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SPIN IN ELECTRONICS

Characterization techniques for nano-sized systems

O. Fruchart

SPINTEC, Univ. Grenoble Alpes / CNRS / CEA-INAC, France

www.spintec.fr

email: olivier.fruchart@cea.fr

Slides: http://fruchart.eu/slides

THE EUROPEAN SCHOOL ON MAGNETISM

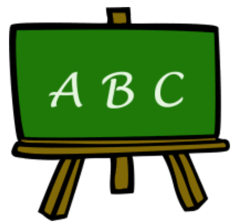
The EUROPEAN MAGNETISM ASSOCIATION

INSTITUT NANOSCIENCES ET CRYOGÉNIE

UNIVERSITÉ Grenoble Alpes

Grenoble INP

Criteria for measurement techniques



Criteria for measurement techniques

What is measured?

- Magnetization, induction, stray field?
- Elemental resolution
- Direct or indirect?
- Quantitative or not?

Environmental conditions

- Temperature
- Field: magnetic field, electric
- Electric current, light etc.
- Strain
- Additional measuring techniques

Which specifications?

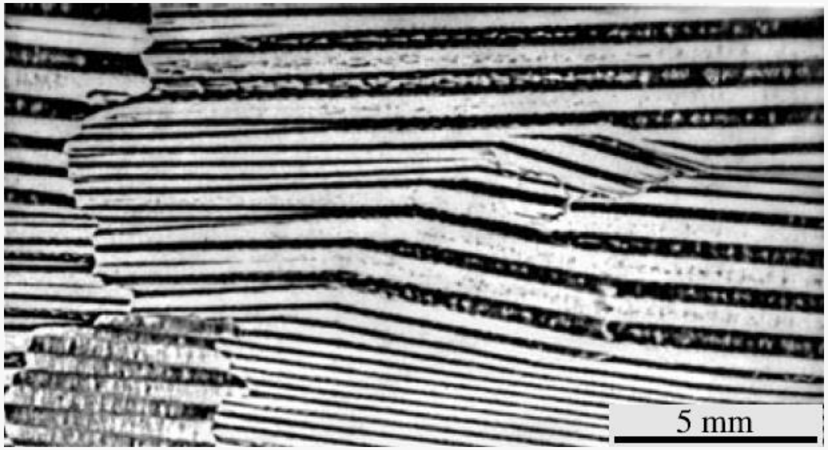
- Magnetization: 1D, 2D, 3D
- Depth resolution: surface or volume?
- Lateral resolution
- Sensitivity
- Time/Spectral resolution

Versatility

- Sample preparation needed
- Time per one measurement
- In situ / ex situ
- Large-scale or in-lab?
- Expensive or cheap?

Magnetic domains

- Numerous and complex shape of domains



History: Weiss domains

Magnetic length scales

- Magnetic energy

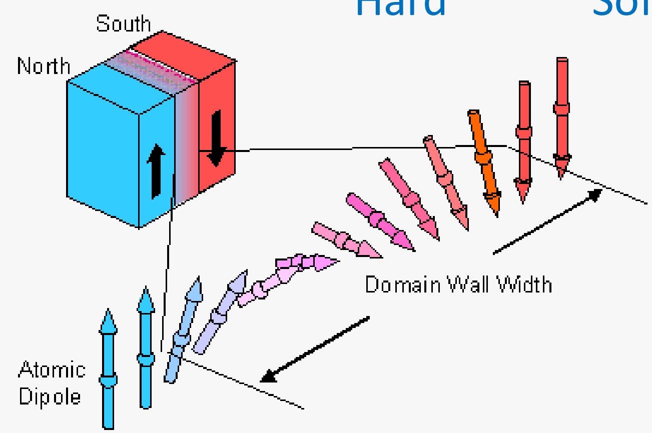
$$E = A\left(\frac{\partial m_i}{\partial x_j}\right)^2 + K\sin^2\theta$$

Exchange: $\mathrm{J/m}$ Anisotropy: $\mathrm{J/m^3}$

- Anisotropy exchange length

$$\Delta_\mathrm{u} = \sqrt{A/K} \quad 1\ \mathrm{nm} \rightarrow 100\ \mathrm{nm}$$

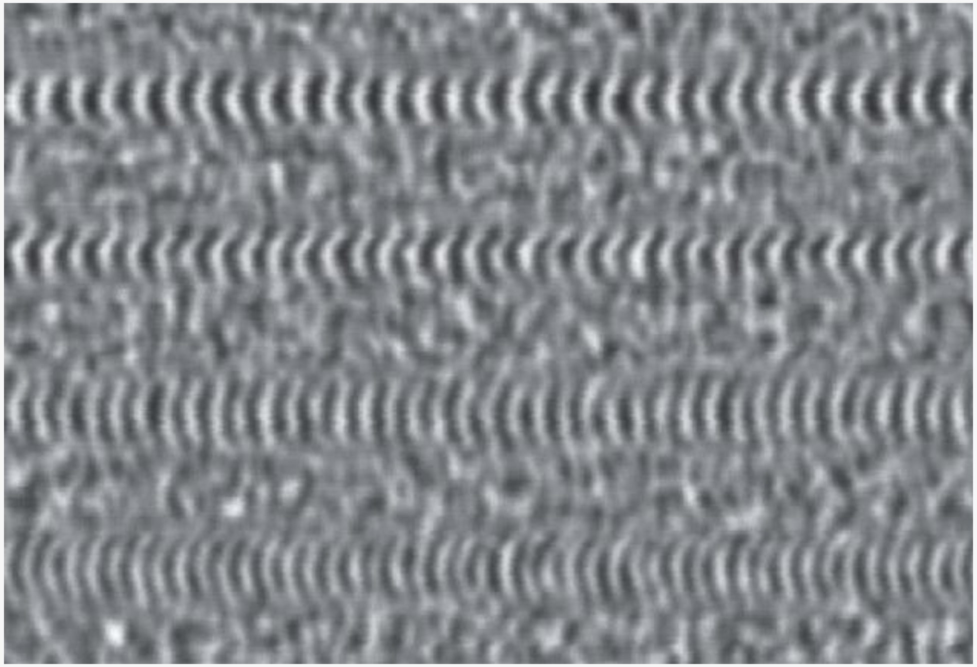
Hard → Soft



MOTIVATION: length scales (technology)

Magnetic bits on hard disk drive

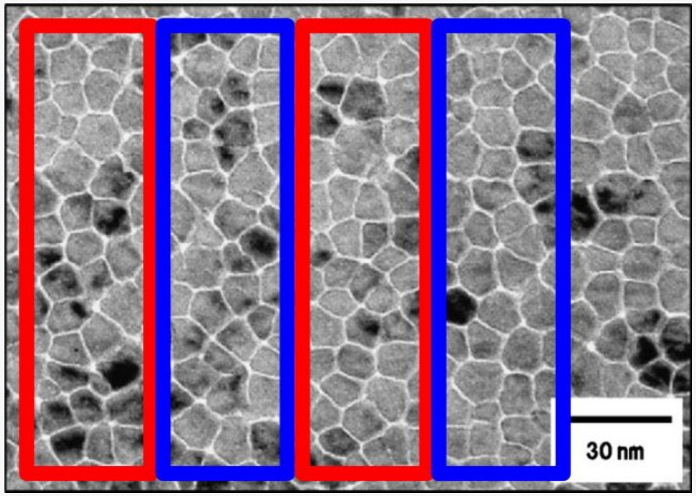
Co-based hard disk media : bits 50nm and below



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

Underlying microstructure

10 nm magnetic grain



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

MOTIVATION: time scale

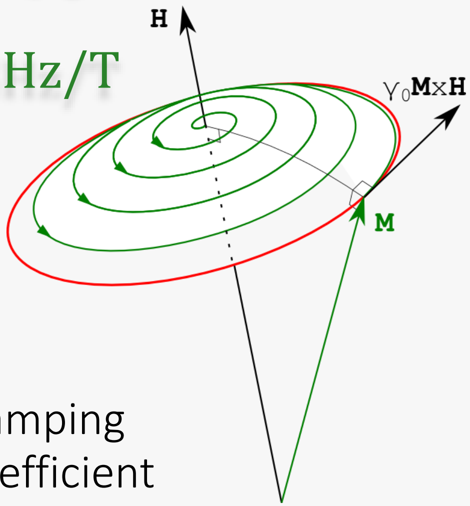
Precessional magnetization dynamics

See lecture Christian Back

$$\frac{\mathrm{d}\mathbf{m}}{\mathrm{d}t} = \gamma_0 \mathbf{m} \times \mathbf{H} + \alpha \mathbf{m} \times \frac{\mathrm{d}\mathbf{m}}{\mathrm{d}t}$$

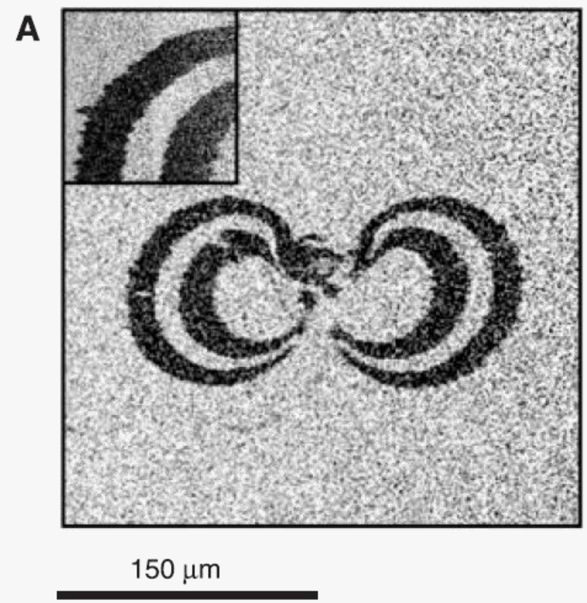
$\gamma_0 = \mu_0 \gamma < \mathbf{0}$ Gyromagnetic ratio

$\gamma_s = 28\ \mathrm{GHz/T}$



$\alpha > 0$ Damping coefficient

Pioneering experiment of precessional magnetization reversal



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

- >1µs : thermally-activated magnetization processes
- 1 ns : precession of magnetization
- 1 ps : ultrafast demagnetization

How to classify techniques?

Spatial resolution

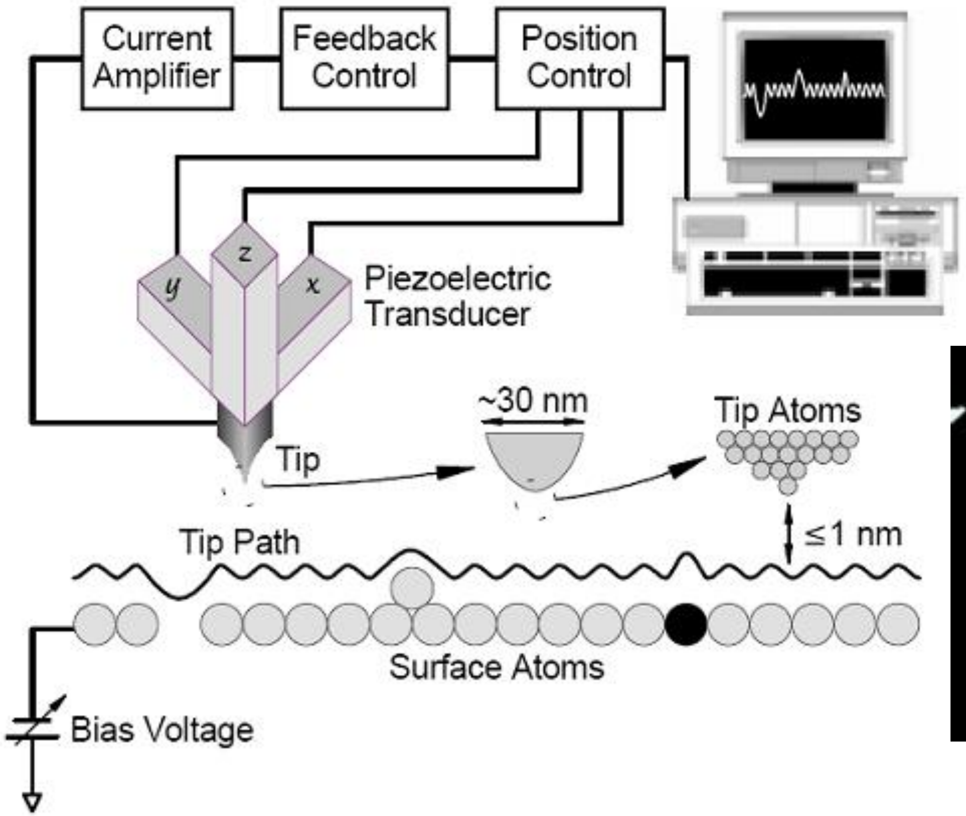
- Global (magnetometry)
- Local (example: small sensors)
- Microscopy
  - Scanning probe
  - Full field

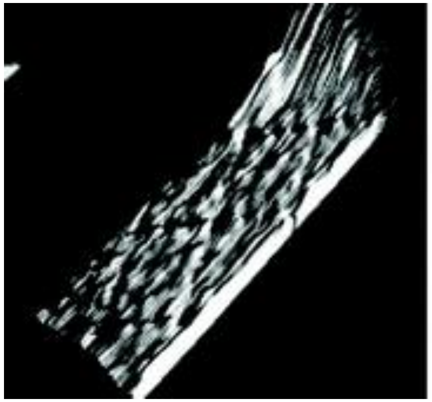
Physical phenomenon

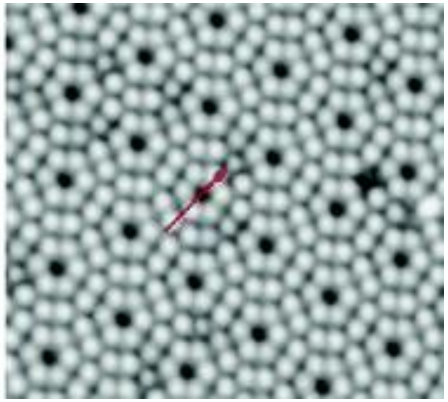
- Probing magnetic field / induction
- Light-matter interaction
- Electron-matter interaction

