

#### **Magnetic Measurements and Imaging**

#### J. McCord

Institute for Materials Science, Kiel University, Germany

## Magnetic measurements and imaging

#### Magnetic material properties (integral)

- Saturation magnetization
- Magnetic anisotropy constant(s)
- Magnetostriction constants
- Precessional frequency
- Magnetic damping parameter



Magnetic imaging (spatial)

C|AU

#### Outline

#### Magnetic fields

- Quasi-static measurements
- Dynamic measurements

#### Far from being complete!

- Quasi-static magnetic domain imaging
- Imaging of magnetization dynamics

#### **Magnetization loops**



- Magnetic field H
- Magnetization M(H) (or flux densitiy B(H))
  - Not an intrinsic property of the material
  - Character of M(H) loop changes with sample preparation and shape

### **Generation of magnetic fields**

- Various methods of producing magnetic fields
- Important parameters
  - Field intensity (what kind of sample is investigated)
  - Volume (depending on sample)
  - Uniformity
- Resistive solenoids
- Electromagnets
- Superconducting coils
- Pulsed magnetic fields
- Permanent magnets

## **Solenoids**

	Single-layer solenoid and its magnetic field distribution.	$\frac{H}{H_{inf}} = 0.5$	$L \rightarrow D$
L/D	H at Center	<i>H</i> at Edge of Middle Half	
5	0.9806 H <sub>inf</sub>	0.9598 H <sub>inf</sub>	$H = \frac{H \cdot I}{L} \left[ \frac{L + 2X}{\sqrt{L + 2X}} + \frac{L + 2X}{\sqrt{L + 2X}} \right]$
0	0.9950	0.9892	$L   2\sqrt{D^2 + (L + 2x)^2} 2\sqrt{D^2 + (L - 2x)^2}  $
20	0.9987	0.9972	
50	0.9996	0.9994	$H_{center}(x=0) = \frac{H \cdot I}{L} \left[ \frac{L}{\sqrt{D^2 + L^2}} \right] \approx \frac{H \cdot I}{L}$

#### Helmholtz coils



- High magnetic field homogeneity over large colume
- Usally small fields (smaller than solenoid)
- Investigations of soft magnetic materials

#### **Electromagnets**

CAU



Electromagnets

Pole piece,

or cap

## **Traditional electromagnets**

- Magnetic DC fields up to 30 kOe (3 T)
  - Higher than saturation polarization of yokes
- Magnetic field depends on
  - Current
  - Permeability and saturation magnetization of yoke and pole tips
  - Gap width
- Joule heating
  - Limited by insulation of copper wires of coils
  - Cooling of coils (water cooling)





#### Limitations of electromagnets



#### http://www.technicoil.com

## High field selonoids (up to 45 T)

- Magnetic DC fields up to 450 kOe (45 T)
  - Stacked Bitter plates

Probe

- High strength low resistivity Cu alloys
- Axial water flow

Coils

**Electric Cables** 

Resistance to Lorentz force and magnetic clamping



Water Pipes





#### https://nationalmaglab.org/

## **Superconducting solenoids**



- Elimination of Joule heating
- Applications (incl. cryostats)
  - Nuclear magnetic resonance (NMR) systems
  - Scientific magnetometers (SQUID)
- Low power to maintain magnetic field
- High inductance slow ramp rates
- Materials (high critical field H<sub>c</sub>!)
  - $Nb_3Sn H_c = 22 T @ 4.2 K$
  - NbZr or NbTi

20 T @ 4.2 KCurrent 120 A Voltage < 10 V Inductance L  $\approx$  240 H





## Pulsed magnetic field magnets (100 T)



- Larce capacity discharge RLC circuit
  - Stored energy of capacity bank ("B")
- Pulsed field with short duration (typ. milliseconds)
- High mechanical strength of coil ("D") needed

 $E_{\rm tot} = 50 \text{ MJ}$  $I_{\rm max} = 100 \text{ kA}$ 

## Pulsed magnetic field magnets



- Non-destructive short pulses
- Mechanicaly reinforced magnet design





## Small coils – high pulsed fields

11

10

9

8

7

6

5

4

3

2

0

0

B (T)

- Bi-polar pulsed magnetic field sources
- Coolant-free
- Compact in size: cm<sup>3</sup>
- High working field: 10 T
- Works in your laboratory!





## Waveguides



- Impedance matched coplanar waveguide (or similar)
- Local magnetic rf field sources



#### Permanent magnet systems



## **Overview on magnetic field generation**

CAU

	Maximum field	Duration	High field laboratories
Electromagnetic flux compression	600 T	10 <sup>-6</sup> s	Tokyo
Pulsed coils (destructive)	300 T	10 <sup>-6</sup> s	Tokyo, Toulouse, Los Alamos
Pulsed coils	> 100 T	10 <sup>-3</sup> to 1 s	Los Alamos
Hybrid magnet (resistive + superconducting)	45 T	static	Tallahassee
Superconducting magnets (conventional)	22 T	static	commercial
Pulsed magnetic field coils	10 T	10 <sup>-5</sup> s	commercial
Coils with yoke, Halbach cylinders	2 T - 3 T	static	commercial
Helmholtz coils	$\approx 0.2 \text{ T}$	static	commercial
Micro strip lines	0.1 T	<100 psec	commercial

Comparison of the generation of magnetic fields.

