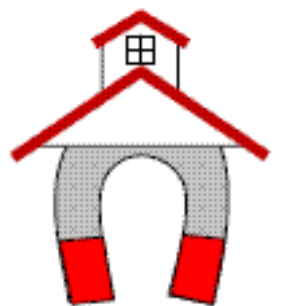


Magnetic Measurements

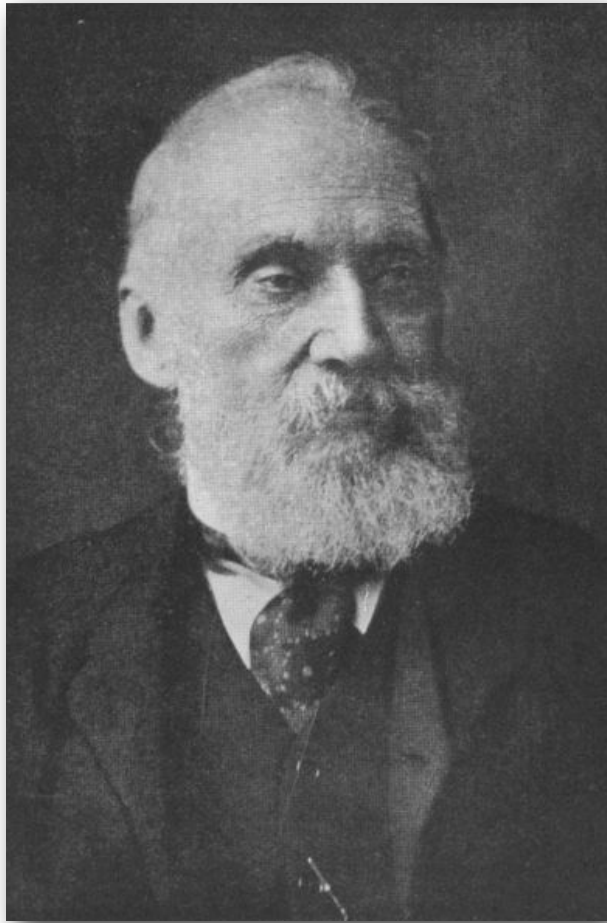
R. Schäfer,

Leibniz Institute for Solid State and Materials
Research (IFW) Dresden, Germany

IEEE Magnetics Summer School 2014, Rio



Why Magnetic Measurements?



"I often say that when you can measure what you are speaking about and express it in numbers you know something about it. But when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meager and unsatisfactory kind."

Lord Kelvin, 1883



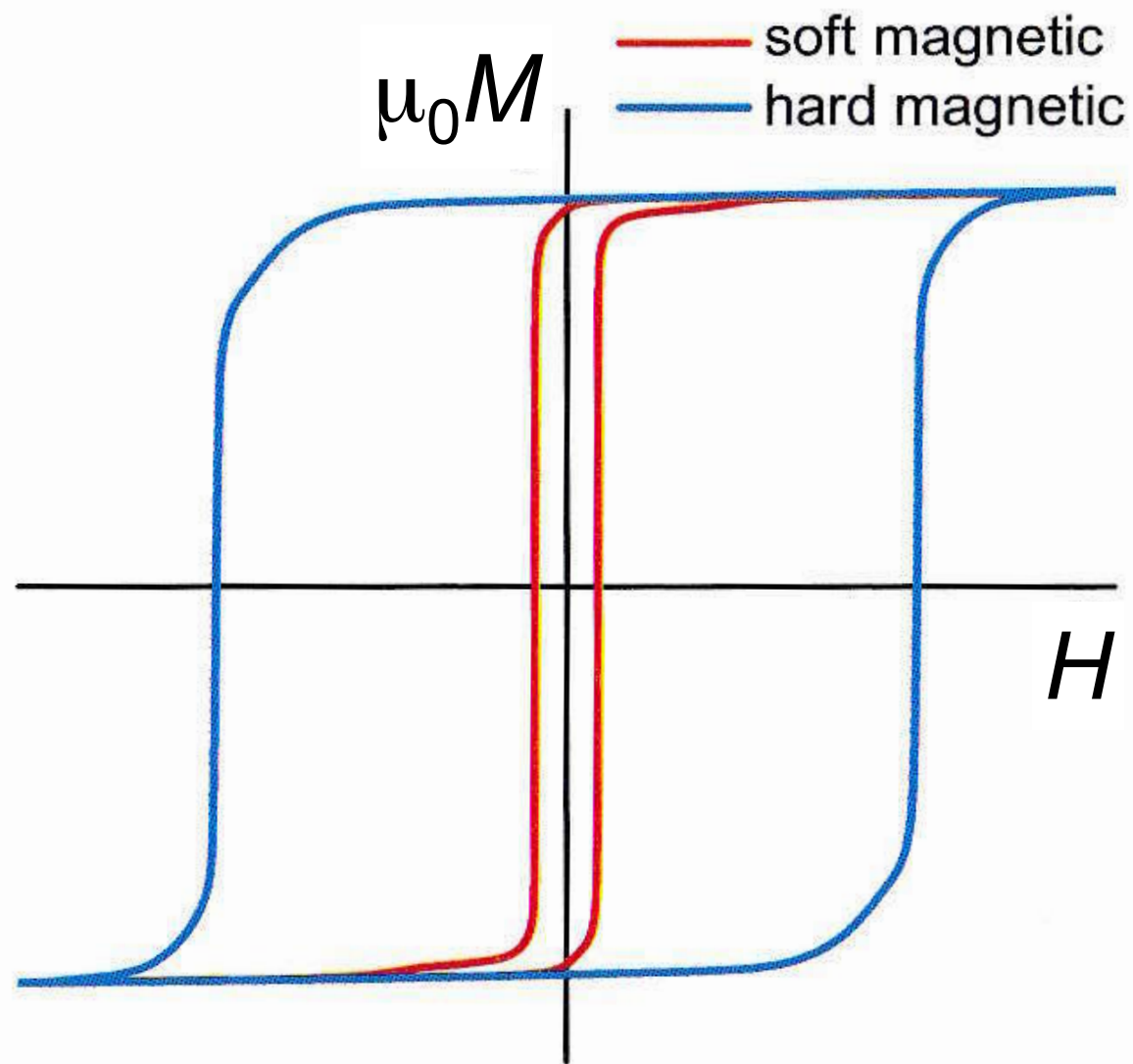
"Magnetism is an experimental science"

Mike Coey, 2010

"No clear understanding of magnetism can be attained without a sound knowledge of the way in which magnetic properties are measured."

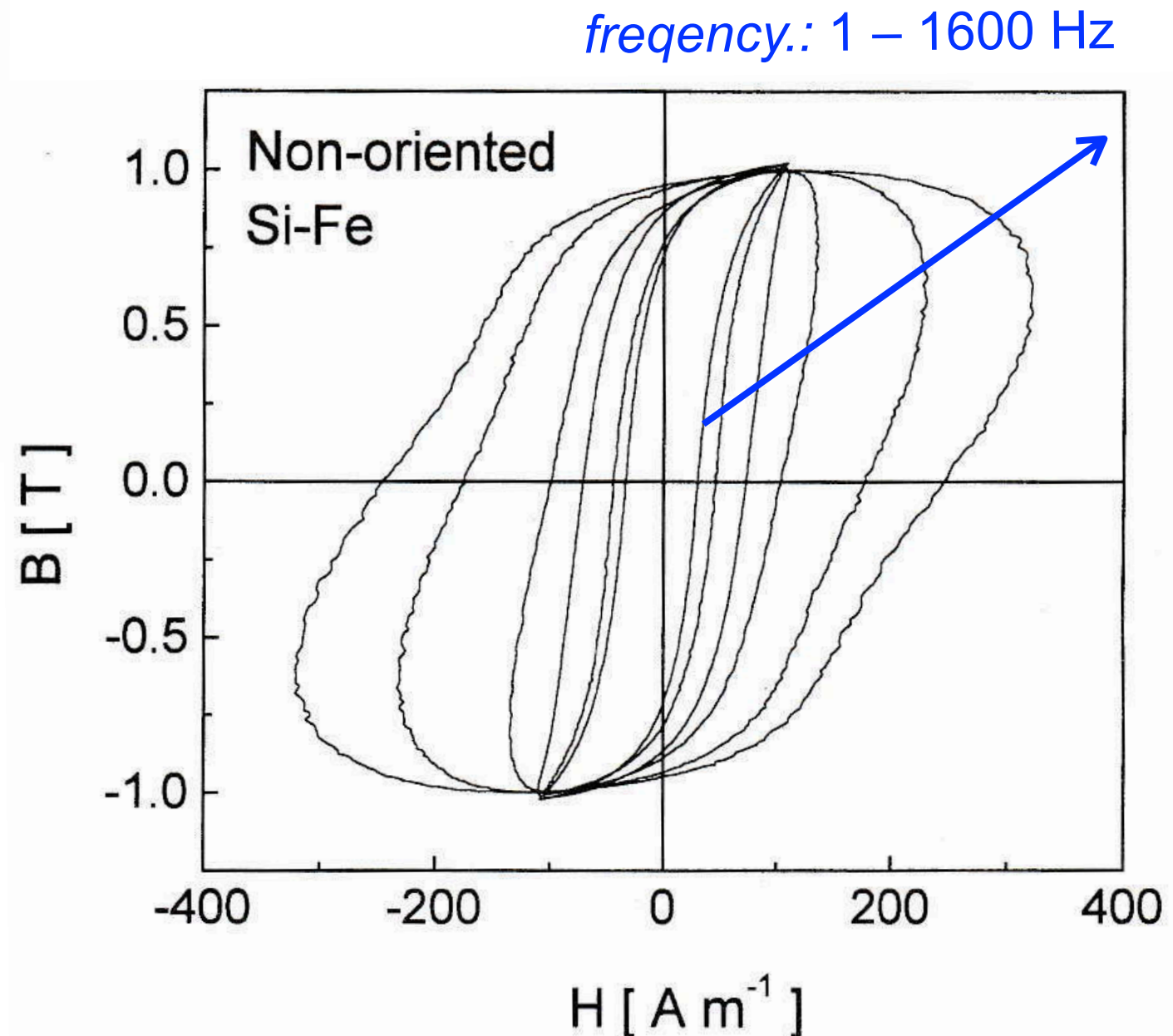
B.D. Cullity & CD Graham: Introduction to Magnetic Materials, 2009

What needs to be measured ?



Hysteresis curve:

- coercivity, remanence
- permeabilities
- energy loss
- interactions
- etc.



M(H) loop and domains

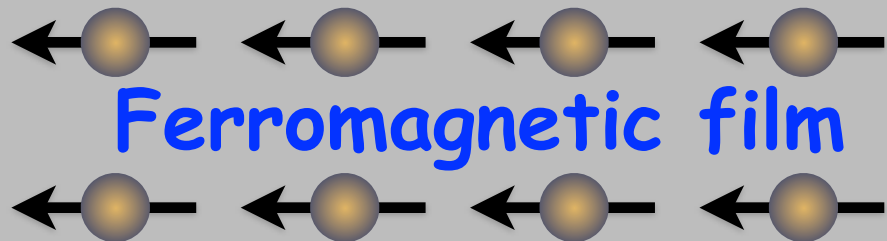
Reversal of Ni₈₁Fe₁₉ (30 nm) / NiO (30 nm)

Exchange bias

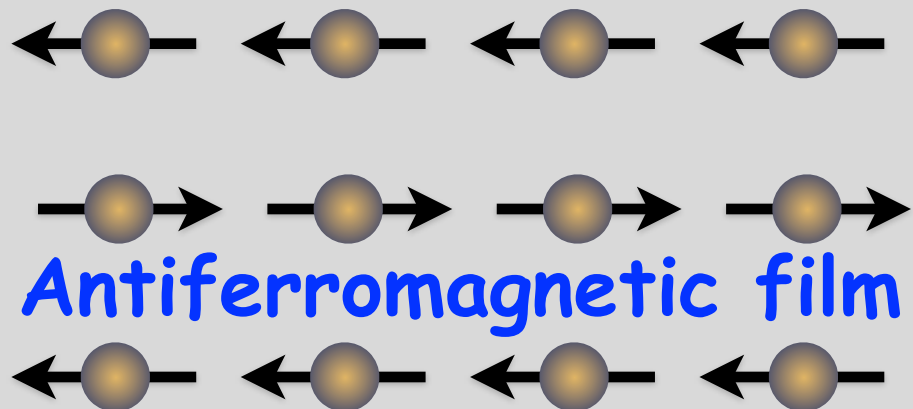
Pinning direction



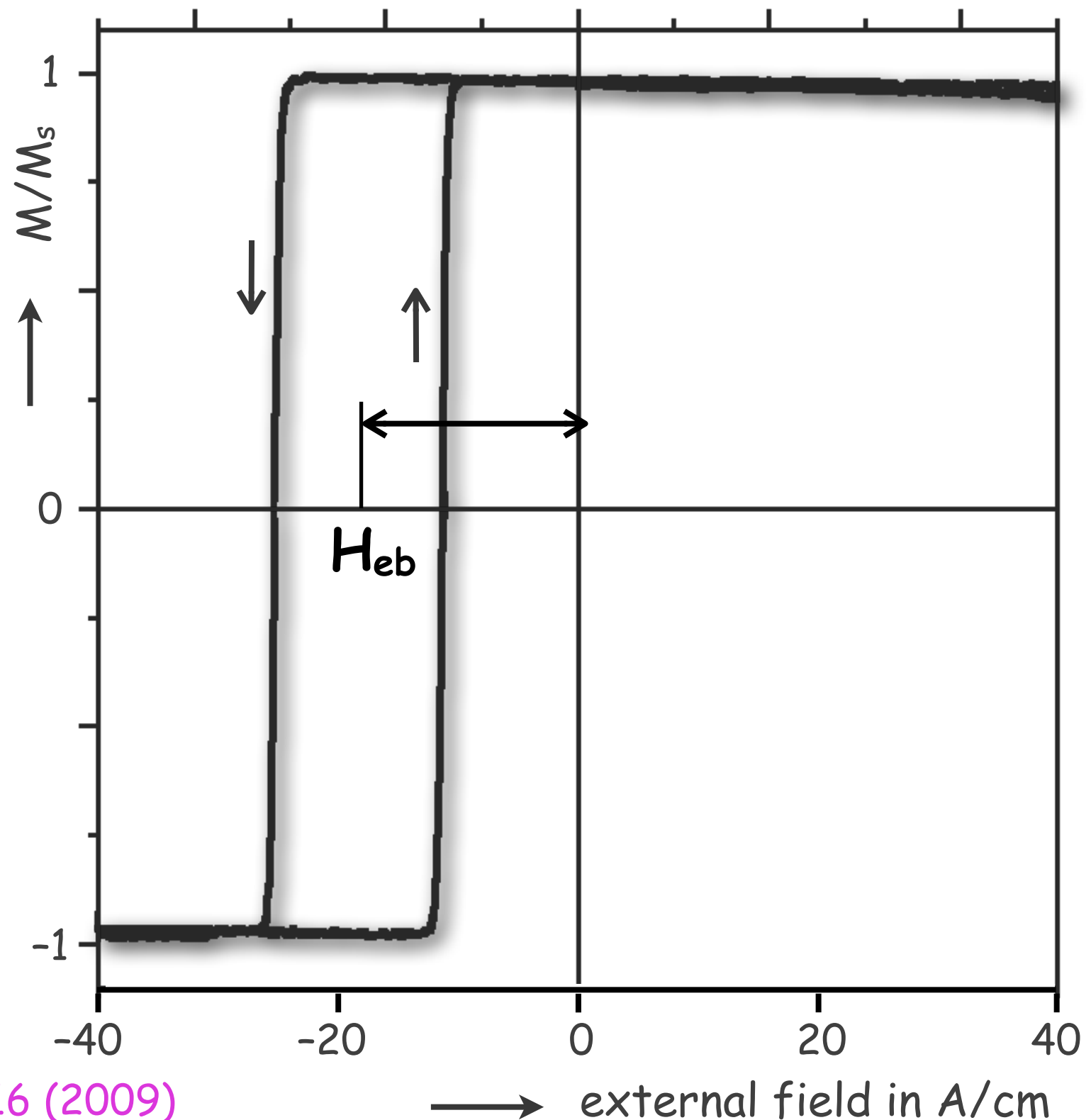
Ferromagnetic film



Antiferromagnetic film

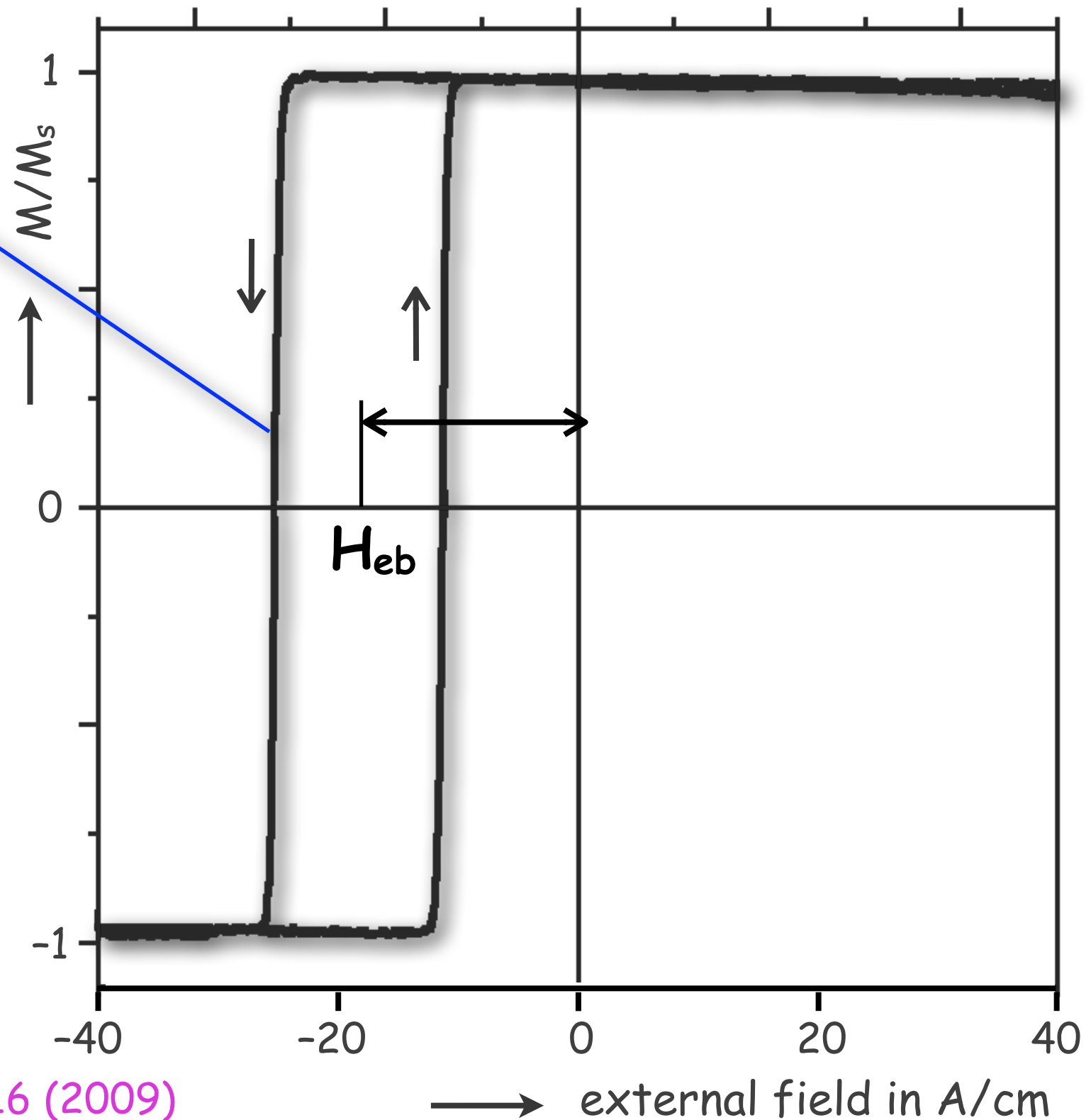
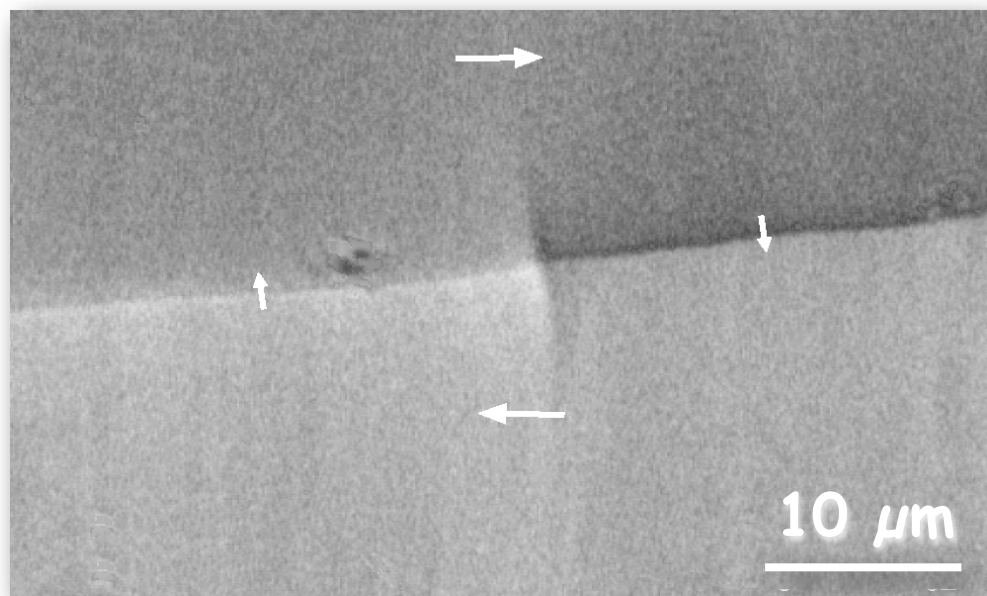


Unidirectional anisotropy



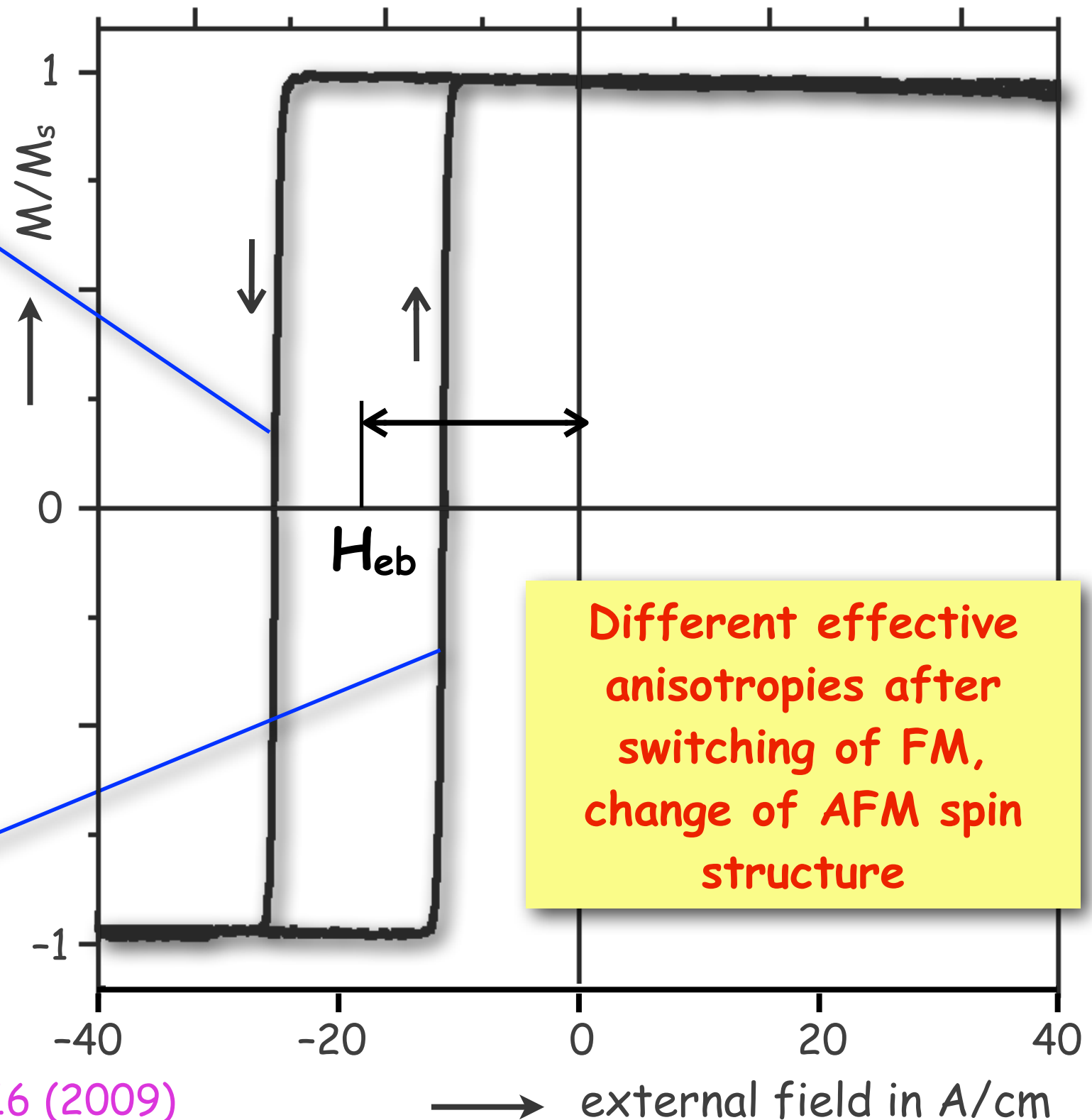
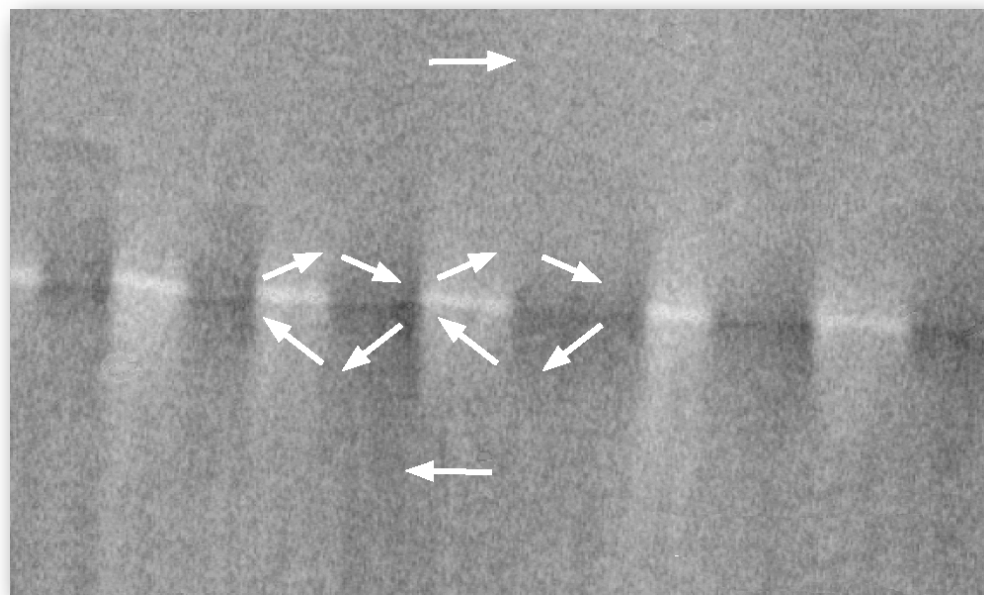
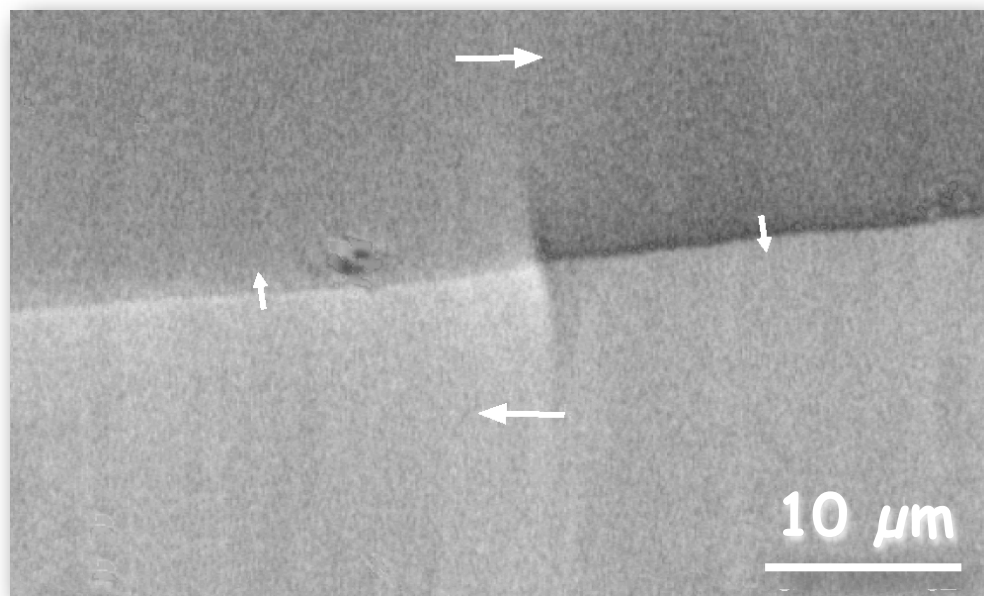
$M(H)$ loop and domains

Reversal of $\text{Ni}_{81}\text{Fe}_{19}$ (30 nm) / NiO (30 nm)



$M(H)$ loop and domains

Reversal of $\text{Ni}_{81}\text{Fe}_{19}$ (30 nm) / NiO (30 nm)



What needs to be measured ?

Magnetization curve

Domain scale
measurements

(Magnetic Imaging)

Saturation magnetization M_s

Crystal or other
anisotropy constants K

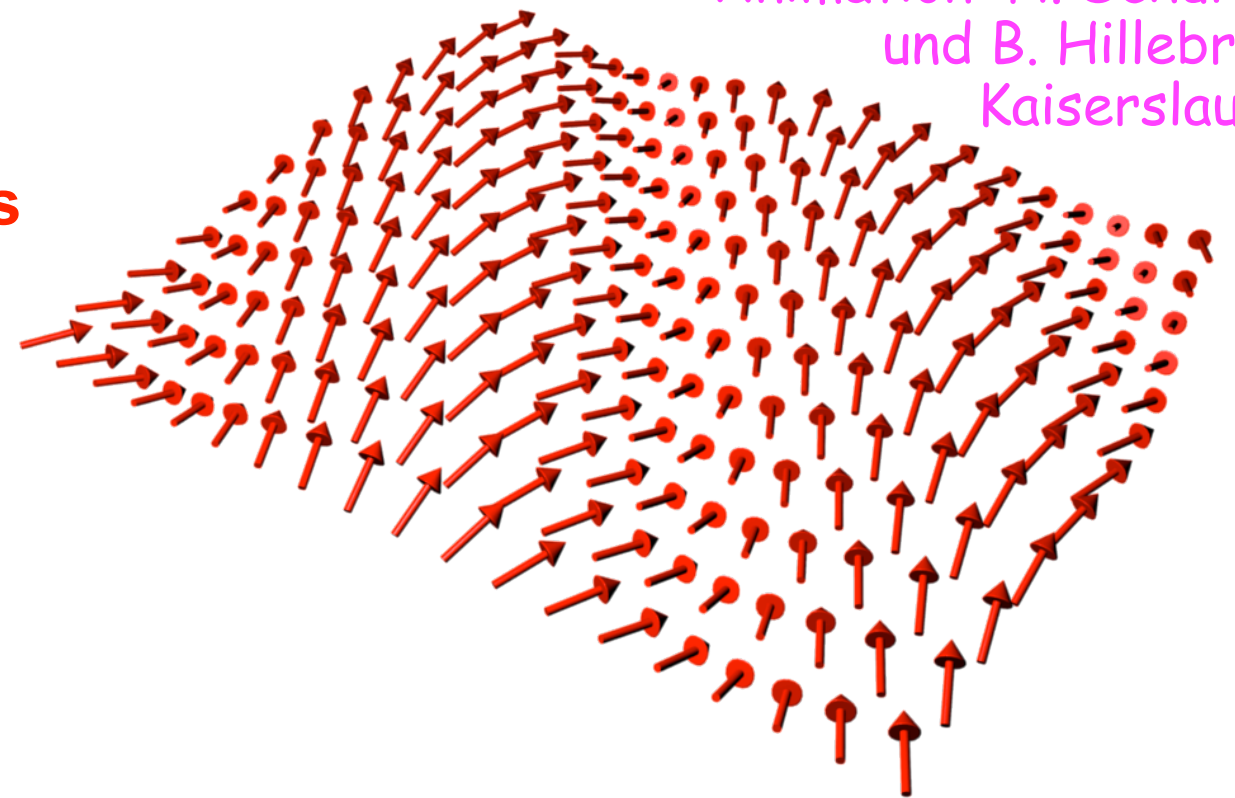
Exchange or
stiffness constant A

Magnetostriction constants λ

Magnetoresistance

Damping constants α
Resonance frequency f_{res}

Animation: H. Schultheiß
und B. Hillebrands
Kaiserslautern



Magnetic order (neutrons)

and more...

Contents of lecture

1. Production of magnetic field
2. Measurement of magnetic field strength
3. Magnetic measurements to determine material parameters & properties
 - 3.1. Magnetic measurements
 - 3.2. Mechanical measurements
 - 3.3. Resonance techniques
 - 3.4. Dilatometric measurements
 - 3.5. Domain methods
4. Domain scale measurements (Magnetic Imaging)

UNITS FOR MAGNETIC PROPERTIES

Quantity	Symbol	Gaussian & cgs emu ^a	Conversion factor, C ^b	SI & rationalized mks ^c
Magnetic flux density, magnetic induction	B	gauss (G) ^d	10^{-4}	tesla (T), Wb/m ²
Magnetic flux	Φ	maxwell (Mx), G·cm ²	10^{-8}	weber (Wb), volt second (V·s)
Magnetic potential difference, magnetomotive force	U, F	gilbert (Gb)	$10/4\pi$	ampere (A)
Magnetic field strength, magnetizing force	H	oersted (Oe), ^e Gb/cm	$10^3/4\pi$	A/m ^f
(Volume) magnetization ^g	M	emu/cm ^{3 h}	10^3	A/m
(Volume) magnetization	$4\pi M$	G	$10^3/4\pi$	A/m
Magnetic polarization, intensity of magnetization	J, I	emu/cm ³	$4\pi \times 10^{-4}$	T, Wb/m ^{2 i}
(Mass) magnetization	σ, M	emu/g	$\frac{1}{4\pi \times 10^{-7}}$	A·m ² /kg Wb·m/kg
Magnetic moment	m	emu, erg/G	10^{-3}	A·m ² , joule per tesla (J/T)
Magnetic dipole moment	j	emu, erg/G	$4\pi \times 10^{-10}$	Wb·m ⁱ
(Volume) susceptibility	χ, κ	dimensionless, emu/cm ³	$\frac{4\pi}{(4\pi)^2} \times 10^{-7}$	dimensionless henry per meter (H/m), Wb/(A·m)
(Mass) susceptibility	χ_p, κ_p	cm ³ /g, emu/g	$\frac{4\pi \times 10^{-3}}{(4\pi)^2} \times 10^{-10}$	m ³ /kg H·m ² /kg
(Molar) susceptibility	$\chi_{\text{mol}}, \kappa_{\text{mol}}$	cm ³ /mol, emu/mol	$\frac{4\pi \times 10^{-6}}{(4\pi)^2} \times 10^{-13}$	m ³ /mol H·m ² /mol
Permeability	μ	dimensionless	$4\pi \times 10^{-7}$	H/m, Wb/(A·m)
Relative permeability ^j	μ_r	not defined		dimensionless
(Volume) energy density, energy product ^k	\mathcal{W}	erg/cm ³	10^{-1}	J/m ³
Demagnetization factor	D, N	dimensionless	$1/4\pi$	dimensionless

- a.* Gaussian units and cgs emu are the same for magnetic properties. The defining relation is $B = H + 4\pi M$.
- b.* Multiply a number in Gaussian units by C to convert it to SI (e.g., $1 \text{ G} \times 10^{-4} \text{ T/G} = 10^{-4} \text{ T}$).
- c.* SI (*Système International d'Unités*) has been adopted by the National Bureau of Standards. Where two conversion factors are given, the upper one is recognized under, or consistent with, SI and is based on the definition $B = \mu_0(H + M)$, where $\mu_0 = 4\pi \times 10^{-7} \text{ H/m}$. The lower one is not recognized under SI and is based on the definition $B = \mu_0 H + J$, where the symbol I is often used in place of J .
- d.* $1 \text{ gauss} = 10^5 \text{ gamma } (\gamma)$.
- e.* Both oersted and gauss are expressed as $\text{cm}^{-1/2} \cdot \text{g}^{1/2} \cdot \text{s}^{-1}$ in terms of base units.
- f.* A/m was often expressed as "ampere-turn per meter" when used for magnetic field strength.
- g.* Magnetic moment per unit volume.
- h.* The designation "emu" is not a unit.
- i.* Recognized under SI, even though based on the definition $B = \mu_0 H + J$. See footnote *c*.
- j.* $\mu_r = \mu/\mu_0 = 1 + \chi$, all in SI. μ_r is equal to Gaussian μ .
- k.* $B \cdot H$ and $\mu_0 M \cdot H$ have SI units J/m³; $M \cdot H$ and $B \cdot H/4\pi$ have Gaussian units erg/cm³.

Magnetic Units

In this presentation:
SI units

$$B = \mu_0(H + M) = \mu_0 H + J$$

$$B, J : [T] = \left[\frac{\text{V sec}}{\text{m}^2} \right]$$

$$H, M : \left[\frac{\text{A}}{\text{m}} \right]$$

from IEEE Magn. Soc. webpage

http://www.ieemagnetics.org/images/stories/magnetic_units.pdf

For reading

B.D. Cullity and C.D. Graham: Introduction to Magnetic Materials.
IEEE Press and Wiley (2009)

S. Tumanski: Handbook of Magnetic Measurements.
CRC Press Taylor & Francis (2009)

F. Fiorillo: Measurement and Characterization of Magnetic Materials. Elsevier
Academic Press (2004)

R. Hilzinger and W. Rodewald: Magnetic Materials.
Edited by Vacuumschmelze GmbH, Publicis Publishing, Erlangen (2013)

D.C. Jiles: Introduction to Magnetism and Magnetic Materials.
Chapman & Hall, London (1995)

G. Bertotti: Hysteresis in Magnetism.
Academic Press, New York (1998)

M. Coey: Magnetism and Magnetic Materials.
Cambridge University Press (2010)

A. Hubert and R.S.: Magnetic Domains. Springer Verlag (1998)

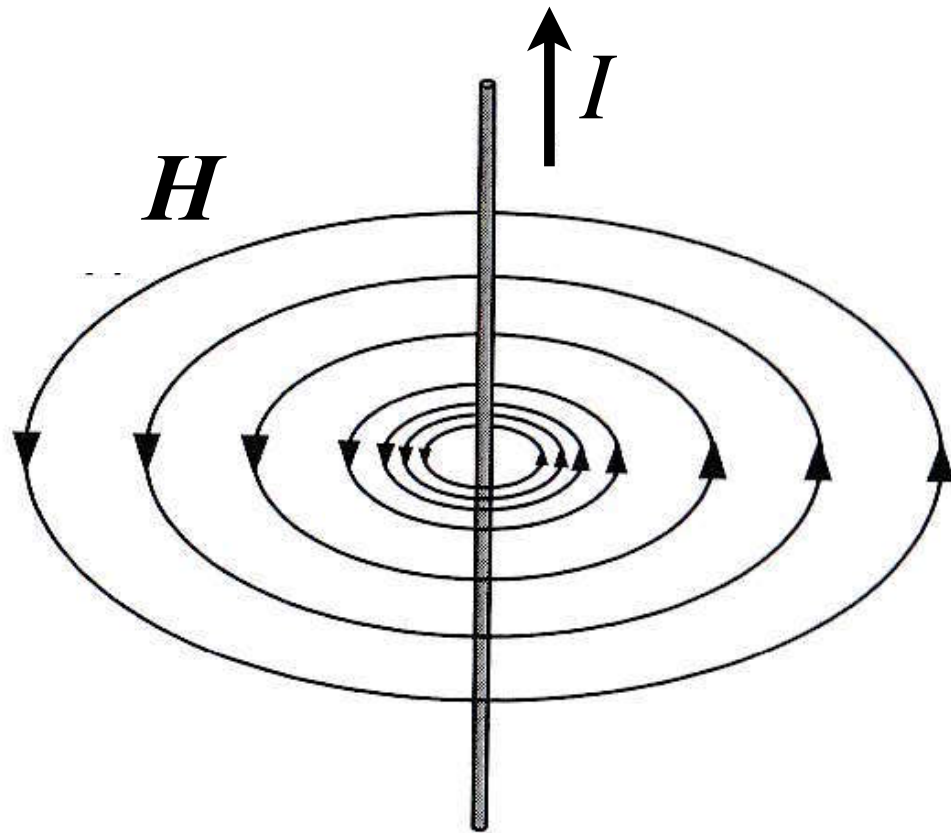
*Many figures in this presentation
are taken from these references -
special thanks to the authors*

1.

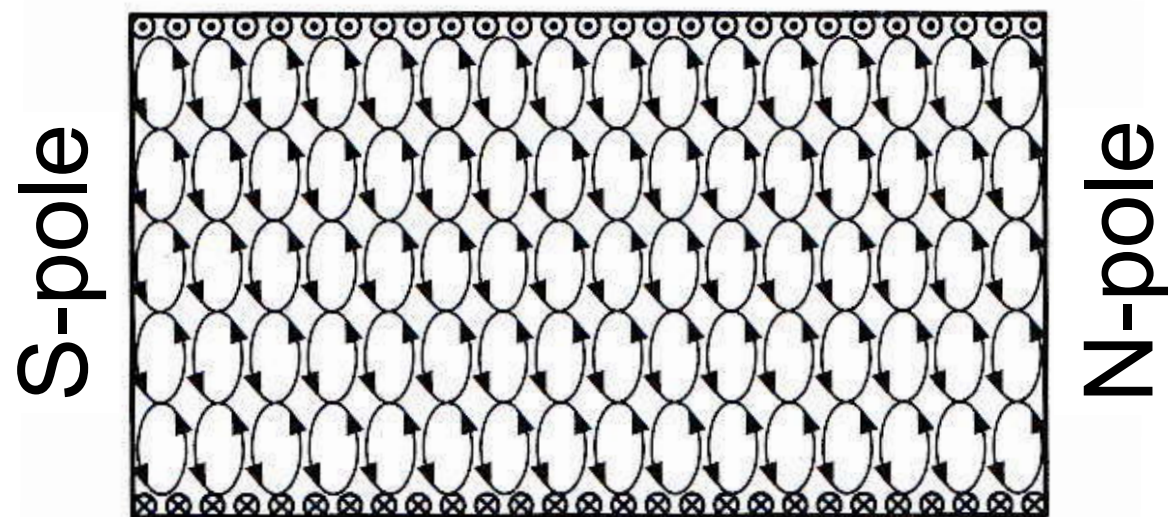
Production of Magnetic Field

1. Production of Magnetic Field

Two possibilities to generate magnetic field:



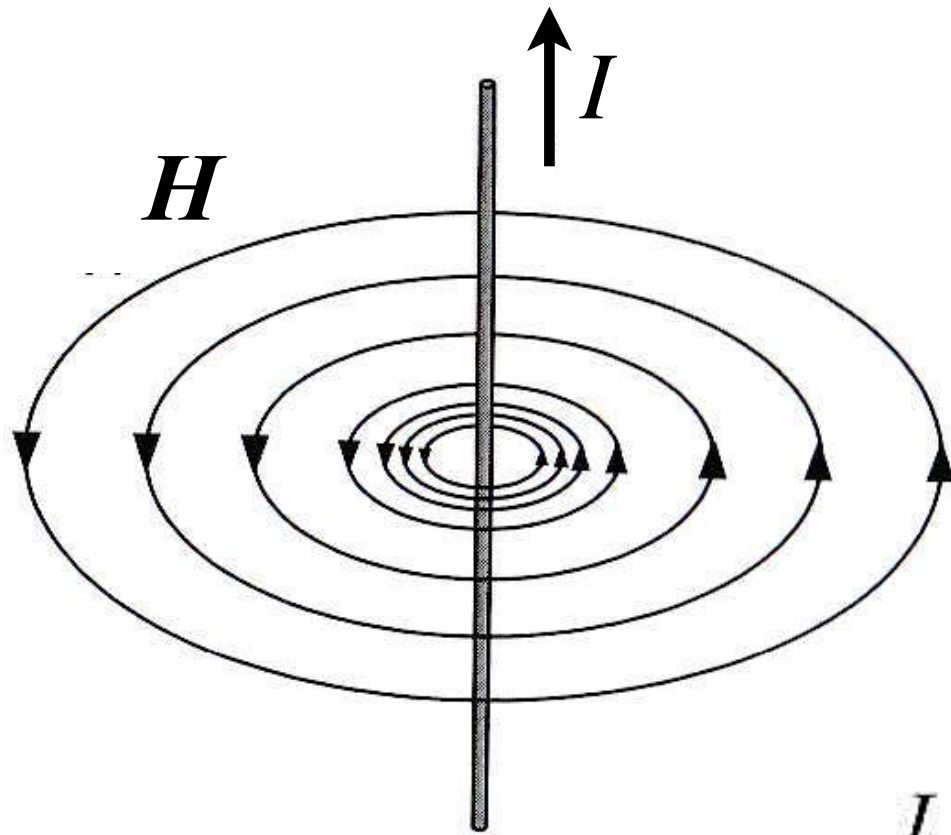
By electrical currents
flowing in conductor
→ electromagnetic coils



By exploiting the ordered array of
quantummechanical electronic
currents circulating in a magnetic
material (→ permanent magnets)

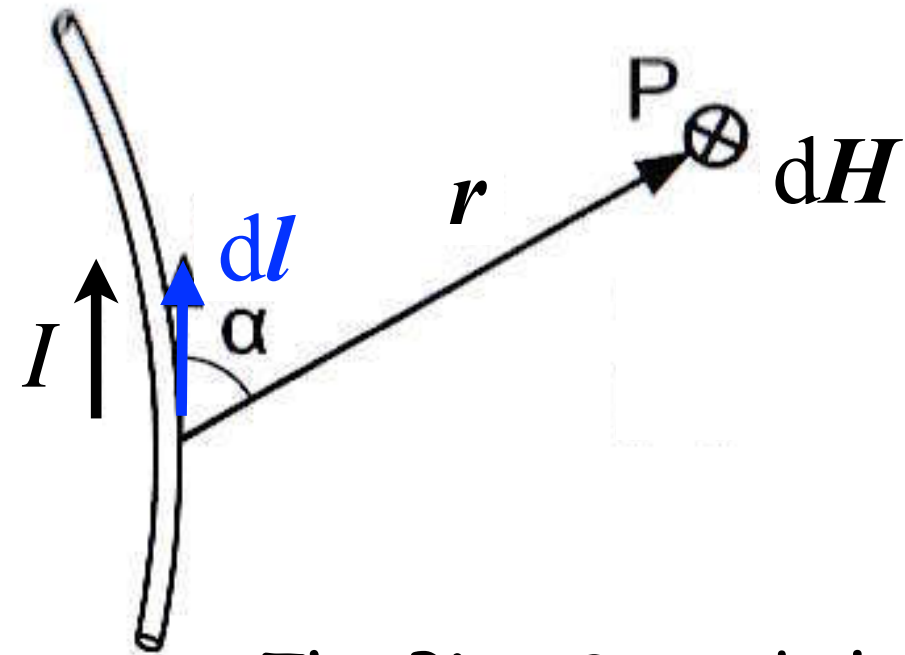
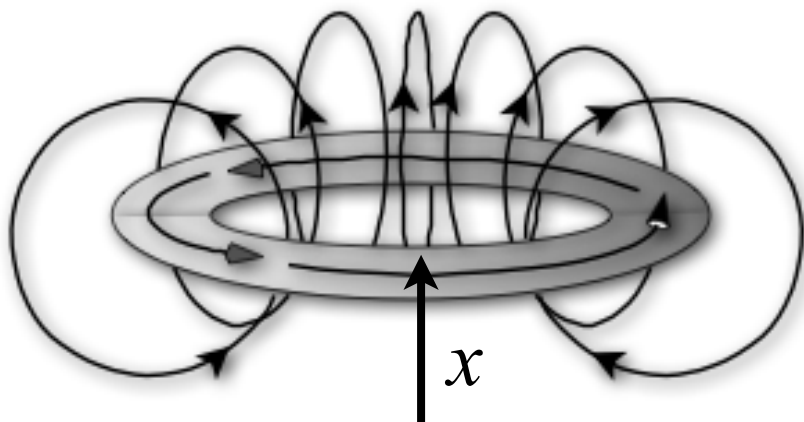
1. Production of Magnetic Field

1.1 Electromagnetic coils



$$\text{rot } \mathbf{H} = \mathbf{j} \longrightarrow H = \frac{I}{2\pi \cdot r}$$

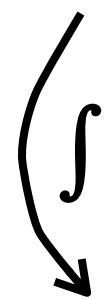
Current loop of radius r



The Biot-Savart's law

$$d\mathbf{H} = \frac{1}{4\pi \cdot r^3} \cdot I \cdot d\mathbf{l} \times \mathbf{r} \quad \text{or}$$

$$dH = \frac{1}{4\pi \cdot r^2} \cdot I \cdot dl \cdot \sin \alpha$$



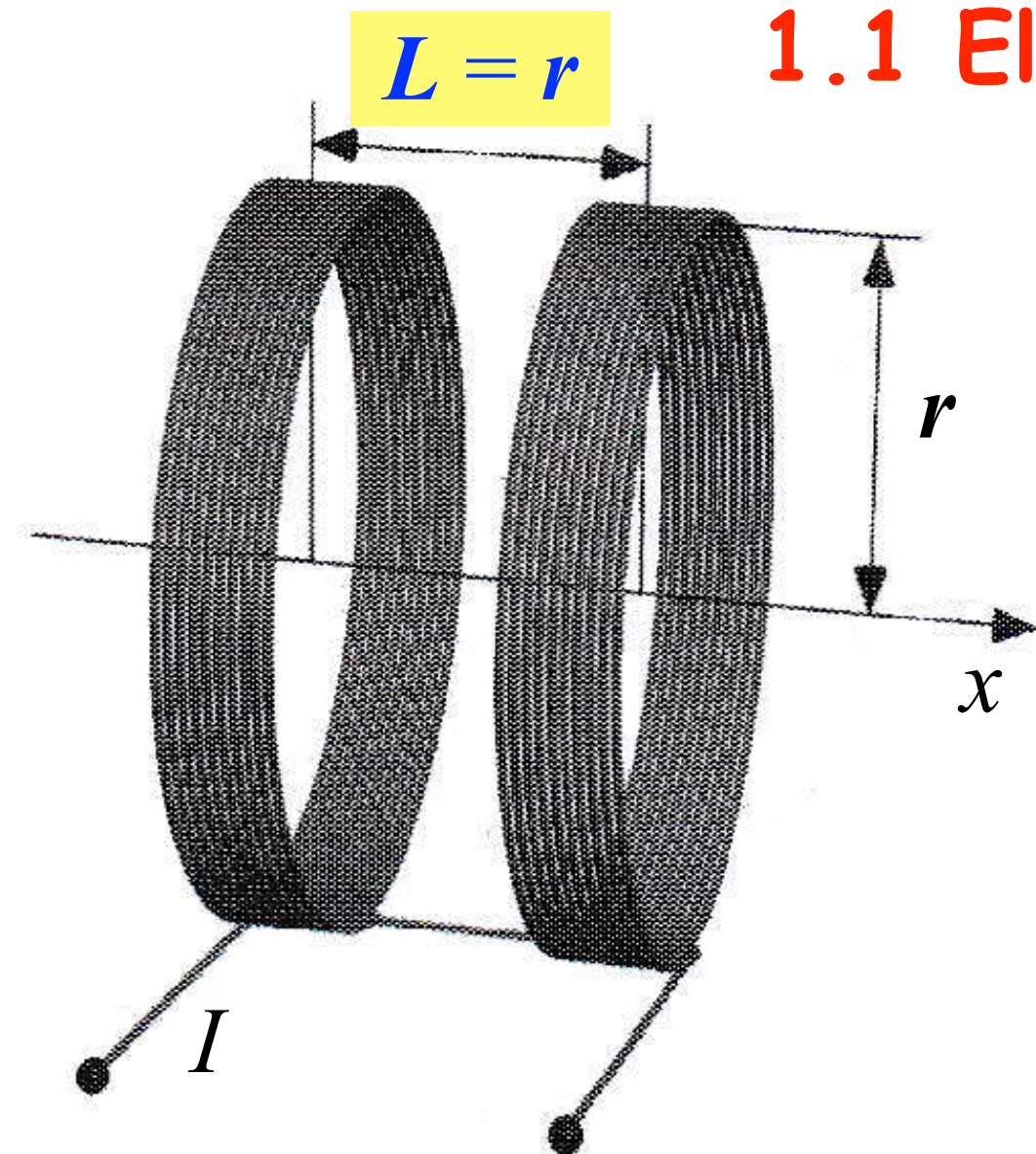
$$H_x(x) = \frac{N \cdot I \cdot r^2}{2(r^2 + x^2)^{3/2}}$$

Field of
coil with
 N windings

1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.)

Helmholtz coils

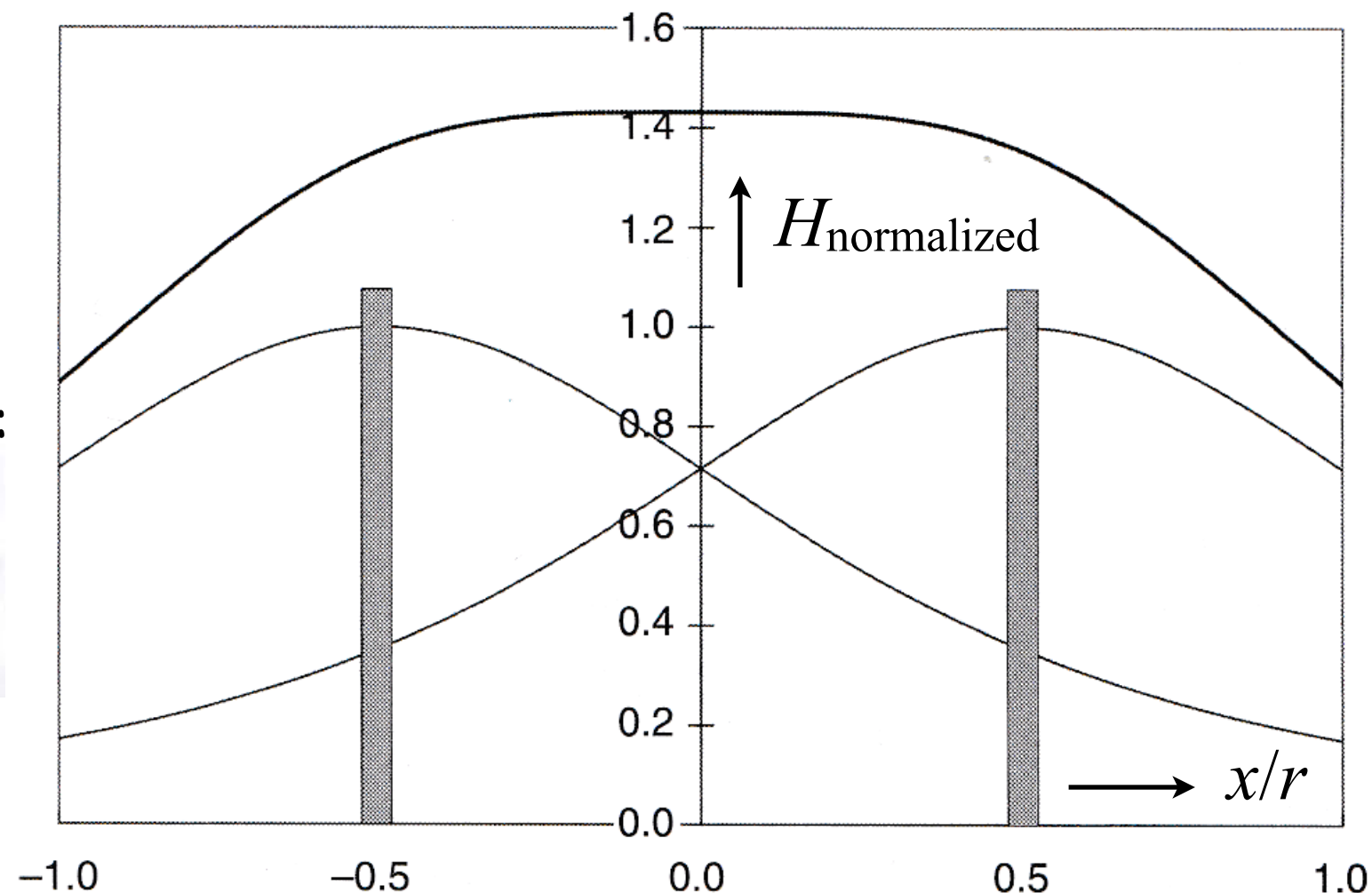


$$H_x(x) = \frac{N \cdot I}{2r} \cdot \left[\left(1 + \frac{x^2}{r^2} \right)^{-\frac{3}{2}} + \left(1 + \frac{(r-x)^2}{r^2} \right)^{-\frac{3}{2}} \right]$$

Axial field component at centre point:

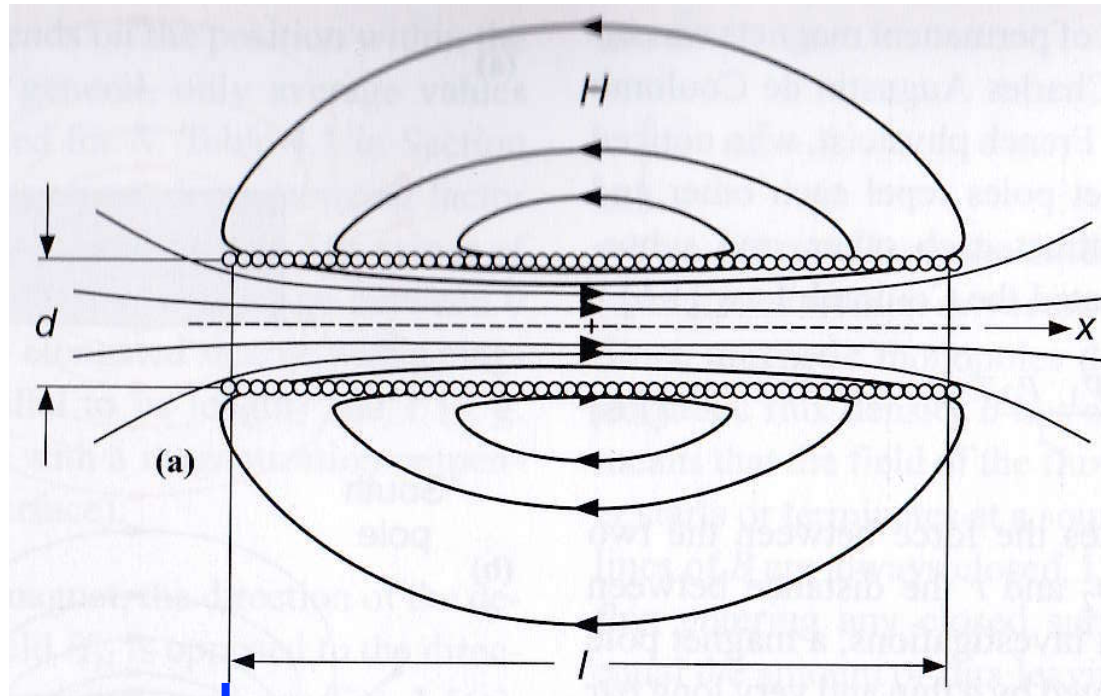
$$H_x(x = r/2) = 0.7155 \cdot \frac{N \cdot I}{r}$$

Homogeneous (small) field
in large volume



1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.)



Solenoid



Solenoid with one layer of winding:

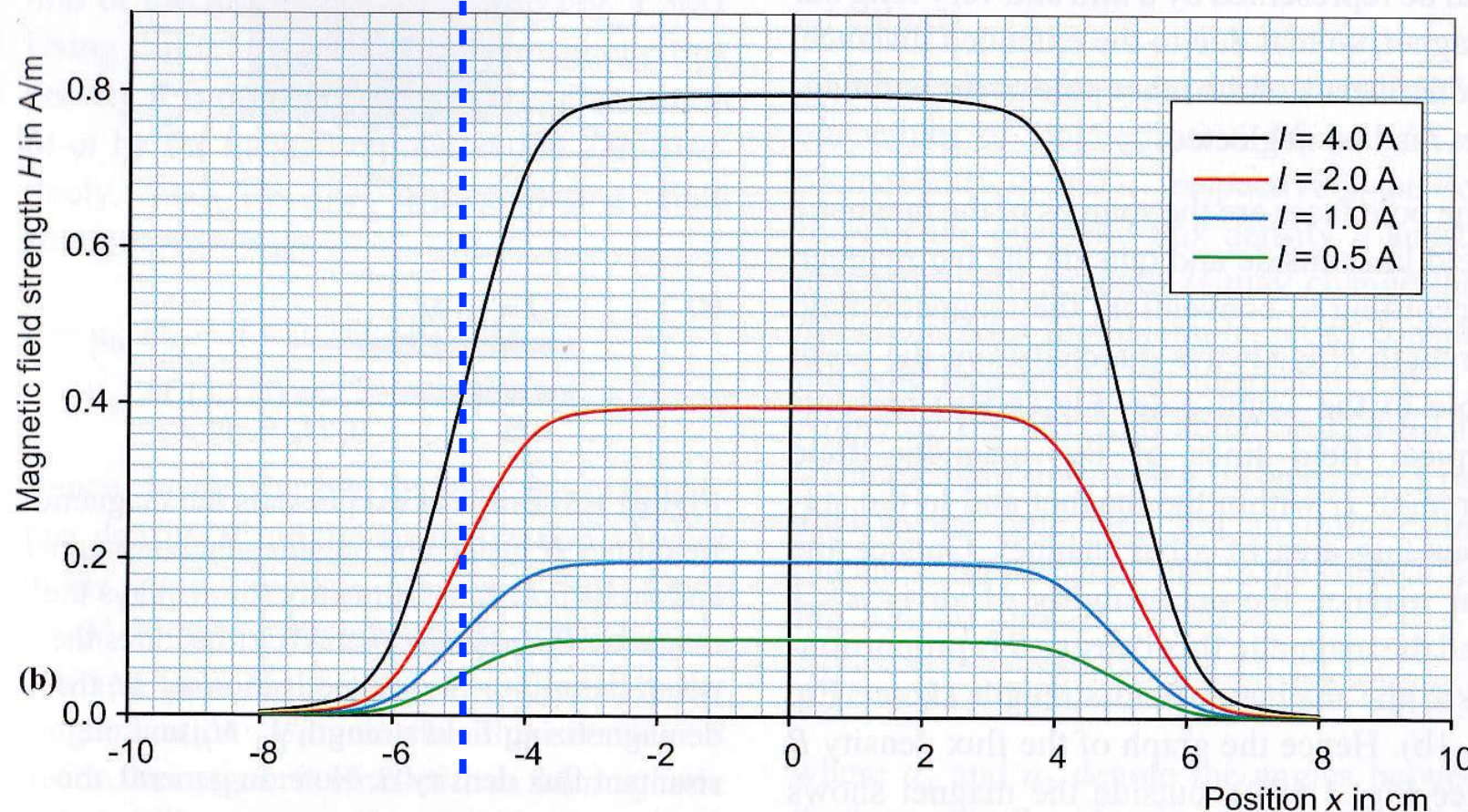
$$H(x) = \frac{N \cdot I}{l} \cdot \left\{ \frac{(l + 2x)}{2\sqrt{[d^2 + (l + 2x)^2]}} + \frac{(l - 2x)}{2\sqrt{[d^2 + (l - 2x)^2]}} \right\}$$

Field at centre:

$$H(x=0) = \frac{N \cdot I}{l} \cdot \frac{l}{\sqrt{[d^2 + l^2]}}$$

Long solenoid ($l \gg d$):

$$H(x=0) = \frac{N \cdot I}{l}$$



1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.)

Remarks:

- Higher field: better increase n/l by adding more layers of winding rather than increasing current

$$H \sim I, \text{ but } \textit{heat} \sim I^2 R$$

Thus doubling number of winding layers and keeping current constant will double H , R , and amount of *heat* ; whereas doubling of current will double H , but will quadruple *heat*

- Typical field: 0.1 T*,
higher field requires cooling

* Although SI unit of field is A/m, it is common to express field strength in units of $\mu_0 H = B$ [Tesla]

Solenoid



Solenoid with one layer of winding:

$$H(x) = \frac{N \cdot I}{l} \cdot \left\{ \frac{(l+2x)}{2\sqrt{[d^2 + (l+2x)^2]}} + \frac{(l-2x)}{2\sqrt{[d^2 + (l-2x)^2]}} \right\}$$

Field at centre:

$$H(x=0) = \frac{N \cdot I}{l} \cdot \frac{l}{\sqrt{[d^2 + l^2]}}$$

Long solenoid ($l \gg d$):

$$H(x=0) = \frac{N \cdot I}{l}$$

1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.)

High-Field Solenoid: Bitter magnet

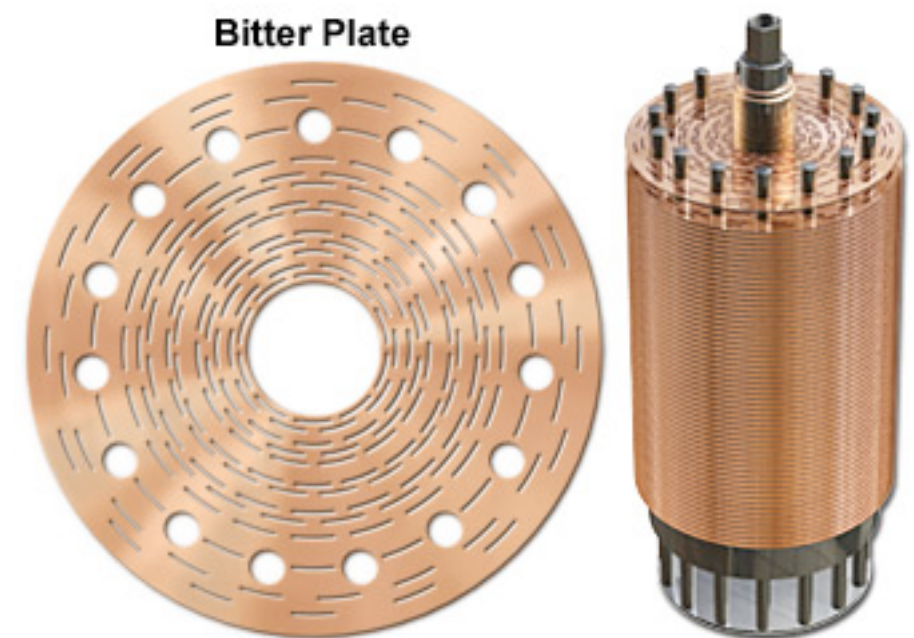
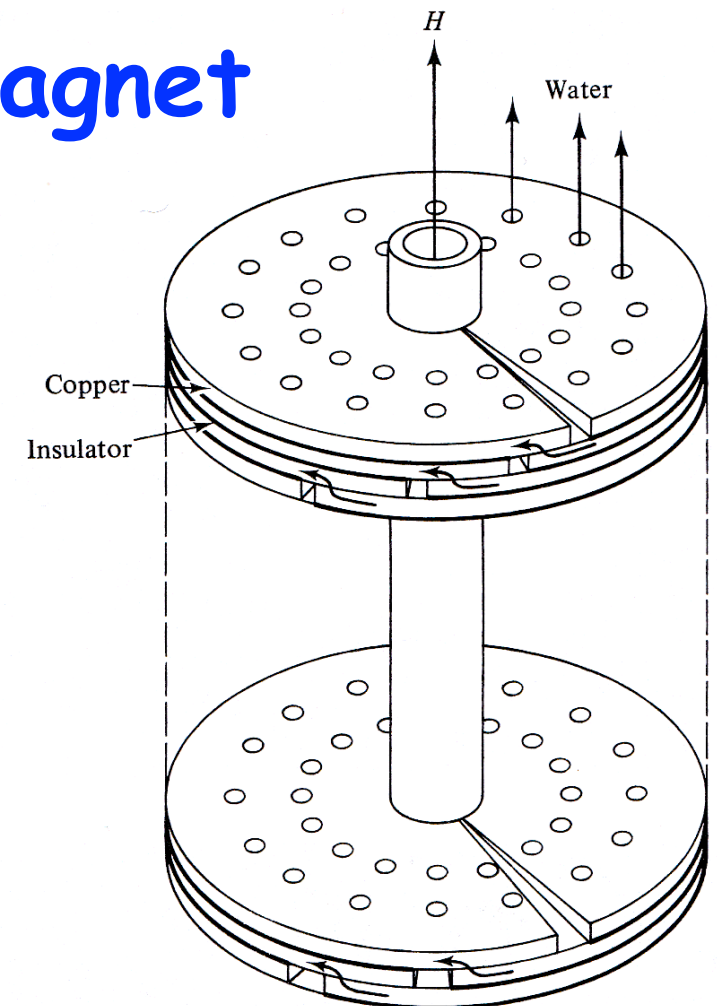
High field: requires large power input

→ two major design problems:

- 1) Large amount of heat (note: maintaining magnetic field by current is process of zero efficiency: all input power goes into heat)
- 2) Mechanical strength to resist large forces acting on current carriers has to be provided

Bitter magnet:

- Winding composed of Cu disks, ~30 cm diameter
- Insulated from each other, clamped together
- Rotated by 20°: overlap = conduction path
- Cooling water pumped through holes,
- Helical current path, acts like solenoid
- Typical field: 45 Tesla in 30 mm bore, requires current of 67.000 A, power input of 20 MW
- Requires large motor-generator sets

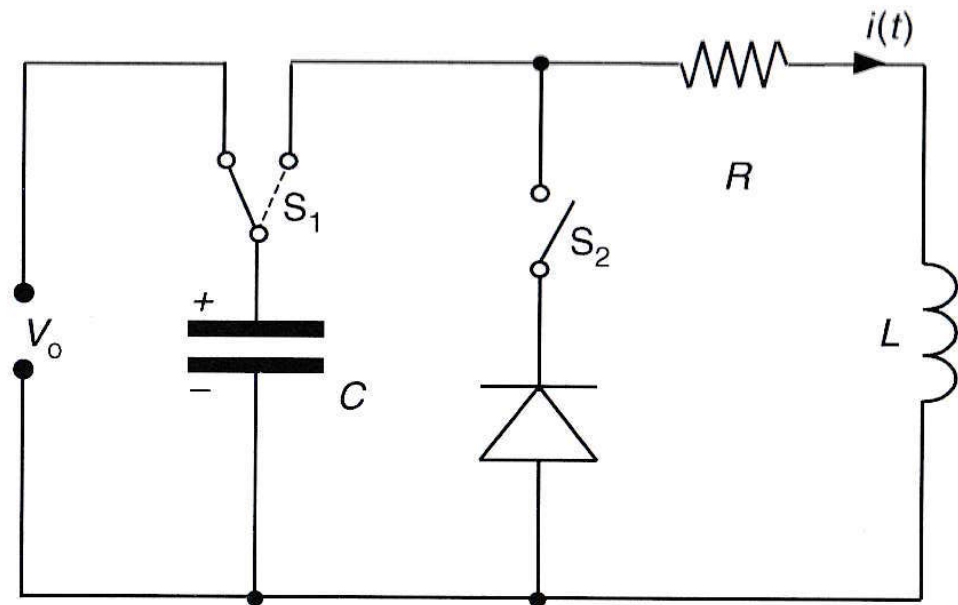


1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.)

High-field Solenoid: Pulsed Fields

Pulsed fields up to about 10 T (peak) can be obtained by conventional solenoids, supplied by a discharging capacitor bank



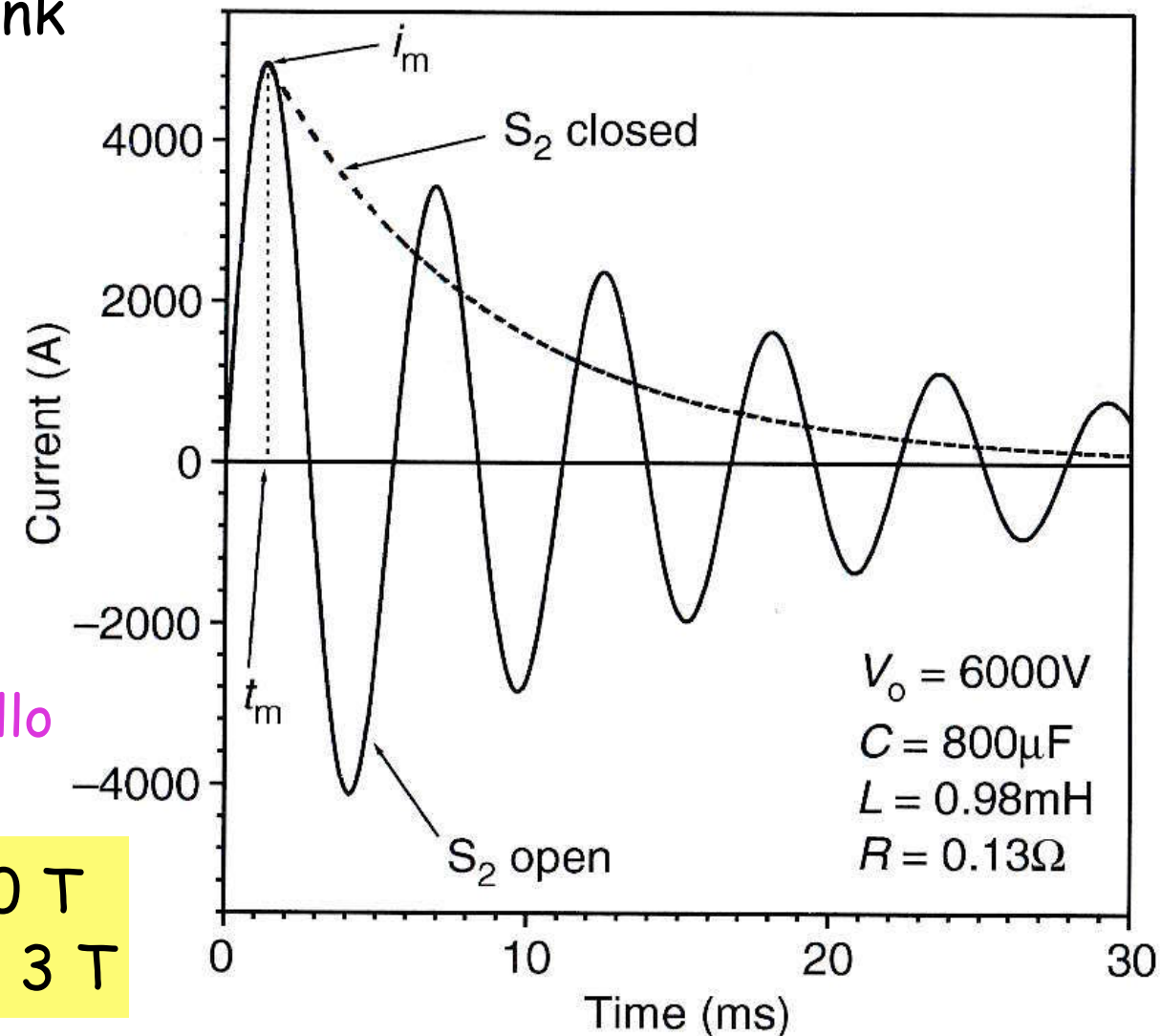
$$L \frac{\partial^2 i(t)}{\partial t^2} + R \frac{\partial i(t)}{\partial t} - \frac{1}{C} i(t) = 0,$$

$$i(t) = \frac{V_0}{L} \exp\left(-\frac{R}{2L}t\right) \frac{\sin \omega t}{\omega}$$



Courtesy F. Fiorillo

- Cooled: up to 20 T
- Uncooled: up to 3 T

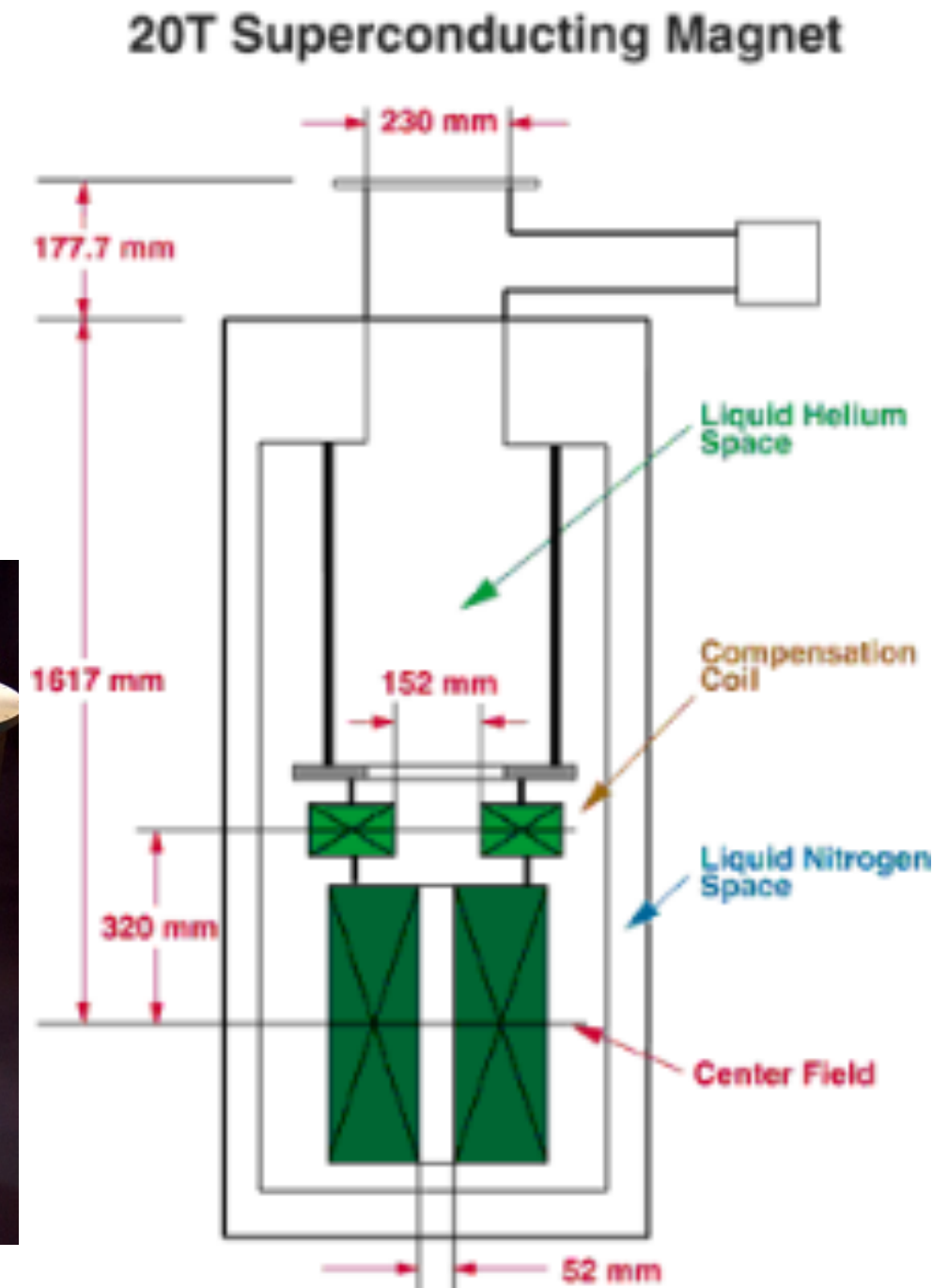


In shown arrangement with given inductance L of solenoid, an oscillating damped discharge is obtained (switch S_2 open). If S_2 is closed at maximum field, the diode prevents capacitor from discharging with reversed polarity and current decays from maximum value with time constant $\tau_1 = L/R$

1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.) Superconducting Solenoids

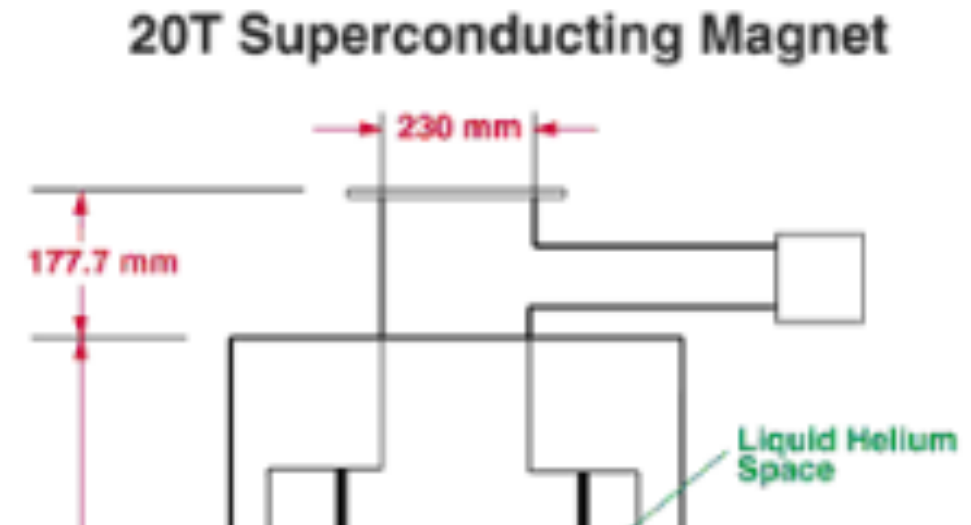
- DC fields up to about 20 T can be obtained by superconducting solenoids, commonly used for fields above 2 T
- Type II superconductor with high critical current and critical field: Nb-Ti or Nb₃Sn
- Cooling of coil by liquid helium (4.2 K), sample temperature up to room temperature
- Shortcut of superconducting coil: persistent mode, no power consumption over months
- Danger: quench by local heating



1. Production of Magnetic Field

1.1 Electromagnetic coils (cont.) Superconducting Solenoids

- DC fields up to about 20 T can be obtained by superconducting solenoids, commonly used for fields above 2 T
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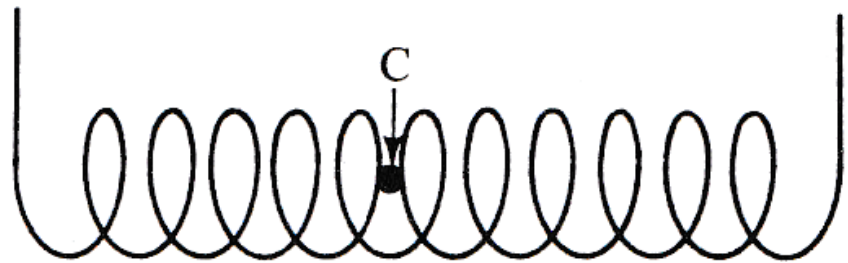


1. Production of Magnetic Field

1.2 Electromagnets

DC fields up to 2 T, most commonly used magnetic field source in labs

Evolution of the electromagnet:



Solenoid, flux density at center C:

$$B = \mu_0 H = \mu_0 \frac{N \cdot I}{l}$$

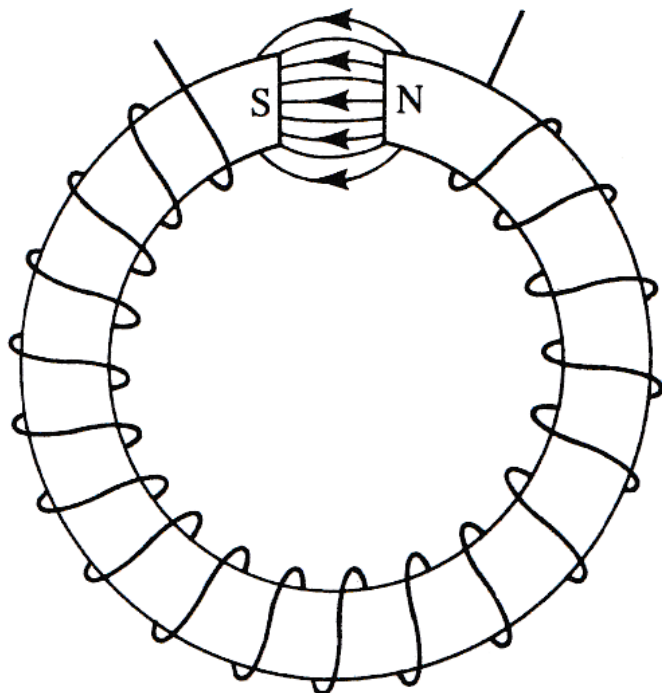
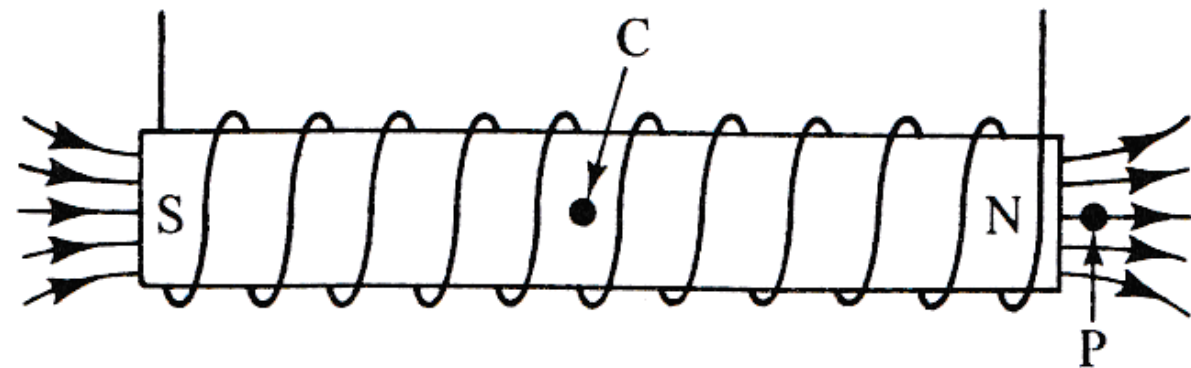
Solenoid with iron rod, flux density at center C:

$$B = \mu_0(H + M) = \mu H$$

Iron has multiplied field due to the current by factor of μ , same field occurs just outside rod at P \rightarrow large field obtained with low current (e.g. $H_{\text{coil}} = 1 \text{ mT}$, $\mu = 2000$: $H_{\text{outside}} = 2 \text{ T}$) Problem: flux lines outside iron diverge and field decreases rapidly

Bended iron rod with gap:

Flux travels directly from pole to pole across gap. Contribution of iron to gap field (if saturated) = 2.15 T

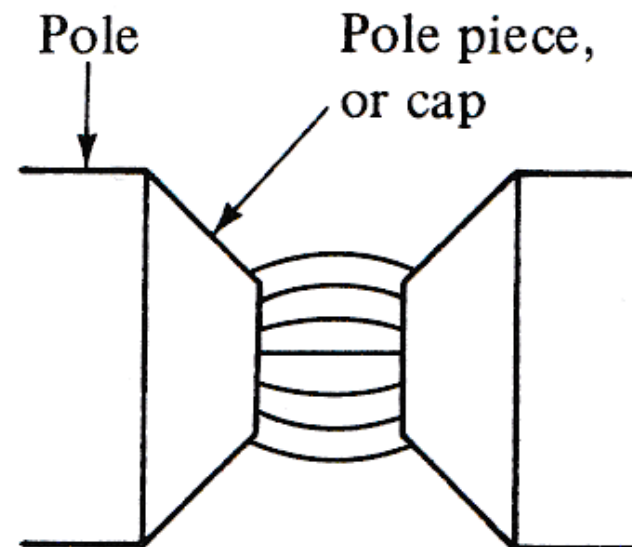
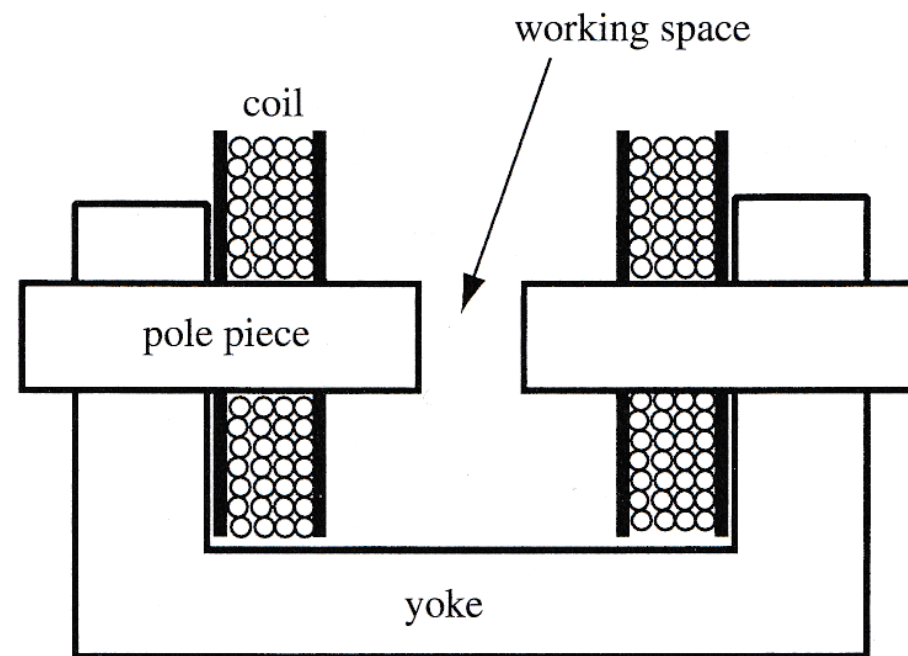
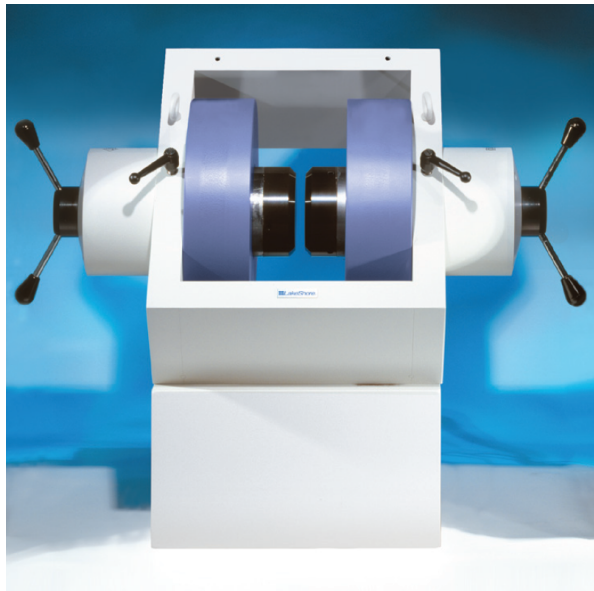


1. Production of Magnetic Field

1.2 Electromagnets (cont.)

DC fields up to 2 T, most commonly used magnetic field source in labs

Evolution of the electromagnet:



Electromagnet:

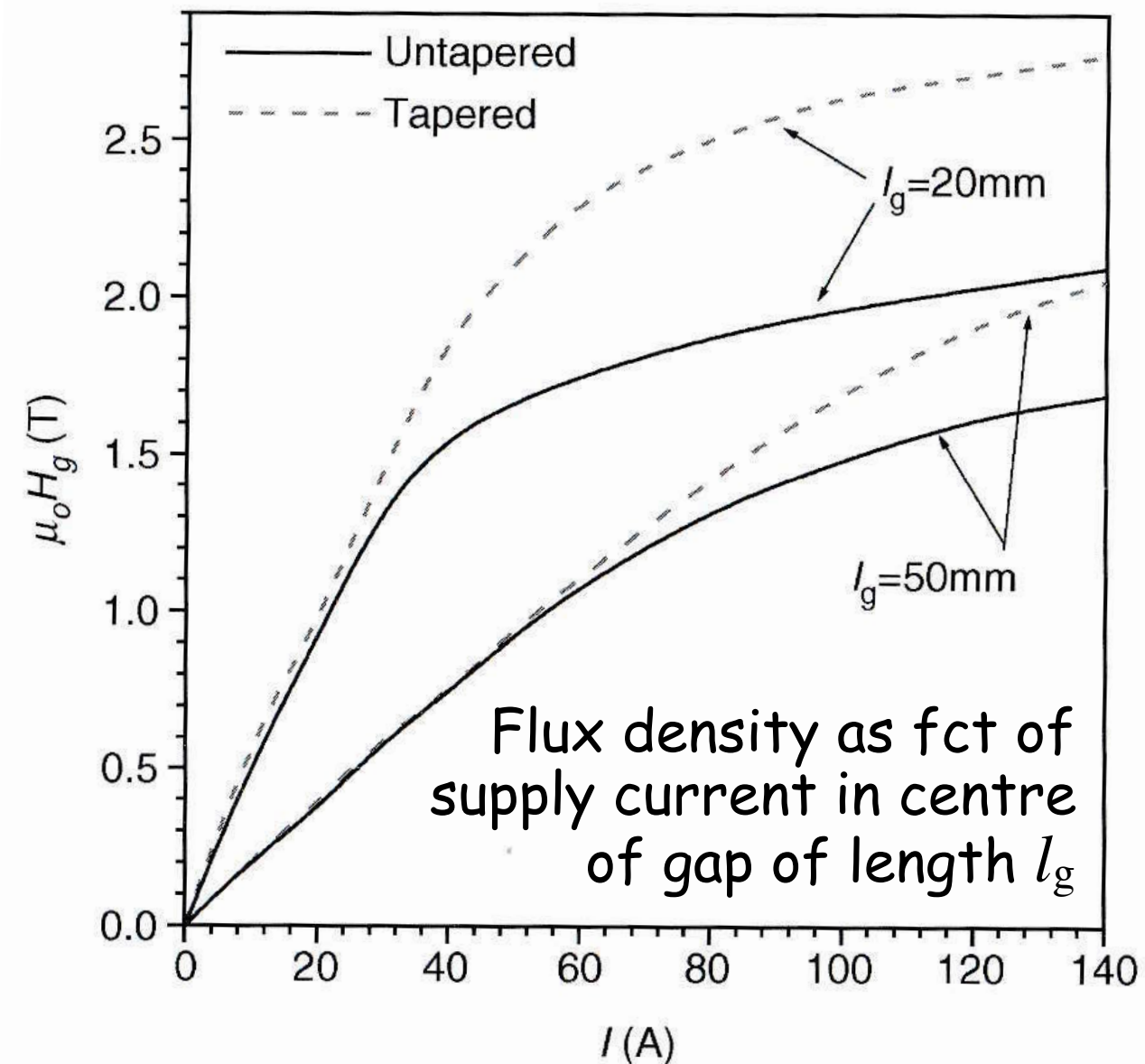
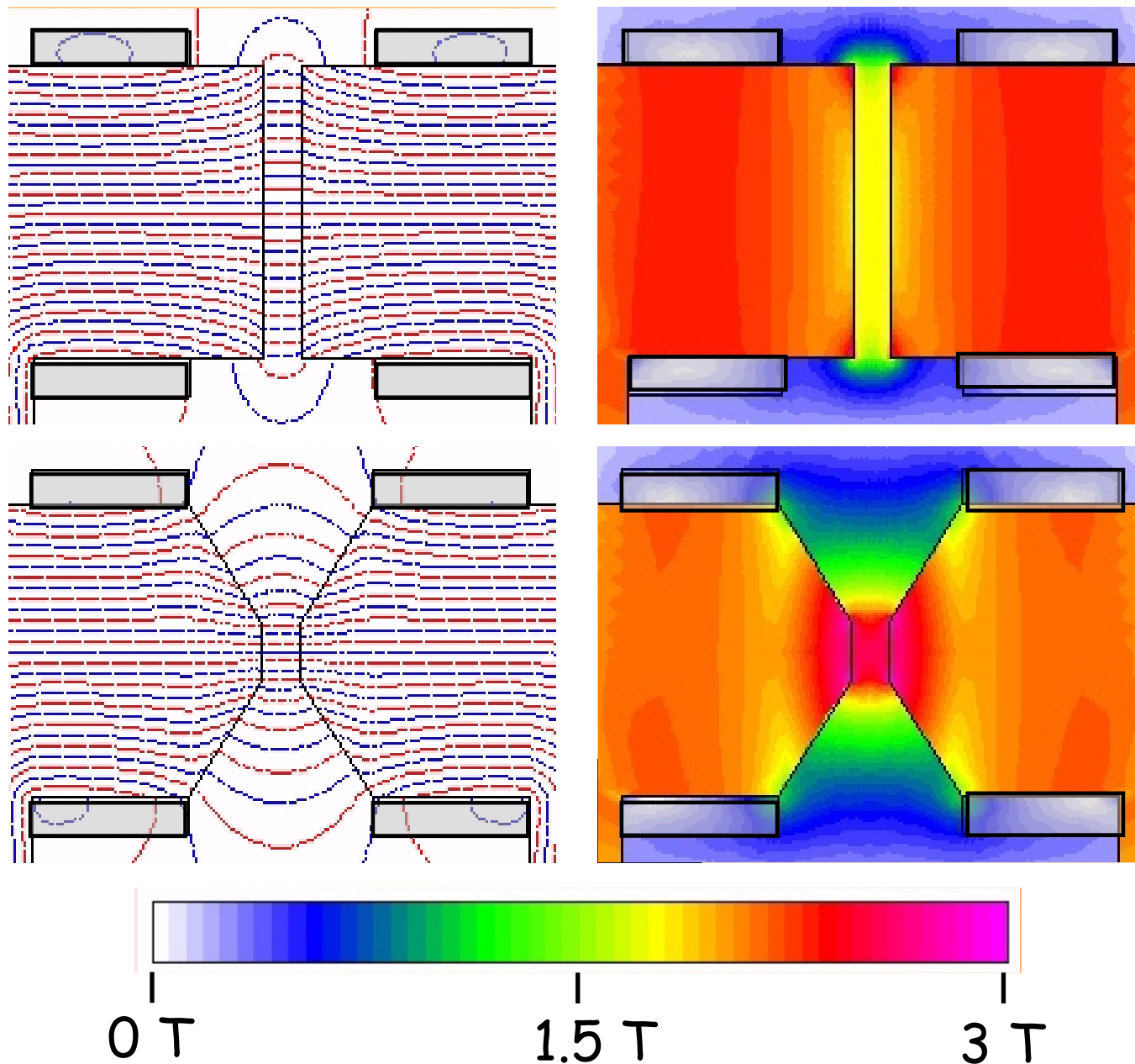
- Windings close to gap
- Core and yoke made of iron, annealed for high permeability
- Windings water-cooled
- Pole diameter: up to 30 cm
- Flat poles for uniform field
- Tapered poles: free poles formed on tapered surfaces contribute to field at gap center, can achieve fields higher than $\mu_0 M_s$ ($> 3\text{ T}$ for gap length of 5-10 mm). Optimum taper angle: $54,74^\circ$
- Pole pieces made of CoFe (M_s about 10% higher than for pure Fe)

1. Production of Magnetic Field

1.2 Electromagnets (cont.)

DC fields up to 2 T, most commonly used magnetic field source in labs

Finite element simulations:
lines and contour maps of induction B



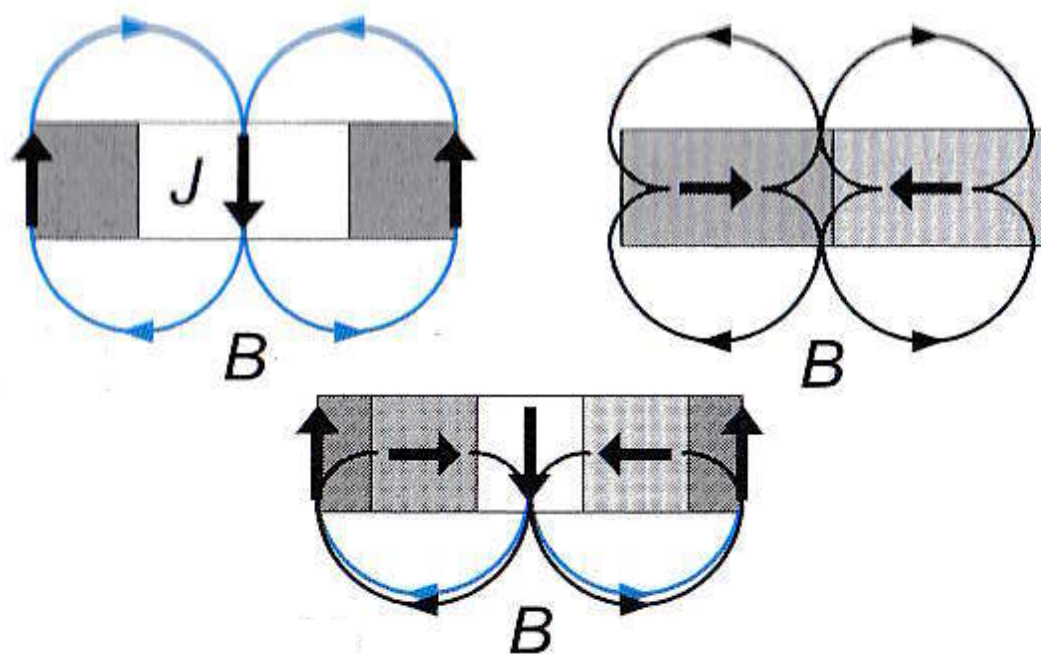
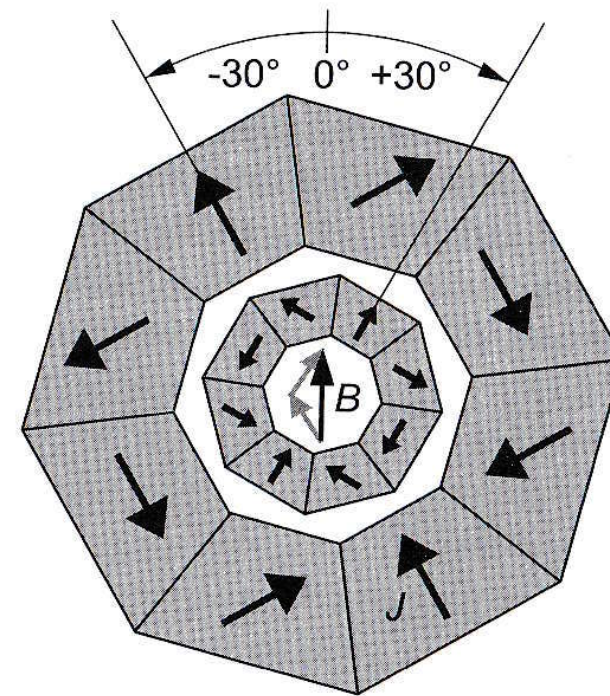
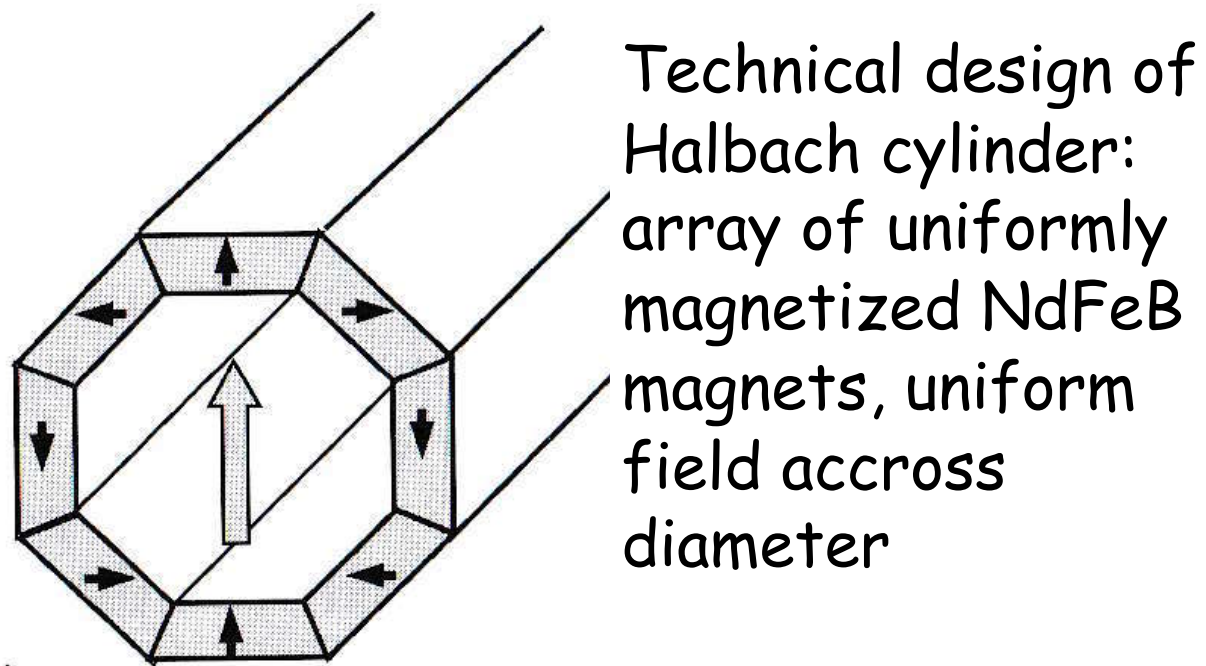
The larger gap, the smaller field.

Courtesy F. Fiorillo

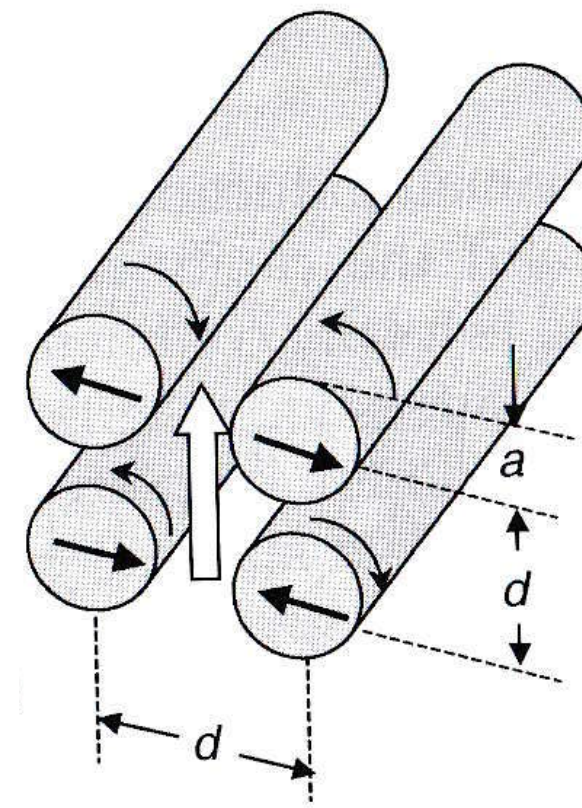
1. Production of Magnetic Field

1.3 Permanent Magnets

Fields up to $\sim 2\text{T}$ by appropriate arrangement of permanent magnets



One-sided field by superposition



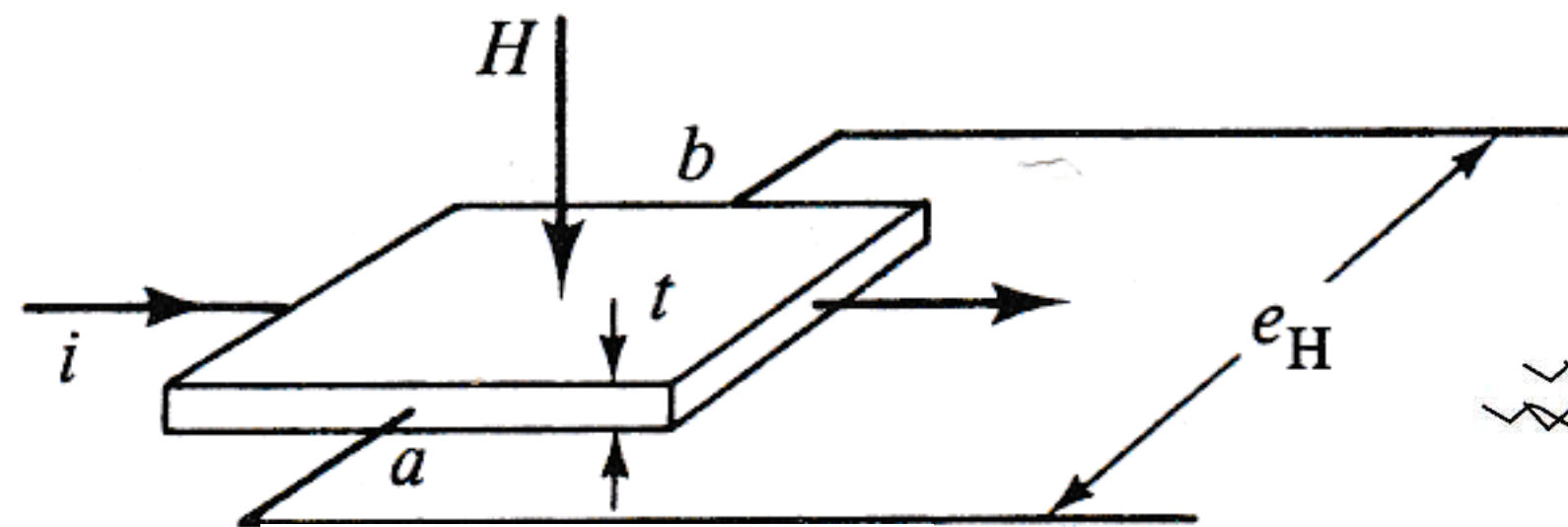
Simplification of above array: continuously varying field by counter rotation of 4 transversely magnetized rods

2.

