Characterization techniques for nano-sized systems

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Criteria for measurement techniques
### Criteria for measurement techniques

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<th>What is measured?</th>
<th>Environmental conditions</th>
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<tr>
<td>Magnetization, induction, stray field?</td>
<td>Temperature</td>
</tr>
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<td>Elemental resolution</td>
<td>Field: magnetic field, electric</td>
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<td>Direct or indirect?</td>
<td>Electric current, light etc.</td>
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<td>Quantitative or not?</td>
<td>Strain</td>
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<td>Additional measuring techniques</td>
<td>Additional measuring techniques</td>
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<table>
<thead>
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<th>Which specifications?</th>
<th>Versatility</th>
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<tr>
<td>Magnetization: 1D, 2D, 3D</td>
<td>Sample preparation needed</td>
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<td>Depth resolution: surface or volume?</td>
<td>Time per one measurement</td>
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<td>Sensitivity</td>
<td>Large-scale or in-lab?</td>
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<td>Time/Spectral resolution</td>
<td>Expensive or cheap?</td>
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</table>
MOTIVATION: length scales (fundamental)

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: \( \frac{J}{m} \)
  - Anisotropy: \( \frac{J}{m^3} \)

- Anisotropy exchange length
  \[ \Delta u = \sqrt{\frac{A}{K}} \]
  - 1 nm → 100 nm
  - Hard → Soft

Diagram showing the concept of domain walls and atomic dipoles.
MOTIVATION: length scales (technology)

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

Underlying microstructure
10 nm magnetic grain


Precessional magnetization dynamics

\[ \frac{dm}{dt} = \gamma_0 m \times H + \alpha m \times \frac{dm}{dt} \]

- \( \gamma_0 = \mu_0 \gamma < 0 \) Gyromagnetic ratio
- \( \gamma_s = 28 \, \text{GHz/T} \)
- \( \alpha > 0 \) Damping coefficient

See lecture Christian Back

Pioneering experiment of precessional magnetization reversal


- >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
The Atomic Force Microscope (AFM)

Laser

Multiquadrant photodiode

Cantilever

Sample

Pizeoelectric stack or tube

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**

\[ Q = \frac{10}{\sqrt{2}} \]

\[ Q = \frac{1}{\sqrt{2}} \]

\[ \omega / \omega_0 \]

**Phase**

\[ Q = \frac{10}{\sqrt{2}} \]

\[ Q = \frac{1}{\sqrt{2}} \]

\[ \omega / \omega_0 \]

The cantilever as an oscillator

\[ m \ddot{z} + \Gamma \dot{z} + k z = F(z) \]

with:

\[ F(z) = F(z_0) + (z - z_0) \partial_z F \]

Mere renormalization:

\[ \omega_{o,\text{eff}} = \omega_o \left( 1 - \frac{1}{2k} \partial_z F \right) \]

Probing forces with the phase shift

- Attractive: red shift
- Repulsive: blue shift
Operation: two-pass technique

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

NB: other measurement modes exist


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**

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

---

Example

Skyrmion lattice in $\text{Fe}_{0.5}\text{Co}_{0.5}\text{Si}$


The Foucault mode

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

Imaging courtesy: A. Masseboeuf
Electron holography

Based on: transmission electron microscopy

Principle

Phase shift due to the magnetic induction

\[ \varphi_{mag}(x) = -\frac{e}{\hbar} \iint B_n(x, z) dx dz \]

Highlights isolines of z component of vector potential

2D maps of induction may be reconstructed

<1-2nm spatial resolution

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

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

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

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

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

Patterned magnetic dot on a Hall cross

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

Example

- Pt\Co(6Å)\AlOx stack
- Difference between Néel & Bloch walls
- Probes Dzyaloshinskii-Moriya physics


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

### 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
Extreme sensitivity

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

**X-ray magnetic dichroism – Microscopy**

**Photo-Emission Electron Microscopy (PEEM)**

**Principle**


**Example**

*FeNi/AlOₓ/Co spin valves*

Field pulse of several 10ns

J. Vogel et al., *PRB 72, 220402(R)* (2005)
**SHADOW XMCD-PEEM**

- Electrons hit the supporting surface and generate photons.
- The photons then interact with the electron beam, creating photo-electrons.