- Criteria for measurement techniques
- **Probing magnetic stray fields**
- Techniques with light-matter interaction
- Techniques with electron-matter interaction

1982 : inventing the scanning tunneling microscope

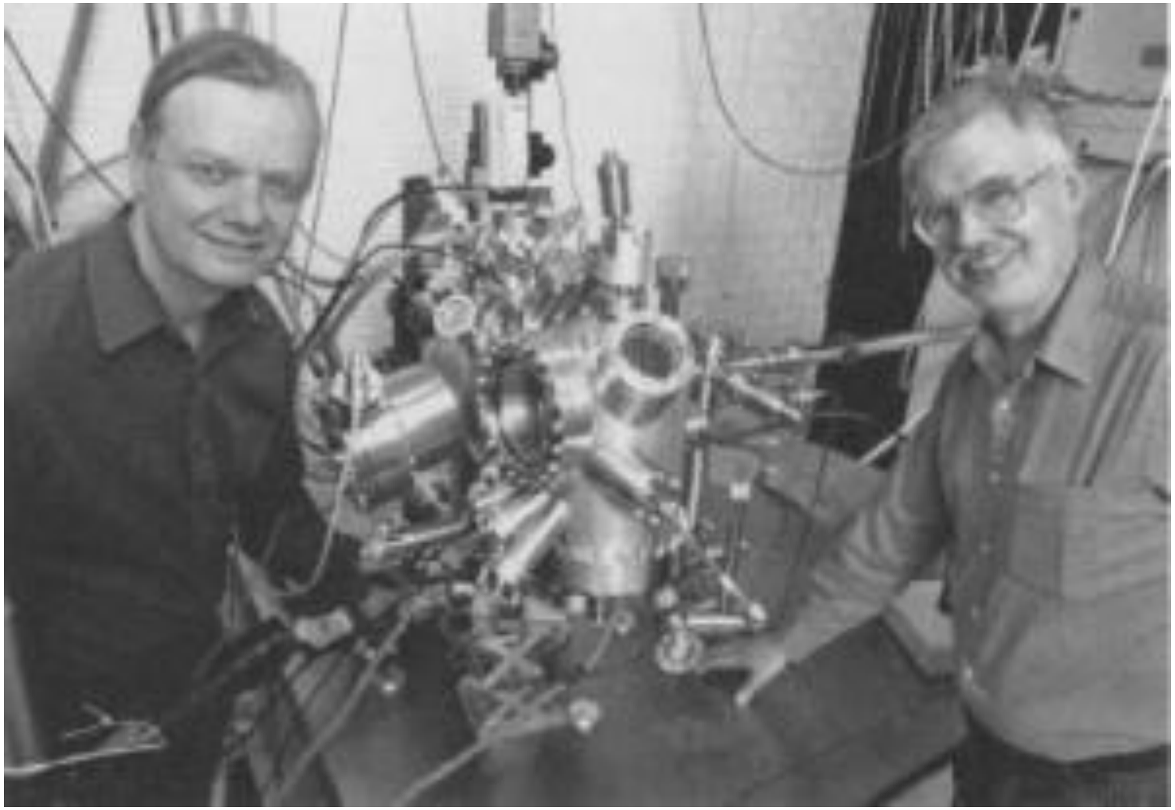






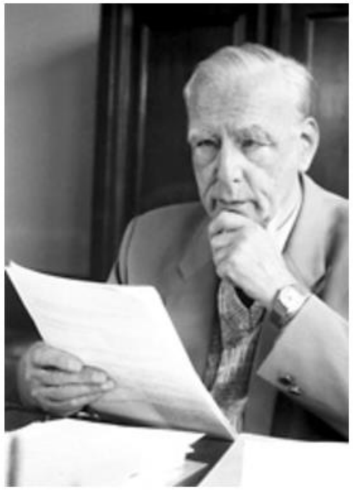
G. Binnig, H. Rohrer, C. Gerber & E. Weibel
Tunneling through a controllable vacuum gap apl 40, 178 (1982)

1982 : inventing the scanning tunneling microscope



G. Binnig, H. Rohrer, C. Gerber & E. Weibel
Tunneling through a controllable vacuum gap apl 40, 178 (1982)

The Nobel Prize in Physics 1986



Ernst Ruska
Prize share: 1/2

Gerd Binnig
Prize share: 1/4

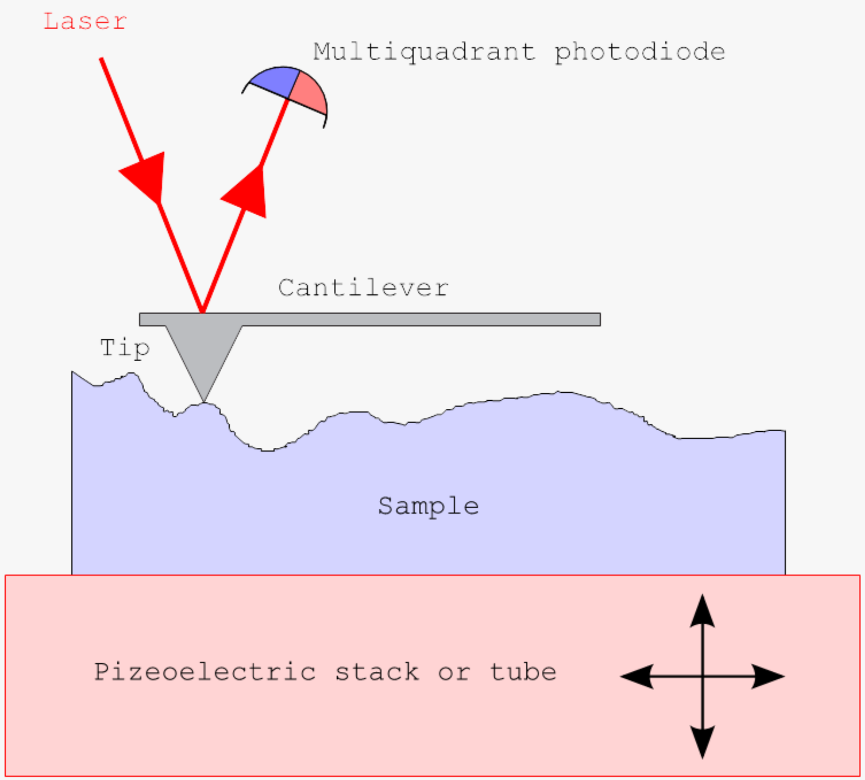
Heinrich Rohrer
Prize share: 1/4

The Nobel Prize in Physics 1986 was divided, one half awarded to Ernst Ruska *"for his fundamental work in electron optics, and for the design of the first electron microscope"*, the other half jointly to Gerd Binnig and Heinrich Rohrer *"for their design of the scanning tunneling microscope"*.

https://www.nobelprize.org

Scanning Probe Microscopy - AFM

The Atomic Force Microscope (AFM)



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

Probing

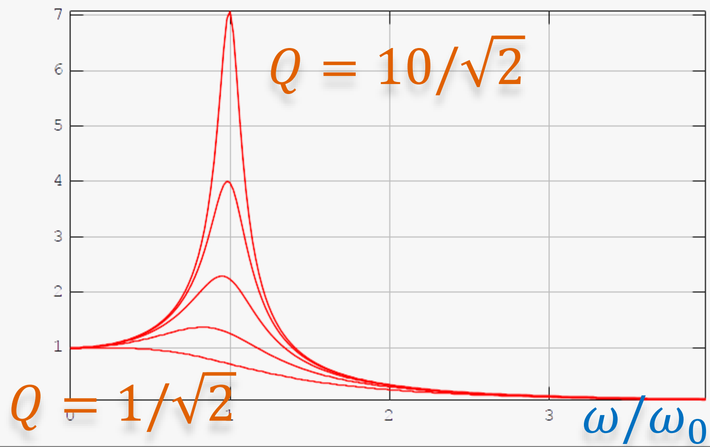
- Mechanical force -> Topography, tribology (adhesion etc.)
- Electric forces -> ferroelectric domains, semiconductor memory cells etc.
- Magnetic force -> magnetic domains

Detection

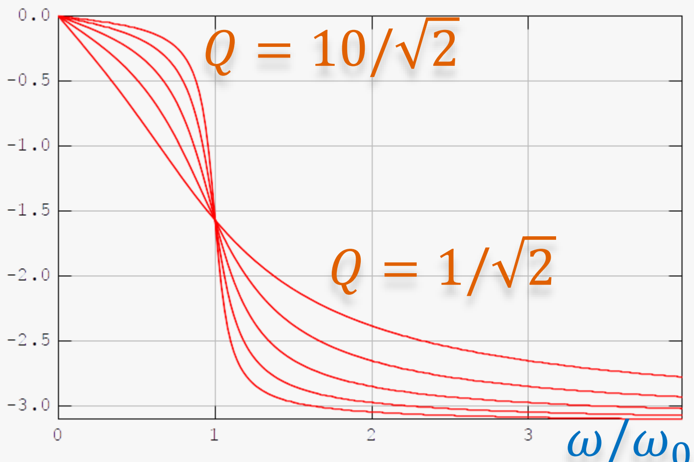
- Laser deflection / interference
- Capacitance

Work under forced ac excitation

Amplitude



Phase



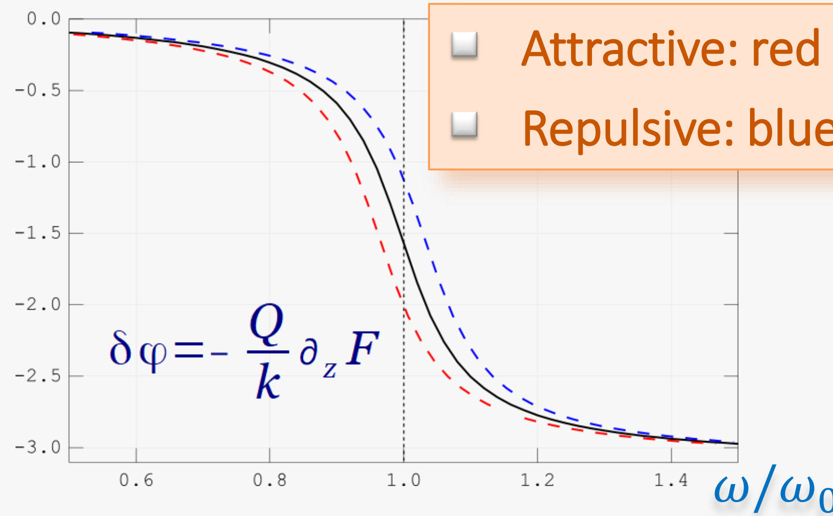
The cantilever as an oscillator

$$m\ddot{z} + \Gamma\dot{z} + kz = F(z)$$

with: $F(z) = F(z_0) + (z - z_0)\partial_z F$

Mere renormalization: $\omega_{0,\mathrm{eff}} = \omega_0\left(1 - \frac{1}{2k}\partial_z F\right)$

Probing forces with the phase shift

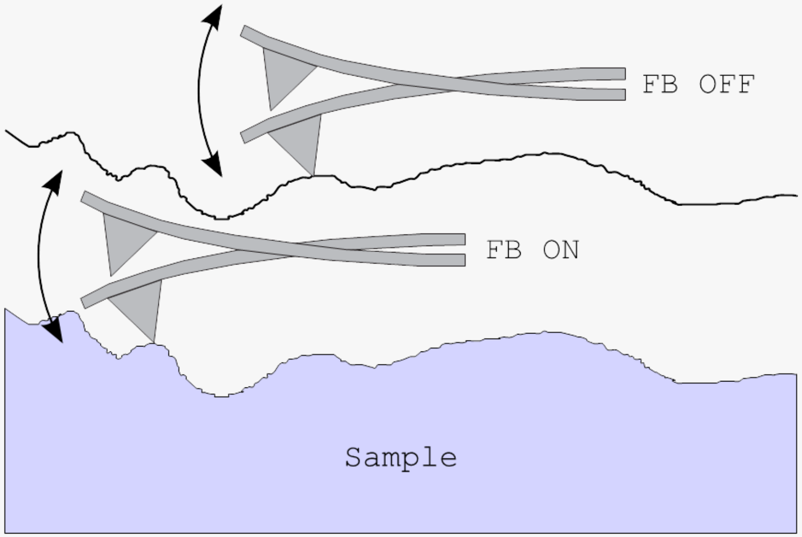


$$\delta\varphi = -\frac{Q}{k}\partial_z F$$

- Attractive: red shift
- Repulsive: blue shift

Magnetic Force Microscopy

Operation: two-pass technique



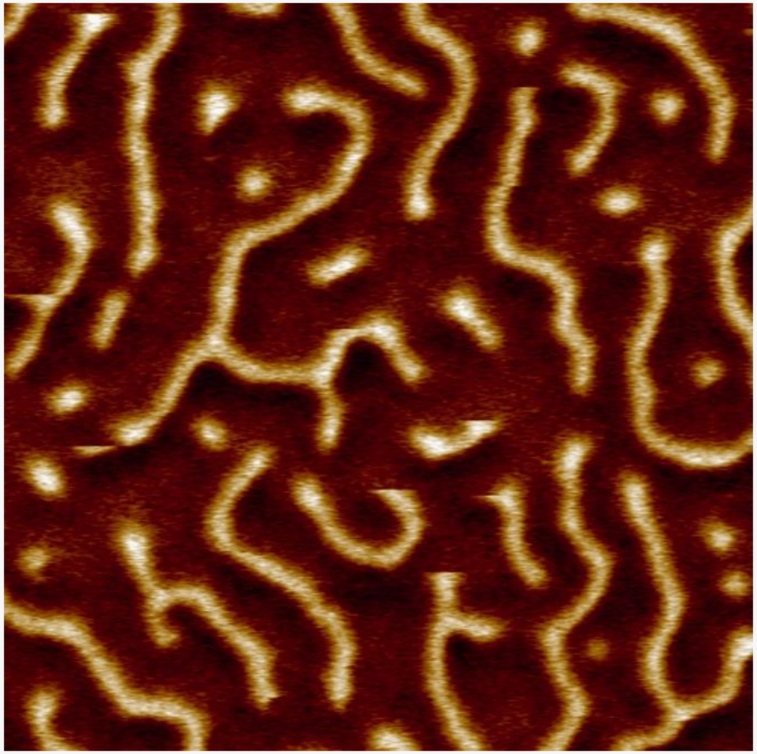
- First pass: measure topography
- Second pass: measure magnetism

NB: other measurement modes exist

Y. Zhu Ed., Modern techniques for charact-erizing magnetic materials, Springer (2005)

http://olivier.fruchart.eu/slides

Example



2x2 µm, Pd\Co\Au multilayer, 80mT
(co-existence of stripes and bubbles)

Sample courtesy: C. Bouard, P. Warin

Magnetic Force Microscopy: criteria

What is measured?

