Basic magnetic measurement methods

Magnetic materials in nanoelectronics

- properties and fabrication

Lecture ☐ ☐ ● ☐

M.Urbaniak
aciej

Magnetic measurements in nanoelectronics

1. Vibrating sample magnetometry and related methods

2. Magnetooptical methods

3. Other methods

Introduction

Magnetization is a quantity of interest in many measurements involving spintronic materials

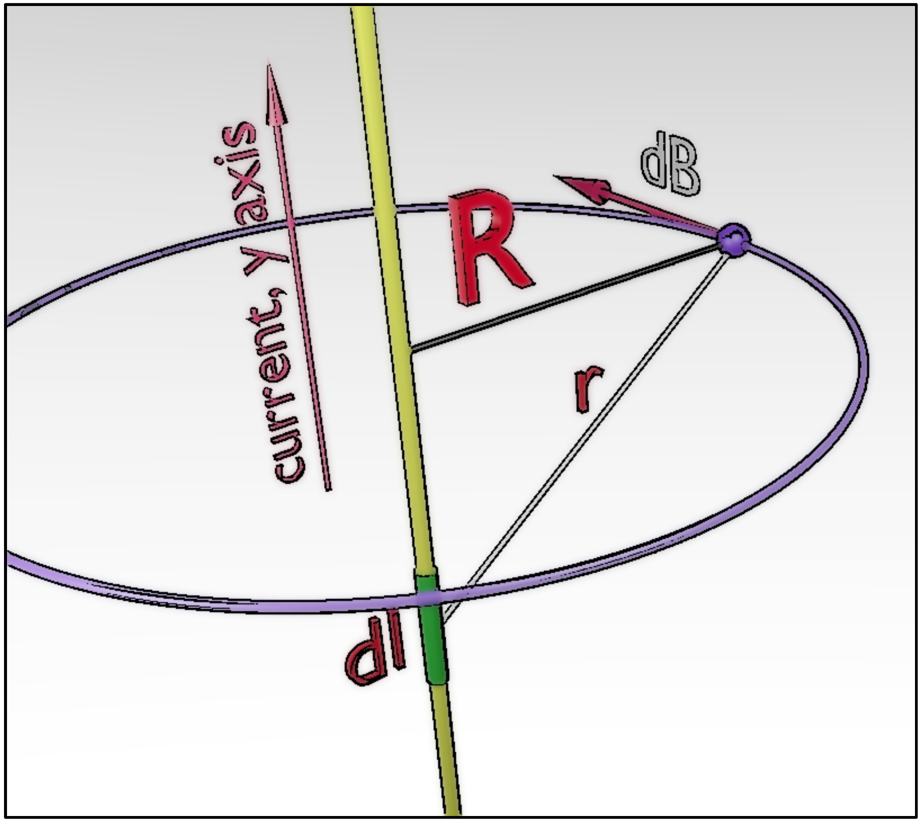
- Biot-Savart law (1820) (Jean-Baptiste Biot (1774-1862), Félix Savart (1791-1841))

Magnetic field (the proper name is magnetic flux density [1]*) of a current carrying piece of conductor is given by:

$$d\vec{B}=\frac{\mu_0 I}{4\pi}\frac{\hat{d}l\times\vec{r}}{|\vec{r}|^3}$$

$\mu_0=4\pi 10^{-7}\,Hm^{-1}$ - vacuum permeability

- The unit of the magnetic flux density, Tesla (1 T=1 Wb/m^2), as a derive unit of Si must be based on some measurement (force, magnetic resonance)



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*the alternative name is magnetic induction

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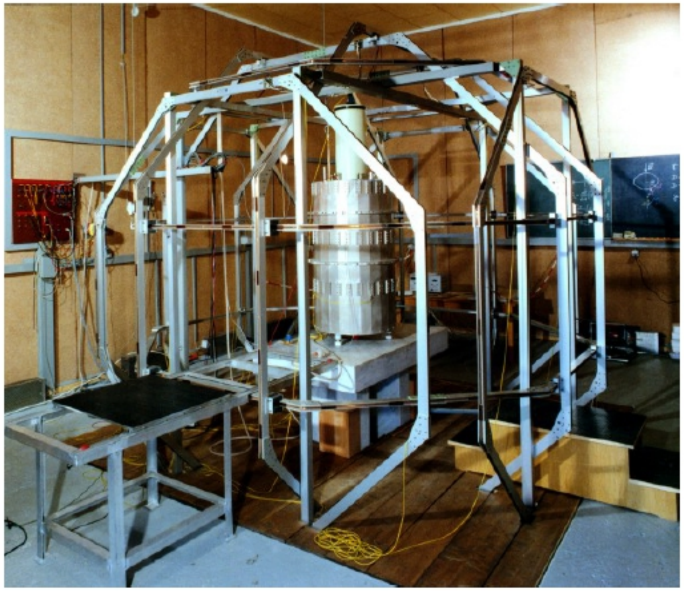
Figure 1:

Arrangement of Braunbek coils for compensation of the three components of the earth's magnetic field



- The Physikalisch-Technische Bundesanstalt (German national metrology institute) maintains a unit Tesla in form of coils with coil constant k (ratio of the magnetic flux density to the coil current) determined based on NMR measurements

graphics from:
http://www.ptb.de/cms/fileadmin/internet/fachabteilungen/abteilung_2/2.5_halbleiterphysik_und_magnetismus/2.51/realization.pdf

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*the alternative name is magnetic induction

Introduction

It can be shown that magnetic flux density can be expressed as*:

$$\vec{B}(\vec{r})=\nabla\times\frac{\mu_0}{4\pi}\int\frac{j(\vec{r}\,')}{|r-r'|}d^3r'$$

This is called **magnetic vector potential**

$\vec{j}(\vec{r}\,')$ - current density [A/m^2]

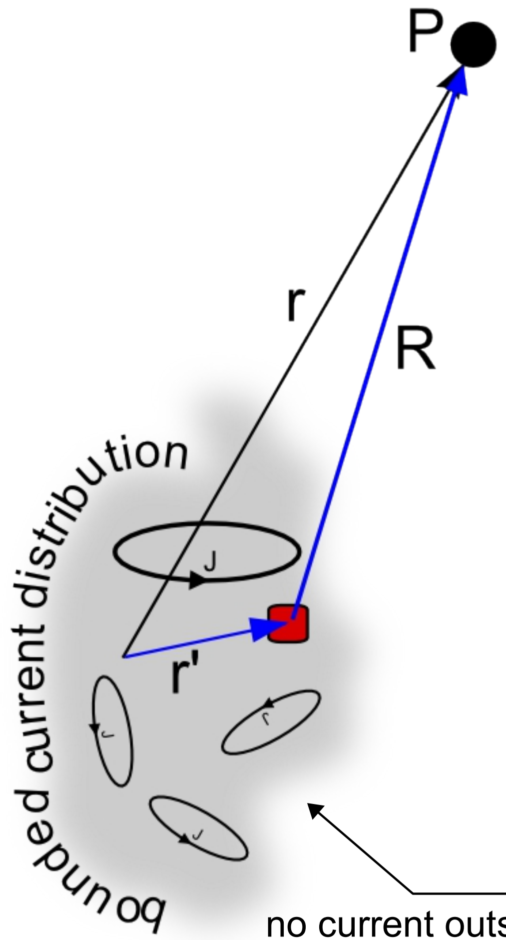
$$\vec{B}(\vec{r})=\nabla\times\vec{A}(\vec{r})$$

It can be further shown that the vector potential created by bounded current density at positions outside the bounding surface and far from it can be approximated by*:

$$\vec{A}(\vec{r})=-\frac{1}{2}\frac{\mu_0}{4\pi}\frac{1}{|\vec{r}|^3}\vec{r}\times\int(\vec{r}\,'\times\vec{j}(\vec{r}\,'))d^3r'$$

This is a multipole expansion of the potential of the current distribution limited to two first terms of the expansion*:

$$\frac{1}{|\vec{r}-\vec{r}\,'|}=\frac{1}{|\vec{r}|}+\frac{\vec{r}\cdot\vec{r}\,'}{|\vec{r}|^3}+\ldots$$



no current outside this boundary

*see for example my lecture *Magnetic field and its sources* from 2012 and references therein (http://www.ifmpan.poznan.pl/urbaniak/Wyklady2012/urbifmpan2012lect1_04powy.pdf)

Introduction

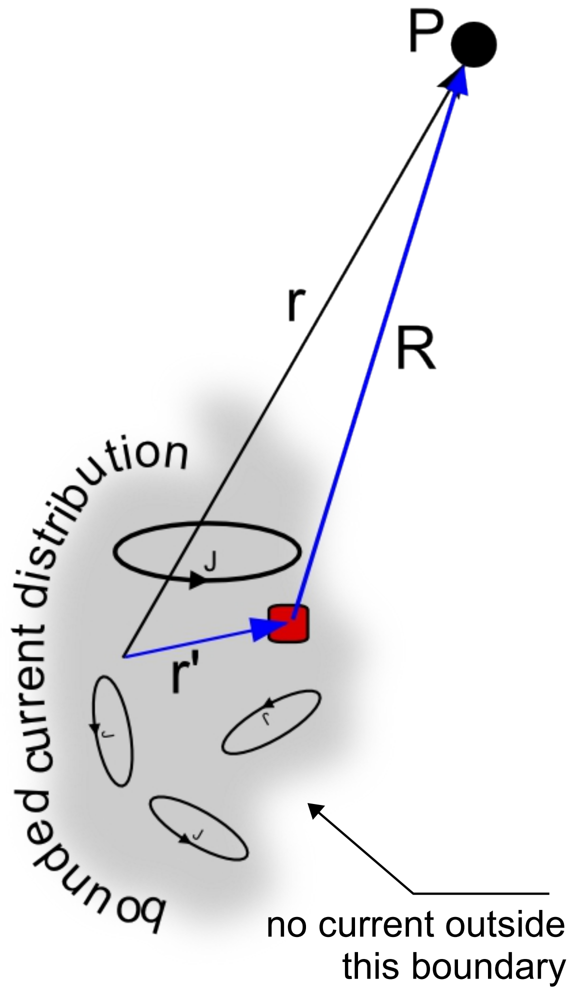
It follows that if a distance ***r*** from the observation point P to the current distribution is much greater than the size of region containing current the material/ sample can be characterized by its second moment:

$$\vec{A}(\vec{r})=-\frac{1}{2}\frac{\mu_0}{4\pi}\frac{1}{|\vec{r}|^3}\vec{r}\times\int(\vec{r}\,'\times\vec{j}(\vec{r}\,'))d^3r'$$

We define **magnetic dipole moment** of current distribution [2]:

$$\vec{m}=\frac{1}{2}\int(\vec{r}\,'\times\vec{j}(\vec{r}\,'))d^3r' \qquad \left[m\cdot\frac{A}{m^2}\cdot m^3=A\cdot m^2\right]$$

- when viewed from far away the details of the current flow within the sample are irrelevant
- the current can be replaced by its dipole moment



Introduction

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The integrand of the above expression is called **magnetization**

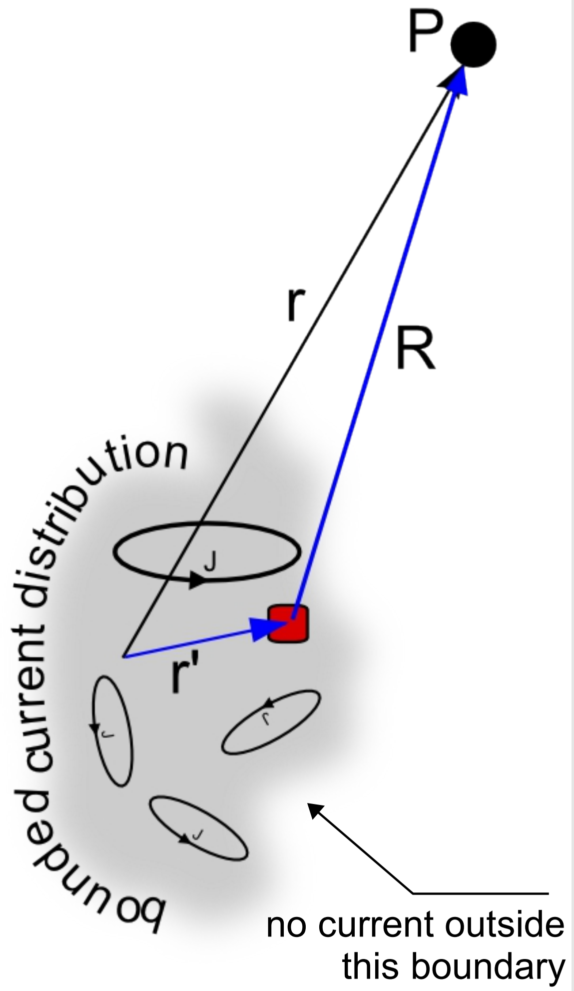
$$\vec{M}(\vec{r})=\frac{1}{2}\vec{r}\times\vec{j}(\vec{r})$$

$M_{Fe} \approx 1.7\times10^6$ A/m

$M_{Co} \approx 1.4\times10^6$ A/m

$M_{Ni} \approx 0.5\times10^6$ A/m

at RT



Introduction

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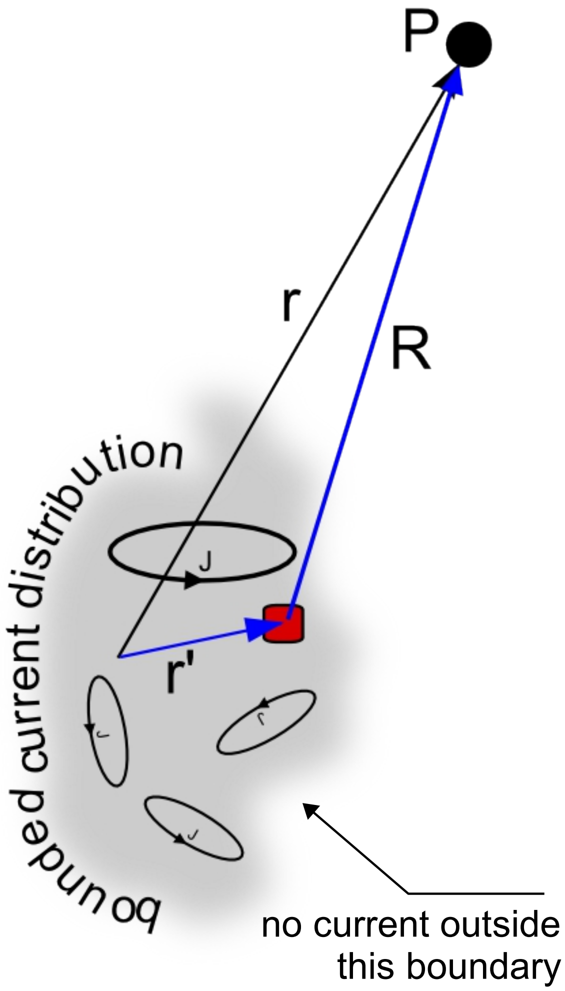
It can be further shown that (still far away from the current) that*:

$$\vec{B}(\vec{r})=\boxed{\frac{\mu_0}{4\pi}\frac{3\hat{r}(\vec{m}\cdot\hat{r})-\vec{m}}{|\vec{r}|^3}}$$

We should compare it with the expression for the field of **electric dipole** [2, 3]:

$$\vec{E}(\vec{r})=\frac{1}{4\pi\varepsilon_0}\frac{3\hat{r}(\vec{p}\cdot\hat{r})-\vec{p}}{|\vec{r}|^3}$$



no current outside this boundary

*see for example my lecture *Magnetic field and its sources* from 2012 and references therein (http://www.ifmpan.poznan.pl/urbaniak/Wyklady2012/urbifmpan2012lect1_04powy.pdf)

Introduction

Unless one is involved in investigations of neutron stars [4], nuclear physics [5],

misaligned magnetic dipole and magnetic quadrupole

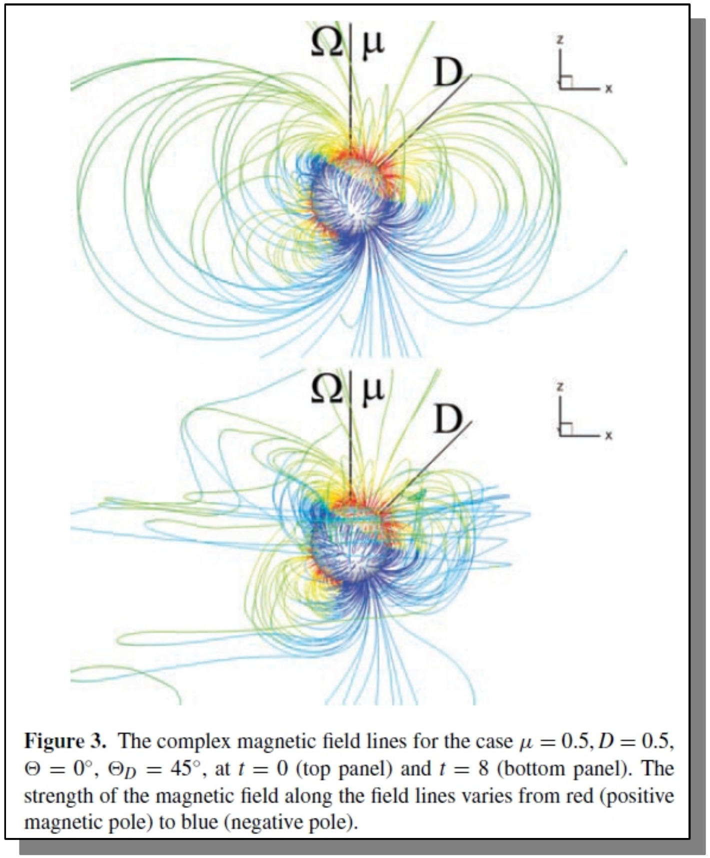
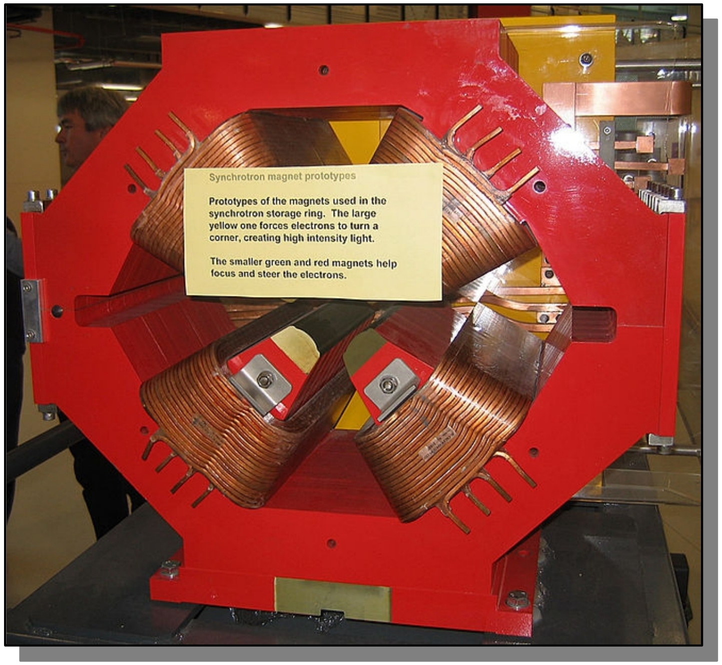


Figure 3. The complex magnetic field lines for the case $\mu = 0.5, D = 0.5$, $\Theta = 0°$, $\Theta_D = 45°$, at $t = 0$ (top panel) and $t = 8$ (bottom panel). The strength of the magnetic field along the field lines varies from red (positive magnetic pole) to blue (negative pole).

graphics from:
M. Long, M. M. Romanova and R. V. E. Lovelace
Mon. Not. R. Astron. Soc. **386**, 1274 (2008)

..., or design of magnets



graphics from Wikimedia Commons;
author: jjron

Quadrupole focusing magnet as used in the storage ring at the Australian Synchrotron, Clayton, Victoria.

...it is usually enough to use magnetic dipole approximation

Introduction

Magnetic field strength ***H***

We distinguish two types of currents contributing to magnetic field:

- **the free currents** – flowing in lossy circuits (coils, electromagnets) or superconducting coils; in general one can influence (switch on/off) and measure free currents
- **the bound currents** – due to intratomic or intramolecular currents and to magnetic moments of elementary particles with spin [6]

It can be shown that*:

$$\vec{A}(\vec{r})=\frac{\mu_0}{4\pi}\int\frac{\vec{j}_{free}(\vec{r}\,')+\nabla'\times\vec{M}(\vec{r}\,')}{|\vec{r}-\vec{r}\,'|}d^3r'$$

The effect of magnetic moment distribution on magnetic field is the same as that of current distribution given by:

$$\vec{j}_{bound}(\vec{r})=\nabla\times\vec{M}(\vec{r})$$

*see for example my lecture *Magnetic field and its sources* from 2012 and references therein (http://www.ifmpan.poznan.pl/urbaniak/Wyklady2012/urbifmpan2012lect1_04powy.pdf)

Introduction

Magnetic field strength *H***

- From Biot-Savart law we have:

$$\nabla\times\vec{B}=\mu_0\vec{j}(\vec{r})=\mu_0\vec{j}_{free}+\mu_0\vec{j}_{bound}=\mu_0\vec{j}_{free}+\mu_0\nabla\times\vec{M} \qquad (1)$$

- We introduce a vector:

$$\vec{H}=\frac{1}{\mu_0}\vec{B}-\vec{M}$$

In cgs system*:

$$\vec{H}=\vec{B}-4\pi\vec{M}$$

- From (1) we have:

$$\nabla\times\vec{B}-\mu_0\nabla\times\vec{M}=\mu_0\nabla\times(\frac{1}{\mu_0}\vec{B}-\vec{M})=\mu_0\vec{j}_{free}$$

- It follows that the rotation of field strength **H is determined solely by the free currents**:

$$\nabla\times\vec{H}=\vec{j}_{free}$$

- In general $\nabla\cdot\vec{H}\neq0$ i.e. magnetic field strength is not source-free.

