

# Introduction to magnetometry techniques

Thomas Hauet

ESM2020, 01/10/2020

### Most of the materials are magnetic







Oscillator in Rolex watch

*	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	ть	Dy	Но	Er	Tm	Yb	Lu
**	Ac	Th	Ра	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr



Magnets

#### Parameters, symbols and units

$$\vec{B} = \mu_0 \left( \vec{H} + \vec{M} \right) = \mu_0 \left( 1 + \chi \right) \vec{H} \quad \text{avec} \quad \chi = \partial M / \partial H$$

Parameters : B (induction) in Tesla (=  $10^4$  Gauss=  $10^4$  Oe) H (field) in A/m (H = 1 A/m => B =  $1.256 \ 10^{-6} \ T$ ) M (magnetization) in A/m ( $=10^{-3}$  emu/cm<sup>3</sup>)  $\mu_0$  (free space permeability) =  $4\pi \cdot 10^{-7}$  H/m  $\chi$  (magnetic susceptibility)



Other material parameters : K (magnetic anisotropy energy) in J/m<sup>3</sup> J (magnetic exchange) in J/m  $\alpha$  (damping) with no unit  $m_{S}$ ,  $m_{orb}$  (spin, orbital atomic moment) in  $\mu$ B/atom



Your advisor needs to switch from CGS to SI : 1 emu/cm<sup>3</sup> = 1 kA/m 1 erg/cm<sup>3</sup> = 0.1 J/m<sup>3</sup>

# Magnetometry

#### Measure of physical parameters

- Magnetic moment
- Magnetic susceptibility
- Electrical resistance
- Specific heat
- Ferromagnetic resonance...

- .... as a function of external parameters
  - DC or AC magnetic field
  - Temperature
  - Pressure
  - Electric field
  - Light...



#### Magnetometry : before to start

What type of sample ? Nanoparticles, molecules, liquids, bulk, thin film, devices, ...

Which properties am I interested in ? Magnetic moment, susceptibility, spin or orbital moment, non-uniform configuration, damping, anisotropy, ...

Which resolution do I need ?

Which excitation do I want to study? Magnetic field, temperature, electric field, ligth, pressure, ...

Under which static condition? Temperature, vacuum, oxidant atmosphere, pressure, magnetic field, ...

Which time scale ? (excitation & measurement) Femtosecond, second, million years, ...

Which space scale ? (sample size & measurement) Nanometer, meter, hundred meter, milliliters...

# Space and time scales



# Magnetometry : Outline

Vibrating sample magnetometry	Magneto-optical Kerr effect	Heat capacity measurement			
Superconducting quantum interference device	ce Torque magnetometry	X-ray circular dichroism			
ballistic electron magnetic microscopy	susceptometry	etic Resonance measurement			
magnetic force microscopy	photoemiss	ion spectroscopy			
magnetoresistivity magnet	ic resonance force microscopy	N-V magnetometry			
magnetic transmission X-ray microscopy	scanning electron micros	scopy with polarization analysis			
scanning near field optical microscopy	spin polarized low energy elec	tron microscopy			
spin polarized scanning tunneling microsco	scanning transm	nission X-ray microscopy			
X-ray photoemission electro	n microscopy Ne	Neutron diffraction			

