Imaging and magnetic characterization of individual nanostructures in a transmission electron microscope (TEM)

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www.elektronenmikroskopie.uni-r.de
Introduction / Motivation

two driving forces lead to miniaturization of magnetic structures

industrial requirements

- magnetic devices like
  - MRAMs (magnetic random access memories)
  - spin valve sensors
  - spintronics (spin transistors etc.)
- reduction in size
- higher speed
- less power consumption

academic curiosity

- magnetism is a collective phenomenon
  - how many atoms needed?
  - onset of ferromagnetism?
  - thermal stability?
  - switching behaviour?
  - geometry effects?
Acknowledgements

- all co-workers for their contributions
- German research society (DFG) and EU for financial support
- Philips / FEI and Gatan company for fruitful cooperation and support
- Prof. Dr. D. Weiss for use of his e-beam lithography facilities and sharing his expertise
- Prof. Dr. H. Lichte (TU Dresden) for advice and help with holography

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TEM in micro- / nanomagnetism research?

Isn't TEM all about imaging crystal lattices and atoms?
NO!
Electron microscopy is ...

... sensing materials properties by electron-matter interaction with high spatial resolution ⇒ "image"

- electric fields
  - i.e. using Coulomb force to interact with specimen
    ⇒ high resolution imaging of nuclei's electrostatic fields

- electronic excitations
  - interband excitations (EELS, EXELFS, EDX, ALCHEMI ...)
  - intraband excitations (EELS, ELNES)
  - plasmonic excitations (EELS, SESAM)

- magnetic fields
  - i.e. using Lorentz force to interact with specimen
    ⇒ imaging of specimen's magnetic fields (internal / external)

- secondaries
  - Auger electrons
  - secondary electrons (with spin !) (PEEM, SMART project)
  - backscattered electrons
  - X-rays
  - ....
What is needed for magnetic imaging in a TEM?

- **Instrumental requirements**
  - no magnetic field @ specimen's location
  - high magnetic sensitivity
  - high lateral resolution
  - possibility to manipulate specimen's induction

- **At your fingertips:**
  - elemental analysis
  - structure determination
  - thickness determination
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Shape!
100 x 100 nm²

Crystallite size, texture

Edge roughness

Corner radii
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**Methodic requirements**
- Fresnel imaging (Lorentz imaging)
- Foucault imaging
- Differential phase contrast imaging
- Holographic imaging
- [EMCD]
  (chiral dichroism w/electrons)

*at your fingertips:*
- elemental analysis
- structure determination
- thickness determination
Specifications for magnetic imaging

- Lateral magnetic resolution down to 5 nm (2 nm achievable)
- Lateral imaging resolution of 0.19 nm
- Magnetic sensitivity: (2.5 ± 0.5) Tnm
  Equivalent thickness of (2.3 ± 0.4) nm Ni_{31}Fe_{19}
- Stray fields are visible
- Strong interaction ⇒ small specimen is sufficient
- Geometry and micromagnetic configuration accessible
- Possibility to explore local composition, local electron spectroscopy on a <10 nm level
Electron – specimen interaction (magnetic fields)

geometrical optics

wave optics

magnetic specimen

region of overlap
some formulae ...

Lorentz angle $\beta_L$:

$$\beta_L = \frac{eBt}{mv} = \frac{eBt}{\sqrt{(2m_0E(1+\frac{E}{2E_0}))}}$$

with $E_0 = m_0c^2$

magnetic phase shift

$$\varphi_m = -\frac{2\pi e}{h} \varphi m = -\frac{2\pi e}{h} B t x$$

conversion recipe

holography $\Leftrightarrow$ DPC

note: $\frac{\partial \varphi_m}{\partial x} \approx \beta_L$
Fresnel imaging

\[ I \approx \left| 1 + \frac{\Delta f \cdot \lambda_{el}}{2\pi} \cdot \frac{d\vec{B}(x,y)}{d(x,y)} \right|^2 \]

⇒ Sensitivity adjustable through \( \Delta f \)
Square Co dots

Co dots, 35 nm thick specimen by R. Sattler
Magnetizing specimen holder
Magnetizing specimen holder

~ 800 turns each
Manipulation with external fields
Vortices in Co structures

circular magnetic structures ⇒ newly found magnetic ground state

J. Raabe et al., J. Appl. Phys. 88, 4437 (2000);
T. Shinjo et al., Science 289, 930 (2000);

specimens: R. Sattler

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Analogon: phase shift vs. refraction

