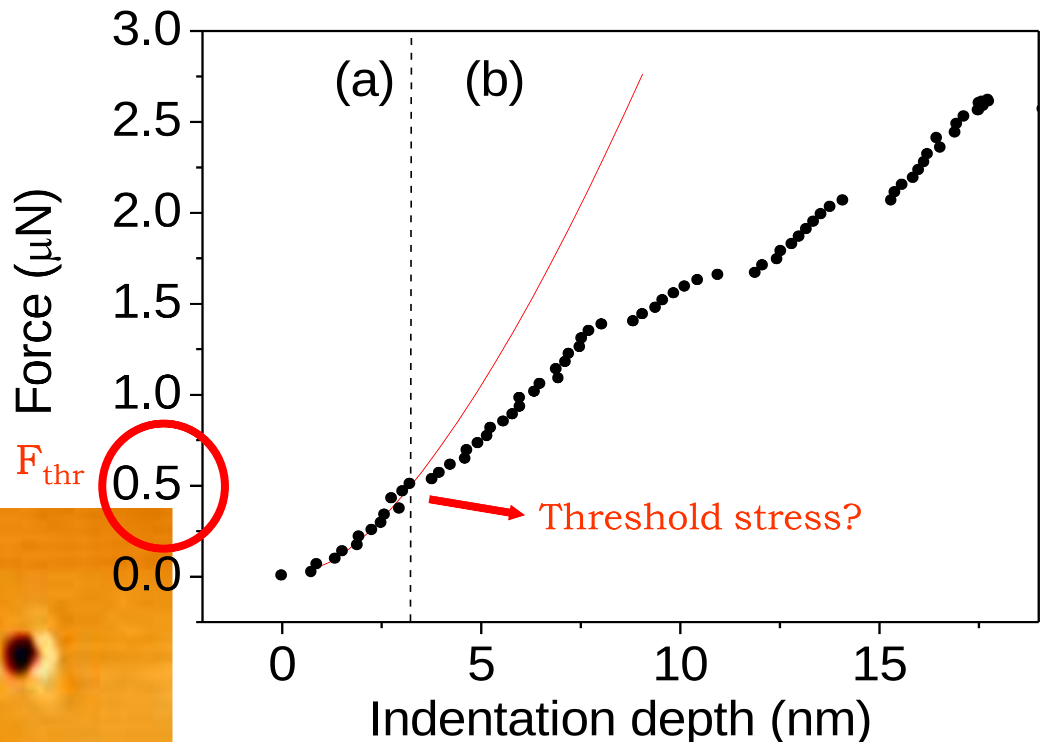


Tin symbol "written" with Sn atoms onto Ge surface by using the AFM tip. First example of manipulation at room temperature.

O. Custance, R. Perez & S. Morita, Nat. Nanotech. 4, 803 - 810 (2009)

# Applications: Initial stages of plasticity in Au(111)




**Region (a): Elastic part.**
**Hertz model**

$$F = \frac{4}{3} E^{*} \sqrt{R} \delta^{3/2}$$

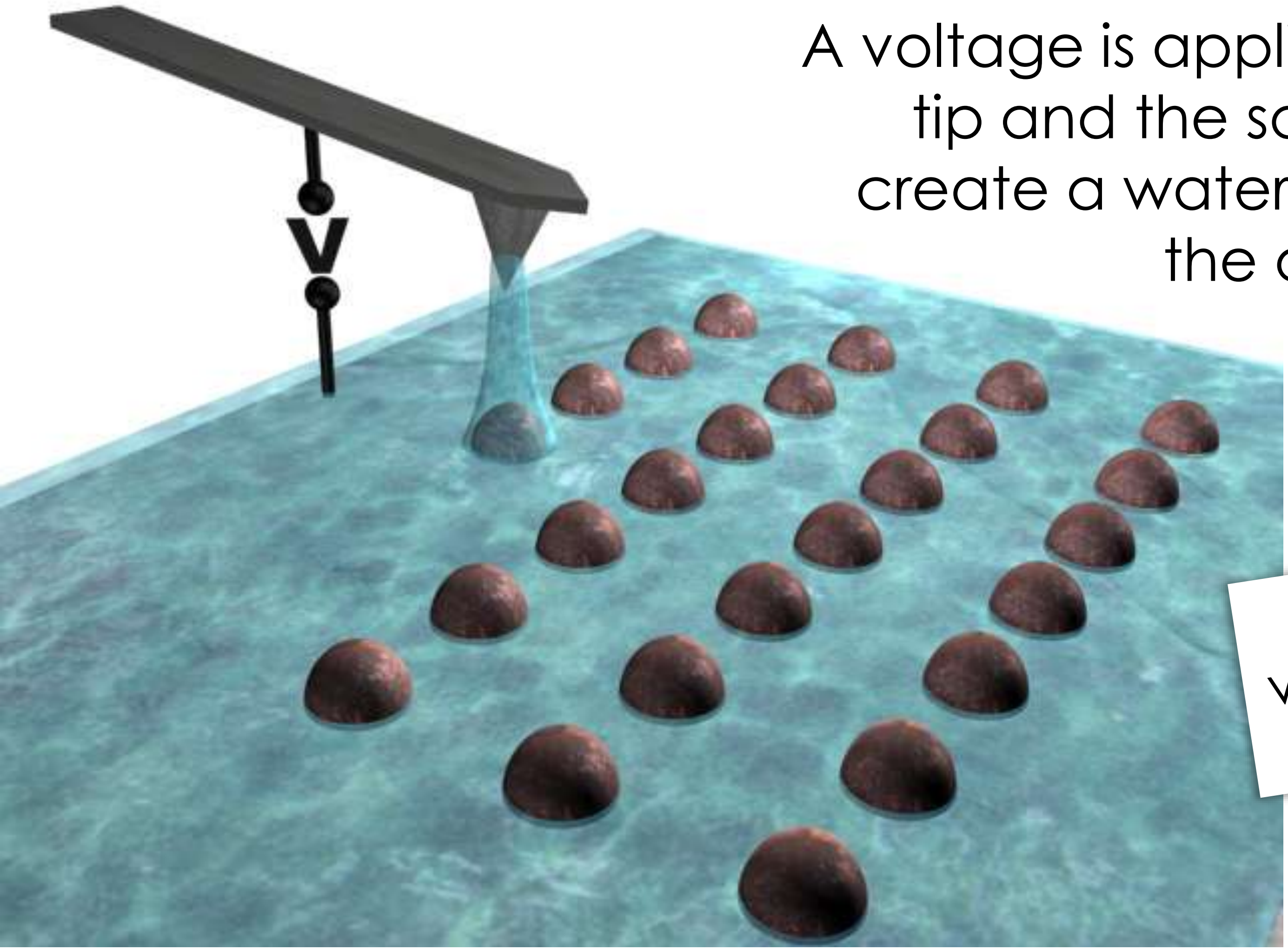
**Region (b): Plastic deformation**

Permanent traces
Discontinuities
**Dislocations**
Cross-slip
Terraces

A. Asenjo et al. Phys. Rev. B, 73, 075431 (2006)

# Applications: Nanolithography

## Local oxidation by using the AFM tip




A voltage is applied between the tip and the sample in order to create a water meniscus where the oxidation occurs.

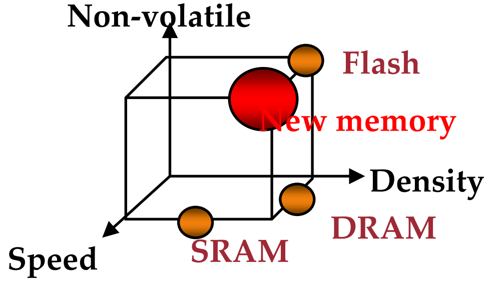
The humidity varies between 30% and 60%.

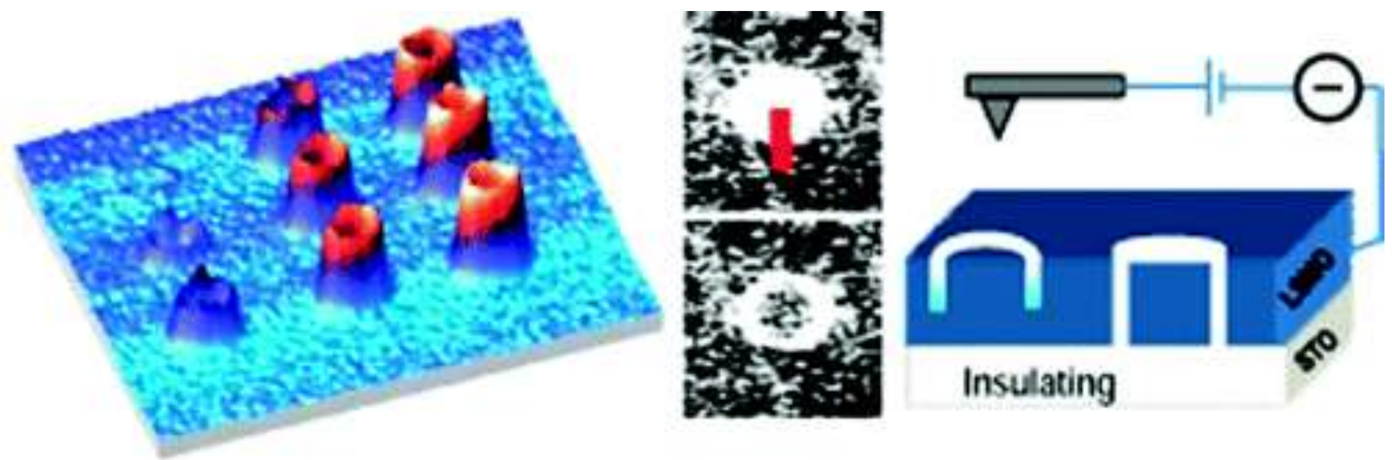
R. García, ICMM-CSIC

# Applications: Local Reversal switching

## Reversal switching in LSMO films for random access memories

- Buffer layer for $YBa_2Cu_3O_7$ coated conductors.
- Magnetic Random Access Memory (MRAM) devices and magnetic sensors.
- Non-volatile resistive random access memories (RRAM)



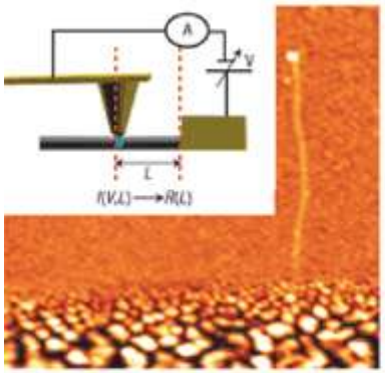

C. Moreno, C. Munuera, S. Valencia, F. Kronast, X. Obradors, and C. Ocal, Nano Lett., 2010, 10 (10), 3828–3835

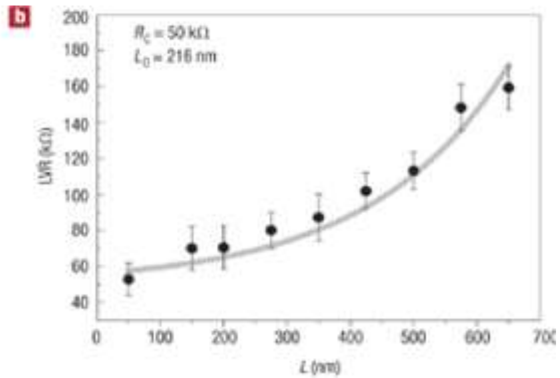
**LSMO film surface lithographed by locally applying a DC voltage with the C-SFM tip.**

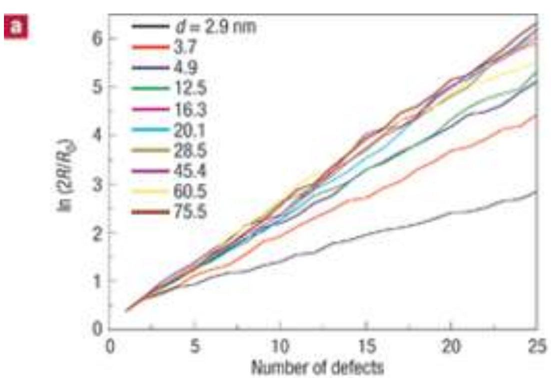
**By writing "circles" we can isolate conducting regions: only where V is applied, the transition occurs. By increasing voltage tip the modified region become more resistive until that the modification ring insolate the central region.**

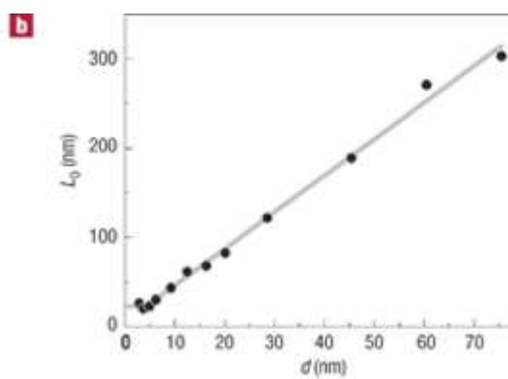
# Applications: Conductive-AFM

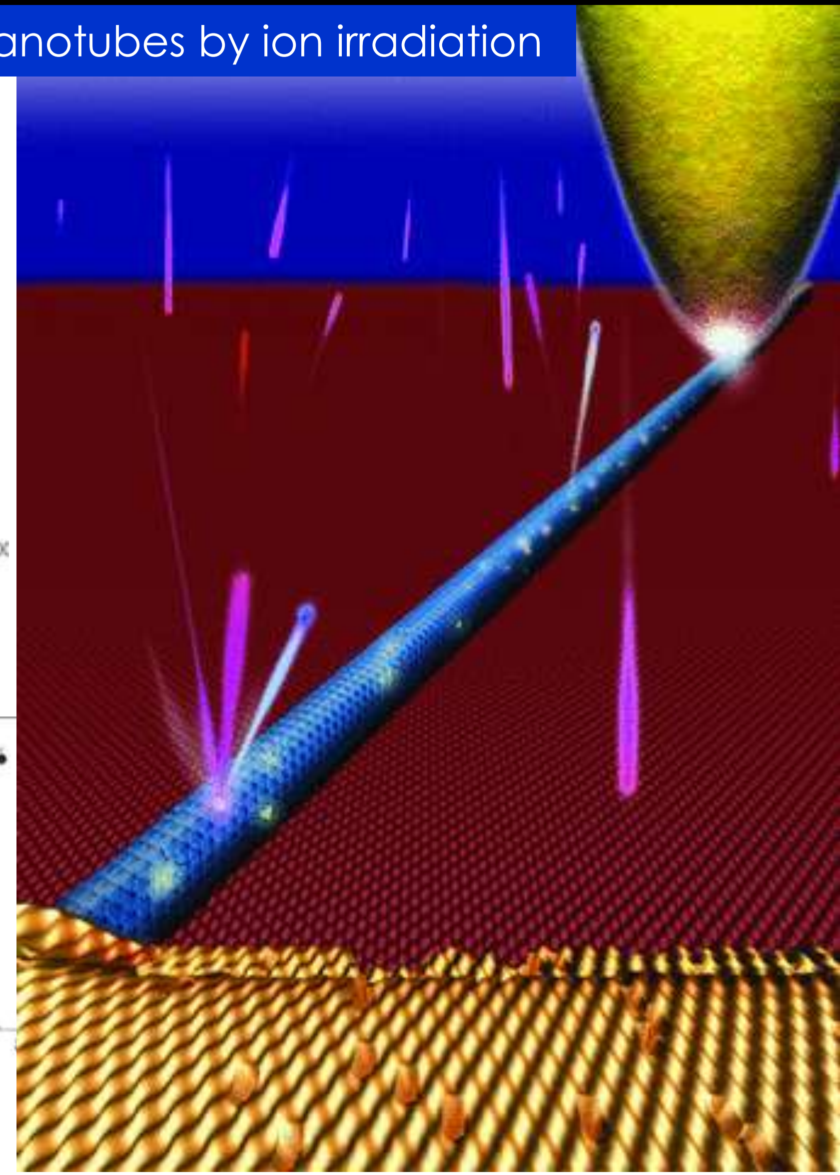
## Tuning the conductance of single-walled carbon nanotubes by ion irradiation



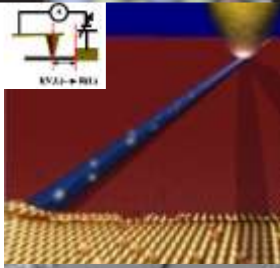



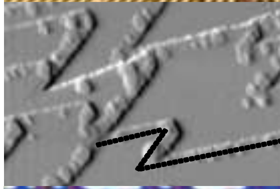
C. Gómez-Navarro, P. J. De Pablo, J. Gómez-Herrero, B. Biel, F. J. Garcia-Vidal, A. Rubio & F. Flores, Nat. Mat. 4, 534 (2005)

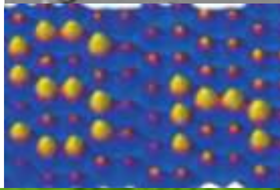
# Applications: contact/non contact

## Contact mode

- Topography. Thin films, nanostructures,…
- Conductivity
- Friction
- Chemical process and growth in real time
- Mechanical properties (friction, adhesion, elastic and plastic properties).

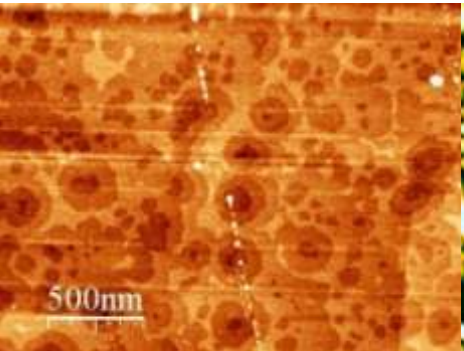



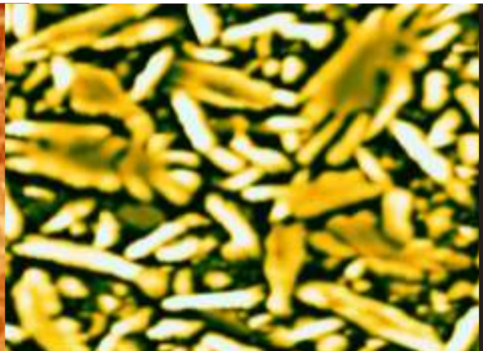


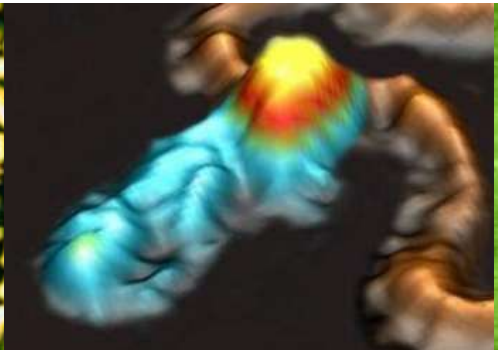


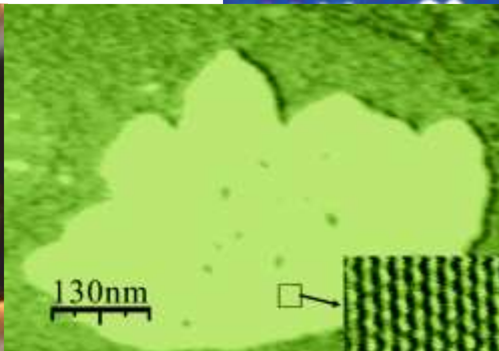
## Non-Contact mode

- **Magnetic** properties, **electrostatic** forces
- Force spectroscopy

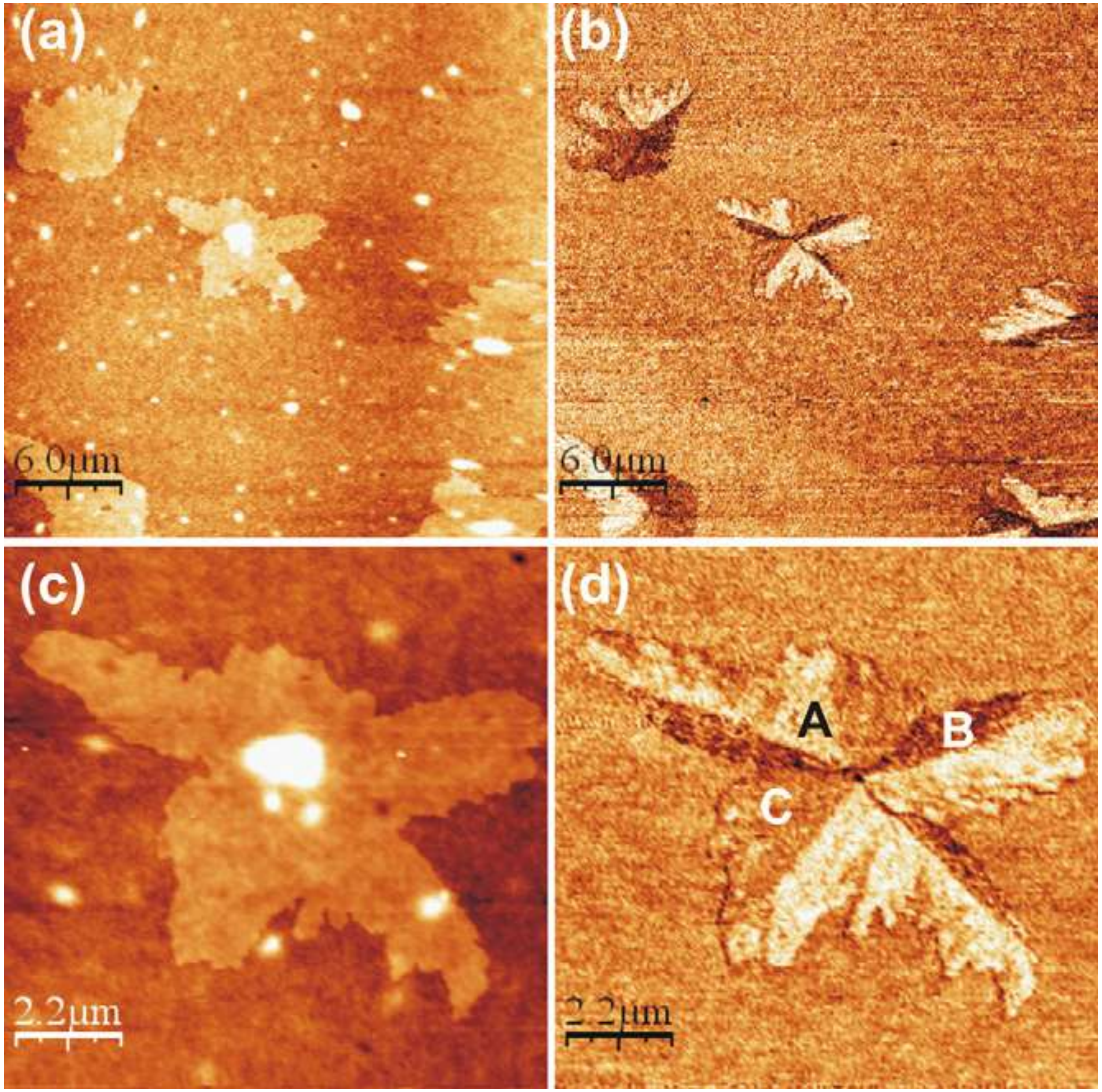


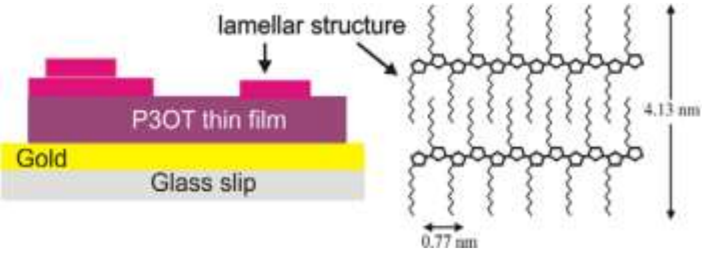







# Applications: Electrostatic interaction




**Kelvin Probe Force Microscopy**

<u>Contact potential</u>

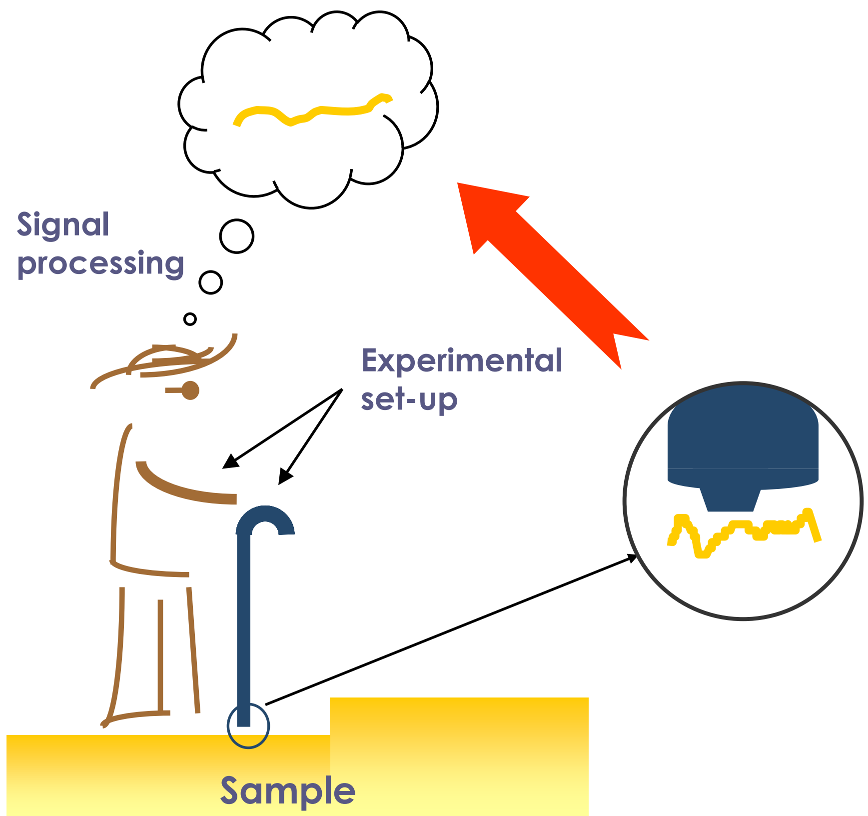
| | |
|---|---|
| A | 411 mV |
| B | 379 mV |
| C | 432 mV |
| D | 386 mV |

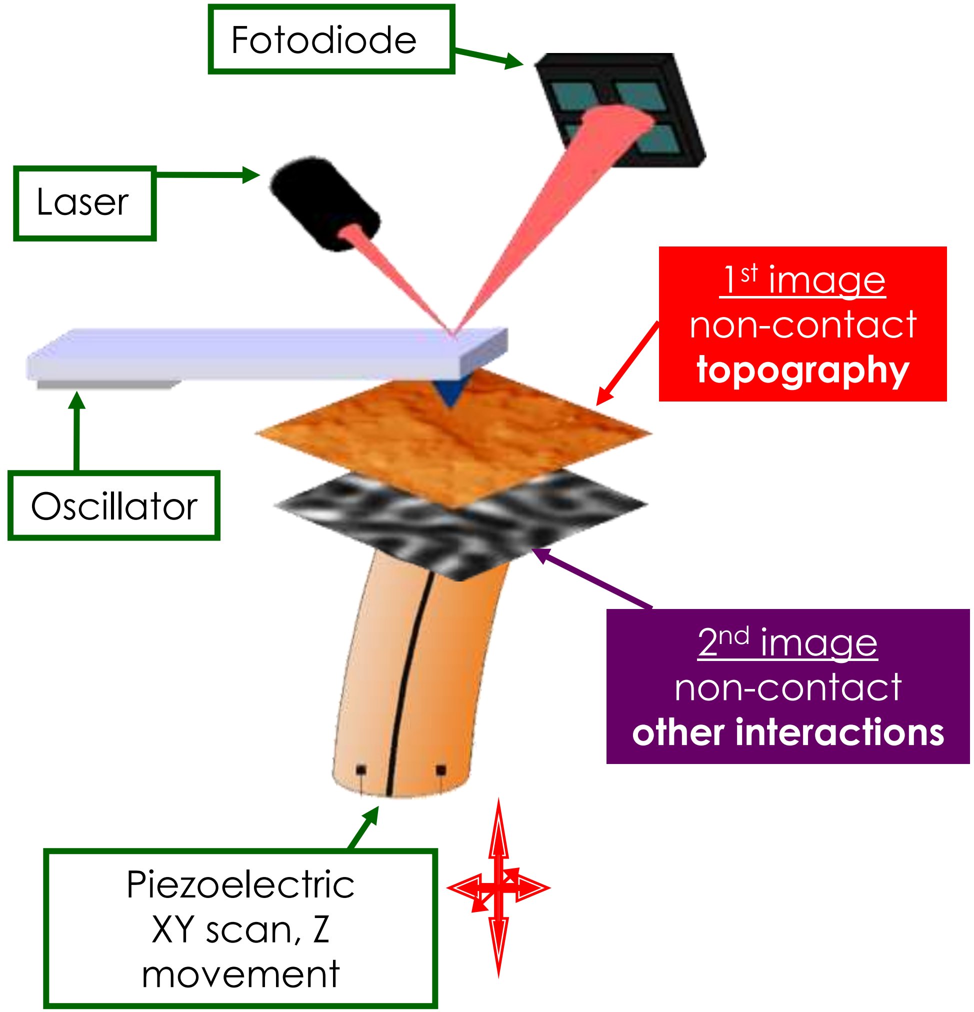
B Pérez-García, J Abad, A Urbina, J Colchero and E Palacios-Lidón, Nanotechnology 19 (2008) 065709

