Experimental techniques for the study of small magnetic objects

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http://neel.cnrs.fr

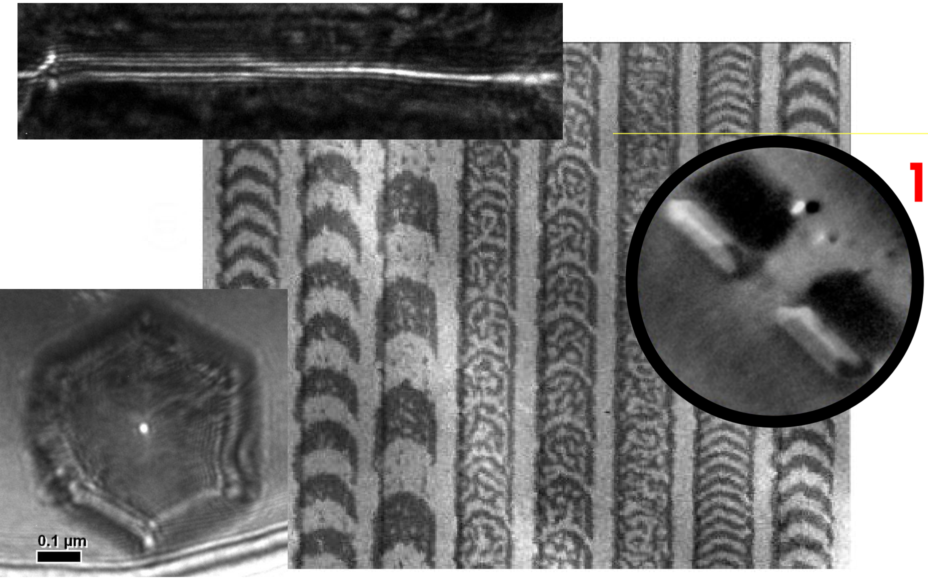
- Large overview of characterization techniques. Therefore remains handwavy
- Not all techniques currently used for nanoalloys (yet?)
- Personal views on prospects
- Experts in the audience: please interrupt any time
- Non-experts in the audience: please interrupt any time

Nano

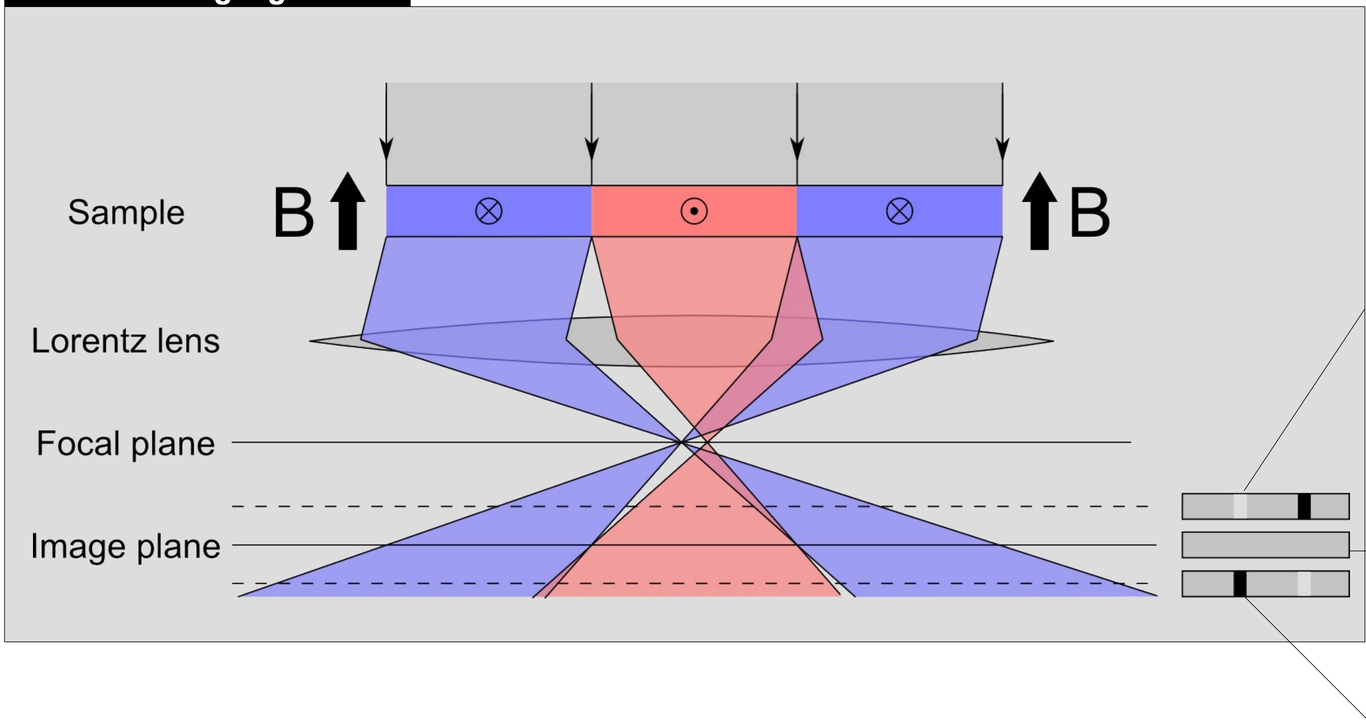
- Sensitivity → Restricted amount of material
- Spatial resolution → Single-object measurements
  → Characterize non-homogeneities
- Depth sensitivity → Relevance of measurements
  → Volume versus surface properties

Alloys

- Chemical sensitivity
- Spatial / depth resolution

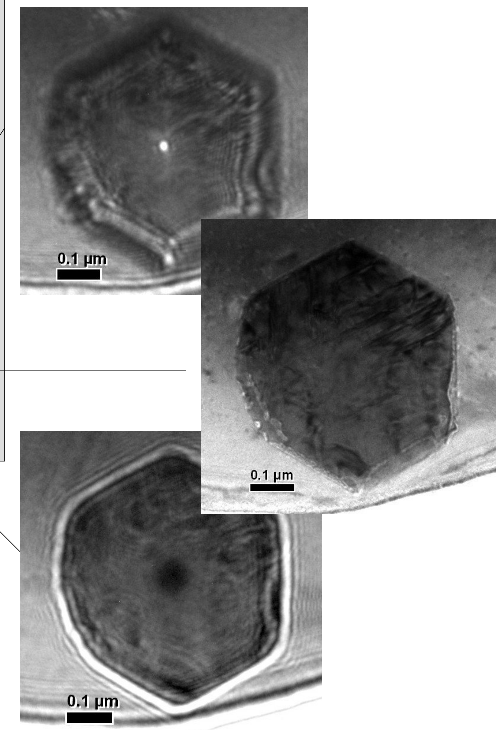

0.1 µm

Fresnel imaging mode



Self-assembled fcc Co dots

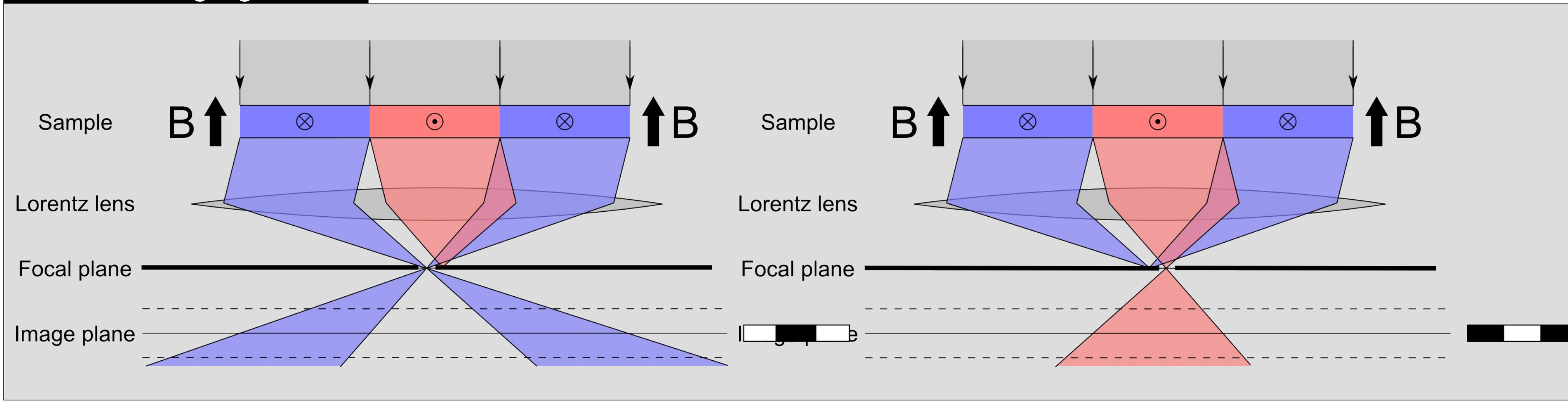
(vortex state)

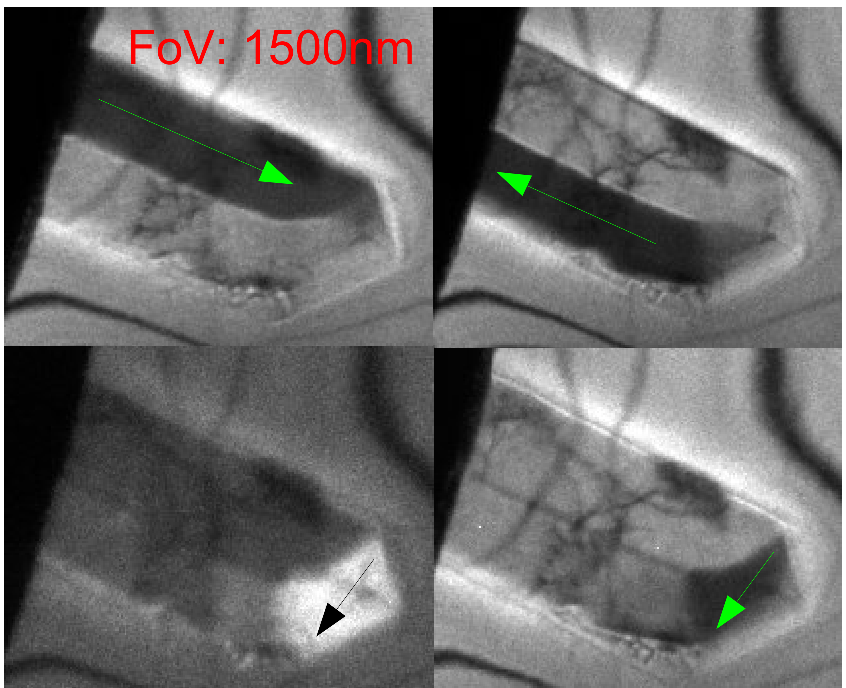


- Sensitive mainly to in-plane components of induction integrated over the sample's + air thickness
- Down to 5nm lateral resolution
- Fresnel: fine resolution of domain walls and vortices

Collaboration:
P. Bayle-Guillemaud,
A. Masseboeuf
(CEA - Grenoble)

Foucault imaging mode





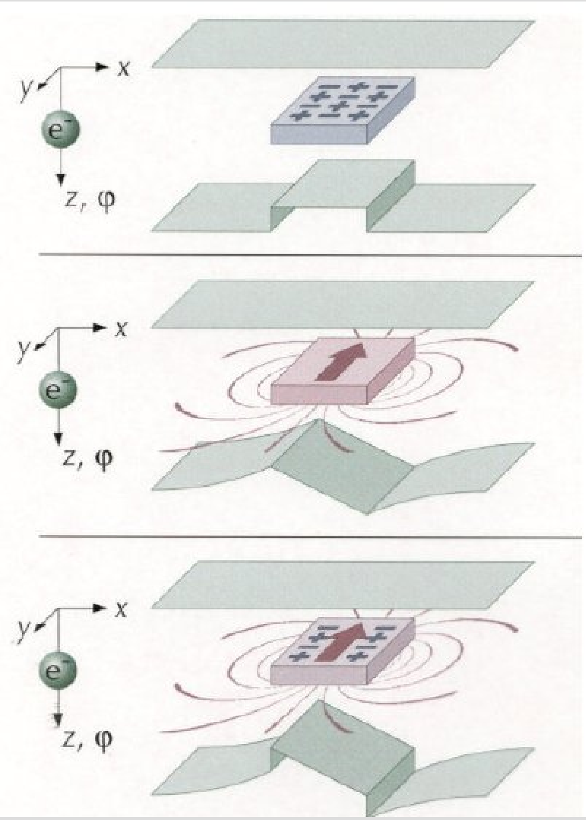
- Foucault: highlights domains
- 2D/3D vectorial maps of induction can be computed (also possible from series of focus

Collaboration:
P. Bayle-Guillemaud, A. Masseboeuf
(CEA - Grenoble)

Lorentz microscopy - Holography

Working principle

Phase-shift of electrons through matter



Phase shift due to the electrostatic potential

$$\boxed{\varphi_{elect}(\mathrm{x}) = \mathrm{C_E} \int V(x,z)dz}$$

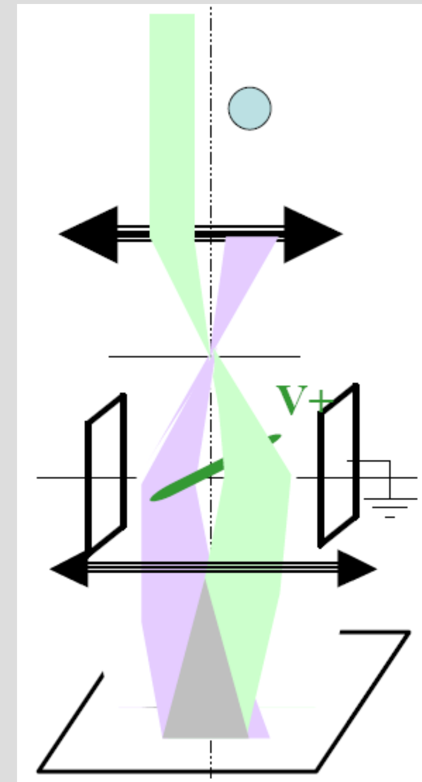
Phase shift due to the magnetic induction

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

Total phase shift

$$\varphi_{Tot}(\mathrm{x}) = \varphi_{elect}(\mathrm{x}) + \varphi_{mag}(\mathrm{x})$$

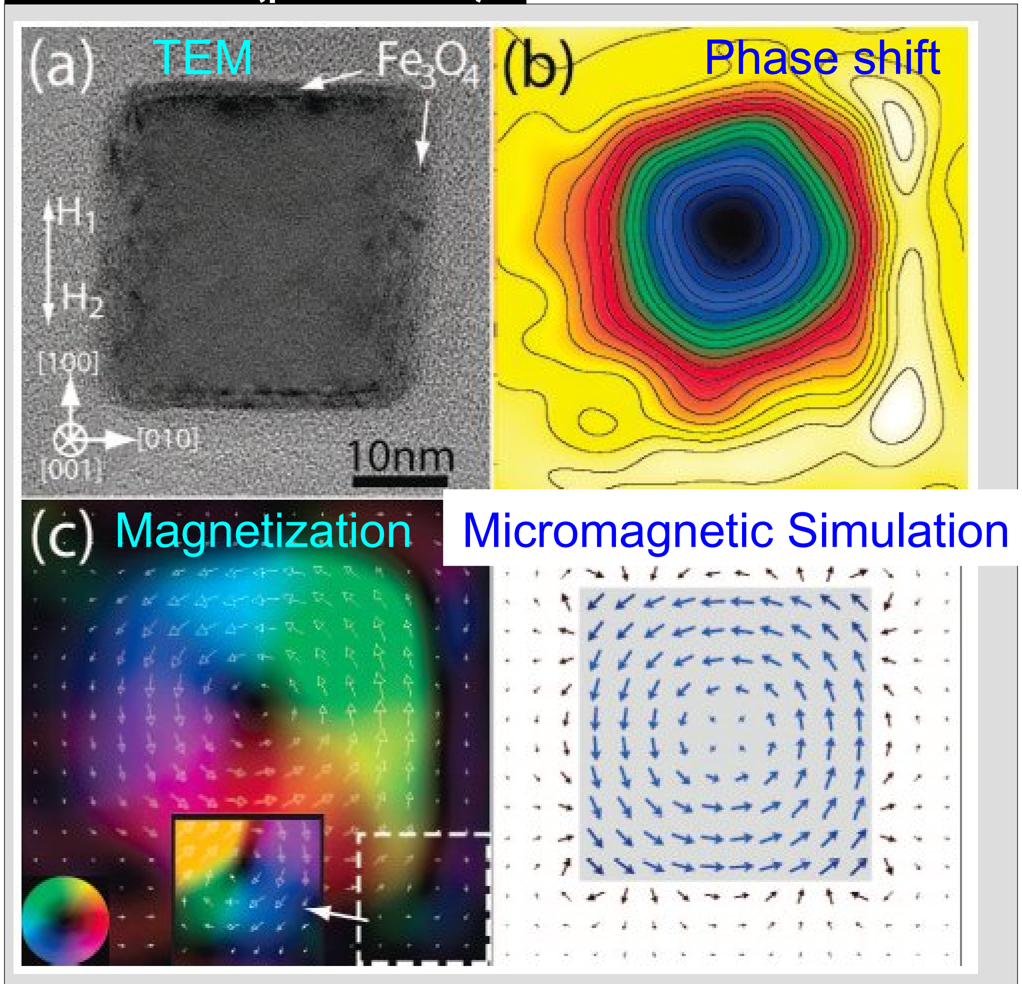
Setup



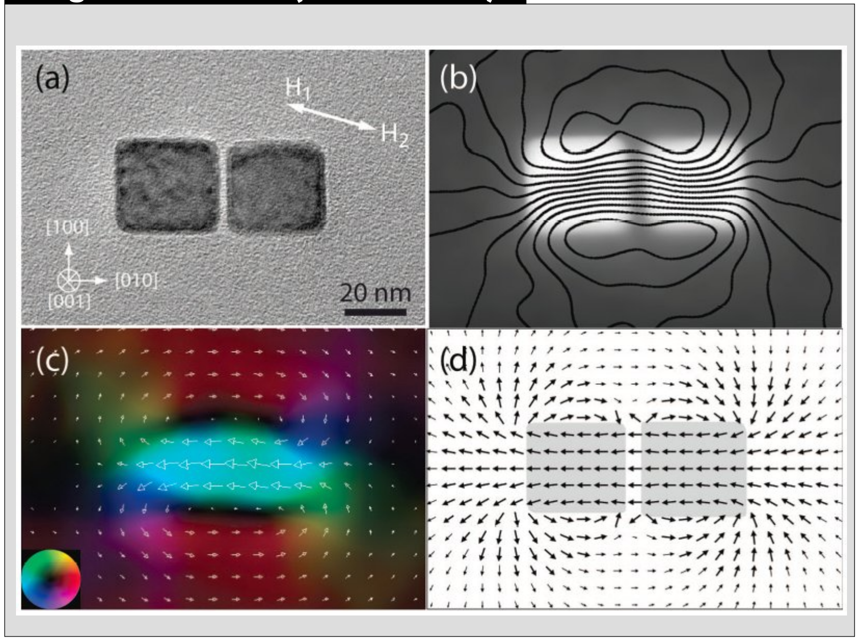
- Computational procedure to retrieve the integrated map of induction
- Compensation measurement needed to get rid of electrical contribution