Wave optical representation: Phase shift due to enclosed flux $\Delta \varphi \propto B t \Delta x$
Action of "water dimples"

reflections of light in a pool
Action of "water dimples"
Action of "water dimples"
Action of "water dimples"
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Contrast from cylindrical magnetic disks

incoming electrons
ferromagnetic disk with flux closure
deflected electrons

Si$_3$N$_4$
Region of increased intensity
dark seam

Si$_3$N$_4$
Region of reduced intensity
bright seam
Moving vortices by magnetic fields

The rigid vortex model

local induction "grows", when external field is parallel

可能性 to move vortices with external fields

Please note: works also with other structures

Guslienko K.-Yu., Novosad V., Otani Y., Shima H., Fukamichi K.,
Internal structure of vortices

Vortices are "costly" in terms of exchange energy.

Less energy, when vortex core is trapped in central hole.
Internal structure of vortices

Vortices are "costly" in terms of exchange energy. Less energy is required when the vortex core is trapped in a central hole.


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creating vortex traps

Ni_{81}Fe_{19}

holes in magnetic disks – drilled using a FIB @

Elettra / Trieste (Italy) – act as traps for magnetic
Golf with vortices ...

-14 Oe
0 Oe
+52 Oe

2 µm

Note: movie can be seen at http://www.physik.uni-regensburg.de/forschung/zweck/Gruppe-ZweckPinning.htm

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What happens during magnetization reversal?

- large specimens (> 0.5 μm): movement of domain walls

- small specimens (< 0.5 μm):
  - different imaging technique (problem: defocus)
  - domain walls too costly in terms of wall energy

- what does a hysteresis loop look like in small particles?
- what micromagnetic configurations can occur?
- what do they depend upon?
Differential phase contrast (DPC)

- STEM mode (Scanning Transmission Electron Microscope)
- Beam shift leaves "diffraction disk" unaffected
- Stationary disk of homogeneous intensity
- Action of magnetism ??

Differential phase contrast (DPC)

difference signals of opposing detectors define coordinate system

induction is projected on coordinate system

obtain two signals proportional to $B_x$ and $B_y$

⇒ two images with directional information

directionality of contrast formation

magnetization of a domain wall

shift of the illuminated area on the detector

images of the x- and the y-component of the magnetic induction

reconstruction of the magnetic induction
Reconstruction from image pairs

20nm thick permalloy (Ni$_{80}$Fe$_{20}$) structures

$\mathbf{B}_{\text{total}} = \sum_{i,j} \mathbf{B}_{i,j}$
Individual hysteresis loops

- Image series acquisition
- Align the image with respect to each other
- Calculate the magnetic induction in the required area of each image
- Mask the required area

B - H

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Prof. Dr. Josef Zweck
Domain structures during hysteretic cycle

Uhlig, T., Zweck, J.
Domain structures during hysteretic cycle

Uhlig, T., Zweck, J.
Hysteresis loop of individual magnetic ring

"Onion state"

20nm thick permalloy ($\text{Ni}_{80}\text{Fe}_{20}$) structures

"Vortex state"

"Onion state"

magnetic induction/a.u.

applied magnetic field/Oe

1μm
hysteresis in magnetic rings: dependency on geometry

please note: effect of geometry w/respect to external field

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hysteresis in magnetic rings: dependency on geometry

please note: effect of geometry w/ respect to external field
Domain walls in magnetic rings

Domain walls in magnetic rings
Reversibility of switching?
Design of switching fields by artificial protrusions

Signal from vortex center and hole

a) edge wave effect signal arises from scattered electrons at specimens outer perimeter

b) edge wave effect signal arises from scattered electrons at specimens hole edge

c) magnetic signal change of induction direction causes deflection of beam & change of detector illumination

Vortex motion & pinning

\[ x = \frac{\Delta x}{R} \]
\[ y = \frac{\Delta y}{R} \]

\[ \text{Relative Vorticity} \]

\[ H_y [\text{Oe}] \]

\[ H_p \]
\[ H_d \]

a) b) c) d) e) f) g) h)

1 \mu m

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Reproducibility: Hall magnetometry (D. Weiss)