# Outline:

1. Scanning Probe Microscopies.
2. Fundaments of MFM. Measuring under standard conditions
3. Improving the hardware:
   a. Variable Field MFM
   b. HV-Low Temperature MFM
   c. MFM probes
4. Developing new operation modes:
   a. 3D modes
   b. KPFM-MFM combination: Co nanostripes, Graphite, Graphene based hall sensor
   c. Dissipation
5. Conclusions

# SPM - Magnetic Force Microscopy



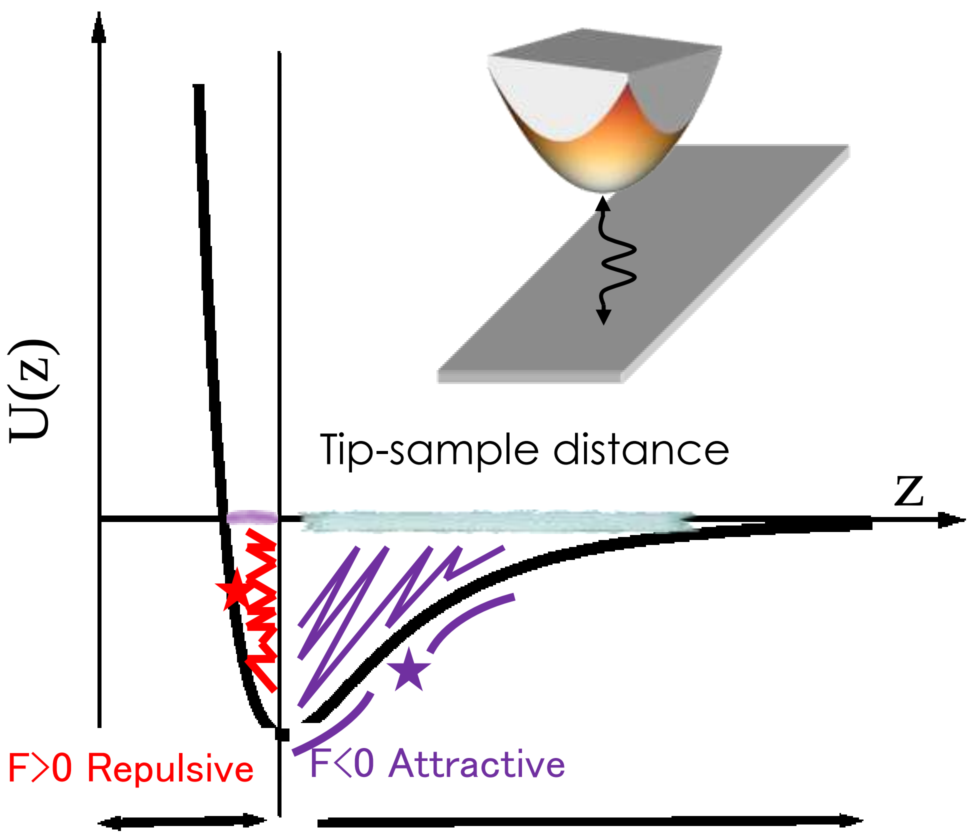



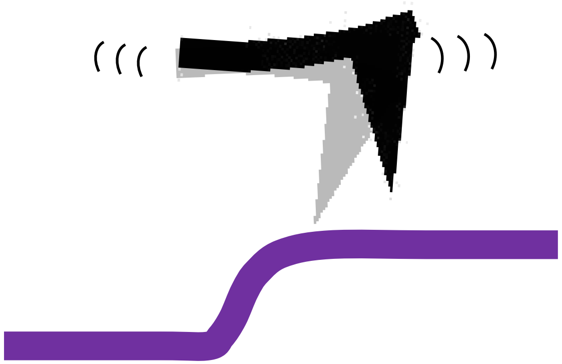

- **Operation modes**
- **Functionalized probes**

# SPM-Operation modes: contact/non-contact

**Assuming the main interaction is van der Waals**, near the surface:

- attractive forces at long distances
- repulsive forces at short distances



Dynamic mode:

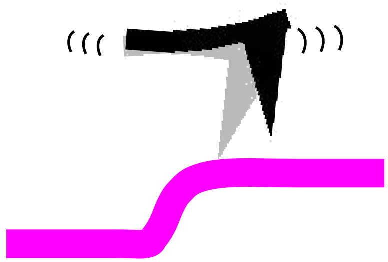
Keeping constant the cantilever oscillation **amplitude** or the **frequency shift**

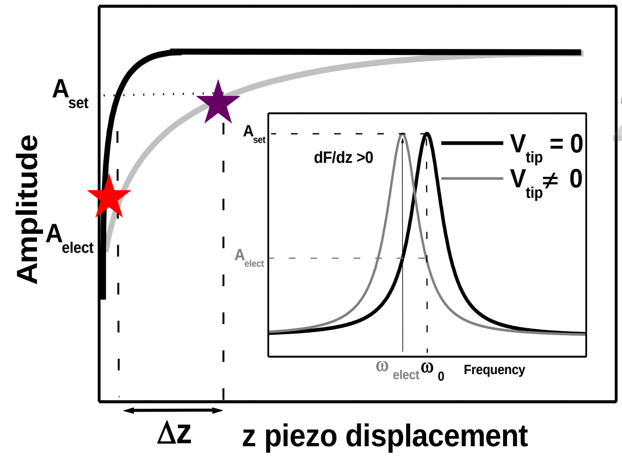
~ constant tip-sample distance

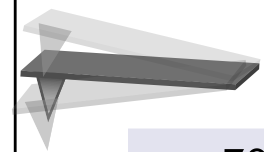
# Operation mode in MFM

## Dynamic mode:
## **A**mplitude **M**odulation

Constant oscillation **amplitude**





ω~ 70 kHz
k~ 3 N/m

$$m\ddot{z}(t) = -kz(t) - \gamma\dot{z}(t) + F_{exc}(t) + F_{int}(z(t), \dot{z}(t), \vec{E}, \vec{B})$$

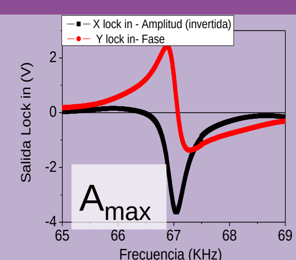
Only for small amplitudes

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

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

Z retrace>10nm → Two scans

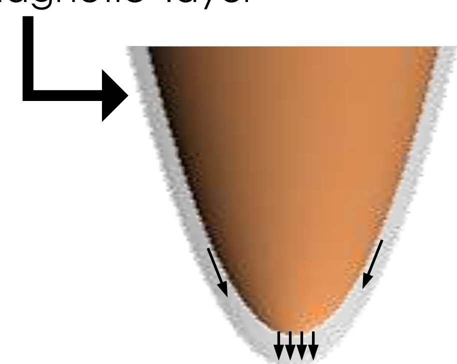
## PLL feedback




Constant phase

MFM signal = frequency shift

Magnetic layer



# MFM images interpretation

Assuming the tip-sample influence <u>is negligible</u>:

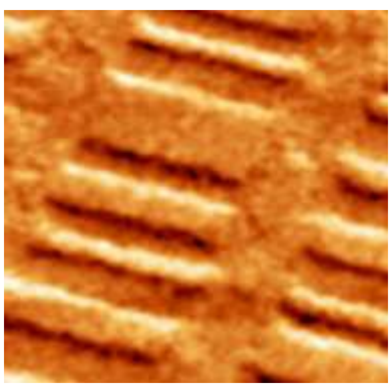
- The MFM contrast is proportional to the magnetic pole density at the surface.
- **Perpendicular anisotropy:** Poles at the center of the domains.
- **In-plane anisotropy:** Poles at the domain walls


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

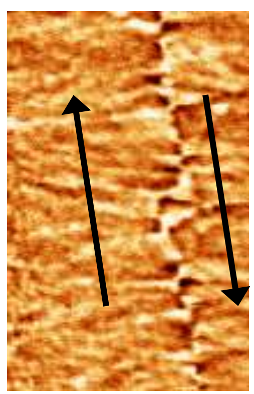
# MFM images interpretation

Assuming the tip-sample influence is negligible:

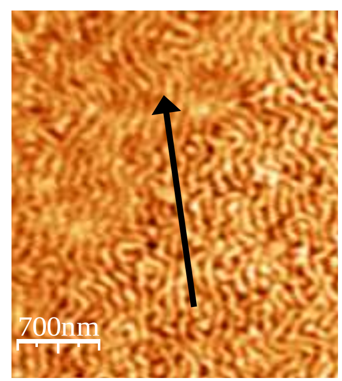
- The MFM contrast is proportional to the magnetic pole density at the surface.
- **Perpendicular anisotropy:** Poles at the center of the domains.
- **In-plane anisotropy:** Poles at the domain walls

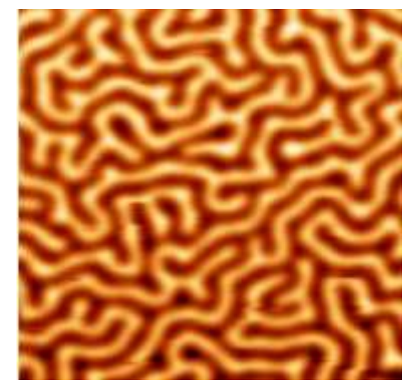
Hard disk image.
10 μm x 10 μm



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. XRCD ~10000 AFM
- Lateral resolution better than 20nm
- Additional information (3D topo,...)
- To study individual elements
- Trouble-free sample preparation, also to check processes during fabrication




Physical methods

Chemical or electrochemical methods

Nanolithography

# CoCrPt thin films, perpendicular anisotropy

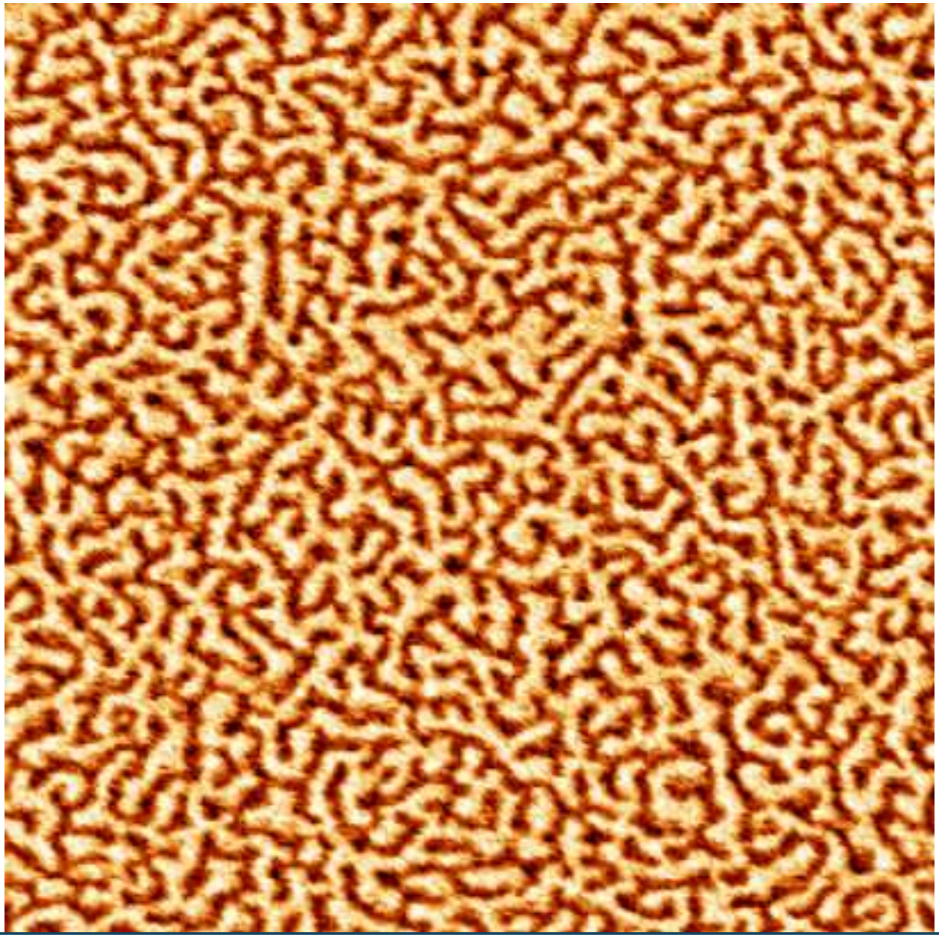


Image Size:
5μm×5μm

**Stripe domains.**

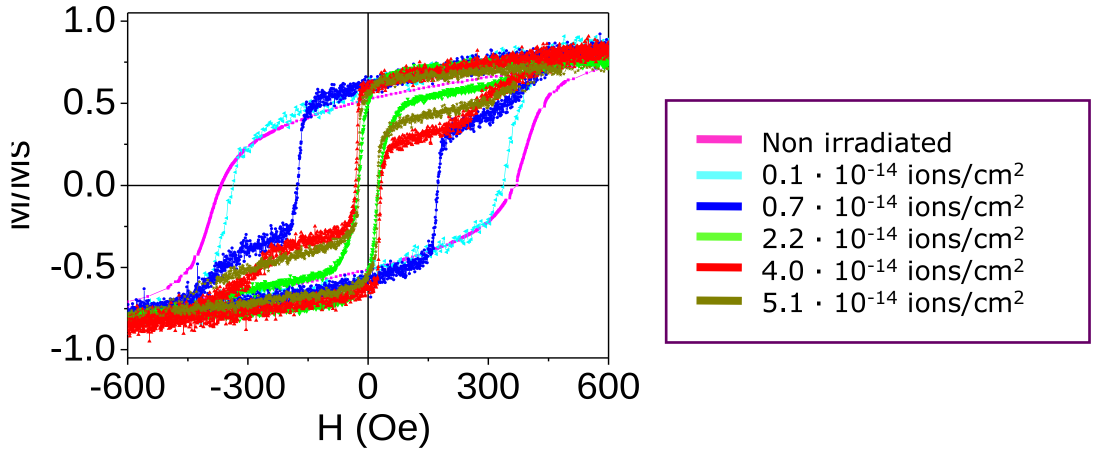
MFM images of 20-nm-thick **CoCrPt** film on a smooth substrate after an out of plane ac-demagnetization process

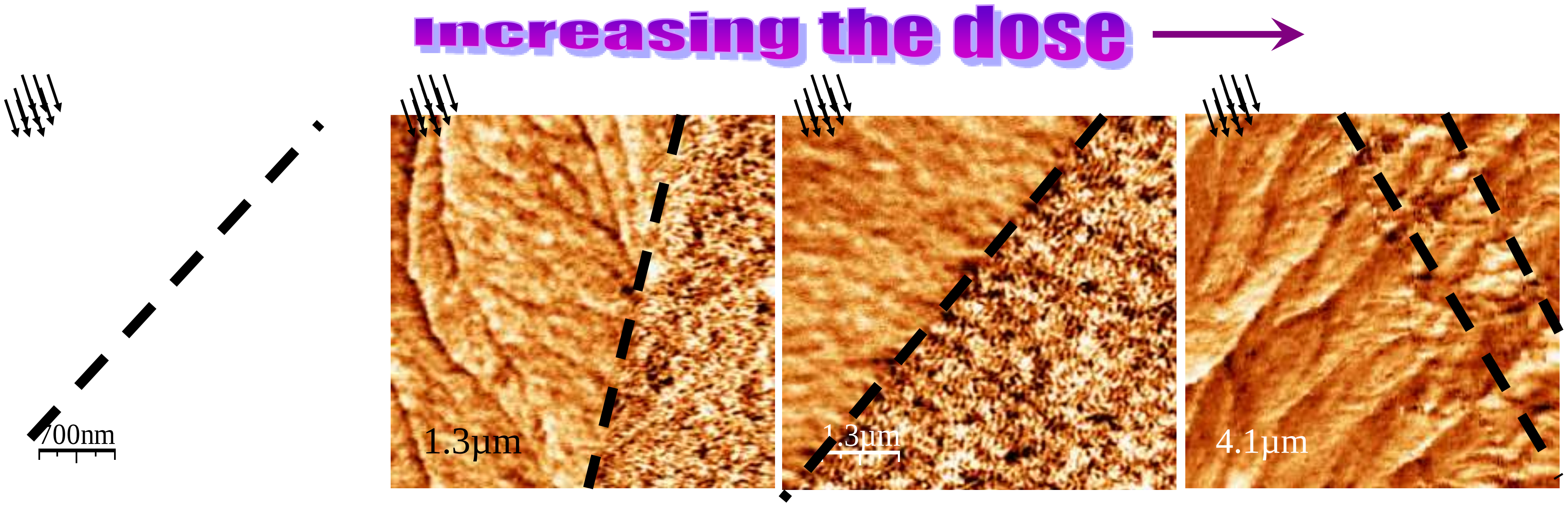
D. Navas et al. In preparation

# FePt thin films, slight out of plane anysotropy

FePt thin film 35 nm thick
Irradiated by $Cl^{2+}$ ions 4 MeV

VSM loops:
In plane hysteresis loops

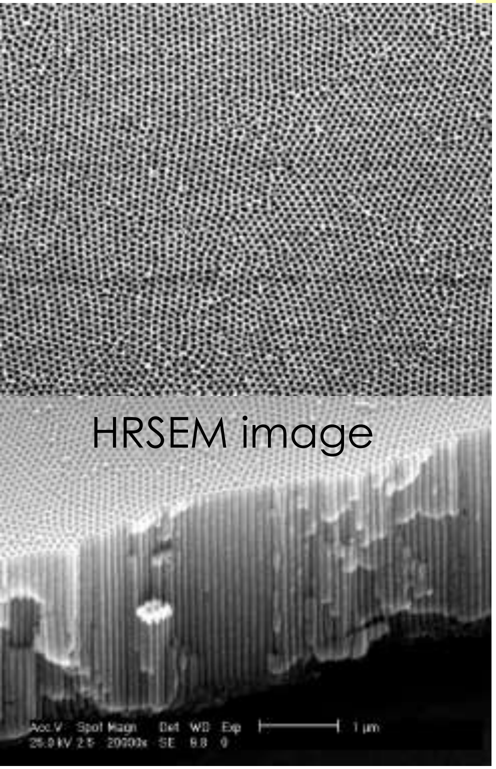


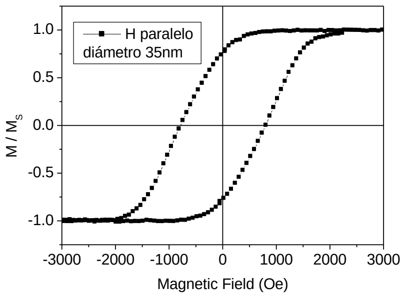




M. Jaafar, R. Sanz, M. Vázquez, A. Asenjo , J. Jensen ,K. Hjort, S. Flohrer, J. McCord and R. Schäfer, Phys. Stat. Sol. (a) 204 (6), 1724 (2007)

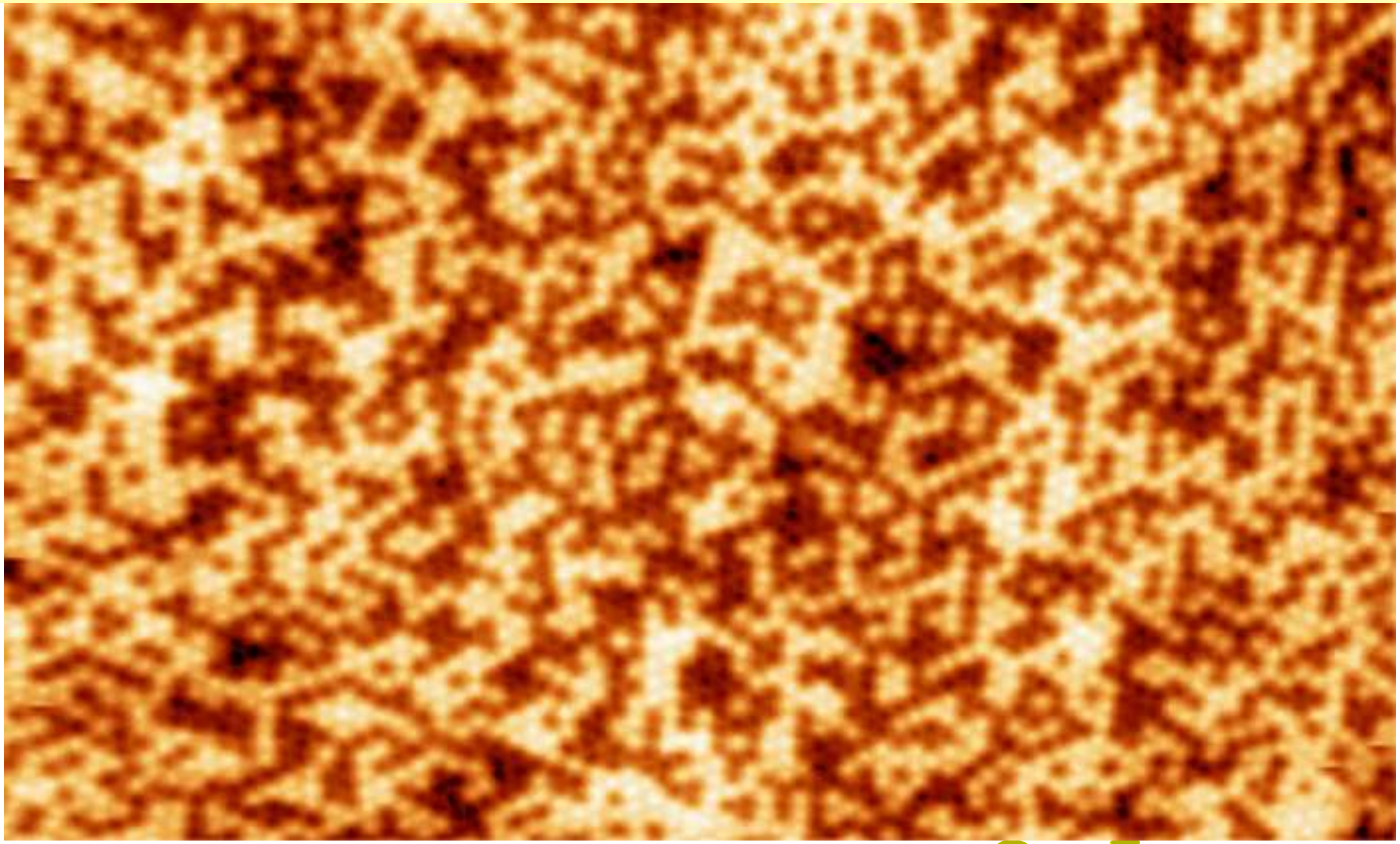
# Magnetic Nanowires Arrays

Anodic Alumina Membranes

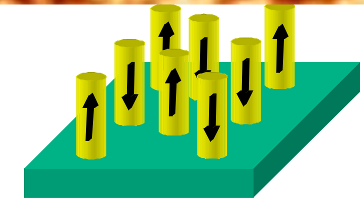



MFM image of an array of Ni nanowires embedded in AAM.
Diameter: 35nm, interpore distance: 105nm.



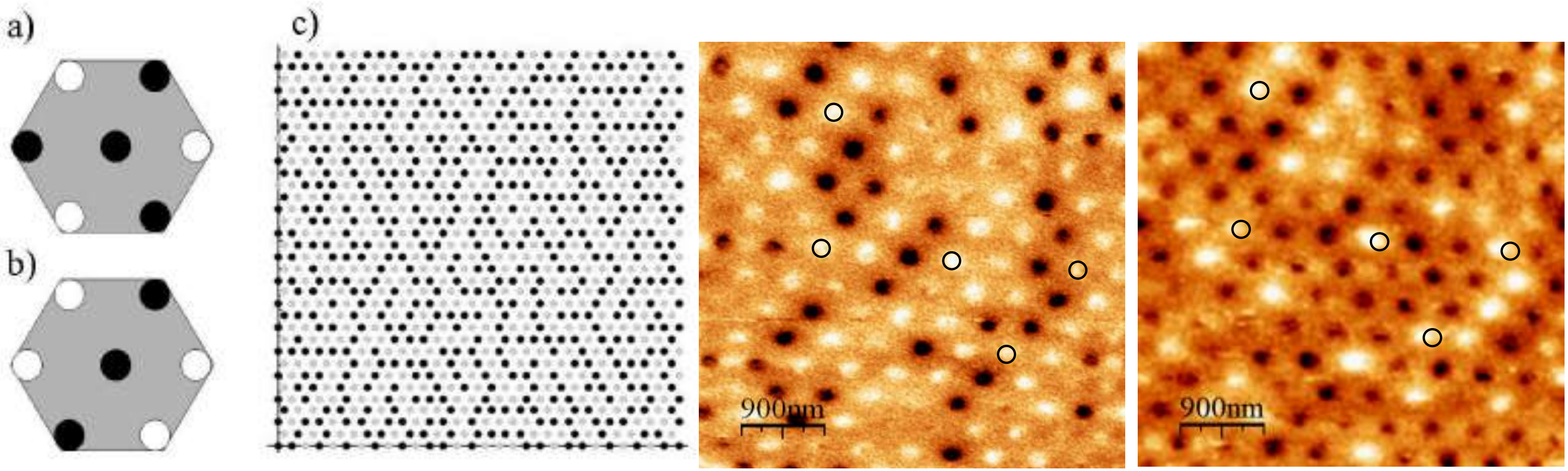
- ***Parallel** to the tip stray field*
- ***Antiparallel** to the tip stray field*



M. Vázquez et al. Euro. Phys. J. B 40(4) (2004)

# Magnetic nanowires, 180nm in diameter

Ni nanowires:
Diameter: 180 nm,  Lattice parameter: 480 nm,  Lenght: 3.6 μm



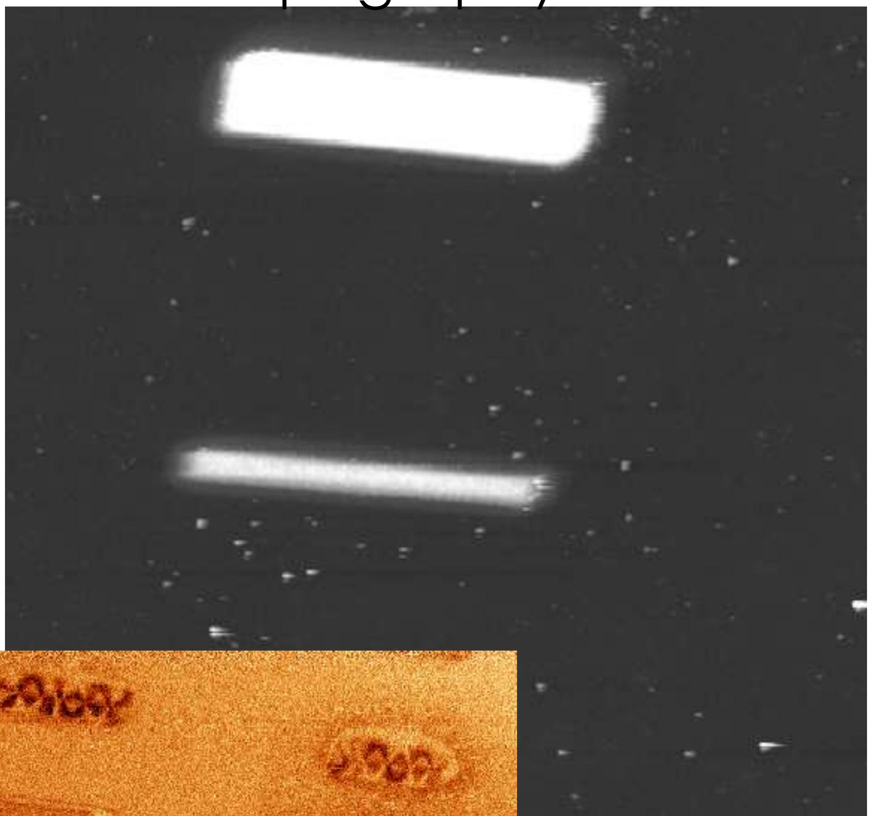

Labyrinth distribution due to dipolar (and multipolar) interactions between nanowires.
Images at different remanent states

J. Escrig, D. Altbir, M. Jaafar, D. Navas, A. Asenjo  and M. Vázquez. Rev. B 75, 184429 (2007)

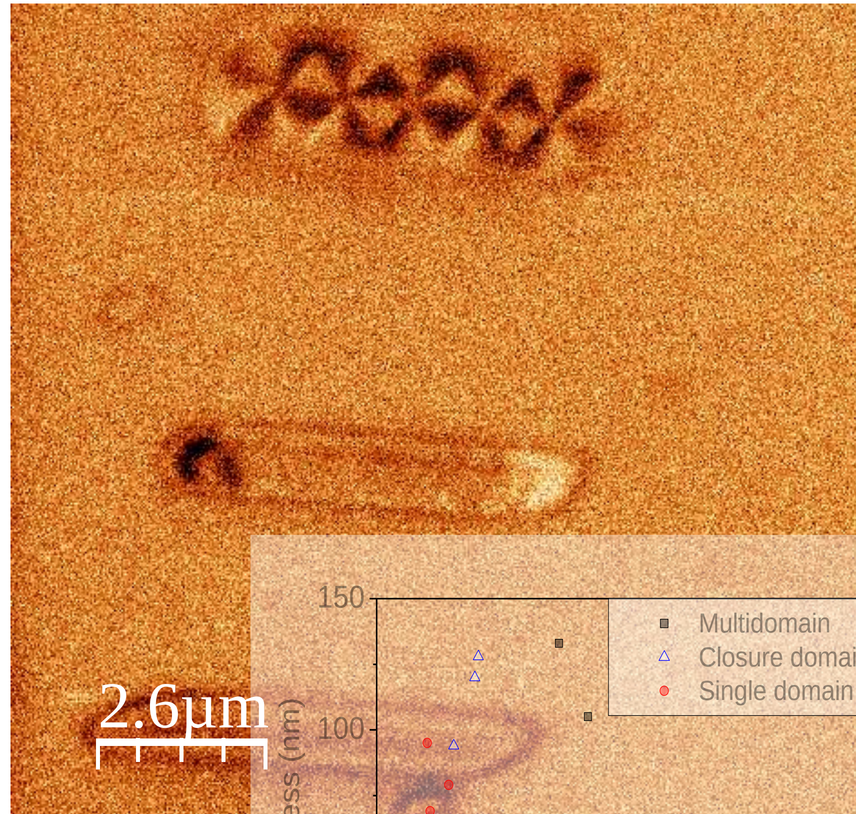
# Co nanostripes: from single to multidomain

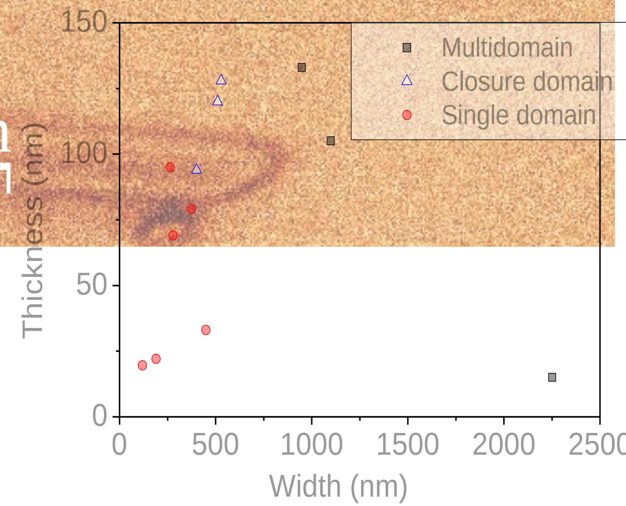
## Relationship between the domain structure and its aspect ratio

Topography



MFM



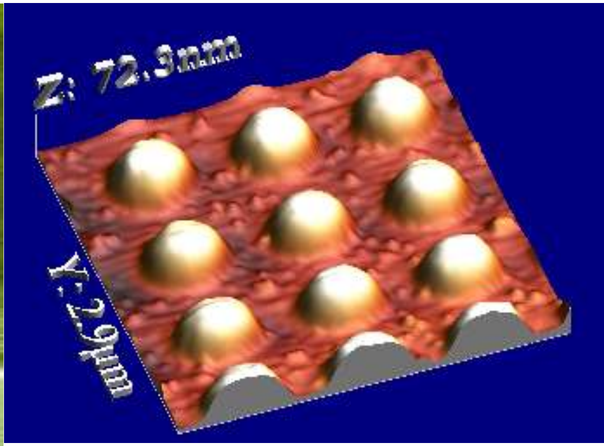

Co nanostripes prepared by Focused Electron Beam Induced Deposition.