Measurement of Magnetic Field Strength

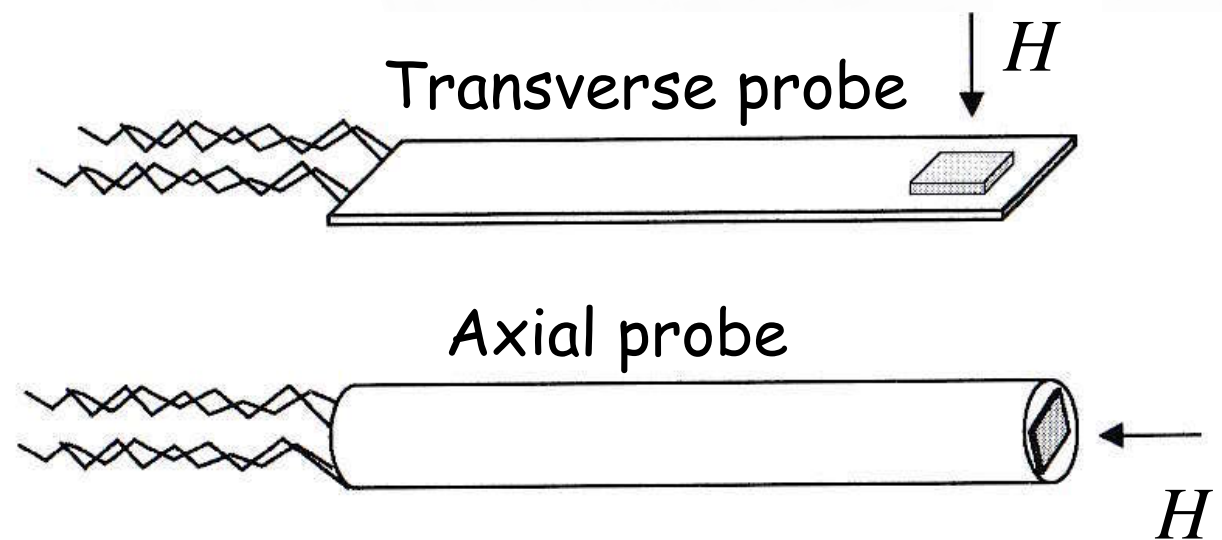
2. Measurement of magnetic field strength

2.1 Hall Probe



$$e_H = R_H \frac{iH}{t} \text{ volt}$$

Hall coefficient



- Hall probe = plate made of InSb or GaAs semiconductor
- H -field perpendicular to plate distorts current path and emf e_H is developed between a and b
- Multirange instruments, sensitive to field range from μT to 3T
- Uncertainty in field reading: 1-5% in hand-held Gaussmeters
- Alternating fields up to some 10 kHz can be measured
- Calibration by accurately known fields required
- Low-field probes: zero must be set with probe in magnetically shielded cylinder to eliminate Earth's field



Gaussmeter

2. Measurement of magnetic field strength

2.2 Fluxmeter

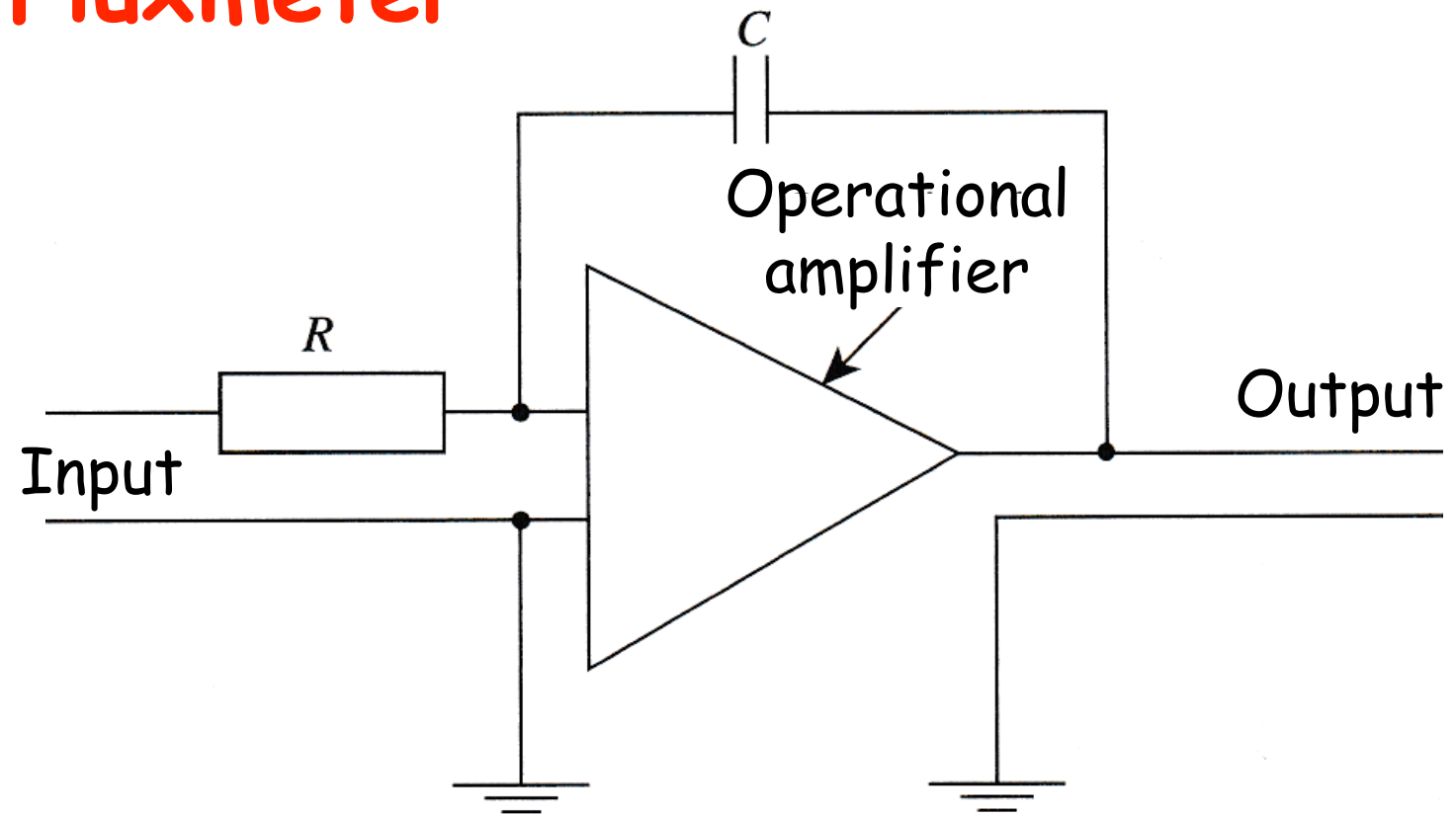
- Faraday's law: a changing magnetic flux φ through a coil of N turns generates a voltage in coil proportional to rate of change of flux:

$$U(t) = -N \frac{d\varphi}{dt} \quad [\text{Volt}]$$

$$U(t) dt = -N d\varphi$$

$$\int_0^t U(t) dt = -N \int_{\Phi_1}^{\Phi_2} d\varphi = -N \Delta\varphi$$

$$U_{\text{out}} = -1/RC \int U_{\text{in}} dt$$



- Instrument to integrate voltage from **pick-up coil** is called fluxmeter
= electronic integrator (based on capacitive feedback around operational amplifier) that provides voltage output
- With $B = \varphi/A$ (flux density in pick-up coil of cross section A):

$$\int U(t) dt = -N A \Delta B \quad [\text{Vs}]$$

Fluxmeter measures
changes in flux density

2. Measurement of magnetic field strength

2.2 Fluxmeter

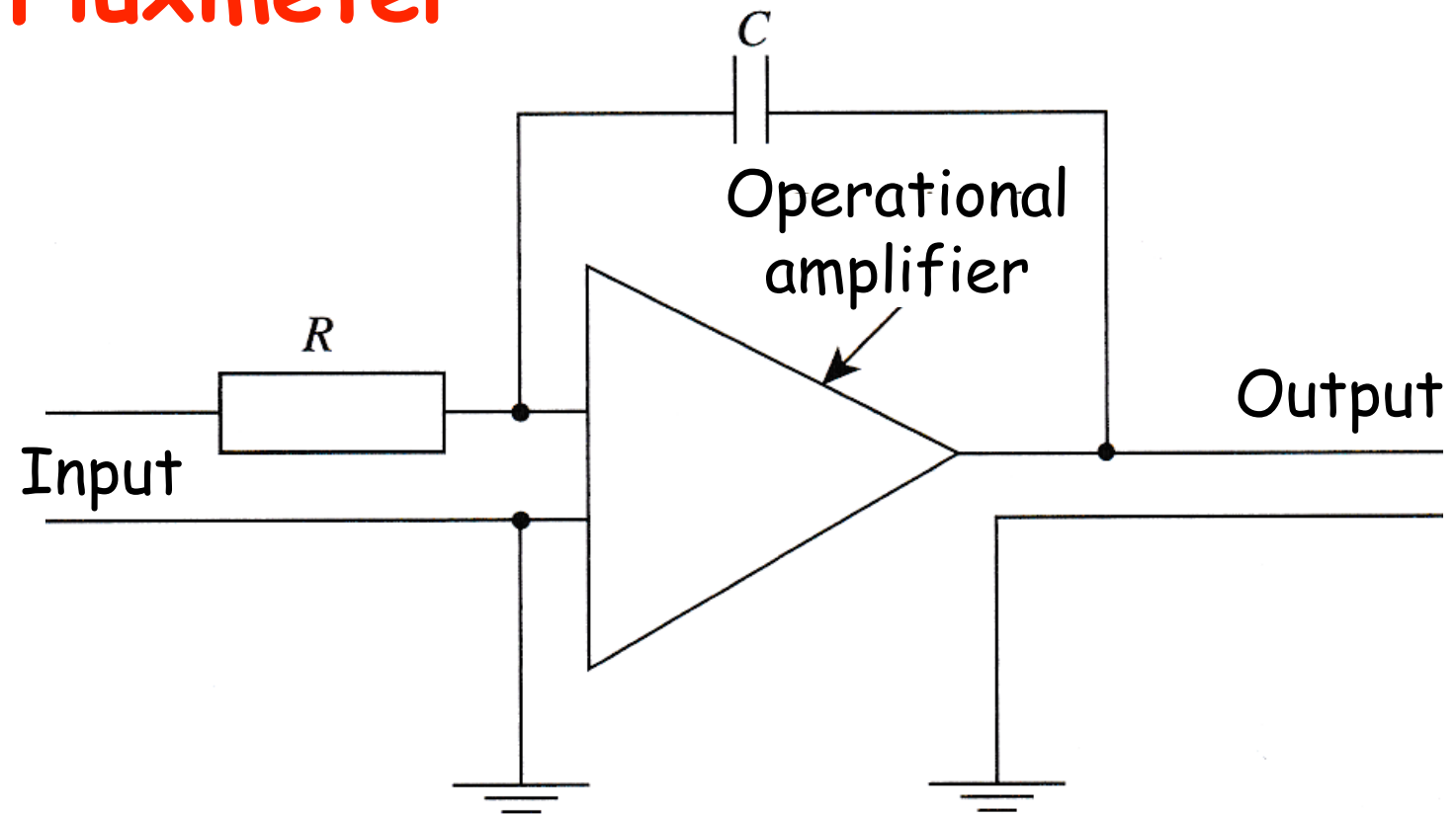
- Faraday's law: a changing magnetic flux φ through a coil of N turns generates a voltage in coil proportional to rate of change of flux:

$$U(t) = -N \frac{d\varphi}{dt} \quad [\text{Volt}]$$

$$U(t) dt = -N d\varphi$$

$$\int_0^t U(t) dt = -N \int_{\Phi_1}^{\Phi_2} d\varphi = -N \Delta\varphi$$

$$U_{\text{out}} = -1/RC \int U_{\text{in}} dt$$



- Instrument to integrate voltage from **pick-up coil** is called fluxmeter
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- With $B = \varphi/A$ (flux density in pick-up coil of cross section A):

$$\int U(t) dt = -N A \Delta B \quad [\text{Vs}]$$

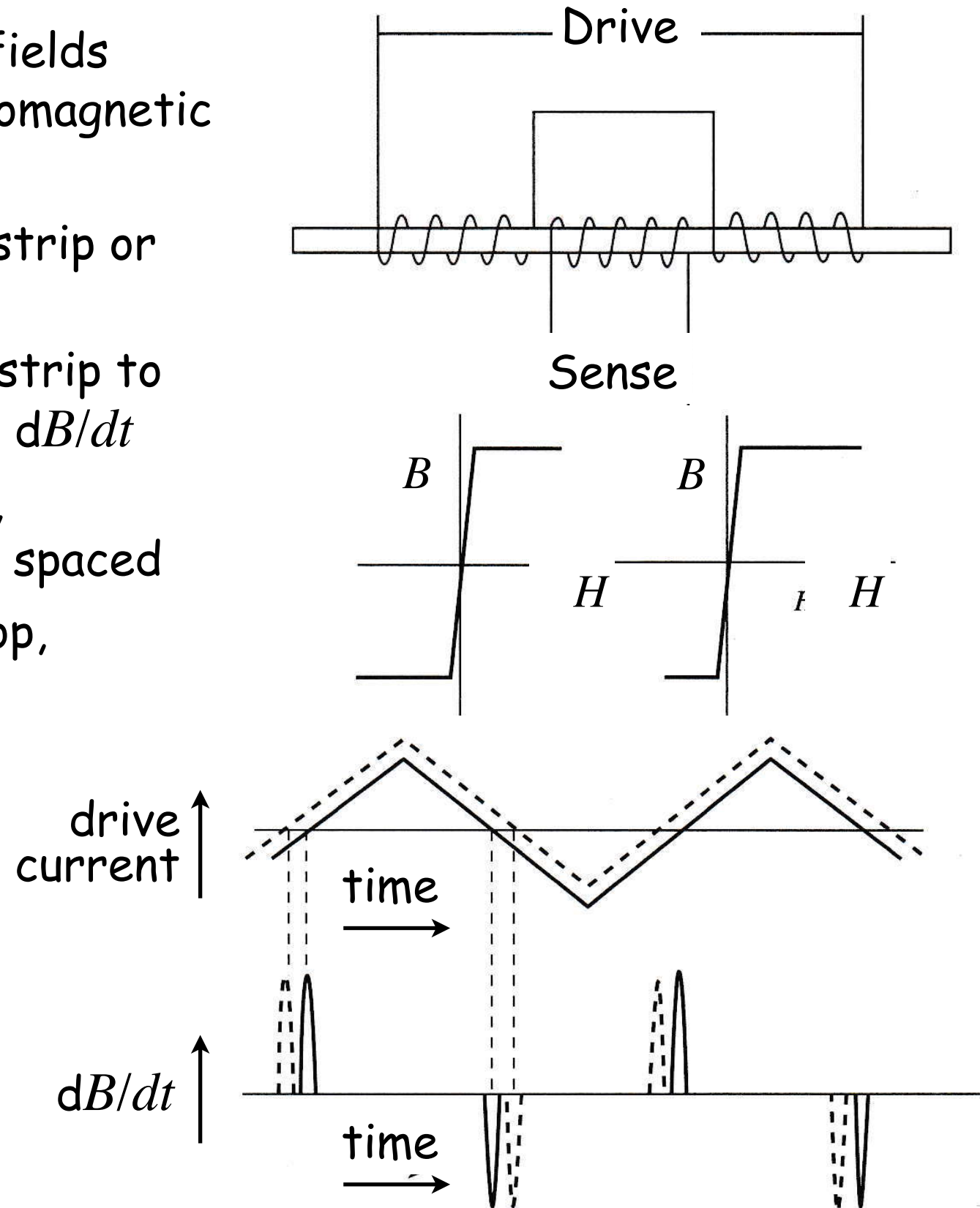
Fluxmeter measures
changes in flux density

Measurement of
constant field:
search coil must be
moved to zero-field
region, or rotated
through 180°

2. Measurement of magnetic field strength

2.3 Fluxgate Magnetometer

- Instrument for accurate measurement of fields comparable to Earth's field (used in e.g. geomagnetic and archeological surveys)
- Principle: high-permeability soft magnetic strip or wire with drive and sense coil
- AC (triangular) current in drive coil drives strip to pos. and neg. saturation, sense coil picks up dB/dt
- No external dc field: symmetric $B(H)$ -loop, positive and negative voltage pulses equally spaced
- With dc field H along strip: asymmetric loop, pulses unevenly spaced
- For triangular drive field: change in pulse spacing is direct measure for dc field
- Typical field range: 0.1 mT



2. Measurement of magnetic field strength

2.4 Magnetic Potentiometer (Rogowski-Chattok coil)

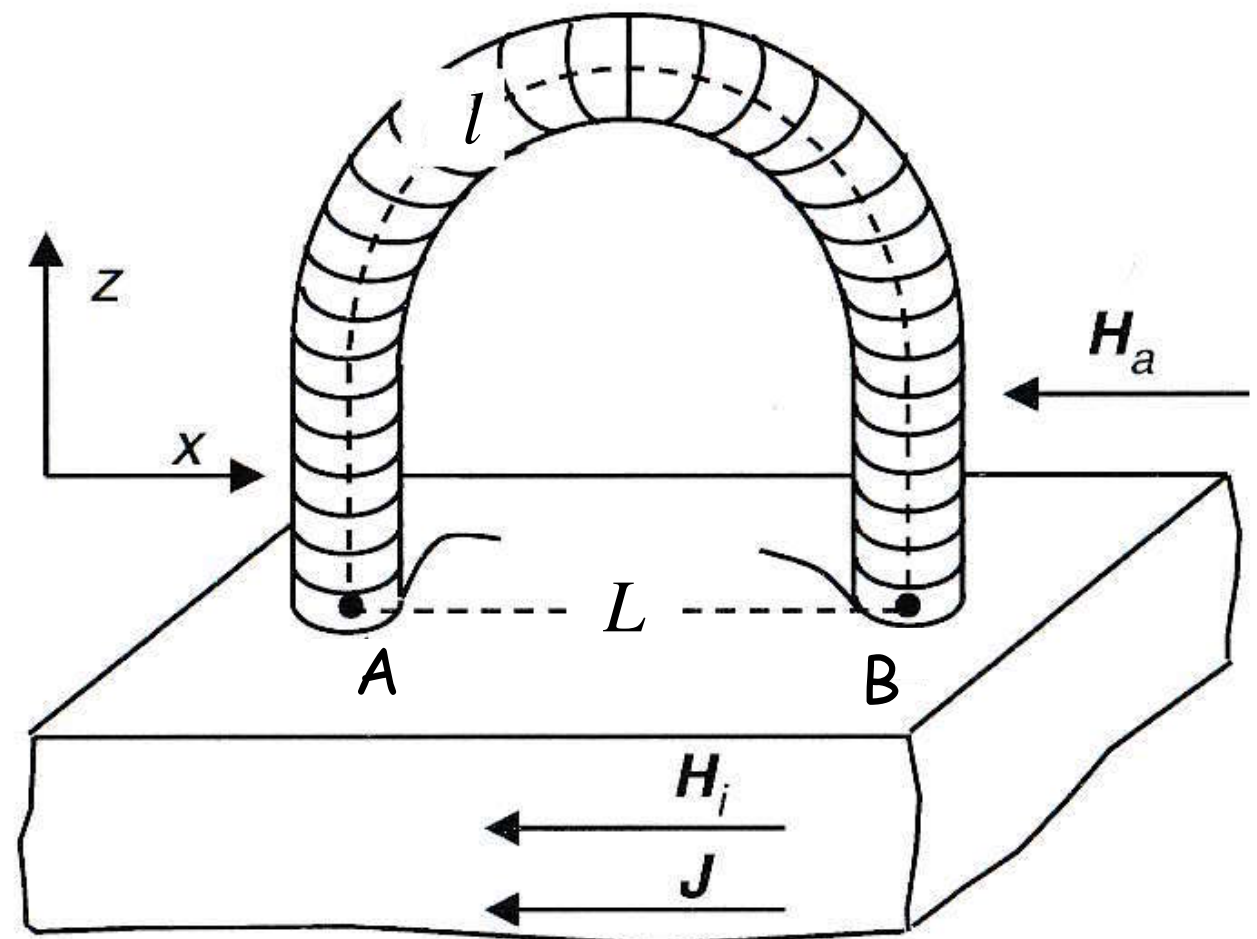
- Tightly wound coil whose ends lie in same plane
- Field at surface of magnetic sample is the same as internal field
- If no current flows in coil: line integral $\oint H dl$ around dotted path must be zero:

$$\int_A^B H dl + \int_B^A H dL = 0$$

- If field is uniform along L :

$$\int_B^A H dL = H \Delta L = - \int_A^B H dl$$

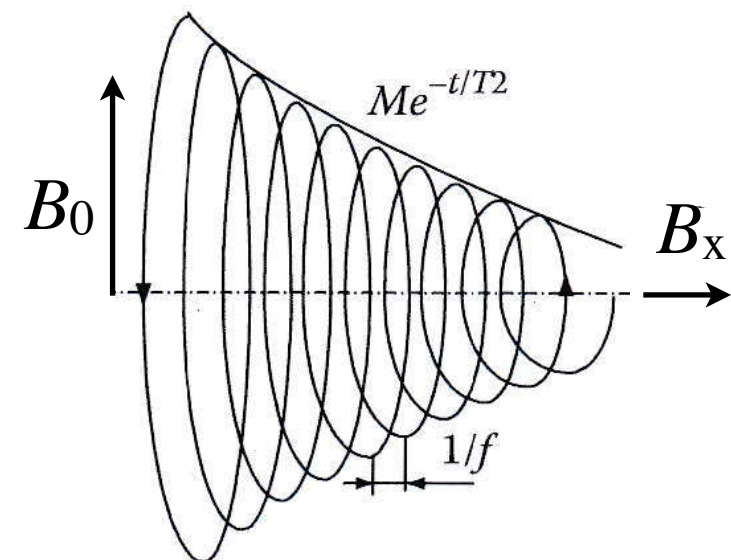
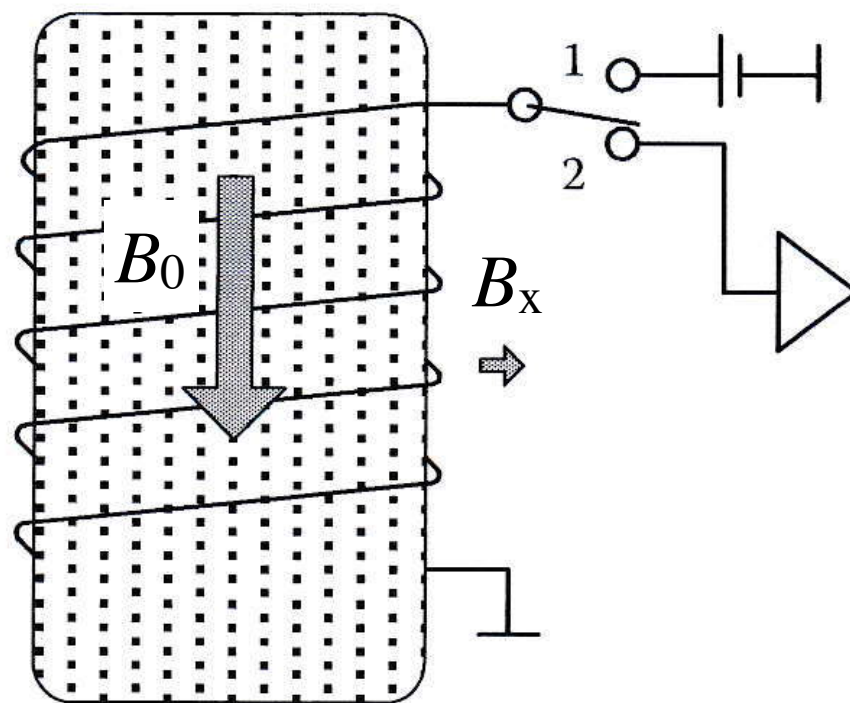
- If output of coil is connected to integrator: the integrated voltage is proportional to change of H along the line L
- Is used to measure magnetic field in flux-closing yokes (like single sheet testers for electrical steel)



2. Measurement of magnetic field strength

2.5 Proton Precession Magnetometer

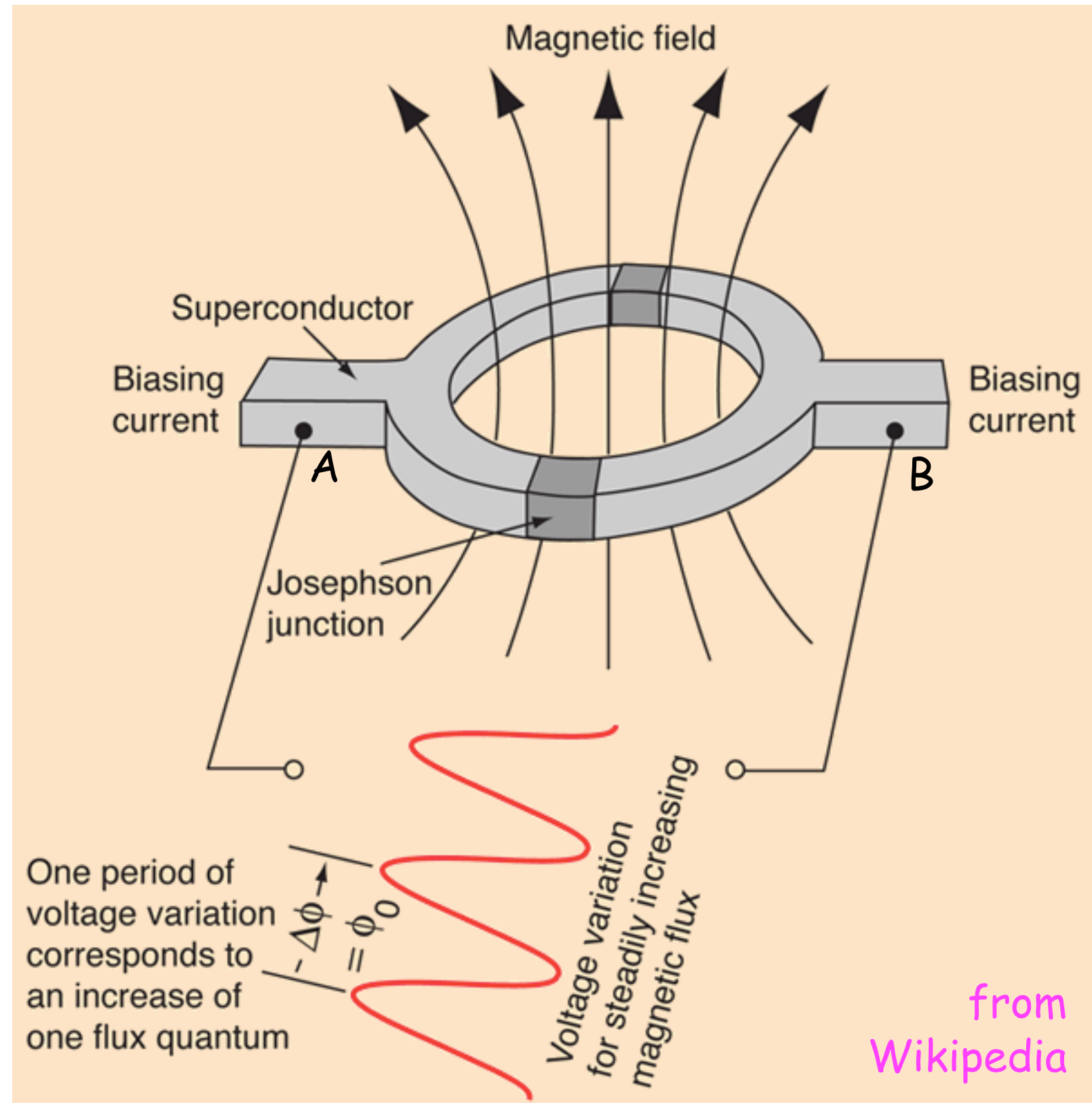
- High-precision measurement of weak magnetic field (like Earth's field, with uncertainty 1 ppm), also used for calibration
- Relies on Nuclear Magnetic Resonance (NMR): applying magnetic field \rightarrow magnetic moment of nucleus rotates in selected quantum directions with resonance frequency which strictly depends on value of field
- To measure weak magnetic field (like Earth's field):
 - (1) sample (e.g. 1 liter of water) is exposed to strong dc magnetic field B_0 (~ 10 mT) perpendicular to the measured field B_x . B_0 aligns certain fraction of proton moments along coil direction
 - (2) B_0 switched-off \rightarrow magnetic moments of protons align along B_x by decaying precession movement. Frequency of precession is measured by measuring frequency of induced voltage in coil. Frequency depends precisely on B_x .
 - Typical precessional frequency: a few kilohertz



2. Measurement of magnetic field strength

2.6 SQUID Magnetometer

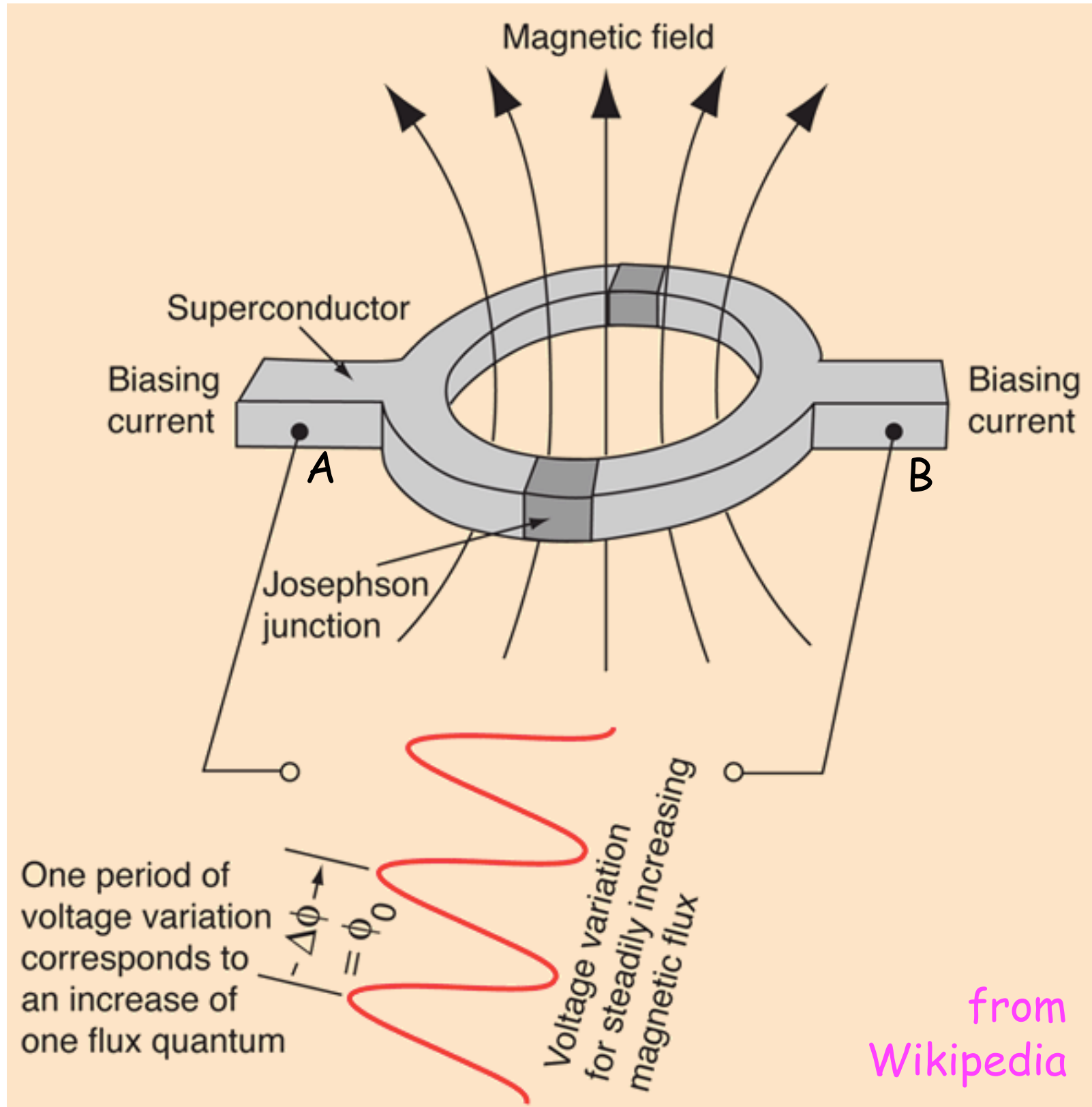
- SQUID: Superconducting Quantum Interference Device magnetometer
- Based on tunneling of superconducting electrons across narrow insulating gap, called Josephson junction
- Ring-shaped device, superconducting current from A to B, equal currents pass through each junction
- Changing magnetic flux through ring: induces „screening“ current in ring (Faraday's law) which generates magnetic field that cancels external flux. Induced current adds to measuring current in one junction, subtracts in other



2. Measurement of magnetic field strength

2.6 SQUID Magnetometer (cont.)

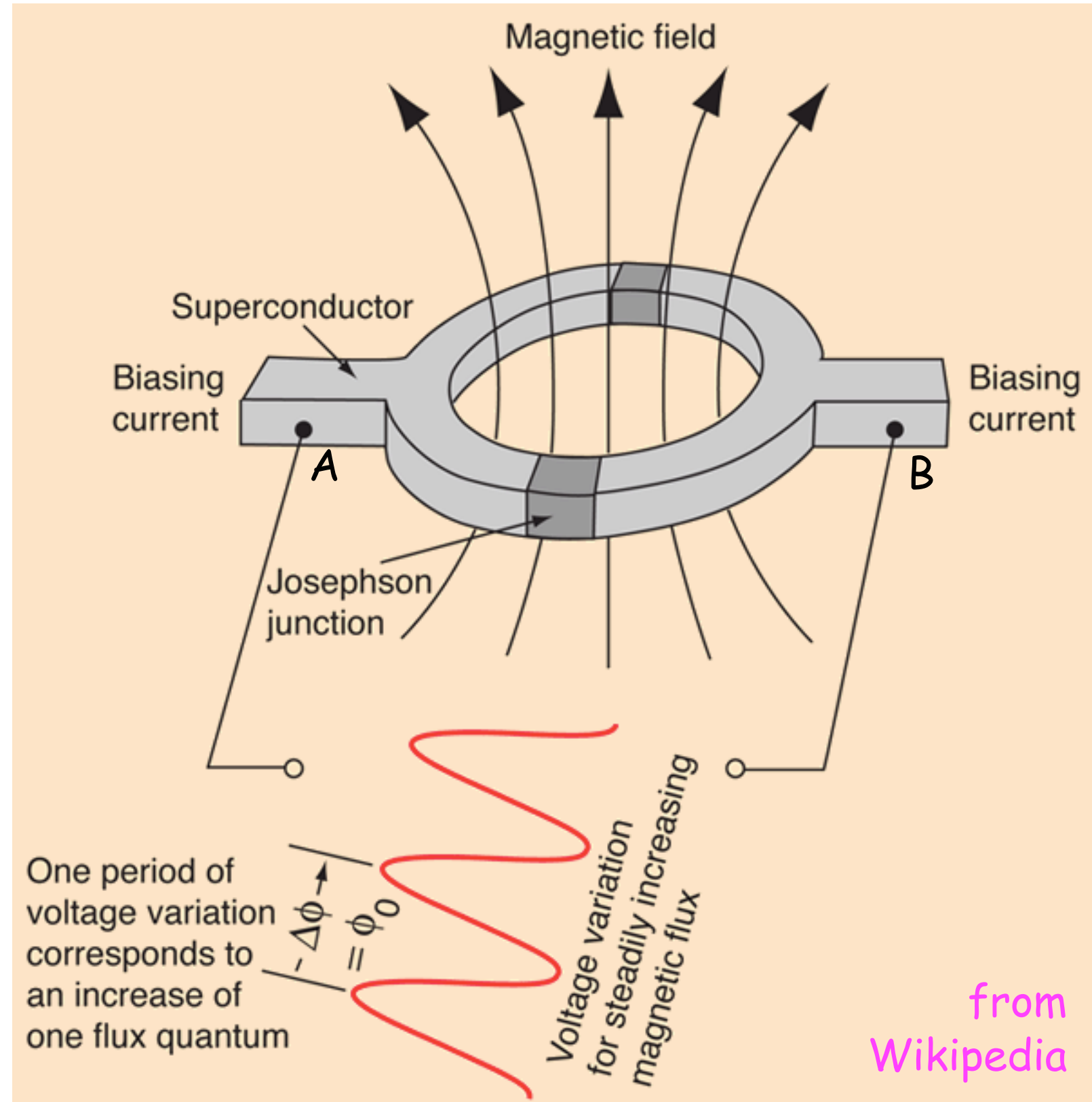
- As soon as current in either branch exceeds the critical current of the Josephson junction, a voltage appears across the junction
- In superconducting ring the magnetic flux is quantized, i.e because of wave nature of superconducting current (quantummechanics) the flux enclosed by the ring must be an integer number of the flux quantum $\Phi_0 = h/2e$



2. Measurement of magnetic field strength

2.6 SQUID Magnetometer (cont.)

- Now suppose the external flux is further increased until it exceeds $\Phi_0/2$. Since the flux enclosed by the loop must be integer number of flux quanta, instead of screening the flux the SQUID now energetically prefers to increase it to Φ_0 . The screening current now flows in the opposite direction.
- → screening current changes direction every time the flux increases by half integer multiples of Φ_0
- critical current oscillates as a function of the applied flux
- voltage between A and B is function of applied magnetic field and a period equal to Φ_0

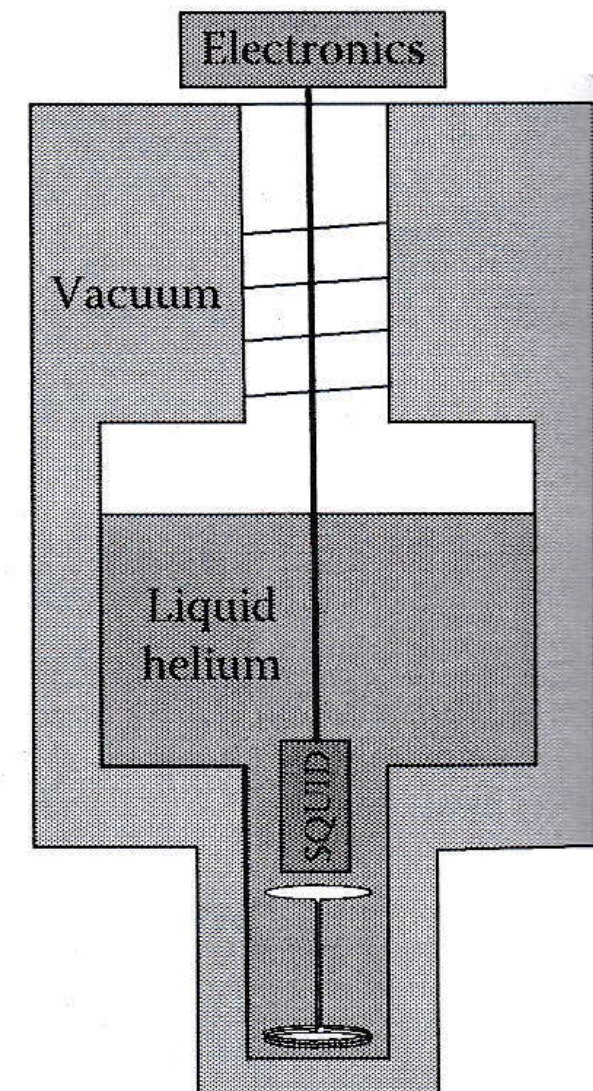
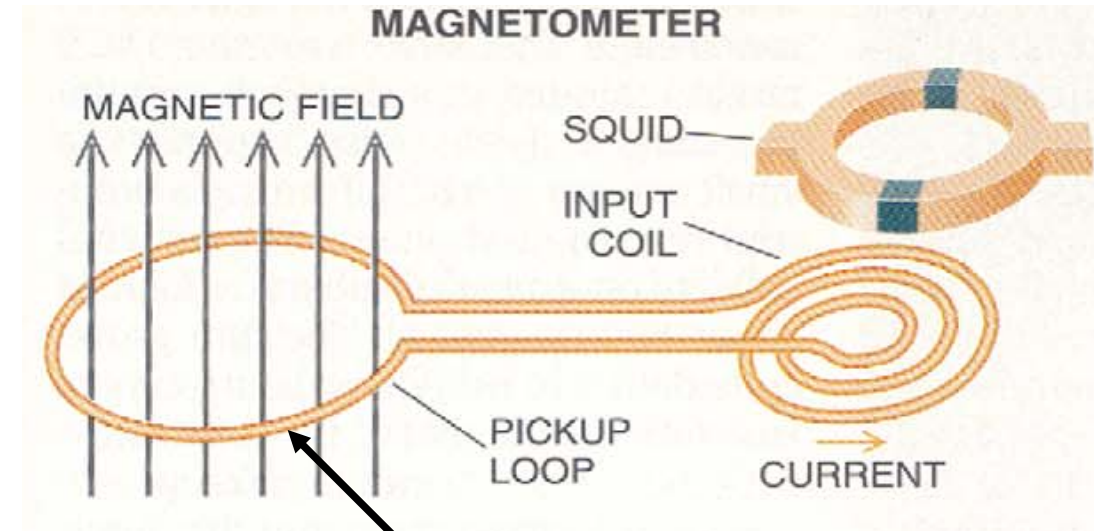


from
Wikipedia

2. Measurement of magnetic field strength

2.6 SQUID Magnetometer (cont.)

- In practise: SQUID is not directly contacted with magnetic source, device is rather linked to transformer coil to measure flux from small sample, i.e. sample magnetization
- SQUID magnetometer is high-sensitivity static fluxmeter
- Sensitivity: femto- to pico-Tesla
- Since SQUID requires low-T operation, it is usually used in conjunction with superconducting coil



3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

3.4 Dilatometric measurements

3.5 Domain methods

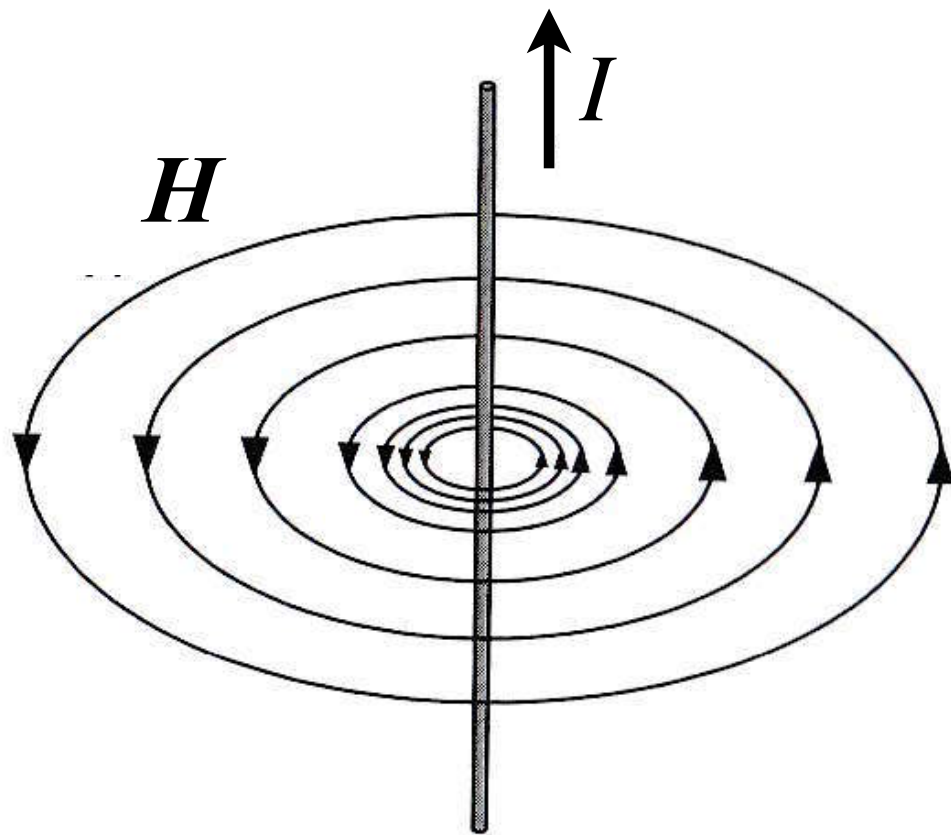
3. Magnetic Measurement ...

General aspects

Magnetic field H

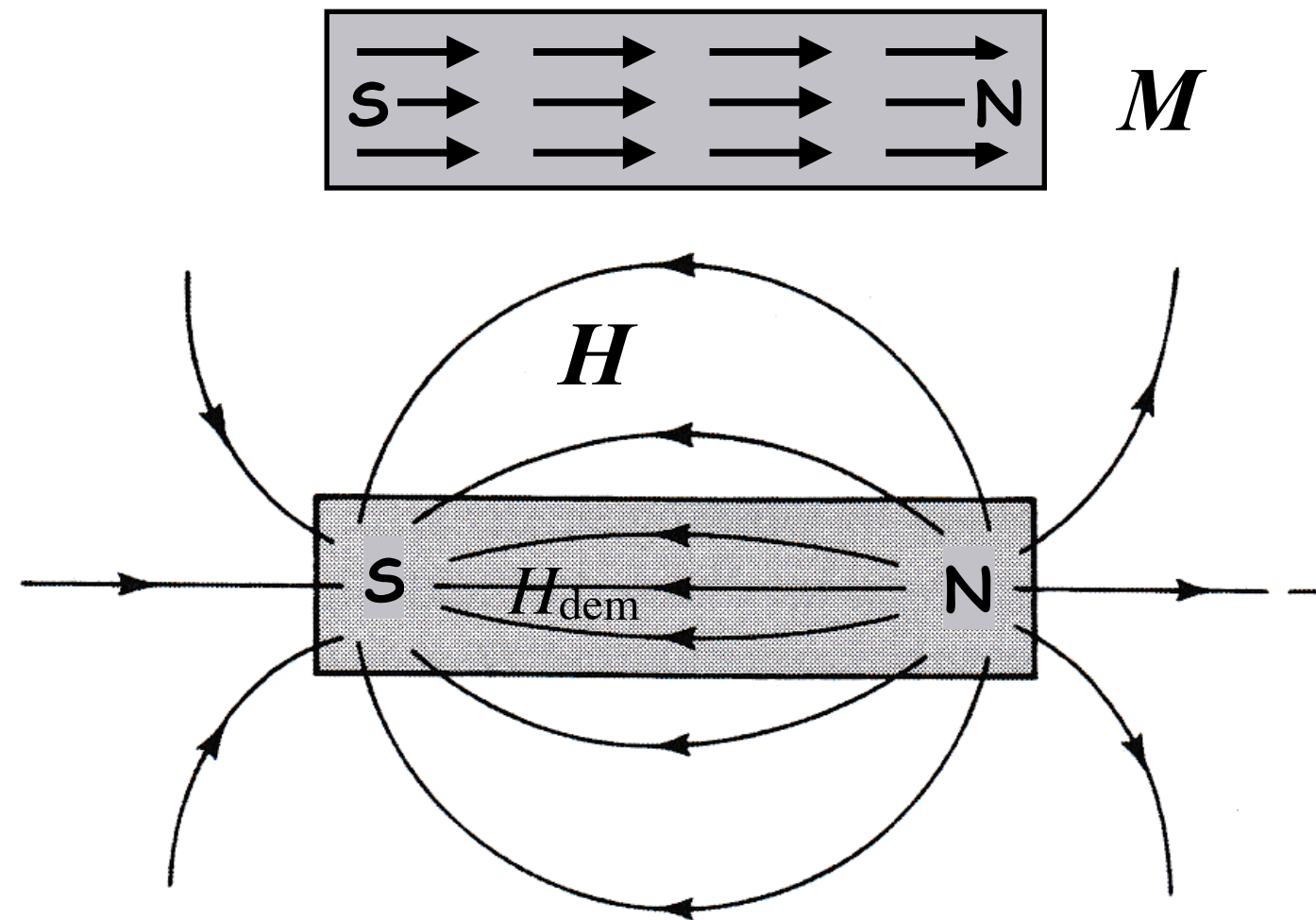
Field produced by currents:

Lines of H are continuous and form closed loops



Field produced by magnetic poles:

Lines of H begin at north poles and end at south poles (here: $H_{\text{applied}} = 0$)



Demagnetizing field $H_{\text{dem}} = -N\bar{M}$: acts opposite to magnetization M that creates it

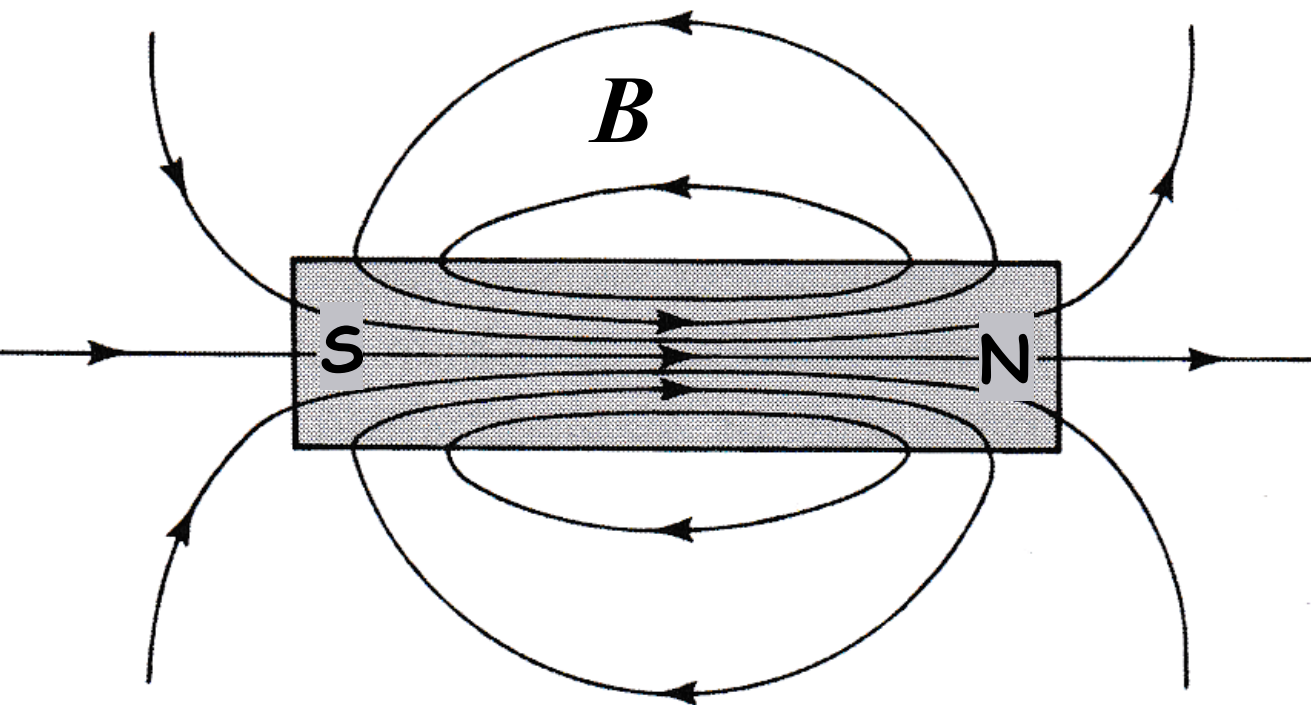
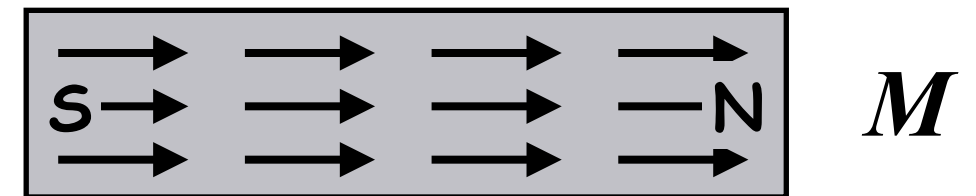
3. Magnetic Measurement ...

General aspects

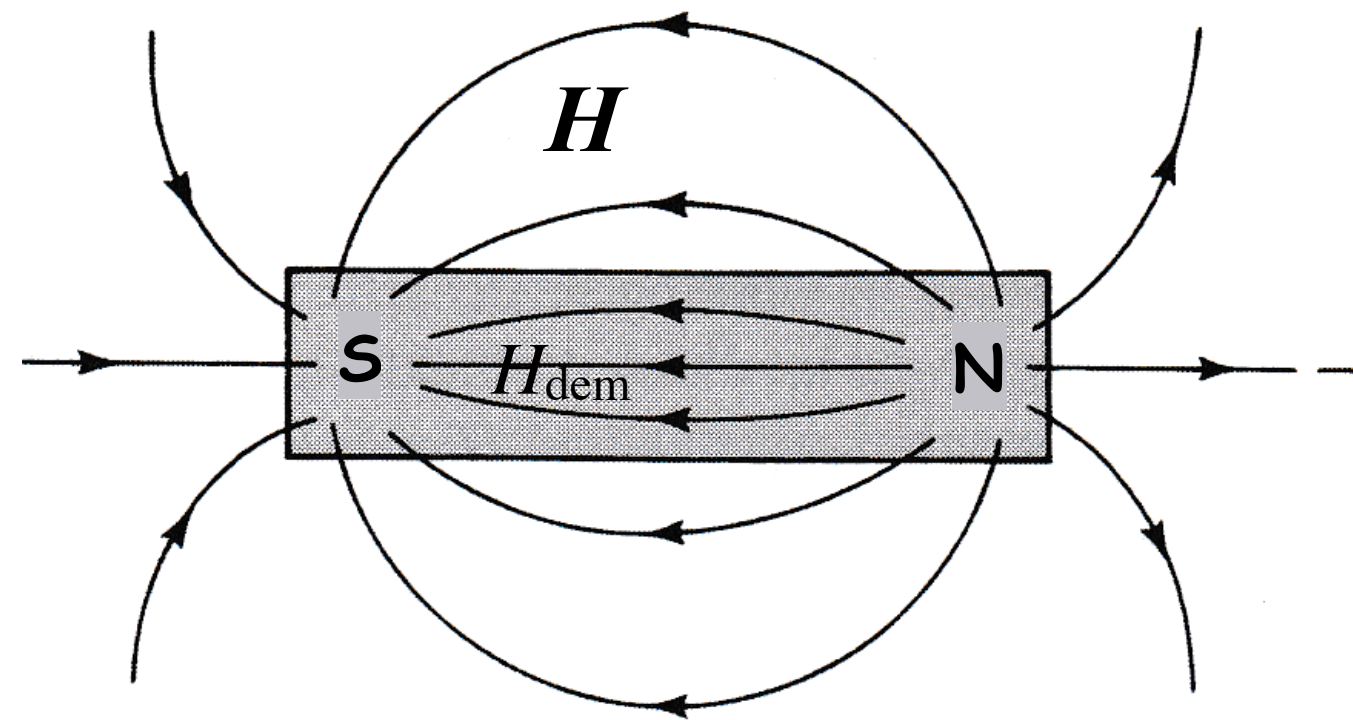
Magnetic field H and Induction B

- $B = \mu_0 (H + M) = \mu_0 (H_{\text{applied}} - H_{\text{dem}} + M)$
- If $H_{\text{applied}} = 0$: H_{dem} is only field acting, and $B = -\mu_0 H_{\text{dem}} + \mu_0 M$
- $\mu_0 H_{\text{dem}}$ can never exceed $\mu_0 M$, i.e. flux density B inside magnet is always smaller than $\mu_0 M$, but in same direction

Field produced by magnetic poles:
Lines of H begin at north poles and end at south poles (here: $H_{\text{applied}} = 0$)



- Lines of B are continuous, from S to N inside magnet, outside $\mu_0 H = B$



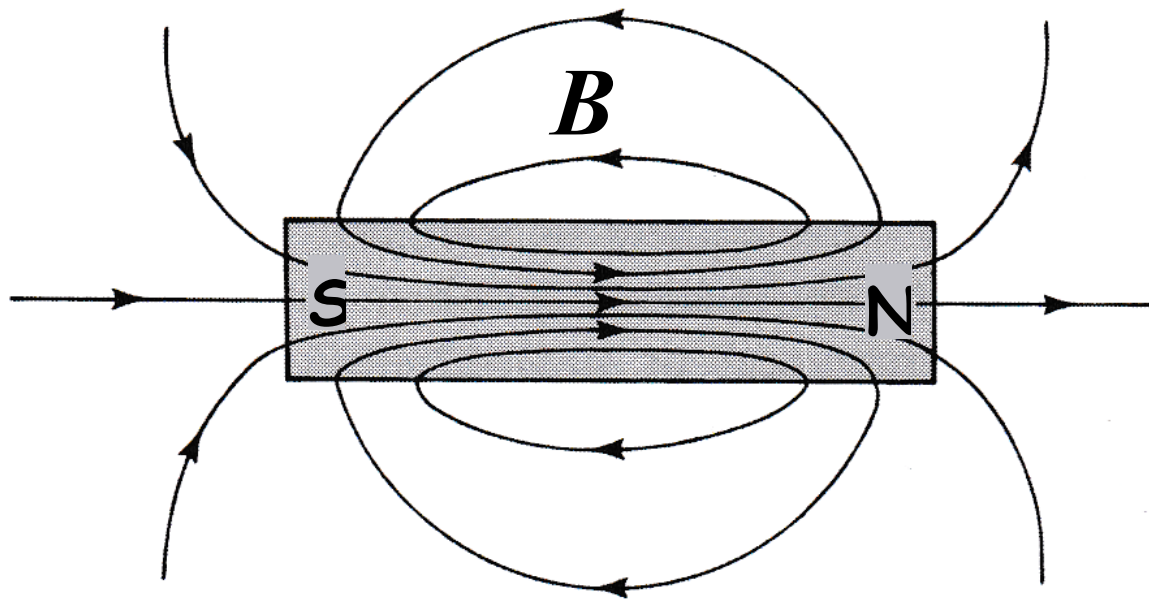
Demagnetizing field $H_{\text{dem}} = -N\bar{M}$: acts opposite to magnetization M that creates it

3. Magnetic Measurement ...

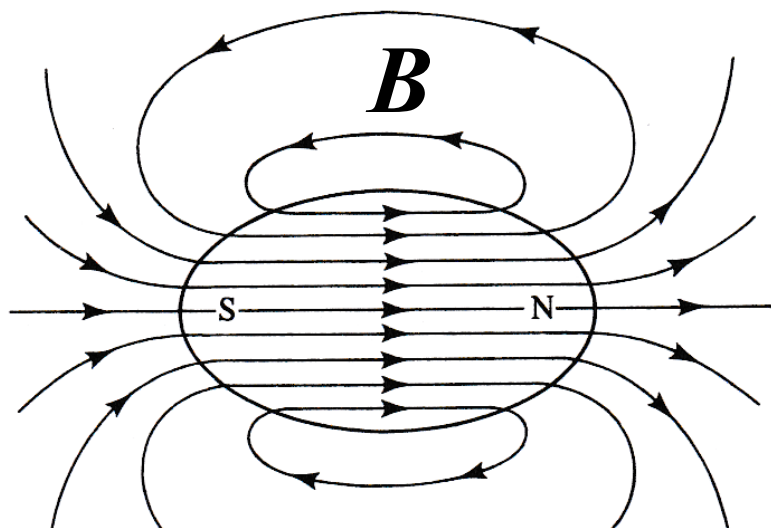
General aspects

Magnetic field H and Induction B

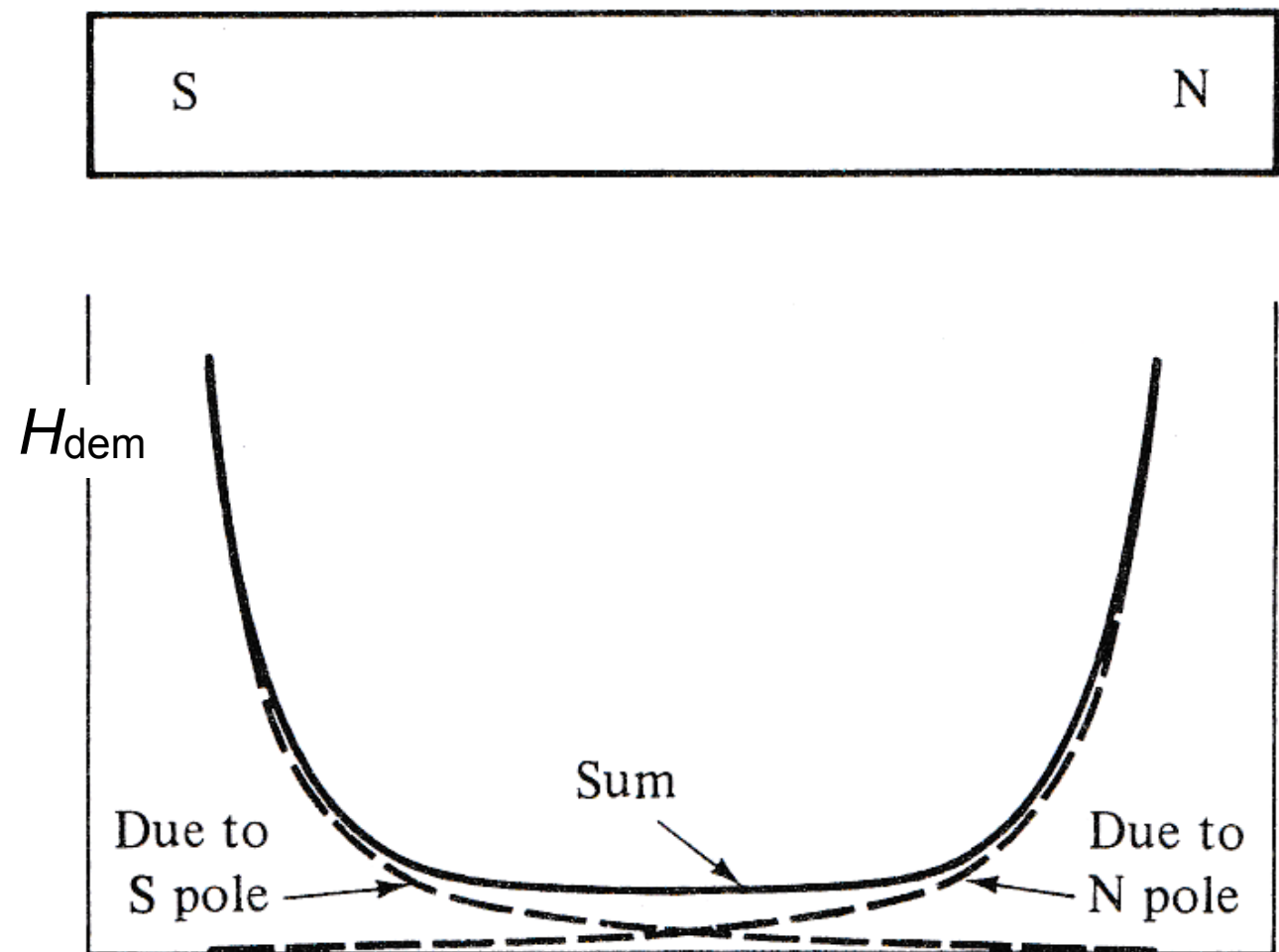
- Flux density of bar magnet is not uniform: B -lines diverge towards the ends \rightarrow flux density is less than in center



- Exception: rotational ellipsoid:



- Reason:
 H_{dem} is stronger near the poles

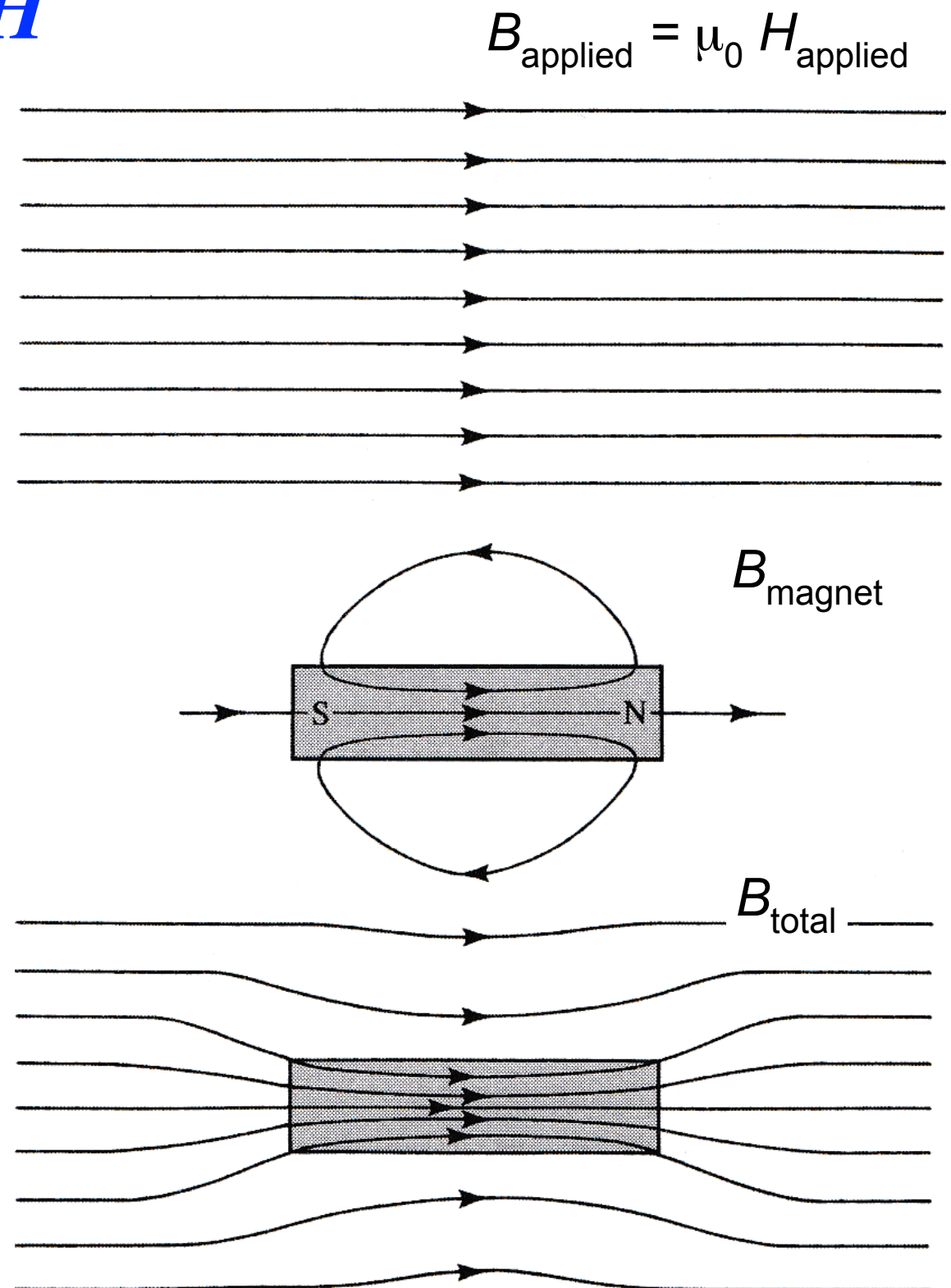


3. Magnetic Measurement ...

General aspects

Finite sample in applied field H

- When soft magnetic body is placed in field, it alters shape of field.
- This is demonstrated by assuming a fully magnetized bar magnet. The total field B_{total} is the vector sum of applied field B_{applied} and B -field of magnet
- The flux tends to crowd into the magnet, as though it were more permeable than surrounding air (\rightarrow term „permeability“). At points outside the magnet: field is reduced
- The same result is obtained if the body that is placed in the field is originally unmagnetized, because the field itself will produce magnetization (for material with $\mu \gg 1$)
- For strongly magnetic materials the disturbance of field is considerable



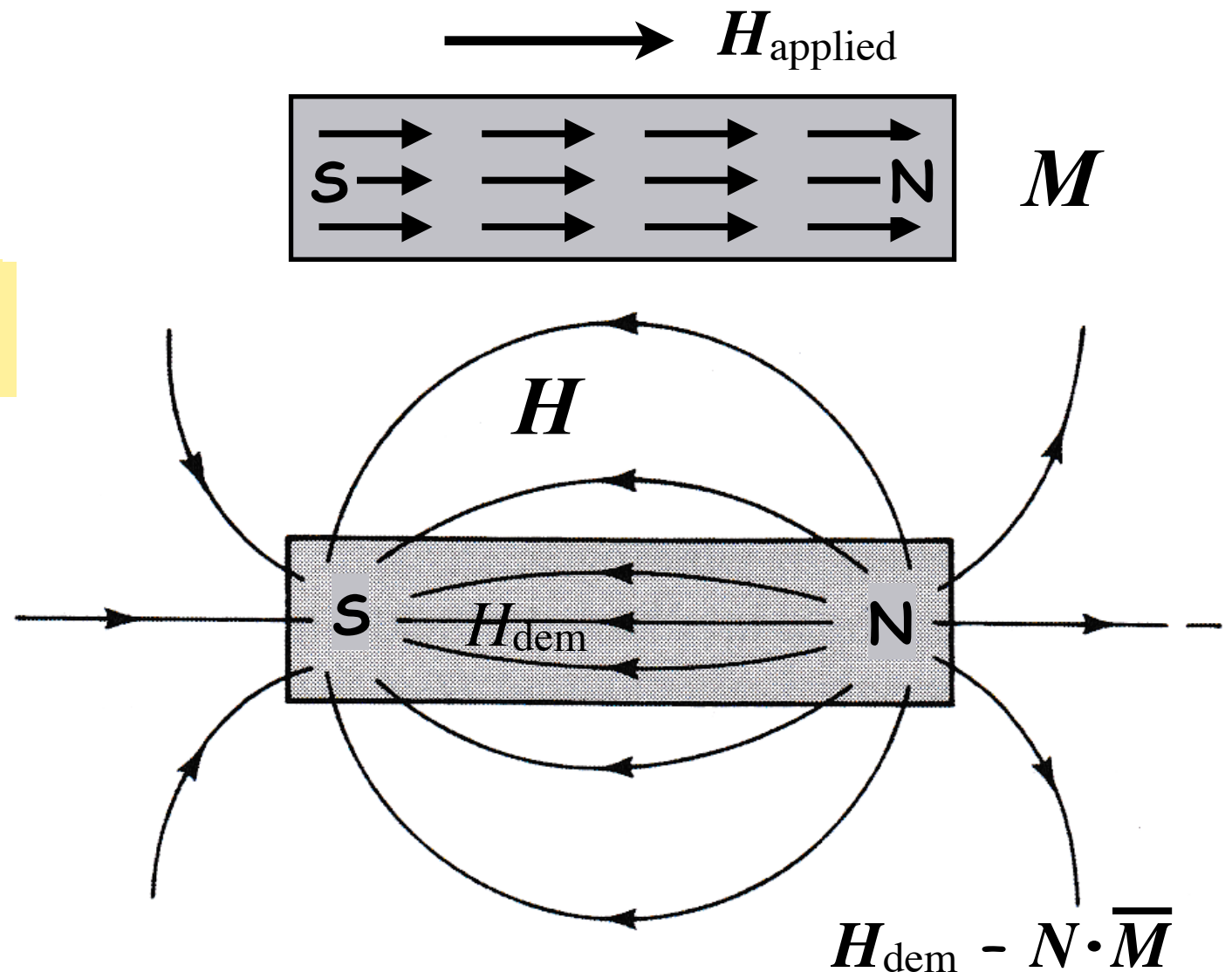
3. Magnetic Measurement ...

General aspects

Closed and open samples

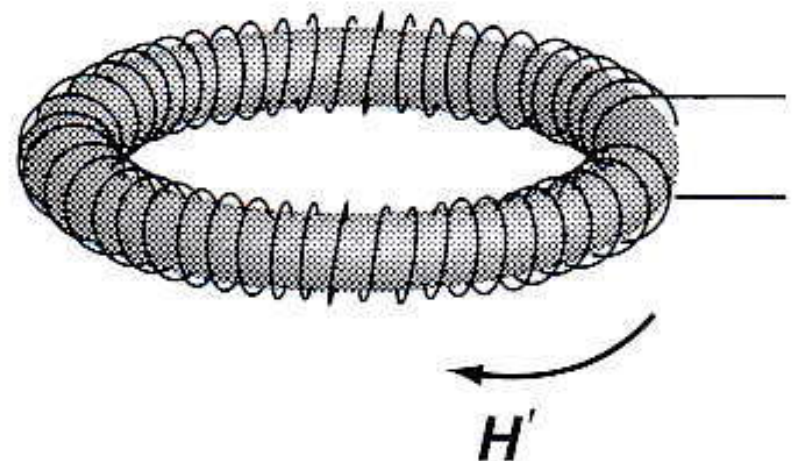
- Open sample

$$\text{Internal field: } H_{\text{in}} = H_{\text{applied}} - N \cdot \bar{M}$$



- Closed sample

$$\text{Internal field: } H_{\text{in}} = H_{\text{applied}} \quad , N = 0$$

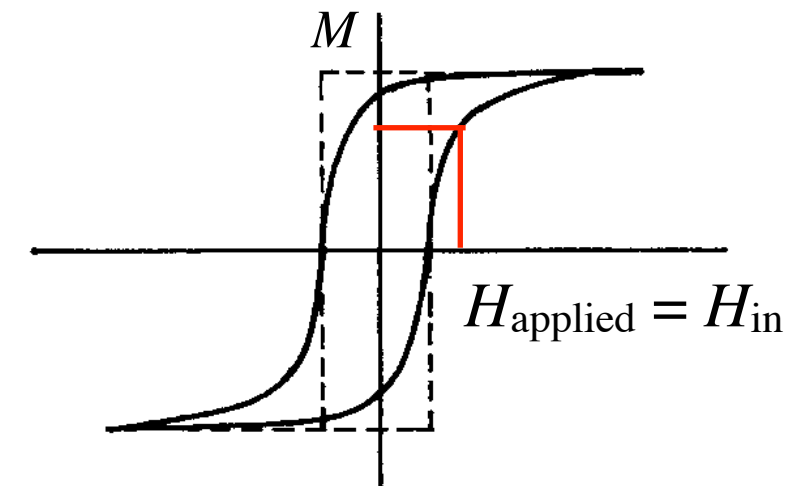
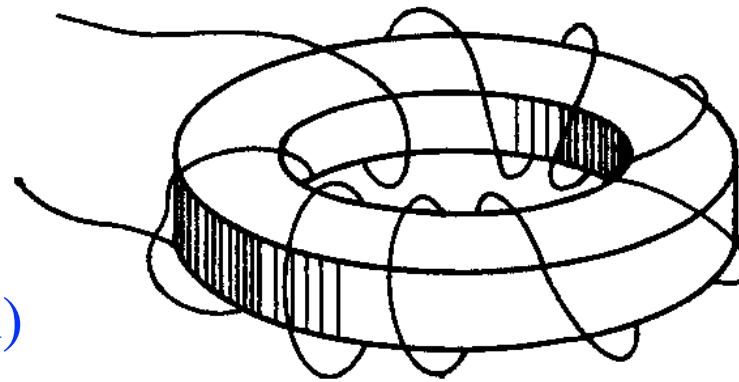
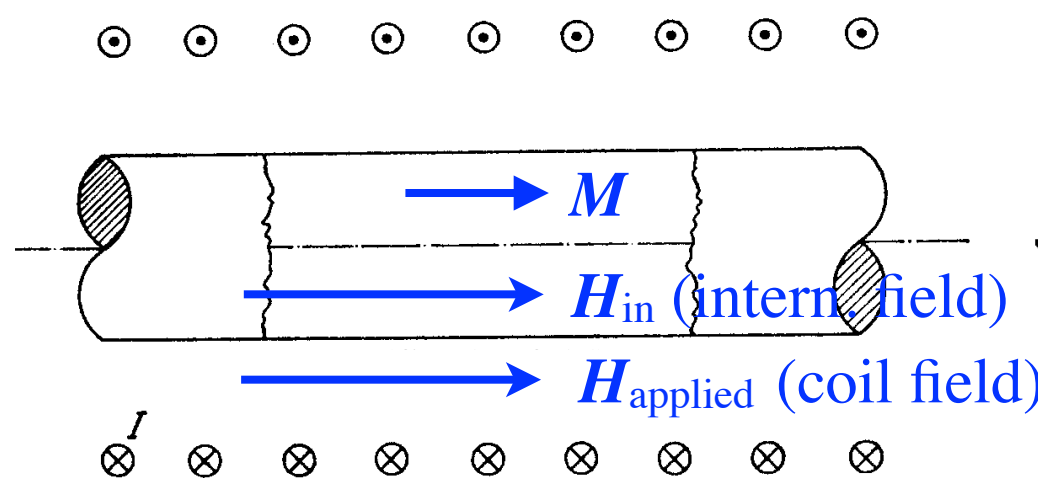


3. Magnetic Measurement ...

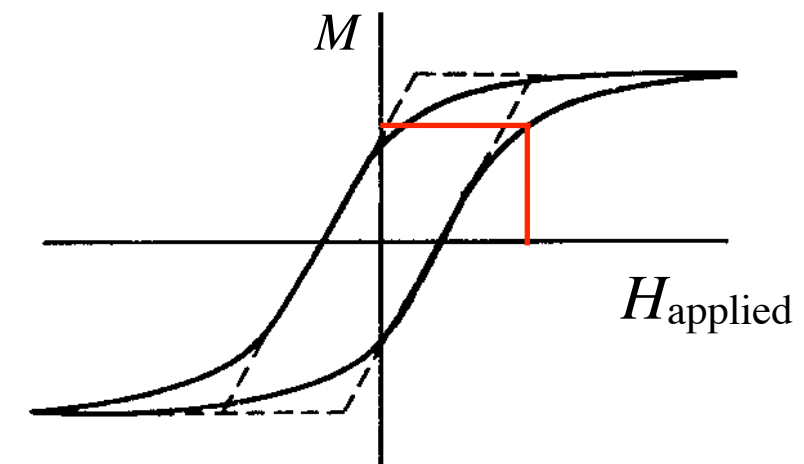
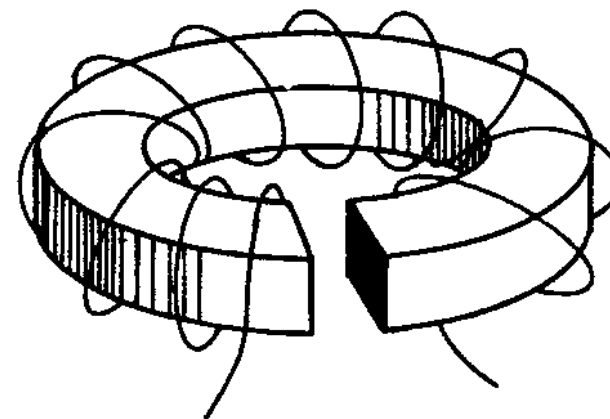
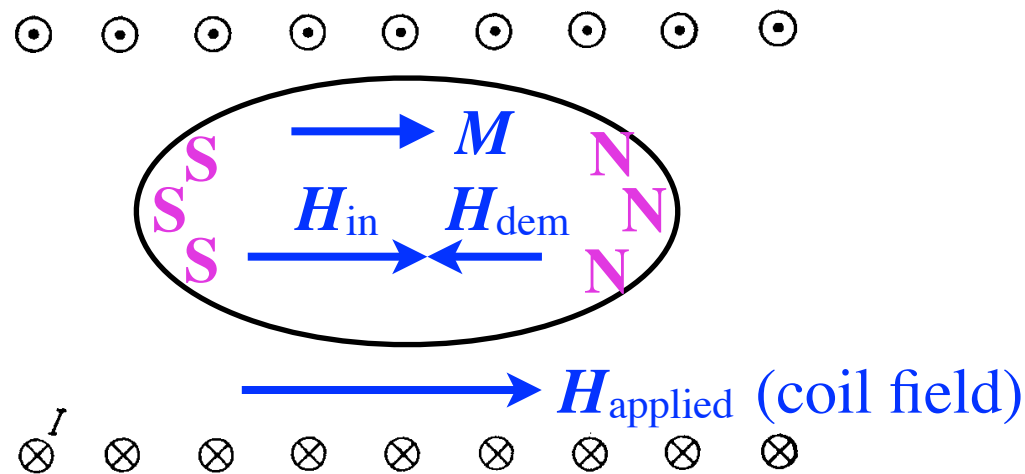
General aspects

Closed and open samples

Demagnetization effect
→ Shearing of magnetization curve



Infinite sample or closed ring: unsheared hysteresis curve: $N = 0$, i.e. $H_{in} = H_{ext}$



Finite sample or open core: sheared hysteresis curve due to demagnetization effect (a higher $H_{applied}$ is needed to achieve a given degree of M)

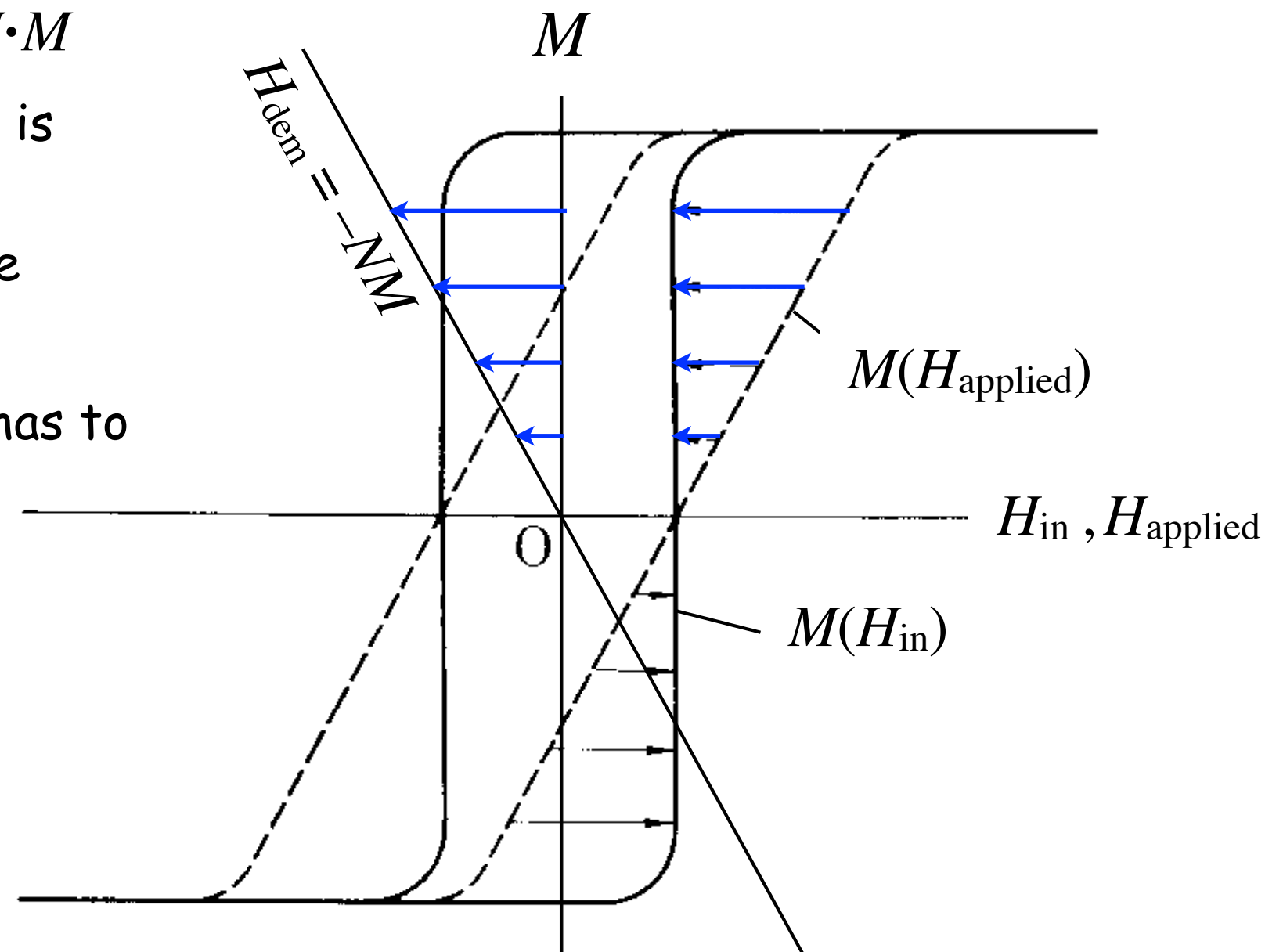
3. Magnetic Measurement ...

General aspects

Closed and open samples

Demagnetization effect
→ Shearing of magnetization curve

- Internal field: $H_{\text{in}} = H_{\text{applied}} - N \cdot M$
- Relevant for magnetic materials is the $M(H_{\text{in}})$ -curve, as it is independent of the sample shape
- If a magnetization curve was measured on a finite sample, it has to be re-sheared.

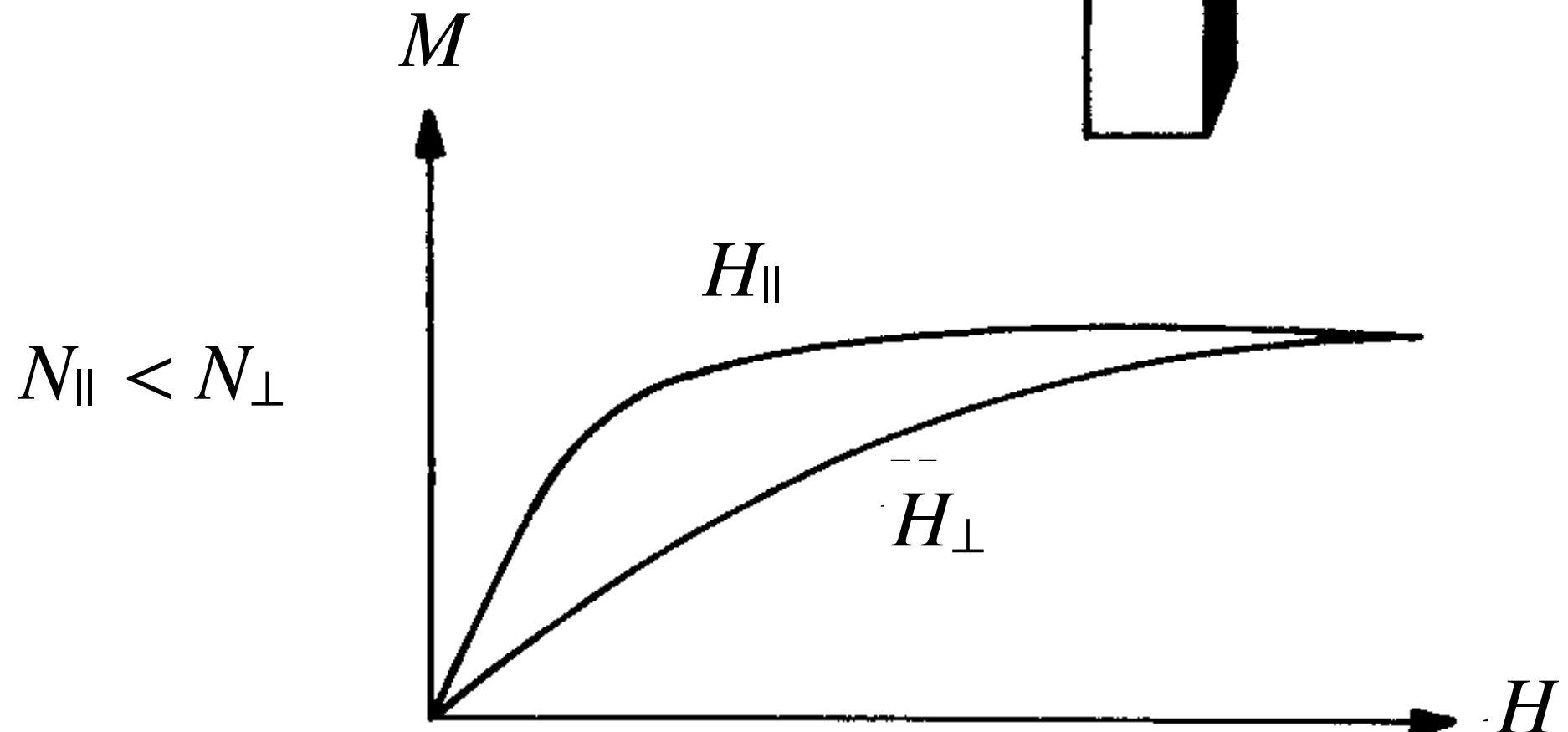
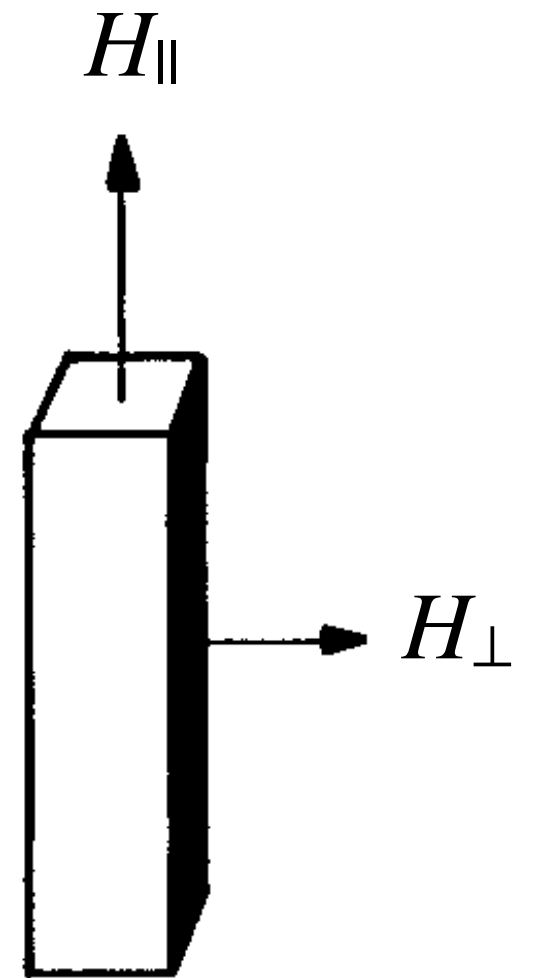


3. Magnetic Measurement ...

General aspects

Influence of demagnetizing factor

- Influence of sample shape on hysteresis curve: Shape anisotropy
- Larger demag. factor \rightarrow stronger shearing of magnetization curve



3. Magnetic Measurement ...

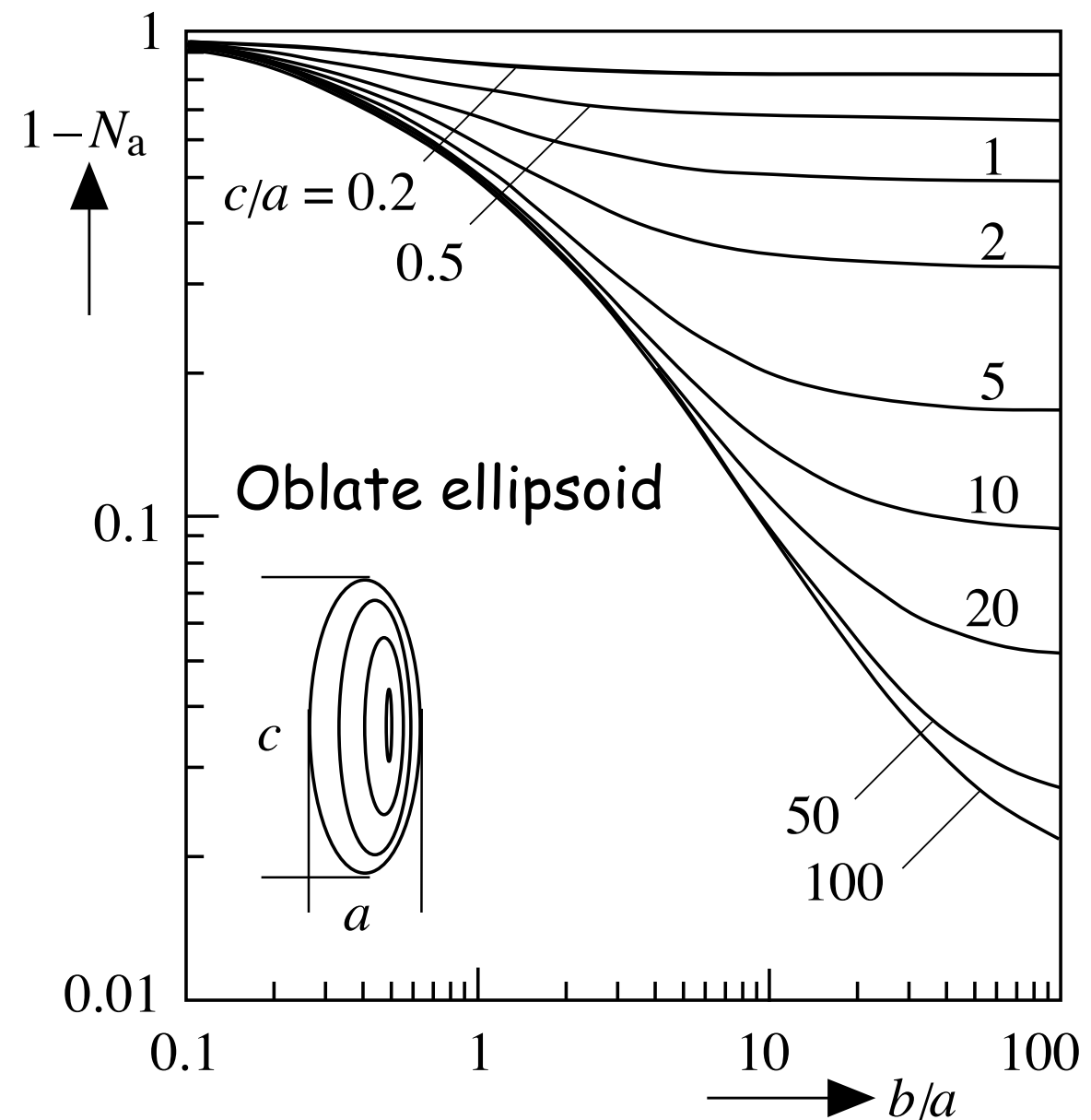
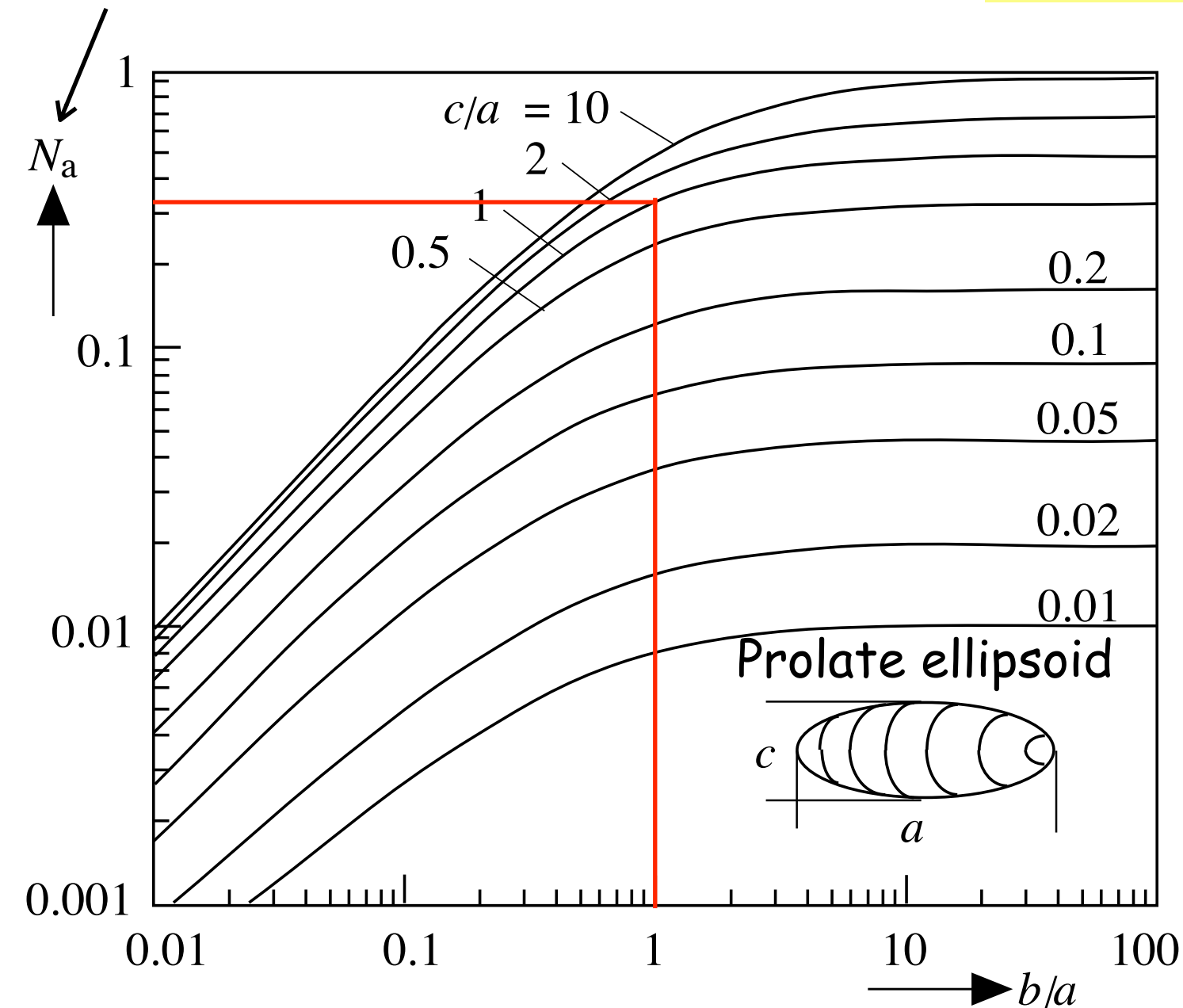
General aspects

Demagnetizing factor

Can only be calculated exactly for rotational ellipsoid

$$N_a + N_b + N_c = 1 \quad (a, b, c: \text{main axes of ellipsoid})$$

N along a -axis



Sphere: $a = b = c \rightarrow N_a = 1/3 = N_b = N_c$

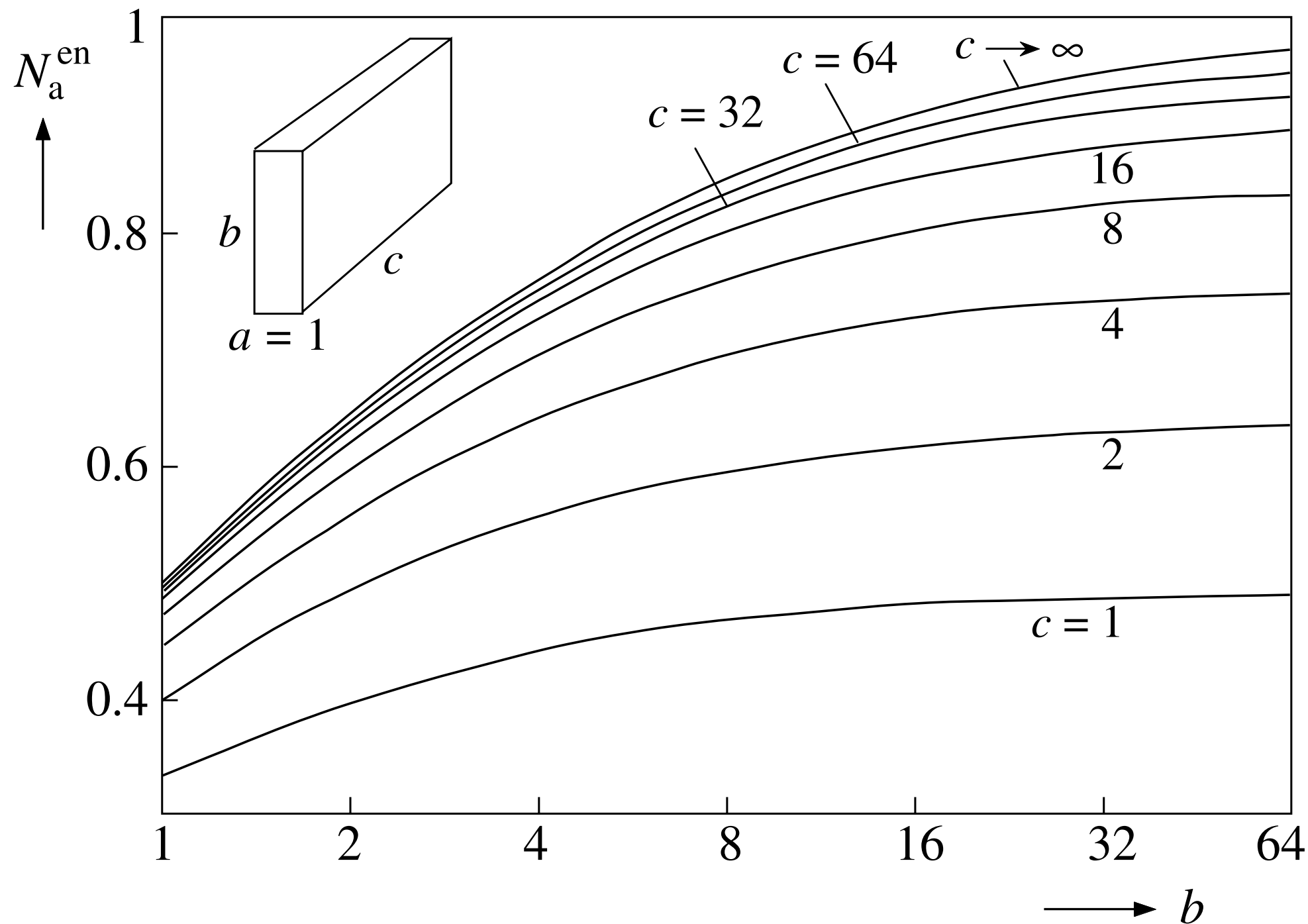
3. Magnetic Measurement ...

General aspects

Demagnetizing factor

Numerical calculation for rectangular body

$$N_a + N_b + N_c = 1 \text{ applies}$$



3. Magnetic Measurement ...

General aspects

Demagnetizing factor

Flat disk

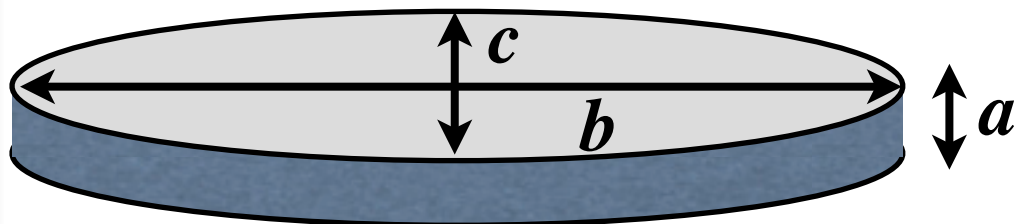
(thickness a ; $b = c \rightarrow \infty$):


$$N_a \approx 1;$$

$$N_b = N_c \approx 0$$



Disk prefers to be magnetized in-plane





Long cylinder
($b = c$; $a \rightarrow \infty$):

$$N_a \approx 0;$$
$$N_b = N_c \approx 1/2$$

↓

Cylinder prefers to be magnetized in longitudinal direction

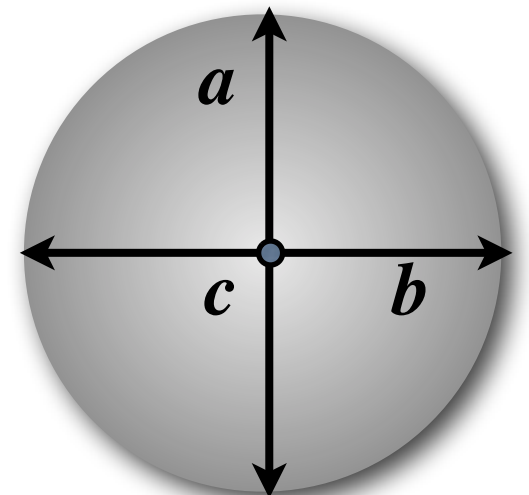
Sphere

($a = b = c$):

$$N_a = N_b = N_c = 1/3$$



no preferred direction for magnetization

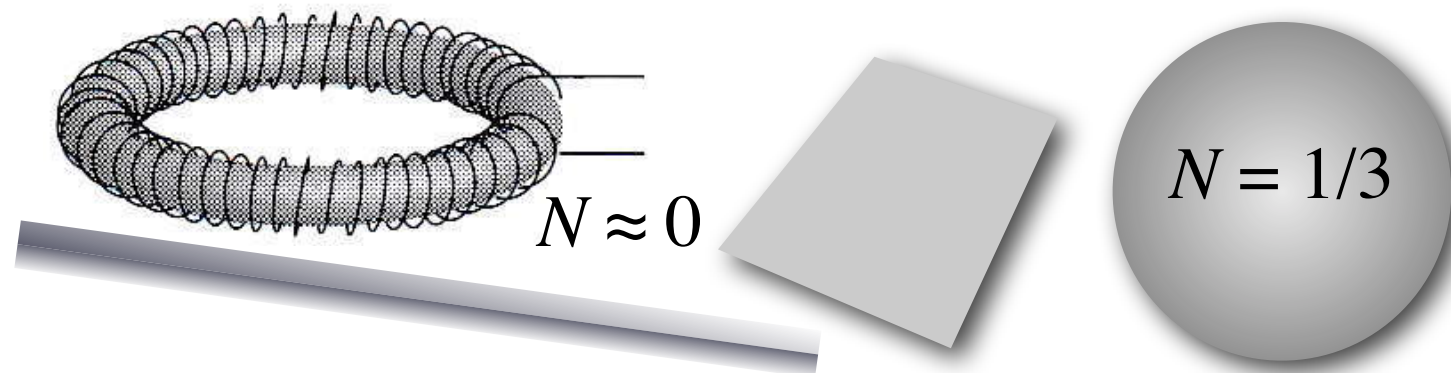
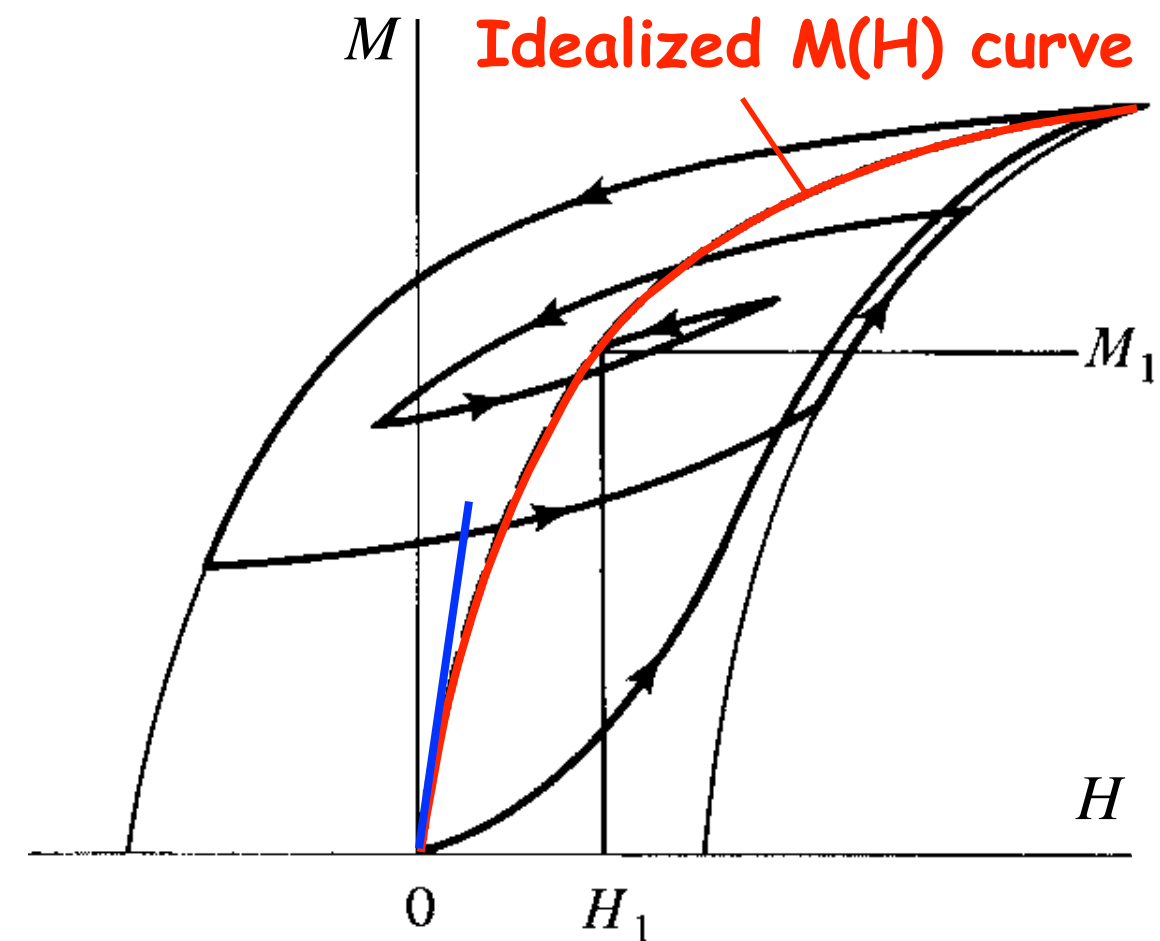
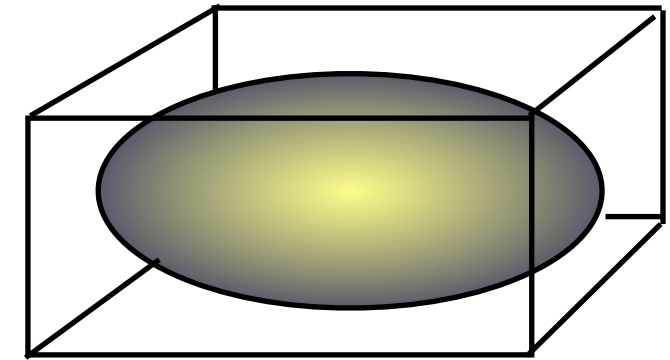


3. Magnetic Measurement ...

General aspects

Demagnetizing factor

- Demagnetizing factor of compact bodies is often well approximated by that of **inscribed ellipsoid**
- Experimental determination of the demagnetization factor:
 - For every dc-field an ac-field of decreasing amplitude is superimposed (helps to overcome barriers in magnetization process)
 - Then approximately that magnetization is achieved, the demagnetizing field of which is equal to the applied field
 - The initial slope of the magnetization curve is $dM/dH = 1/N$
- If possible: **avoid** demagnetization effect by choosing proper sample geometry for magnetic measurement



3. Magnetic Measurement ...

General aspects

Conclusion (demagnetization problematics):

The long-range nature of the demagnetizing field makes the measured property of any test specimen geometry-dependent

Demagnetizing field should be avoided when characterizing **soft** magnetic materials

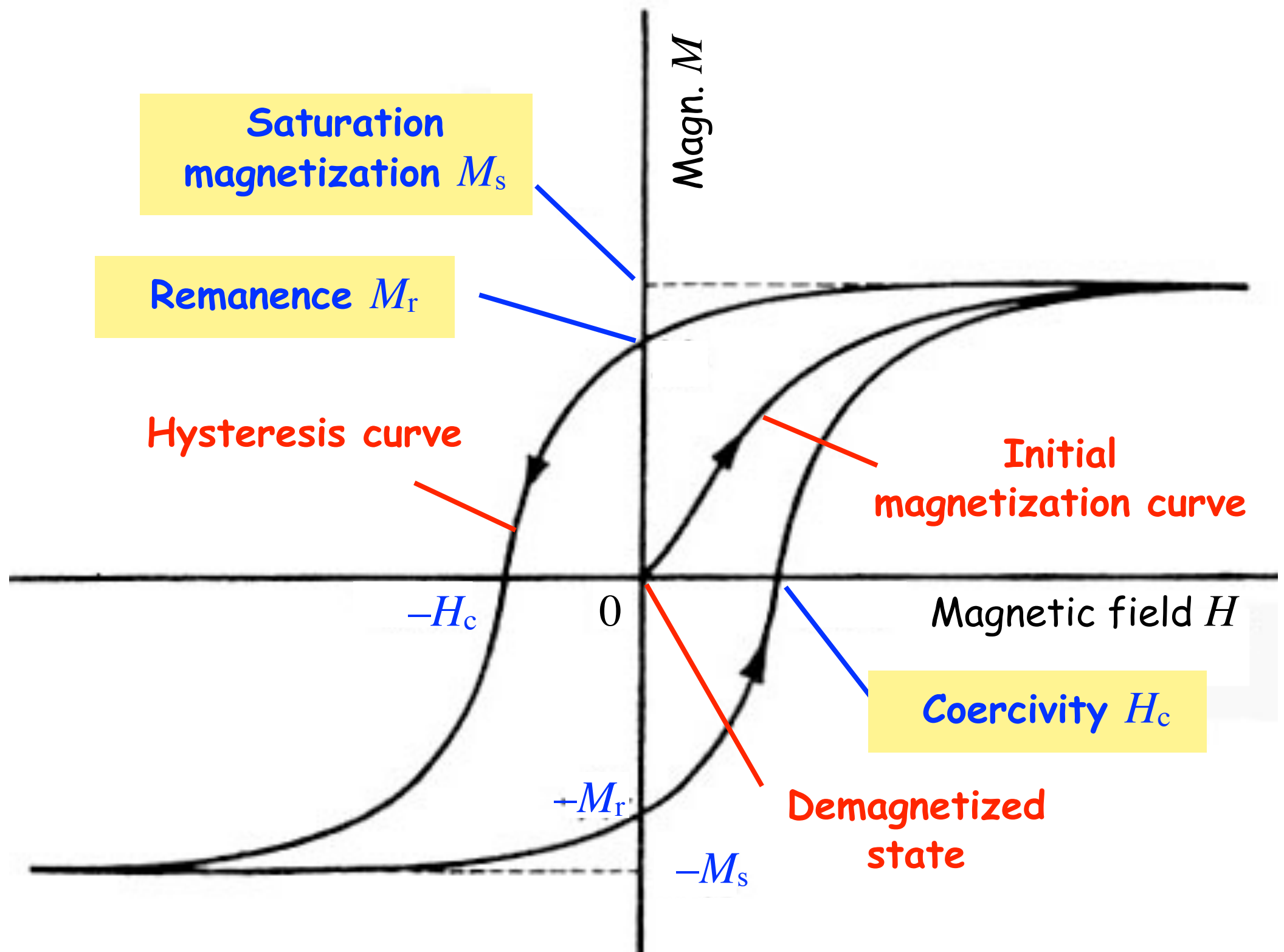
Demagnetizing field can be tolerated when characterizing **hard** magnetic materials, provided it is accurately known and is possibly uniform

For finite, non-ellipsoidal samples the magnetization is non-uniform due to demagnetizing effects → has to be considered when placing pick-up coil for inductive measurements

3. Magnetic Measurement ...

General aspects

Hysteresis curve:



3. Magnetic Measurement ...

General aspects

Hysteresis curve: B and M

- Difference between $M(H)$ and $B(H)$

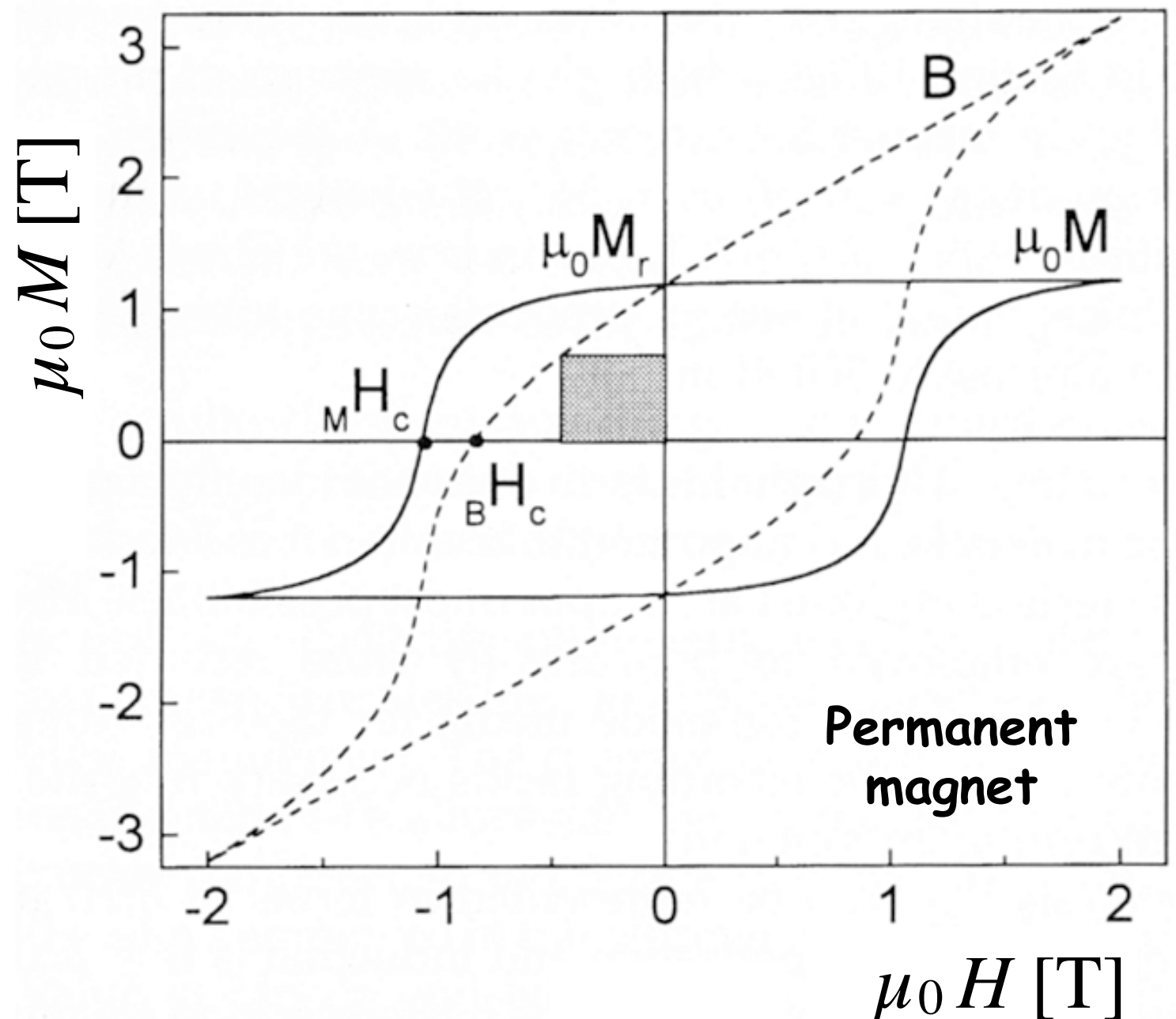
$$B(H) = \mu_0 (H + M)$$

- Soft magnets:
Fields involved in hysteresis loop are much smaller than corresponding magnetization values

$$\rightarrow B \cong \mu_0 M$$

\rightarrow difference between $B(H)$ and $M(H)$ negligible

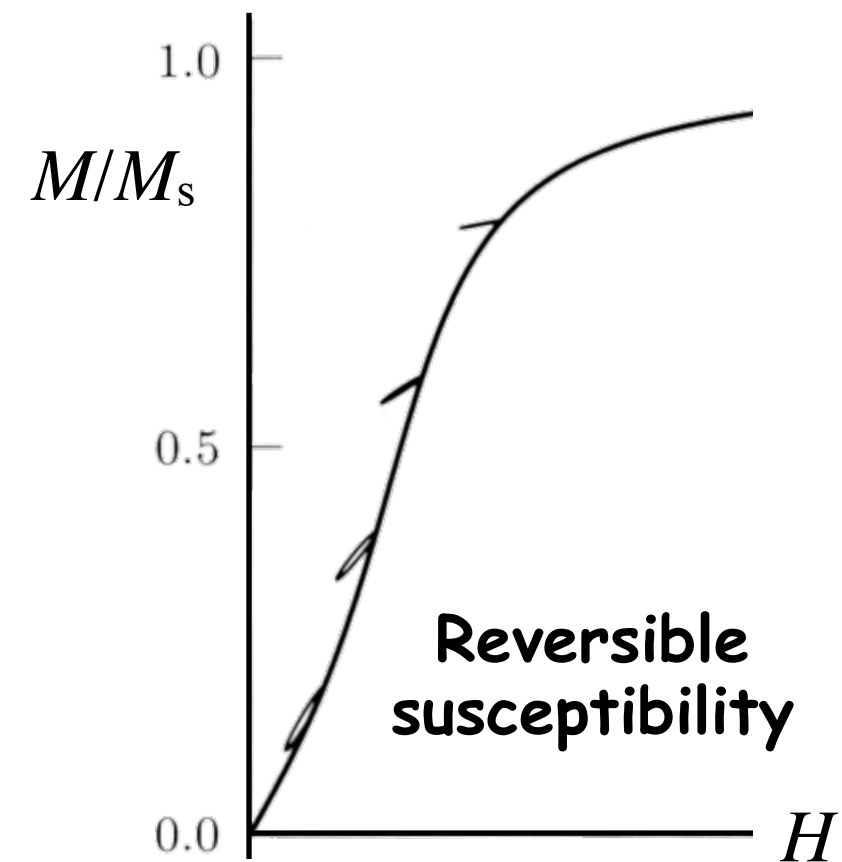
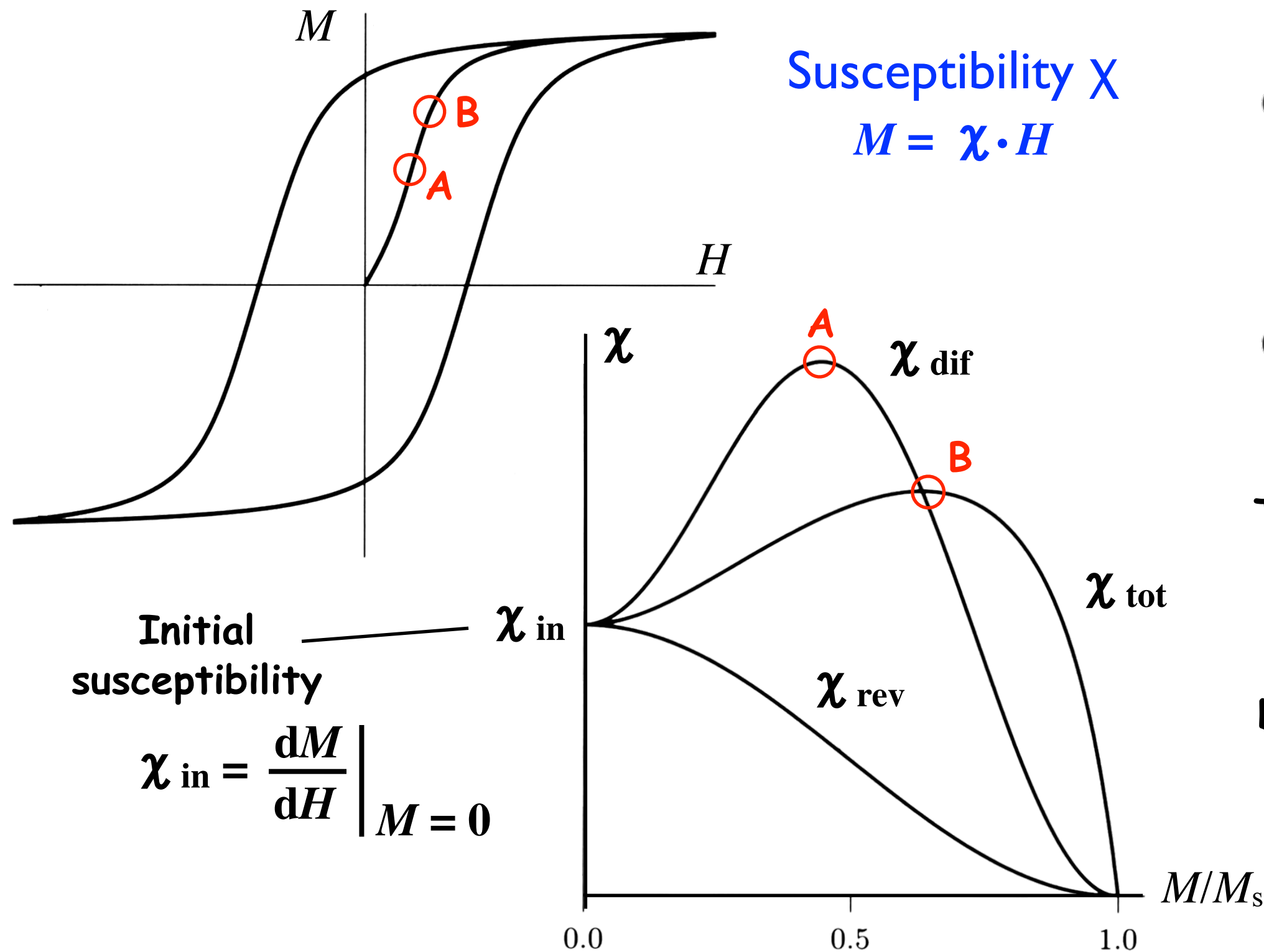
- Hard magnets:
 H and M have comparable orders $\rightarrow B(H)$ significantly different from $M(H)$



3. Magnetic Measurement ...

General aspects

Hysteresis curve: Susceptibility



Total susceptibility:

$$\chi_{tot} = \frac{M}{H}$$

Differential suscept.::

$$\chi_{dif} = \frac{dM}{dH}$$

3. Magnetic Measurement ...

General aspects

Hysteresis curve: Anisotropy

- Magnetic anisotropy is defined as energy differences needed for saturation along different axes → Magnetic anisotropy can be determined from $M(H)$ -curve for single crystals by comparing magnetization curves along hard- and easy directions
- Example: cubic magnetocrystalline anisotropy (case of iron)

$$e_{Kc} = K_{c1} \cdot (m_1^2 m_2^2 + m_1^2 m_3^2 + m_2^2 m_3^2) + K_{c2} m_1^2 m_2^2 m_3^2$$

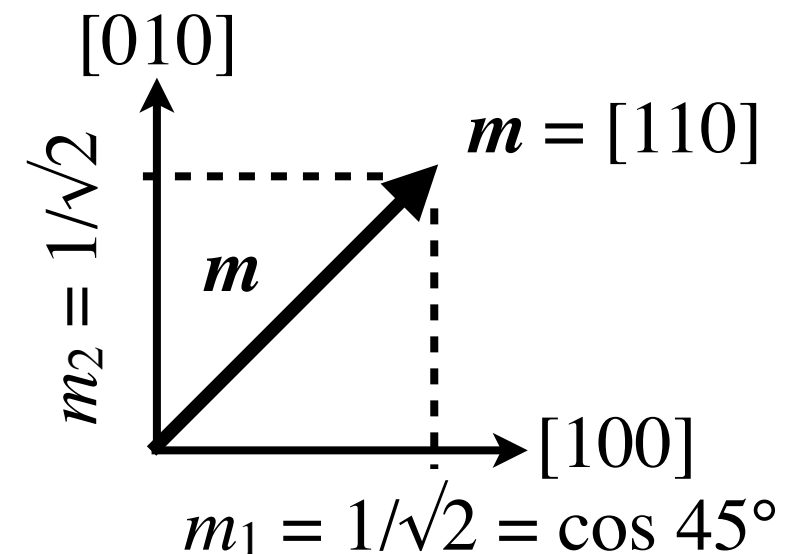
m_i = Magnetization components along cubic axes (direction cosine)

K_{ci} = Anisotropy constants

$$\mathbf{m} = [100]: \quad e_{Kc} = 0$$

$$\mathbf{m} = [110]: \quad m_1 = m_2 = 1/\sqrt{2}$$
$$e_{Kc} = K_{c1} (1/2 + 0 + 0) = K_{c1}/4$$

$$\mathbf{m} = [111]: \quad m_1 = m_2 = m_3 = 1/\sqrt{3}$$
$$e_{Kc} = K_{c1} (1/9 + 1/9 + 1/9) + 1/27 K_{c2}$$
$$= 1/3 K_{c1} + 1/27 K_{c2}$$



3. Magnetic

Hysteresis curve: An

- Magnetic anisotropy is defined for different axes → Magnetic anisotropy in single crystals by comparing magnetization curves for different crystallographic directions.
- Example: cubic magnetocrystalline anisotropy

$$e_{Kc} = K_{c1} \cdot (m_1^2 m_2^2 + m_1^2 m_3^2 + m_2^2 m_3^2)$$

m_i = Magnetization component along the i -th axis

K_{ci} = Anisotropy constant

$$\mathbf{m} = [100]: \quad e_{Kc} = 0$$

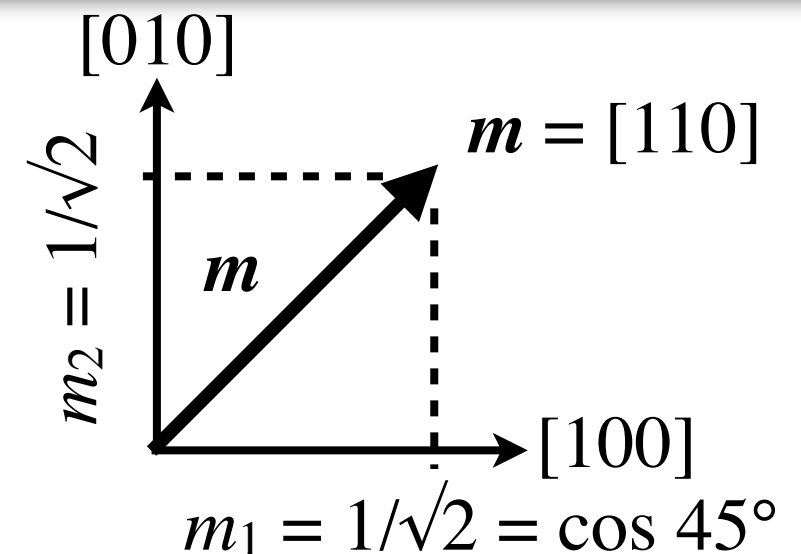
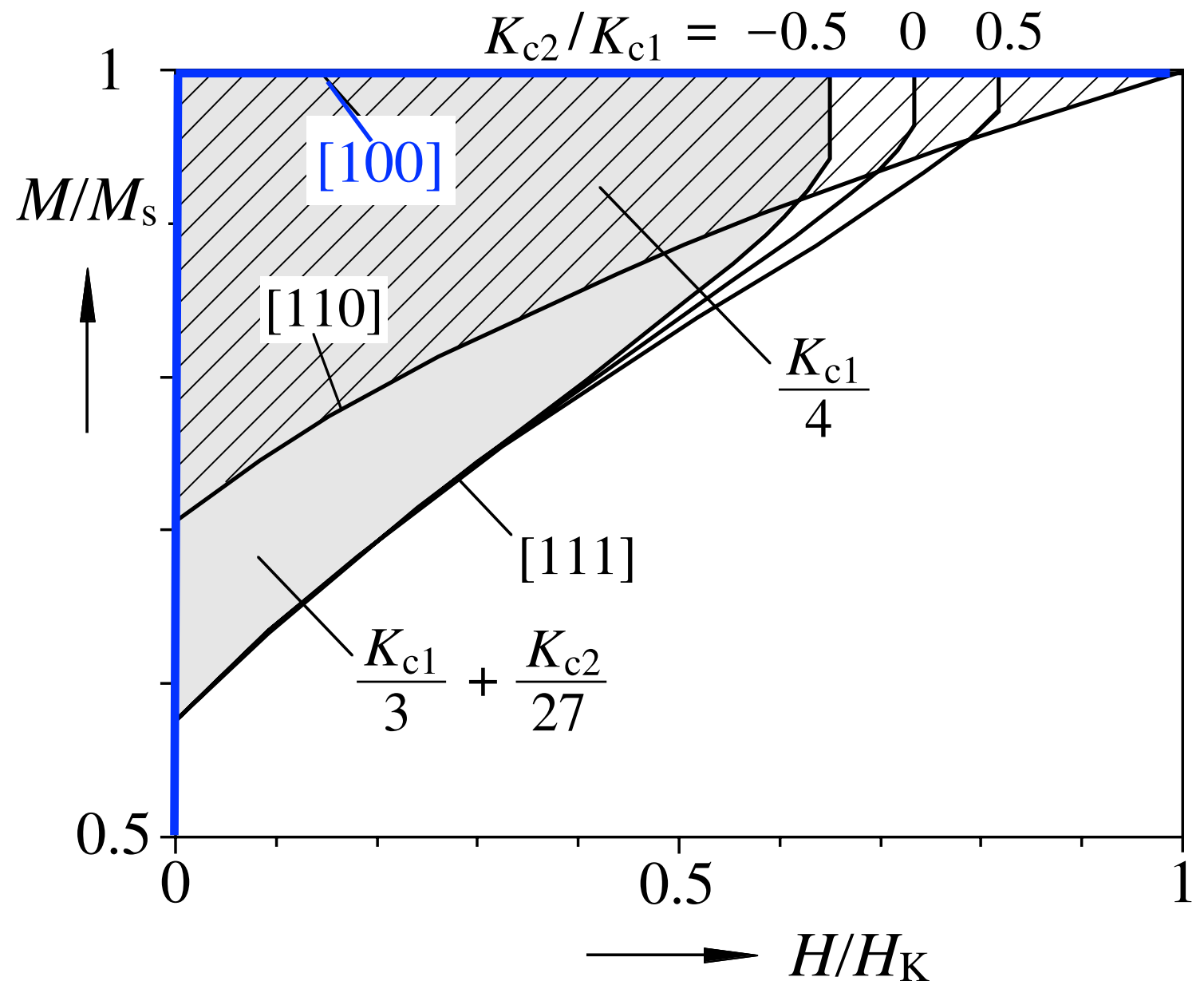
$$\mathbf{m} = [110]: \quad m_1 = m_2 = 1/\sqrt{2}$$

$$e_{Kc} = K_{c1} (1/2 + 0 + 0) = K_{c1}/4$$

$$\mathbf{m} = [111]: \quad m_1 = m_2 = m_3 = 1/\sqrt{3}$$

$$e_{Kc} = K_{c1} (1/9 + 1/9 + 1/9) + 1/27 K_{c2}$$

$$= 1/3 K_{c1} + 1/27 K_{c2}$$



3. Magnetic Measurement ...

General aspects

Hysteresis curve: Anisotropy

- Magnetic anisotropy is defined as energy differences needed for saturation along different axes → Magnetic anisotropy can be determined from $M(H)$ -curve for single crystals by comparing magnetization curves along hard- and easy directions
- Example: cubic magnetocrystalline anisotropy (case of iron)

$$e_{Kc} = K_{c1} \cdot (m_1^2 m_2^2 + m_1^2 m_3^2 + m_2^2 m_3^2) + K_{c2} m_1^2 m_2^2 m_3^2$$

m_i = Magnetization components along cubic axes (direction cosine)

K_{ci} = Anisotropy constants

- Limitations:
 - Hysteresis effects make the determination of the area ambiguous. Relying on the idealized magnetization curve offers fair solution to this problem
 - Non-ideal behaviour in approach to saturation. Internal stresses, inclusions and shape irregularities lead to a rounding of the magnetization curve. These effects depend on the magnetization direction because the magnetization deviations around such irregularities are influenced by anisotropy. The direct determination of the anisotropy from the magnetization curves is therefore often unreliable

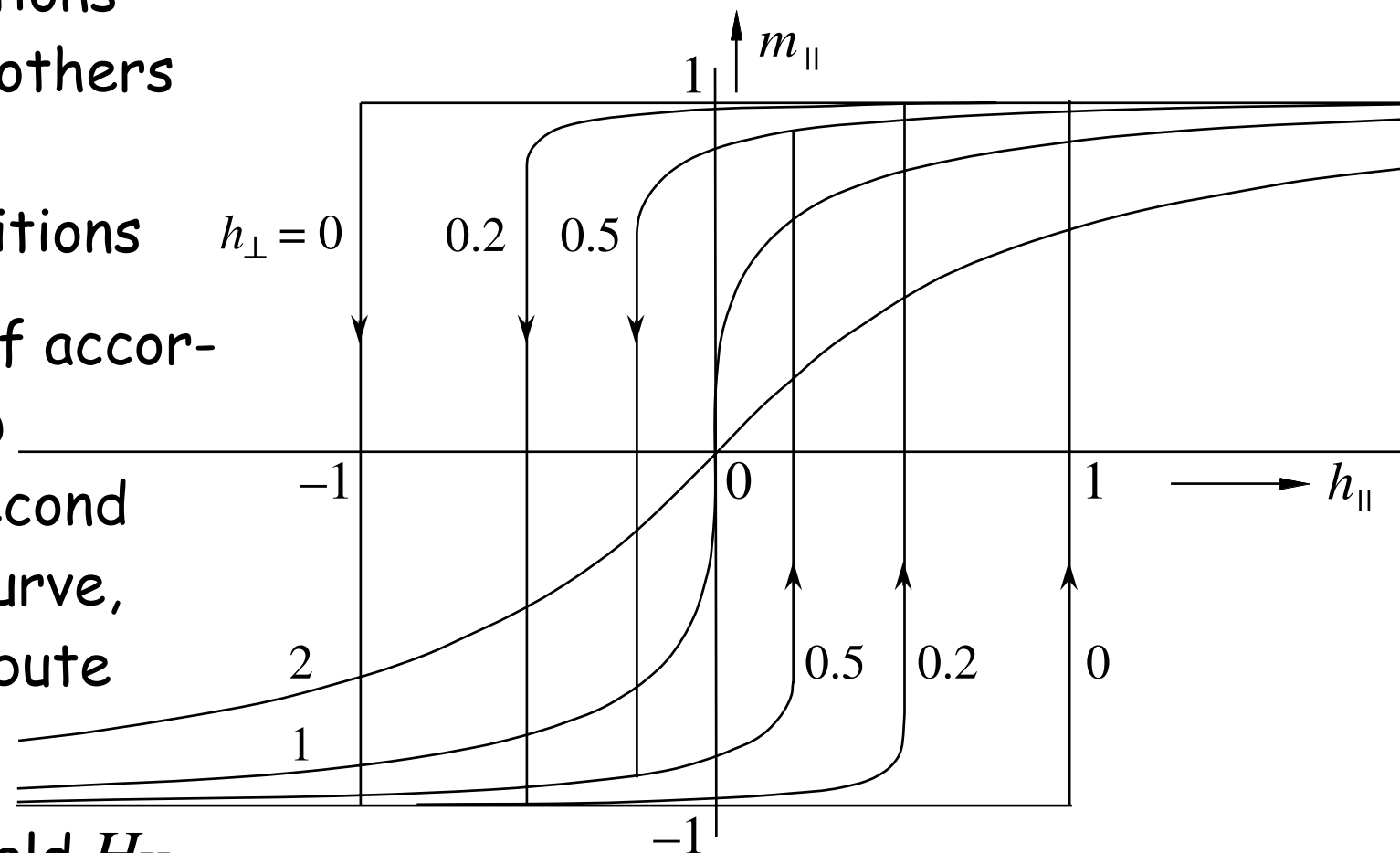
3. Magnetic Measurement ...