**this section is taken from K.J. Ebeling and J. Mähnß [3]

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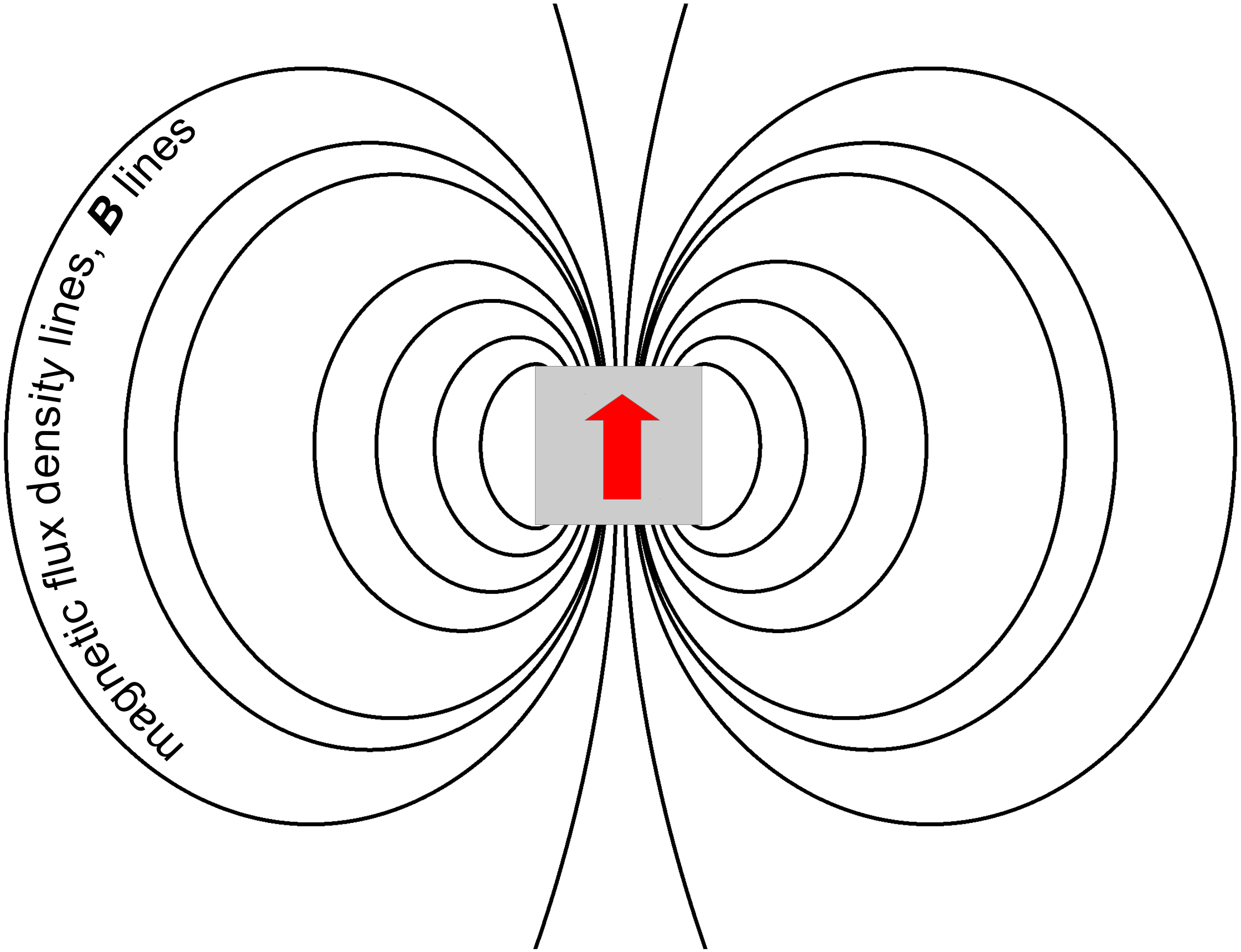
*it is an obsolete system but there are still some active users (http://bohr.physics.berkeley.edu/classes/221/1112/notes/emunits.pdf)

Introduction

Magnetic field strength ***H***



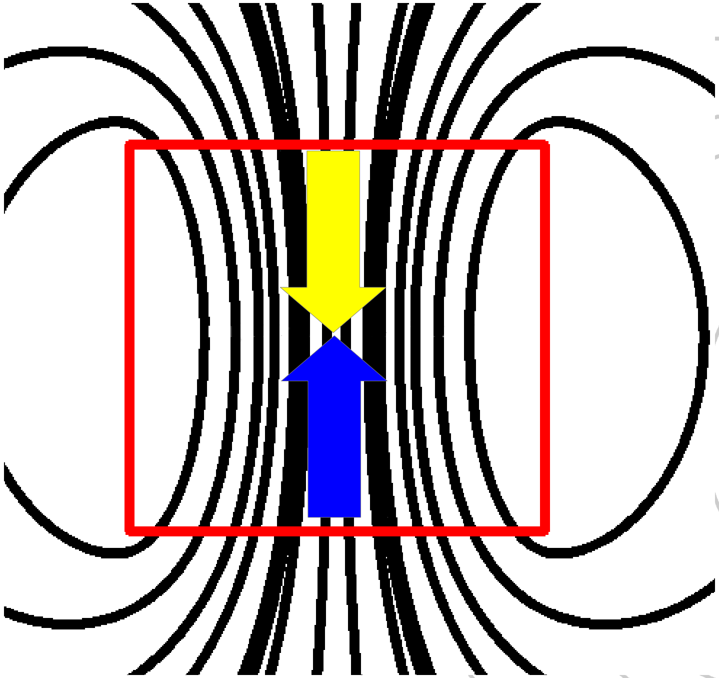
B field lines of a rectangular magnet, **magnetized** upward, and infinite in third dimension

- In general $\boxed{\nabla \cdot \vec{H} \neq 0}$ i.e. magnetic field strength is not source-free.

Introduction

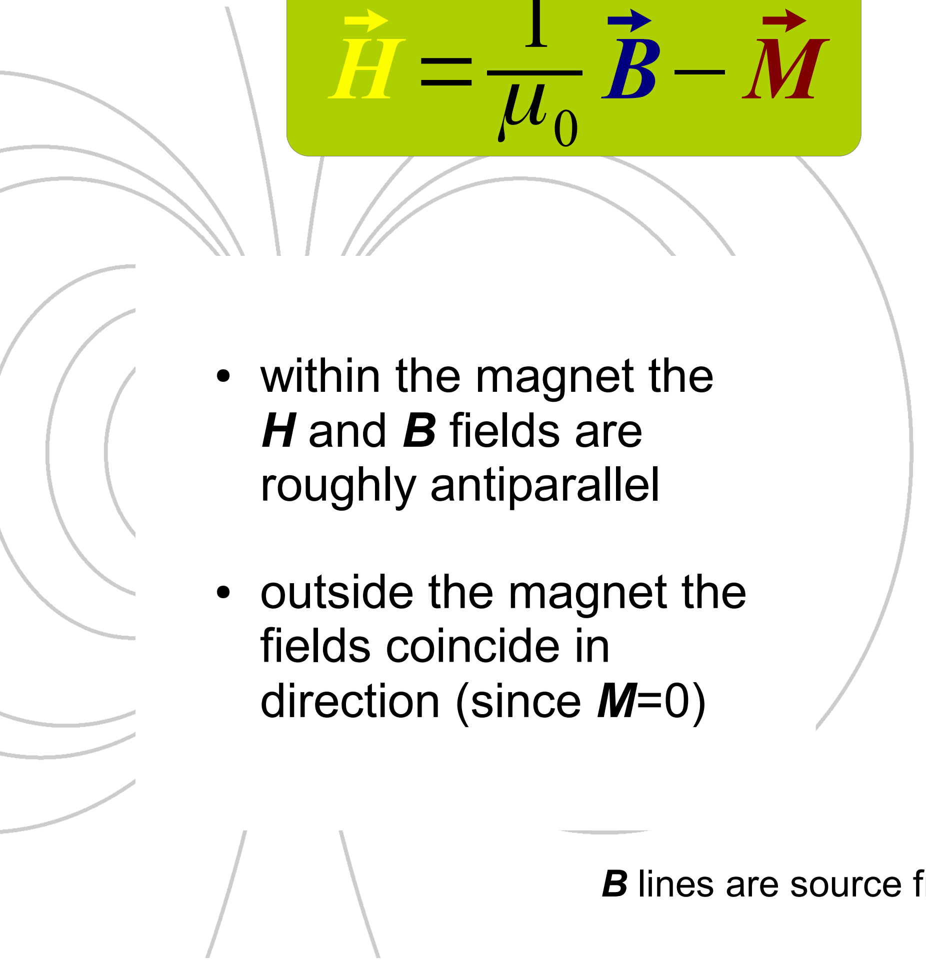
Magnetic field strength *H*

$$\vec{H}=\frac{1}{\mu_0}\vec{B}-\vec{M}$$



B field lines of a rectangular magnet, **magnetized** upward, and infinite in third dimension

- within the magnet the ***H*** and ***B*** fields are roughly antiparallel
- outside the magnet the fields coincide in direction (since ***M***=0)



B lines are source free

- In general $\nabla\cdot\vec{H}\neq 0$ i.e. magnetic field strength is not source-free.

Introduction

Magnetoreception

- “The magnetic field of the Earth provides a pervasive and reliable source of directional information that certain animals can use as an orientation cue while migrating, homing, or moving around their habitat.”- Sönke Johnsen and Kenneth J. Lohmann
- Diverse animal species (bees, salamanders, turtles, birds etc.) possess magnetoreceptory senses
- Humans do not seem to have the ability to sense either direction or the intensity of magnetic field.

Figure 2. Bacterial magnetoreceptors. Transmission electron micrograph of the bacterium *Magnetospirillum magnetotacticum* showing the chain of magnetosomes inside the cell. The magnetite crystal incorporated in each magnetosome is about 42 nm long. (Image courtesy of Dennis Bazylinski.)

- Magnetobacterias possess **magnetosomes containing magnetic materials** (single domain size range; for example magnetite Fe_3O_4)
- The torque on chain of magnetosomes is strong enough to turn the entire bacteria along magnetic field direction (field inclination). They use this to sense what direction is “down” - they prefer deeper, less oxygenated mud.
- In higher animals the mechanical sensors in cells are supposed to detect the rotation of magnetosomes.

S. Johnsen, K.J. Lohmann, Physics Today March 2008

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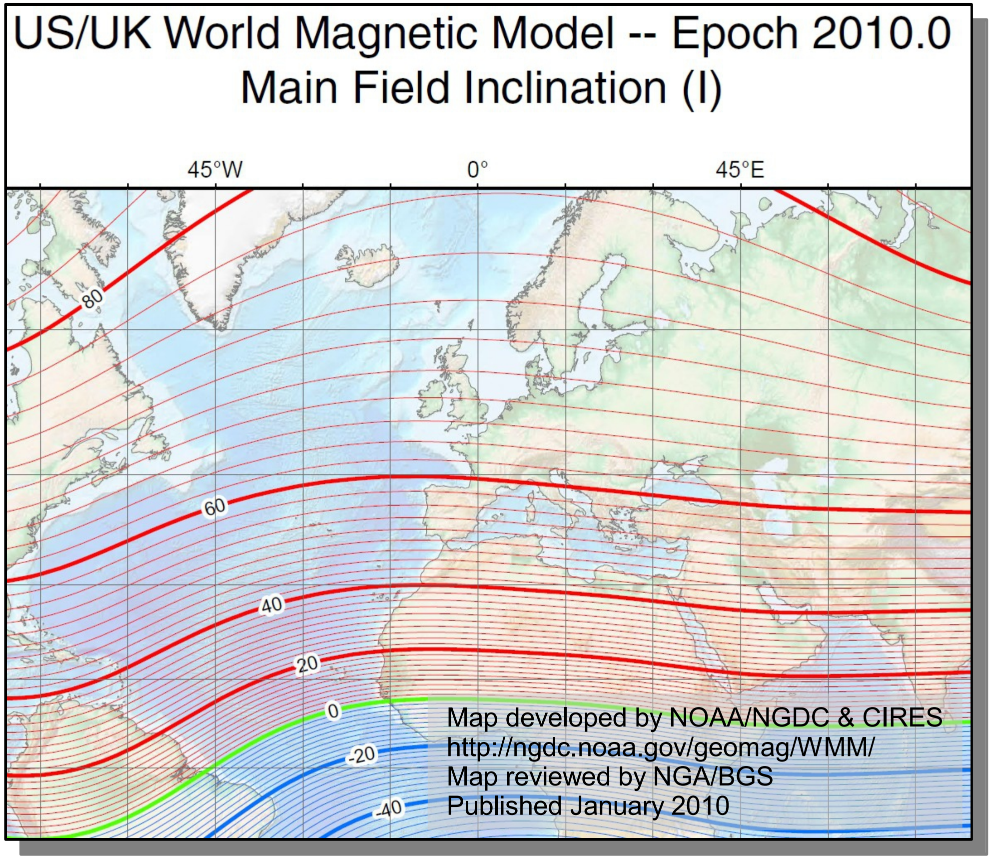

S. Johnsen, K.J. Lohmann, Physics Today March 2008

right image from:http://www.ngdc.noaa.gov/geomag/WMM/data/WMM2010/WMM2010_I_MERC.pdf

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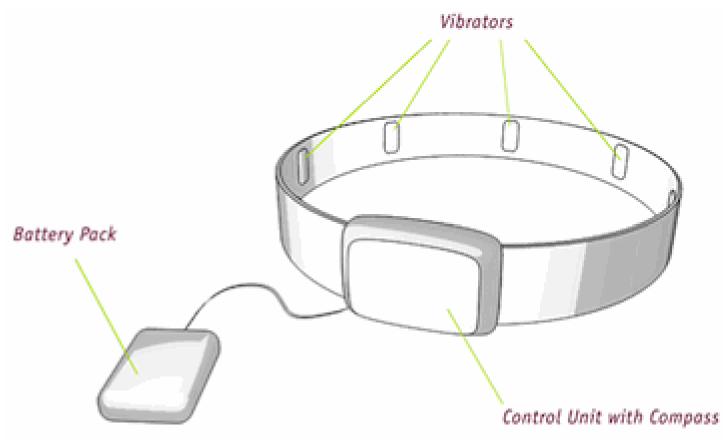
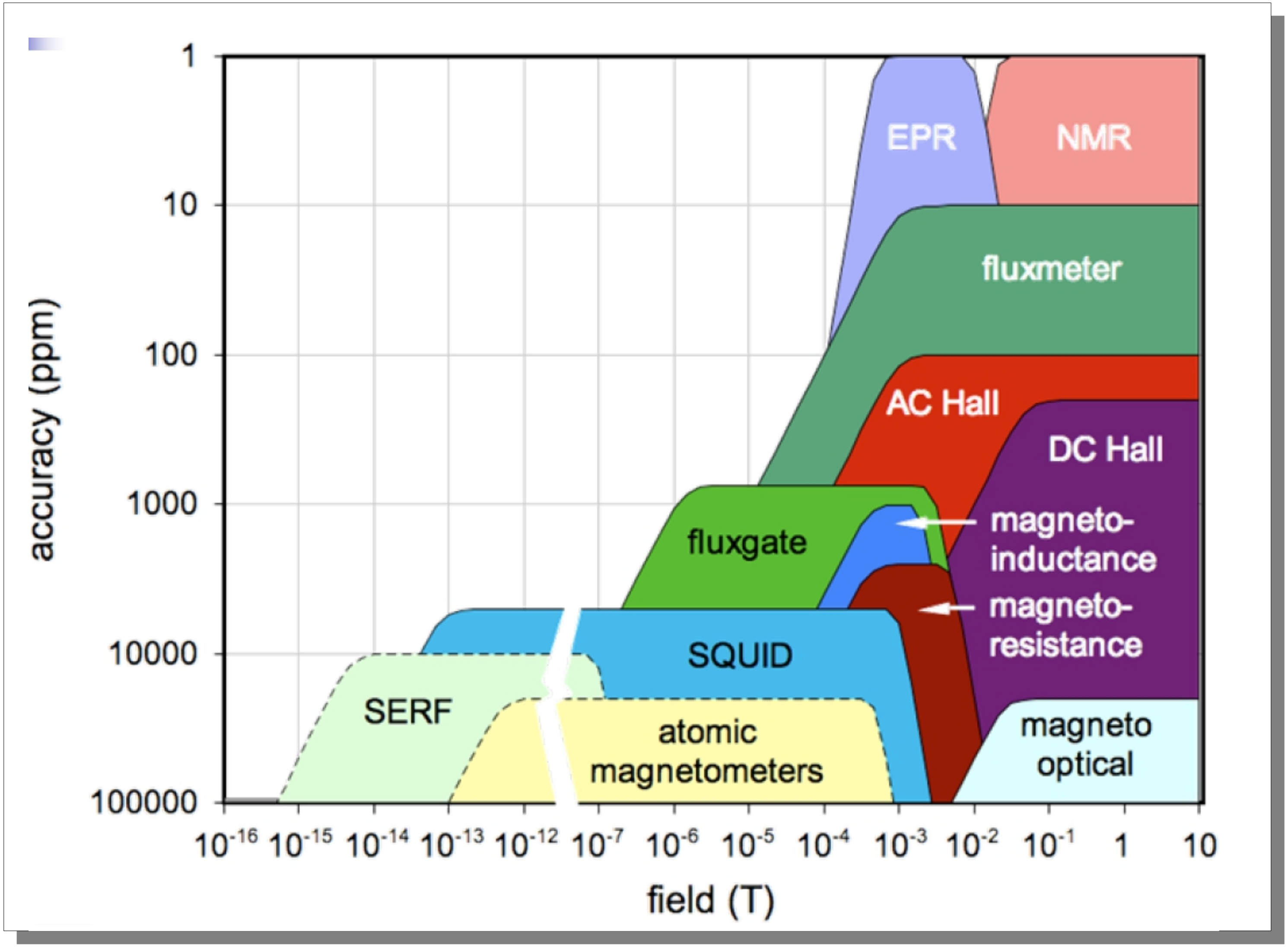


The research is underway to incorporate magnetic field sensing into humans (“*The **feelSpace** belt is a wearable sensory augmentation device that projects the direction of north onto the waist of the user using thirty vibrating actuators.*”)

graphics from:
http://feelspace.cogsci.uni-osnabrueck.de/

Introduction

Measurement of magnetic field strength



methods most relevant in spintronic measurements:
AC and DC Hall probes

The sensitivity range of the probe should cover whole range of fields in which magnetic configuration significantly changes

graphics from *Field Measurement Methods* lecture delivered during The Cern Accelerator School; Novotel Brugge Centrum, Bruges, Belgium, 16 - 25 June, 2009; author: Luca Bottura

Introduction

- Magnetic sensor can be divided according to different criteria:

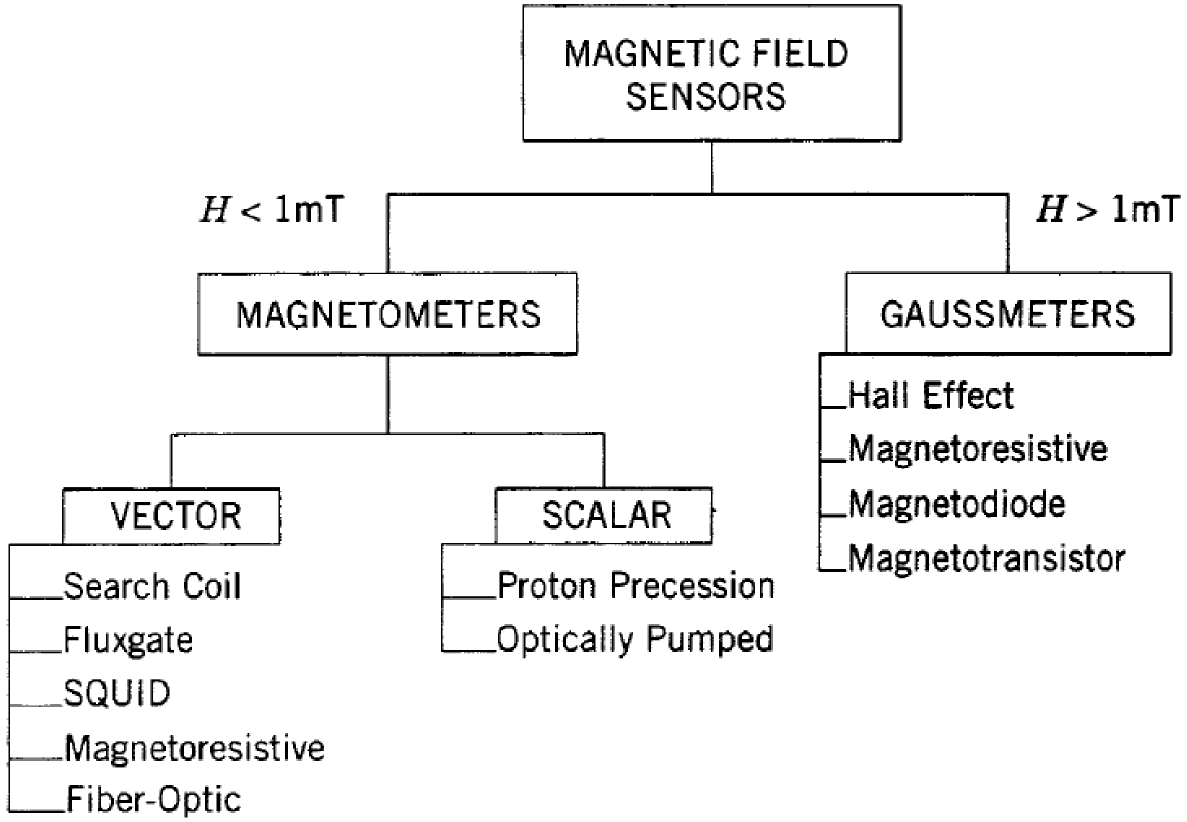


FIGURE 48.1 Magnetic field sensors are divided into two categories based on their field strengths and measurement range: magnetometers measure low fields and gaussmeters measure high fields.