- Stray field?
- Indirect?
- Hardly quantitative

Environmental conditions

- Temperature
- Field: magnetic field, electric
- Electric current, light etc.
- Strain
- Additional measuring techniques

Which specifications?

- Depth resolution: surface/volume
- Lateral resolution: 25-50nm
- Sensitivity: medium (1nm thickness)
- Time/Spectral resolution: slow

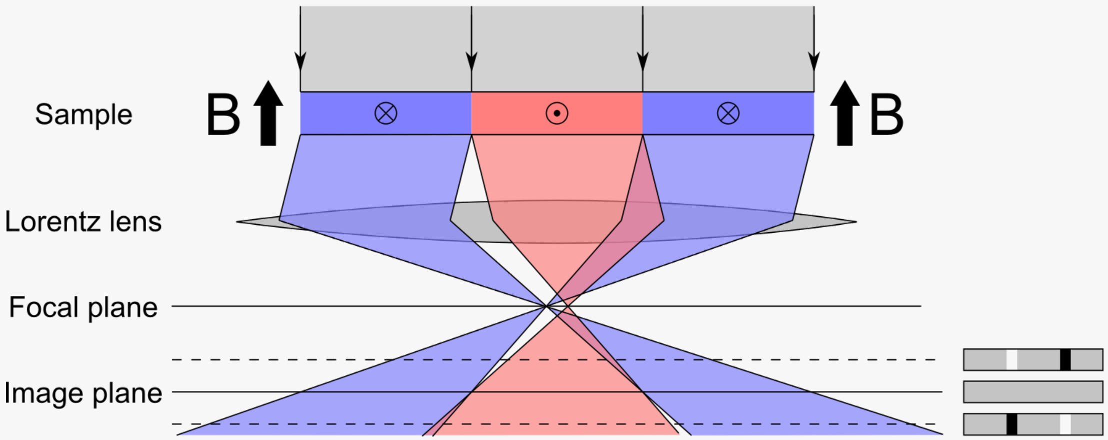
Versatility

- No sample preparation needed
- Time per measurement: few mn
- Ex situ
- In-lab, cheap
- May influence sample

Lorentz microscopy

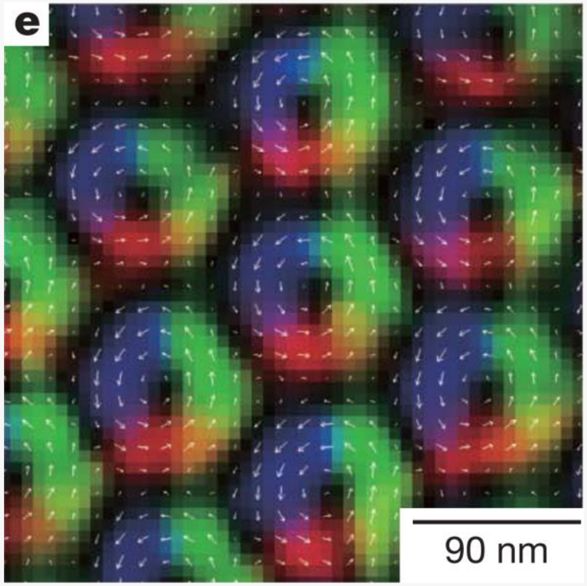
Based on: transmission electron microscopy

The Fresnel mode



Y. Zhu Ed., Modern techniques for charact-erizing magnetic materials, Springer (2005)

Example

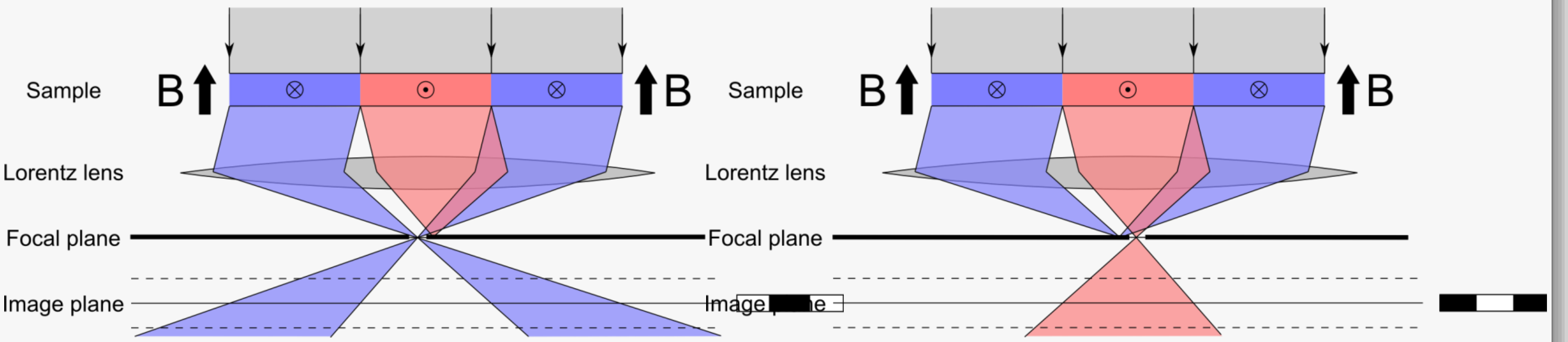


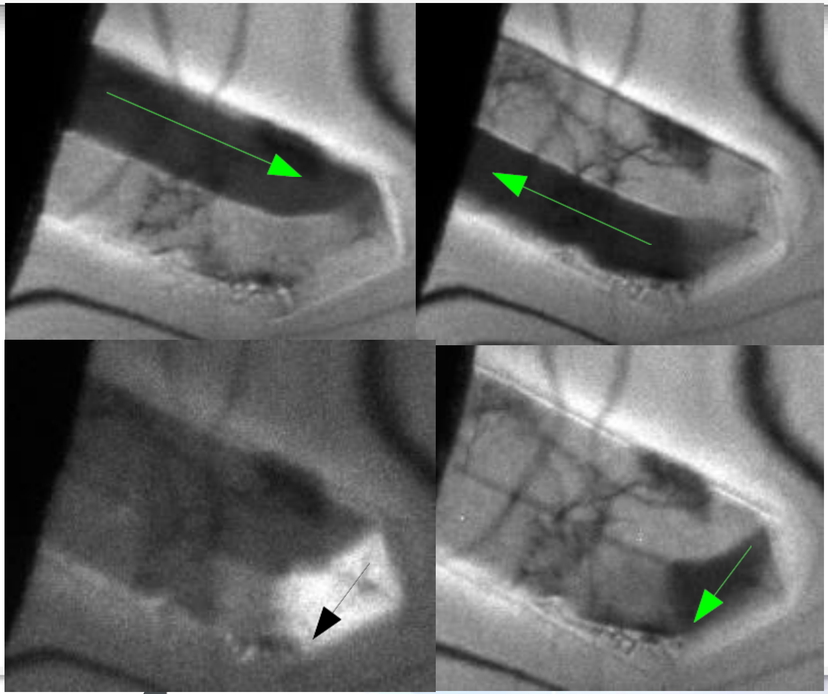
Skyrmion lattice in $Fe_{0.5}Co_{0.5}Si$

X. Z. Yu et al., Nature 465, 901 (2010)

- Highlights gradients of magnetization: domain walls, vortices etc.
- Probes induction: magnetization + stray field
- 2D maps may be reconstructed
- < 5nm spatial resolution

The Foucault mode



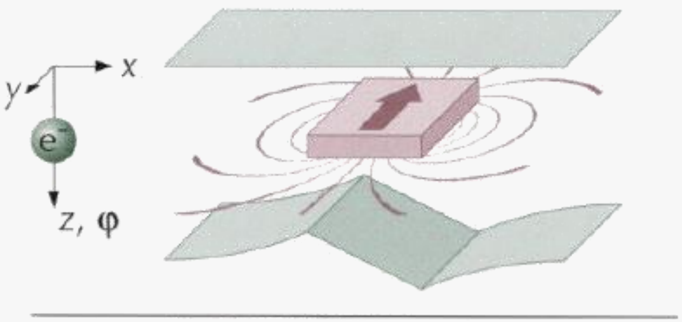


- Highlights magnetic domains
- 2D maps of induction may be reconstructed
- <5nm spatial resolution

Imaging courtesy: A. Masseboeuf

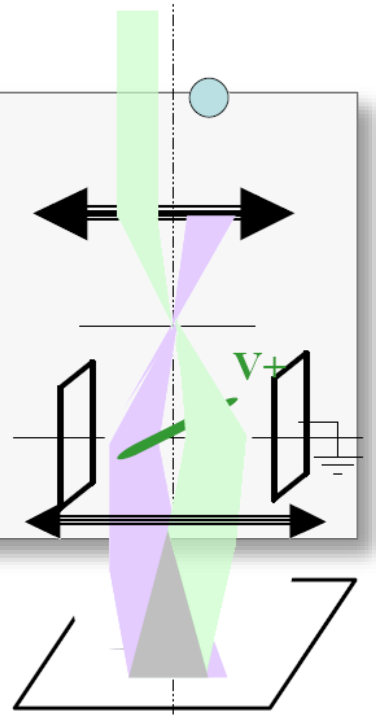
Based on: transmission electron microscopy

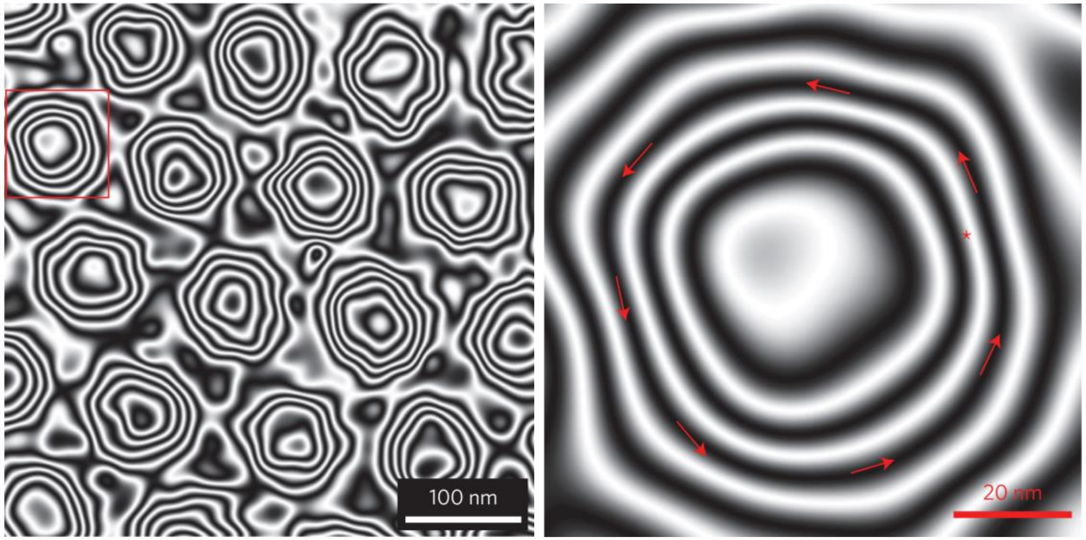
Principle



Phase shift due to the magnetic induction

$$\varphi_{mag}(\mathrm{x}) = -\frac{e}{\hbar}\iint B_{\mathrm{n}}(\mathrm{x},\mathrm{z})\mathrm{dxdz}$$





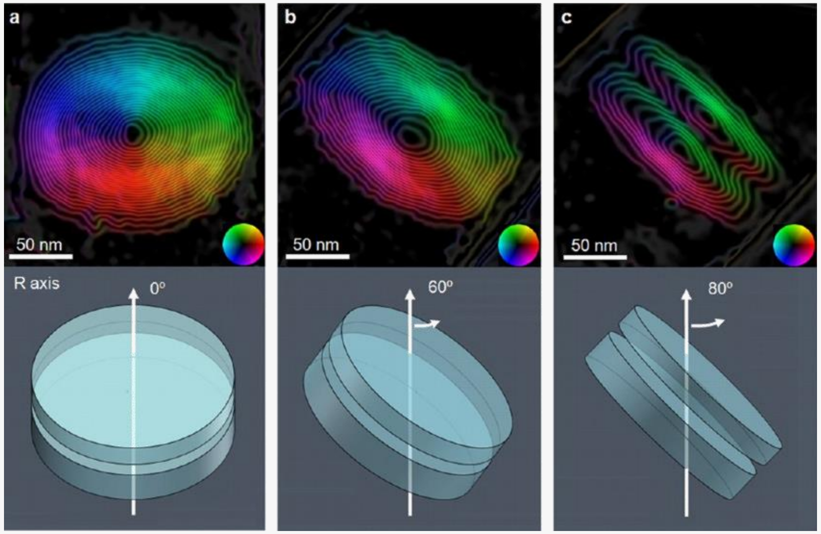
H. S. Park et al., Nat. Nanotech 9, 337 (2014)

- Highlights isolines of z component of vector potential
- 2D maps of induction may be reconstructed
- <1-2nm spatial resolution