General aspects

Hysteresis curve: Anisotropy

Singular Point Detection in polycrystals

- Detects singularities in magnetization curve of polycrystalline sample which are caused by singular contributions from certain grains
- Compare magnetization curves of uniaxial particles: There are field orientations for which curves are smooth, for others they show characteristic jumps = first-order magnetization transitions
- In polycrystalline sample: jumps of accordingly oriented grains will show up as singularities (maxima) in the second derivative of the magnetization curve, while the other grains only contribute to a smooth background



→ Determination of anisotropy field H_K

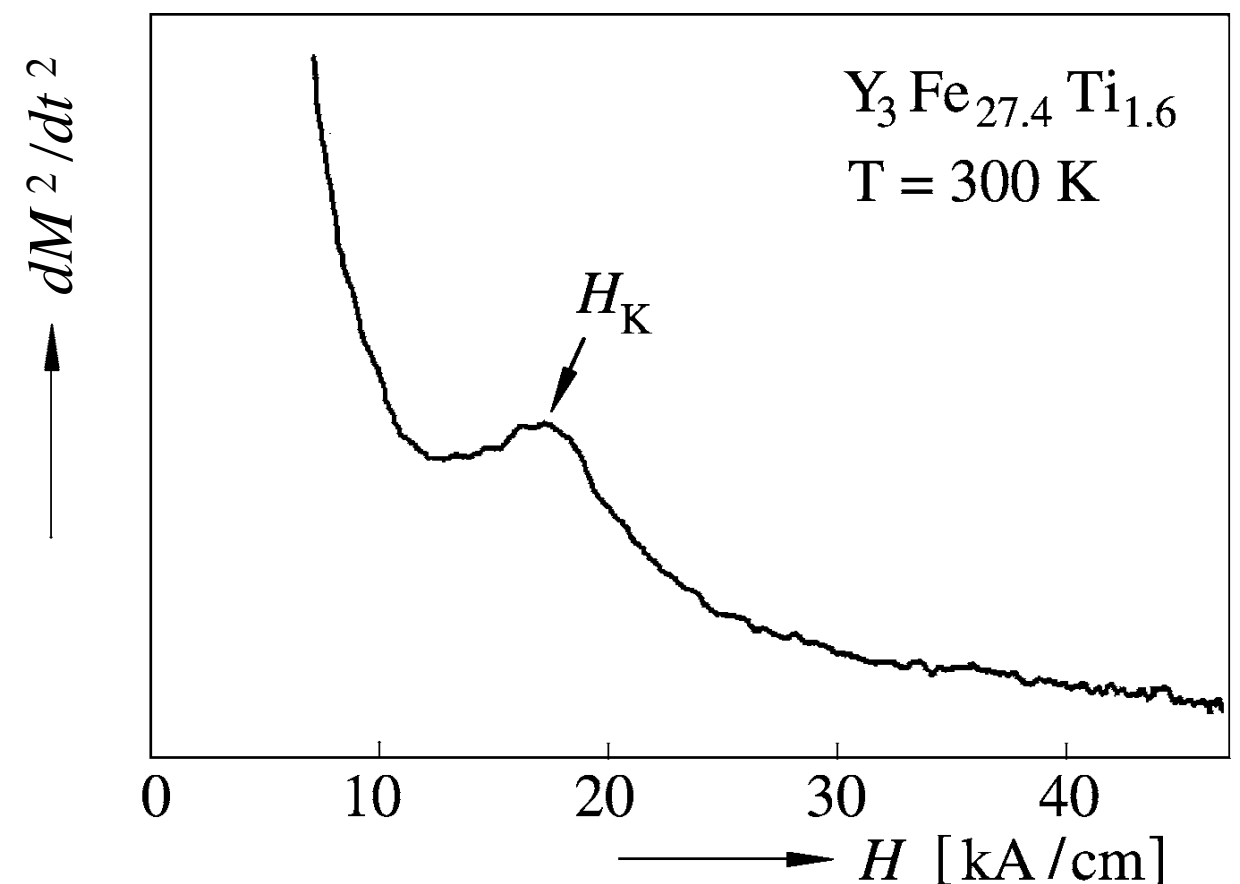
3. Magnetic Measurement ...

General aspects

Hysteresis curve: Anisotropy

Singular Point Detection in polycrystals

- Detects singularities in magnetization curve of polycrystalline sample which are caused by singular contributions from certain grains
- Compare magnetization curves of uniaxial particles: There are field orientations for which curves are smooth, for others they show characteristic jumps = first-order magnetization transitions
- In polycrystalline sample: jumps of accordingly oriented grains will show up as singularities (maxima) in the second derivative of the magnetization curve, while the other grains only contribute to a smooth background
→ Determination of anisotropy field H_K



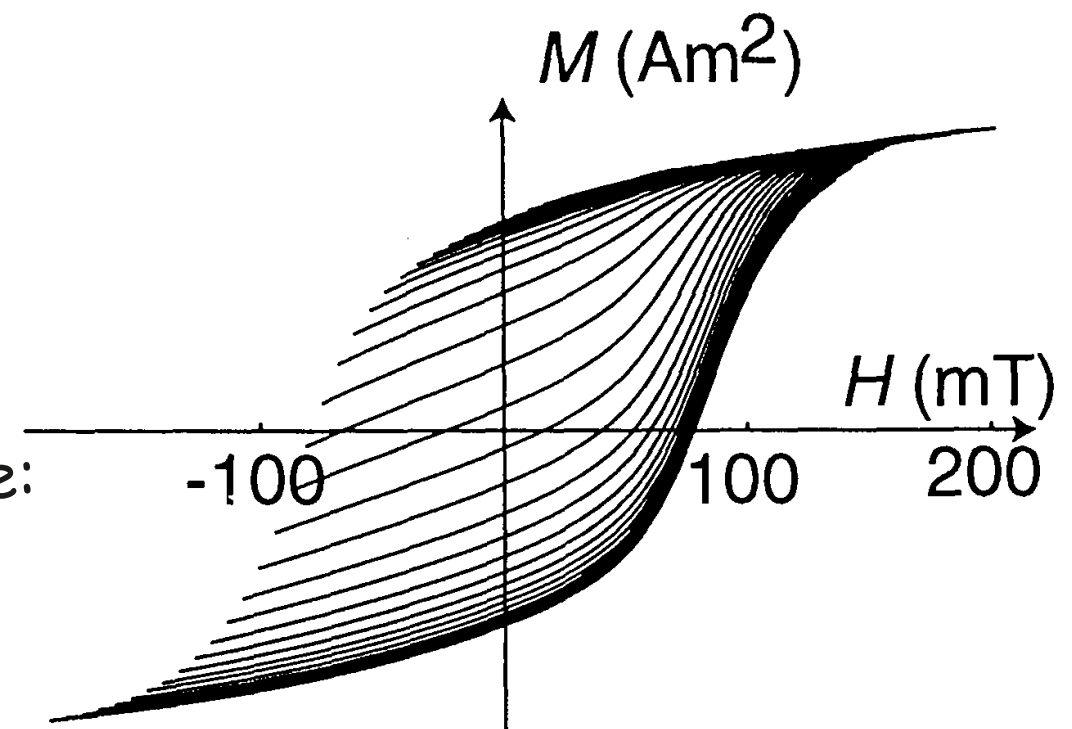
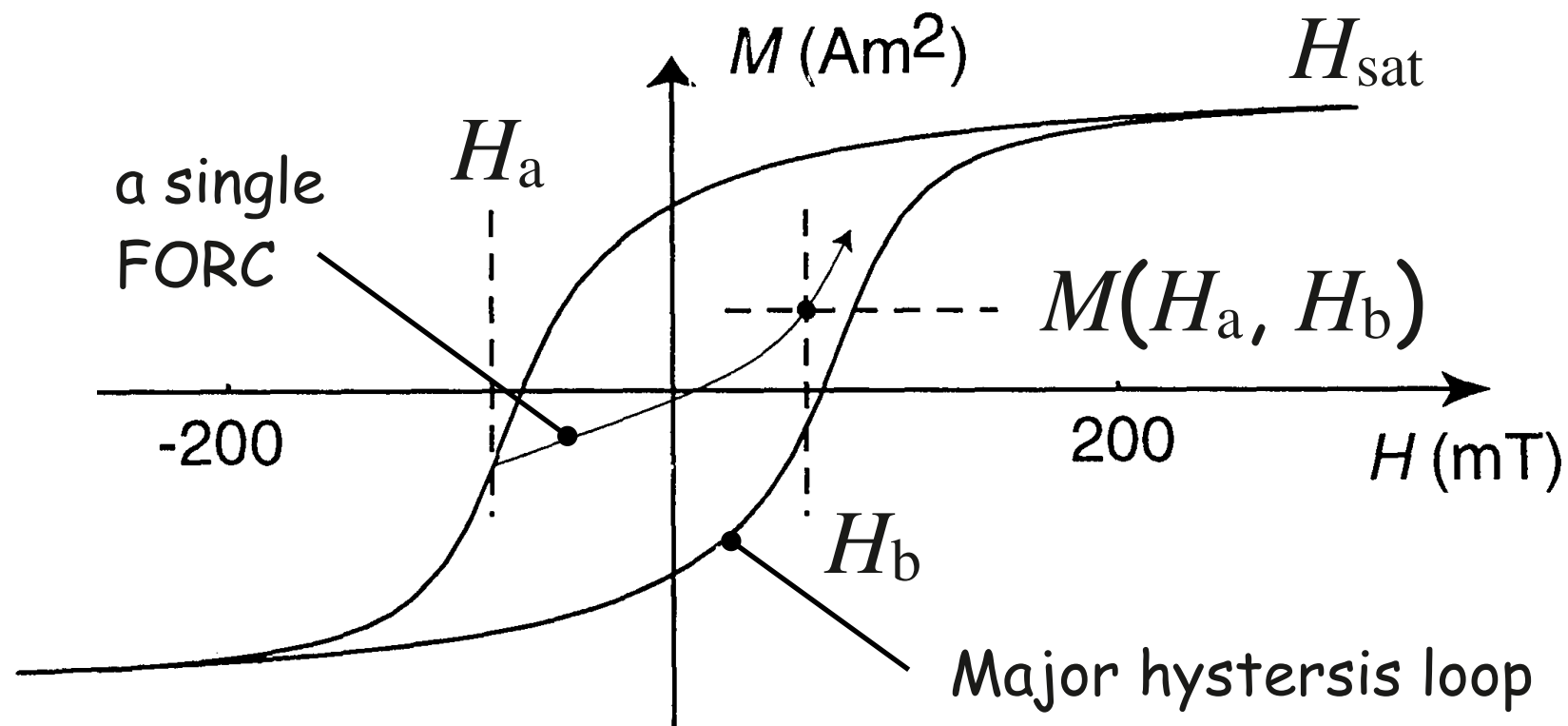
Courtesy R. Grössinger, Vienna

3. Magnetic Measurement ...

General aspects

FORC: First Order Reversal Curve

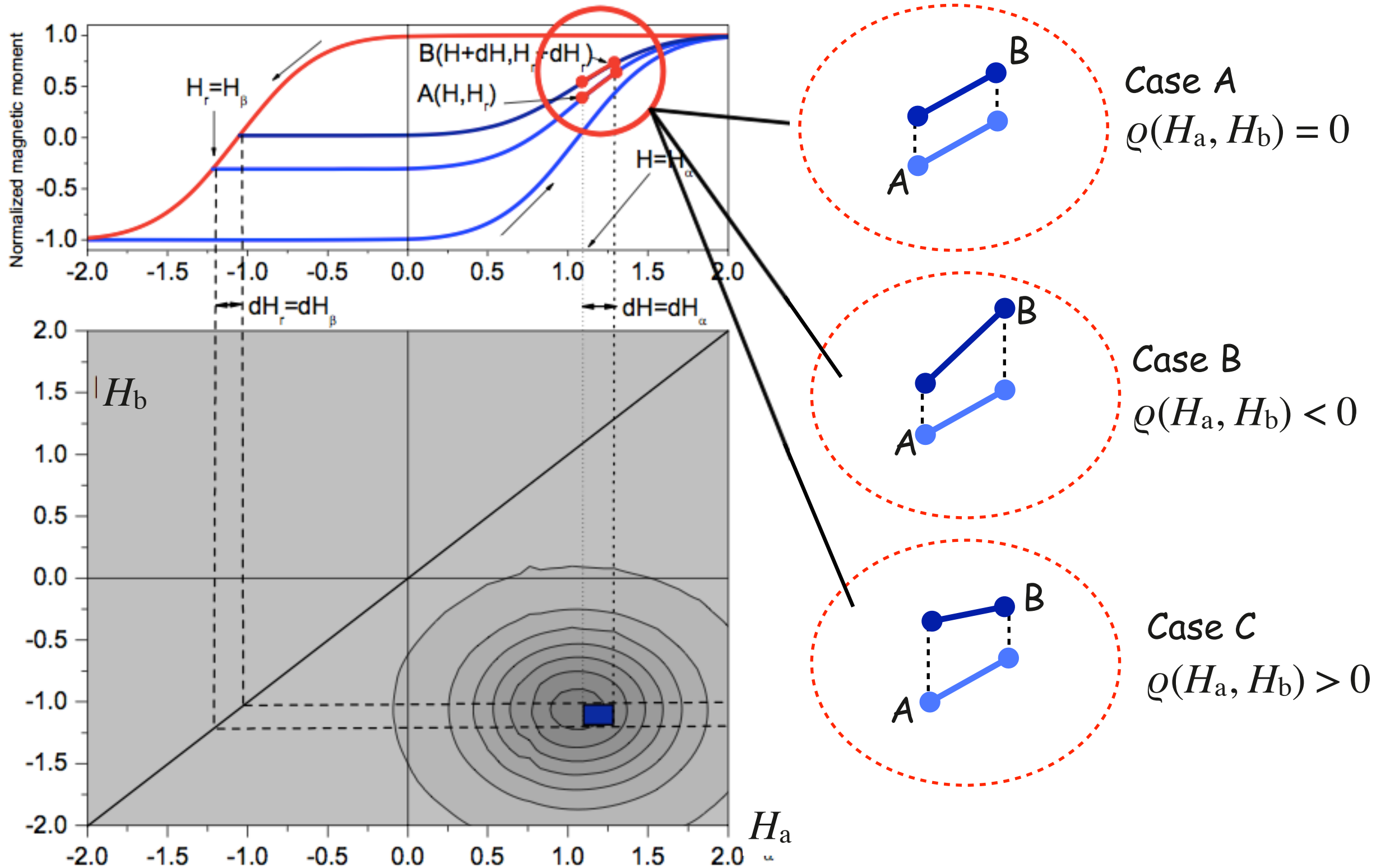
- A FORC is measured by saturating sample in field H_{sat} , decreasing the field to a reversal field H_a , then sweeping field back to H_{sat} in a series of equal field steps H_b . The magnetization curve between H_a and H_b is a FORC
- This process is repeated for many values of H_a yielding a series of FORCs, and the measured magnetization at each step as a function of H_a and H_b gives $M(H_a, H_b)$ distribution
- The **FORC distribution** $\varrho(H_a, H_b)$ is defined as the mixed second derivative of the $M(H_a, H_b)$ - surface:
$$\varrho(H_a, H_b) = - \partial^2 M(H_a, H_b) / \partial H_a \partial H_b$$
- $\varrho(H_a, H_b)$ is plotted as contour- or 3D-plot



3. Magnetic Measurement ...

General aspects

FORC: First Order Reversal Curve



3. Magnetic Measurement ...

General aspects

FORC: First Order Reversal Curve

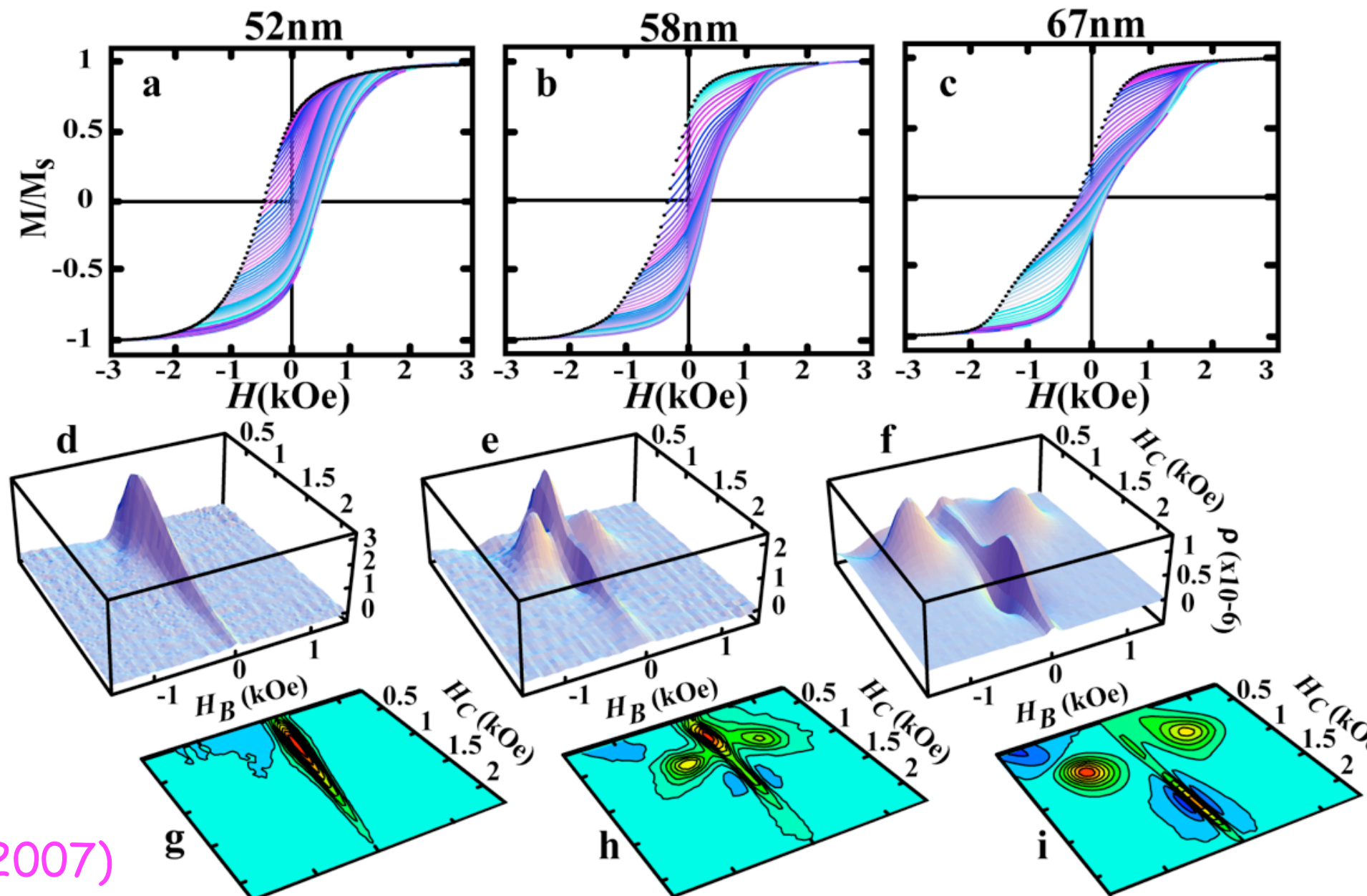
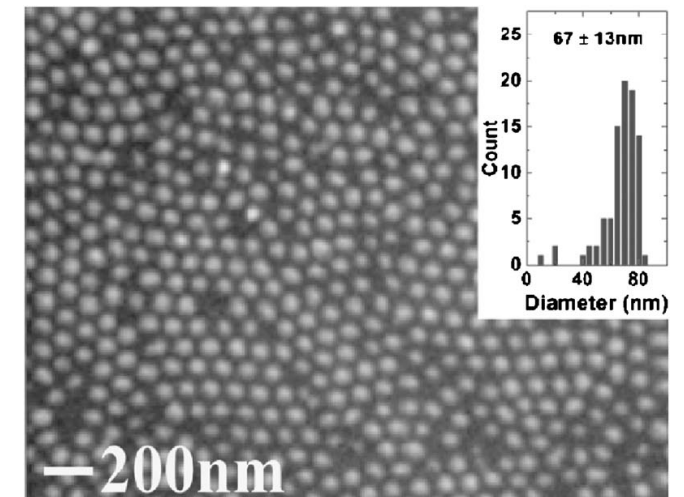
- Alternatively Q can be plotted in coordinates of (H_C, H_B) , where H_C is the local coercive field and H_B is the local interaction or bias field
This transformation is accomplished by a rotation of the coordinate system defined by: $H_B = (H_a + H_b) / 2$ and $H_C = (H_a - H_b) / 2$
- FORC distribution eliminates purely reversible components of magnetization process. Thus any **non-zero** Q corresponds to **irreversible** switching processes
- FORC thus provide insight into relative proportions of reversible and irreversible components of the magnetization process. Examples:
 - Exchange-coupled nanocomposite permanent magnet material: investigation of magnetostatic- and exchange interactions between hard and soft phases
 - Exchange-biased spin-valves: studies of the switching distribution and exchange bias in materials where the switching of the free layer magnetization is strongly influenced by the magnetic state of the fixed layer
 - Arrays of magnetic nanowires, nanodots or nanoparticles: investigation of irreversible magnetic interactions or processes in the array due to coupling between adjacent wires, dots or particles

3. Magnetic Measurement ...

General aspects

FORC: First Order Reversal Curve

- Example: nanodot array of different dot diameter.
Distinctly different reversal mechanisms, despite only subtle differences in the major hysteresis loops.



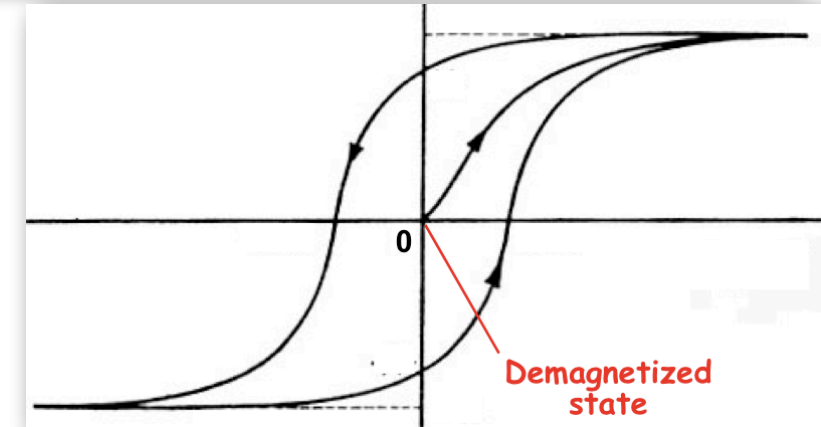
Courtesy Kai Liu, Davis
Phys. Ref. B 75, 134405 (2007)

3. Magnetic Measurement ...

General aspects

Demagnetization

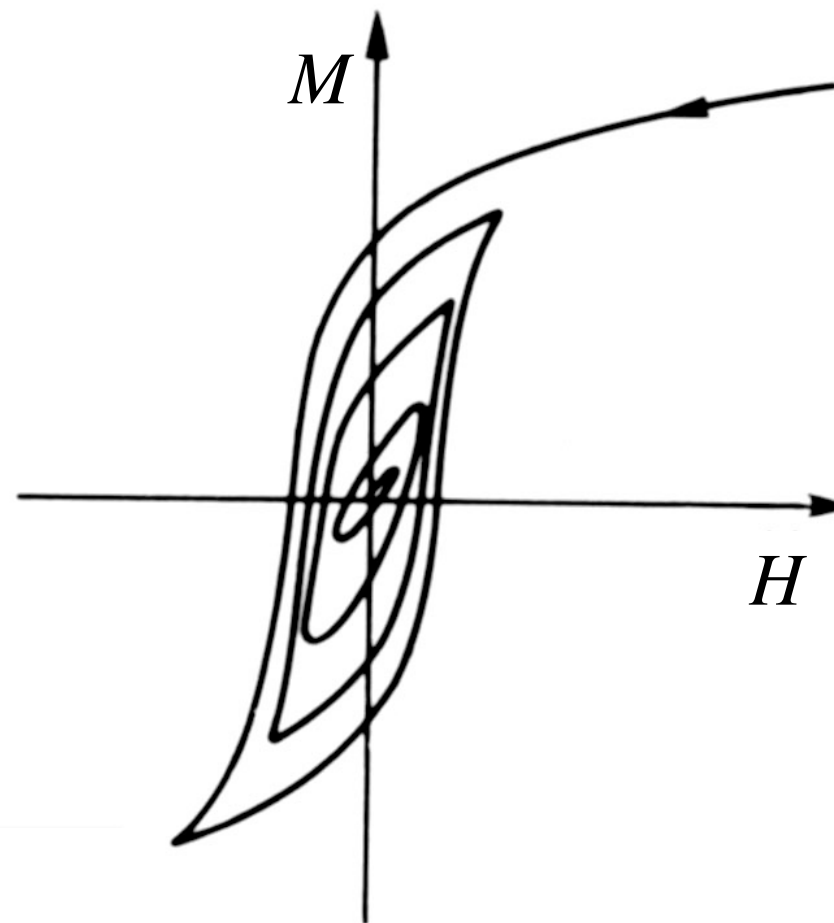
3 possibilities, to „demagnetize“ a magnet ($\bar{M} = 0$) :



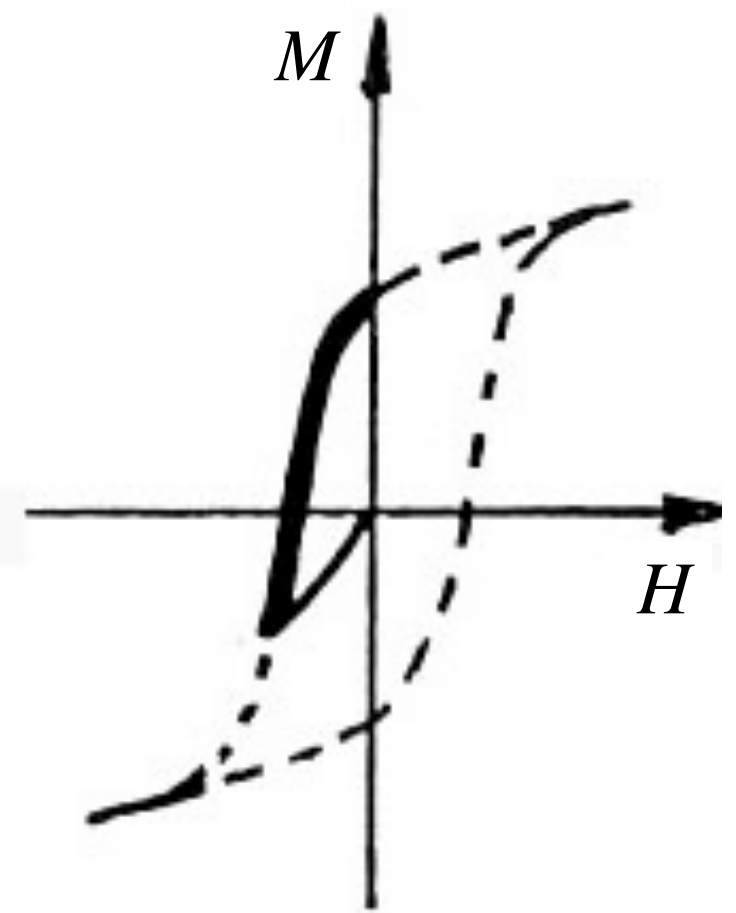
1. Thermal demagnetization

Heating above Curie temperature and cooling in absence of magnetic field

2. Cyclic (ac) demagnetization



2. dc-field demagnetization



In principle any point within or on the hysteresis loop can be obtained by choosing the right field history

3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

3.4 Dilatometric measurements

3.5 Domain methods

3.1 Magnetic Measurements

a) Inductive methods

Sample surrounded by coil, in which voltage is induced when magnetization of sample is changed or when sample is moved. Voltage is integrated → signal proportional to magnetization

b) Magnetometric methods

For finite samples: demagnetizing field, which is proportional to mean magnetization ($H_{\text{dem}} = -N\bar{M}$), is measured

c) Optical magnetometry

Surface magnetization is measured by magneto-optic effect, useful for thin films where signal of inductive or magnetometric methods are too weak

3.1 Magnetic Measurements

a) Inductive methods

Extraction Method

- Based on flux change in pick-up coil when sample is extracted from coil, or when specimen and pick-up coil together are extracted from field

Total flux through pick-up coil:

$$\Phi_1 = BA = \mu_0(H + M)A = \mu_0(H_a - H_d + M)A = \mu_0(H_a - N_d M + M)A$$

If sample is removed from pick-up coil, the flux through the coil becomes:

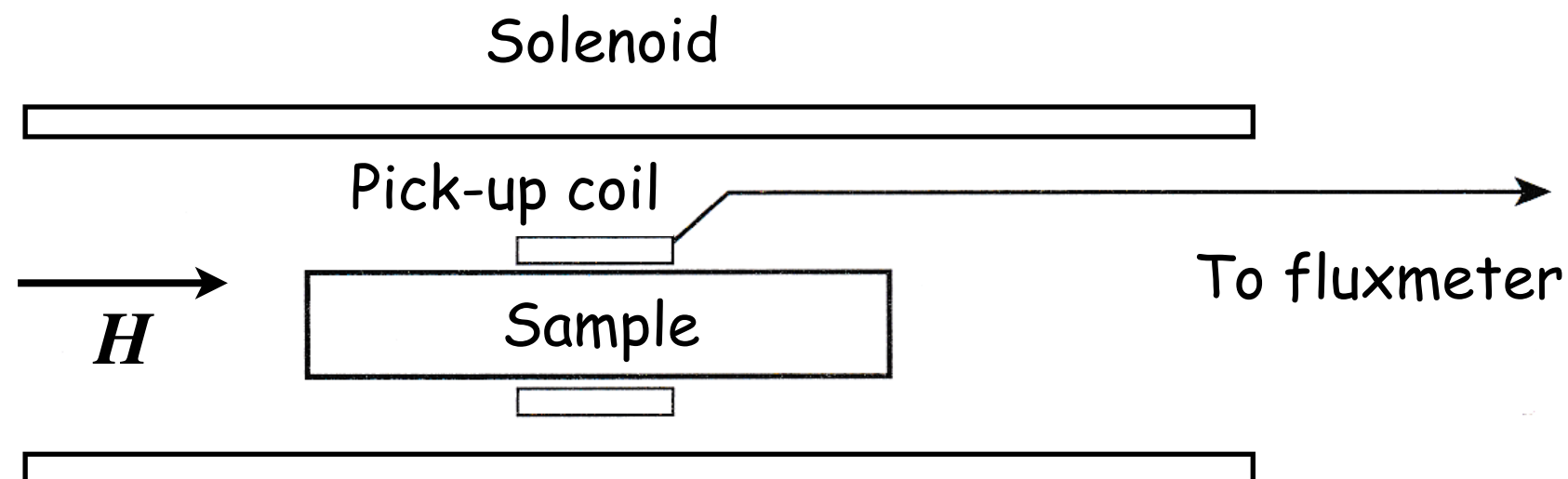
$$\Phi_2 = \mu_0 H_a A$$

A: specimen or pick-up coil area

Fluxmeter will record a value proportional to flux change:

$$\Phi_1 - \Phi_2 = \mu_0(1 - N_d)MA$$

- Extraction method **measures M** directly, rather than B
- Vibrating sample magnetometer may be regarded as kind of partial extraction method



3.1 Magnetic Measurements

a) Inductive methods

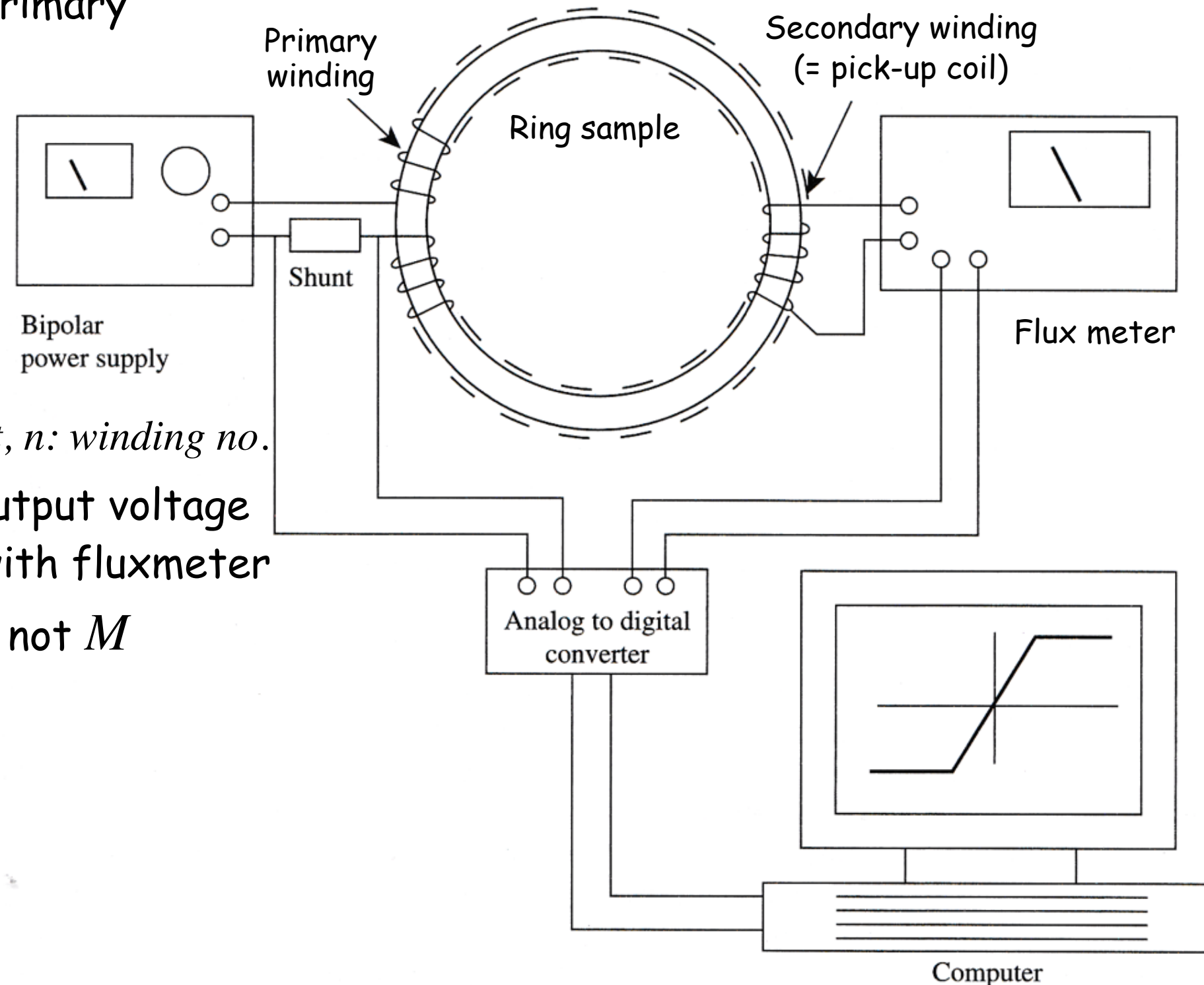
Measurement of closed circuit samples

- Conventional setup with primary and secondary winding
- Procedure: vary current through primary winding and measure its magnitude by voltage drop across shunt:

$$H = nI/L_{\text{Fe}} \quad I: \text{current}, n: \text{winding no.}$$

simultaneously integrate output voltage from secondary winding with fluxmeter

- Measures flux **density** B , not M



Measurement of magnetic field strength

Fluxmeter

- Faraday's law: a changing magnetic flux φ through a coil of N turns generates a voltage in coil proportional to rate of change of flux:

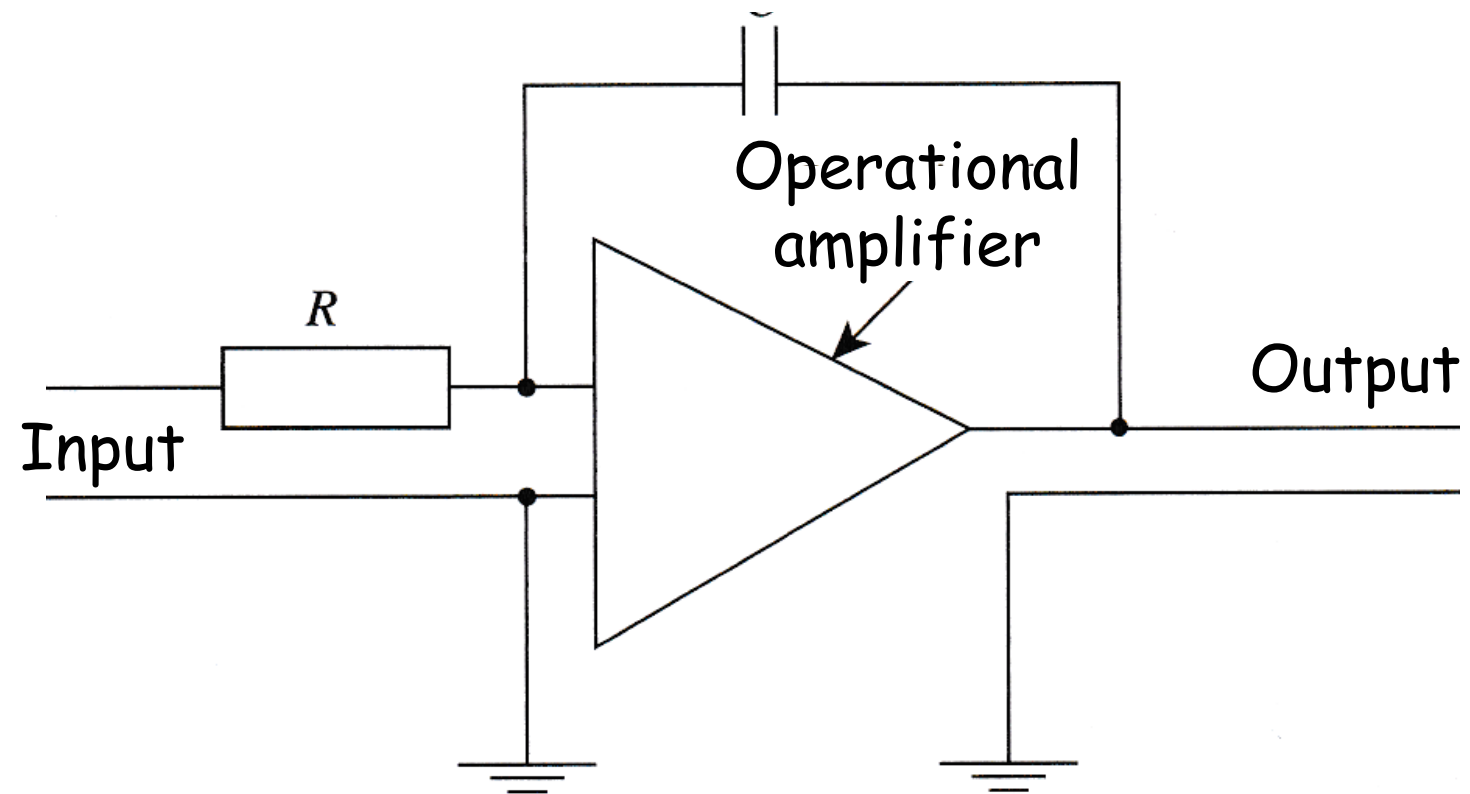
$$U(t) = -N \frac{d\varphi}{dt} \quad [\text{Volt}]$$

$$U(t) dt = -N d\varphi$$

$$\int_0^t U(t) dt = -N \int_{\Phi_1}^{\Phi_2} d\varphi = -N \Delta\varphi$$

- Instrument to integrate voltage from pick-up coil is called fluxmeter = electronic integrator (based on capacitive feedback around operational amplifier) that provides voltage output
- With $B = \Phi/A$ (flux density in pick-up coil of cross section A):

$$\int U(t) dt = -N_{\text{pick-up}} A_{\text{pick-up}} \Delta B \quad [\text{Vs}]$$

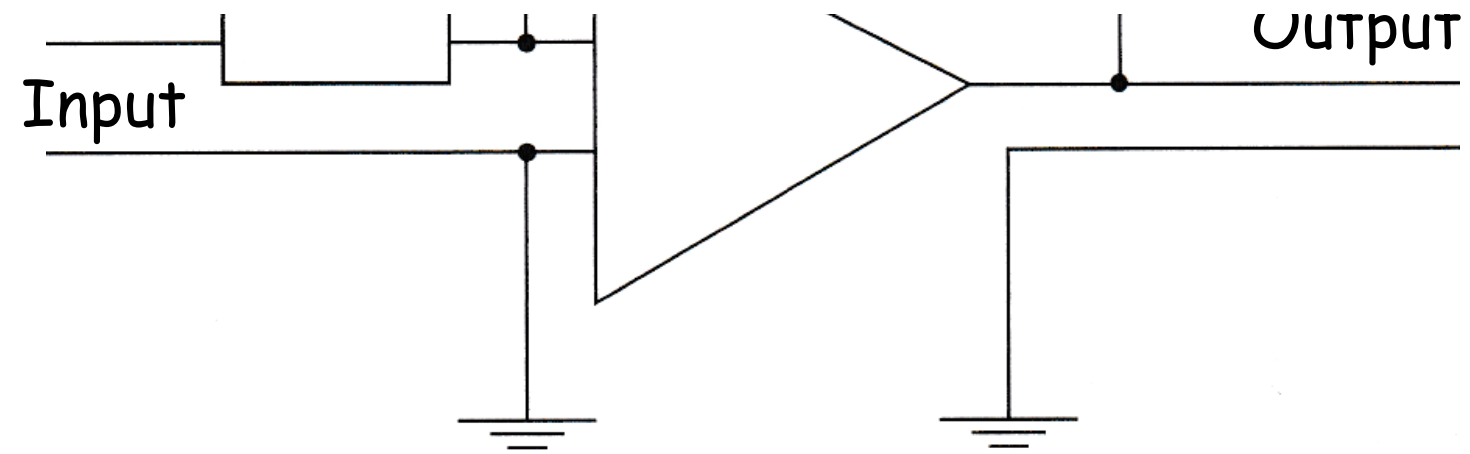


$$U_{\text{out}} = -1/RC \int U_{\text{in}} dt$$

Fluxmeter measures changes in flux density

Measurement of magnetic field strength

Fluxmeter



induced voltage is proportional to rate of change of flux:

$$U(t) = -N \frac{d\phi}{dt} \quad [\text{Volt}]$$

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Fluxmeter measures changes in flux density

- Pick-up coil filled with (e.g.) iron:

$$\int U(t) dt = -N_{\text{pick-up}} A_{\text{Fe}} \Delta B \rightarrow \Delta B = - \frac{1}{N_{\text{pick-up}} A_{\text{Fe}}} \int_0^t U(t) dt$$

3.1 Magnetic Measurements

a) Inductive methods

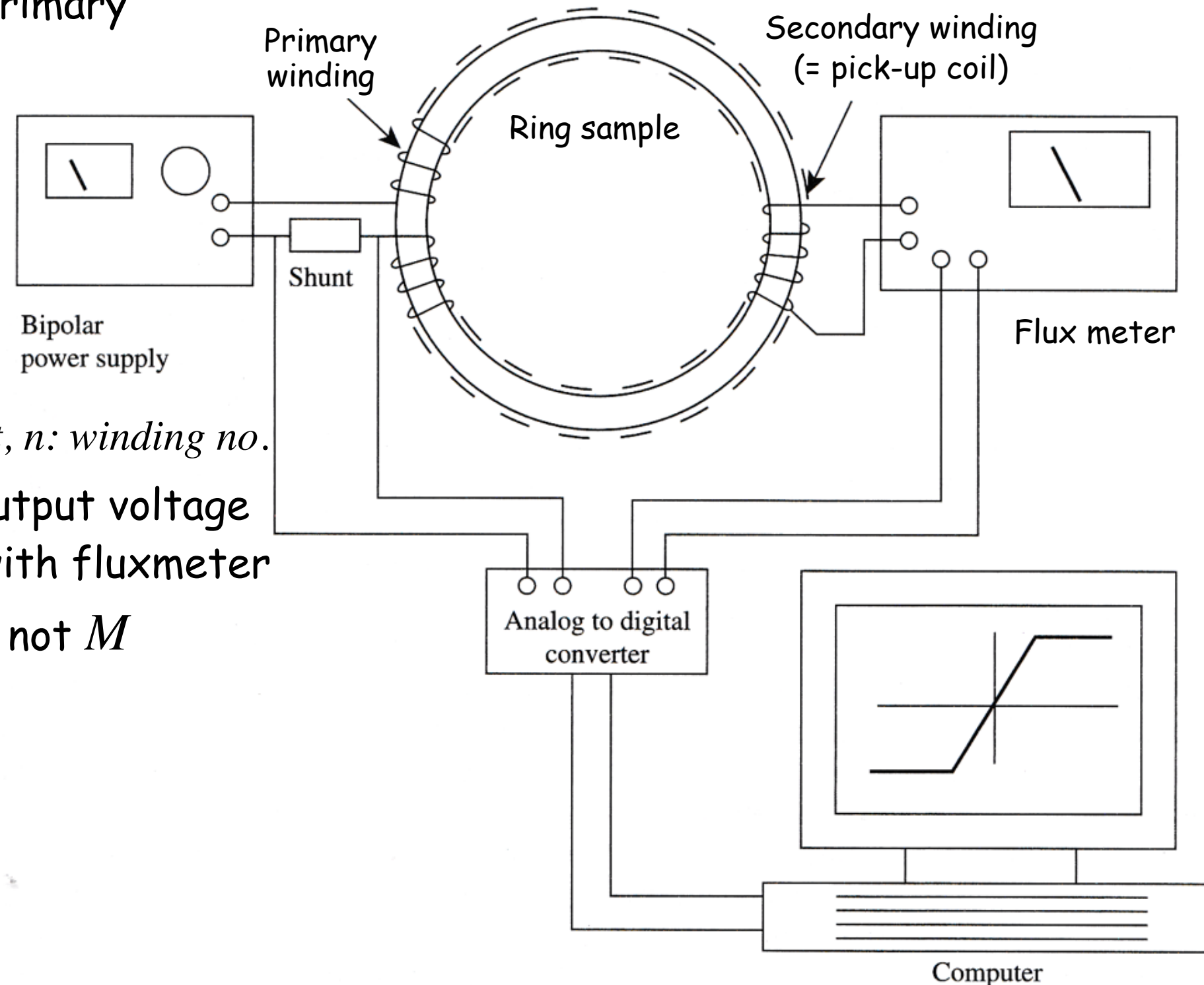
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simultaneously integrate output voltage from secondary winding with fluxmeter

- Measures flux density B , not M

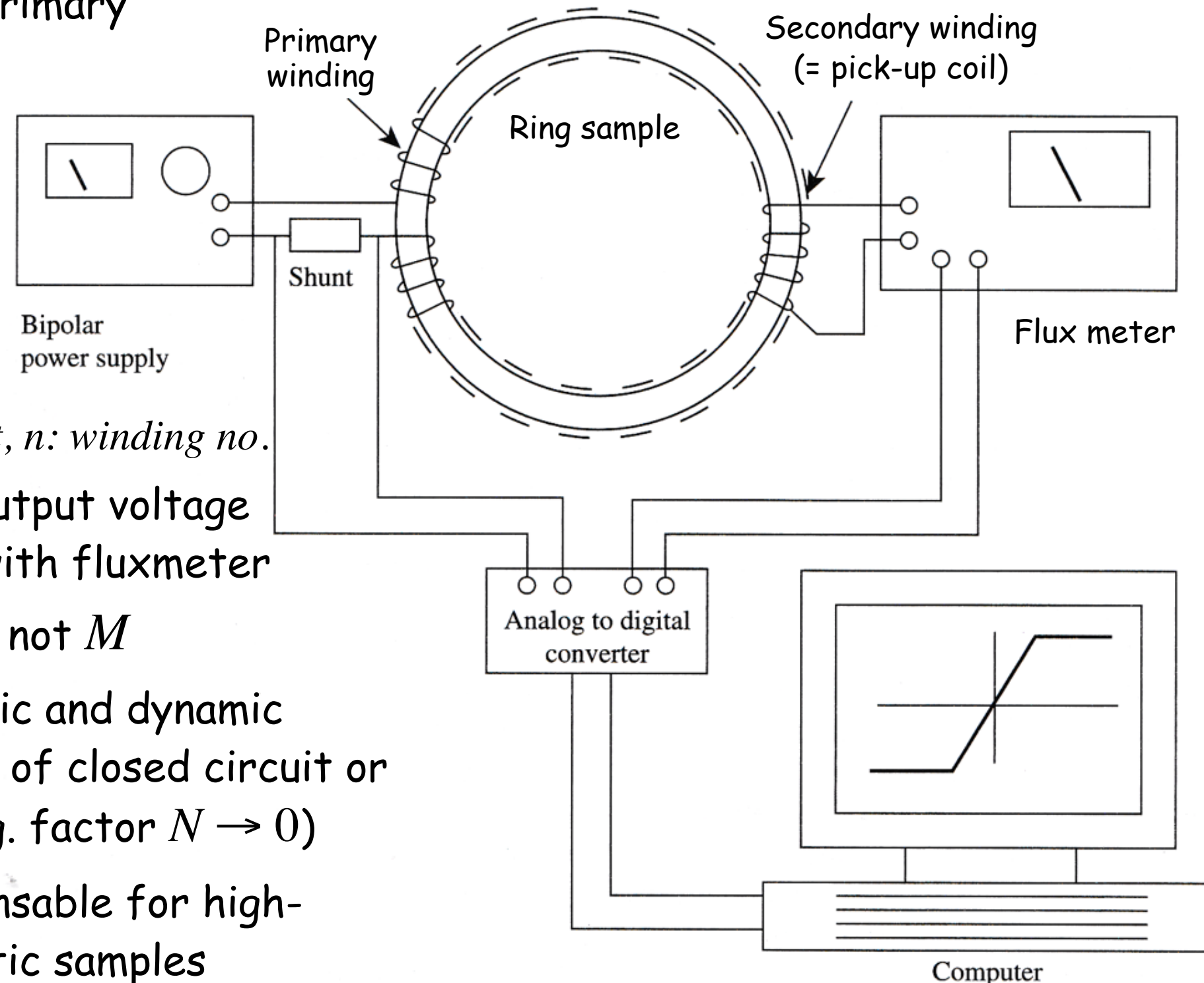


3.1 Magnetic Measurements

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Measurement of closed circuit samples

- Conventional setup with primary and secondary winding
- Procedure: vary current through primary winding and measure its magnitude by voltage drop across shunt:
$$H = nI/L_{Fe}$$
I: current, n: winding no.
simultaneously integrate output voltage from secondary winding with fluxmeter
- Measures flux density B , not M
- Can be used for quasistatic and dynamic hysteresis measurements of closed circuit or elongated samples (demag. factor $N \rightarrow 0$)
- Such geometry is indispensable for high-permeability, soft magnetic samples

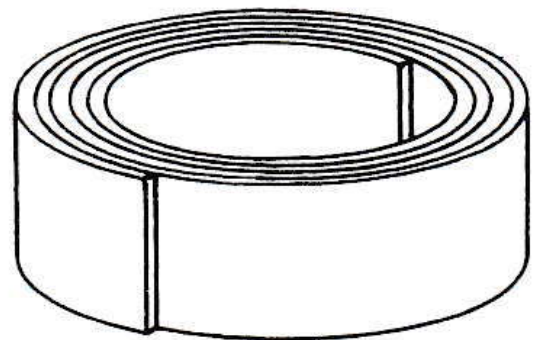


3.1 Magnetic Measurements

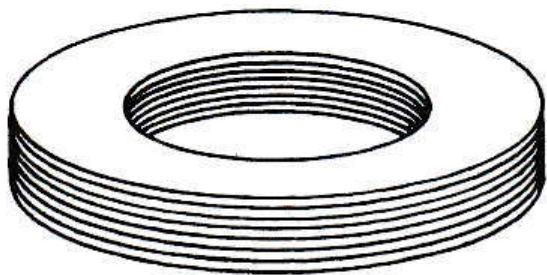
a) Inductive methods

Measurement of closed circuit samples

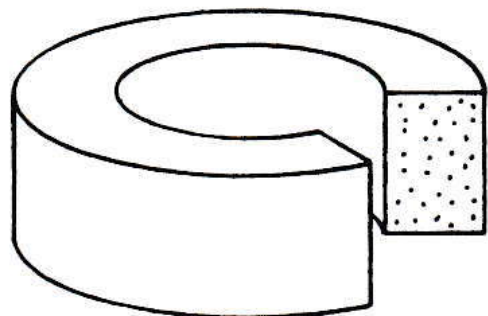
- Examples of closed samples



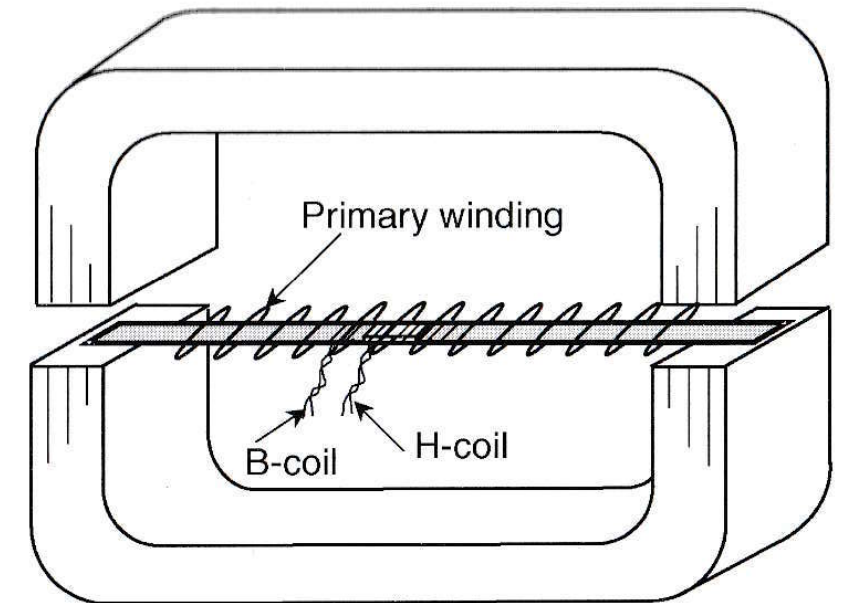
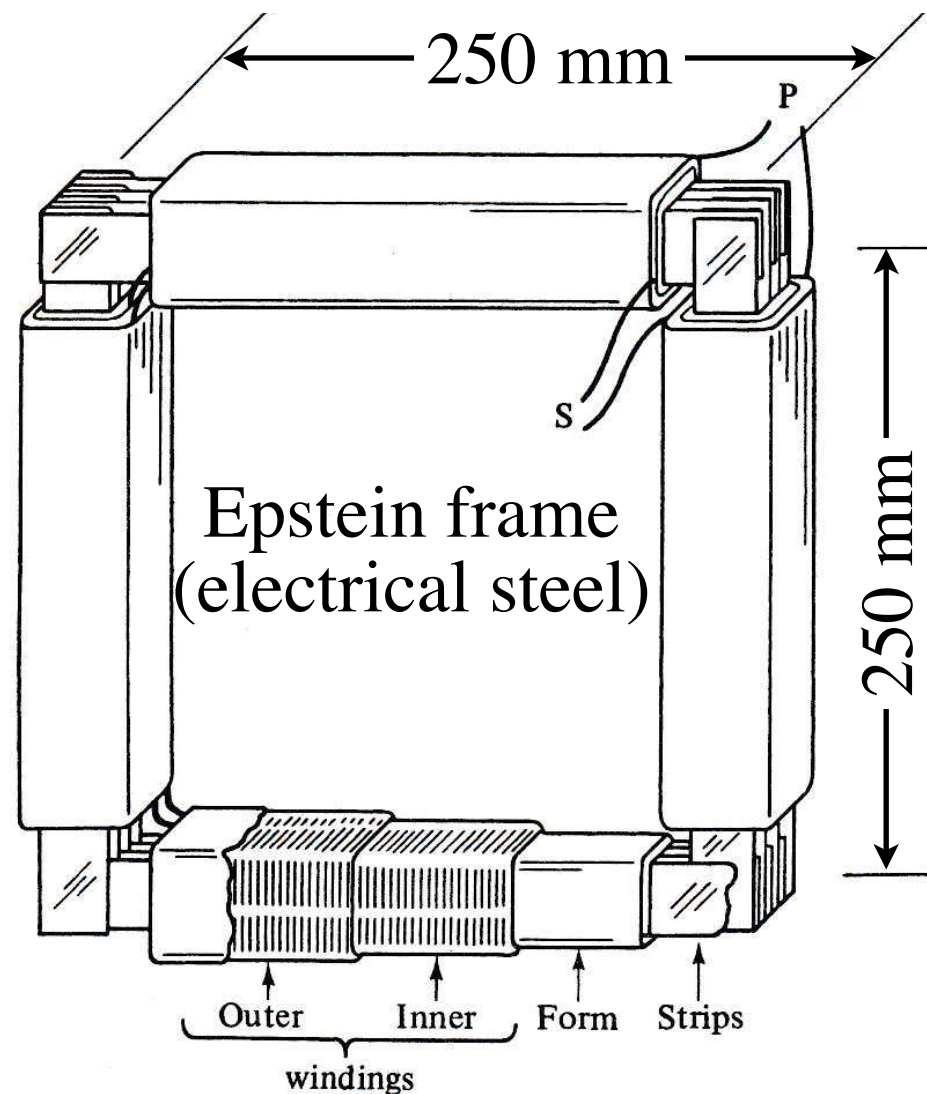
Tape-wound core



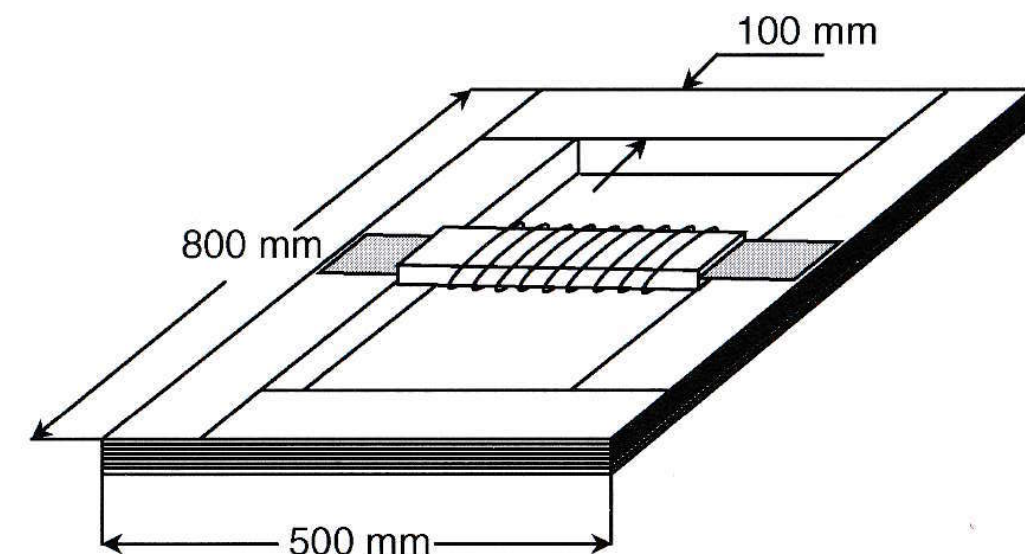
Stacked lamination



Powder core



Laminated yoke for sheet samples (permeameter)



Single sheet tester

3.1 Magnetic Measurements

a) Inductive methods

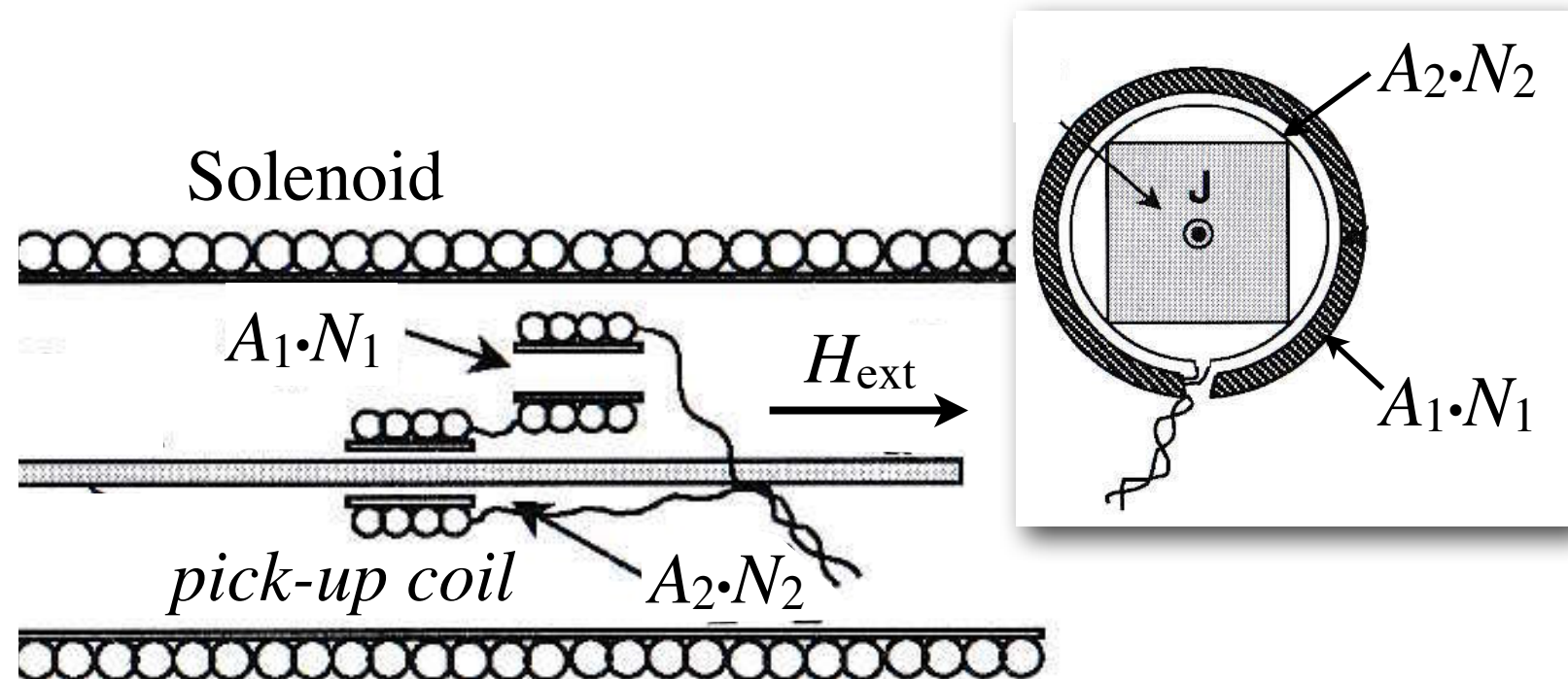
Measurement of closed circuit samples

- $B = \mu_0 H + J \longrightarrow$

If $J(H)$ is to be measured instead of $B(H)$: **Air flux compensation** required to subtract effect of applied field

- Compensation coil arrangement: 2 coils with equal winding areas $A_1 \cdot N_1 = A_2 \cdot N_2$ ($A_{1,2}$: cross sectional area of coils, $N_{1,2}$: number of turns) are connected electrically in opposition \rightarrow difference signal is proportional to magnetization alone, i.e. without specimen in pick-up coil: no flux recorded by fluxmeter, with specimen in pick-up coil: fluxmeter reads $J = B - \mu_0 H$

- Two possibilities: (i) two identical coils arranged side by side; (ii) external layers of the winding of a coil can be connected in such a way that their winding area is equal and opposite to the core winding area

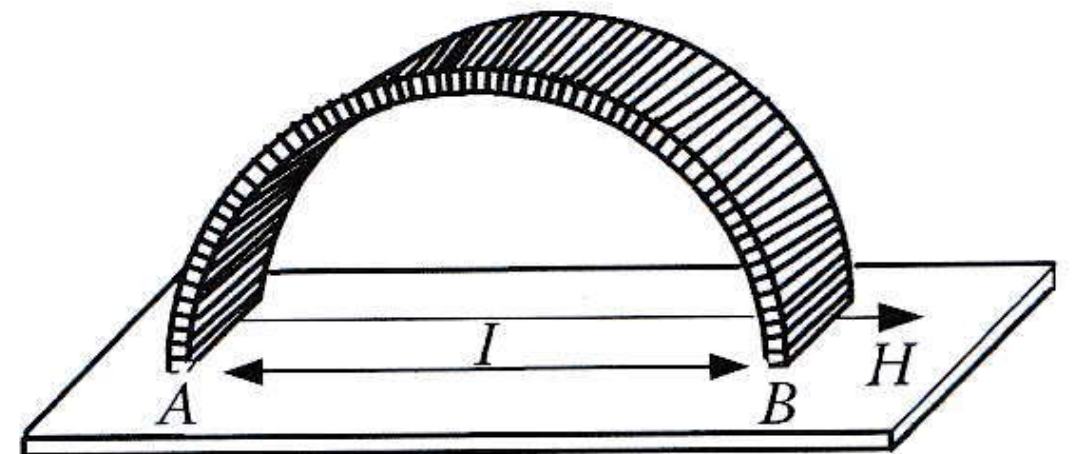
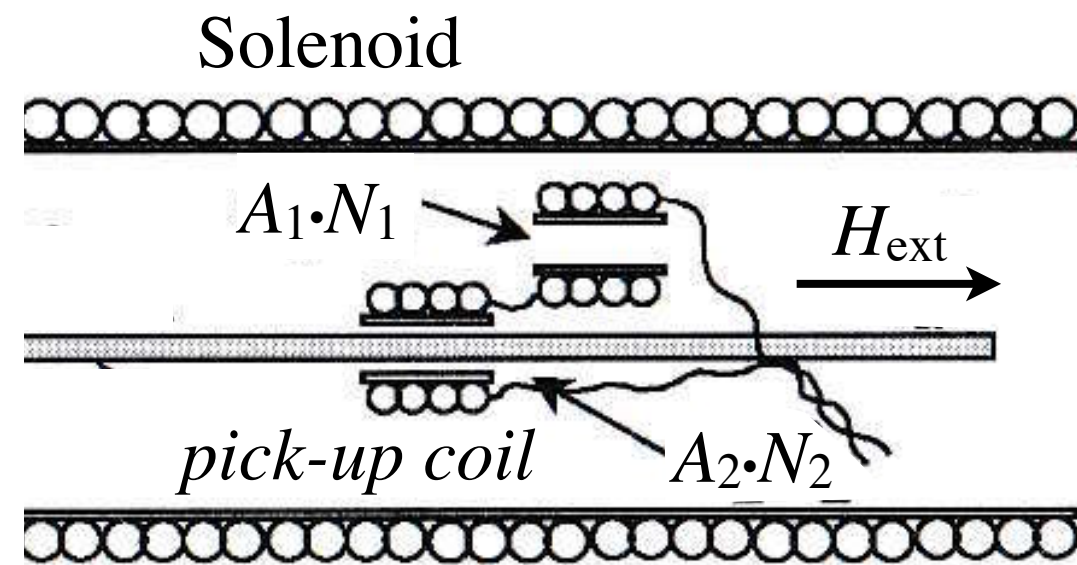


3.1 Magnetic Measurements

a) Inductive methods

Measurement of closed circuit samples

- Measurement of field strength:
 - Signal induced in compensation coil can also be used to measure the **effective internal field**
 - Maxwell's equations: tangential component of magnetic field must be equal on both sides of sample surface (if no current is flowing in sample surface)
 - → A coil placed close to the surface may therefore measure the internal field
 - With this technique the unsheared magnetization curve can be measured even for short samples
 - Alternative: Rogowski-Chattok coil



3.1 Magnetic Measurements

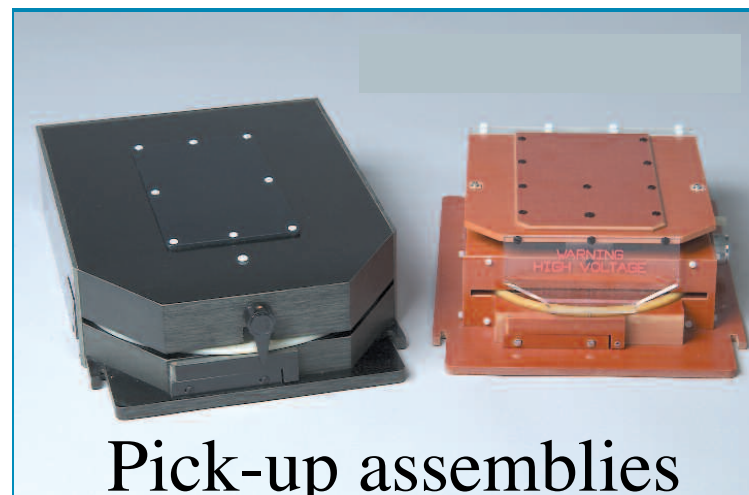
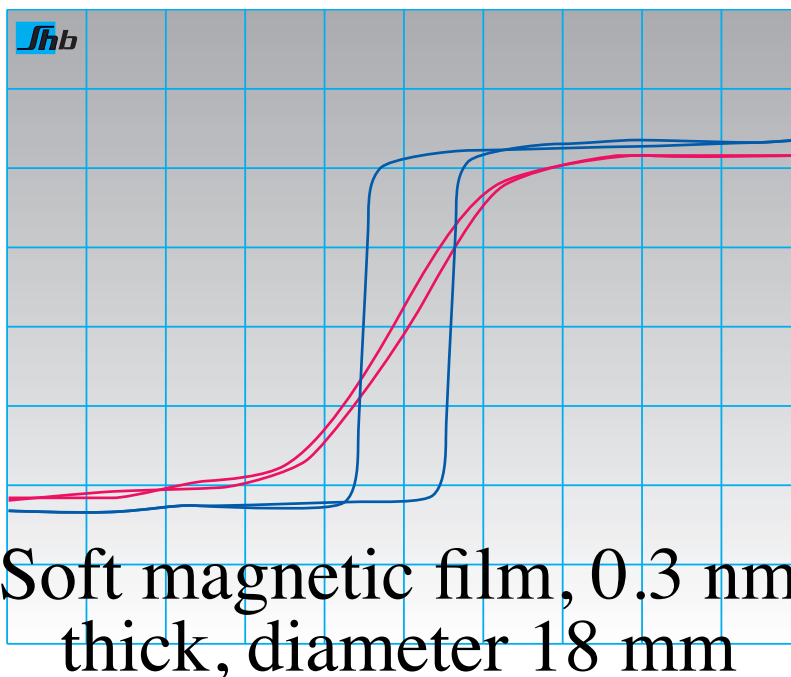
a) Inductive methods

Loop tracer for magnetic films

- Highly sensitive commercial instrument to inductively measure $M(H)$ loops in soft magnetic films
- Helmholtz coils: field up to 100 mT
- Frequency 1 - 10 Hz
- Alternative to Vibrating Sample Magnetometer

MESA

<http://www.shbinstruments.com>

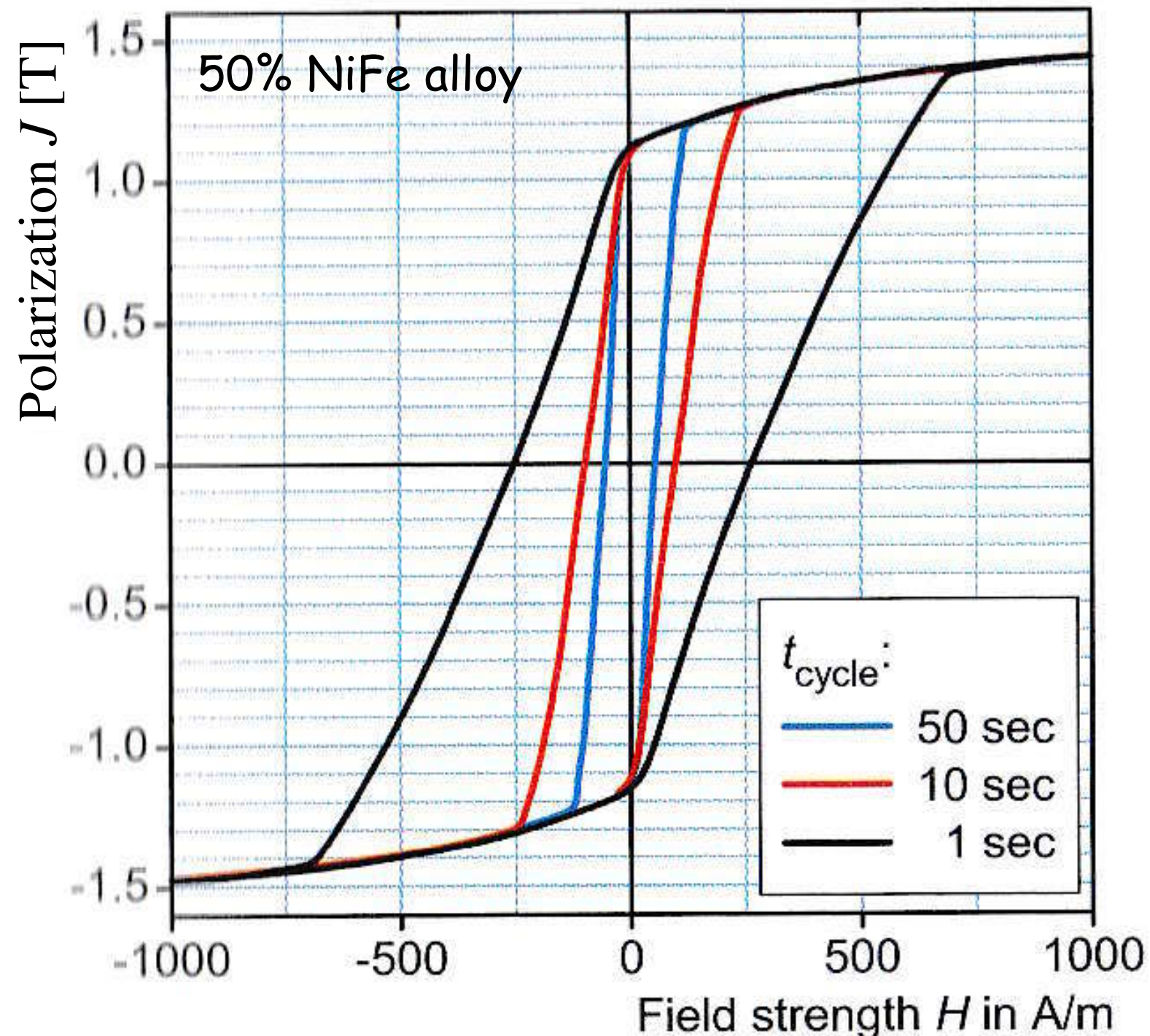


3.1 Magnetic Measurements

a) Inductive methods

(Quasi-)static magnetization

- Quasistatic $M(H)$ measurement: rate of magnetization dB/dT and time for complete hysteresis cycle have to be low enough to eliminate all dynamic effects (like eddy currents, relaxation processes etc.)
- Quasistatic loop is narrower than AC loop;
- Quasistatic coercivity is always lower than dynamic coercivity

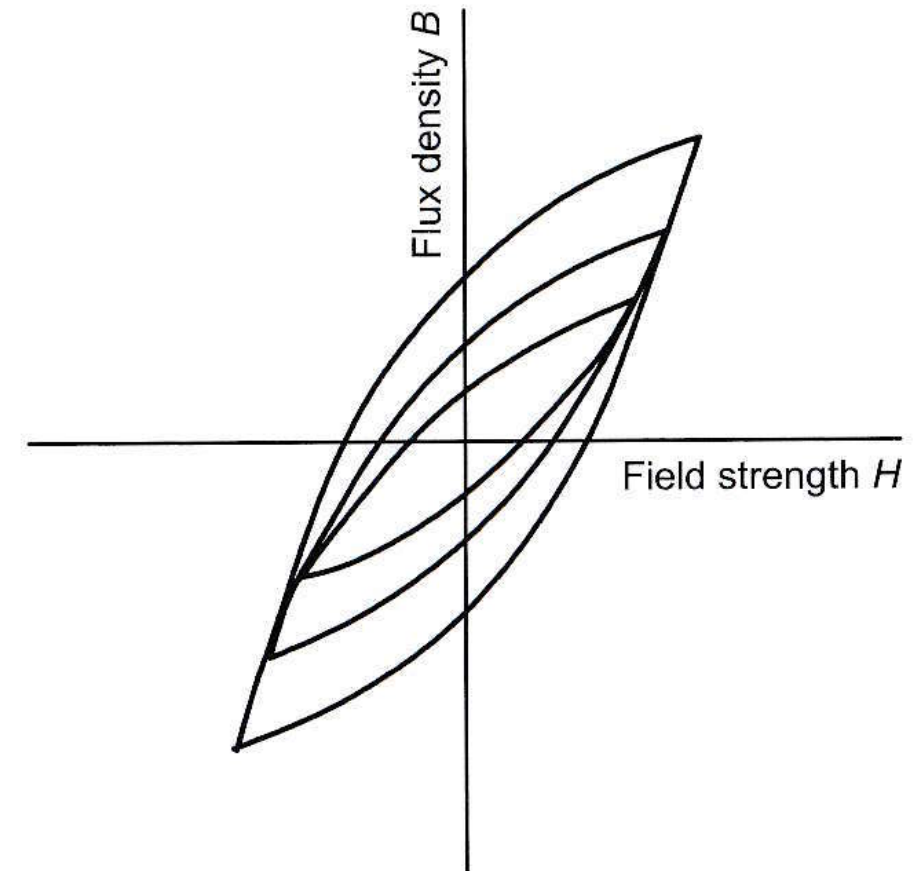


3.1 Magnetic Measurements

a) Inductive methods

Dynamic magnetization

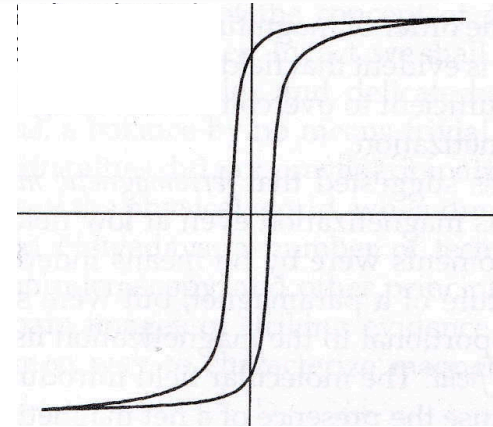
- AC magnetization at **low** excitation level:
 - Field amplitude below coercive field (Rayleigh region)
 - Relationship between AC flux density and AC field strength can be represented by ratio factor (permeability) and phase angle (magnetic loss angle due to eddy currents)
 - Can be described in terms of classical eddy current theory



3.1 Magnetic Measurements

a) Inductive methods

Dynamic magnetization



- AC magnetization at **high** excitation level:
 - AC excitation into region of maximum permeability and up to saturation
 - Severe distortions of magnetization due to non-linear behaviour of ferromagnetic material (S-like or rectangular hysteresis loops). Reason:
 - At high excitation level: material is subjected to rapid changes of $H(t)$ or $B(t)$
 - Local magn. flux density cannot follow changes due to eddy currents
 - Consequence: Magnetization curve depends on frequency and mode of excitation
 - 2 modes of dynamic magnetization:
 - **Voltage -controlled** magnetization: induced voltage is controlled to be sinusoidal (by feedback loop) \rightarrow sinusoidal flux density $B(t) \rightarrow$ magnetizing current $I(t)$ becomes dependent. Recommended as IEC standard
 - **Current-controlled** magnetization: magnetizing current is controlled to be sinusoidal (by high-impedance power source) \rightarrow sinusoidal field strength $H(t) \rightarrow U(t)$ and $B(t)$ become dependent.

↓ cont.

3.1 Magnetic Measurements

a) Inductive methods

Dynamic magnetization

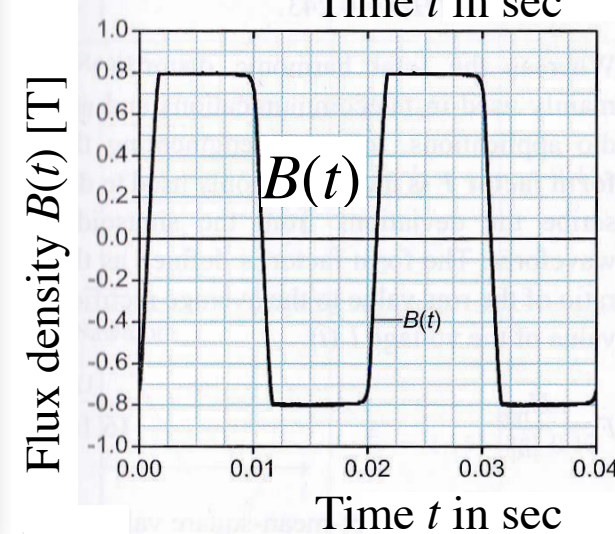
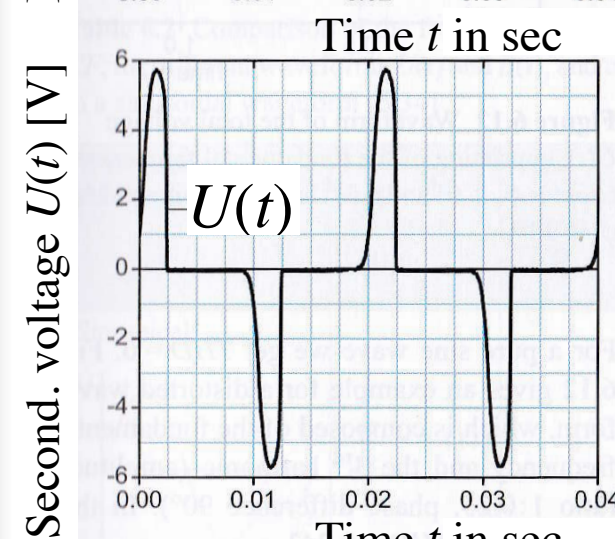
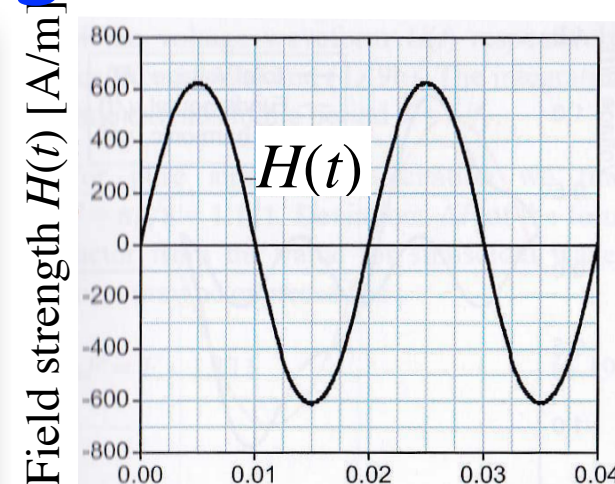
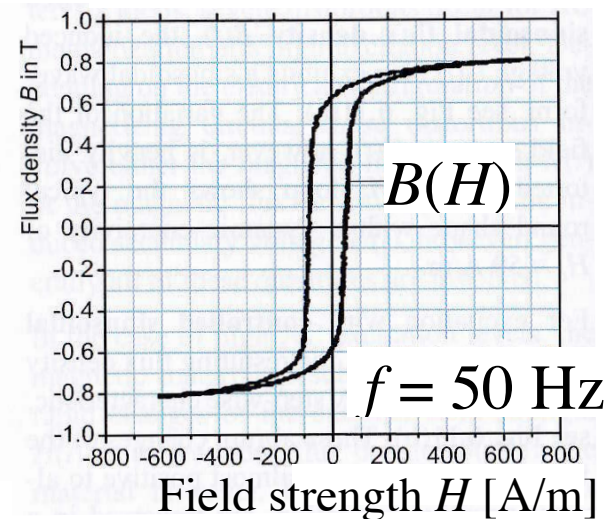
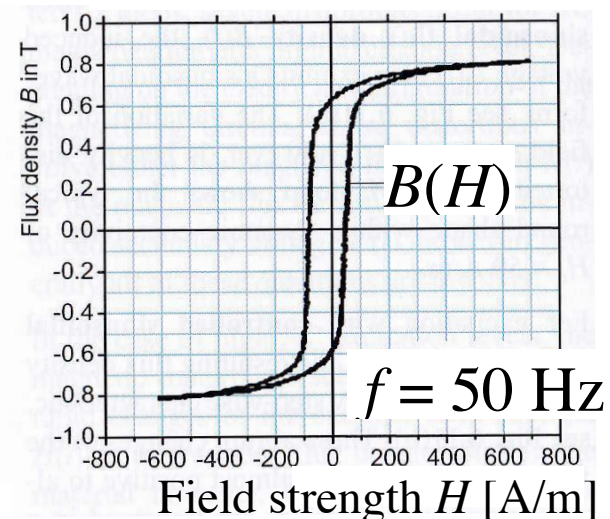
high excitation level

Controlled sinusoidal
flux density $B(t)$

$$U(t) = (-)N_2 \cdot A_{\text{Fe}} \frac{dB}{dt}$$

→ Induced voltage
 $U(t)$ also exhibits
sinusoidal waveform

→ $H(t)$ heavily
distorted

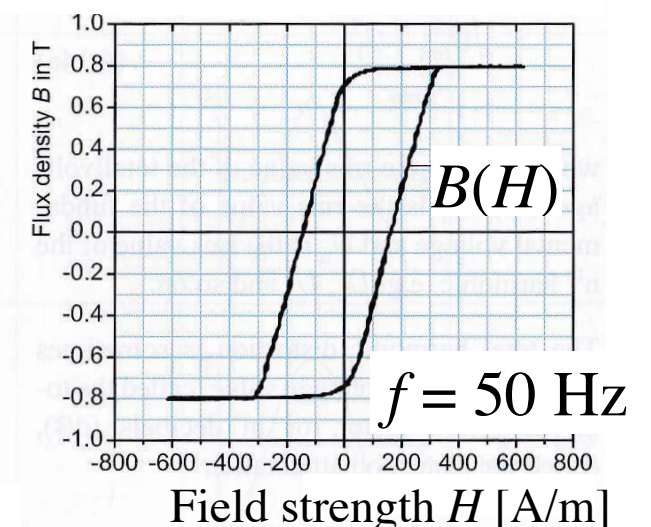


Controlled sinusoidal
field strength $H(t)$

→ Step-wise char-
acteristics of $B(t)$.
Abrupt changes of
 $B(t)$ are traversed in
short time interval

→ Spikes in induced
voltage $U(t)$

→ Large eddy
currents, sheared loop

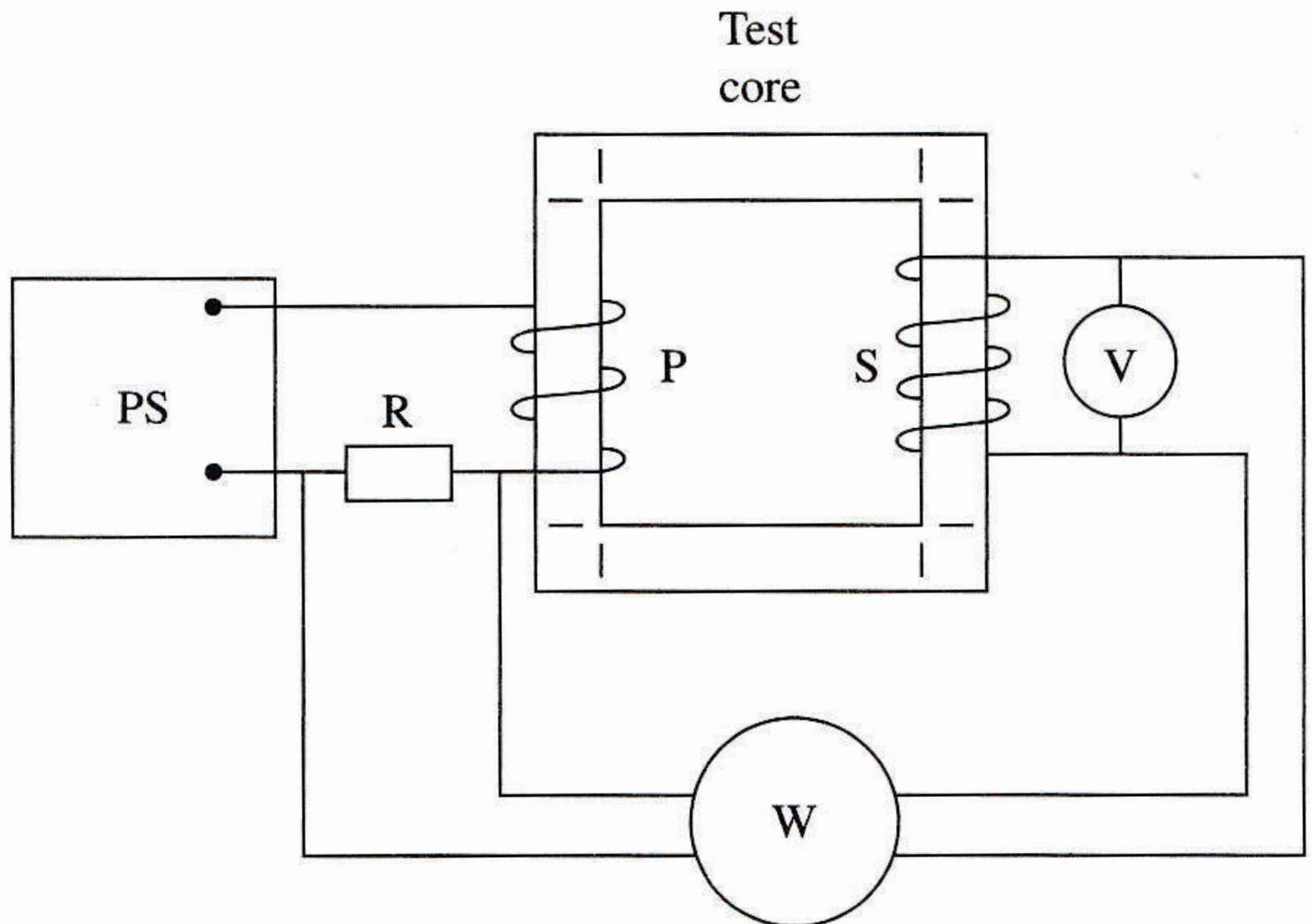


3.1 Magnetic Measurements

a) Inductive methods

Loss measurement

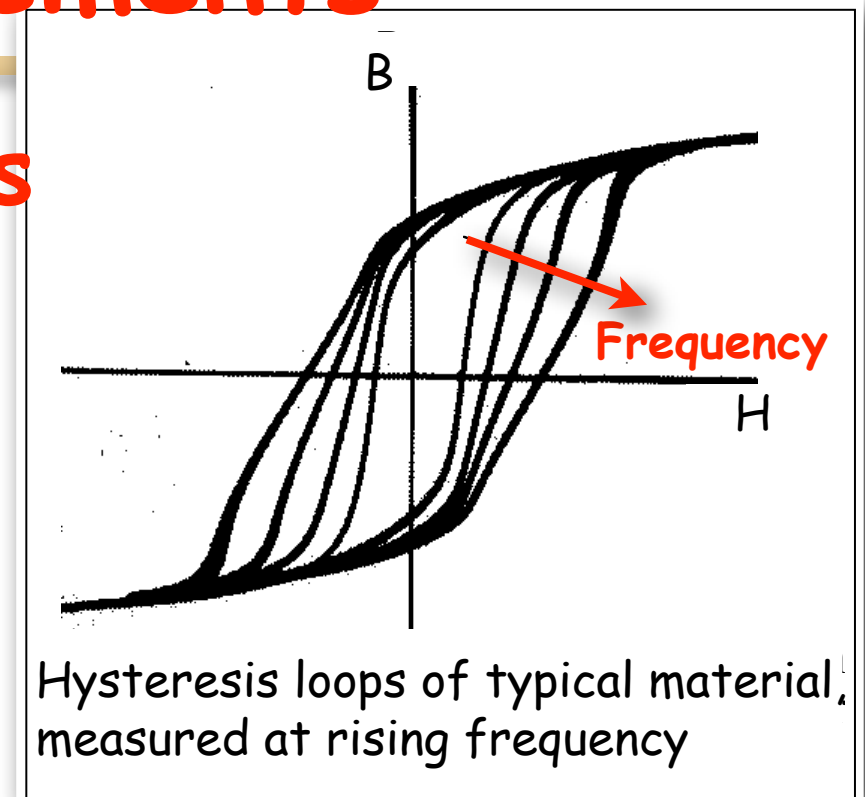
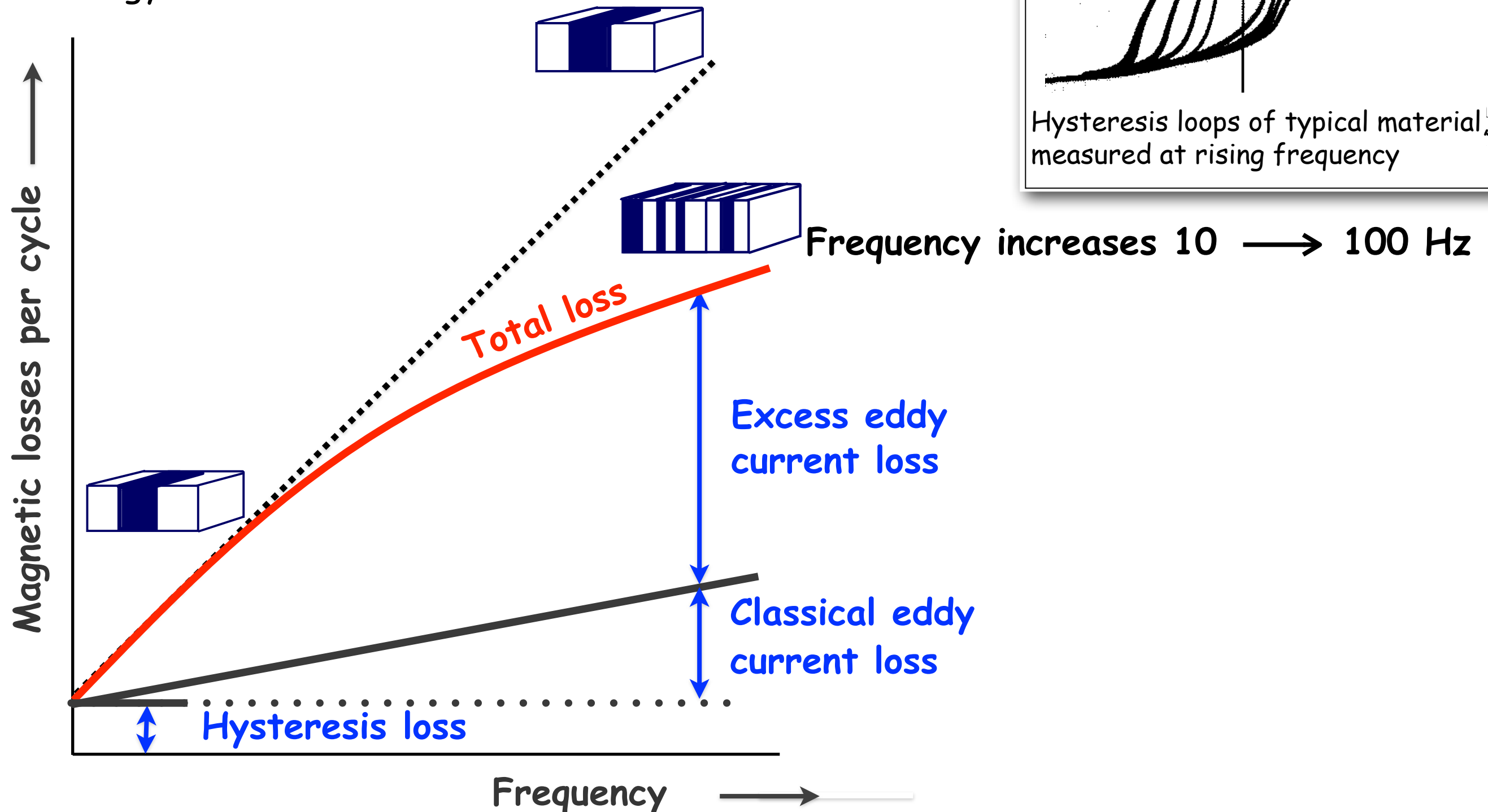
- Wattmeter method (e.g.)
 - Measures core loss (not copper loss)
 - Losses are measured at certain maximum flux density and given frequency
 - Total weight of sample is recorded, and losses are reported in W/kg



3.1 Magnetic Measurements

a) Inductive methods Loss measurement

- Energy loss

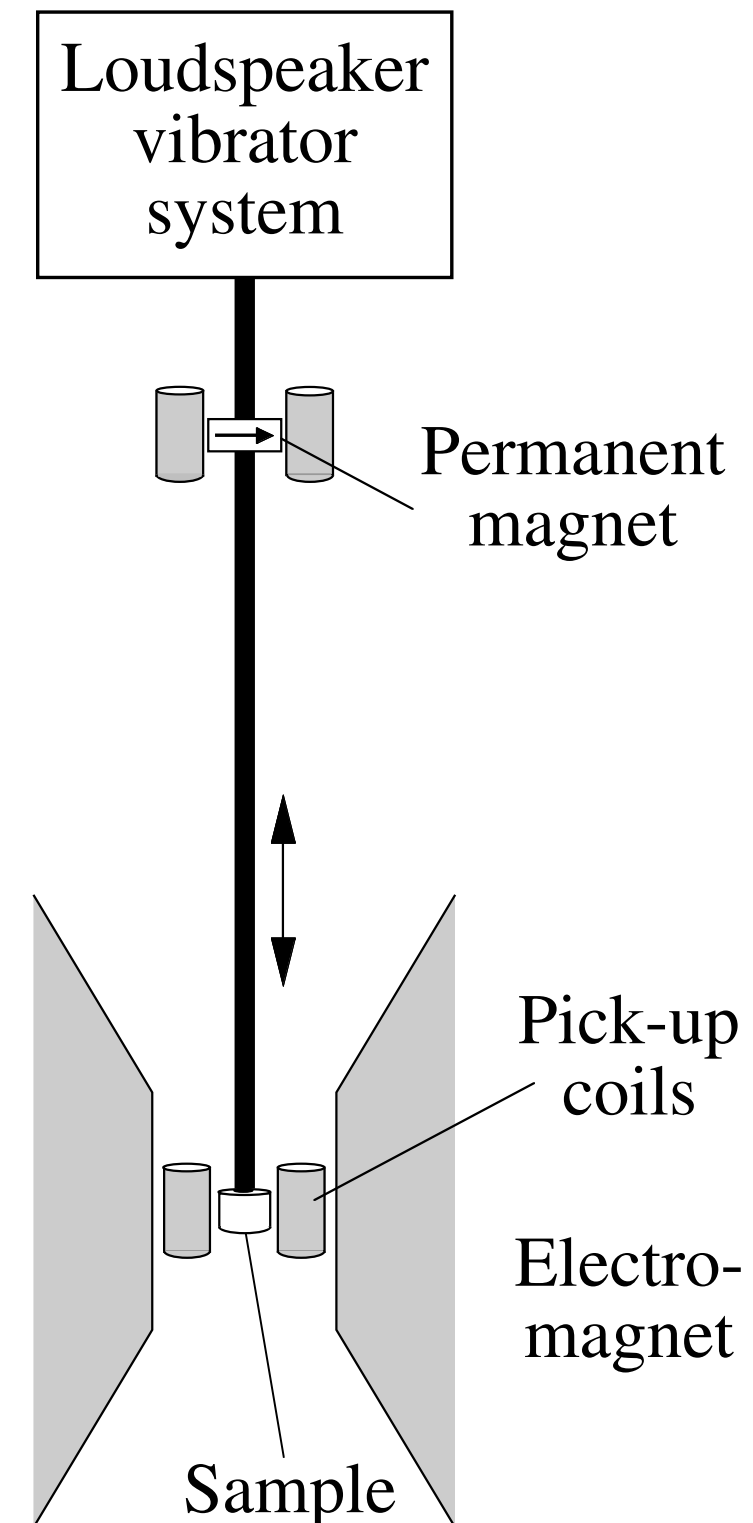


3.1 Magnetic Measurements

a) Inductive methods

Vibrating Sample Magnetometer (VSM)

- Sample placed inside magnet and vibrated perpendicular to field direction (frequ. ~ 100 Hz, vibration ampl. ~ 0.1 mm)
- Oscillating magnetic field of moving sample induces alternating voltage in pick-up coil, whose magnitude is proportional to magnetic moment of sample
- The pick-up signal is amplified with lock-in amplifier and compared with the signal induced in a pair of reference coils by a permanent magnet or by some variable capacitor setup (only sensitive to vibration frequency)
- Strong external fields can be applied (superconducting magnets for hard magnetic materials)
- Since sample is well-separated from the pick-up coils, it can be surrounded by cooling or heating devices
- The sample magnetization is static in the VSM, so that no eddy current effects have to be considered
- The shape of the sample is largely arbitrary



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Loudspeaker



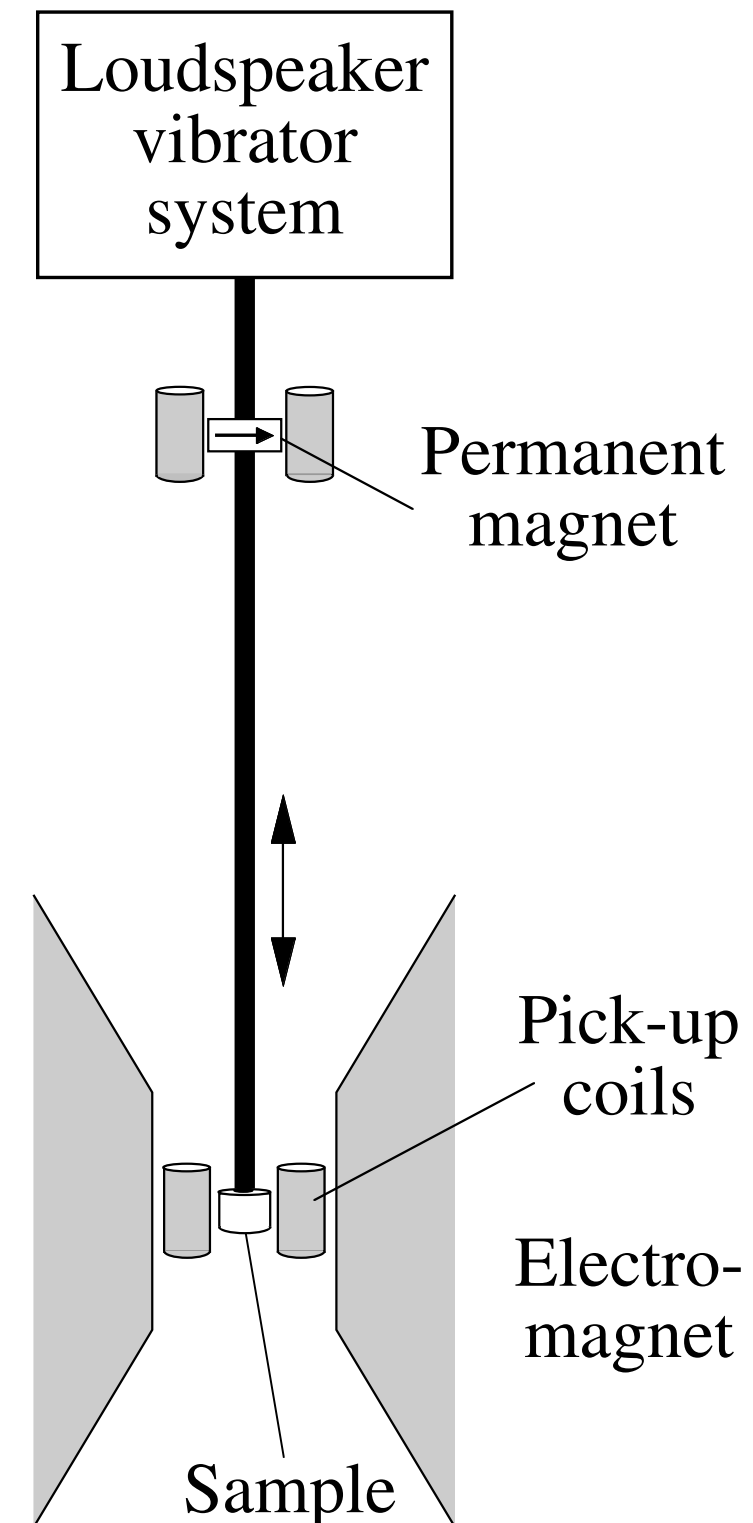
Courtesy Lakeshore

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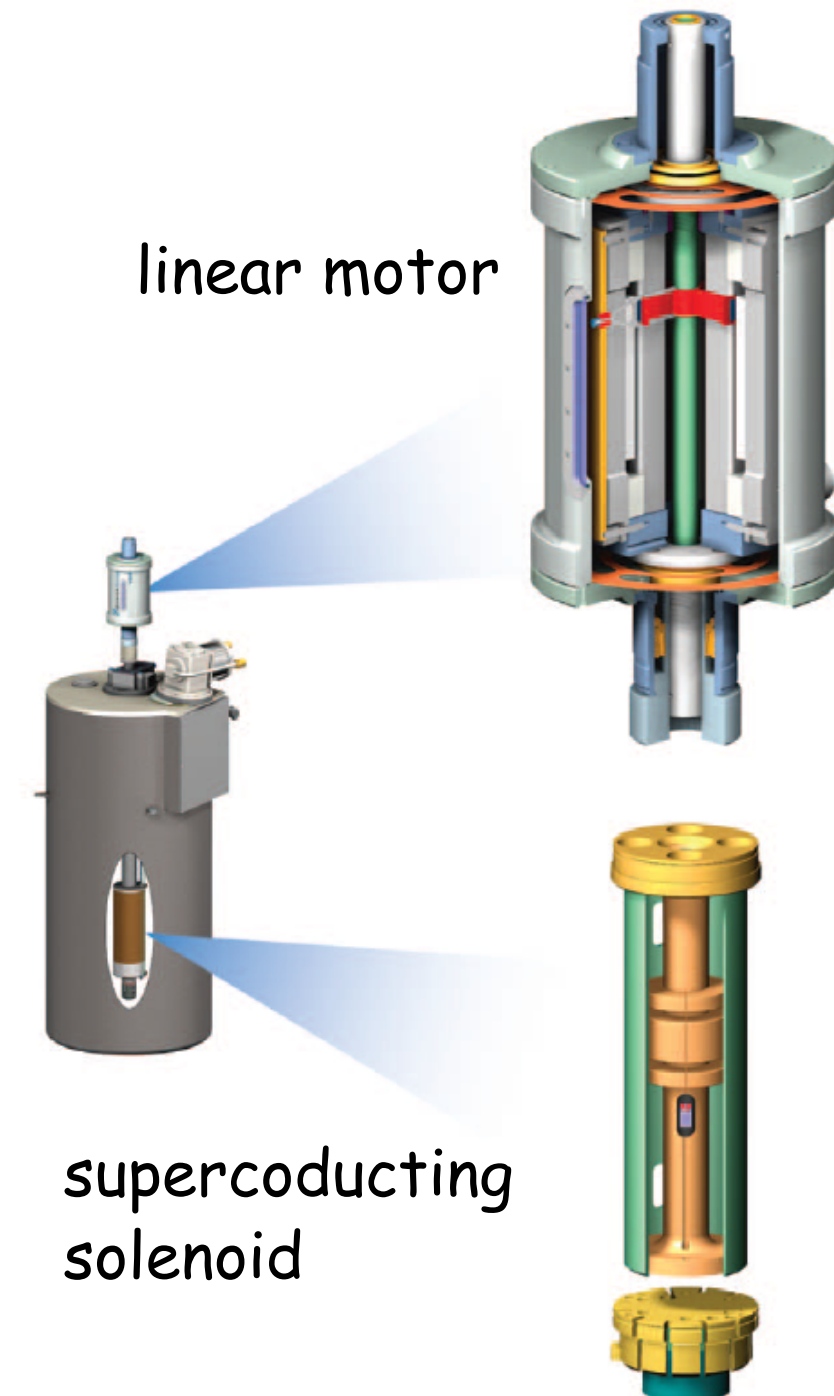


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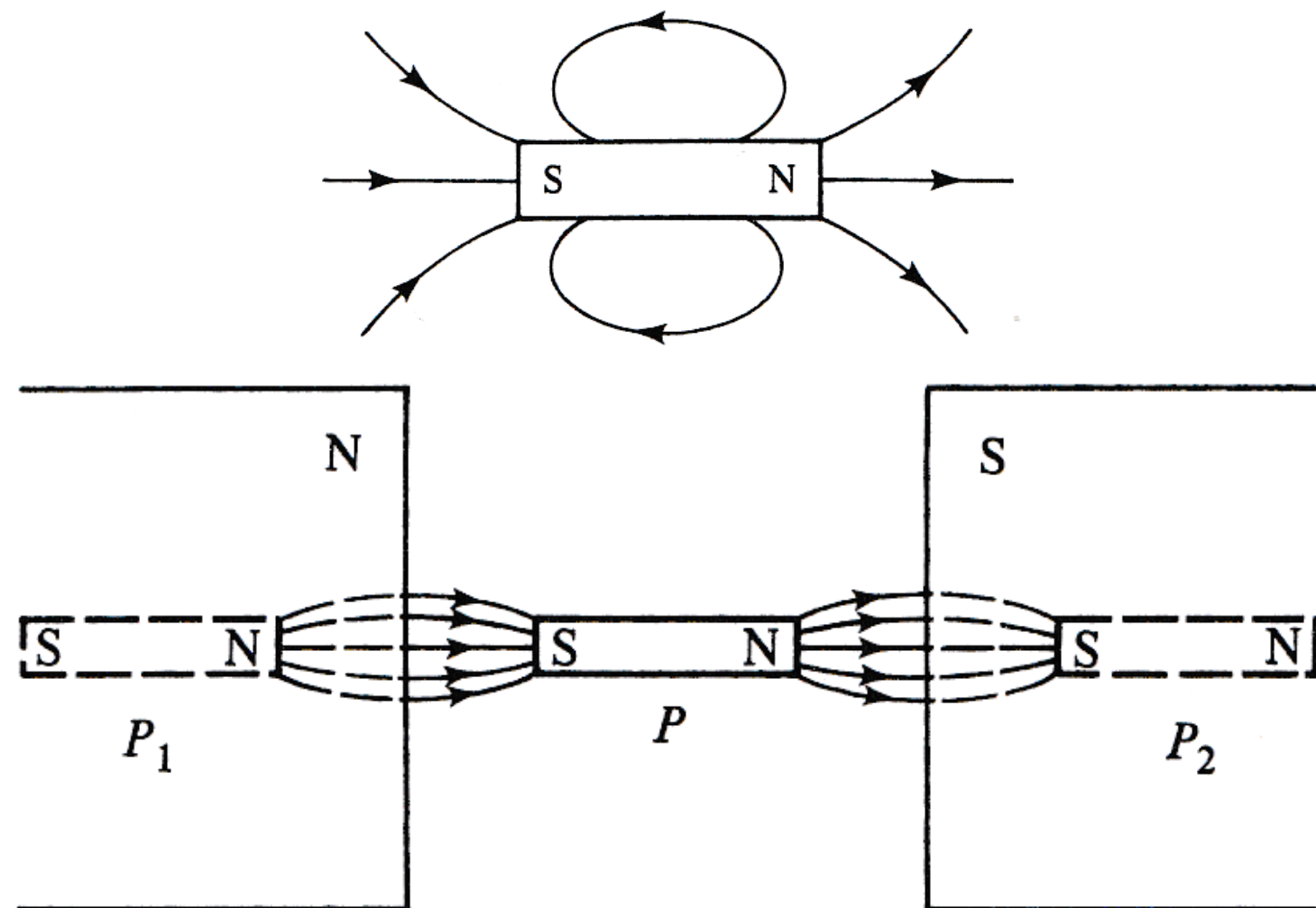
Courtesy Quantum Design.