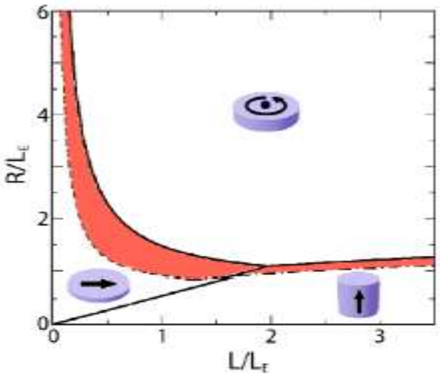
**J.M. de Teresa** and **R. Ibarra**, ICMA-CSIC, INA

# Ni (111) Nanostructures

**VORTEX**

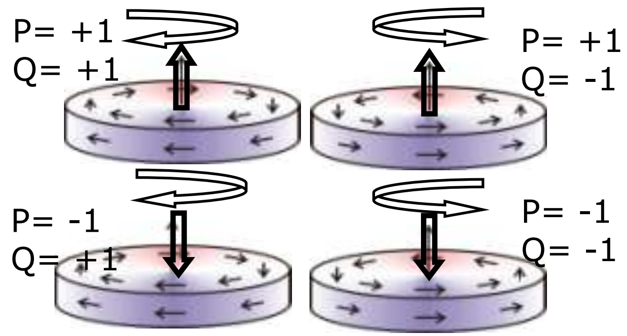
Lateral dimensions~500 nm;
thickness~50nm; distance~800 nm



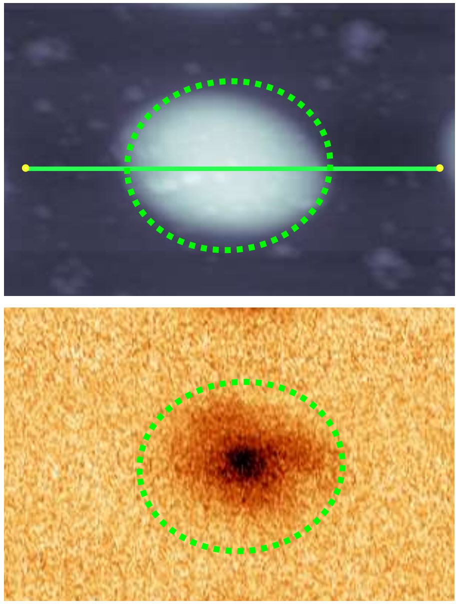



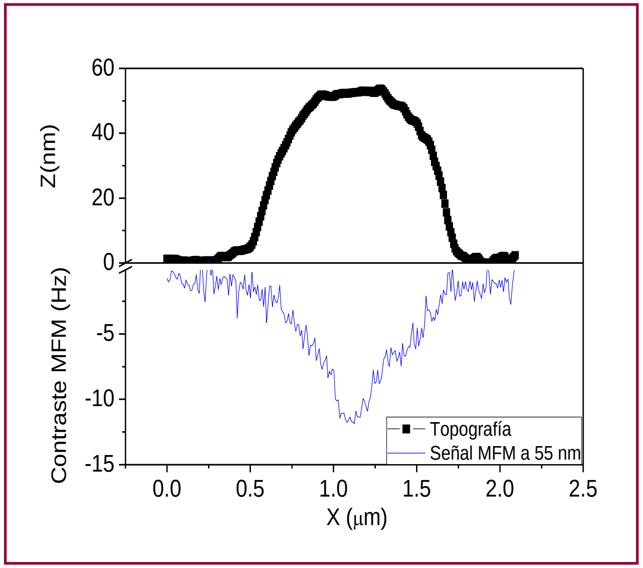

**Energetic balance:**
exchange enegy,
magnetostatic,
magnetocristalline,
shape and induced
anysotropy

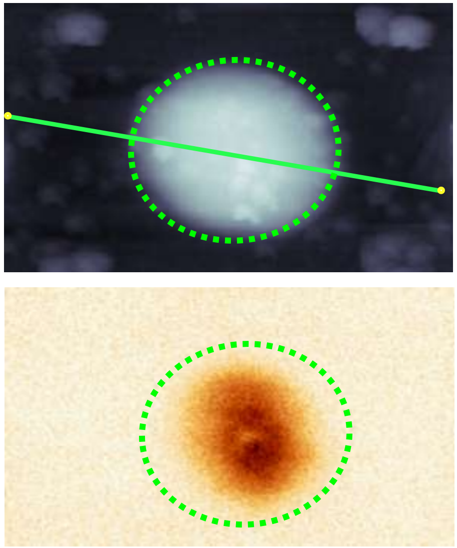
**Chirality** – closure flux direction
**Polarity** – Core magnetization

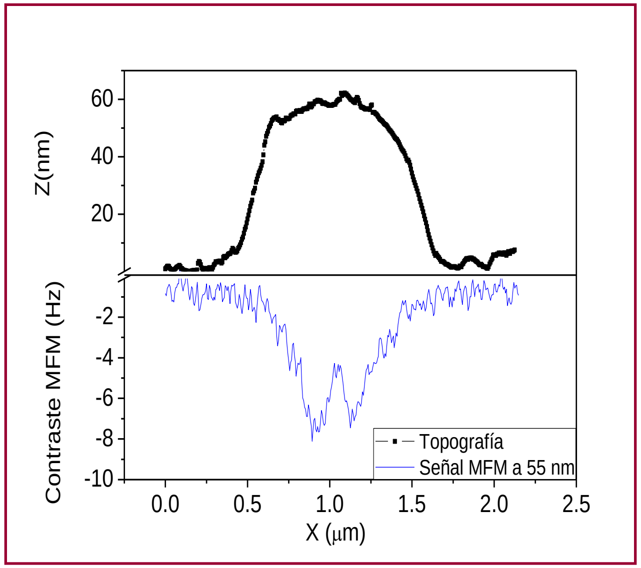


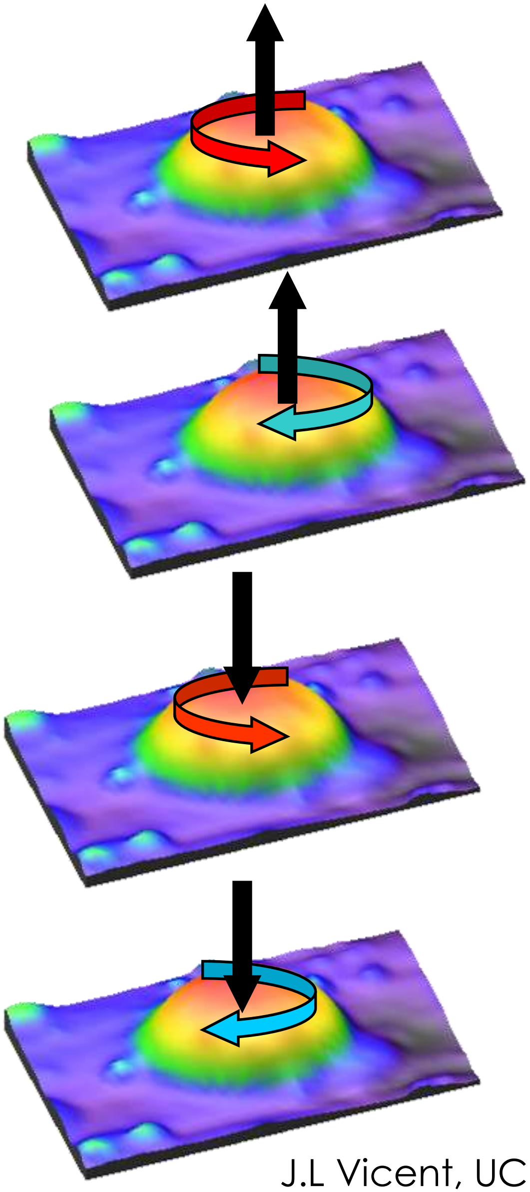
# Ni (111) Nanostructures





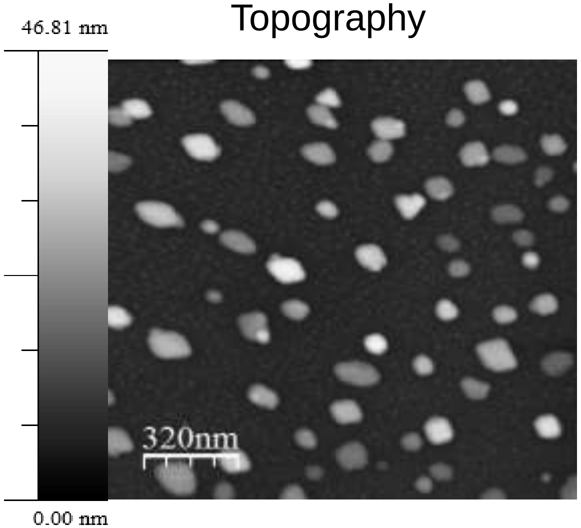


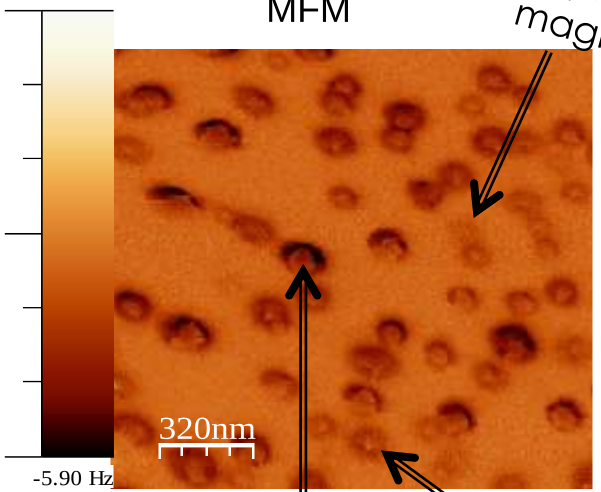



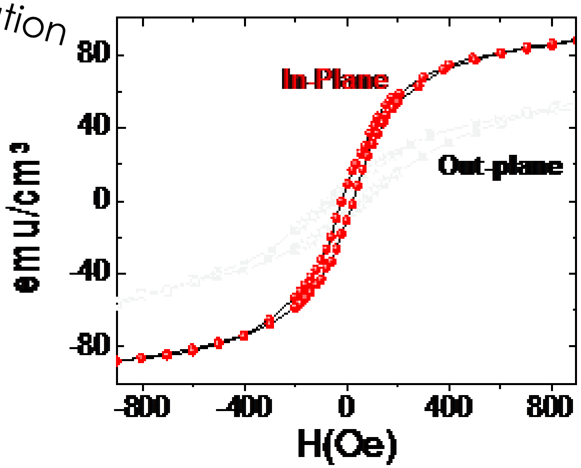
J.L Vicent, UCM

# Smaller structures. LSMO nanoislands

LSMO/YSZ prepared by CSD methods. Ferromagnetic at RT (Tc=360K)
X. Obradors' group, ICMAB-CSIC
Slight changes in shape give different magnetic configuration



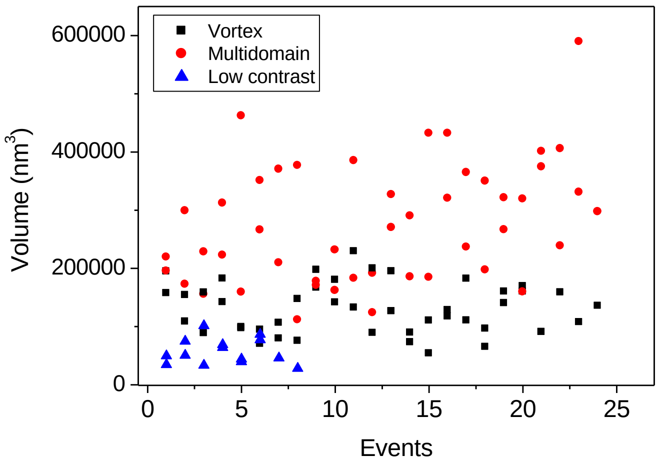

**Special MFM probes**

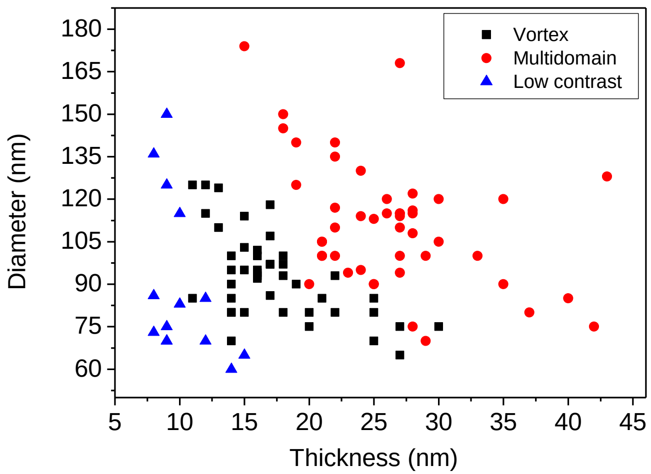
J. Zabaleta et al J. Appl. Phys, 111(2) 2012

# Smaller structures. LSMO nanoislands

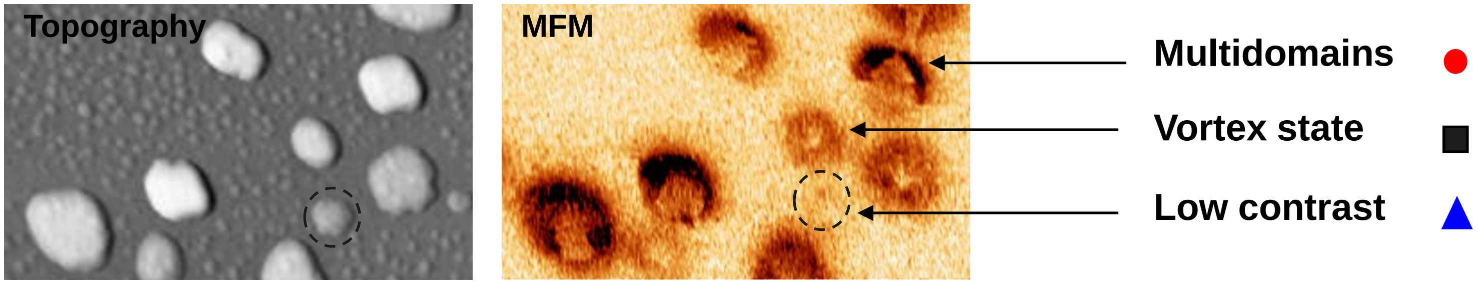
## LSMO/YSZ prepared by CSD methods. Ferromagnetic at RT (Tc=360K)

There is a relationship between the size and the magnetic configuration




Different kind of structures




J. Zabaleta et al J. Appl. Phys..111(2) 2012 , X. Obradors`s group ICMAB-CSIC

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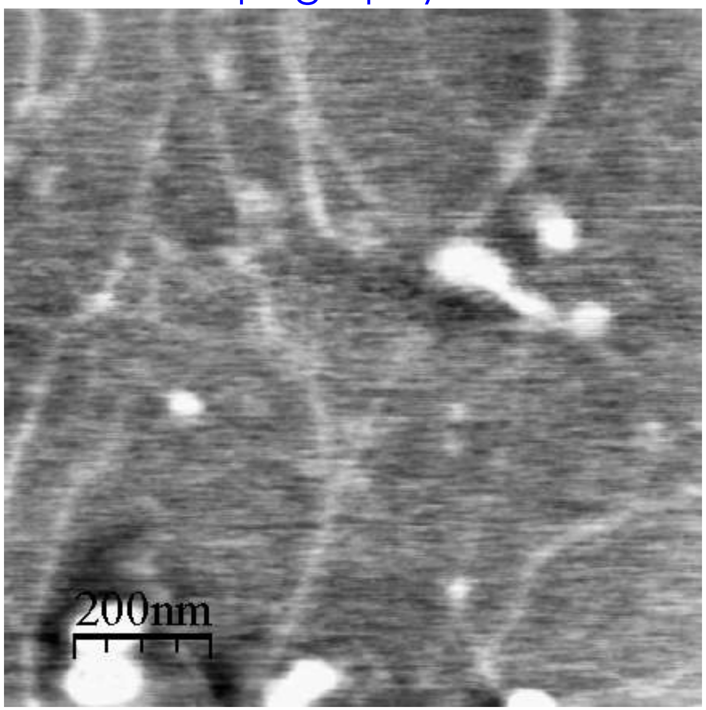
    c. Dissipation
5. Conclusions

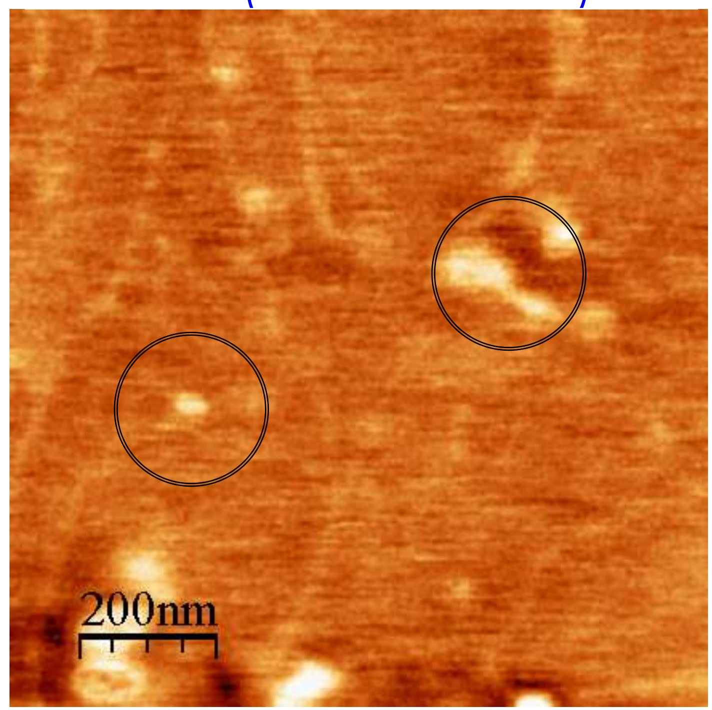
# Some challenges in MFM: measuring low moment-low coercivity nanoparticles

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

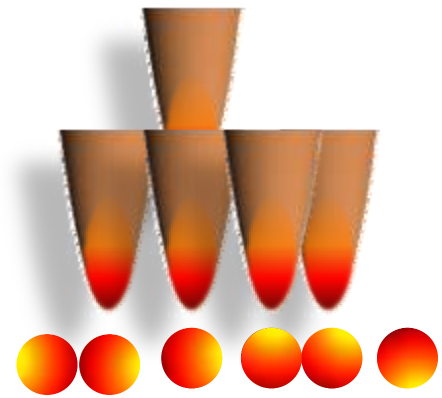
Topography




MFM (z retrace=30nm)



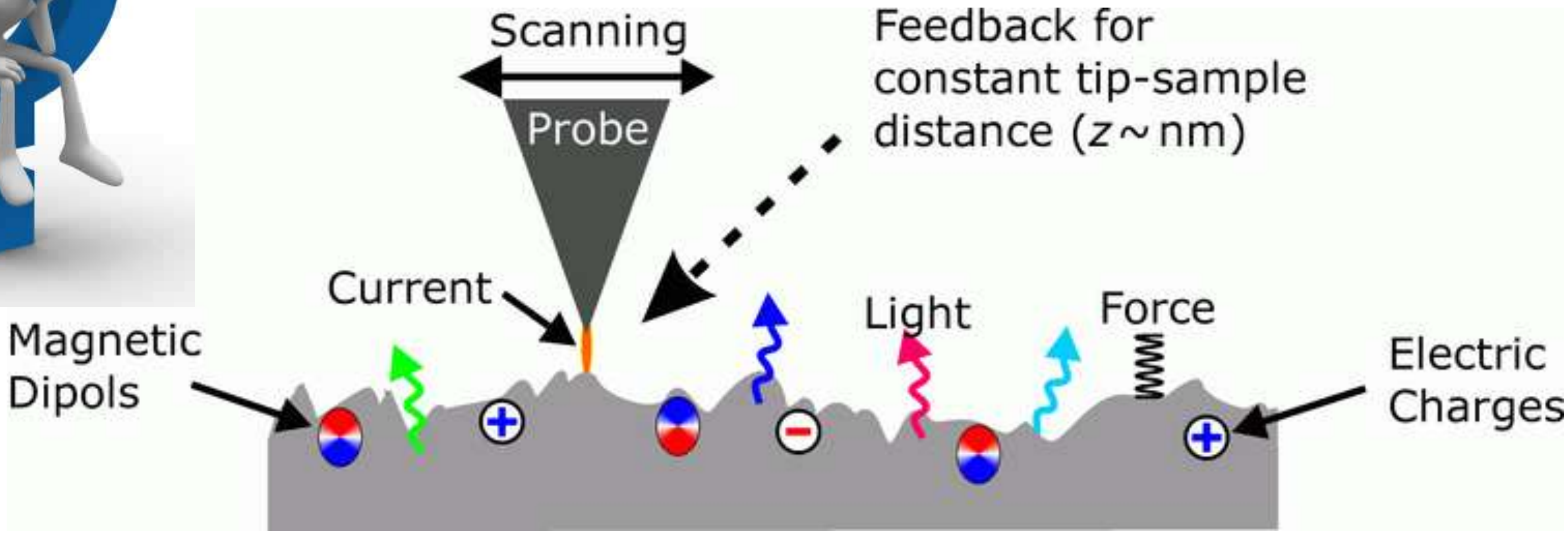

Low coercivity NP



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

# To take into account…

Different interactions can produce changes in amplitude and frequency



# What are the applications? Why MFM?

- Low cost technique. XRCD ~10000 AFM
- Lateral resolution better than 20nm
- Additional information (3D topo,...)
- To study individual elements
- Trouble-free sample preparation, also to check processes during fabrication


- What's the meaning of the contrast?
- What's the influence of the tip stray field?
- What's the higher lateral resolution?
- Is it magnetic?