Laboratory

#### Magnetic field measurements

#### Measurement of magnetic field strengths

- Hall probes ("Gaussmeters")
- Magneto-restive devices
- Flux gate sensors
- Induction coils
- SQUID sensors
- Only very rarely the magnetic field can be calculated!
- Most apparatus need to be calibrated!

V.19

### Hall probes



$$V_{H} = \mu \frac{W}{I} V_{0} B_{z}$$

carrier mobility  $\mu$ length l width w supply voltage  $V_0$ 

	carrier mobility
InSb	80.000 cm²/Vs
InAs	33.000 cm²/Vs
GaAS	8.500 cm²/Vs
Si	2.000 cm <sup>2</sup> /Vs



- Experience Lorentz force at charge carriers with perpendicular magnetic field
  - Results in Voltage V<sub>H</sub>
     perpendicular to current and field
  - Temperature dependent
  - High spatial resolution

## Hall probes

#### Specs

- Typical range from 0.01 mT to 30 T
- Accuracy better than 0.1 %
- DV to 30 kHz
- Micro Hall probes for characterization of nanomagnetic phenomena
- Scanning Hall probe microscopy (SHPM)







#### Measurements of magnetization loops (quasi-static)

- Vibrating sample magnetometer (VSM)
- Alternating gradient force magnetometer (AGM)
- SQUID magnetometer
- First order reversal curves (FORC)
- BH Looper (inductive)
- Magneto-optical methods

#### Measurements of magnetization dynamics

Ferromagnetic resonance

- Methods for determining magnetization are divided into closedcircuit and open circuit measurements
  - Sample is (or not) a part of a complete magnetic circuit

Method	Open/closed circuit	Typical sensitivity
Vibrating sample	Open	$10^{-9} \text{ Am}^2$
Alternating gradient force	Open	$10^{-10} \text{ Am}^2$
BH-Looper	Open	$10^{-9} \text{ Am}^2$
SQUID	Open	$10^{-11} \text{ Am}^2$
Magneto-optics	Open	$10^{-11} \text{ Am}^2$
Hysteresigraph	Closed	$10^{-4}  \text{Am}^2$
Faraday	Open	$10^{-6} \text{ Am}^2$

Comparison of methods for measuring magnetization loops of magnetic materials (adapted from M. Coey, Magnetism and Magnetic Materials, Cambridge University Press 2010)

- Magnetic poles induced on the sample surface
- Demagnetization factor N depending on sample shape
- N for elliposids exactly defined

```
N_a + N_b + N_c = 1
(a, b, c: ellipsoid axes)
```

Ellipsoid with dimensions a and c.

- "Flat disk"  $N_{\rm c} \approx 1$ ;  $N_{\rm a} = N_{\rm b} \approx 0$
- Sphere  $N_{\rm a} = N_{\rm b} = N_{\rm c} = 1/3$
- "Cylinder"  $N_{\rm c} \approx 0$ ;  $N_{\rm a} = N_{\rm b} \approx \frac{1}{2}$

Sheared magnetization loops  $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow \downarrow$   $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow \downarrow$   $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow$   $\downarrow \downarrow \downarrow \downarrow \downarrow \downarrow$   $\downarrow \downarrow \downarrow \downarrow \downarrow$  $\downarrow \downarrow \downarrow \downarrow \downarrow$ 

**Closed circuit** 

CAU

В

#### J. Appl. Phys. 79 (8), 15 April 1996

#### The vibrating sample magnetometer: Experiences of a volunteer (invited)

S. Foner

Francis Bitter National Magnet Laboratory and Department of Physics, MIT, Cambridge, Massachusetts 02139

#### Scientific inventions can happen at home!

That summer we were living in a small farm-hand's house about 1 km from the laboratory and just off the runway at Hanscom Field, a Strategic Air Command (SAC) base at that time. While shaving one evening I decided to try ac induction for magnetic measurements. With some Duco cement, a small \$2.00 (in 1955) replacement loudspeaker, a conical paper cup, and a paper straw (the latter components were light and conveniently available at night from the lunch room), the first working model VSM was assembled.

#### Flux detection method!

A simple, inexpensive, and versatile instrument!

#### **Basic components of a VSM**





#### Vibrating sample magnetometer

Mechanical details ⊛ of a VSM 2a |2C 2a \* 2a **Scalar coils Vector coils** Moment(emu) Examples of VSM 0.003 coil configurations 0.002 0.001 0.000 -0.001 0 -0.002 -2000 -1000 4000 5000 4000 3000 0 1000 2000 3000 Field(Oe) coercivity(Hci): 1.69 kiloOe Magnetization: 0.00294 emu Retentivity(Mr): 0.00279 emu Switching Field Distribution 2: 0 Squarness Ratio: 0.947 S\*: 0.951

Magnetization loop for hard disk CoPt magnetic film deposited on a rigid disk substrate. M(H) loop parameters are indicated in the figure.

C|AU

#### Example VSM loops – vector magnetometry

CAU



- Soft magnetic thin film with anisotropy dispersion
- Also earth field present!