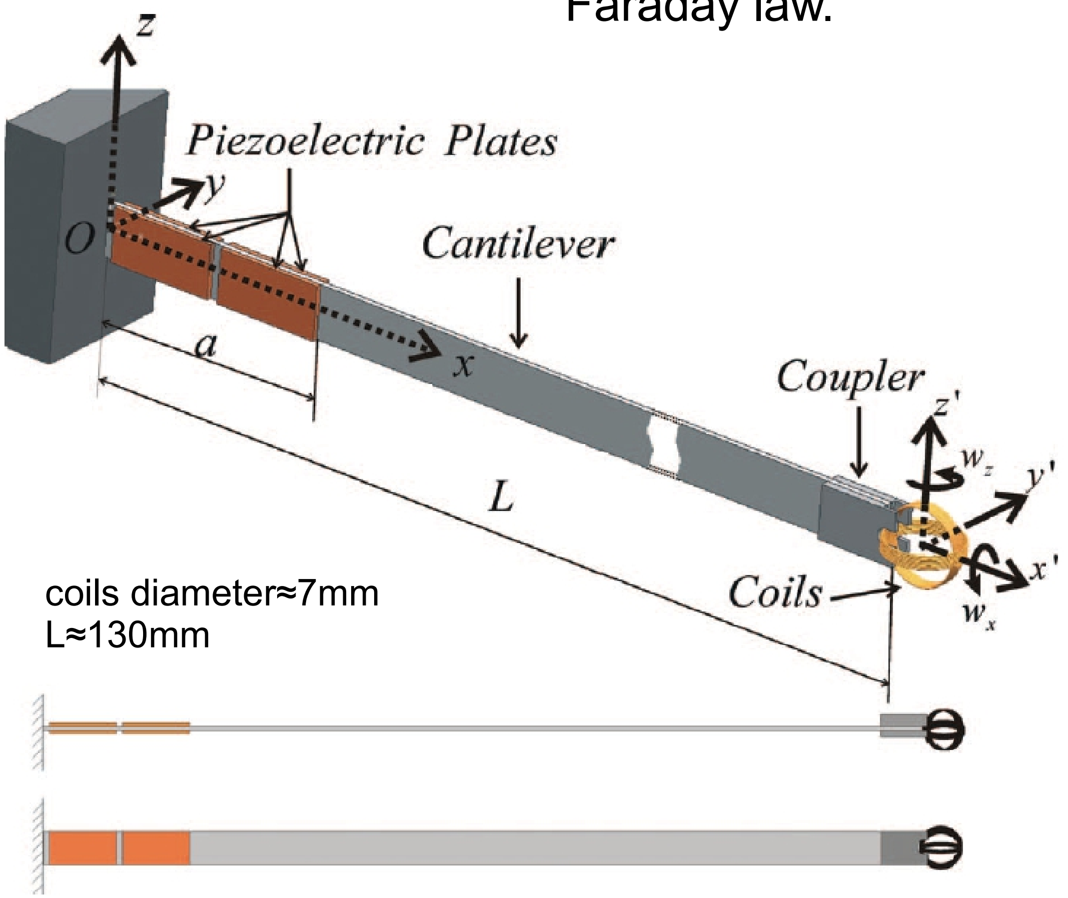
- Distinction magnetometer-gaussmeter is rather arbitrary and not commonly used.

graphics from [7]: S.A. Macintyre, Magnetic Field Measurement

Introduction

Vibrating coils magnetometers:

- Coil magnetometers are usually used to measure varying field.
- The situation can be reversed: direct use of Faraday law.



coils diameter≈7mm
L≈130mm

FIG. 1. (Color online) Schematic diagram of device configuration.

- Piezoelectric sheets are used to excite the cantilever bending
- Two individual sensing coils are orthogonally fastened at the tip of cantilever which bends and twists
- Rotation of the coils allows the measurements of field components perpendicular to rotation axis
- High spatial resolution of measurements - coils virtually at the same position (**3-axis measurement**)

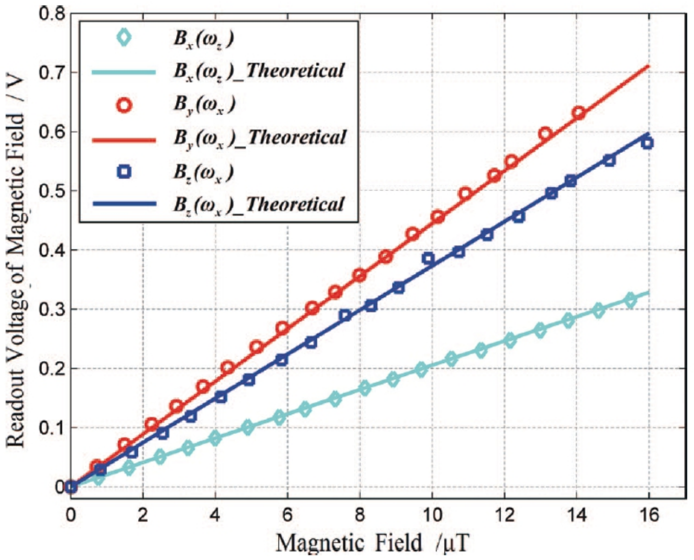


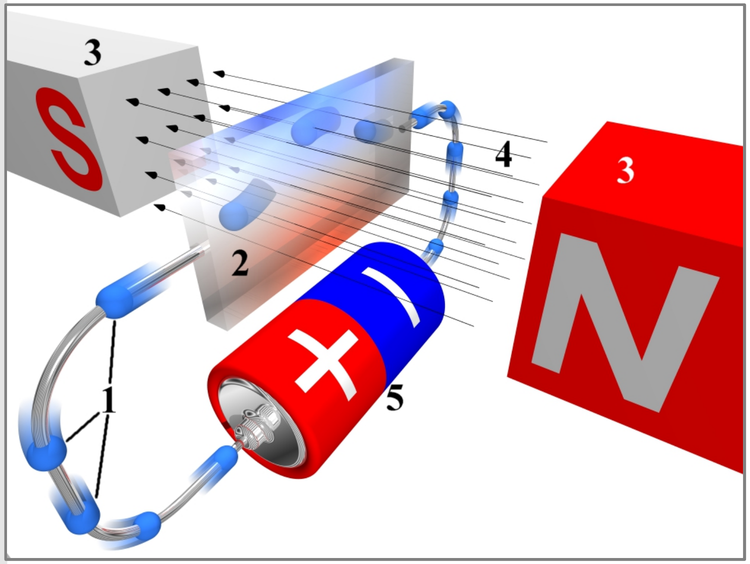
FIG. 6. (Color online) Relationships of measured readout voltages and magnetic fields in the single-mode.

Jing Yin, Cheng Liang Pan, Hong Bo Wang, and Zhi Hua Feng, Rev. Sci. Instrum. 82, 124702 (2011)

Introduction – Hall magnetometer

- Lorentz force acting on electrons in a circuit deflects them perpendicularly to drift direction:

$$\vec{F}_{Lorentz}=q\vec{E}+q\vec{v}\times\vec{B}$$

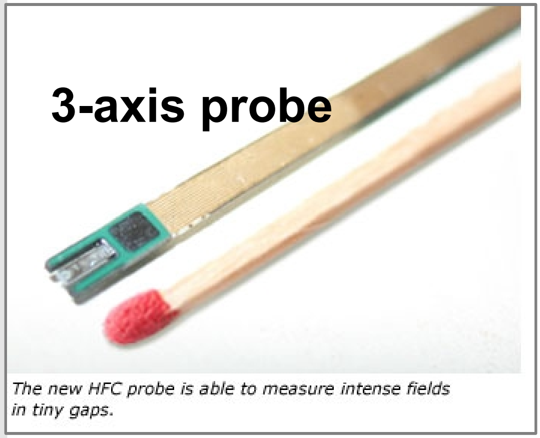


- The build-up of charges on outer limits of the circuit induces Hall voltage which depends on the field strength and is used to sense it.
- The Hall voltage is given by (t-film thickness, R_H- Hall coefficient*):

$$U_y=R_H\frac{I}{t}B_z$$

- The main figure of interest is field sensitivity of the sensor** (for a given driving current I_c):

$$\gamma_b=\frac{U_y}{B_z}=\frac{R_H I_c}{t}$$



The new HFC probe is able to measure intense fields in tiny gaps.

image from http://metrolab.com/

Hall sensors are relatively easy to miniaturize

- Semiconductors are used to obtain high sensitivity combined with temperature stability (InAs)
- The Hall sensors have a limited use at high fields and low temperatures (conductivity quantization)

image from Wikimedia Commons; authors: Peo (modification by Church of emacs)

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*for InAs R_H is about 0.0001 m^3/As **some tenths of mV per kA/m for I_c of several mA (www.lakeshore.com/products/Hall-Magnetic-Sensors/pages/Specifications.aspx)

Introduction – classification of magnetic materials

All materials can be classified in terms of their magnetic behavior falling into one of several categories depending on their bulk magnetic susceptibility χ.

$$\chi=\frac{\vec{M}}{\vec{H}}$$

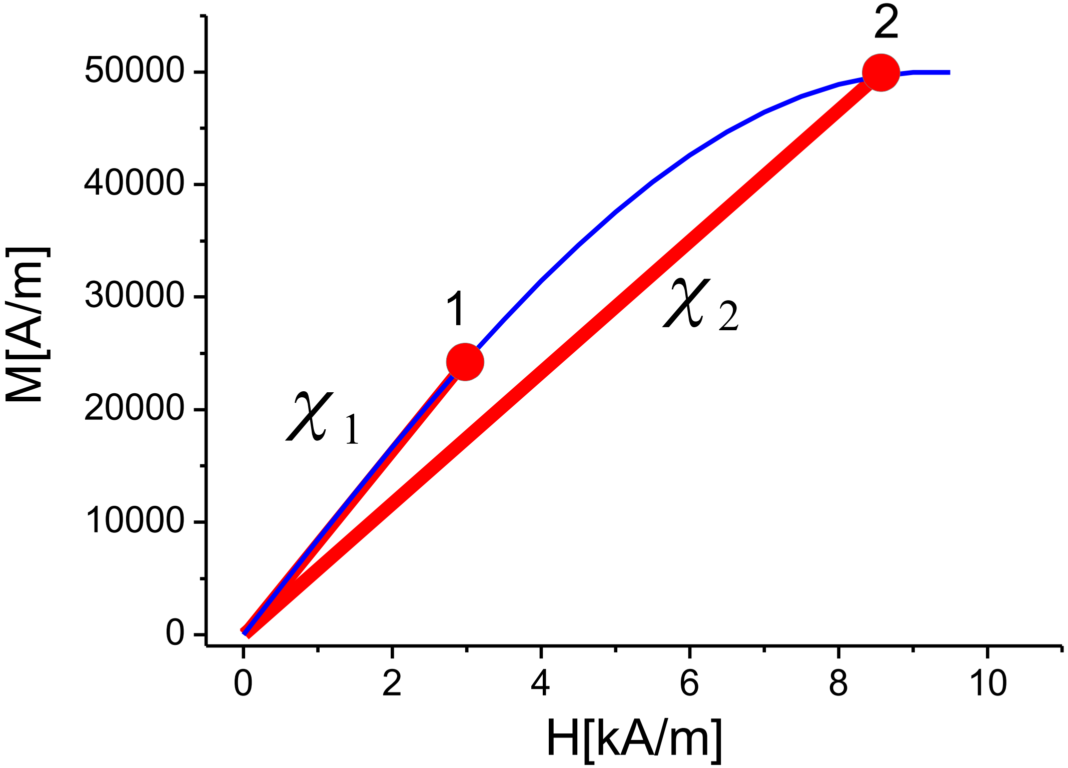
In general the susceptibility is a position dependent tensor

In some materials the magnetization is not a linear function of field strength. In such cases the differential susceptibility is introduced:

$$\chi_d=\frac{d\,\vec{M}}{d\,\vec{H}}$$

We usually talk about isothermal susceptibility:

$$\chi_T=\left(\frac{\partial\vec{M}}{\partial\vec{H}}\right)_T$$



Theoreticians define magnetization as:

$$M=-\left(\frac{\partial\vec{F}}{\partial\vec{H}}\right)_T$$

$F=E-TS$ - Helmholtz free energy

Introduction – classification of magnetic materials

It is customary to define susceptibility in relation to volume, mass or mole (or spin):

$$\chi = \frac{\vec{M}}{\vec{H}} \quad [\text{dimensionless}], \qquad \chi_{\rho} = \frac{(\vec{M}/\rho)}{\vec{H}} \left[\frac{m^3}{kg}\right], \qquad \chi_{mol} = \frac{[\vec{M}/(mol/V)]}{\vec{H}} \left[\frac{m^3}{mol}\right]$$

The general classification of materials according to their magnetic properties

μ<1	$\chi<0$	diamagnetic*
μ>1	$\chi>0$	paramagnetic**
μ≫1	$\chi\gg0$	ferromagnetic***

dia /daɪəmæɡˈnɛtɪk/ -Greek: “from, through, across” - repelled by magnets. We have from:

$$\vec{F} = \frac{1}{2\mu_0}\chi V \nabla(B^2)$$

the force on diamagnet is directed antiparallel to the gradient of ***B*²** i.e. away from the magnetized bodies

- water is diamagnetic $\chi \approx -10^{-5}$ (see “levitating frog” in my lecture 2 from 2012)

** para- Greek: beside, near; for most materials $\chi \approx 10^{-5} - 10^{-3}$ [1].

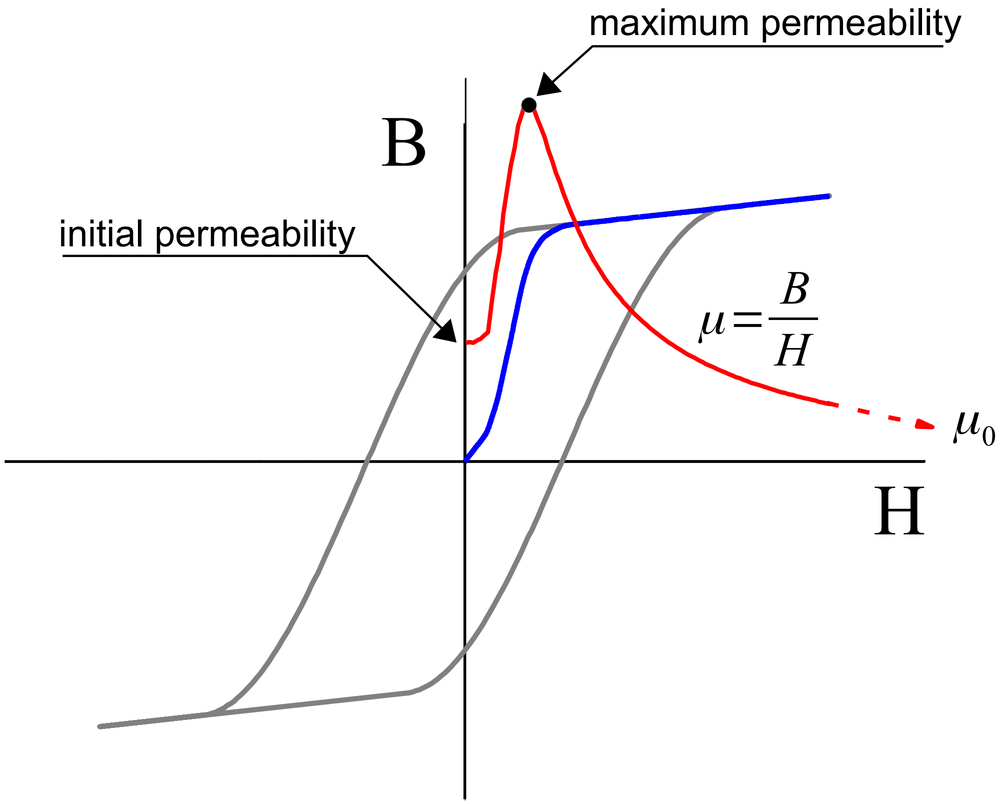
***susceptibility ranges from several hundred for steels to 100,000 for soft magnetic materials (Permalloy)

*see for example my lecture at www.ifmpan.poznan.pl/~urbaniak/Wyklady2012/urbifmpan2012lect2_04.pdf

Introduction – classification of magnetic materials

- Feebly magnetic material – a material generally classified as “nonmagnetic” whose maximum normal permeability is less than 4 [5].
- Ferromagnetic materials can be classified according to the magnetic structure on atomic level:

1. Ferromagnets
2. Antiferromagnets
3. Ferrimagnets
4. Asperomagnets -random ferromagnets
5. Sperimagnets – random ferrimagnets



Introduction – classification of magnetic materials

In general the susceptibility is frequency dependent and the magnetization depends on the preceding field values [3]:

$$\vec{M}(t)=\int f(t-t')H(t')dt'$$

It is customary to introduce a complex susceptibility:

$$\chi=\chi_{real}+i\,\chi_{imag}$$

$$e^{ix}=\cos(x)+i\sin(x)$$

Then we have:

$$\vec{M}=R.e(\chi\vec{H})=R.e[(\chi_{real}+i\,\chi_{imag})\vec{H}_0 e^{-i\omega t}]=\vec{H}_0(\chi_{real}\cos(\omega t)+\chi_{imag}\sin(\omega t))$$

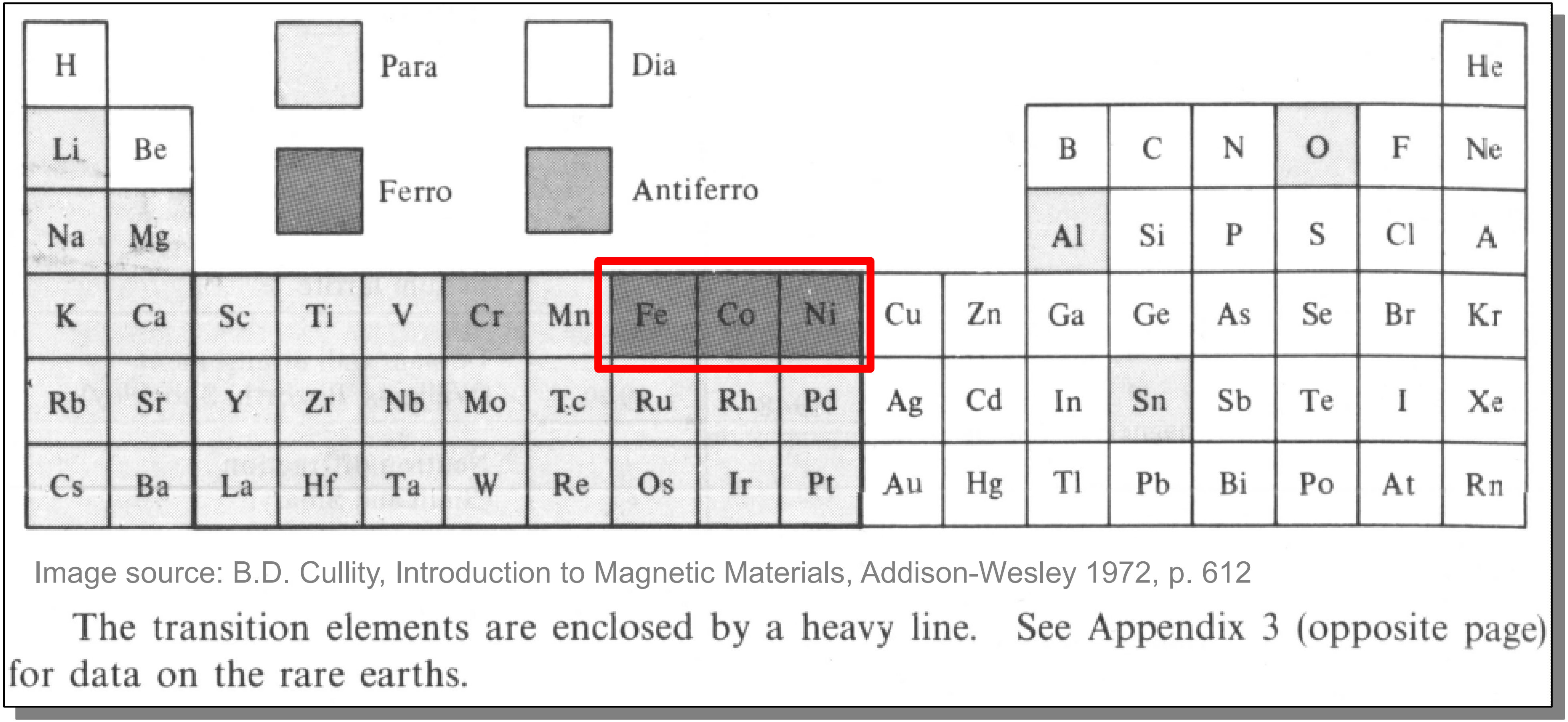
in phase with excitation

out of phase

The imaginary part of susceptibility is responsible for magnetic losses [11, p. 254].