**Example**

- **Experimental Image**: Domain wall in a 100nm-diameter electroplated cylindrical nanowire.
- **Simulation Image**: Illustration of a Bloch point in a domain wall, highlighting the unique singularity in micromagnetism.

- **Bloch point in a domain wall** – The only singularity in micromagnetism.
- **“Topological protection”** of domain walls.

References:

- S. Jamet et al., PRB92, 144428 (2015)
- S. Da Col et al., PRB 89, 180405 (2014)
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
**X-ray magnetic dichroism – TXM**

Transmission X-ray Microscopy (PEEM)

### Principle

- **Condenser zone plate**
- **Pinhole**
- **Micro zone plate**
- **Soft x-ray sensitive CCD**

#### Example

- **FeNi square dot, 6µm.**
- **Stroboscopic time resolution**


- **230 MHz**
- **1.9 GHz**
- **2.4 GHz**

- **Compatible with magnetic field**
- **Ongoing development of tomography**

X-ray magnetic dichroism – Holography

Principle

Example

Lens-less imaging
Requires microfabrication

X-ray magnetic dichroism – Scattering

Principle

Perpendicularly-magnetized stripes, 250nm wide

AFM

MFM


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
Spin-Polarized STM

Principle

Spin-dependent spectroscopy

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

Antiferromagnetic Fe/W(001)


Frustration at ferro/antiferro interface

Mn/Fe(001)

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.
Spin-Polarized Low-Energy Electron Microscopy

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

**Principle of LEEM**

- Electron gun
- Aperture
- Condenser lens
- Sample
- Objective lens
- Screen

**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


Electron spin polarization and control

Features

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


Spin-Polarized Low-Energy Electron Microscopy

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

After annealing at 350°C

Field of view: 7 µm

SEMPA = Scanning Electron Microscopy with Polarization Analysis

**Principle**

- **Focused primary electron beam**
- **Spin analyzer**
- **Secondary electrons**
- **Magnetic domains**

![Diagram of SEMPA principle](image)


**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?
Example

Fe/W(001)

Field of view: 1.5 µm


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

<table>
<thead>
<tr>
<th></th>
<th>Sp-STM</th>
<th>MFM</th>
<th>NV</th>
<th>BEMM</th>
<th>SEMPA</th>
<th>SPLEEM</th>
<th>TEM</th>
<th>XMCD-PEEM</th>
<th>XMCD-microscopy (Fresnel ZP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resolution</td>
<td>&lt;1nm</td>
<td>15nm</td>
<td>5-10 nm</td>
<td>1-5nm</td>
<td>10nm</td>
<td>10nm</td>
<td>1-2nm</td>
<td>25nm → 10nm</td>
<td>15nm</td>
</tr>
<tr>
<td>Sensitivity</td>
<td>High</td>
<td>Med</td>
<td>High</td>
<td>Med</td>
<td>Med</td>
<td>High</td>
<td>Low</td>
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</tr>
<tr>
<td>In-field</td>
<td>YES</td>
<td>Limited</td>
<td>Limited</td>
<td>YES</td>
<td>local</td>
<td>No?</td>
<td>Limited</td>
<td>No?</td>
<td>YES</td>
</tr>
<tr>
<td>Versatile*</td>
<td>No</td>
<td>YES</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>UHV</td>
<td>Limited</td>
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<tr>
<td>Dynamics</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>New</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Element-sensitive</td>
<td>Limited</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Limited</td>
<td>Limited</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Probes</td>
<td>$m_i$</td>
<td>$H_d$</td>
<td>$H_d$</td>
<td>$m_i$</td>
<td>$m$</td>
<td>$m$</td>
<td>$m_{x,y}$</td>
<td>$m_k$</td>
<td>$m_k$</td>
</tr>
</tbody>
</table>

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

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**Conclusions**
- Worse and best come together
- Need for combining various instruments
Combine different techniques...

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

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