Courtesy: B. Warot-Fonrose, CEMES-Toulouse

Vortex state (plane view)



Single-domain (side view)



Notice (both cases):

Probes magnetization AND stray/internal fields

E. Snoeck et al., Nano letters 8 (12), 4293 (2008)

Features

- Ultimate lateral resolution ~5nm
- Minimum thickness: a few nm (best: holography) to ten nm

- Rate (video).
- Preparation (spreading, thinning) but versatile ('ex situ')

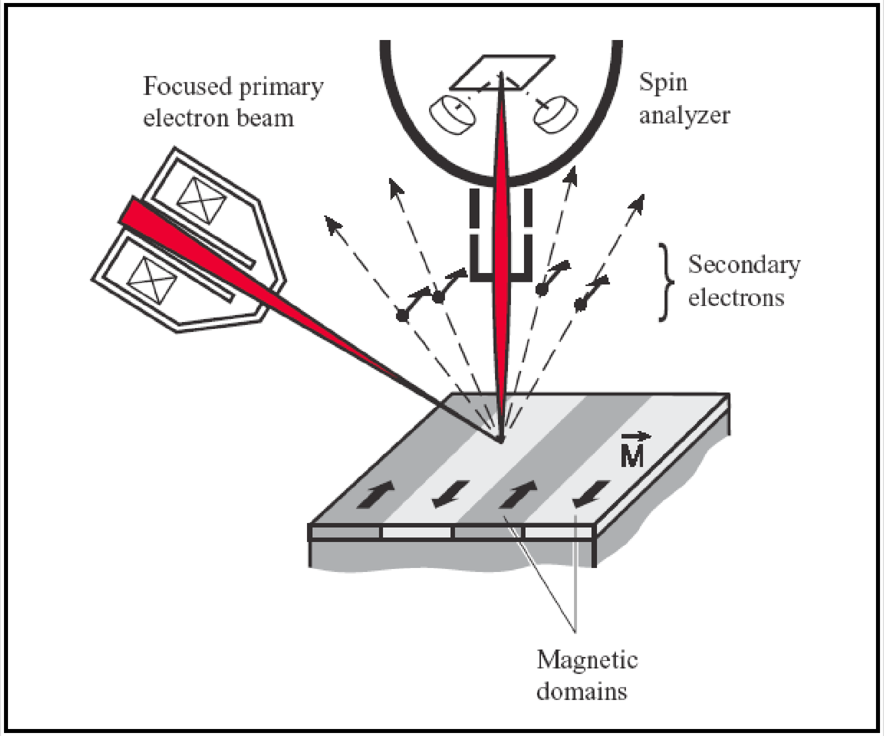
- Sensitive to total induction: magnetization AND demagnetizing / stray fields
- Probes the two in-plane components of induction; tiltable sample holder.
- Field applied through dedicated Lorentz lens: mainly perpendicular

Trends / future?

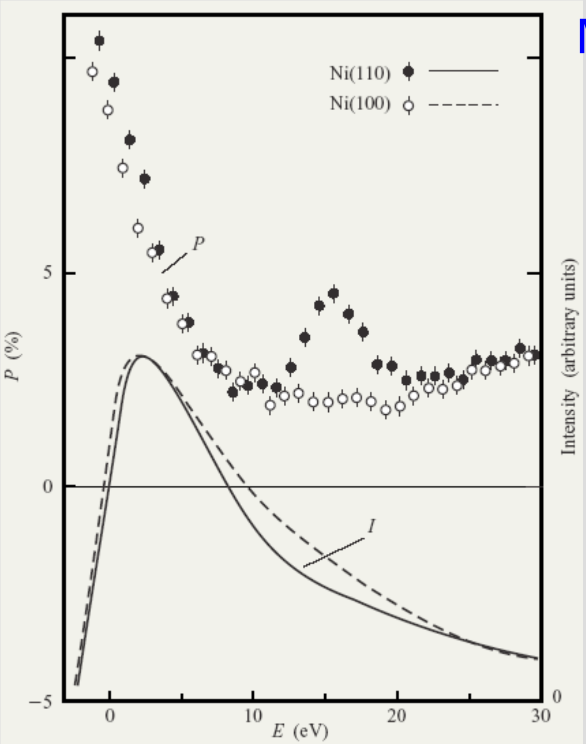
- Micromagnetic simulations are required to fully benefit from the resolution
- More combined HREM / Lorentz studies
- 'Environmental': moderate stand-alone external field; electrical connections etc.
- Magnetization dynamics

SEMPA = Scanning Electron Microscope with Polarization Analysis

Setup



Polarization of secondary electrons



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

Spin detectors

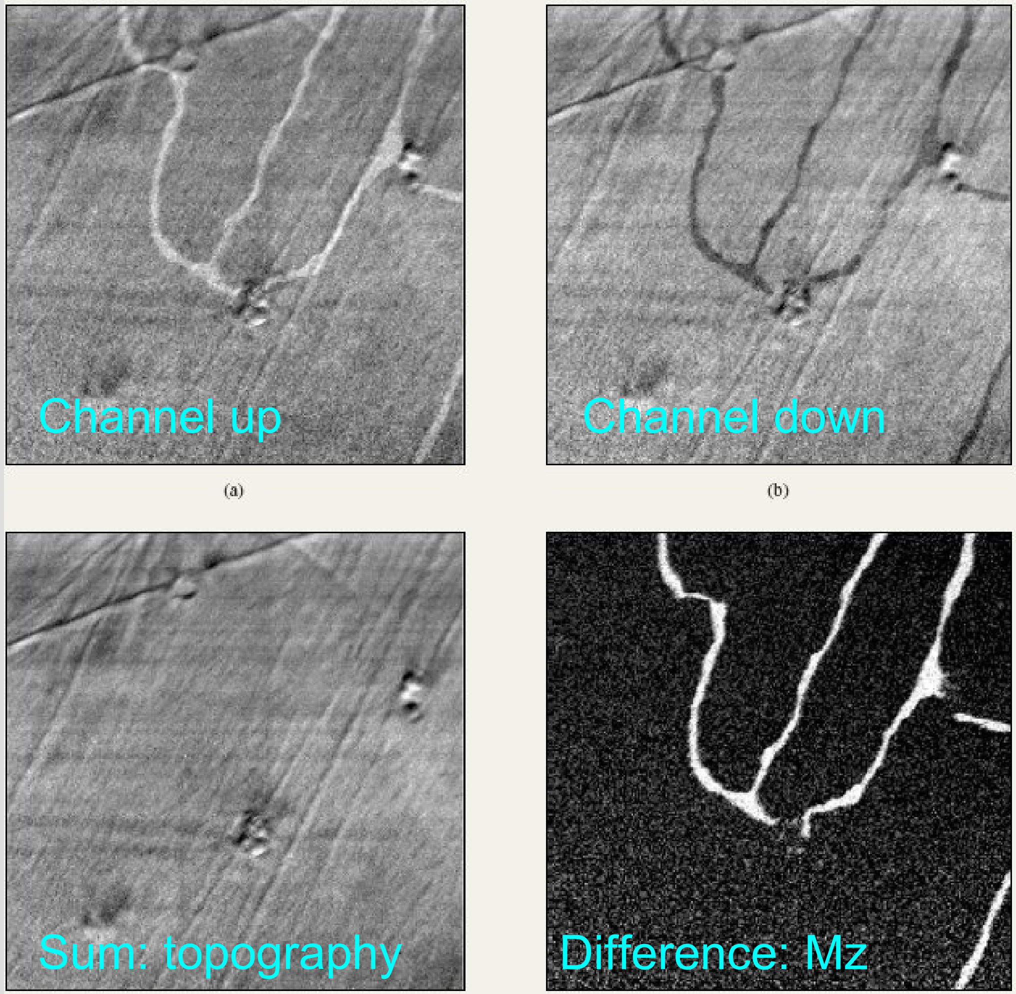
General feature: low efficiency or low energy window

⇨ Mott detector, LEED detector (W(001), Low-energy diffuse scattering

R. Allenspach, Spin-polarized scanning electron microscopy, IBM J. Res. Develop. 44, 553 (2000)

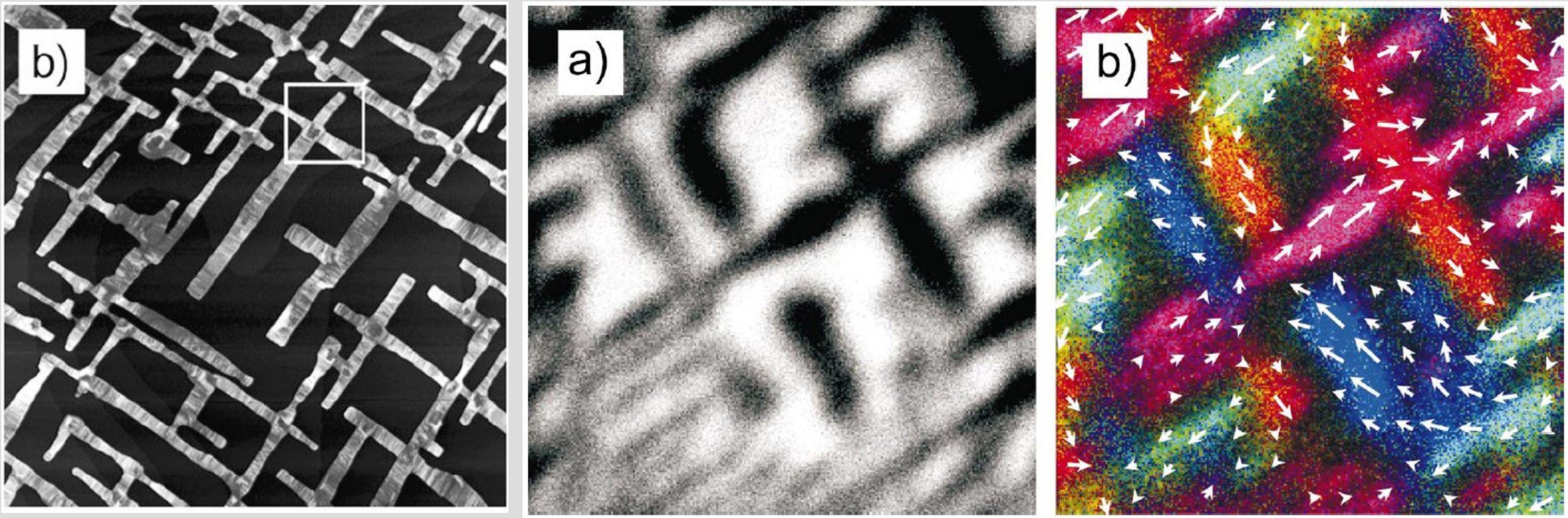
Reconstruction of magnetization map

Ex: 3ML Fe/Cu(001)



R. Allenspach, Spin-polarized scanning electron microscopy, IBM J. Res. Develop. 44, 553 (2000)

Example: Fe/W(001)



Fields of view: 1.5 μm

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

Features

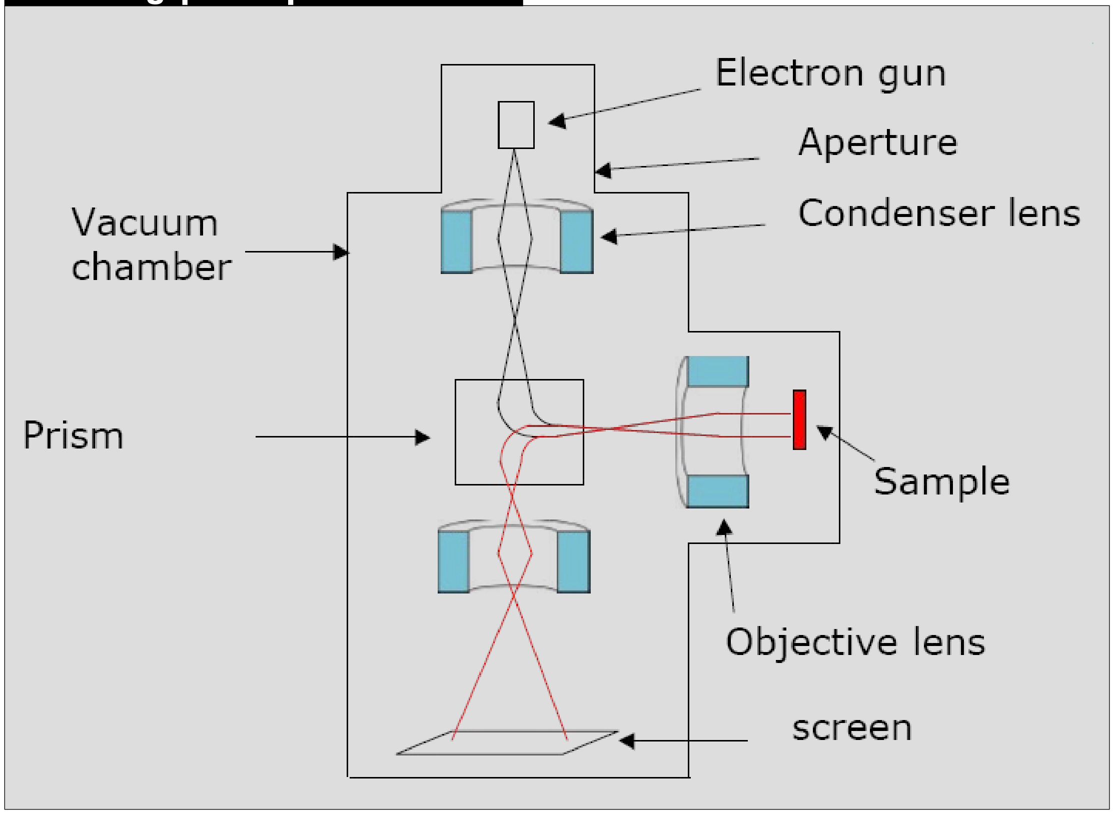
- Potential lateral resolution ~ 5nm
- Surface sensitive (<1nm)
- Low rate (scanning, efficiency of spin detectors)
- External field require special setups

Prospects

- More in-field setups
- See improved spin detectors
- Use energy filtering for elemental sensitivity

