Magnetic Imaging Techniques

Laura Heyderman ETH Zurich & Paul Scherrer Institut, Switzerland



Types of Magnetism

Ferromagnetism

Helimagnetism

Antiferromagnetism

Ferrimagnetism

Spin-Canted Magnetism

Paramagnetism



Types of Magnetism















Types of Magnetism

Paramagnetism



Ferromagnetism



Antiferromagnetism



Ferrimagnetism



Spin-Canted



Helimagnetism





5

Imaging Techniques



Magnetic flux density (or induction): B = μ₀H + J H = magnetic field (external + stray field) J = magnetic polarization (magnetization μ₀M = q/V magnetic moment / volume) Maxwell's equation divB = 0, therefore: divH = -divM → any divergence of M creates a stray field H (zero external field)

Out-of-plane

Imaging techniques provide information on stray field, magnetization (magnitude and direction), magnetic induction (includes stray field and magnetization) at material surface or through a thin film

In-plane

$$\odot \otimes \odot \otimes$$





Imaging Techniques

Bitter Technique

Scanning Probe Microscopy

Electron Microscopy

Kerr Microscopy

X-Ray Microscopy Techniques



lacksquare

ASSEMBLED



Scanning Probe Microscopy



Scanning Probe Microscopy



If an atom was as large as a ping-pong ball...

...the tip would have the size of the Matterhorn!



E

- Stray field interaction between film and magnetic tip
- Forces are on the order of 10⁻¹⁰ N
- Employ a cantilever (CL) "spring"
- The force sensing carried out in two ways:
 - Static force sensing: CL brought near to surface and bends down or up (interaction attractive or repulsive). But CL might "snap" onto the surface.
 - Dynamic force sensing: CL is oscillated at a certain frequency, typically at its resonance frequency or a bit off (5%)



- > The cantilever is oscillated with a fixed amplitude (nm).
- > The sample is scanned by the XYZ scan piezo.
- The measurement signal is the frequency shift (shift of cantilever's resonance frequency), measured with a quadrant diode detector or laser interferometer.



SFM Dynamic Operation Modes

Measurement Signal:

Massless spring system → harmonic oscillator



near part of Taylor Expansion

$$f_{P} = \frac{1}{2\pi} \sqrt{\frac{1}{m} \left(c_{L} - \frac{\partial F}{\partial z} \right)}$$

$$\delta f \equiv f_P - f_0 \approx -\frac{f_0}{2c_L} \frac{\partial F}{\partial z}$$

interaction force ∂F ∂z gradient

nce frequency \rightarrow -ve freq. shift nce frequency \rightarrow +ve freq. shift Peter Kappenberger, Hans Hug **EMPA** Attractive forces \rightarrow lower resonance frequency \rightarrow -ve freq. shift Repulsive forces \rightarrow higher resonance frequency \rightarrow +ve freq. shift



The High Resolution MFM



6

High Resolution MFM: Requirements

- High aspect ratio tip with small apex diameter, small cone angle.
- Ultra-thin & smooth ferromagnetic coating (3-6nm).
- High measurement sensitivity limited only by thermal noise of cantilever due to gas molecules hitting cantilever. Therefore need to go to vacuum (results in a large Q, i.e. a low damping).



T.R. Abrecht, J. Appl. Phys. 69(2), p668 (1994)

Peter Kappenberger, Hans Hug EMPA



High Resolution MFM: Example

High resolution MFM images of hard disk media (sample by Seagate Research):



A. Moser et al. J. Magn. Magn. Mater. (2005)

ETH



In-Plane Structures with CNT Tip



Y. Lisunova, J. Heidler, I. Levkivskyi,I. Gaponenko, A. Weber, Ch. Caillier,L.J. Heyderman, M. Kläui and P. ParuchNanotechnology Accepted (2013)

High resolution with no vacuum!

Scale Bar: 100 nm



ETH GEO Magnetic Islands with EUV-IL





Co/Pd Multilayer on SiO_x Pillars

Period = 50 nm \rightarrow 263 Gbit/in² Diameter = 28.4 nm, σ = 5 %

H = -6.5 kOe

-7 kOe



MFM measurements Switched Islands:•





Mean switching field: 7200 Oe SFD (σ /mean) = 11.5 %



F. Luo, L. J. Heyderman, H. H. Solak, T.Thomson, M. E. Best APL (2008)



CoPt Multilayer in Hysteresis Loop



> Magnetization of layers from top to bottom.