3.1 Magnetic Measurements

a) Inductive methods

Vibrating Sample Magnetometer (VSM)

- Direct method. Ideally, no calibration would be needed to derive the magnetization of the sample if pick-up coil geometry and sample volume are known and if magnetic field could be generated by a simple air coil
- However: for electromagnets the pole material interacts with the measuring process: "mirror images" of the sample are formed by the presence of soft magnetic iron yokes
- → Mirror images also induce voltage in pick-up coil
- Strength of mirror images depends on permeability of iron yoke that in turn depends on the induction level in magnet
- → VSM must be calibrated by replacing sample by nickel sample of the same size and shape, and to rely on the accurately known saturation magnetization of nickel

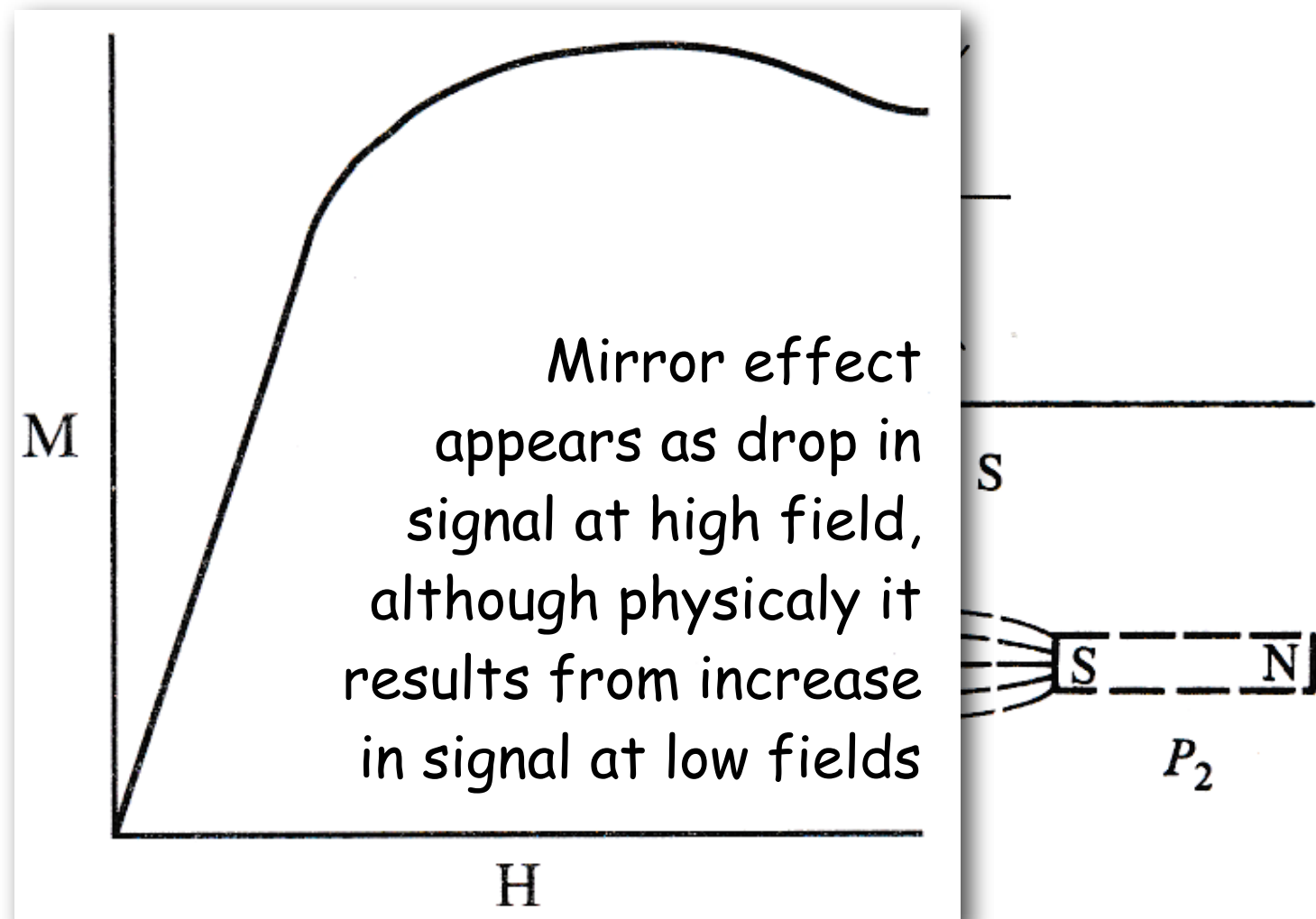


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- → Mirror images also induce voltage in pick-up coil
- Strength of mirror images depends on permeability of iron yoke that in turn depends on the induction level in magnet
- → VSM must be calibrated by replacing sample by nickel sample of the same size and shape, and to rely on the accurately known saturation magnetization of nickel

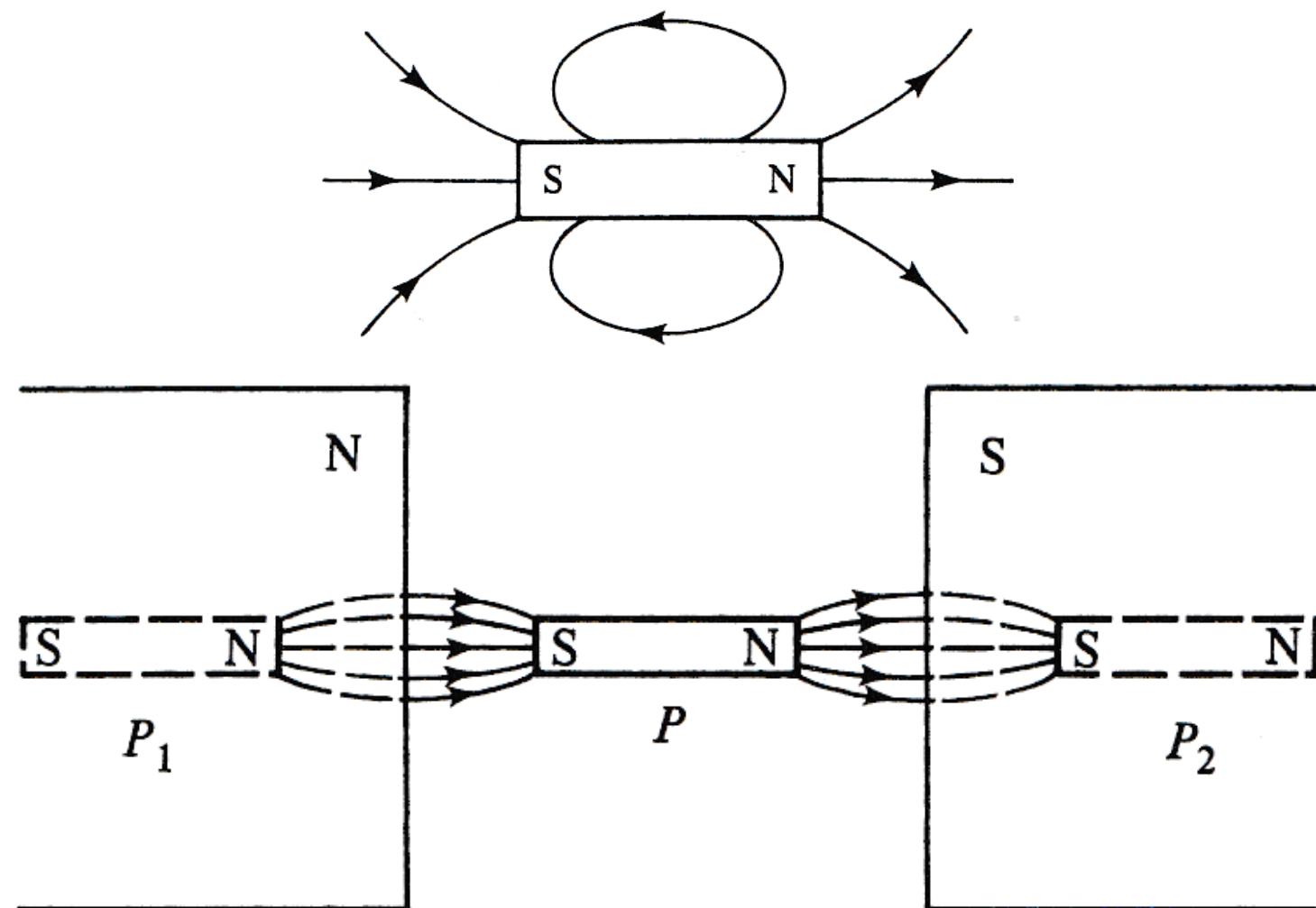


3.1 Magnetic Measurements

a) Inductive methods

Vibrating Sample Magnetometer (VSM)

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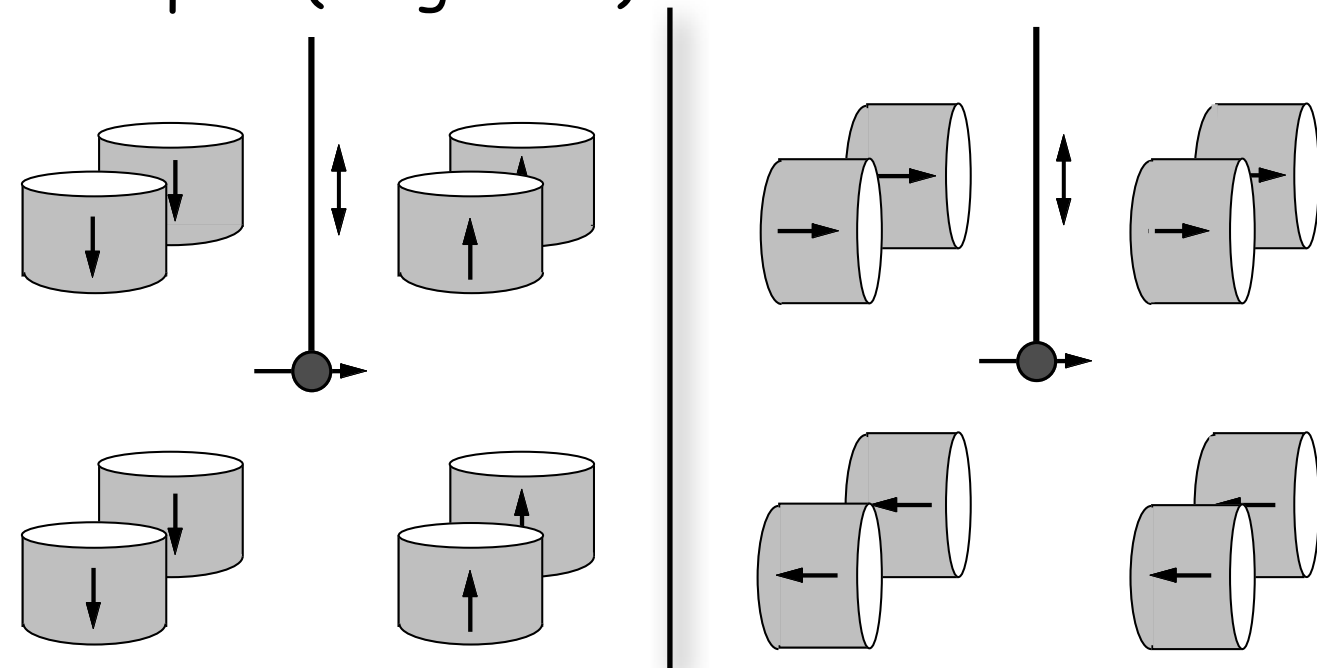


3.1 Magnetic Measurements

a) Inductive methods

Vibrating Sample Magnetometer (VSM)

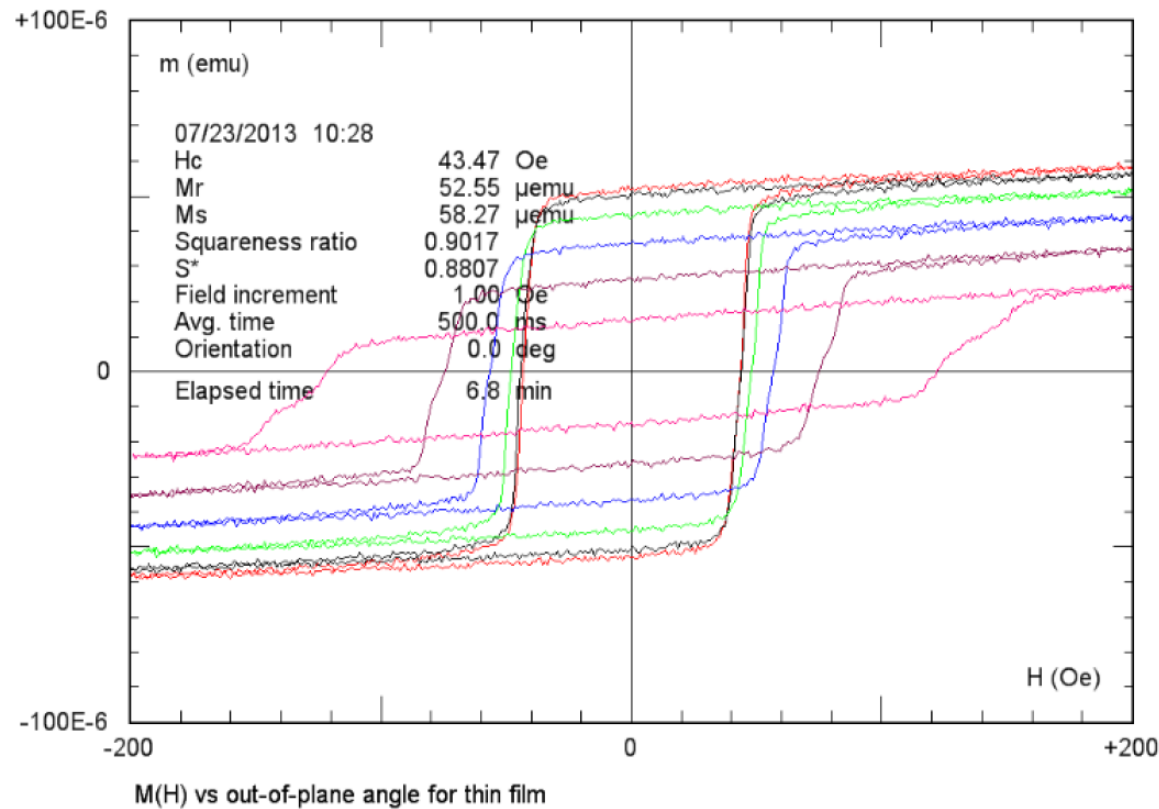
- VSM measures magnetic moment and therefore **magnetization M** - whereas fluxmeter methods measure flux density B
- VSM is best method to measure the saturation magnetization M_s of any material because high field can be applied to reach saturation
- VSM less useful for measurement of other parameters of magnetization curve in soft magnetic materials because of demagnetization effects (real field unknown).
Exception: thin films. For high-anisotropy or hard magnetic materials, however, the VSM is the preferred instrument for many kinds of magnetic measurements
- Sensitivity: 10^{-5} emu = 10^{-8} Am² → small samples (< 1 gramm)
- Pick-up coil arrangements to measure longitudinal and transverse magnetization components
- SQUID magnetometer: high-sensitive variant of VSM. Pick-up signal transformed to SQUID device outside magnet



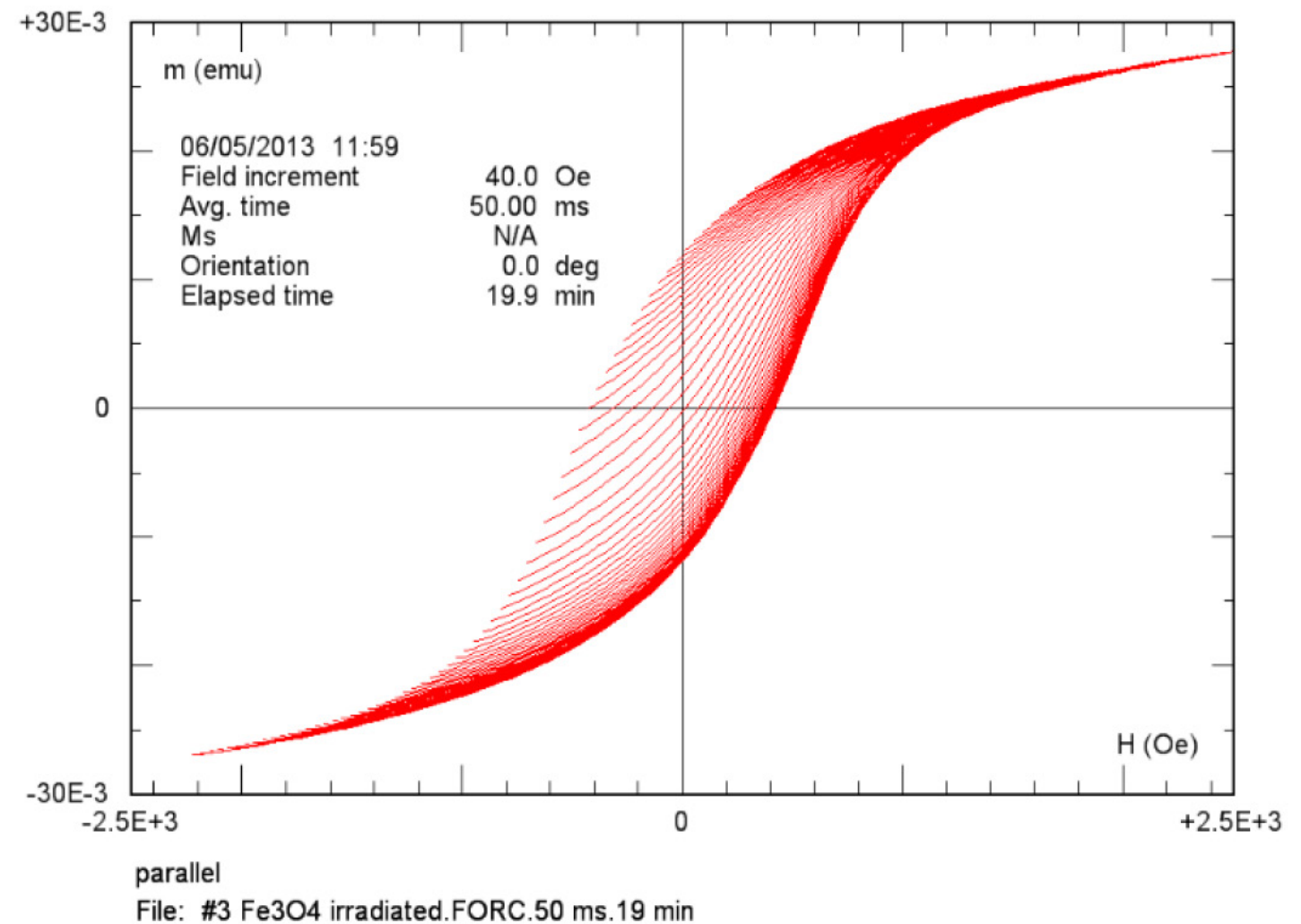
3.1 Magnetic Measurements

a) Inductive methods

Vibrating Sample Magnetometer (VSM)



Examples



Courtesy Lakeshore

3.1 Magnetic Measurements

a) Inductive methods

Sample surrounded by coil, in which voltage is induced when magnetization of sample is changed or when sample is moved. Voltage is integrated → signal proportional to magnetization

b) Magnetometric methods

For finite samples: demagnetizing field, which is proportional to mean magnetization ($H_{\text{dem}} = -N\bar{M}$), is measured

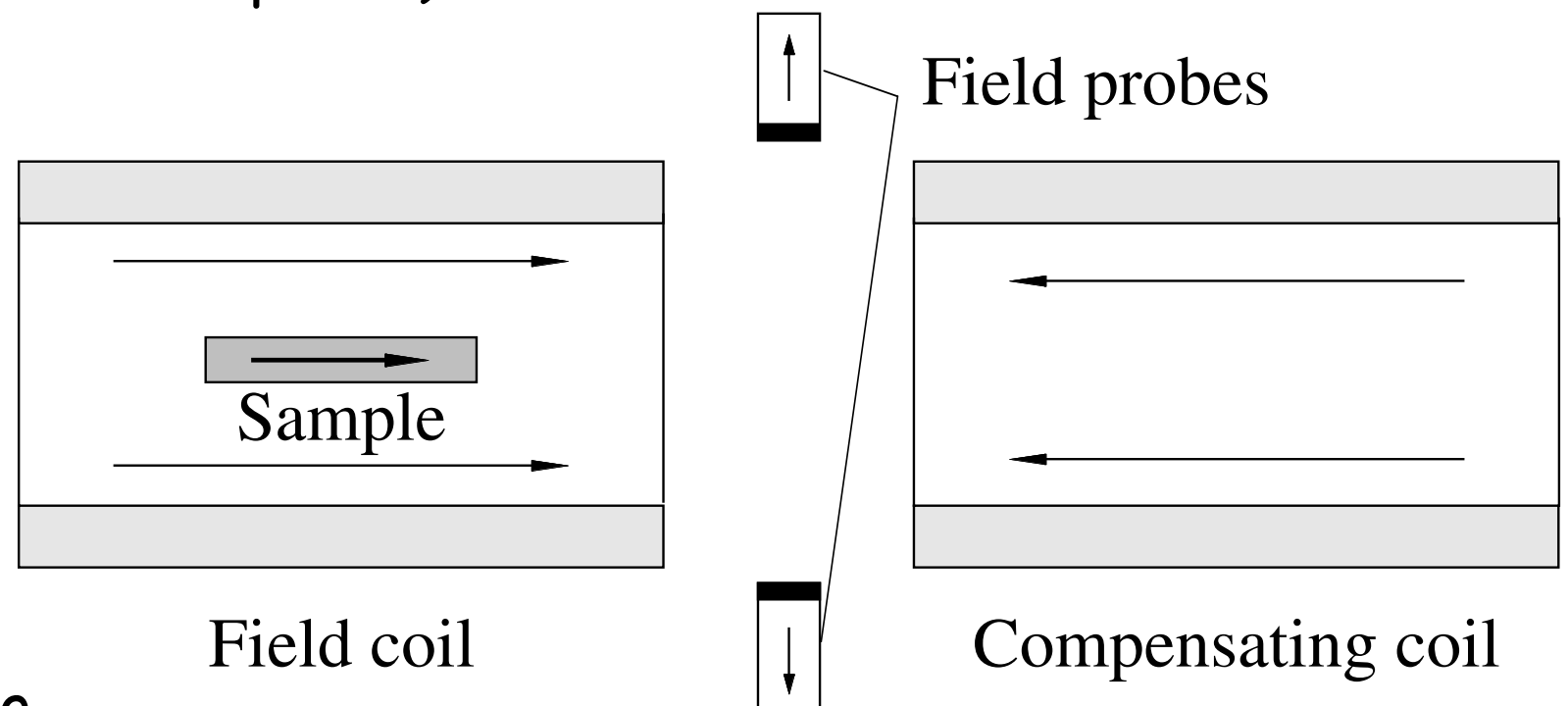
c) Optical magnetometry

Surface magnetization is measured by magneto-optic effect, useful for thin films where signal of inductive or magnetometric methods are too weak

3.1 Magnetic Measurements

b) Magnetometric measurements

- Magnetometer measures dipolar field generated by magnetized sample with the help of field detection device (like Hall probe)
- In shown arrangement the difference signal of the two probes is proportional to the magnetic moment of the sample, and insensitive to the driving field
- Probes must be placed sufficiently far away from sample, so that only its dipolar field is detected → samples should be small and short, but can be of any shape → hard and high-anisotropy materials better suited than soft magnetic materials where demagnetization will dominate the intrinsic properties of short samples
- Advantages: (i) any sample shape, (ii) arbitrarily slow magnetization processes can be followed, (iii) sample can be easily exposed to various environmental conditions such as high or low temperature or mechanical stress.



3.1 Magnetic Measurements

c) Optical magnetometer

Magneto-optical Kerr effect

$$\mathbf{D} = \boldsymbol{\varepsilon} \mathbf{E} = \varepsilon \begin{pmatrix} 1 & -i Q m_3 & i Q m_2 \\ i Q m_3 & 1 & -i Q m_1 \\ -i Q m_2 & i Q m_1 & 1 \end{pmatrix} \mathbf{E}$$

$$= \varepsilon \mathbf{E} + i \varepsilon Q \mathbf{m} \times \mathbf{E}$$

\mathbf{E} : electric vector of light wave

\mathbf{D} : dielectric displacement vector

(= vector of light after reflection)

m_i : components of magnetization vector (cubic crystal)

$\boldsymbol{\varepsilon}$: dielectric tensor

Q : material constant ($\sim M_s$, complex, determines strength of rotation)

3.1 Magnetic Measurements

c) Optical magnetometer

Magneto-optical Kerr effect

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$$= \varepsilon \mathbf{E} + i \varepsilon Q \mathbf{m} \times \mathbf{E} \longrightarrow \text{concept of Lorentz force}$$

\mathbf{E} : electric vector of light wave

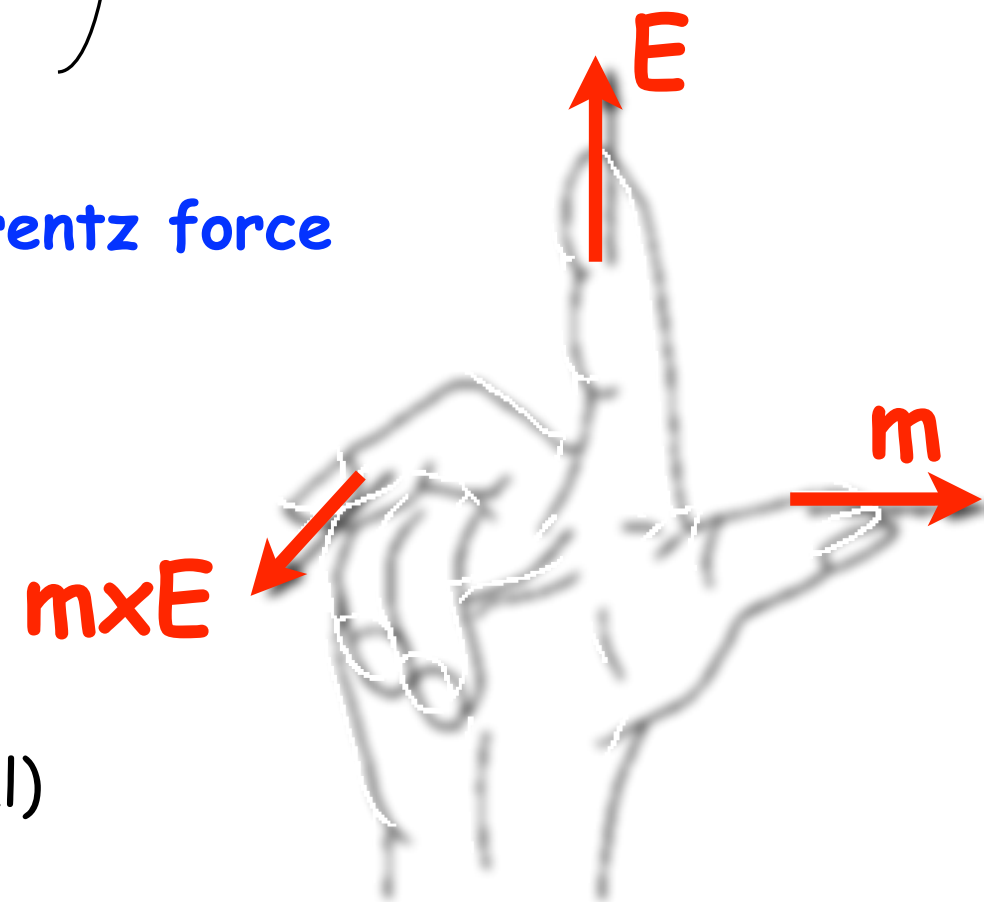
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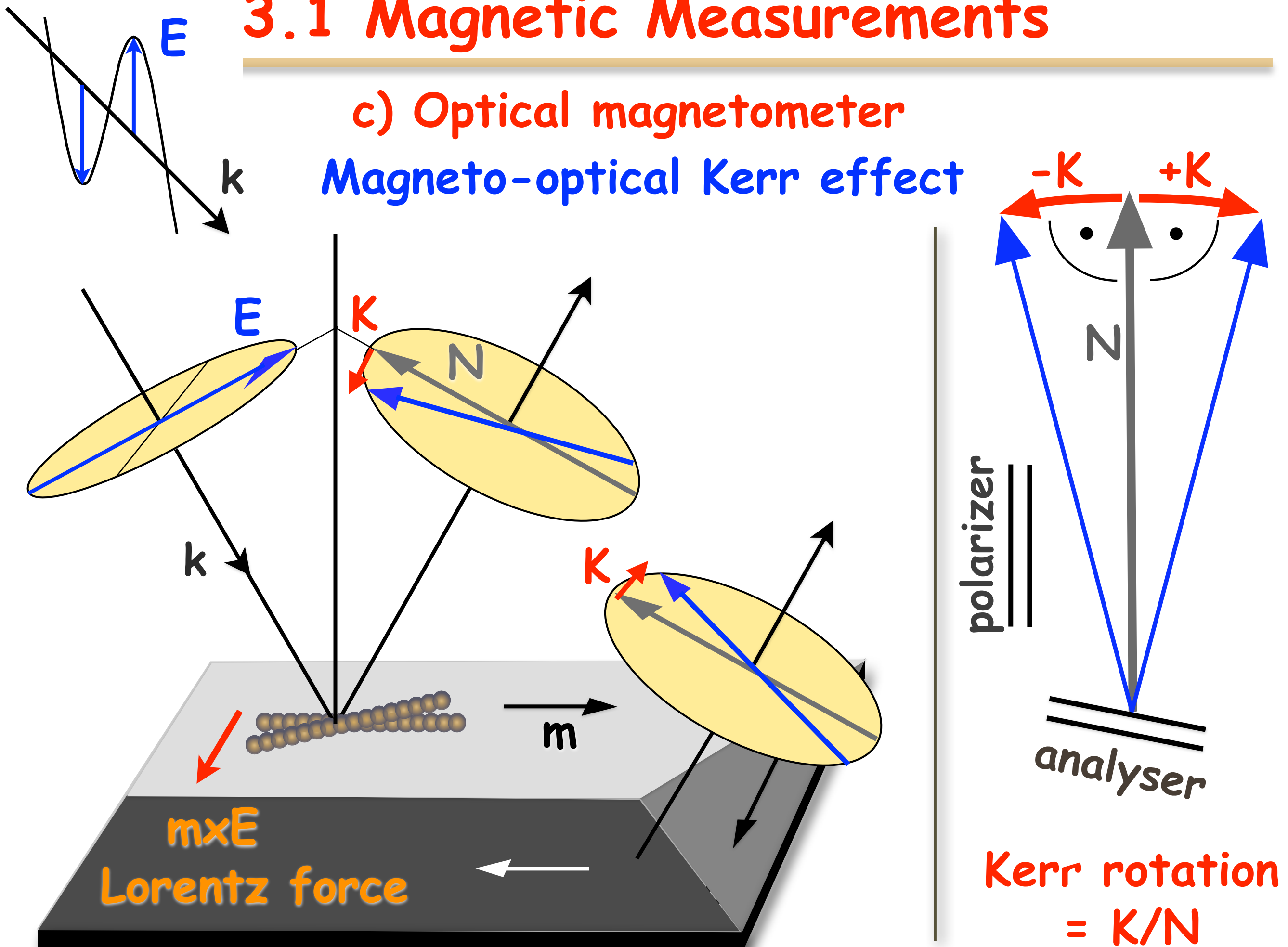
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3.1 Magnetic Measurements

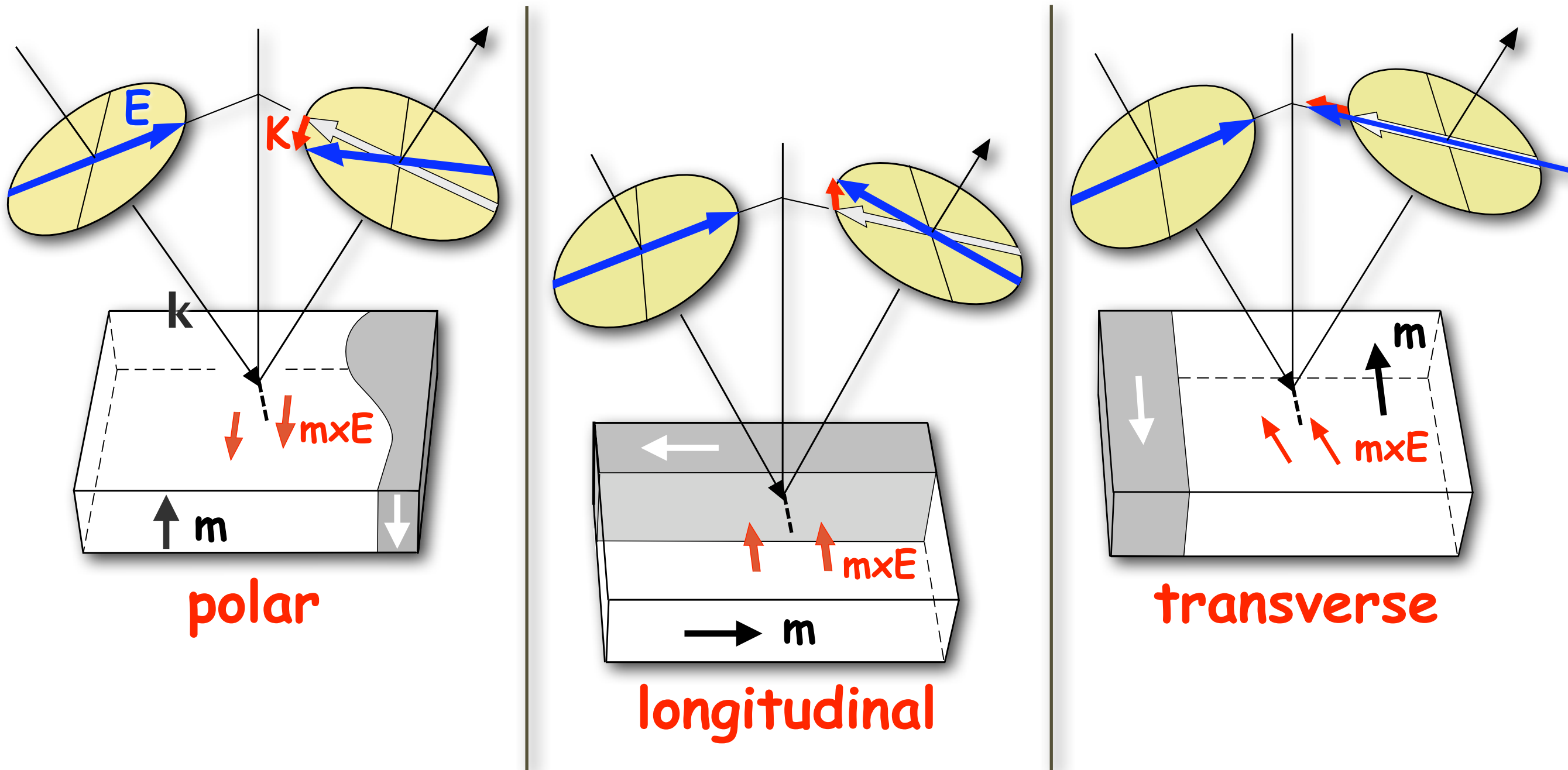
c) Optical magnetometer

Magneto-optical Kerr effect



3.1 Magnetic Measurements

c) Optical magnetometer Magneto-optical Kerr effect

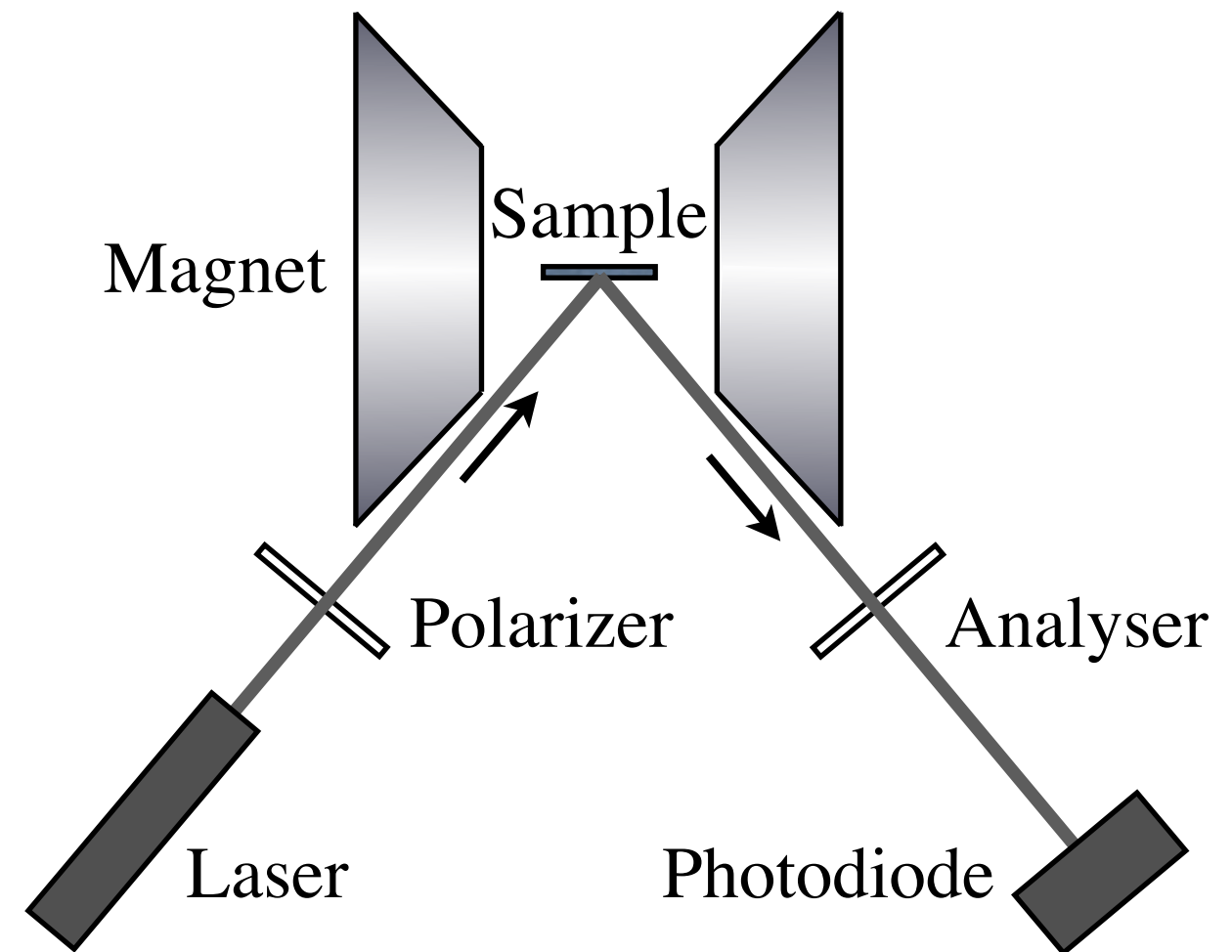


The Kerr effect causes a rotation of light, which is proportional to the magnetization component parallel to the reflected light beam

3.1 Magnetic Measurements

c) Optical magnetometer

- Magneto-optical Kerr effect is linear function of magnetization and therefore well-suited for magnetometry
- For non-transparent material optical magnetometry makes sense only for thin films for which surface magnetization is representative
- Advantages: (i) direct, (ii) quasi-static and dynamic measurements, (iii) Space-resolved measurements are possible by scanning over the surface. (iv) Optical measurements can be performed on-line during preparation or treatment of a material for example inside vacuum chamber
- Noise suppression: feed split-off part of laser light as reference signal into amplifier. If polarization of light is modulated by a spinning analyser or electro-optical device, the magnetic signal can be detected by a lock-in amplifier, thus achieving virtually unlimited sensitivity

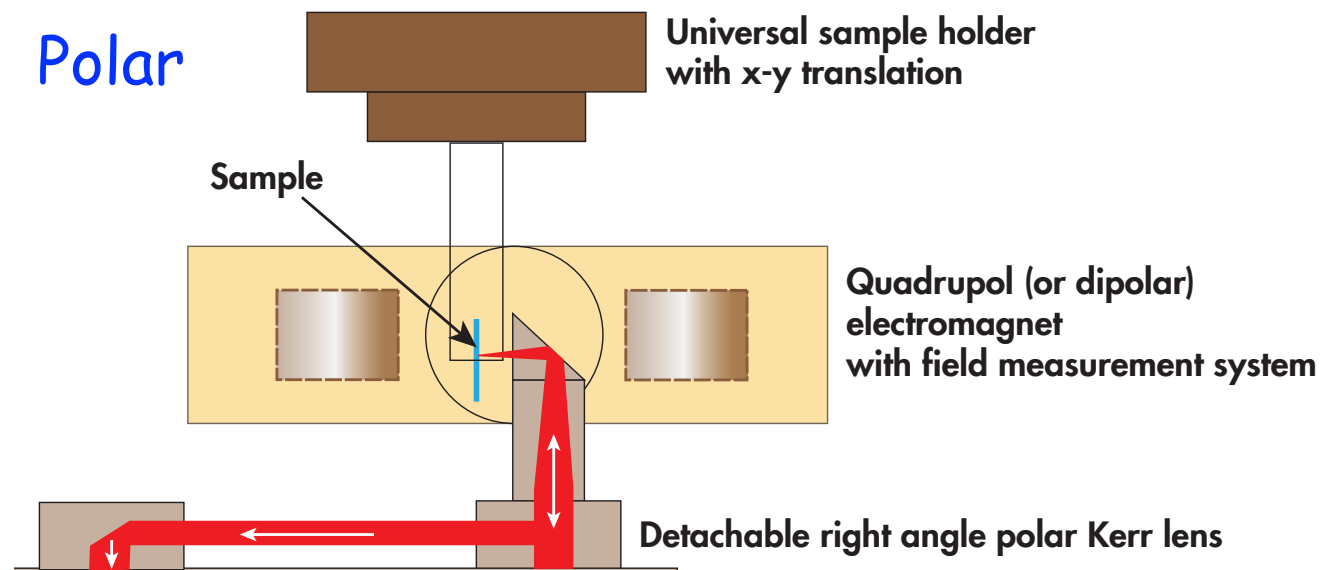


3.1 Magnetic Measurements

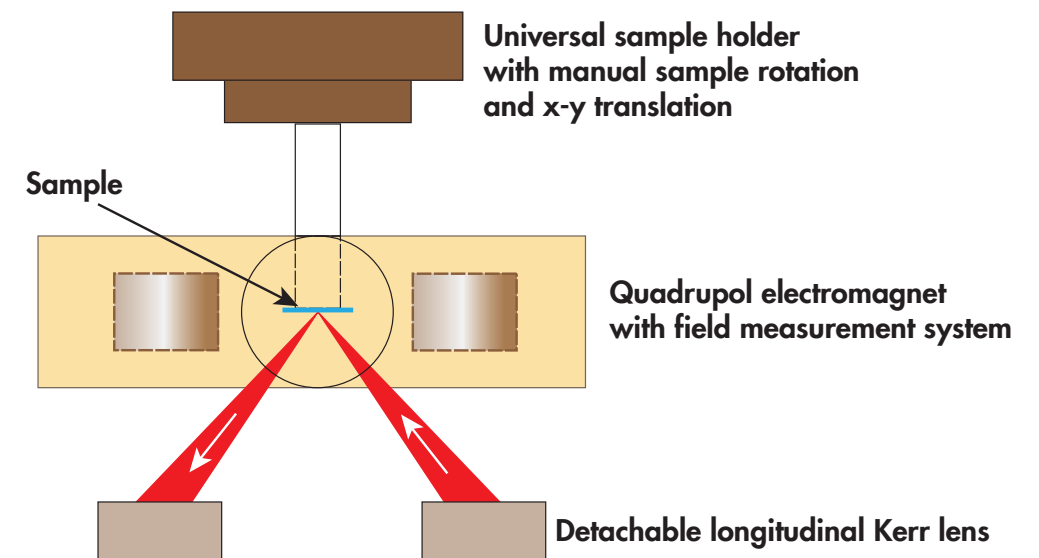
c) Optical magnetometer

NANOMOKE3®
<http://www.lot-qd.de>

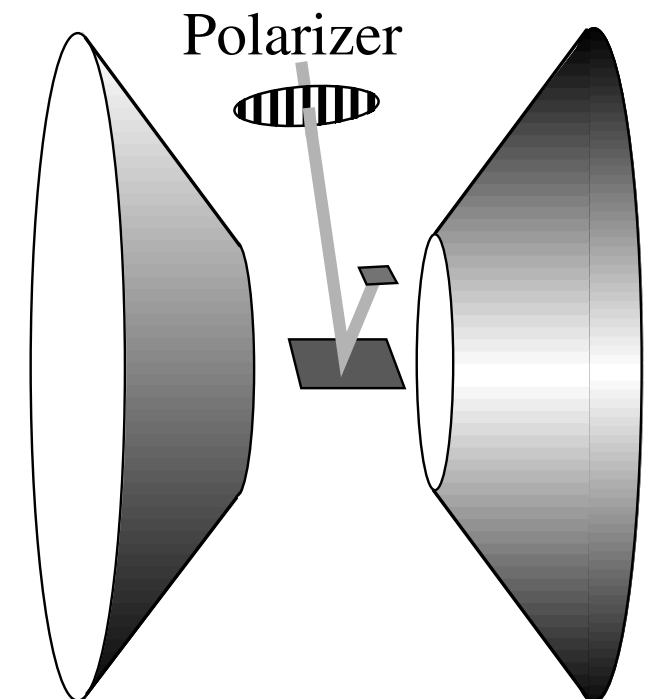
- Polar Kerr magnetometer (P-MOKE)



Longitudinal



- Use of transverse Kerr effect (T-MOKE)
 - Polarizer set parallel to the plane of incidence and analyser omitted
 - M -component perpendicular to the plane of incidence causes variation of the reflected intensity, which can be detected electronically
 - Fits nicely into electromagnet



3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

3.4 Dilatometric measurements

3.5 Domain methods

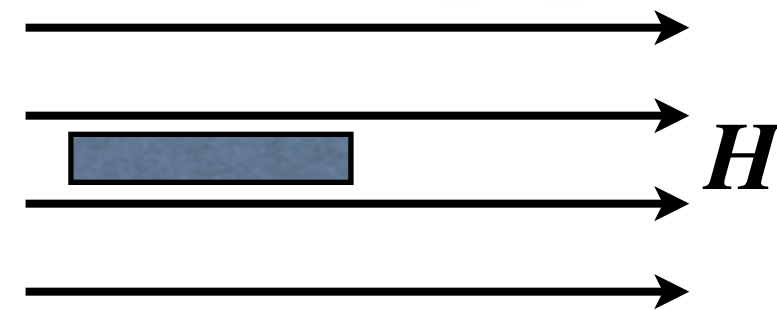
3.2 Mechanical Measurements

Principle:

Measurement of mechanical forces on magnetic sample

Two possibilities:

- A uniform field H , acting on uniformly magnetized sample of magnetization M and volume V , generates a mechanical torque $T_m = \mu_0 V H \times M$
- Gradient of non-uniform field generates a mechanical force $F_m = \mu_0 V \text{grad}(M \cdot H)$



a) Torque magnetometer

Most direct method to measure anisotropy

b) Field gradient methods

Faraday Balance and Alternating Gradient Magnetometer

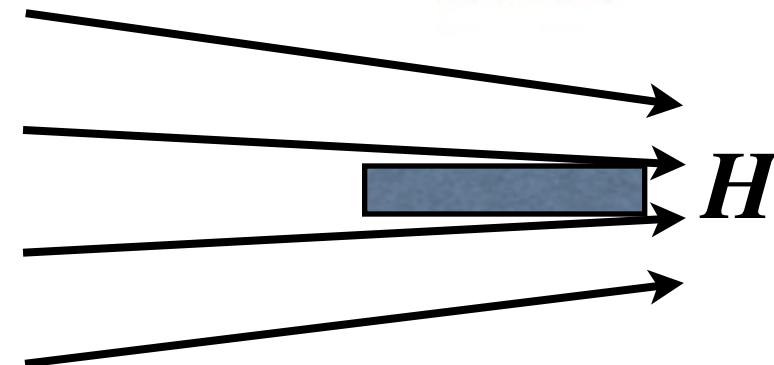
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a) Torque magnetometer

Most direct method to measure anisotropy

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Faraday Balance and Alternating Gradient Magnetometer

3.2 Mechanical Measurements

a) Torque magnetometer

- Torque measurements offer most direct methods for measuring anisotropies
- Requires uniform, single-crystalline samples, preferably of spherical or disk shape
- Example: crystal with cubic anisotropy:

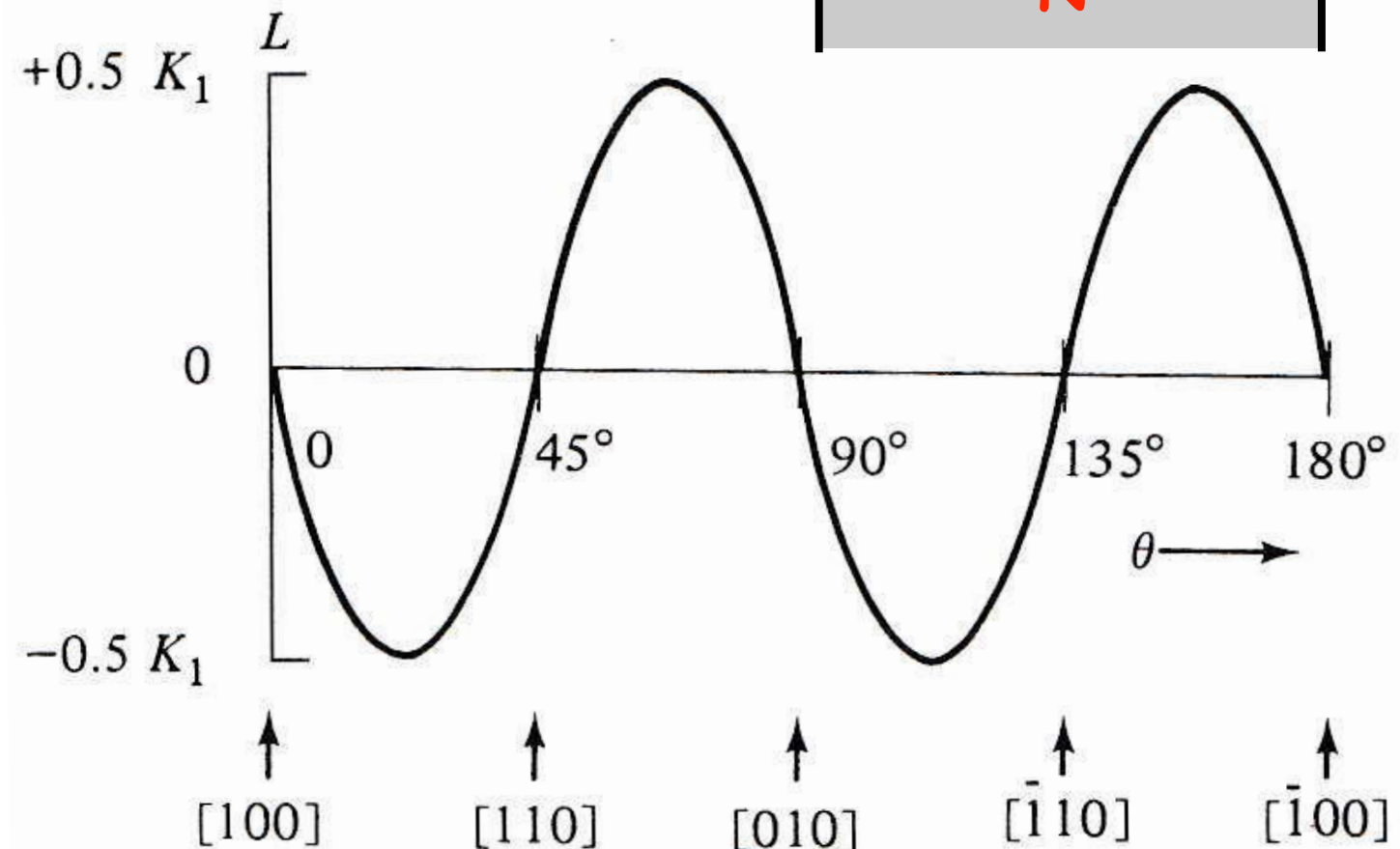
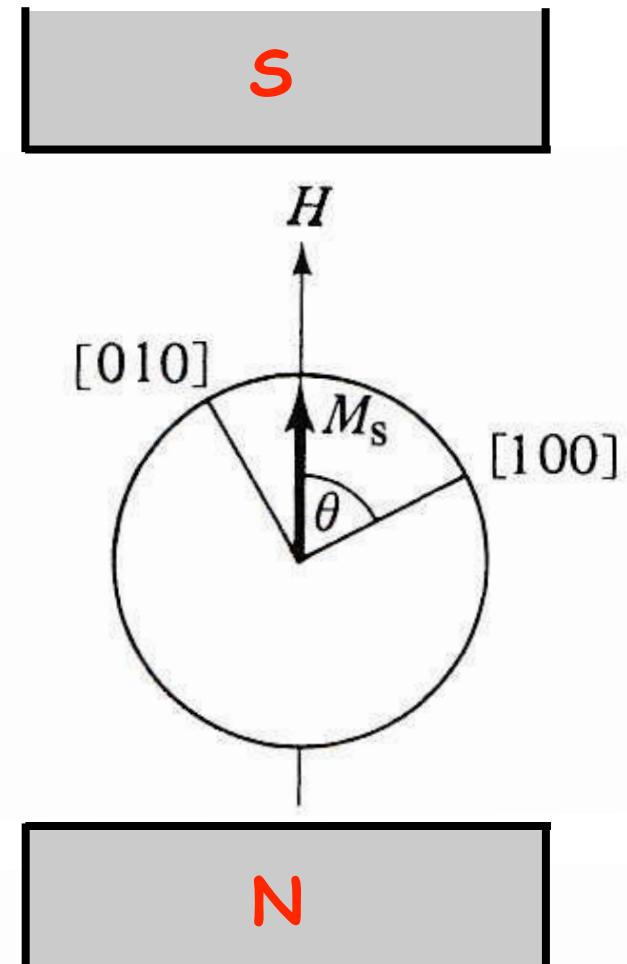
Anisotropy energy:

$$E = K_0 + K_1 \sin^2\theta \cos^2\theta,$$

Torque (assumption: $M_s \parallel H$):

$$\begin{aligned} L &= -\frac{dE}{d\theta} = -K_1 \sin 2\theta \cos 2\theta \\ &= -\frac{K_1}{2} \sin 4\theta. \end{aligned}$$

- Anisotropy constant can be measured by torque measurement

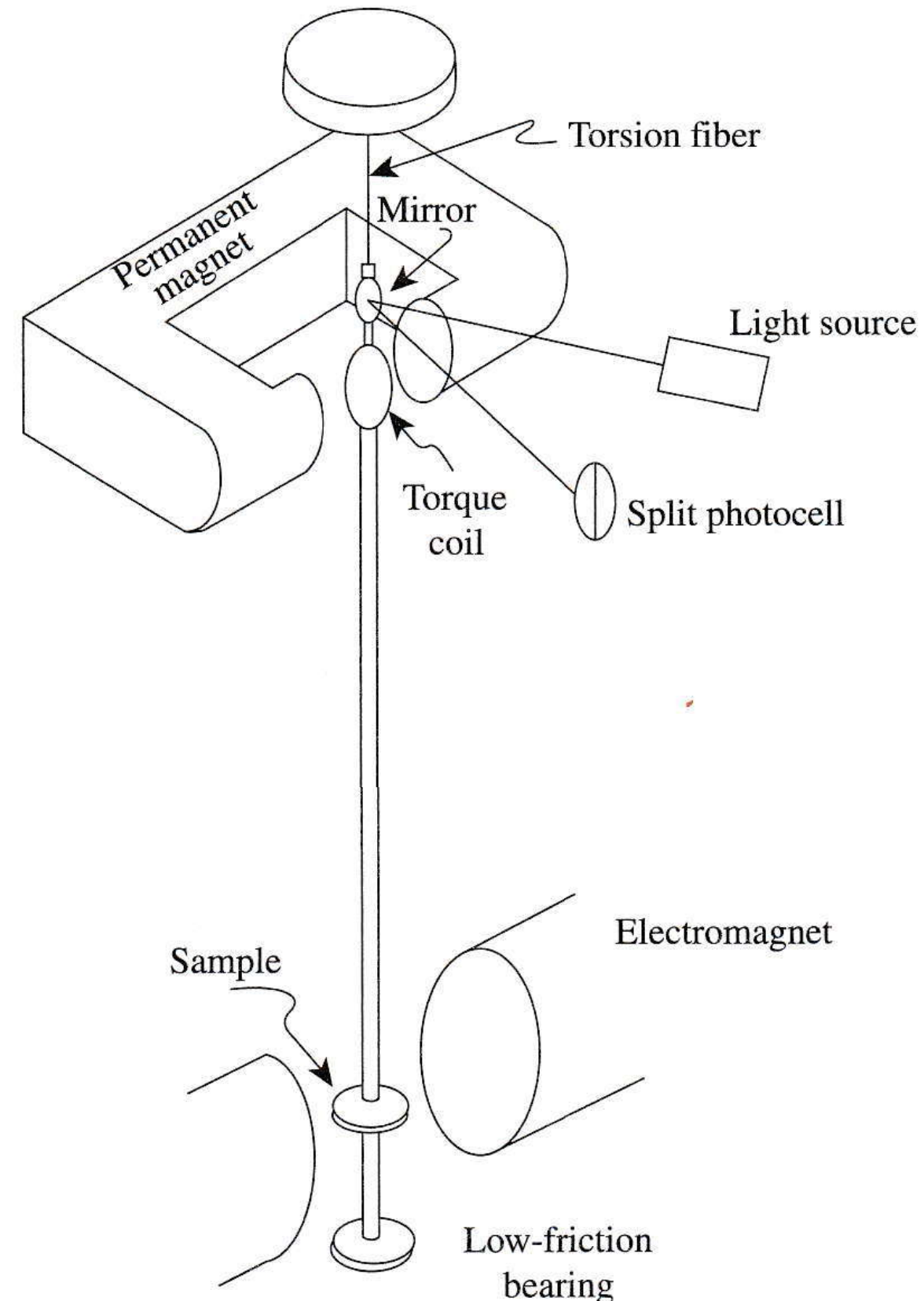


3.2 Mechanical Measurements

a) Torque magnetometer

Example: Torque meter with active sensing

- Sample hung from sensitive torsion fiber, placed in electromagnet that can be rotated
- Torque coil, placed in field of permanent magnet. Current through torque coil: coil experiences torque proportional to current
- Sensing circuit (light beam, mirror, photocell) provides feedback signal that drives a current through the torque coil to balance the anisotropy torque of sample
- Value of current through torque coil is proportional to torque on sample

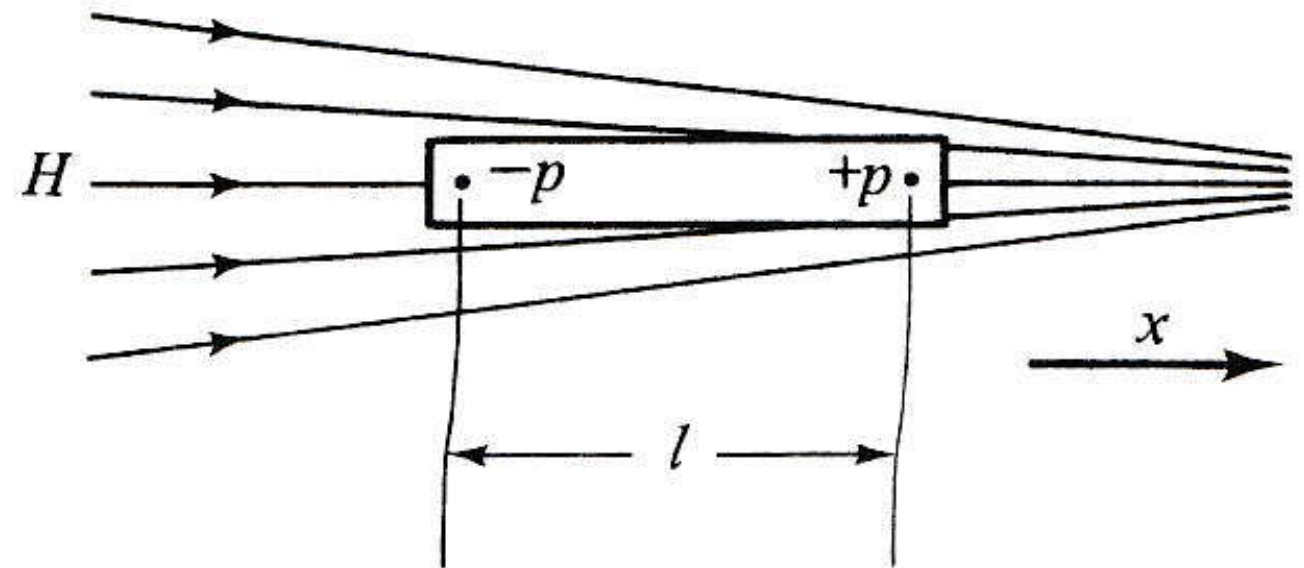


3.2 Mechanical Measurements

b) Field gradient methods

Faraday Balance

- Measures static force on sample in magnetic field gradient:
- A non-spherical body in homogeneous field will rotate till its long axis is parallel to field (compass needle)
- Field gradient: in body of positive susceptibility χ poles of strength p are produced
- Since field is stronger at north pole than at south pole: net force F_x to right, with m = magnetic moment and v = volume of body
- Body will move toward region of greater field strength (to right)



$$\begin{aligned} F_x &= -pH + p \left(H + l \frac{dH}{dx} \right) \\ &= pl \frac{dH}{dx} = m \frac{dH}{dx} = Mv \frac{dH}{dx} \\ &= \chi v H \frac{dH}{dx} = \frac{\chi v}{2} \frac{dH^2}{dx} \end{aligned}$$

3.2 Mechanical Measurements

b) Field gradient methods

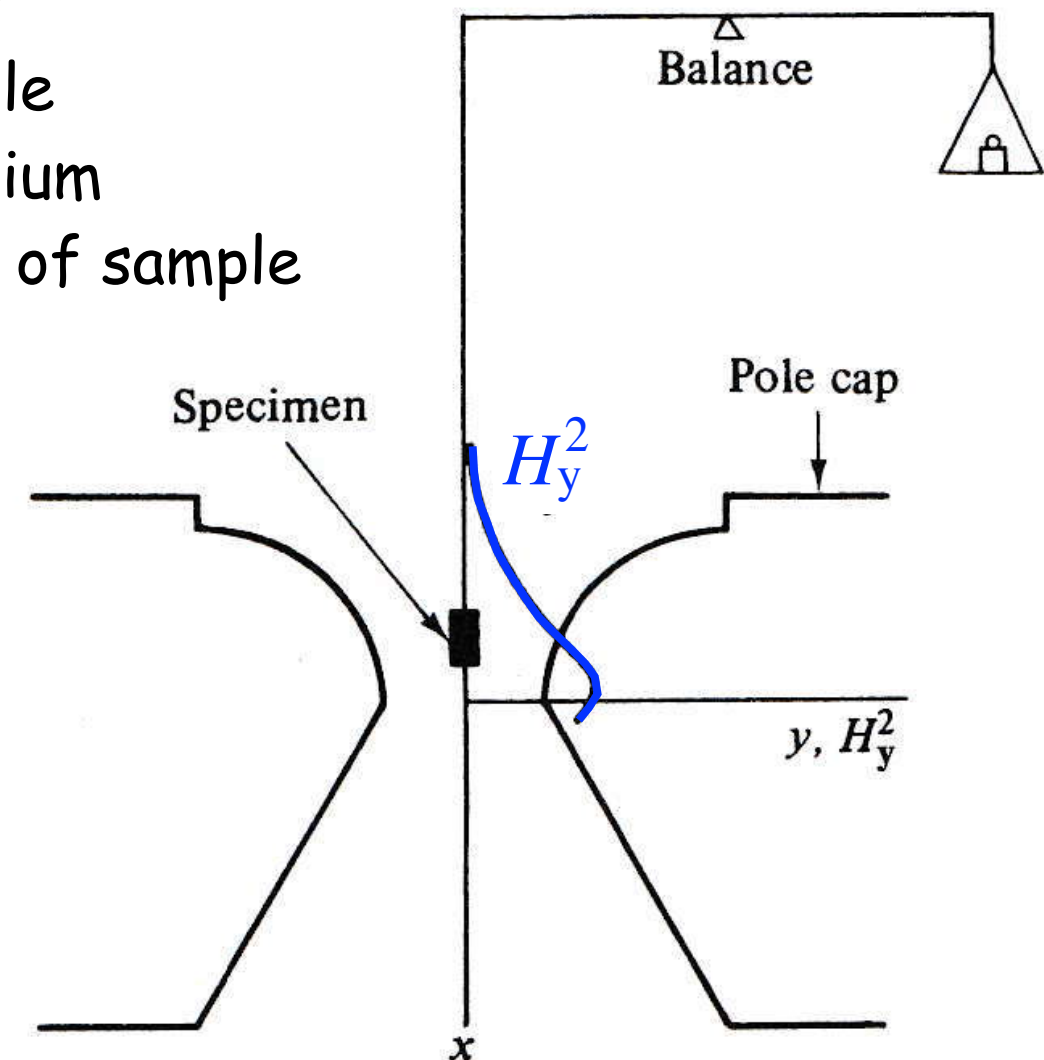
Faraday Balance

- Measures static force on sample in magnetic field gradient
- Uniform field is generated by electromagnet, and the gradient field is produced by an additional coil-set, optimized for a uniform magnetic gradient along y -axis
- If sample is mounted elastically, it is displaced by force :

$$F_x = (\chi - \chi_0) v H_y \frac{dH_y}{dx}$$

χ : susceptibility sample
 χ_0 : susceptibility medium
 vH_y : magn. moment m of sample

- Force can be detected and compensated by a calibrated electromagnetic counter-force. Compensation current is measure of force. Only quantity needed: sample volume
- Highly sensitive, can be applied to all kinds of magnetic substances
- In ferromagnetism it is best suited to measure the saturation magnetization with high precision

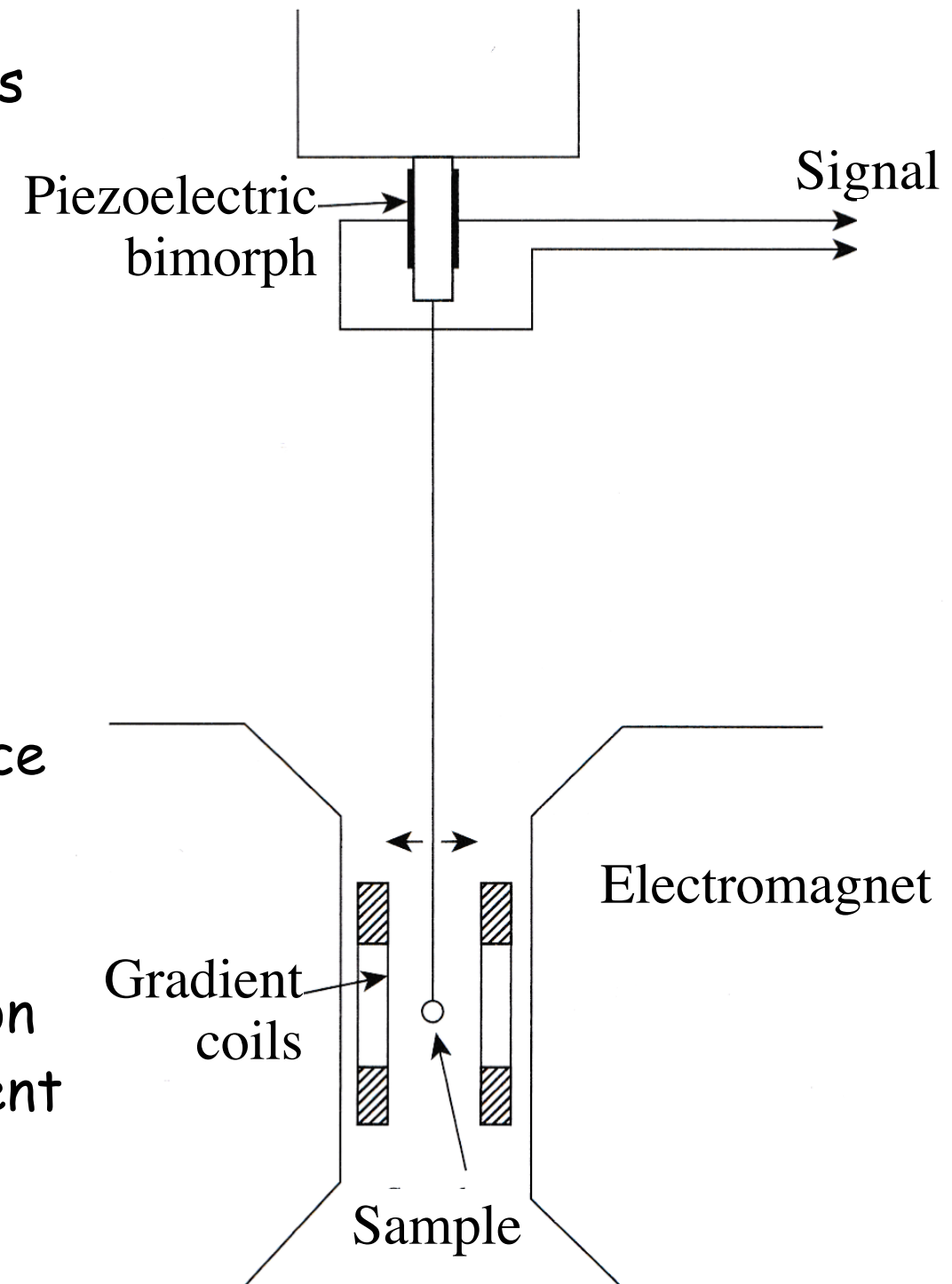


3.2 Mechanical Measurements

b) Field gradient methods

Alternating Gradient Magnetometer (AGM)

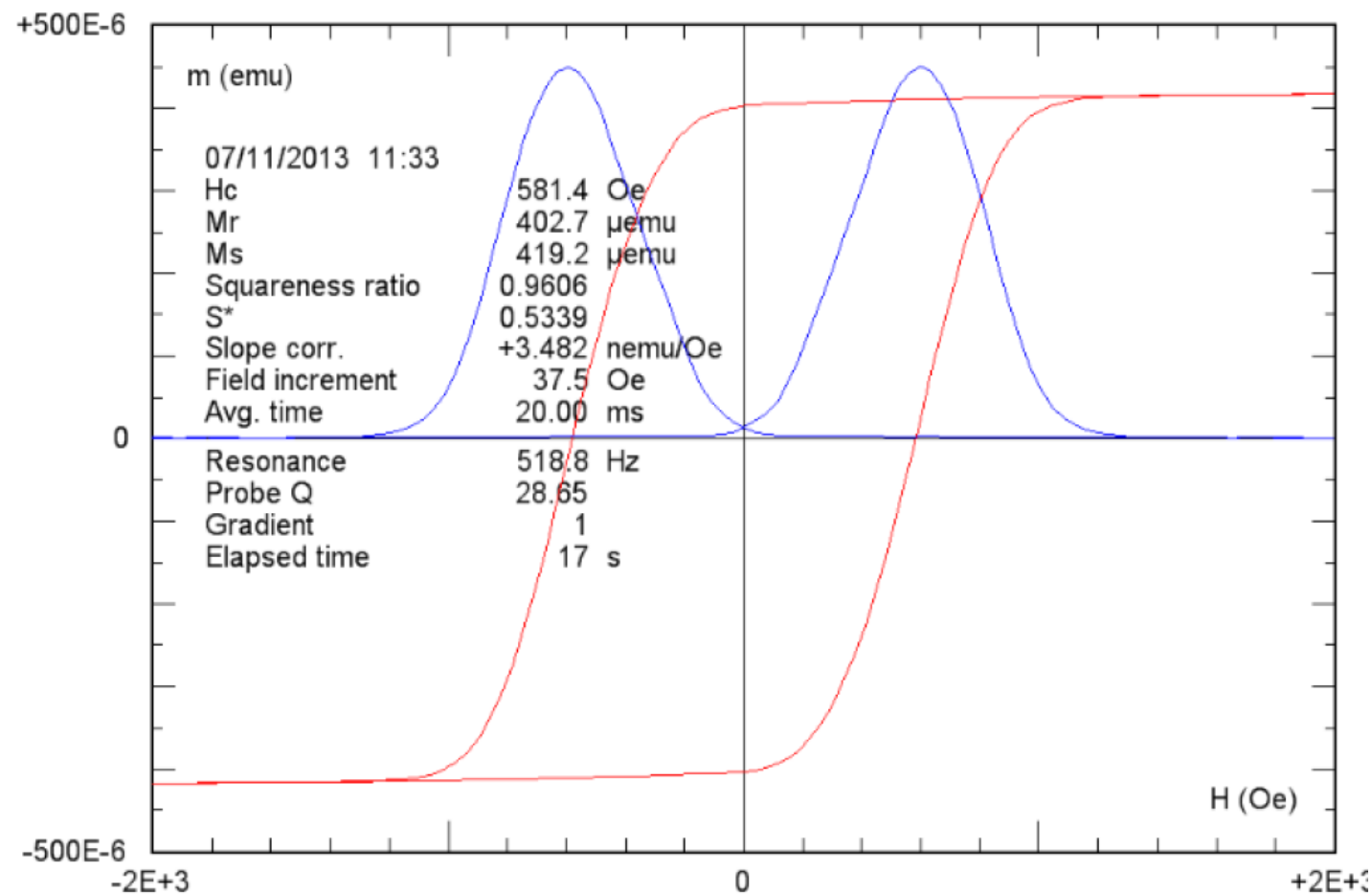
- AGM (or Vibrating Reed Magnetometer) resembles superficially the VSM
- VSM: sample agitated mechanically and electric signal is derived from motion. AGM: **magnetically** excited signal is recorded
- Sample mounted on elastic cantilever or "reed", which is excited into resonant vibration by alternating gradient field (produced by gradient coils in addition to dc magnet) by choosing resonance frequency
- Vibration is recorded by piezoelectric pick-up system: generates voltage proportional to vibration amplitude = proportional to sample magnetic moment
- Sensitivity: 10^{-6} emu = 10^{-9} Am² → higher than VSM



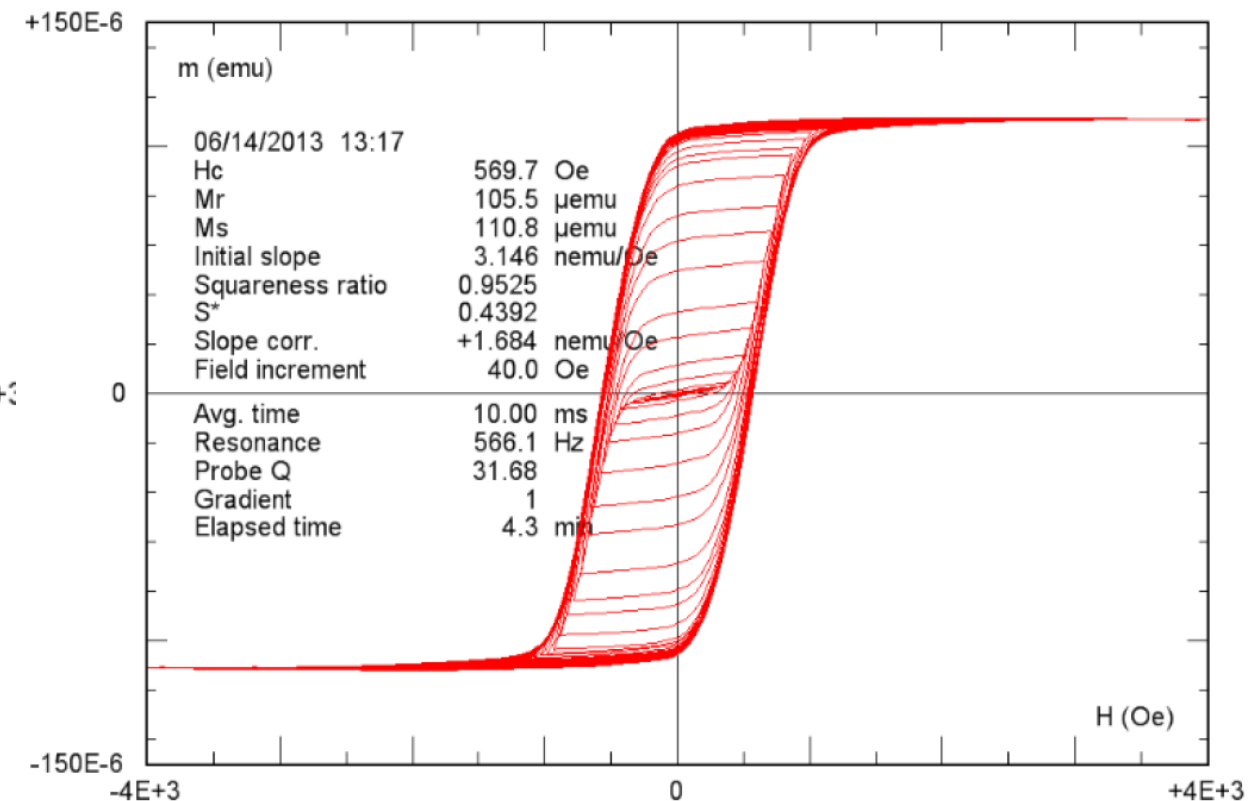
3.2 Mechanical Measurements

b) Field gradient methods

Alternating Gradient Magnetometer (AGM)



Examples



Description: [Not assigned]
File: Ni nanowire array hysteresis

Description: [Not assigned]
File: Ni nanowire array.1 mm.100 uemu.80 minor loops.10 ms ms

Courtesy Lakeshore

3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

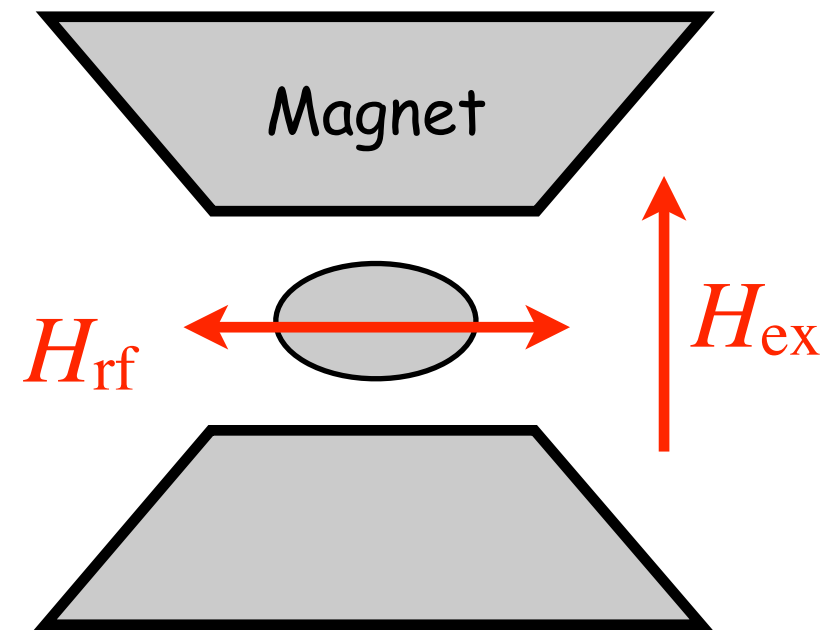
3.4 Dilatometric measurements

3.5 Domain methods

3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

- FMR is dynamic measurement method at microwave frequency (GHz-regime, order of Larmor frequency)
- Because of high-frequency alternating magnetic field: eddy current shielding in metals is nearly complete (penetration depth of field ~ 200 nm)
 - resonance methods are not applicable to bulk metallic samples
 - can only be applied to non-conducting oxidic materials, thin films, and powdered materials
- Typical resonance experiment:
 - Sample magnetized in strong **static** field H_{ex} to enforce uniformly magnetized state
 - To induce resonance phenomenon: alternating field with fixed GHz-frequency is superimposed at right angle to magnetization direction → stimulates precession of magnetization vector
 - The static field amplitude H_{ex} is swept till resonance is achieved at $H_{\text{ex}} = H_{\text{res}}$



3.3 Resonance Techniques

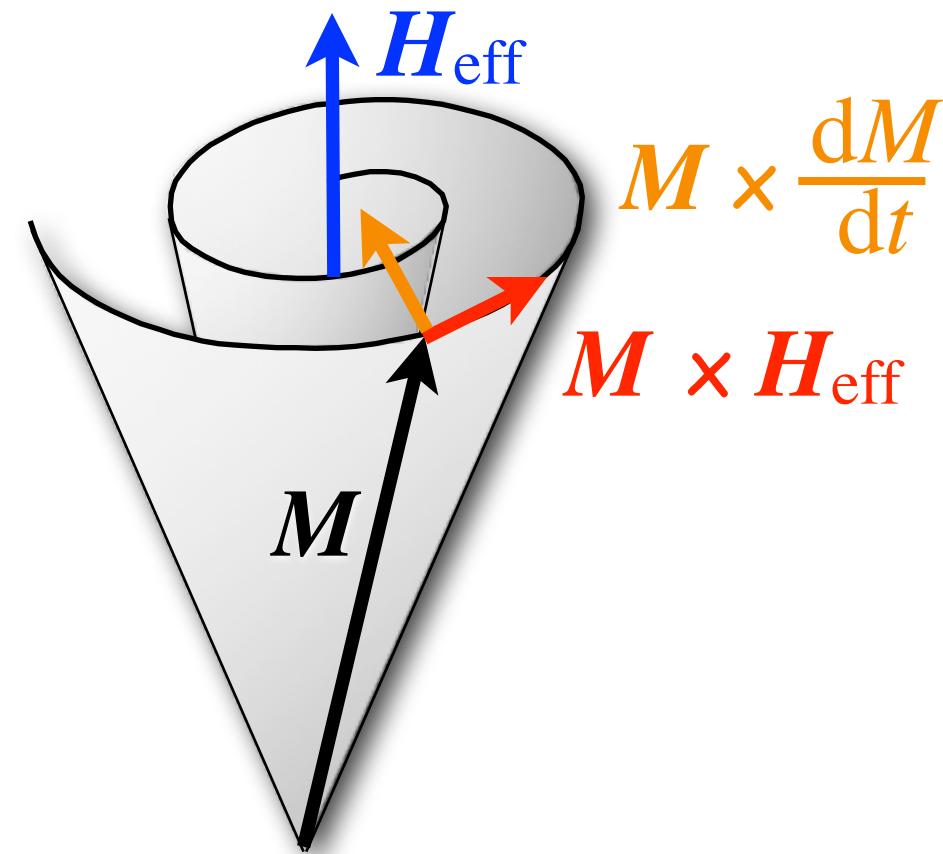
Ferromagnetic Resonance (FMR)

- Based on LLG equation:

$$\frac{d\mathbf{M}}{dt} = -\gamma_0 [\mathbf{M} \times \mathbf{H}_{\text{eff}}] + \frac{\alpha}{M_s} [\mathbf{M} \times \frac{d\mathbf{M}}{dt}]$$

Describes (damped) precession of \mathbf{M} around effective field \mathbf{H}_{eff} , caused by gyrotropic reaction of magnetic moment due to its angular momentum

- If the precession is uniform (does not depend on the position in sample): **ferromagnetic resonance**
- At higher frequencies, non-uniform precession modes may be excited:
 - If their wavelength is comparable with sample size: **magnetostatic modes**
 - Resonance phenomena at shorter wavelengths depend on the exchange stiffness constant and are called **spin-wave modes**
 - Finally, there are **surface modes** of magnetic resonance which can be excited for example by light instead of an alternating field. Then spectroscopy replaces the standard inductive detection methods



→ **Lecture of B. Hillebrands**

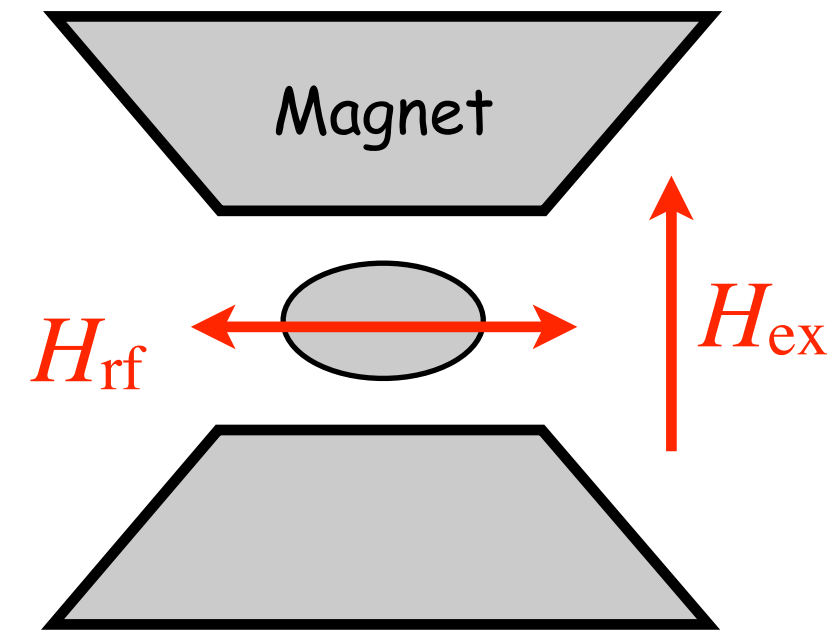
3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

- FMR experiment:
 - Static field H_{ex} with superimposed microwave field H_{rf} of fixed frequency
 - Static field amplitude H_{ex} is swept till resonance is achieved at $H_{\text{ex}} = H_{\text{res}}$ (sweeping of field is easier than sweeping microwave frequency)
- Resonance frequency ω_{res} and effective resonance field H_{res} are given by:

$$\omega_{\text{res}} = \gamma H_{\text{res}} \quad \text{with} \quad \gamma = \mu_0 g e / 2m_e$$

$$H_{\text{res}} = \sqrt{\left[\underbrace{\frac{2K_u}{\mu_0 M_s}}_{\text{Anisotropy field}} + \underbrace{H_{\text{ex}}}_{\text{External field}} + \underbrace{M_s (N_b - N_a)}_{\text{Demagnetizing field}} \right] \left[\frac{2K_u}{\mu_0 M_s} + H_{\text{ex}} + M_s (N_c - N_a) \right]}$$

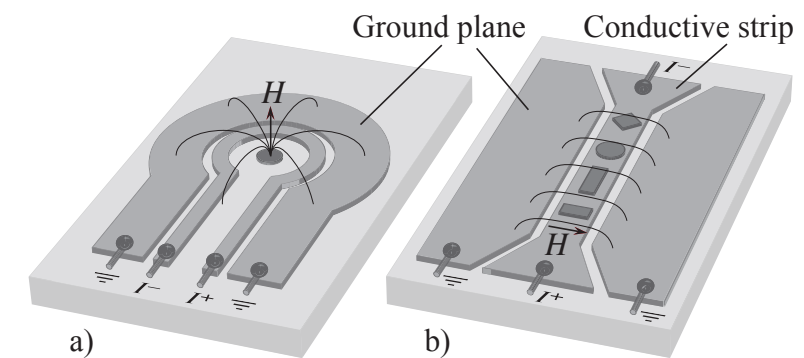
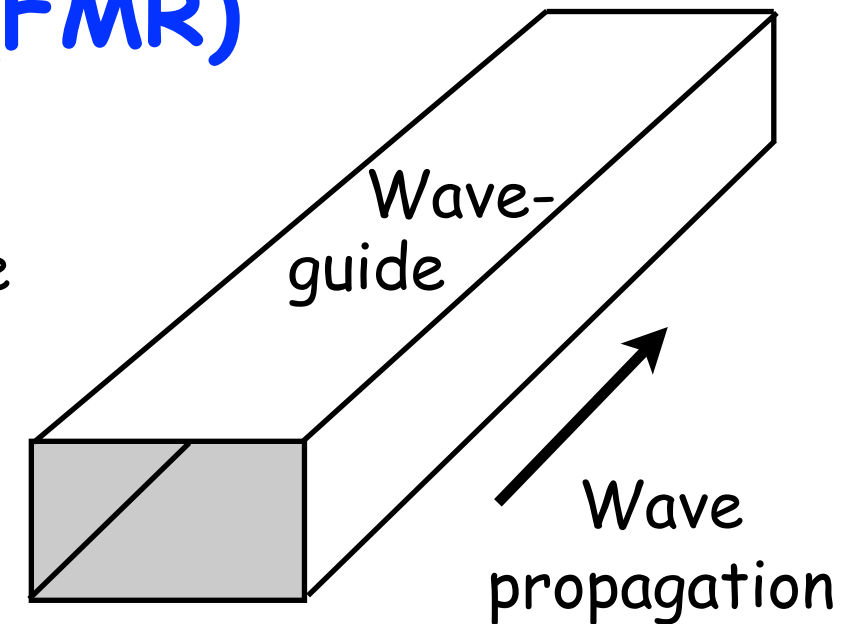


- If M_s and N_i are known \rightarrow anisotropy K_u can be derived from measuring H_{res}

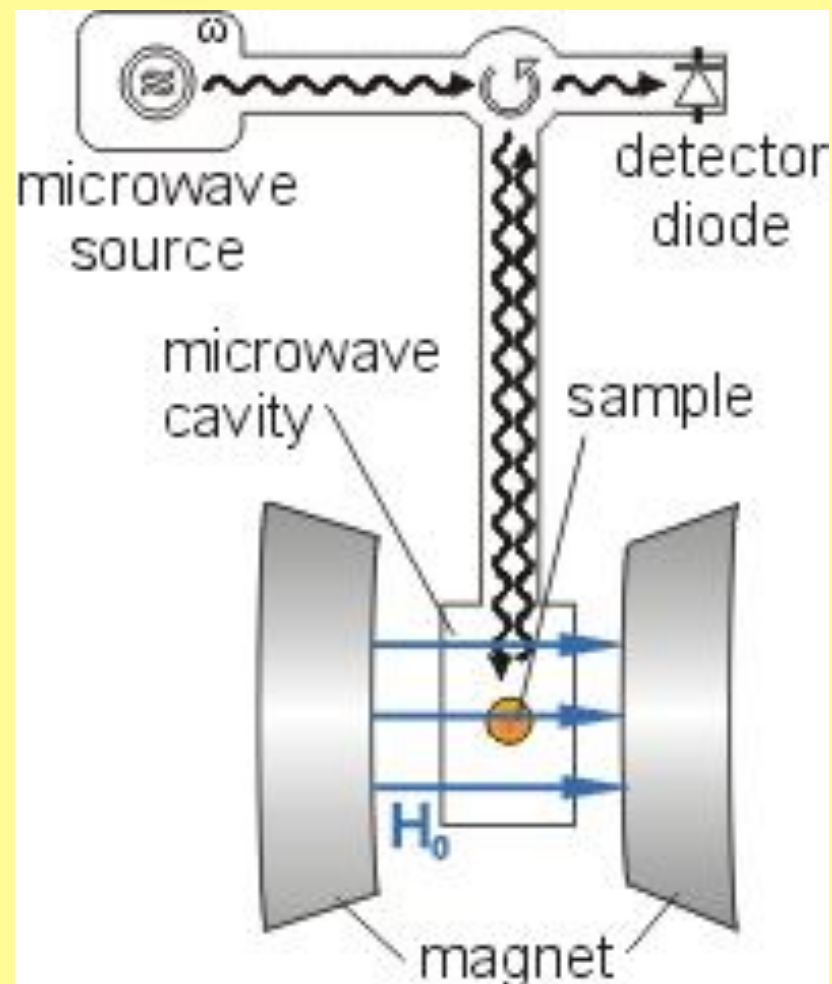
3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

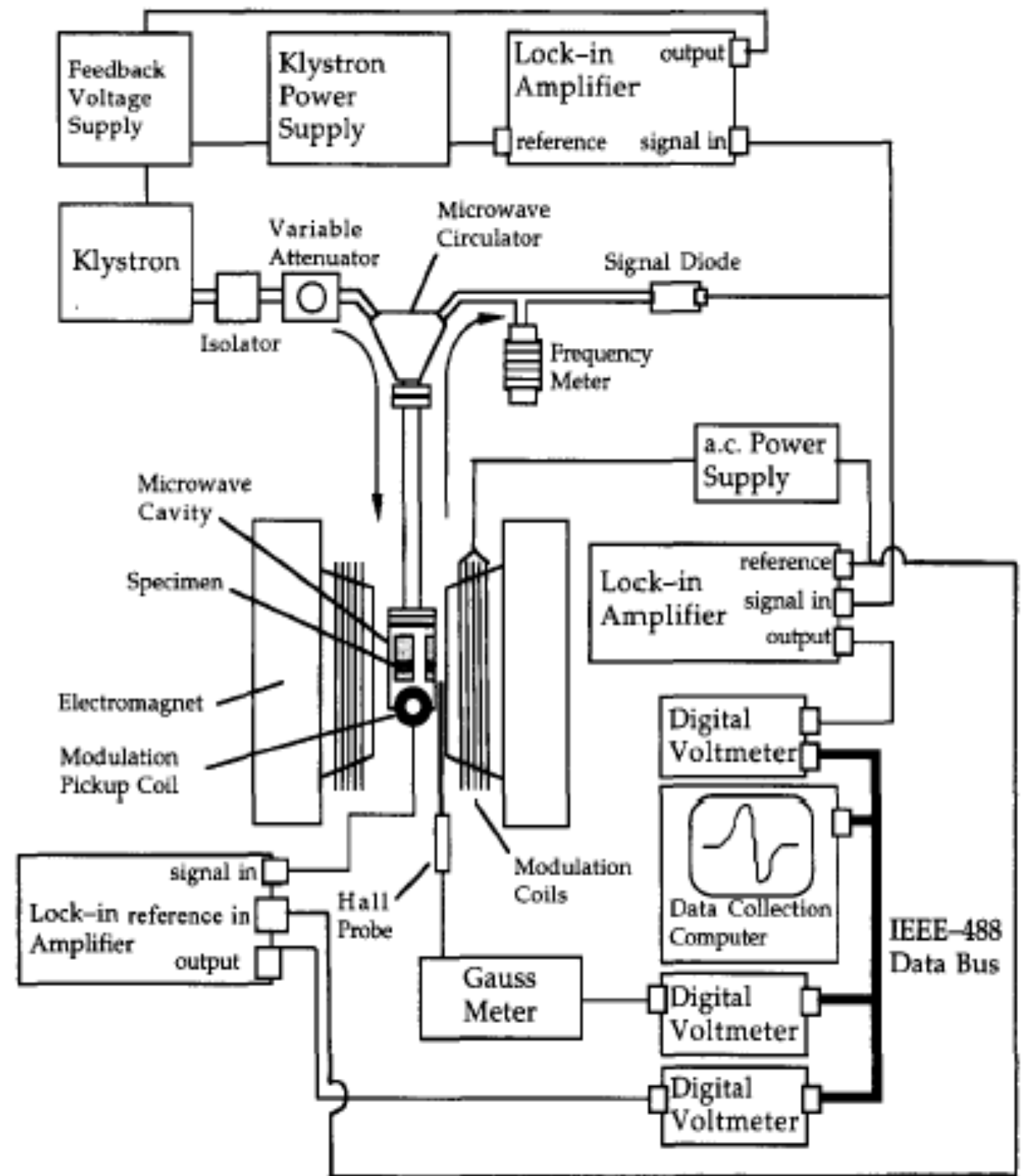
- FMR experiment:
 - Microwave field is generated by Klystron or Gunn diode and conducted to sample by wave guide (hollow metal tube)
 - Sample centered in microwave cavity (closed hollow metal structure), located in electromagnet
- Microwave frequency fixed and determined by klystron and cavity. Alternatively sample can be placed on micro-stripline



3.3 Resonance Techniques



Courtesy Wolfgang Kuch

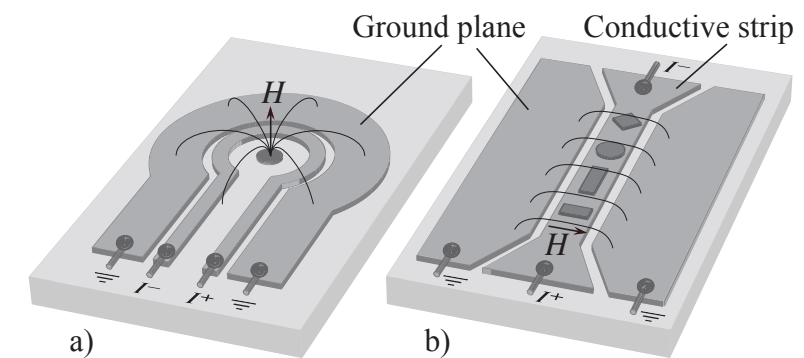
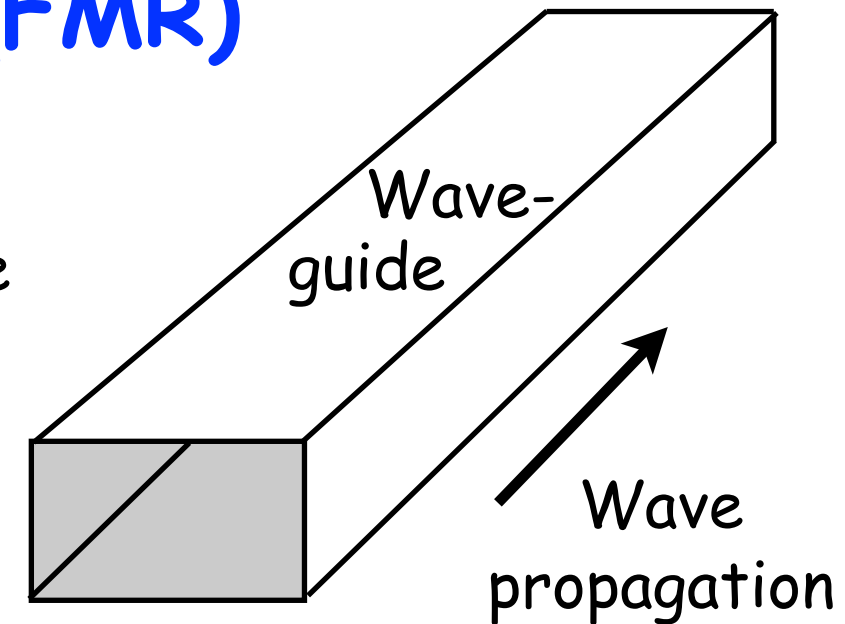


Courtesy Zbigniew Celinski

3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

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3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

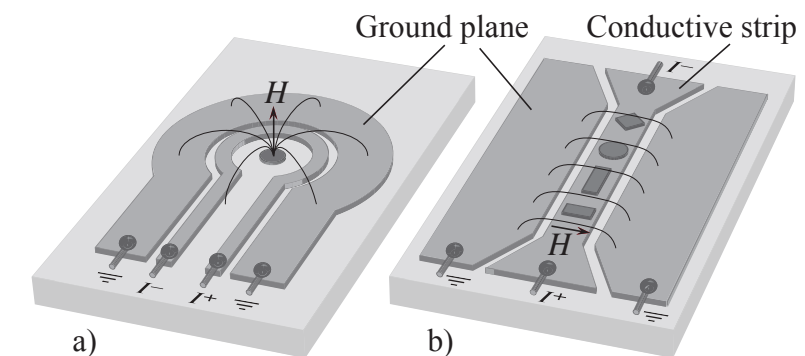
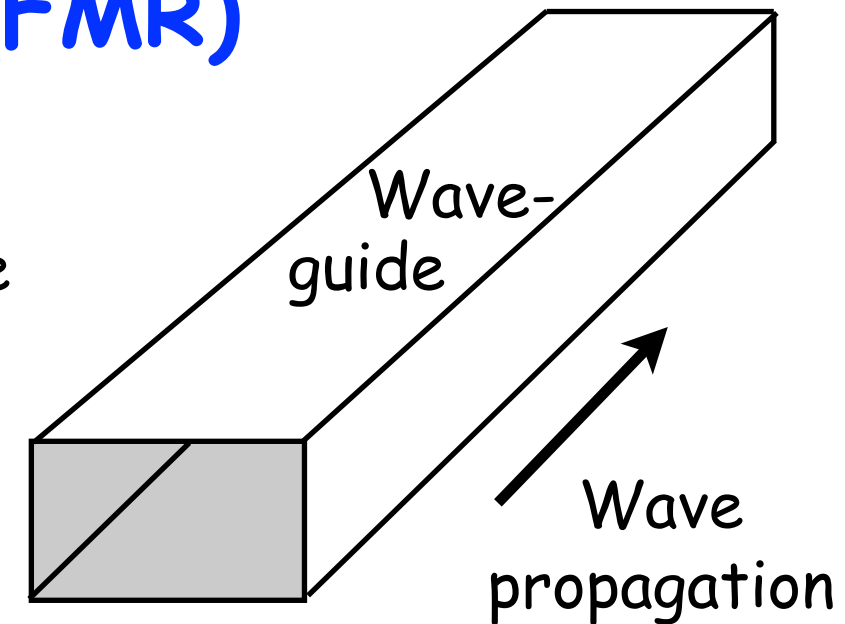
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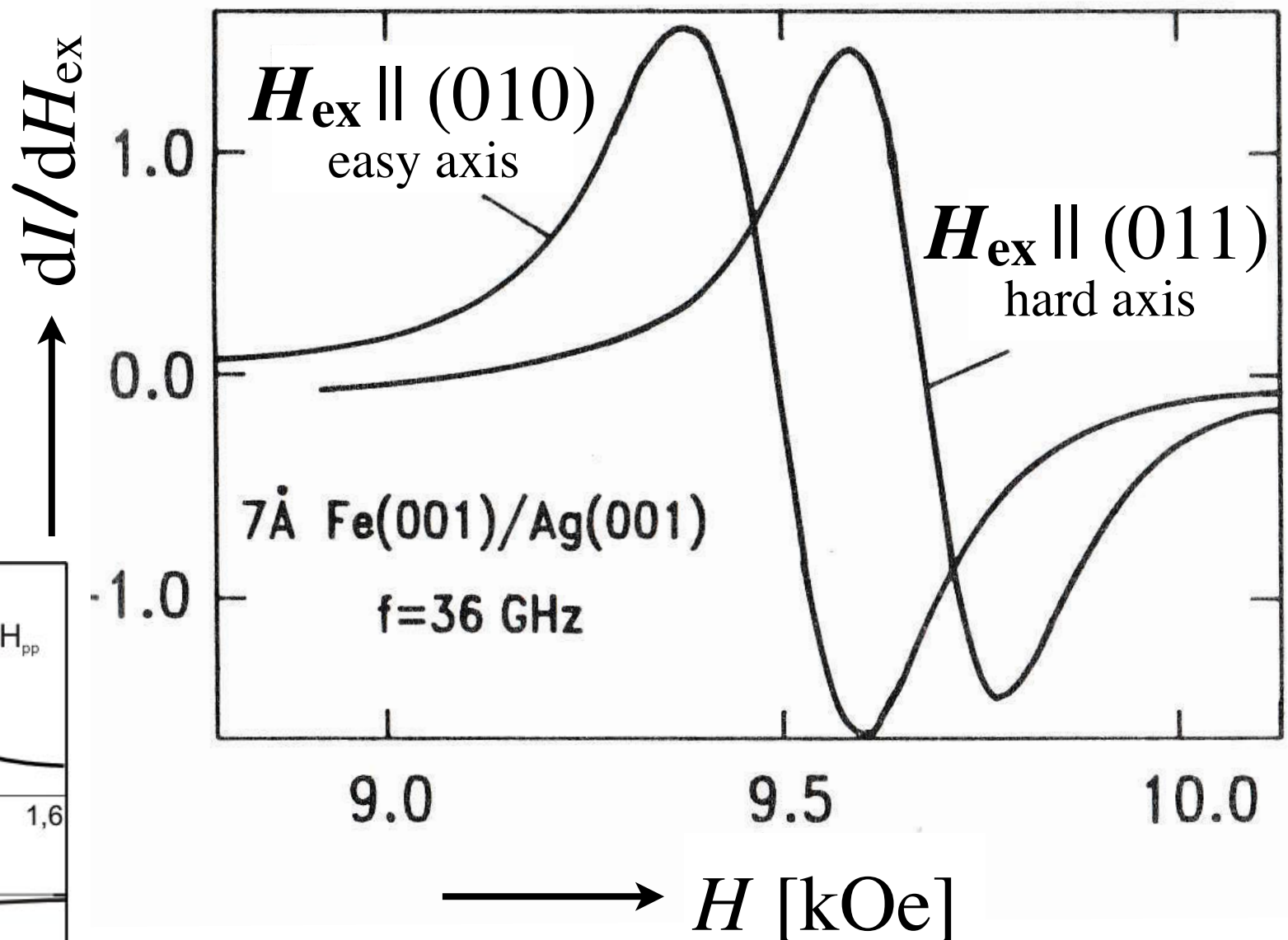
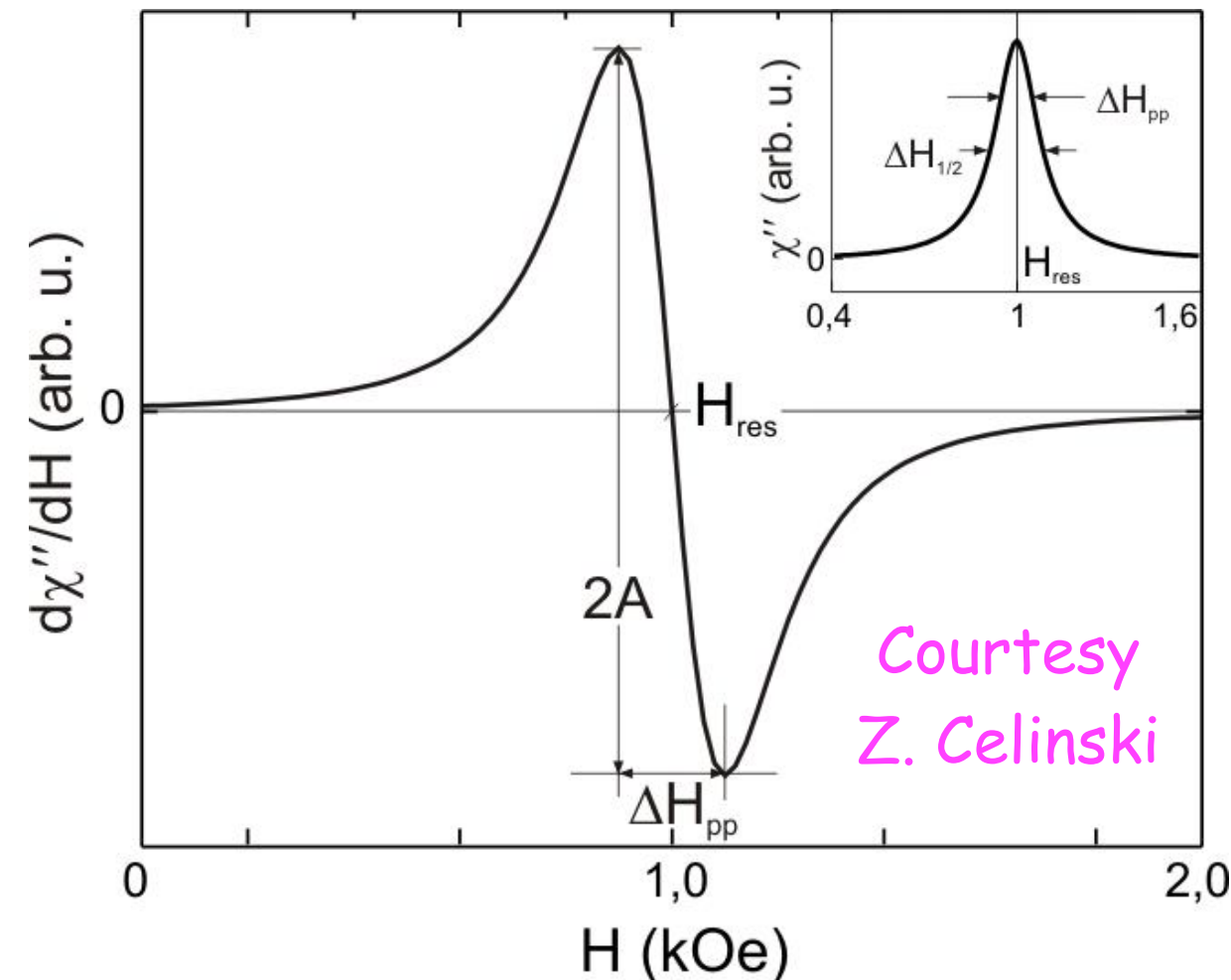
- Incident microwave signal couples to sample and is partially absorbed in dependence of field strength H_{ex}
- If resonance condition is fulfilled → max. absorption of microwave power by sample
- Crystal detector measures reflected microwave signal. This signal is input into lock-in amplifier where it is compared with reference signal
- Output of lockin vs. external field: corresponds to derivative of absorption curve, i.e the change of absorbed intensity I as function of H_{ex} : dI/dH_{ex}



3.3 Resonance Techniques

Ferromagnetic Resonance (FMR)

- Resonance field H_{res} and half power line width ΔH can be determined
- $H_{\text{res}} \rightarrow$ magnetic anisotropy
- $\Delta H \rightarrow$ relaxation
- Intensity $\rightarrow M_s$



B. Heinrich et al., PRL 59, 1756 (1987)

3.3 Resonance Techniques

Spinwave Resonance

Light Scattering experiments

3.3 Resonance Techniques

Spinwave Resonance

Light Scattering experiments

→ Lecture of B. Hillebrands

3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

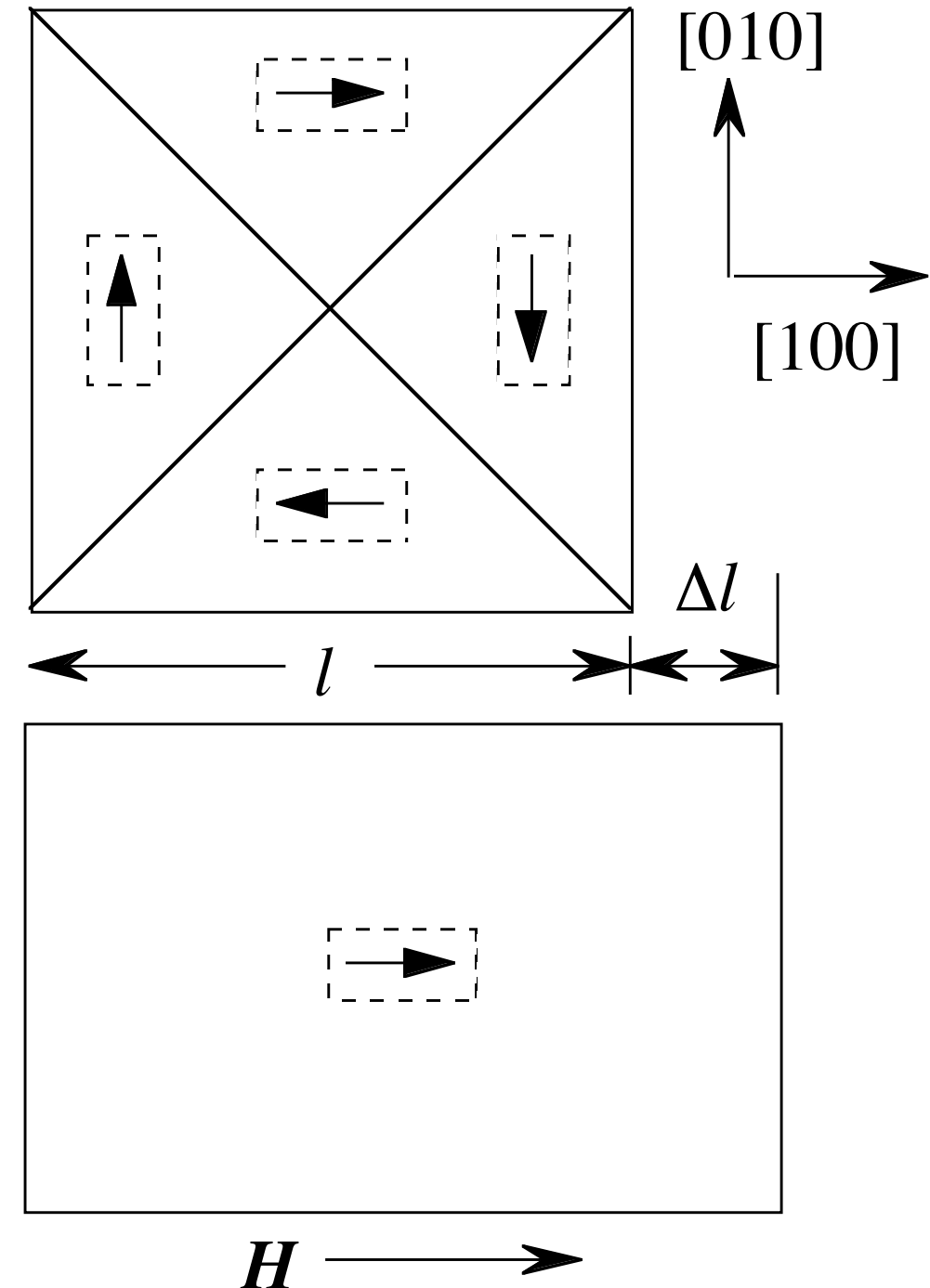
3.4 Dilatometric measurements

3.5 Domain methods

3.4 Dilatometric Measurements

Magnetostriction

- Example: cubic crystal (iron) with 4 domain phases in ground state → no net-magnetization
- Each domain elongates crystal along M -direction → cubic lattice gets tetragonally distorted
- Application of magnetic field, saturation → elongation of total crystal
- Relative length change $\Delta l / l = \lambda_s$
 λ_s = magnetostriction coefficient
- $\lambda_s > 0$: elongation in magnetization direction,
 $\lambda_s < 0$: compression
- Note: 180° -domains are elongated along same axis
- λ for iron: $20 \cdot 10^{-6}$ → very small effect

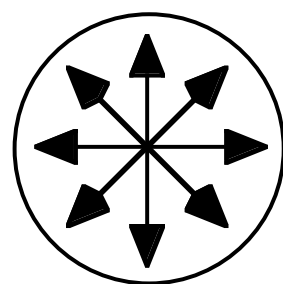


3.4 Dilatometric Measurements

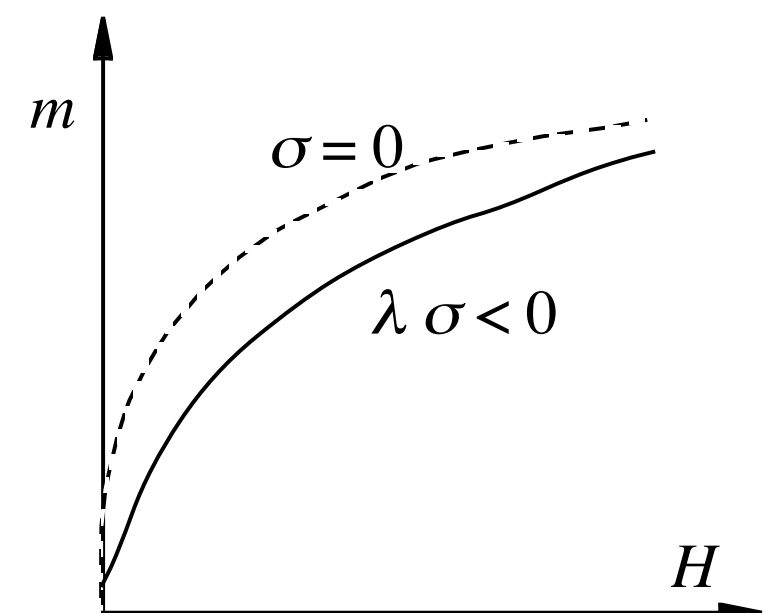
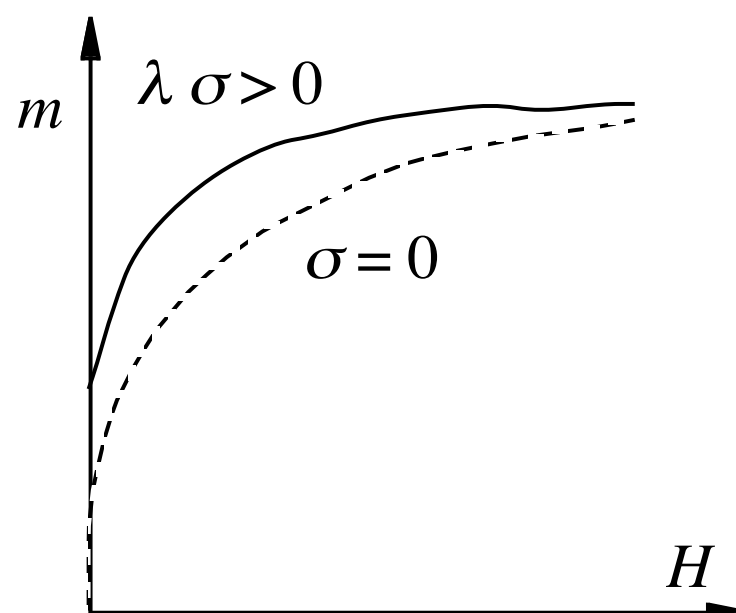
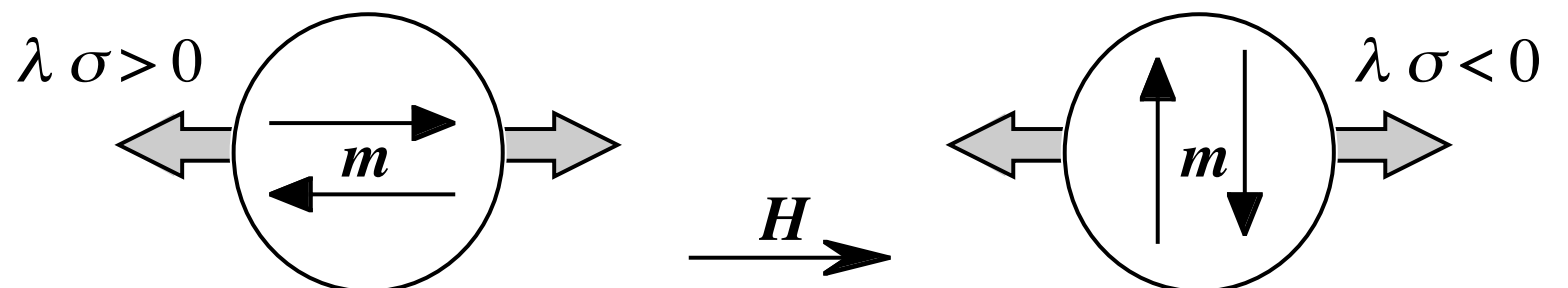
Magnetostriction measurement

- Saturation magnetostriction constants can be determined both directly or indirectly:
- **Indirect methods:** stress sensitivity of a suitable magnetic property is analysed, more suitable for thin films and wires

Example: magnetization curve and resonance measurements, if performed as function of external stress, can be evaluated in terms of magneto-elastic coefficients



$$\langle \mathbf{m} \rangle = 0$$
$$\sigma = 0$$



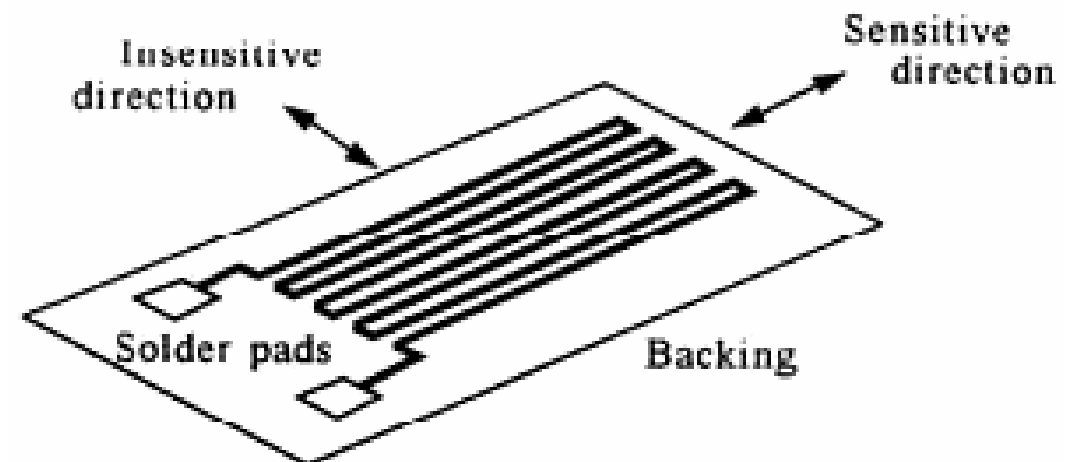
3.4 Dilatometric Measurements

Magnetostriction measurement

- Saturation magnetostriction constants can be determined both directly or indirectly:
- **Direct measurements:** evaluate elongation of a magnet depending on the magnetization direction, preferred for bulk samples of sufficient size

Strain Gauges

- Plastic foil on which structured metal films act as sensors, cemented on sample
- Based on (small) change of el. resistance by elongation, measured with bridge circuit
- Strain gauge can be applied locally on favourable small region of single crystal or even on grain in a coarse-grained sample. Active area down to 1 mm^2
- It is always advisable to apply strain gauges on both sides of sample, and to connect them in series to avoid influence from sample bending induced by one-sided heating by the measuring current



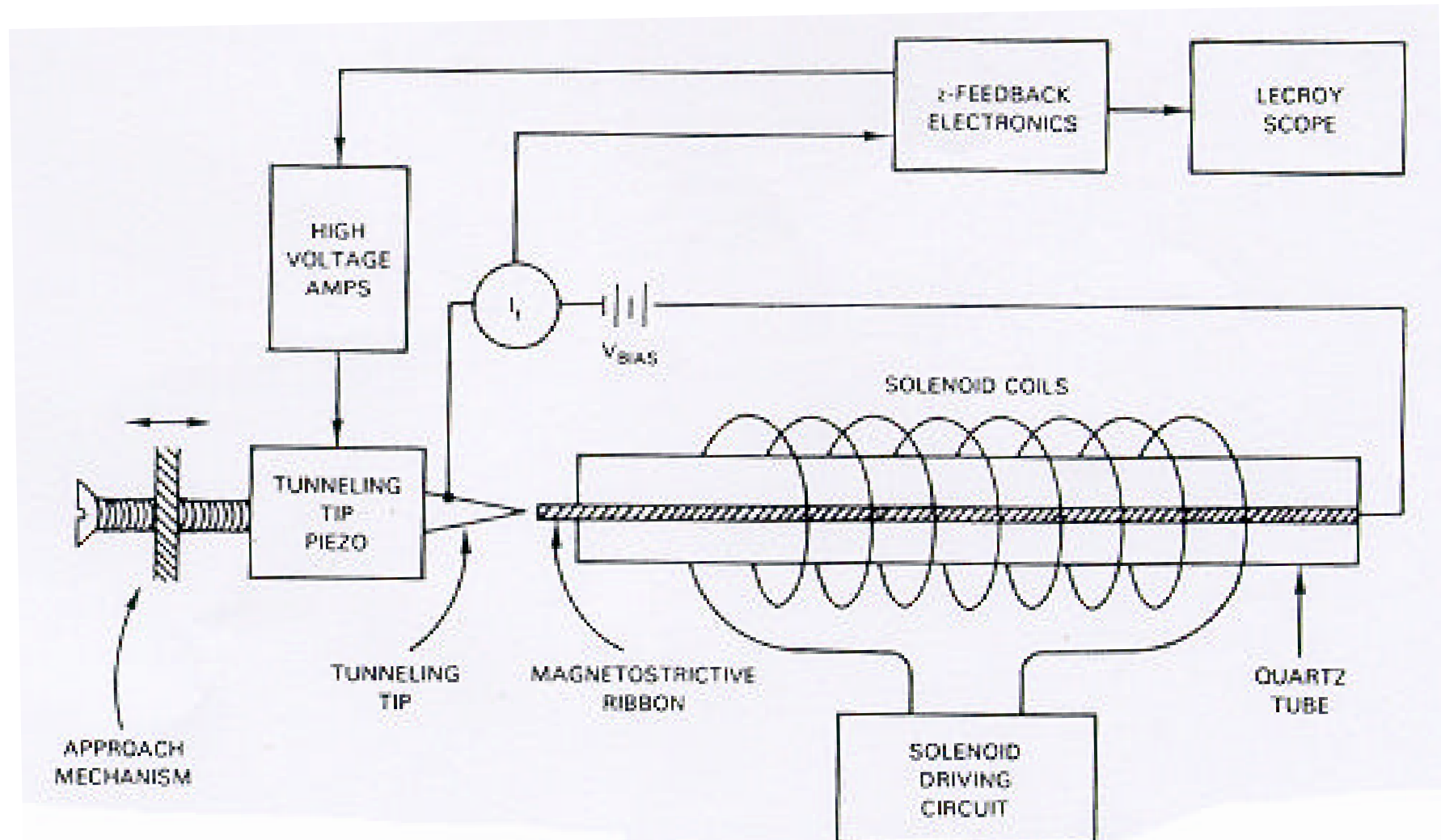
3.4 Dilatometric Measurements

Magnetostriction measurement

- Saturation magnetostriction constants can be determined both directly or indirectly:
- **Direct measurements:** evaluate elongation of a magnet depending on the magnetization direction, preferred for bulk samples of sufficient size

Dilatometers

- Capacitive sensors, optical interferometers, tunnelling sensor, piezo sensor...
- With normal samples of centimetre dimension a resolution in the nanometre range is required



3.4 Dilatometric Measurements

Magnetostriction measurement

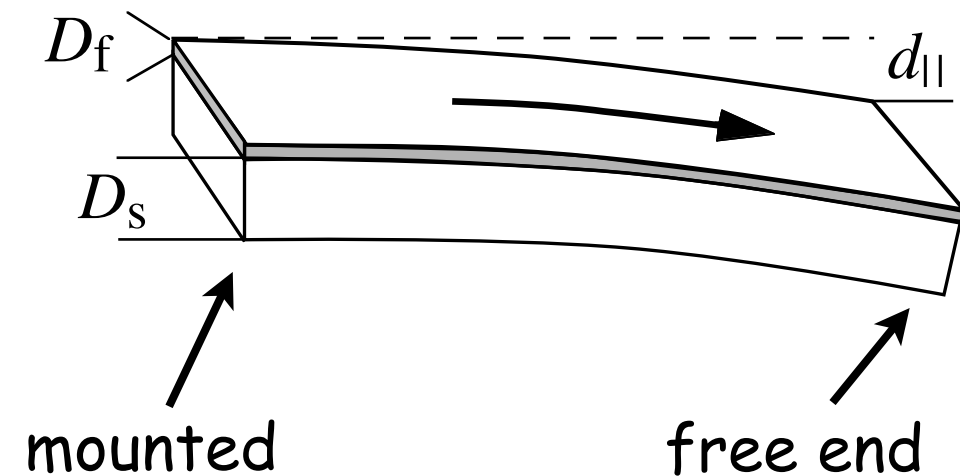
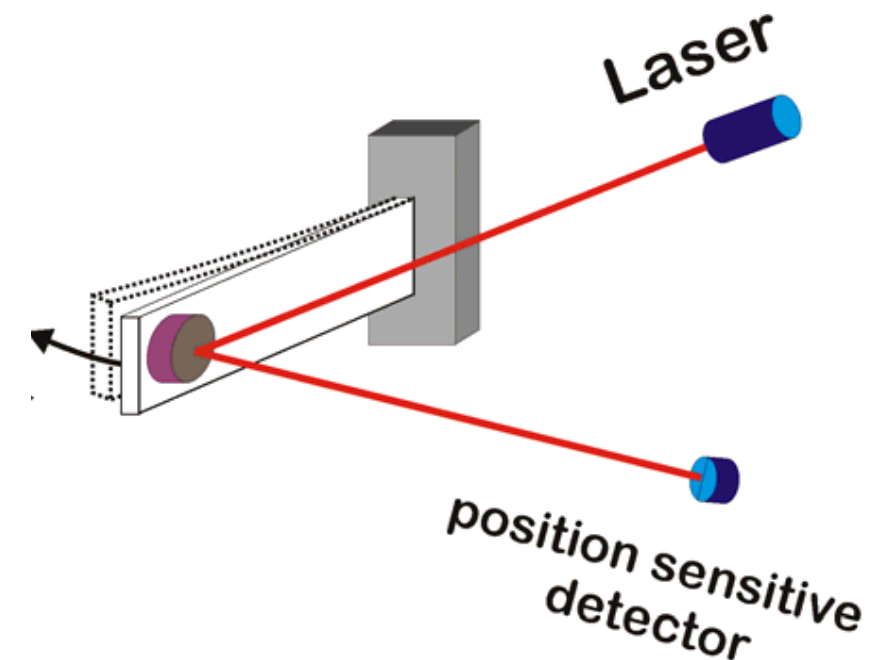
- Saturation magnetostriction constants can be determined both directly or indirectly:
- **Direct measurements:** evaluate elongation of a magnet depending on the magnetization direction, preferred for bulk samples of sufficient size

Cantilever for films

- Magnetostrictive bending of a sample-substrate-composite in magnetic field is optically detected as measuring signal by reflected laser beam, a quadrant detector and lock-in technique
- Sample exposed to rotating saturation field
- Deflection difference d_f of free end of cantilever for longitudinal and transverse magnetization:

$$d_f = d_{||} - d_{\perp} = \frac{3D_f L^2}{D_s^2} \frac{E_f(1 + \nu_s)}{E_s(1 + \nu_f)} \cdot \frac{3}{2} \lambda_s \sin^2 \vartheta$$

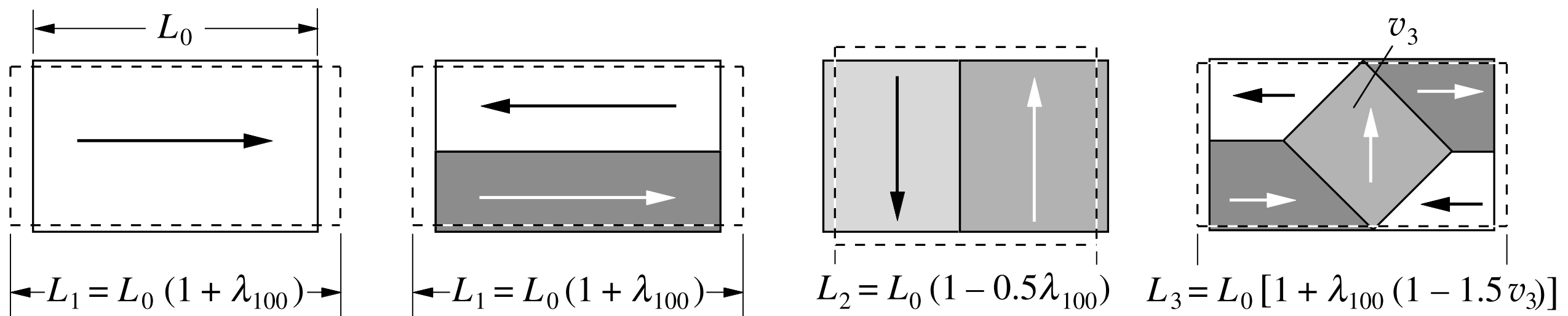
E_x and ν_x : Young's moduli and Poisson's ratios of film and substrate, θ : magnetization angle ($\theta = 0$ for magnetization along cantilever)



3.4 Dilatometric Measurements

Magnetostriction measurement, remark

- Every direct determination of magnetostriction constant requires measurement of length change between two different **saturated** states — usually parallel and perpendicular to measuring direction. Experiment with field applied along only one axis of crystal yields no useful information on magnetostriction constants, because then only some arbitrary demagnetized state and the saturated states are compared



3.