 $M_x$  hysteresis and  $M_y$ -hysteresis loops of a Permalloy film with slightly varying magnetic field angles applied close to the hard axis of magnetic anisotropy.



IEEE Summerschool 2017 McCord

## Alternating gradient force magnetometer (AGM)



## **SQUID** based magnetometry

SQUID **Superconducting Quantum** SAMPLE SPACE **Interferences Device** SAMPLE CHAMBER TUBE ISO-THERMAL SHEET WITH INNER VACUUM JACKET WALL HEATER **ANNULAR COOLING REGION** Based on two parallel OUTER VACUUM JACKET WALL SUPERINSULATION CCW Josephson junctions Measures magnetic fields changes of the order of a CW quantum flux SAMPLE MULTIFILAMENT EMU-1.526500E - 002 CW SUPERCONDUCTING WIRE PICK UP COILS DEV 0.000000E (en composite form) 5 3 VOLTAGE (V) COMPOSITE FORM CCW FOR SOLENOID -1 2<sup>nd</sup> order gradiometer 0 2 3 5 SCAN (cm) superconducting M. McElfresh, Fundamentals of **SQUID Response** magnetism and magnetic detection coil) (voltage vs. sample position) measurement systems (1994)

#### micro-SQUID



CAU



- Measuring single nano objects
  - Switching of the magnetization of single Ni wires (diameter 65 nm)

W. Wernsdorfer, Phys. Rev. Lett. 77, 1873 (1996)



## First order reversal curves (FORC)

JOURNAL OF APPLIED PHYSICS

VOLUME 85, NUMBER 9

1 MAY 1999

 $M(H_{\rm a},H_{\rm b})$ 

C|A|U

# Characterizing interactions in fine magnetic particle systems using first order reversal curves

Christopher R. Pike<sup>a)</sup> Department of Geology, University of California, Davis, California 95616

Andrew P. Roberts Department of Oceanography, University of Southampton, Southampton Oceanography Centre, European Way, Southampton SO14 3ZH, United Kingdom

Kenneth L. Verosub Department of Geology, University of California, Davis, California 95616

(Received 1 June 1998; accepted for publication 1 February 1999)

- Minor loop measurements
- Saturation field H<sub>sat</sub>
- Reversal field H<sub>a</sub>
- Field swept back H<sub>a</sub> to H<sub>b</sub>
- *M*(*H*<sub>a</sub>,*H*<sub>b</sub>)



## **FORC – Forc distribution** p

$$M(H_a, H_b) \longrightarrow \rho(H_a, H_b) = -\frac{1}{2} \frac{\partial^2 M(H_a, H_b)}{\partial H_a \partial H_b}$$
  
Fransformation (common)

Distribution coercive fields  $H_c = (H_b - H_a)/2$ 

- One measurement can take days!
- Distribution of interaction or reversal fields  $H_{\rm u} = (H_{\rm b} + H_{\rm a})/2$



## **BH-Looper - Hysteresis loop tracer**

-4 -3 -2 -1 0 1 2 3 4

 $H_{\rm ext}$  (kA/m)

CAU

- Sample in AC field
- Stray field picked up in vicinity of sample
- **Balance induction**

 $U_{\text{sample}} = U_{\text{sense}} - U_{\text{balance}}$ 

0.0

**Real time hysteresis measurements** (10 Hz) 1.0 0.5





BH-Looper principle - A pickup coil senses the flux density of a magnetic sample. A balance coil picks up the induction due to the drive field.

## **Magneto-optical magnetometry**

#### Mostly based on the magneto-optical Kerr effect (MOKE)

- Plane of polarization of light is rotated when light is reflected from a magnetic material surface
- Other effects exist!

#### Surface sensitive

- Depth of information approx. 30 nm
- Very high sensitivity for magnetic thin films

#### Also used for magnetic domain imaging!

Geometry	Physical phenomena	Dep. with <i>M</i>
M    k	Magnetic circular birefringence (MO Earaday Effect and MO Kerr Effect)	linear
M    k	Magnetic circular dichroism	linear
$M \perp k$	Magnetic linear birefringence (MO Voigt Effect)	quadratic
$M \perp k$	Magnetic linear dichroism	quadratic
$\partial M \perp k$	Gradient contrast (MO Gradient Effect)	differential
## **Description of magnetooptical effects**

$$D = \varepsilon_{total} E$$

$$\varepsilon_{total} = \varepsilon \begin{pmatrix} 1 & -iQm_{3} & iQm_{2} \\ iQm_{3} & 1 & -iQm_{1} \\ -iQm_{2} & iQm_{1} & 1 \end{pmatrix} + \begin{pmatrix} B_{1}m_{1}^{2} & B_{2}m_{1}m_{2} & B_{2}m_{1}m_{3} \\ B_{2}m_{1}m_{2} & B_{1}m_{2}^{2} & B_{2}m_{2}m_{3} \\ B_{2}m_{1}m_{3} & B_{2}m_{2}m_{3} & B_{1}m_{3}^{2} \end{pmatrix}$$
Faraday or Kerr effect Voigt effect

"Circular birefringence" ~ M "Linear birefringence" ~  $M^2$ 

- Electric vector of light wave E
- Dielectric tensor  $\varepsilon$
- Dielectric displacement vector D
- Magnetization vector components *m*
- Complex Voigt constant Q
  Faraday and Kerr effect
- **Complex material constants B1, B2** Voigt effect

#### **MO effects – MOFE and MOKE**



#### Faraday effect (MOFE)

Longitudinal Faraday and Kerr effect under an angle of incidence  $\theta_{inc}$  relative to the surface normal. The case of spolarization is sketched. By the MO interaction with the magnetic medium the linearly polarized incoming light ( $E^{inc}$ ) is transformed to an elliptically polarized light  $E^{trans}$  and  $E^{refl}$ . The resulting Faraday rotation  $\theta_{f}$  and ellipticity  $e_{f}$ , respectively, Kerr rotation  $\theta_{k}$ and ellipticity  $e_{k}$  are shown.  $\theta_{refl}$  is the angle of reflection of light.