# Some challenges in MFM

- Lateral resolution
- Low magnetic moment
- Tip influence
- Other tip-sample interactions
- Additional information

Special MFM probes

Variable Field MFM

KPFM-MFM

New MFM modes

# Outline:

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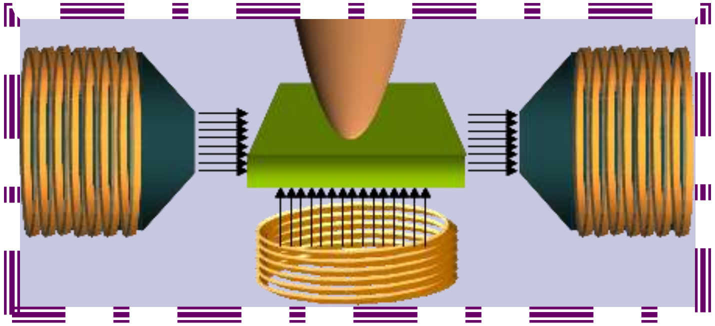
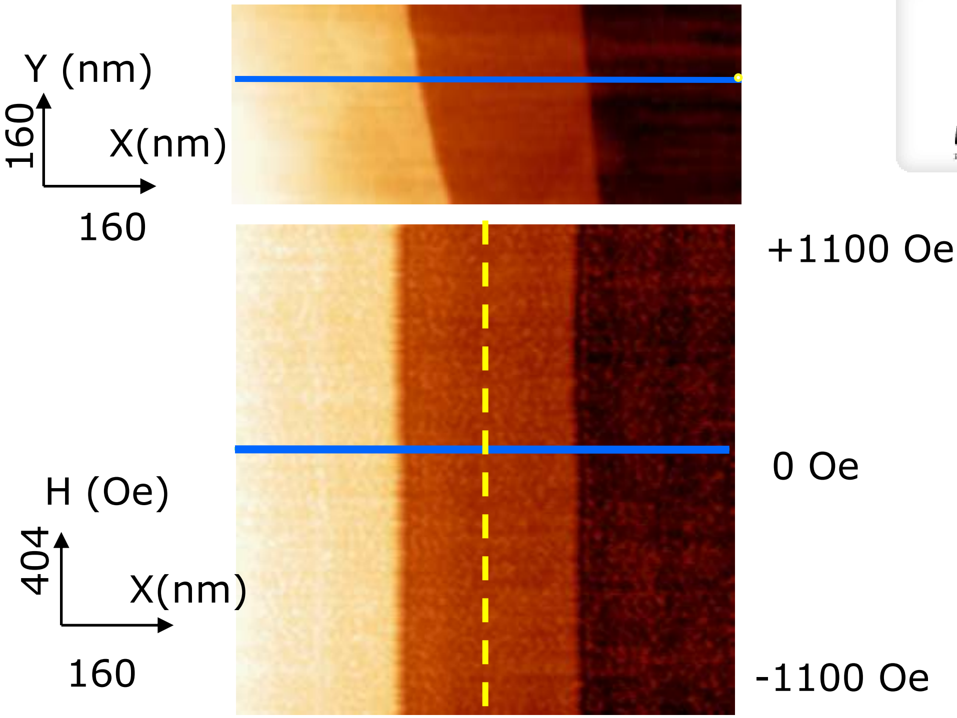
# Variable Field Magnetic Force Microscope

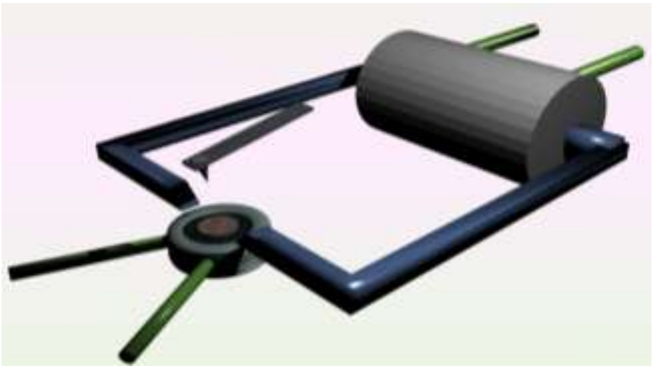


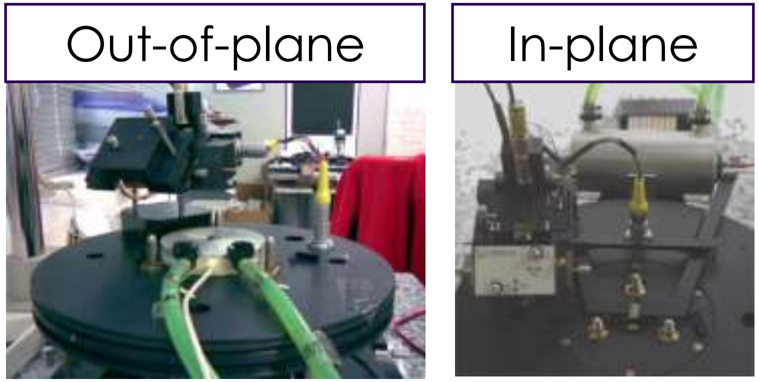
- Out-of-plane magnetic field up 1.5 kOe
- In-plane magnetic field up to 2 kOe

**Mechanical stability** → 0. 014 nm/Oe

**Thermal stability** →Δ T = 2 °C (Field max, t = 180 min)



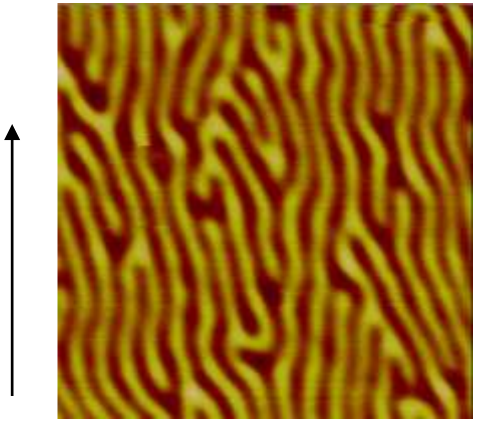





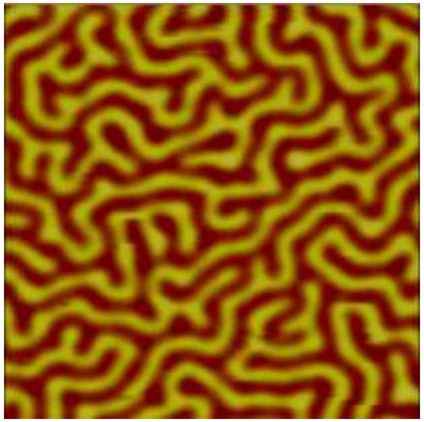

M Jaafar, J Gómez-Herrero, A Gil, P Ares, M Vázquez and A Asenjo, Ultramicroscopy (2009)

# MFM and magnetic field

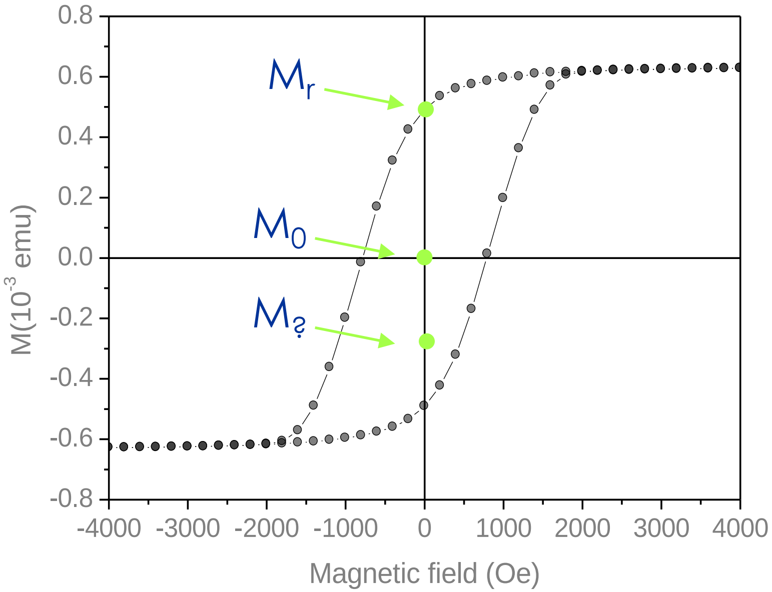
Sample: FePd thin film high perpendicular anisotropy



After saturate



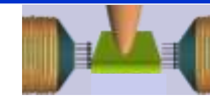
Demagnetized



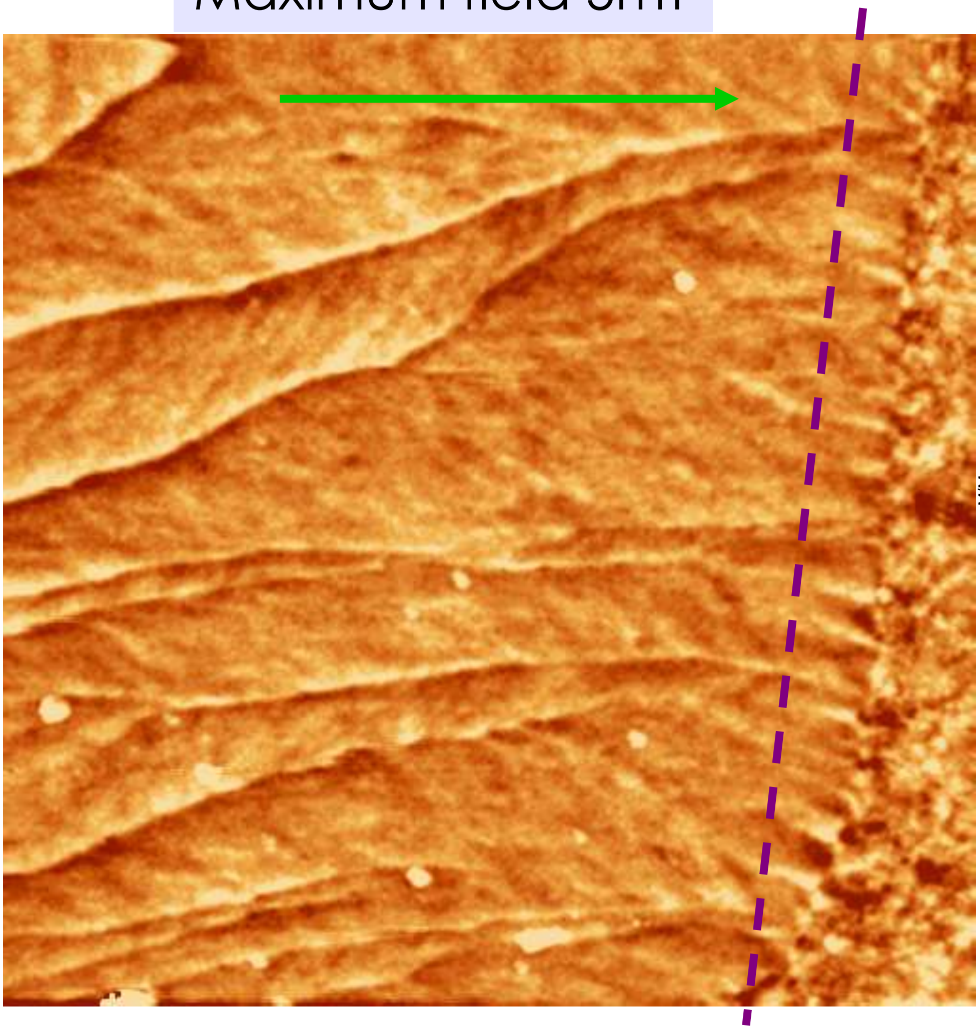

Domain configuration depends on the previous magnetic story

Magnetization Reversal process at the nanoscale

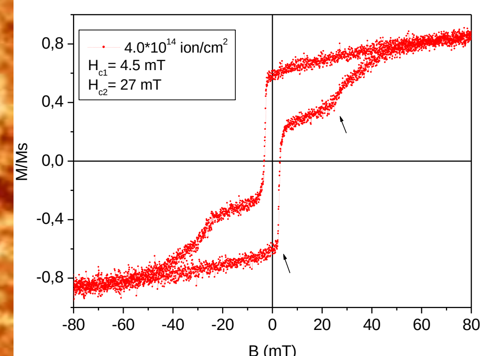
# VFMFM in irradiated FePt

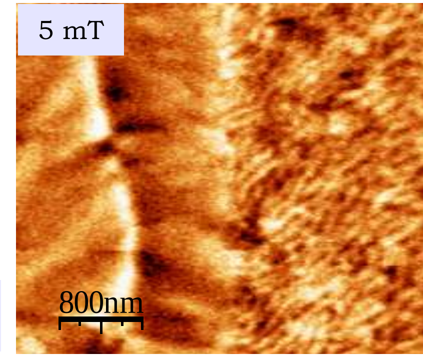


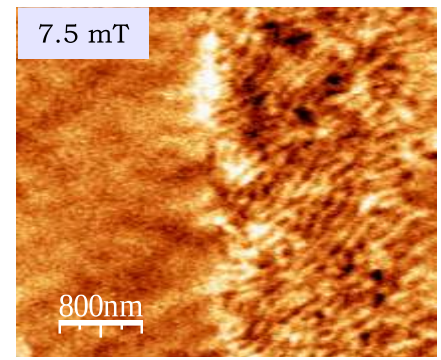
Maximum field 5mT

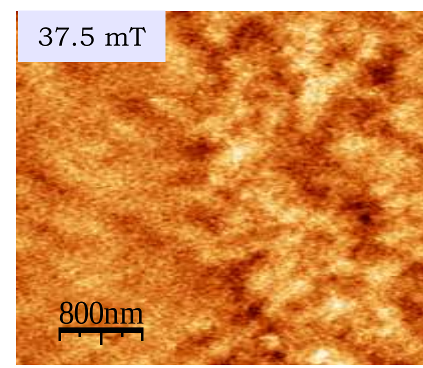


VSM in plane loops



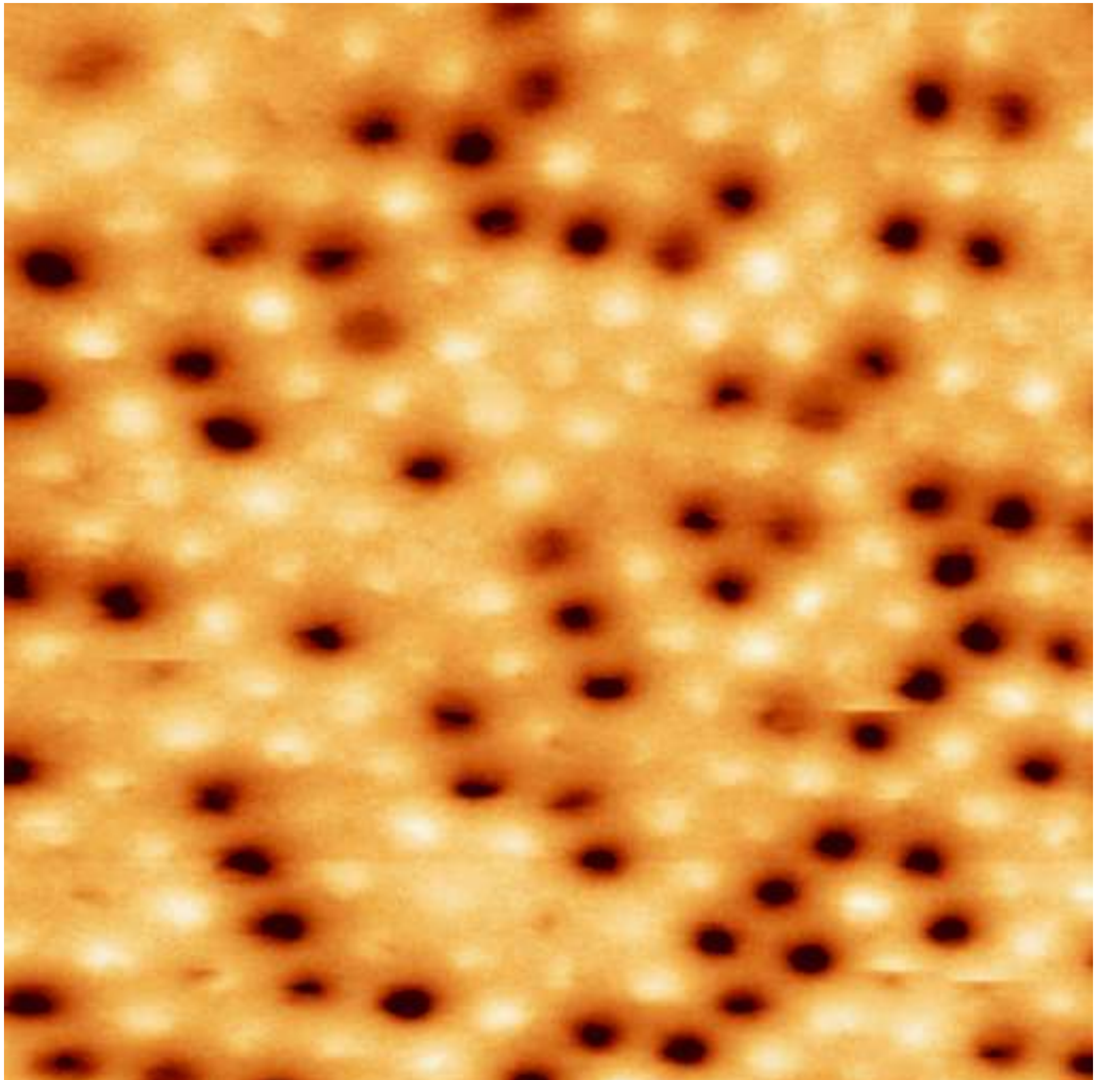



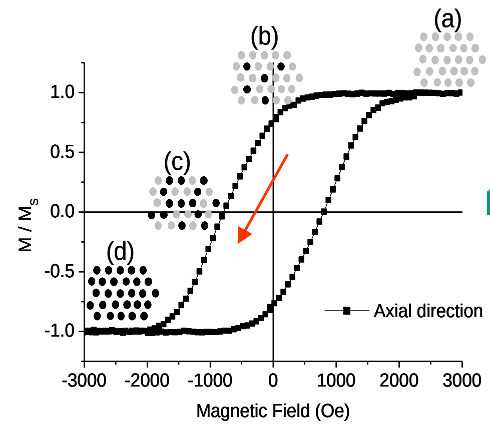



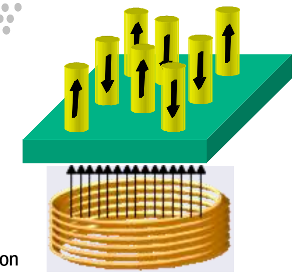



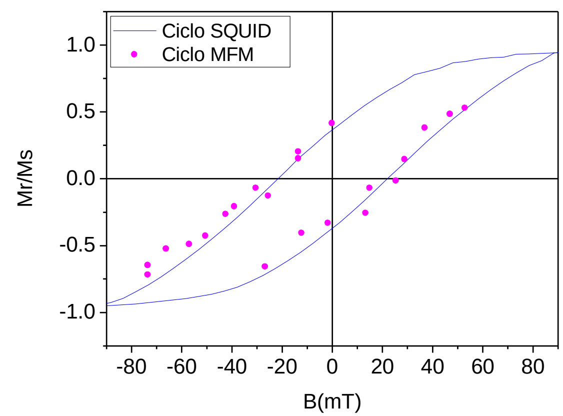

M. Jaafar, R. Sanz, M. Vázquez, A. Asenjo , J. Jensen ,K. Hjort, S. Flohrer, J. McCord and R. Schäfer, Phys. Stat. Sol. (a) 204 (6), 1724 (2007)

# VFMFM: Ni nanowires, 180nm in diameter







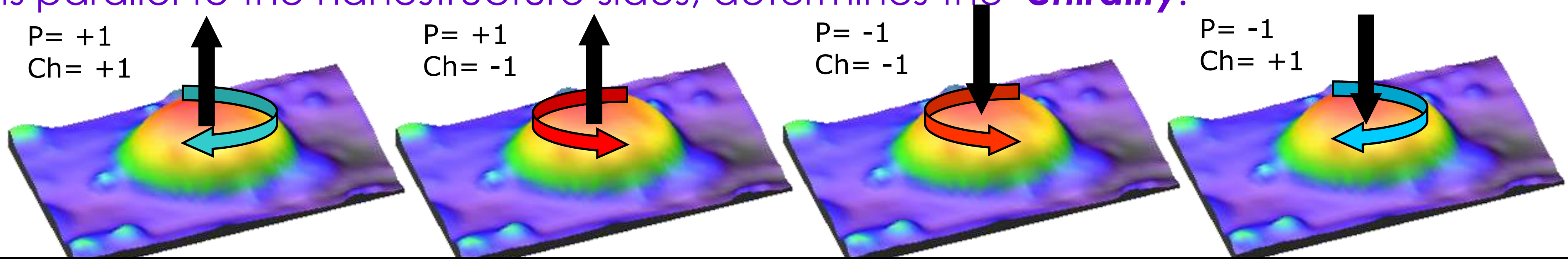

$$M_r/M_s = (N_{brigth} - N_{dark}) / N_{total}$$

Reversal magnetization process of individual nanowires. Hysteresis loops of the sample

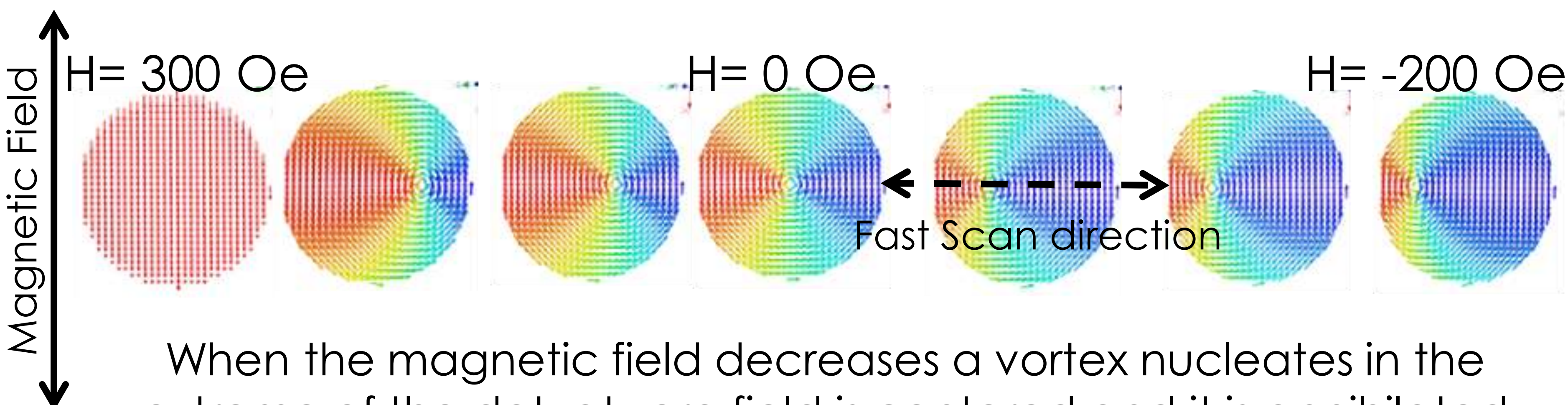
M Jaafar, J Gómez-Herrero, A Gil, P Ares, M Vázquez and A Asenjo, Ultramicroscopy (2009)

# Vortex configuration in Py dots

We can distinguish two regions: **core and external region.** The core, at the center of the dot, presents out-of-plane magnetization in up or down direction that determines the ***polarity***. The external region where the magnetization flux, is parallel to the nanostructure sides, determines the ***chirality***.

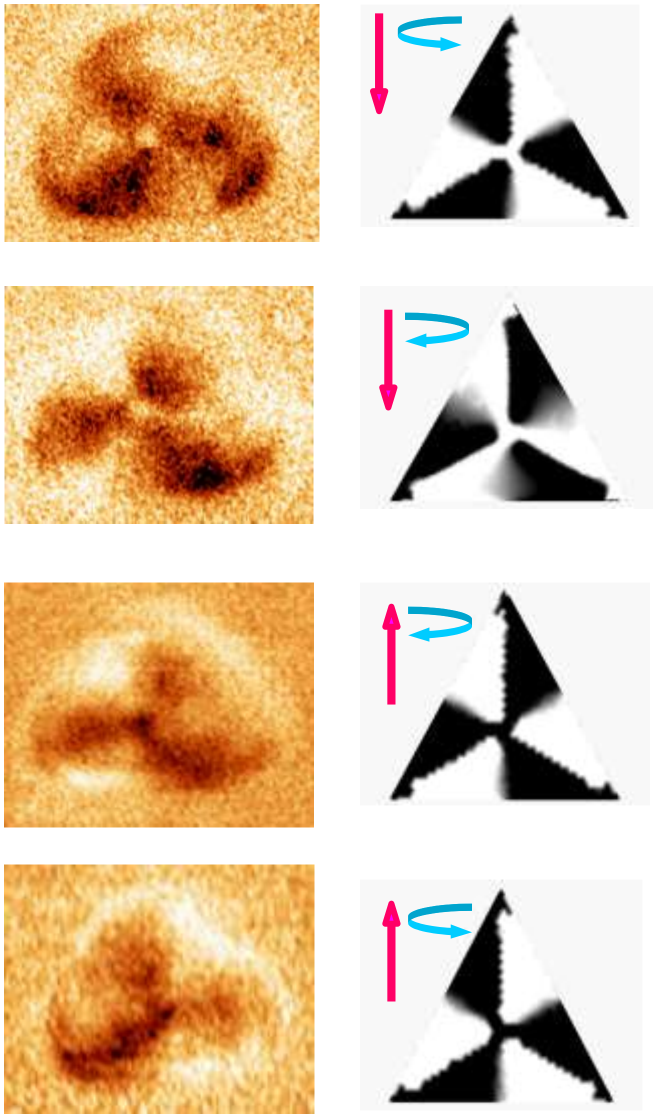



# Reversal Magnetization process



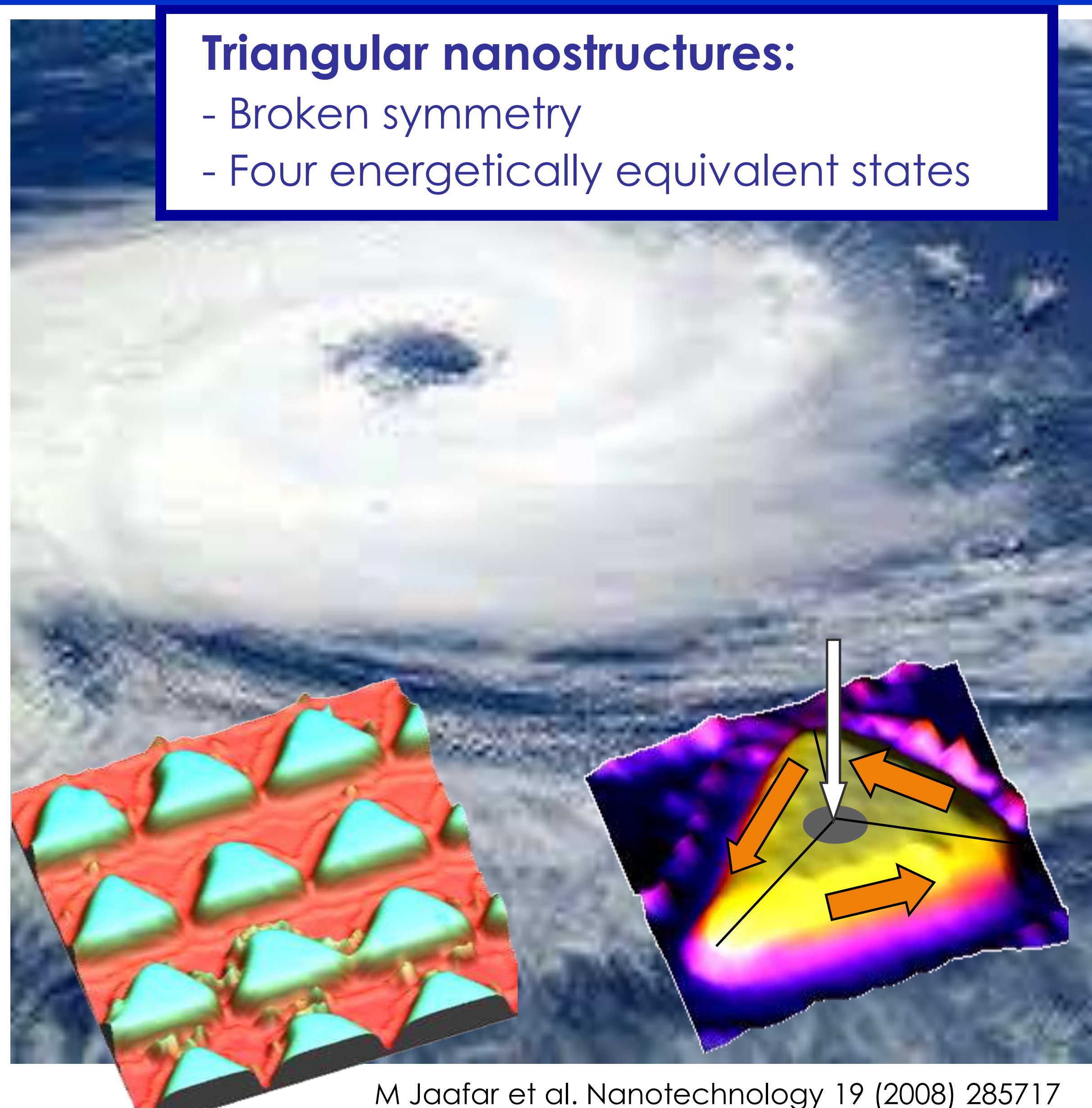

When the magnetic field decreases a vortex nucleates in the extreme of the dot, at zero field is centered and it is annihilated when increasing the field in the opposite direction.