Magnetic Measurements to determine material parameters & properties

3.1 Magnetic measurements

3.2 Mechanical measurements

3.3 Resonance techniques

3.4 Dilatometric measurements

3.5 Domain methods

3.5 Domain Methods

Principle:

Under favourable circumstances material constants may be derived directly from observed domains. Such approach requires an equilibrium situation for which a reliable theoretical treatment is possible.

To qualify as suitable domain structure for quantitative evaluation, a pattern must fulfil a number of requirements:

- It must be sufficiently simple to permit a complete analysis.
- It must contain at least one feature (such as an angle or a characteristic length) which can reach its optimum value independent of its environment.
- This feature must be sensitive to an interesting material parameter.

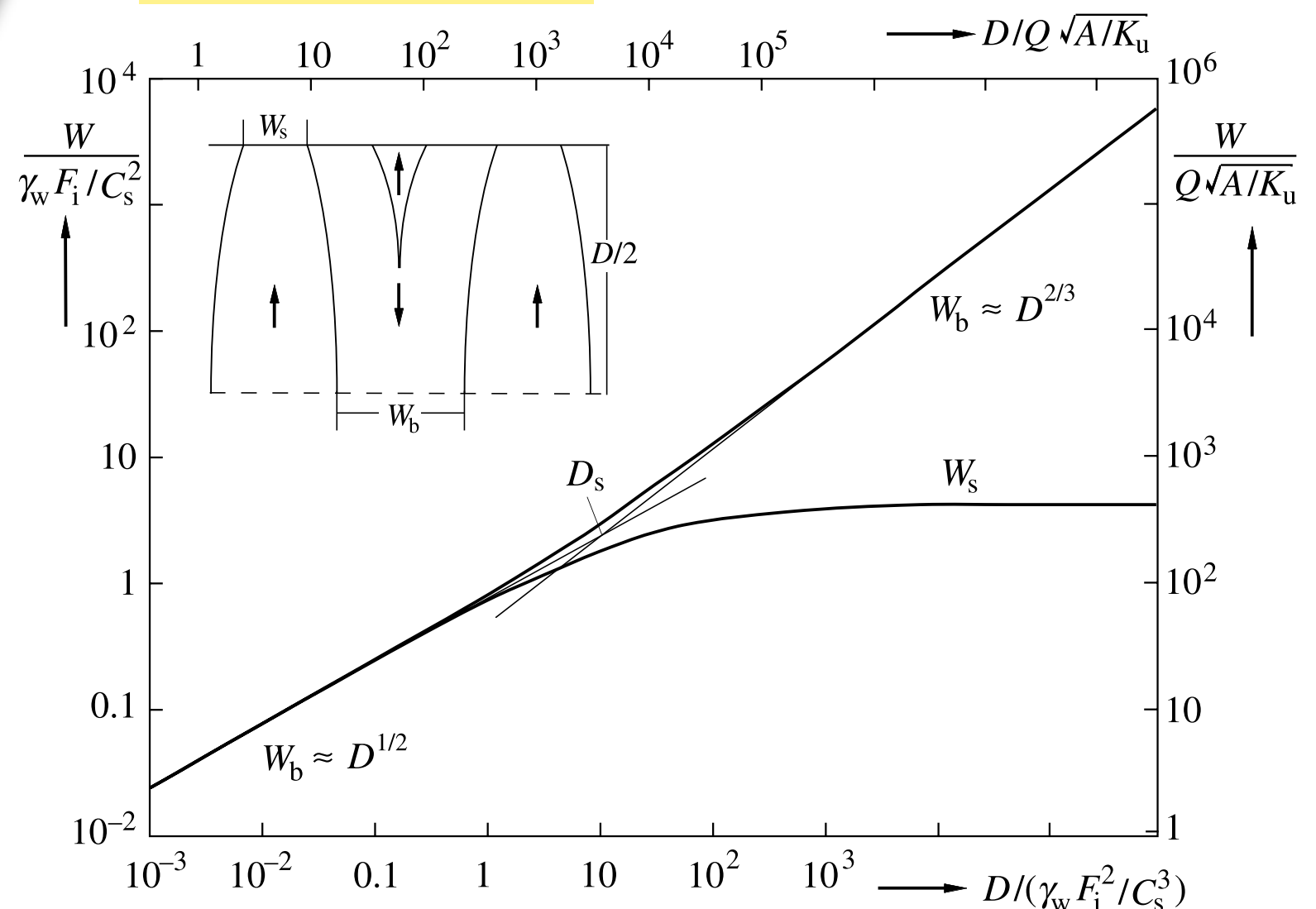
3.5 Domain Methods

Example: Surface domain width in bulk uniaxial crystals

NdFeB, top- and side views $20\ \mu\text{m}$

For sufficiently thick crystals: surface domain width W_s constant, independent of sample dimensions, depends only on wall energy γ_w and stray field energy constant $K_d = \mu_0 M_s^2 / 2$:

$$W_s = 108 \gamma_w / K_d$$

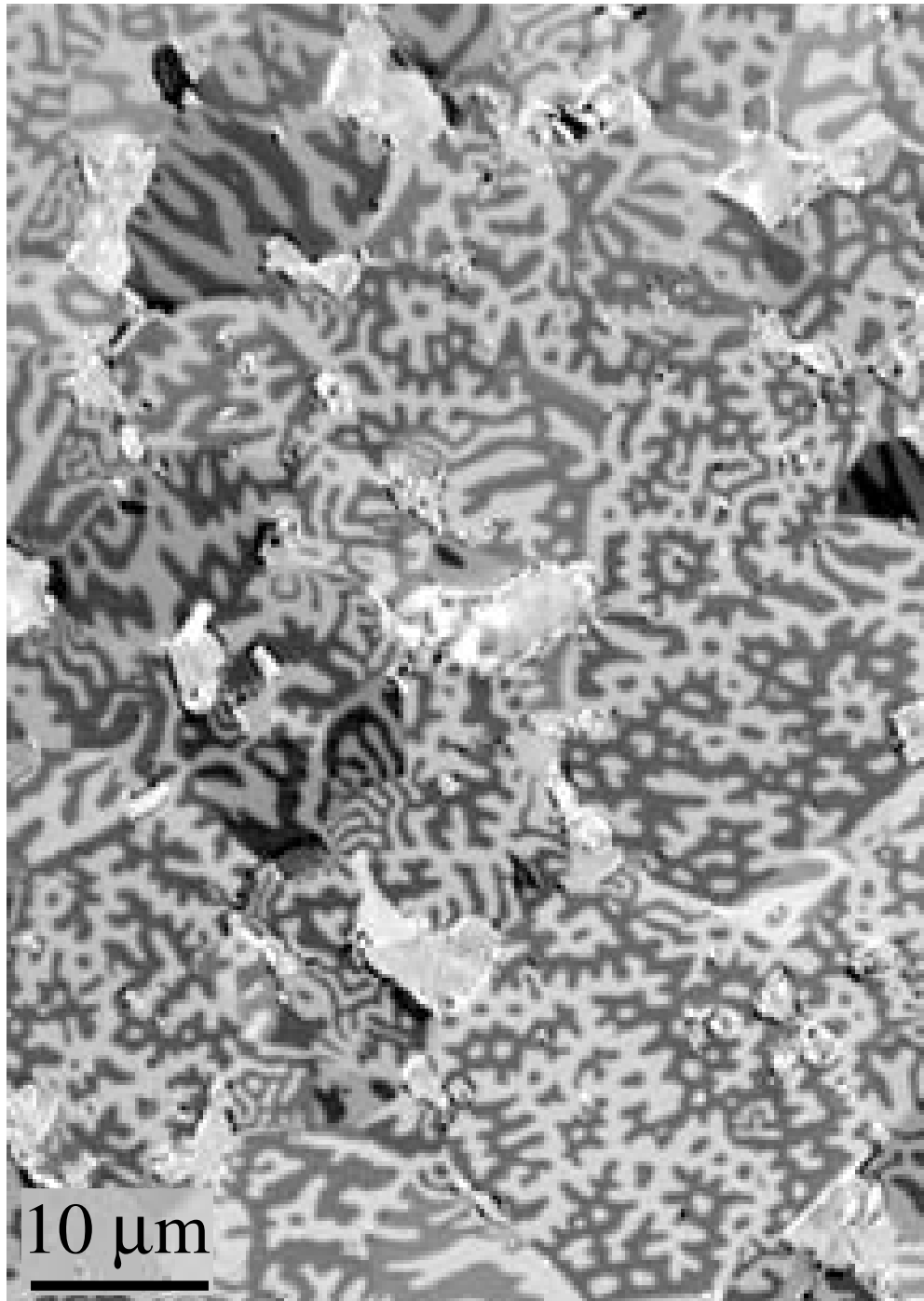


Anisotropy axis

3.5 Domain Methods

Example: Surface domain width in bulk uniaxial crystals

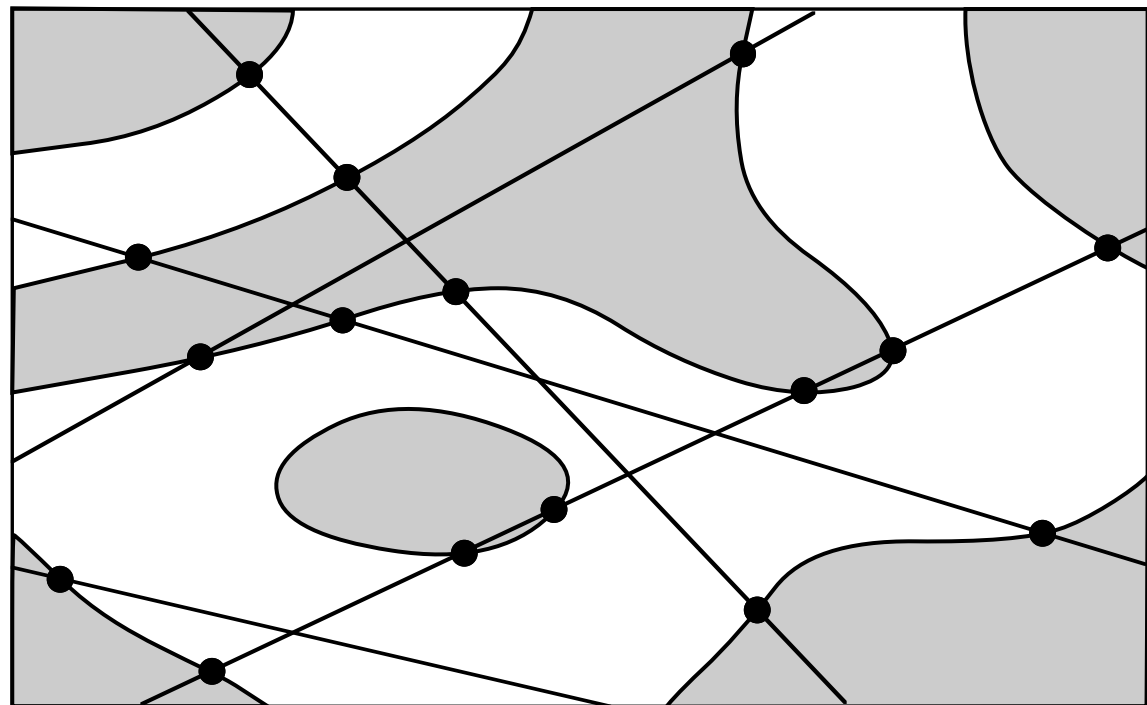
NdFeB magnet,
c-axis perpendicular



For sufficiently thick crystals: surface domain width W_s constant, independent of sample dimensions, depends only on wall energy γ_w and stray field energy constant $K_d = \mu_0 M_s^2 / 2$:

$$W_s = 108 \gamma_w / K_d$$

$$\gamma_w = \pi \sqrt{A / K_u}$$

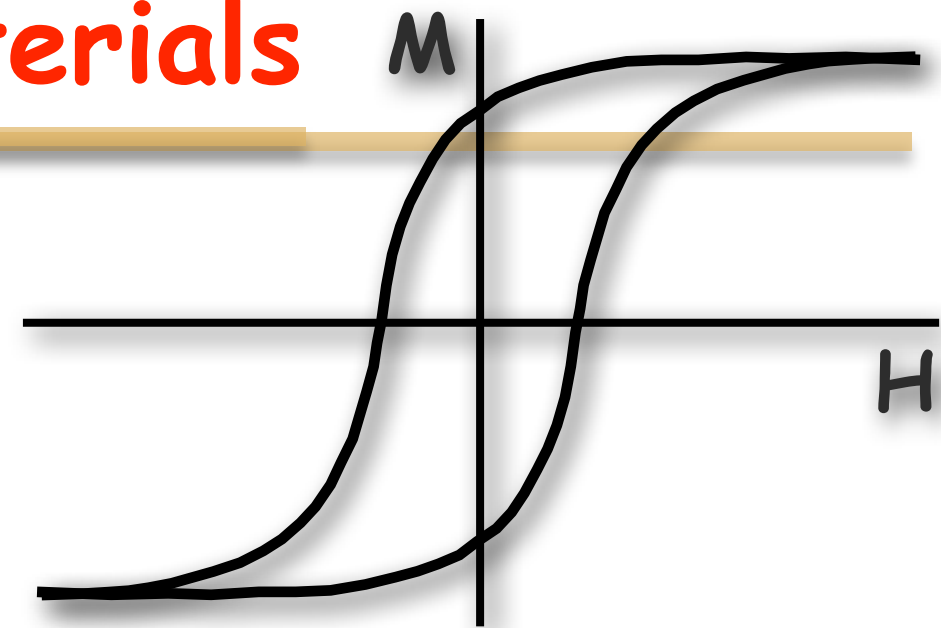


$$\text{Domain width} = \frac{2 \cdot \text{Total test line length}}{\pi \cdot \text{Number of intersections}}$$

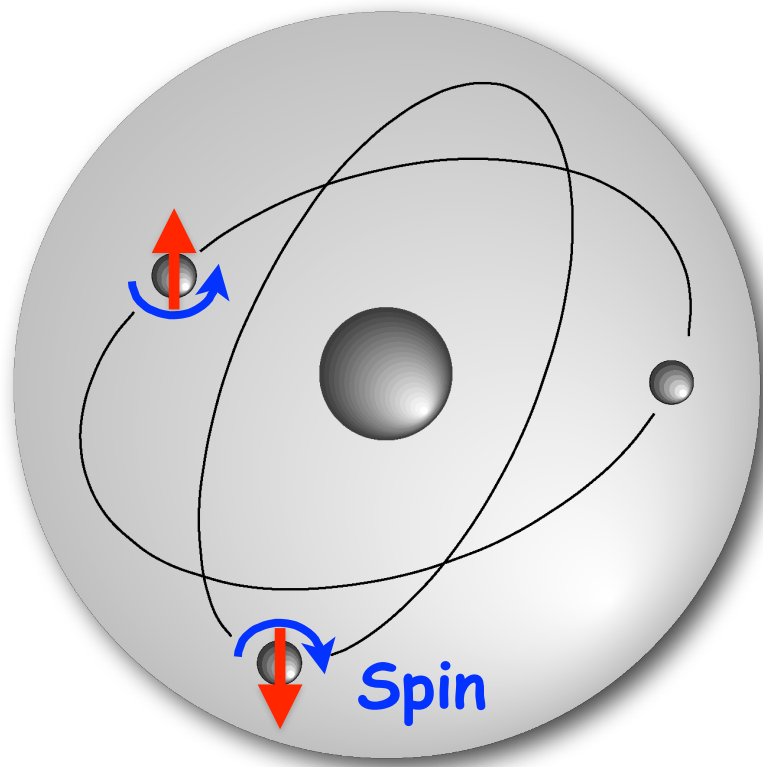
4.

Domain scale
measurements
(Magnetic Imaging)

Descriptive levels of magn. materials

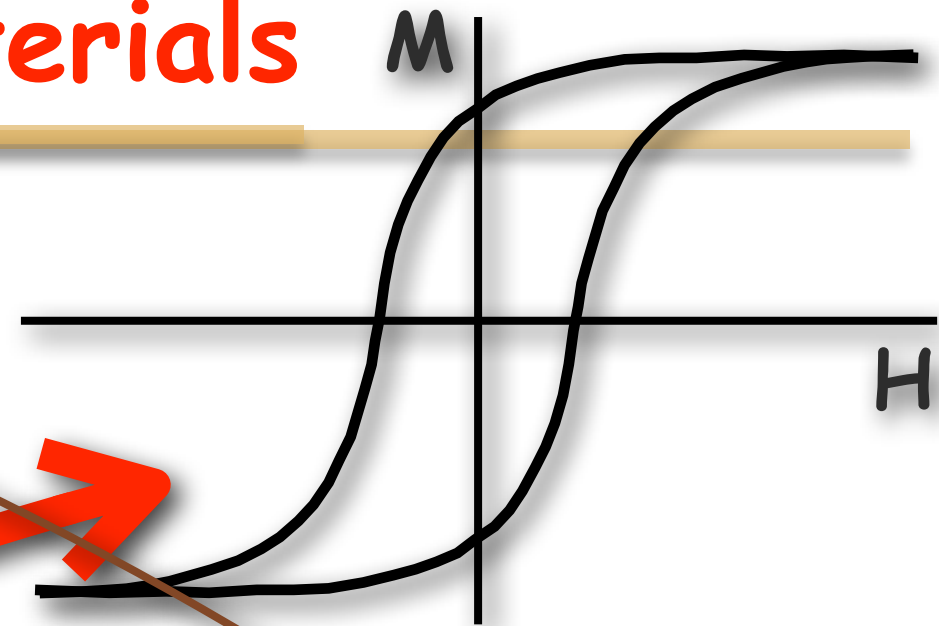


5. Magnetization curve



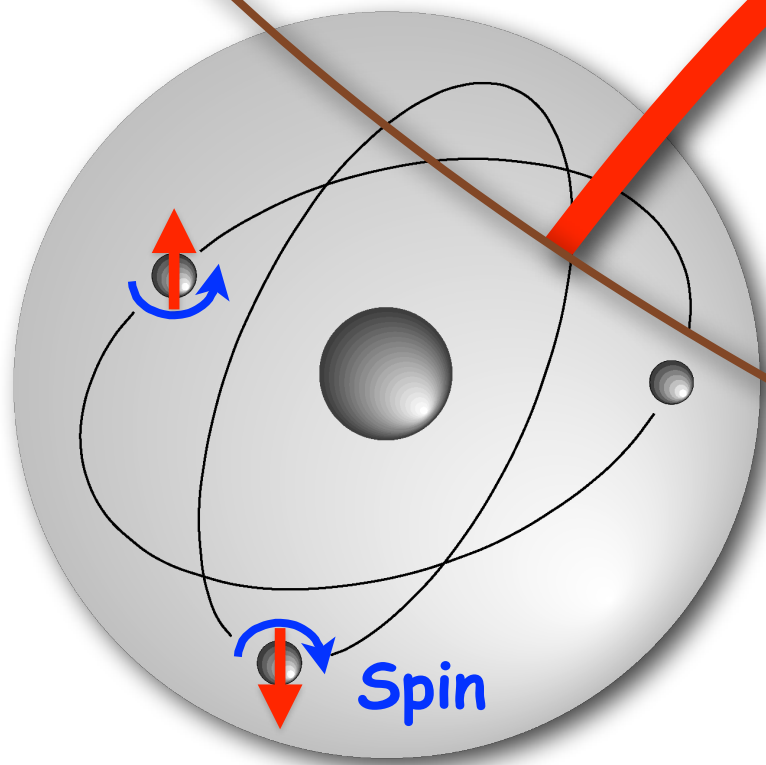
1. Atomic Foundation

Descriptive levels of magn. materials



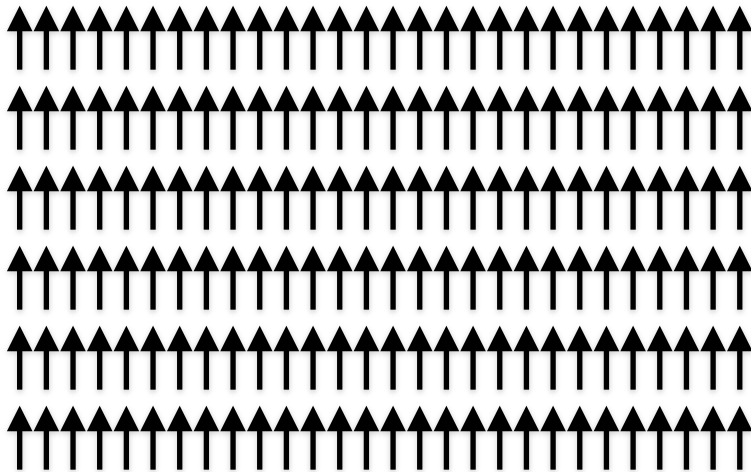
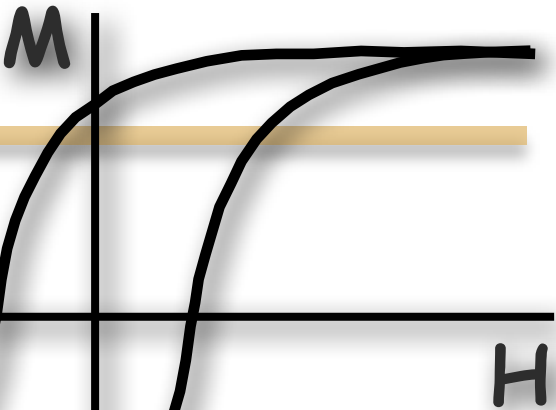
5. Magnetization curve

Magnetic Microstructure Analysis



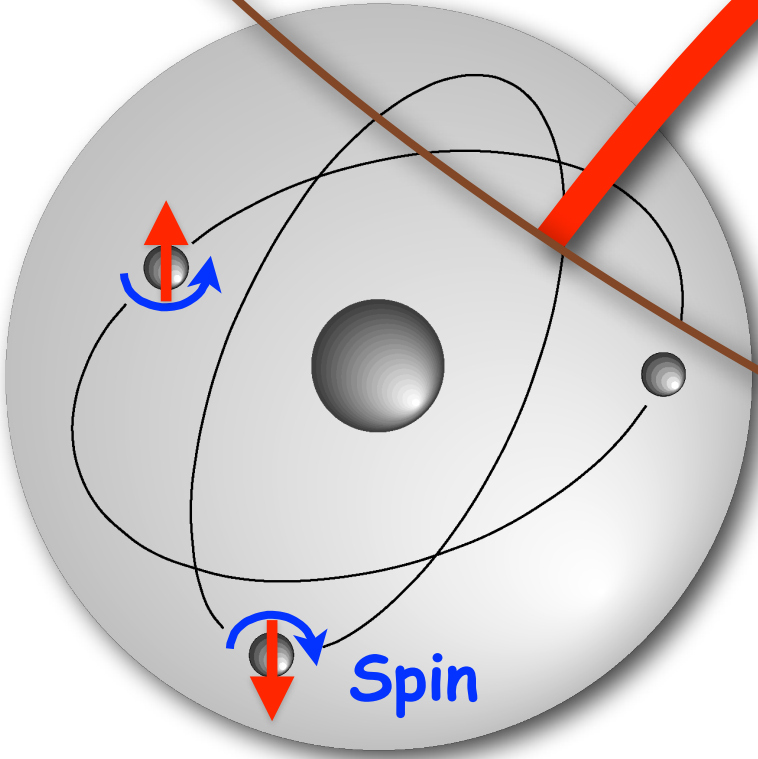
1. Atomic Foundation

Descriptive levels of magn. materials



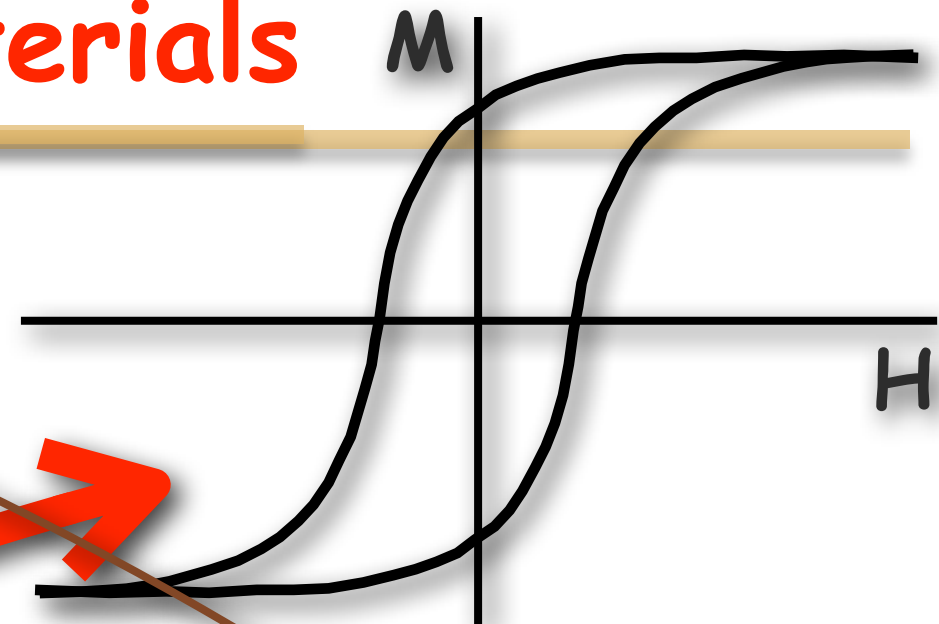
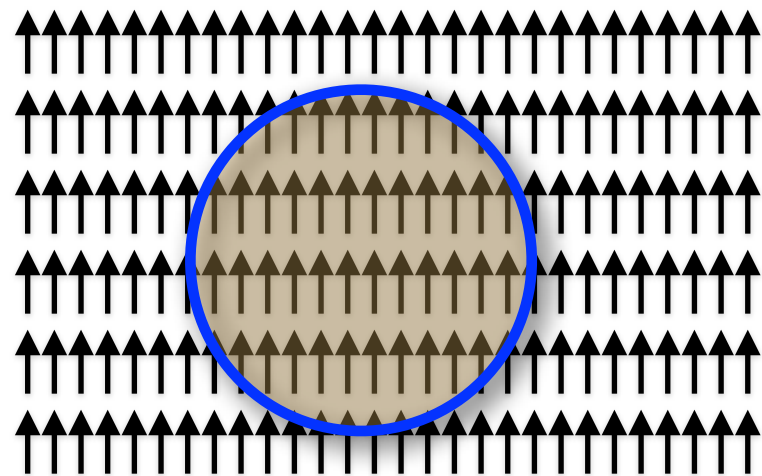
5. Magnetization curve

Magnetic Microstructure Analysis



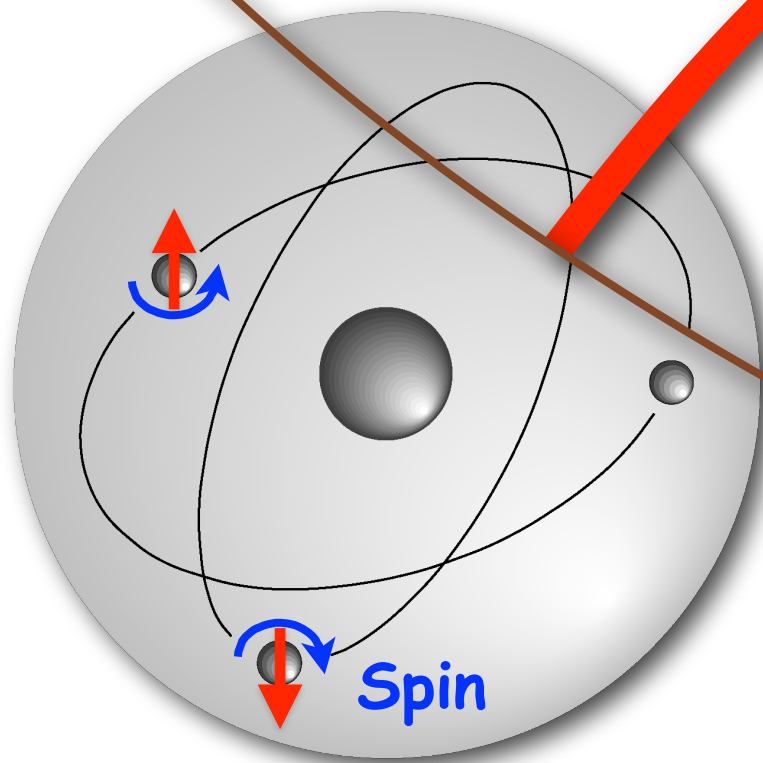
1. Atomic Foundation

Descriptive levels of magn. materials



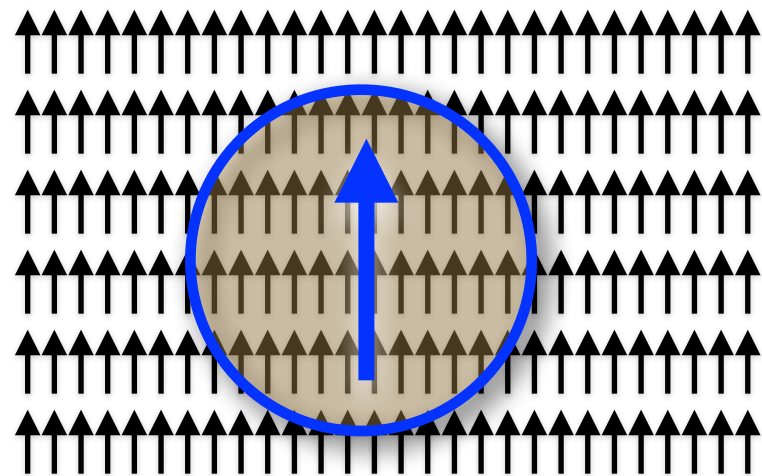
5. Magnetization curve

Magnetic Microstructure Analysis

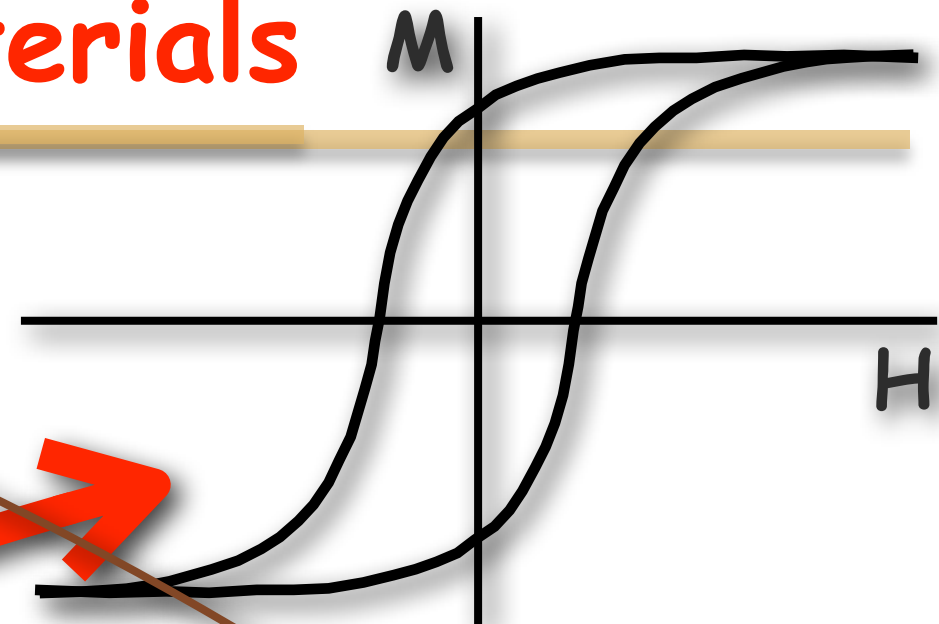


1. Atomic Foundation

Descriptive levels of magn. materials

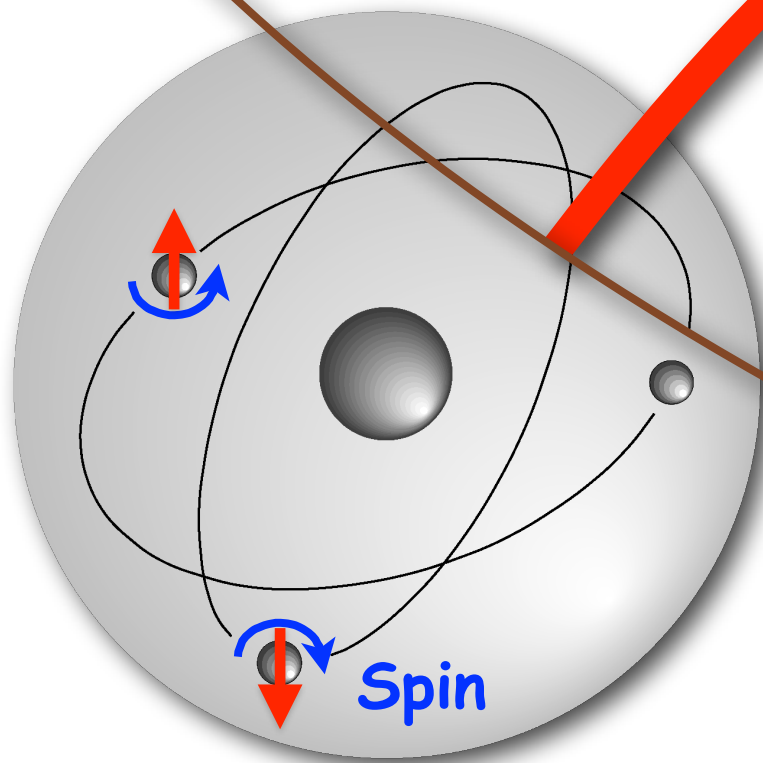


$$m = M/M_s$$



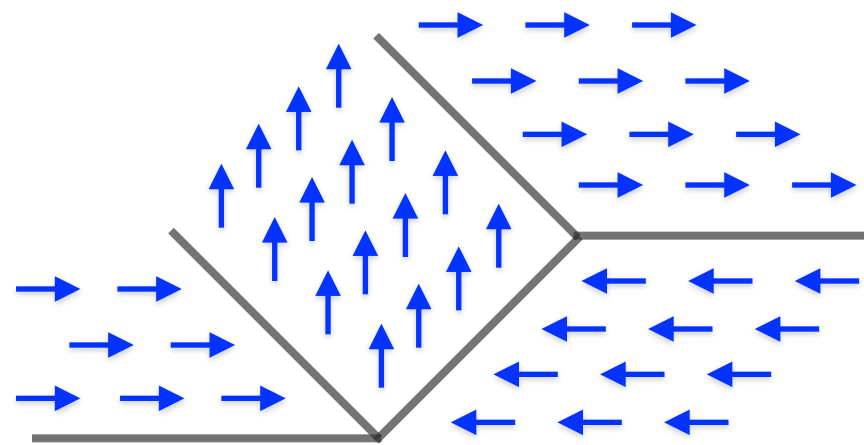
5. Magnetization curve

Magnetic Microstructure Analysis

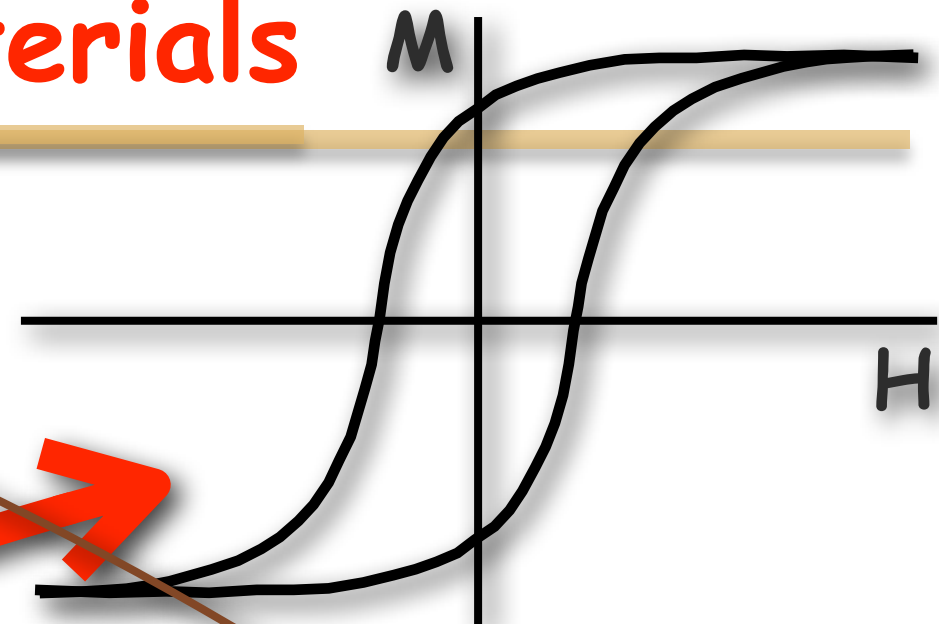


1. Atomic Foundation

Descriptive levels of magn. materials

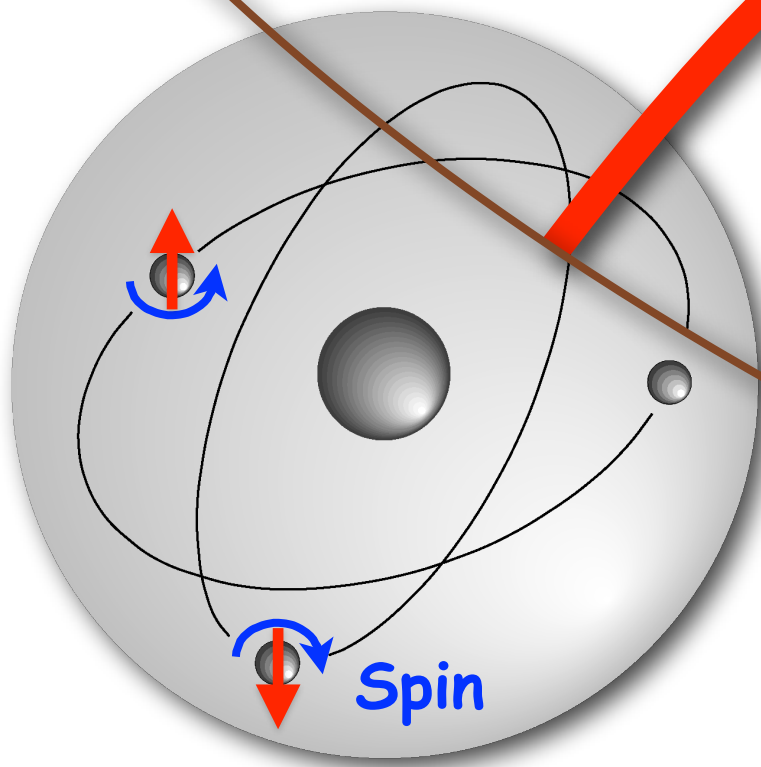


$$m(r), m^2 = 1$$



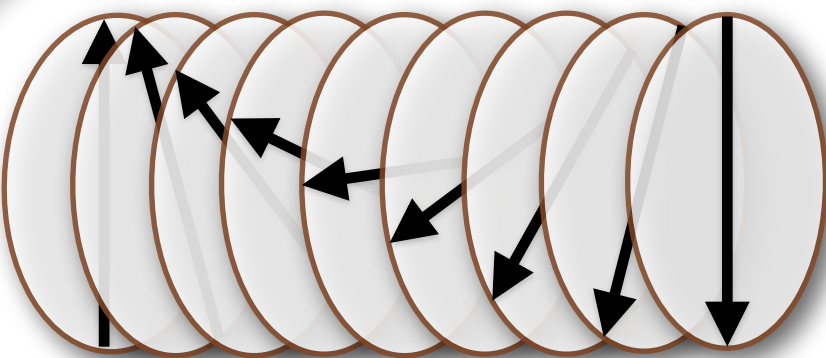
5. Magnetization curve

Magnetic Microstructure Analysis

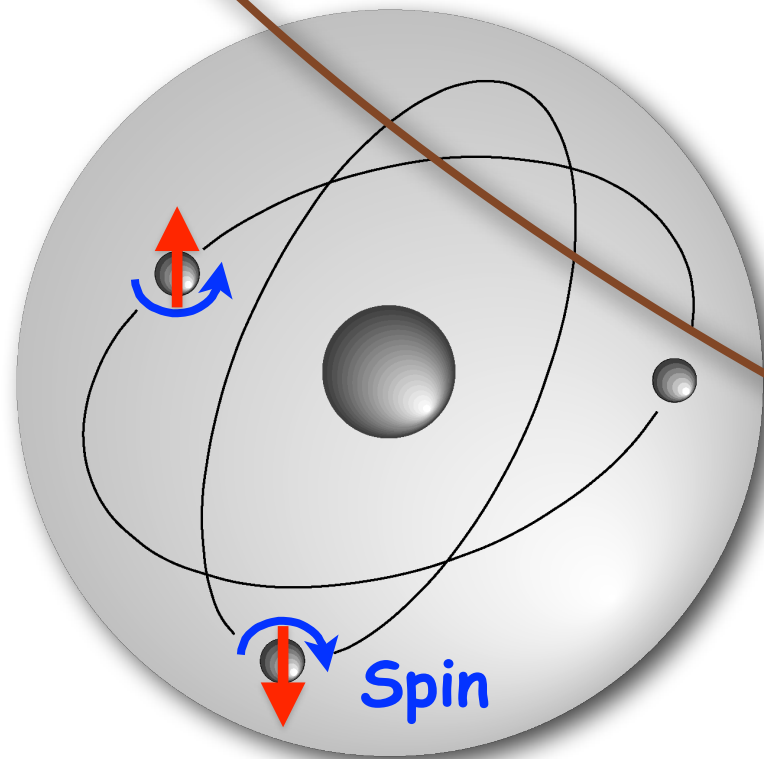


1. Atomic Foundation

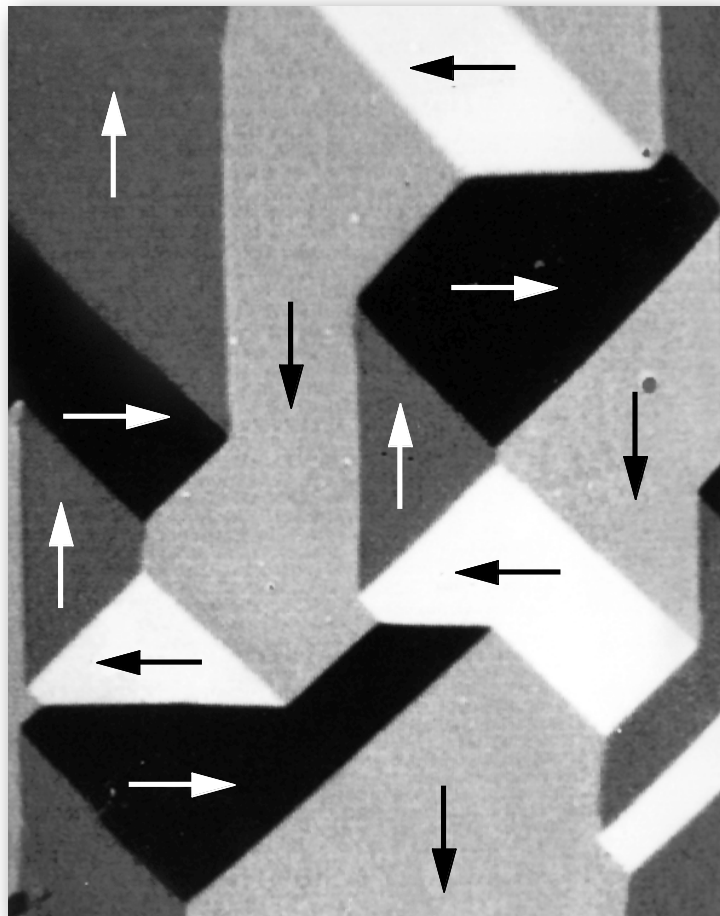
Descriptive levels of magn. materials



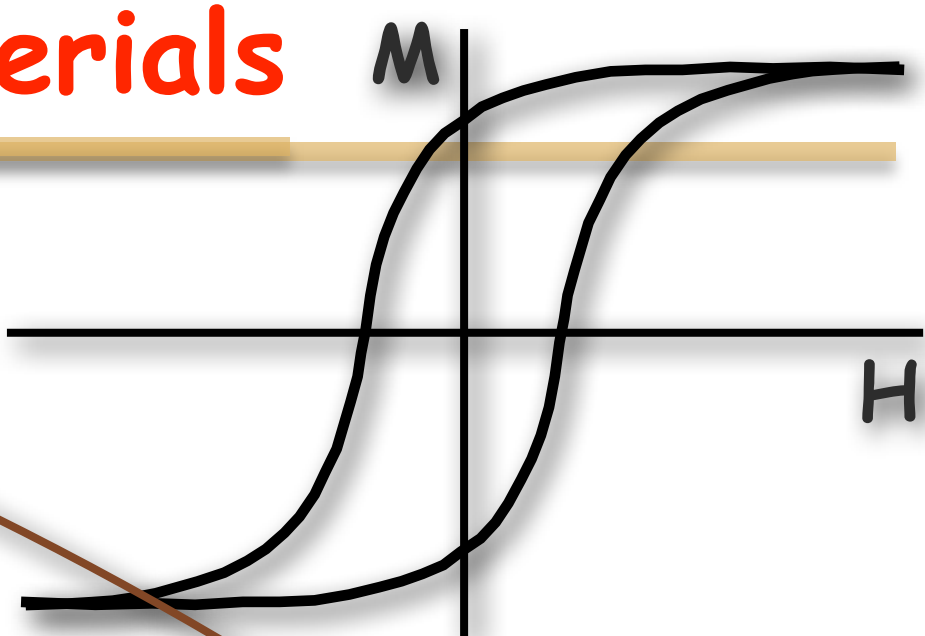
2. Micromagnetic Analysis



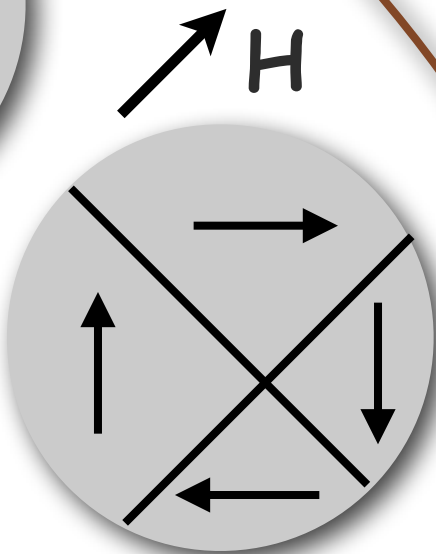
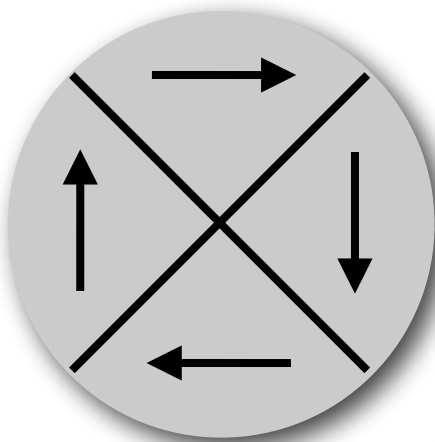
1. Atomic Foundation



3. Domain Analysis



5. Magnetization curve



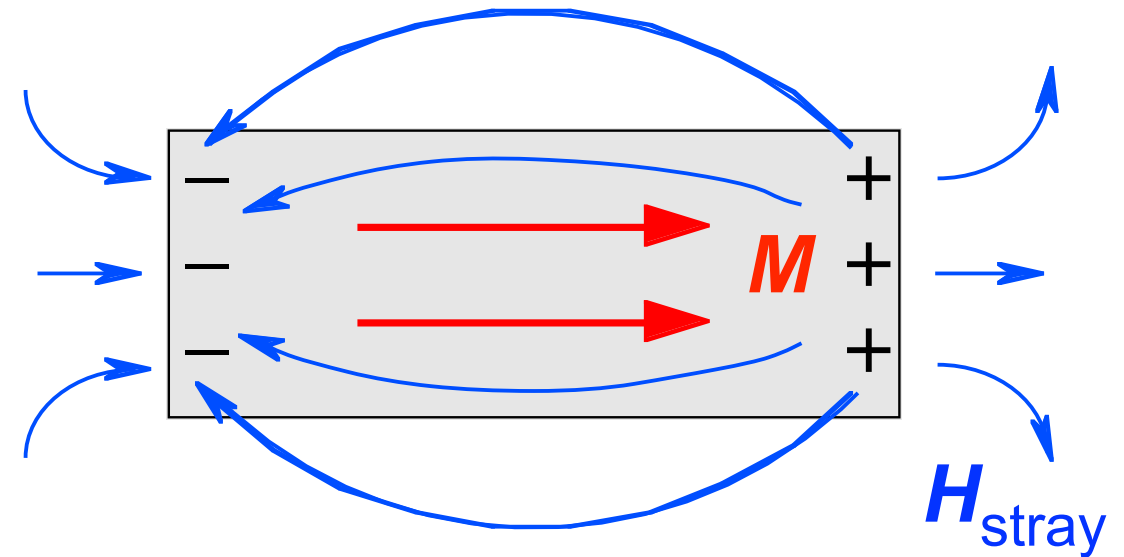
4. Phase Analysis

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

$$\text{div } \mathbf{B} = 0$$

$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



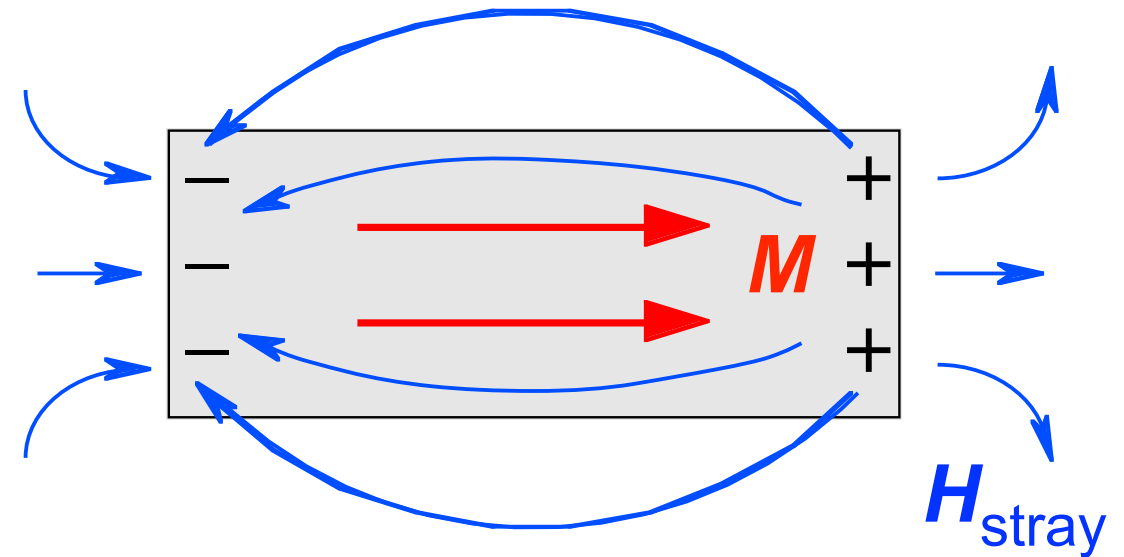
- Sensitive to $\mathbf{H}_{\text{stray}}$
 - Bitter technique
 - Magnetic force microscopy
 - Hall probe microscopy
- Sensitive to \mathbf{M}
 - Magneto-optical microscopy
 - X-ray spectroscopy
 - Polarized electrons (SEMPA, SPT)
- Sensitive to \mathbf{B}
 - Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

$$\text{div } \mathbf{B} = 0$$

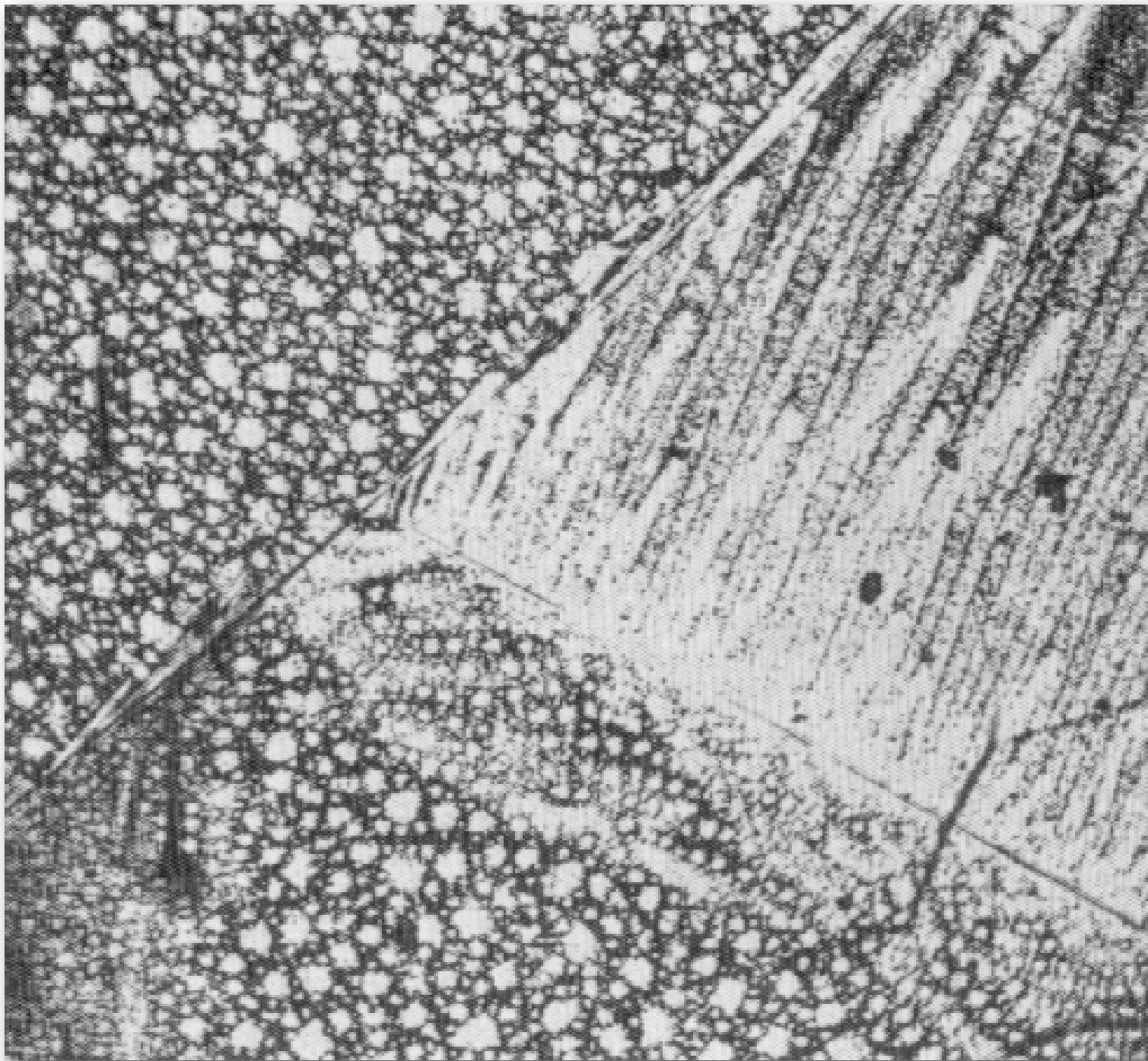
$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



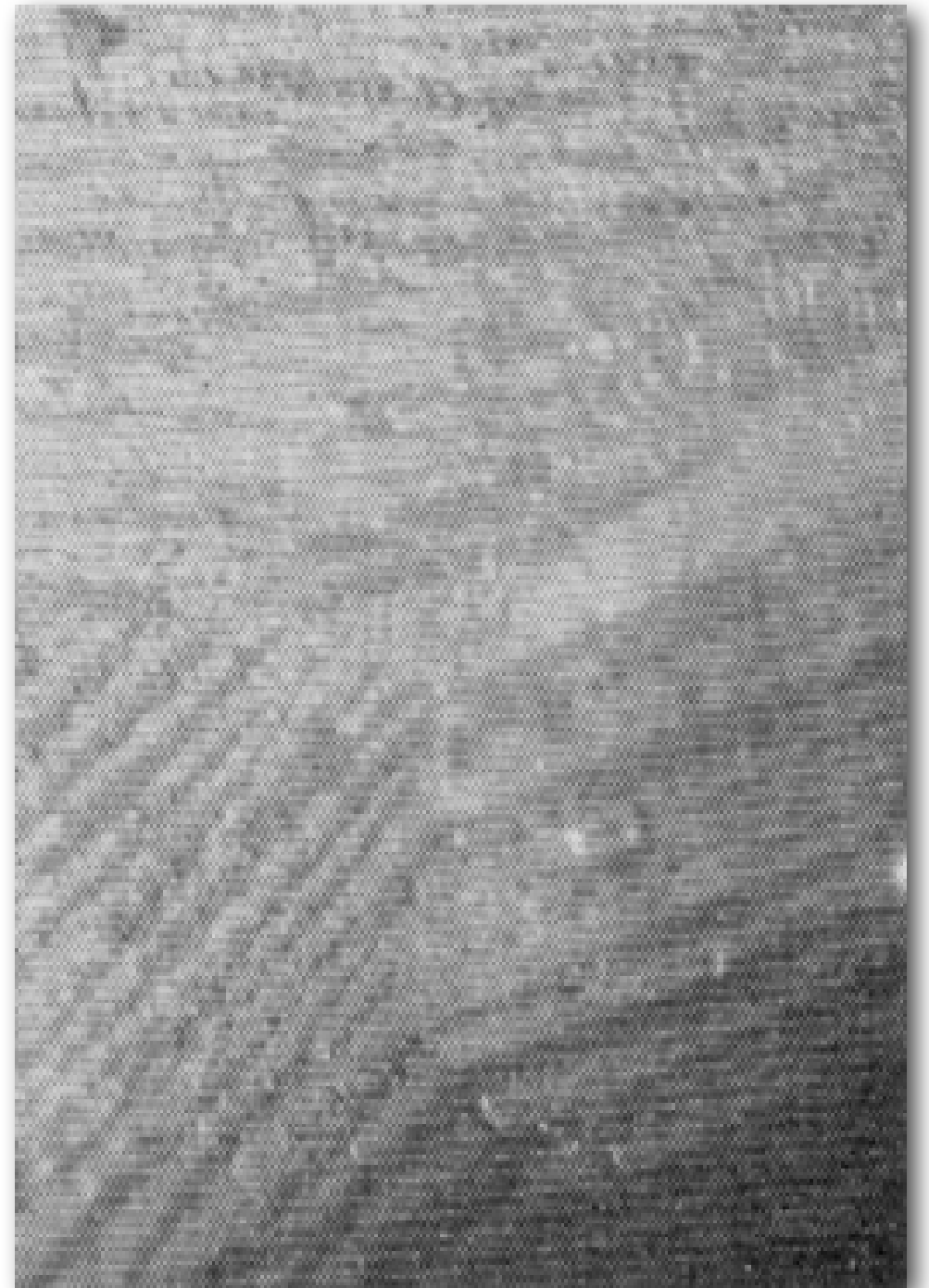
- Sensitive to **H_{stray}**
 1. Bitter technique
 2. Magnetic force microscopy
 3. Hall probe microscopy
- Sensitive to **M**
 - Magneto-optical microscopy
 - X-ray spectroscopy
 - Polarized electrons (SEMPA, SPT)
- Sensitive to **B**
 - Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

1. Bitter technique

History: first imaging of domains by F. Bitter, 1931)



Cobalt

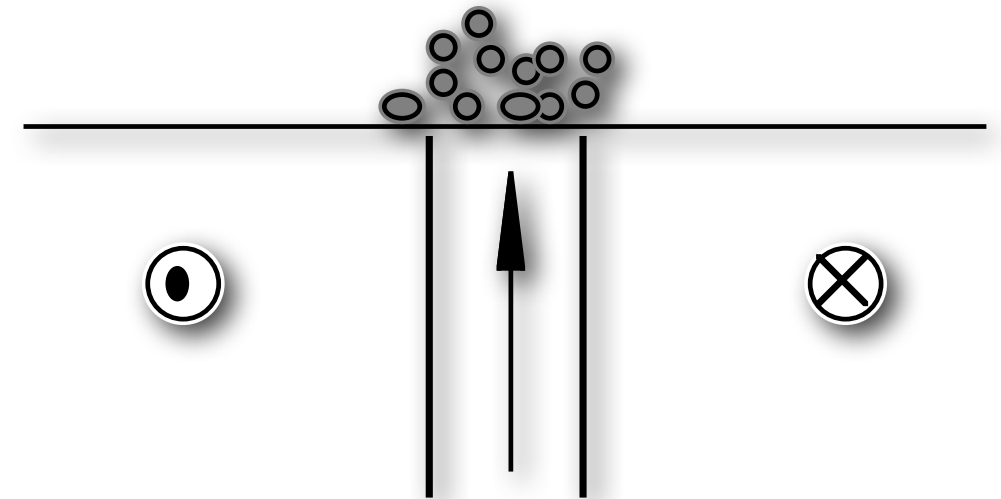


FeSi

1. Bitter technique

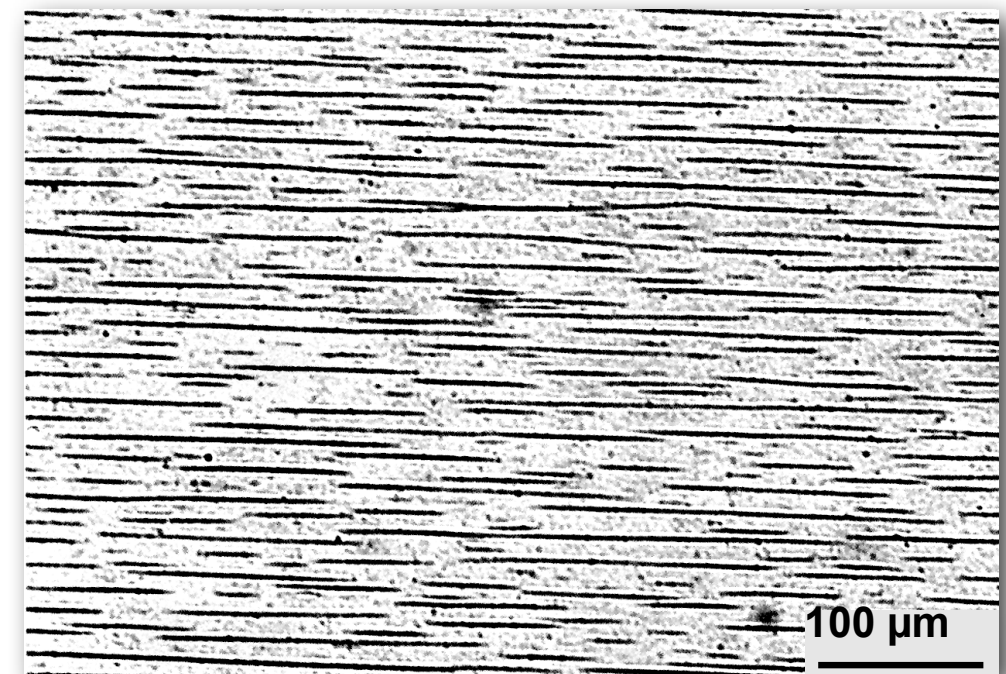
Principle

- Magnetic colloid: Magnetite particles (diameter about 10 nm) in water
- Accumulation in stray field at sample surface



Sensitivity

- Reversible agglomeration in weak magnetic field
 - Increase of volume, elongated shape
 - Large susceptibility
 - Large sensitivity to stray fields in order of a few 100 A/m



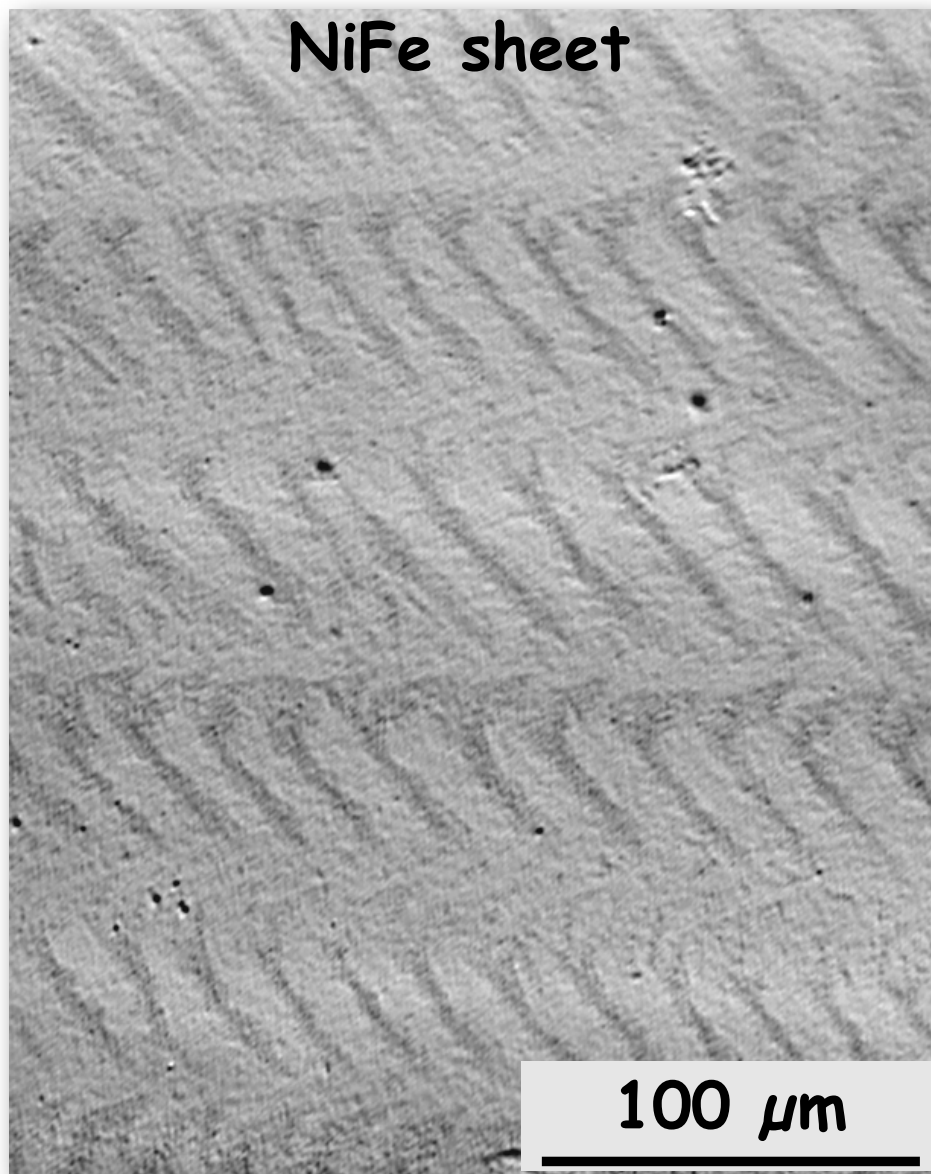
Agglomeration in magnetic field
(560 A/m)

1. Bitter technique

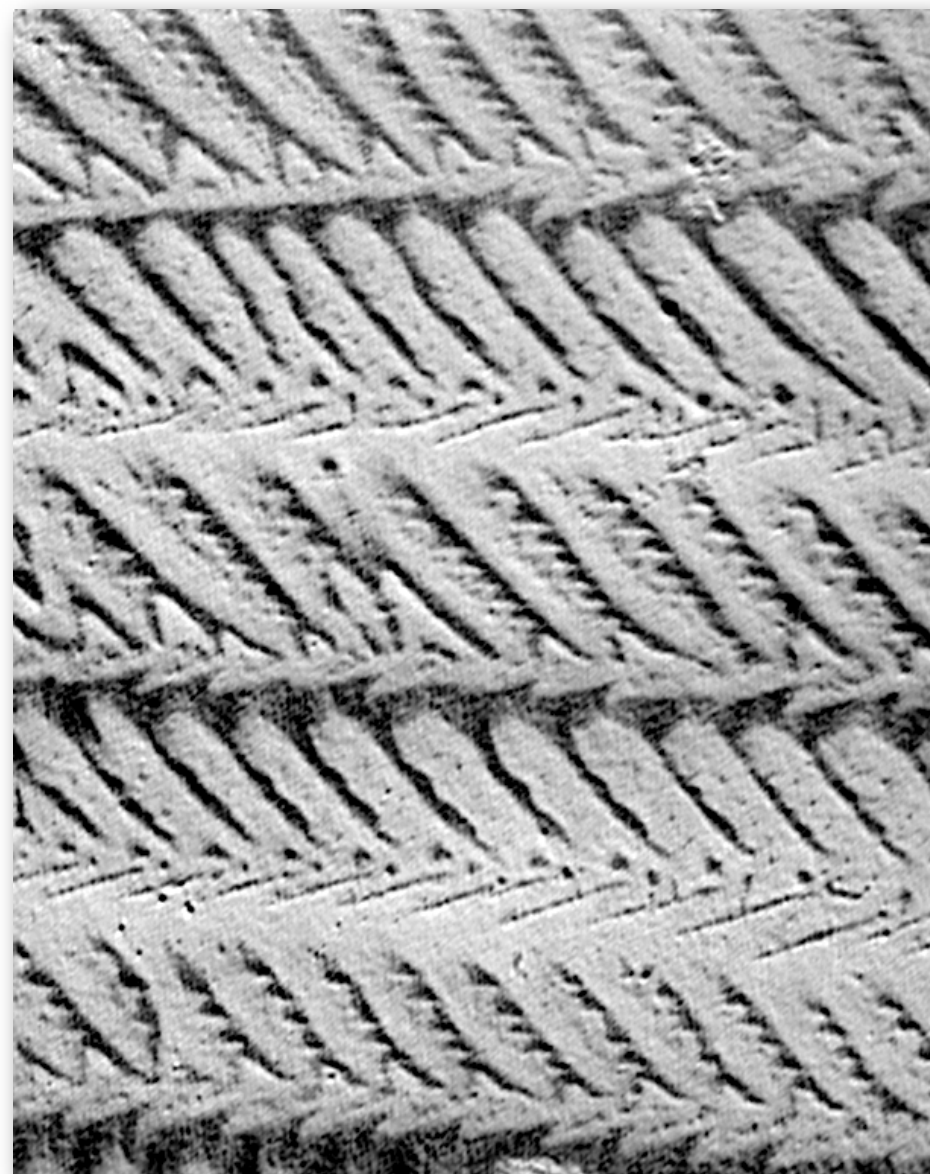
Sensitivity

Increase of sensitivity in weak perpendicular field

→ Domain imaging in soft magnetic materials



Without auxiliary field



With perpendicular field

● 1.5 kA/m

1. Bitter technique

Dry colloid technique

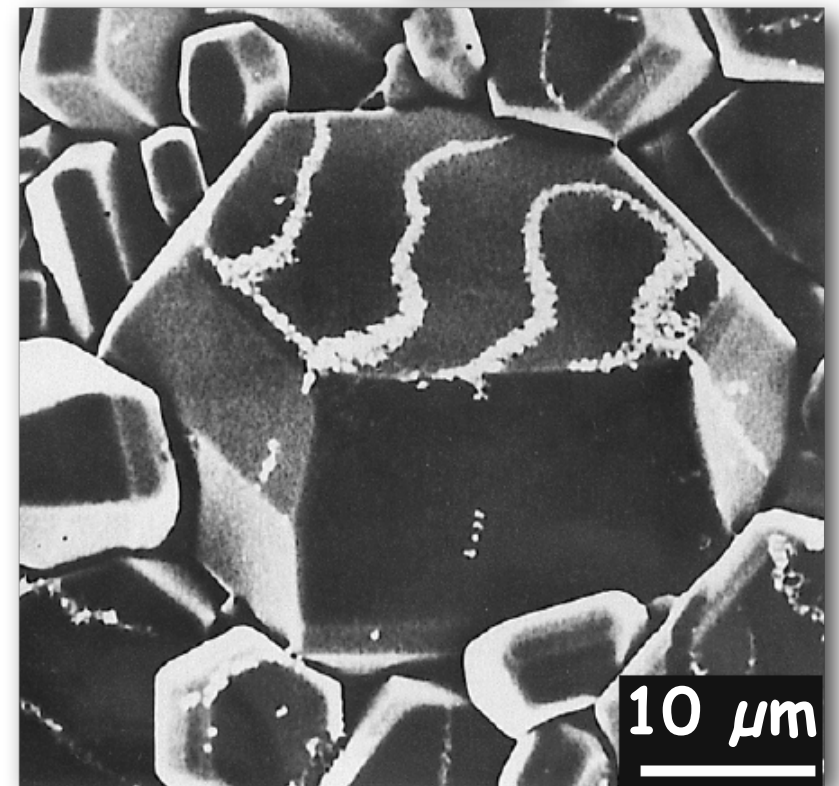
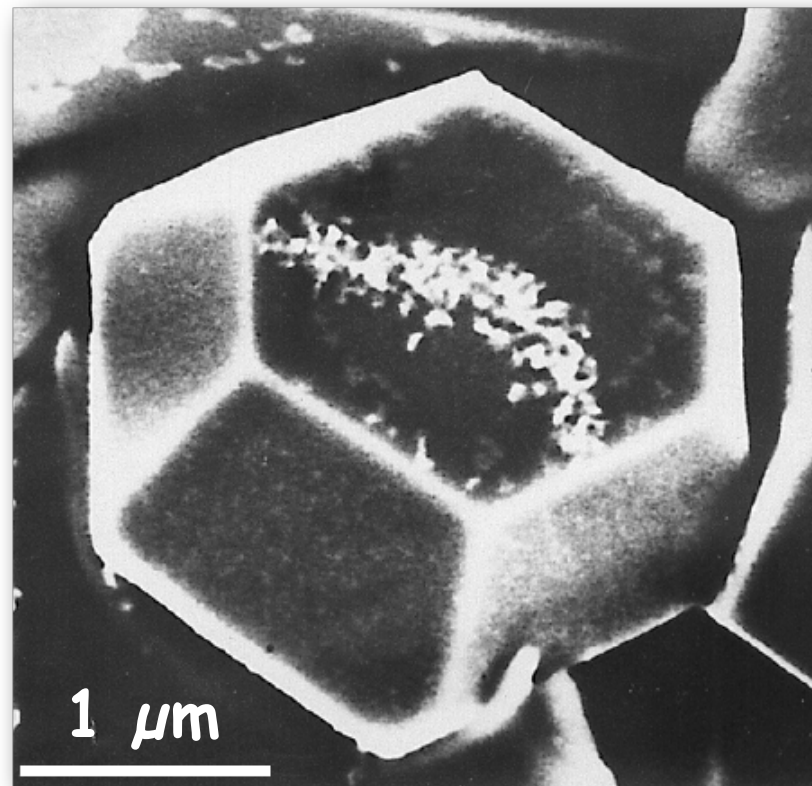
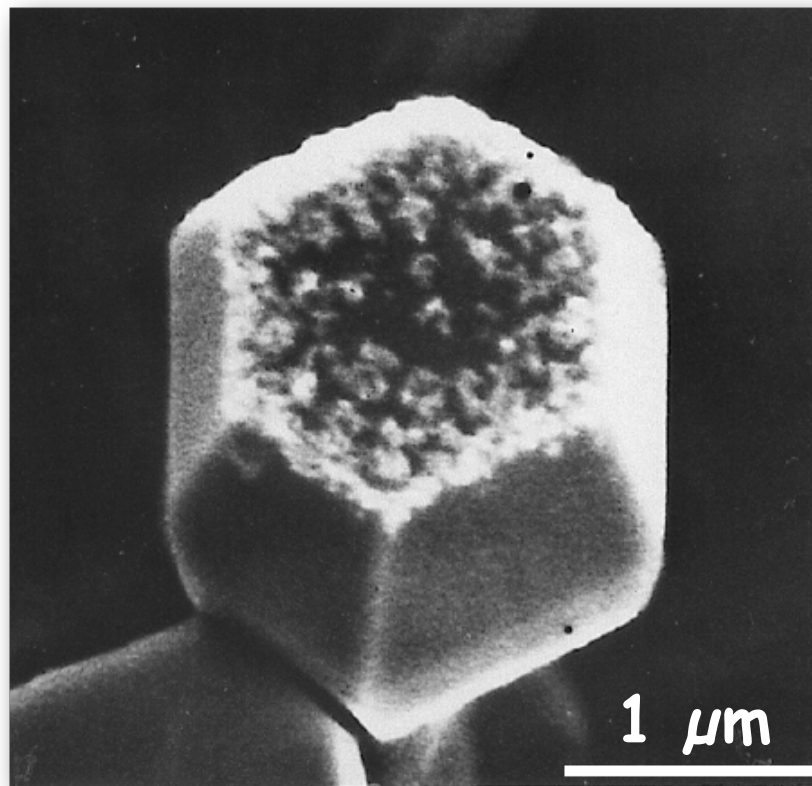
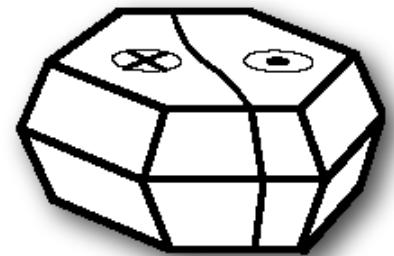
Allowing colloid to dry on surface

Adding agent

→ Strippable film

→ Imaging in electron microscope

Ba-Ferrite particles (courtesy K. Goto, Sendai)



Dry colloid technique:

Static domain observation
on rough, 3-dimensional surfaces
at high resolution of some 10 nm

1. Bitter technique

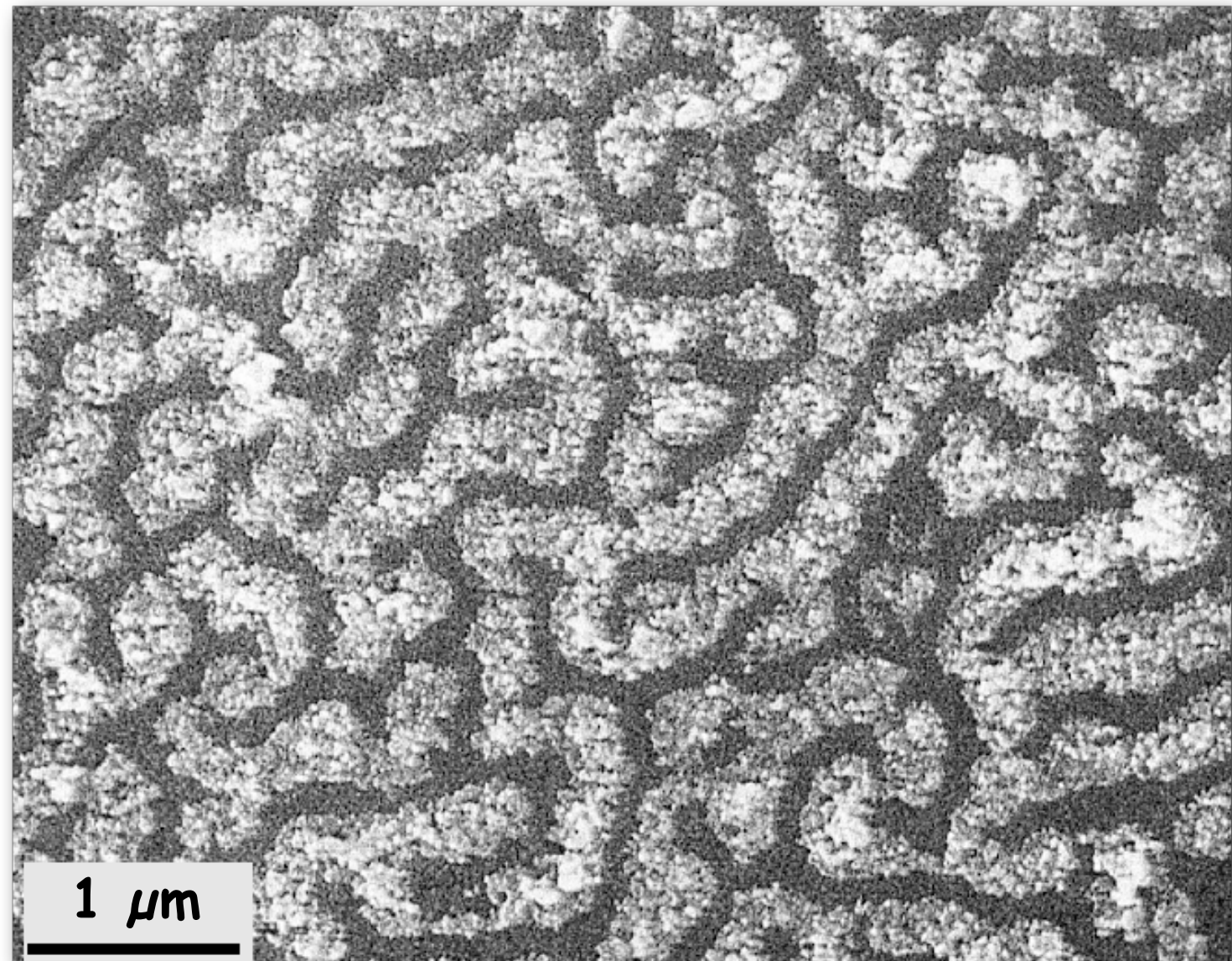
Dry colloid technique

Allowing colloid to dry on surface

Adding agent

- Strippable film
- Imaging in electron microscope

CoCr recording medium
(courtesy J. Simsová, Prague)



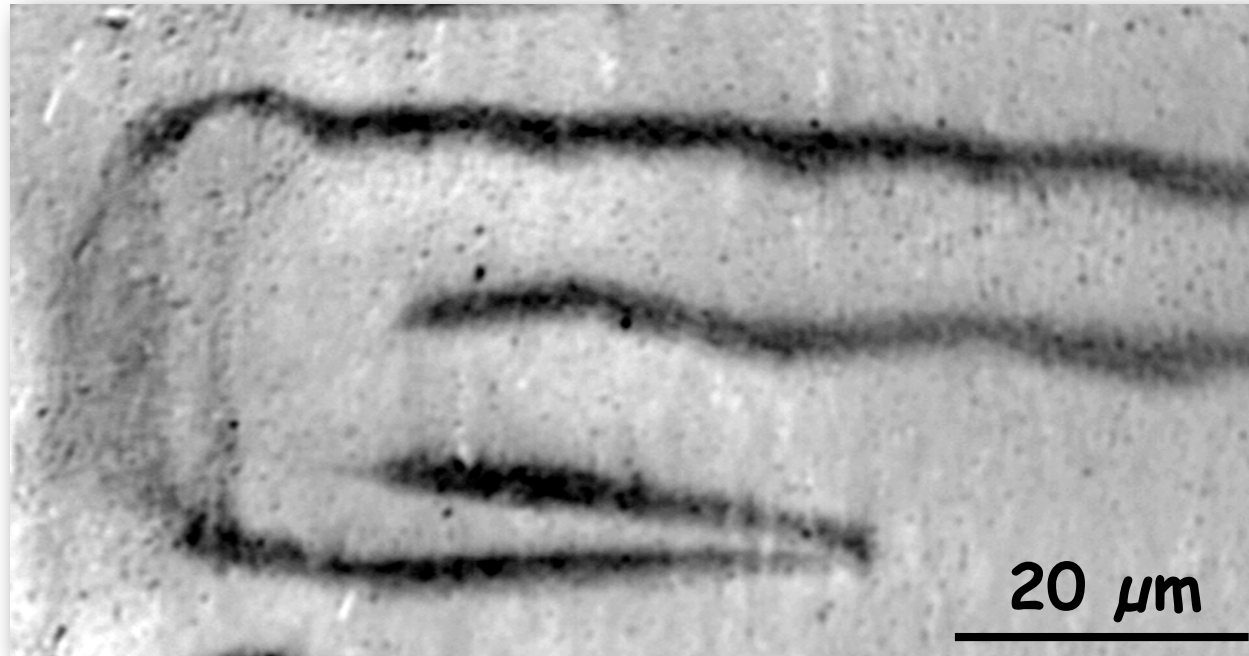
Dry colloid technique:

Static domain observation
on rough, 3-dimensional surfaces
at high resolution of some 10 nm

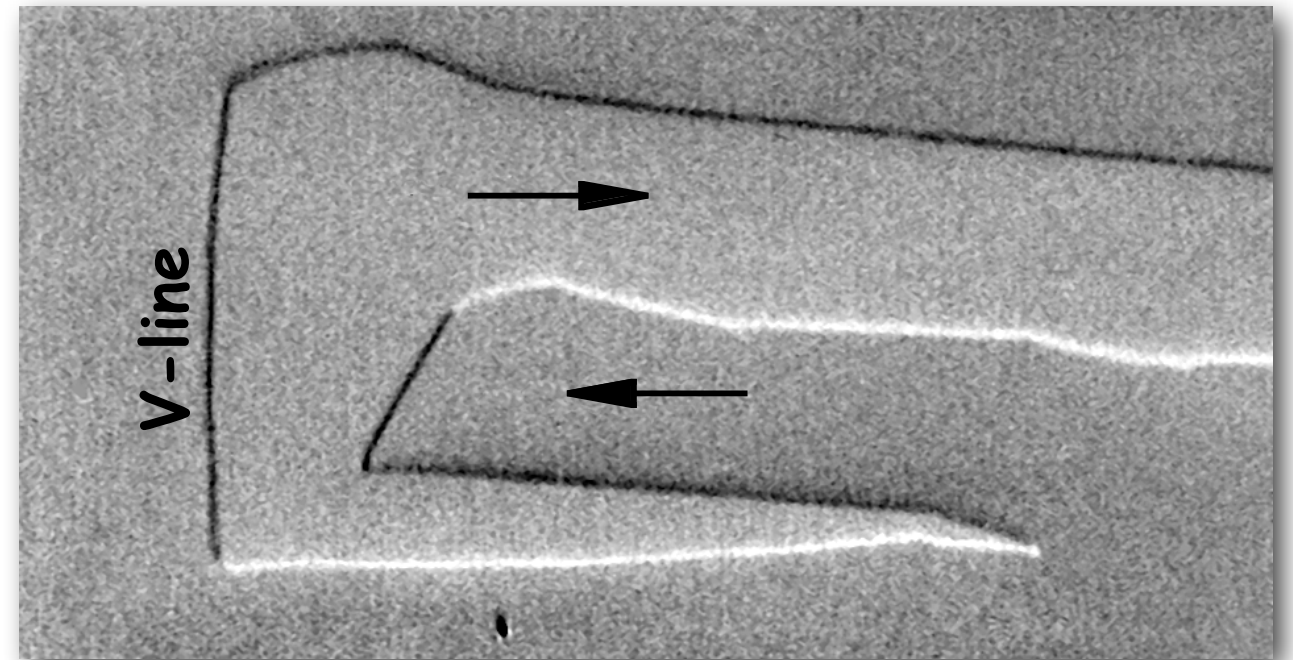
1. Bitter technique

Visible and invisible features

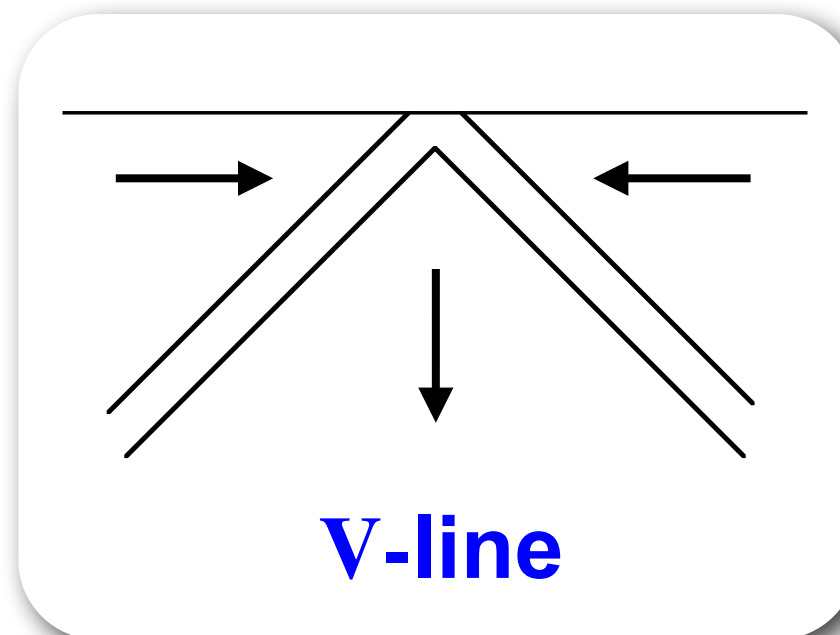
V-lines



Bitter image



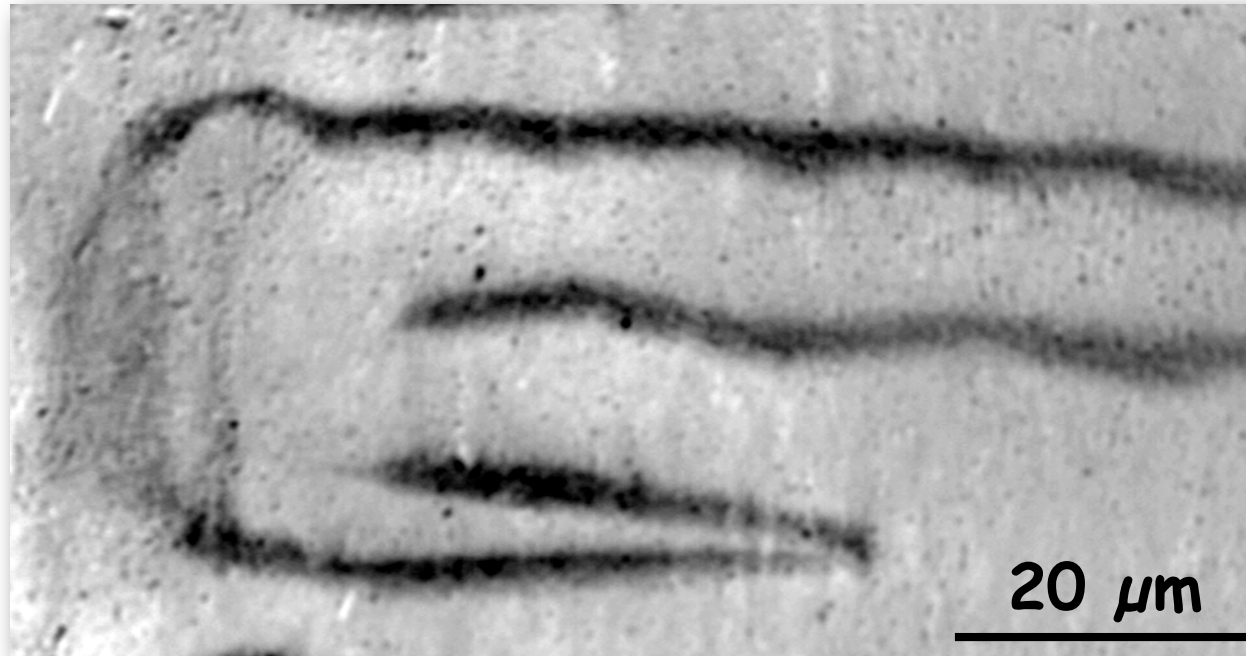
Kerr image



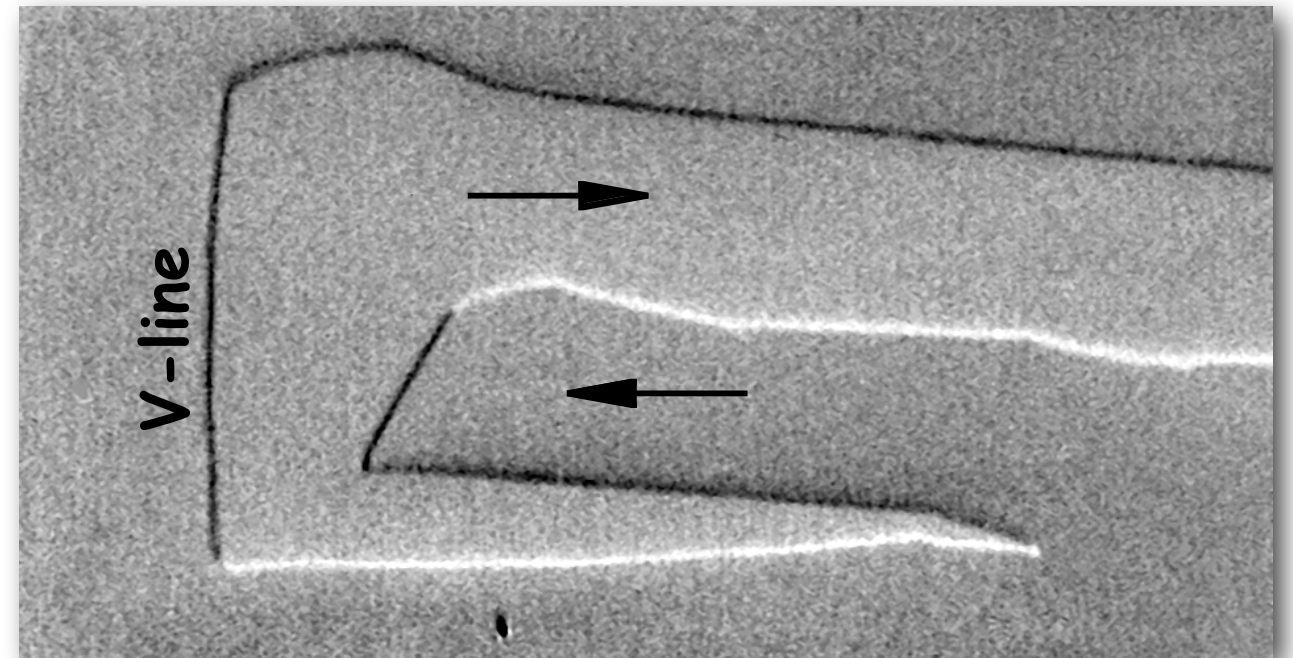
1. Bitter technique

Visible and invisible features

V-lines

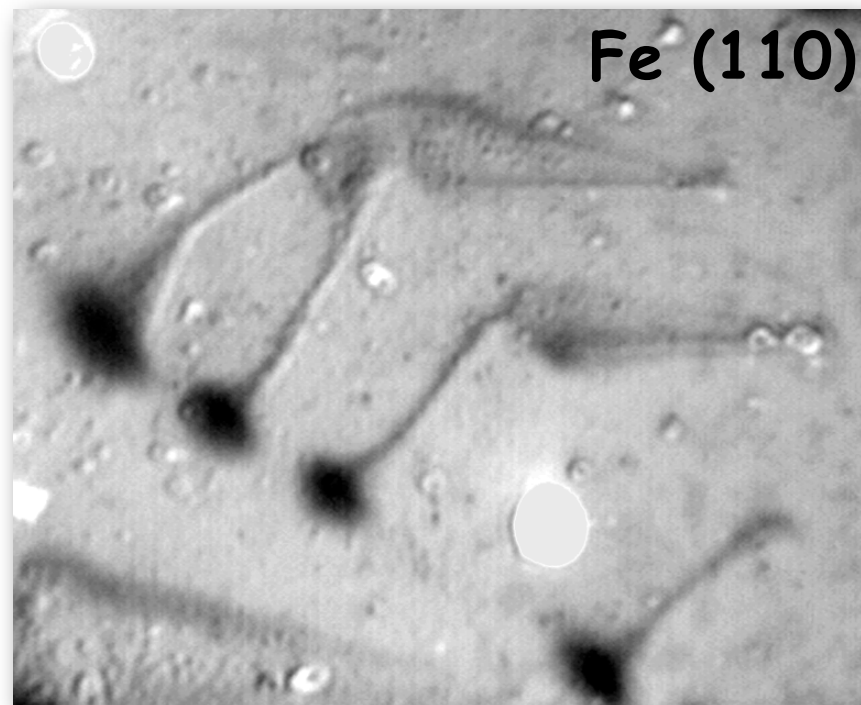


Bitter image

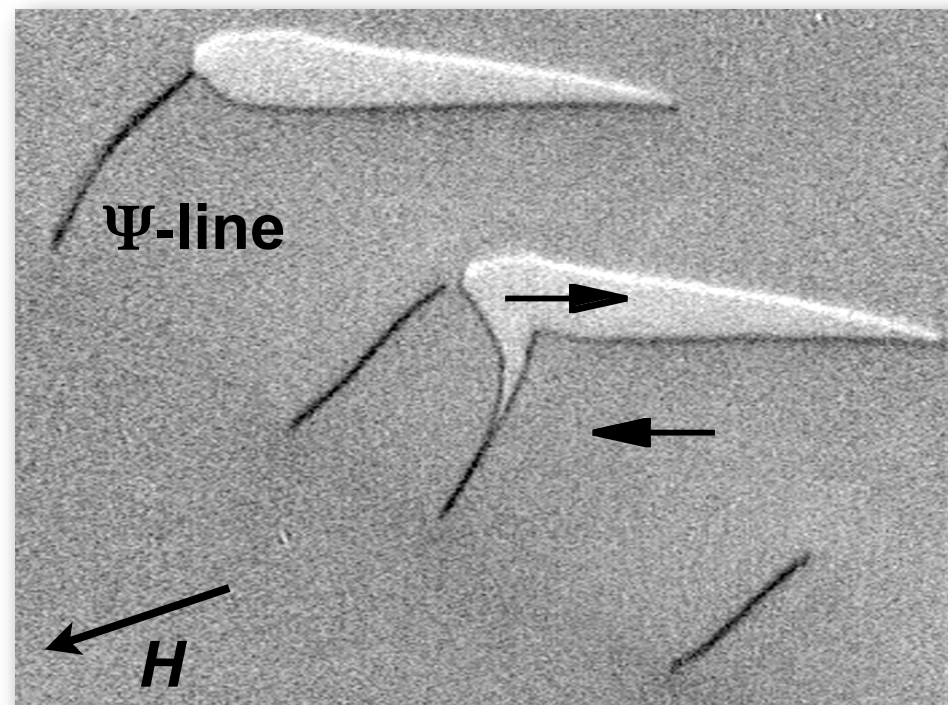


Kerr image

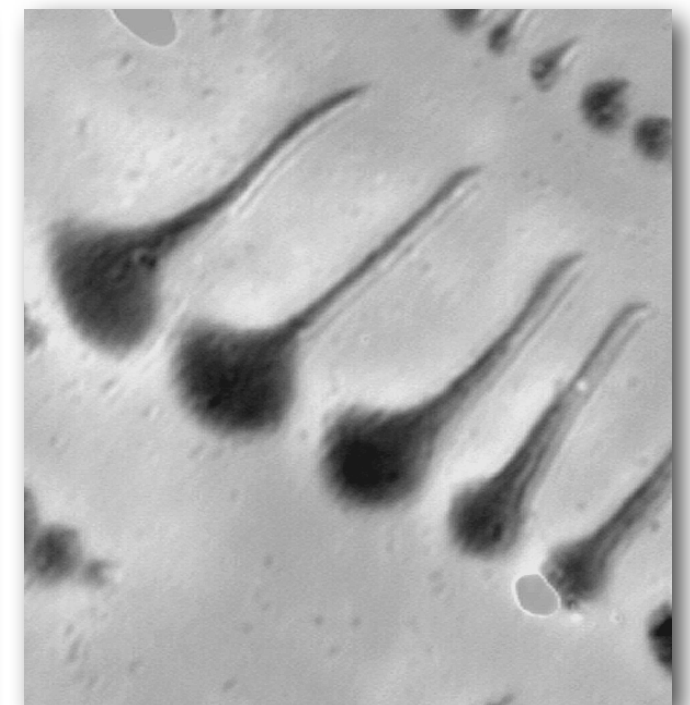
Ψ -lines



Bitter image



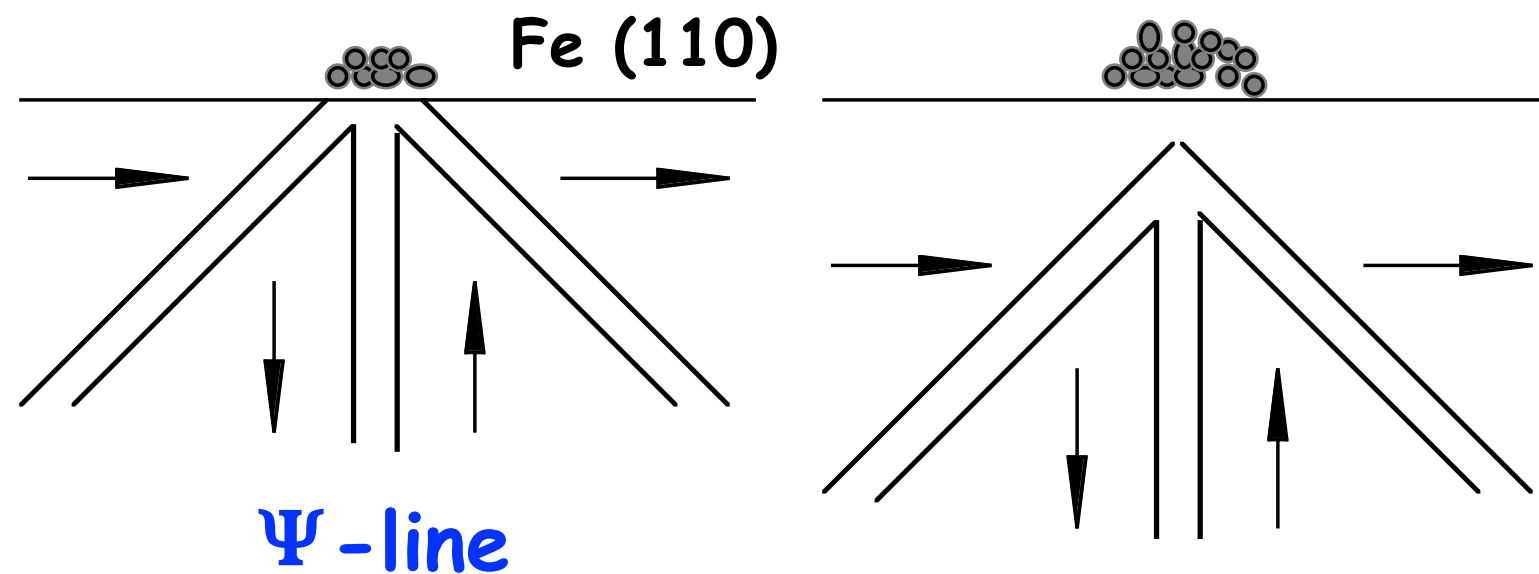
Kerr image



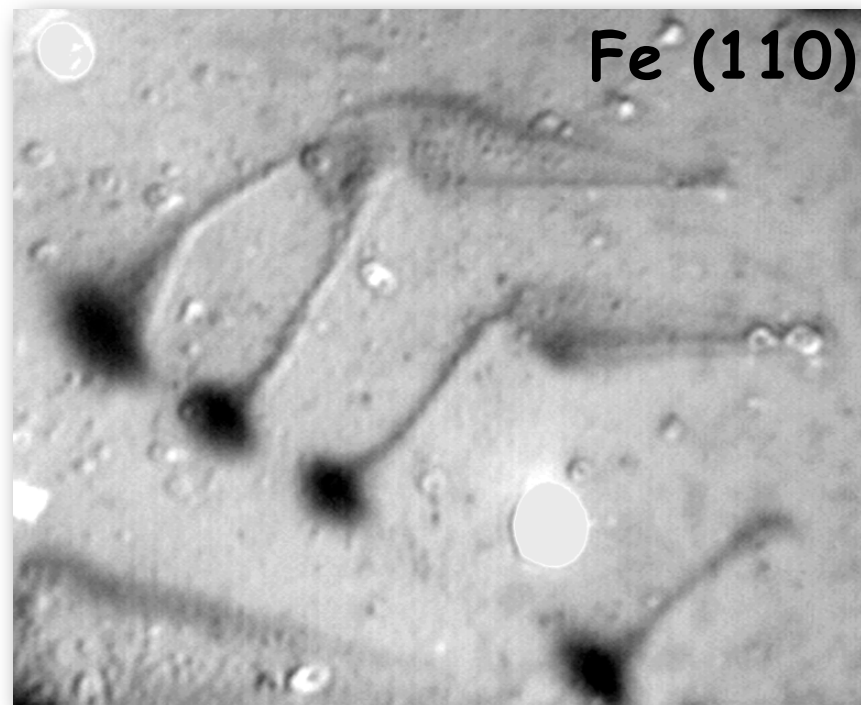
Bitter image

1. Bitter technique

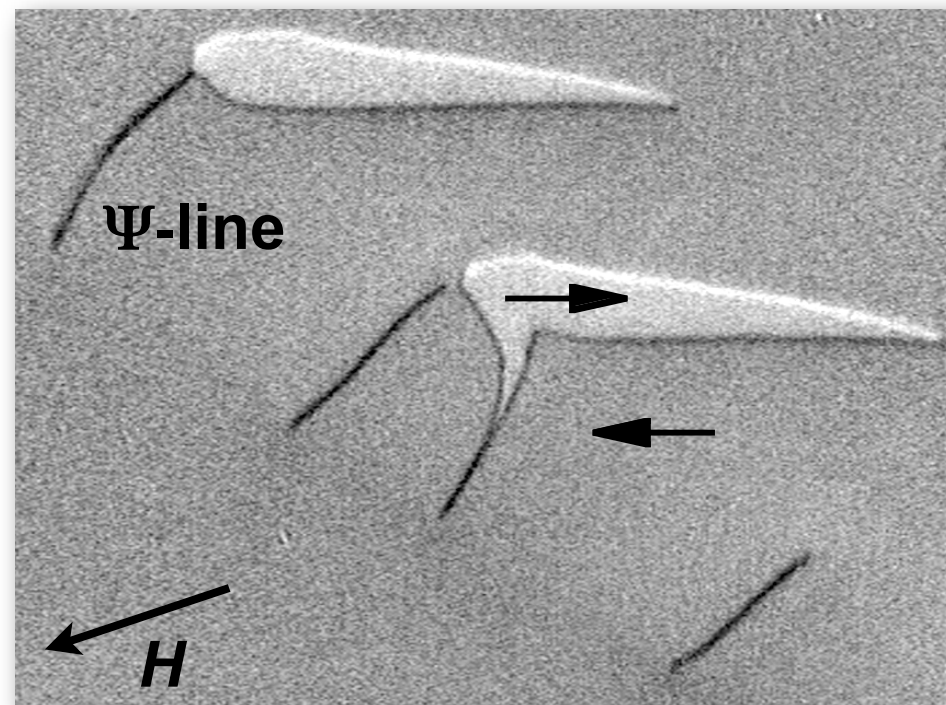
Visible and invisible features



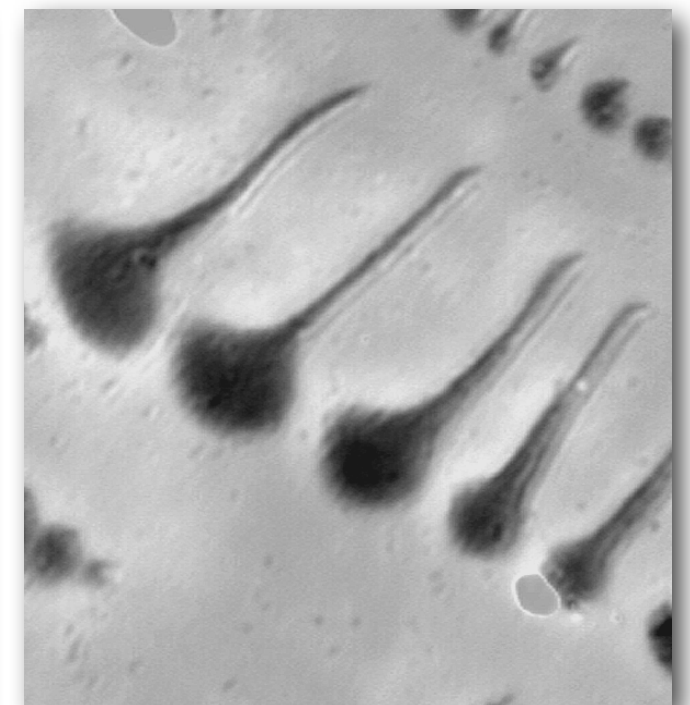
Ψ -lines



Bitter image



Kerr image

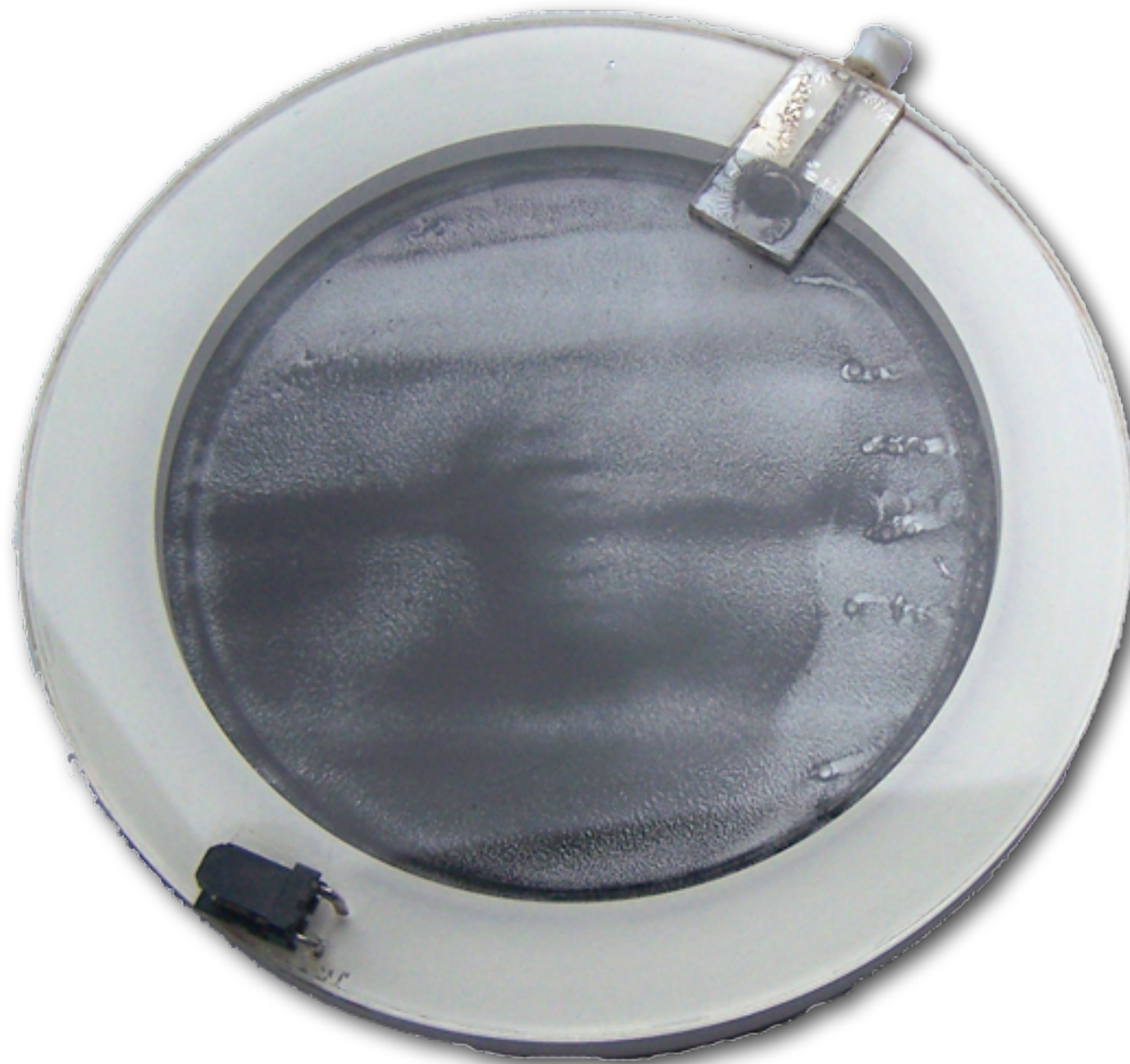


Bitter image

1. Bitter technique

Toner powder emulsion

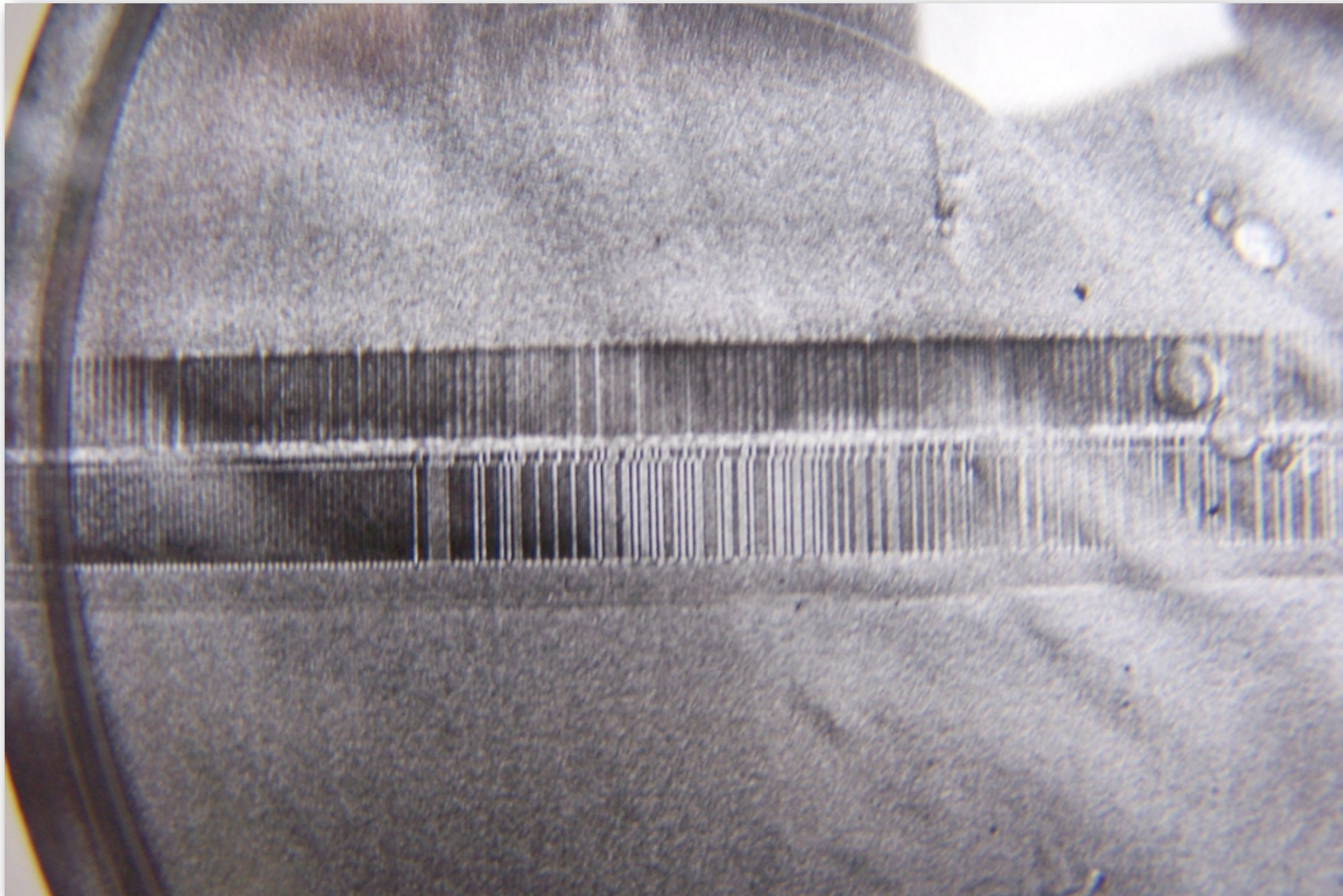
Laser printer toner + water + household detergent