Introduction

Magnetic periodic table of elements



Para

Dia

Ferro

Antiferro

H																	He
Li	Be											B	C	N	O	F	Ne
Na	Mg											Al	Si	P	S	Cl	A
K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
Cs	Ba	La	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn

Image source: B.D. Cullity, Introduction to Magnetic Materials, Addison-Wesley 1972, p. 612

The transition elements are enclosed by a heavy line. See Appendix 3 (opposite page) for data on the rare earths.

Ferromagnetic materials

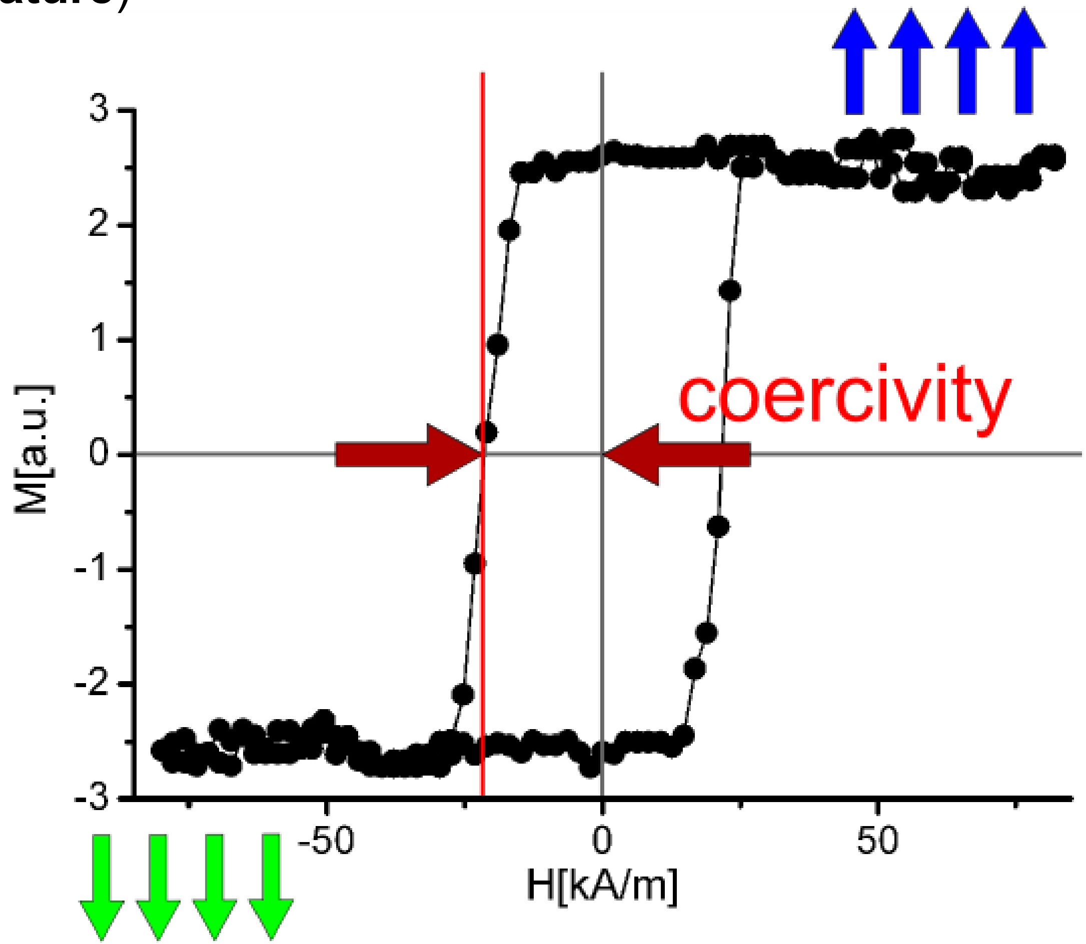
Most notable features of ferromagnetic materials:

- high initial susceptibility/permeability
- they usually retain magnetization after the removal of the external field – **remanence**
- the magnetization curve (B-H or M-H) is nonlinear and hysteretic
- they lose ferromagnetic properties at elevated temperatures (**Curie temperature**)

The notable examples [4]:

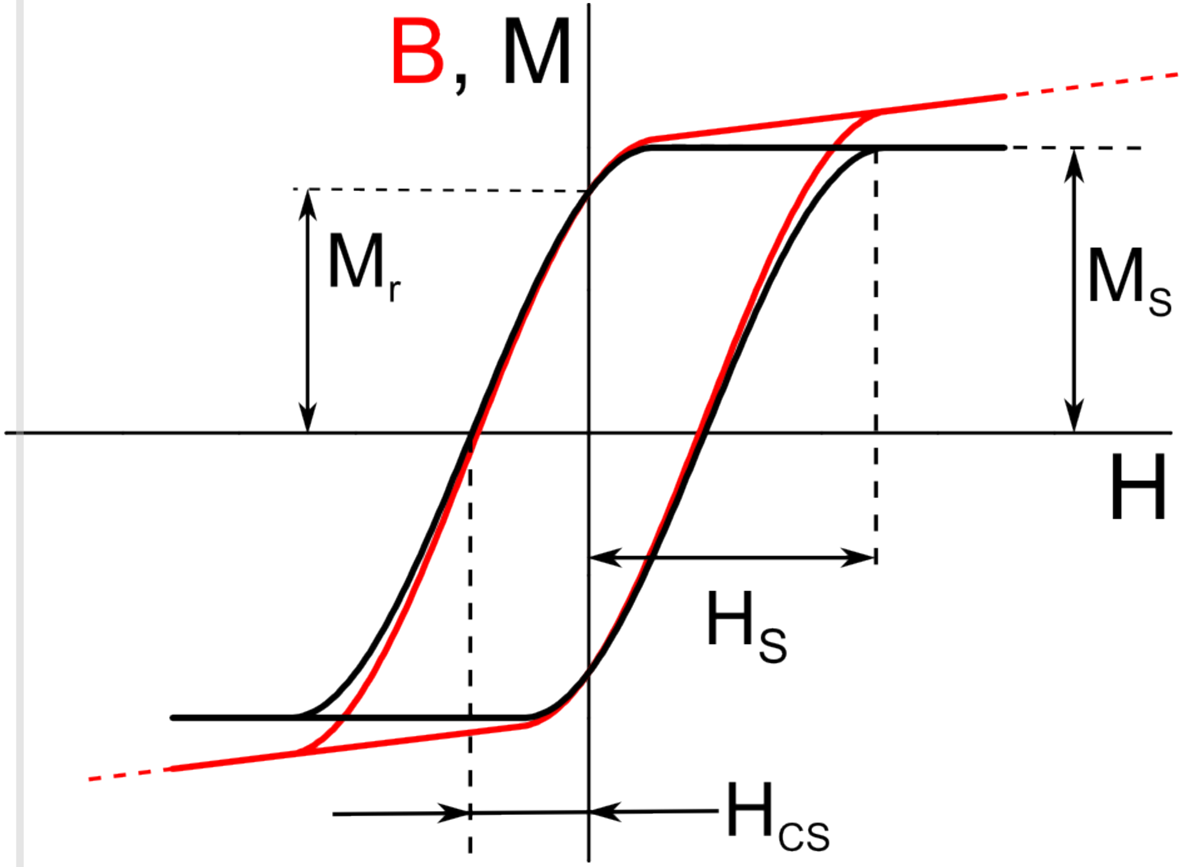
	M_s[kA/m] @RT	T_c[K]
Fe	1714	1043
Co	1433	1403
Ni	485	630

M_s[Am^2/kg] @RT
217.75
161
54.39



Hysteresis nomenclature

The magnetic hysteresis can be presented both as ***B***(***H***) and ***M***(***H***)* dependencies.



intrinsic induction:

$$\vec{B}_i = \vec{B} - \mu_0 \vec{H} = \mu_0 \vec{M}$$

coercive field strength – field required to reduce the **magnetic induction** to zero after the material has been symmetrically cyclically magnetized.

intrinsic coercive field strength – field required to reduce the **intrinsic induction** to zero after...

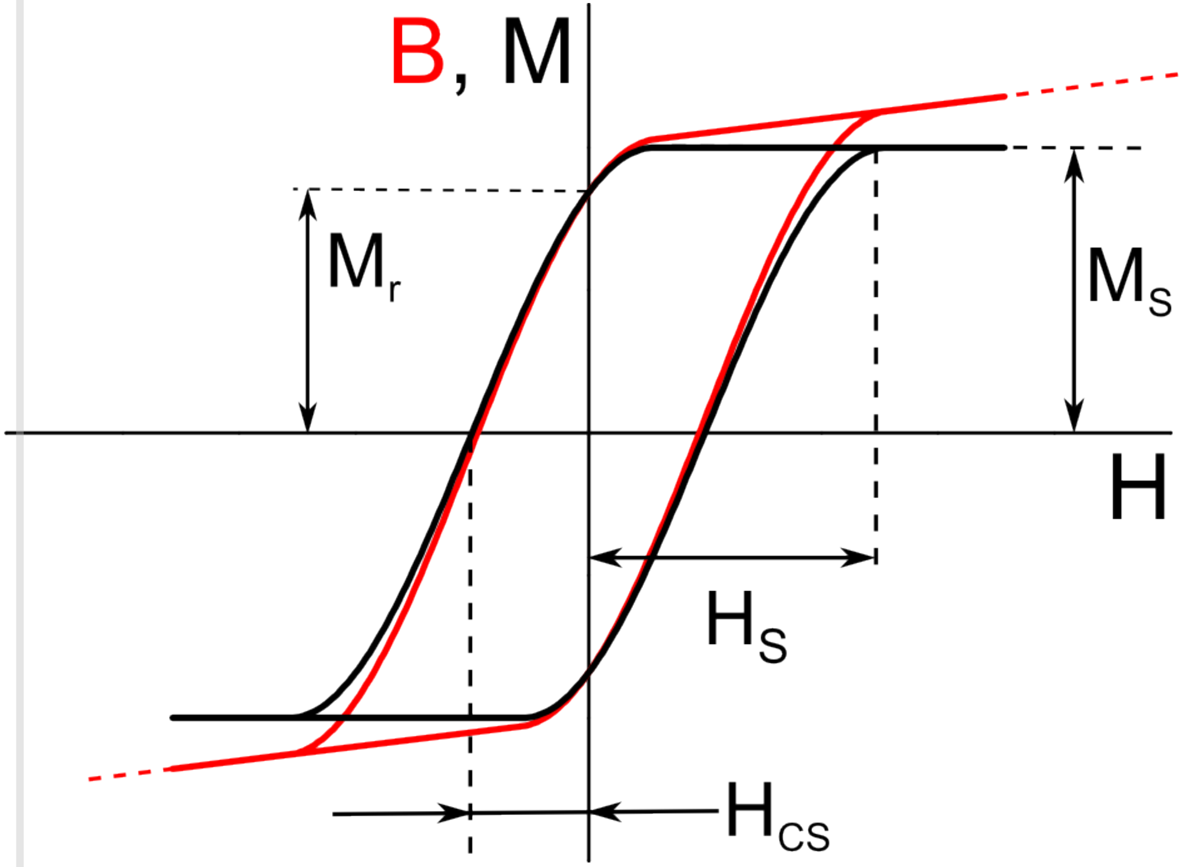
coercivity, H_{cs}—the maximum value of coercive field strength that can be attained when the magnetic material is symmetrically cyclically magnetized to *saturation induction*, B_S.

---

*μ_0 ***M***(H) dependence is called a intrinsic hysteresis loop [8]

Hysteresis nomenclature

The magnetic hysteresis can be presented both as ***B***(***H***) and ***M***(***H***) dependencies.



saturation induction, *B*s—the maximum intrinsic induction possible in a material

saturation magnetization, **M***s:*

$$\vec{M}_s = \vec{B}_s / \mu_0$$

demagnetization curve—the portion of a dc hysteresis loop that lies in the second (or fourth quadrant). Points on this curve are designated by the coordinates, ***B***d and ***H***d.

remanence, *B*$_{dm}$—the maximum value of the remanent induction for a given geometry of the magnetic circuit.

*for the glossary of magnetic measurements terms see ASTM, A 340 – 03a, 2003 [8]

Hysteresis loss

From Faraday's law it follows [4] that the change in a current in circuit 1 produces *emf* in the second circuit:

$$emf_{21} = -M_{21}\frac{dI_1}{dt}$$

At any instant of time the following relation is fulfilled:

$$emf^{appl.} + emf^{ind.} = IR$$

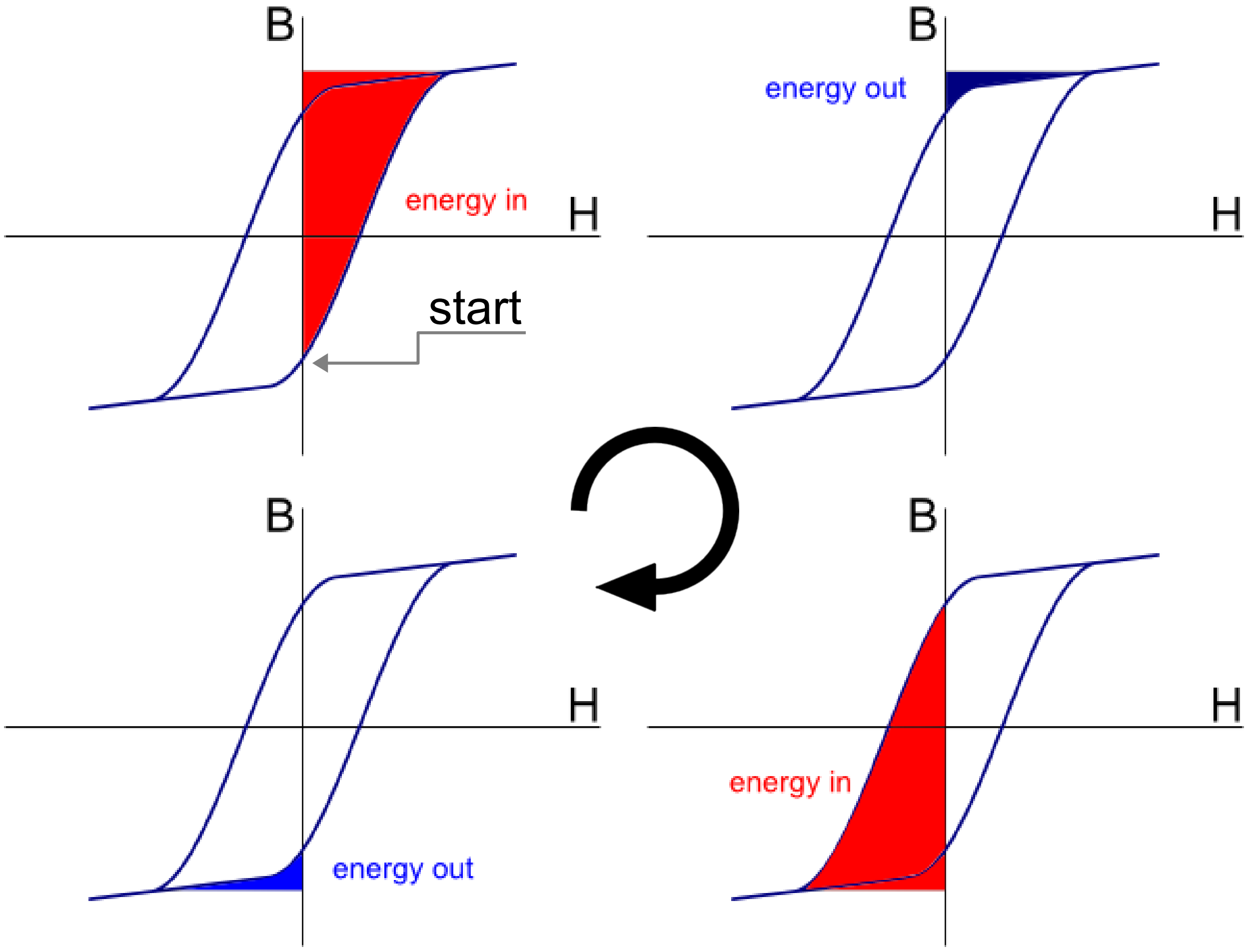
It can be shown [4] that the total energy required to establish a currents in an ensemble of coils fixed in space is:

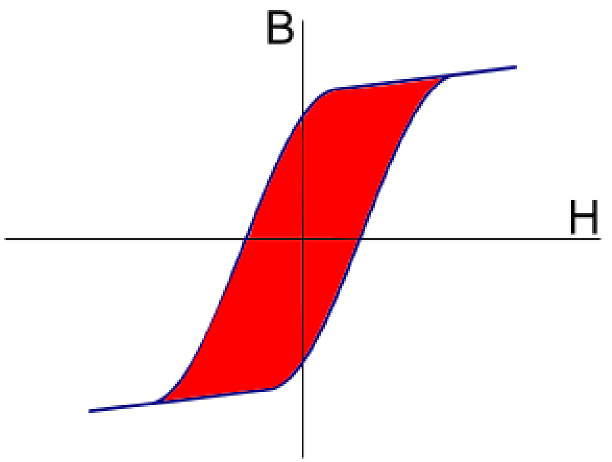
$$W = \frac{1}{2}\sum_i \Phi_i I_i, \qquad where\ \Phi_i = \int_S \vec{B}\cdot dS \quad is\ the\ flux\ \ enclosed\ \ by\ \ i-th\ \ circuit$$

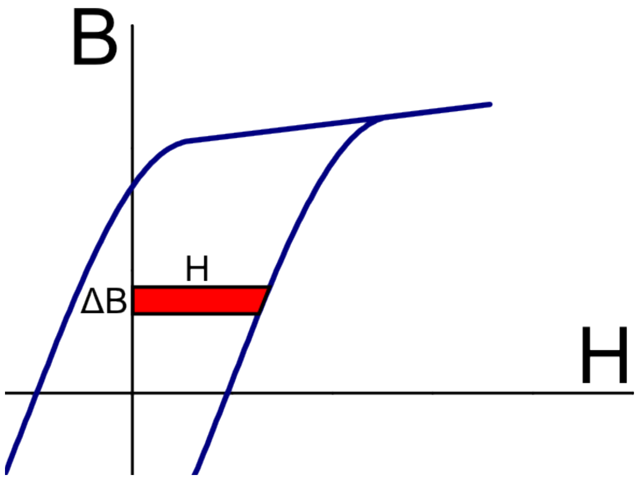
Further it can be shown that the total energy may be expressed by:

$$W = \int \Delta\vec{B}\cdot\vec{H}\,dV$$

Hysteresis loss

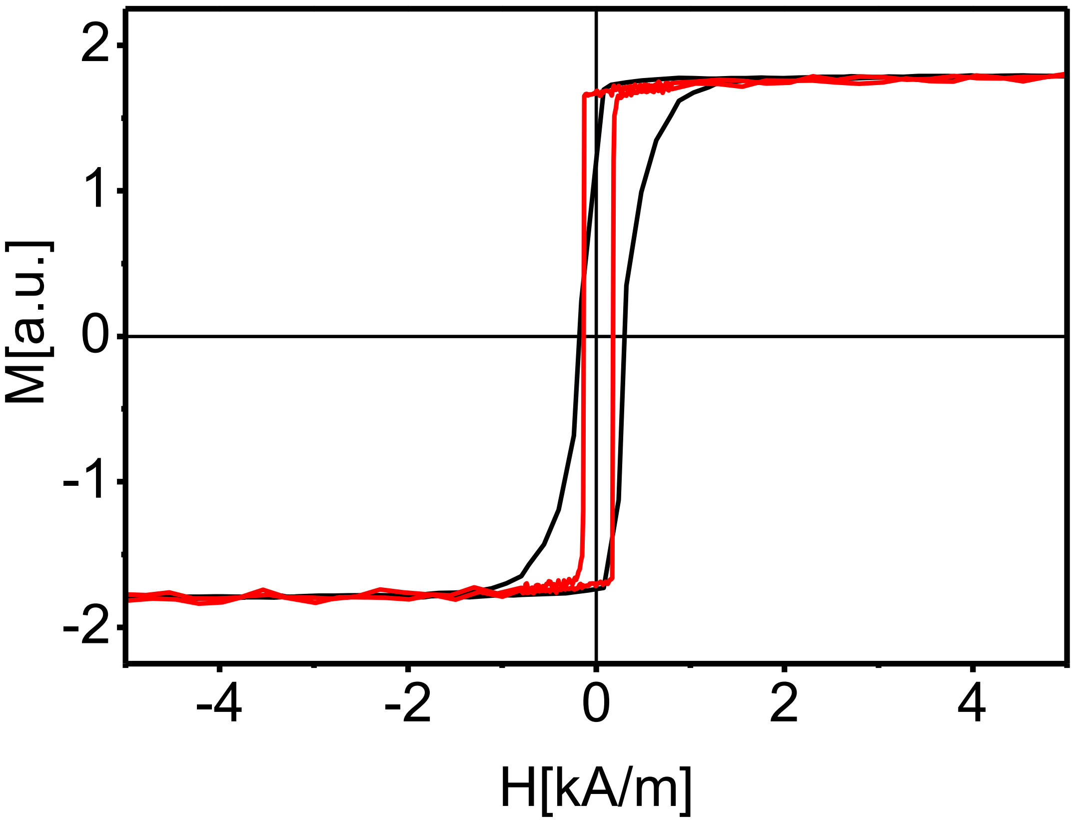






$$W = \int \Delta\vec{B}\cdot\vec{H}\,dV$$

Hysteresis – not an intrinsic property



- Hysteresis is not an intrinsic property of the material
- The character of magnetization depends on sample preparation and the shape of the sample.