#### **Kerr effect (MOKE)**

J.McCord, Journal of Physics D: Applied Physics 48, 333001 (2015)

CAU



The three basic configurations of the (a) polar, (b) longitudinal, and (c) transverse magnetooptical Kerr effect. The unit vector of magnetization m is lying along the corresponding sensitivity axes (as indicated).

> J.McCord, Journal of Physics D: Applied Physics 48, 333001 (2015)

#### **Typical simple MOKE magnetometry setup**

. . 121 less on • 25 • mm • av 1 de. Net rate

J. M. Teixeira et al. Review of Scientific Instruments 82, 043902 (2011)

### **Typical detection schemes**



Direct detection (upper) and bi-channel homodyne (lower) schemes use low-noise lasers (PBS: polarizing beam-splitting cube).

Practical opto-electronics, Springer (2014) V.V. Protopopov, Magneto-optics

CAU



V.V. Protopopov, Magneto-optics

Angular dependence of MO signals for direct and bi-channel detection schemes.

CAU

#### J. Hamrle et al., Phys. Rev. B 66, 224423



Example of polar Kerr rotation hysteresis loops measured at several photon energies E on the  $(TbFe/Si_3N_4)_4$  sample. Each step in the hysteresis loop corresponds to a MOKE signal coming from a given TbFe stack.



### High resolution MOKE magnetometry

CAU



- Permalloy
  - thickness t = 5 nm
  - width w = 200



Phys. 36 (2003) 2175–2182



#### **Magnetization dynamics**

 Described by Landau-Lifschitz-Gilbert equation of magnetization dynamics

$$\frac{d}{dt}\vec{M} = -\gamma \vec{M} \times H_{\text{eff}} + \frac{\alpha}{M_{\text{s}}} \left(\vec{M} \times \frac{d}{dt}\vec{M}\right)$$
Precession

Relaxation

Precession of magnetization including damping

$$f_{\rm res} = \frac{\gamma \mu_0}{2 \pi} \sqrt{M_{\rm s} H_{\rm eff}} \sim \sqrt{H_{\rm eff}}$$

**Precessional frequency** *f*<sub>res</sub>

$$\alpha_{\rm eff} = \frac{2}{\tau \gamma \mu_0 M_{\rm s}} \sim \frac{1}{\tau}$$

Damping parameter  $\alpha$  (small fields)



C|AU

## **Ferromagnetic resonance (FMR)**

#### **FMR** measurement modes

- Magnetic field sweep with constant frequency
  - Standard FMR technique
  - High sensitivity through resonant cavity (resonator)

- Frequency sweep with constant magentic field
  - Broadband sources needed
  - Allows for zero field measurements

#### Material from K. Lenz, HZDR,







#### **Cavity based FMR**

#### CAU

Sample

TE<sub>011</sub>

erexses

#### Resonator with microwave bridge

- Sensitivity: ~10<sup>10</sup> spins
- x-band FMR @ 9 GHz
- Up to 500 GHz



#### FMR data

- Microwave absorption ~ susceptibility
- rf-susceptibility (imaginary part)

$$\chi(\omega) = \frac{\gamma M_s}{\gamma H_{eff} + i\alpha\omega} \left( 1 + \frac{\omega^2}{(\gamma M_s + \gamma H_{eff} + i\alpha\omega)(\gamma H_{eff} + i\alpha\omega) - \omega^2} \right)$$



Measurement signal is a Lorentz curve

- Resonance field H<sub>res</sub>
- Line width  $\Delta H$
- Amplitude A

 $H_{\rm res} \rightarrow {\rm anisotropy}$ 

 $\Delta H \rightarrow$  damping parameter

Material from K. Lenz, HZDR, Germany

#### **Broadband FMR**

- Tunable microwave source (Vector Network Anlyzer (VNA))
- Excitation through wave guide (antenna)
- Measuring of transmission and reflection coefficients
  - Transmission  $S_{ij}$  (i  $\neq$  j) and reflection coeff.  $S_{ii}$  (i = j)





### FMR & MOKE

CAU



IEEE Summerschool 2017 McCord

#### ... definitely not a complete list ...

Method	Remarks	Typical sensitivity
Vibrating sample	Slow	$10^{-9} \text{Am}^2$
Alternating gradient force	Fast	$10^{-10} \text{ Am}^2$
BH-Looper	Even faster	$10^{-9} \text{Am}^2$
SQUID	Highest sensitivity	$10^{-12} \text{Am}^2$
Magneto-optics	Fast and high surface sensitivity	10 <sup>-15</sup> Am <sup>2</sup>
FMR	Dynamic properties	$10^{-16} \text{ Am}^2$

... papers, papers, and papers ...