1. Bitter technique

Toner powder emulsion

Laser printer toner + water + household detergent

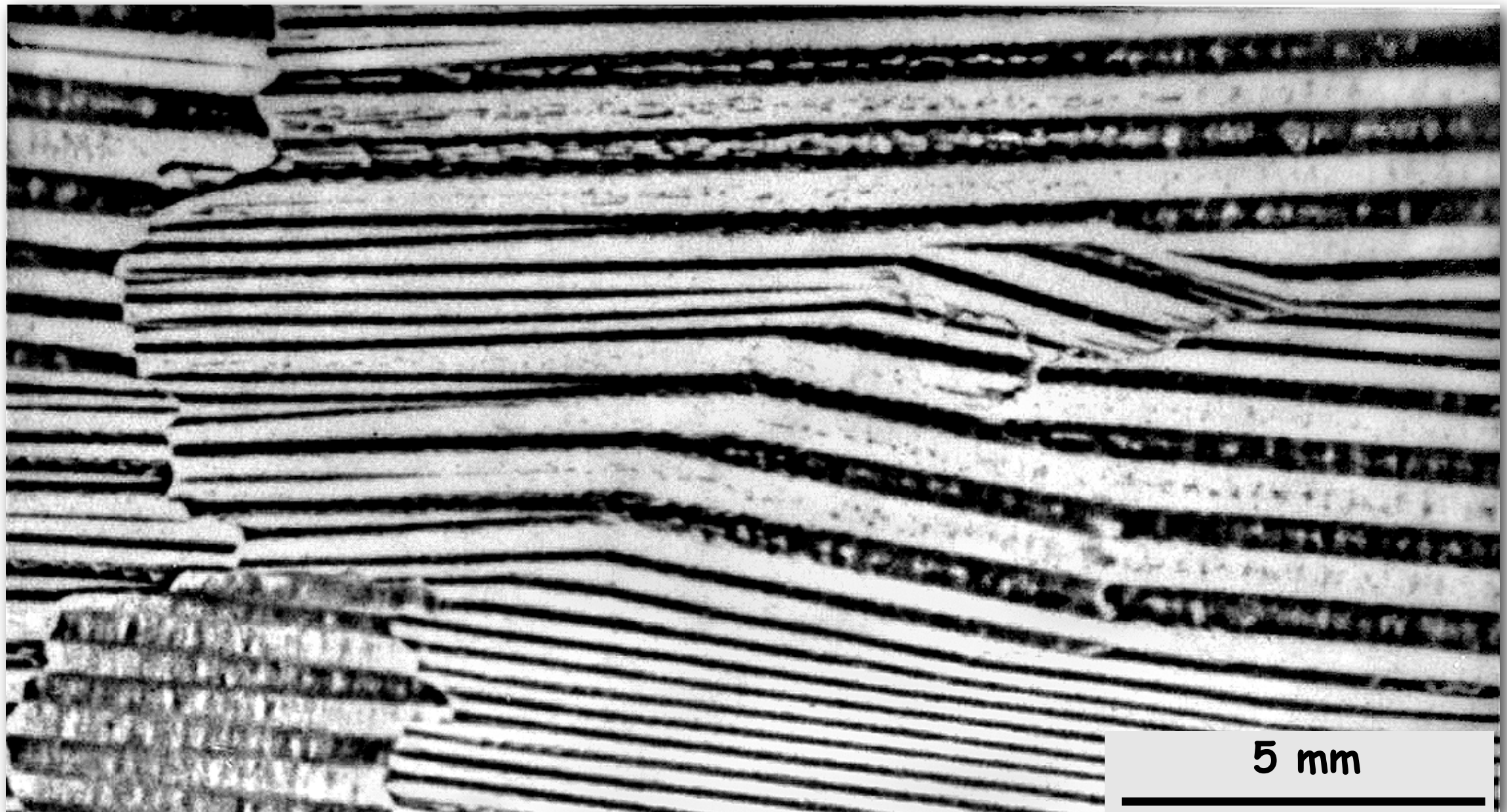


1. Bitter technique

Toner powder emulsion

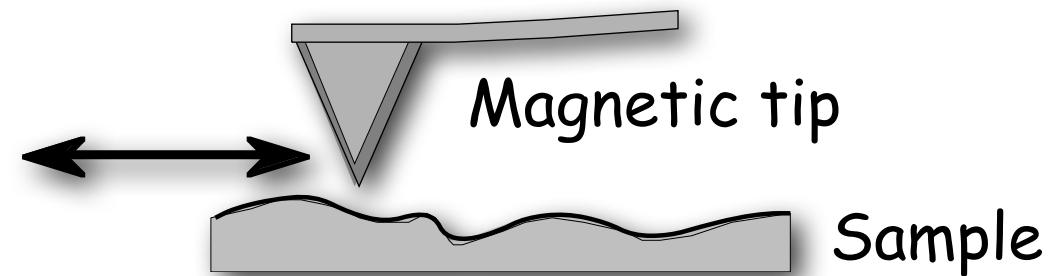
Laser printer toner + water + household detergent

Transformer steel (courtesy S. Arai, Nippon steel)



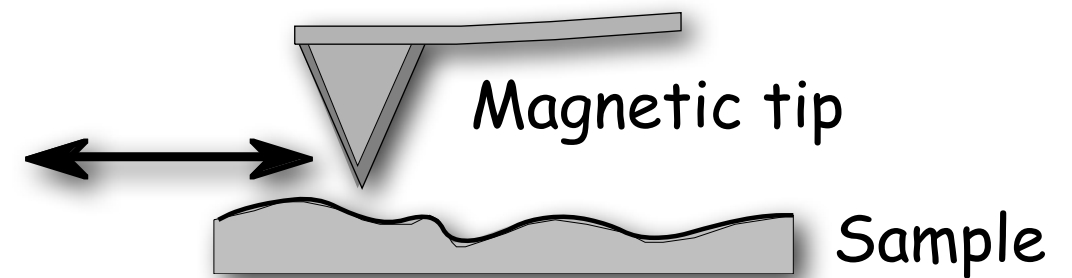
2. Magnetic Force Microscopy

- Spin-off of scanning tunnelling microscope
- Tip-shaped probe at free end of cantilever (= flexible beam). Tip position detected (e.g.) by optical interference between tip of a fibre and cantilever
- 2 modes:
 - Static mode: MFM is run at constant force (equivalent to constant deflection of the cantilever) and the height necessary to obtain this state is used as the imaging information
 - Dynamic mode: Cantilever is operated at frequency close to its mechanical resonance, and change in resonance amplitude or shift in phase due to stray field interaction are detected. Since a magnetic force gradient is equivalent to an additional contribution to the spring constant of the cantilever, profiles of constant force gradient are recorded this way

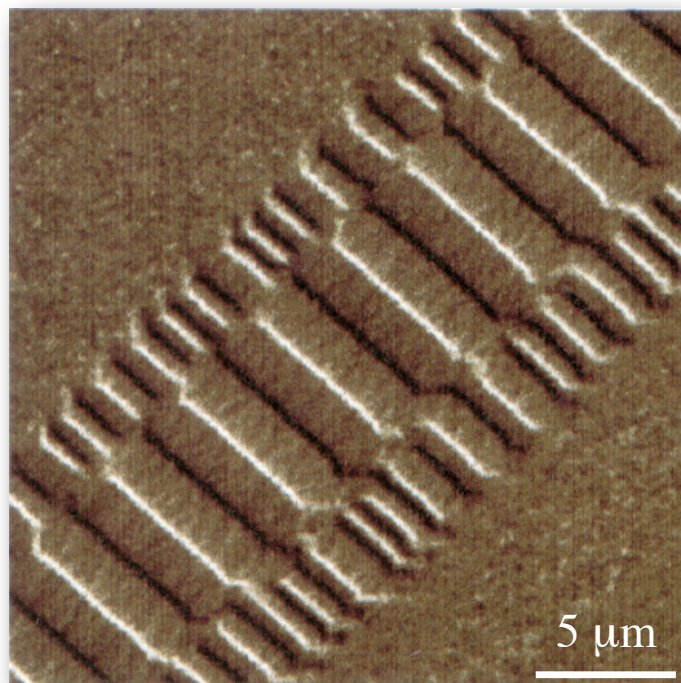


2. Magnetic Force Microscopy

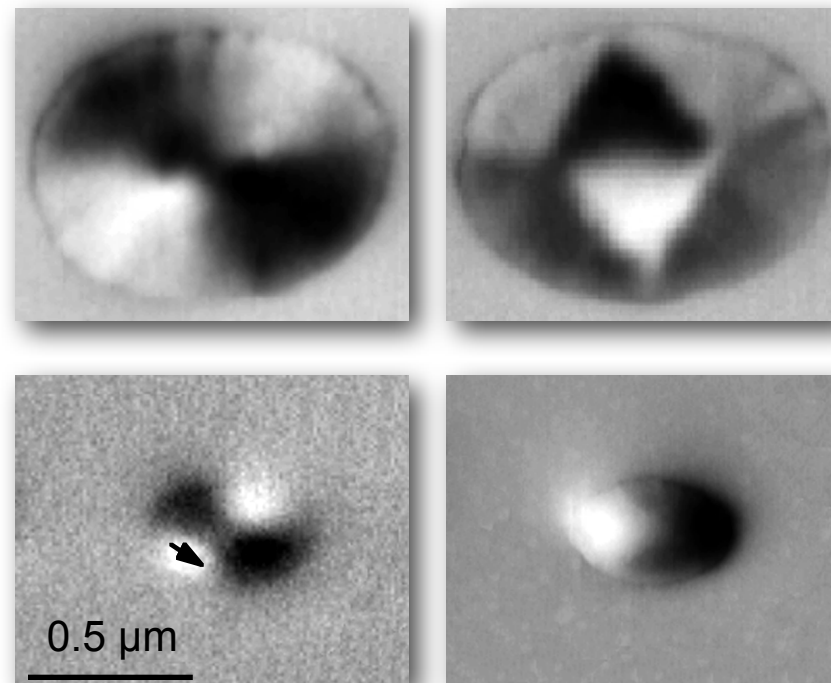
- Spin-off of scanning tunnelling microscope
- Tip-shaped probe at free end of cantilever (= flexible beam). Tip position detected (e.g.) by optical interference between tip of a fibre and cantilever
- 2 modes:
 - Static mode: MFM is run at constant force (equivalent to constant deflection of the cantilever) and the height necessary to obtain this state is used as the



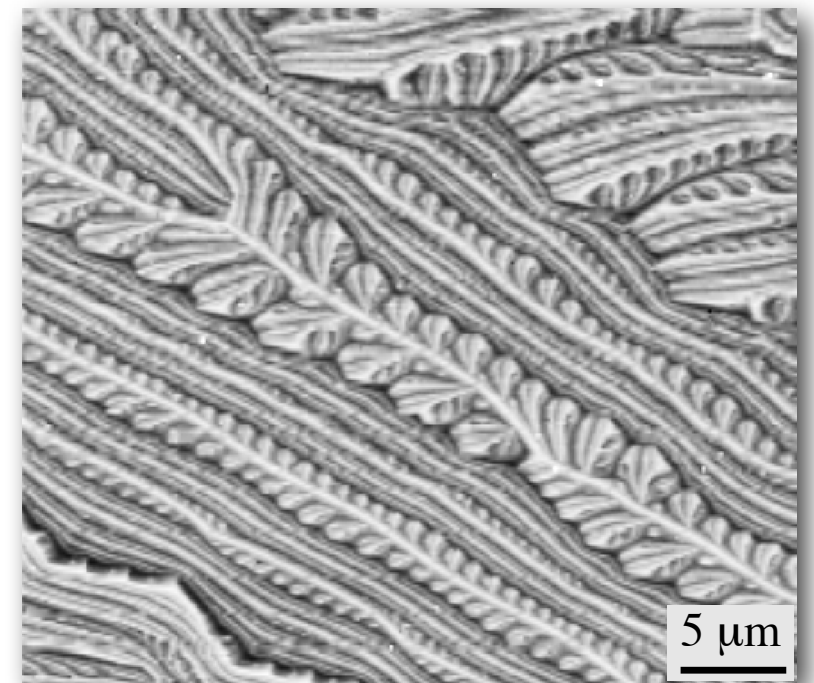
Data track on hard disk



Co elements



(111) Fe surface



Courtesy: A. Fernandez

Courtesy: J. Miltat

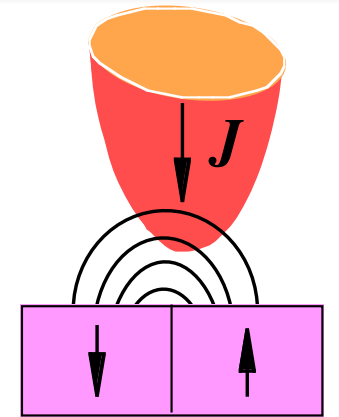
2. Magnetic Force Microscopy

Conventional interpretation of MFM: $F = -\partial E_{\text{inter}} / \partial z$; $\partial F / \partial z = -\partial E_{\text{inter}}^2 / \partial z^2$

$$E_{\text{inter}} = - \int_{\text{tip}} \mathbf{J}_{\text{tip}} \cdot \mathbf{H}_{\text{sample}} dV = - \int_{\text{sample}} \mathbf{J}_{\text{sample}} \cdot \mathbf{H}_{\text{tip}} dV$$

$\mathbf{H}_{\text{tip}} = -\text{grad } \Phi_{\text{tip}}$, partial integration

$\Phi_{\text{tip}} =$ tip potential



tip magnetization =
stray field sensor

Alternative interpretation *Hubert, Rave, Tomlinson: Phys. Stat. Sol. B204, 817 (1997)*

$$E_{\text{inter}} = \int_{\text{surface}} \sigma_{\text{sample}} \Phi_{\text{tip}} dS + \int_{\text{sample}} \rho_{\text{sample}} \Phi_{\text{tip}} dV$$

$\sigma_{\text{sample}} = \mathbf{n} \cdot \mathbf{J}_{\text{sample}}$ (surface charge)

$\rho_{\text{sample}} = -\text{div } \mathbf{J}_{\text{sample}}$ (volume charge)

Force

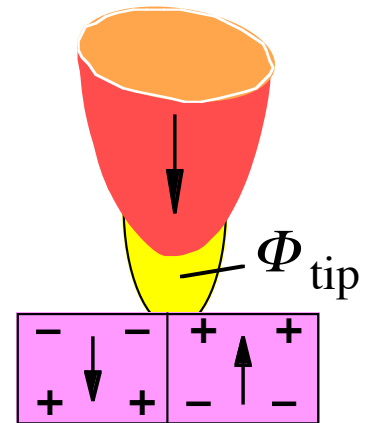
$$F = -\frac{\partial E_{\text{inter}}}{\partial z} = - \int \left(\cancel{\frac{\partial \sigma}{\partial z}} \Phi + \sigma \frac{\partial \Phi}{\partial z} \right) dS - \int \left(\cancel{\frac{\partial \rho}{\partial z}} \Phi + \rho \frac{\partial \Phi}{\partial z} \right) dV$$

for weak interaction

$$\approx - \int \sigma \frac{\partial \Phi}{\partial z} dS - \int \rho \frac{\partial \Phi}{\partial z} dV$$

Force gradient

$$\frac{\partial F}{\partial z} = -\frac{\partial E_{\text{inter}}^2}{\partial z^2} \approx - \int \sigma \frac{\partial^2 \Phi}{\partial z^2} dS - \int \rho \frac{\partial^2 \Phi}{\partial z^2} dV$$



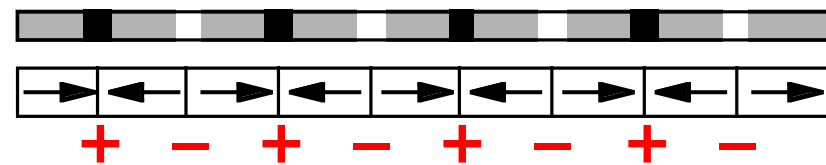
tip potential =
charge sensor

In the limit of weak interaction: MFM is *Charge Microscopy*

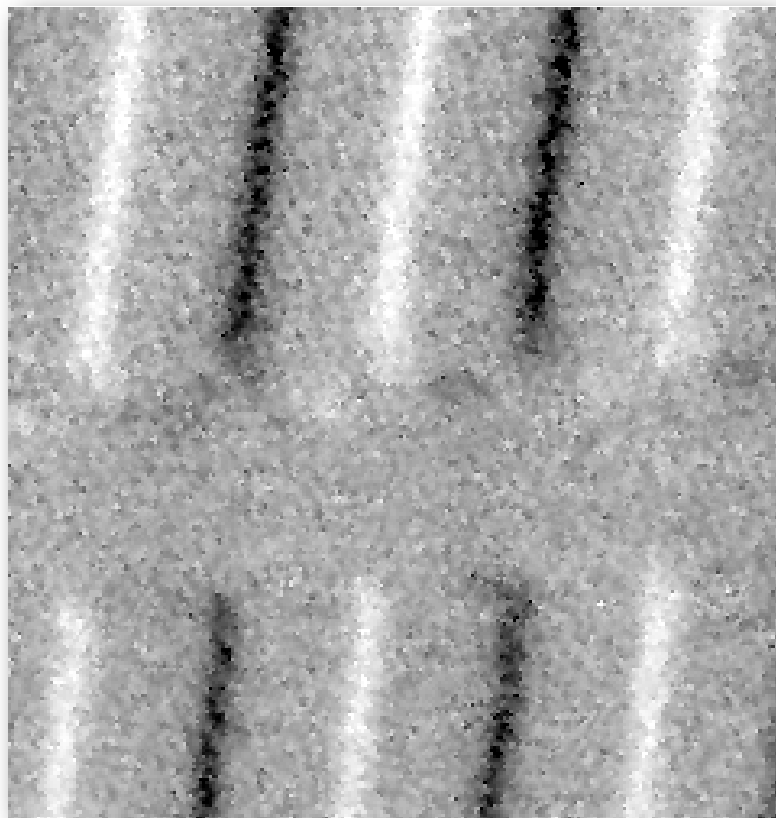
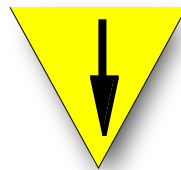
2. Magnetic Force Microscopy

Charge contrast

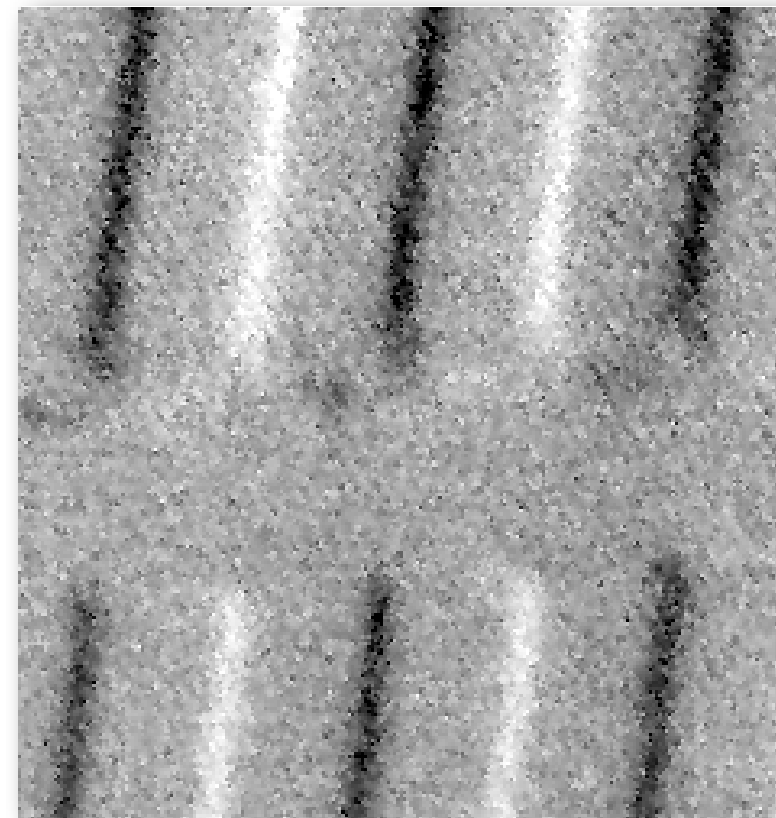
Longitudinal data track



Tip down



Tip up

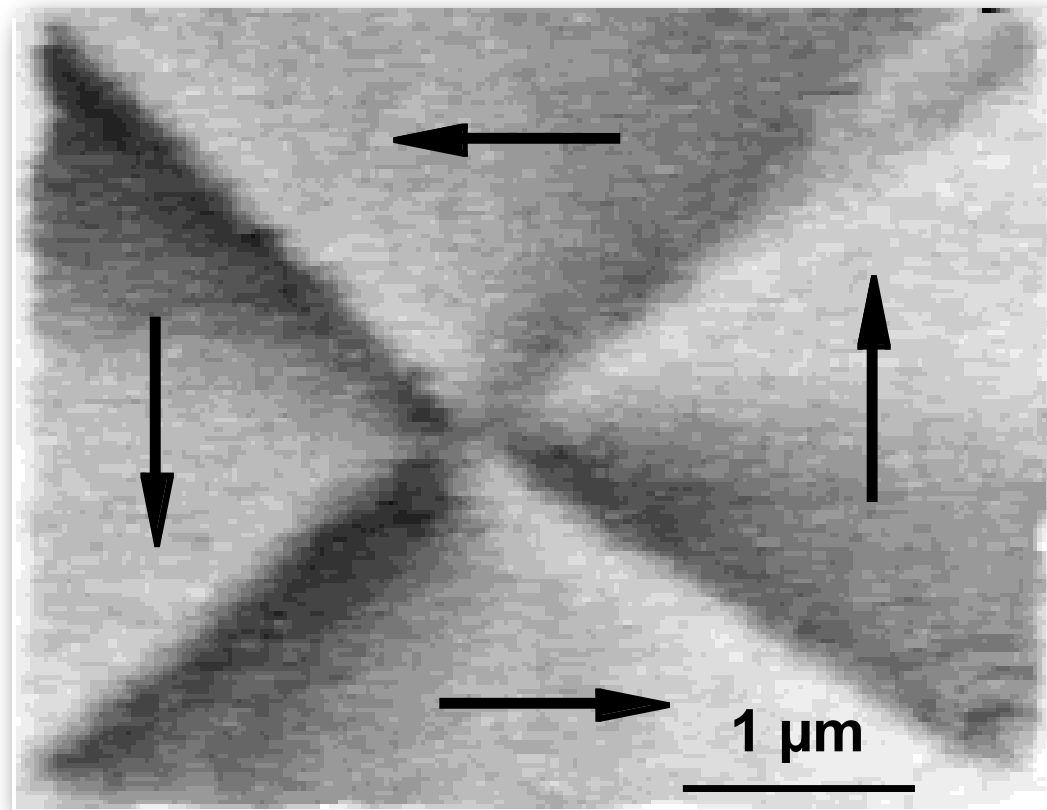


10 μm

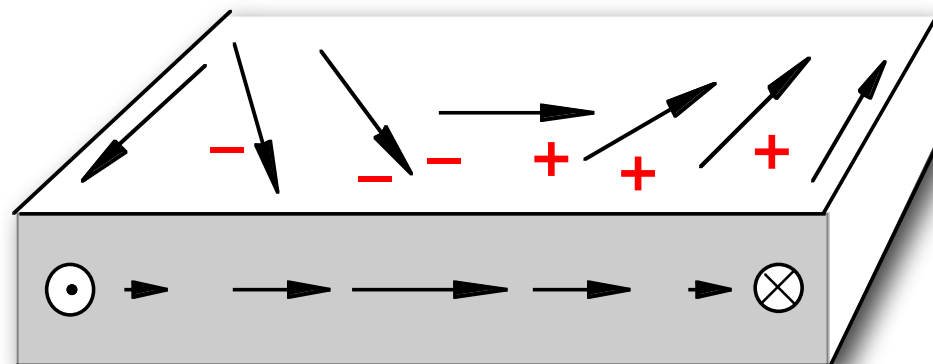
2. Magnetic Force Microscopy

Charge contrast

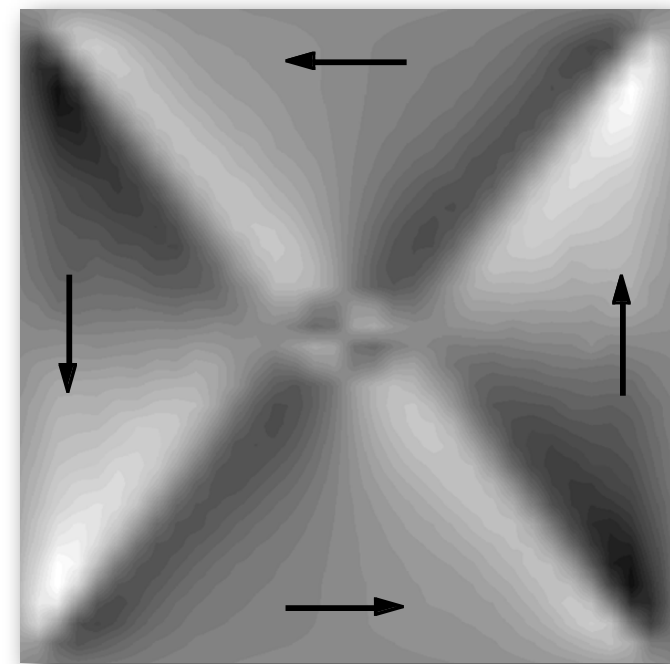
MFM image



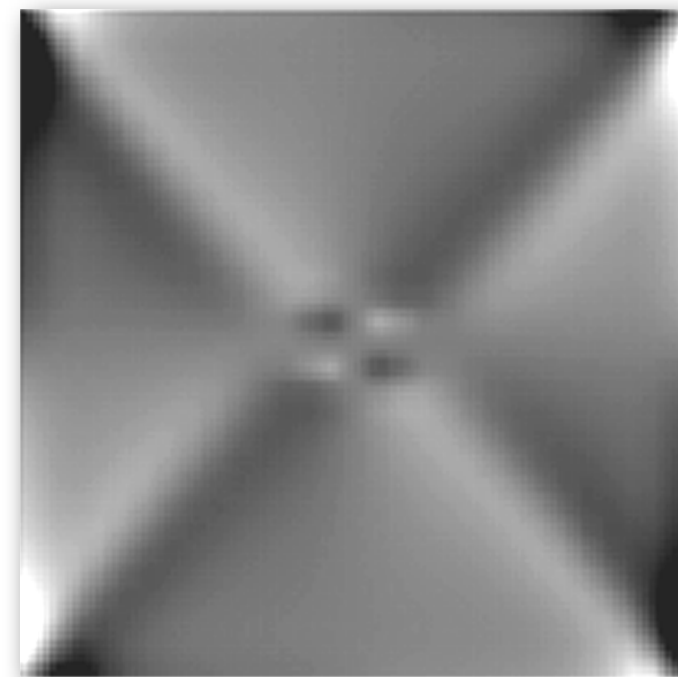
FeTnN element (30 nm)
(courtesy J. Miltat)



Simulated
MFM image



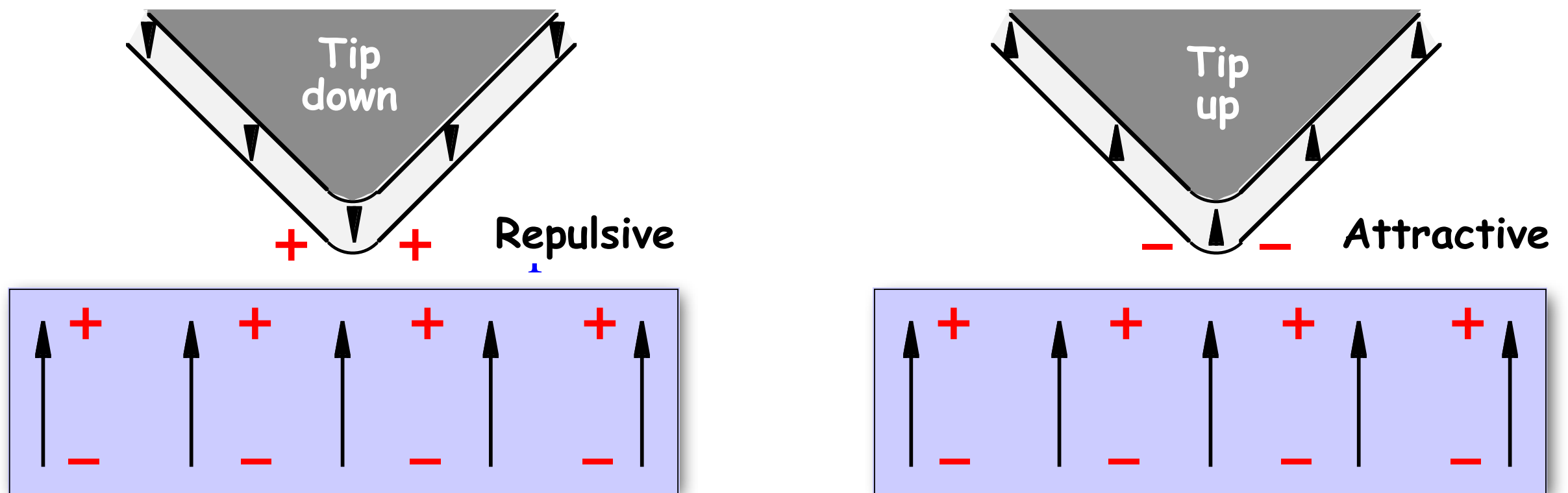
Simulated
charge
distribution



A. Hubert, W. Rave, S. Tomlinson:
Phys. Stat. Sol. B 204, 817 (1997)

2. Magnetic Force Microscopy

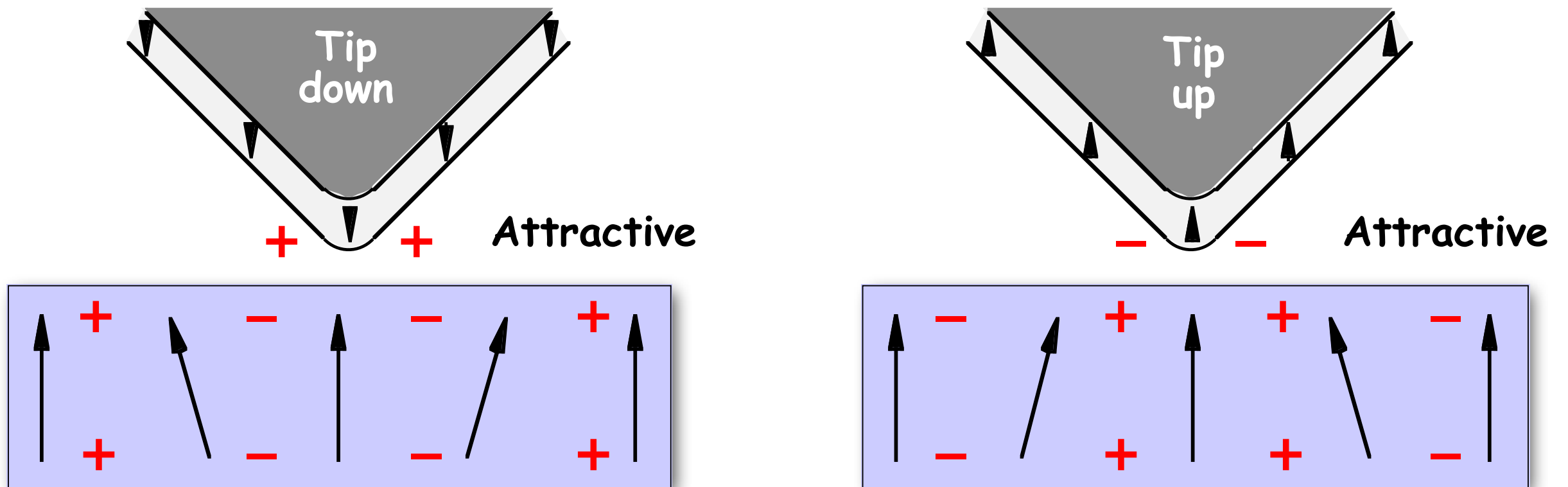
Charge contrast



Charge contrast is inverted when tip polarity is inverted

2. Magnetic Force Microscopy

Induced charges: susceptibility contrast

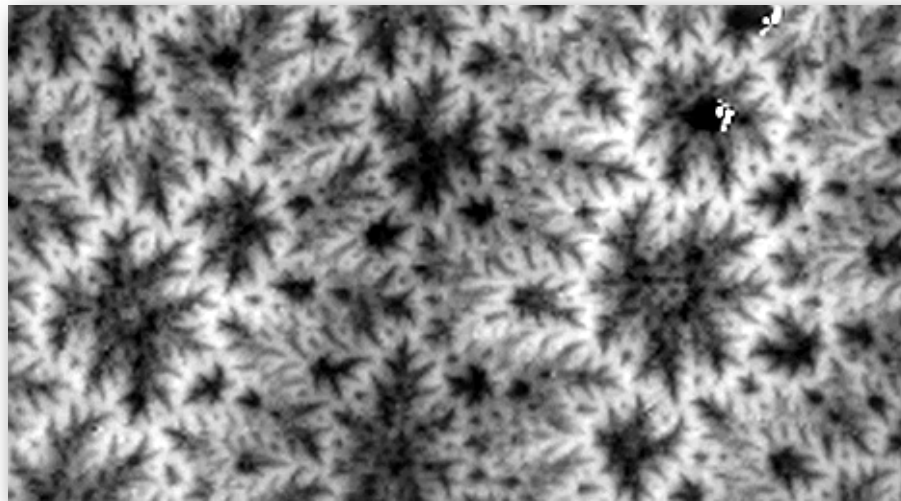
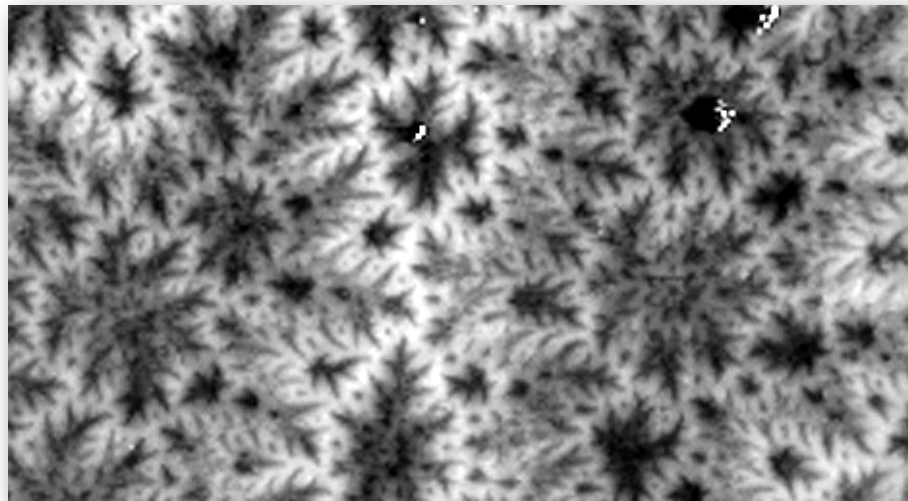


- Tip induces charges of opposite polarity in each case
- Always attractive interaction (independent of tip polarity)
- Strength of attraction depends on local susceptibility

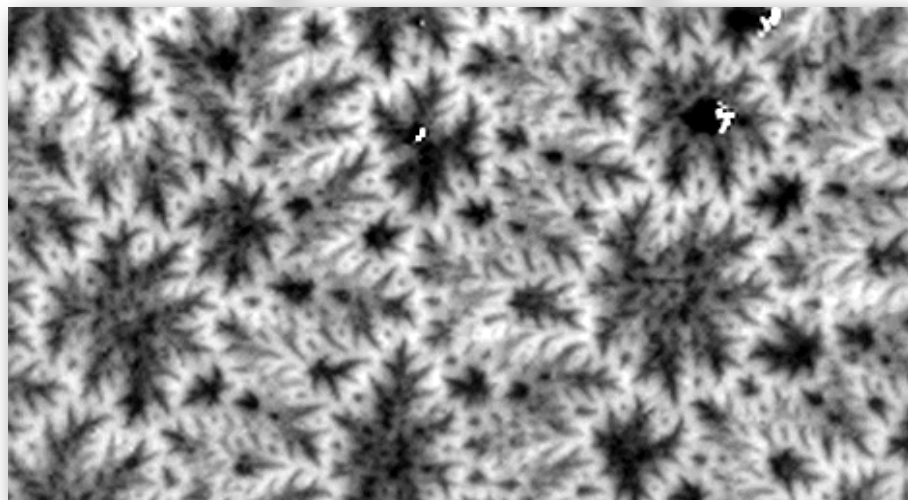
2. Magnetic Force Microscopy

Charge & susceptibility contrast

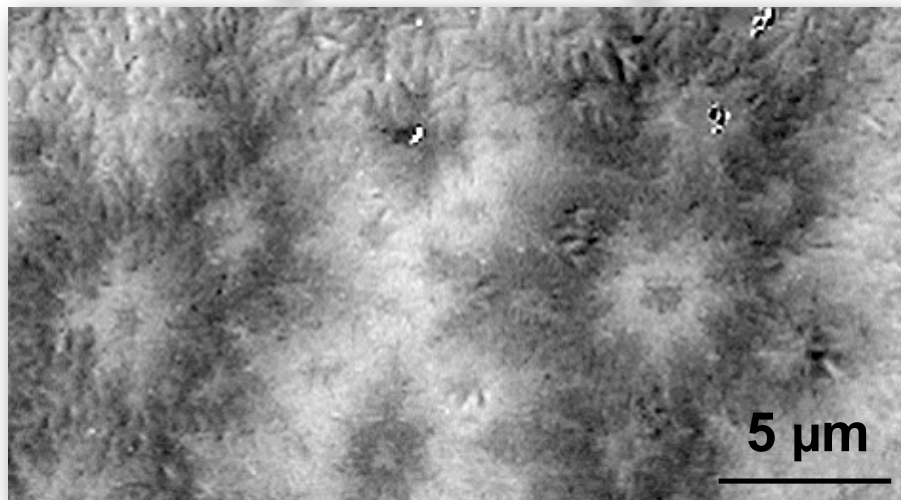
- Charge contrast is inverted by inversion of tip magnetization,
- Susceptibility contrast: not inverted
- → Separation of charge and susceptibility contrast by difference and sum images



+



-

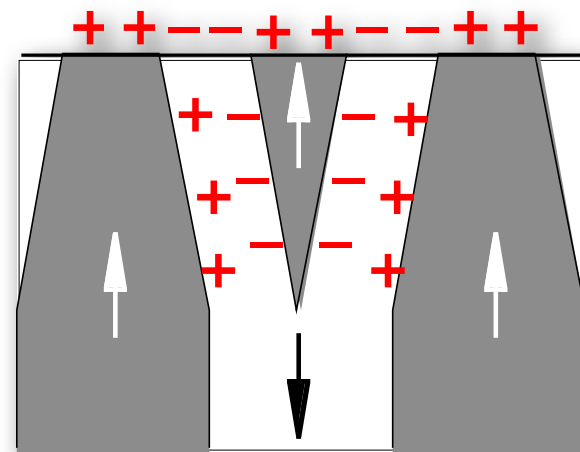


Co crystal

2. Magnetic Force Microscopy

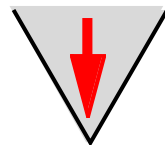
Charge & susceptibility contrast

E. Zueco et al.:
JMMM 190, 42 (1998)

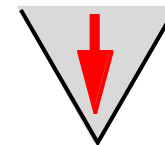
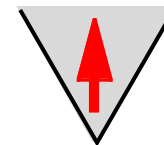


Stronger attraction
Weaker attraction

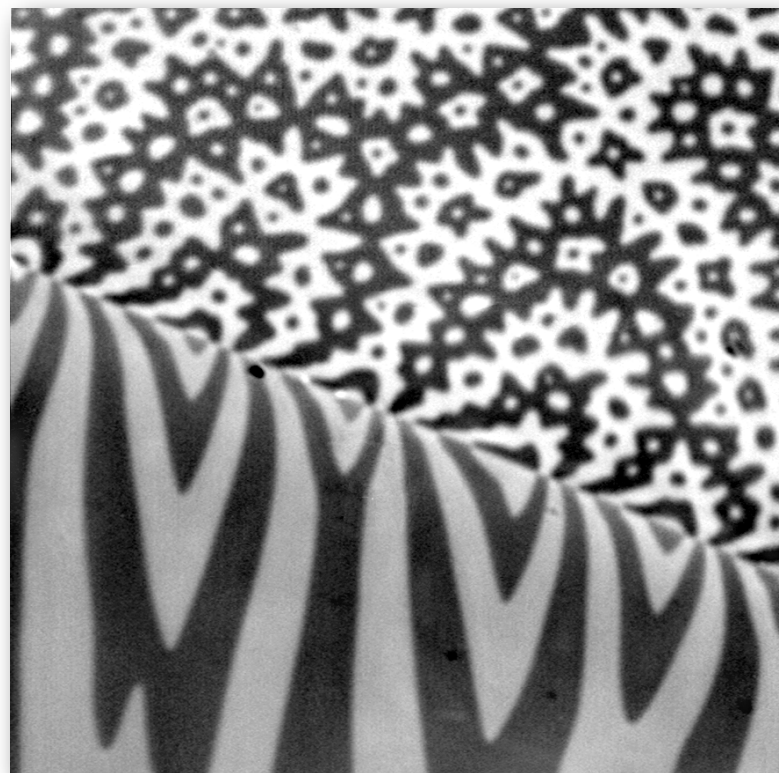
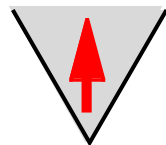
NdFeB twin boundary



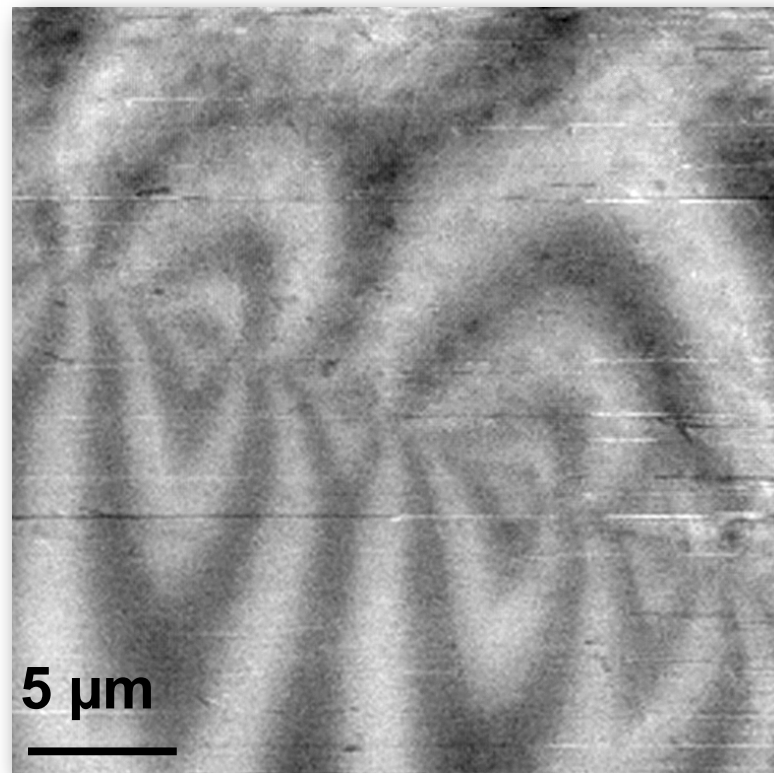
—



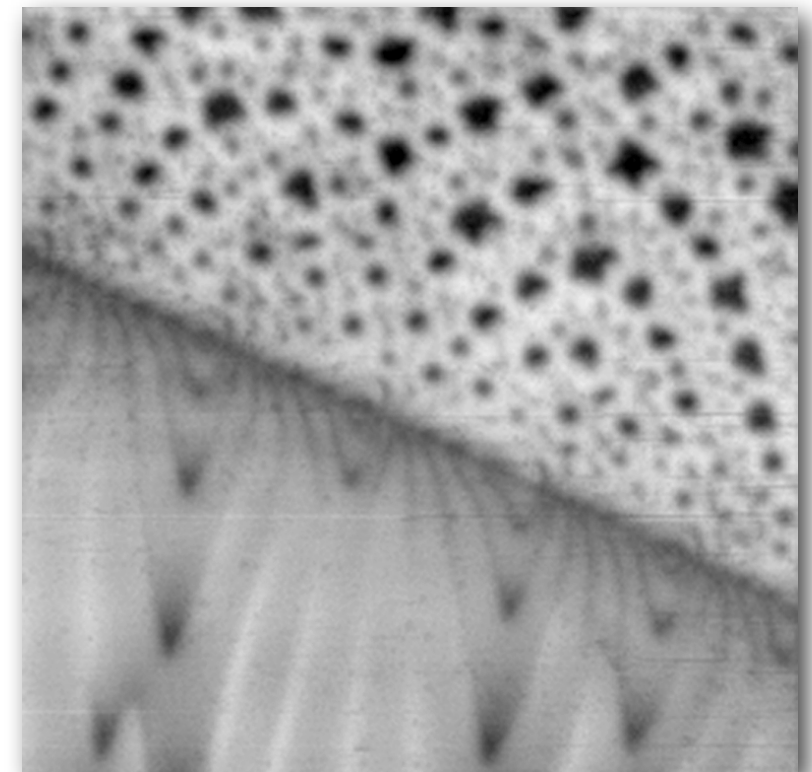
+



Kerr image



MFM: charge contrast

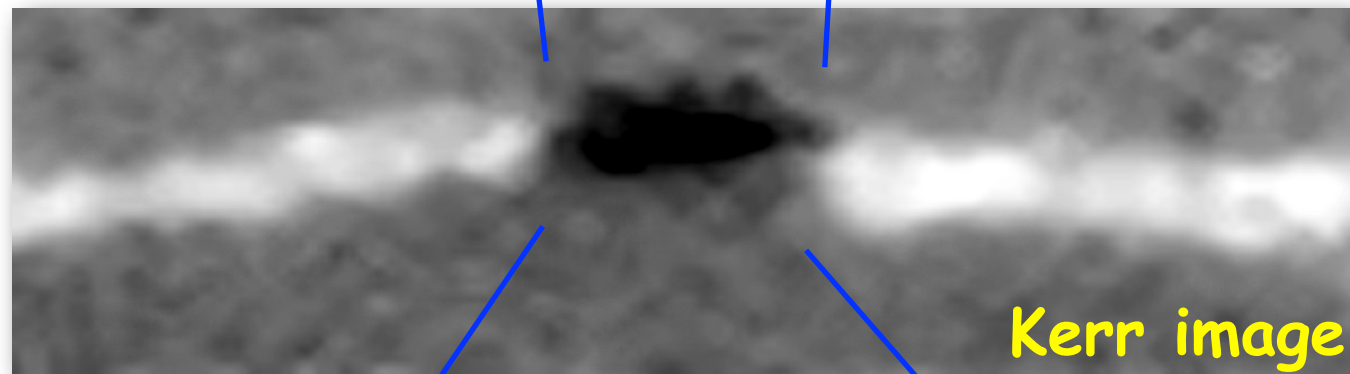
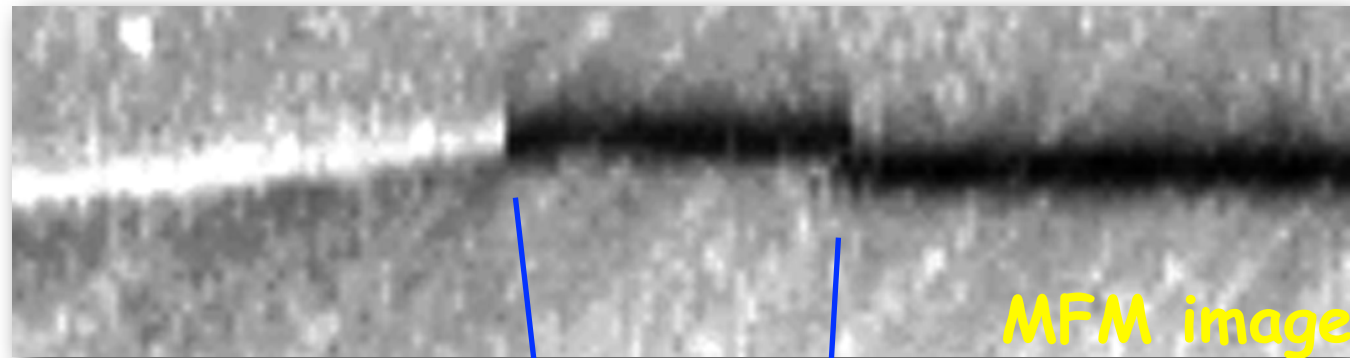


Susceptibility contrast

2. Magnetic Force Microscopy

Depth sensitivity

Fe whisker

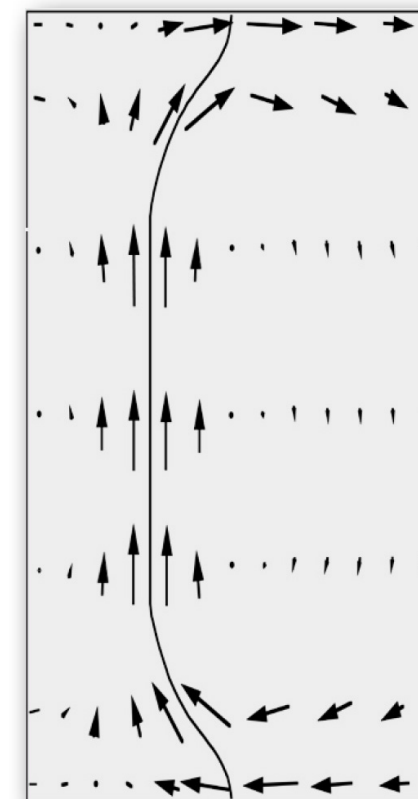
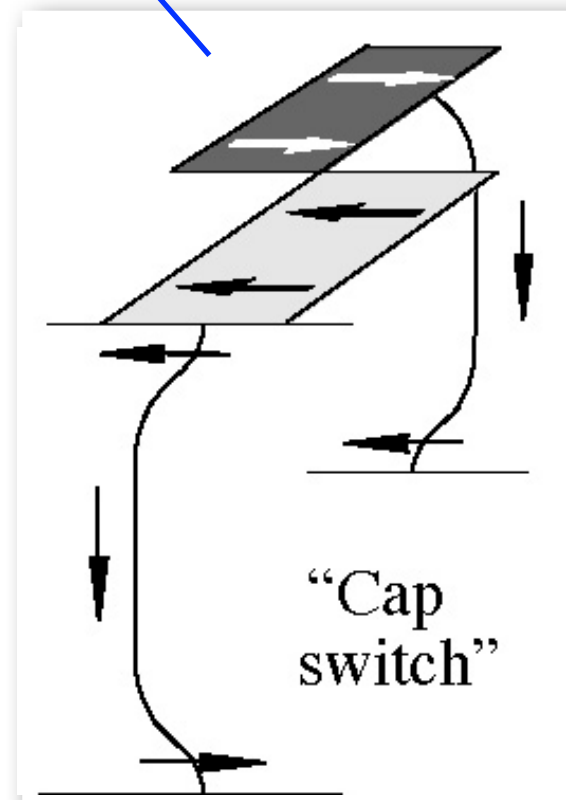
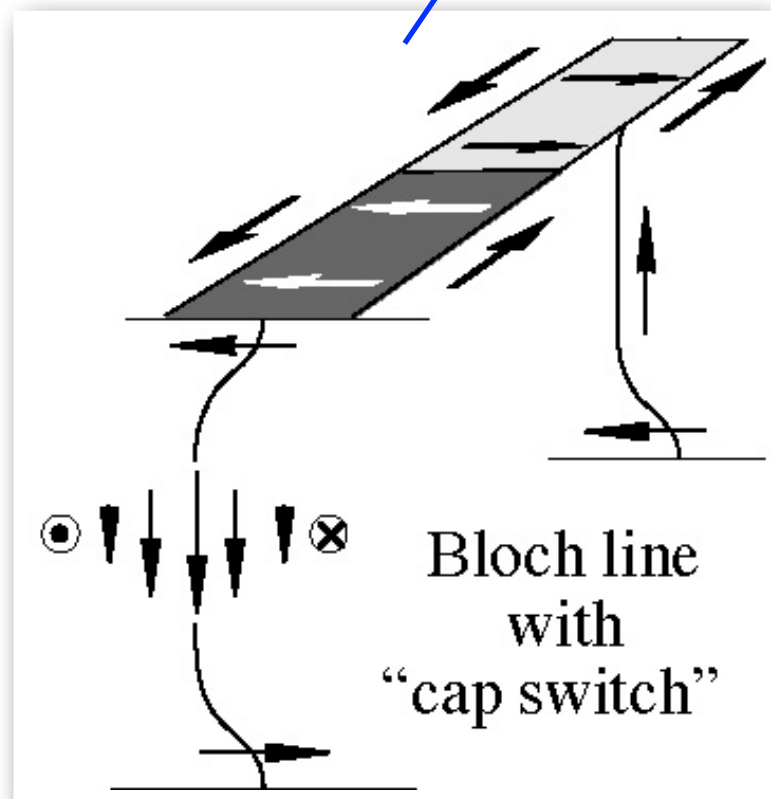


MFM:

sensitive to interior magnetization of the wall

Kerr:

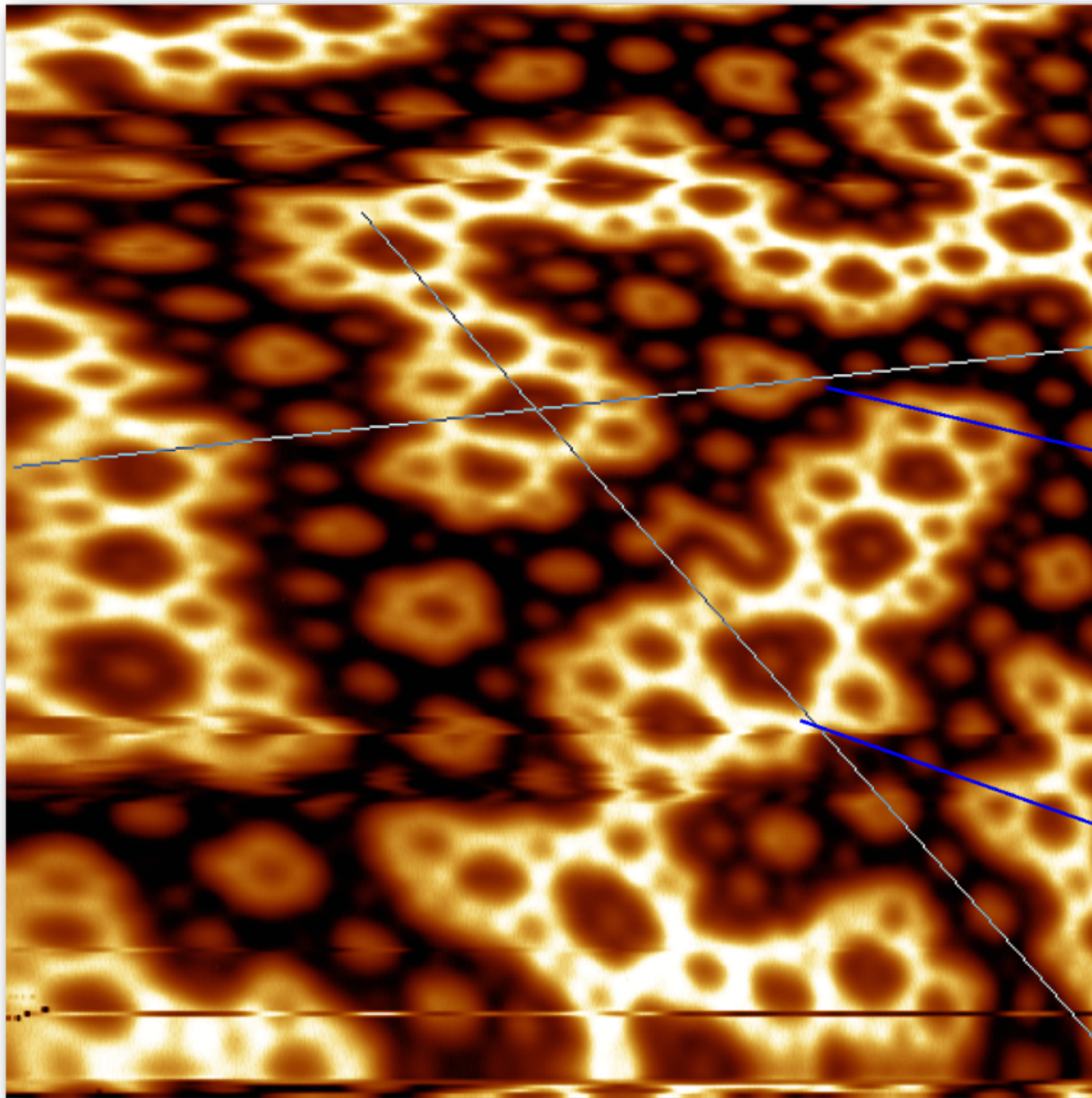
sensitive to surface magnetization



E. Zueco et al.,
JMMM 196, 115 (1999)

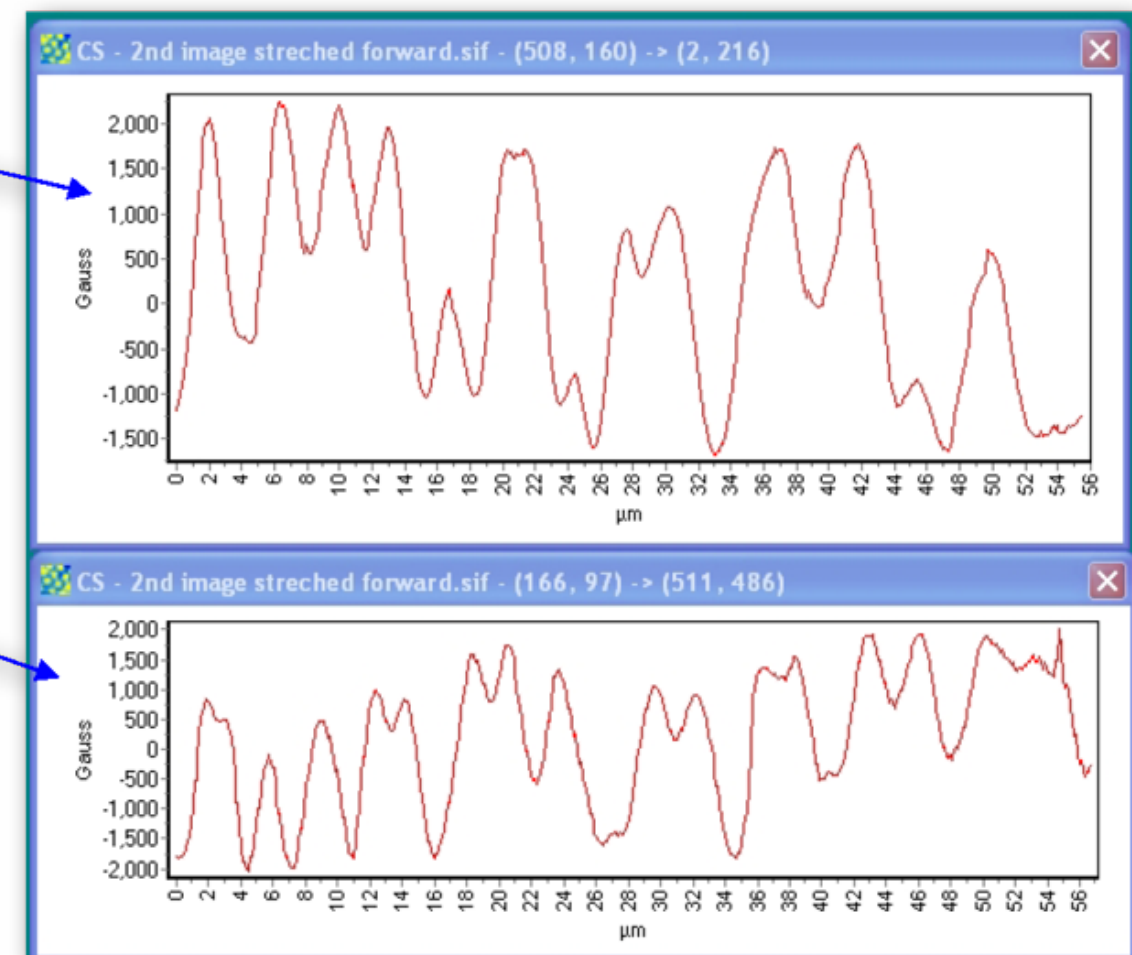
3. Hall-Probe Microscopy

56 μm



NdFeB crystal

Micro-Hallprobe
is scanned across surface



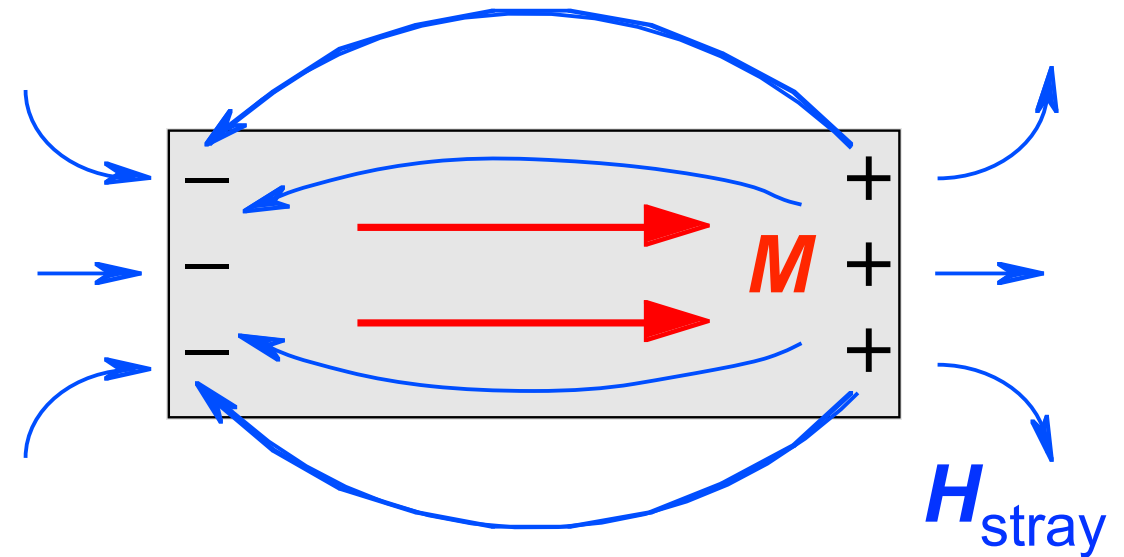
Together with
J. McCord and U. Wolff, IFW

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

$$\text{div } \mathbf{B} = 0$$

$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



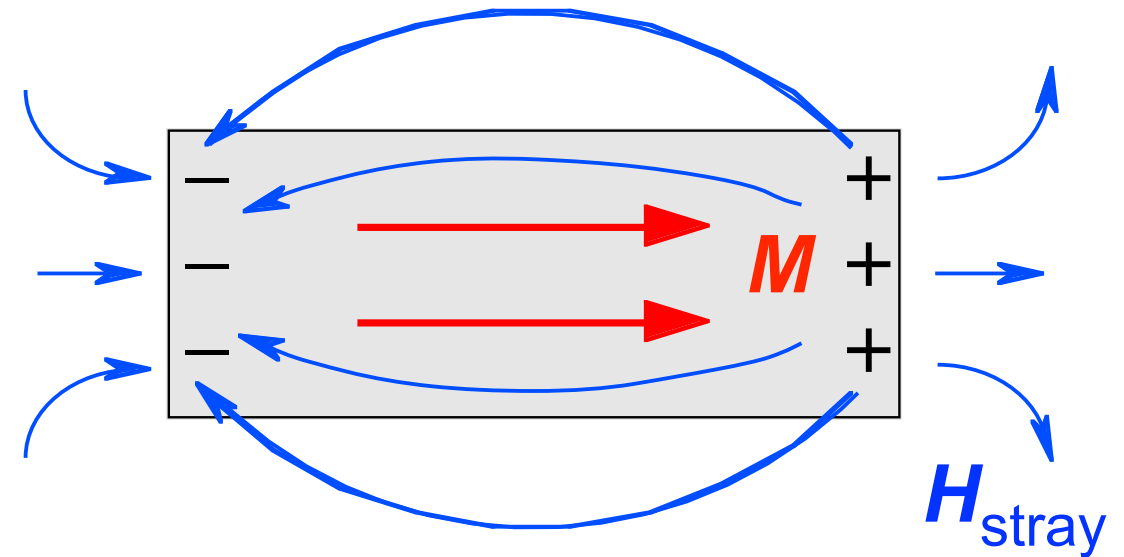
- Sensitive to $\mathbf{H}_{\text{stray}}$
 1. Bitter technique
 2. Magnetic force microscopy
 3. Hall probe microscopy
- Sensitive to \mathbf{M}
 - Magneto-optical microscopy
 - X-ray spectroscopy
 - Polarized electrons (SEMPA, SPT)
- Sensitive to \mathbf{B}
 - Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

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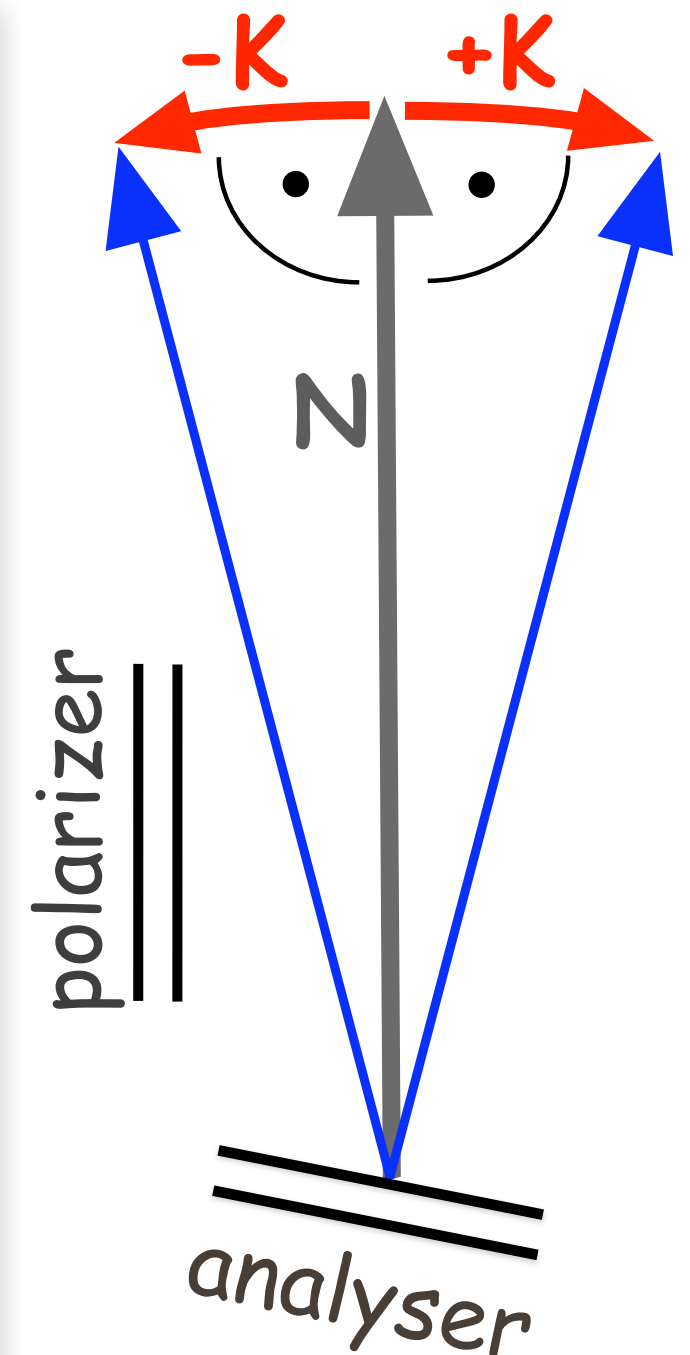
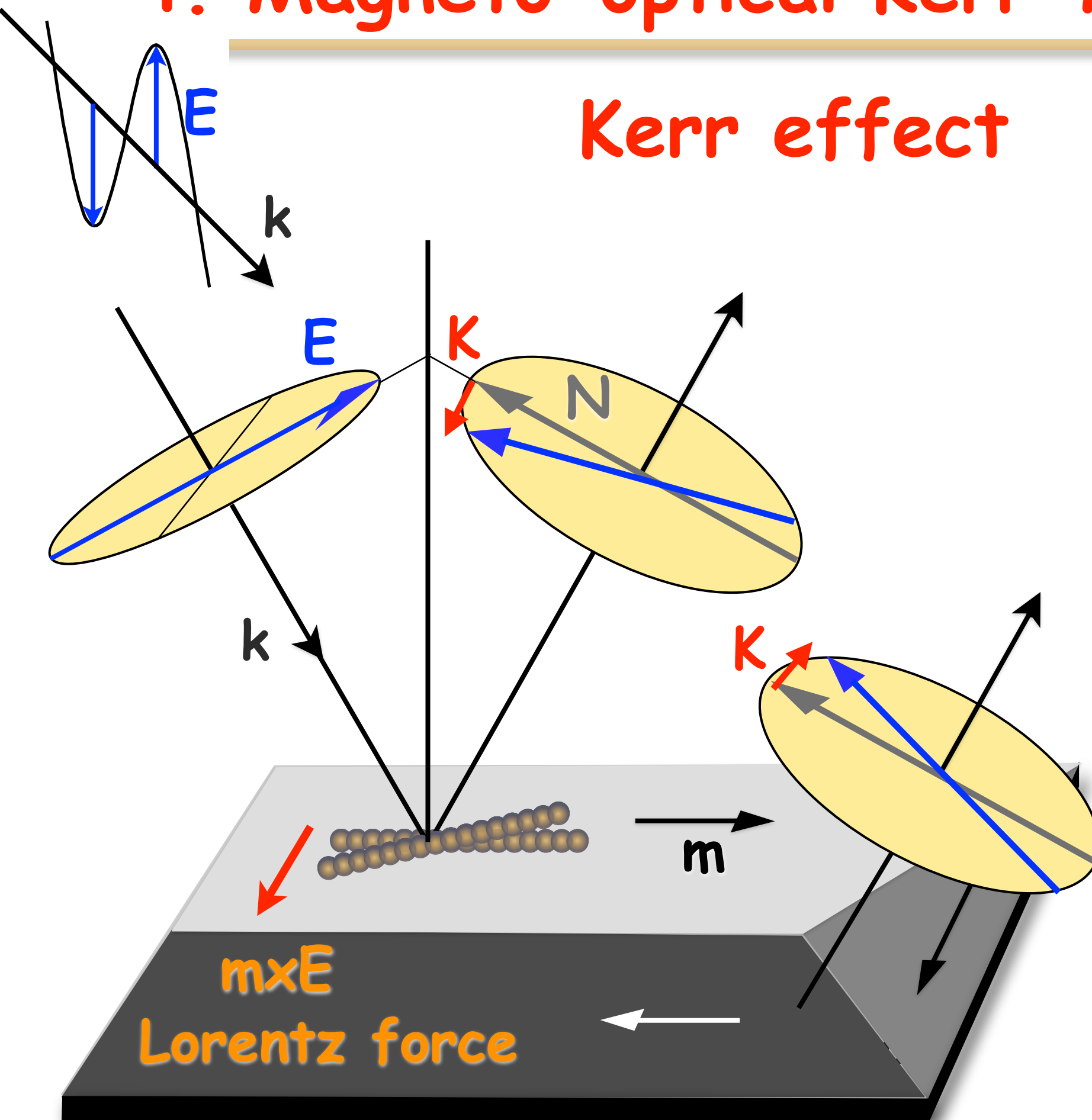
$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



- Sensitive to $\mathbf{H}_{\text{stray}}$
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 2. Magnetic force microscopy
 3. Hall probe microscopy
- Sensitive to \mathbf{M}
 4. Magneto-optical microscopy
 5. X-ray spectroscopy
 6. Polarized electrons (SEMPA, SPT)
- Sensitive to \mathbf{B}
 - Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

4. Magneto-optical Kerr-Microscopy

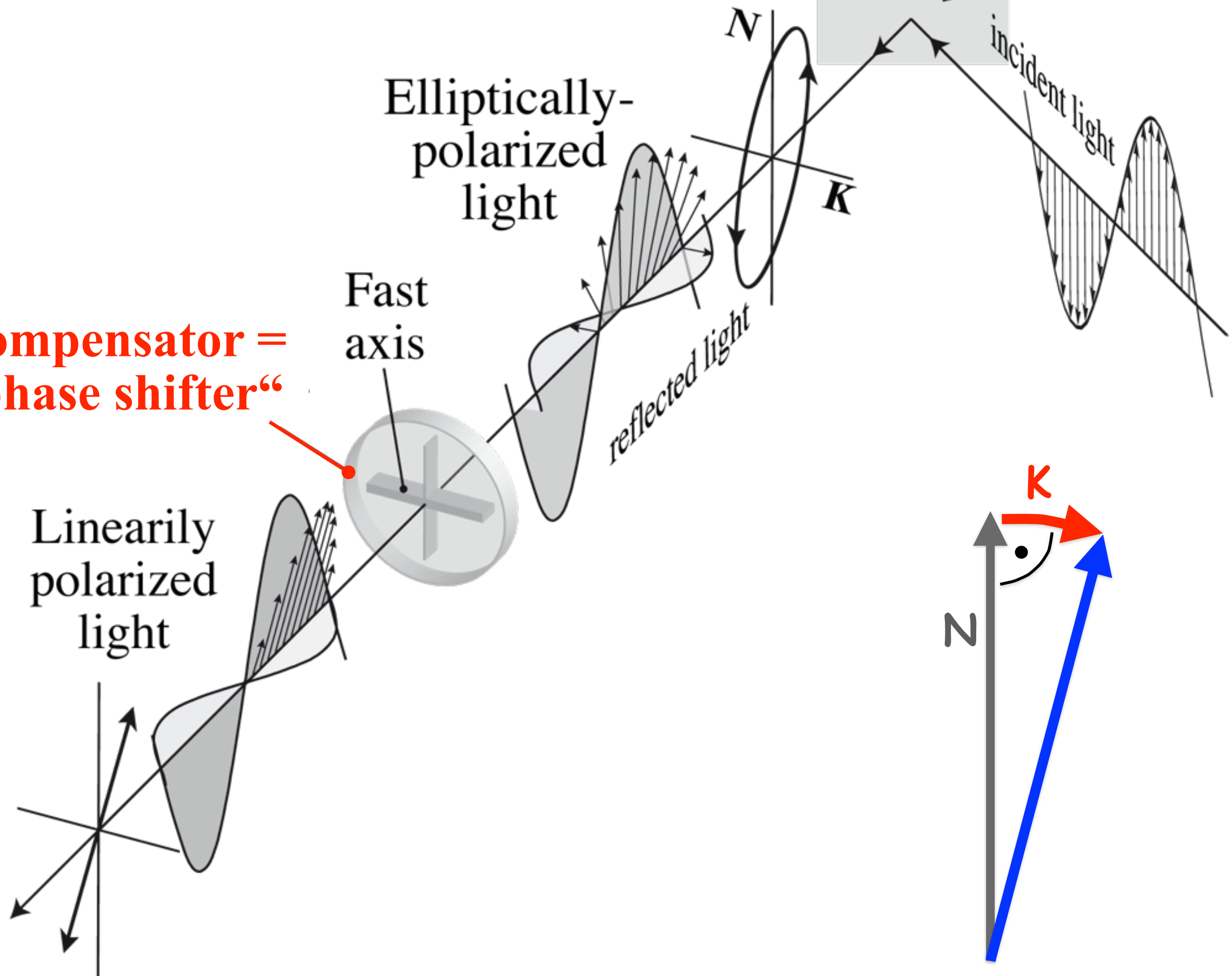
Kerr effect



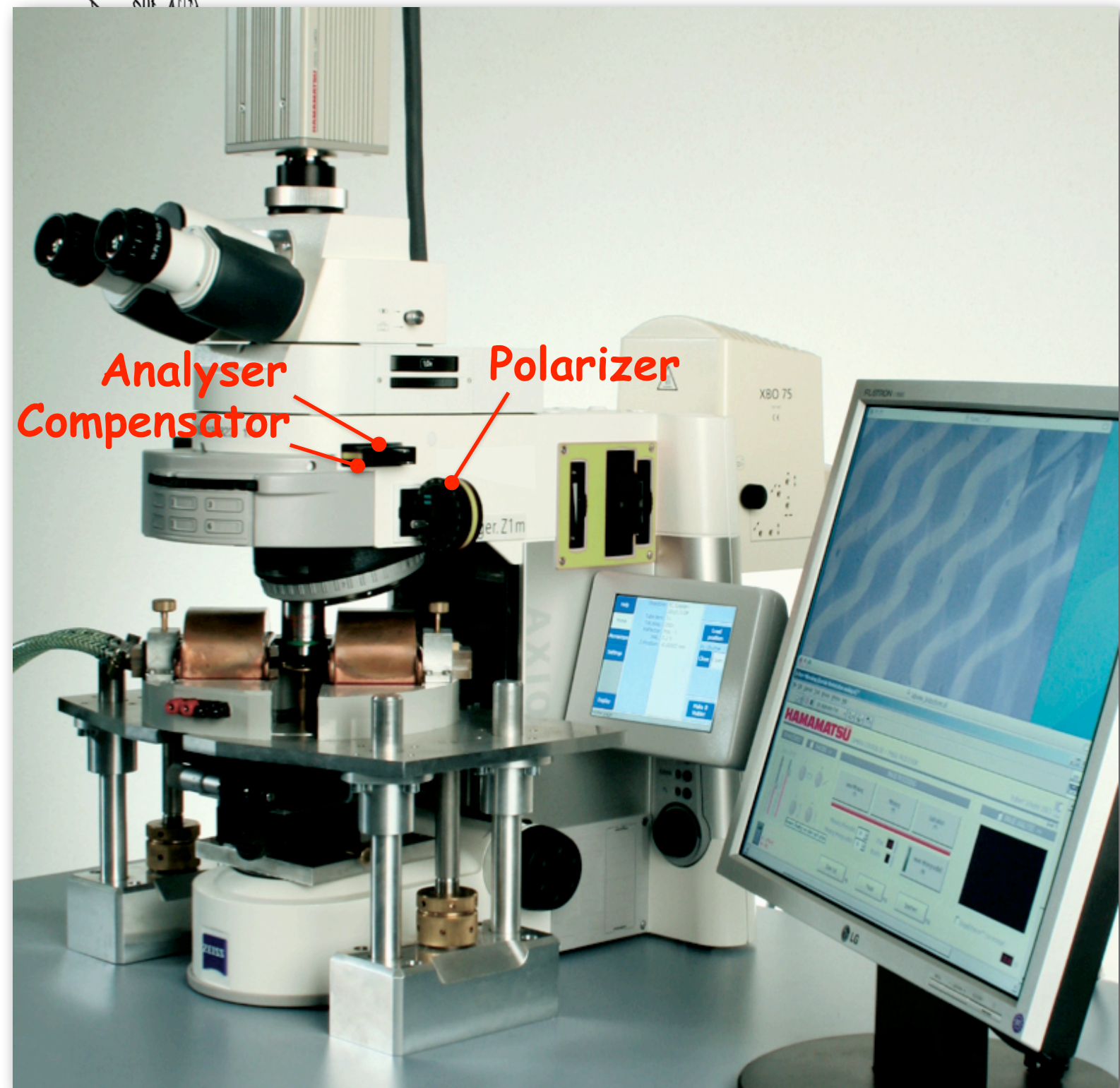
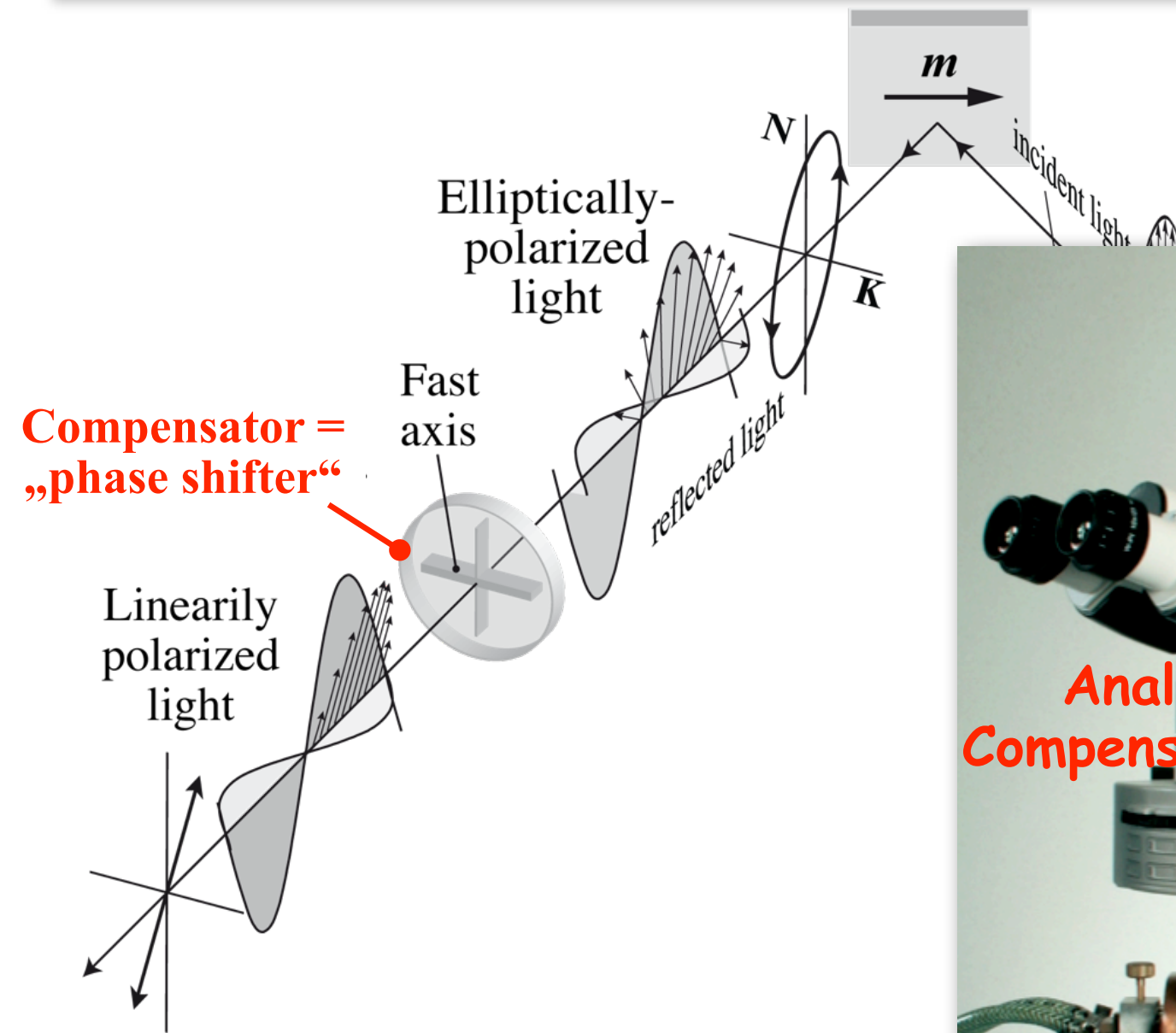
Kerr rotation
 $= K/N$

4. Magneto-optical Kerr-Microscopy

Compensator = „phase shifter“

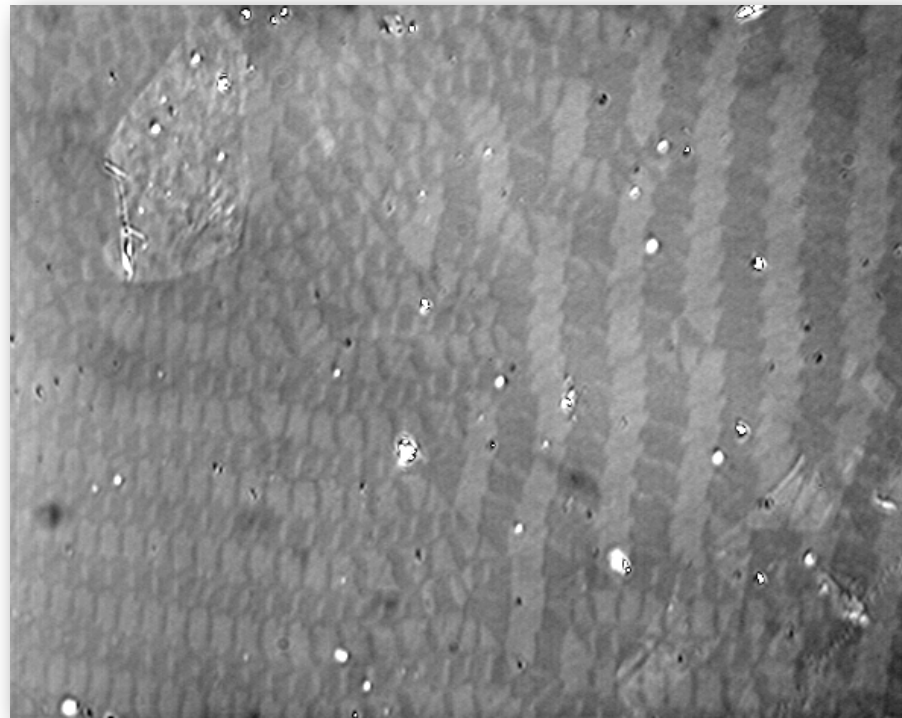


4. Magneto-optical Kerr-Microscopy

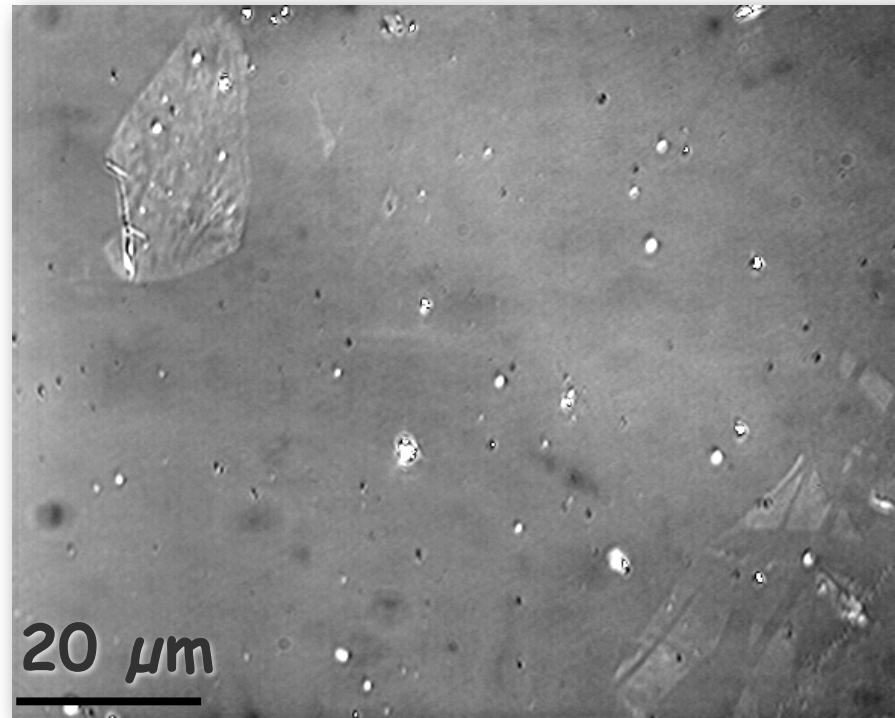


4. Magneto-optical Kerr-Microscopy

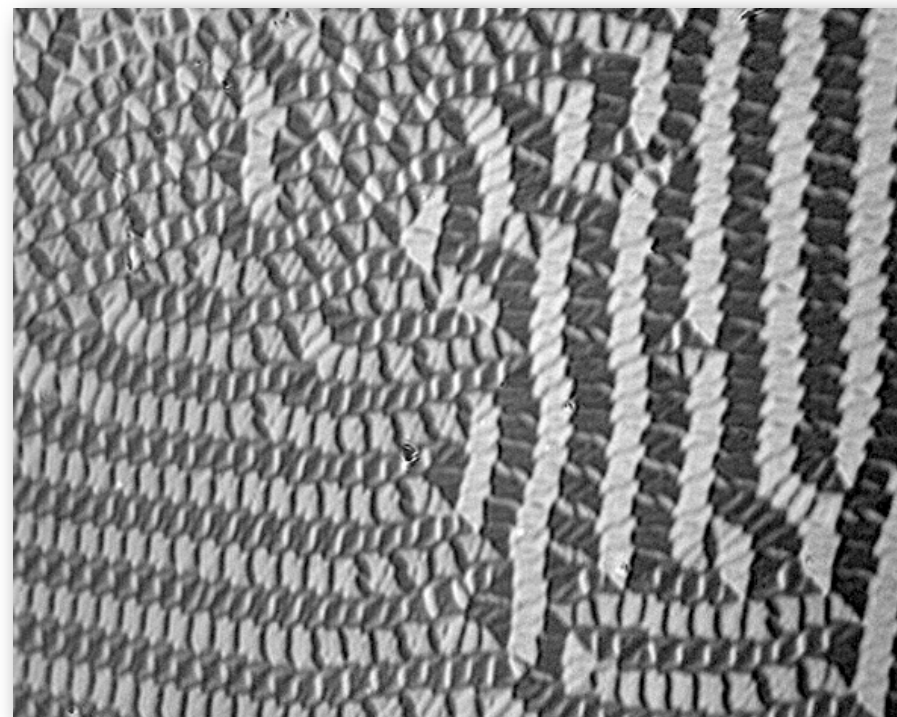
Difference image technique



Original image

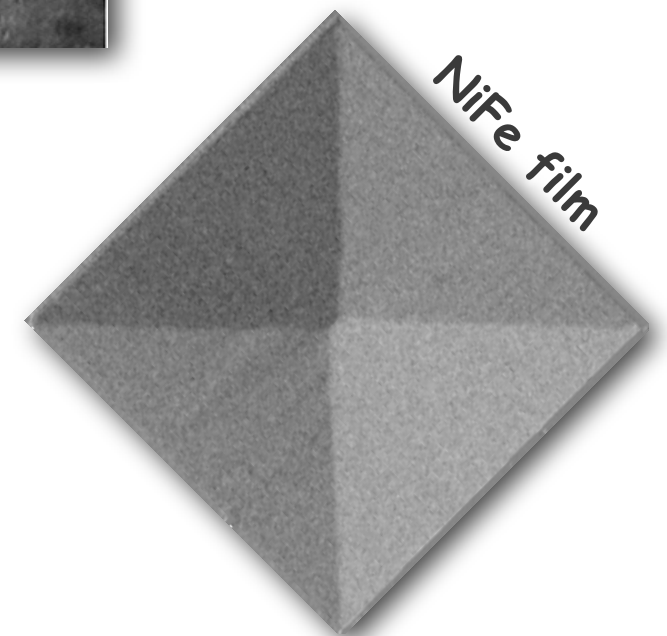


Reference image



Difference image

amorphous
ribbon



Important:
Difference Imaging
in real time !

4. Magneto-optical Kerr-Microscopy

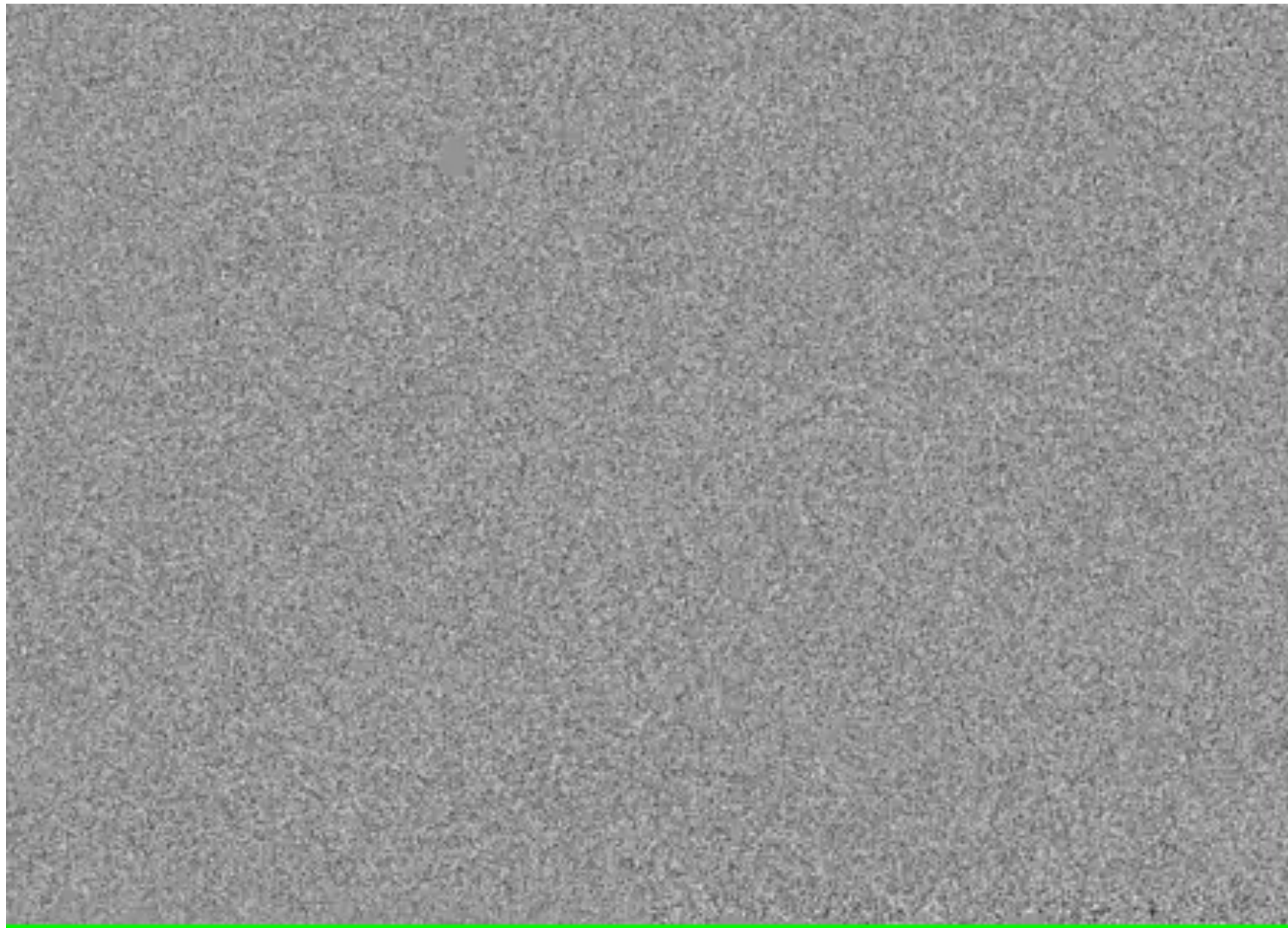
Vectorial imaging



Sensitivity



Sensitivity

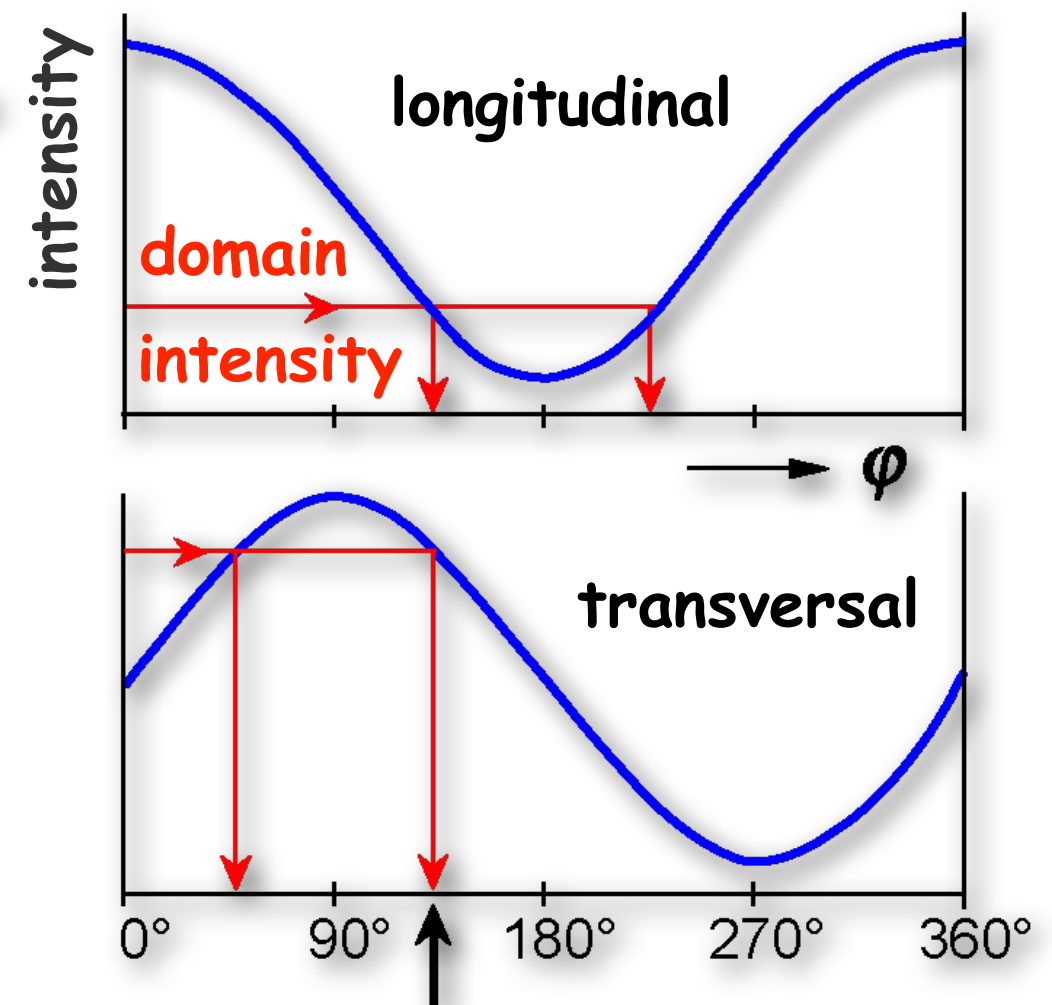
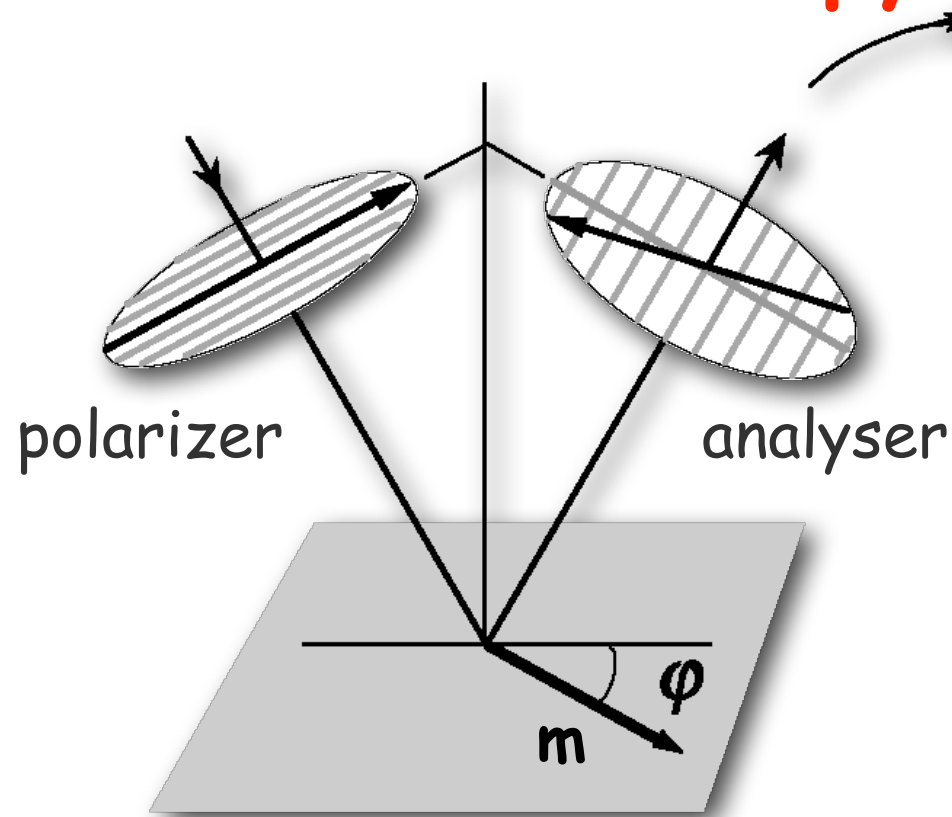


Courtesy J. McCord, Kiel

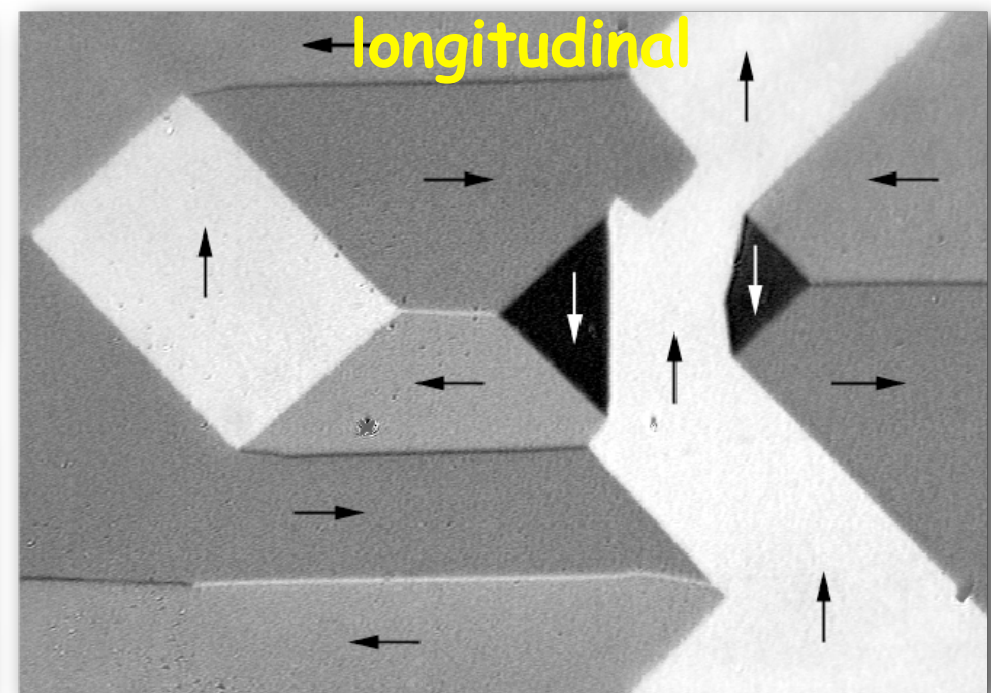
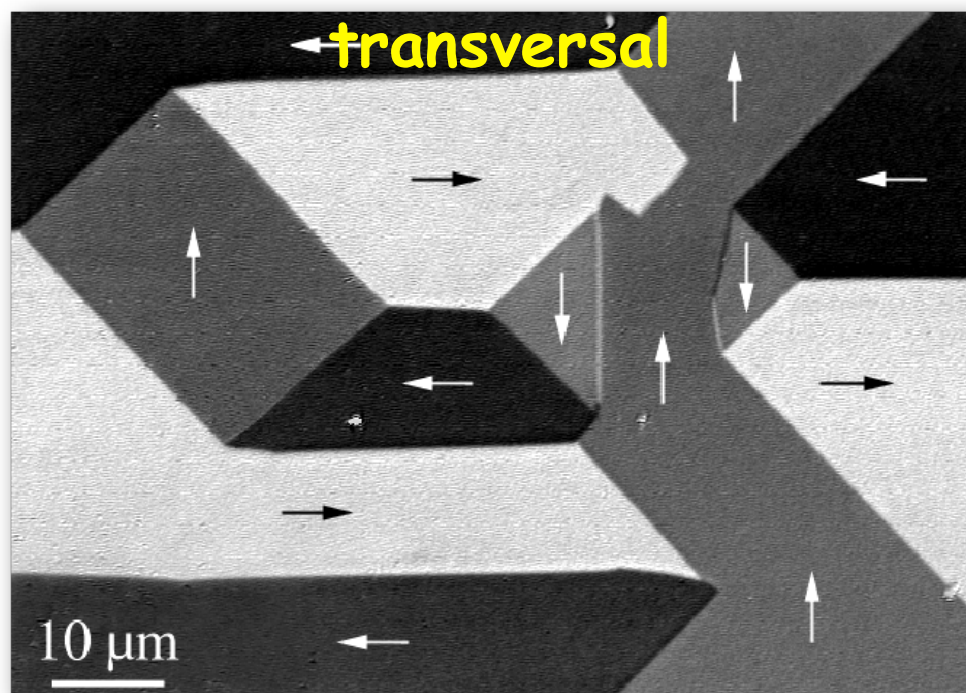
<http://www.tf.uni-kiel.de/matwis/nmm/en/movies/laminated>

4. Magneto-optical Kerr-Microscopy

Quantitative Kerr microscopy



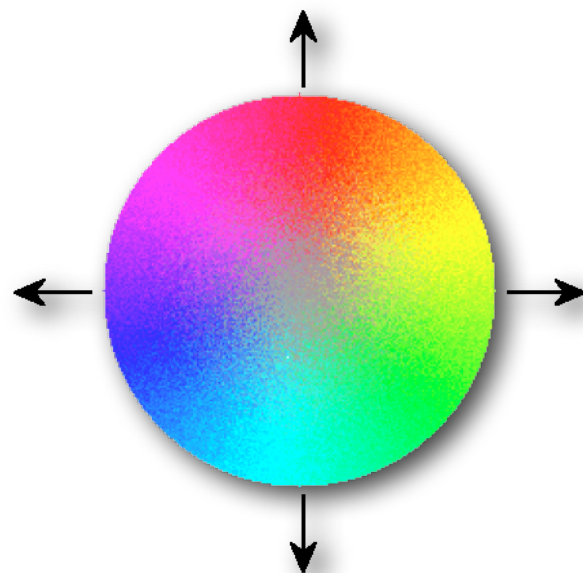
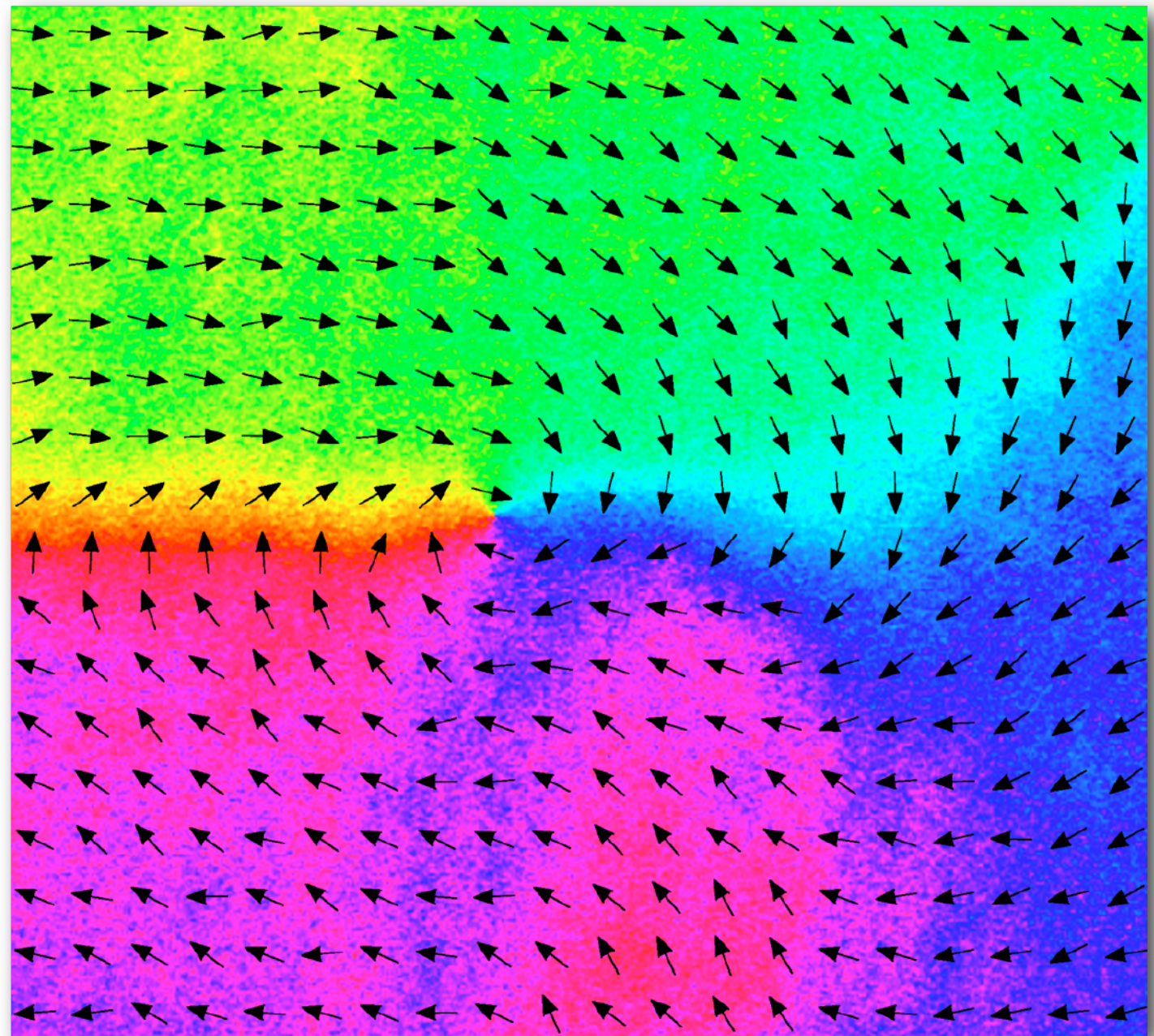
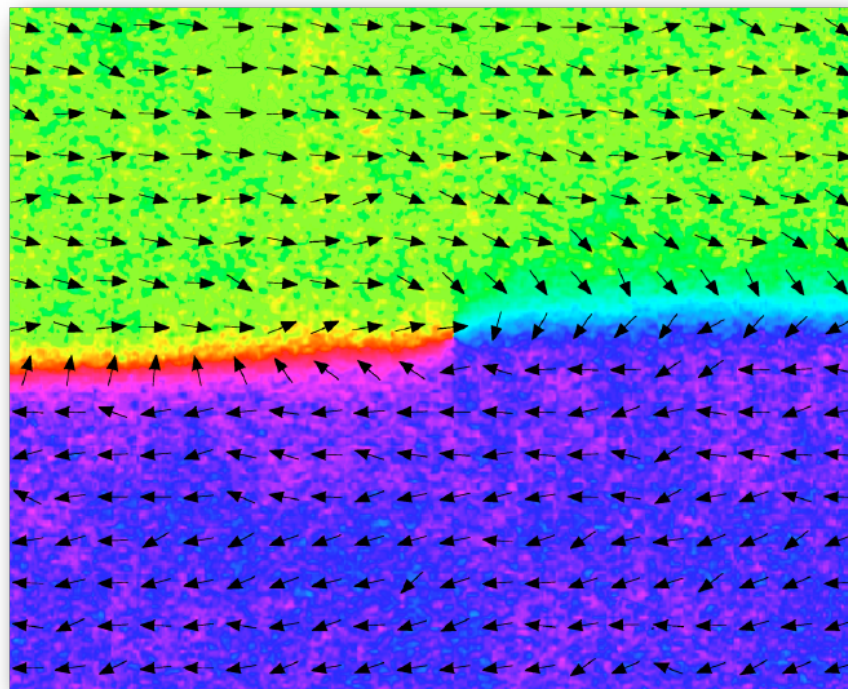
Domains on
(100)-FeSi
sheet



4. Magneto-optical Kerr-Microscopy

Quantitative Kerr microscopy

Domains in magnetostriction-free amorphous ribbon
as-quenched state after annealing in rotating field