# VFMFM. Ni (111) Nanostructures



**Triangular nanostructures:**

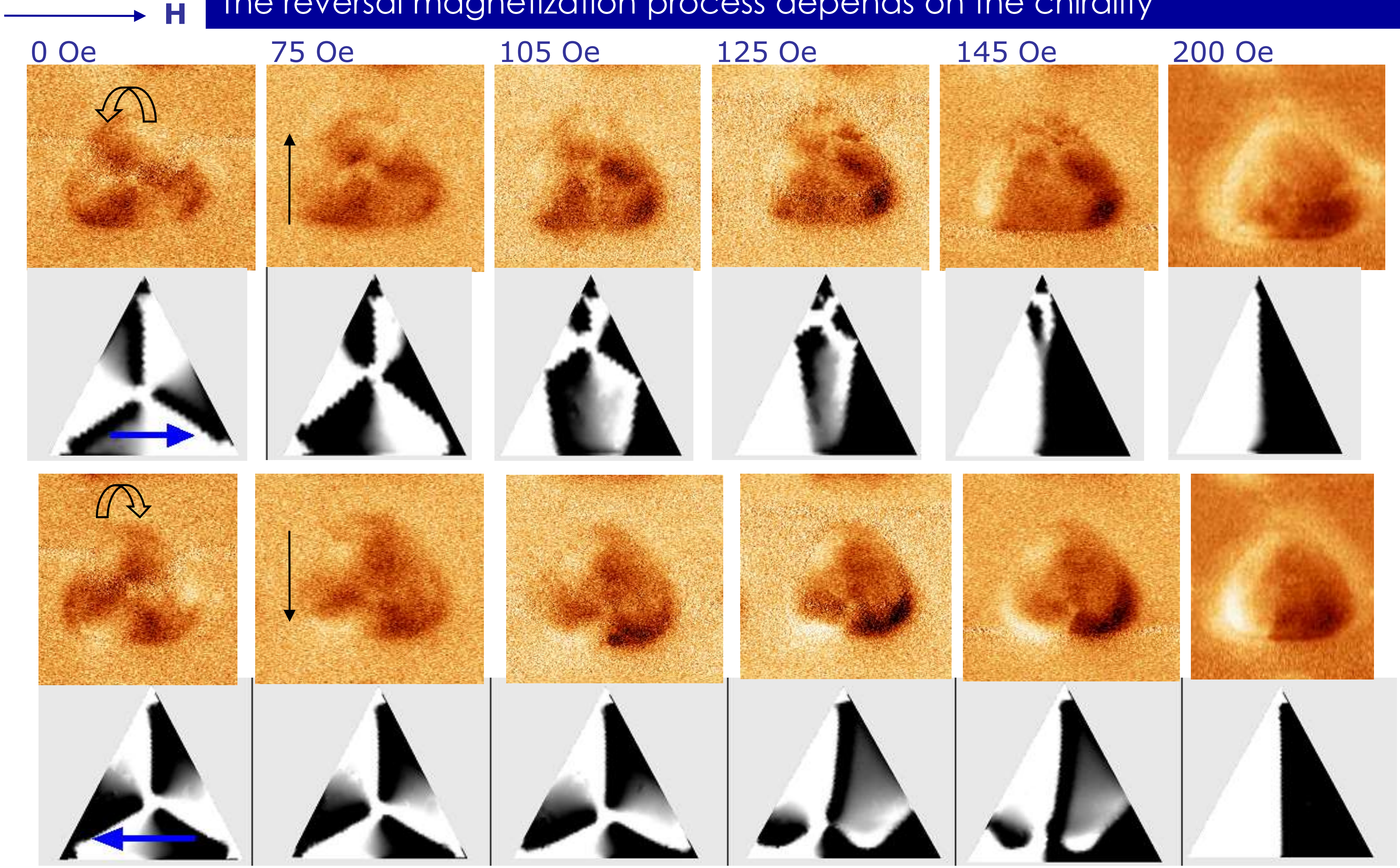
- Broken symmetry
- Four energetically equivalent states



M Jaafar et al. Nanotechnology 19 (2008) 285717

# VFMFM. Ni (111) Nanostructures

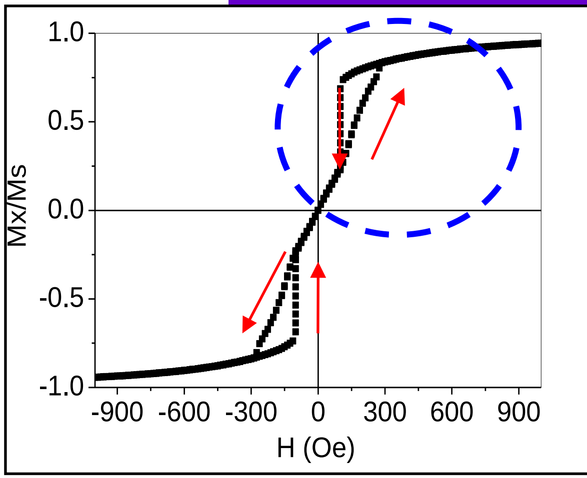
The reversal magnetization process depends on the chirality

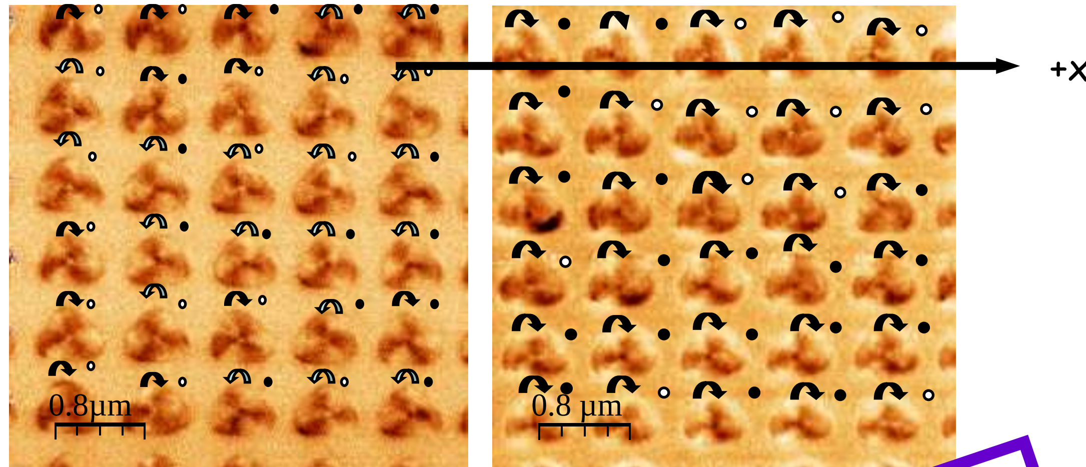



M Jaafar et al. Nanotechnology 19 (2008) 285717

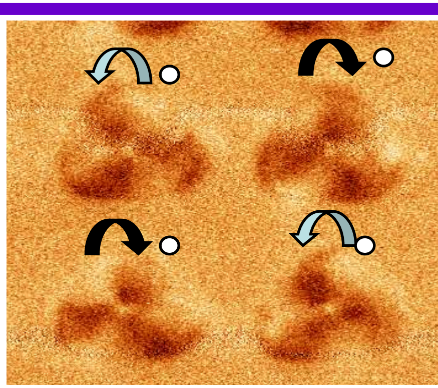
# VFMFM. Ni (111) Nanostructures

**Chirality** control by DC **in-plane** magnetic field

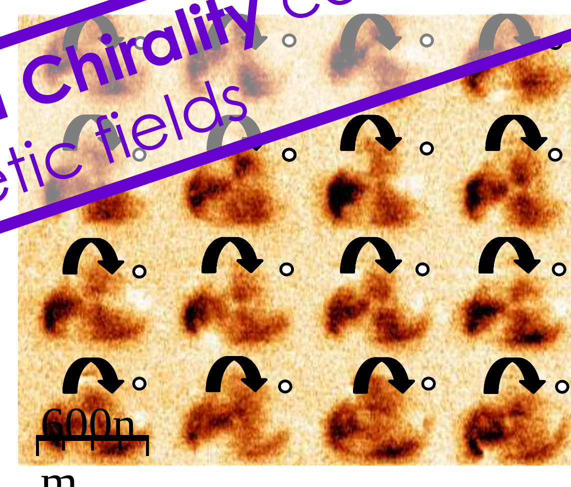



**Polarity** control by DC **out of plane** magnetic field



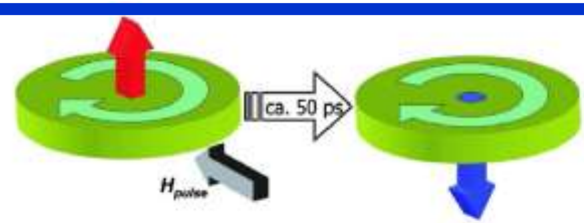
**Polarity and Chirality** control by DC magnetic fields

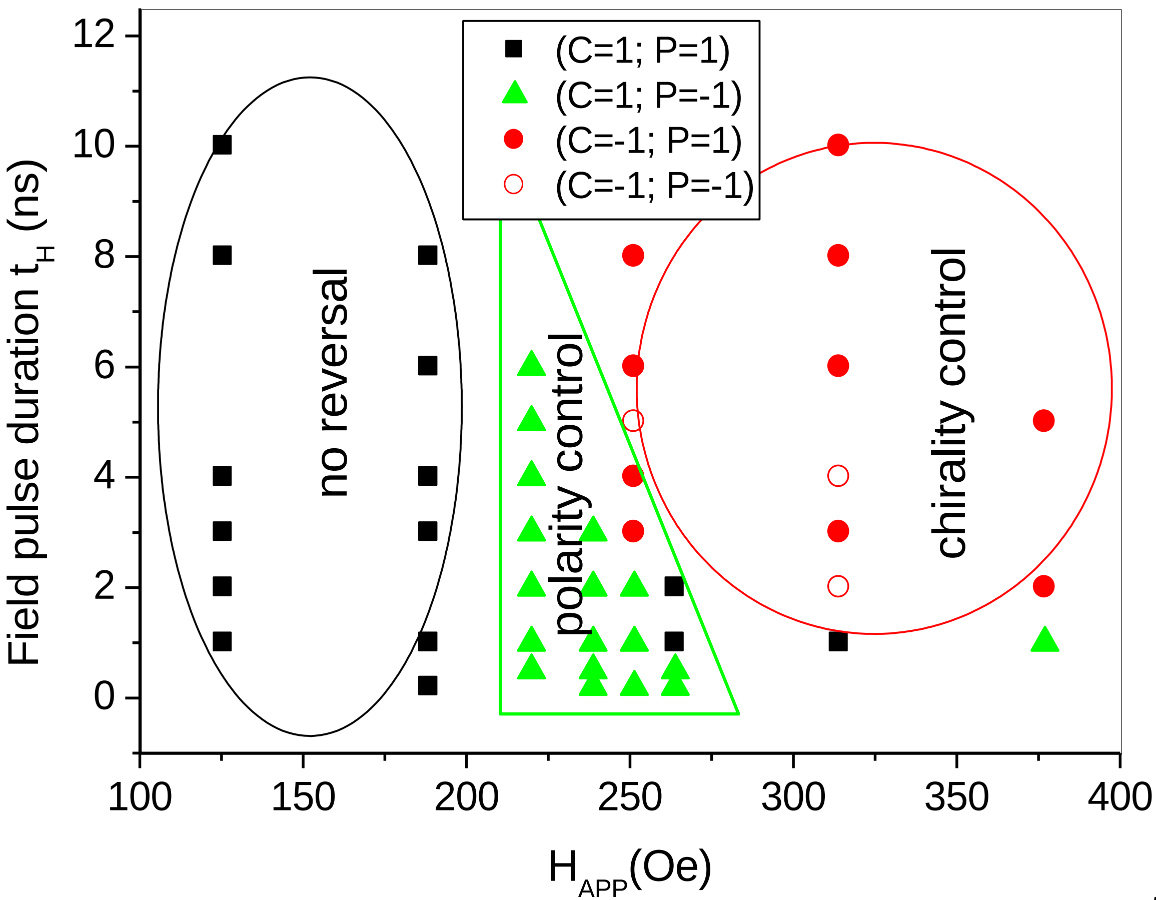


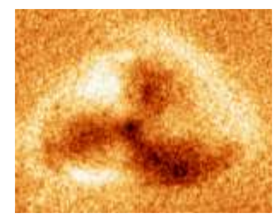

M. Jaafar et al. Phys. Rev. B, 81, 054439 (2010)

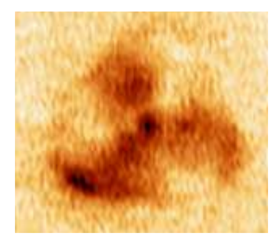
# VFMFM. Ni (111) Nanostructures

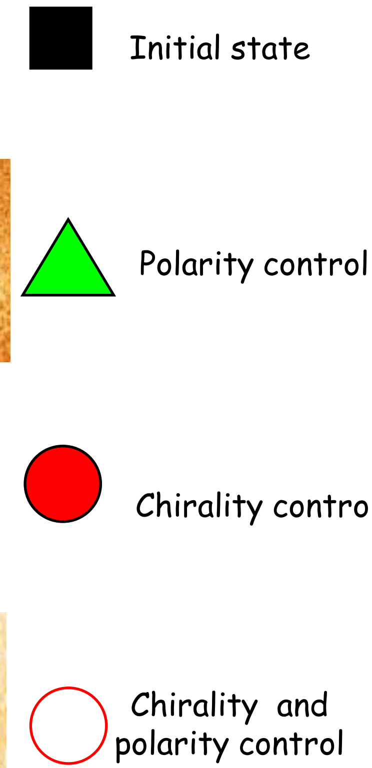
## Micromagnetic Simulations, Phase Diagram. *O. Fesenko ICMM-CSIC*








M. Jaafar et al. Phys. Rev. B, 81, 054439 (2010)

# Outline:

1. Scanning Probe Microscopies.
2. Fundaments of MFM. Measuring under standard conditions
3. Improving the hardware:
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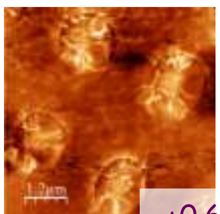
   c. Dissipation
5. Conclusions

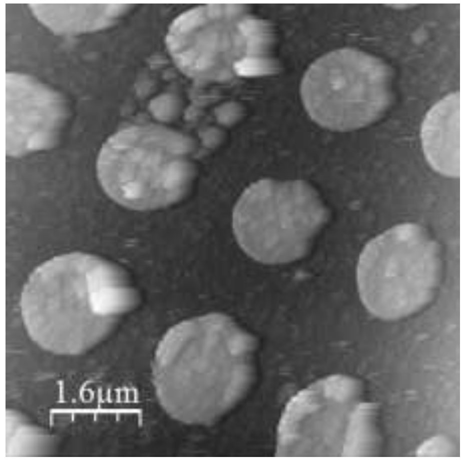
# Variable Temperature MFM: copper shield

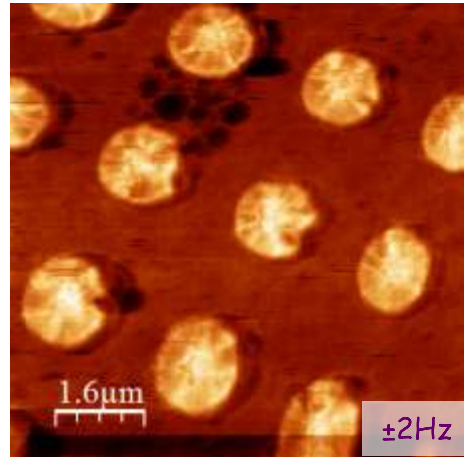
## Shielding the AFM with a cold trap screen to avoid water condensation.

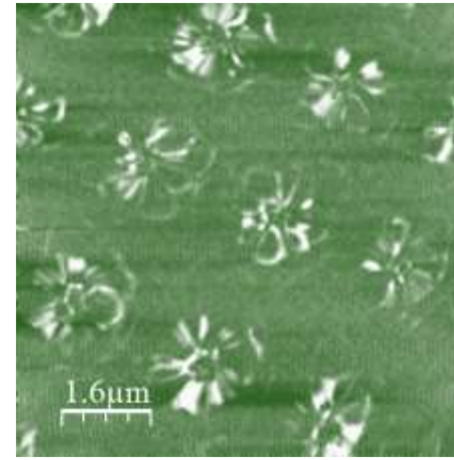
25°C



±0.6Hz



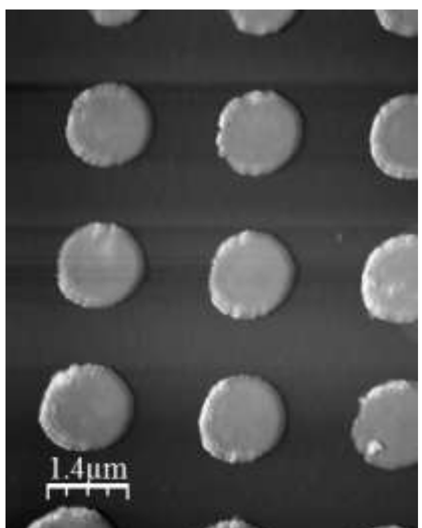



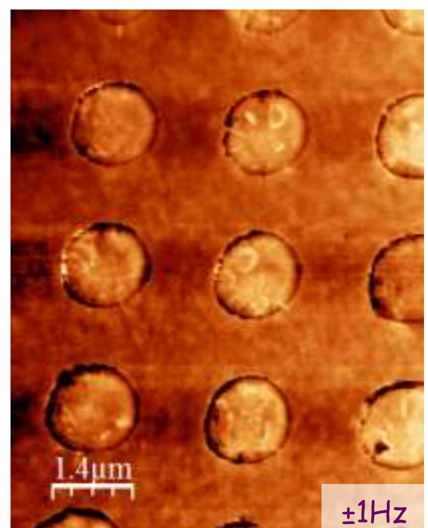

topo

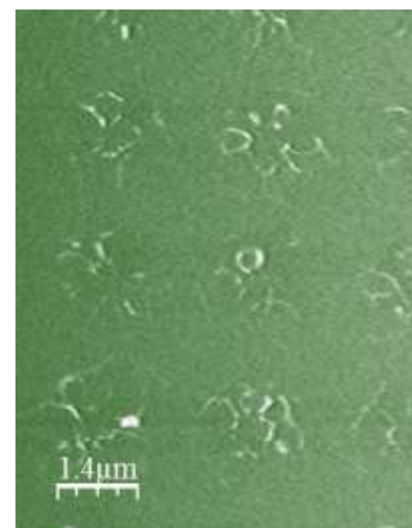
Δf (retrace)

ΔVosc (retrace)

-60°C



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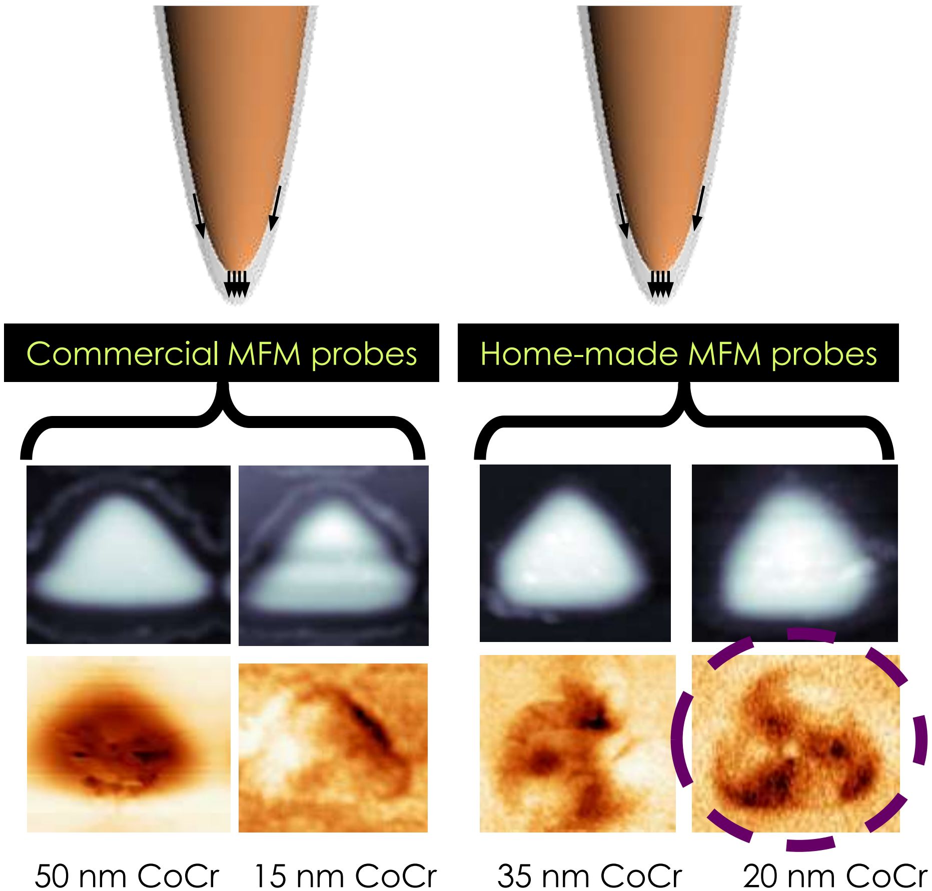
# Importance of the tips.

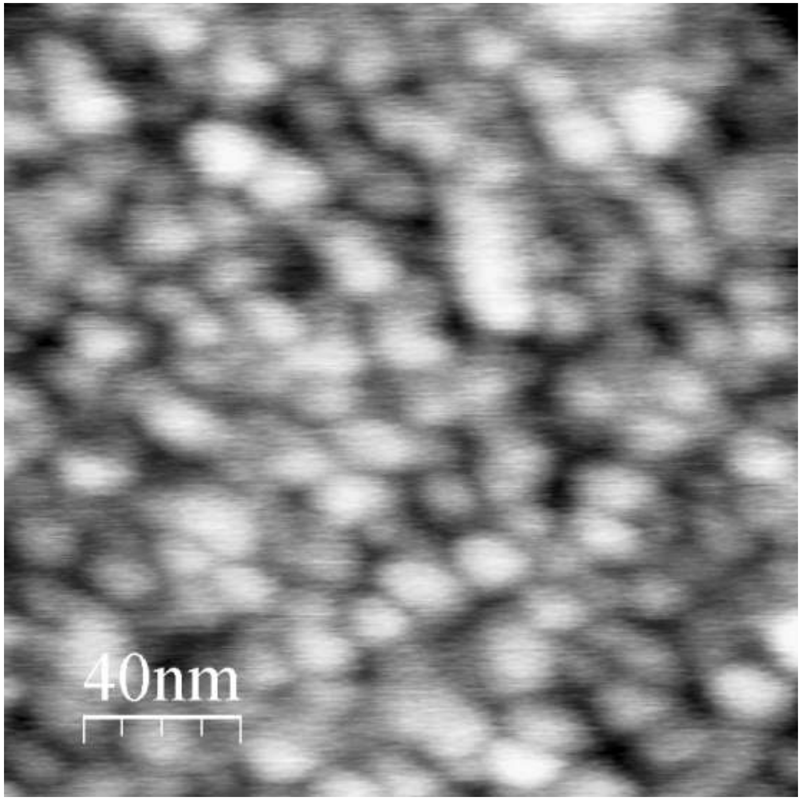
Home-made MFM probes by coating the commercial tips with a magnetic layer.



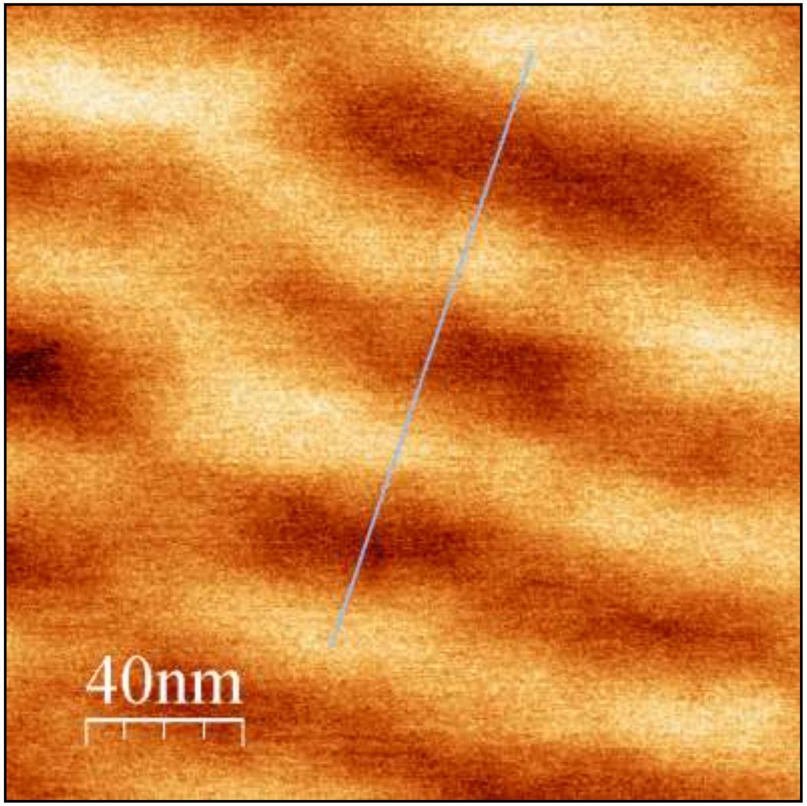

**Ni triangules, side 500nm**

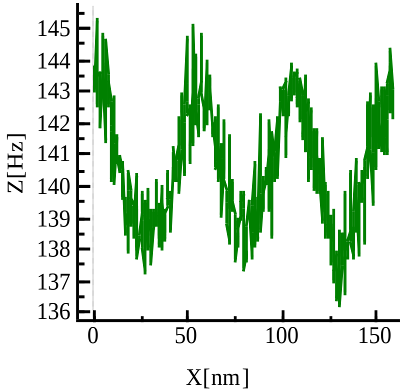
M. Jaafar , A. Asenjo, M. Vázquez, *IEEE Nano* **7** (2008) 245

# High resolution / low sensitivity



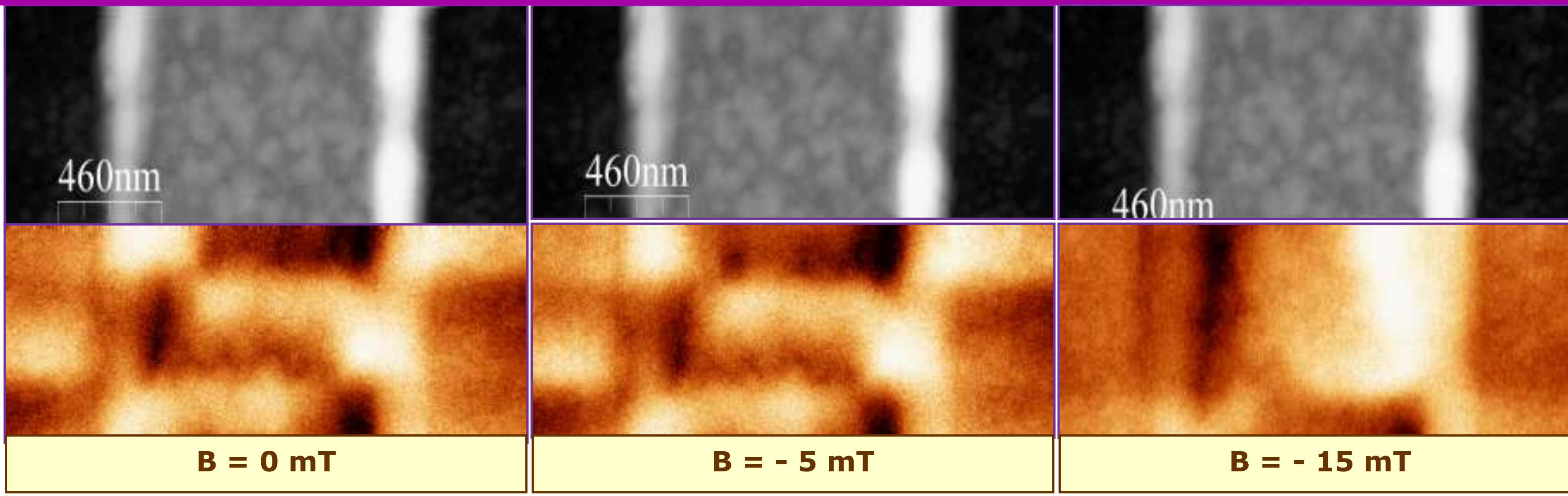

Homemade Tip 20 nm Co

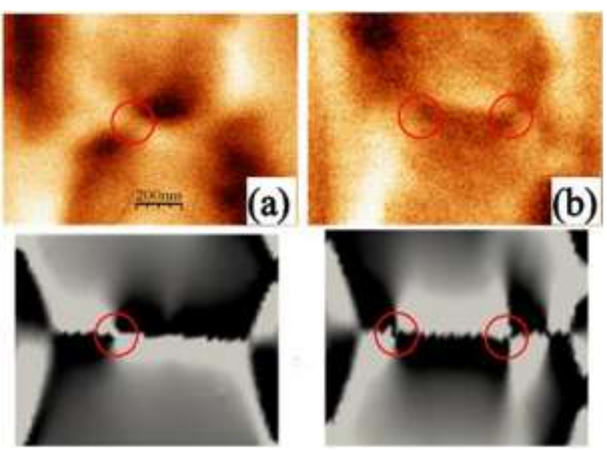


# Special MFM probes for soft materials

## Vortex-Domain Walls on Co stripes




High resolution MFM images (up) and results of micromagnetic simulation (down) for vortex (a) and double vortex (b) DWs in nanostripe