- Review of Scientific Instruments (AIP Publishing)
- Web-presence of vendors
- Coey, John MD. Magnetism and magnetic materials. Cambridge University Press, 2010.
- Cullity, Bernard Dennis, and Chad D. Graham. Introduction to magnetic materials. John Wiley & Sons, 2011.

#### Outline

- Magnetic fields
- Quasi-static measurements
- Dynamic measurements
- Quasi-static magnetic domain imaging
- Imaging of magnetization dynamics

#### Again, far from being complete!

#### **Magnetic domains**

CAU

### **External field energies** Zeemann energy – M(H)Shape E-field induced anisotropy $K_{\mu}(E)$ Local demagnetization field Magnetostrictive self energy **MO Kerr effect image** Film thickness and layering Anisotropy Domain wall structures Magnetic uniaxial anisotropy $K_{\rm u}$ Unidirectional anisotropy $K_{ud}$ Stress induced anisotropy $K_{\sigma}$ **Parameters may vary laterally!**

# Magnetic measurements and imaging

- Magnetic material property measurements
- Magnetic imaging
  - Quasi-static and dynamic imaging methods
- Vary by ...
  - ... contrast mechanism (M, dM/dx, ...)
  - ... spatial resolution
  - ... temporal resolution

... it is all about magnetic domains ...



#### Imaging methods





R. Celotta et al., Techniques to Measure Magnetic Domain Structures. Characterization of Materials. 1–15. Wiley (2012)

Images from several magnetic domain observation techniques from bits written on a magnetic storage media. The track width is about 10  $\mu$ m wide. The bit length ranges from 10.0 to 0.2  $\mu$ m. The bit length and spacing of the large bits in the XMCD electron yield image is 10  $\mu$ m.

## Magnetic domain imaging techniques

CAU



Magnetic force microscopy (MFM)X-Ray microscopyScanning electron microscopy (SEMPA)Transmission electron microscopy (TEM)

## Imaging methods

CAU

	Bitter pattern	MFM	SEM-I Secondary	TEM Fresnel and Economit	TEM DPC	TEM Holography	SEM-II Back- scattered	SEMPA	SPLEEM	MOKE (Kerr microscopy)	XPEEM	STXM <sup>(a)</sup>
Contrast mechanism	$\nabla \mathbf{B}_{ext}$	$\nabla \mathbf{B}_{ext}$	∇B <sub>ext</sub>	B	В	<b>Β</b> ,Φ <sub><i>B</i></sub>	В	М	М	М	М	М
Evaluation of the magnetization, M(r)	Indirect	Indirect	Q	Indirect	Q	Q	Q	Q	Q	Q	Q	Q
Spatial resolution (nm) Typical Limit	$\frac{300}{80}$	$\frac{60}{20}$	$\frac{1000}{800}$	$\frac{50}{\sim 10}$	$\frac{10}{2}$	$\frac{20}{5}$	$\frac{2000}{1000}$	$\frac{150}{20}$	$\frac{40}{20}$	$\frac{800}{250}$	$\frac{300}{50}$	$\frac{30}{15}$
Depth of information (nm)	500– 1000	20–500	10–50 nm	Thickness integrated <150 nm	Thickness integrated <100 nm	Thickness integrated <100 nm	10,000	1–2	<1	<20 (metals)	<5 nm	Thickness integrated <100 nm
Time for image acquisition	30 msec	5–20 mins	10-60 sec	50 msec- 60 sec	5-60 secs	50 msec- 10 sec	10-60 sec	1–100 min	~1sec	1 msec-5 sec	~1sec	~1sec
Limits on applied magnetic fields	None	<500 kA/m	Not advised	~100- 500 kA/m	~100– 500 kA/m	50 kA/m	Not advised	Not advised	Not advised	None	Not advised	None
Imaging conditions	None	In air	HV	HV	HV	HV	HV	UHV	UHV	In air	UHV	None
Max thickness	None	None	None	<150 nm	<100 nm	<100 nm	None	None	None	None		60–100 nm
Sample smoothness	R	R	NR	Preferred	Preferred	Preferred	NR	NR	R	R	NR	NR
Sample clean surface	NR	NR		Preferred	Preferred	Preferred	NR	R	R	NR	Preferred	NR

K. Krishnan, Fundamentals and applications of magnetic materials, OXFORD University Press (2016)

### Elements of magnetic force microscopy



Material from L. Abelmann, Univ. Twente, Netherlands & KIST Europe, Germany

## **MFM - Dynamic mode operation**

C|AU

#### Harmonic oscillator



Probing of magnetic interaction



## **Basic principles of MFM**

- Stray field interaction between film and magnetic tip
- Small forces on the order of 10<sup>-10</sup> N detected with cantilever
  - Dynamic force sensing: Cantilever is oscillated at a frequency close to its resonance frequency – detection of frequency shift
- Lift mode (non contact mode)
  - Tapping mode (AFM mode)
  - Subsequent MFM trace



## Transitions in perpendicular recording media





A. Moser et al., JMMM 287 (2005) 298–302

kfci (kilo flux changes per inch)