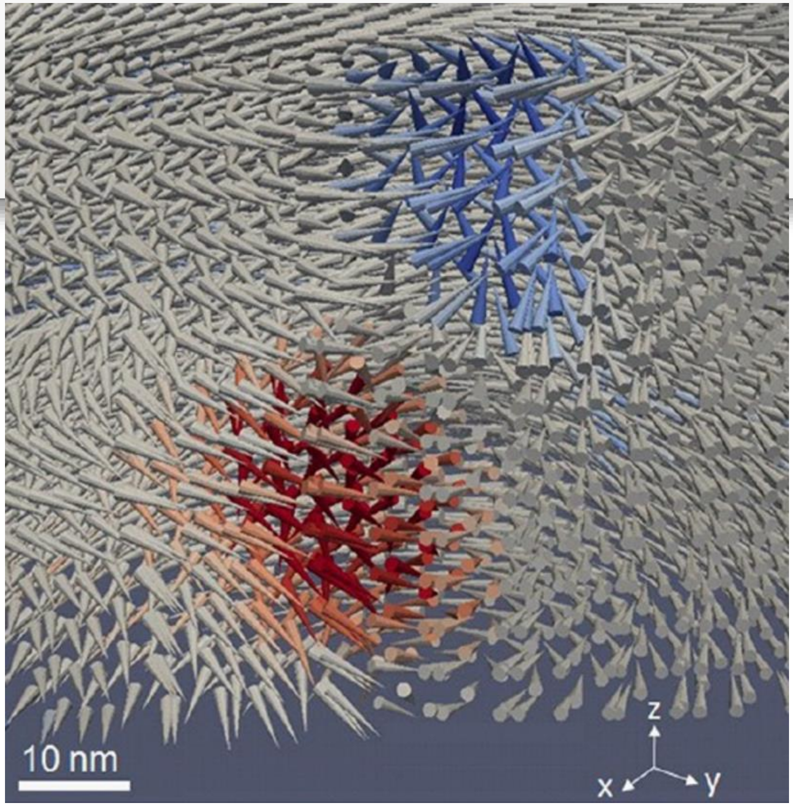
Electron tomography

Principle

- Gather 2D set of images at different (θ,ψ) tilts
- Reconstruct 3D magnetization pattern with iteration algorithm
- Note: no bijection, unlike the structural case



T. Tanigaki et al., Nanolett 15, 1309 (2015)



- 3D maps of induction may be reconstructed
- <2nm spatial resolution
- Cutting edge
- Still debated…

TEM-based microscopy – Criteria

What is measured?

- Induction ✓
- No elemental resolution ✓
- Direct ✓
- Quantitative ✓

Environmental conditions ✓

- Temperature
- Field: magnetic field, electric
- Electric current, light etc.
- Strain
- Additional measuring techniques

Which specifications?

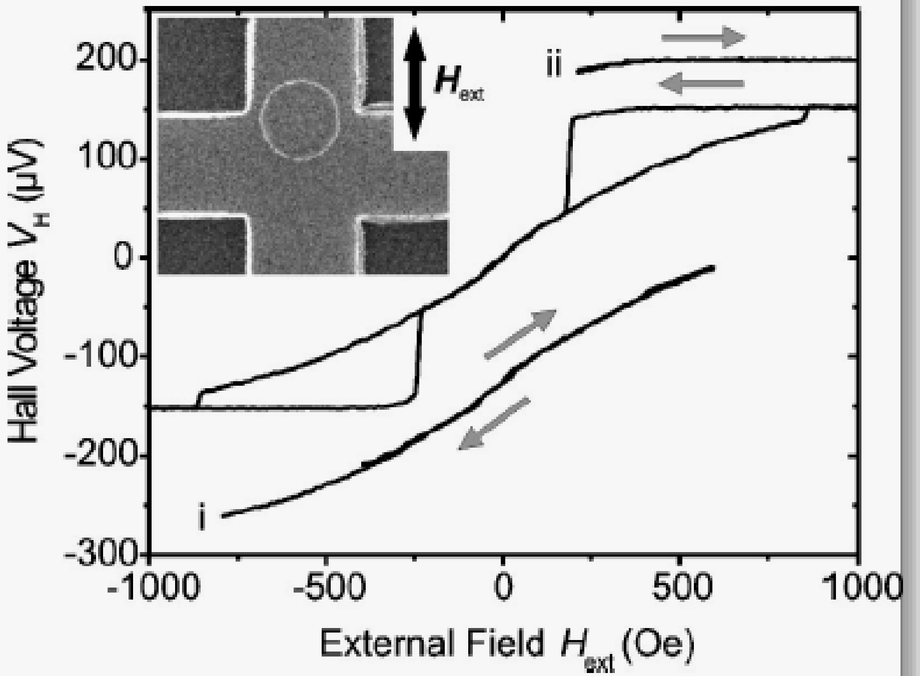
- Magnetization: 1D-3D ✓
- Depth resolution: integrated, <100nm ✓
- Lateral resolution: <5nm ✓
- Sensitivity: >1nm ✓
- Time resolution: cutting-edge ✓

Versatility

- Sample preparation needed ✓
- Time per measurement: seconds ✓
- In situ / ex situ ✓
- Large-scale or in-lab? ✓
- Expensive or cheap? ✓

Magneto-resistive sensors

Example



Patterned magnetic dot on a Hall cross

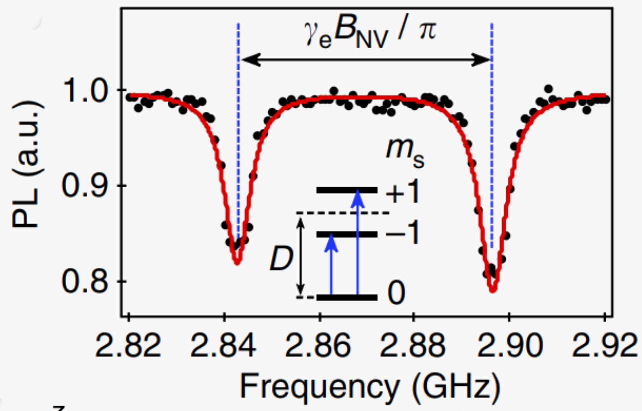
M. Rahm et al., Appl. Phys. Lett. 82, 4110 (2003)

- Measurement of stray field
- High sensitivity (local magnetometer)
- May be turned into scanning probe
- Versatile for measurement, not for fabrication
- Medium dynamics

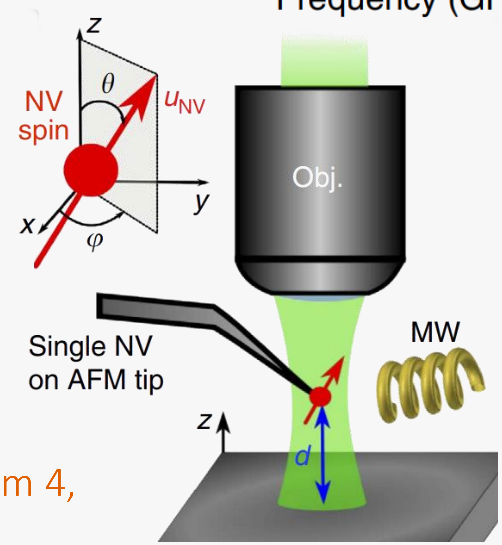
NV center microscopy

Principle

- Resonant excitation of an NV center in diamond with triplet state: sensitive to Zeeman splitting



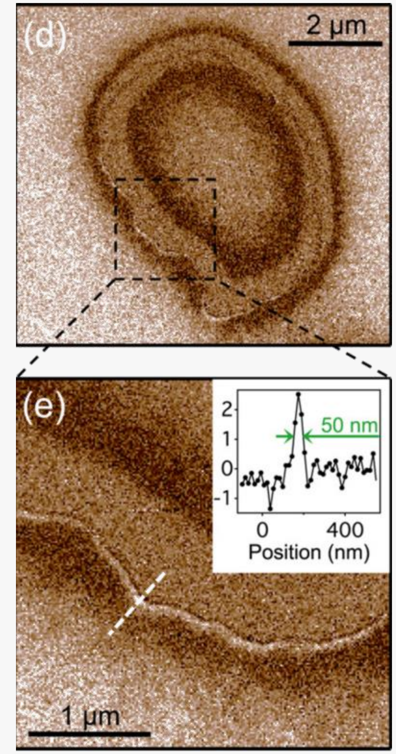
- Mounted on AFM tip + scanning + confocal microscope



L. Rondin et al., Nat. Comm 4, 2279 (2013)

- Extreme sensitivity (µT)
- Needs modeling (stray field + quantization axis)

Example



J.-P. Tetienne et al., JAP 115, 17D501 (2014)

Pt\Co(6Å)\AlOx stack

- Difference between Néel & Bloch walls
- Probes Dzyaloshinskii-Moriya physics

OUTLINE

- Criteria for measurement techniques
- Probing magnetic stray fields
- **Techniques with light-matter interaction**
- Techniques with electron-matter interaction

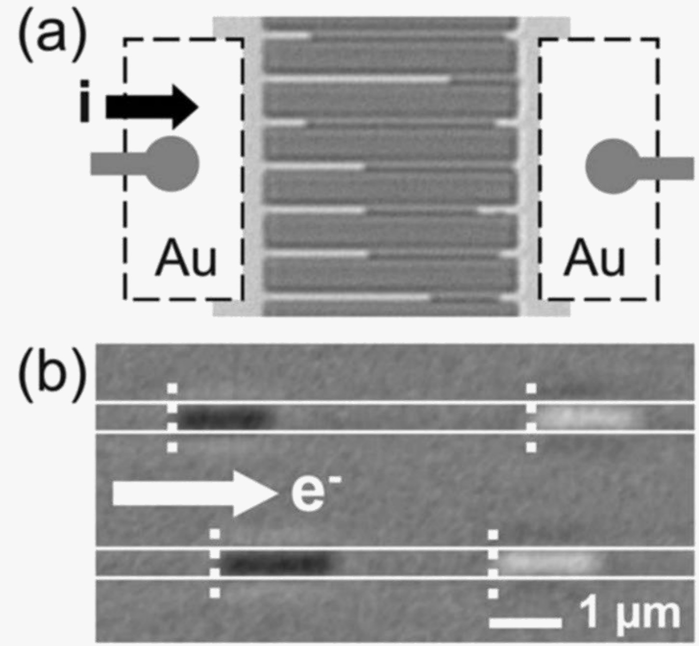
Kerr microscopy

Principle

- Magnetization-dependent dichroism and birefringence of polarized light
  - Kerr= reflection (metals)
  - Faraday = transmission (insulators)
- Wavelength dependent

- Limited by light wavelength (except near-field microscopy)
- Compatible with time resolution, environments, magnetic field

Example



Current-induced wall motion. Pt\Co(6Å)\AlOx

- Physics: interplay with Dzyaloshinskii-Moriya physics, and interfacial effects (Rashba, spin-Hall)

T. A. Moore et al., APL 93, 262504 (2008)

Criteria for measurement techniques

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What is measured?
- Magnetization
- Spectroscopy
- Indirect?
- Not quantitative

Environmental conditions
- Temperature
- Field: magnetic field, electric
- Electric current, light etc.
- Strain
- Additional measuring techniques

Which specifications?
- Magnetization: 1D, 2D, 3D
- Depth resolution: surface/volume
- Lateral resolution
- Sensitivity
- Time/Spectral resolution

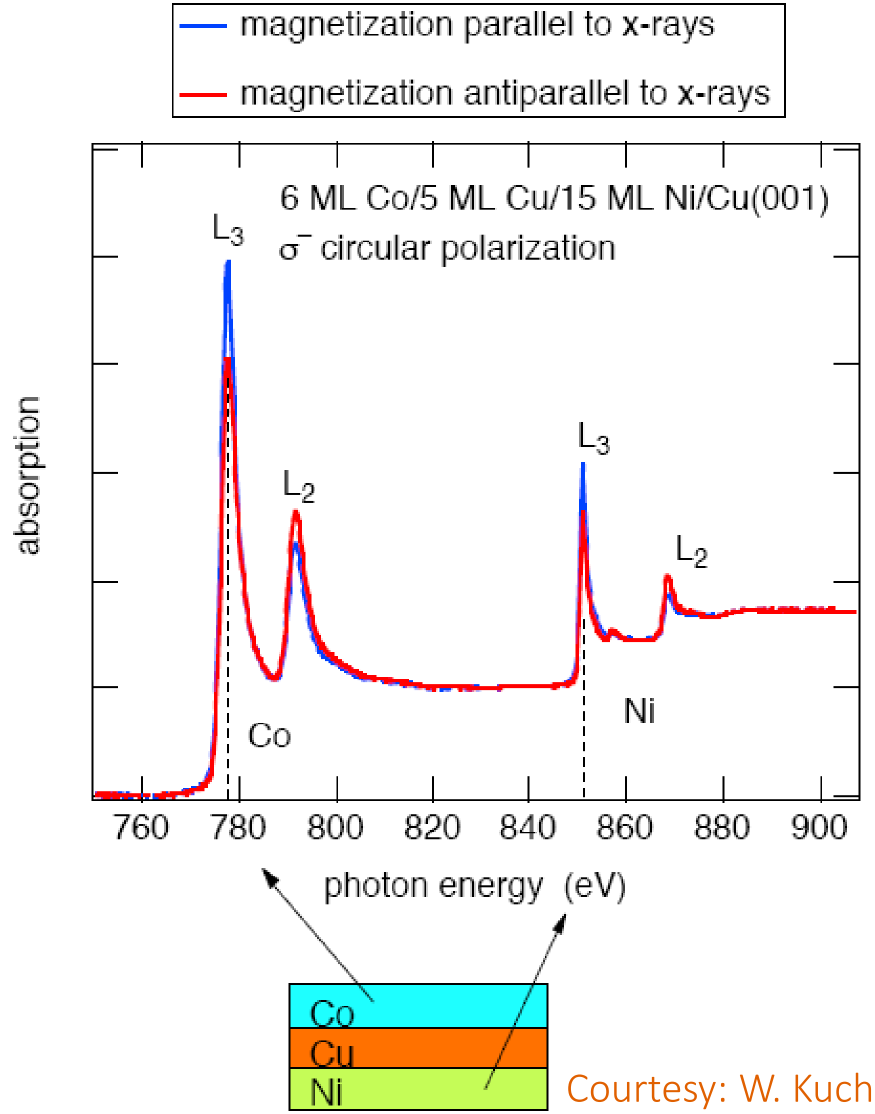
Versatility
- No sample preparation
- Time per measurement: second
- Ex situ
- In-lab?
- Cheap?