SPLEEM = Spin-Polarized Low-Energy Electron Microscope
= Spin-Polarized LEEM

Working principle of LEEM



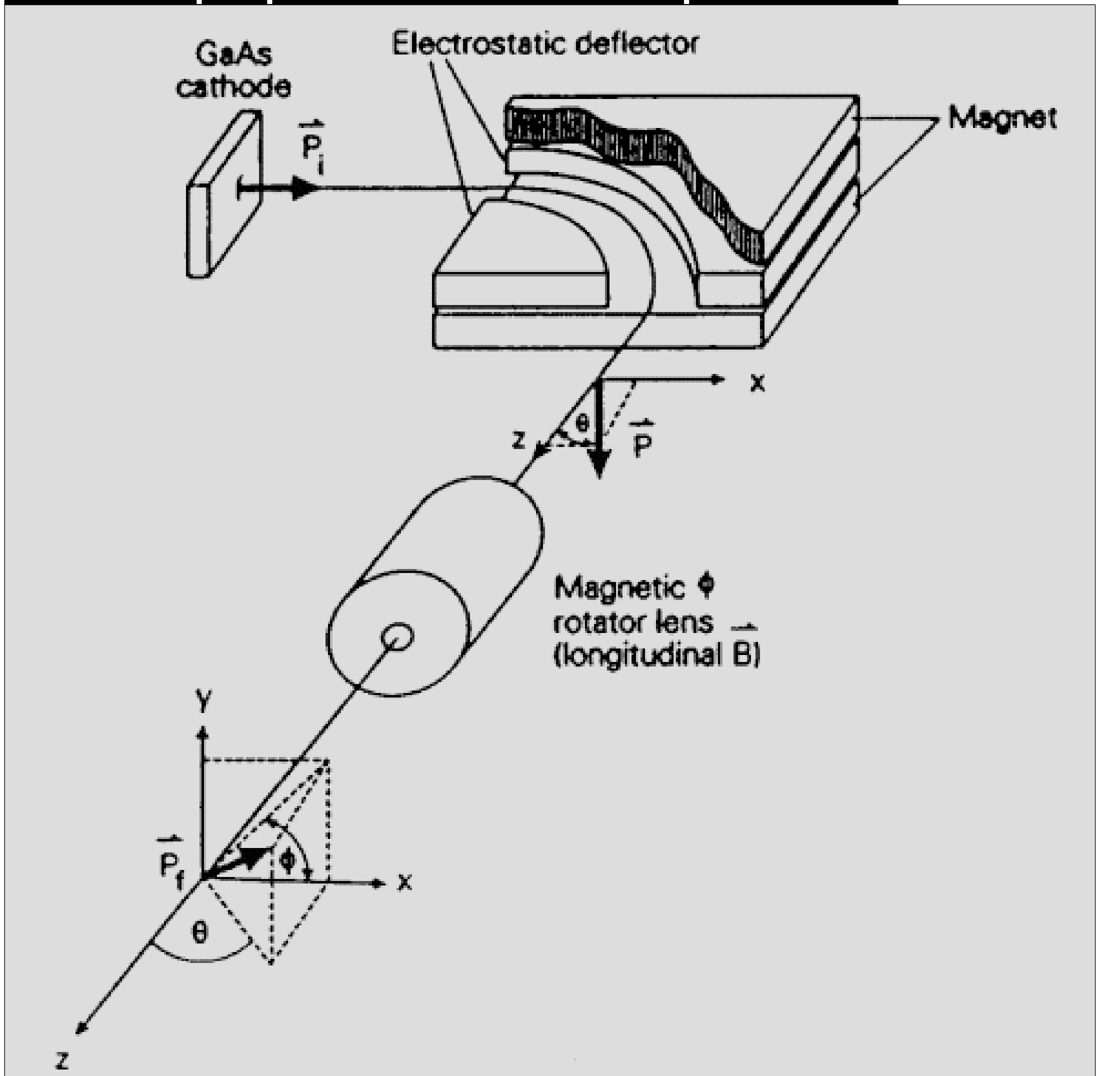
Features

- Full-field (video imaging 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

See: growth of Fe/W(110)

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

Electron spin polarization and manipulation

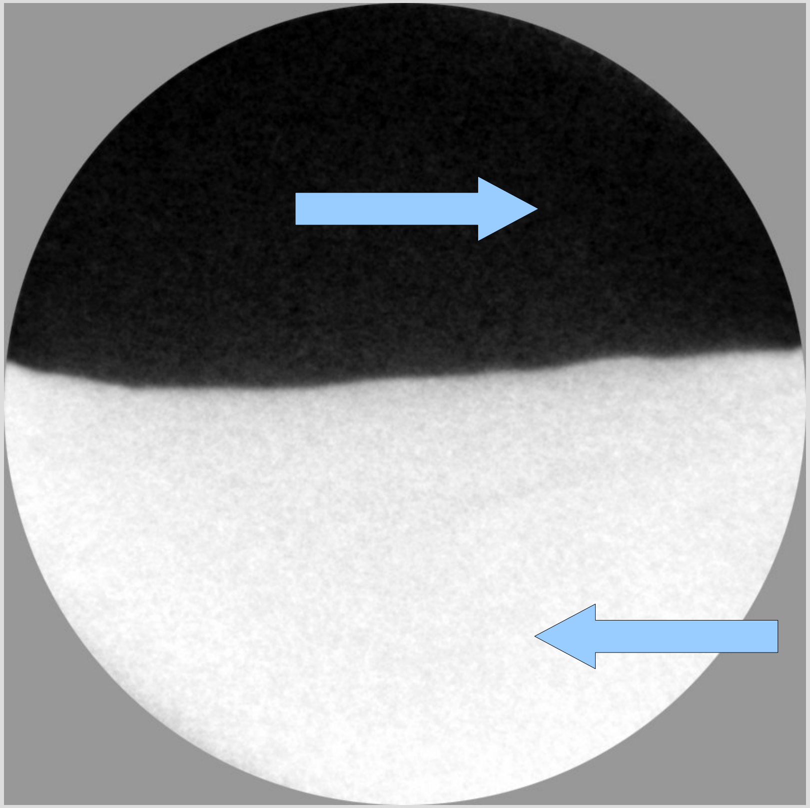


Features

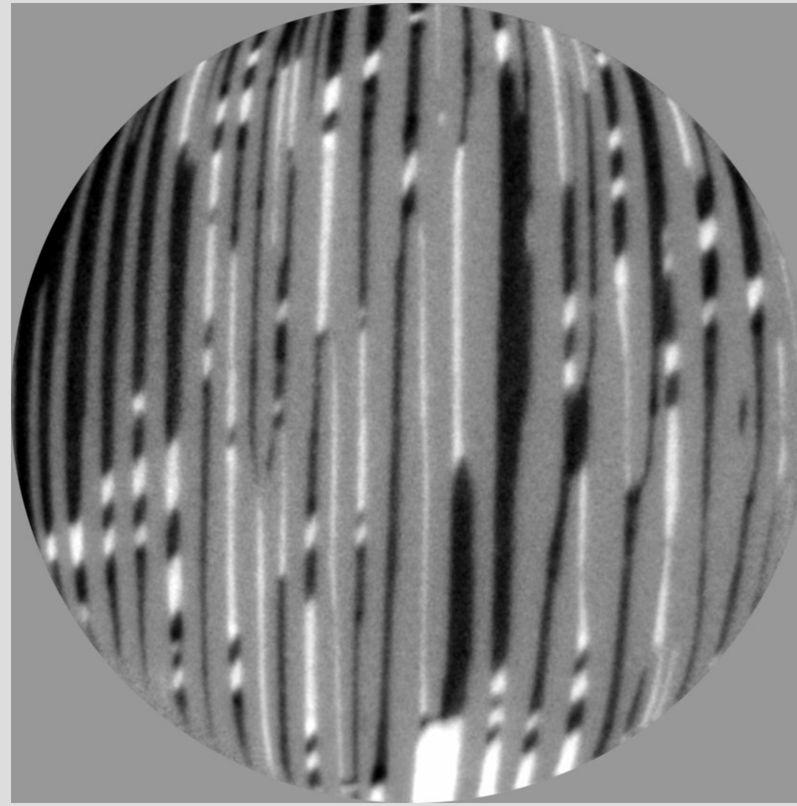
- Polarization over 80% achievable
- 3D manipulation of spin direction using combined magnetic/electrostatic optics
- 2D maps of magnetization with 3 components

SPLEEM: example

14MLFe/W(110), deposited at RT



After annealing at 350°C



Field of view: 7 μm

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

Features

- UHV technique
- Full-field (video imaging rate)

- 5-10nm lateral resolution
- Vertical resolution: atomic steps

- Surface sensitivity
- 2D maps of magnetization with 3 components
- Some elemental or thickness resolution through working energy

- No applied field

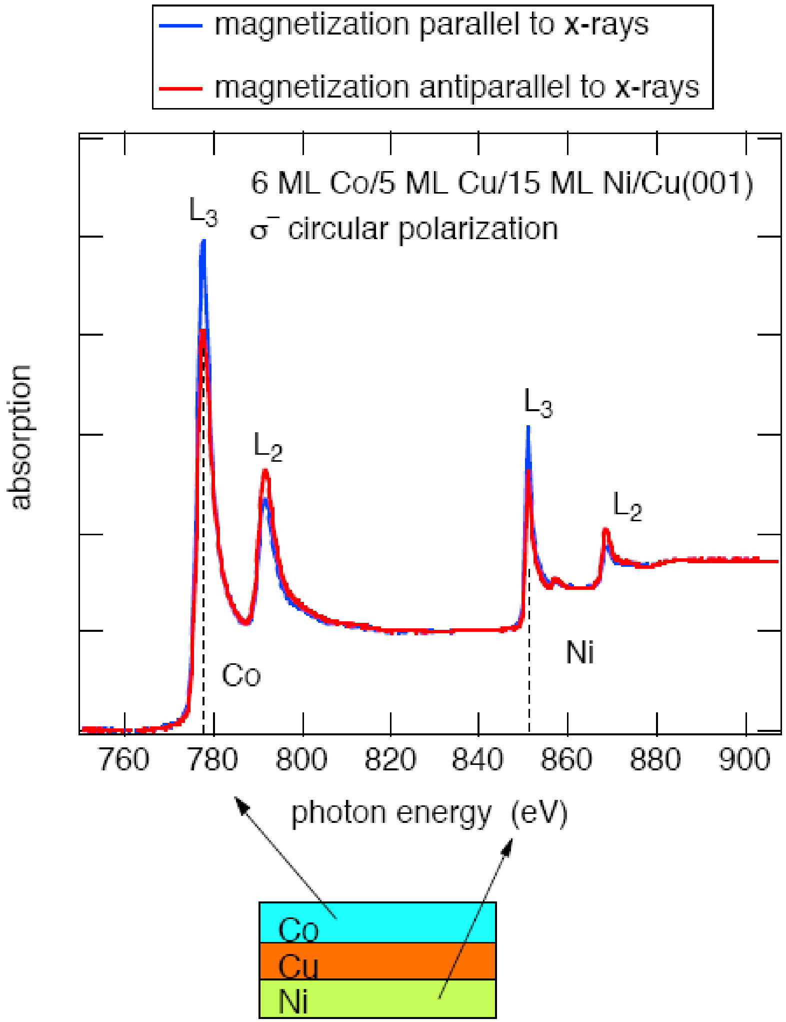
Prospects / future / dreams

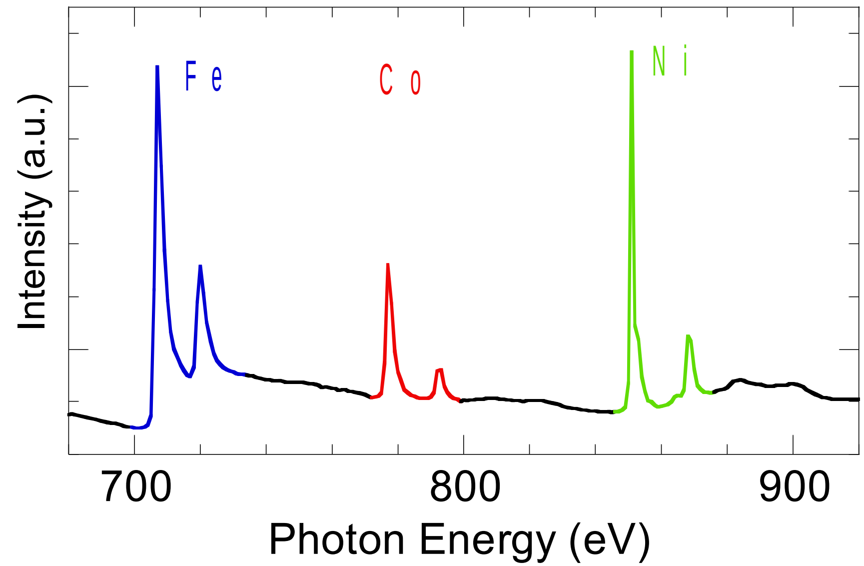
- Use dark-field imaging for magnetism?
- Investigate samples from ex situ?

XMCD magnetometry

XMCD = X-ray Magnetic Circular Dichroism

XMLD = X-ray Magnetic Linear Dichroism

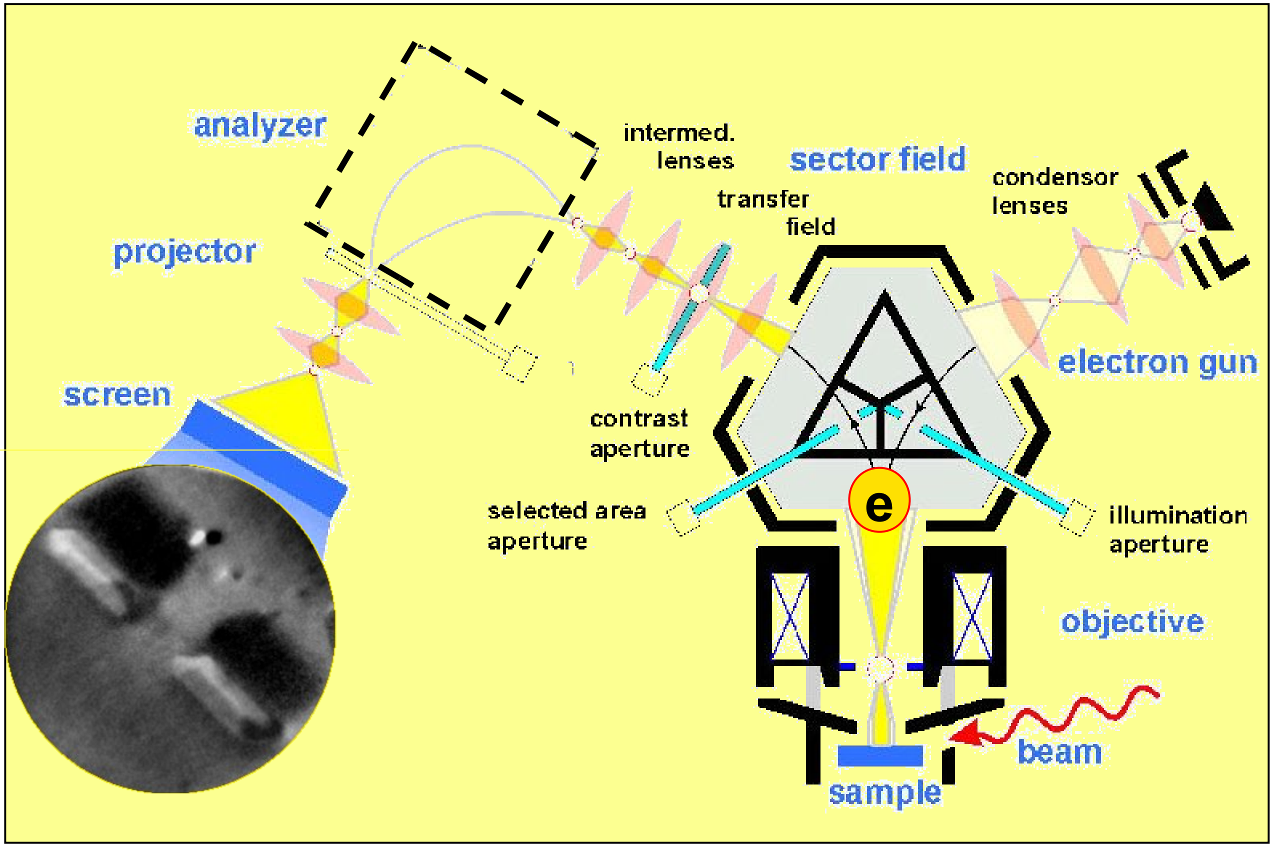


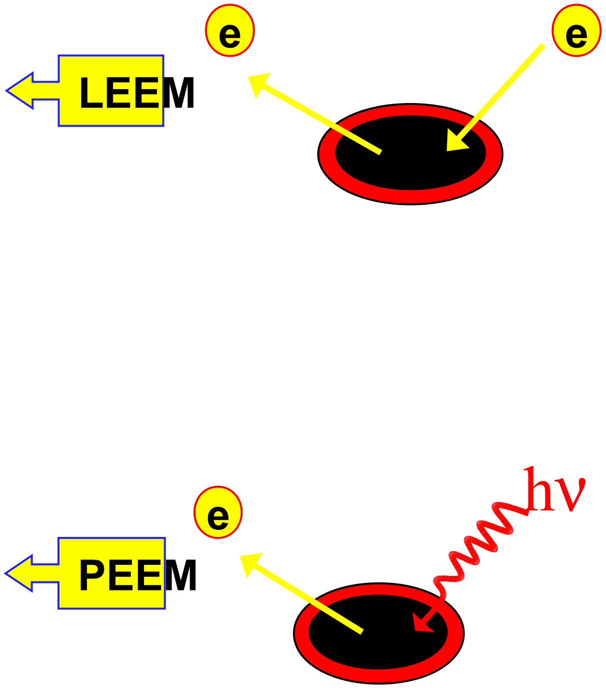


Courtesy: W. Kuch

XMCD-PEEM: X-ray Magnetic Circular Dichroism PhotoElectron Emission Microscope

Based on LEEM instrument: Low-Energy Electron Microscope

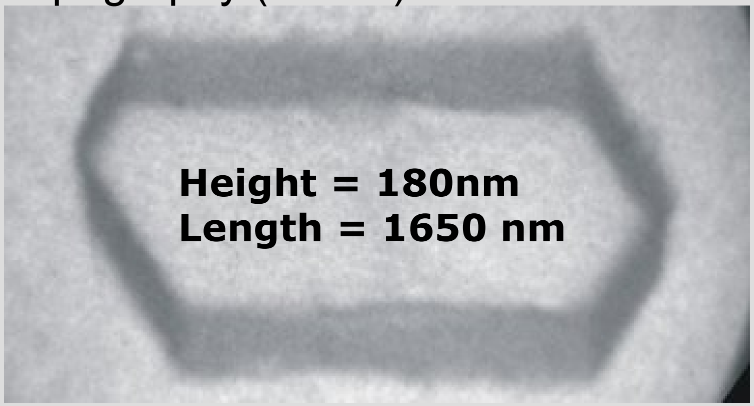




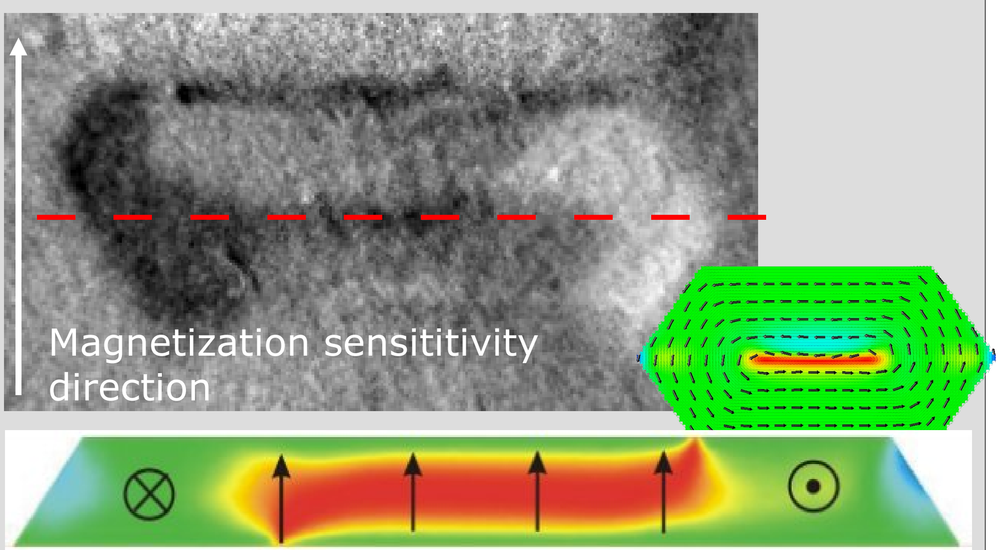
Note: simpler XMCD-PEEM instruments exist, not based on dual LEEM/PEEM.