## Electrical switching of a vortex core seen by MFM



- Maps the derivative of the magnetic stray field
- Spatial resolution down to 10 nm (vacuum)
- Non-destructive
- Mostly sensitive to the z-component of stray field (depending on tip)
- No sample preparation needed
- Surface should be relatively flat
- Tip quality is critical
- Tip-sample interaction: induced change of magnetic state
- Limitations to measure magnetically soft samples

#### **TEM–** Lorentz microscopy

	TEM Fresnel and Foucault	TEM DPC
Contrast mechanism	В	В
Evaluation of the magnetization, M(r)	Indirect	Q
Spatial resolution (nm) $\frac{\text{Typical}}{\text{Limit}}$	$\frac{50}{\sim 10}$	$\frac{10}{2}$
Depth of information (nm)	Thickness integrated <150 nm	Thickness integrated <100 nm
Time for image acquisition	50 msec- 60 sec	5-60 secs
Limits on applied magnetic fields	~100– 500 kA/m	~100– 500 kA/m
Imaging conditions	HV	HV
Max thickness	<150 nm	<100 nm
Sample smoothness	Preferred	Preferred
Sample clean surface	Preferred	Preferred

$$F = |e|(\mathbf{v} \times \mathbf{B})$$

**Lorentz force** 



$$\beta_L = \frac{e\lambda(\mathbf{B} \times \mathbf{n})}{h} t \approx 100 \,\mu rad$$

**Lorentz deflection angle** 

#### LTEM– Lorentz microscopy

CAU



Schematic of ray diagram indicating the paths followed by electrons passing through a magnetic specimen, together with the contrast that would be seen in the image for the Fresnel and Foucault modes of LTEM

after A.K. Petford-Long & J.N. Chapman, Lorentz microscopy, in Magnetic Microscopy of Nanostructures, Springer (2005)

#### Domain walls in thin films (Fresnel mode)

CAU



10 µm

, Material from L. Heyderman, ETH Zürich, Switzerland

IEEE Summerschool 2017 McCord

## LTEM imaging – Fresnel mode

#### T = 250K ~130 Oe (remanance)





# Helical structures in FeGe



Material from S. McVitie, Univ. Glasgow, UK

#### LTEM imaging – Fresnel mode

796 Oe

# Helical structures and skyrmions in FeGe



Material from S. McVitie, Univ. Glasgow, UK

### LTEM imaging – Fresnel mode

1938 Oe

# Skyrmion lattice in FeGe



Material from S. McVitie, Univ. Glasgow, UK

**IEEE Summerschool 2017** 

#### **Differential Phase Contrast**



A.K. Petford-Long & J.N. Chapman, Lorentz microscopy, in Magnetic Microscopy of Nanostructures, Springer (2005)

#### **Lorentz STEM – Differential Phase Contrast**

CAU

In-plane magnitude

#### Skyrmion lattice



Material from S. McVitie, Univ. Glasgow, UK
### Analysis of Skyrmion structure



- Lorentz deflection angle +/- 4 µradians equiv. to B<sub>s</sub>~0.2 Tesla
- Six-fold symmetry

Material from S. McVitie, Univ. Glasgow, UK

# LTEM summary

- High spatial resolution (better than 5 nm possible)
- Information on domain and domain wall structures
- Thin films
- Sensitive to induction (sample magnetization & stray fields)
- Quantitative information
- Real time studies (magnetic field & temperature)
- Complementary structural information
- Limited to thin samples
- Magnetization components parallel to the electron beam invisible
- Sample preparation is critical!

# **SEM** with polarization analysis



CIAU

#### SEMPA images all three components of magnetization vector and intensity













#### **Derive In-plane magnetization direction**





Intensity

Material from J. Unguris, NIST, USA

CAU

#### In-plane spin analyzers measure chirality:









#### Out-of-plane analyzers measure vortex core polarity







Chung et al, Ultramicroscopy 110, 177-181 (2010).



Contrast is reduced by non-local secondary electrons generated by backscattered electrons.

#### Material from J. Unguris, NIST, USA

# Depth profiling magnetization in Co/Ru/Co

#### Ion mill with Ar ions



#### Material from J. Unguris, NIST, USA

IEEE Summerschool 2017 McCord

# Ferromagnetic & ferroelectric structure in multiferroics

**CoFe/BiFeO**<sub>3</sub> (R. Ramesh, Berkeley)

SEM back scattered electron signal

SEMPA

Material from J. Unguris, NIST, USA

Unguris et al, APL Materials 2 (2014)

Trassin et al, Phys. Rev. B 87 (2013)

Zhou et al, Nature Comm. 6 (2015)

IEEE Summerschool 2017 McCord

CIAU

C|AU

### Patterned CoFeB (2 nm) on Ta wires

- Direct visualization of Spin Hall effect induced switching
- Pt and Ta have opposite spin Hall effects and therefore show opposite switching



I. Gilbert *et al.*, *Phys. Rev. B* 94, 094429 (2016).

#### Material from J. Unguris, NIST, USA

IEEE Summerschool 2017 McCord



- High spatial resolution (better than 5 nm possible)
- Information on domain and domain wall structures
- Sensitive to induction surface magnetization
- Quantitative information
- Complementary structural information
- Limited to clean surfaces in ultra high vacuum