$[Ni_{80}Fe_{20}(2\ nm)/Au(5\ nm)]_{15}$

$Ni_{80}Fe_{20}(38\ nm)$

$Ni_{80}Fe_{20}$- soft magnetic material, $H_C \approx 160$ A/m

Minor hysteresis loops

- Minor hysteresis – external field does not saturate the sample
- First order reversal curves allow the characterization of interactions between magnetic particles in particulate media (magnetic recording)

J. Appl. Phys., Vol. 85, No. 9, 1 May 1999

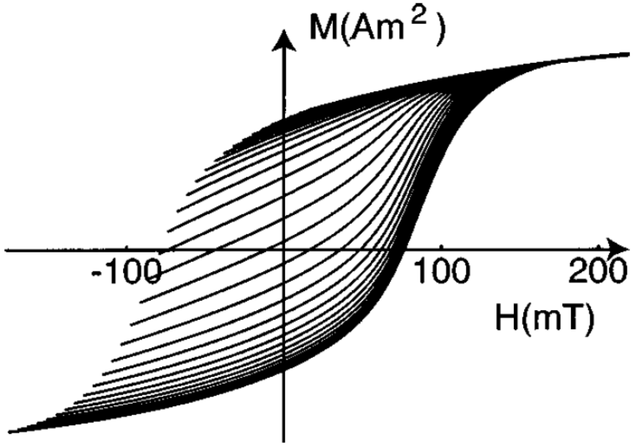
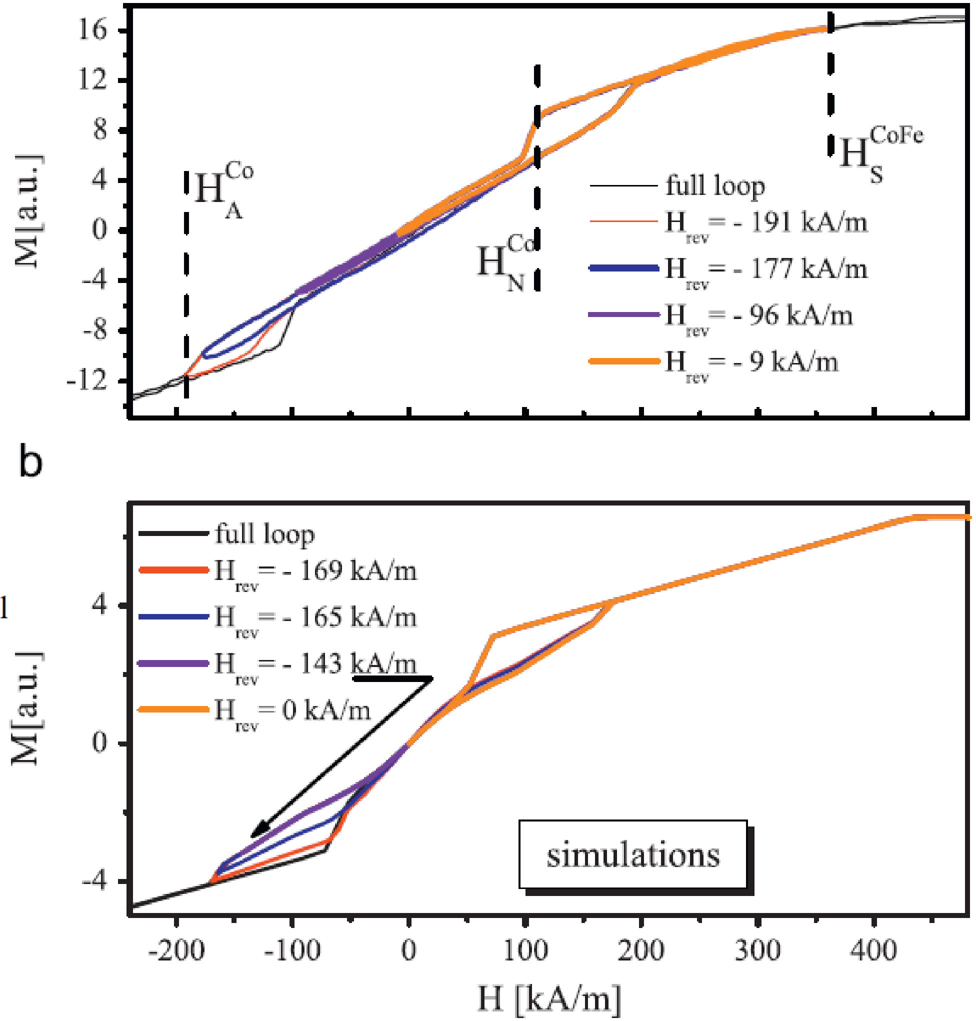


FIG. 1. A set of first order reversal curves (FORCs) for a piece of a typical floppy magnetic recording disk.

Christopher R. Pike, Andrew P. Roberts, and Kenneth L. Verosub



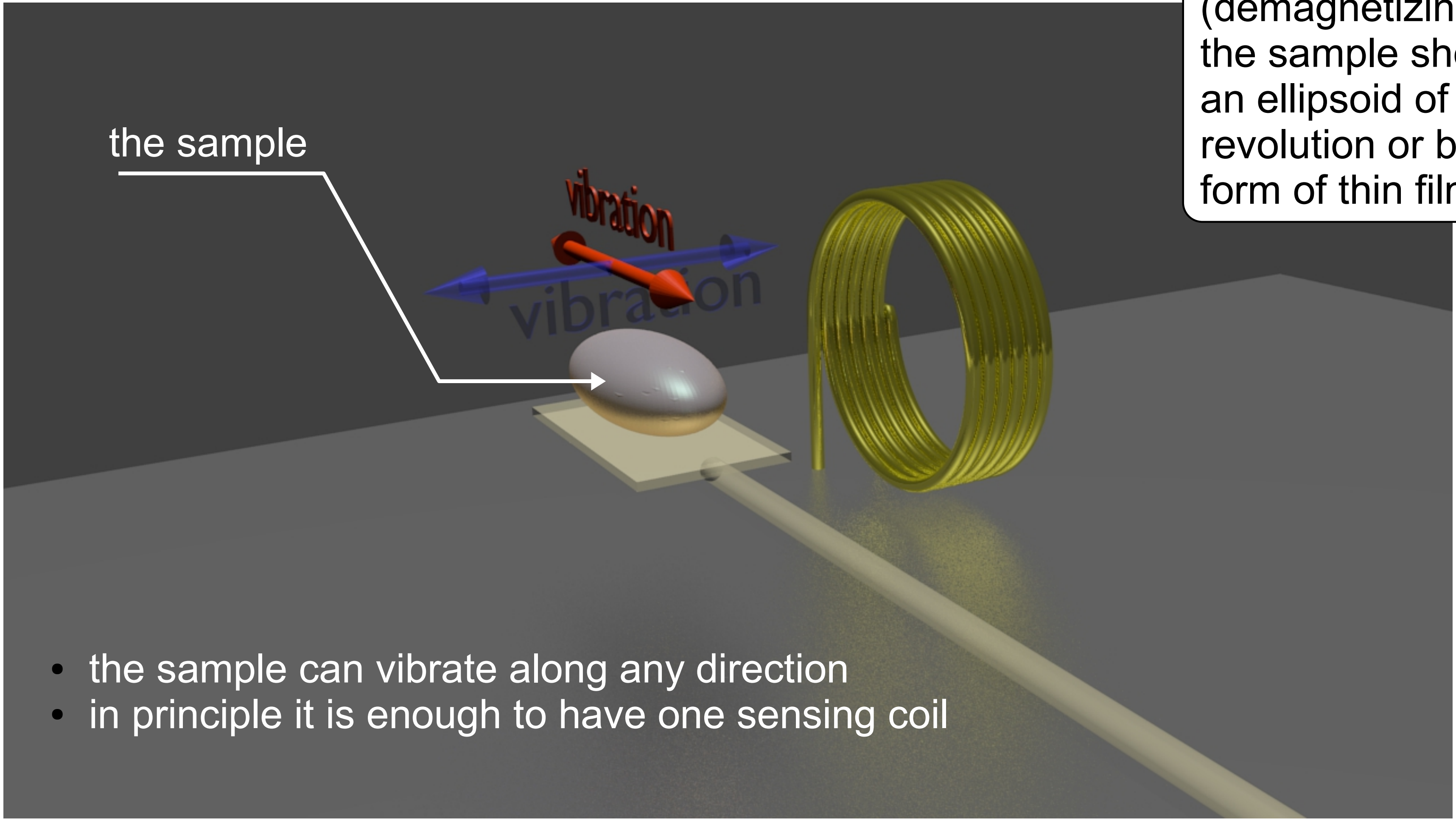
Major and minor hysteresis loops obtained for [CoFe(1.2nm)/ Au(1.2 nm)/Co(0.6nm)/Au(1.2nm)]$_{10}$ ML

Induction methods magnetometry

- In induction methods the Faraday induction is used to measure the magnitude of magnetic moment of the specimen [12].
- The method is based on the Maxwell equation:

$$\nabla\times\vec{E}=\frac{\partial}{\partial t}\vec{B}$$

For the reasons which will become clear later (demagnetizing field) the sample should be an ellipsoid of revolution or be in the form of thin film



- the sample can vibrate along any direction
- in principle it is enough to have one sensing coil

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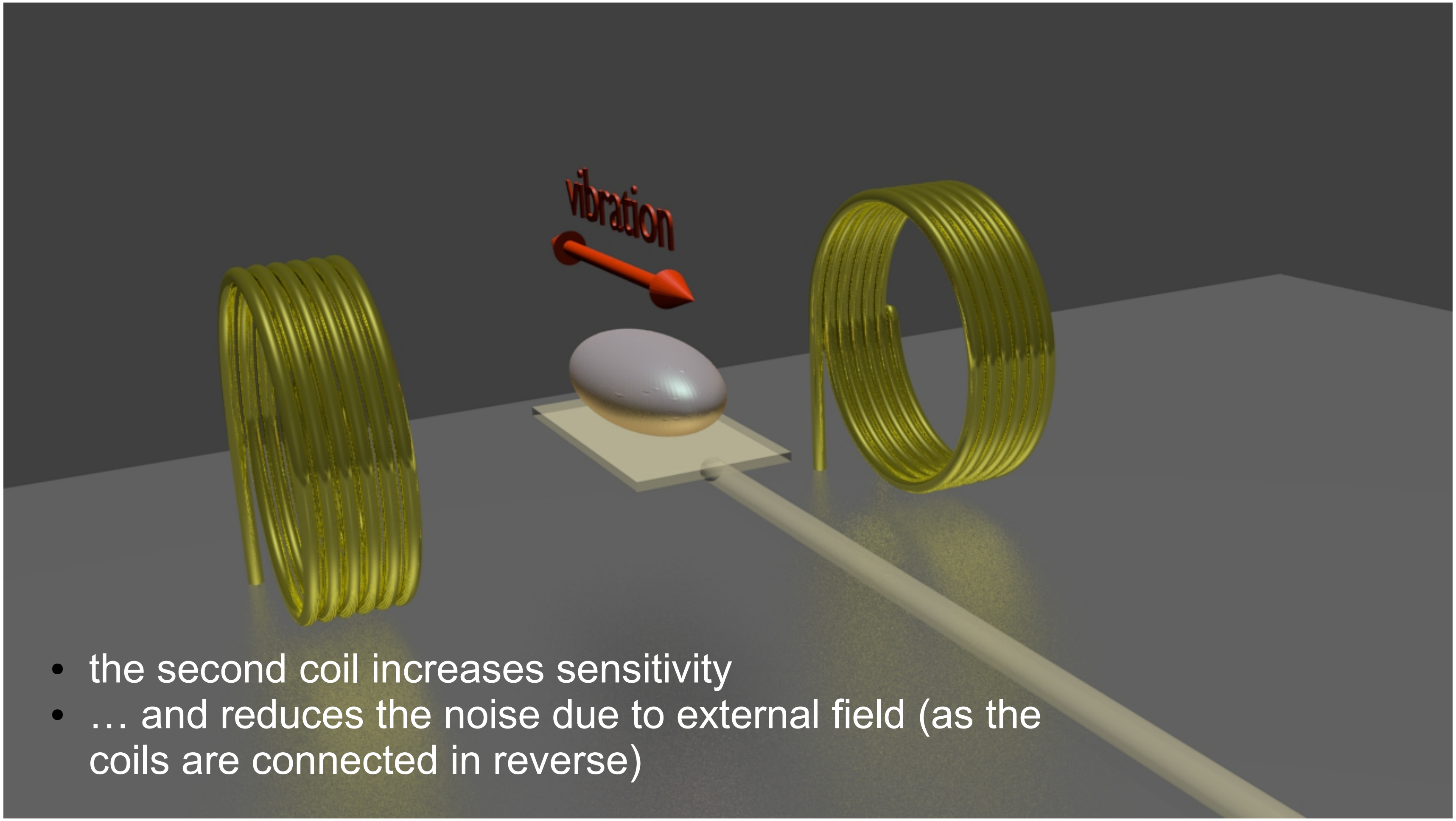

- the second coil increases sensitivity
- … and reduces the noise due to external field (as the coils are connected in reverse)

Induction methods magnetometry

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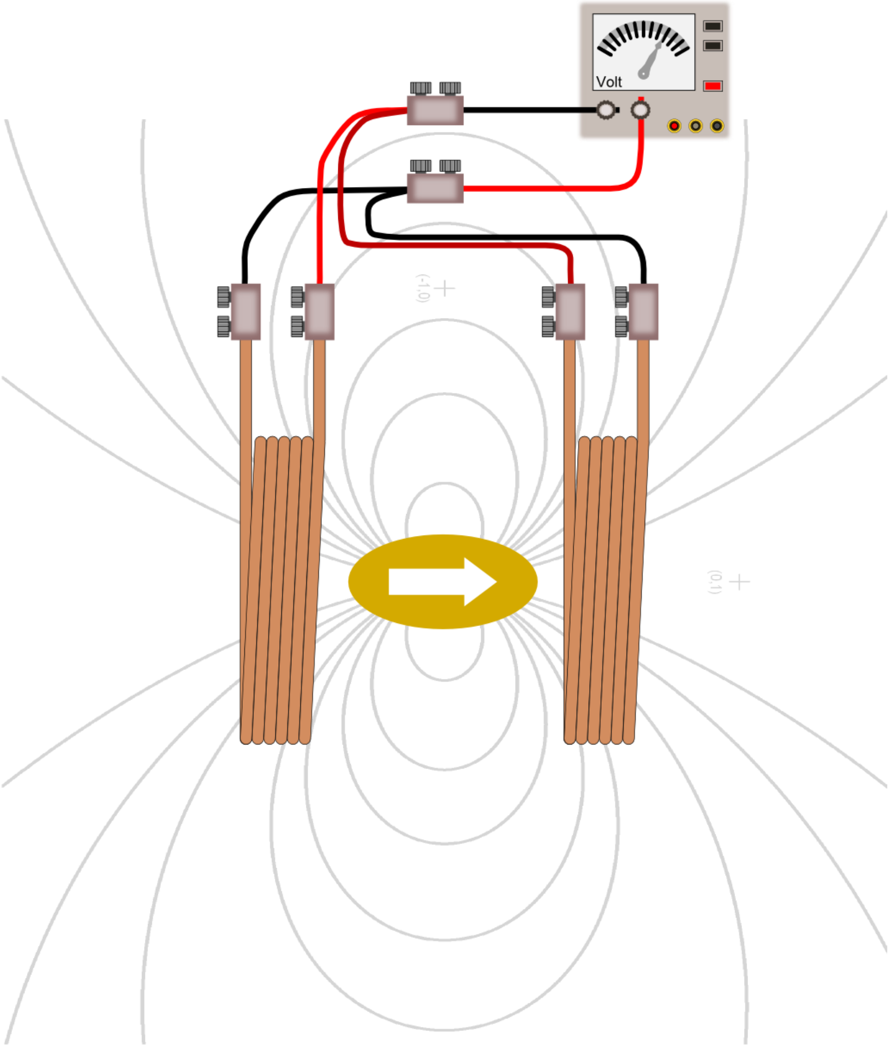
- the sample moving to the right induces some a voltage in the right pick-up coil (the current flow direction is determined by Lenz's law)
- the voltage induced in the left coil has an opposite sign
- if the coils are connected in reverse both voltages add increasing the sensitivity
- temporal variations of nearly homogeneous external field (from electromagnet or distant sources of EM – fields) cause the opposite voltages in the two coils and cancel out in ideal case.



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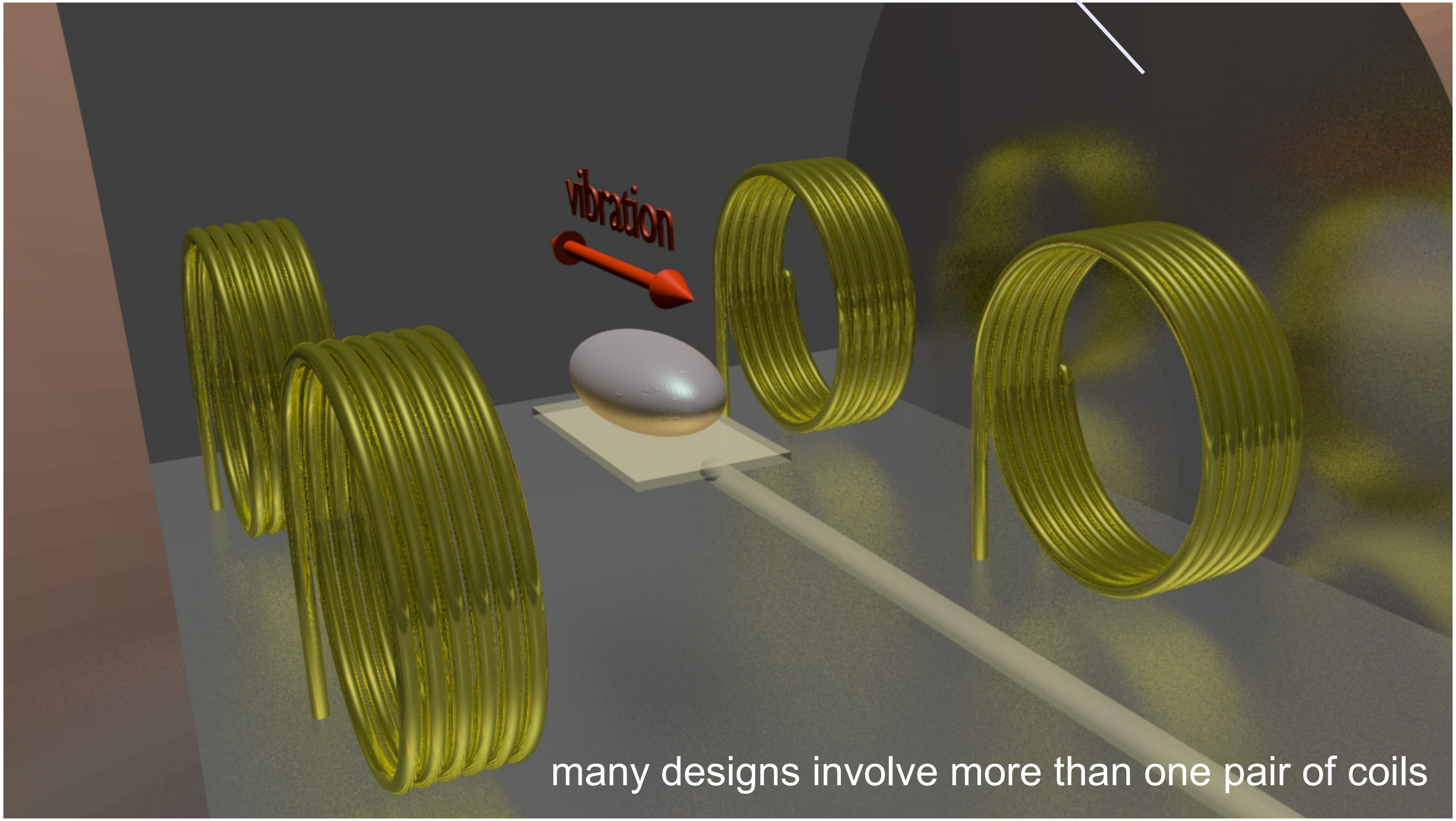


pole piece of an electromagnet

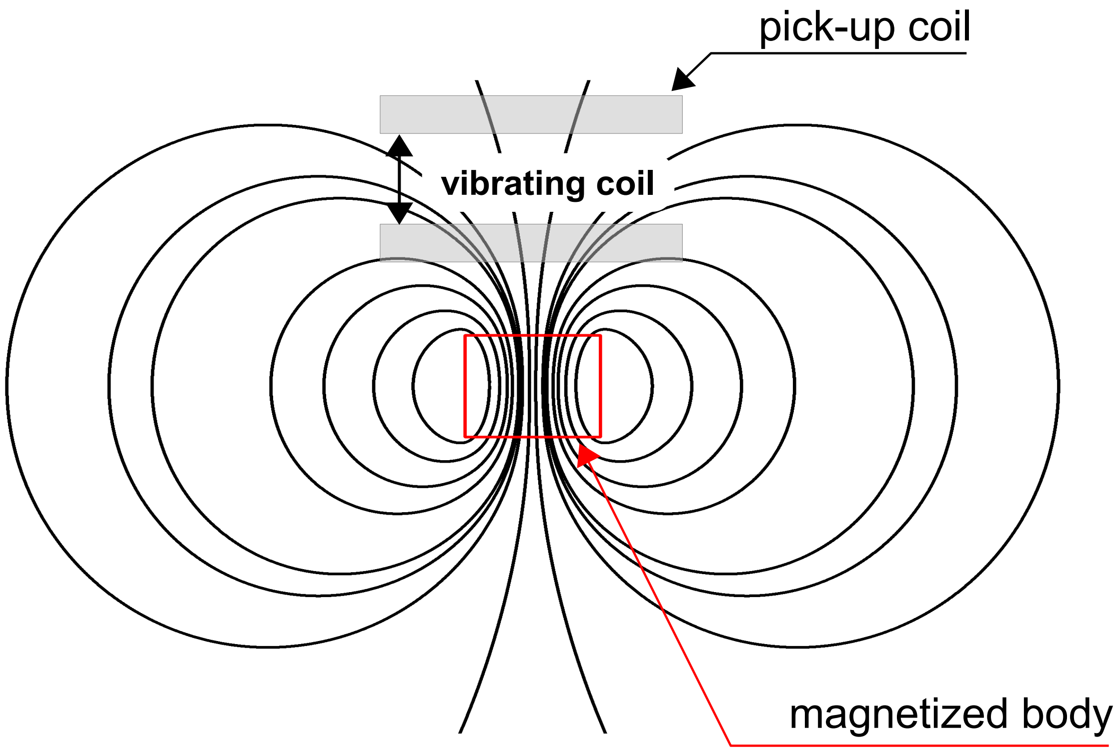
many designs involve more than one pair of coils