Surface sensitivity

Topography (LEEM)



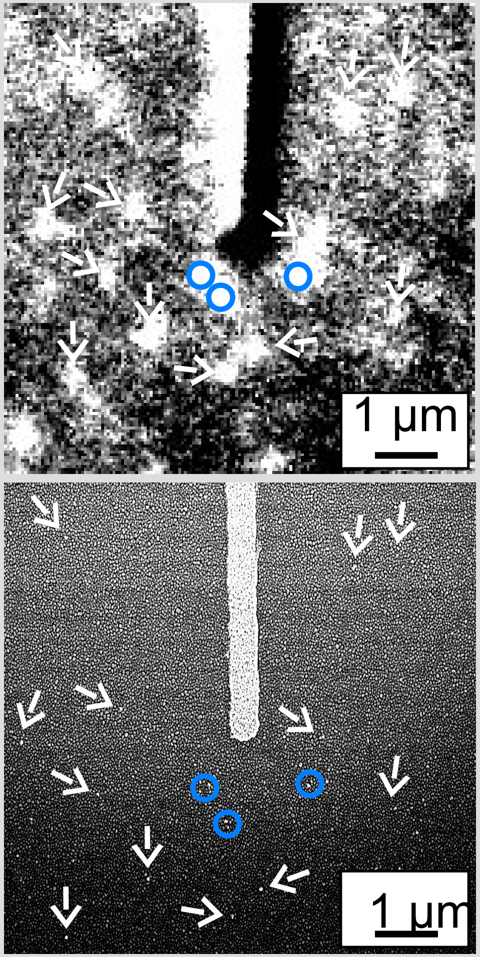
Surface magnetization (XMCD-PEEM)



R. Hertel et al., Phys. Rev. B 72, 214409 (2005)

For nanoparticles?

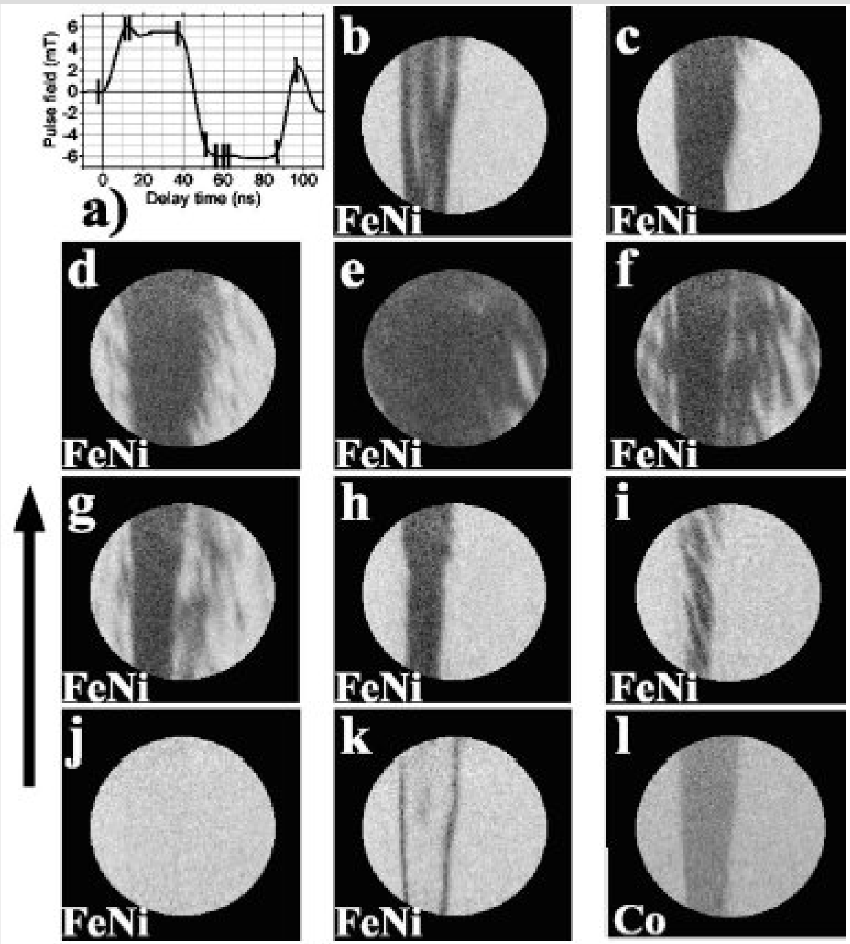
Co, diameter 8nm



A. Fraile Rodriguez, JMMM316, 426 (2007)

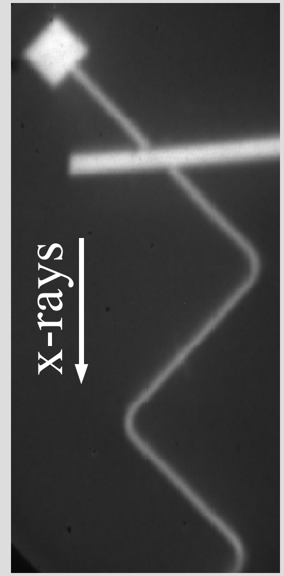
Field-driven magnetization reversal

FeNi/Al2O3/Co spin valves

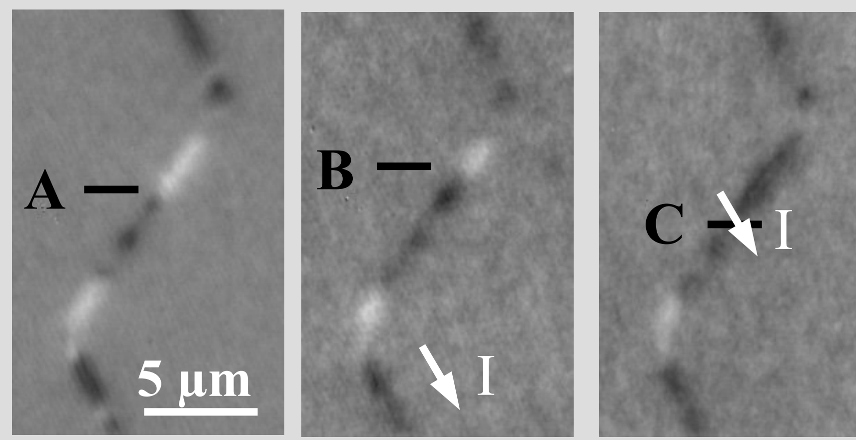


J. Vogel et al. (2005)

Spin-polarized driven domain wall motion



400 nm wide stripes:
FeNi(4nm)/Cu(8nm)/
Co(7nm)



S. Pizzini et al., APEX, in press (2009)

Features

- UHV technique, however nanofabricated and capped samples OK
- Full-field (medium imaging rate)

- 25nm lateral resolution

- Surface sensitivity
- 2D maps of magnetization with 1 component only
- Elemental resolution

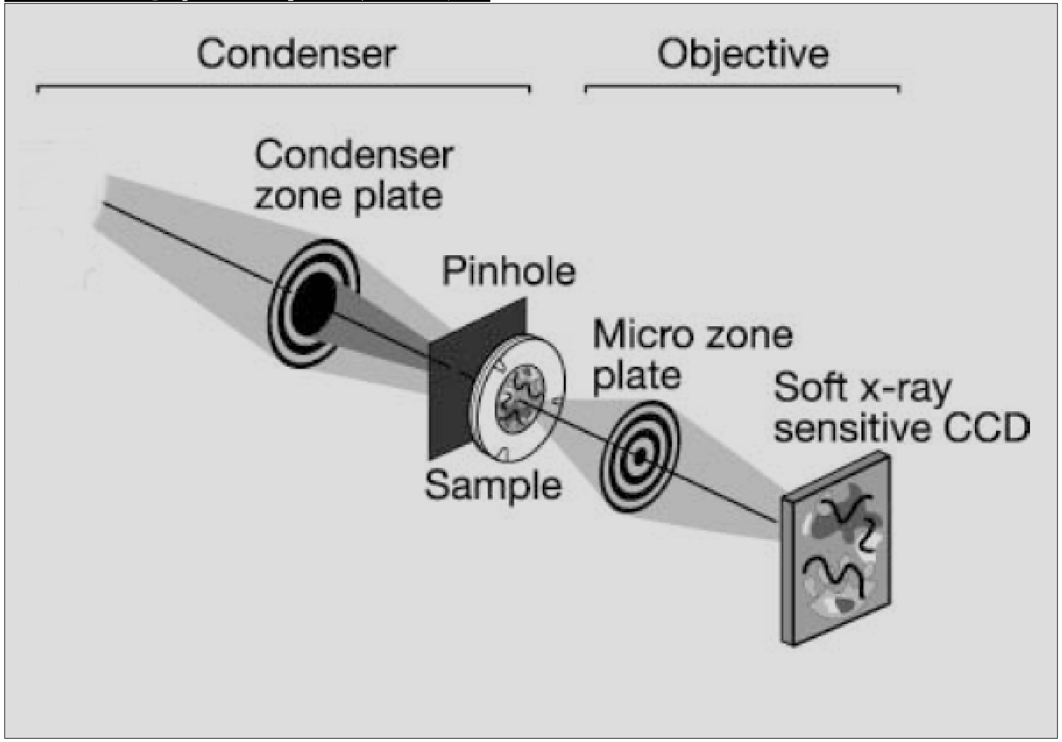
- Compatible with time resolution
- No significant applied field

Prospects / future

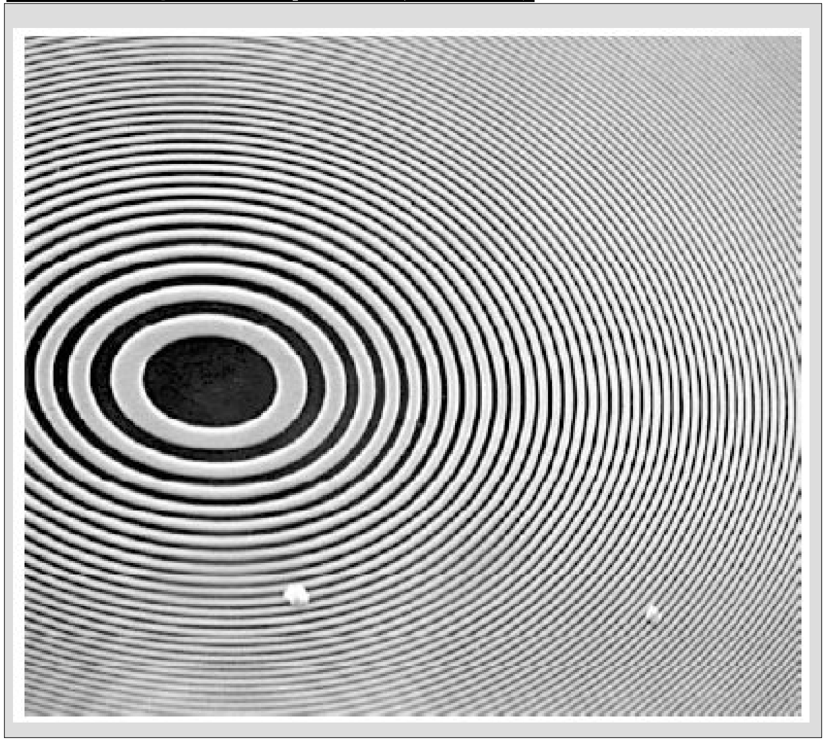
- Resolution improved to 10nm (energy filtering; abberation correction)
- Manipulator for multi-component imaging

(S)TXM = (Scanning) Transmission X-ray Microscope

Working principle (TXM)



Focusing zone plate (detail)



P. Fischer et al., Rev. Sci. Instrum. 72, 2322 (2001)

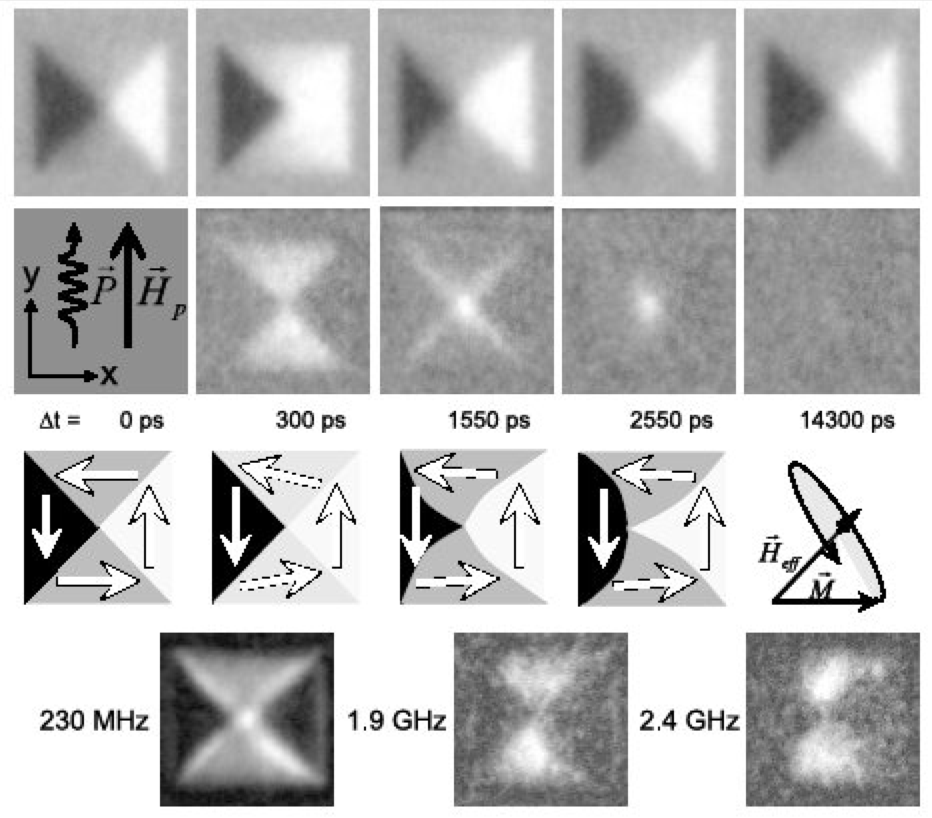
Static imaging

CoPtCr recording media

P. Fischer et al., Rev. Sci. Instrum. 72, 2322 (2001)

Pump-probe time-resolved measurements

6µm Permalloy squares

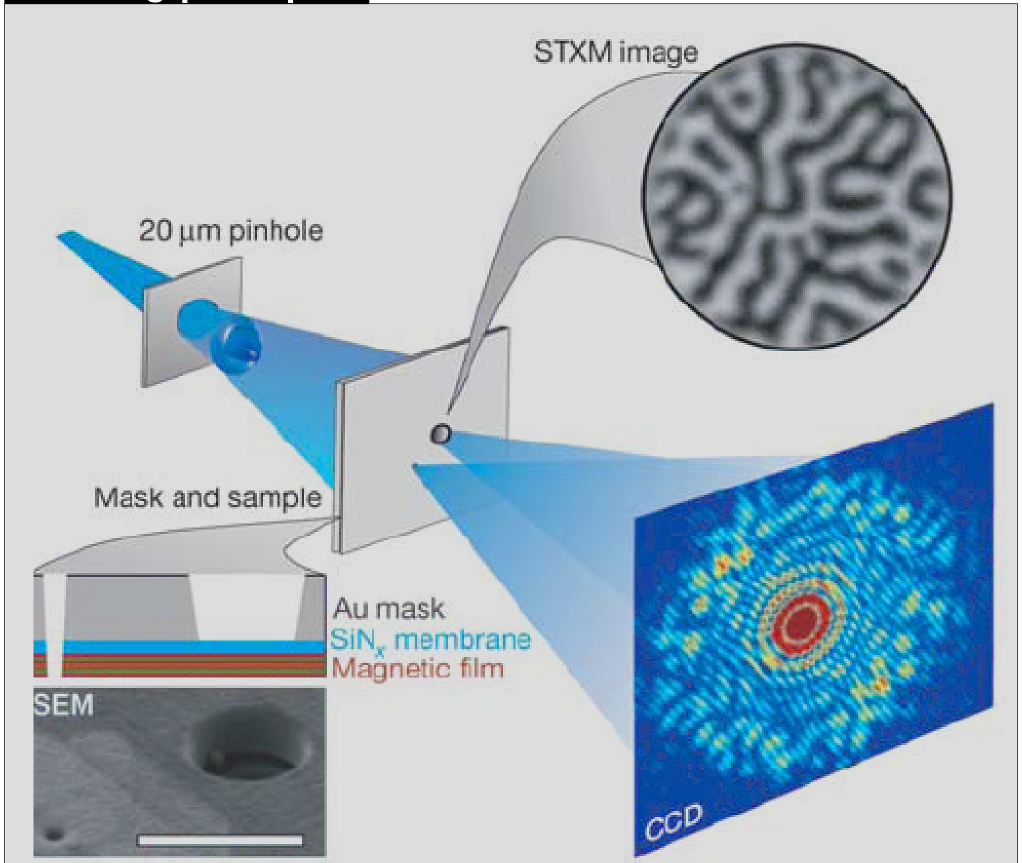


J. Raabe et al., Phys. Rev. Lett. 94, 217204 (2005)

Features

- Compatible with nanofabricated and capped samples
- Down to 15nm lateral resolution
- Volume sensitivity (transmission)
- 2D maps of magnetization with 1 component only. Samples can be tilted
- Elemental resolution
- Compatible with time resolution
- Compatible with applied field