X-ray magnetic dichroism

Principle

- Magnetization-dependent dichroism of polarized X rays
  - XMCD: Circular dichroism. Probes ferromagnetism
  - XMLD: Linear dichroism. May probe domains in antiferromagnets

- Magnetometry or microscopy
- Element selective
- Compatible with time resolution
- Requires synchrotron radiation
- May be highly sensitive



Courtesy: W. Kuch

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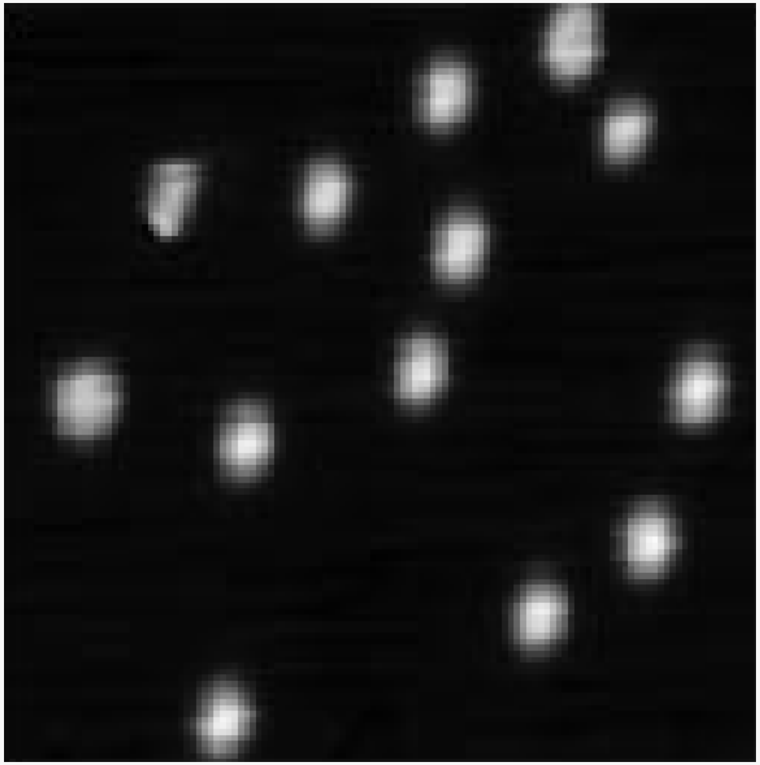
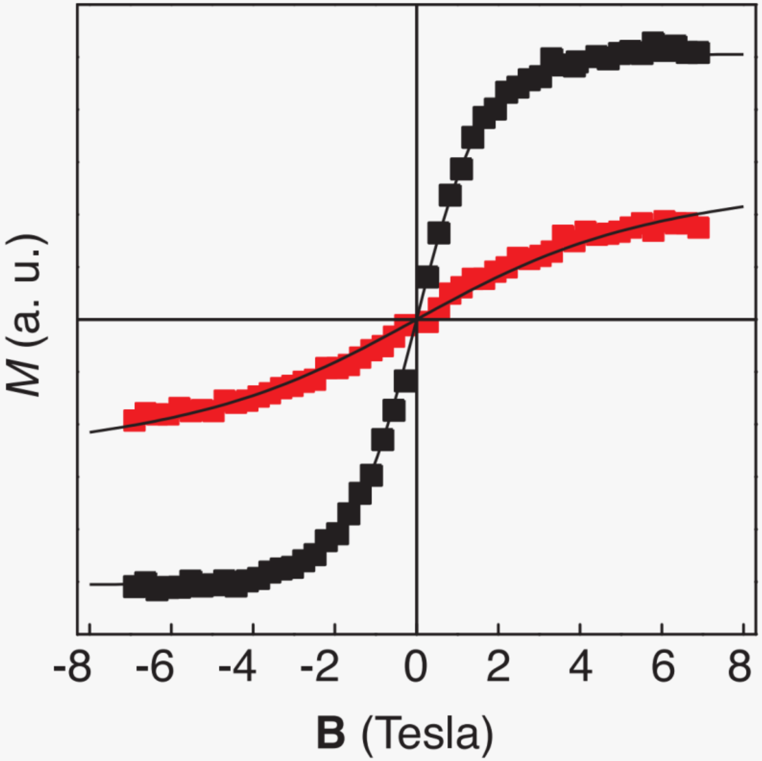
Extreme sensitivity



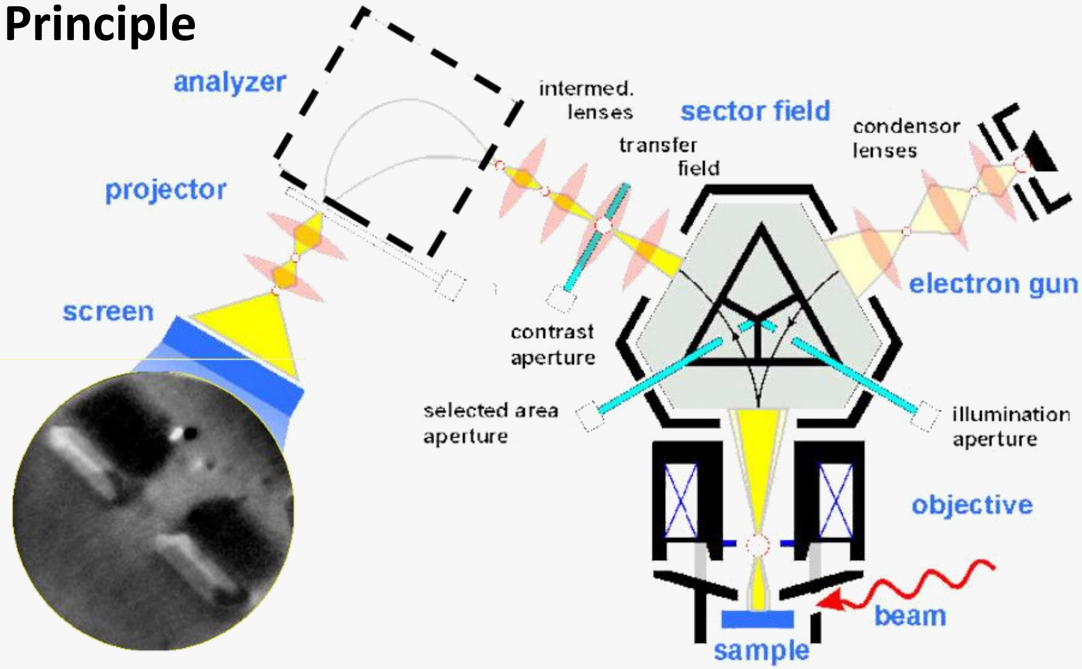


Single Co adatoms on Pt(111)
(STM, 8.5 x 8.5nm)

P. Gambardella et al.,Science 300, 1130 (2003)

Photo-Emission Electron Microscopy (PEEM)

Principle

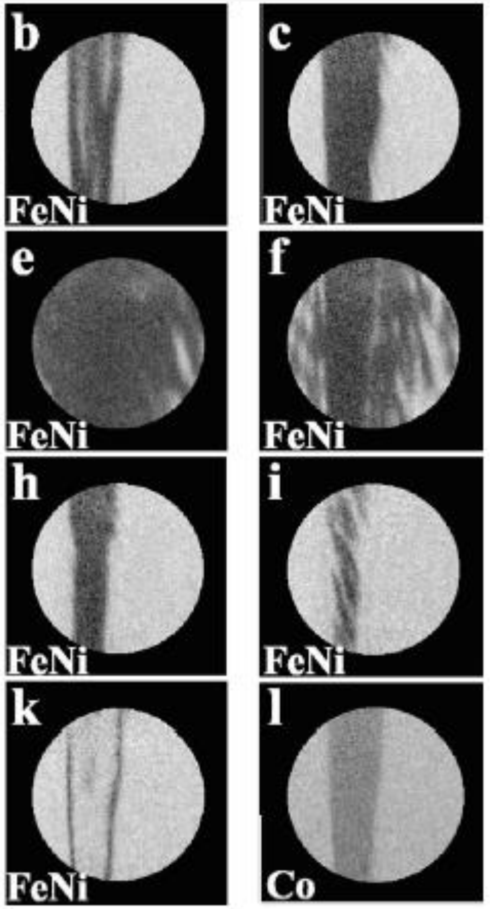


E. Bauer, Surface Microscopy with Low Energy Electrons, Springer, (2014)

Example

FeNi/AlO$_x$/Co spin valves

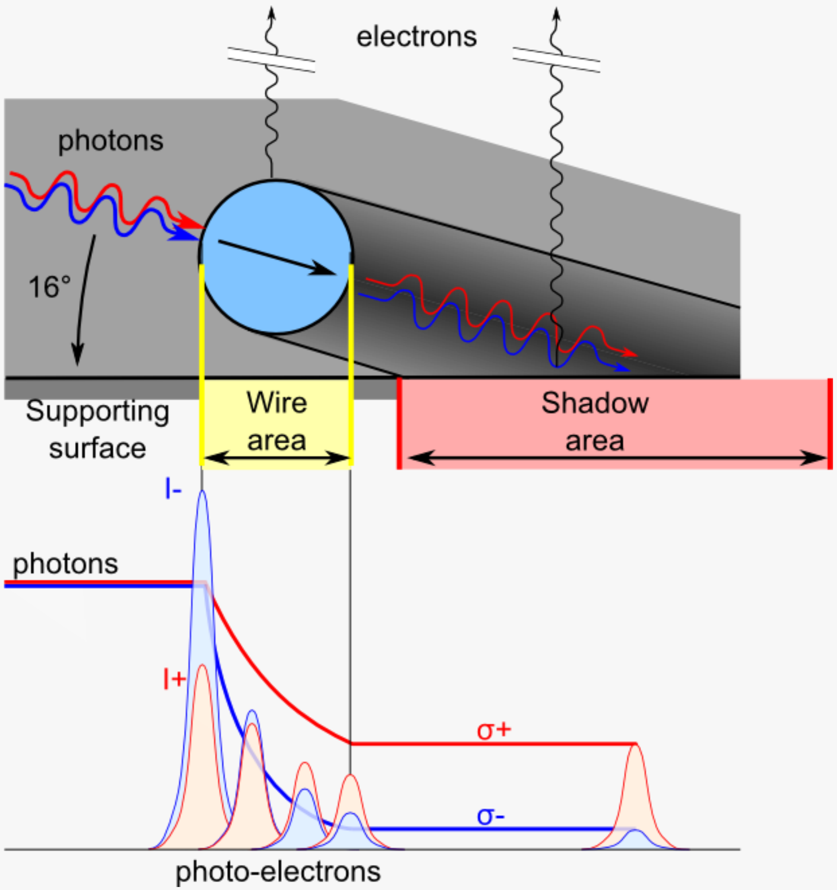
Field pulse of several 10ns



J. Vogel et al., PRB 72, 220402(R) (2005)

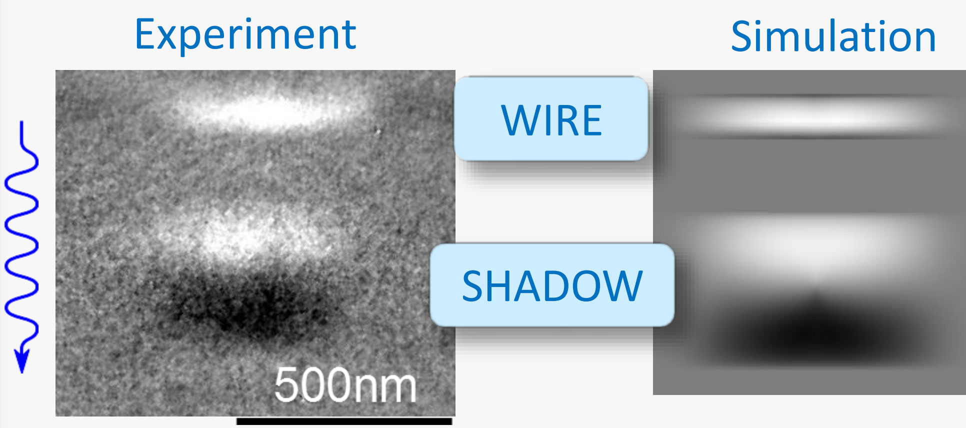
X-ray magnetic dichroism – PEEM going 3D

SHADOW XMCD-PEEM



S. Jamet et al., PRB92, 144428 (2015)

EXAMPLE



Domain wall in 100nm-diameter electroplated cylindrical nanowire

- Bloch point in a domain wall – The only singularity in micromagnetism
- "Topological protection" of domain walls

S. Da Col et al., PRB 89, 180405 (2014)

(XMCD)-PEEM – Criteria

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What is measured?

- Magnetization component
- Elemental resolution
- Direct
- Quantitative

Environmental conditions

- Temperature
- Field: magnetic field (not PEEM)
- Electric current, light etc.
- Strain
- Additional measuring techniques

Which specifications?