Induction methods magnetometry

- In induction methods the Faraday induction is used to measure the magnitude of magnetic moment of the specimen [12].
- The method is based on the Maxwell equation:

$$\nabla \times \vec{E} = \frac{\partial}{\partial t} \vec{B}$$

- The electromotive force generated in the pick-up coils is proportional to the magnetization of the sample; it depends too on the orientation of the magnetic moment relative to the coils:



- Depending on position of the coils the integral of the induction through the surface bounded by the coils changes; the voltage (or integral of ***E*** along the coil perimeter) depends on the rate of change of induction ***B***:

$$U = \oint \vec{E}\, dl = \iint \frac{\partial}{\partial t} \vec{B}\ dS$$

*field lines of the magnet of height 3 and width 4 which is infinite in the direction perpendicular to the image

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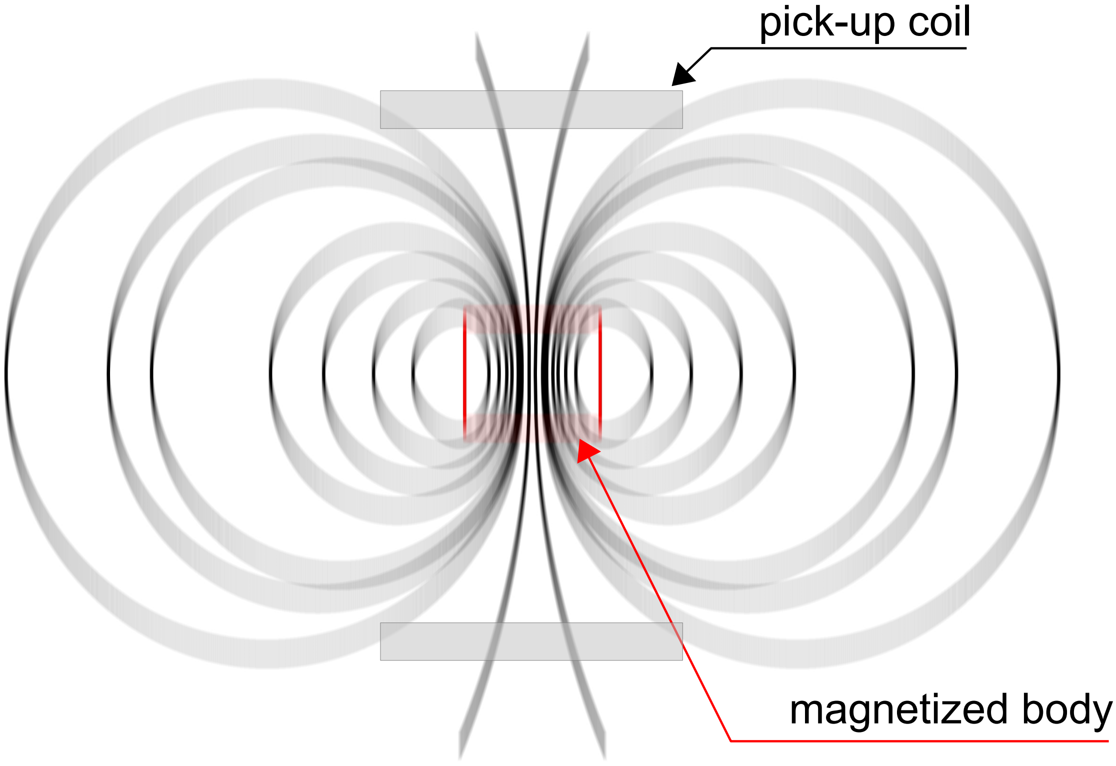


- In measurement one usually uses static pick-up coils while the sample (and its magnetic field) vibrates
- To minimize the influence of the external sources of magnetic field pairs of coils ares used: the variations of the external field add to the signal in one coil and subtract from the signal of the other coil.

*field lines of the magnet of height 3 and width 4 which is infinite in the direction perpendicular to the image

Vibrating sample magnetometer

- **VSM** is a device used to measure magnetic moment and hysteresis
- It uses the electromagnetic induction and lock-in principle of measurement [13]

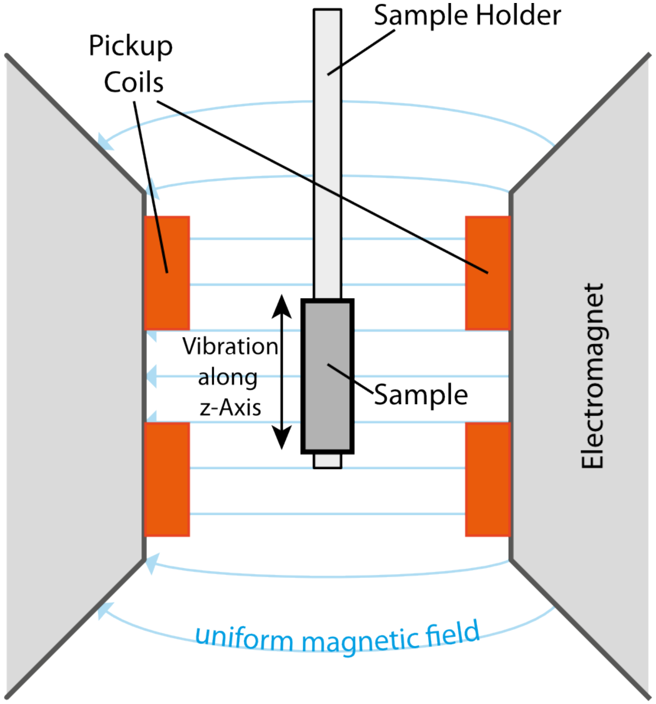
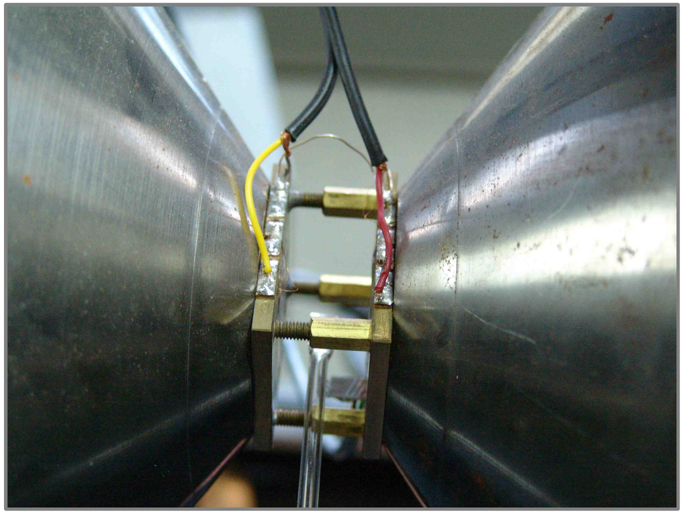


Image source - Wikimedia Commons; author: R0oland



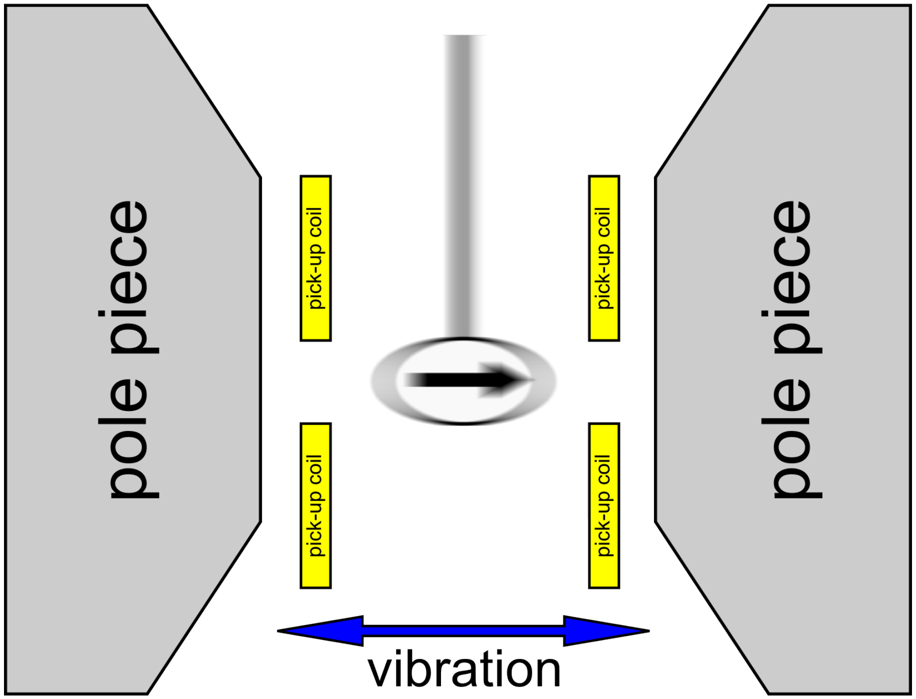
“nonmagnetic” sample holder

- Commercially available VSM magnetometers have sensitivity below 10^{-9} Am^{-2} (depending on acquisition time)

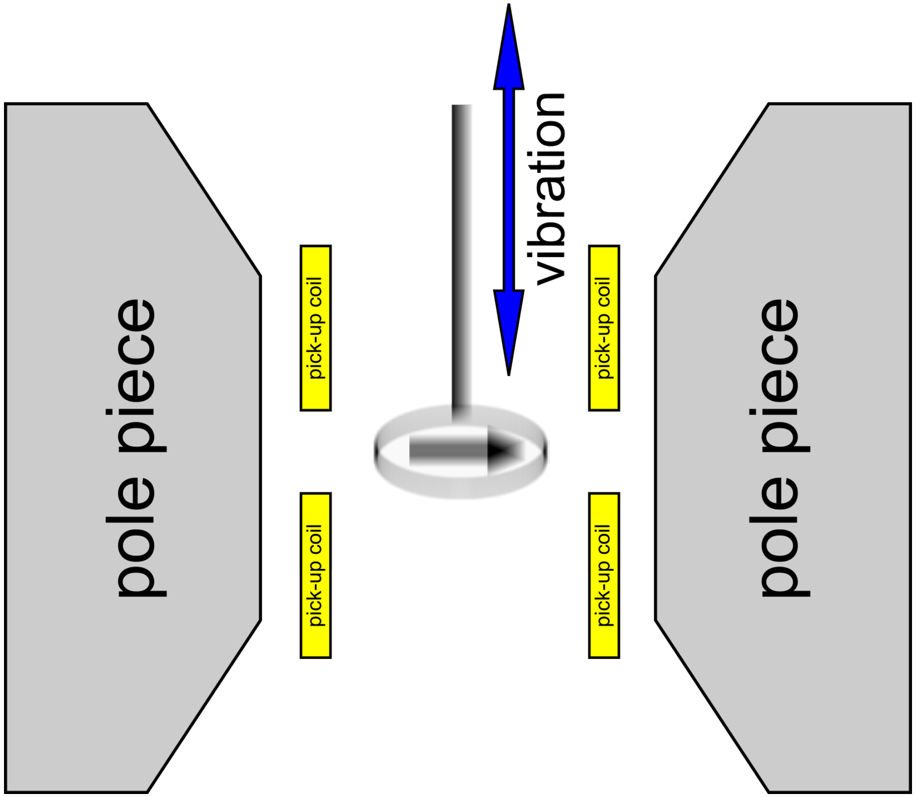
VSM (properly calibrated) measures the absolute value of magnetic moment of the sample

Vibrating sample magnetometer

- VSM can be used in different configurations of sample vibration relative to the external field direction:



parallel configuration
(vibration parallel to the field)



transverse configuration

A. Zieba and S. Foner, Rev. Sci. Instrum. 53, 1344 (1982)

Vibrating sample magnetometer – principle of operation

Lock-in principle of measurement allows the measurement of signals weaker then the noise. We start from the trigonometric identity:

$$\cos(\alpha)\cos(\beta)=\frac{1}{2}\cos(\alpha-\beta)+\frac{1}{2}\cos(\alpha+\beta)$$

- We assume that the sample vibrates with frequency ω giving the signal of that frequency and amplitude A in pick-up coils (through magnetic induction)
- We mix* (multiply) that signal with the signal of frequency ω_1 taken from the generator that drives the sample (we can have some phase difference φ). Using the above identity we have:

$$A\cos(\omega t)B\cos(\omega_1 t+\phi)=\frac{1}{2}AB\cos(\omega t-\omega_1 t-\phi)+\frac{1}{2}AB\cos(\omega t+\omega_1 t+\phi)$$

But $\omega_1=\omega$ (the same generator) so we obtain:

$$A\cos(\omega t)B\cos(\omega_1 t+\phi)=\underbrace{\frac{1}{2}AB\cos(\phi)}_{\text{constant in time}}+\underbrace{\frac{1}{2}AB\cos(2\omega t+\phi)}_{\text{fast varying component}}$$

Using **low pass-filter** we can filter out the varying component.
There remains only constant voltage which is proportional to the signal from the sample and which is maximum if the phase difference is a multiple of π:

$$\frac{1}{2}AB\cos(\phi)$$

*mixing can also mean adding signals - additive mixers in audio electronics

Vibrating sample magnetometer – principle of operation

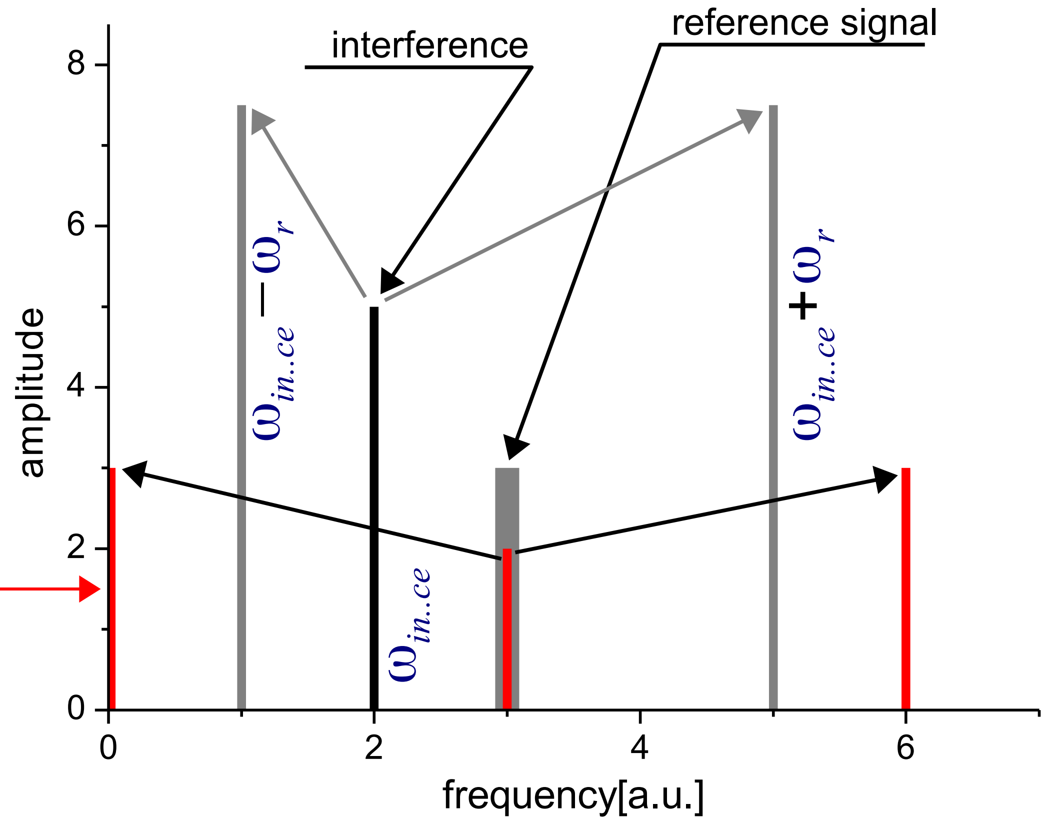
The signal from the pick-up coils can be interfered by external sources of electromagnetic radiation (50 Hz and its harmonics from power lines, car ignition circuits etc.). The signal can be expressed now as:

$$S_{coil}=\sum_i A_i\cos\left(\omega_i t+\phi_i\right)$$

Multiplying again by the reference signal (from generator) we get:

$$S_{coil}B\cos\left(\omega_r t+\phi\right)=\sum_i\left(\frac{1}{2}A_iB\cos\left(\left(\omega_i-\omega_r\right)t-\phi_i\right)+\frac{1}{2}A_iB\cos\left(\left(\omega_i+\omega_r\right)t+\phi_i\right)\right)$$

Only those signals which have a frequency equal to the reference frequency contribute to constant voltage received from mixer



constant signal from the sample

Vibrating sample magnetometer – the sensitivity function

- Sensitivity function ***G***(***r***) represents the spatial distribution of detection coil sensitivity – the dependence of VSM signal on sample position. The function G is calculated for given direction of sample motion and given set of detection coils.
- For the moment moving with velocity v(t) the signal induced in coils is:

$$U(t)=\mu G(\vec{r})v(t)$$

- To obtain time dependence of the signal the above expression must be integrated over the volume of the sample for given amplitude and frequency of oscillations.

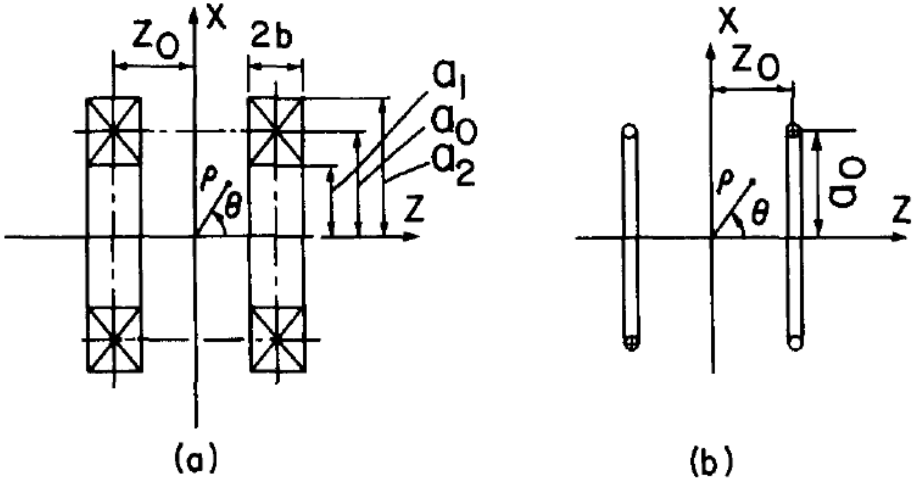


FIG. 1. Axial detection coils: (a) thick rectangular cross-section coils, (b) thin coils. The spherical coordinates ρ and θ give the position of vibrating dipole.