Y. P. Ivanov, Ó. Iglesias-Freire, O. Chubykalo-Fesenko and A. Asenjo, PRB, B 87, 184410 (2013)

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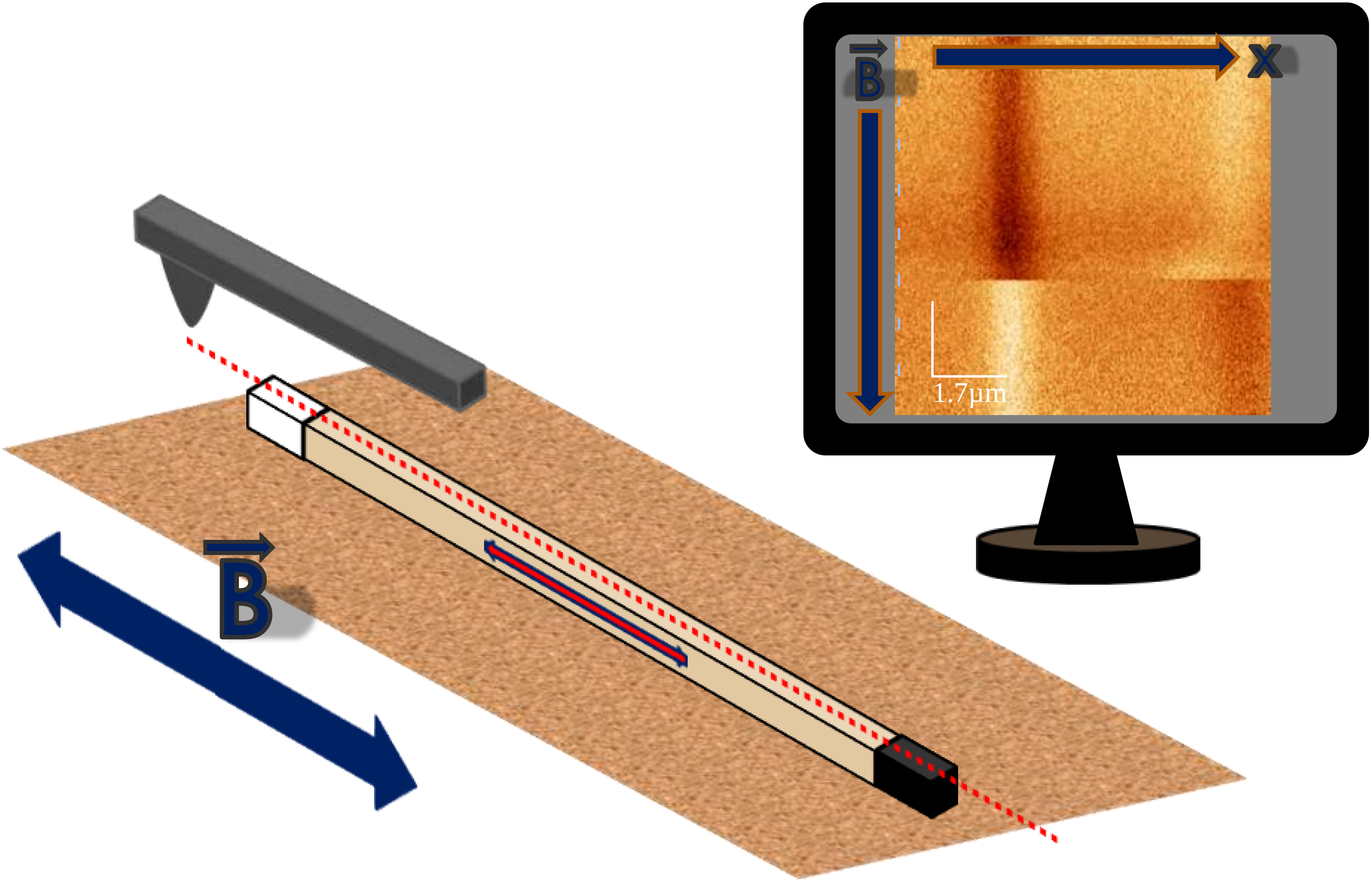
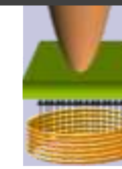
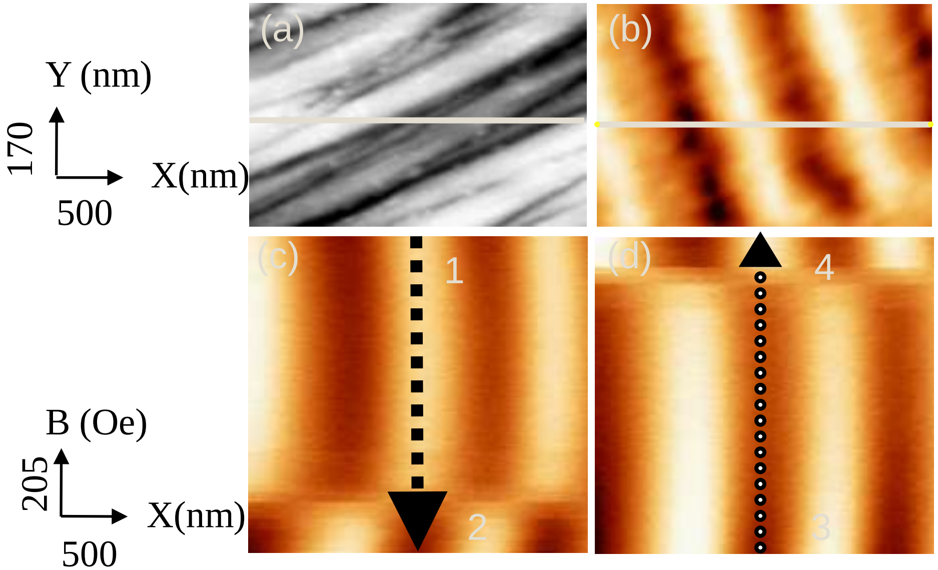
# VFMFM: 3D mode images.



# *In situ* hysteresis loops of MFM probes



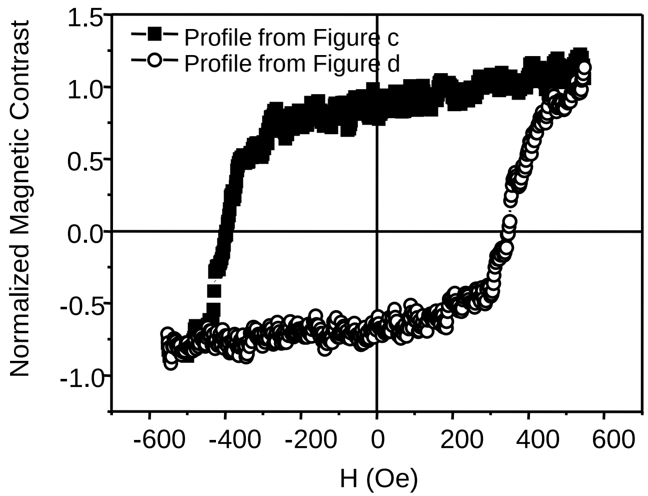
New method to characterize the tips, faster and more precise

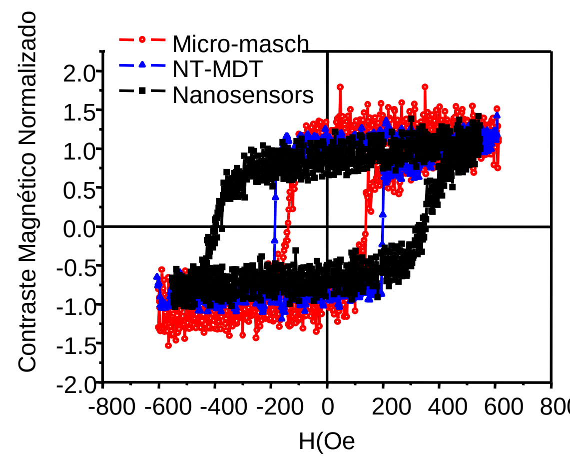



$$\frac{\partial F}{\partial z} \propto m_{tip} \times m_{sample}$$

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



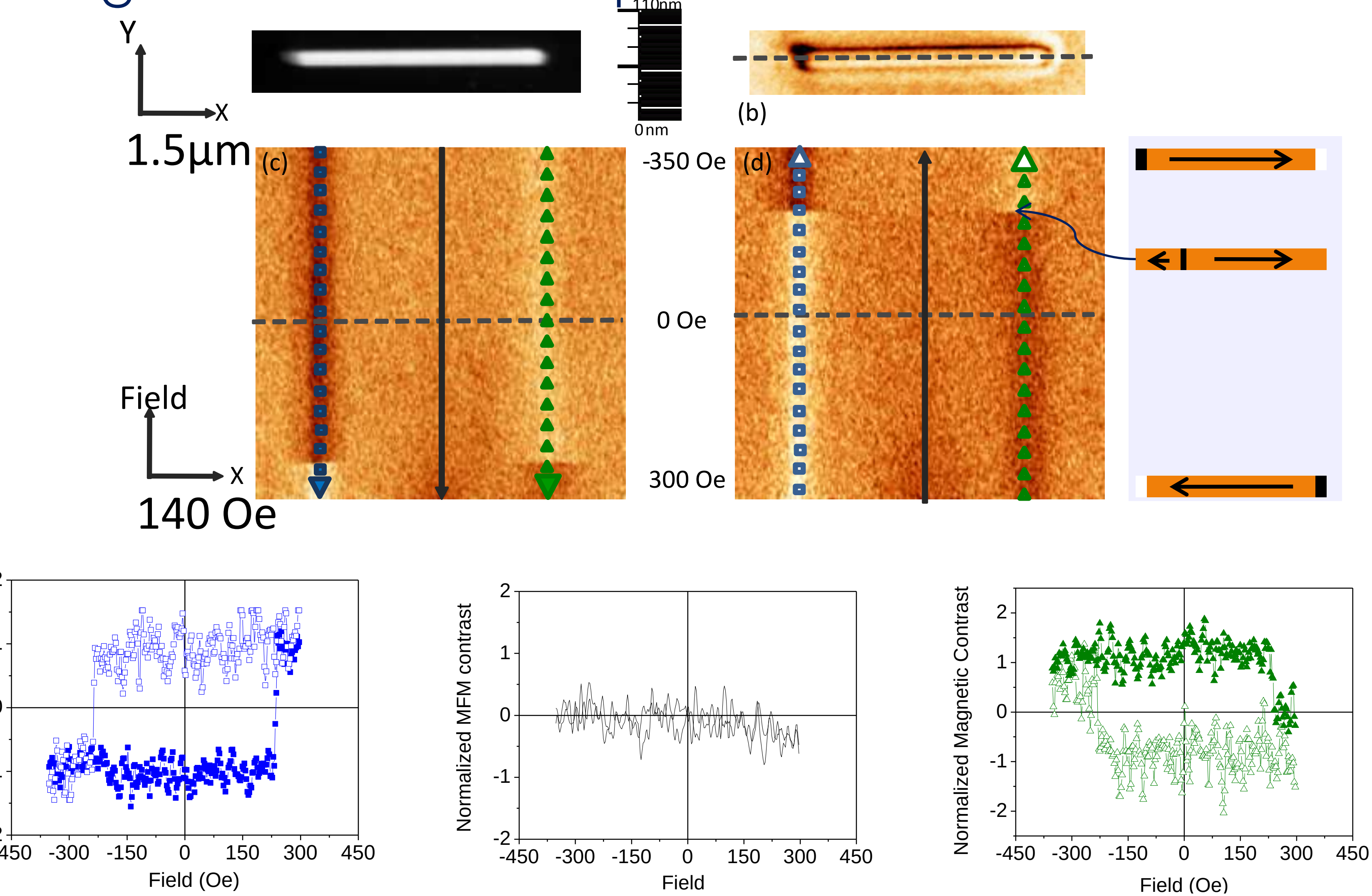




*M. Jaafar, J. Gómez-Herrero, A. Gil, P. Ares, M. Vázquez and A. Asenjo, Ultramicroscopy 109 (2009) 693*

# 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

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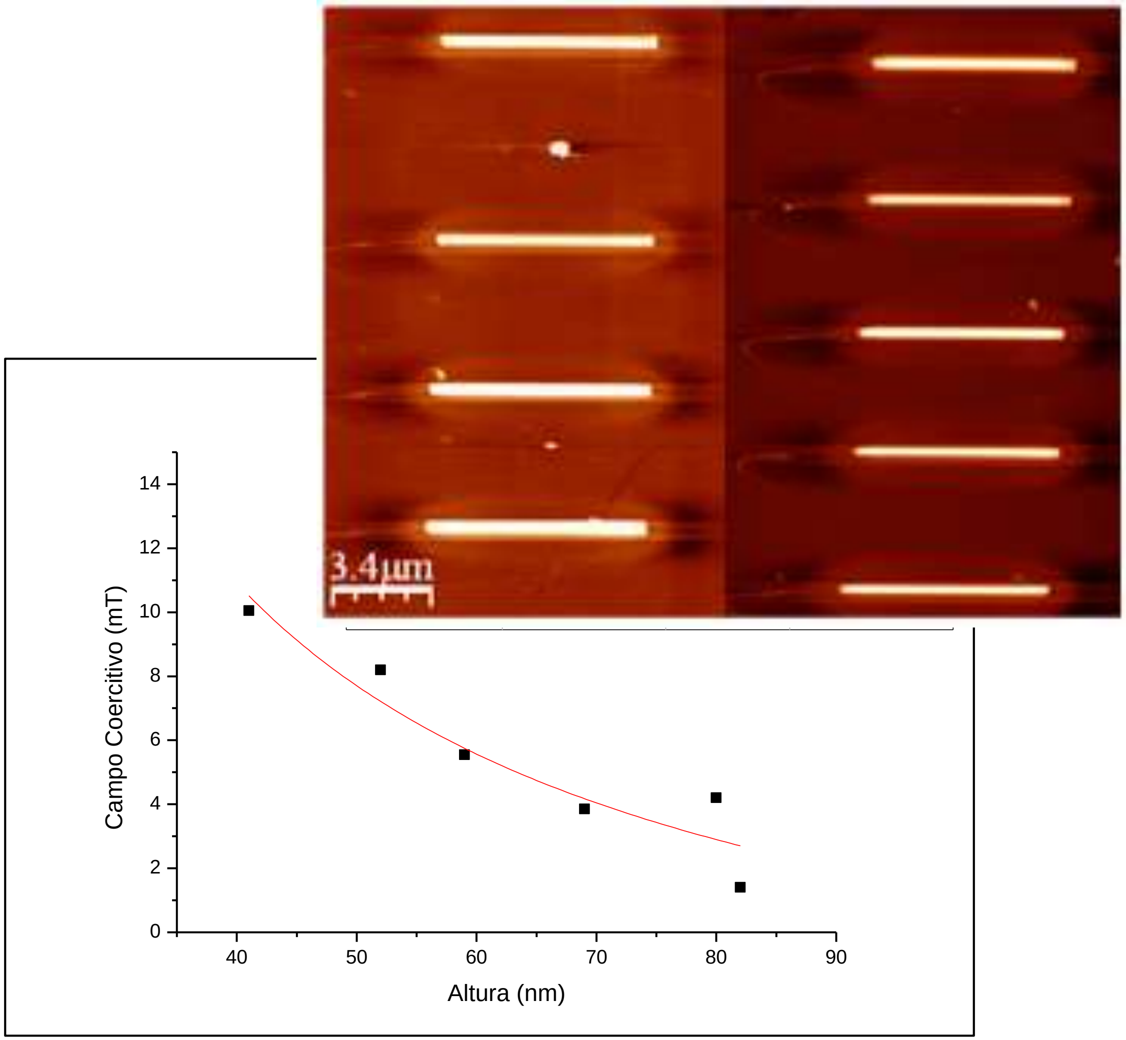
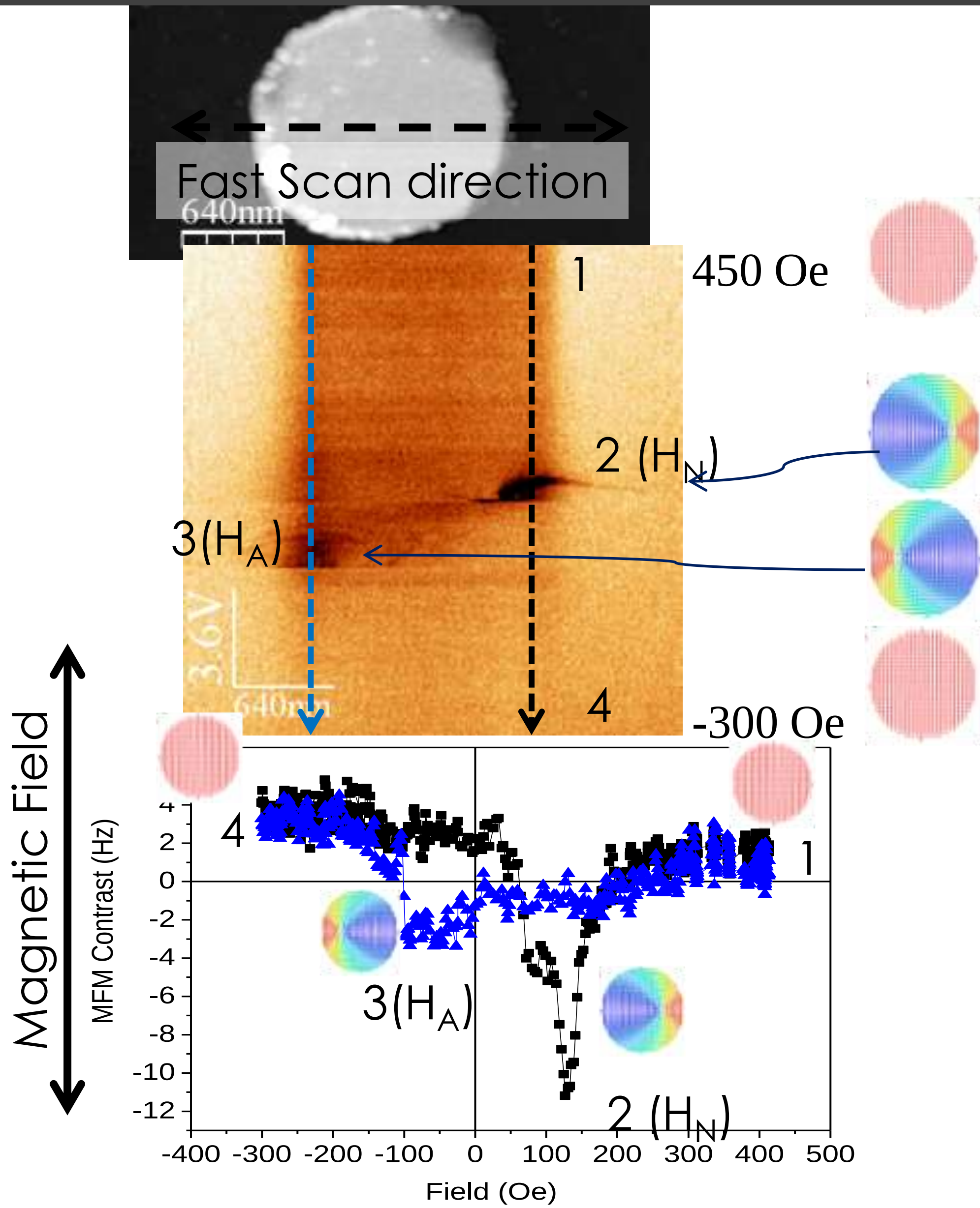
## Single domain nanostripes

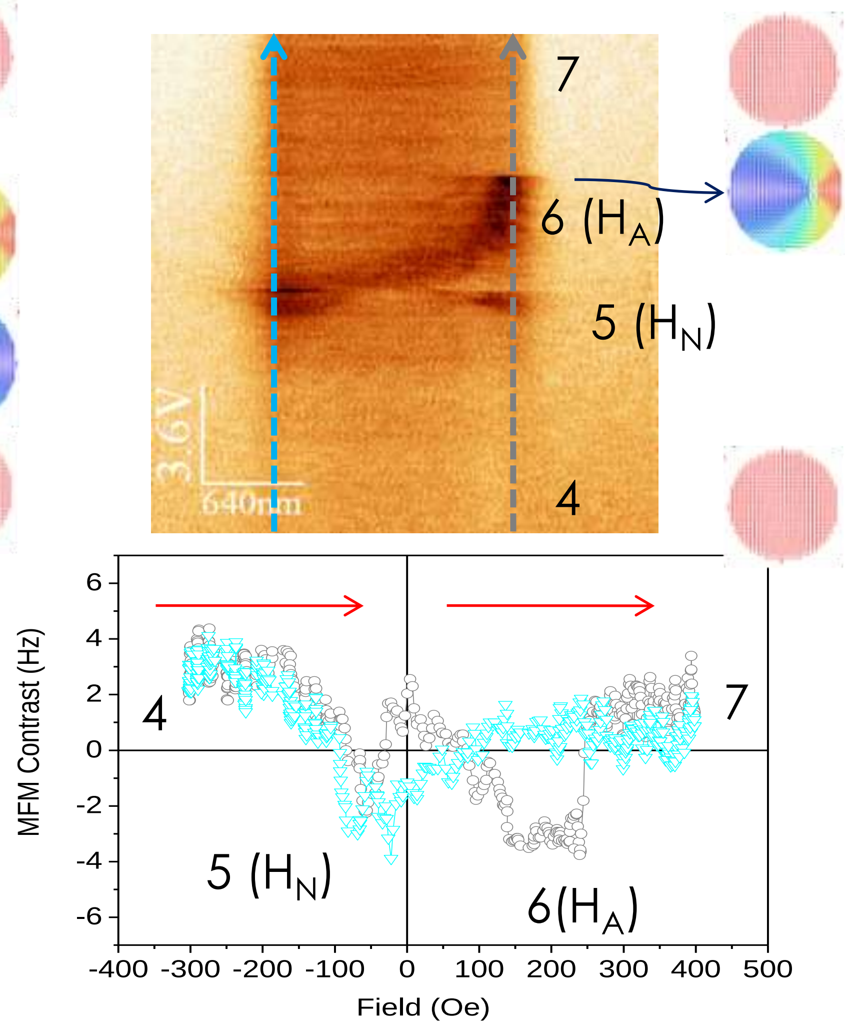



M. Jaafar, O. Iglesias-Freire, L. Serrano-Ramón, M. R. Ibarra, J. M. de Teresa and A. Asenjo, BJ Nano 2 (2011) 552

# *In situ* hysteresis loops of a single Py dot




## Double vortex?



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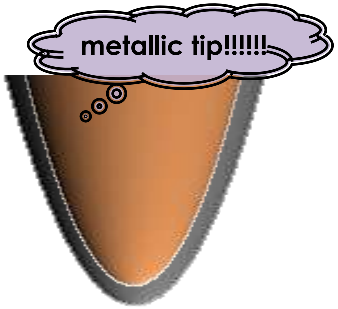
# KPFM and MFM combination

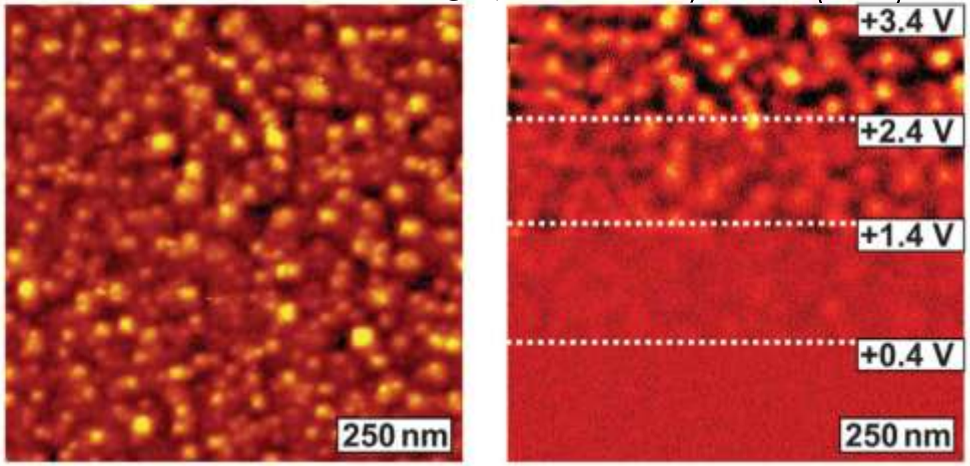
## Electrostatic Forces

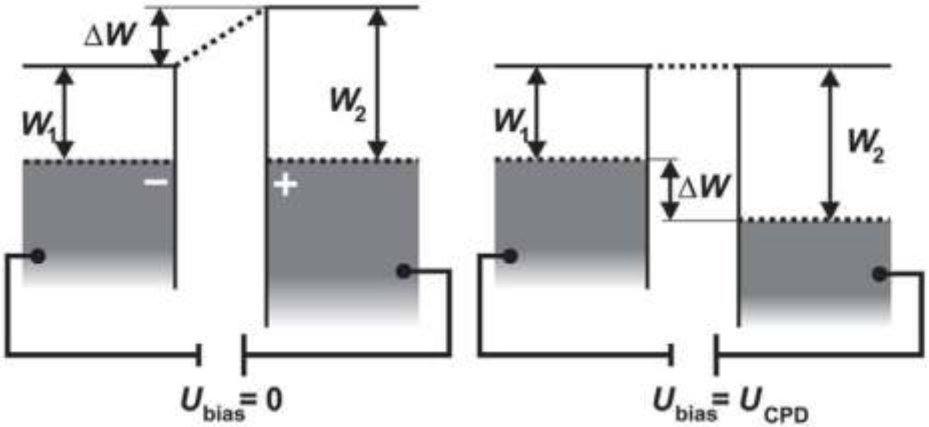



Different materials, different <u>work functions</u>, $W_1 \neq W_2$

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

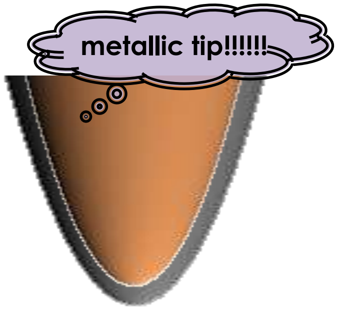
A. Schwarz and R. Wiesendanger, Nanotoday, 3, 1-2 (2008) 28



# KPFM and MFM combination

## Kelvin Probe Force Microscopy




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

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

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

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

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

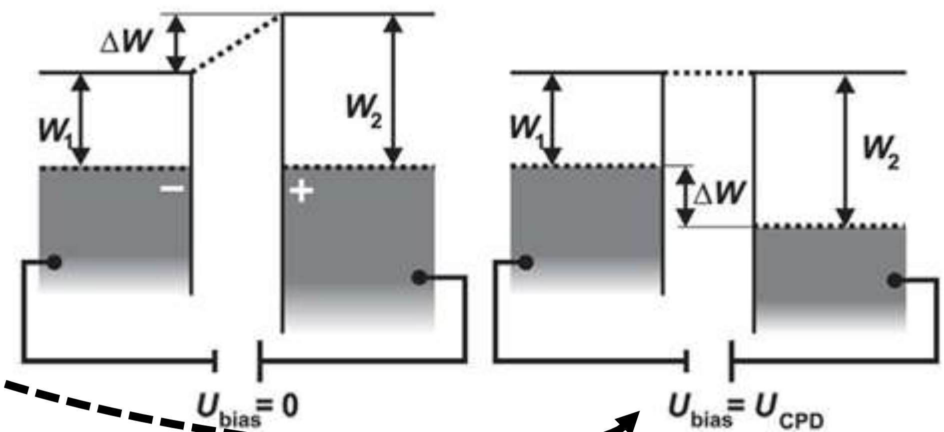
| Mode | Frequency |
|---|---|
| KPFM (air) | 7 kHz |
| Resonant Frequency | 70 kHz |
| KFPM (HV, 2nd mode) | 400 kHz |

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

Different materials, differ work functions, $W_1 \neq W_2$

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

We need to employ **Kelvin Probe Force Microscopy.**

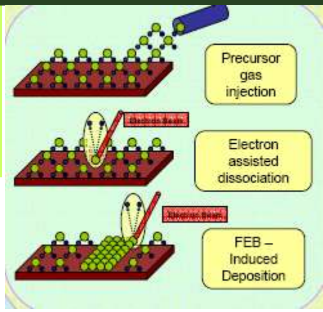



M. Jaafar, O. Iglesias-Freire, L. Serrano-Ramón, M. R. Ibarra, J. M. De Teresa and A. Asenjo, BJNano., 2, 552-560 (2011)
D. Martínez – Martín, M. Jaafar, J. Gómez – Herrero, R. Pérez and A. Asenjo, Phys. Rev. Lett. 105, 257203 (2010)

# KPFM and MFM combination

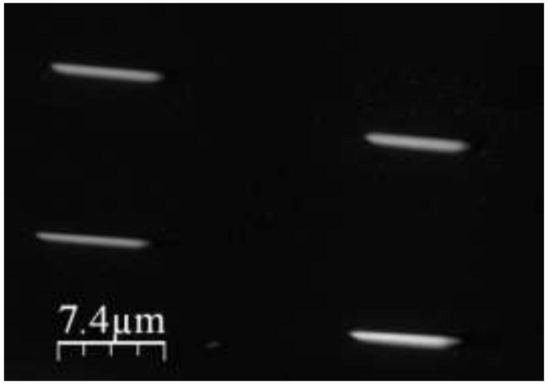
## Co nanostripes/$SiO_2$ prepared by Focused Electron Beam

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.