# Outline : 1h30 talk



# Many available data in ESM library

<u>Fichier Édition Affichage Histori</u>	ique <u>M</u> arque-pages <u>O</u> utils Aid <u>e</u>			- @ X
G AC susceptometry - Recherche	× 🔯 A simple torque magnetometer × 🍈 European School on Magnetism × 🚽	F		
← → ♂ ☆	👽 🔏 magnetism.eu/esm/repository-topics.html	♡ ☆	Rechercher	± II\ ⊡ © ≡
<ul> <li>Les plus visités Débuter av</li> <li>[2003] Sen-organization on se</li> <li>[2003] Chemical synthesis of</li> </ul>	ec Firefox ( Authentification auto ( Web of Science [v.5.30 unaces: an overview. <u>O. FRUCHART</u> [ ADSTRACT ] Sinces ] magnetic metal nanoparticles : <u>B. CHAUDRET</u> [ Abstract ]			^
Magnetic microscop Dresden, Germany [ Abstra [2019] Electron holography at [2019] Holography, tomograp [2019] Scanning probe micro [2017] Characterization techn [2015] Magnetic imaging tech [2013] Magnetic imaging tech [2007] Magneto-optical micro [2007] STM and spectroscop	ct ] nd tomography: <u>AxeL LUBK</u> , Dresden, Germany [ Abstract ] hy (Electron, X-ray): X-rays: <u>Jan Lüning</u> , Berlin, Germany [ Abstract ] scopy for magnetism: <u>OLIVIER FRUCHART</u> , Grenoble, France [ Abstract ] liques for nano-sized systems: <u>OLIVIER FRUCHART</u> , Grenoble, France [ Abstract ] niques: <u>LAURA HEYDERMAN</u> , Zürich, Switzerland [ Abstract   Intro   Kerr   Scan niques: <u>LAURA HEYDERMAN</u> , Zürich, Switzerland [ Abstract   Slides ] scopy and its applications: <u>RUDOLF Schäfer</u> , <i>IFW</i> , Dresden, Germany [ Abstract y of nanosized ferromagnetic structures: <u>GULLEMIN RODARY</u> , MPI-Halle, Germany	http://magnet Slides ] ning probe   PEEM ]   Slides pt.1-2 ] [ Slides ]	ism.eu/esm/repository	v-topics.html
<ul> <li>[2005] Magneto-optical micro</li> <li>[2005] Scanning probe techni</li> <li>[2005] STM and spin-polarize</li> <li>[2005] Magnetic imaging by L</li> <li>[2005] Electron microscopies</li> <li>[2003] Analysis of nanostruct</li> <li>[2003] High spatial-resolution</li> <li>[2003] Magnetic force micros</li> <li>Magnetometry</li> <li>[2019] General and torgue micros</li> </ul>	scopy (incl. time resolved): <u>J. McCorp</u> [ Abstract   Slides (12.6MB) ] iques (MFM, SNOM, etc.): <u>L. ABELMANN</u> [ Abstract   Slides (3.5MB)   Supplement ed-STM: <u>W. WULFHEKEL</u> [ Abstract   Slides pt1 (5.6MB)   Slides pt2 (2.3MB) ] .EEM, X-PEEM, X-ray microscopy, and X-ray holography: <u>W. Kuch</u> [ Abstract   Slides (Lorentz, holography, SEMPA, etc.): <u>B WAROT-FONROSE</u> [ Abstract   Slides (11.5 ures by electron Microscopy (EM) and Scanning Probe Microscopy (SPM) (structure analysis of magnetic properties (special SPM and EM techniques as SHPFM and L copy (MFM) and spin polarized scanning tunneling microscopy (SPSTM): <u>A. THIAVI</u> aganetometry: <b>VITTORIO BASSO</b> , <i>Torino</i> , <i>Italy</i> [ Abstract ]	ntary information (AVI) (2.6MB) ] ides (5.4MB) ] MB) ] al and compositional analysis): <u>M. HIETSCH</u> .orentz-Microscopy) <u>M. HIETSCHOLD</u> [ Abst LLE [ Abstract   Slides ]	Neutrons [2019] Neutron scattering: <u>VIRGINIE SIMONET</u> , Gre [2017] Magnetic diffraction with neutrons, non-reso [2017] Neutron diffraction for magnetic structure de [2011] Spin waves: theory and neutron scattering: [2011] Neutrons for magnetism: <u>STEPHEN BLUNDE</u> [2005] Neutrons and magnetism: <u>A. WILDES</u> [ Abs [2005] Magnetic scattering: X-ray and neutrons: <u>CI</u>	noble, France [ Abstract ] mant and resonant X-ray scattering etermination: <u>VIRGINIE SIMONET</u> , Gr <u>SYLVAIN PETIT</u> , CEA-Saclay, France <u>LL</u> , Oxford, UK: [ Abstract   Slides tract   Slides~pt1 (23.0MB)   Slid <u>1. VETTIER</u> [ Abstract   Slides (1.6
<ul> <li>[2019] Indirect techniques: ca</li> <li>[2018] Magnetometry: <u>PAoLo</u></li> <li>[2018] Tutorial: experimental</li> <li>[2005] Advanced Magnetometry: n</li> <li>[2003] Nanomagnetometry: n</li> </ul>	alorimetry, dilatometry, transport: <u>VITTORIO BASSO</u> , <i>Torino, Italy</i> [ Abstract ] <u>VAVASSORI</u> , San Sebastian, Spain [ Abstract   Slides ] methods for magneto-optical Kerr effects measurements: <u>PAOLO VAVASSORI</u> , San Setry: <u>D. SANDER</u> [ Abstract   Slides (2.3MB)] nicroSQUID, Hall microprobe: <u>E. BONET</u> [ Abstract   Slides ]	Sebastian, Spain [ <mark>Slides</mark> ]	Precessional techniques [2019] Magnetic resonance: <u>Laurent Ranno</u> , Gre [2011] Muons for time-dependent studies in magne [2011] Local and resonance techniques: FMR, EPF [2005] Using muons for magnetization studies: <u>D. A</u>	enoble, France [ <mark>Abstract</mark> ] etism: <u>Andreica, D.</u> , <i>UBB - Cluj-Naj</i> R, NMR, Mössbauer: <u>Philippe Men</u> Andreica
Optics and X-ray to [2019] Magneto-optics: <u>Rubo</u> [2019] Holography, tomograp [2019] X-ray techniques: sync [2019] Time-resolved pump-p [2018] X-ray magnetic dichroi [2018] Synchrotron radiation:	ols (structural and magnetic)         DLF SCHÄFER, Dresden, Germany [ Abstract ]         hy (Electron, X-ray): X-rays: Jan Lüning, Berlin, Germany [ Abstract ]         chrotron, FEL, HHG: Jan Lüning, Berlin, Germany [ Abstract ]         orobe techniques: THEO RASING, Nijmegen, The Netherlands [ Abstract ]         ism: EBERHARD GOERING, Stuttgart, Germany [ Abstract   Slides (Pt.1, Pt.2) ]         MARCIN SIKORA, Kraków, Poland [ Abstract   Slides ]		Electron spectroscopies • [2005] XPS, including spin-polarized XPS: <u>M. Neur</u> • [2005] Surface magnons by EELS: <u>J. KIRSCHNER</u>	<u>мапя [ Slides</u> (3.5MB) ]. [ Abstract   Slides (1.9MB) ]
📕 🔎 🗊 🤦	📄 🔟 🚳 💴 📴		ف 🕸 🗞 🕅	≥ 📕 🐼 🖅 🖵 ⊄× 13:49 💭

## Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - VSM, SQUID, Torque, MOKE, XMCD
- III. AC macroscopic magnetometry techniques
  - AC susceptometry

#### IV. Imaging techniques



Scanning probe microscopies, Photon-out

#### Help from European/French magnetometry networks



Special thanks to F. Mazaleyrat, Ph. Vanderbemden, L. Ranno, C.-S. Chang, B. Hillebrands, O. Fruchart

www.magnetometry.eu

www.magnetometrie.cnrs.fr

## Outline

#### I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - > VSM, SQUID, Torque, MOKE, XMCD, Photo-emission spectroscopy

#### III. AC macroscopic magnetometry techniques



AC susceptometry, FMR

#### IV. Imaging techniques



Scanning probe microscopies, Photon-out, Electron-out

# Magnetic field amplitude scale

Human brain	10 <sup>-15</sup> T		
Earth's magnetic field	5x 10 <sup>-5</sup> T		
Refrigerator magnet		0.05 T	
Computer	usual magnetometers	0.1 T	
MRI scanner		1 - 7 T	
Laboratoire de Champs Mag	37 T		
Pulsed field magnet		100 T	
White dwarf star		10 000 T	
Pulsar		100 000 000 T	

### How to generate a magnetic field





1820 - Hans Christian Oersted



#### **Electromagnets :**

Requieres electric energy

1 coil with R=25mm and 40 kA generates 1 Tesla

- Non-uniform or uniform fields
- Field can be tuned easily with current
- Can produce **DC, AC or pulsed fields** (μs, ns)



#### Permanent magnets :

- Assembly of dipoles  $\vec{B} = \frac{\mu_0}{4\pi} \left[ \frac{3(\vec{\mathcal{M}} \cdot \vec{r'}) \cdot \vec{r'}}{r'^5} \frac{\vec{\mathcal{M}}}{r'^3} \right]$
- DC Magnetic flux with no energy
- Non-uniform and uniform fields
- Big advantage for small sample space

### Electromagnets : homogeneous field up to 3 Tesla





Field along z-axis for N turns, current I and length L

$$B_{z}(z) = \frac{NI}{L} \left(\cos \theta_{1} + \cos \theta_{2}\right)$$

**Coilset in Helmoltz geometry** 



Coil radius R, Coil distance b, Helmholtz condition:  $b = R \rightarrow$  quadratic term vanishes

$$d = \sqrt{R^2 + \frac{1}{4}b^2}$$

### Fields above 3 Tesla



Limitations :

**1)** Energy dissipation  $P = \int \rho(x) \mathbf{J}^2(x) d^3x \propto \mathbf{B}^2$  **2)** Force de Lorentz  $\mathbf{F} = \int \mathbf{J}(x) \times \mathbf{B}(\mathbf{x}) d^3x \propto \mathbf{B}^2$ 

#### Superconducting electromagnets



## Outline

I. Few preliminary details about the sample environment

- Magnetic field, **Temperature**
- II. Static macroscopic magnetometry techniques
  - > VSM, SQUID, Torque, MOKE, XMCD
- III. AC macroscopic magnetometry techniques



#### IV. Imaging techniques



Scanning probe microscopies, Photon-out

#### Quick reminder on heat transfers

**Radiation :** In average the coldest region gets more photons from the hotest region than it loses itself

Stefan-Boltzmann's Law defines the relationship between the emission of black bodies with its temperature

Surface density of Radiative power=  $\sigma \in T^4$ 

 $\sigma$  : Stefan constant,  $\epsilon$  : émissivity, T : temperature

Ways to limit radidative heat transfer : - reflective materials (metals with low roughness)

- thermal Screening by absorbant materials (N2)

#### Quick reminder on heat transfers

**Radiation :** In average, the coldest region gets more photons from the hotest region than it loses itself

Ways to limit radidative heat transfer : - reflective materials (metals with low roughness)

- thermal Screening by absorbant materials (N2)

**Conduction :** Heat transfer, without moving material, between two media of different temperature (vibrations)

Fourier law's : heat flux density  $= -\lambda \overrightarrow{grad} T$ Thermal conductivity

Ways to limit heat transfer through conduction : - Materials with low thermal conductivity (thermal insulation)

- Removing materials between two region (vacuum)

### Quick reminder on heat transfers

#### **Radiation :** In average, the coldest region gets more photons from the hotest region than it loses

Ways to limit radidative heat transfer : - reflective materials (metals with low roughness)

- thermal Screening by absorbant materials (N2)

**Conduction :** Heat transfer, without moving material, between two media of different temperature (vibrations)

Ways to limit heat transfer through conduction : - materials with low thermal conductivity (thermal insulation)

- absence of materials between two regions (vaccuum)