A. Zieba and S. Foner, Rev. Sci. Instrum. 53, 1344 (1982)

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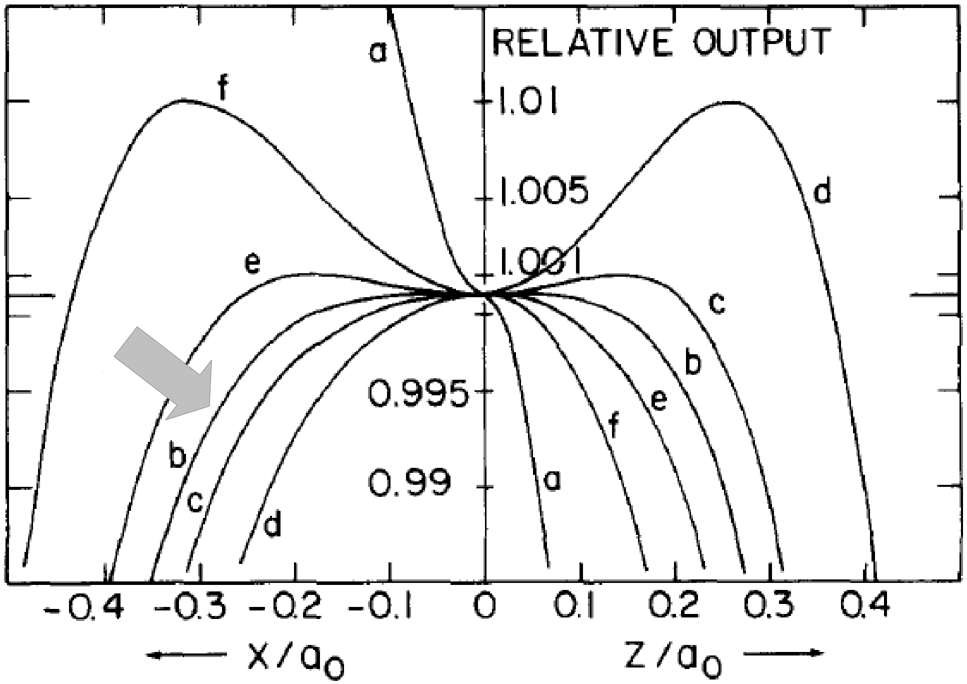


FIG. 3. Relative sensitivity function vs displacement of small sample along the z axis (right-hand side of the figure), and along the x axis (in the perpendicular symmetry plane, left-hand side of the figure) for thin-coil pairs with intercoil distances z_0/a_0 equal to: (a) ½; (b) $\sqrt{3}/2 \cong 0.8660$; (c) 0.8841; (d) 0.9244; (e) 0.8444; (f) 0.7992. The curves (c) and (d) correspond to coils with elongated homogeneity along the z axis, and curves (e) and (f) have elongated homogeneity in the perpendicular plane. Note that the elongated homogeneity corresponds to overcompensation of 0.1% for (c) and (e) and 1% for (d) and (f), respectively.

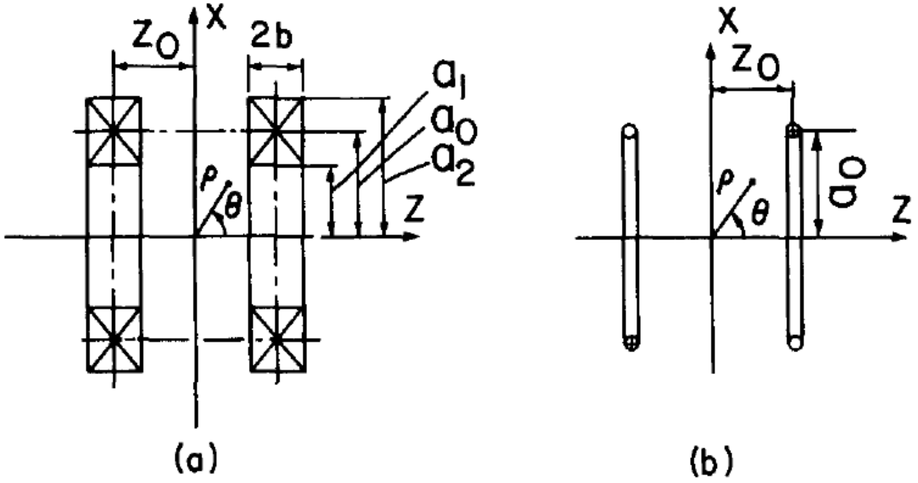


FIG. 1. Axial detection coils: (a) thick rectangular cross-section coils, (b) thin coils. The spherical coordinates ρ and θ give the position of vibrating dipole.

- For *thin coils* spaced 0.866..times their diameter apart the sensitivity function is not maximal but is very flat at center of the coils system – the signal does not depend much on the dipole position.

A. Zieba and S. Foner, Rev. Sci. Instrum. 53, 1344 (1982)

Minor hysteresis loops

- There are several common coils configurations each of which is characterized by different sensitivity function.

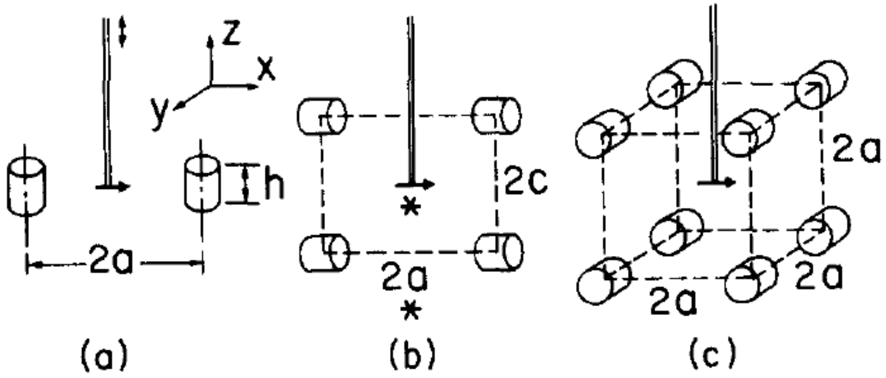


FIG. 5. Three principal transverse detection coil configurations for a VSM with sample vibration perpendicular to the direction of the dipole moment and exhibiting the saddle point at the symmetry center. The axes of the coils are directed along the *z*, *x*, and *y* axes, respectively. The asterisks in Fig. 5(b) indicate the position of the accidental saddle points for that geometry when the two upper coils are removed.

- Depending on the shape of the sample one usually need corrections factors to obtain the signal independent of the shape.

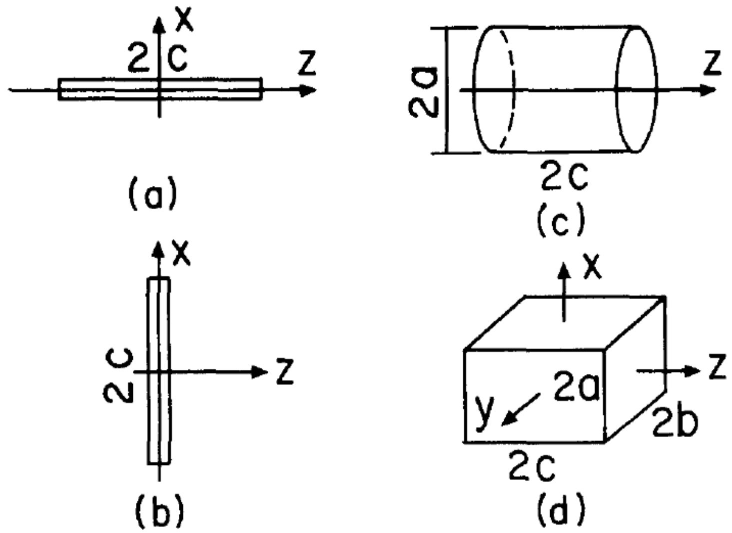
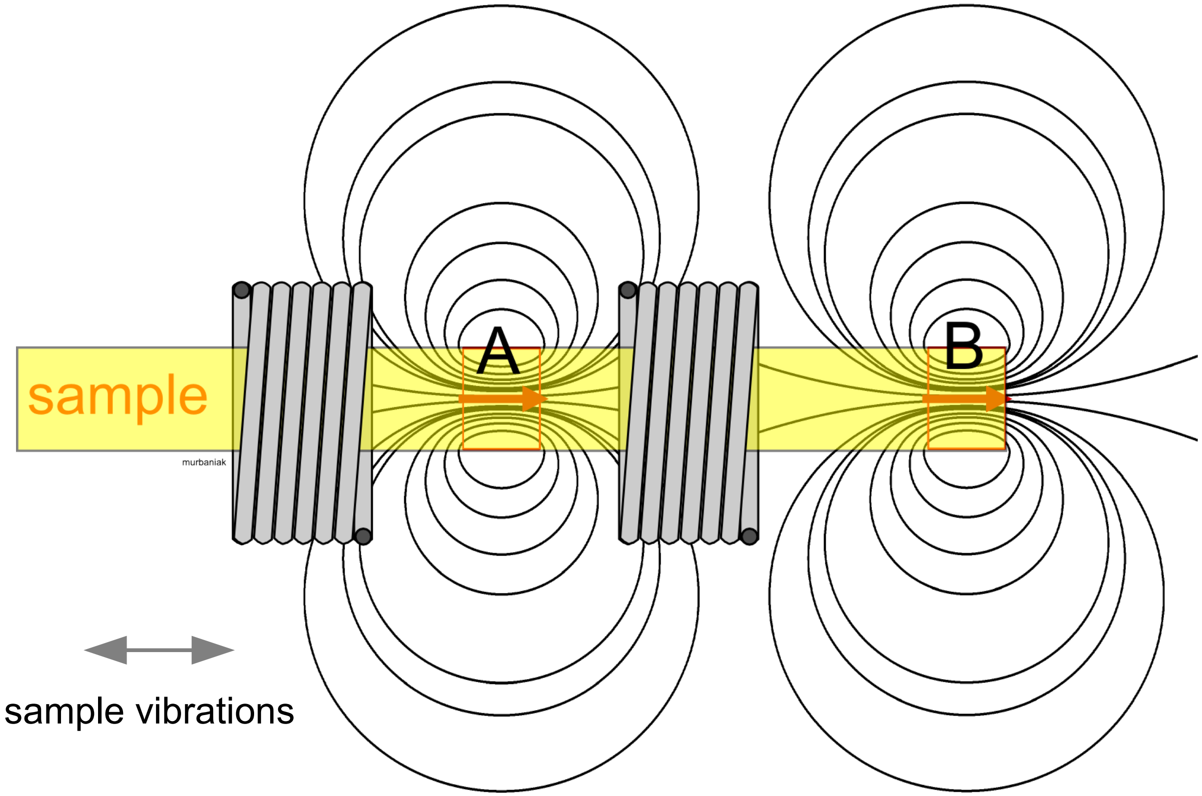


FIG. 7. Examples of regular sample shapes: (a) thin rod (arbitrary cross section) parallel to the *z* axis; (b) thin rod perpendicular to the *z* axis; (c) cylinder; (d) rectangular parallelepiped.

A. Zieba and S. Foner, Rev. Sci. Instrum. 53, 1344 (1982)

Vibrating sample magnetometer – sample size

- The ideal measurement of the M(H) dependence is performed using ellipsoidal (or spherical) samples; in that case the sample can be represented by the point dipole.
- It is often desirable to maintain the integrity of the sample for further measurements so the size of the sample cannot be made small compared to detection coils.



- Parts **A** and **B** of the sample (yellowish bar) contribute oppositely to the signal in the right coil
- For infinitely long sample the signal would be zero
- The sample size should be possibly small (which increases the signal to noise ratio)

Vibrating sample magnetometer – sample size

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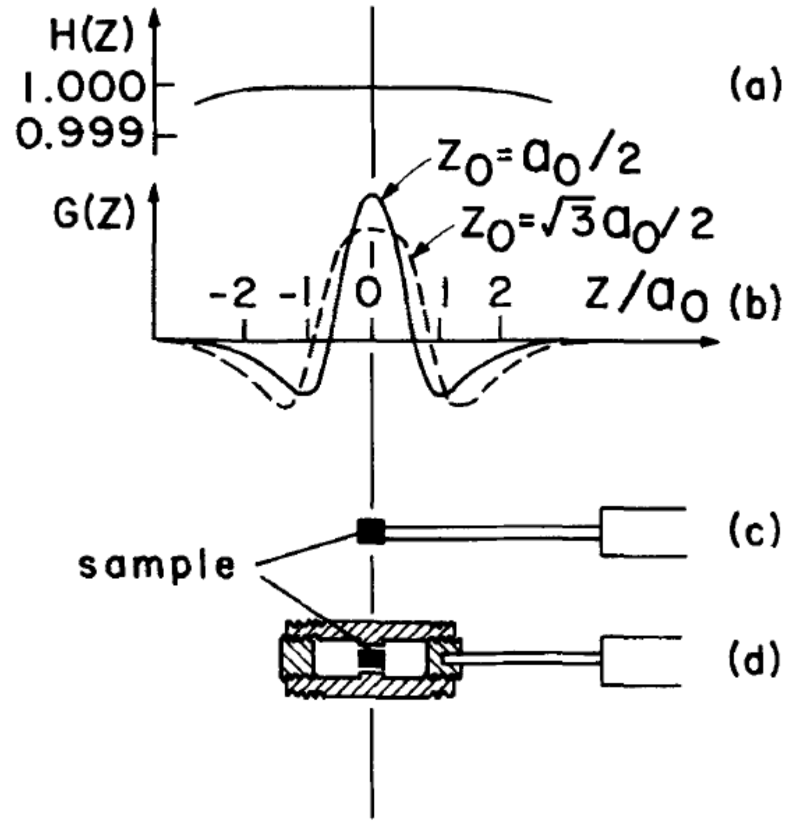


FIG. 11. Sketch comparing relative spatial distribution of: (a) relative external field $B(z)$; (b) sensitivity function $G(z)$ for $z_0 = a_0/2$ and $z_0 = a_0\sqrt{3}/2$; (c) sample support rod; (d) pressure clamp and support rod.

- For typical two coils configurations the material beyond about $z/a_0=1$ produces an output of opposite sign to that of $z/a_0<1$*.
- Large support rod structures may interfere with the signal of the sample – the symmetric arrangement of holders will reduce/cancel that effect

*$2a_0$ – distance between coils

A. Zieba and S. Foner, Rev. Sci. Instrum. 53, 1344 (1982)

Vibrating sample magnetometer

- VSM is a standard method of measuring hysteresis of thin magnetic films (other popular method is a Kerr effect magnetometry)
- The VSM may use permanent magnets configurations like Halbach cylinders (see L.2) instead of electromagnets.

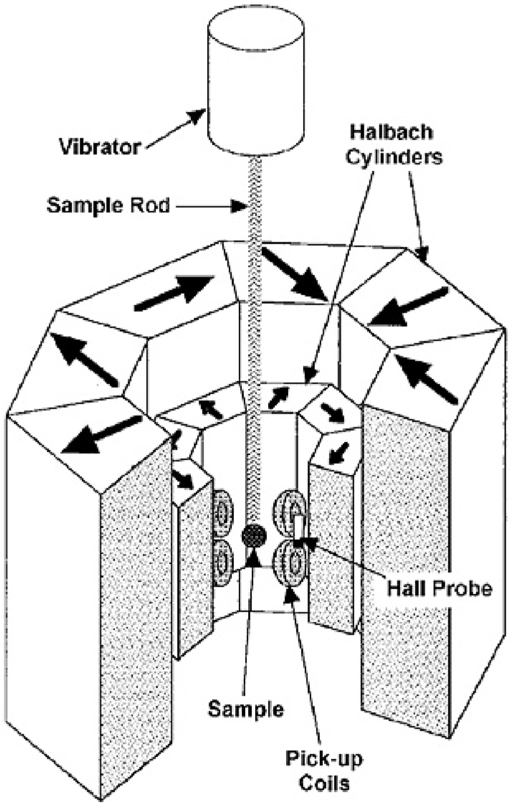


image source: http://www.tcd.ie/Physics/Magnetism/Research/instrumentation.php

- There is an interesting development of VSM called an alternating gradient magnetometer (**AGM**):

-the sample is placed in the static magnetic field which is locally modified by a small varying field of current coils

-this field creates field gradient which exerts a sinusoidally varying force on the sample

-the displacement of the sample is sensed by the piezoelement which is a part of the sample holder

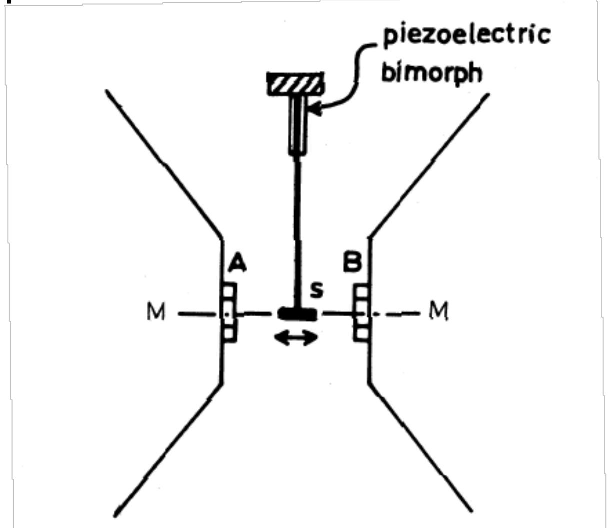


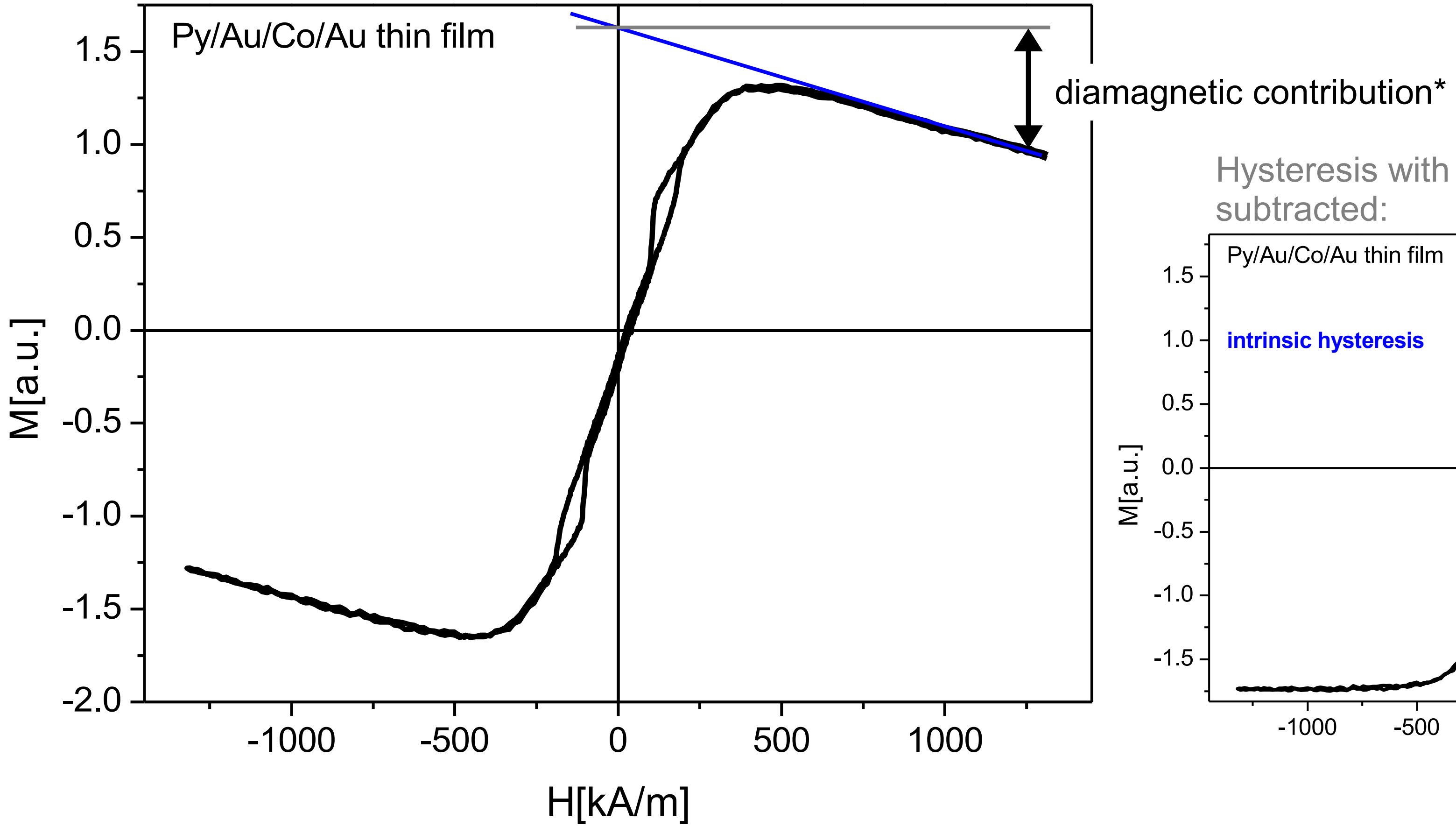
Fig.2 Simple AGM. M, M=electromagnet pole pieces; A, B=gradient field coils, connected series opposing; S=sample

-AGM is not necessarily suitable for soft magnetic materials

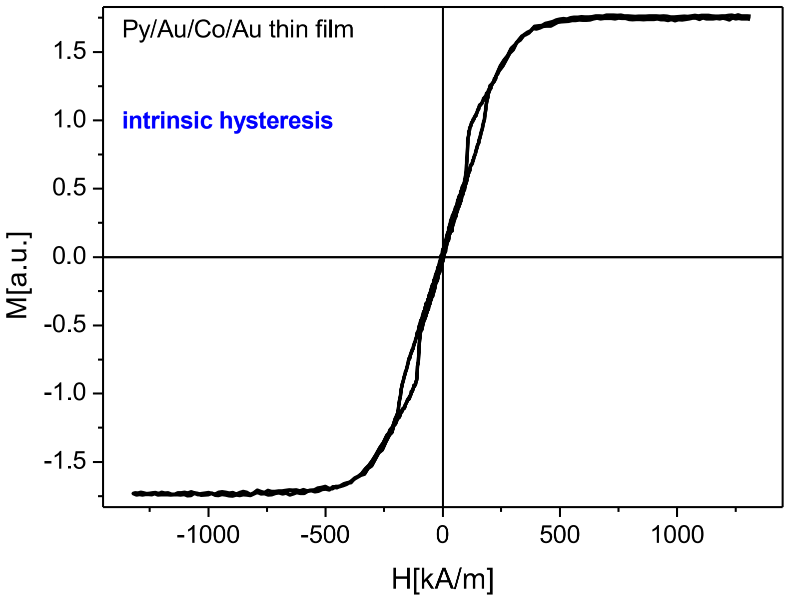
[7] C.D. Graham, J. Mater. Sci. Technol. **16**, 100 (2000)

Vibrating sample magnetometer

- The shape of the sample influences the measurement of hysteresis with VSM (demagnetizing field) – intrinsic field differs from the applied field; the effect of demagnetizing field can be properly taken into account in ellipsoidal samples (see L2) or in their limiting cases (elongated rod, thin film)
- With VSM measurements of small volume samples (like thin films) it is often necessary to subtract the diamagnetic contribution from the sample holder



Hysteresis with diamagnetic contribution subtracted:



Vibrating sample magnetometer with SQUID

- The superconducting quantum interference device (SQUID) can be used as a flux to voltage converter in the VSM magnetometer.