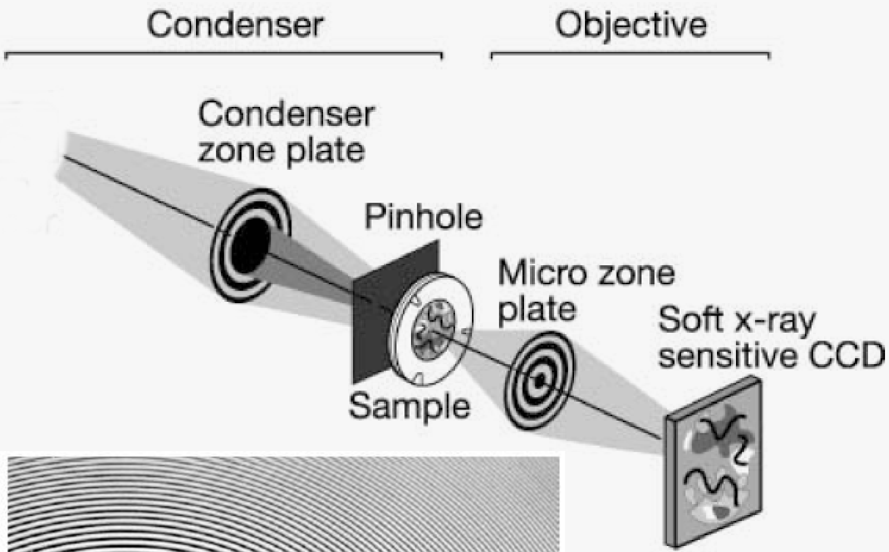
- Magnetization: 1D, 2D
- Depth resolution: surface/volume
- Lateral resolution 25 nm
- Sensitivity: <single layer
- Time/Spectral resolution

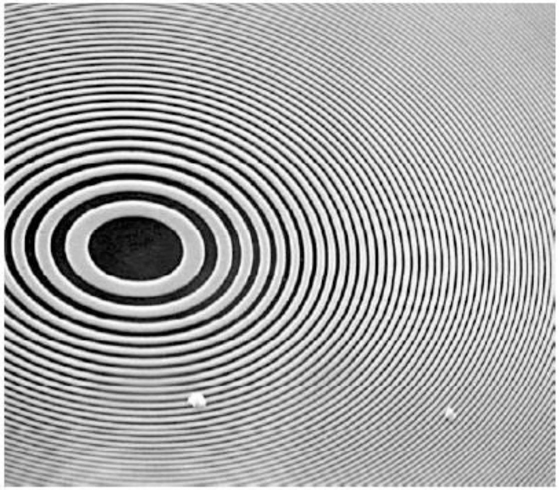
Versatility

- No sample preparation
- Time per measurement: s – min
- In situ
- Synchrotron radiation

Transmission X-ray Microscopy (PEEM)

Principle

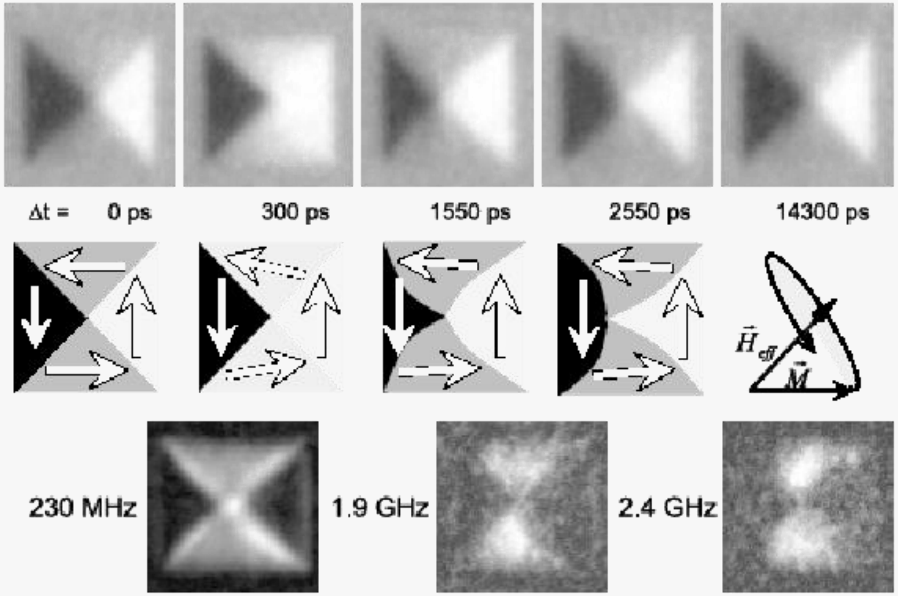




Zone plate

P. Fischer, Front. Phys. 2 (2015)

Example



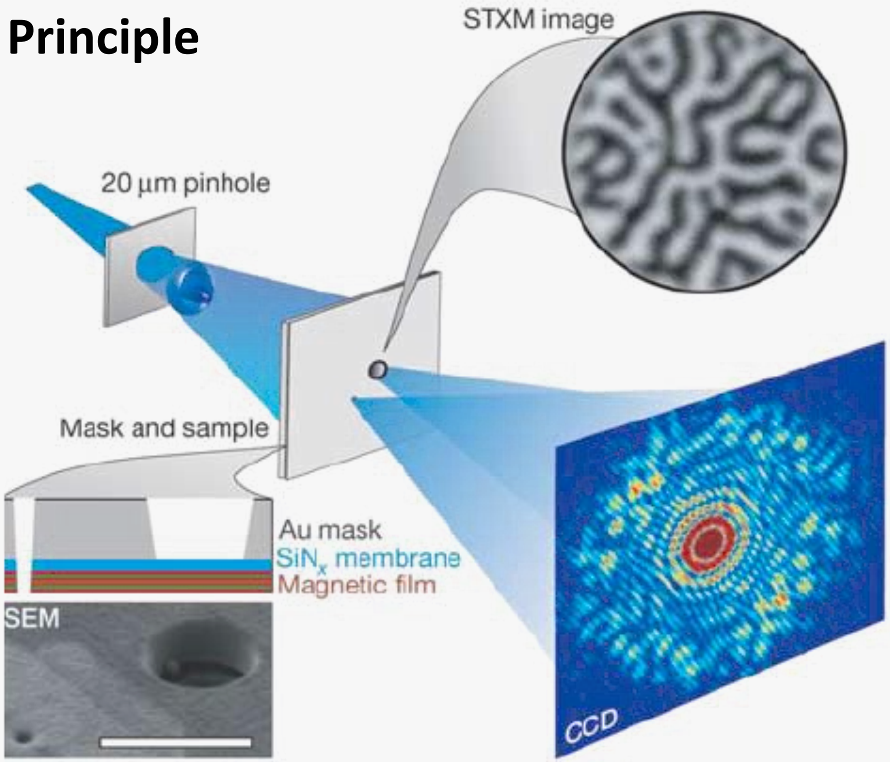
FeNi square dot, 6µm.
Stroboscopic time resolution

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

- Compatible with magnetic field
- Ongoing development of tomography

X-ray magnetic dichroism – Holography

Principle

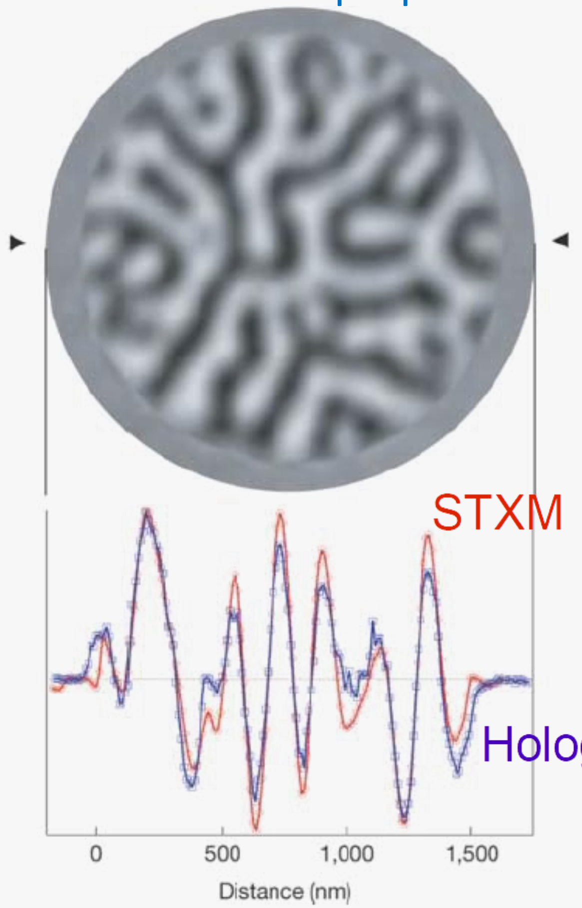


S. Eisebitt et al., Nature 332, 885 (2004)

- Lens-less imaging
- Requires microfabrication

Example

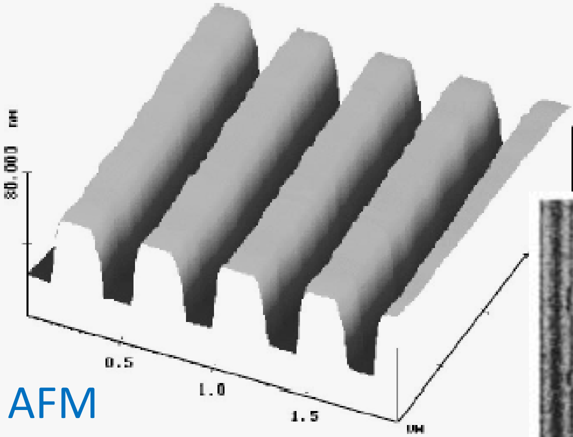
[Co(4Å)/Pt(7Å)]50 with perpendicular magnetization



X-ray magnetic dichroism – Scattering

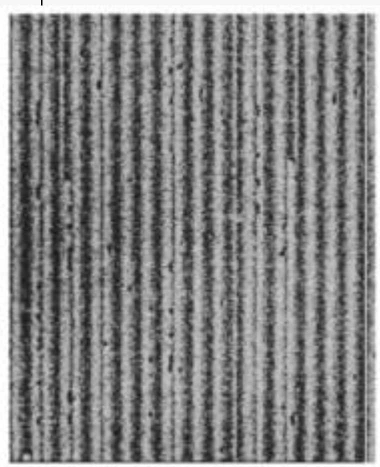
Principle

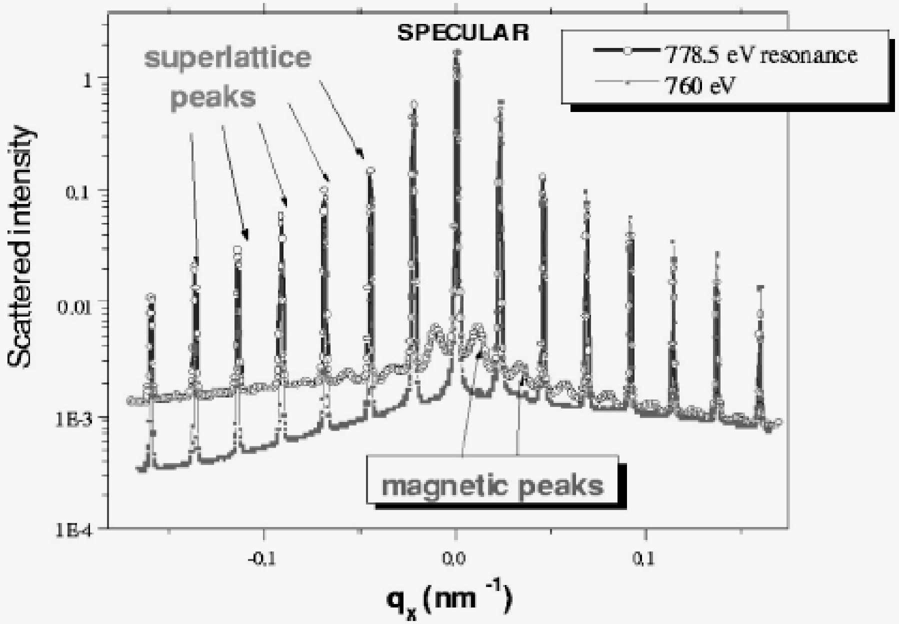
Perpendicularly-magnetized stripes, 250nm wide



AFM

MFM





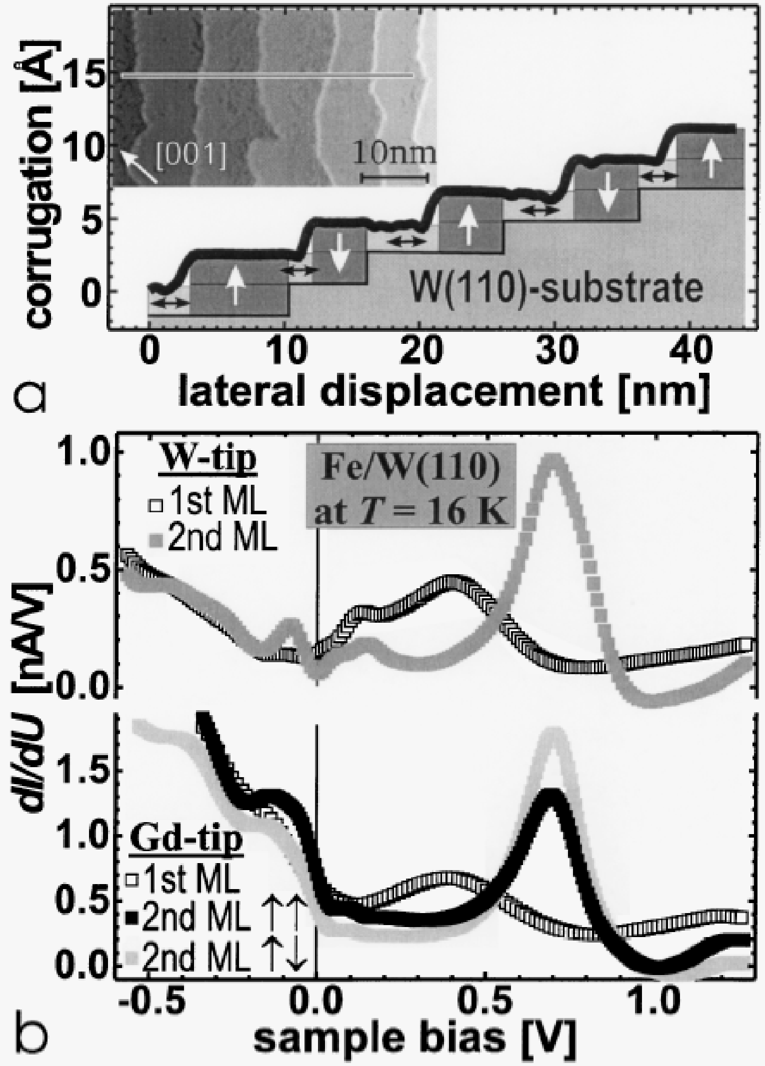
K. Chesnel et al., Phys. Rev. B 66, 024435 (2002)