- > Which grey level corresponds to which layer?
- ➤Take a calibrated tip and compare simulation with image.

layer 1 layer 2 layer 3 layer 4 layer 5 layer 6 layer 7 layer 8 layer 9 layer 10

Peter Kappenberger, Hans Hug EMPA



Simulation of MFM Images



12

Topography Separation

Magnetic contrast inverts when rotating the tip magnetization by 180°. Topographic contrast remains (van der Waals is always attractive).

⇒ Acquire two images with opposite tip magnetization states.



Peter Kappenberger, Hans Hug

EMD/

MFM Summary

- Maps the magnetic stray field (or derivative)
- Resolution: typically 20-30 nm, high resolution: 10 nm
- Non-destructive
- Especially sensitive to z-component of stray field: ideal for perpendicular anisotropy materials (e.g.magnetic media) but can also image domain walls in in-plane samples
- Requires virtually no sample preparation
- Surface should be relatively flat
- Tip quality is critical
- Influence of tip: difficult to measure magnetically soft samples



Electron Microscopy





Transmission Electron Microscopy

Transmission Electron Microscope



ETH



L. J. Heyderman

John Chapman

3

Lorentz Microscopy Classical Theory



- Electron voltage 100 200 keV
- Particle representation, corresponding to classical beam optics, the electrons are deflected by the Lorentz force:

F = q(v x B)

- q = electron charge
- v = electron velocity
- B = magnetic flux density
- With deflection angle: $\beta = (e \lambda B t)/h$
- Only components of B perpendicular to the electron beam are effective
- Includes stray fields outside sample



Fresnel and Foucault Microscopy



UNIVERSITY of GLASGOW



John Chapman, University of Glasgow

- Field Emission Gun source: small source angle
- Electron transparent phase shifting aperture: holes in SiN membrane, 50 nm thick, 200 keV, gives π phase shift of all magnetic spots
- Interference fringes parallel to induction direction with periodicity h/eBt



4 x 20 μm

t • In-line Holography !







Differential Phase Contrast





Fresnel Microscopy of Different Domain Walls





Fresnel Microscopy: Domain Walls in Multilayers

Single wall

Twin wall





5 um 2 Layers 3 Layers 4 Layers

Microstructure and Magnetic Information



Diffraction Pattern



Domain walls in $SrRuO_3$

A. F. Marshall et al. J. Appl. Phys. (1999)



Off-axis Electron Holography



E

R.E. Dunin-Borkowski, et al. Micr. Res. and Technique (2004)

- apply a voltage to an electron biprism
- overlap of part of a coherent electron wave that has passed through a sample with the part that has passed through vacuum only.
- Analysis of resulting interference pattern allows the phase shift of the electron wave to be recovered and a quantitative map of the in-plane induction can be obtained.
- This provides a high spatial resolution information about domains, domain walls and stray field interactions.



Off-axis Electron Holography

- High resolution ($\approx 5 \text{ nm}$)
- Magnetic induction: internal spin orientation + external stray field



R.E. Dunin-Borkowski, et al. Micr. Res. and Technique (2004)

Lorentz Microscopy Summary

- high spatial resolution (< 5 nm has been demonstrated)
- information on domain and domain wall structures
- straightforward image interpretation (usually)

- sensitive to induction (so contrast from sample magnetisation and stray fields)
- quantitative information on spatial distribution of integrated induction components
- suitable for real time studies involving field & temperature variation
- availability of complementary (perfectly registered) nanostructural information
- Sample must allow transmission of electrons



But.....

- phase shifts can be of magnetic or electrostatic origin, leading to severe problems when the latter contribution dominates
- no information about components of induction parallel to the direction of electron travel
- for multilayers, no way of separating contributions to images arising from individual layers
- no contrast from antiferromagnets
- time resolution is poor (typically ~1s, best ~20ms)

Kerr Microscopy


Michael Faraday (1791-1867)

small change of polarization plane due to magnetooptic interaction in transmission, circular birefringence, ~ M

> John Kerr (1824-1907)

John Kerr (1824-1907)

small change of polarization plane due to magnetooptic interaction in reflection, circular birefringence, ~ M

Rudolf Schäfer — IFW Dresden

1845





Rudolf Schäfer — IFW Dresden





Rudolf Schäfer — IFW Dresden





Rudolf Schäfer — IFW Dresden

Kerr Effect – Lorentz Concept



E

Rudolf Schäfer — IFW Dresden

Linearly polarized light will induce electrons to oscillate parallel to its plane of polarization – the plane of the electric vector E of light