Working principle



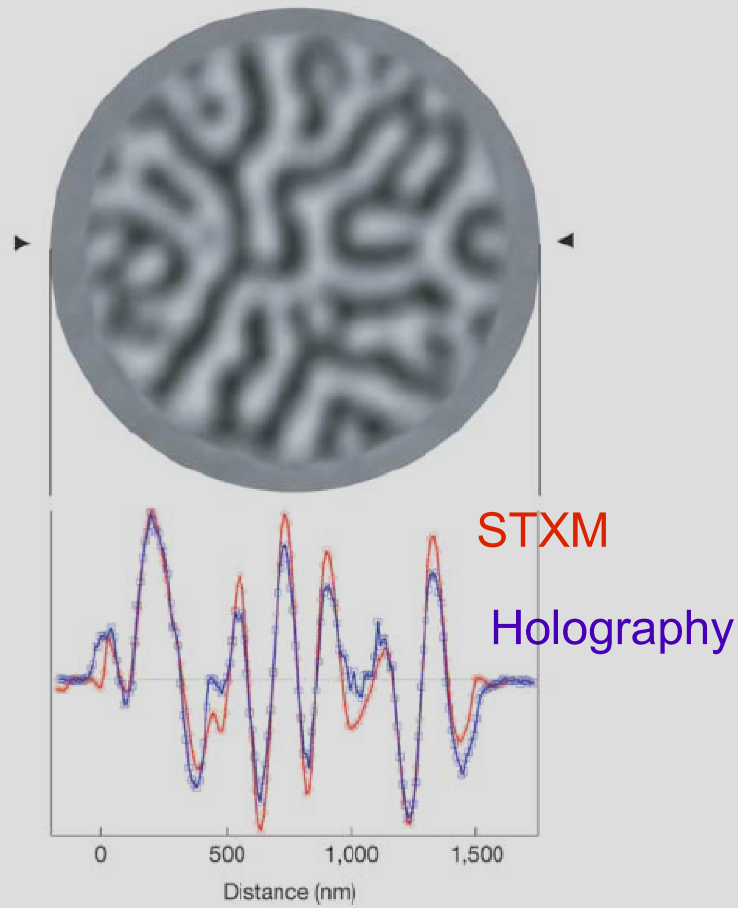
S. Eisebitt et al., Nature 332, 885 (2004)

Features

- Resolution improving
- Requires nanofabrication

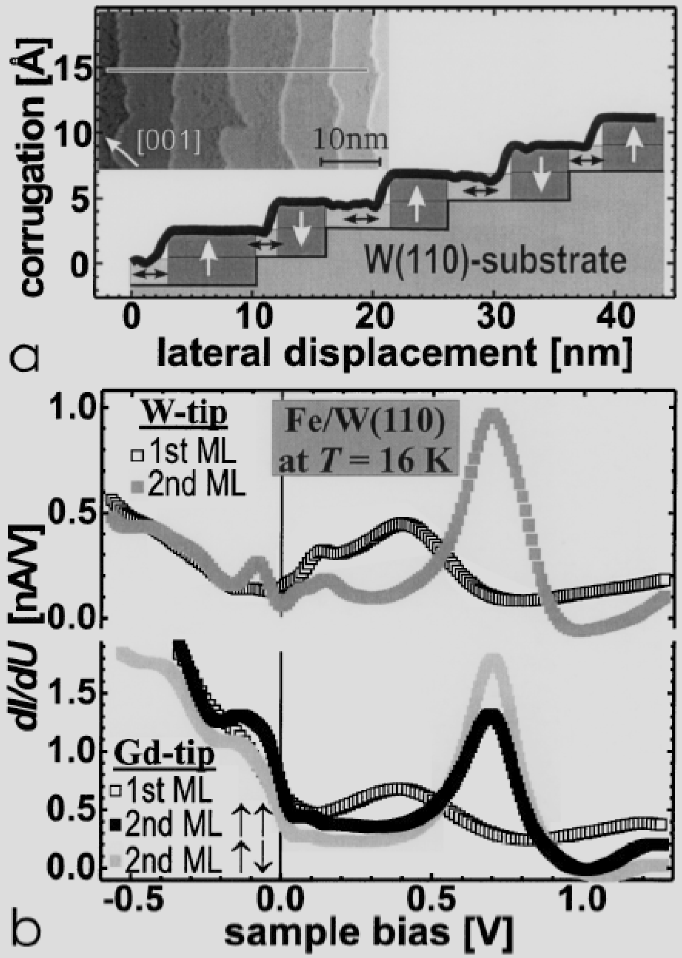
Example

[Co(4Å)/Pt(7Å)]$_{50}$ with perpendicular magnetization



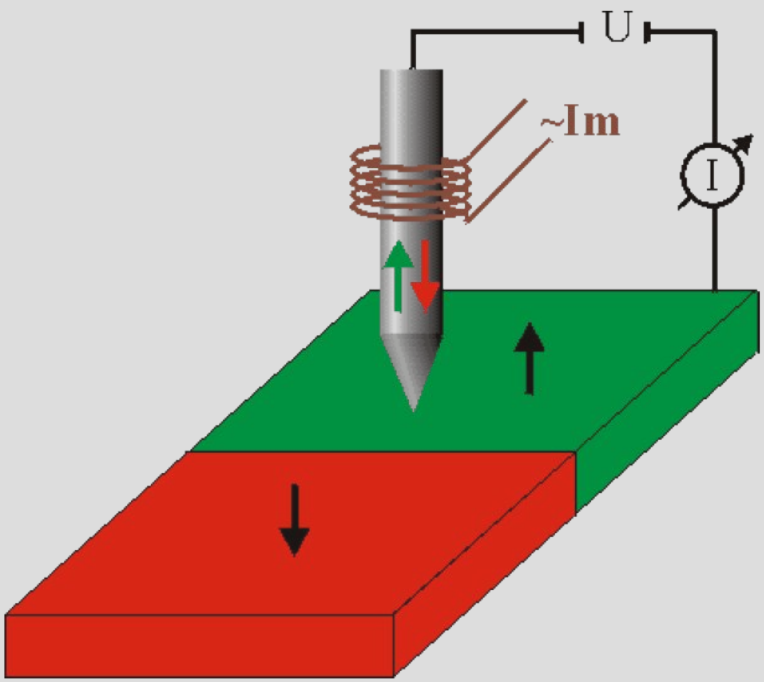
spSTM = Spin-Polarized Scanning Tunneling Microscope

Spin-dependent spectroscopy



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

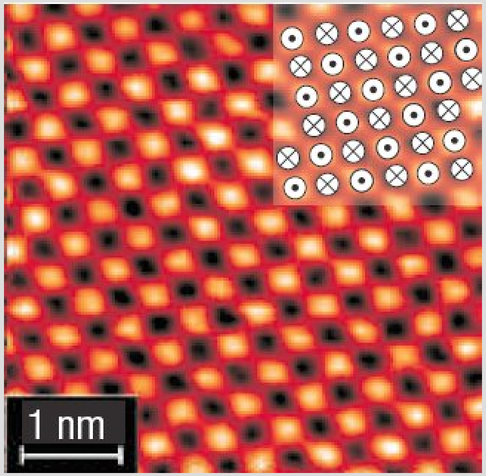
Modulation of tip magnetization + lock-in tunneling current



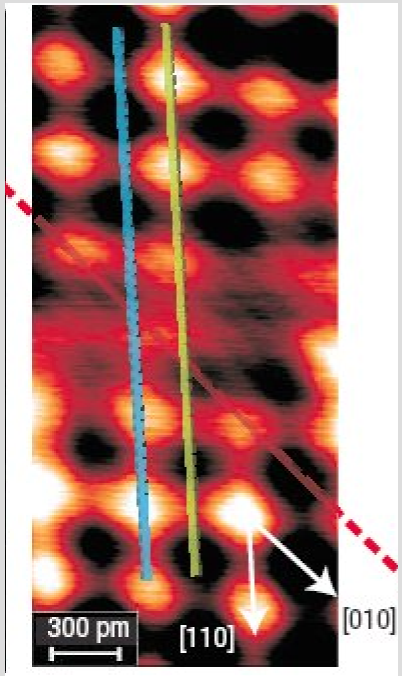
W. Wulfhekel et al., APL 75, 1944 (1999)

Ultimate magnetic resolution

Antiferromagnetic Fe(1ML)/W(001)



Antiferromagnetic domain

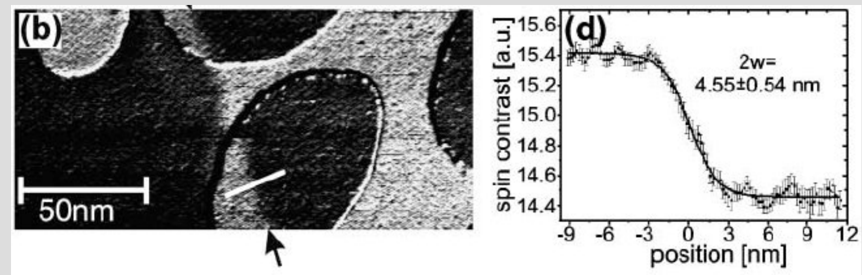


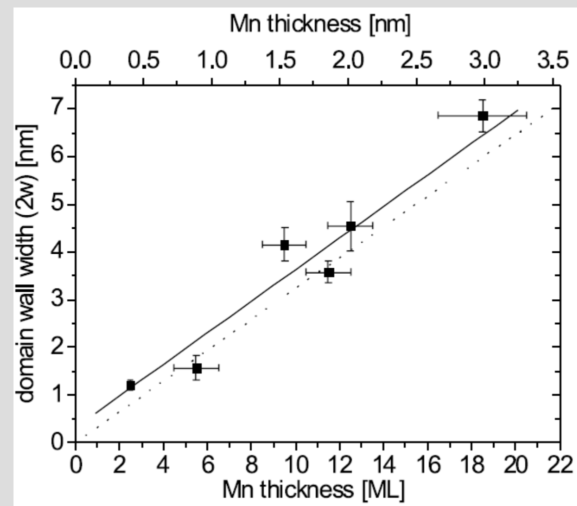
Antiferromagnetic domain wall

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

Frustration at a ferro/antiferro interface

Mn/Fe(001)





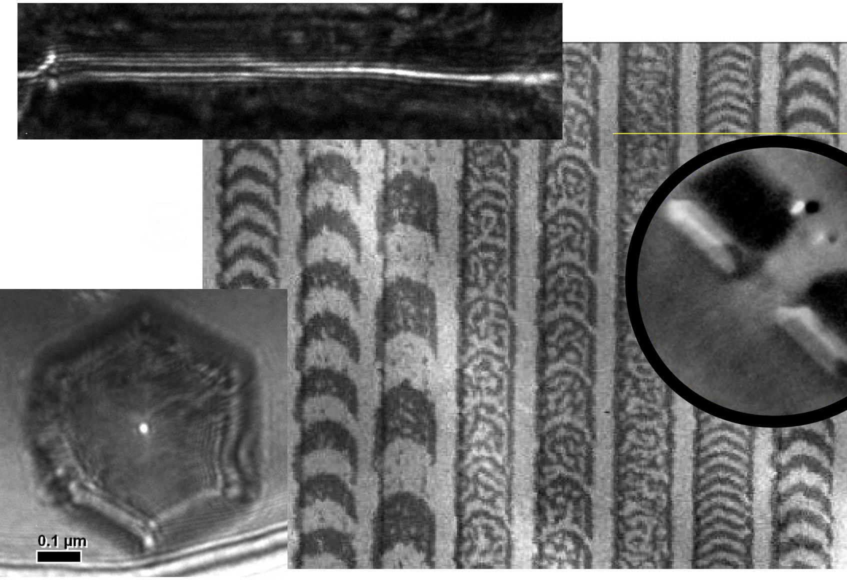
U. Schlickum, Phys. Rev. Lett. 92, 107203 (2004)

Features

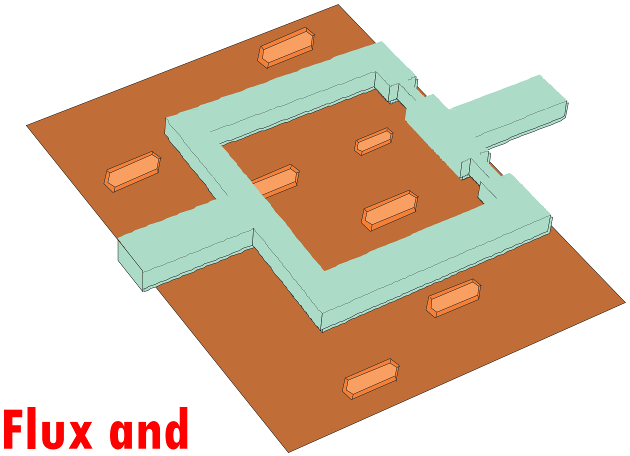
- UHV technique,
  very few samples from ex situ
- Slow
- Ultimate lateral resolution
- Compatible with field and low temperature

Prospects / future

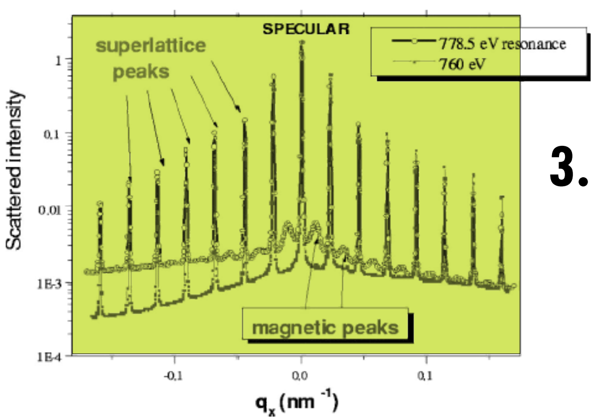
- Magnons and anisotropy with spectroscopy
- Local manipulation with spin-polarized current or electrical field



1. Microscopy

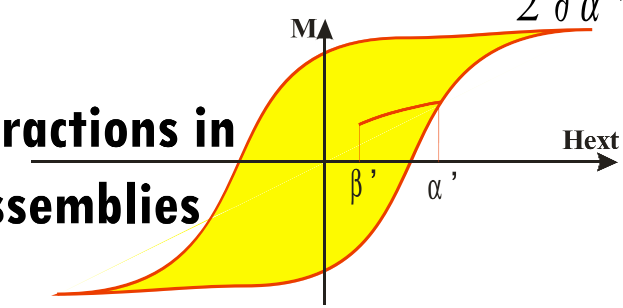


2. Flux and direct local probes



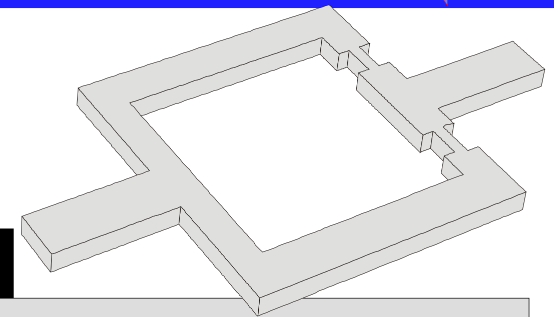
3. Reciprocal space

$$\mu(\alpha',\beta') = \frac{1}{2}\frac{\partial^2 f_{\alpha',\beta'}}{\partial\alpha'\partial\beta'}$$