Off-specular reflectivity to probe lateral order

- Lens-less spatial information
- Another important geometry: reflectivity for in-depth profiles

- Criteria for measurement techniques
- Probing magnetic stray fields
- Techniques with light-matter interaction
- **Techniques with electron-matter interaction**

Principle



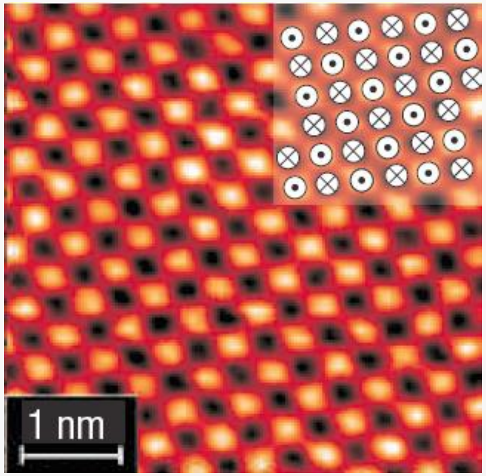
Spin-dependent spectroscopy

O. Pietzsch et al., PRL 84, 5212-5215 (2000)

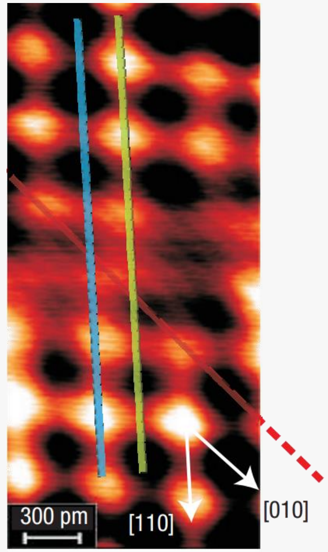
Spin-Polarized STM

Ultimate spatial resolution

Antiferromagnetic Fe/W(001)



Antiferromagnetic domain

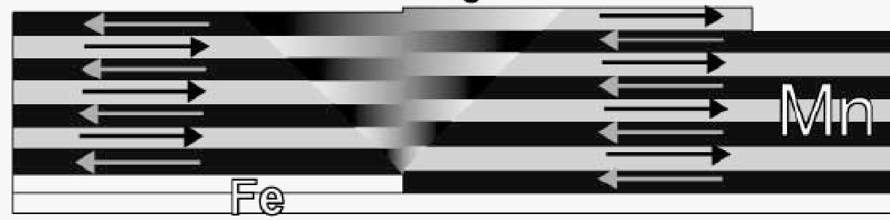


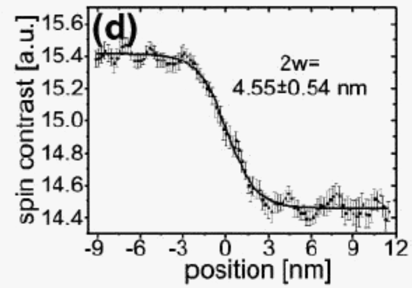
Antiferro. Domain wall

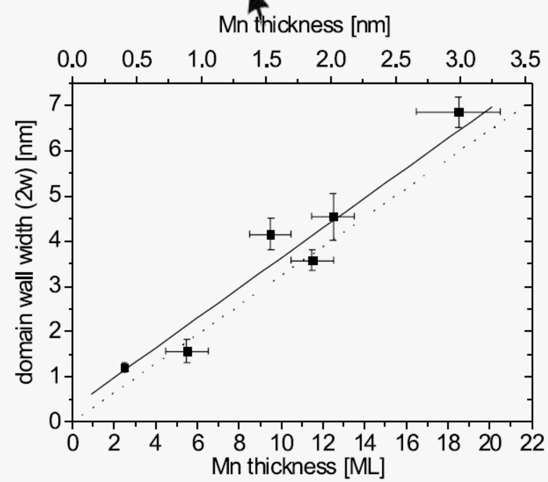
M. Bode et al., Nat. Mater. 5, 477-481 (2006)

Frustration at ferro/antiferro interface

Mn/Fe(001)







U. Schlickum, Phys. Rev. Lett. 92, 107203 (2004)

Spin-polarized STM – Criteria

What is measured?

- Related to magnetization and element
- Indirect?
- Not quantitative

Environmental conditions

- Low temperature only
- Field: magnetic field
- Light

Which specifications?

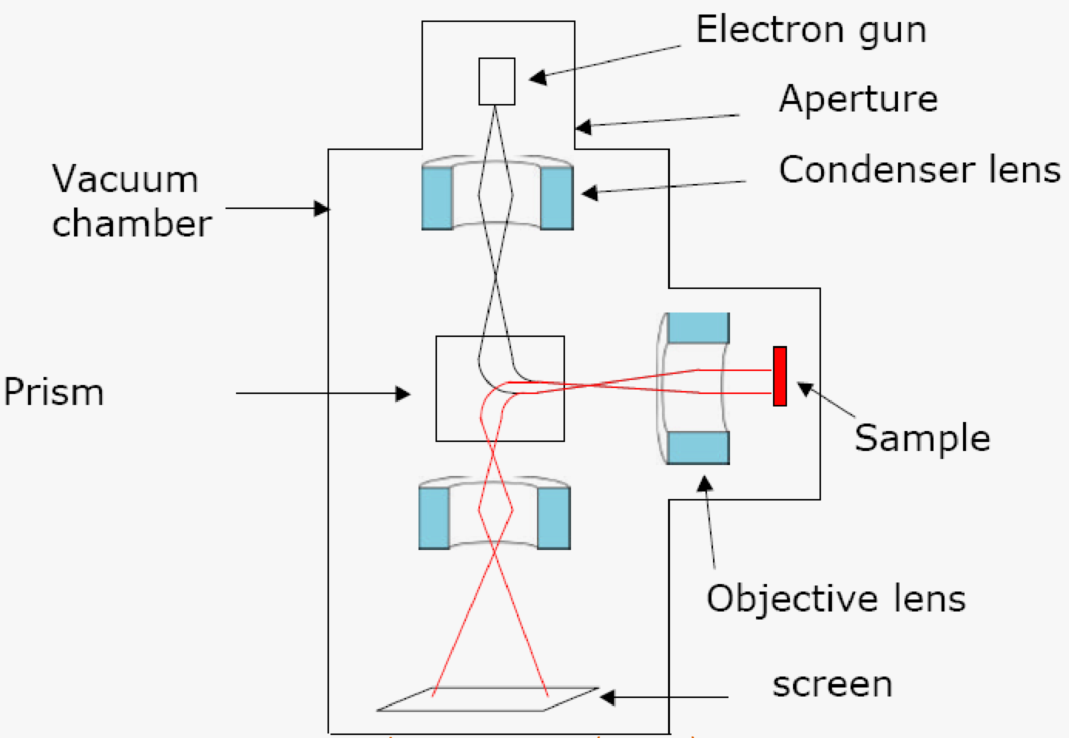
- Magnetization: 3D
- Depth resolution: surface.
- Lateral resolution: atom
- Sensitivity: high
- Time/Spectral resolution: emerging

Versatility

- Ultra-high vacuum and single crystal
- Time per measurement: minutes
- Mid-scale in-lab.

SPLEEM = Spin-Polarized Low-Energy Electron Microscopy (LEEM)

Principle of LEEM



E. Bauer, Rep. Prog. Phys 57, 895 (1994)

E. Bauer Surface Microscopy with Low Energy Electrons Springer, (2014)

Features

- Full-field (video rate)
- 5-10nm lateral resolution
- Resolution of atomic steps
- Some elemental or thickness
- Resolution through working energy
- High voltage column, however low energy electrons on sample

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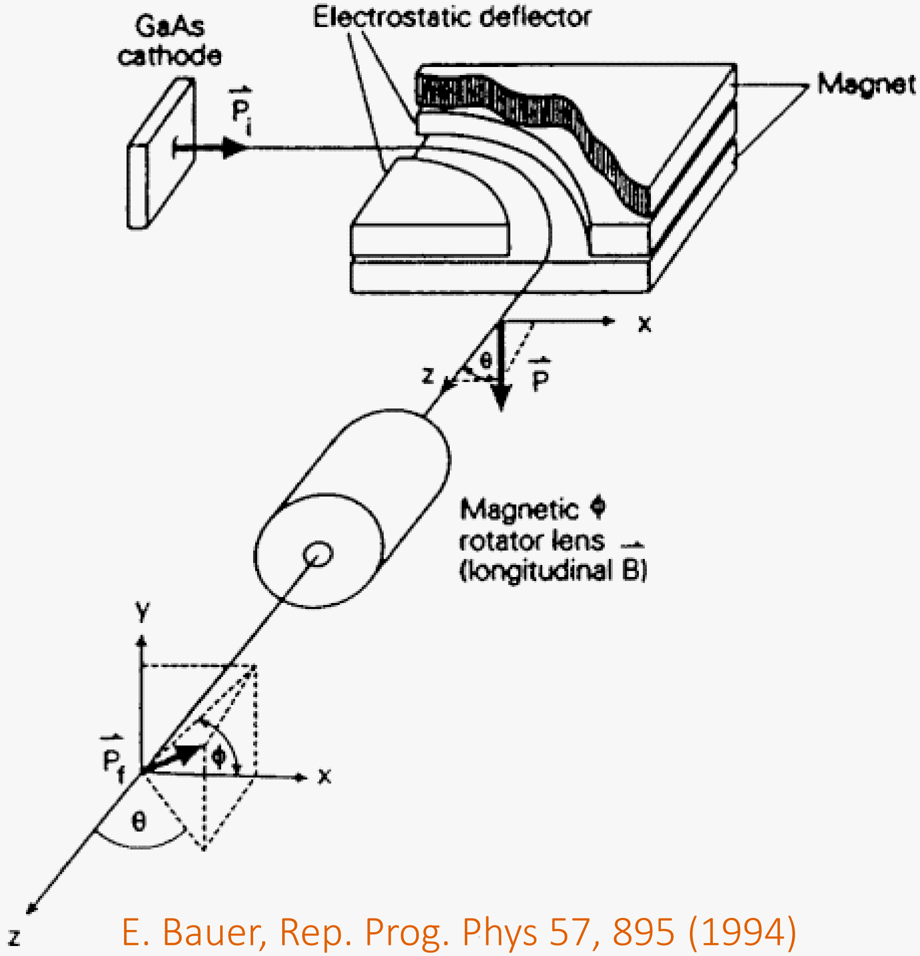
Electron spin polarization and control



E. Bauer, Rep. Prog. Phys 57, 895 (1994)

E. Bauer Surface Microscopy with Low Energy Electrons Springer, (2014)

Features

- Polarization over 80% achievable
- 3D manipulation of spin direction using combined magnetic/electrostatic optics
- 2D maps of magnetization with 3 components

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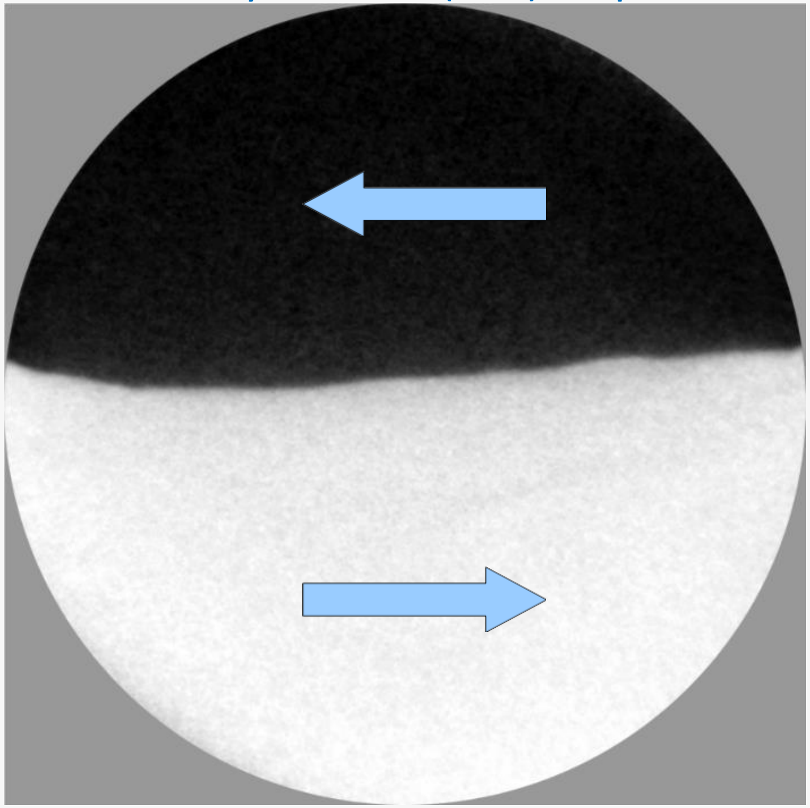
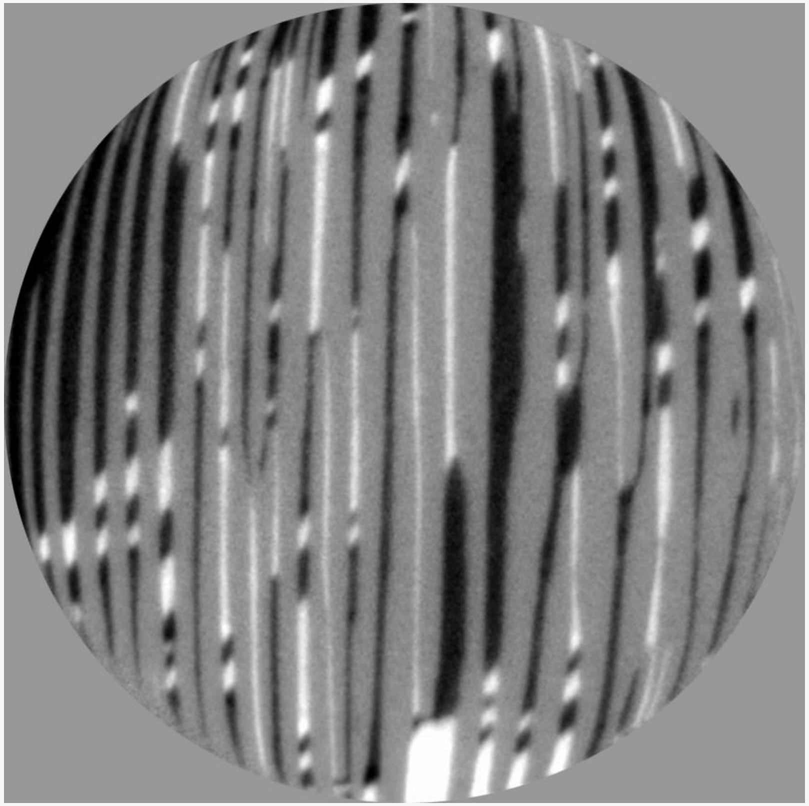
Example