#### **Convection :** Heat transfer by moving matter



Ways to limit convective heat transfer : - to limit matter motion

- absence of matter between the two regions (vacuum)

## General design of a cryostat

1) Low temperature source

- Cold flux
- 2) High temperature source
  - Hot flux
- 3) Control of the environment
  - Insulated system
- 4) Measure of tempearture
  - Well chosen thermometer







Conduction - convection - Radiation



 




# Other typical temperature control set ups



## Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques

**VSM, SQUID**, Torque, MOKE, XMCD, Photo-emission spectroscopy

#### III. AC macroscopic magnetometry techniques



AC susceptometry, FMR

#### IV. Imaging techniques



Scanning probe microscopies, Photon-out, Electron-out

#### Macroscopic moment measurement



# Induction to detect guitar string motion (1950's)



Figure 1 - The Frying Pan, guitare bawaïenne solidbody, Rickenbaker 1931. [Pierrick Lotton, musique & technique, 2009]



Gibson Les Paul 1952



Figure 2 — Pickup développé par G. Beauchamp pour Rickenbaker.



# First vibrating sample magnetometer (1959)

Rev. of Scientific Instr. 1959 Brevet déposé en 1960



Simon FONER 1925 † 2007

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

#### The vibrating sample magnetometer: Experiences of a volunteer

#### S. Foner

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

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. Initial



### Two VSM geometries



## **Axial VSM equations**

 $B_{z} = \frac{\mu_{0}}{4\pi} \left[ \frac{3\mathcal{M}h^{2}}{(h^{2}+r^{2})^{5/2}} - \frac{\mathcal{M}}{(h^{2}+r^{2})^{3/2}} \right]$ 

$$\phi = \int B_z dS = \int_0^R \frac{\mu_0}{4\pi} \left[ \frac{3\mathcal{M}h^2}{(h^2 + r^2)^{5/2}} - \frac{\mathcal{M}}{(h^2 + r^2)^{3/2}} \right] 2\pi r dr$$
$$= \frac{\mu_0 \mathcal{M}}{2} \left[ -\frac{3h^2}{(h^2 + R^2)^{3/2}} + \frac{1}{(h^2 + R^2)^{1/2}} \right]$$

Taylor : 
$$\phi = \frac{\mu_0 \mathcal{M}}{2R} \left[ -3 + \frac{9h^4}{2R^4} + 1 - \frac{h^2}{2R^2} + \frac{3h^4}{8R^4} + o(\frac{h^6}{R^6}) \right]$$
  
H<

If the sample vibrates along z:  $h(t) = h + a \sin(\omega t)$ ,  $a \ll h$ 

$$\frac{\mathrm{d}\phi}{\mathrm{d}t} = \frac{-\mu_0 \mathcal{M}}{4R^3} \frac{\mathrm{d}}{\mathrm{d}t} \left(h^2 + 2ah\sin(\omega t) + a^2\sin^2(\omega t)\right)$$

Induced fem for n spires : 
$$\mathcal{E} = -n \frac{d\phi}{dt} = \frac{n\mu_0 \mathcal{M}ha\omega}{2R^3}$$

If the sample is considered as a single magnetic dipole :



$$\vec{B} = \frac{\mu_0}{4\pi} \left[ \frac{3(\vec{\mathcal{M}} \cdot \vec{r'}) \cdot \vec{r'}}{r'^5} - \frac{\vec{\mathcal{M}}}{r'^3} \right]$$

### **Radial VSM equations**







Both VSMs carry good sensitivity (down to 10<sup>-9</sup> A.m<sup>2</sup>) because of the synchronous detection (Lock-In)

### Two VSM geometries : usual tools



#### Radial VSM

Up to 2.7 Tesla 300K up to 1200K Easy sample rotation

MicroSense (USA),




#### Example 1 : anisotropies in crystals

Fe bcc atomic lattice anisotropy leads to prefered directions for the atomic magnetic moments. This translate into an anisotropy of potential energy :

$$E_{mc} = K_1 \left( \alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2 \right) + K_2 \left( \alpha_1^2 \alpha_2^2 \alpha_3^2 \right);$$
where  $\alpha_i = \cos \theta_i : \theta_i$  -angle between axis and M

From the litterature :





37

# Example 2 : study of structural transitions



A. Kyianytsia et al., APL 117, 122411 (2020)

38

# SQUID magnetometer



# Magnetic flux through a superconducting coil



Perfect Superconducting coil

Electrons are coupled in Cooper pairs  
Superconducting current: 
$$\vec{j} = \frac{ne}{m} (\hbar \vec{\nabla} \varphi - \frac{2e}{c} \vec{A})$$
  
If there is no current:  $\oint \vec{j} \vec{d} \vec{l} = 0 = \hbar . 2\pi . N + 2e \iint \vec{B} \vec{S}$   
 $\iint \vec{B} \vec{S} = N . \frac{h}{2e} = N \Phi_0$ 

1 quantum  $\Phi_0 = \frac{h}{2e} = 2.10^{-15} \text{ Tm}^2$  (=Wb Weber)



lf

Magnetic flux in the coil is quantified
when superconducting, magnetic field cannot vary inside the coil