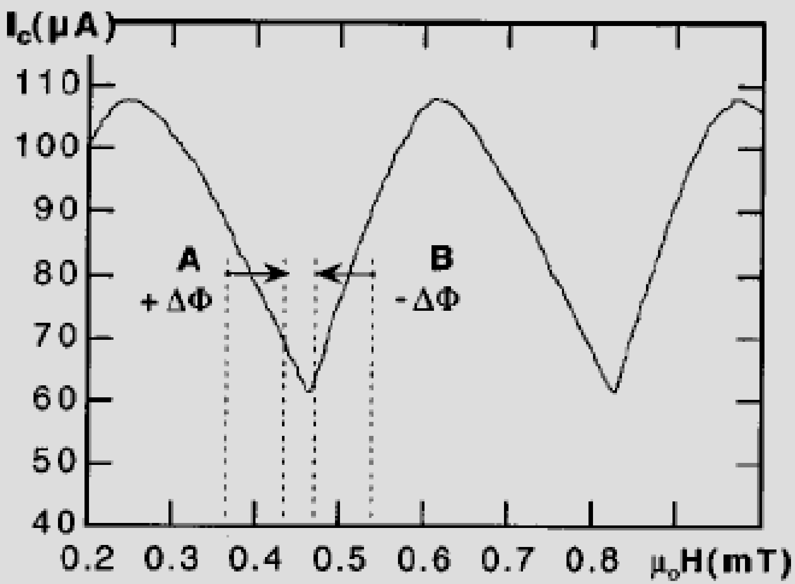


4. Interactions in assemblies

Micro-SQUID: a microfabricated loop with Josephson junction

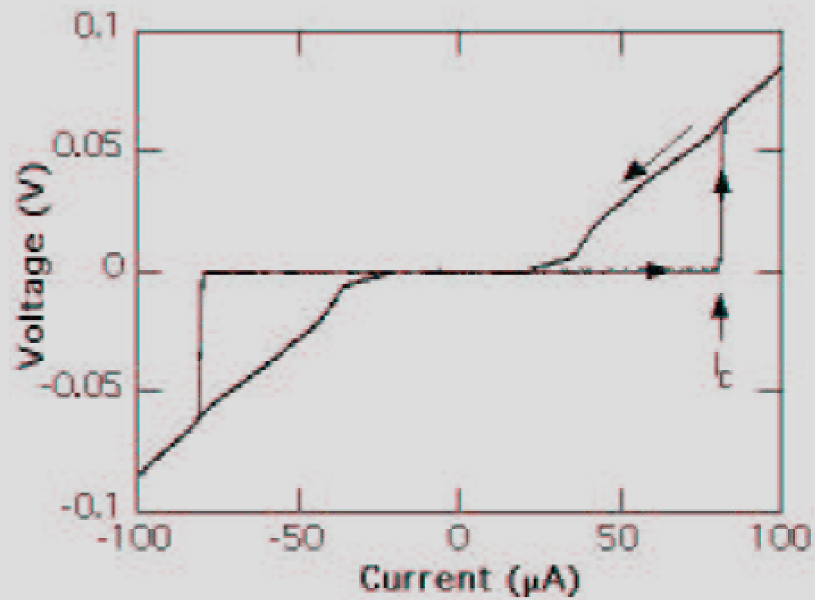


Critical current versus flux



⇨ Flux in the loop monitored through the measurement of the critical current

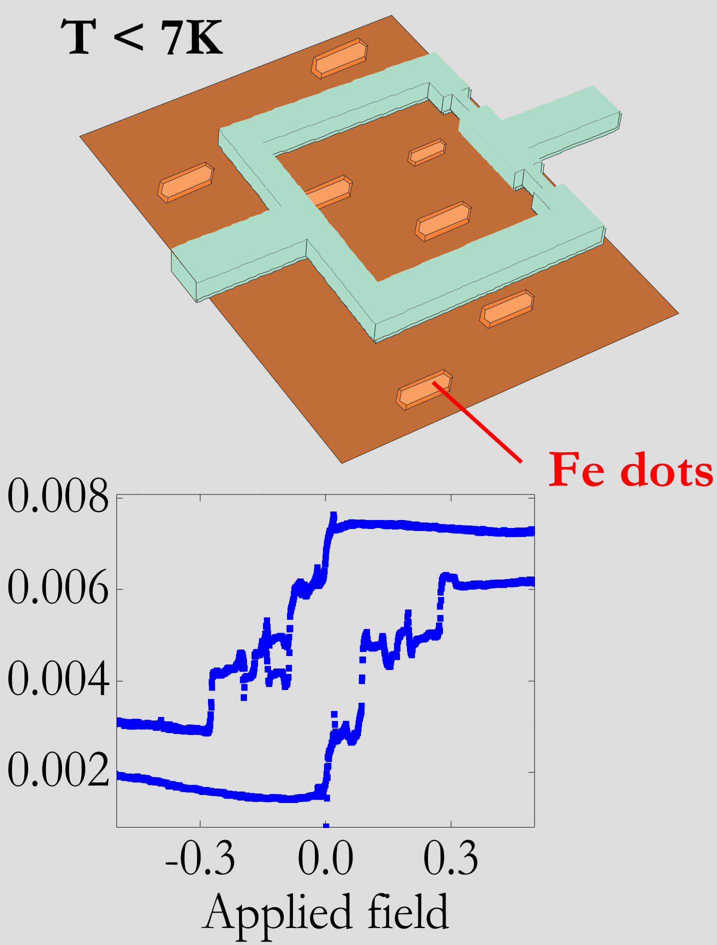
V(I) characteristics



⇨ Critical current measured by triggering the S → N transition in the loop

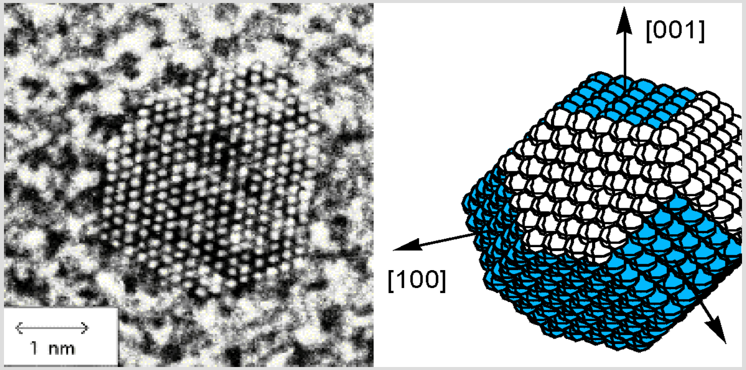
W. Wernsdorfer et al., Adv. Chem. Phys. 118, 99-190 (2001)

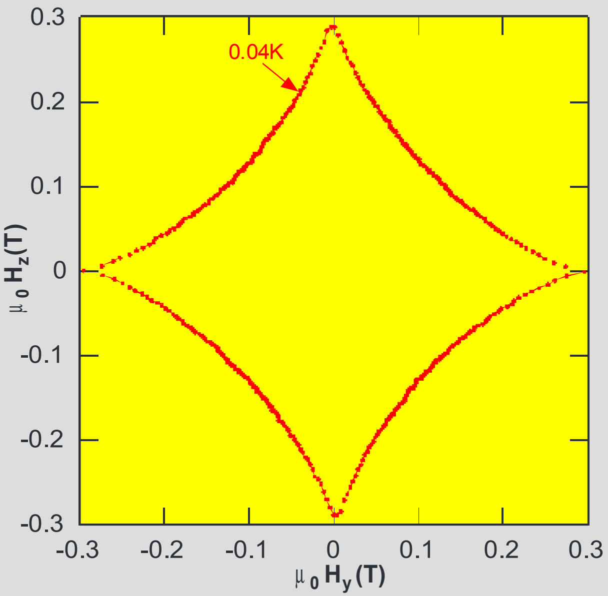
Mode: flux monitoring



O. Fruchart et al., J. Magn. Magn. Mater. 198-199, 228 (1999)

Cold mode: switching monitoring





W. Wernsdorfer et al., Phys. Rev. Lett. 78, 1791 (1997)

M. Jamet et al., Phys. Rev. Lett., 86, 4676 (2001)

Features

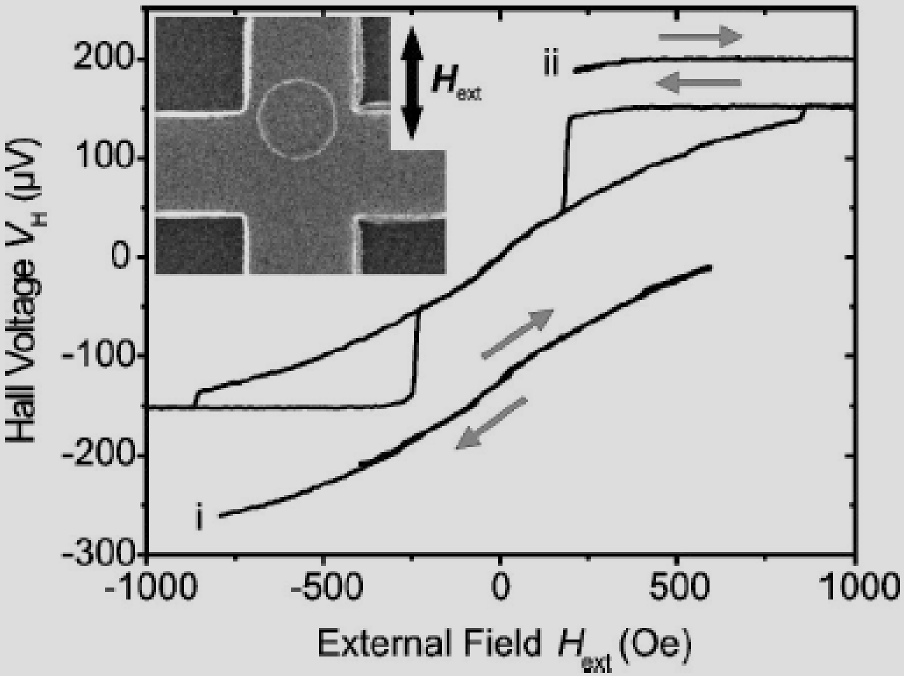
- Monitors flux or magnetization switching
- Sensitivity $10^3 \mu_B$
- Limited to low temperature

Prospects / future

- Improved resolution ; nanotube -SQUID

Working principle and example

Local flux monitored via a nanofabricated micro-Hall cross of a 2D electron gas



M. Rahm et al., Appl. Phys. Lett. 82, 4110-4112 (2003)

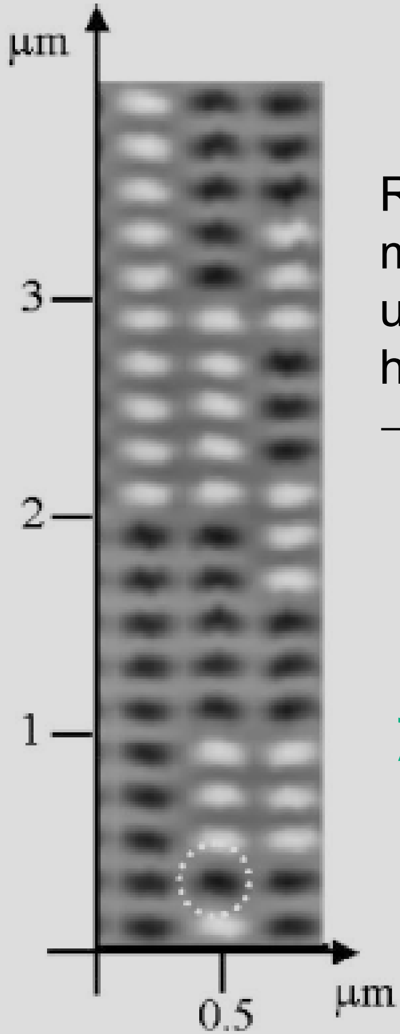
Features

- Monitors flux
- Sensitivity $10^4 \mu_B$
- No temperature limitation

Prospects

- Magnetization dynamics?

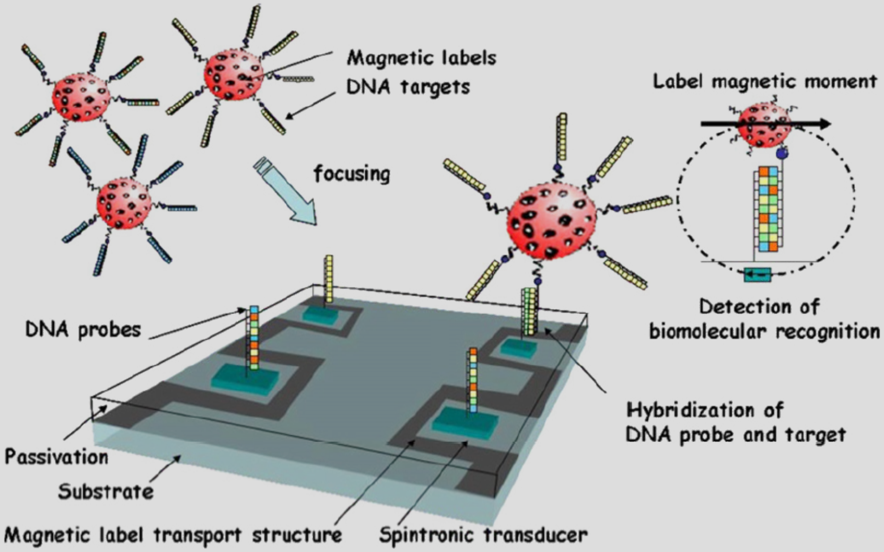
Hard-disk-drive GMR heads



Read-Write a patterned media [CoPt]n/Si(111) using a hard-disk drive head on a translator
→ magnetic microscopy and manipulation

J. Moritz et al., Appl. Phys. Lett. 84, 1519 (2004)

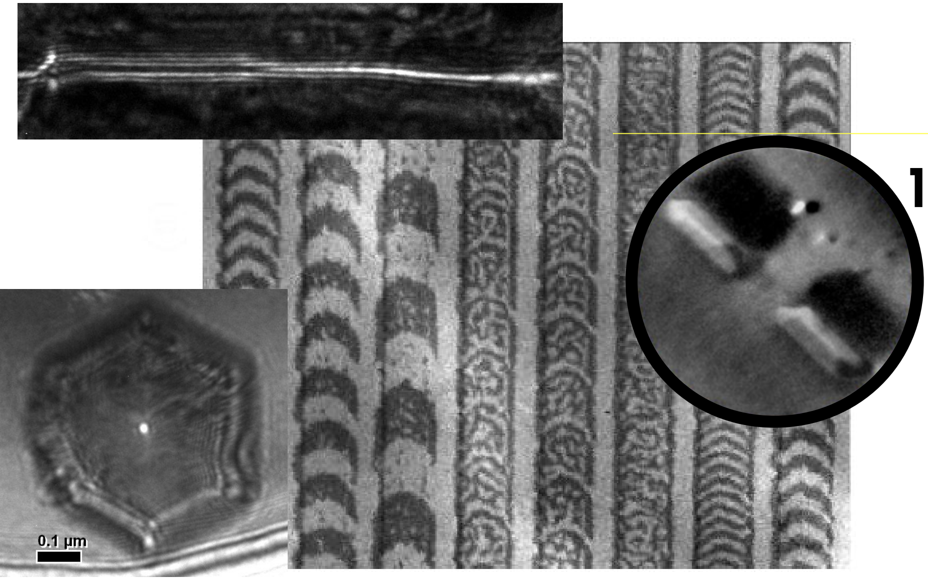
GMR / TMRelements for biochips



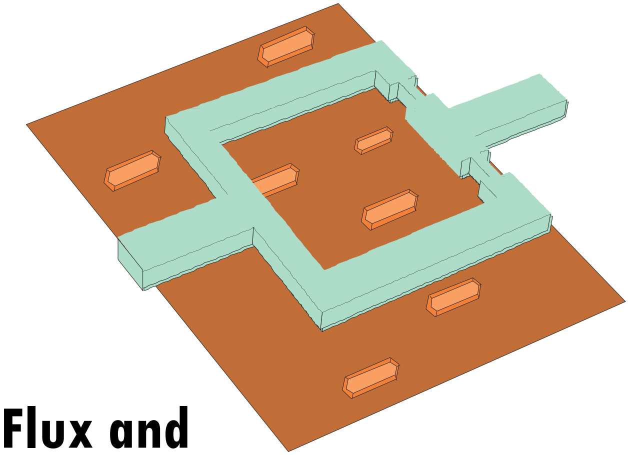
Sensitivity: 10pT DC

P. P. Freitas, Magnetoresistive sensors, J. Phys. Condens. Matter 19, 165221 (2007)

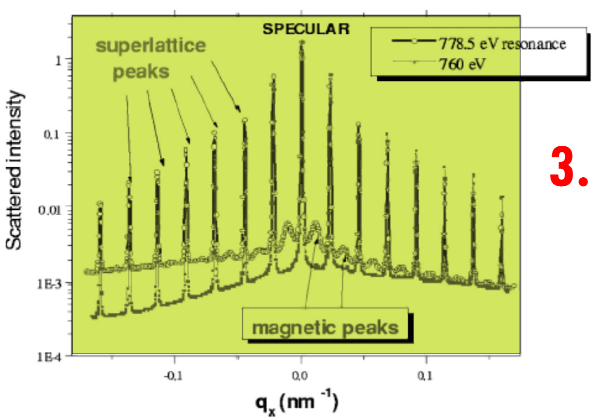
Also: direct magneto-transport: GMR, Extraordinary Hall Effect (EHE) etc.