Secondary motion is proportional to $-M \ge E$, and generates the Kerr amplitude, A_K , for reflection \rightarrow rotation of polarisation

Opposite M direction → opposite Kerr rotation



Kerr Microscopy

- Based on small rotations of the polarization plane of light
- Linearly polarized light will induce electrons to oscillate parallel to its plane of polarization – the plane of the electric vector E of light
- Regularly reflected light is polarised in the same plane as the incident light: A_N component
- Lorentz force induces a small component of vibrational motion perpendicular to original motion and to direction of magnetisation
- Secondary motion which is proportional to –M x E, and generates the Kerr amplitude, A_K, for reflection

Image Signal

If total signal amplitude:

 $A_{TOT} = A_N + A_K$ (normal & Kerr) Then Kerr Rotation:

 $\phi_{\mathsf{K}} = \mathsf{A}_{\mathsf{K}} / \mathsf{A}_{\mathsf{N}}$

Start with $\alpha_S = \phi_K$; extinguishes light from one domain; one domain appears dark and the other light

Actually better to rotate the analyser beyound the extinction point.

In practice, adjust polarizer and analyser until an image of satisfactory contrast and brightness is obtained.

http://www.physik.fu-berlin.de/~bauer/habil_online/node9.html



Hubert & Schäfer, Magnetic Domains



Polarising Microsope

http://physics.nist.gov/Divisions/Div841/Gp3/Facilities/kerr.html



http://www.fkf.mpg.de/kern/facilities/kerr/kerr.html



Digital Image Enhancement

Digital difference technique: non-magnetic background image is digitally subtracted



Images of 500 $\mu\,\text{m}$ wide NiFe square

0 Oe field, no scanned slit



0 Oe field



with confocal scanned slit

100 Oe field



Resolution approx. 200 nm

http://physics.nist.gov/Divisions/Div841/Gp3/Facilities/kerr.html

Resolution of optical microscopy (E. Abbe 1840 - 1905)





Twin Walls



Ferromagnetic Thin Film Non-magnetic Spacer Layer Ferromagnetic Thin Film

Image shows domain wall (black line) and quasi-domain wall (white line) in top layer of sandwich film



Time-Resolved Kerr Microscopy



Stroboscopic technique:

- Short field pulses (e.g. 20 ns, 10 kA/m) with copper microstrip line
- With a defined time delay, the magnetization is probed
- Reasonable signal-to-noise ratio by integrating the optical signal
- Accumulation over repeatable magnetization processes is required.
- Gated & intensified CCD camera providing temporal resolution down to 200 ps

Chumakov, McCord, Schäfer, Schultz

Time-Resolved Kerr Microscopy

Applied Field: quasi-static reversal



Applying a field pulse: magnetization not able to instantaneously follow magnetic field.

To reach new magnetization direction, will have to spin about the field axis: precessional motion that is gradually opposed by damping.

Reversal looks very different!



- Flat and smooth surface
- Resolution approx. 200 nm
- Magnetization can be observed directly
- Quantitative measurements possible but need to take care with calibration
- Observation does not influence magnetization
- Dynamic processes can be observed at high speed
- Sample may be easily manipulated: fields, high or low temperature, mechanical stress
- Surface magnetization: penetration depth of 10-20 nm
- Can look at back and front of sample

X-Ray Microscopy Techniques

ETH



The Swiss Light Source, Paul Scherrer Institut



- The electrons are accelerated close to the speed of light in a linear accelerator and injected into the storage ring
- Bending magnets or insertion devices (wigglers or undulators) cause electrons to bend or wobble through the section and emit light.



Reference energy: 2.4 GeV Circumference: 288 m Current: 350 mA (400 mA)



X-ray Absorption Spectroscopy





valence band

- Density of unoccupied states above Fermi level
- Each element: own characteristic peaks



PEEM & TXM





Photoemission Electron Microscopy



Slow electrons: mean free path is submono to several monolayers (few nm's)

Surfaces, thin films and interfaces

....consequences for electron optics.