### **Magneto-optical microscopy**

CAU

	MOKE (Kerr micros	copy)
Contrast mechanism	М	
Evaluation of the magnetization, M(r)	Q	
Spatial resolution (nm) $\frac{\text{Typical}}{\text{Limit}}$	$\frac{800}{250}$	
Depth of information (nm)	<20 (m	etals)
Time for image acquisition	1 msec-	-5 sec
Limits on applied magnetic fields	None	
Imaging conditions	In air	
Max thickness	None	
Sample smoothness	R	
Sample clean surface	NR	Topical Review J. McCord.
		J. Phys. D: Appl. Phys. 48,



333001 (2015)

# **Spatial resolution of MO imaging**

δ μm	Α μη	$f_{ext}$ $h_{ext}$ $h_{e$
Stripe domains in	n Interaction domains	Magnetic domains in
Ni <sub>81</sub> Fe <sub>19</sub>	in Nd <sub>2</sub> Fe <sub>14</sub> B	a angle sensor
Domain width 250	nm Domain width 400 nm	Stripe width 300 nm
		Localization accuracy
	(sample O. Gutfleisch	10 nm
200 nm	TU Darmstadt)	(sample R. Mattheis,
solution		IPHT Jena)

J. McCord, J. Phys. D: Appl. Phys. 48, 333001 (2015)

# Stochastic domain wall propagation in field sensors

CAU



#### Extracting domain wall position after CW and CCW rotation



CAU

J. McCord, J. Phys. D: Appl. Phys. 48, 333001 (2015)



Current induced magnetic domain wall displacements (a) MOKE images displaying domain wall displacements in  $Pt/Co/Al_2O_3$  magnetic nanowires induced by nanosecond current pulses. (b) Current induced domain motion versus total current pulse length for Ir/Co/Ni/Co and Au/Co/Ni/Co stacks.

# LARGE view MOKE microscopy



#### 20 mm

FeCoSiB 4 µm

CAU

#### Noise mechanism in FM/FE composite magneto-electric sensors

J. McCord, J. Phys. D: Appl. Phys. 48, 333001 (2015)

# Multi-effect MO imaging (advanced)

Dual path Köhler illumination including dichroic mirrors (DM)



# Quantitative magnetic domain imaging

CAU

 Multicomponent & multiple effect imaging



(a), (b) Concurrently obtained Kerr images of a single crystal iron film with orthogonal sensitivity directions, and (c) obtained quantitative domain image. Concurrently obtained (d) diagonally aligned Kerr and (e) Voigt image. (f) Vector representation of magnetic domain pattern.

J. McCord, J. Phys. D: Appl. Phys. 48, 333001 (2015)

### Imaging with magneto-optical detector films

CAU





Scheme of imaging magnetic domains of a ferromagnetic sample (FM) with a magneto-optical indicator film (MOIF).

(a) MOIF image of a region of an exchange spring sample with an opening. (b) Inversion of magnetization of a GdCoCu single crystal below and above the compensation temperature. (c) Domain structure from a Ni<sub>2</sub>MnGa crystal across a twin boundary (TB).

- Flat and smooth surface required
- Spatial resolution approx. 200 nm
- Magnetization can be observed directly
- Quantitative measurements possible
- Almost no influence on magnetization (but light induced heat)
- Straightforward sample manipulation (fields, temperature, stress)
- Surface sensitive (approx. 30 nm)

# X-ray microscopy techniques

CAU

XPEEM STXM<sup>(a)</sup>

Contrast mechanism	М	М
Evaluation of the magnetization, M(r)	Q	Q
Spatial resolution (nm) $\frac{\text{Typical}}{\text{Limit}}$	$\frac{300}{50}$	$\frac{30}{15}$
Depth of information (nm)	<5 nm	Thickness integrated <100 nm
Time for image acquisition	$\sim 1 sec$	$\sim 1 sec$
Limits on applied magnetic fields	Not advised	None
Imaging conditions	UHV	None
Max thickness		60–100 nm
Sample smoothness	NR	NR
Sample clean surface	Preferred	NR

### Synchrotron based

 Undulators and bending magnets cause electrons emit light



#### Big illuminator!



### Magnetic x-ray amplitude contrast



Transition of electrons following dipole selection rules from 2p-3d levels in L-edge x-ray absorption





### X-ray Magnetic Circular Dichroism (XMCD)

#### Material from P. Fischer, Lawrence Berkeley Natl. Lab, USA

IEEE Summerschool 2017 McCord

# Magnetic soft x-ray spectro microscopies



#### **Real-space!**

### **TXM and X-PEEM**





# XMCD spectro microscopy



#### (Co 0.3nm/Pt 0.5nm)x30





# X-ray imaging of vortex states

CAU



# Magnetic soft x-ray Tomography - 3D imaging



R. Streubel, F. Kronast, P. Fischer, D. Parkinson, O.G. Schmidt, D. Makarov, Nature Communication 6 7612 (2015)

#### Remanent state after applying magnetic field (200 kA/m) along 180°

and more windings

b





3D view

CAU





- Current pulses of  $\pm 3.9 \cdot 10^{11}$  Am<sup>-2</sup>.
- Forward and backward motion of skyrmion bubbles (static images)