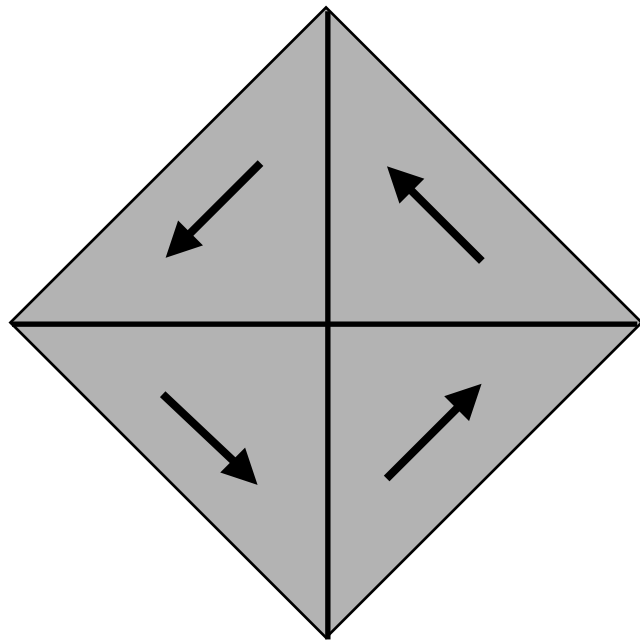
5 μm

4. Magneto-optical Kerr-Microscopy

MOKE-Magnetometry and domains

Permalloy (NiFe) film
207 nm thick

57 μm

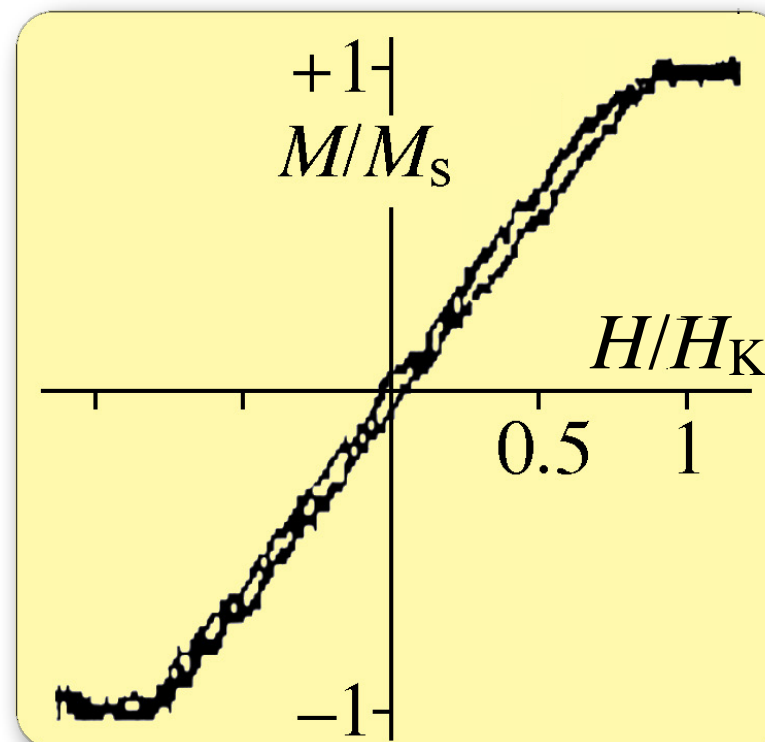


Magnetic field H

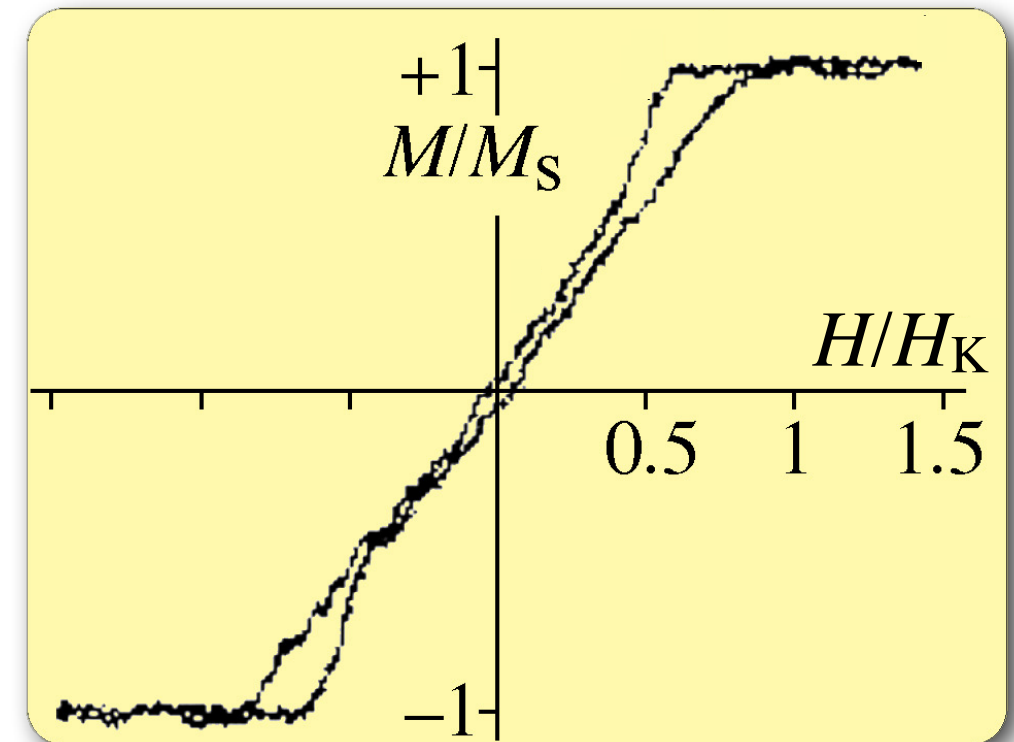


MOKE
 $M(H)$ loops

$H_{\text{max}} = H_K$



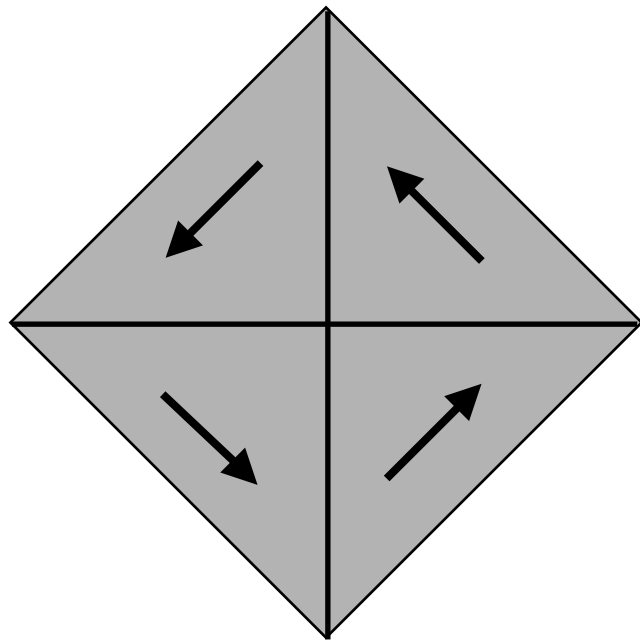
$H_{\text{max}} > H_K$



4. Magneto-optical Kerr-Microscopy

MOKE-Magnetometry and domains

Permalloy (NiFe) film
207 nm thick

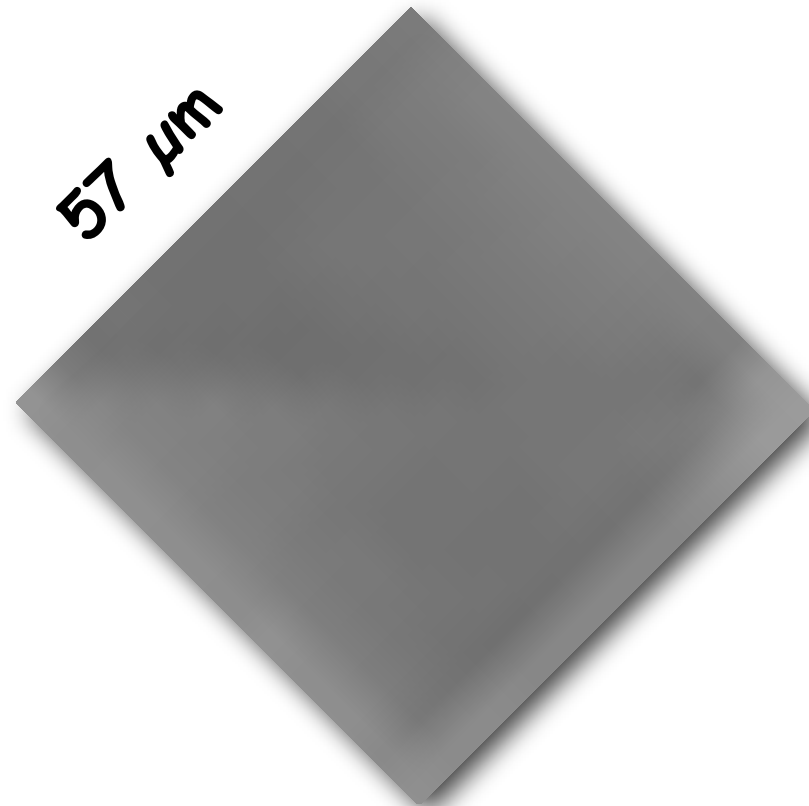


Magnetic field H

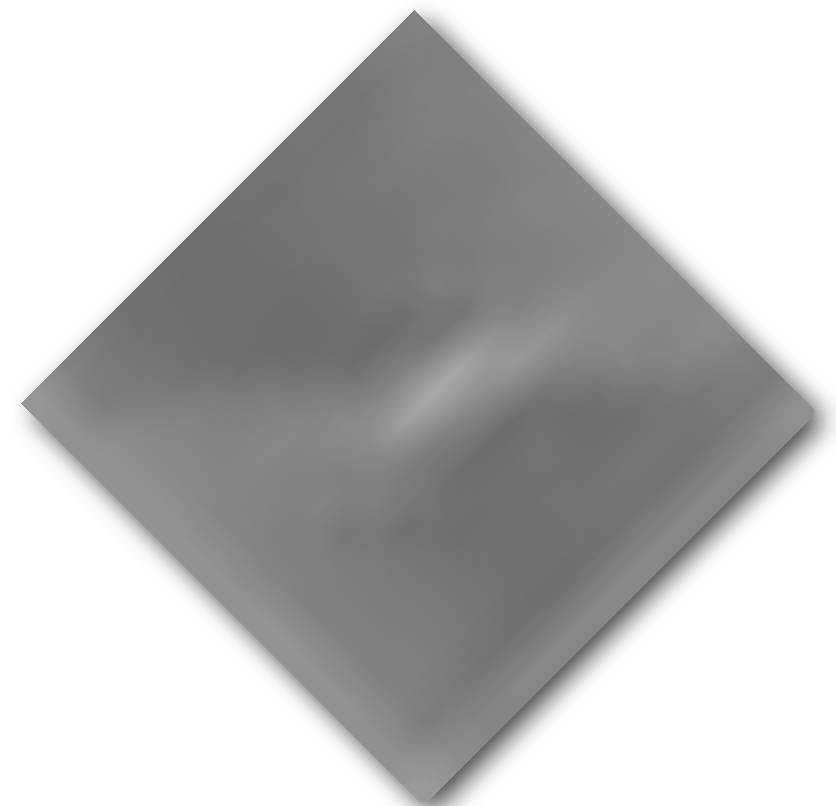
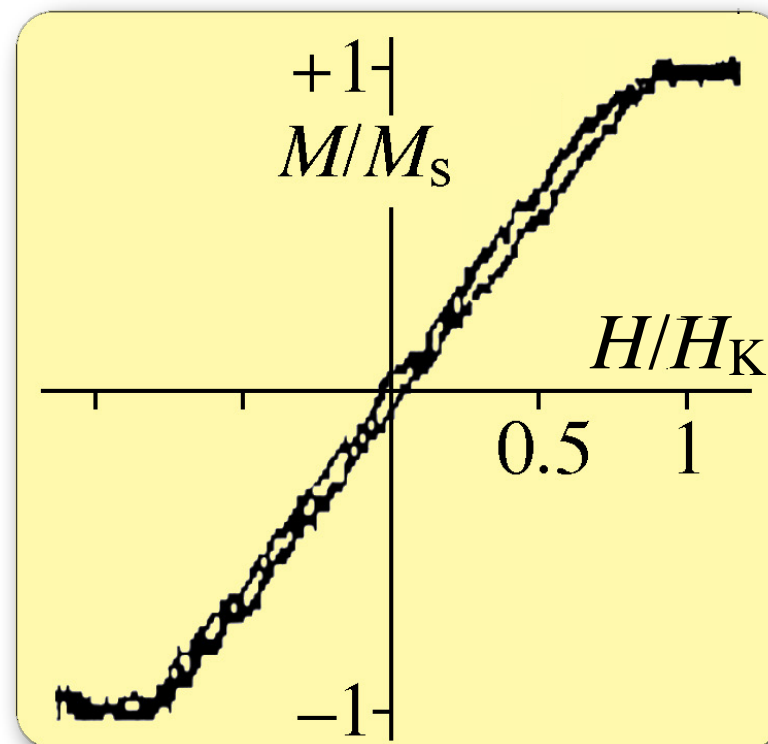


MOKE
 $M(H)$ loops

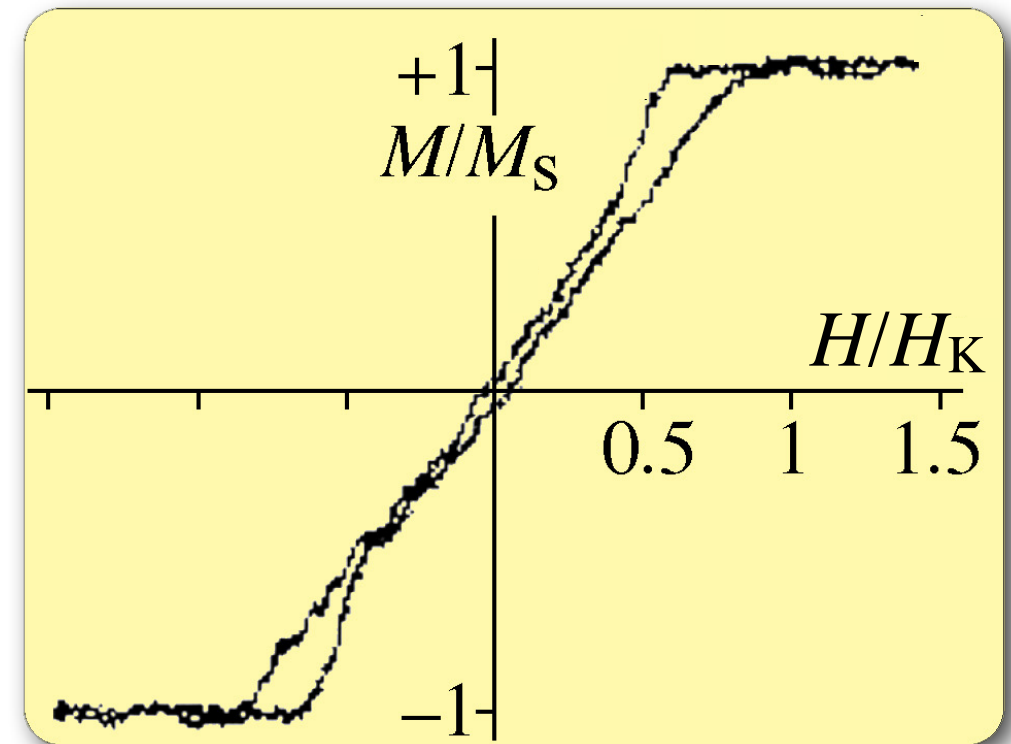
57 μm



$H_{\text{max}} = H_K$



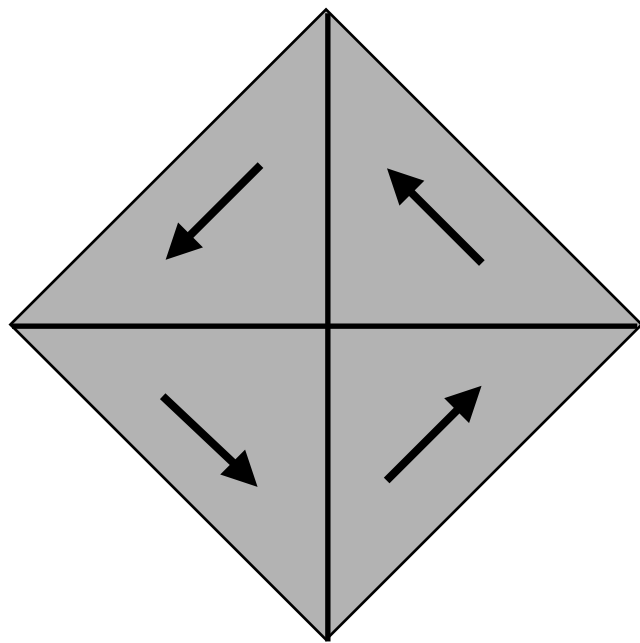
$H_{\text{max}} > H_K$



4. Magneto-optical Kerr-Microscopy

MOKE-Magnetometry and domains

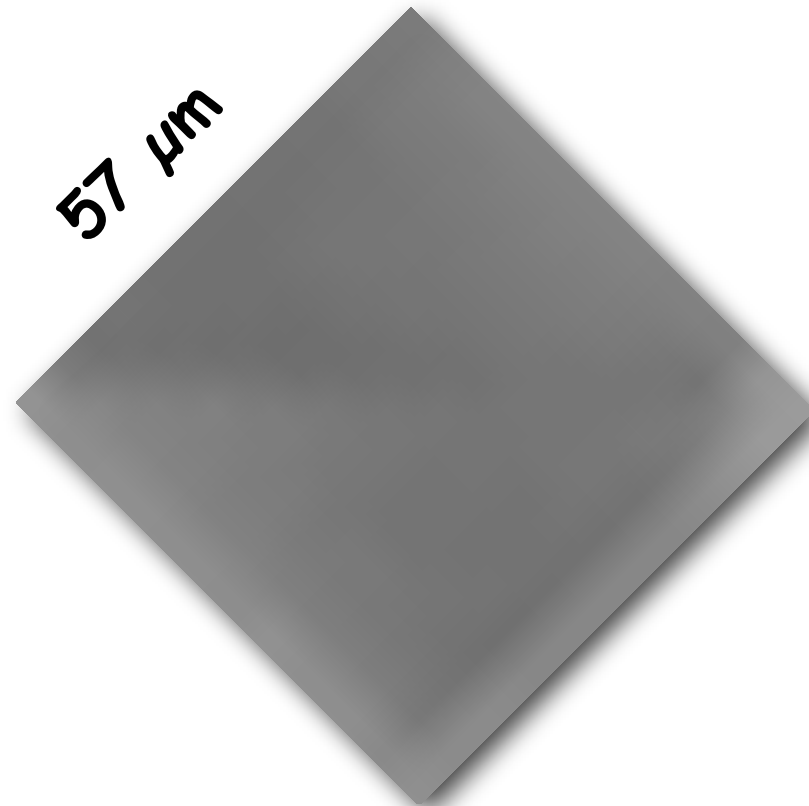
Permalloy (NiFe) film
207 nm thick



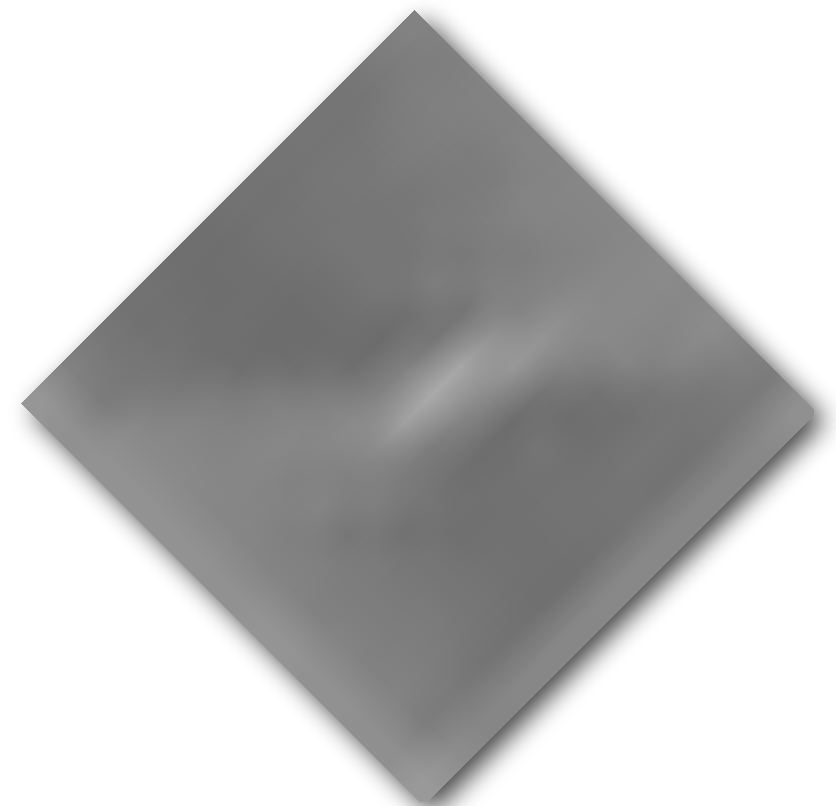
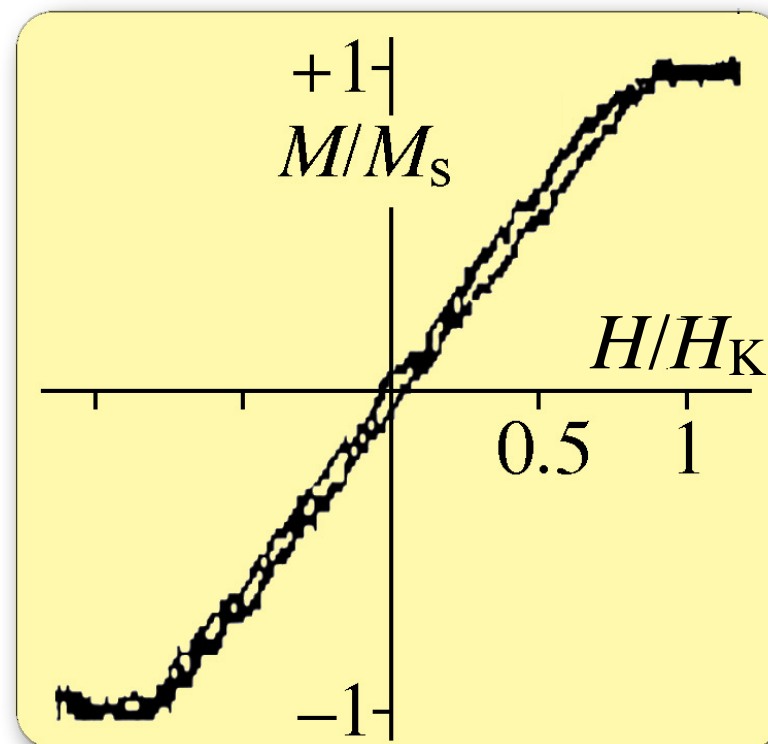
Magnetic field H



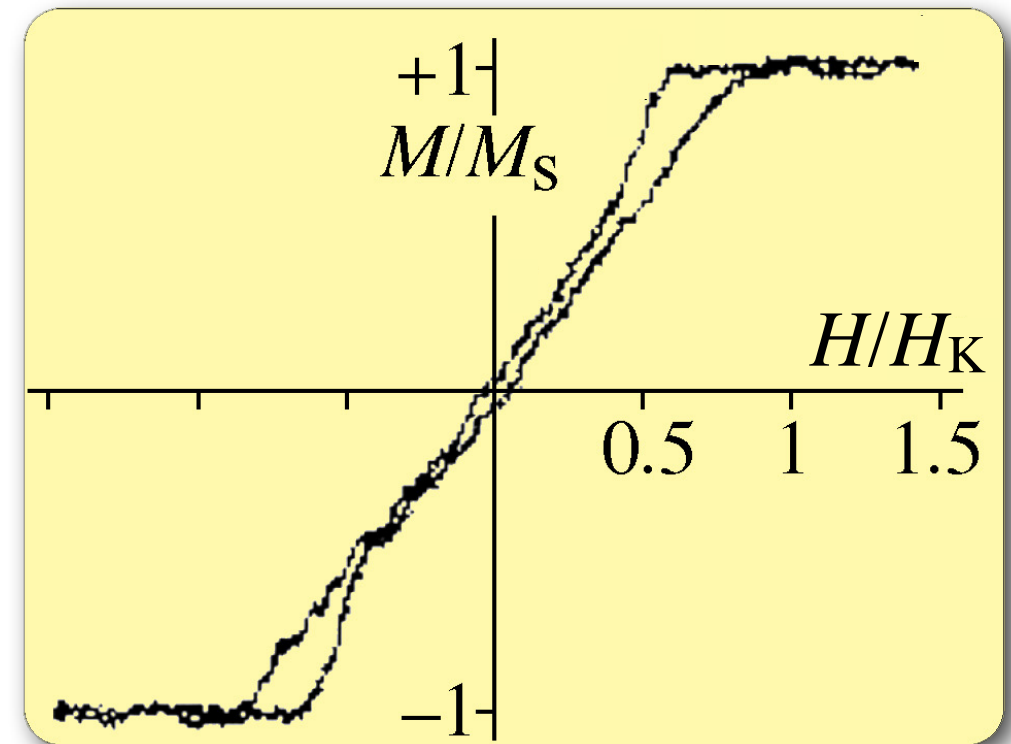
MOKE
 $M(H)$ loops



$H_{\max} = H_K$



$H_{\max} > H_K$

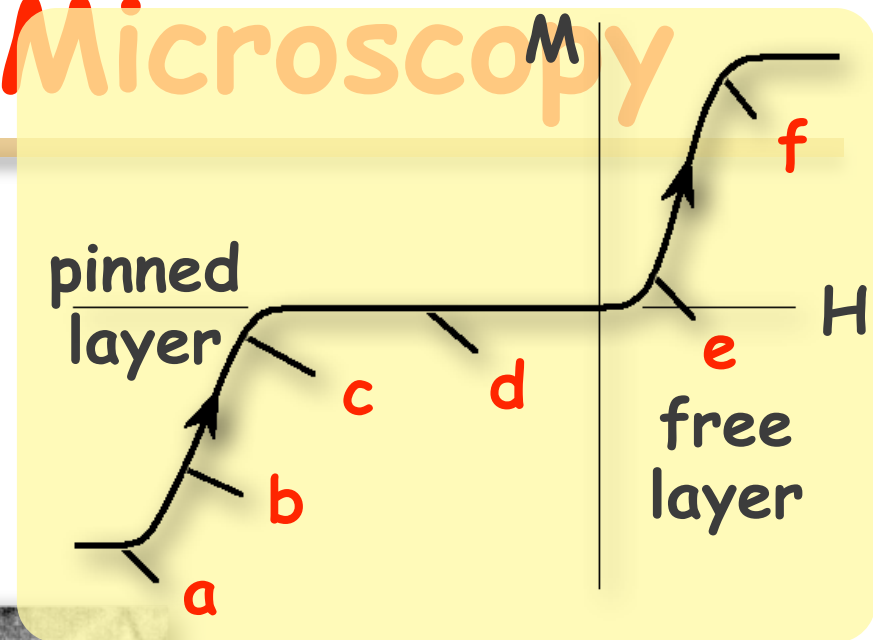
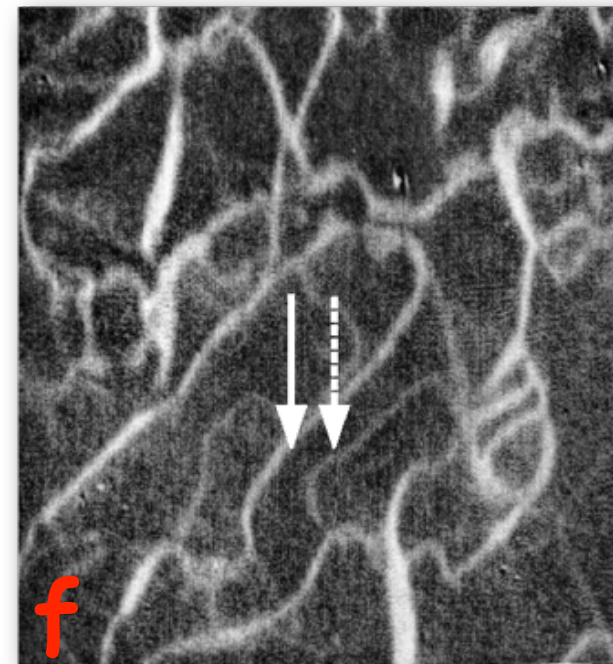
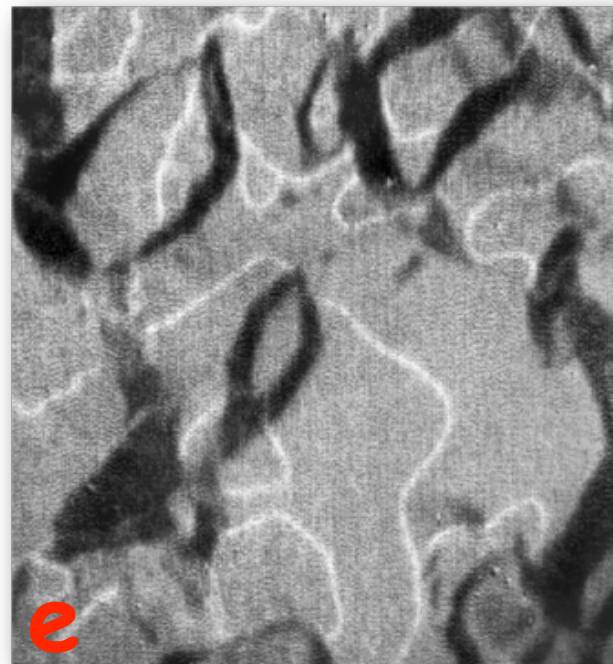
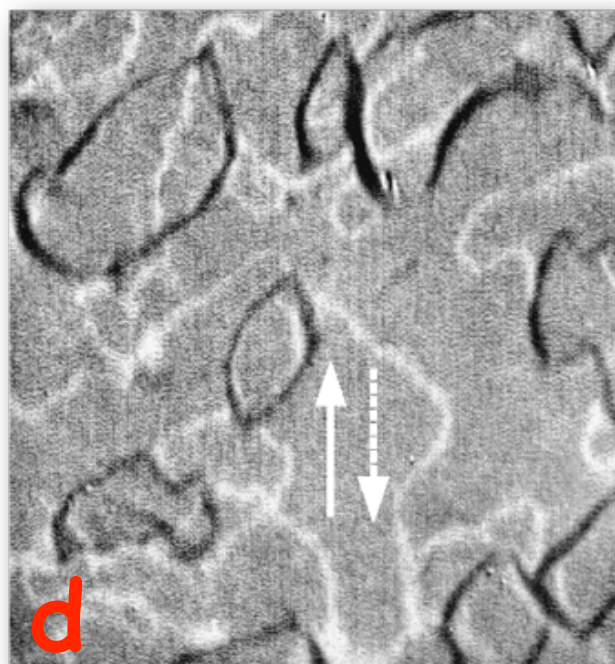
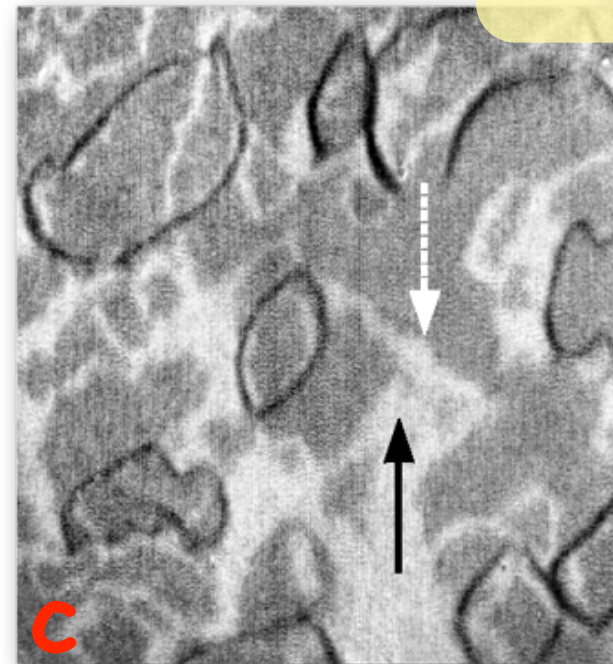
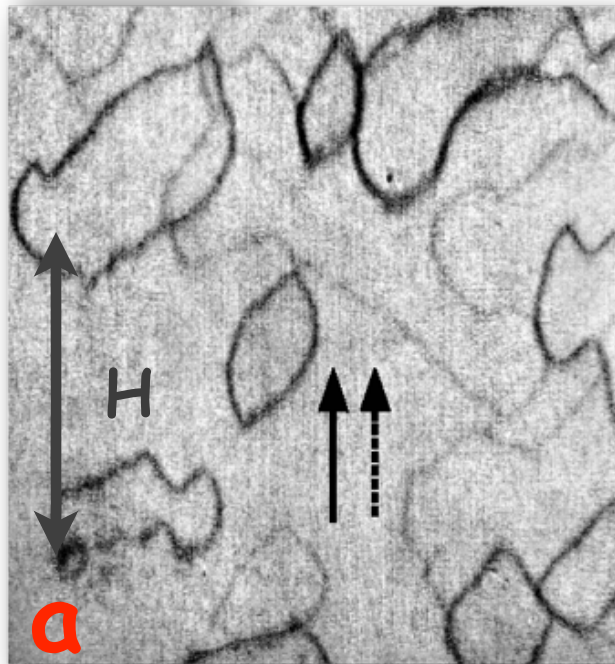


4. Magneto-optical Kerr-Microscopy

Depth sensitivity of Kerr microscopy

Information depth in metals: ~20 nm

20 μm



NiFe (3 nm)

Ru (3 nm)

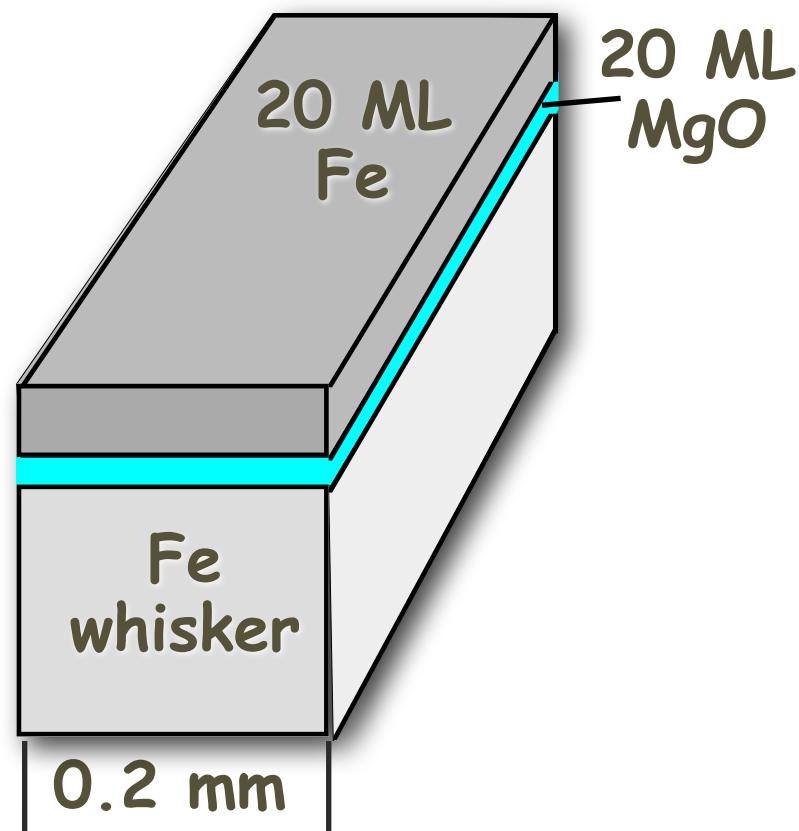
NiFe (10 nm)

FeMn

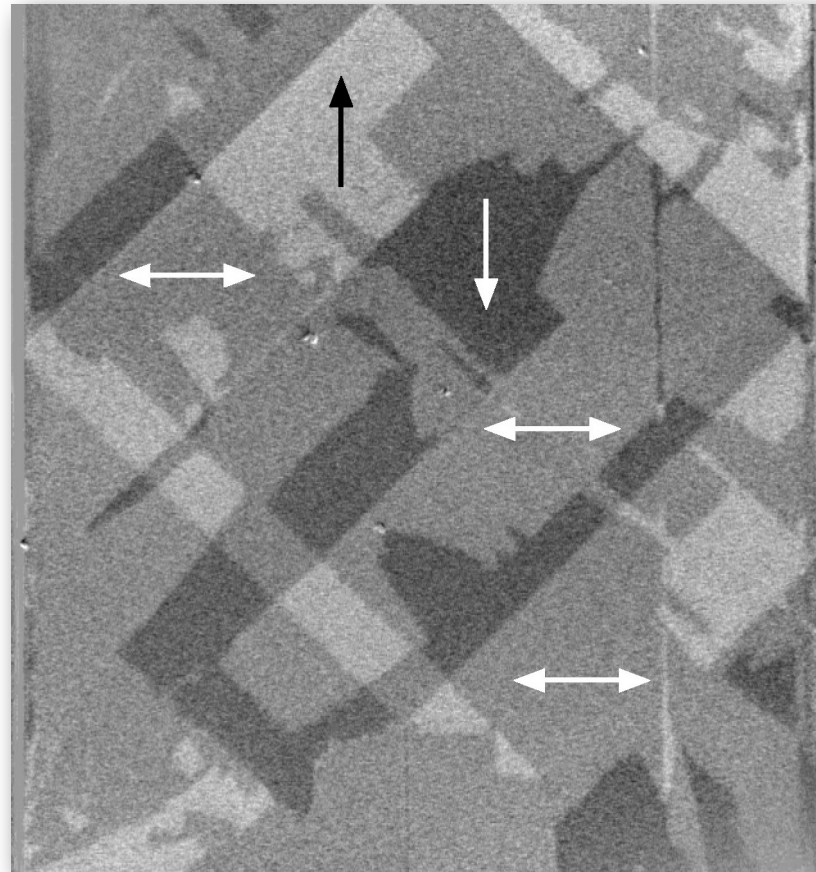
Sample:
S. Parkin, IBM

4. Magneto-optical Kerr-Microscopy

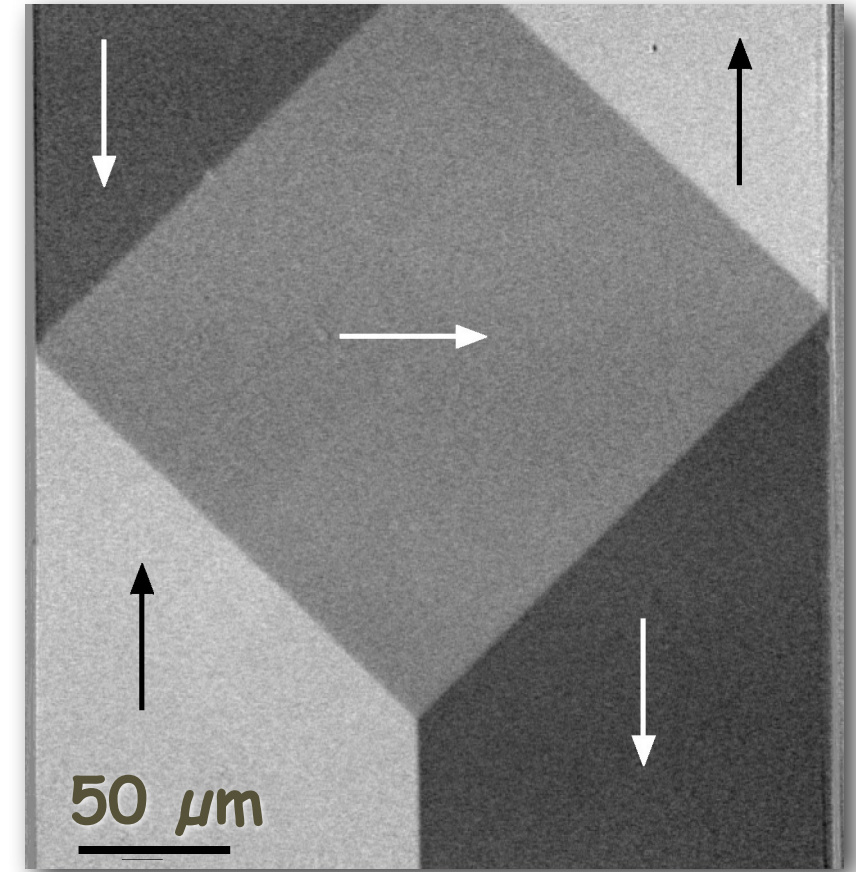
Layer-selective Kerr microscopy



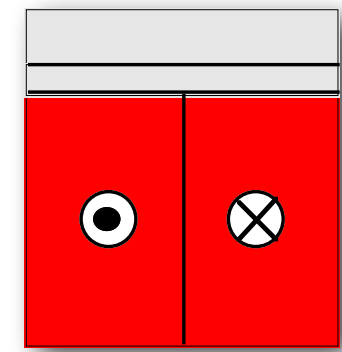
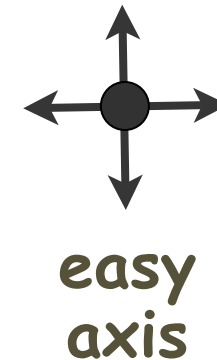
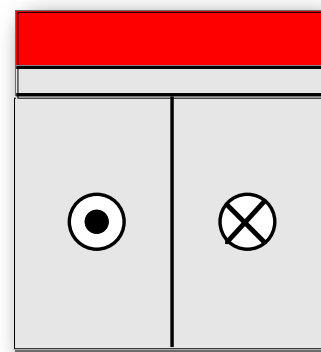
Domains in Fe-film



Whisker domains



Depth-selectivity by tuning phase of Kerr amplitude with compensator



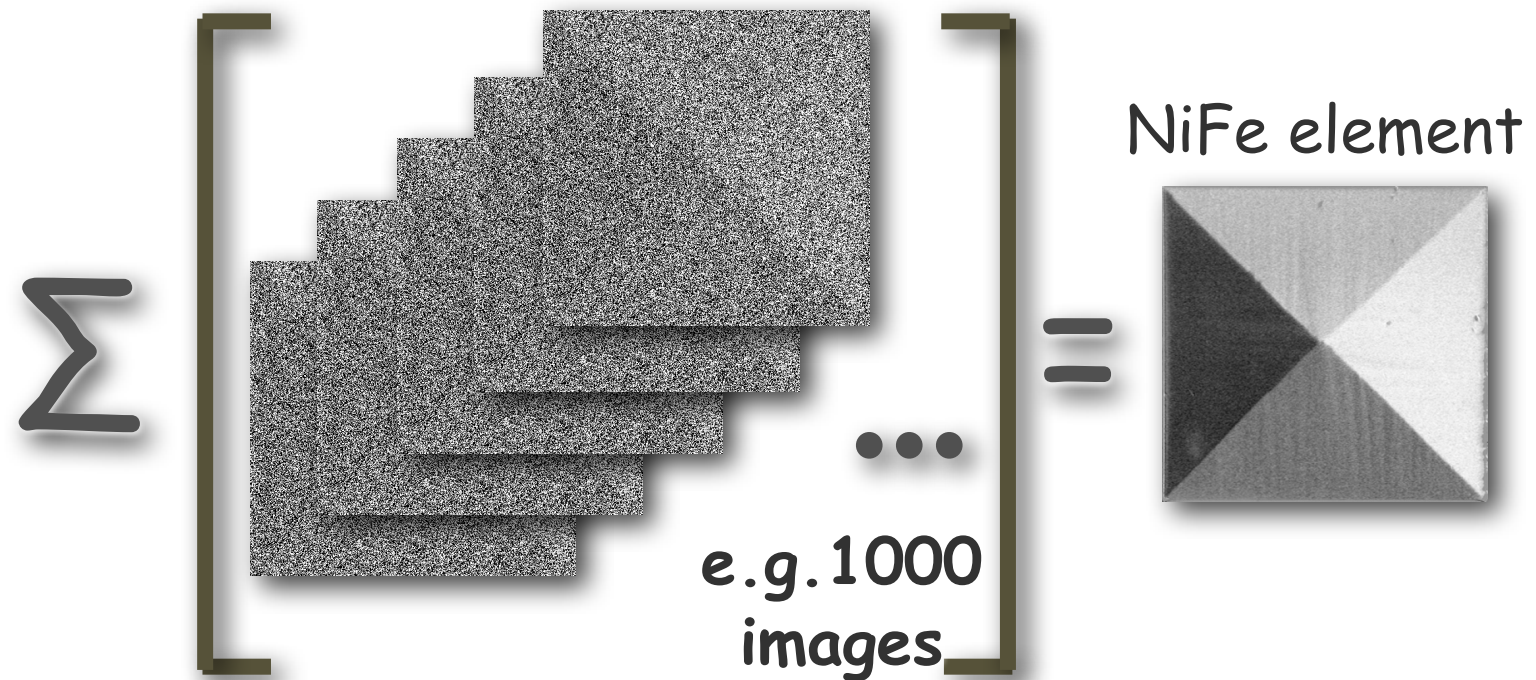
R. S., R. Urban, D. Ullmann, H. L. Meyerheim, B. Heinrich, L. Schultz, J. Kirschner, PRB 65, 144405 (2003)

4. Magneto-optical Kerr-Microscopy

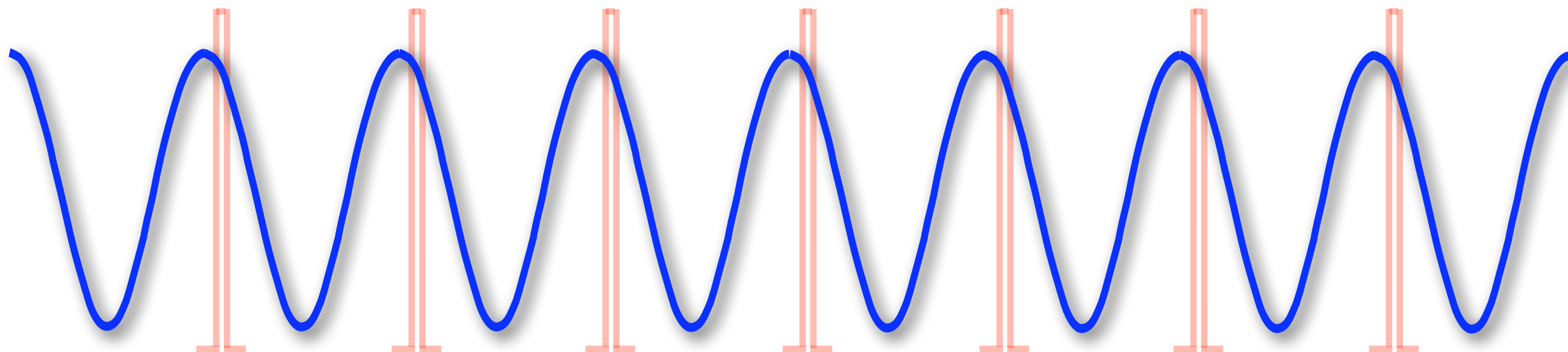
Time-resolved (stroboscopic) imaging

illumination intensity and repetition rate are limited

- no single-shot imaging possible
- accumulation of large number of independent events necessary (at fixed time delay)
- requires repetitive magnetization processes !!



probing with defined time delay



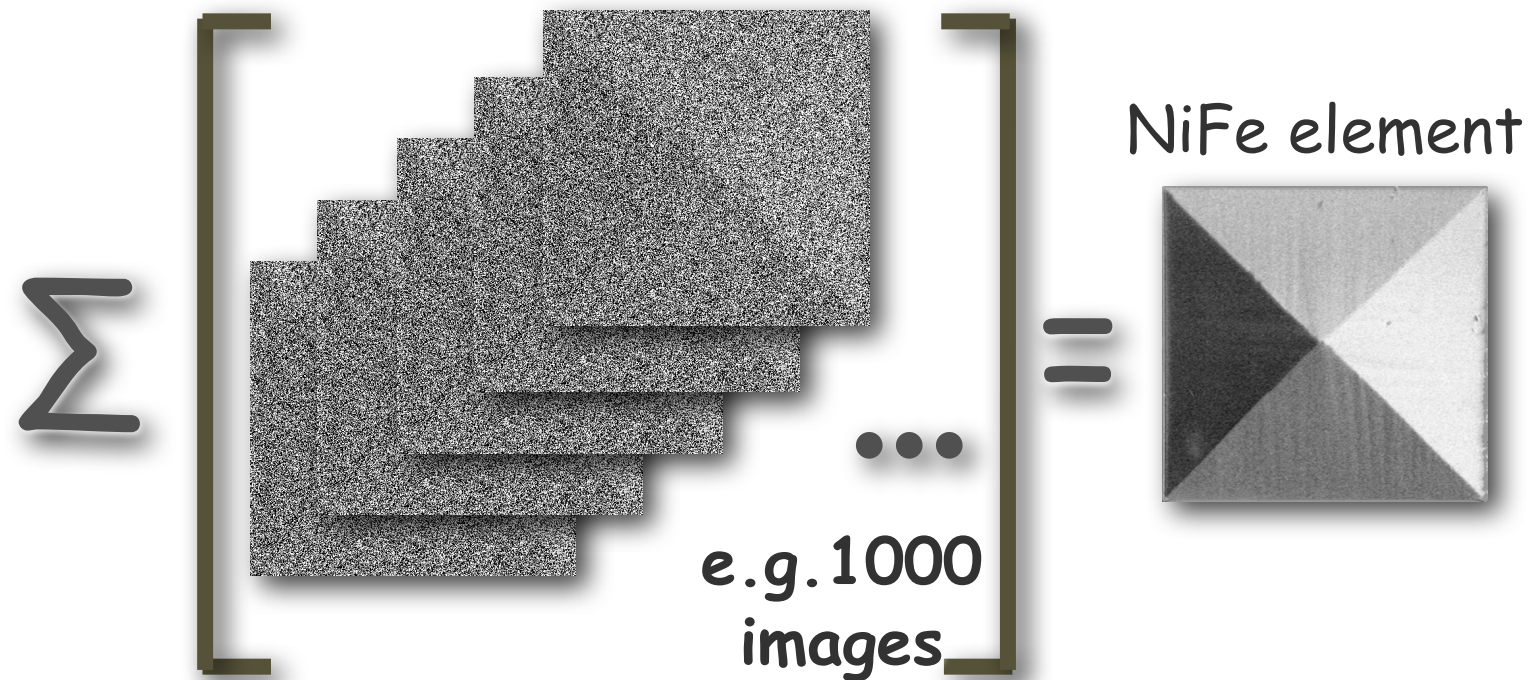
periodic magnetic field excitation

4. Magneto-optical Kerr-Microscopy

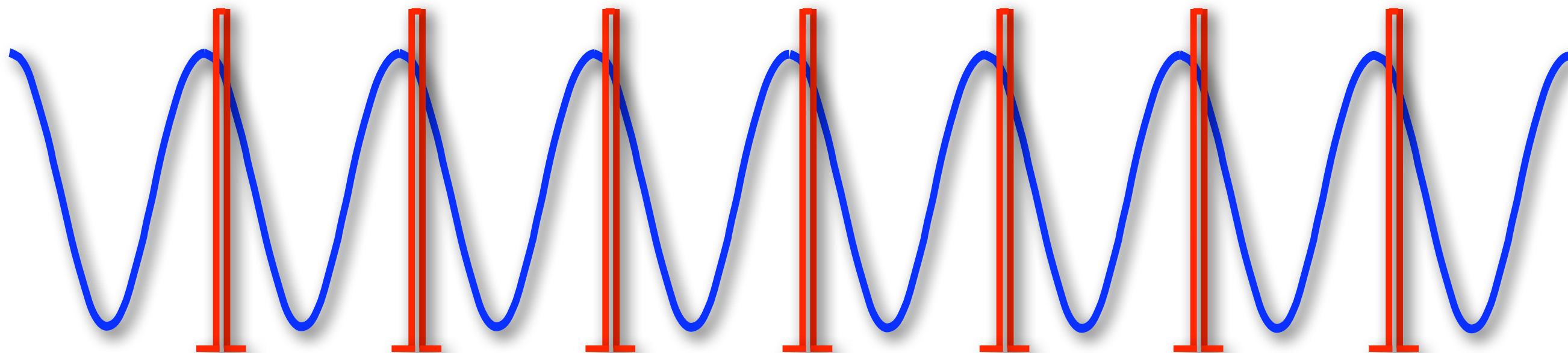
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probing with defined time delay



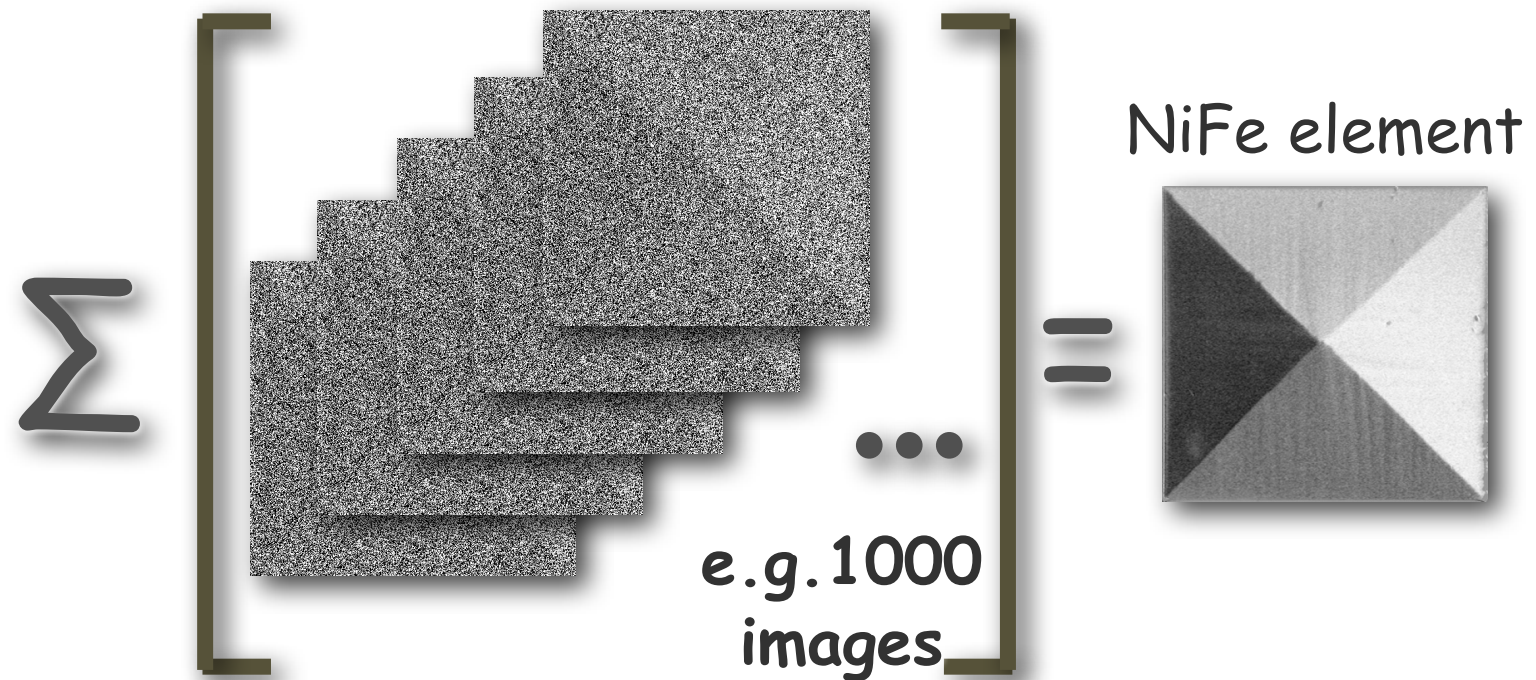
periodic magnetic field excitation

4. Magneto-optical Kerr-Microscopy

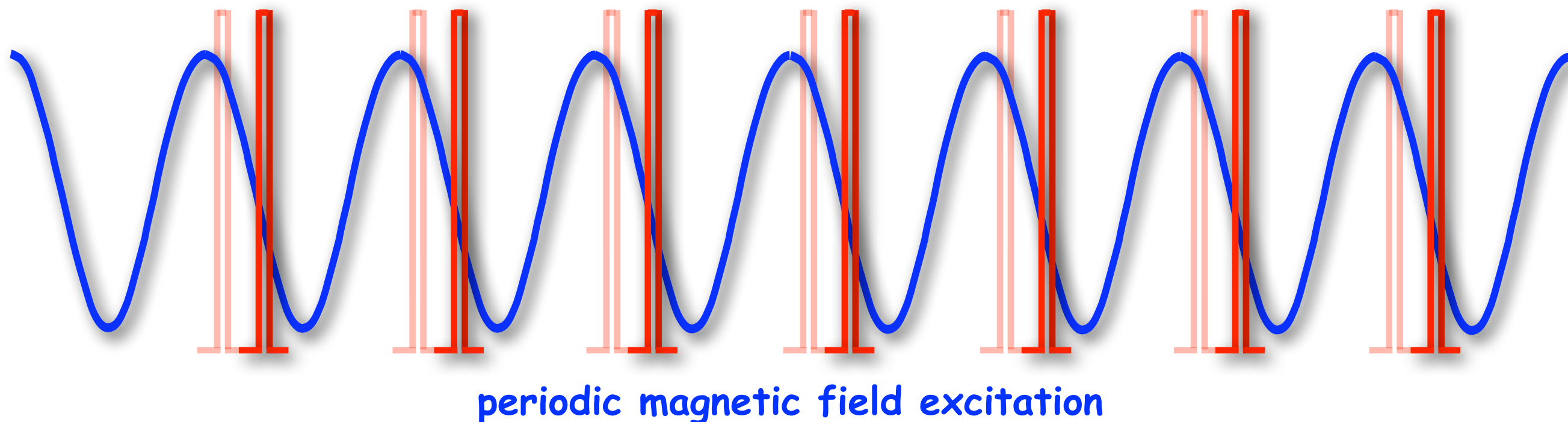
Time-resolved (stroboscopic) imaging

illumination intensity and repetition rate are limited

- no single-shot imaging possible
- accumulation of large number of independent events necessary (at fixed time delay)
- requires repetitive magnetization processes !!

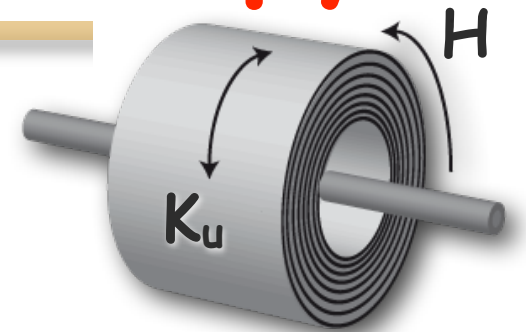


probing with defined time delay

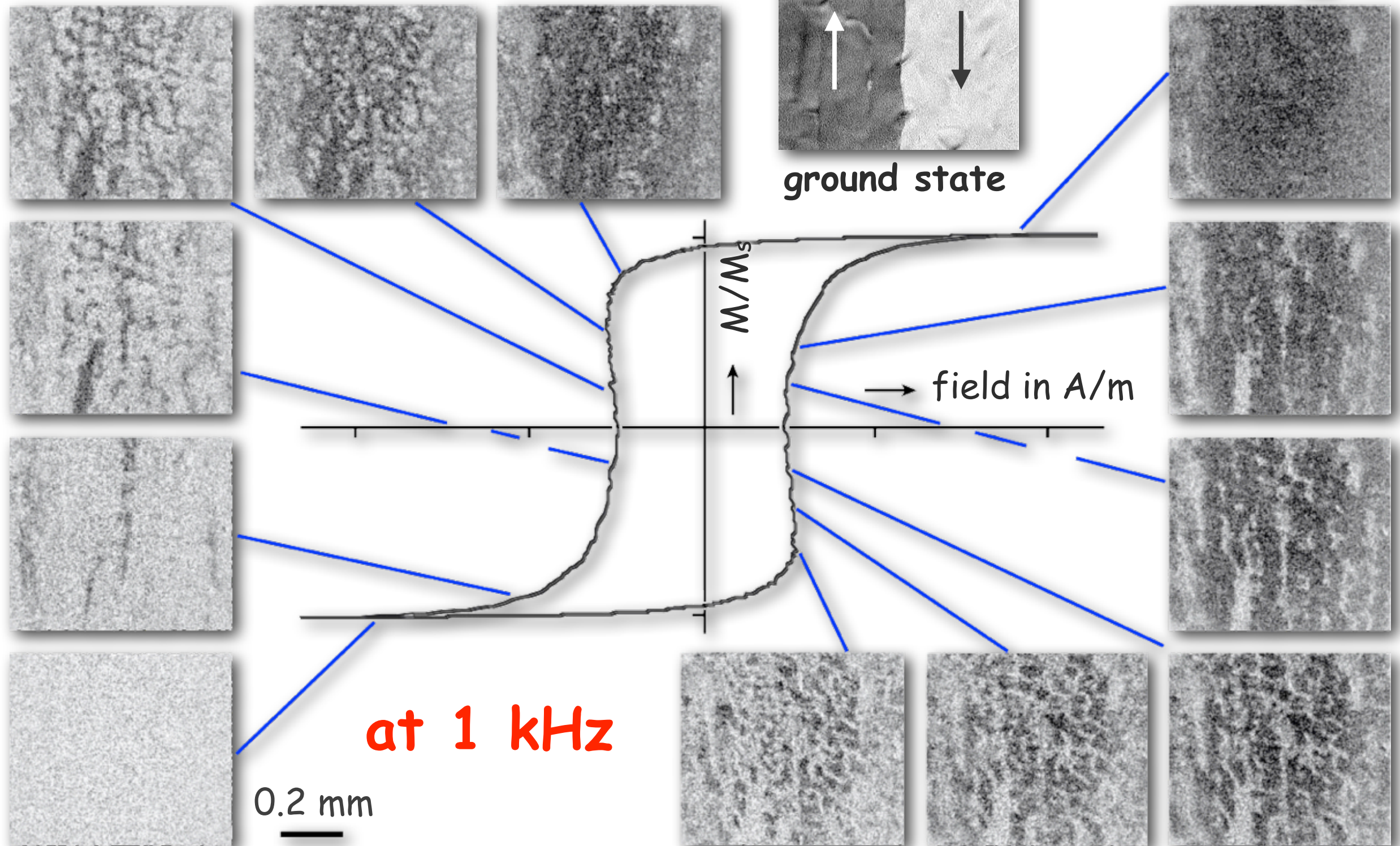


4. Magneto-optical Kerr-Microscopy

Time-resolved (stroboscopic) imaging



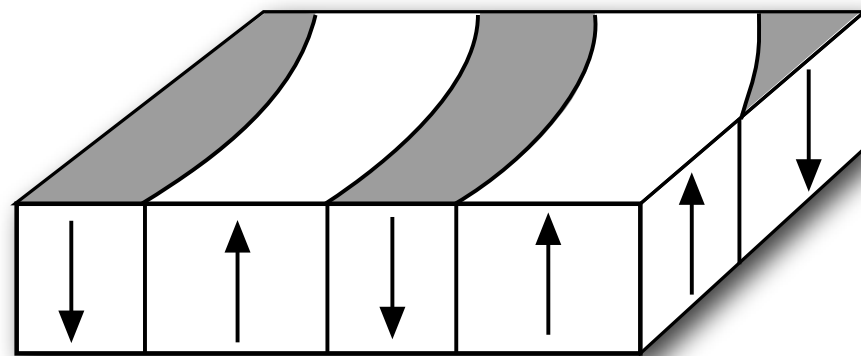
Nanocrystalline core with weak K_u



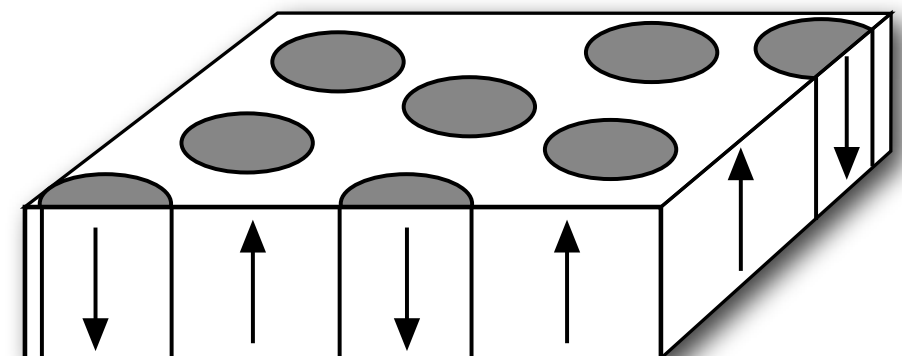
4. Magneto-optical Kerr-Microscopy



Band domains

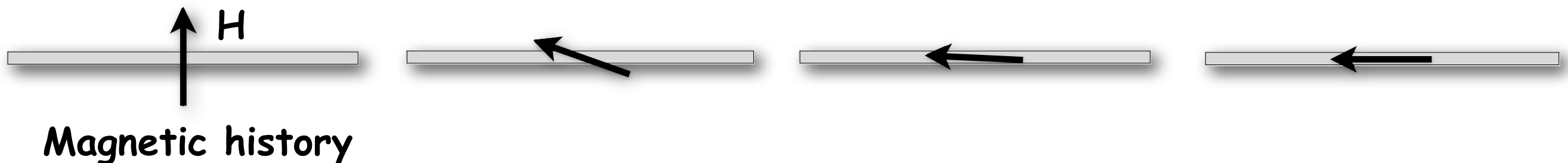
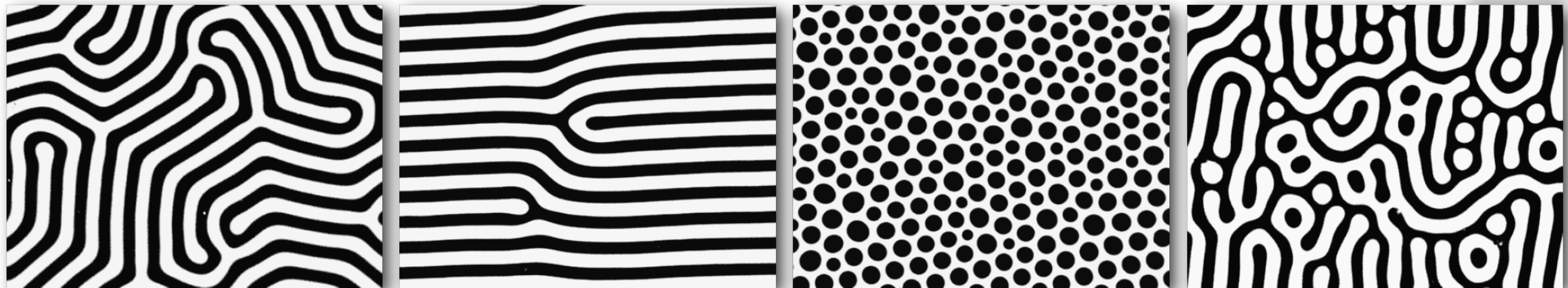


Bubble domains



Bubble garnet film

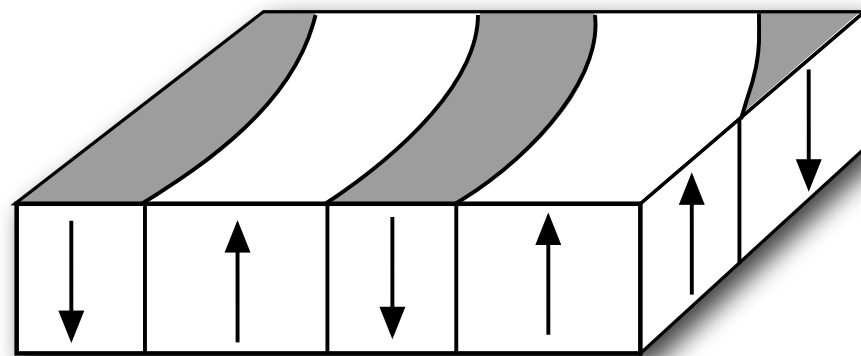
20 μm



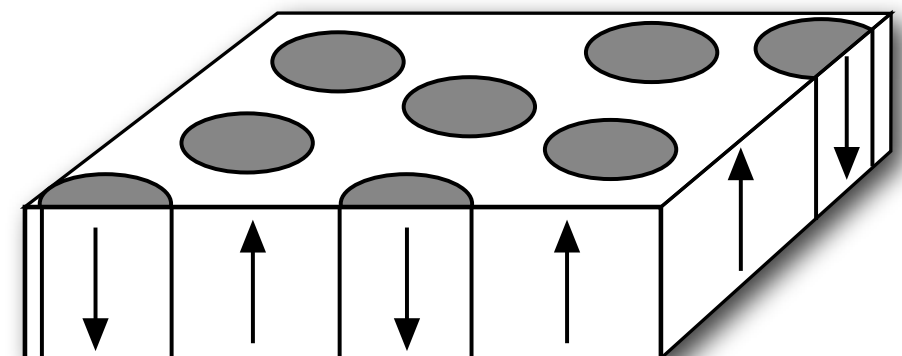
4. Magneto-optical Faraday-Microscopy



Band domains

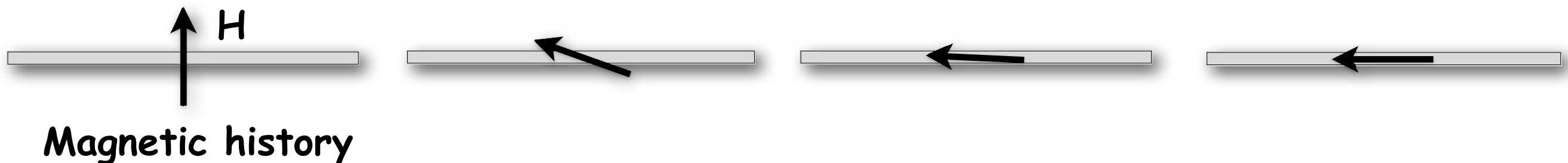
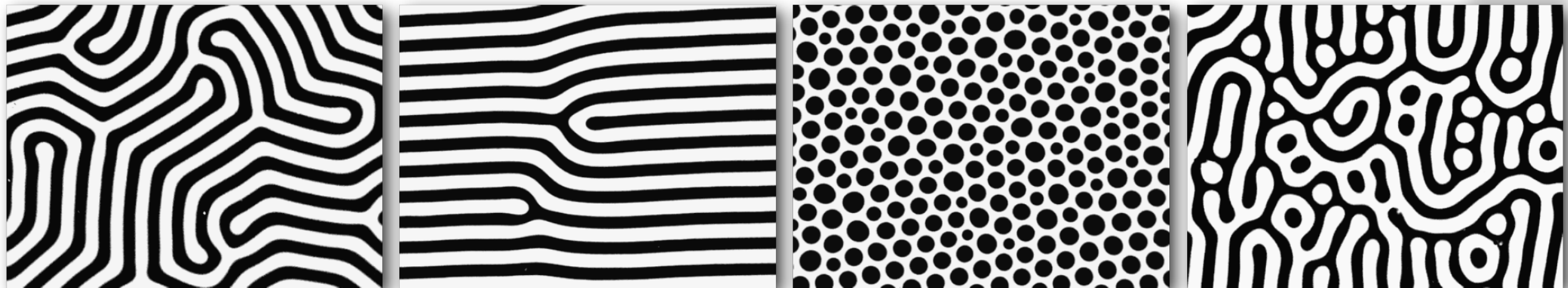


Bubble domains



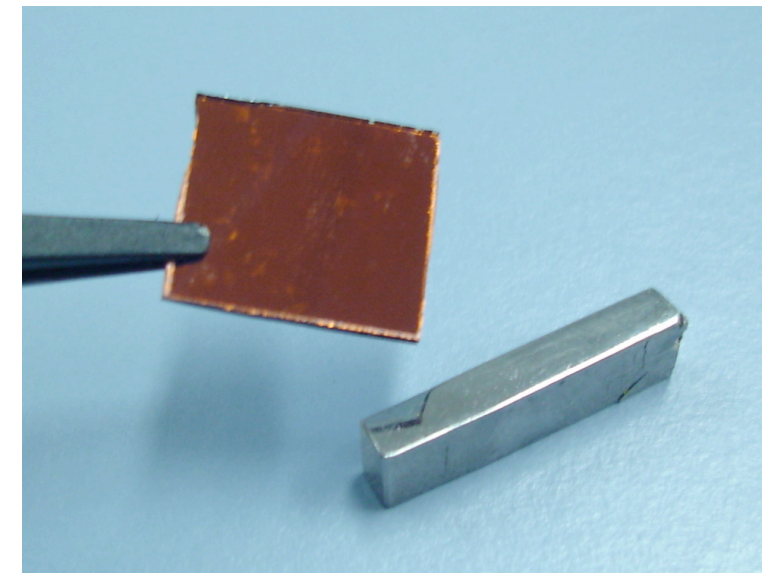
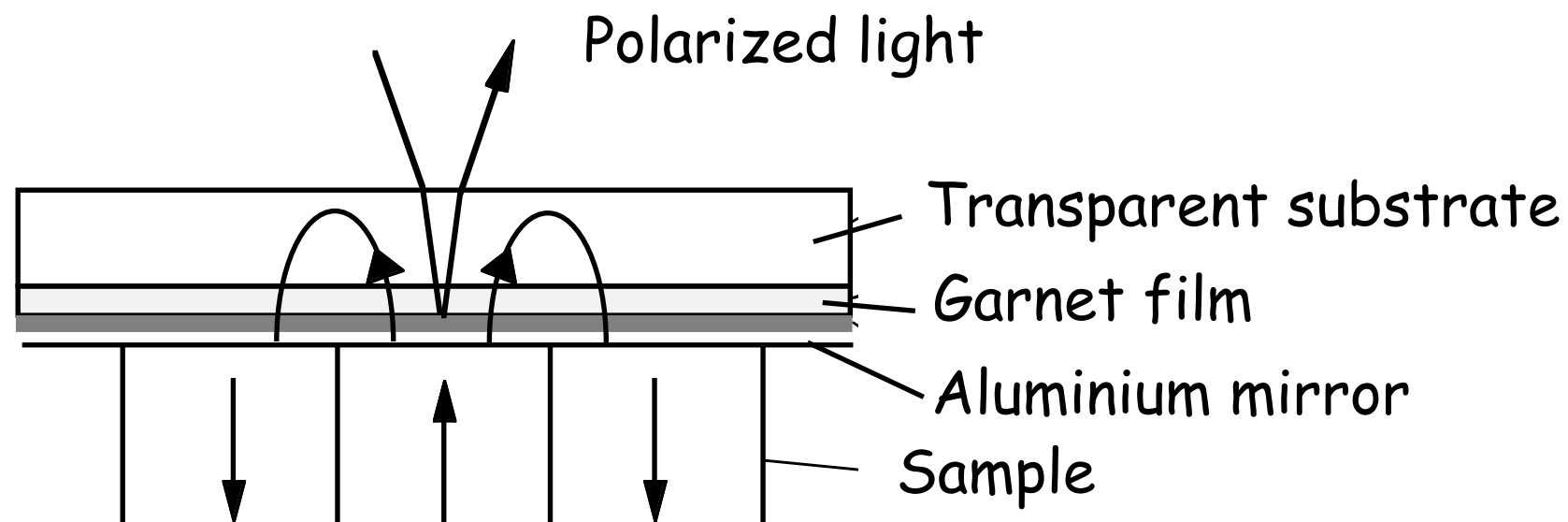
Bubble garnet film

20 μm



4. Magneto-optical Faraday-Microscopy

Indicator film technique: stray field imaging

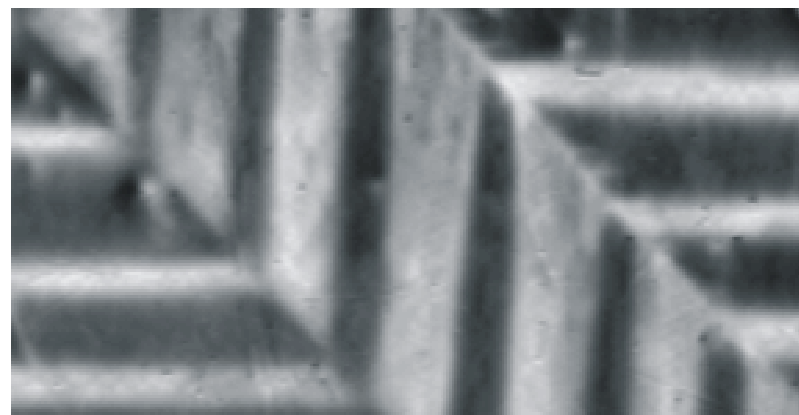


Metallographic contrast



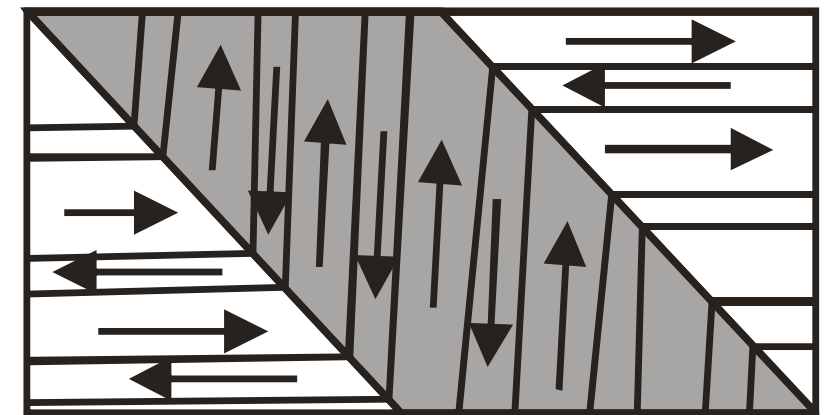
200 μm

With indicator film



Bulk NiMnGa single crystal

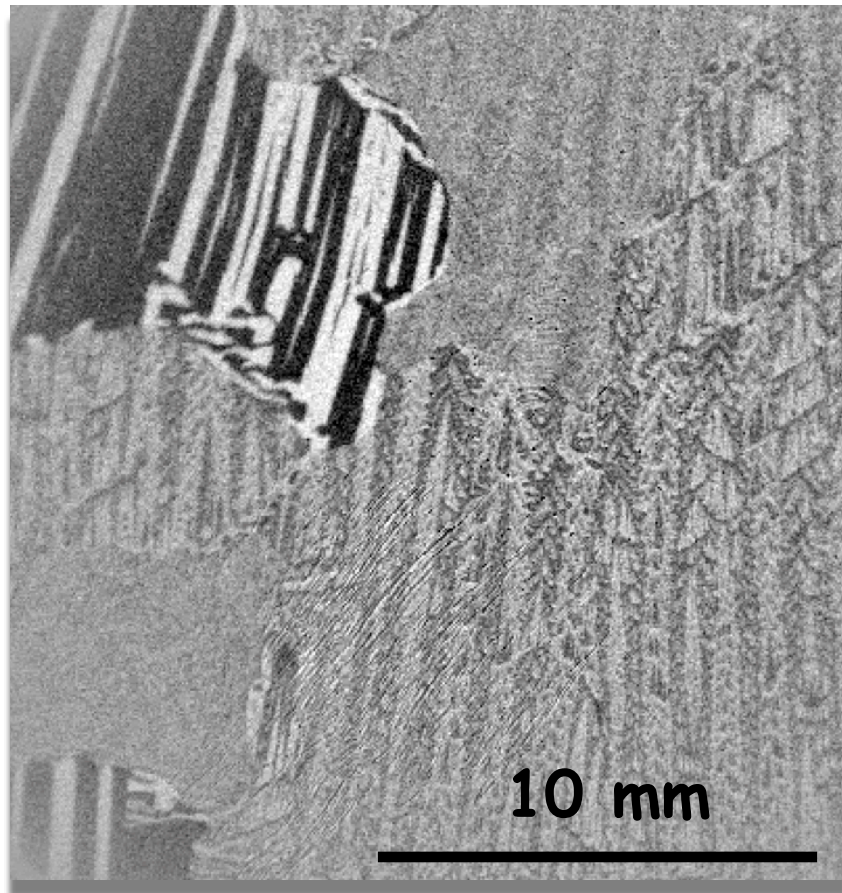
(shape memory material, does not show direct Kerr contrast)



4. Magneto-optical Faraday-Microscopy

Indicator film technique: stray field imaging

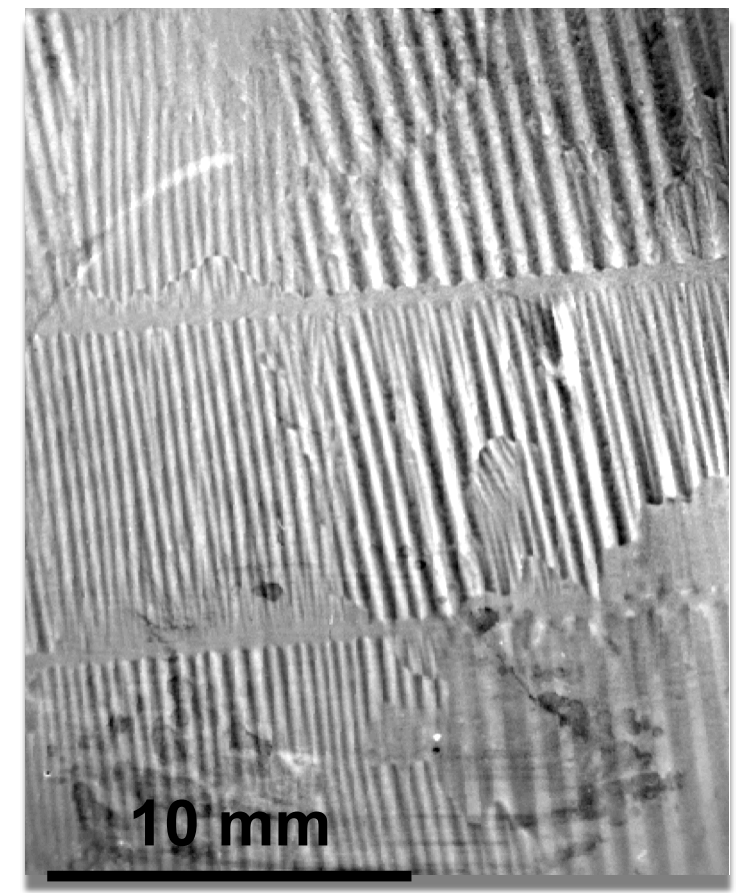
Grain oriented electrical steel:



Direct Kerr contrast
(sample polished)



MOIF contrast
(sample polished)



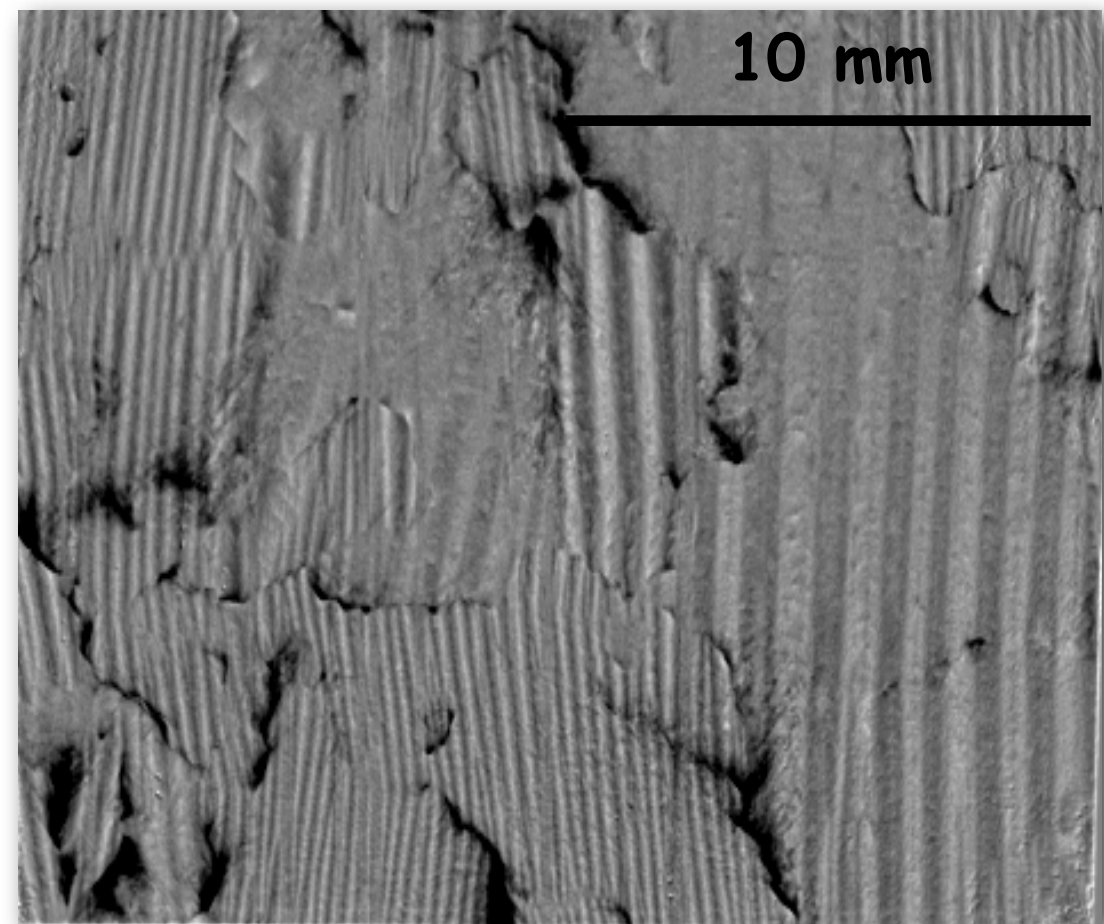
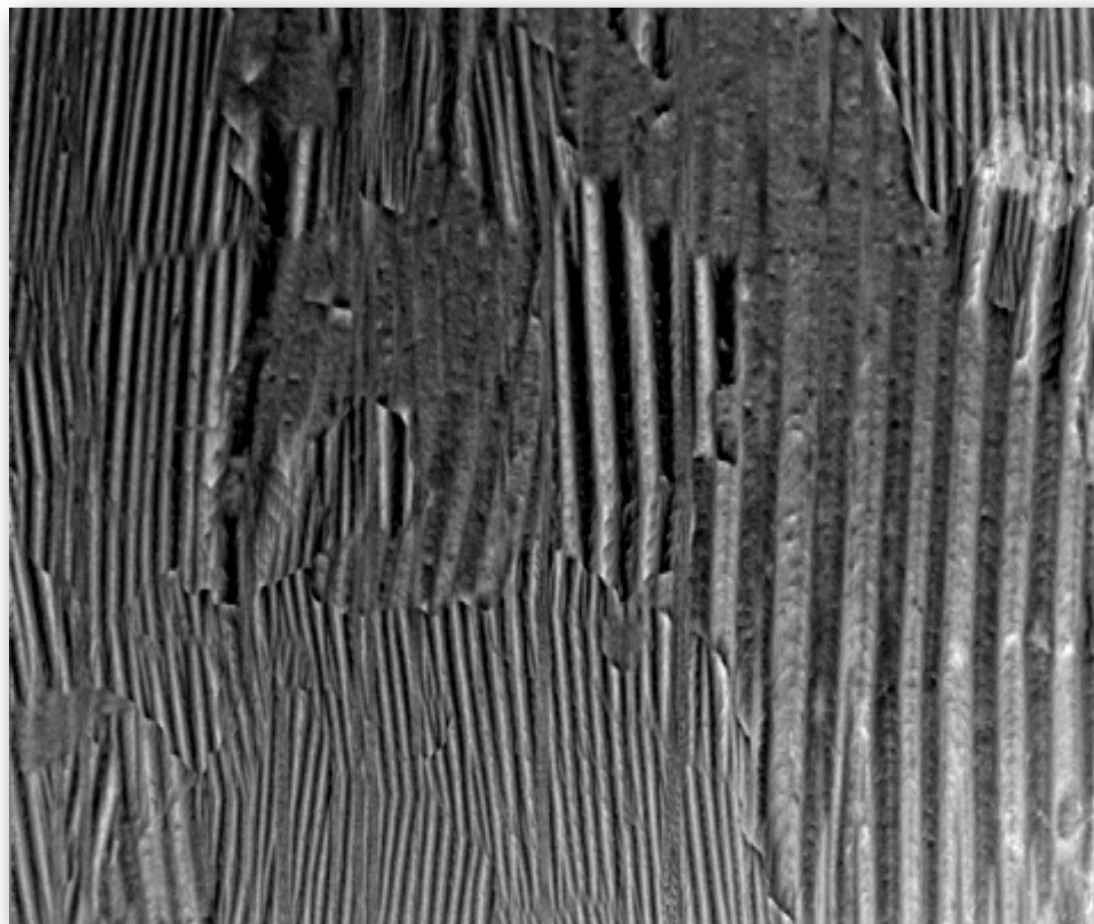
MOIF contrast
(with insulation coating)

Domain contrast even through coating

4. Magneto-optical Faraday-Microscopy

Indicator film technique: stray field imaging

Grain oriented electrical steel:



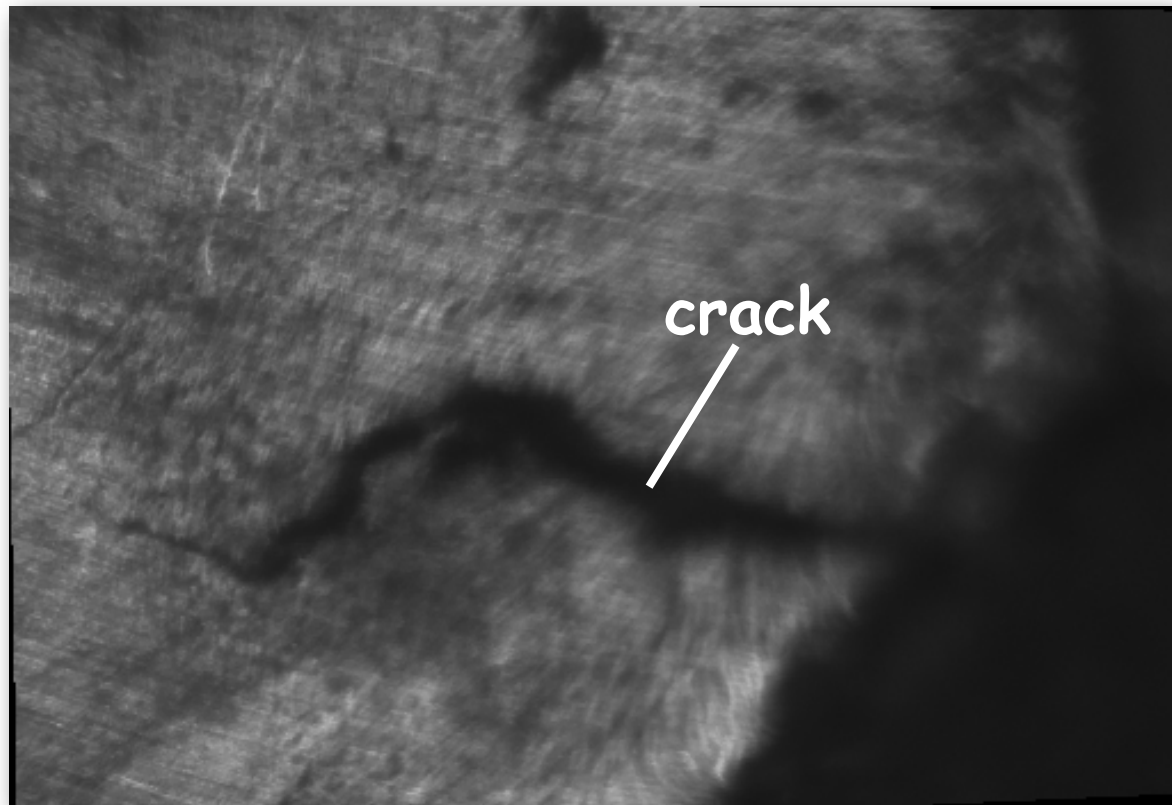
MOIF contrast (with insulation coating)

Magnetic Pole contrast at grain boundaries

4. Magneto-optical Faraday-Microscopy

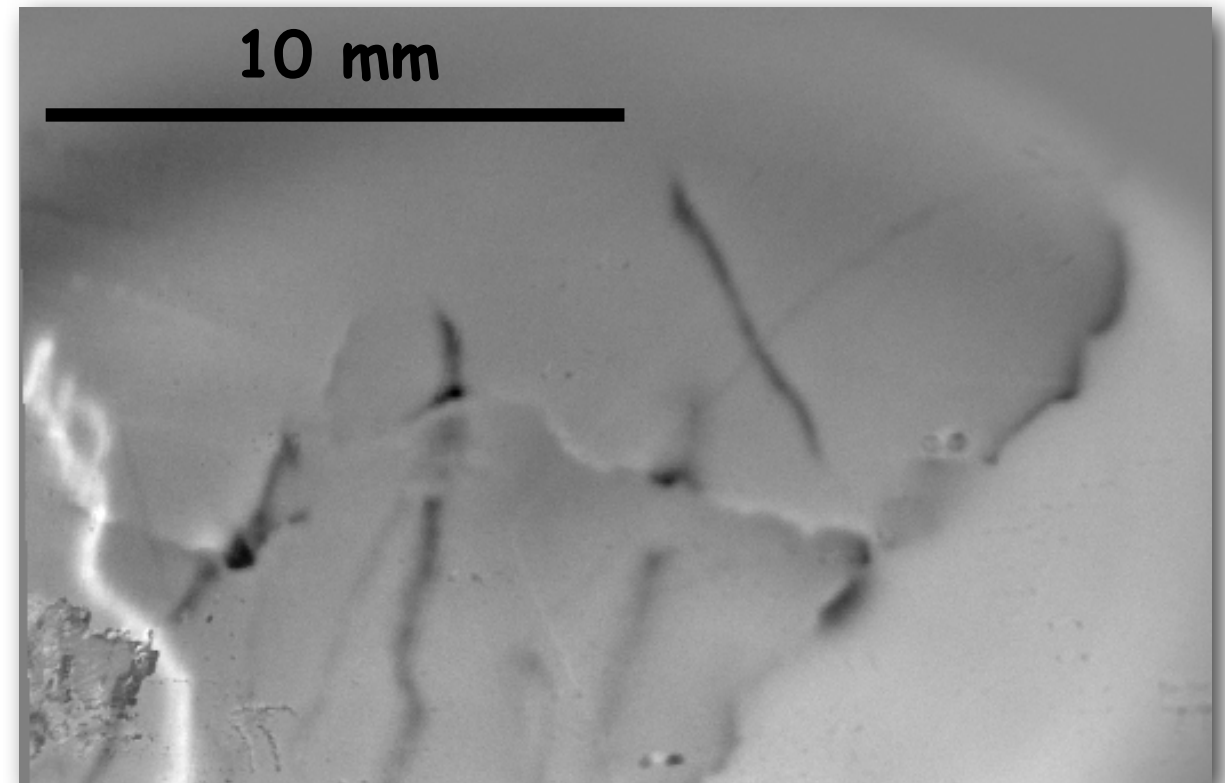
Indicator film technique: stray field imaging

Direct image of surface



Rail track with crack

MOIF image



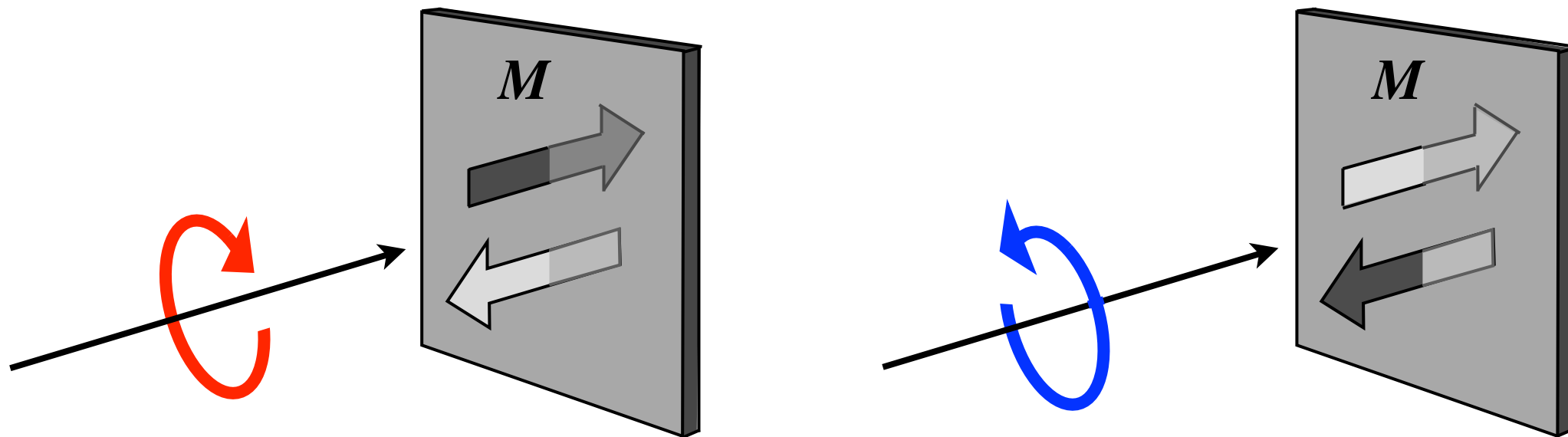
Imaging of defects for non-destructive testing

Together with G. Y. Tian, Chengdu

5. X-Ray Spectroscopy

X-Ray Magnetic Circular Dichroism (XMCD)

XMCD: Absorption of circularly polarized x-rays depends on orientation of magnetization M with respect to helicity of the X-rays, change of sign by reversing M



5. X-Ray Spectroscopy

X-Ray Magnetic Circular Dichroism (XMCD)

XMCD: Absorption of circularly polarized x-rays depends on orientation of magnetization \mathbf{M} with respect to helicity of the X-rays, change of sign by reversing \mathbf{M}

Physical origin: If energy of absorbed photon exceeds binding energy of an inner core level (e.g. $p_{1/2}$ and $p_{2/3}$ states, separated by spin-orbit coupling)

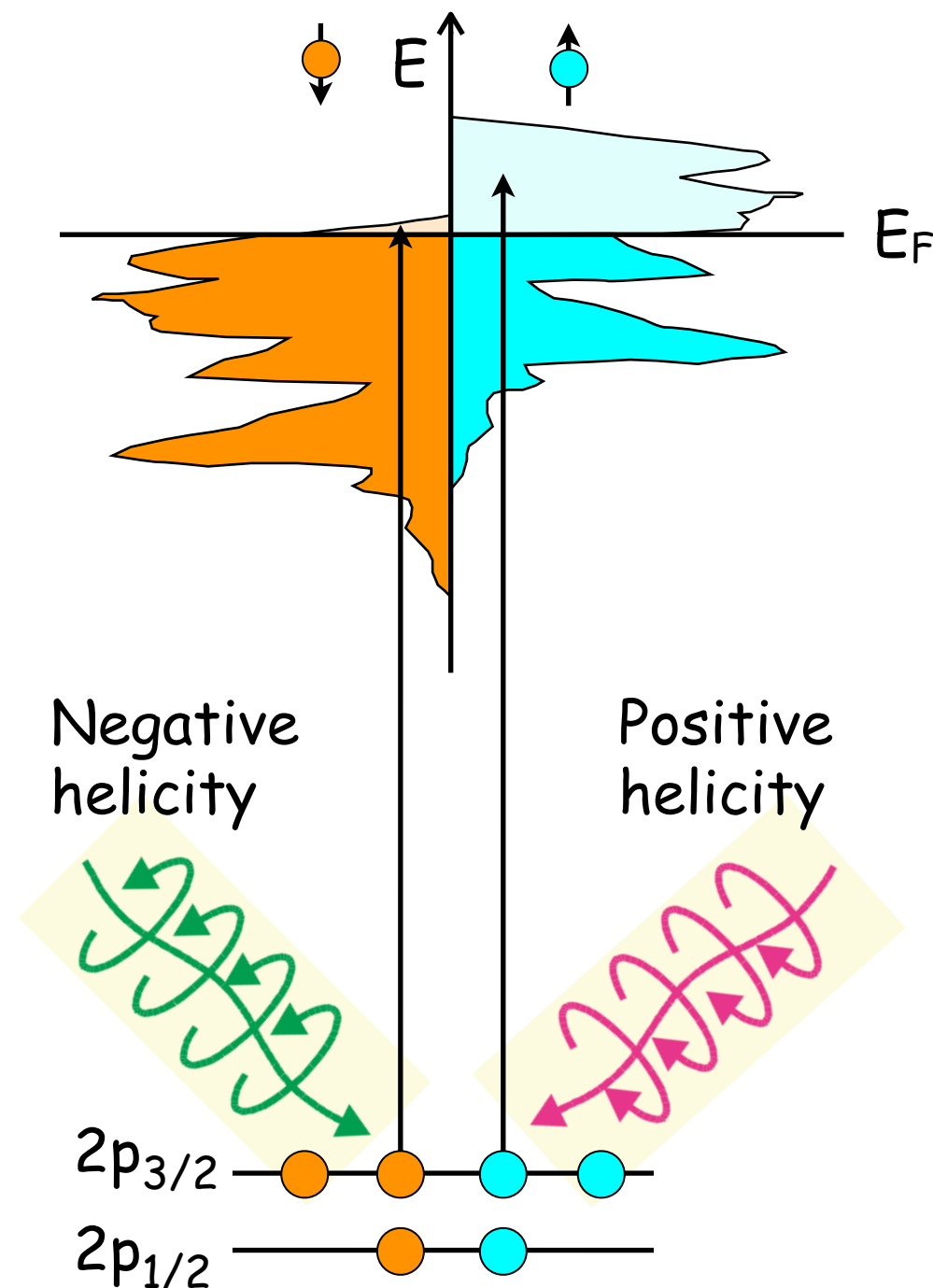
→ Transition into unoccupied spin-split states above Fermi level (e.g. into 3d band)

Initial states are well defined inner-core levels

→ **XMCD is element selective**

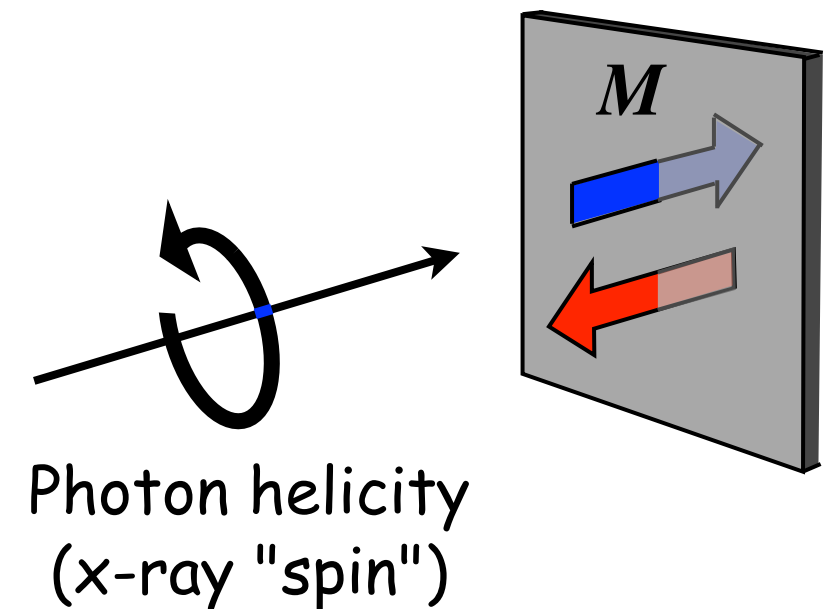
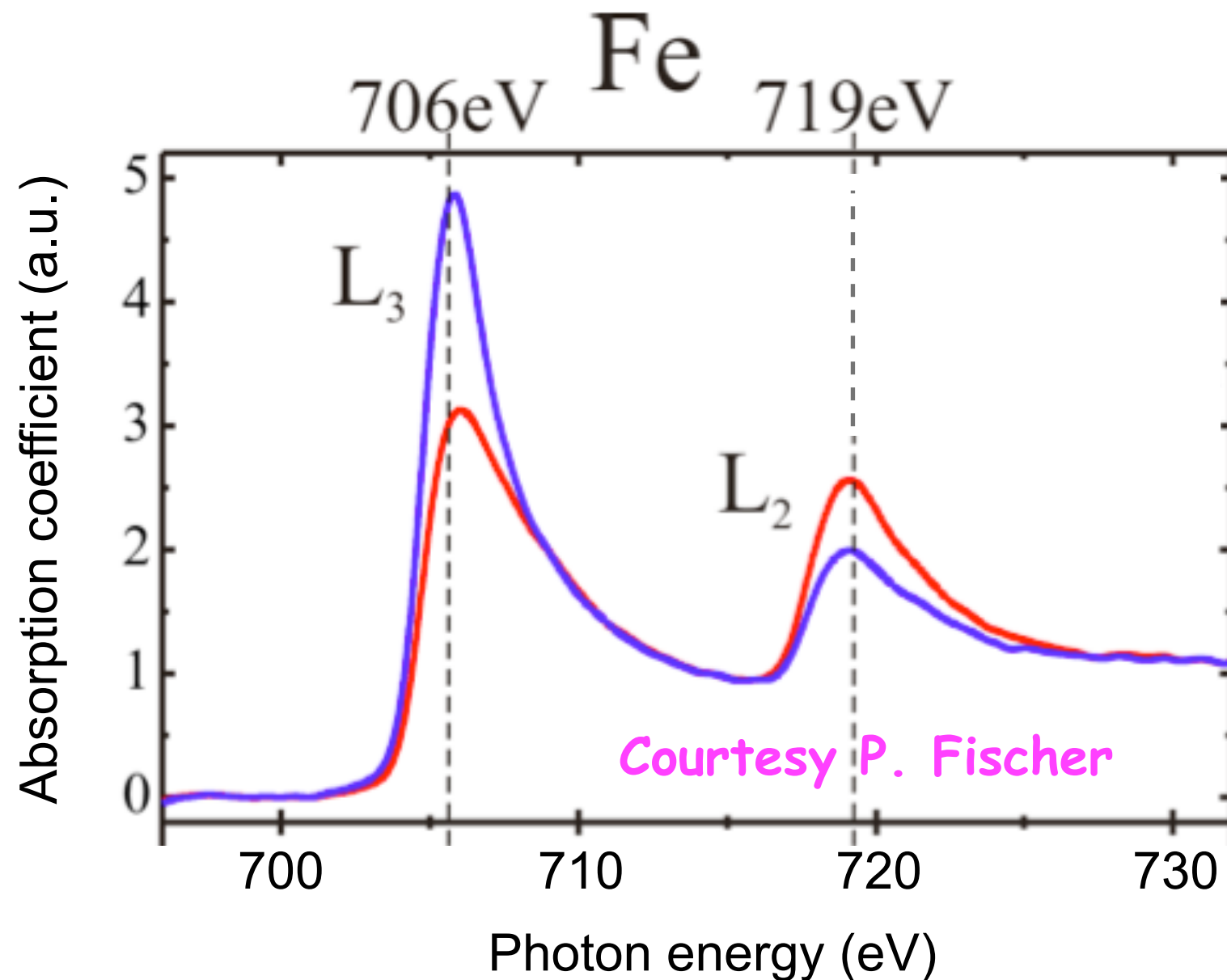
Fermi's golden rule: transition probability of absorption process is related to density of unoccupied states, which are different for minority and majority bands due to exchange interaction

→ X-MCD signal is proportional to magnetic moment of absorbing atom → **Sensing of magnetization of sample**



5. X-Ray Spectroscopy

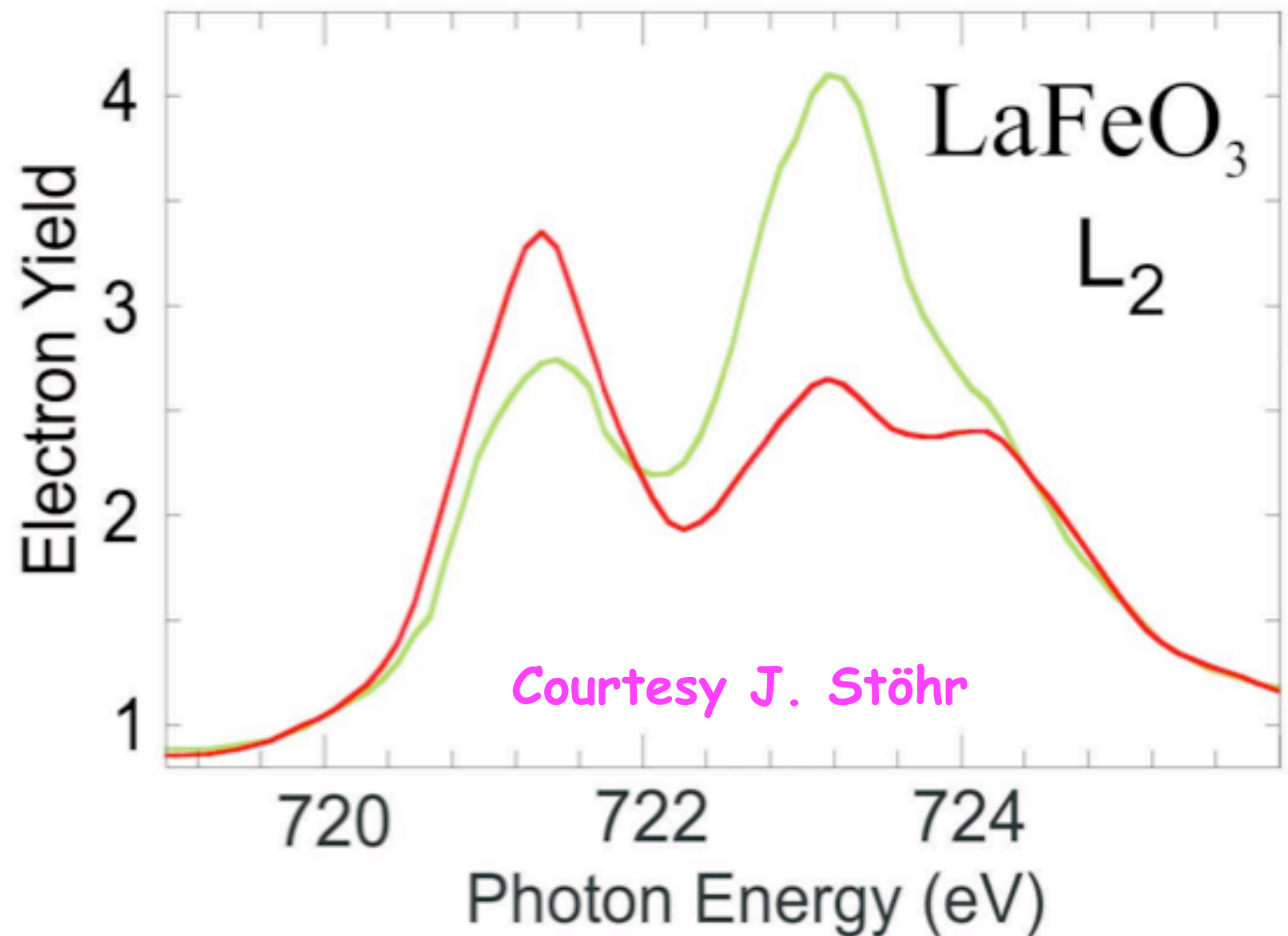
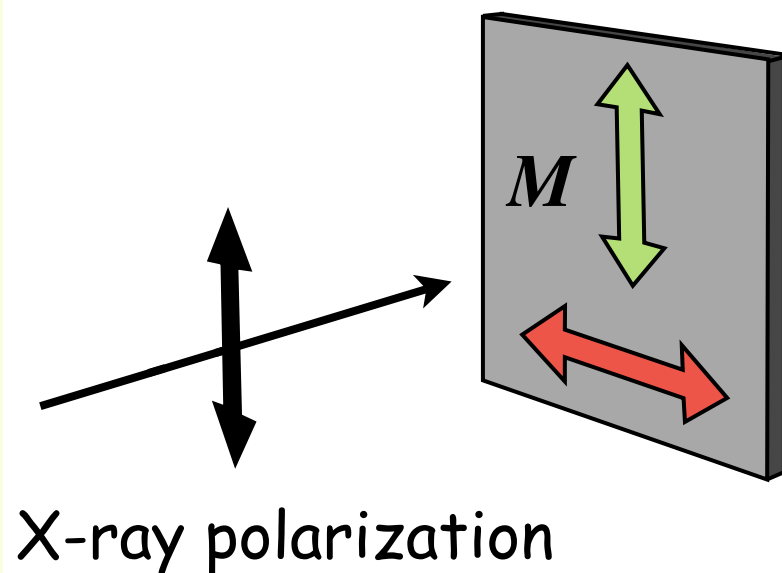
X-Ray Magnetic Circular Dichroism (XMCD)



XMCD effect is localized around L_2 (transition from $2p_{1/2}$ core level to unoccupied 3d states) and L_3 (transition from $2p_{3/2}$ core level) absorption edges, which are separated by the spin-orbit coupling

5. X-Ray Spectroscopy

X-Ray Magnetic Linear Dichroism (XMLD)

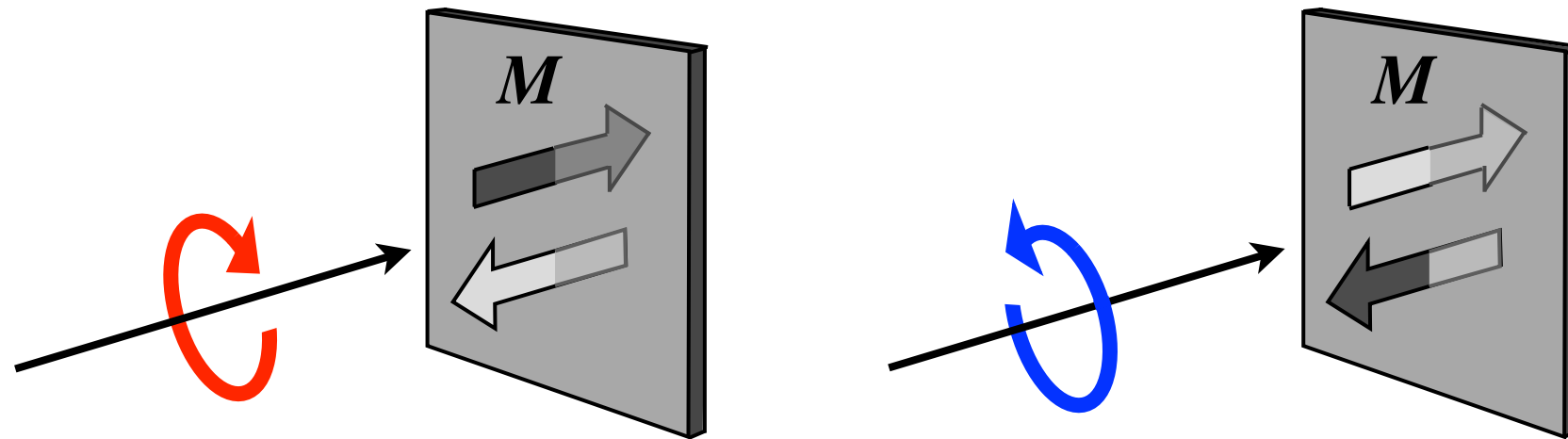


Oxides are usually antiferromagnetic: studied with X-Ray Magnetic **Linear** Dichroism (XMLD) spectroscopy

The XMLD effect is large only in cases where the absorption edge exhibits multiplet structure

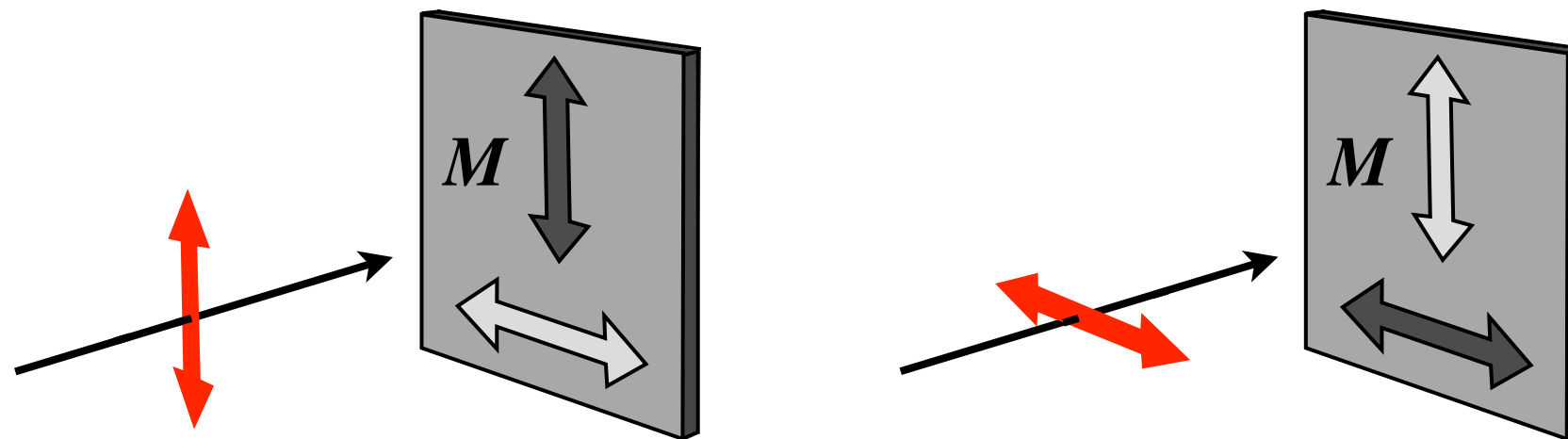
5. X-Ray Spectroscopy

XMCD



XMCD detects the difference in absorption for the projection of the sample's magnetization onto the propagation direction of circularly polarized X rays. XMCD distinguishes between magnetization parallel and antiparallel to the light **propagation** direction

XMLD

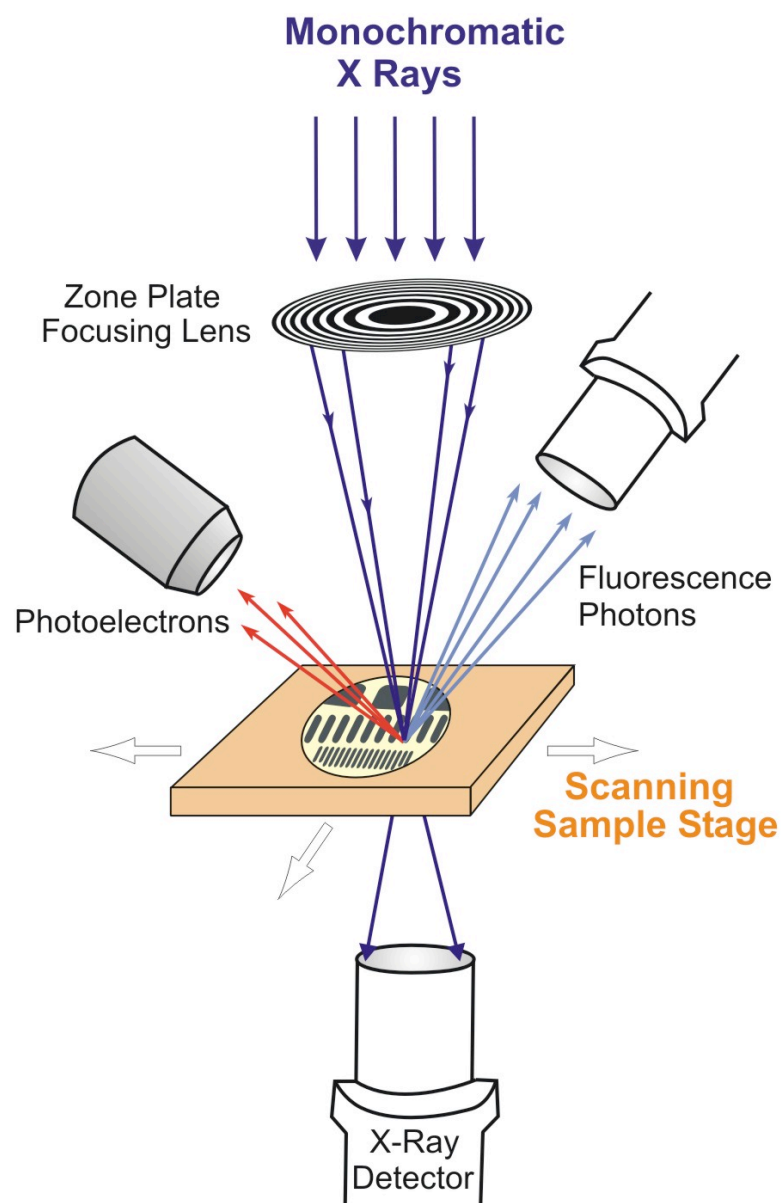


XMLD detects the difference in absorption of the axis of magnetization aligned parallel or perpendicular to the E-field of the X-rays. XMLD distinguishes between the sample being magnetized parallel or perpendicular to the light **polarization** direction

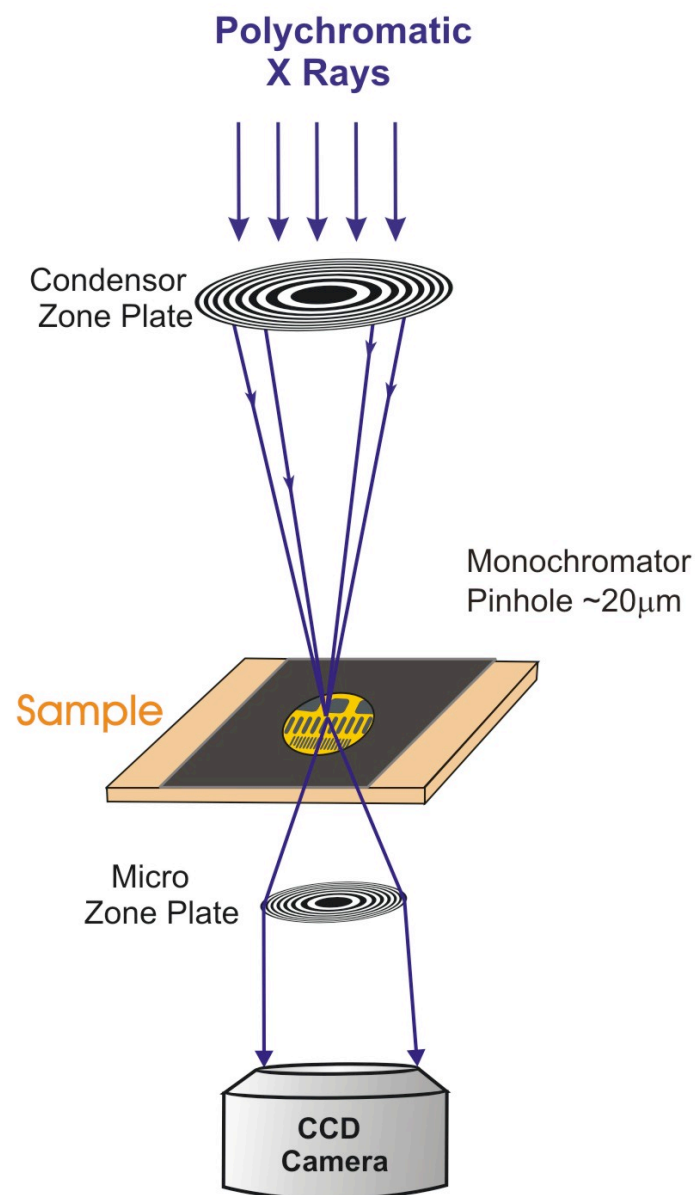
5. X-Ray Spectroscopy

X-Ray microscopy methods

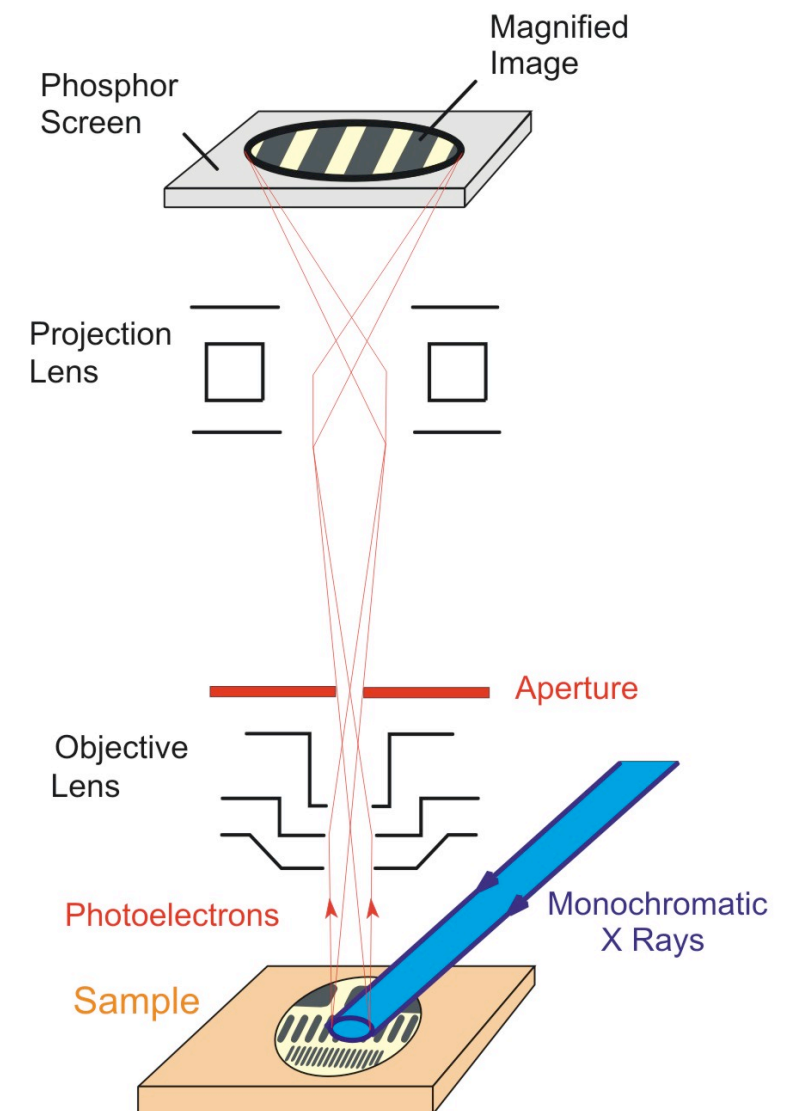
Scanning transmission X-ray Microscopy



Transmission X-ray Microscopy



X-ray Photoemission Electron Microscopy (XPEEM)