Frithjof Nolting, Swiss Light Source



The Surface and Interface Microscopy (SIM) Beamline

SIM Beamline, Swiss Light Source

Close-up of the PEEM

The Photoemission Electron Microscope (PEEM)



		Slow	Electrons	10
Probe : slow electrons Imaging: high energy electrons (more stable and maintain spatial information) Lens Equivalent has two functions: accelerating field due to potential & focussing function				
0 eV Sample "integral part of lens	20 keV High Volt Ojective	20 ł tage / Lens	 High volta reduced to externa magnetic reduced spread ar electron b diameters 	ge: sensitivity al fields energy nd smaller beam
Immersion lens:electrons have before and after the lens different velocity (different wavelength)Cathode lens:Sample is cathode electron microscope is anode				rent Hevderman H



-. J. Heyderman



transmission of electrons with higher energy

Energy distribution is narrowed but transmission (intensity) is reduced. Therefore need to find compromise.



Effect of aperture size on resolution

ETH

- Spatial resolution depends on aperture size limits pencil angle of transmitted electrons and transmission
- Highest resolution is achieved with 12 μm aperture for PEEM2





Spatial Resolution for Magnetic Imaging

PEEM with X-rays: 50-20 nm spatial resolution

Aberration-corrected instruments using an electron mirror:

SMART (spectromicroscope for all relevant techniques) at BESSY II, Berlin, Germany collaboration of seven Universities in Germany PEEM III

at ALS, Berkeley, USA mainly ALS

down to a few nm spatial resolution



Photoemission Electron Microscope Arantxa Fraile-Rodriguez SIM beamline (SLS) **Frithjof Nolting**

X

analyzer

- elemental composition
- chemistry
- structural parameters
- electronic structure
- magnetic properties

16°

topography









Photoemission Electron Microscope Arantxa Fraile-Rodriguez SIM beamline (SLS) **Frithjof Nolting**

analyzer

- elemental composition
- chemistry
- structural parameters
- electronic structure
- magnetic properties
- topography





Topographical Contrast



Microfocussing due to distortion of the local electric fields



Photoemission Electron Microscope *Arantxa Fraile-Rodriguez Frithjof Nolting*



X-Ray Magnetic Circular Dichroism (XMCD)



- L-edge absorption in d band transition metal
- Magnetic metal: d valence band split into spin-up and spin-down with different occupation
- Absorption of right/left circular polarisation: light mainly excites spin-up/down photoelectrons
- Spin flips forbidden: measured resonance intensity reflects number of empty d-band states of a given spin
- Can determine sizes and directions of atomic magnetic moment







. J. Heyderman




















Rectangle, Square, Disk





Ring





Ring of Nanomagnets







J. Heyderman





Magnetic Structure: changing phase, changes polarisation

symmetric



Undulator







shift π/2 asymmetric









Strain and Magnetic Domains





Antidot Arrays – Basic Domain Configuration



Remanent Hysteresis Loop in Antidot Arrays



Observe magnetisation reversal in applied magnetic field:





L. J. Heyderman, F. Nolting, D. Backes, S. Czekaj, L. López-Díaz, M. Kläui, U. Rüdiger et al

Cobalt Antidot Arrays





Iron Nanoparticles Coupled to Cobalt Thin Film



Noncollinear alignment for particles > 6 nm



A. Fraile Rodríguez, A. Kleibert, J. Bansmann, A. Voitkans, L. J. Heyderman, and F. Nolting, PRL (2010)



Time Resolved Imaging



Image excitations in magnetic nanostructures

Precession frequency & damping

Pump-probe experiment

SLS: X-ray stroboscope

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

ETH Why perform time-resolved imaging?



Are all four feet of a horse off the ground at the same time during a gallop.

Galloping horse, animated in 2006, using photos by Eadward Muybridge, Wikipedia





L. J. Heyderman





L. J. Heyderman



Py Square: Excitation $ec{P}$ \vec{H}_{Pulse} 0ps

Permalloy (Ni₈₁Fe₁₉) t=30nm H_p~80 Oe ^{5040906_015}

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

Py Square: Excitation



- Element selective (multilayer, coupled systems)
- Surface/interface sensitive (sampling depth a few nm)
- Antiferromagnetic and Ferromagnetic domains
- Spatial resolution: 50-20 nm, future aberration corrected: few nm's
- Time resolved measurements
- Temperature 120 K 1000 K
- Submonolayer sensitivity
- Combination with other analytical techniques: LEEM & LEED
- In-situ and ex-situ sample preparation
- Sample size 3 to 15 mm diameter, 0.2 mm 2 mm thick Challenges (limitations):
- UHV compatible (<10⁻⁷ mbar)
- Smooth surface (< 1 μm, hard to say)
- X-ray damage