1. Microscopy

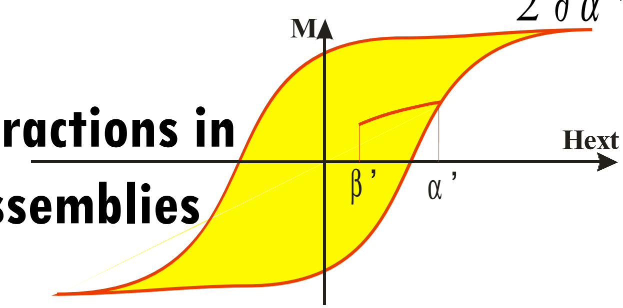


2. Flux and direct local probes



3. Reciprocal space

$$\mu(\alpha', \beta') = \frac{1}{2}\frac{\partial^2 f_{\alpha',\beta'}}{\partial \alpha' \partial \beta'}$$

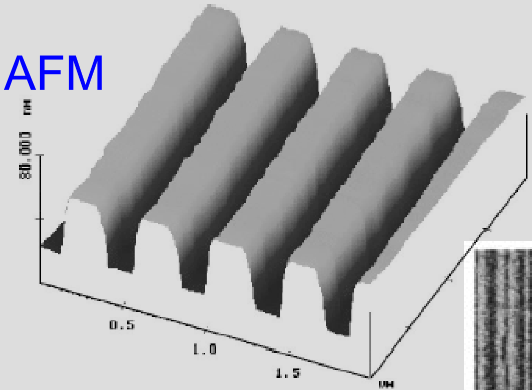


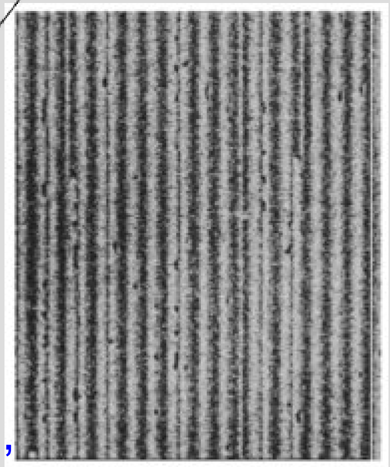
4. Interactions in assemblies

Idea: probe depth and lateral magnetic correlations with reflectivity and scattering

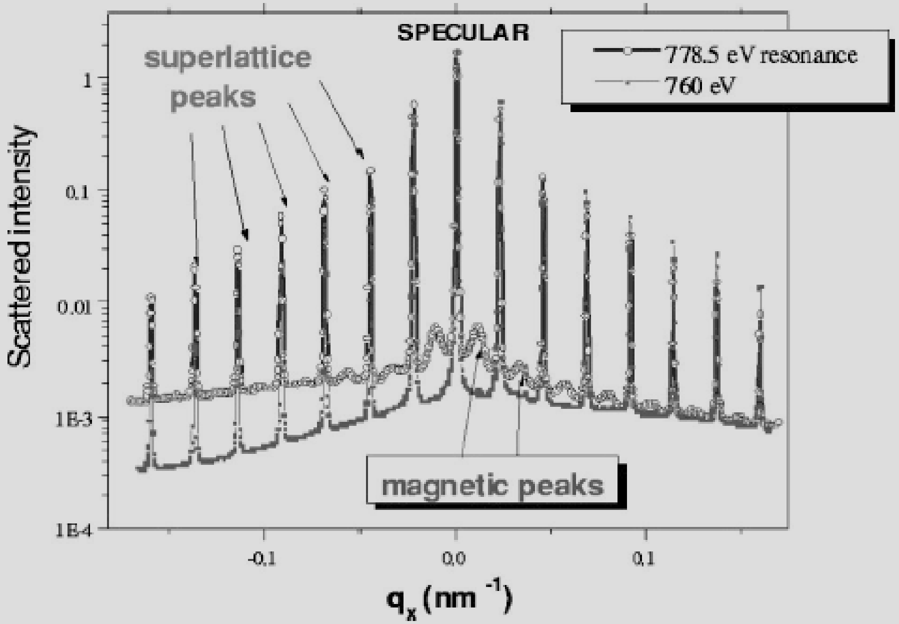
Test case: perpendicularly-magnetized stripes, 250nm wide

AFM





MFM, demagnetized state



Off-specular reflectivity to probe lateral order

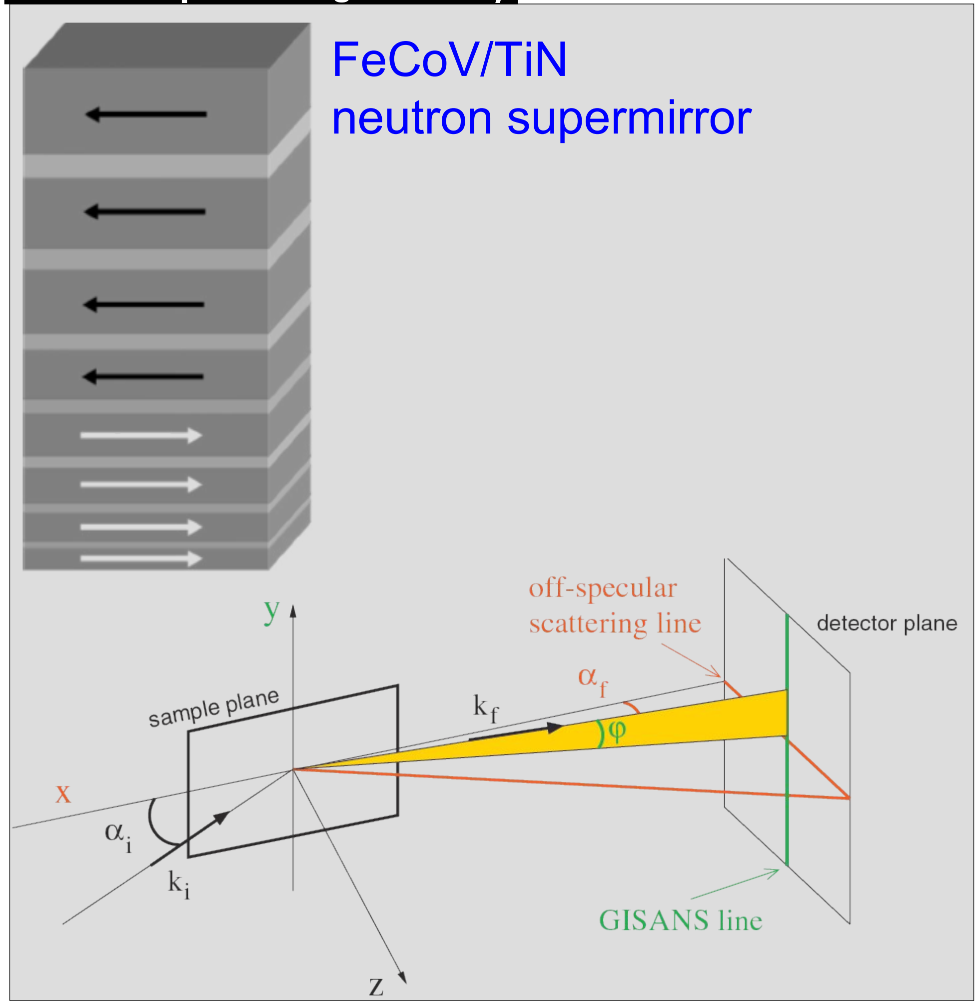
K. Chesnel et al., Phys. Rev. B 66, 024435 (2002)

Prospects

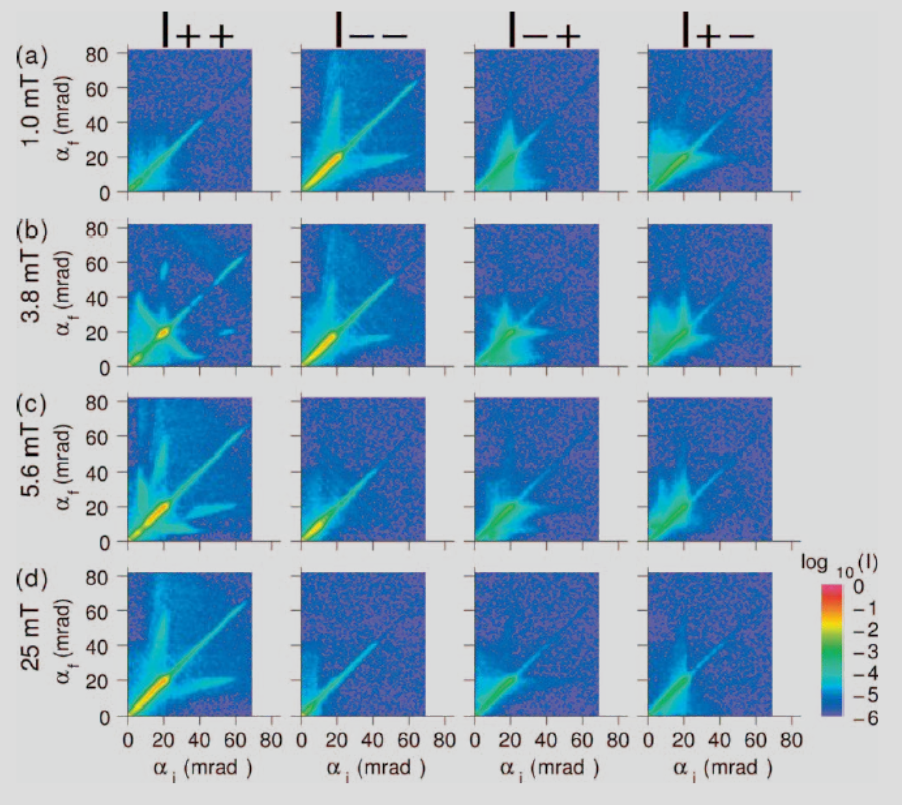
- Probe lateral order on muchy smaller structures (no other microscopy tool available)
- Use self-organization as a tool to learn about magnetic domains at the nanoscale?

GISANS: Grazing Incidence Small Angle Neutron Scattering

Test sample and geometry

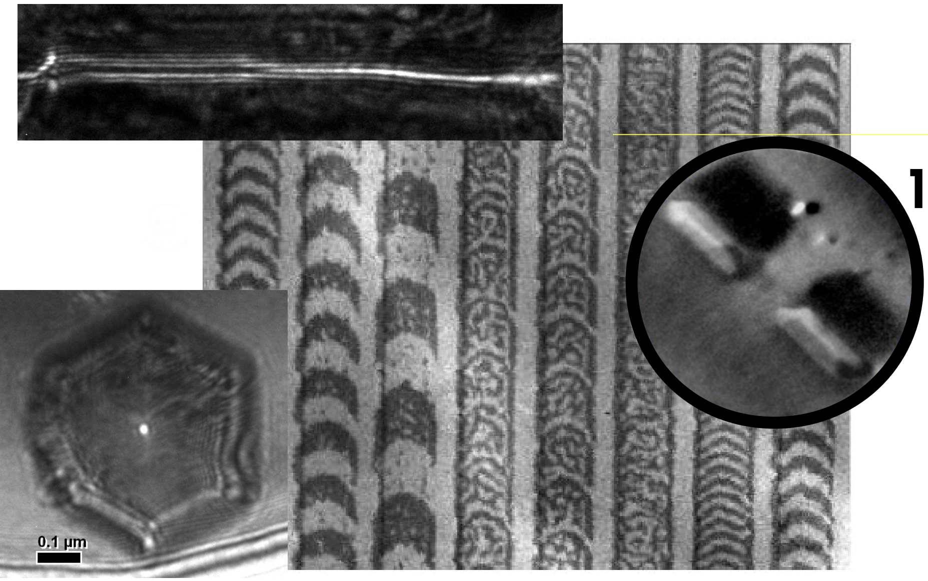


Results

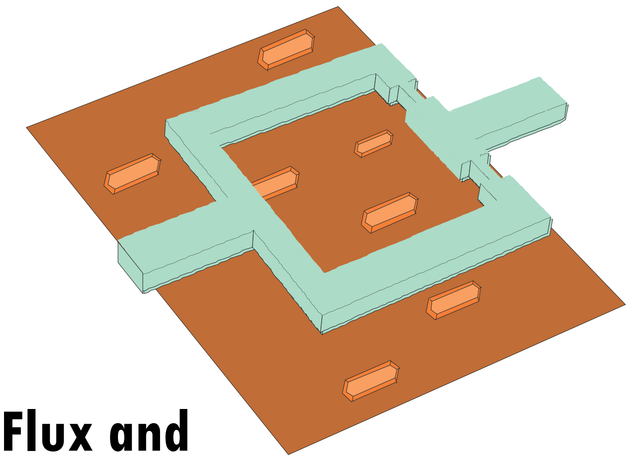


E. Kentzinger et al., Phys. Rev. B 77, 104435 (2008)

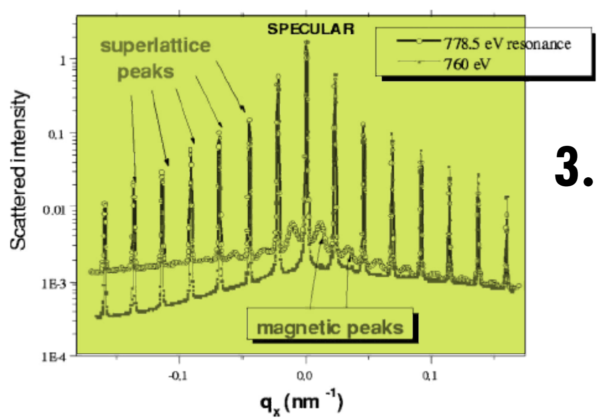
- Recent improvement in neutron flux
- Apply to nuried material and assembles of nanoparticles



1. Microscopy

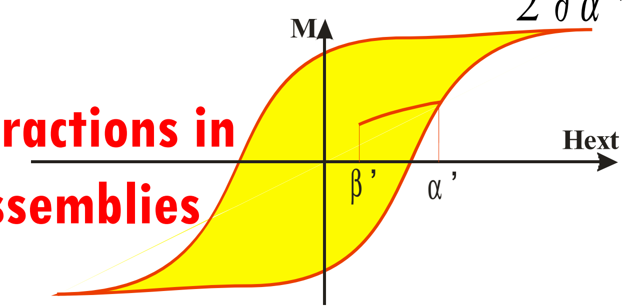


2. Flux and direct local probes



3. Reciprocal space

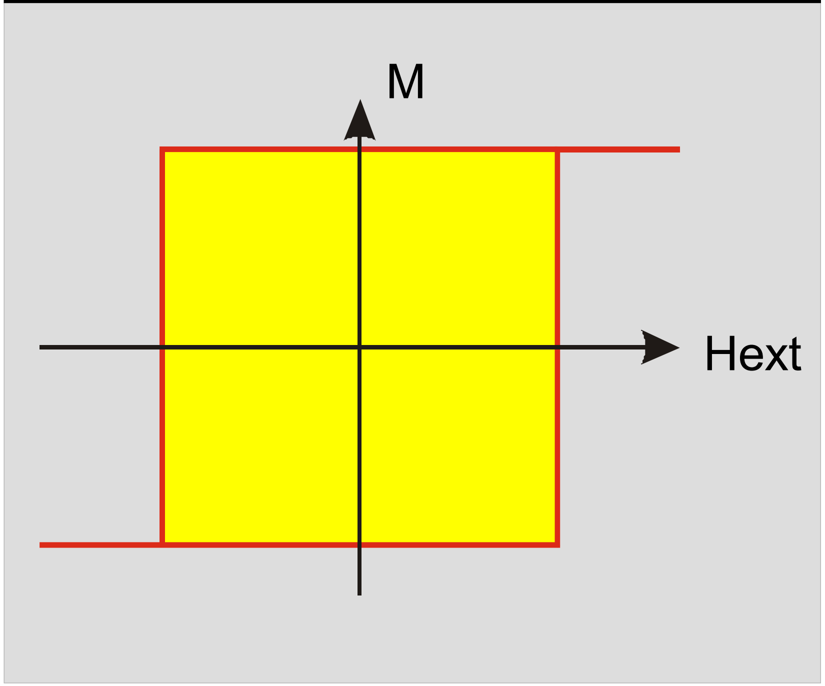
$$\mu(\alpha',\beta') = \frac{1}{2}\frac{\partial^2 f_{\alpha',\beta'}}{\partial\alpha'\partial\beta'}$$