# Magnetic flux through a superconducting coil



Perfect Superconducting coil

Electrons are coupled in Cooper pairs

Superconducting current:  $\vec{j} = \frac{ne}{m} (\hbar \vec{\nabla} \varphi - \frac{2e}{c} \vec{A})$ 

If there is no current :  $\int \vec{j} \vec{dl} = 0 = \hbar . 2\pi . N + 2e \iint \vec{BS}$ 

$$\iint \vec{B}\vec{S} = N.\frac{h}{2e} = N\Phi_0$$

1 quantum  $\Phi_0 = \frac{h}{2e} = 2.10^{-15} \text{ Tm}^2$  (=Wb Weber)

#### But magnetic field can penetrate through defect :



# DC SQUID : 2 interfering Josephson junction





#### DC CHARACTERISTICS AND NOISE PERFORMANCE



Measured Voltage-Current (V-I) and Voltage-Flux (V- $\Phi$ ) characteristics of a typical M800 dc SQUID magnetometer operated 4.2 K.

#### **Example : detecting Ni nanoparticles**



SQUID-VSM : Small signal measurement



Method for detection of Ni nanoparticles after they are used to grow carbon nanotubes

B.Vigolo et al. IJL Nancy European Patent PCT/FR2011/052519 (2011)

### VSM and SQUIDs limiting factors



Final Lock-in voltage = Gain x Moment x Sample geometry factor x Coil geometry factor x Amplitude x Frequency

- The parameters in green are accounted for <u>through</u> <u>calibration with a standard</u> of known moment for a particular VSM coil set.
- This leaves sample moment coupled together with its geometry factor. This is very important to consider when interpreting the measured moment.

# Experimental limiting factors : sample geometry

#### Final Lock-in voltage =

Gain x Moment x Sample geometry factor x Coil geometry factor x Amplitude x Frequency



Example of a **thin film sample geometry** effect for SQUID VSM (4 mm x 4 mm x 200 nm Ni film on silicon)

# Experimental limiting factors : sample geometry

#### Final Lock-in voltage =

Gain x Moment x Sample geometry factor x Coil geometry factor x Amplitude x Frequency



**QD Software : Corrected Moment = Measured Moment / Correction Factor** 

BMPMS 3 Sample Geometry Simul	ator	ر <sup>0.0060</sup> ر	Ni film	n Ms SV	'SM	■ in	nlano		]
Reference Geometry (Pd Cylinder) Height (mm)	Sample Geometry Thin Film (Field ⊥ ab)	- 0.0058 - -	•	•		• 0 • ir • o	ut-of-pl n-plane ut-of-pl	ane correct ane co	ted rrected
Diameter (mm) 2.80	a (mm) 4	0.0056 -		•					
DC Scan Length (mm) 30	b (mm) 4	(nm) 0.0054 -			•	•	•		
	Radial Offset (mm) 0	0.0052 -						-	•
VSM Amplitude (mm) 8	Estimated Correction Factors VSM Measurement 1.112	0.0050 -							
DC Scan Length (mm) 30	DC Scan 1.077	- 0.0048 -	Ĭ			<b>₽</b> ▼	<b>₽</b> ▼	•	•
	Calculate	+ 0	1	2 3	4	5	6	7	8
		1		Ar	mplitude	e (mm)			

# Experimental limiting factors : background



Final Lock-in voltage = Gain x Moment x Sample geometry factor x Coil geometry factor x Amplitude x Frequency

> contains any magnetic signal from the environment (background)

Here, same piece of Kapton tape and sample holder measured without the sample to obtain background signal.

#### But it can be worst... and if you don't check, you don't find out







In the case of a nonuniform background signal, the software is still trying to fit the curve whatever the curve is.

# Experimental limiting factors : trapped field

The superconductive magnet can have trapped flux especially when the magnet has been loaded at fields larger ~0,5 T





The System does not measure the actual field. The reported field is calculated based on the current placed in the magnet.

#### Experimental limiting factors : temperature



When measuring while temperature is sweeping, there will be thermal lag between the measured and the actual sample temperature.



Don't sweep faster than 3K/min even if your tool can go 20K/min

### Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - > VSM, SQUID, **Torque, MOKE, XMCD**
- III. AC macroscopic magnetometry techniques



#### IV. Imaging techniques



Scanning probe microscopies, Photon-out

#### Torque magnetometry





Sample position (here out-of-plane uniaxial anisotropy)

#### Torque measured via piezoelectricity

*Torque*:  $\tau = \vec{M} \times \vec{B} = -\frac{dE}{d\theta}$ 

Uniaxial anisotropy  $E_a = K_1 \sin^2\theta$ 



Torque  $-dE_{tot}/d\theta = -dE_a/d\theta = -K_1 \sin 2\theta$ 



https://www.youtube.com/user/QuantumDesignUSA

#### Torque magnetometry

 $-\kappa_1$ 



Torque: 
$$\tau = \vec{M} \times \vec{B} = -\frac{dE}{d\theta}$$

Uniaxial anisotropy  $E_a = K_1 \sin^2\theta$  $K_1$ E 0 45° 90\* 135° 180° Torque  $-dE_{tot}/d\theta = -dE_a/d\theta = -K_1 \sin 2\theta$ +K., Easy 20.00 1 0 Hard ania Easty