ETH

- Image in applied magnetic field below 50 Oe
- High voltage often leads to discharges (20 keV, at 2 mm distance)
- Charging effects due to electrical insulating sample (can get around this)



Transmission X-Ray Microscopy



Magnetic Soft X-ray microscopy, ALS



Peter Fischer, ALS



.. J. Heyderman

Peter Fischer, ALS



Fresnel Zone Plate Lenses

 $\Delta r = 25nm$ courtesy of E. Anderson (LBNL)

diffractive optics: concentric rings

- spatial resolution ~ Δr
- focal length $\sim N(\Delta r)^2/\lambda$
- spectral bandwidth $\Delta\lambda/\lambda \sim 1/N$





nature

Vol 435 30 June 2005 doi:10.1038/nature03719

LETTERS

Soft X-ray microscopy at a spatial resolution better than 15 nm

Weilun Chao^{1,2}, Bruce D. Harteneck¹, J. Alexander Liddle¹, Erik H. Anderson¹ & David T. Attwood^{1,2}



Magnetic absorption contrast



Polarization properties at bending magnet



Modulation of circ. polarization







- reducing non-magnetic background
- increase magnetic contrast

B.-S. Kang et al., J Appl. Phys **98** (2005) 093907



Magnetic imaging at 15 nm resolution





D.-H. Kim et al., J. Appl. Phys. 99, 08H303 (2006)

ETH Reversal at the nanogranular scale

- CoCrPt alloy films: possible high-density magnetic recording media:
 - strong perp. anisotropy
 - Iow media noise: exchange decoupled at grain boundaries
- Dark & white areas correspond to regions where the Co magnetization is pointing in and out.



Details of magnetization reversal for each grain: closely related to the size, irregularity, and stability of written domains.

D.-H. Kim et al., J. Appl. Phys. 99, 08H303 (2006)
Layer resolution via element specificity Pt/[Pt 0.75nm/Co 0.35nm]₅₀/Pt 3nm/Tb₄₅Fe₅₅ 25nm)/Pt 5nm





w/ S.Mangin, A. Berger, E. Fullerton (HITACHI/Almaden) (2005)

Time Resolved Imaging with STXM ¹⁴



PolLux Beamline, Swiss Light Source



. J. Heyderman



Towards fundamental time scales



Puzic et al.

JAP 2005

L. J. Heyderman





Summary

- Element selective (multilayer, coupled systems)
- Sample can be in air or HV
- Spatial resolution currently down to below 15 nm
- Sensitivity to out-of-plane magnetisation, but sample can be tilted
- Image in applied fields
- Time resolved measurements

Some Challenges (limitations):

- X-ray damage
- Sample must allow transmission of x-rays



Conclusion



Information on Different Methods

A. Hubert and R. Schäfer, Magnetic Domains The Analysis of Magnetic Microstructures

Magnetic Microscopy of Nanostructures An overview of techniques to image the magnetic structure on the nano-scale H. Hopster and H. P. Oepen

Internet, for example:

Techniques to Measure Magnetic Domain Structures, R.J. Celotta, J. Unguris, M.H. Kelley, and D.T. Pierce, Methods in Materials Research (2000)



Comparison Between Different Techniques

- Contrast Origin: B, M, H_{ext}
- > In-Plane or Out-of-Plane components
- > Quantitative or Qualitative
- > Best Resolution, but better Typical Resolution
- > Information depth
- > Sensitivity, Acquisition Time
- > Vacuum Equipment: none, HV, UHV
- Sample requirements: thickness, surface roughness, clean surface, insulators ?
- > In-situ experiments: maximum field, heating, stress
- > Additional information: crystallography, topography, chemical, electronic
- Commercial Availability, Cost & Complexity Manpower



Future Challenges

μm



- Currently sub100ps (≈10ps): precession relaxation dynamics (LLG).
- Limited flux of photons: repeatable phenomena (stroboscopic pump-probe).

ns



exchange interactions $t(fs) \sim \frac{4}{E(eV)}$



fs

• Future challenge: fs time scale (times associated with exchange interactions, spin fluctuation rates)

ps

- nm spatial resolution in single shot experiment.
- Need high flux (10¹²ph/s) X-ray source
- Lensless imaging and Full-field X-ray microscopy



magnetic tunnel junction

Peter Fischer, ALS

nm