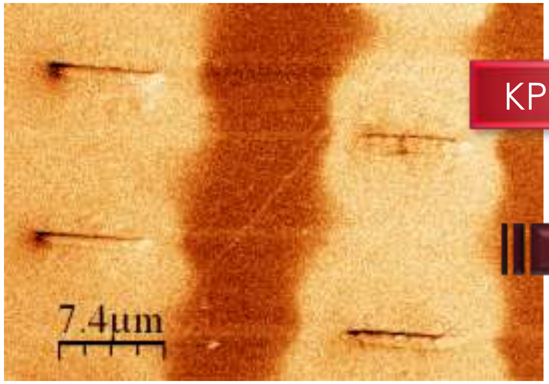



Heterogeneous electrostatic interaction between tip and sample that can be interpreted as magnetic interaction

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



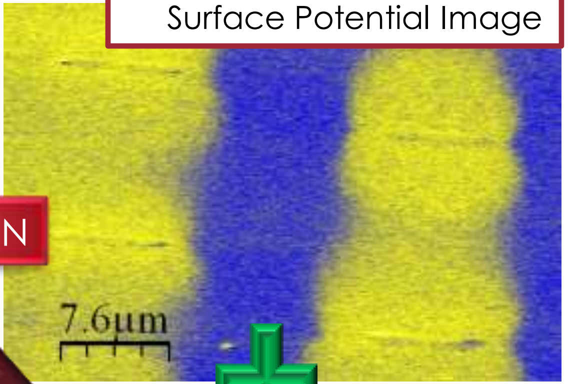

Topography

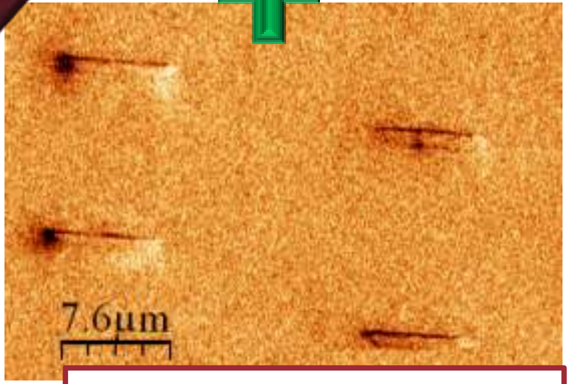



Frequency Shift (MFM probe)

KPFM ON

Surface Potential Image



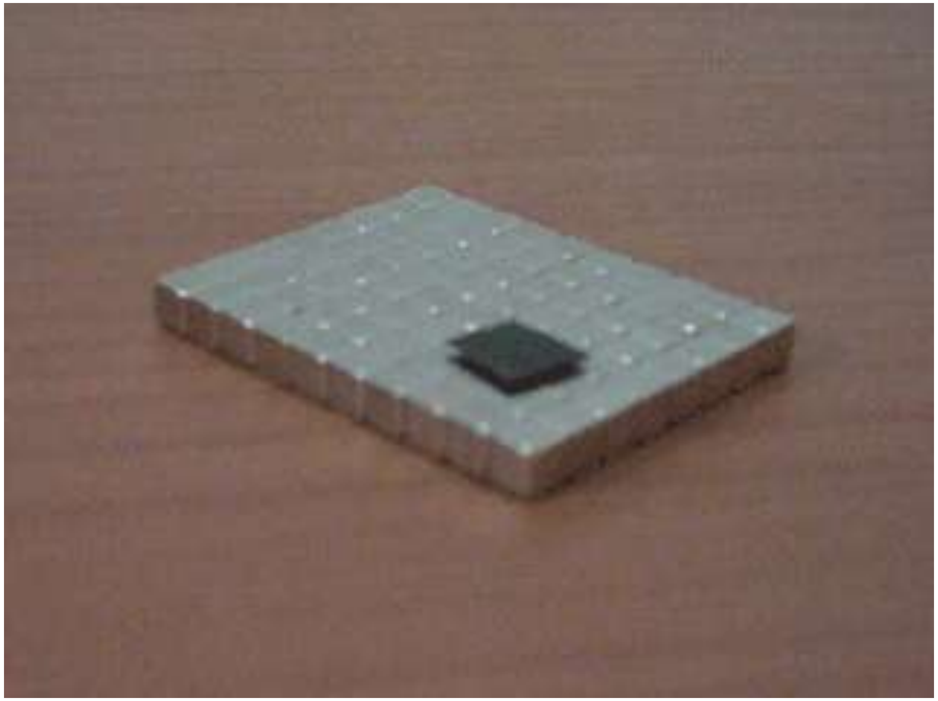

Frequency Shift (MFM image)

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

# However the graphite is …

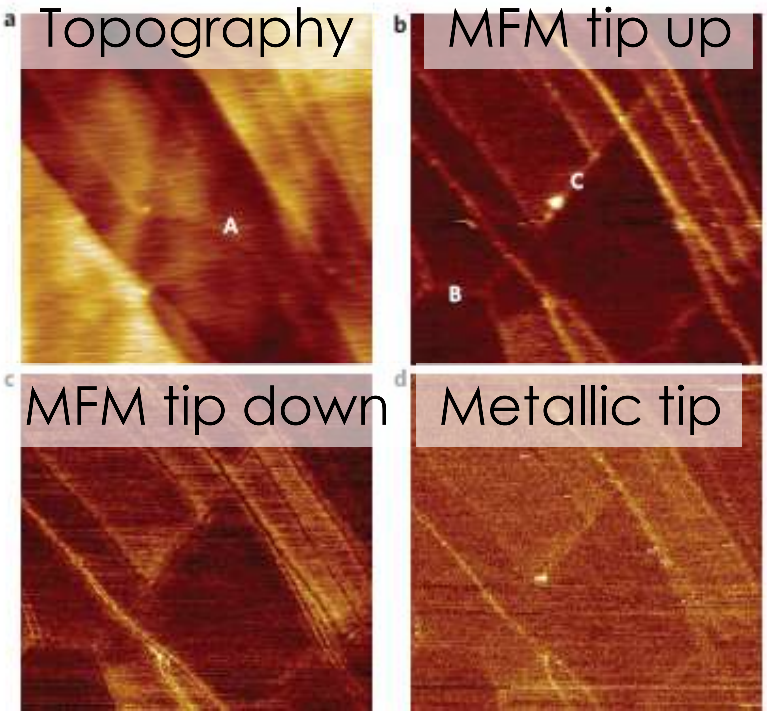
Macroscopically: **Diamagnetic material**

Graphite levitating on a magnet



**Controversial** topic, different results and interpretations

# MFM in Graphite



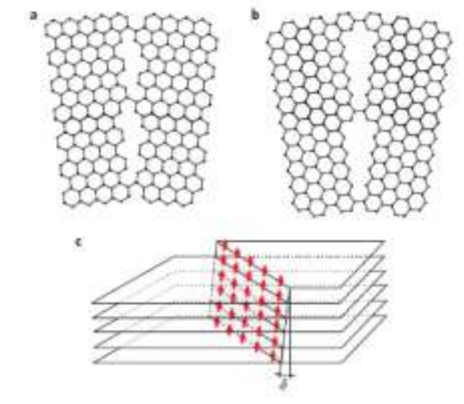
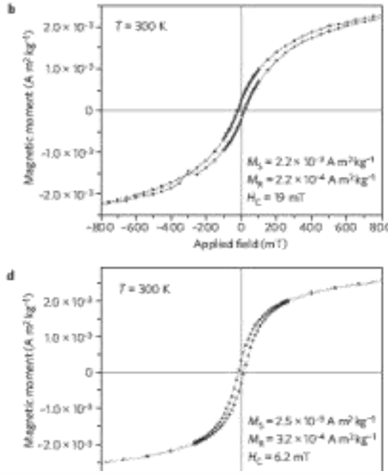




**Figure 4 | Schematic models of two basics shapes of grain boundaries in graphite. a,** Armchair direction with periodicity *D*. **b,** Zigzag direction with



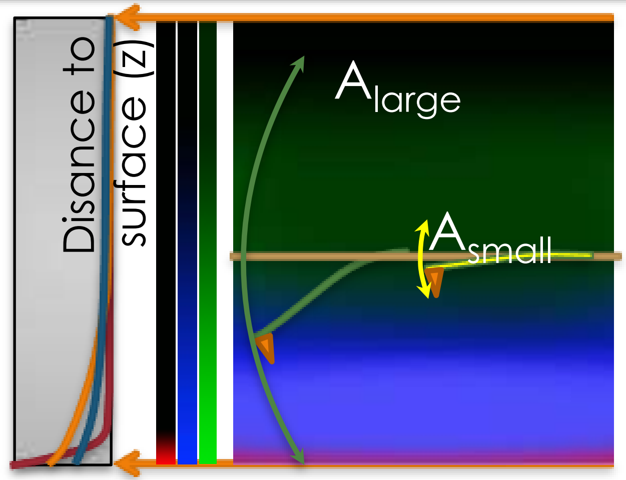

Tip: Nanosensors PPP- MFMR

Amplitude: more than 100nm!!!

Retrace: 50nm!!!

"Room-temperature ferromagnetism in **graphite** driven by two-dimensional networks of point defects"

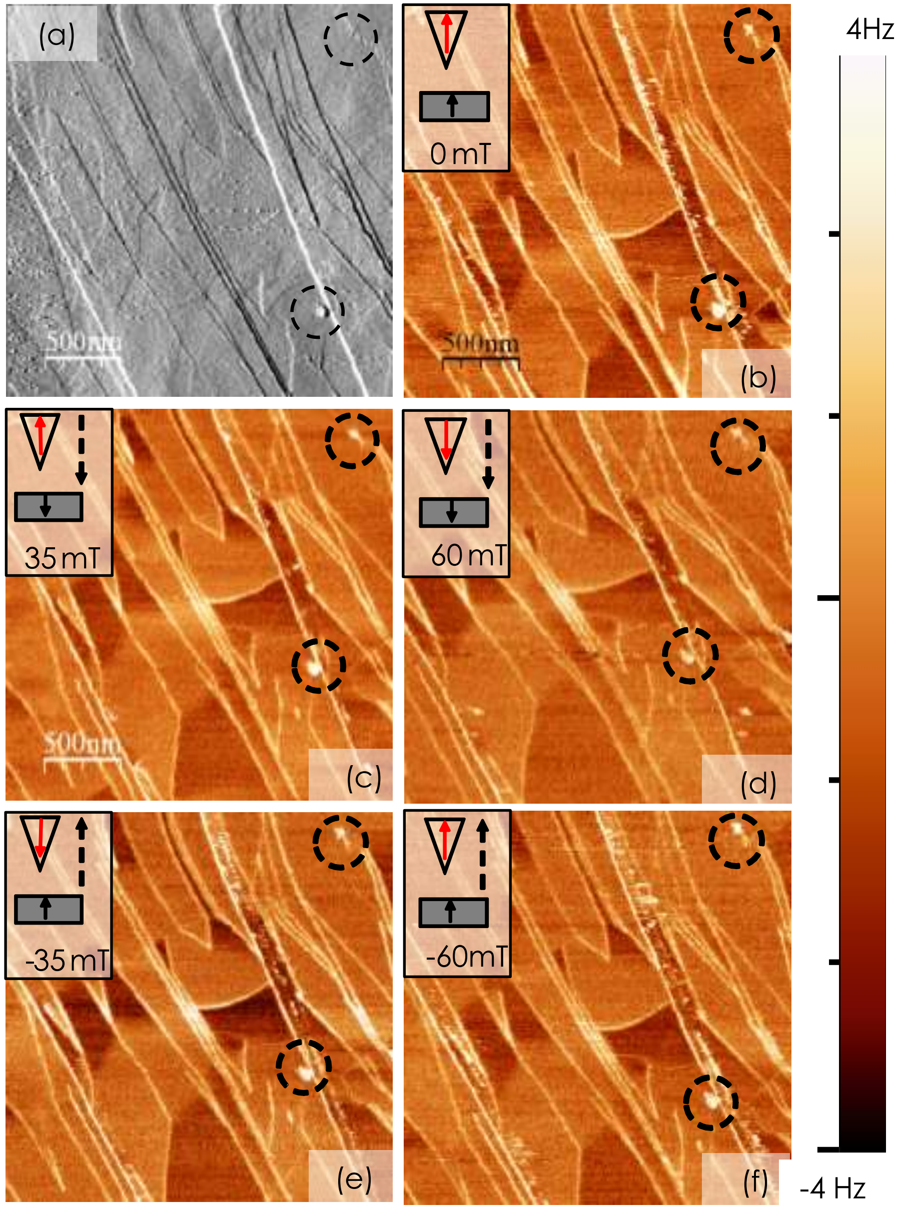
Cervenka et al. *Nature Physics* **5**, 840 (2009)



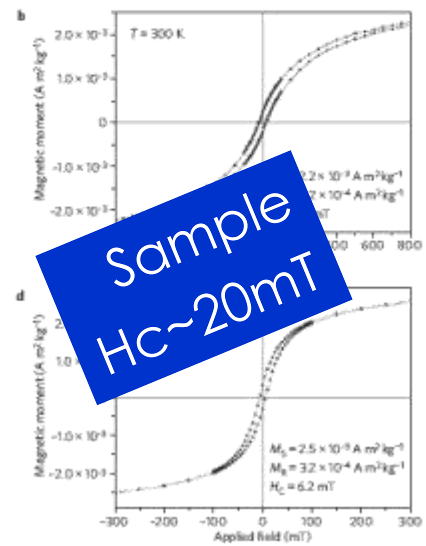

Z= 50 nm

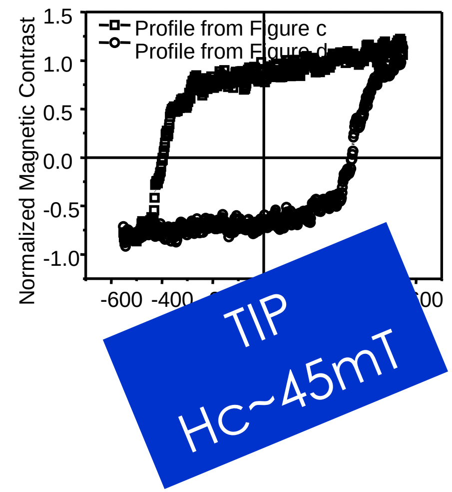
Ferromagnetic domains located in the grain boundaries

# Variable Field MFM measurements




**In situ variation of the magnetic field**




**Nothing changes**

Amplitude, 7nm

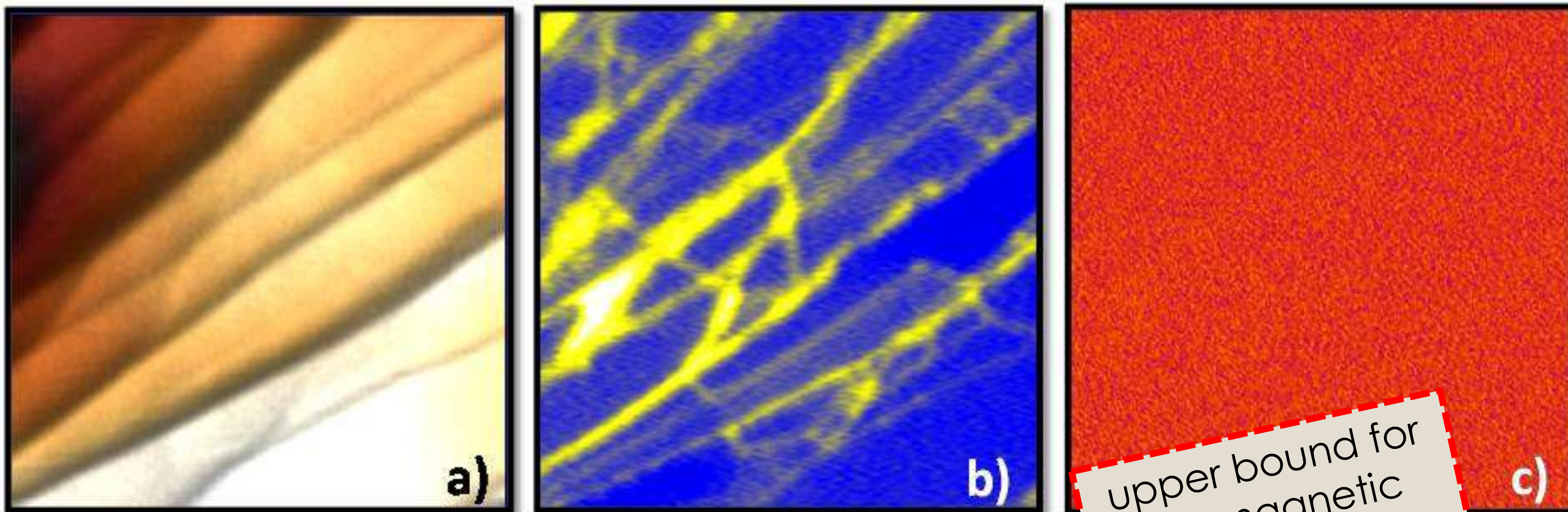
Retrace , 50nm

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

# KPFM and MFM in Graphite

KPFM ON

Topography | KPFM image, 1st scan | Frequency shift at 50nm




upper bound for the magnetic force gradient

Amplitude, 4 nm

**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)

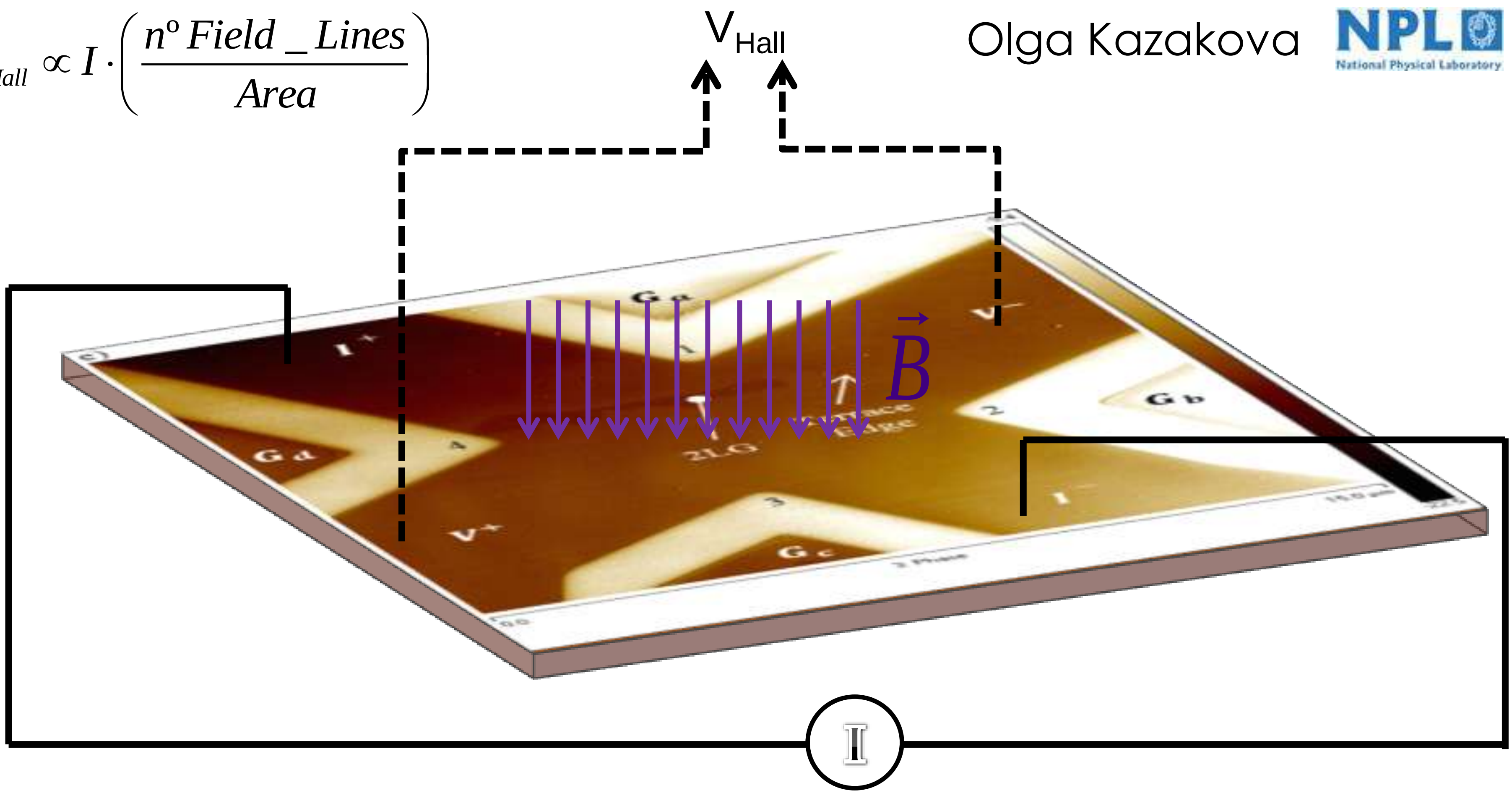
# Hall effect graphene-based sensor

**Device fabricated out of epitaxially grown graphene on SiC (0001) substrate**

$$V_{Hall} \propto I \cdot \left( \frac{n^{o}\, Field\_Lines}{Area} \right)$$

Olga Kazakova

NPL National Physical Laboratory



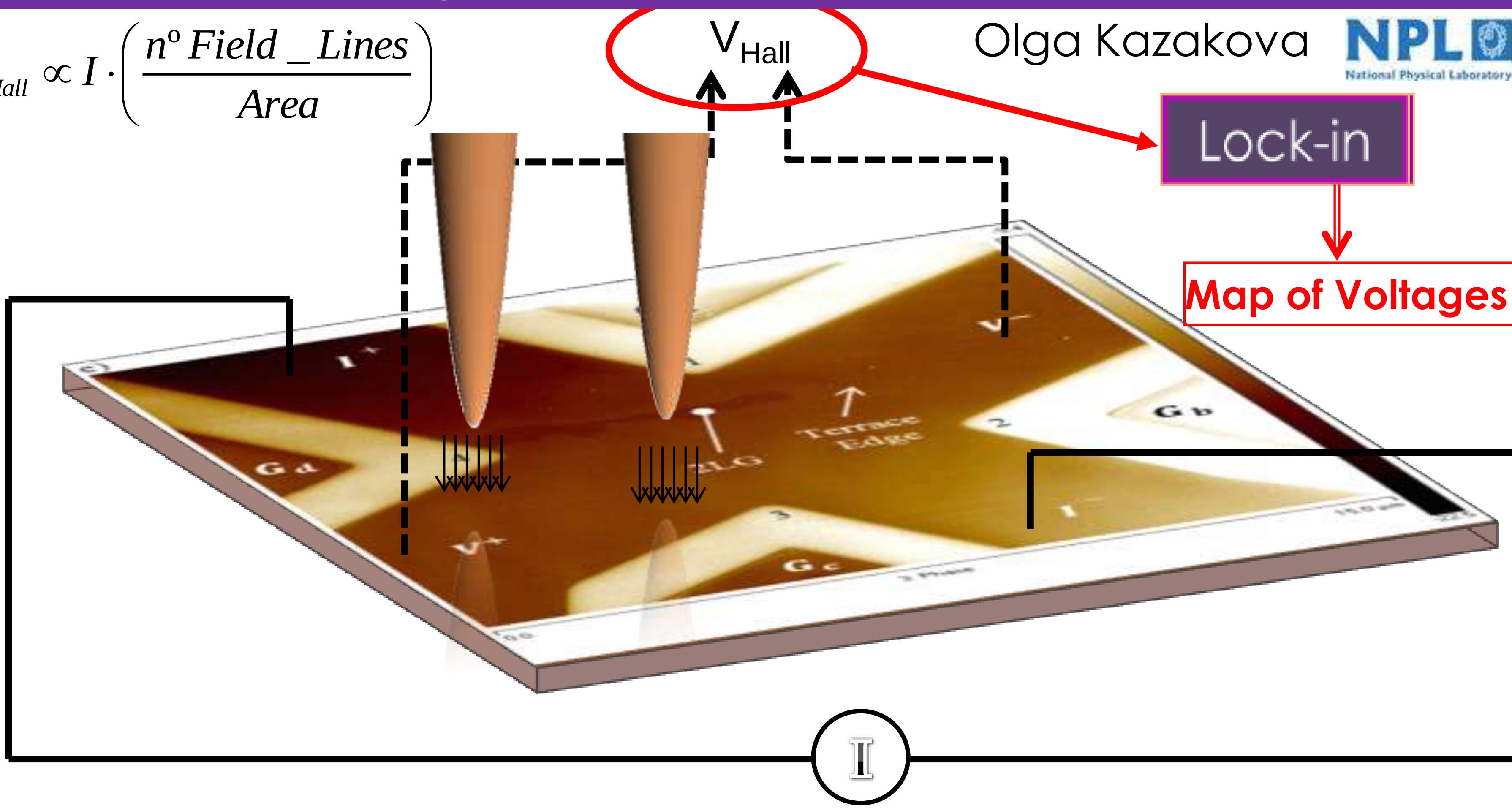
# Hall effect graphene-based sensor

**Device fabricated out of epitaxially grown graphene on SiC (0001) substrate**

The objective:
To evaluate the magnetic field of MFM tips

$$V_{Hall} \propto I \cdot \left( \frac{n^{o}\, Field\_Lines}{Area} \right)$$

Olga Kazakova



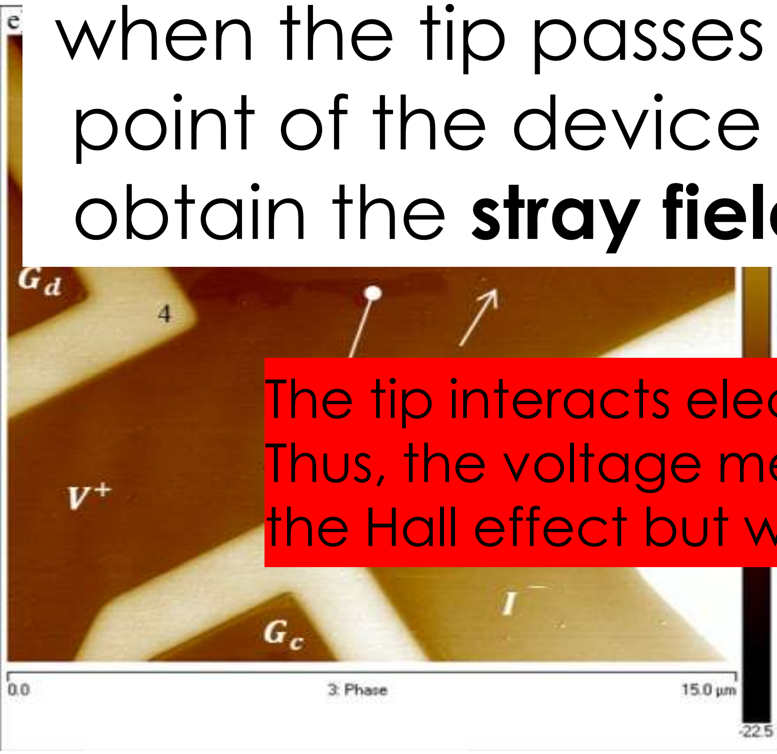
# Hall effect graphene-based sensor

**Device fabricated out of epitaxially grown graphene on SiC (0001) substrate**

The objective:
To evaluate the magnetic field of MFM tips

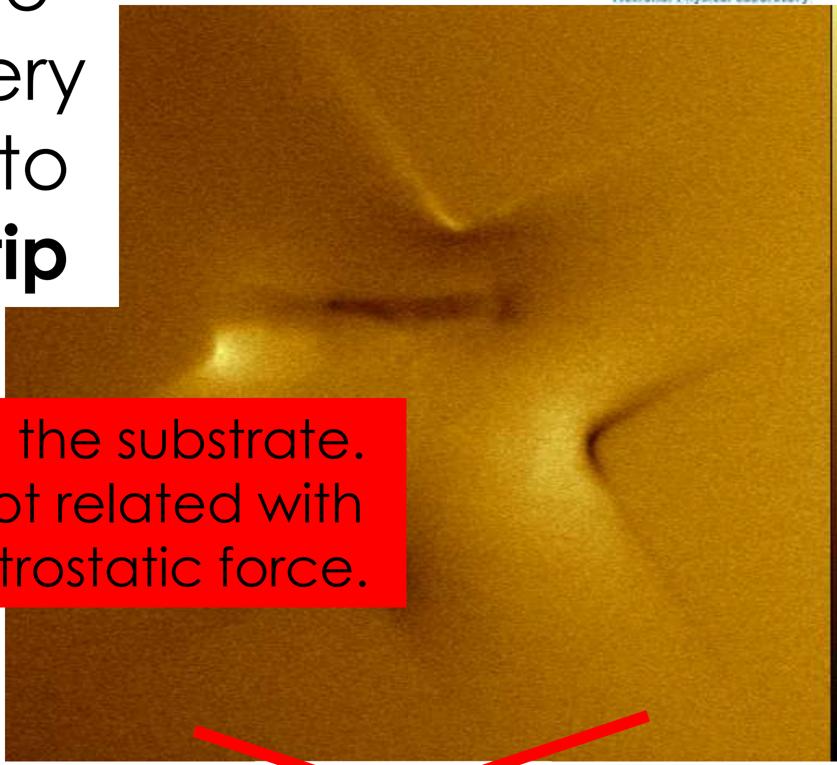
Olga Kazakova

We measure the hall voltage when the tip passes over every point of the device in order to obtain the **stray field of the tip**




The tip interacts electrically with the substrate. Thus, the voltage measured is not related with the Hall effect but with the electrostatic force.