1816

Bergholz, Elmers, Gradmann, PRL 63,566 (1989) / Appl. Phys. A 51, 255 (1990)

#### Torque magnetometry : non-uniform configuration



P. Vallobra, JAP 120, 013903 (2016)

### Magneto-optical Kerr effect magnetometry



# Magneto-optical Kerr effect magnetometry



#### B. Hillebrands

# Magneto-optical Kerr effect magnetometry



## MOKE example : sensitive to surfaces



Example : study of magnetic anisotropy in multilayer thin film Ni(0.6nm)/Co(0.6nm)/Ni(x)



MOKE is only sensitive to about 20-50 nm thickness at the materials surface

# MOKE magnetometry : imaging and dynamics

(a) (b) digital camera Köhler illumination IP primary image retarder plate tube tube lens polarizer lens analyzer Bertrand lens AP beamsplitter HP LED IP aperture back focal plane (AP) plane (AP) ΥY objective lens (c)image plane (IP) specimen illumination observation **Picosecond SOT switching** 6 ps observed by MOKE current (a.u.) 0.5 J. Gorchon arXiv:1912.01377 0 10 20 delay (ps) Hz 0.0 10µm -45µm SiO LT-GaAs -Hx photoswitch magnet

MOKE can be focused and so it can be used to perform magnetic microscopy

Laser can be pulsed so that MOKE can be used to probe magnetic changes at different timescales down to femtosecond

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

# MOKE magnetometry : microscopic model



Hulme, Proc. R. Soc. London **A135**, 237 (1932) Bruno, PRB **53**, 9214 (1995) Transition energy  $\approx$  eV

#### Electron (I, m, s) transition rules :

- Energy: E<sub>f</sub> E<sub>i</sub> = ħω
   (absorbed photon energy = difference between final and initial state)
- Electron spin : ∆s=0

(spin of electron is preserved for electric dipole transitions)

• Orbitum momentum :  $\Delta I=\pm 1$ 

(photon has angular momentum 1ħ). Therefore only  $s \leftrightarrow p$ ,  $p \leftrightarrow d$  etc. are allowed

Orbital momentum along z-axis : ∆m=±1

(determines if photon is right/left polarized)

#### P. Bruno, PRB 53, 9214 (1996)

# X-ray magnetic circular dichroism (XMCD)



- XMCD disentangles spin and orbital magnetic moment
- XMCD disentangle the moment from specific atoms in an alloy or a multilayer
- XMCD requieres to work in synchrotron to get enough X-ray flux (700 - 900 eV for Fe,Co,Ni)



# X-ray magnetic circular dichroism (XMCD)





# Example of XMCD data



S. Andrieu, PRMater 2, 064410 (2018)

### Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - > VSM, SQUID, Torque, MOKE, XMCD
- III. AC macroscopic magnetometry techniques
  - AC susceptometry
- IV. Imaging techniques



Scanning probe microscopies, Photon-out

# AC susceptometry : measure of susceptibility X



#### Coil sets in a PPMS



Frequency range 0.1Hz-1kHz AC amplitude 0.01 à 1 mT



Physical Property Measurement System AC Measurement System Option User's Manual

#### Measurement method in a PPMS



By default, the sample undergoes a five-point measurement process that utilizes the calibration coil to increase measurement accuracy. The first reading is made with the sample positioned in the center of the bottom detection coil. Then the sample is positioned in the center of the top detection coil, and then in the center of the bottom coil again. During all three readings, the signals from the detection coil array are amplified, low-pass filtered, and digitized by an analog-to-digital converter (A/D). These signals are stored as waveform blocks in the data buffer. All 128 buffer points are used to record each response waveform. The points are fitted and compared to the driving signal to determine the real and imaginary components of the response when the sample is in the center of each detection coil. (Imaginary components are in phase with the driving signal and real components are 90° out of phase with the driving signal.) Subtracting one reading from the other gives a sample vector in the complex plane.

When the bottom-top-bottom coil readings are complete, the sample is placed at the center of the detection coil array so that it is between the two detection coils. Two more readings are taken with the calibration coil switched into the detection circuit with opposing polarities. The real and imaginary components of each response waveform are obtained by again fitting the data and comparing it to the driving signal. The two calibration readings are subtracted to yield a calibration vector in the complex plane. Subtracting the two calibration readings subtracts out the sample signal, leaving only environmental and instrumental factors that affect the reading.