Rigid vortex model: linearity range

Position / %

Feldstärke / kA/m

Disk center

Range of linearity
Jump-like motion of a vortex

- $\Delta_y/R$
- $\Delta_x/R$

Movement occurs in tiny steps as field is varied
Jump-like motion of a vortex

- Movement perpendicular to field
  - Jump width up to ~ 100 nm
- Movement parallel to field

relative vortex position

coordinates

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Pinning @ local anisotropy

vortex has to match "energy landscape" given by local crystal (and other) anisotropies

- jump-like motion instead of continuous motion

low vortex energy w/respect to local anisotropy distribution

high vortex energy w/respect to local anisotropy distribution
Holography – a reminder

reference wave

object wave
Reminder: electron holography

equidistant spacing of interference fringes (cos² type)
Reminder: electron holography

electron interference fringes
Reminder: electron holography
Reminder: electron holography

non-equidistant spacing of interference fringes due to phase contour of object wave
Electron hologram of magnetic cylinder
Electron hologram of magnetic cylinder
Electron holography setup

following

Gabor's

idea, using a

Möllenstedt-Düker

type electron biprism

electron beam

specimen

lens

electron biprism

intermediate image (first hologram)

magnifying lens

hologram
Information propagation in holography

- Magnetic induction
- Phase shift of electron wave
- Shift of interference fringes
- Fourier transform
- Phase image from inverse Fourier transform
- Phase information in side bands

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interparticular stray fields

Phase x2, false colours

200nm
Permalloy with $t = 8$ nm

Phase x2, grey values
interparticular stray fields

Phase x2, false colours

200nm
Permalloy with t = 8 nm
interparticular stray fields

Michelangelo Buonarotti (1475 – 1564)

Sistine chapel, Rome (Italy)

Phase x2, false colours

200nm
Permalloy with t = 8 nm
Micromagnetic structure in small particles

phase shift due to

electrostatic potential  magnetic induction

incoming plane wave
specimen
exit wave front

incoming plane wave
specimen
exit wave front

=
Micromagnetic structure in small particles

Technique after Huhle and Dunin – Borkowski,
 specimen: H. Brückl, Bielefeld university

Dunin-Borkowski
R.E., Newcomb S.B.,
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M., Electron
Microscopy and
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Proceedings, 2001:
485-8, IOP
Publishing, Bristol,
UK

Zhao B.,
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Muhl T., Monch I.,
Vinzelberg H.,
Ritschel, M., Graff,
A., Huhle M., Lichte
H., Schneider C.M.,
AIP Conference
Proceedings, (633):
583-7 (2002)

sum = height profile

difference = induction
Micromagnetic structure in small particles

thickness profile

magnetic induction, superimposed on particle geometry

100 nm
Holography: remagnetization cycle

circular dot, $\varnothing = 600\text{nm}$, 20nm Permalloy

<table>
<thead>
<tr>
<th>7 kA/m</th>
<th>8 kA/m</th>
<th>16 kA/m</th>
<th>24 kA/m</th>
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<table>
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<th>-40 kA/m</th>
<th>-34 kA/m</th>
<th>-16 kA/m</th>
<th>0</th>
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Holography: nano hysteresis loops

complex evolution throughout magnetization reversal cycle

particle size ca. 250 nm

examination of one single particle only!
Holography: nano hysteresis loops

complex evolution throughout magnetization reversal cycle

particle size ca. 250 nm

examination of one single particle only!
Switching of individual particles

- produced in one single run
- nominally identical particles
- different switching behaviour
- 200 nm size!
REAL single domain behaviour!

Heumann, M., Uhlig, T., Zweck, J.,

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REAL single domain behaviour!

thermally activated switching

Heumann, M., Uhlig, T., Zweck, J.,
Perception of results

20nm thick permalloy (Ni$_x$Fe$_{1-x}$) structures

- Label i
- Label ii
- Label iii

- Magnetic induction (B) vs. applied magnetic field (H)
- 100nm
- 0.24 kA/m
- 0.32 kA/m

Dr. Thomas Uhlig

Dr. Martin Heumann

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Take home message

- magnetic information on a 10 nm level accessible
  - 5 nm in range for future investigations
- hysteresis loops of individual particles on a 100 nm length scale are possible
- design of magnetic properties by geometry optimization is possible
- true single domain switching & transition to superparamagnetism (thermal switching)
- non-continuous, step-like motion of vortices & validity of rigid vortex model