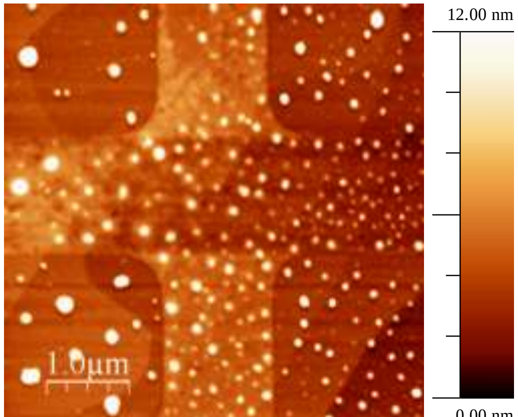
EFM image



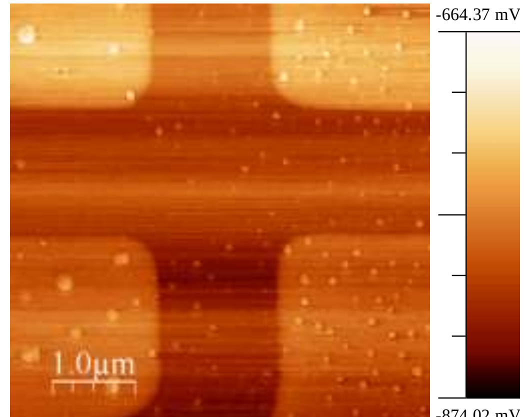
~~$V_{hall}$ map~~

# Hall effect graphene-based sensor

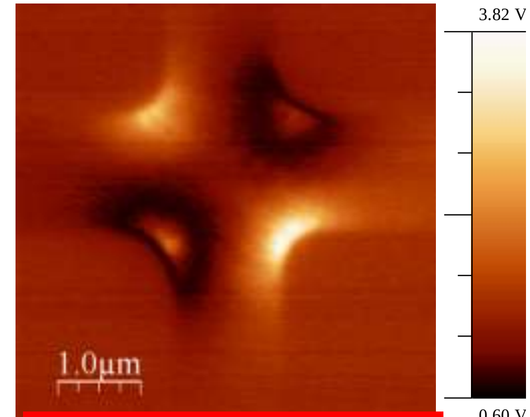
KPFM is switched off




Topography



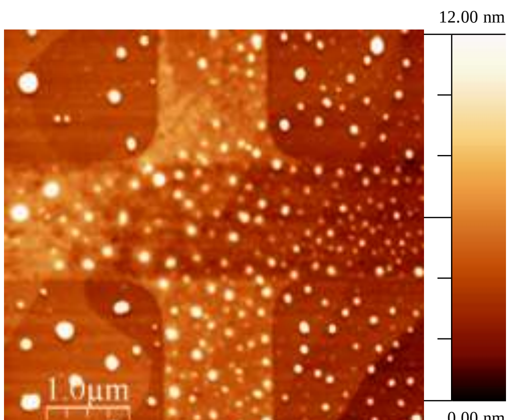

Electrostatic signal



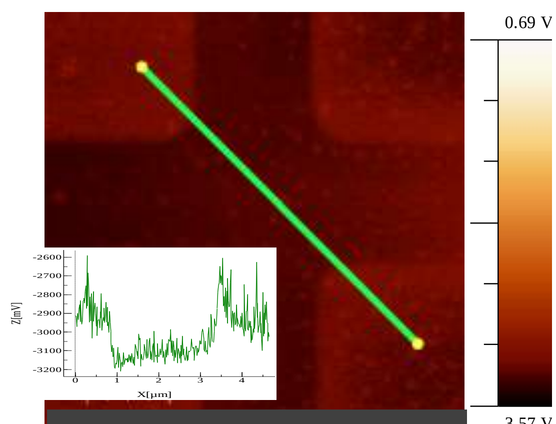

Hall effect signal
Lock-in amplitude

Tip magnetized out of plane. $I_{bias}$ = **+50 µA**

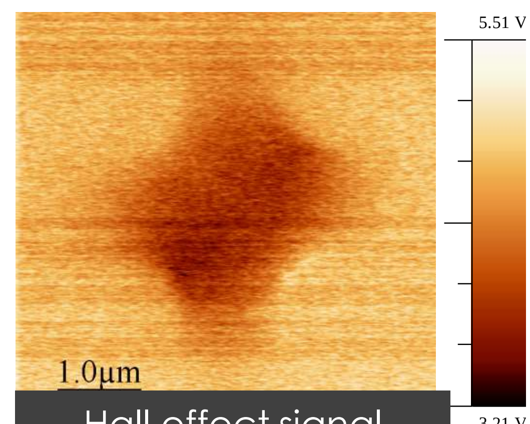
KPFM is switched on




Topography




Kelvin

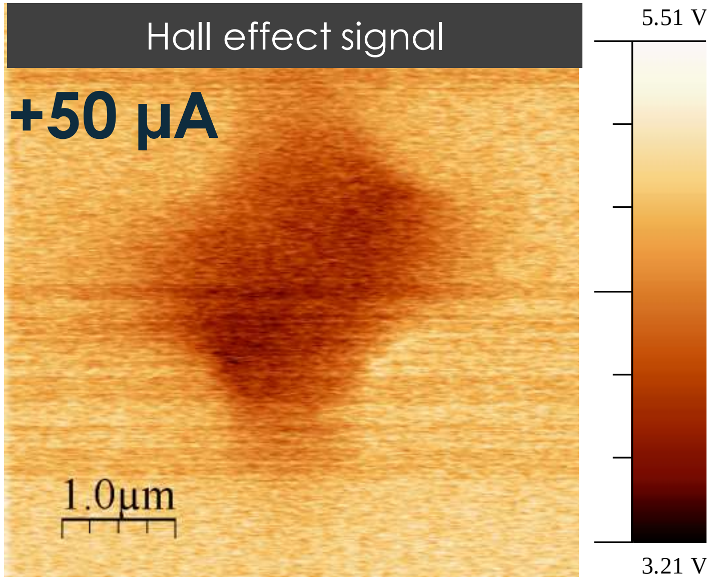


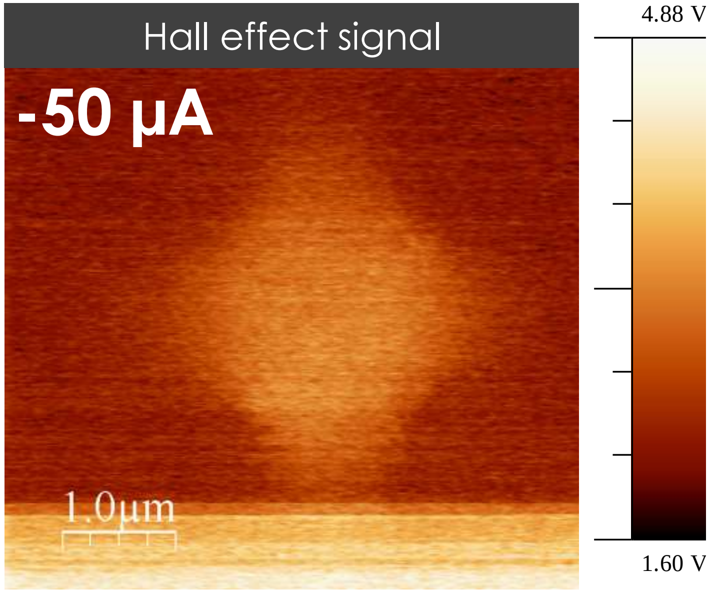

Hall effect signal
Lock-in amplitude

# Hall effect graphene-based sensor

Hall voltage changes when the current direction changes.

The MFM tip's magnetization remains constant.



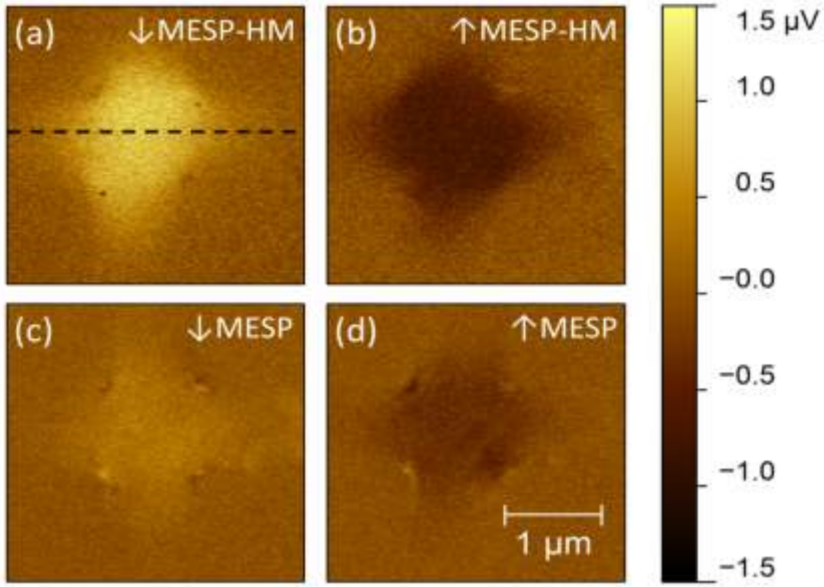

$$V_{Hall} \propto I \cdot \left( \frac{n^{o}\, Field\_Lines}{Area} \right)$$

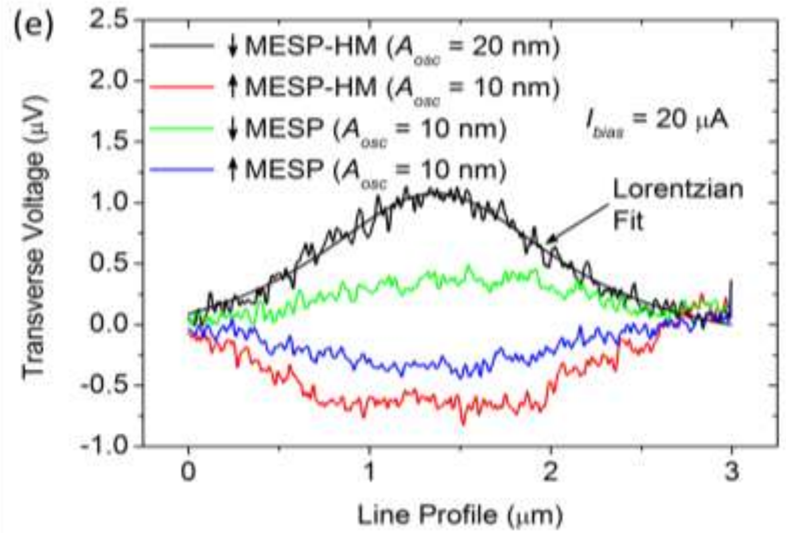
V. Panchal, O. Iglesias-Freire, A. Lartsev, R. Yakimova, A. Asenjo and O. Kazakova, IEEE Trans on Mag, 49 (2013) 3520

# Hall effect graphene-based sensor

Hall voltage changes when the MFM tip's magnetization direction changes.

The Hall voltage depends on the tip stray field and on the tip oscillation's amplitude.



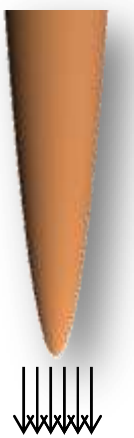

$$V_{Hall} \propto I \cdot \left( \frac{n^{o}\, Field\_Lines}{Area} \right)$$

V. Panchal, O. Iglesias-Freire, A. Lartsev, R. Yakimova, A. Asenjo and O. Kazakova, IEEE Trans on Mag, 49 (2013) 3520

# Hall effect graphene-based sensor

## Micromagnetic Simulations, OOMMF software

Tip parameters: 40 nm-thick polycrystalline cobalt layer , saturation magnetization $M_s$=1400 kA/m, exchange stiffness A=3×$10^{-11}$J/m, magnetocrystalline anisotropy is negligible and a cell size of 1 nm.



1.- Stray field at different tip-sample distances

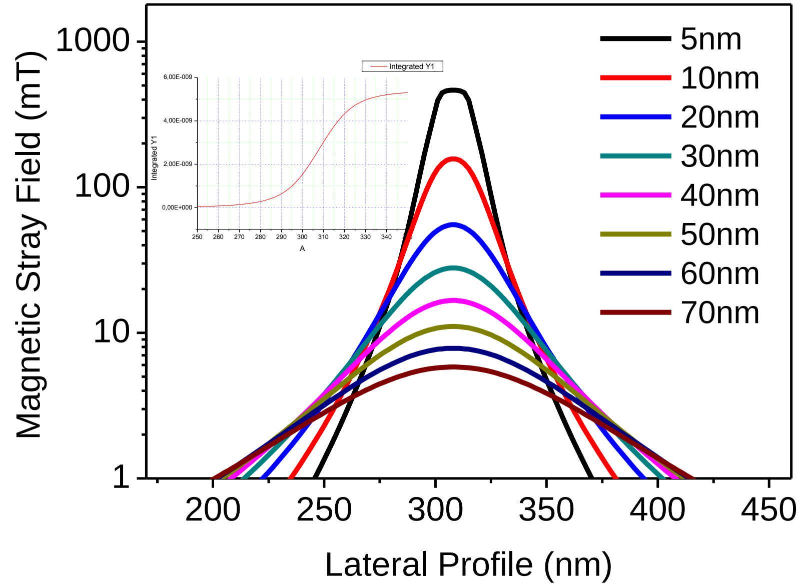
2.- Integrated field versus edge distance

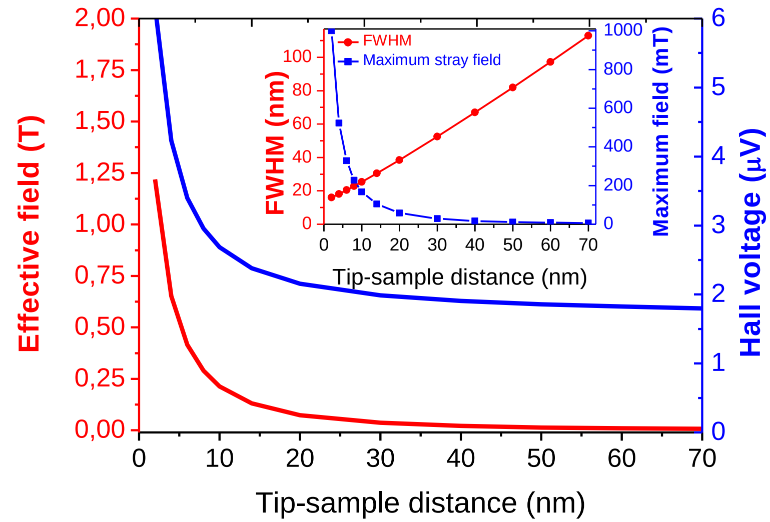
3.- Effective radius

4.- Expected $V_{hall}$ values for different $A_{osc}$

5.- $B_{probe}$ estimation

| Probe | $A_{OSC}$ | EXPERIMENTAL $V_{XY}$ | SIMULATED $V_{XY}$ | $B_{PROBE}$ |
|---|---|---|---|---|
| **MESP** | 10/20 nm | ~0.38/__µV | 0.74/0.87µV | 153 / 70mT |
| **MESP-HM** | 10/20 nm | 0.64/1.08µV | 0.96/1.24 µV | 183/ 76 mT |




V. Panchal, O. Iglesias-Freire, A. Lartsev, R. Yakimova, A. Asenjo and O. Kazakova, IEEE Trans on Mag, 49 (2013) 3520

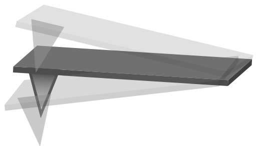
# Outline:

1. Scanning Probe Microscopies.
2. Fundaments of MFM. Measuring under standard conditions
3. Improving the hardware:
   a. Variable Field MFM
   b. HV-Low Temperature MFM
   c. MFM probes
4. Developing new operation modes:
   a. 3D modes
   b. KPFM-MFM combination: Co nanostripes, Graphite, Graphene based hall sensor
   c. Dissipation
5. Conclusions

# Dissipation in MFM

Cantilever movement

***Low field***



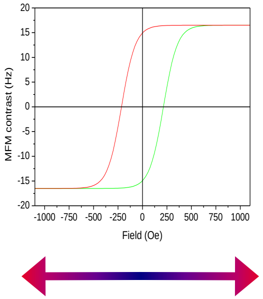
***High field***

+

**"High"** stray field of the sample/tip

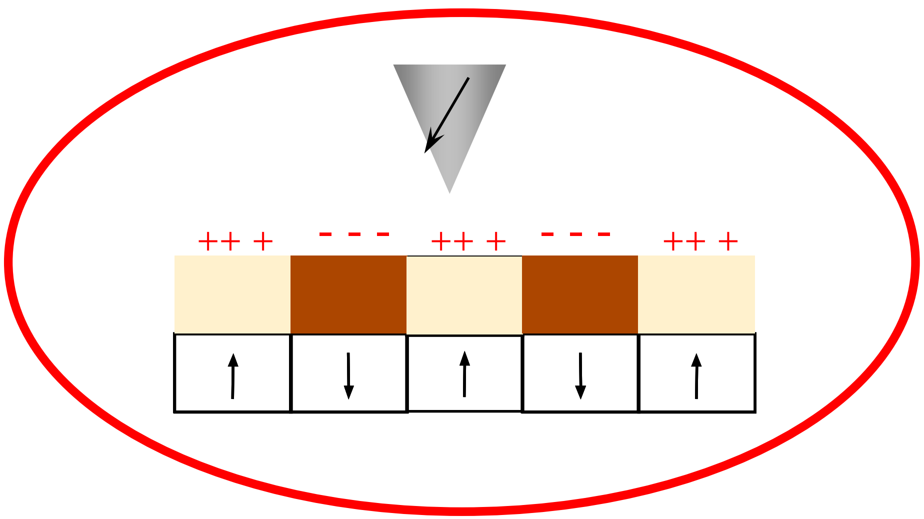
+

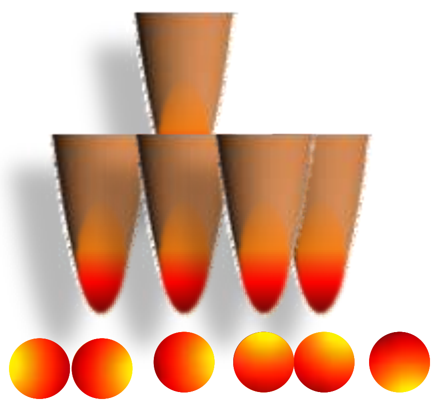
Hysteresis




=

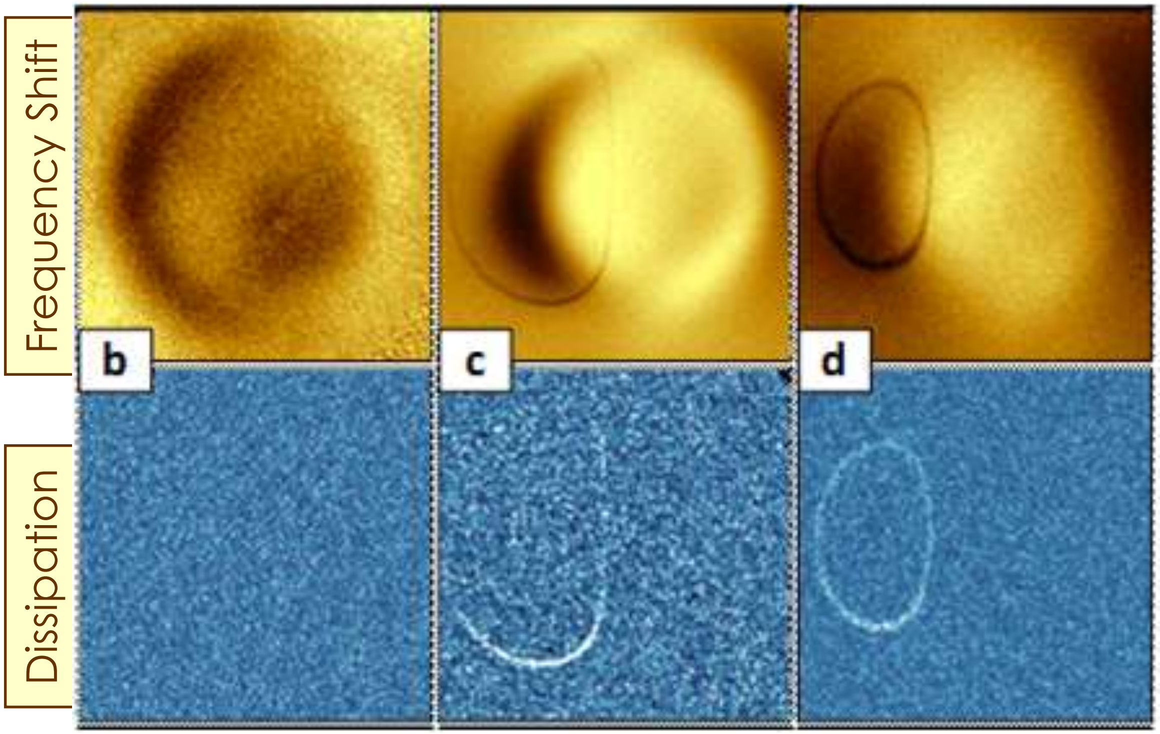
## Dissipation of energy!!!!!



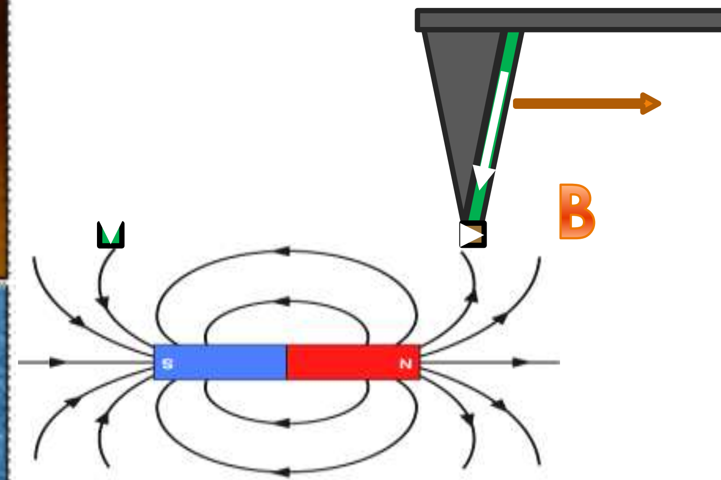



# Tips for mapping the magnetic field

Retrase=60nm Retrase=190nm



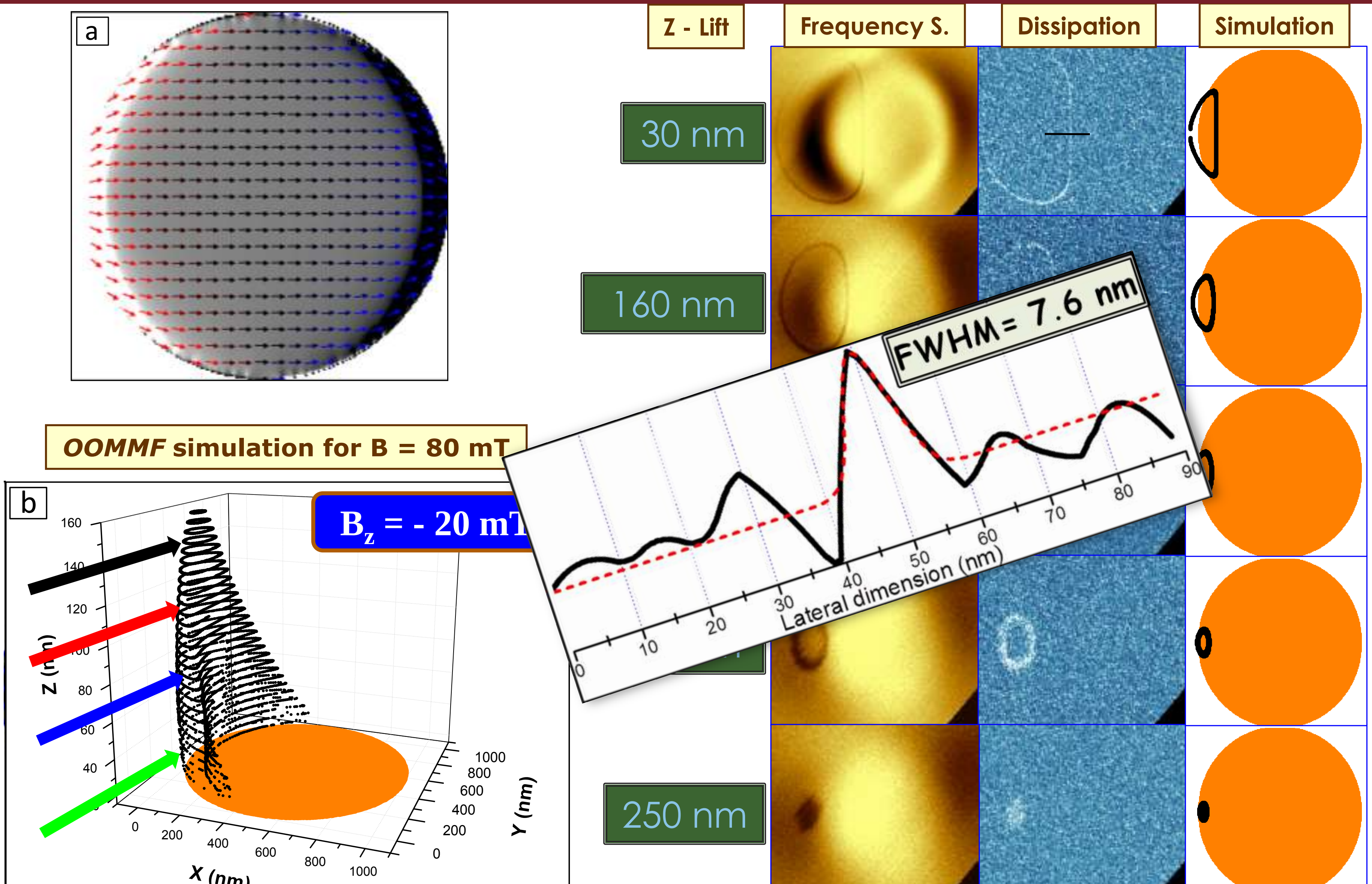

B=800 Oe B=800 Oe




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




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

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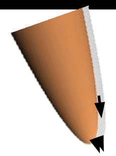
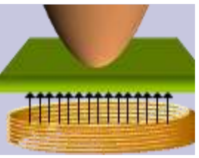
5. Conclusions

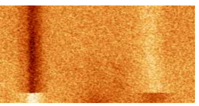
# Conclusions



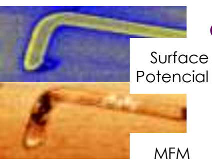
- **Special tips** are presented which allow to improve lateral resolution and to study soft magnetic samples.



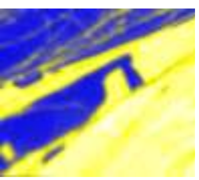
- VF-MFM to characterize the **MFM probes** and the reversal **magnetization process** of individual **elements** at the nanoscale.



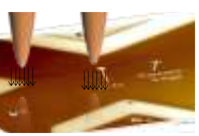
- A novel method to precisely **quantify critical fields.**




- **KPFM/MFM** combination mode is useful to separate electrostatic and magnetic contrasts.



- **KPFM /MFM** combination allow us to give an **upper bound** for the magnetic signal in graphite **16 μN/m** (**six times** lower than the theoretical prediction).



- The **stray field of the MFM tips** can be measured by using a graphene based device and the KPFM/MFM combination .

- The **dissipation of energy**, a new way to gain information about magnetic behaviour with high resolution (less than 7nm).

- **HV-LT MFM (higher Q factor), single pass MFM**

# In collaboration with….

Hall effect graphene-based sensor

V. Panchal
**O. Kazakova**

NPL National Physical Laboratory

Py and Co dots

A. Awad
**F. G. Aliev**

UAM Universidad Autónoma 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*

Agustina Asenjo
Óscar Iglesias-Freire
Eloy Pérez
Miriam Jaafar

**Nanomagnetism and Magnetic Materials Group**

**Manuel Vázquez**
Rafael P. del Real
Cristina Bran

ICMM

**Oksana Fesenko**
Yurii Ivanov

KPFM-MFM in Graphite

D. Martín Martínez
**J. Gómez-Herrero**

**R. Pérez**

UAM Universidad Autónoma de Madrid

$Fe_3O_4$ nanoparticles

C. Moya
**X. Batlle**
**A. Labarta**

Universitat de Barcelona

IN2UB

Technical support

NANOTEC ELECTRONICA