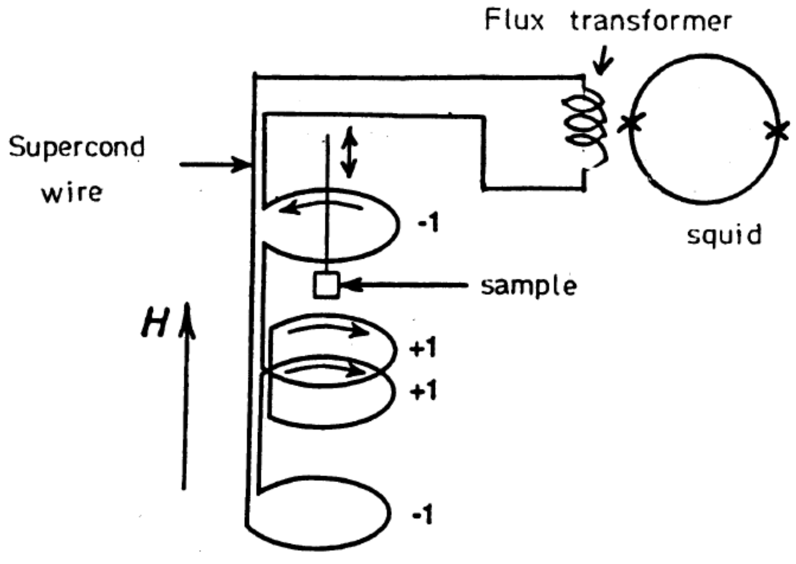


Fig.3 Diagram of SQUID magnetometer

Image from: [7] C.D. Graham, J. Mater. Sci. Technol. **16**, 100 (2000)

- The sample vibrates between pick-up coils placed in an external magnetic field
- The coils are connected so as to insure that the change in the applied field produces no net flux in the coils (second order gradiometer [7])
- The coils are *coupled inductively to SQUID* element which converts flux changes caused by the movement of the sample to voltage
- The voltage is measured with lock-in principle
- The sensitivity of commercial VSMs wit SQUID can exceed 10^{-11}Am^2*.
- Resolution is field dependent (noise from field source)

It corresponds roughly to
0.1mm×0.1mm×1nm piece of iron

*MPMS SQUID VSM from **Quantum Design**

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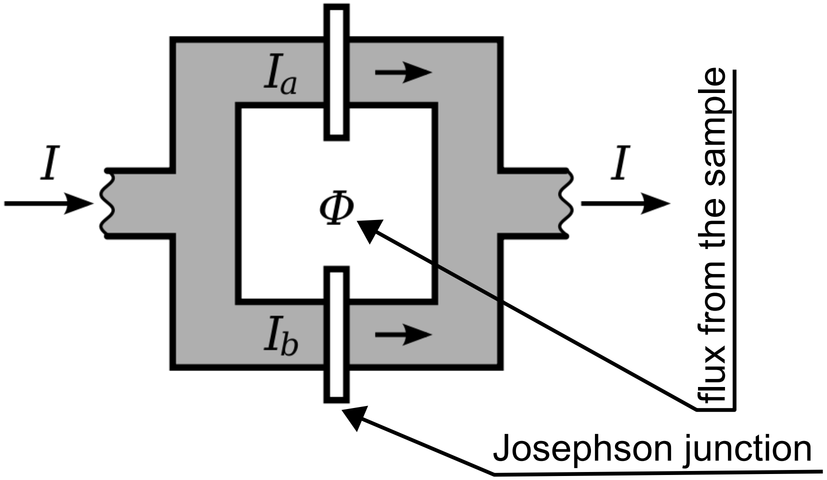


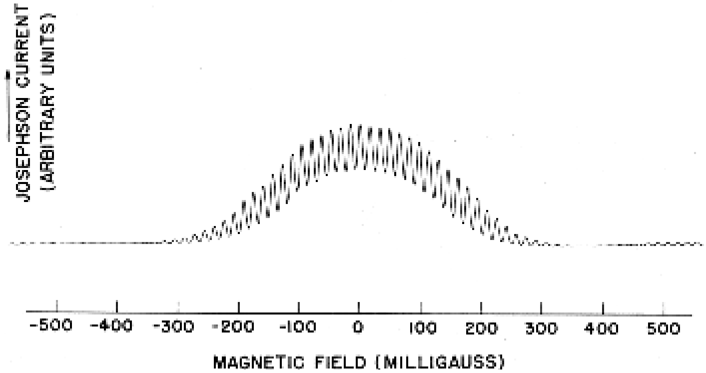
- Magnetic flux passing through a superconducting current circuit is quantized:

$$\phi_0 = \frac{h}{2e} \approx 2.067 \times 10^{-15} T\, m^2$$

- The total current going through the junction can be shown to be [8]:

$$J_{tot} = J_0 \cos\left(\frac{2\pi e}{h}\phi\right)$$

- The current through the junction oscillates as the function of the flux through the superconducting coil. Since the junctions have a resistance we can measure the voltage drop across the device:



Counting oscillations one can determine the flux through the Josephson device

[8] SQUIDs: A Technical Report, http://rich.phekda.org/squid/technical/index.html#toc

Image source - Wikimedia Commons; author: Miraceti

Vibrating sample magnetometer with SQUID

- The magnetometers with SQUID are the most sensitive devices of this kind. They can measure field as low as 10^{-14} T* which is *less than fields associated with human brain activities*.
- The resolution of the device can be much better than the magnetic flux quantum.
- The sensitivity of the device allow the measurement of hysteresis loops of single particles:

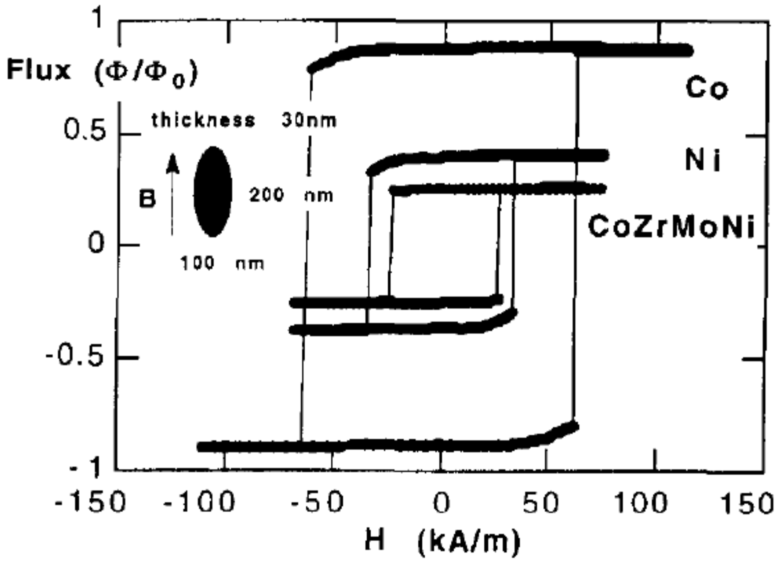


Fig. 3. Hysteresis loops of type A – Co, Ni and CoZrMoNi particles, ellipticity 200×100 nm, thickness 30 nm, $T = 0.2$ K.

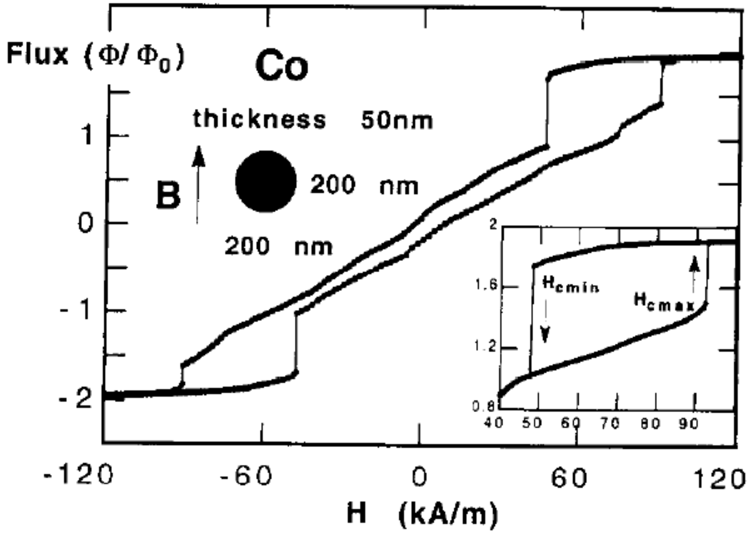


Fig. 6. Hysteresis loop of type C – Co particles, ellipticity 200×200 nm, thickness 50 nm, $T = 0.2$ K. The inset shows a minor loop of this particle.

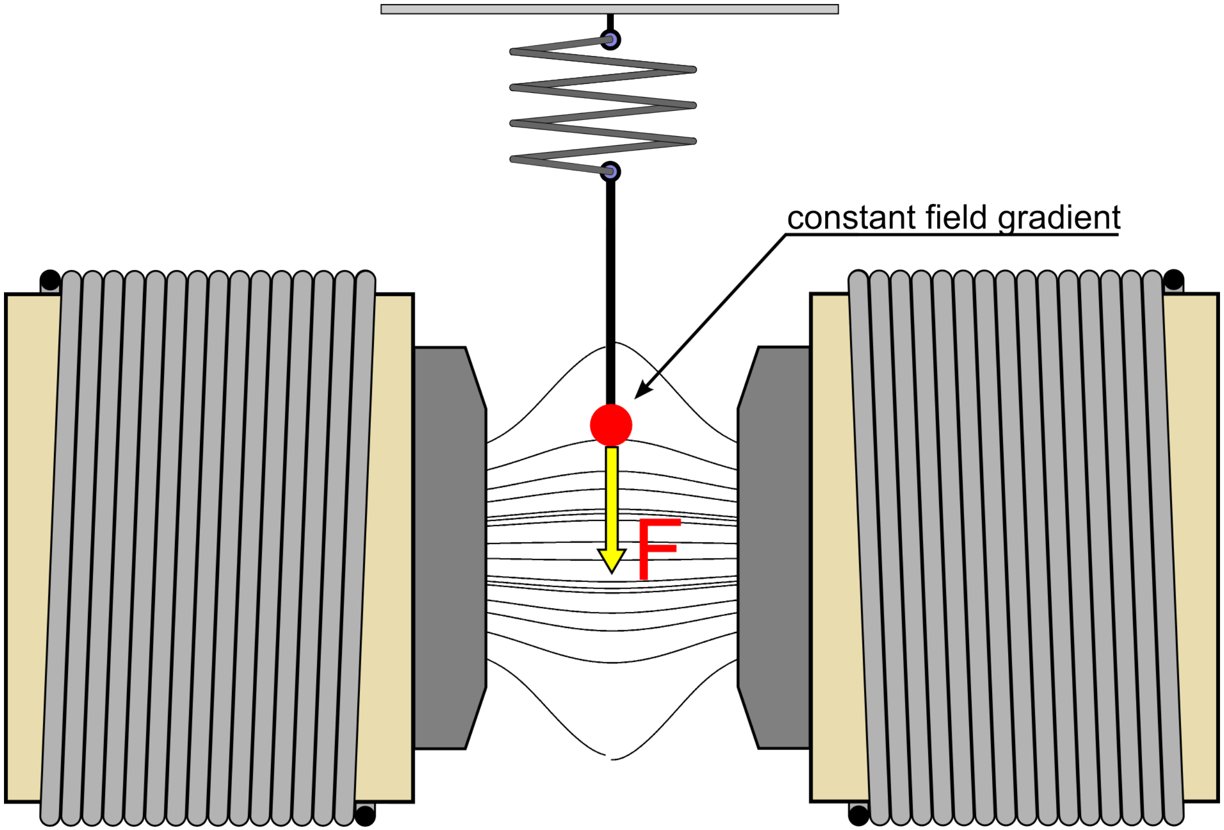
Resolution of $10^{-4}\Phi_0$ – behavior of about 10^6 spins.

W. Wernsdorfer et al., Journal of Magnetism and Magnetic Materials 145 (1995) 33-39

*http://hyperphysics.phy-astr.gsu.edu/hbase/solids/squid.html#c2

Magnetic scales

- The magnetic moment and susceptibility can be measured with magnetic scales [10].
- They utilize the force exerted by a magnetic field with a gradient (see L.2) on magnetized body.
- The **Faraday method** utilizes the force on a small sample placed in virtually constant field gradient:

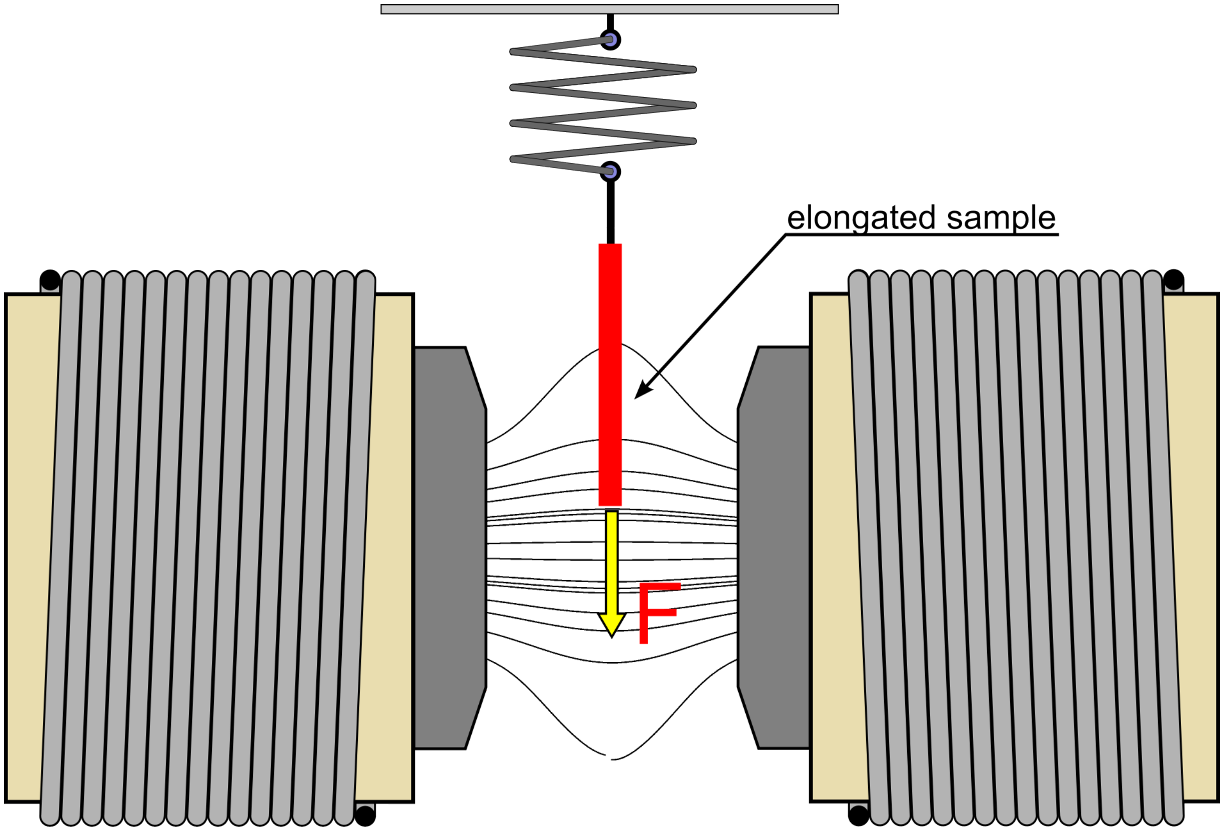


The force on magnetic particle is (L.2) (*V* - volume):

$$\vec{F}=\frac{1}{2\mu_0}\chi V\nabla(B^2)$$

Magnetic scales

- The **Gouy method** utilizes the force on a long bar sample with one end placed in electromagnet and the other one outside, in very small field.
- The method is used mainly for diamagnetic and paramagnetic substances.



The force on magnetic particle is (L.2) (*V* - volume):

$$\vec{F}=\frac{1}{2\mu_0}\chi V\nabla\left(B^2\right)$$

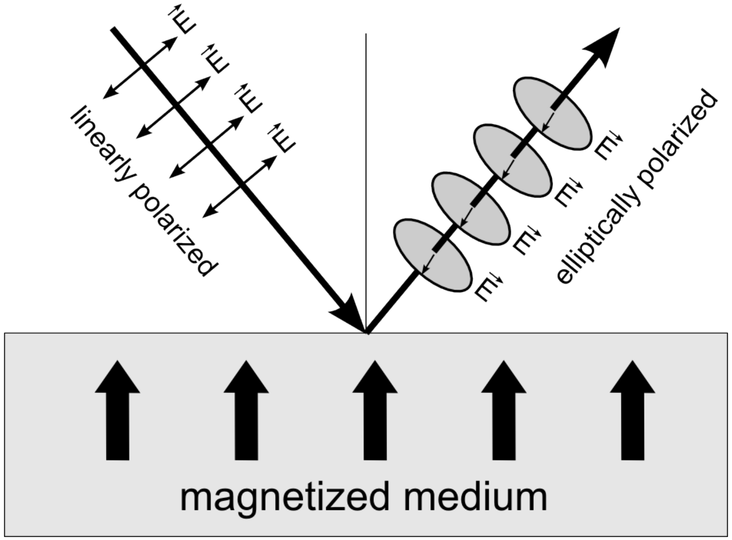
Integrating this along the length of the sample we get [10]:

$$\vec{F}=\frac{1}{2}\chi V\left(H_{max}^2-H_{min}^2\right)\approx$$

$$\frac{1}{2}\chi V H_{max}^2$$

Kerr effect magnetometer

Kerr effect – change of polarization of light reflected from the surface of magnetized material*



- Using polarizer for incident light and analyzer for reflected light one obtains voltage which is, for small angles of rotation *proportional to the magnetization*:

$$I = I_0 \cos^2(\theta) \qquad \text{Malus' law}$$

$$\cos^2(\theta) = 1 - \theta^2 + \ldots$$

- The effect can be used to measure magnetic hysteresis of thin films (or near surface layers of bulk materials).
- The penetration depth is determined by skin depth for a given radiation frequency** and resistivity of the material.
- The Kerr magnetometers can be extremely sensitive (1.2×10^{-18} Am^2 **!** [9])

$$^{**}\delta = \sqrt{\frac{2\rho}{2\pi\mu_0\mu_r f}}$$

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• POV-Ray	www.povray.org
• Blender	www.blender.org
• SketchUp	sketchup.com.pl

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