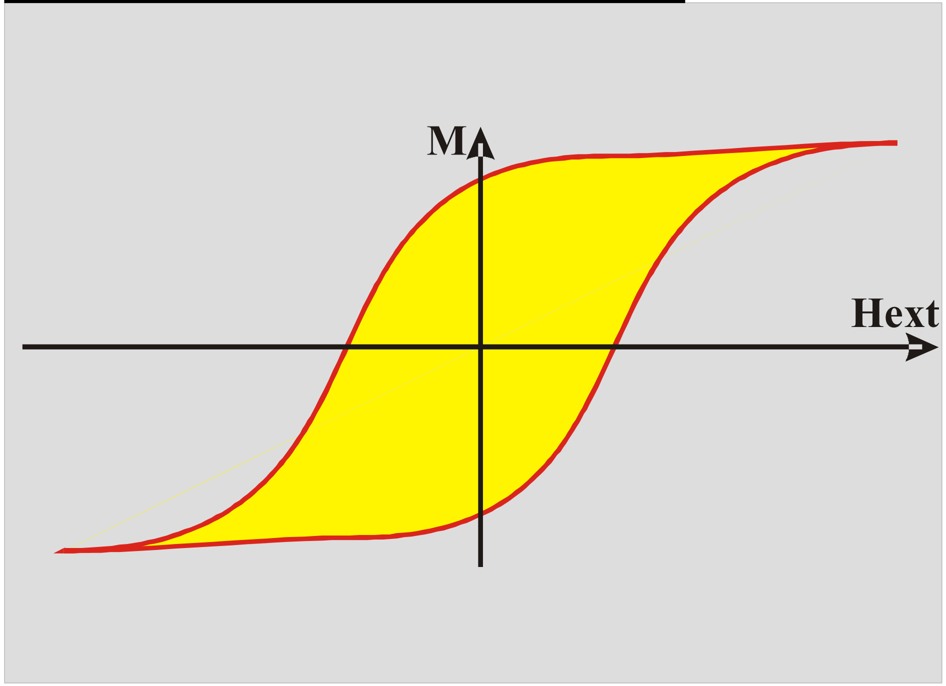
4. Interactions in assemblies

Hysteresis loops to estimate interactions in assemblies

Expected hysteresis loop for macrospins



Hysteresis for assemblies of dots



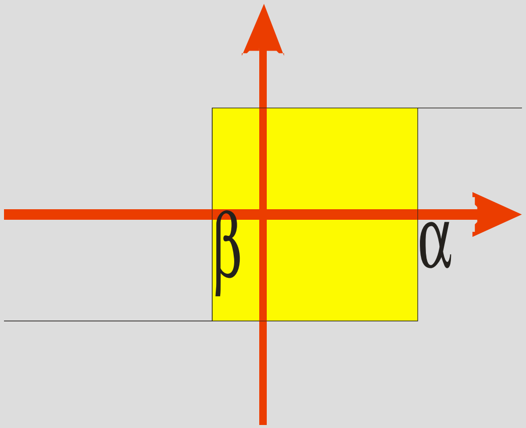
Possible effects

- Distribution of coercive fields
- (Dipolar, RKKY) interactions

Preisach model

G. Biorci et al., Il Nuov. Cim. VII, 829 (1958)

I. D. Mayergoyz, Mathematical models of hysteresis, Springer (1991)

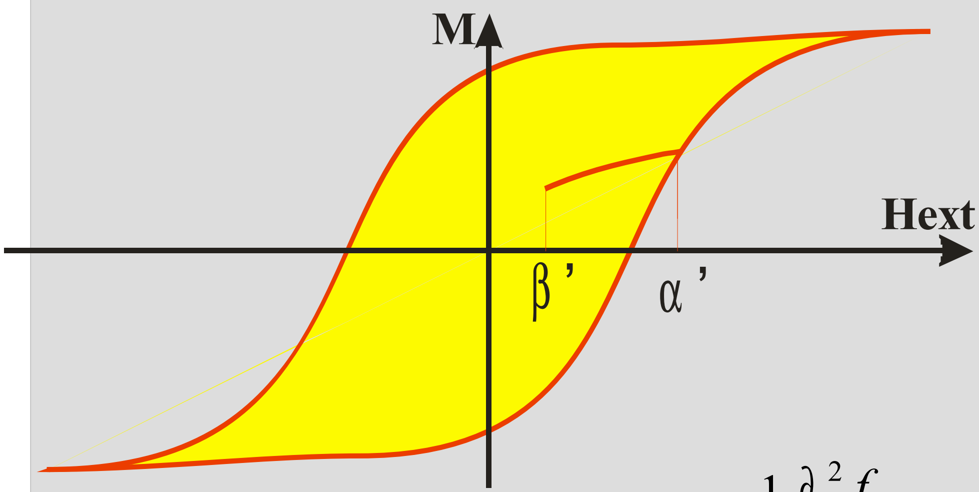


- Distribution function

$$\mu(\alpha, \beta) \text{ with } \alpha > \beta$$

- No true link between real particles and μ

Solving



$$\mu(\alpha', \beta') = \frac{1}{2}\frac{\partial^2 f_{\alpha',\beta'}}{\partial \alpha' \partial \beta'}$$

- Long experiments (1D set of hysteresis curves)
- Better suited to bulk materials with strong interactions

Henkel plots

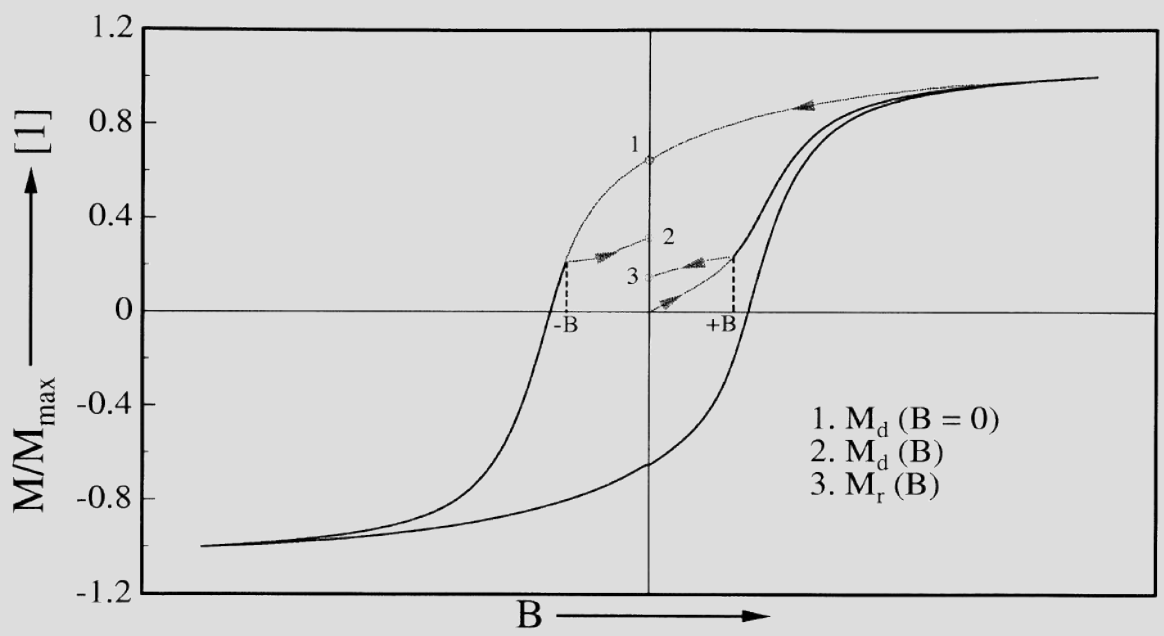


Fig. 1. Explanation of how to measure the two different remanent magnetisations M_r and M_d.

O. Henkel,
Phys. Stat. Sol. 7, 919 (1964)

S. Thamm et al.,
JMMM184, 245 (1998)

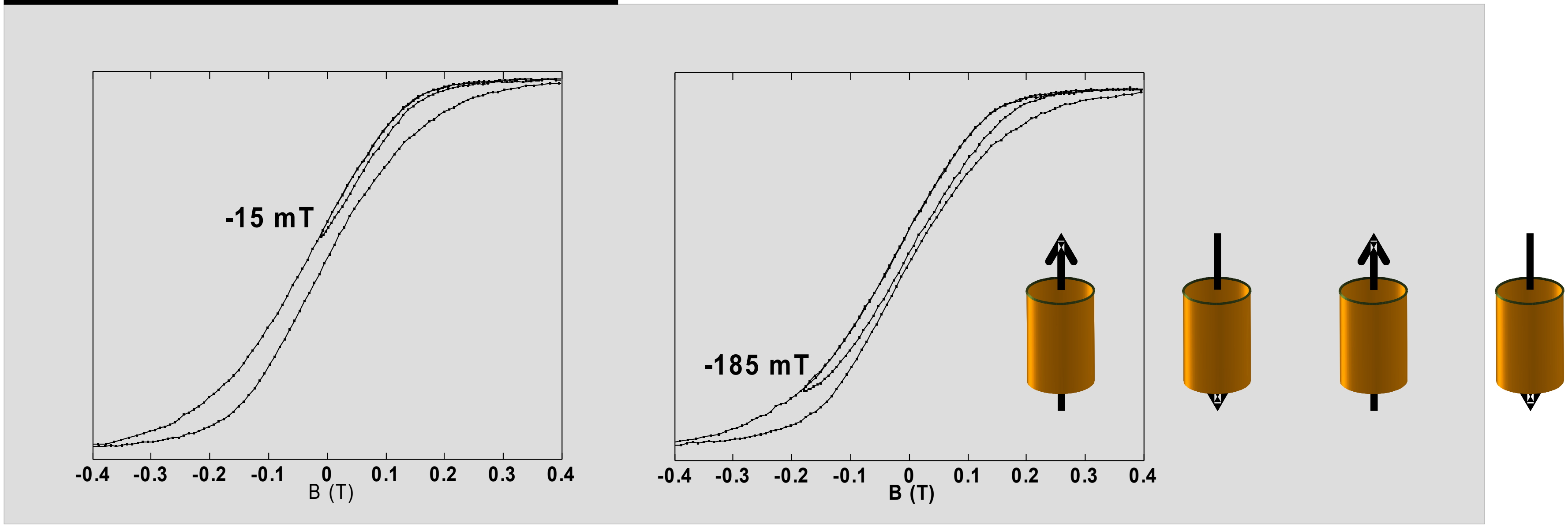
Measure of dipolar interactions

$$\Delta M_H(x) = M_d(x) - [1 - 2M_r(x)]$$

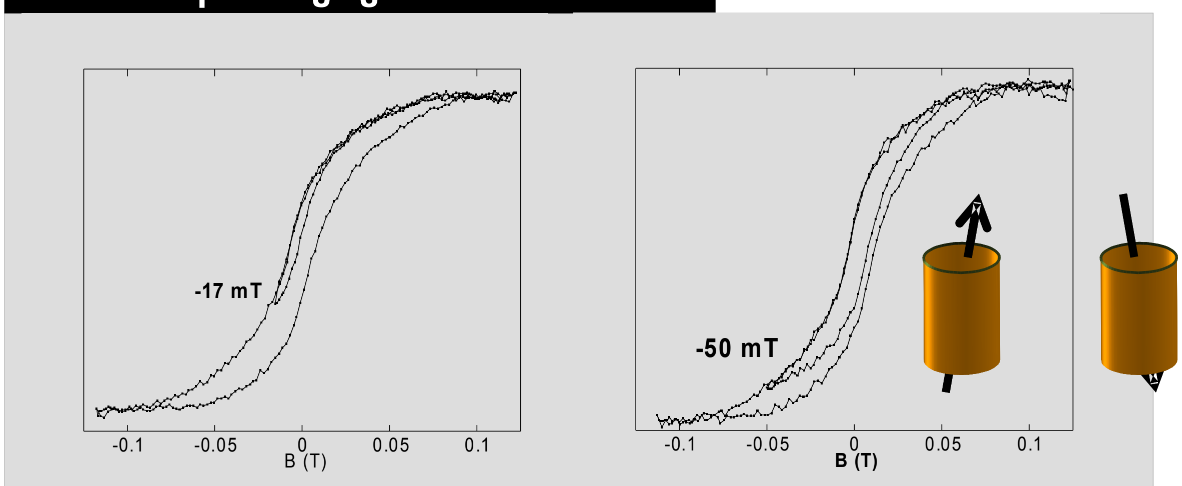
- Long experiments (ac demagnetization)
- Better physical meaning than Preisach

Minor loops: negative interactions

Example: dipolar interactions



Minor loops: negligible interactions



- Faster than Henkel
- Other applications: characterization of exchange bias

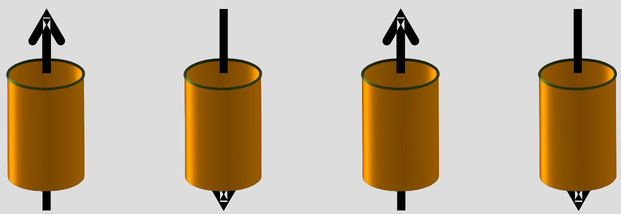
Superparamagnetic regime: plot of inverse susceptibility

- **Brillouin 1/2 function**

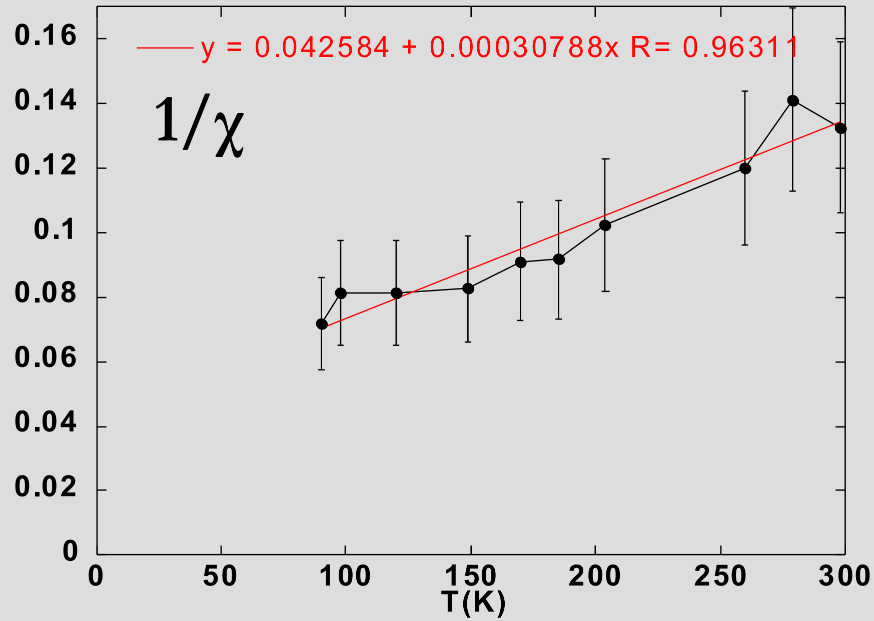
$$m = B_{1/2}\left(\mu_0 \mu_{Co} N H_{eff.} / kT\right)$$

- **Effective field**

$$H_{eff.} = H + r M_s m$$



(Demagnetizing dipolar interactions)



- **First order expansion:**

susceptibility

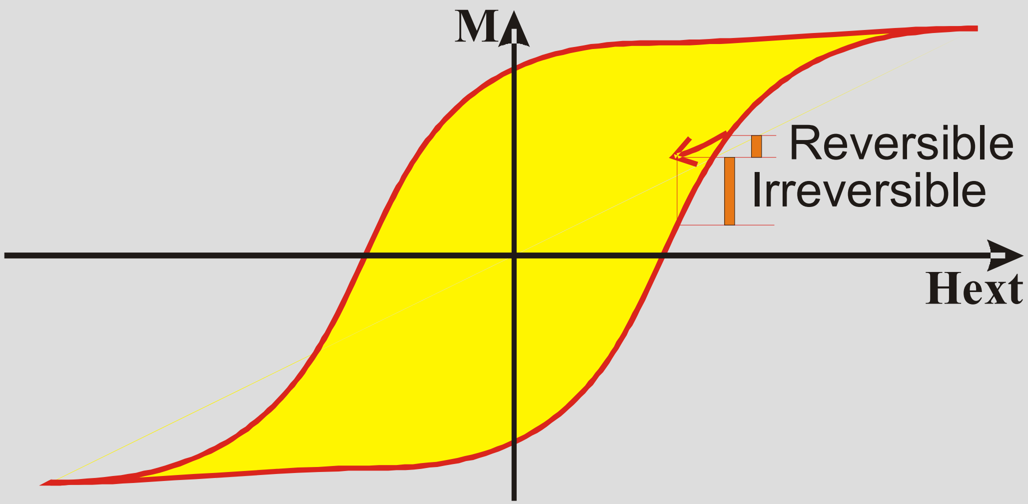
$$\frac{d(\mu_0 H)}{dm} = \frac{1}{\chi} = -\mu_0 M_S r + \frac{k}{\mu_{Co} N} T$$

$$a \quad + \quad b \, . \, T$$

O. Fruchart *et al.*, PRL 23, 2769 (1999)

- No need of hysteresis
- Analogy with Curie-Weiss law

Distribution of properties



$$\rho\,(H_r) = \left.\frac{dm}{dH}\right|_{\text{irreversible}}$$

Hc(T) for a given population of the distribution can be studied at a given stage of the reversal (10%, 20% etc.)

Effect of distributions and dipolar interactions are sometimes difficult to disentangle

MFM-based techniques
- High-resolution MFM
- MRFM (O. Klein et al., Saclay)
- NMR

Other microscopy techniques
- Dichroism in TEM
- BEM: Ballistic Electron Microscope

•Direct transport techniques
- GMR
- Extraordinary Hall Effect

Probes of electronic properties, excitations
- Spin-resolved Photo-emission and inverse photo-emission
- SPEELS: Spin-polarized electron energy loss spectroscopy
- Supra/I/Ferro to measure the spin polarization

Discussion of the estimation of lateral resolution

etc.

[1] ***Magnetic domains***, A. Hubert, R. Schäfer, Springer (1999)

[2] J. Stöhr, H. C. Siegmann, ***Magnetism***, Springer (2006)

[3] H. Kronmüller, S. Parkin Eds., ***Handbook of Magnetism and Advanced Magnetic Materials***, vol.3: Noval techniques for characterizing and preparing samples, Wiley (2007).

[4] H. Hopster, H. P. Oepen, Eds., ***Magnetic Microscopy of Nanostructures***, Springer (2005).

[5] E. Beaurepaire, F. Scheurer, G. Krill, J.-P. Kappler, ***Magnetism and Synchrotron Radiation***, Lecture Notes in Physics, Springer (2001).

[6] European School on Magnetism 2005: New experimental approaches to Magnetism, http://esm.neel.cnrs.fr/2005-constanta/