Physical Property Measurement System AC Measurement System Option User's Manual

Measurement

Calibration

(2)

(3)

(4)

(5)

#### AC susceptometry equations



Ph. Vanderbemden

#### AC susceptometry equations

When M(t) is assumed to follow a pure sinewave (i.e. only one « fundamental » signal at one frequency ω) the complex AC susceptibility reads:



#### AC susceptometry equations

Most of the time M does not linearly vary with H :



BUT M(t) is still a periodic signal of the same period as the AC field. Therefore, thanks to the Fourier theorem, M(t) reads

 $\mathsf{M}(\mathsf{t}) = \mathsf{H}_1 \sum_{n=1}^{\infty} \left( \chi'_n \cos(n\omega t) + \chi''_n \sin(n\omega t) \right)$ 

<u>If M(t) is a pure sinewave</u>:  $\chi'_1$  and  $\chi''_1$  only  $\rightarrow \chi'(=\chi'_1)$  and  $\chi''(=\chi'_1)$ <u>If M(t) is distorted</u>:  $\rightarrow$  the harmonic susceptibilities might be  $\neq 0$ 

Harmonics originate from the <u>non-linearity</u> of the M-H process

#### Examples of AC susceptometry data



# Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - VSM, SQUID, Torque, MOKE, XMCD, Photo-emission spectroscopy
- III. AC macroscopic magnetometry techniques



- AC susceptometry, FMR
- IV. Imaging techniques



Scanning probe microscopies, Photon-out, Electron-out



#### microscopy techniques : a rough map



NV = NV center of diamond probe
MFM = magnetic force microscopy
MRFM = magnetoc resonance force microscopy
MTXM = magnetic transmission X-ray microscopy
SEMPA = scanning electron microscopy with polarization analysis
SNOM = scanning near field optical microscopy

SP-LEEM = spin polarized low energy electron microscopy SP-STM = spin polarized scanning tunneling microscopy STXM = scanning transmission X-ray microscopy X-PEEM = X-ray photoemission electron microscopy SHPM = scanning all probe microscopy
#### Which technics fits your need ?



The 1st parameter to check may be the size of your object...

## Which technics fits your need ?

Spatial scale	Microscopy technic	aues					
		Technique	Probed	Spatial Resolution	Temporal Resolution	Info. Depth	Comments
— 1 mm	Magnetic domains Network of sen	SORS Lorentz Microscopy	stray field + sample induction	10 nm	1 ns	sample average	Thin samples, Quantitative info. with differential phase contrast microscopy.
——100 μm	Hall/GMR se	Electron Holography	stray field + sample induction	$5\mathrm{nm}$	$10\mathrm{ms}$	sample average	Quantitative info. through mathematical image reconstruction.
10 μm	microsco	ру ѕемра	magneti- zation	$20\mathrm{nm}$	700 ps*	$1\mathrm{nm}$	Quantitative info., Long acquisitions, UHV required.
— 1 µm	Micro/Nano Kerr microso structures	SP-STM	magneti- zation	atomic	120 ps*	surface	UHV required, Usually low temperature, Long acquisitions.
— 100 nm	Domain wall IXM SNOM	MFM	stray field	10-100 nm	low	$1000\mathrm{nm}$	Potentially invasive, Long acquisitions, Few sample requirements.
— 10 nm		TXM	magneti- zation	$25\mathrm{nm}$	$50\mathrm{ps}$	sample average	Synchrotron technique, Quick overview images.
		STXM	magneti- zation	$25\mathrm{nm}$	$50\mathrm{ps}$	sample average	Synchrotron technique, High repetition rates.
— 1 nm	Nanoparticle, molecules SP-STM	PEEM	magneti- zation	$25\mathrm{nm}$	$50\mathrm{ps}$	$5\mathrm{nm}$	Synchrotron technique, Discharges possible due to high potential.
───100 pm							

#### The 1st parameter to check may be the size of your object...

R.M. Reeve et al., arXiv:1806.07767v2

### Which technics fits your need ?

... But you also need to check other criteria...

#### Spatial scale



	Sp-STM	MFM	NV	ВЕММ	SEMPA	SPLEEM	TEM	XMCD -PEEM	XMCD- microscopy (Fresnel ZP
Resolution	<1nm	15nm	5-10 nm	1-5nm	10nm	10nm	1-2nm	25nm → 10nm	15nm
Sensitivity	High	Med	High	Med	Med	High	Low	High	High
In-field	YES	Limited	Limited	YES	local	No?	Limited	No?	YES
Versatile*	No	YES	Yes	No	Yes	UHV	Limited	Yes	Limited
Dynamics	No	No	No	No	No	No	New	Yes	Yes
Element- sensitive	Limited	No	No	No	No	Limited	Limited	Yes	Yes
Probes	$m_i$	$\mathbf{H}_{d}$	$\mathbf{H}_{d}$	$m_i$	m	m	$m_{x,y}$	$m_{\mathbf{k}}$	m <sub>k</sub>

#### What is measured?

- Magnetization, induction, stray field?
- Elemental resolution
- Direct or indirect?
- □ Quantitative or not?

#### **Environmental conditions**

- Temperature
- □ Field: magnetic field, electric
- 🔲 Electric current, light etc.
- 🗆 Strain
- Additional measuring techniques

#### Which specifications?

- Magnetization: 1D, 2D, 3D
- □ Depth resolution: surface or volume?
- Lateral resolution
- Sensitivity
- □ Time/Spectral resolution

#### Versatility

- Sample preparation needed
- Time per one measurement
- 💷 🛛 In situ / ex situ
- Large-scale or in-lab?
- Expensive or cheap?

#### Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - VSM, SQUID, Torque, MOKE, XMCD
- III. AC macroscopic magnetometry techniques



- AC susceptometry, FMR
- IV. Imaging techniques



Scanning probe microscopies, Photon-out



### Magnetic force microscopy



#### **AFM characterizes topography**

## Magnetic force microscopy (MFM)



- First pass: measure topography
- Second pass: measure magnetism NB: other measurement modes exist

Y. Zhu Ed., Modern techniques for characterizing magnetic materials, Springer (2005) **Example:** magnetic domain configuration of [Co/Pd] multilayer on patterned substrate







L. Piraux et al., APL 101, 013110 (2012)

## Magnetic force microscopy : criteria





Tip-sample interactions can impact the magnetic configuration of your sample



#### Scanning GMR/Hall probe microscopy



J. Moulin (phD thesis 2020)

#### Scanning GMR/Hall probe microscopy

Advantage : To get a direct access to magnetic field instead of its second derivative as in MFM



## Spin-polarized Scanning Transmission Microscope



S. Rohart (ESM2017)

## Spin-polarized Scanning Transmission Microscope



#### Outline

I. Few preliminary details about the sample environment

- Magnetic field, Temperature
- II. Static macroscopic magnetometry techniques
  - VSM, SQUID, Torque, MOKE, XMCD
- III. AC macroscopic magnetometry techniques



- AC susceptometry
- IV. Imaging techniques



Scanning probe microscopies, **Photon-out** 



#### Using XMCD contrast to image





X-ray absorption depends on the angle of incidence regarding to the magnetic moment direction

=> Magnetic contrast

<u>BONUS</u>: Absorption depends on electronic transitions => Chemical selectivity

#### X-ray miscroscopy

#### Transmission X-ray microscopy (TXM)

(ALS, SLS, BESSY II, ANKA)



**Advantages :** large image (10 μm) with good, 50ps time resolution

**Disadvantages :** mostly 300K, small field, lenses

Distance between 2 circles => resolution (here 15 nm)



### X-ray miscroscopy



Still 50 ps time resolution and 25 nm resolution **BUT Only one lense AND Scanning** => More flux locally Scanning Sample Stage

### X-ray miscroscopy

#### X-ray Holography



Advantage : 400K-10K, more space around the sample Disadvantage : sample preparation, limited image size (< 2 μm) Loss in resolution (50nm) Phase retrieval

> Dynamics in MTXM, STXM, Holography requieres stromboscopy



Detector



**Excitation :** Current pulsed at t=0ps

Micromagnetic simulation (Scheinfein code)



D.P. Bernstein et al., PRB(R) 83, 180410 (2011)



**Excitation :** Current pulsed at t=0ps

Preset t = 0 pst = 50 psCo 100nm  $t = 250 \, ps$  $t = 550 \, ps$ Set



D.P. Bernstein et al., PRB(R) 83, 180410 (2011)

(Scheinfein code)



**Excitation :** Current pulsed at t=0ps



Micromagnetic simulation (Scheinfein code)



D.P. Bernstein et al., PRB(R) 83, 180410 (2011)



**Excitation :** Current pulsed at t=0ps



0 ps

550 ps



100 nm

250 ps

Micromagnetic simulation (Scheinfein code)

D.P. Bernstein et al., PRB(R) 83, 180410 (2011)

150 ps

1200 ps



**Excitation :** Current pulsed at t=0ps

Preset t = 0 ps t = 50 ps **100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 100 10** 

Micromagnetic simulation (Scheinfein code)



D.P. Bernstein et al., PRB(R) 83, 180410 (2011)

#### X-ray microscopy : criteria

![](_page_93_Figure_1.jpeg)

#### Magnetic measurement : Summary

![](_page_94_Picture_1.jpeg)

I. Few preliminary details about the sample environment

Magnetic field	l, Temperature

II. Static macroscopic magnetometry techniques

VSM, SQUID, Torque, MOKE, XMCD

III. AC macroscopic magnetometry techniques

![](_page_94_Picture_7.jpeg)

#### IV. Imaging techniques

Scanning probe microscopies, Photon-out

#### **CONCLUSION :**

Know your tool box and you will find the **rigth** tool**S** to answer your need

#### Magnetometers are not only lab tools

#### Here I focused on material characterization in lab but magnetometers can be used in many ways

![](_page_95_Figure_2.jpeg)

#### Few references

- SQUID R.L. Fagaly Review of Scientific Instruments 77, 101101 (2006)
   M. Buchner et al. J. Appl. Phys. 124, 161101 (2018);
   D.-X. Chen, et al., REVIEW OF SCIENTIFIC INSTRUMENTS 82, 045112 (2011)
- VSM S. Foner, Review of Scientific Instruments 30, 548 (1959)V. Lopez, Review of Scientific Instruments 89, 034707 (2018)
- Torque S. Brialmont et al., Rev. Sci. Instrum. 90, 085101 (2019)
- AC susceptometry
   M. Nikolo, Am. J. Phys., 63, 57 (1995)

   F. Gömöry, Supercond. Sci. Technol. 10 (1997) 523
- **MOKE** J. McCord, J. Phys. D: Appl. Phys. 48, 333001 (2015)
- Magnetic ImagingR.M. Reeve et al. arXiv:1806.07767v2W. Kuch, Book "Magnetism: A Synchrotron Radiation Approach", p275-320 (2006)

And many more at : http://magnetism.eu/esm/repository-topics.html

![](_page_96_Picture_8.jpeg)