[W(5 nm)/CoFeB(0.8 nm)/MgO(2 nm)]<sub>10</sub>/Ta(5 nm)

Material from M. Kläui, Johannes Gutenberg-University Mainz, Germany

# **XMCD-PEEM - magnetic and topographic information**



#### Positive helicity



#### Negative helicity



#### Normalized difference signal



 $l(\sigma^{+})$ 

*l(*σ⁻)

W. Kuch, K. Fukumoto,J. Wang, MPI-MSP,C. Quitmann, F. Nolting,T. Ramsvik, PSI-SLS,unpublished.

20 μm

# Co/Ni/Cu(001)

Ni L3 edge

 $\frac{I(\sigma^{+})-I(\sigma^{-})}{I(\sigma^{+})+I(\sigma^{-})}$ 

Material from W. Kuch, FU Berlin, Germany

### Element selectivity - local magnetic coupling

CAU



#### Material from W. Kuch, FU Berlin, Germany

# Summary on soft x-ray microscopy

#### Element selectivity (multilayers)

- Sample can be in air or vacuum
- Spatial resolution below 15 nm
- Sensitivity to out-of-plane magnetization (transmission, but tilting possible)
- Imaging with applied magnetic fields
- X-ray damage
- Sample must allow transmission of x-rays

# Fourier transform holography (FTH) with soft x-rays



- **Coherent X-rays**
- Germany **Object and reference locked (high stability to detect small signals)**

### **Image formation**





Reference aperture makes the image easily interpretable.

One directly obtains an image of the object!



# **Functionality of magnetic nanomaterials**

CAU

[111]

misaligned grain

[111]

[100]



Prototype hard drive data storage media (Hitachi GST) Magnetic contrast via XMCD (Co L<sub>3</sub>)

#### **Magnetization switching in external B-field**





50 nm



B. Pfau et al, APL 99, 062502 (2011) & APL 105, 132407 (2014)

#### Material from S. Eisebitt, Max-Born-Institut, Germany

IEEE Summerschool 2017 McCord

- Current spatial resolution 20 nm (best) 50 nm (every day)
- Atomic, chemical, magnetic sensitivity via resonant scattering
- Extreme stability to track small effects
- Requires coherent x-ray illumination

# Imaging magnetization dynamics

#### Mostly stroboscopic imaging



#### Magneto-optical methods capable of dynamic imaging

- X-ray based methods
- MOKE microscopy

# Stroboscopic imaging (differential mode)

CAU



#### **Accumulation of many repeatable events!**

# Time-resolved magnetooptical microscopy



- Stroboscopic pump-probe
- 10<sup>7</sup> pump-probe events/image
- Only fully repeatable dynamics
## Time-resolved quantitative MO imaging @ 2 GHz

#### R. Holländer et al., JMMM 432, 283–290 (2017)



#### **Dynamic response of Landau structure**

CAU

#### Excitations of spin waves in FeFeSiB @ 100 MHz





# Domain wall induced generation of spin waves from local edge resonances

B. Mozooni, J. McCord, APL 107, 042402 (2015)

IEEE Summerschool 2017 McCord

## Excitations of spin waves in Ni<sub>81</sub>Fe<sub>20</sub> @ 4 GHz





Antennaless generation of spin waves from local edge resonances

M. Lohmann et al., accepted for JMMM (2017)

## Time resolved soft x-ray microscopy



- Stroboscopic pump-probe
- 10<sup>8</sup> pump-probe events/image
- Only fully repeatable dynamics

Material from P. Fischer, Lawrence Berkeley Natl. Lab, USA

### Spin-orbit torque driven skyrmion dynamics

CAU



Material from P. Fischer, Lawrence Berkeley Natl. Lab, USA

## Time-resolved x-ray holography

#### **Skyrmions as information carriers**

#### **Skyrmion fine structure**



FOV Ø 1 μm resolution < 20 nm



**Skyrmion intrinsic GHz dynamics** F. Büttner et al., Nature Physics **11**, 225 (2015).

#### **Skyrmion motion**



F. Büttner et al. (submitted, arXiv 1705.01927)

15 repeats of Pt(2.7 nm)/Co<sub>60</sub>Fe<sub>20</sub>B<sub>20</sub>(0.8 nm)/MgO(1.5 nm)

Material from S. Eisebitt, Max-Born-Institut, Germany

## Brillouin light scattering (BLS) microscopy

CAU



## **Brillouin light scattering microscopy**

CAU



#### Imaging of spin-wave phenomena (up to THz!).

Material from H. Schultheiss, HZDR, Germany

#### ... papers, papers, papers ...

- McCord, Jeffrey. "Progress in magnetic domain observation by advanced magneto-optical microscopy." *Journal of Physics D: Applied Physics* 48.33 (2015): 333001.
- Fischer, Peter, and Hendrik Ohldag. "X-rays and magnetism." *Reports on Progress in Physics* 78.9 (2015): 094501.
- Krishnan, Kannan M. Fundamentals and applications of magnetic materials. Oxford University Press, 2016.
- A. Hubert, R. Schäfer. Magnetic domains: the analysis of magnetic microstructures. Springer Science & Business Media, 2008.