14 atomic layers Fe/W(110), deposited RT



After annealing at 350°C



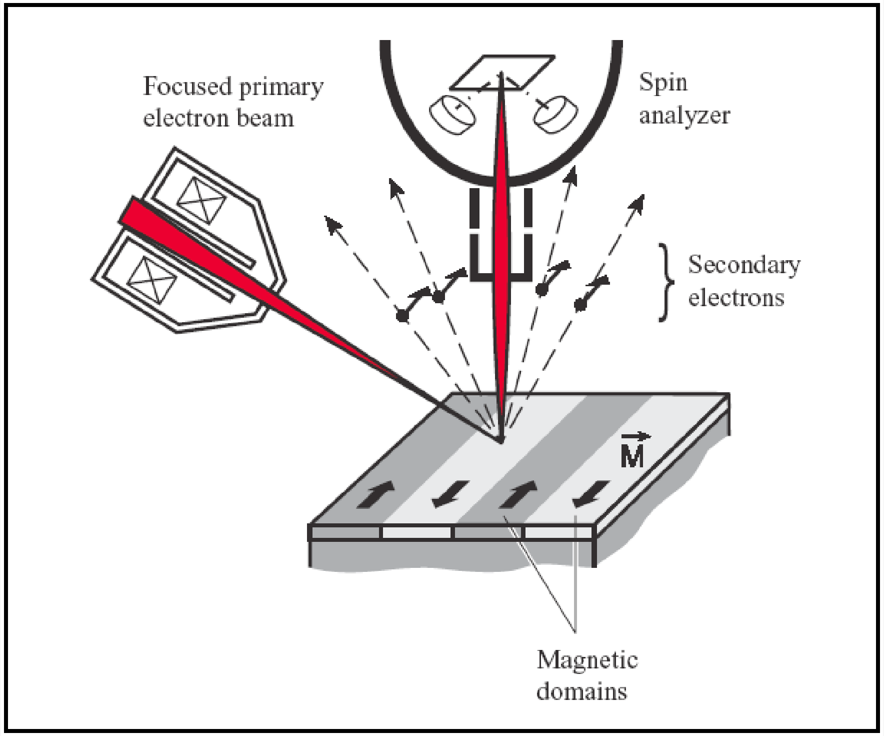
Field of view: 7 µm

N. Rougemaille & A. K. Schmid, J. Appl. Phys. 99, 08S502 (2006)

SEMPA or spin-SEM

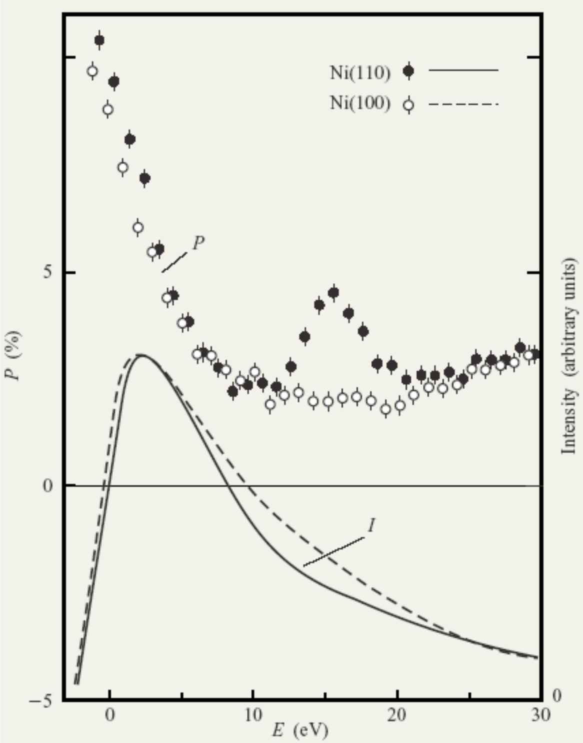
SEMPA = Scanning Electron Microscopy with Polarization Analysis

Principle



R. Allenspach, IBM J. Res. Develop. 44, 553 (2000)

Polarization of secondary electrons



Max values
Fe: 50%
Co: 35%
Ni: 10%

Spin detectors

- Mott detector: low efficiency
- LEED detector (W(001) etc.): high efficiency

SPLEEM – Criteria

What is measured?

- Magnetization
- Spectroscopy features
- Indirect, related to spin reflectance
- Fairly quantitative

Environmental conditions

- Temperature
- Field: no magnetic field, electric current

Which specifications?

- Magnetization: 2D map of 3D vector
- Depth resolution: surface.
- Lateral resolution: 10nm
- High sensitivity
- Time/Spectral resolution: compatible

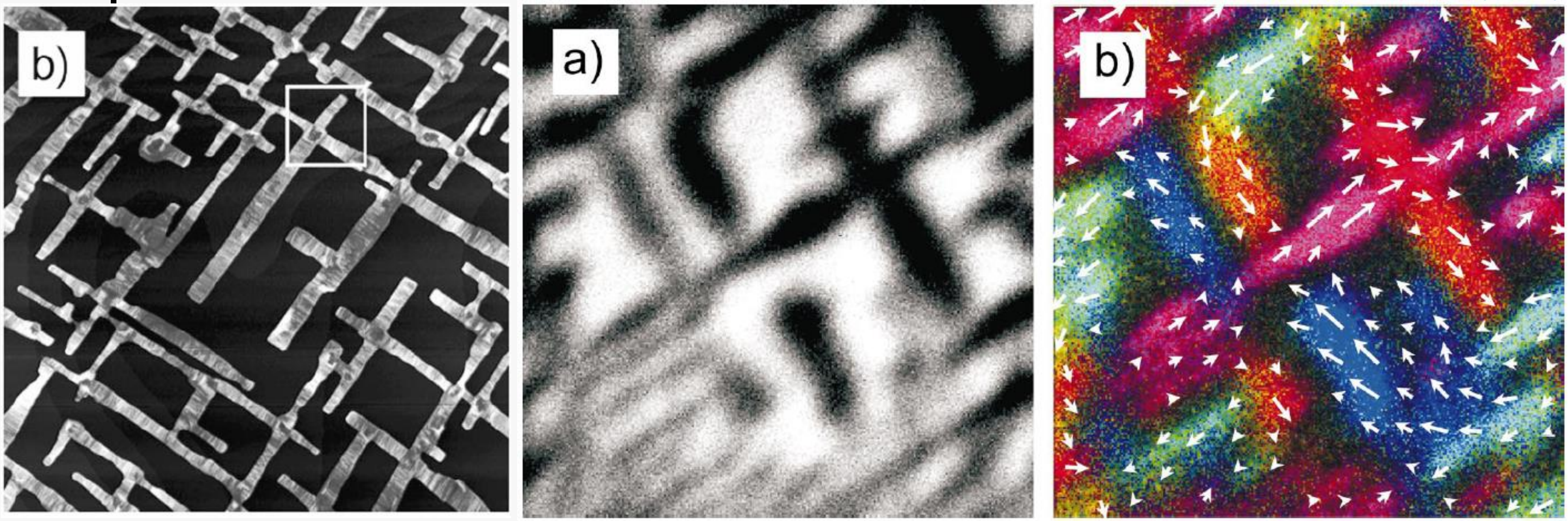
Versatility

- Ultra-high vacuum
- Single-crystalline surface
- Time per one measurement
- Mid-scale in-lab?

SEMPA or spin-SEM

Example

Fe/W(001)



Field of view: 1.5 µm

W. Wulfhekel et al., Phys. Rev. B 68, 144416 (2003)

Features

- Potential high spatial resolution (5nm)
- Surface sensitive (<1nm)
- Low rate (scanning, efficiency of Mott detector)
- Hardly compatible with magnetic field

SEMPA – Criteria

What is measured?

- Magnetization
- Basic spectroscopy
- Direct
- Quantitative

Environmental conditions

- Temperature
- Field: magnetic field, electric
- Electric current, light etc.

Which specifications?

- Magnetization: 3D
- Depth resolution: surface.
- Lateral resolution
- Sensitivity
- Compatible with time resolution

Versatility

- Epitaxial sample required
- Time per measurement: 10min
- Ultra-High Vacuum
- In-lab however complex

Some magnetic microscopies with resolution <50nm

	Sp-STM	MFM	NV	BEMM	SEMPA	SPLEEM	TEM	XMCD -PEEM	XMCD-microscopy (Fresnel ZP)
Resolution	<1nm	15nm	5-10 nm	1-5nm	10nm	10nm	1-2nm	25nm → 10nm	15nm
Sensitivity	High	Med	High	Med	Med	High	Low	High	High
In-field	YES	Limited	Limited	YES	local	No?	Limited	No?	YES
Versatile*	No	YES	Yes	No	Yes	UHV	Limited	Yes	Limited
Dynamics	No	No	No	No	No	No	New	Yes	Yes
Element-sensitive	Limited	No	No	No	No	Limited	Limited	Yes	Yes
Probes	m_i	$\mathbf{H}_\mathrm{d}$	$\mathbf{H}_\mathrm{d}$	m_i	$\mathbf{m}$	$\mathbf{m}$	$m_{x,y}$	$m_\mathbf{k}$	$m_\mathbf{k}$

*Versatile may mean: Sample preparation, measurement of brought-in samples etc.

Conclusions

- Worse and best come together
- Need for combining various instruments

Combine different techniques...

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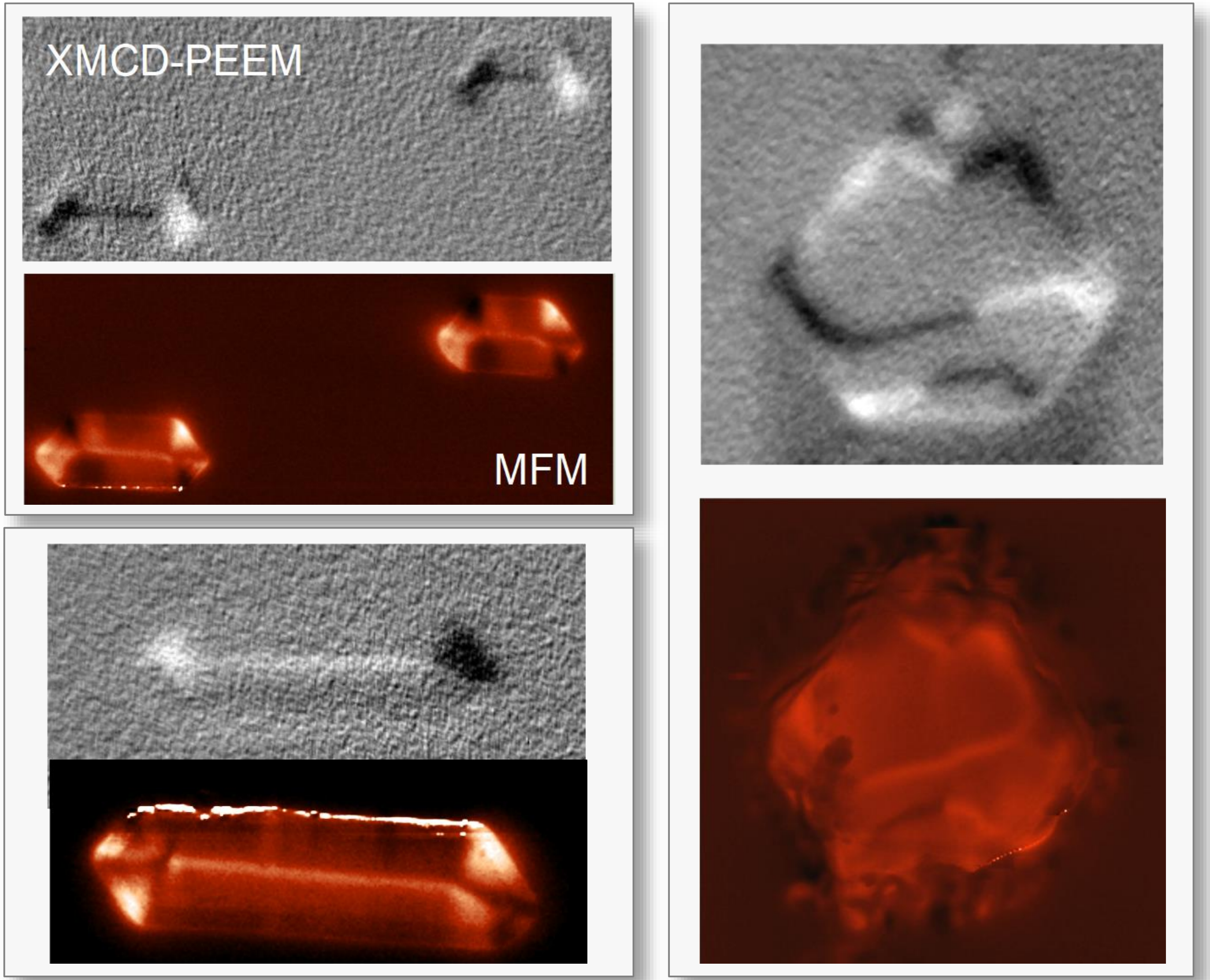
Self-assembled Fe(110) and Co(111) micron-sized dots



- Do you see the same?..

Suggested reading: lectures of Hans Hug (EMPA) at ESONN school: https://www.esonn.fr/2016-lectures

End of the general presentation

www.spintec.fr

email: olivier.fruchart@cea.fr

Slides: http://fruchart.eu/slides