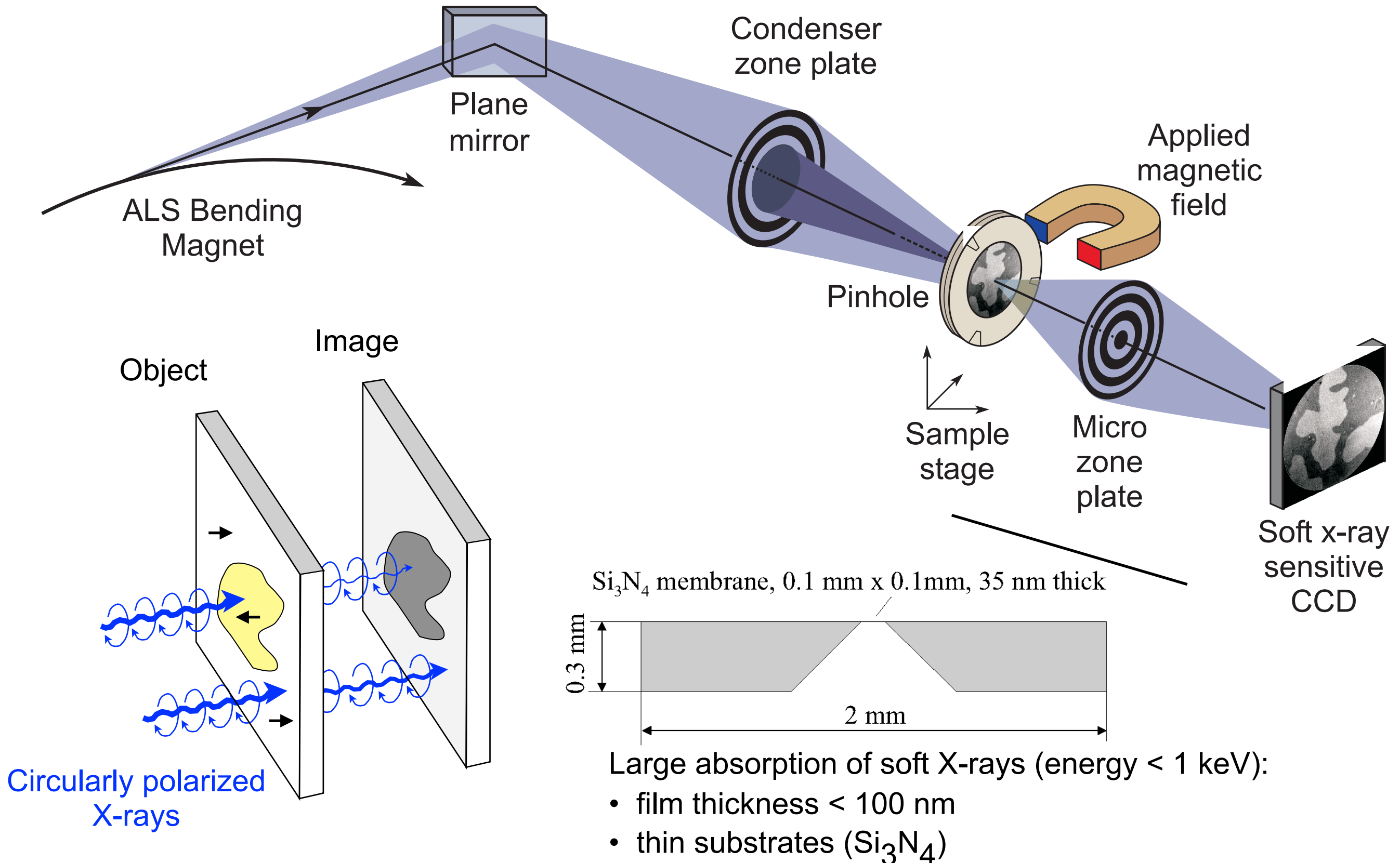


Resolution in 20 nm range

Courtesy J. Stöhr

5. X-Ray Spectroscopy

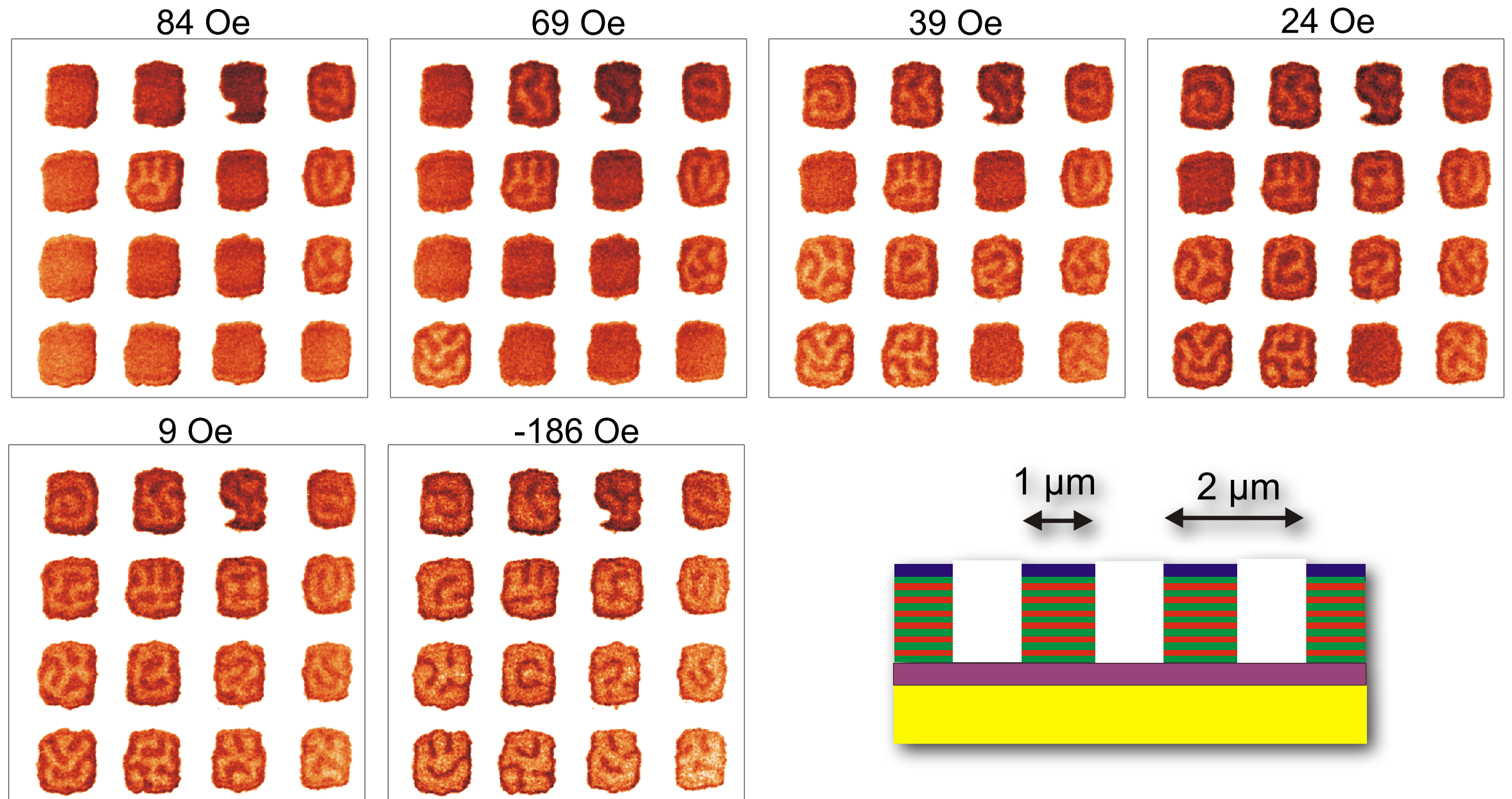
5.1 Transmission X-Ray microscopy



5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy

Switching of Fe/Gd multilayered dots

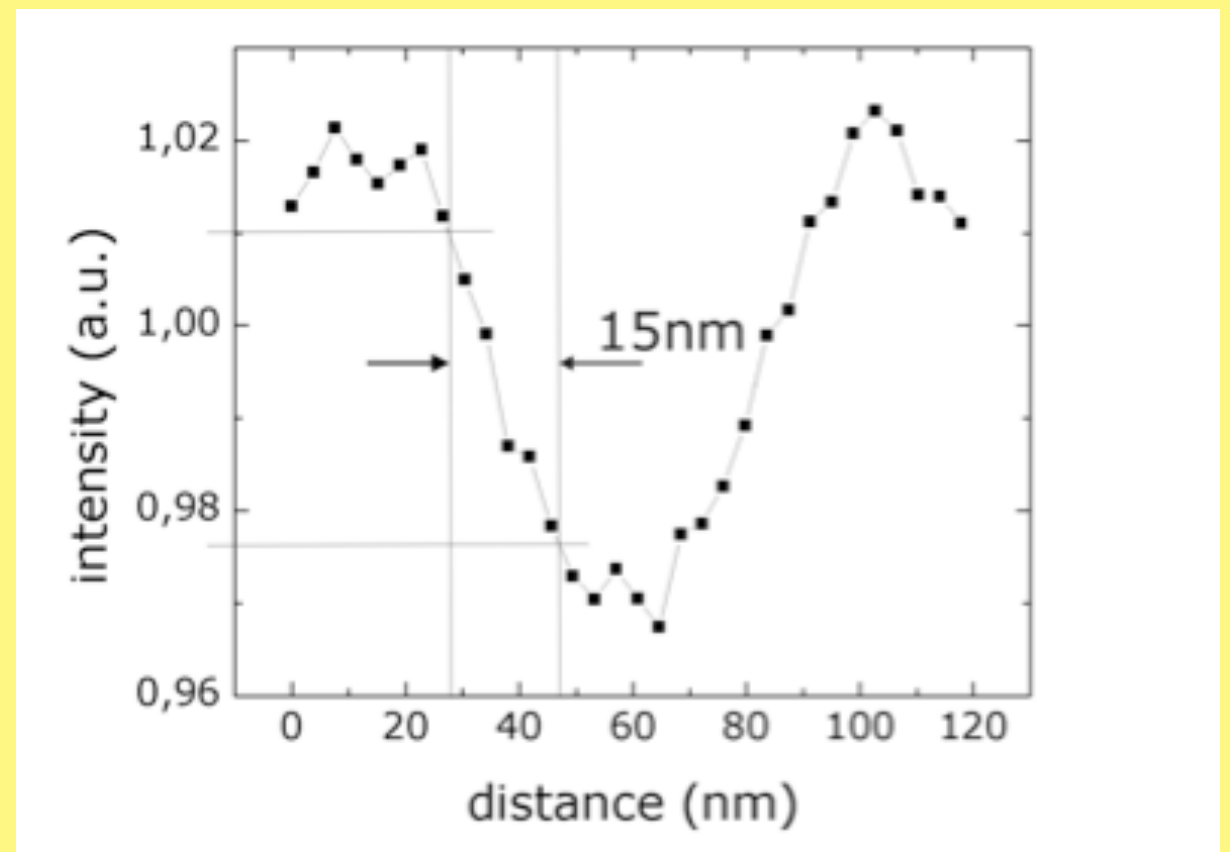
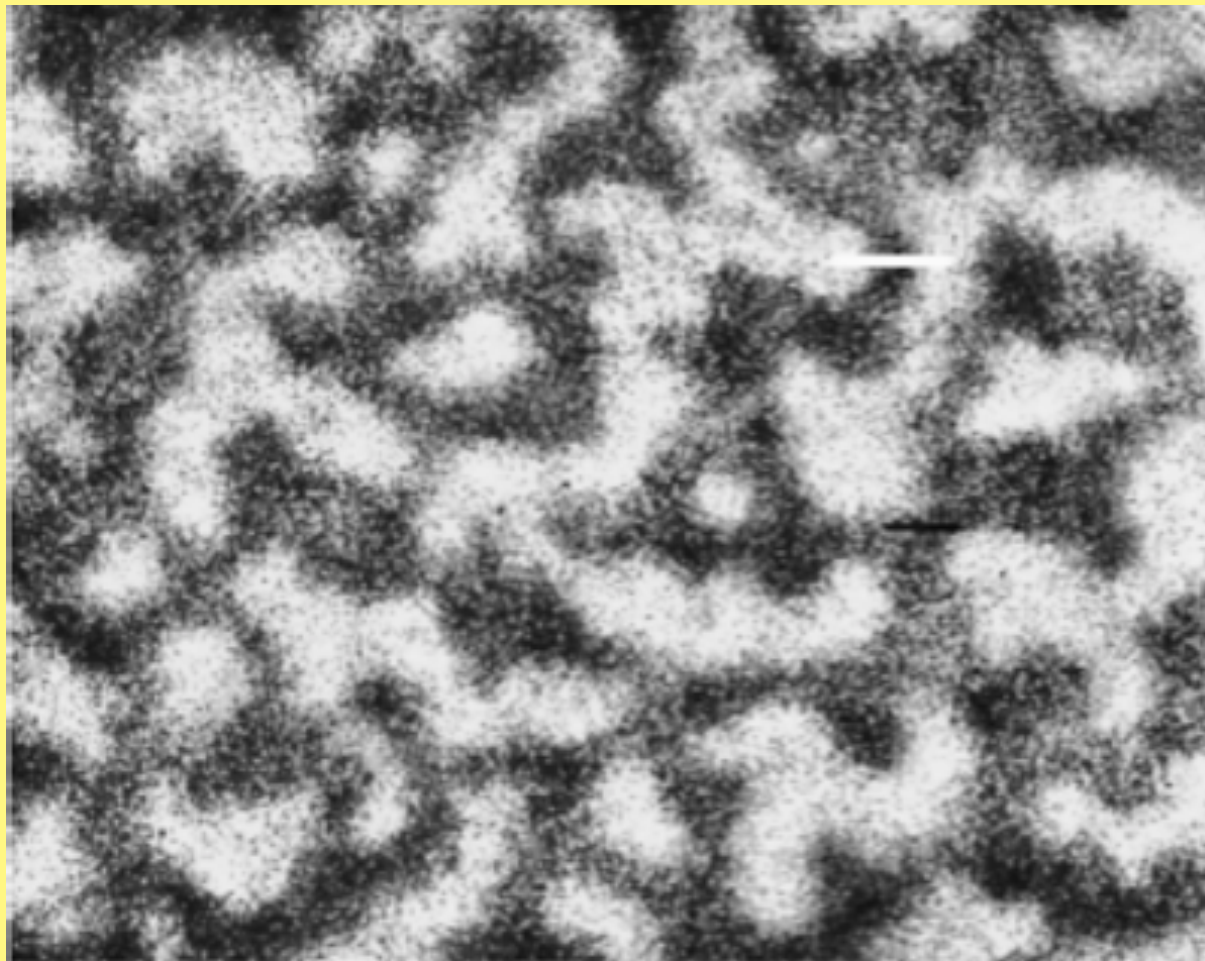


Courtesy P. Fischer, Th. Eimüller

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy

Intensity profile across the boundary of a magnetic domain in an amorphous GdFe layer showing a lateral resolution of less than 15 nm

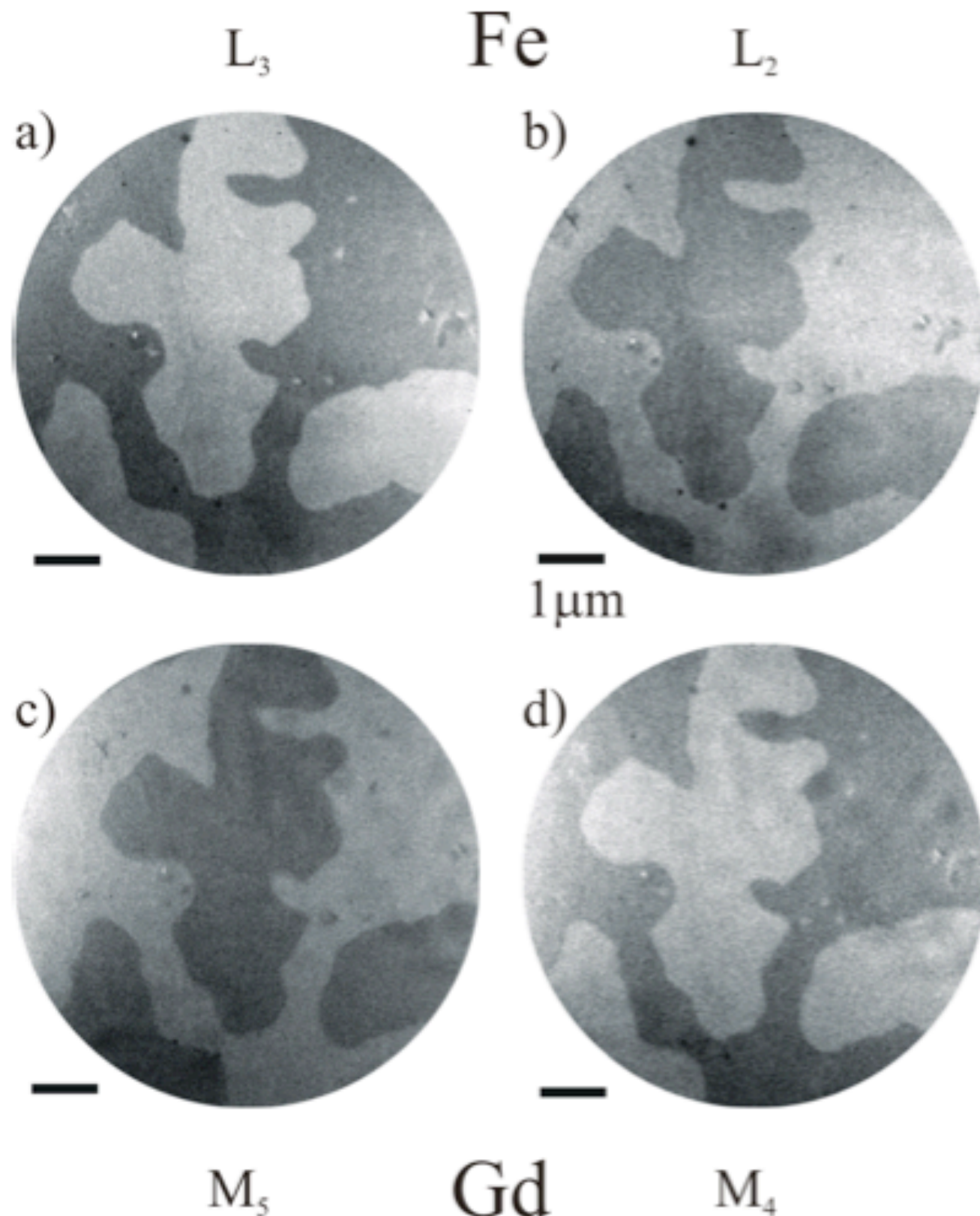


Courtesy P. Fischer

Courtesy P. Fischer, Th. Eimüller

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy



- MTXM images of an amorphous $Gd_{25}Fe_{75}$ layer recorded at the spin-orbit-coupled Fe L_3 (a) and L_2 (b) as well as at the Gd M_5 (c) and M_4 (d) absorption edges
- Contrast inversion: magnetic moments on Cd and Fe couple antiparallel

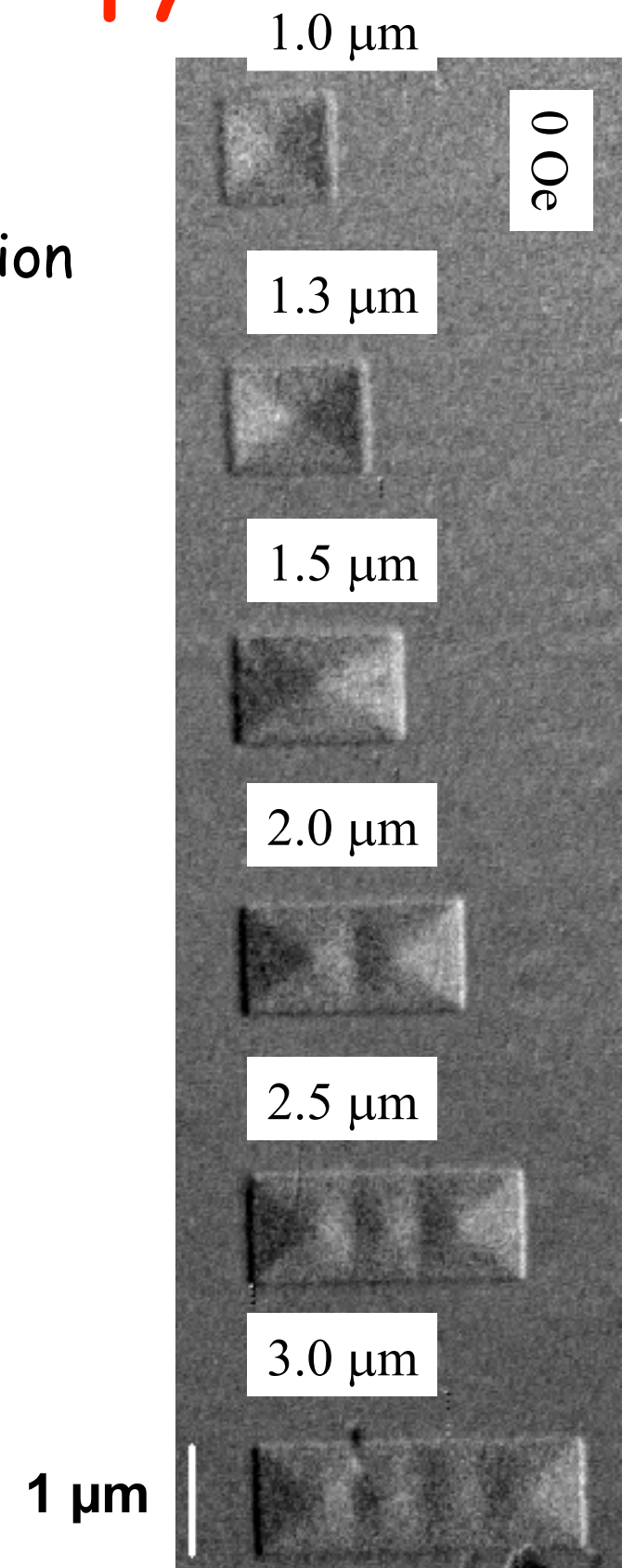
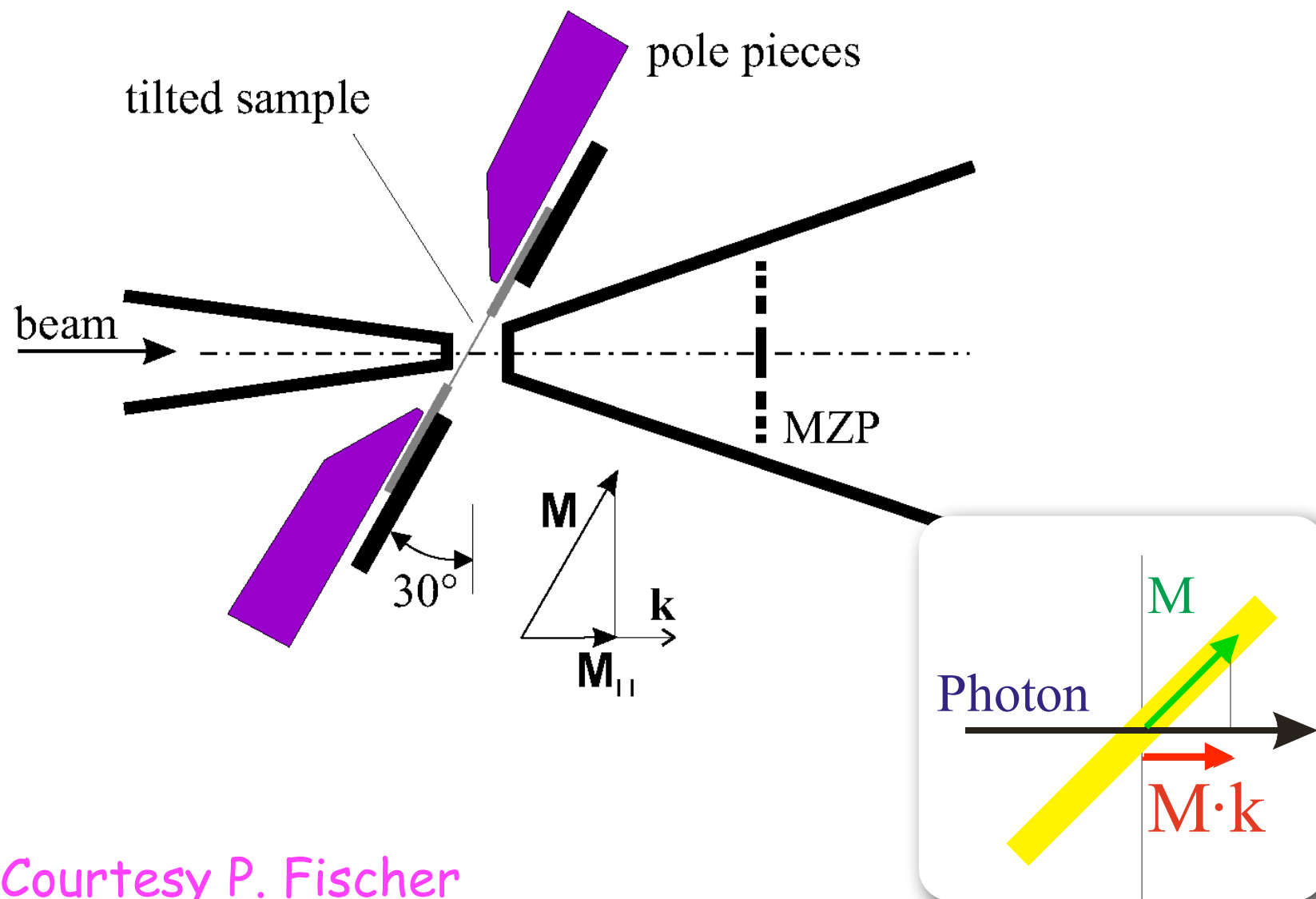
Courtesy P. Fischer

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy

In-plane imaging

Dichroic contrast: given by projection of \mathbf{M} on photon propagation direction. In-plane imaging by tilting the sample (30°):

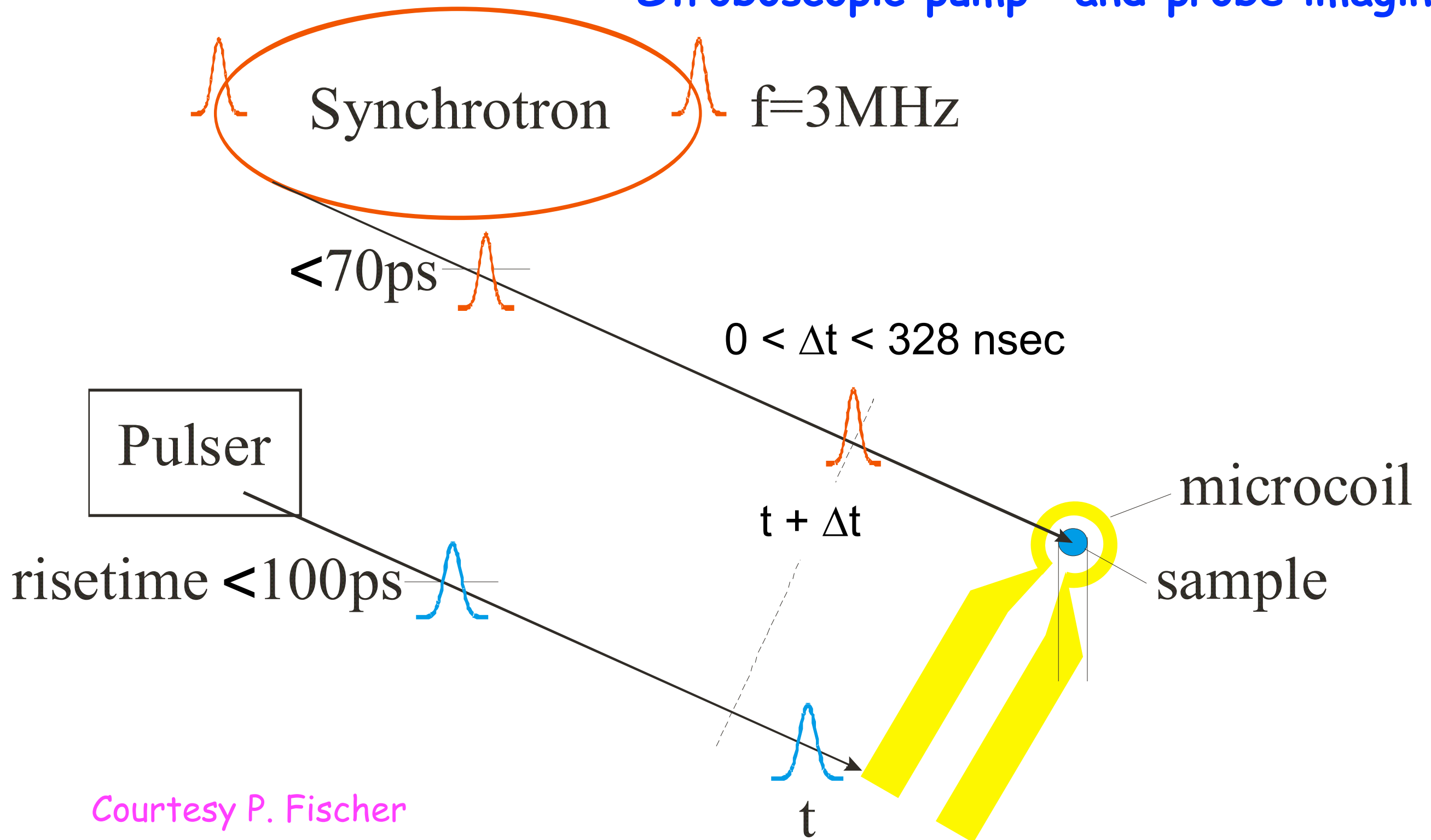


Courtesy P. Fischer

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy

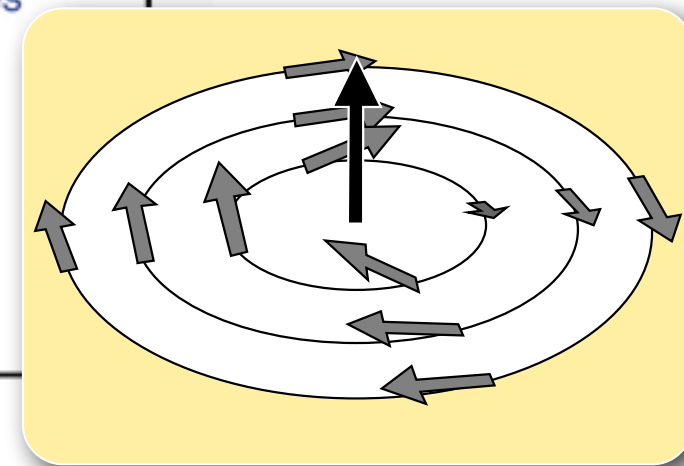
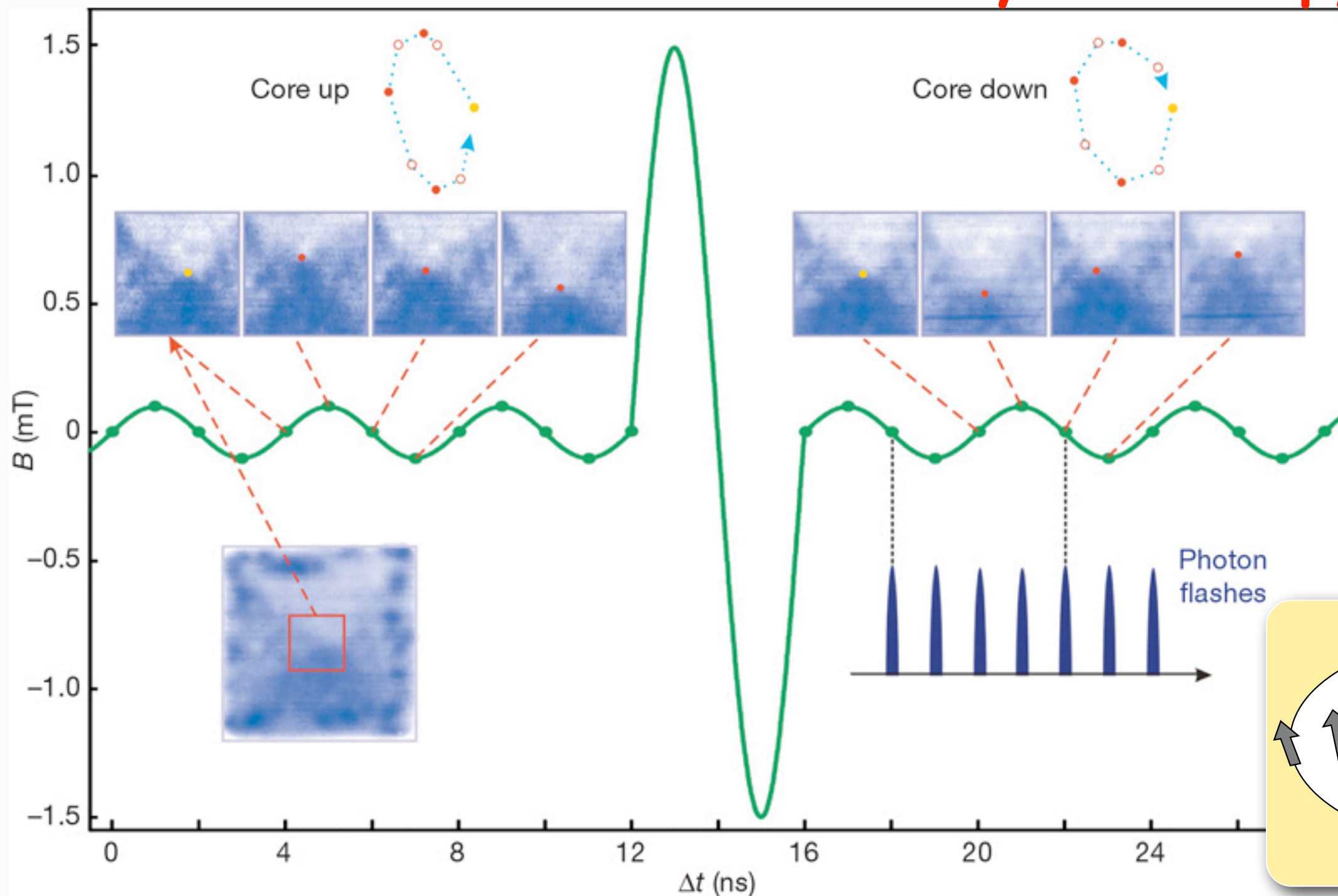
Stroboscopic pump- and probe imaging



Courtesy P. Fischer

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy



Excitation of vortex structure in ac field (frequency 250 MHz, amplitude 0.1 mT). After a 4 ns 'single period' burst (amplitude 1.5 mT) the vortex core polarity is inverted

B. Van Waeyenberge et al., Nature 444, 461 (2006)

5. X-Ray Spectroscopy

5.1 Transmission X-Ray microscopy

Advantages

- Lateral resolution at approx. 15 nm
- Imaging in applied magnetic fields (currently some kOe)
- Quantitative (contrast proportional to M)
- Probing of in- and out-of-plane magnetization
- Time resolution in the sub-ns regime
- Element specific imaging
- High sensitivity to few nm layers (for Fe: < 2 nm)

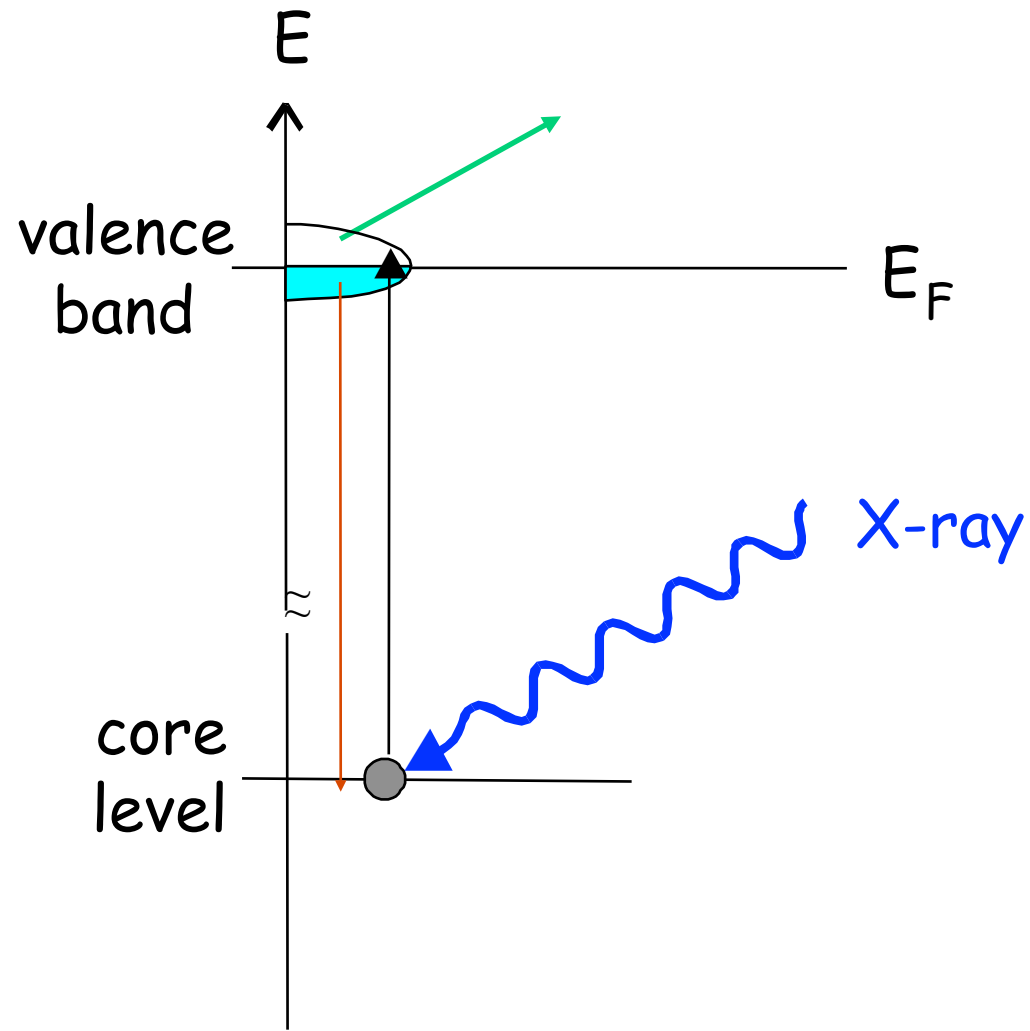
Disadvantages

- Sample must be transparent ($d < 100$ nm)
- Thin substrates
- Synchrotron radiation necessary

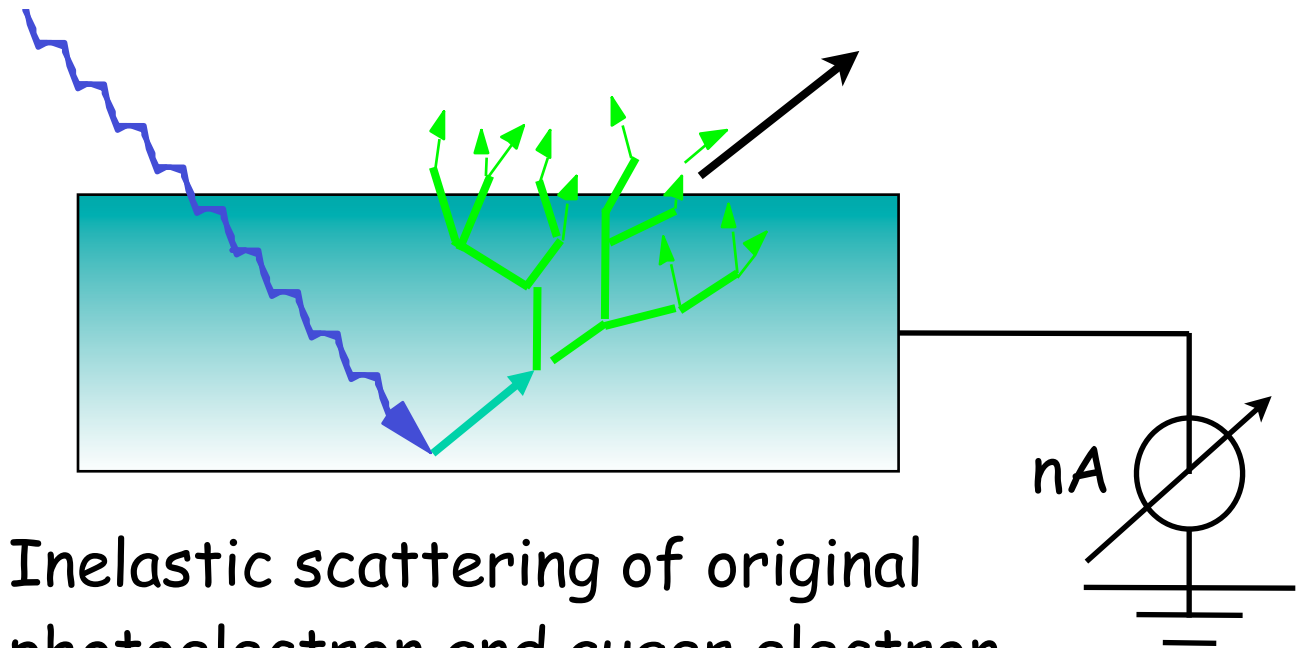
-
- Excitation of core electron into empty valence state by incoming X-ray
 - Recombination (e.g.) by Auger decay

5. X-Ray Spectroscopy

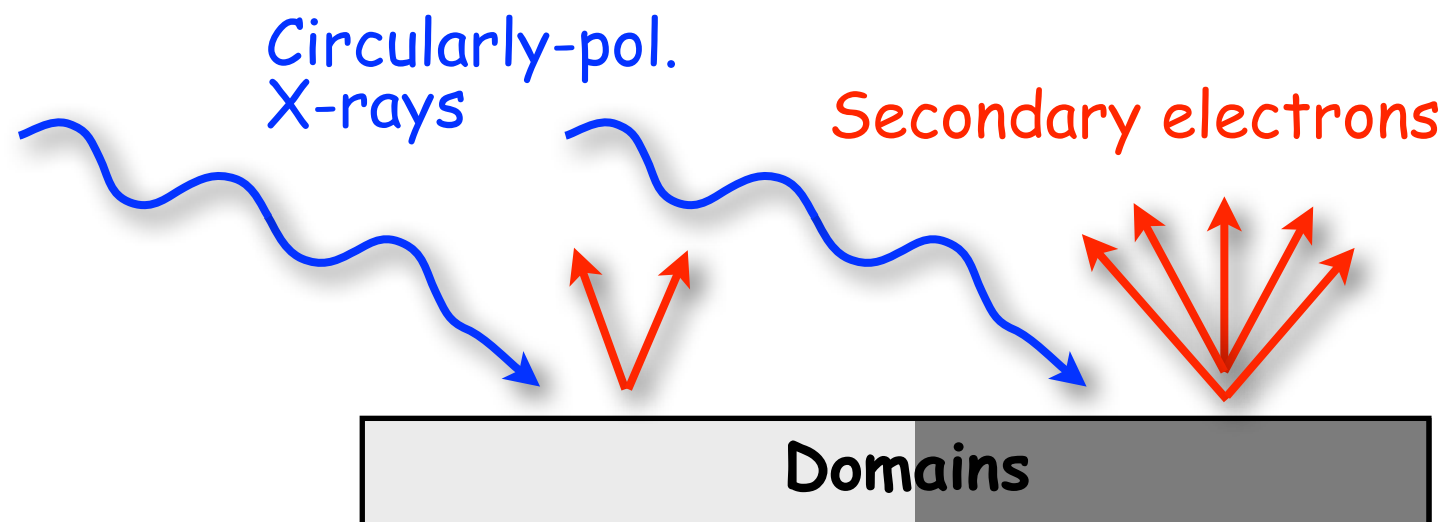
5.2 X-ray Photoemission Electron Microscopy (X-PEEM)



- Excitation of core electron into empty valence state by incoming X-ray
- Recombination (e.g.) by Auger decay

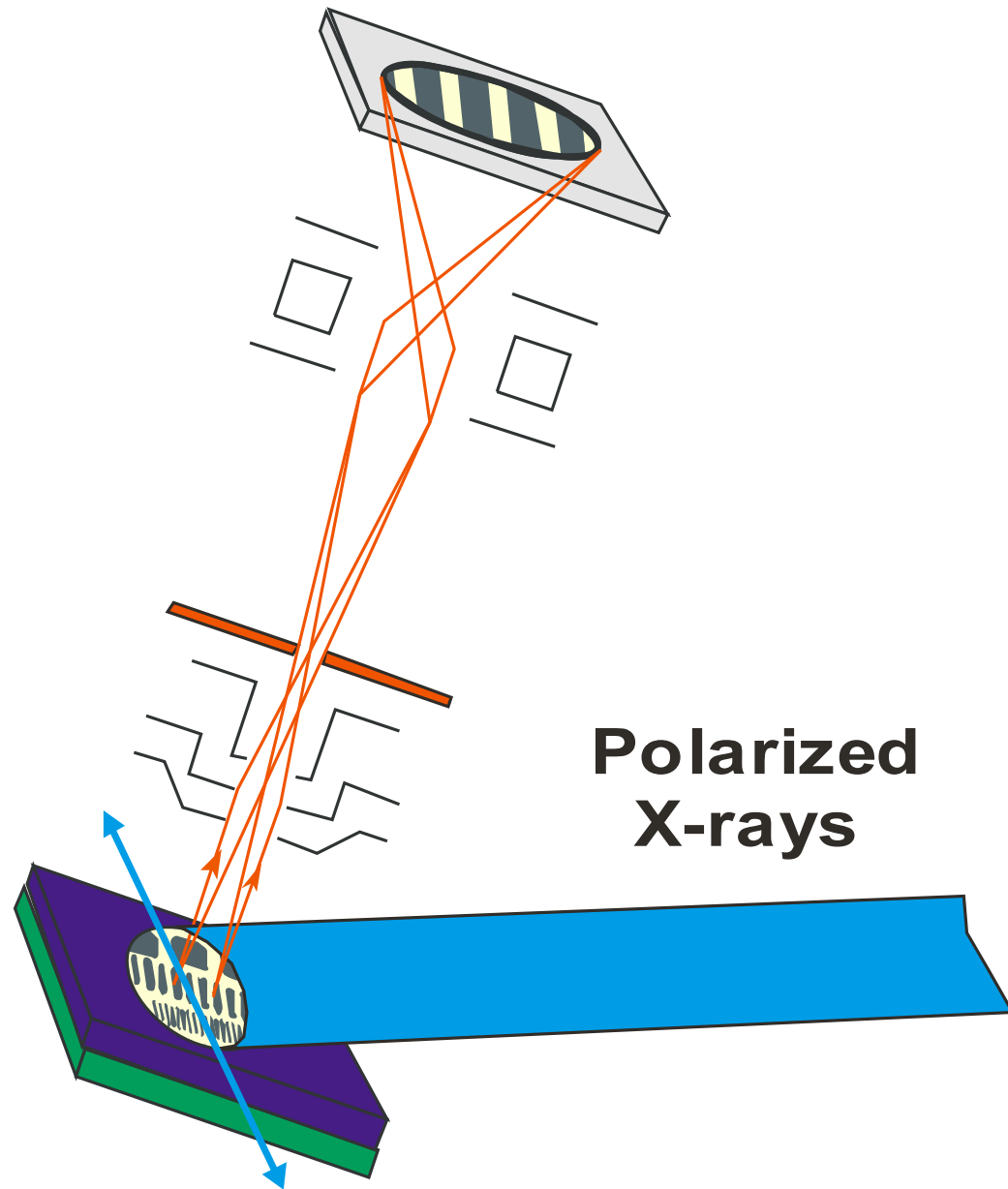


- Inelastic scattering of original photoelectron and Auger electron leads to emission of secondary electrons
- Electron yield \sim X-ray absorption coefficient
- Probing depth \sim electron escape length $\exp(-\lambda t)$ with $\lambda \sim 2 \text{ nm}$



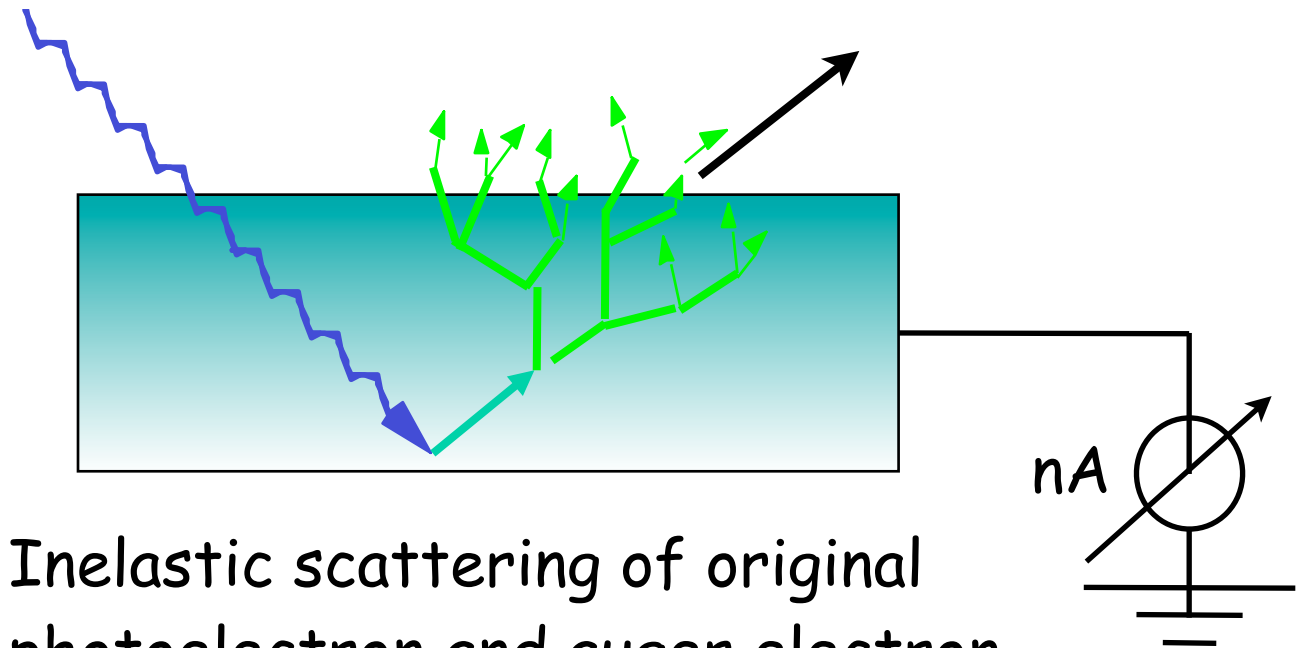
5. X-Ray Spectroscopy

5.2 X-ray Photoemission Electron Microscopy (X-PEEM)

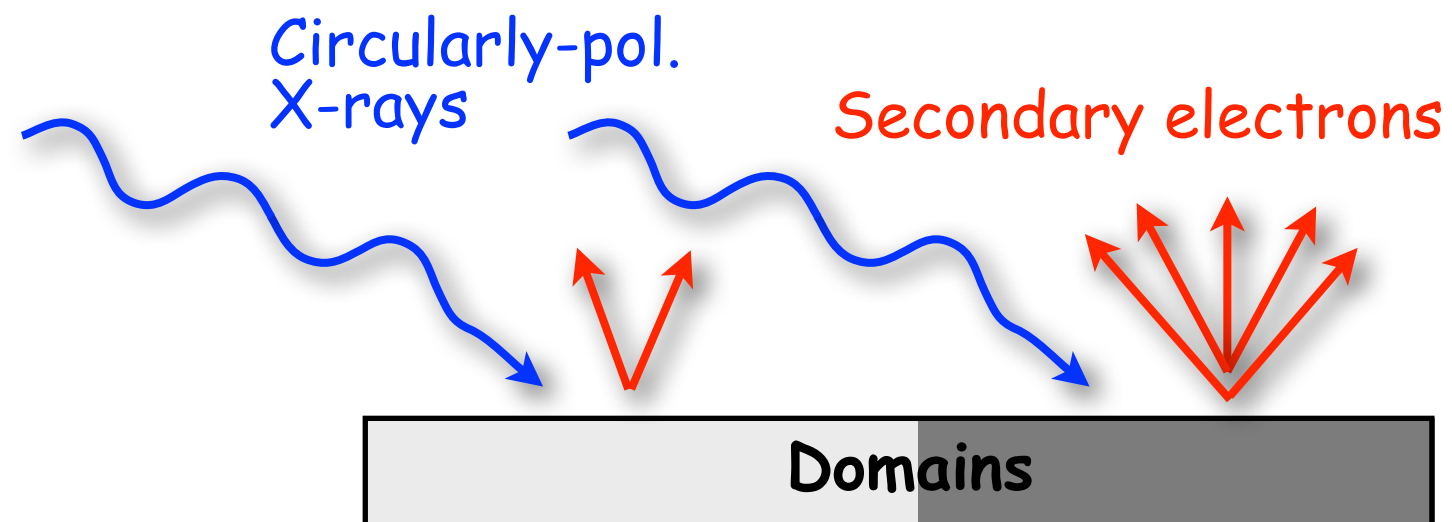


- Full Field Imaging
- 20 - 50 nm Resolution
- Linear and circular polarization

Courtesy H.Ohldag

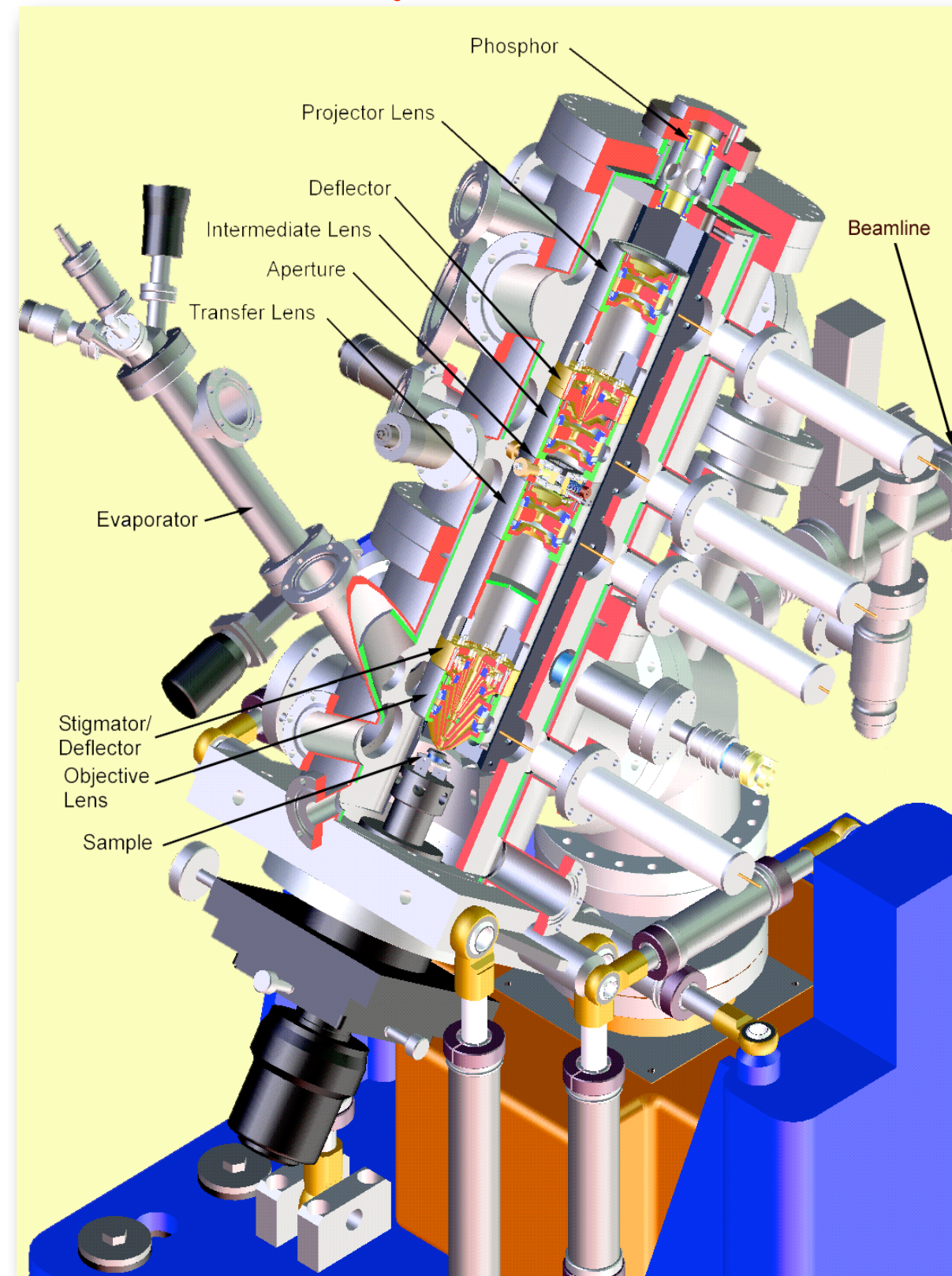


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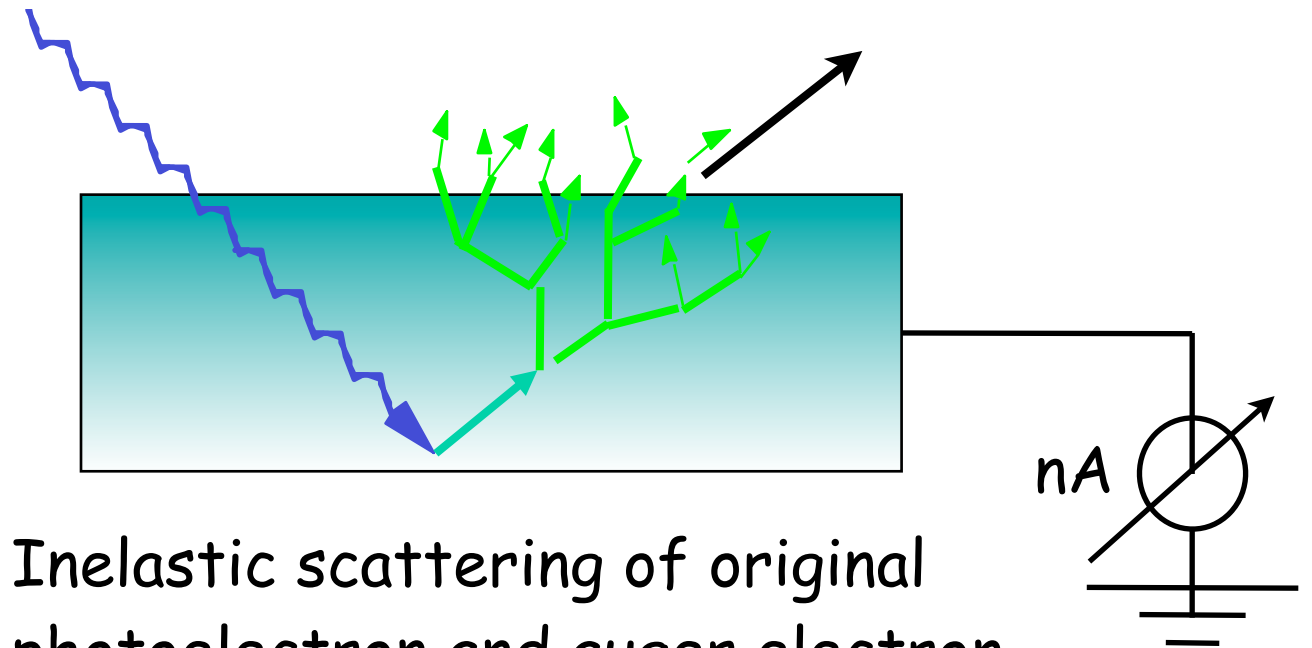


5. X-Ray Spectroscopy

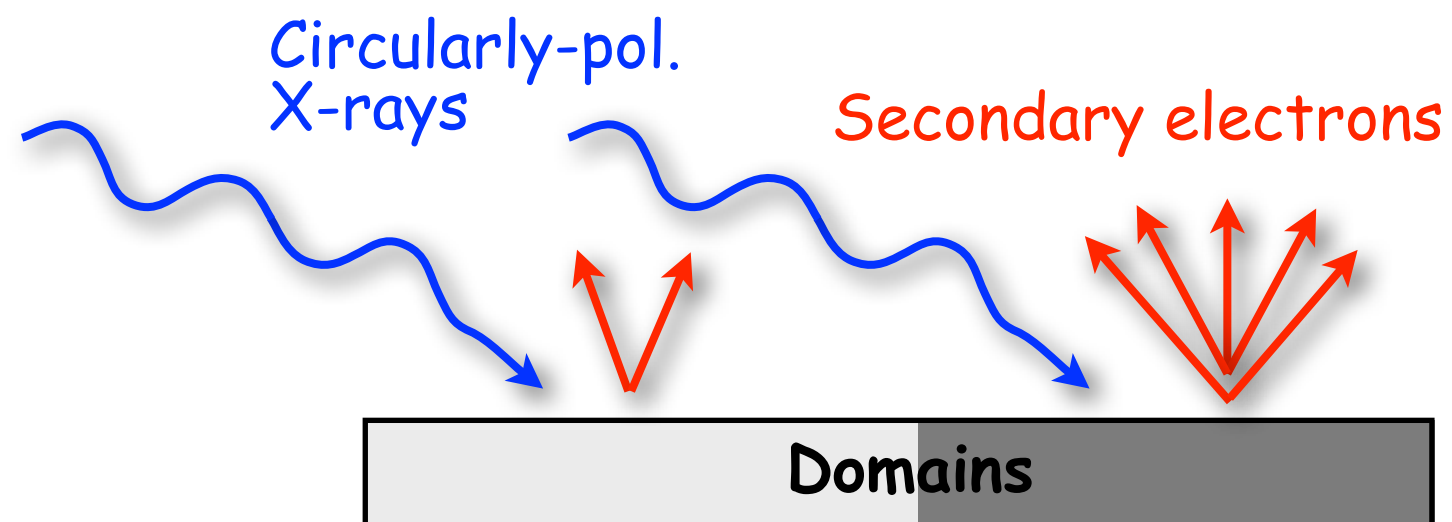
5.2 X-ray Photoemission Electron Microscopy (X-PEEM)



Courtesy H.Ohldag

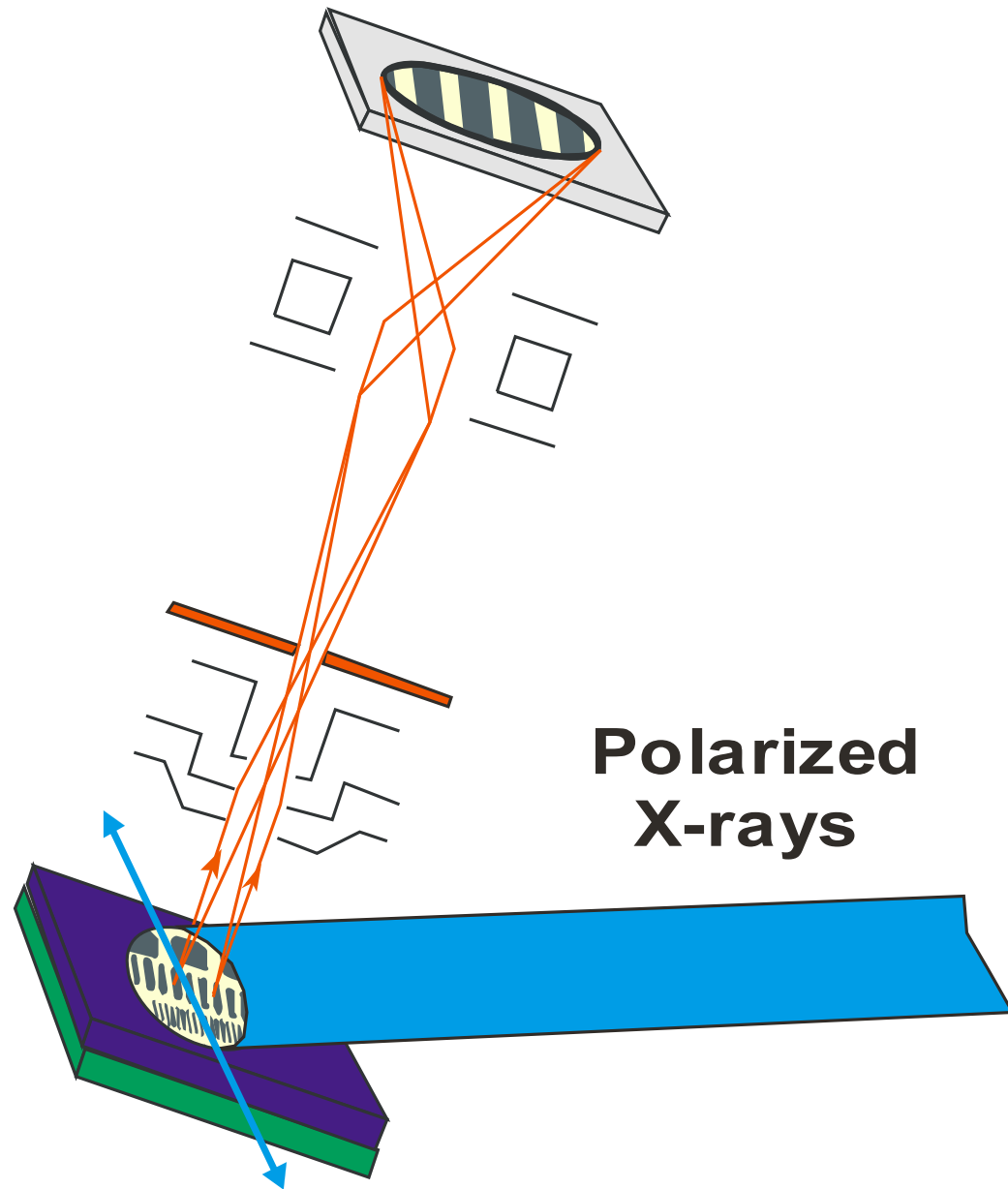


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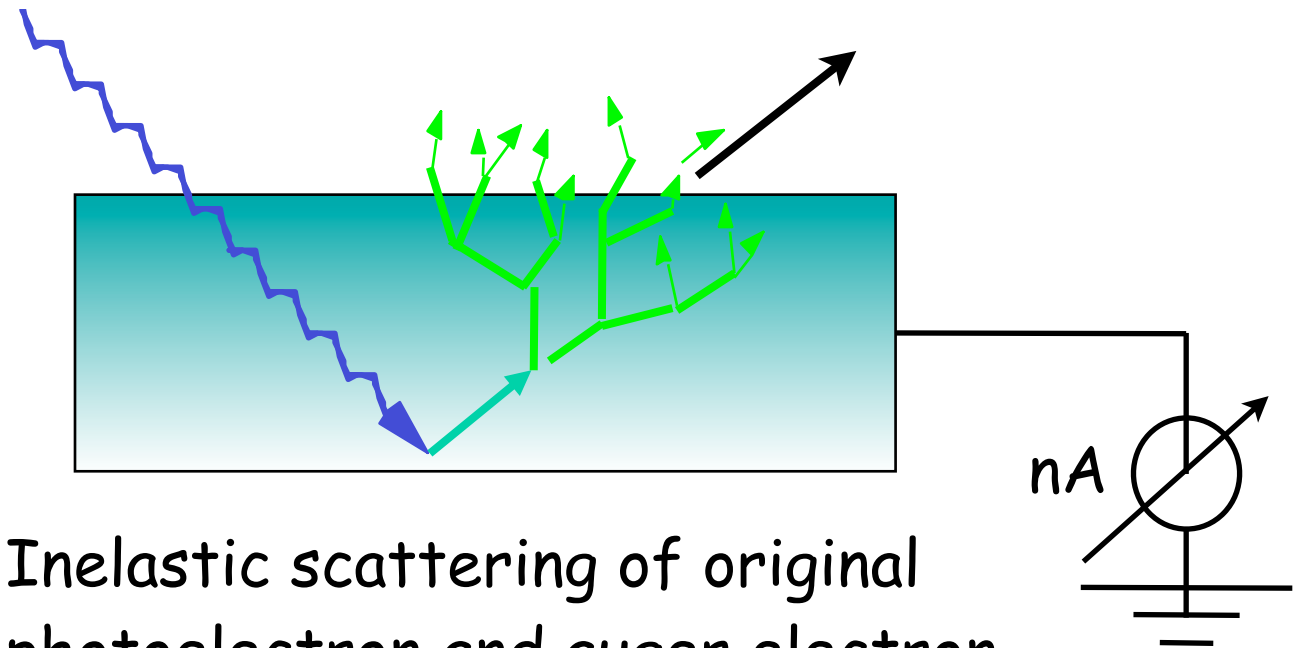
5. X-Ray Spectroscopy

5.2 X-ray Photoemission Electron Microscopy (X-PEEM)

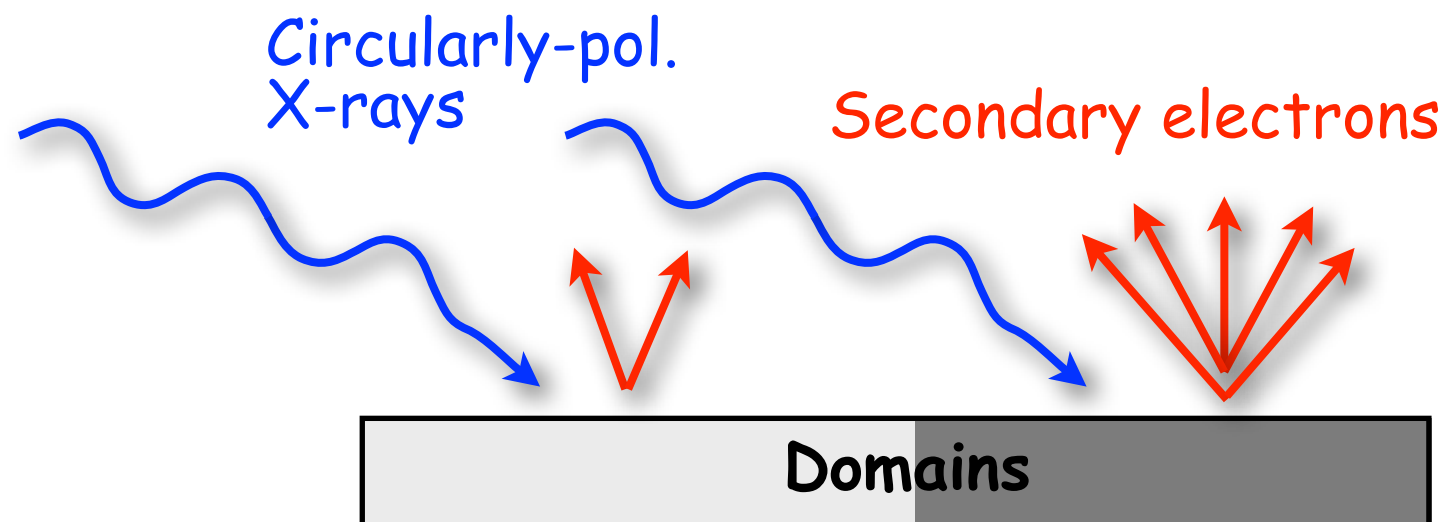


- Full Field Imaging
- 20 - 50 nm Resolution
- Linear and circular polarization

Courtesy H.Ohldag



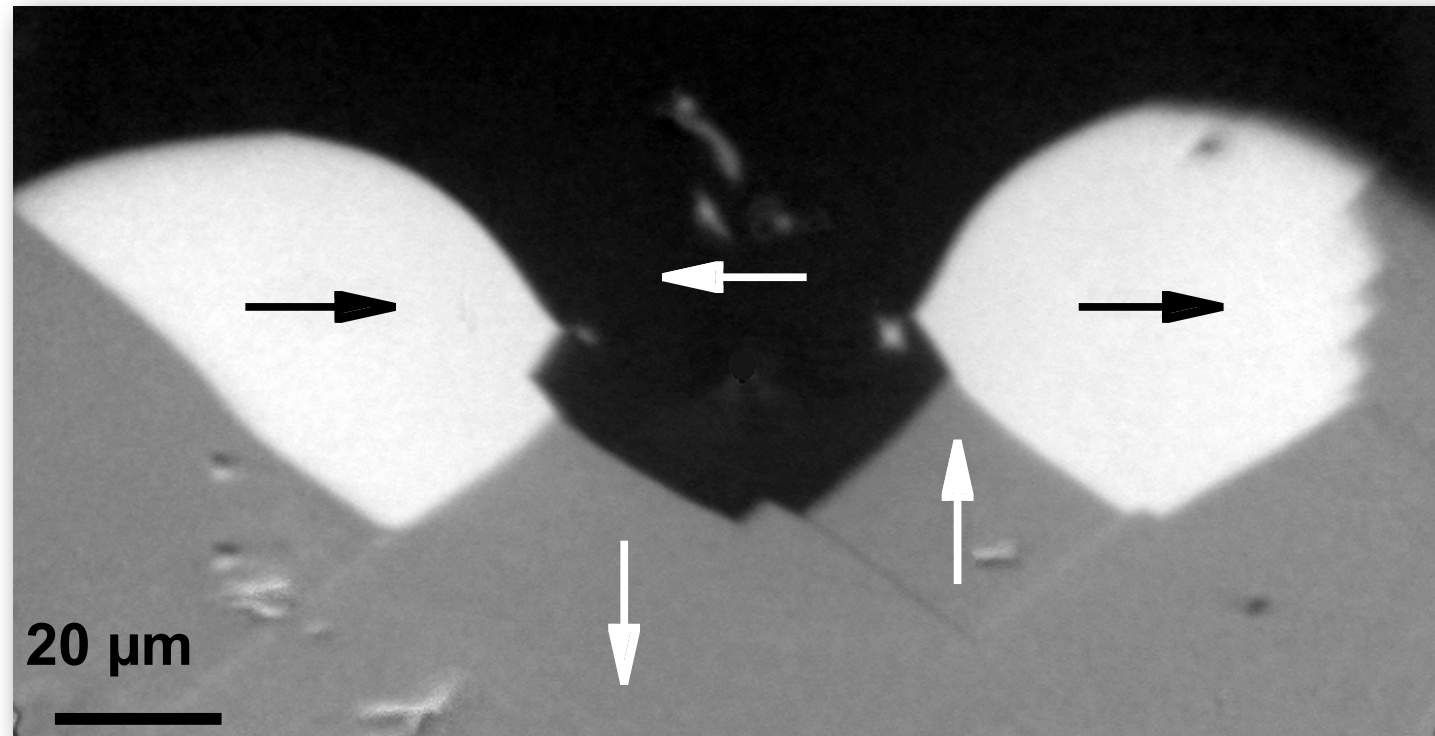
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5. X-Ray Spectroscopy

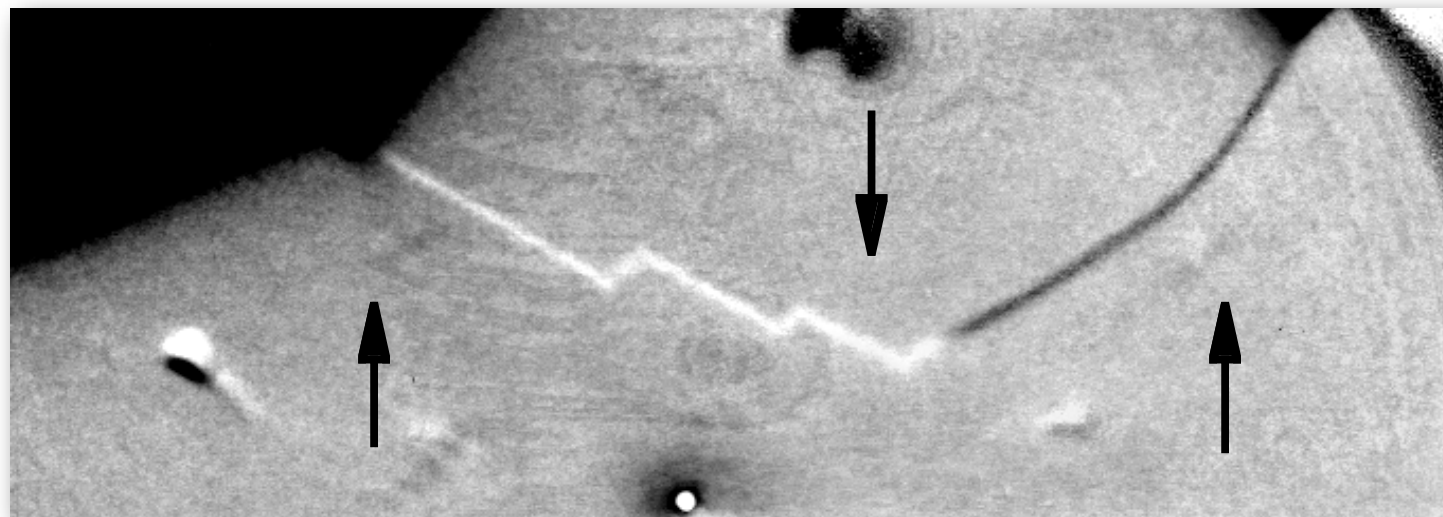
5.2 X-ray Photoemission Electron Microscopy (X-PEEM)

Iron whisker



Excitation by circularly-polarized electrons (MXCD)

\longleftrightarrow
Sensitivity



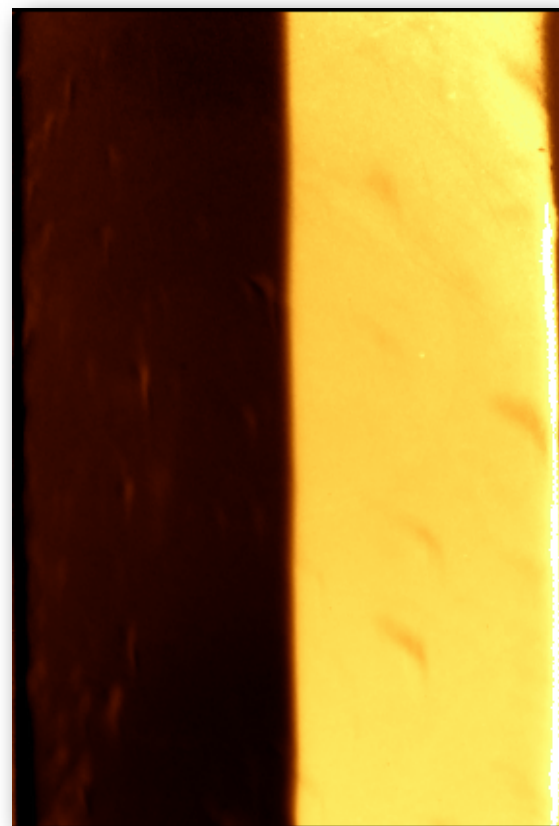
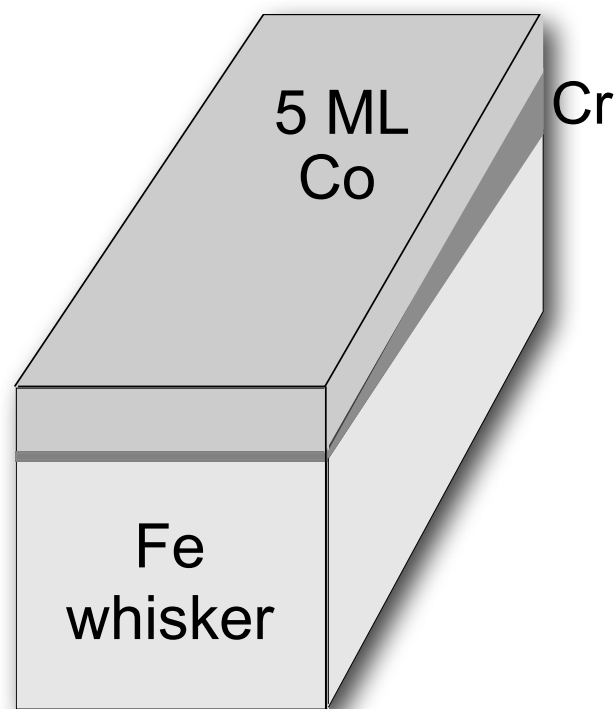
Courtesy C.M. Schneider

5. X-Ray Spectroscopy

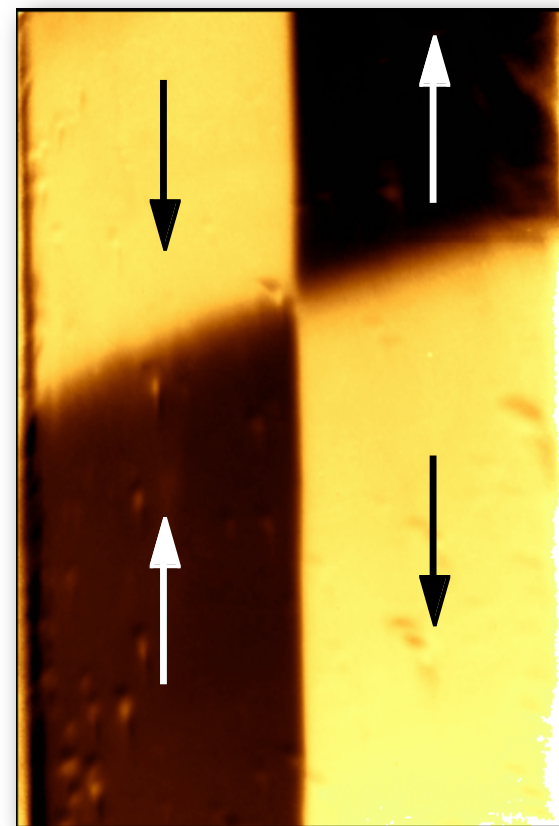
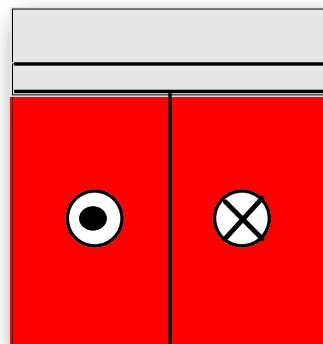
5.2 X-ray Photoemission Electron Microscopy (X-PEEM)

Depth-selectivity by element-specific PEEM imaging

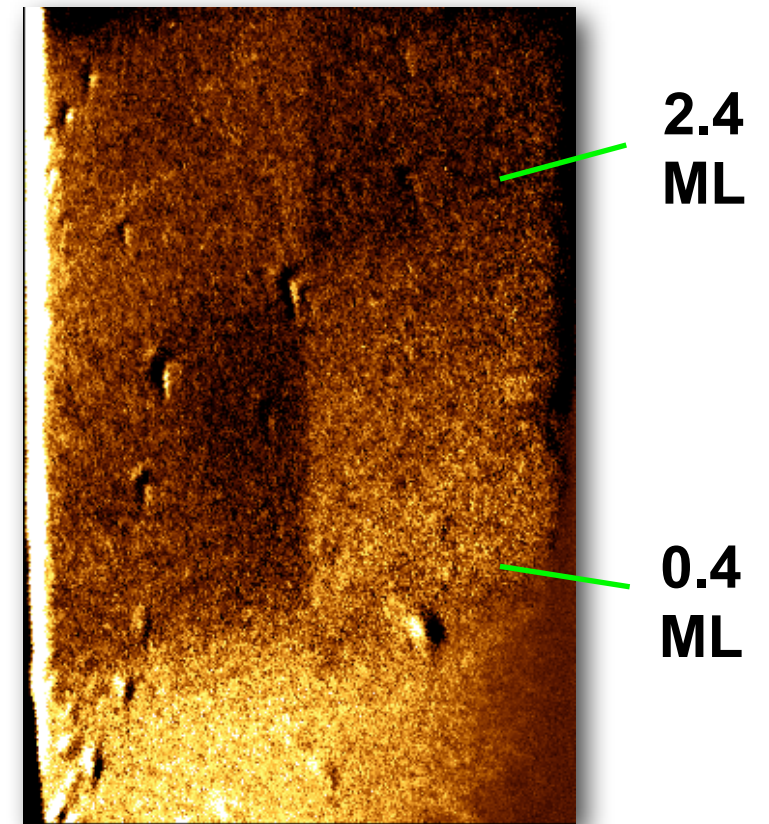
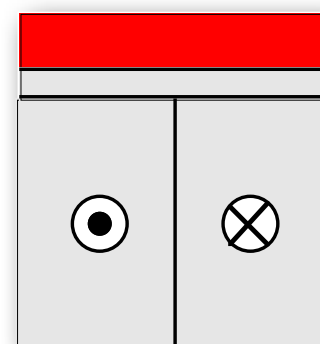
Fe-Cr-Co layer system



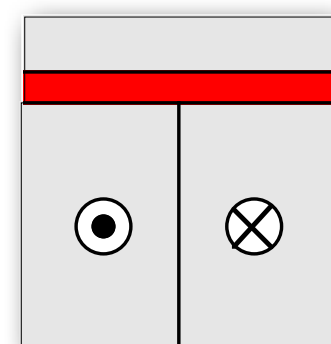
Fe whisker



Co



Cr



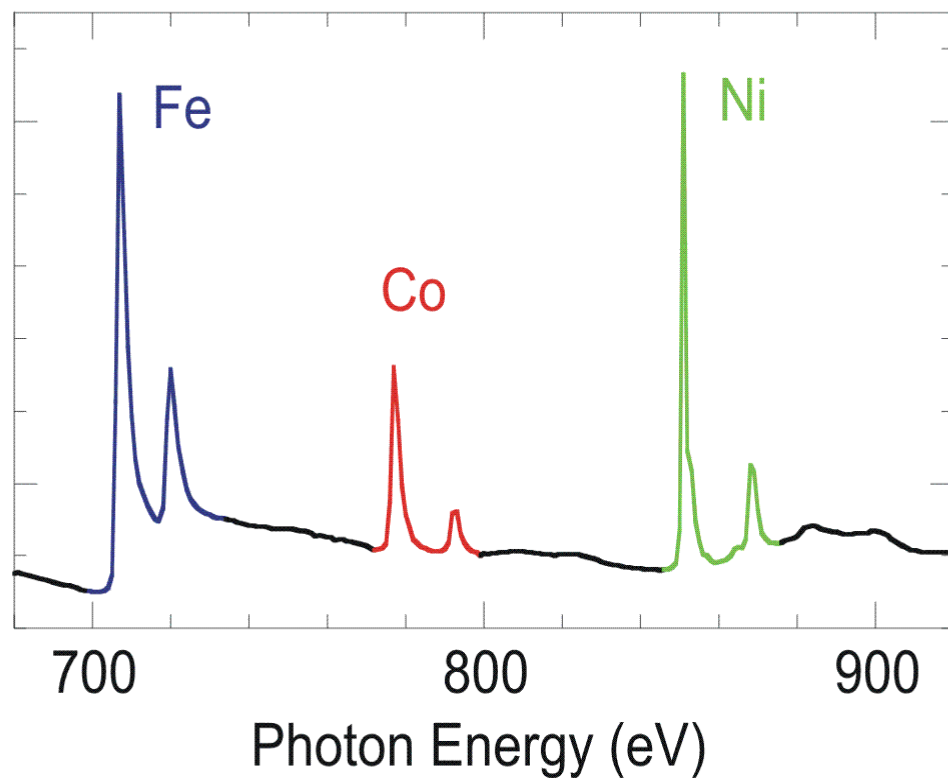
courtesy
C.M. Schneider

5. X-Ray Spectroscopy

5.2 X-ray Photoemission Electron Microscopy (X-PEEM)

Layer-selective imaging

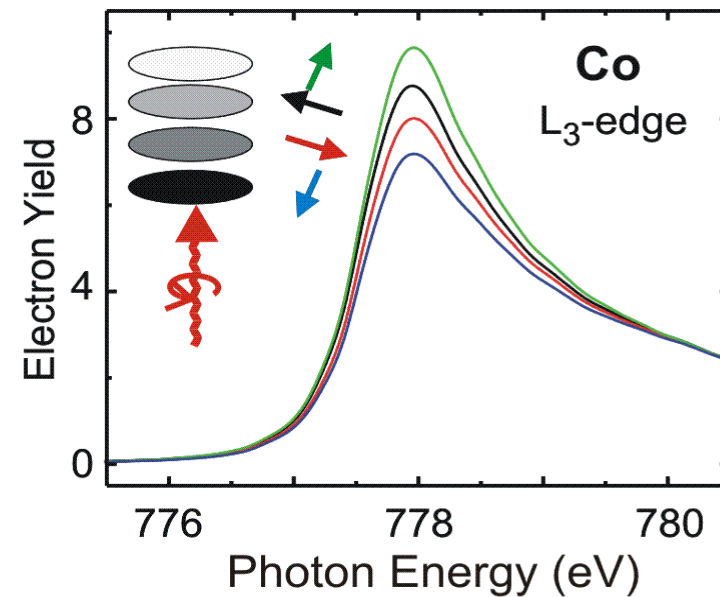
Tune x-ray **energy**
for elemental specificity



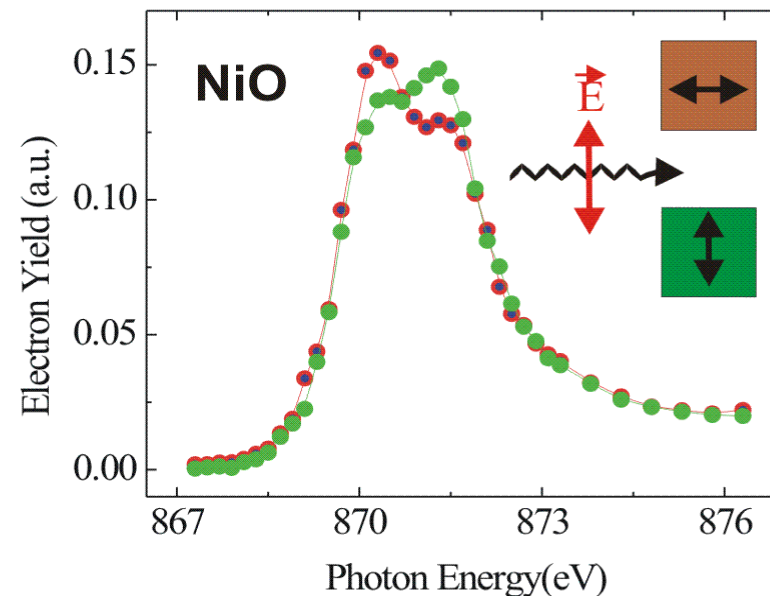
Courtesy J. Stöhr

Tune x-ray **polarization**
for magnetic specificity

Circular Dichroism - Ferromagnets



Linear Dichroism - Antiferromagnets

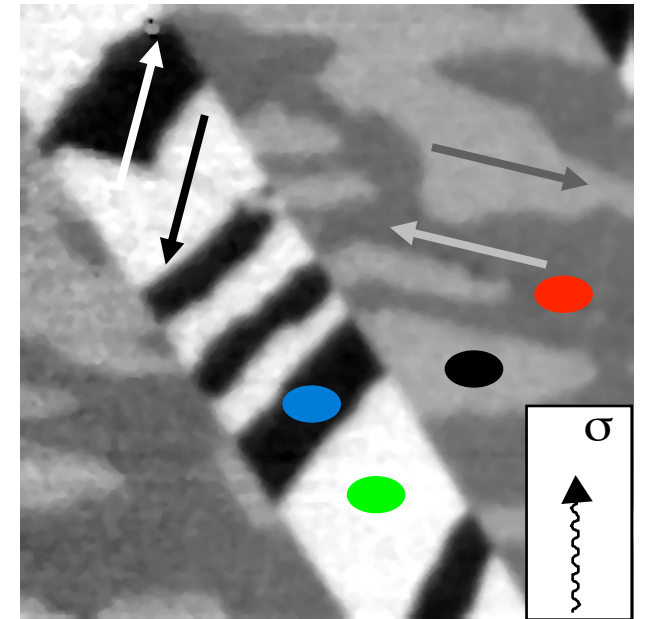
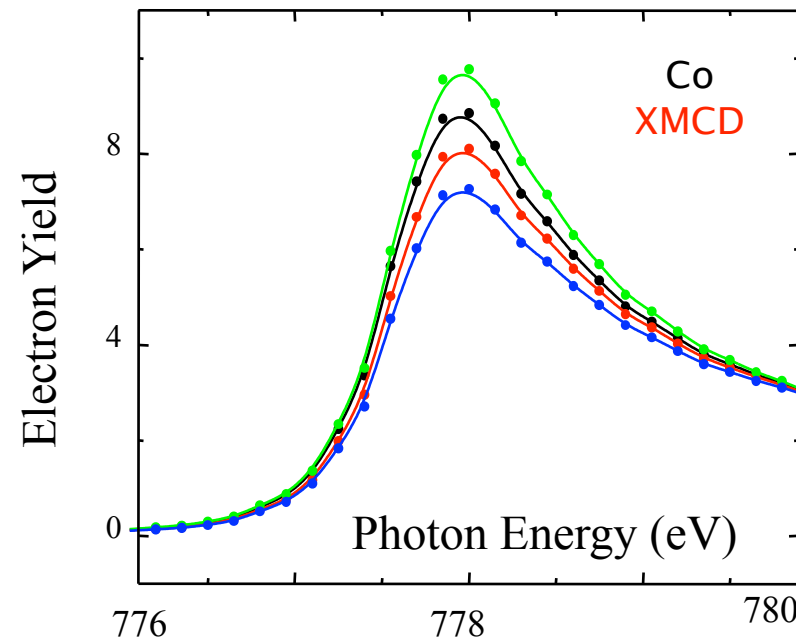
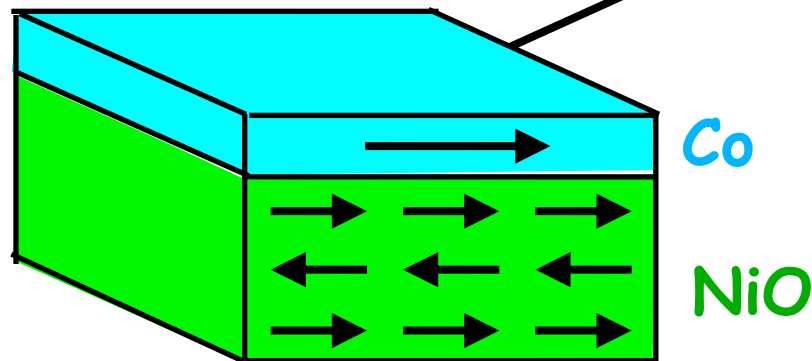


5. X-Ray Spectroscopy

5.2 X-ray Photoemission Electron Microscopy (X-PEEM) Layer-selective imaging

Tune to **Co** edge, use circular polarization: ferromagnetic domains

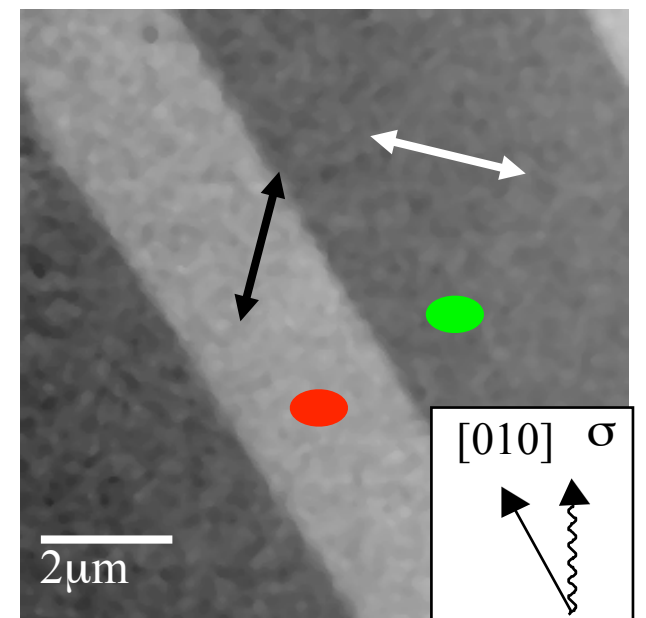
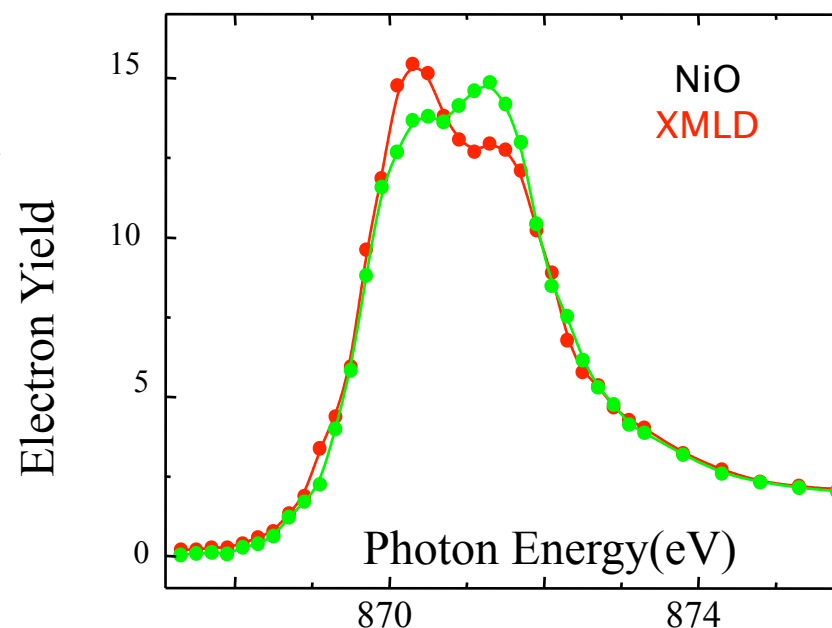
Circular polarization:
Sensitive to direction of
magnetic moment



Tune to **Ni** edge, use linear polarization: antiferromagnetic domains

Linear polarization:
Sensitive to axis of
magnetic moment

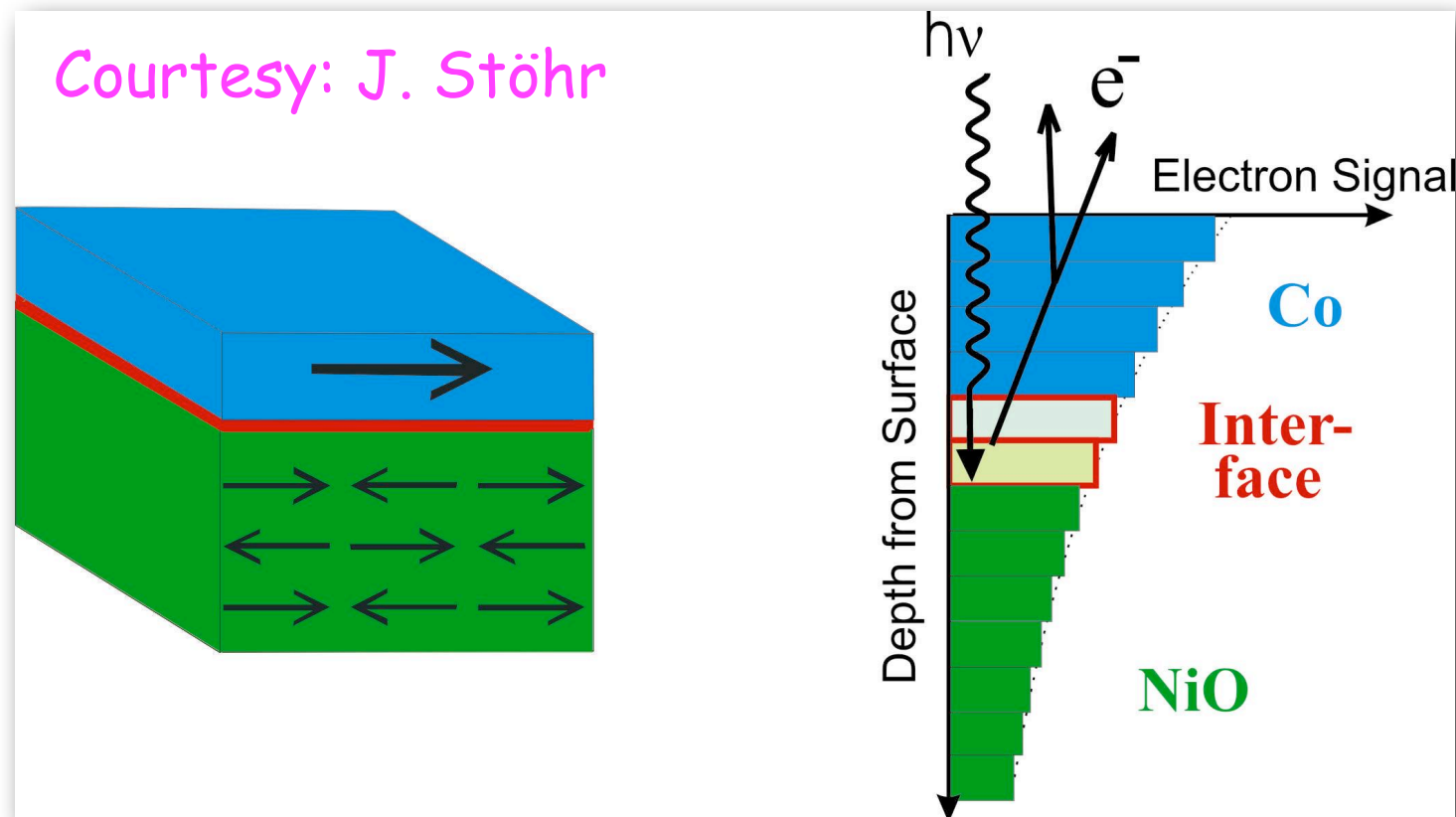
*H. Ohldag et al.,
PRL 86, 2878 (2001)*



5. X-Ray Spectroscopy

Interface studies of Ferromagnets on Antiferromagnets

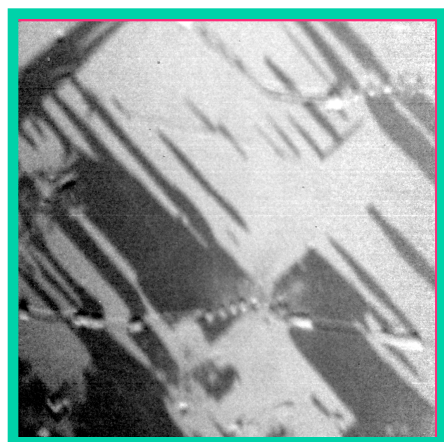
Courtesy: J. Stöhr



FM Co - tune to Co edge -
circular polarization

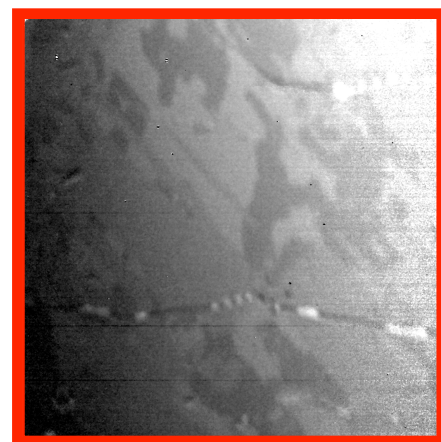
AFM NiO - tune to Ni edge -
linear polarization

FM Ni(O) - tune to Ni edge -
circular polarization



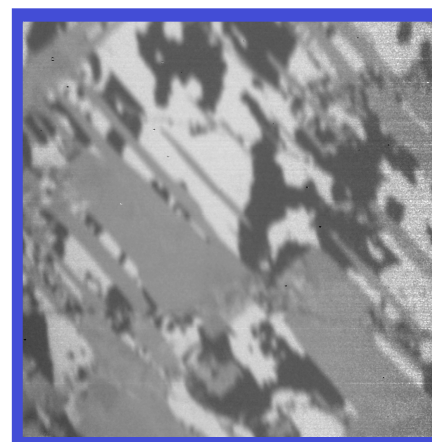
AFM: NiO

Linear pol.
Ni edge



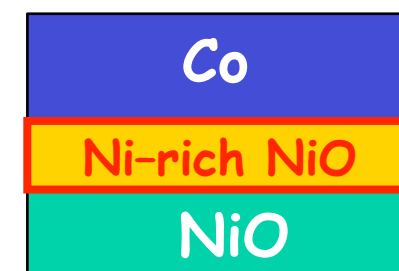
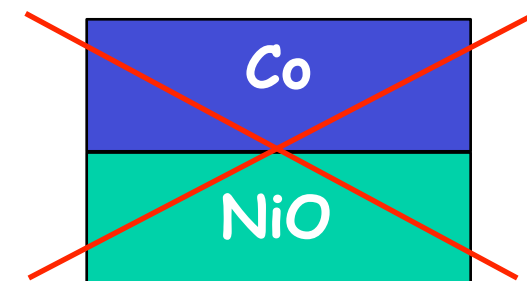
FM:
Ni-rich NiO

Circular pol.
Ni edge



FM: Co

Circular pol.
Co edge



Interfacial
spins

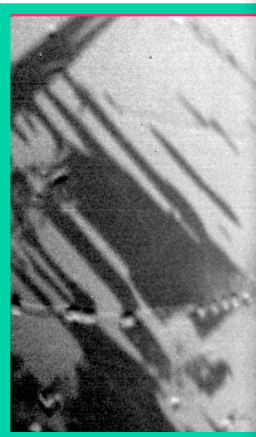
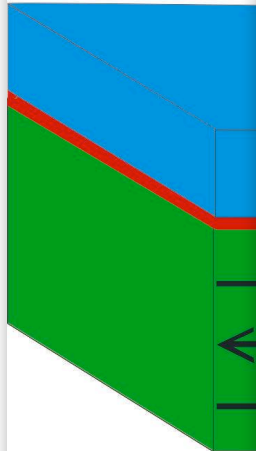
5. X-Ray Spectroscopy

Interface studies of Ferromagnets on Antiferromagnets

Courtesy: J. Stöhr

$h\nu$
 \uparrow e^-

FM Co - tune to Co edge -
circular polarization



Chemically induced
interfacial Ni spins
provide the magnetic link

AFM: NiO

Linear pol.
Ni edge

FM:
Ni-rich NiO

Circular pol.
Ni edge

FM: Co

Circular pol.
Co edge

Co

Ni-rich NiO

NiO

Interfacial
spins

5. X-Ray Spectroscopy

Comparison of X-ray microscopy with PEEM imaging

Equal

- XMCD as contrast
- element-specific
- quantitative
- in- and out-of-plane

Advantages
of PEEM

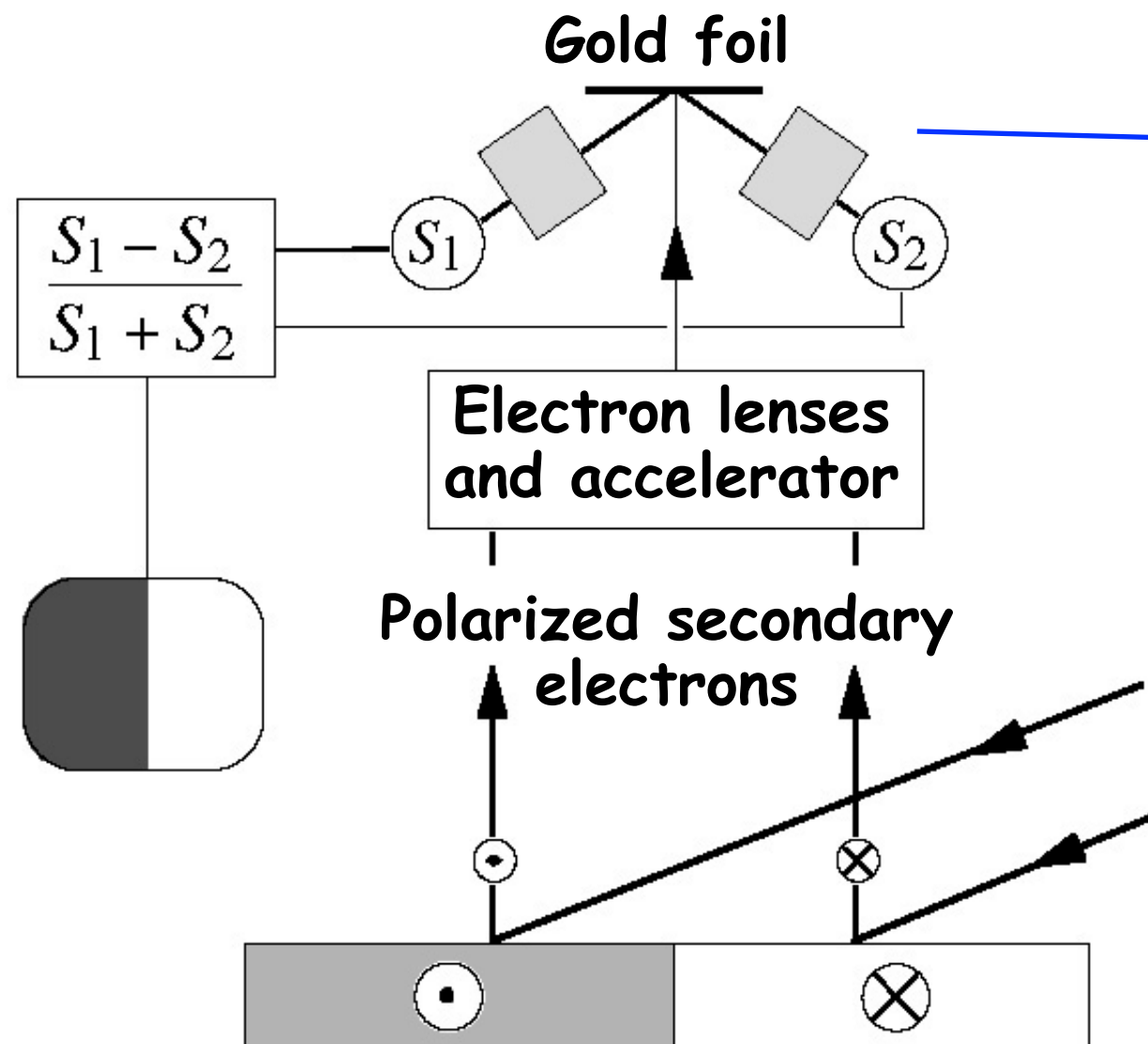
- no sample thinning
- higher sensitivity
- higher spectral resolution

Disadvantages
of PEEM

- imaging in fields difficult
- time consuming adjustment
- lower standard resolution
- UHV conditions

6. Electron Polarization Analysis

6.1 Scanning Electron Microscopy with Polarization Analysis (SEMPA)



Mott detector:

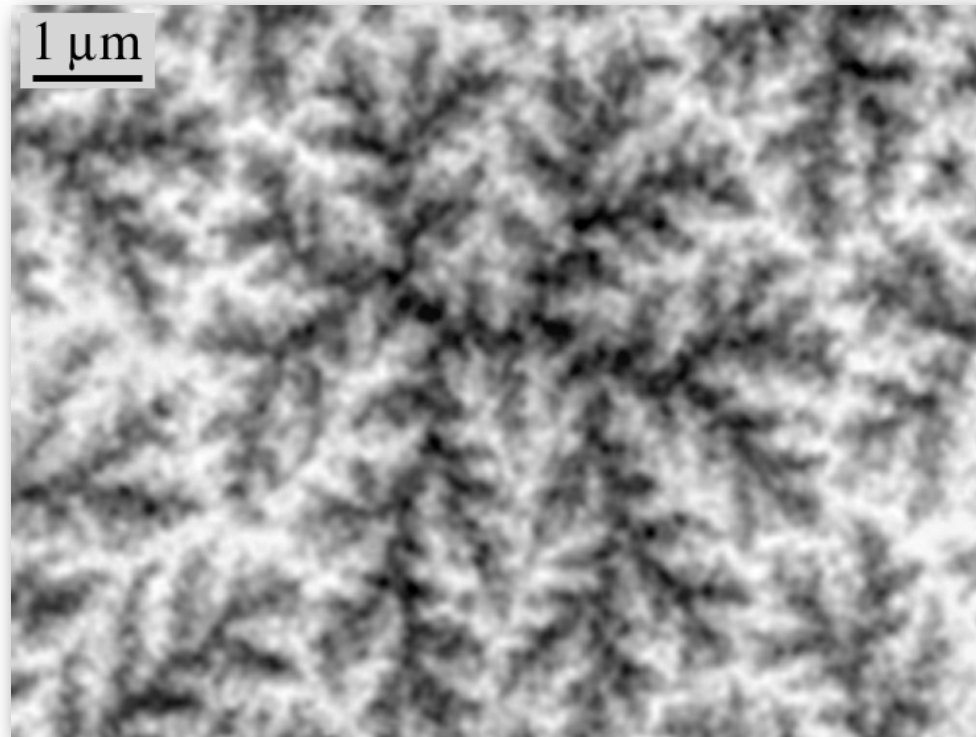
Scattering of polarized electrons by gold foil is asymmetric (spin-orbit coupling effects)

- Secondary electrons are spin polarized, moment along magnetization direction
- Surface sensitive (secondary electrons emerge from top nanometer)
- Quantitative (independent measurement of 3 magnetization components)
- Resolution in 10 nm range

6. Electron Polarization Analysis

6.1 Scanning Electron Microscopy with Polarization Analysis

Basal plane of
Co crystal

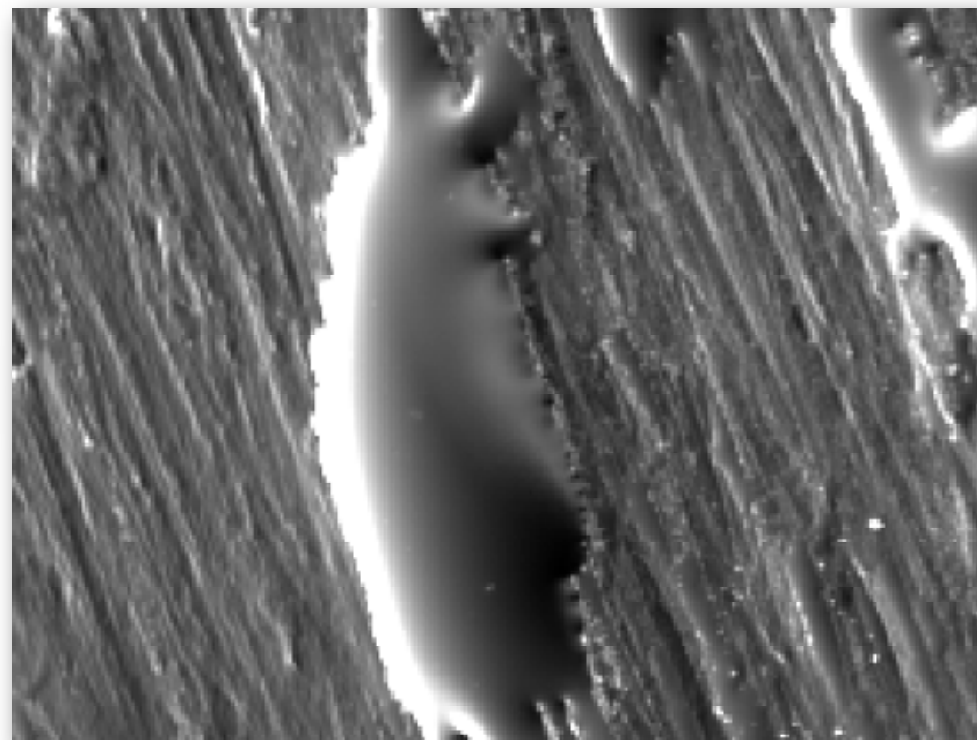


Polar components

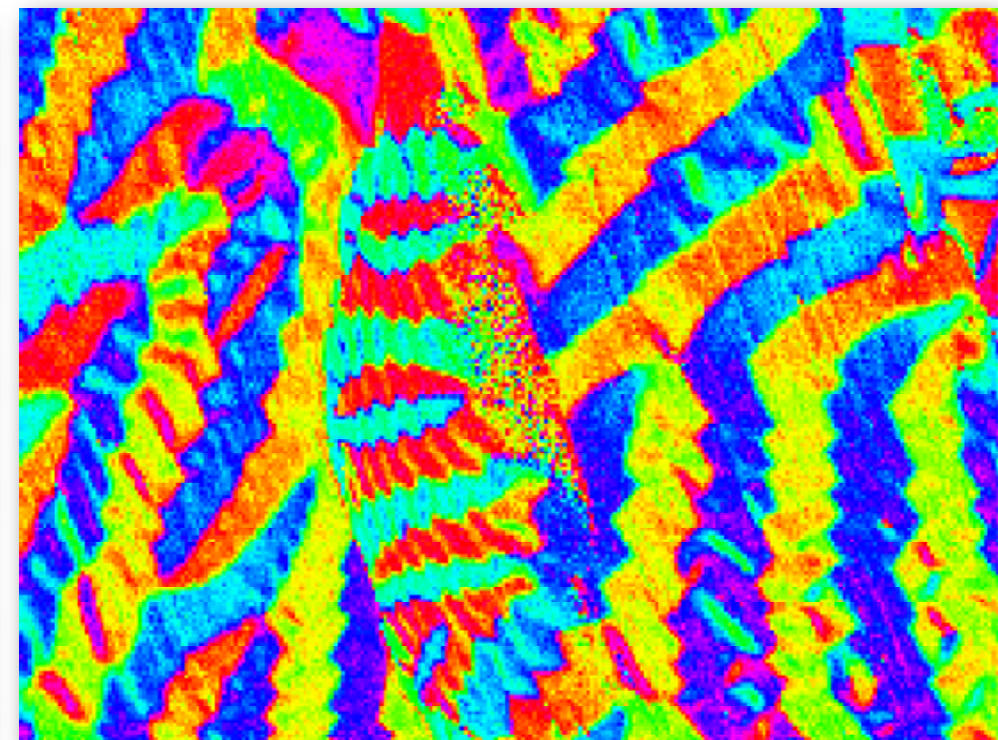


In-plane components

"Wheel side" of
amorphous ribbon



Topography



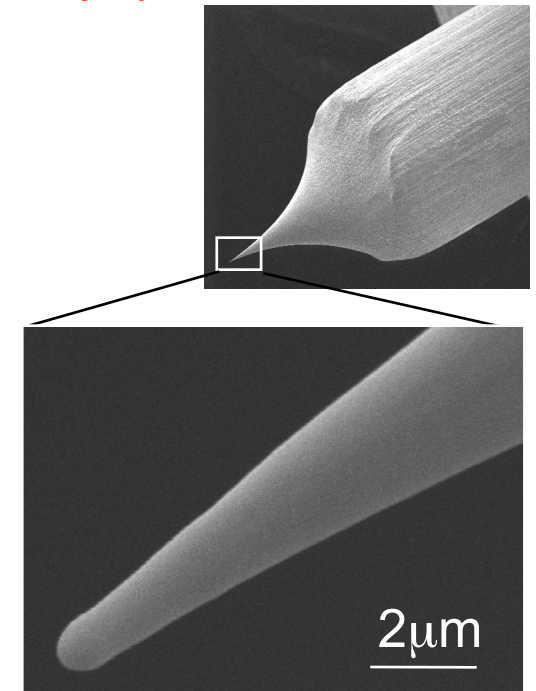
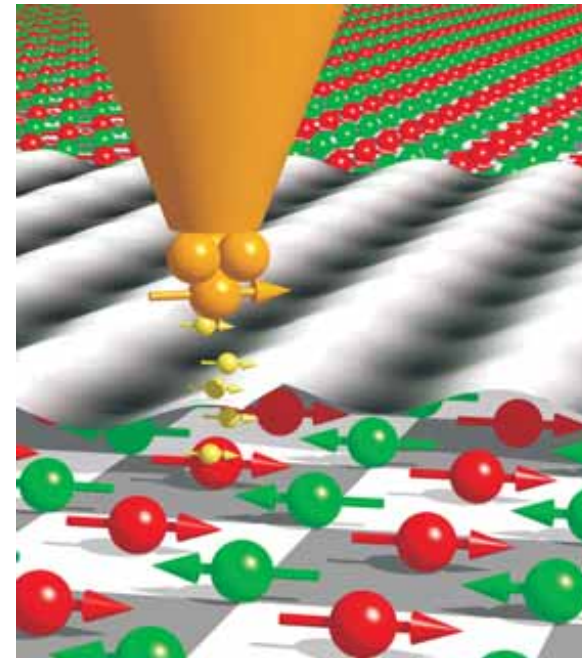
In-plane components

courtesy
J. Unguris

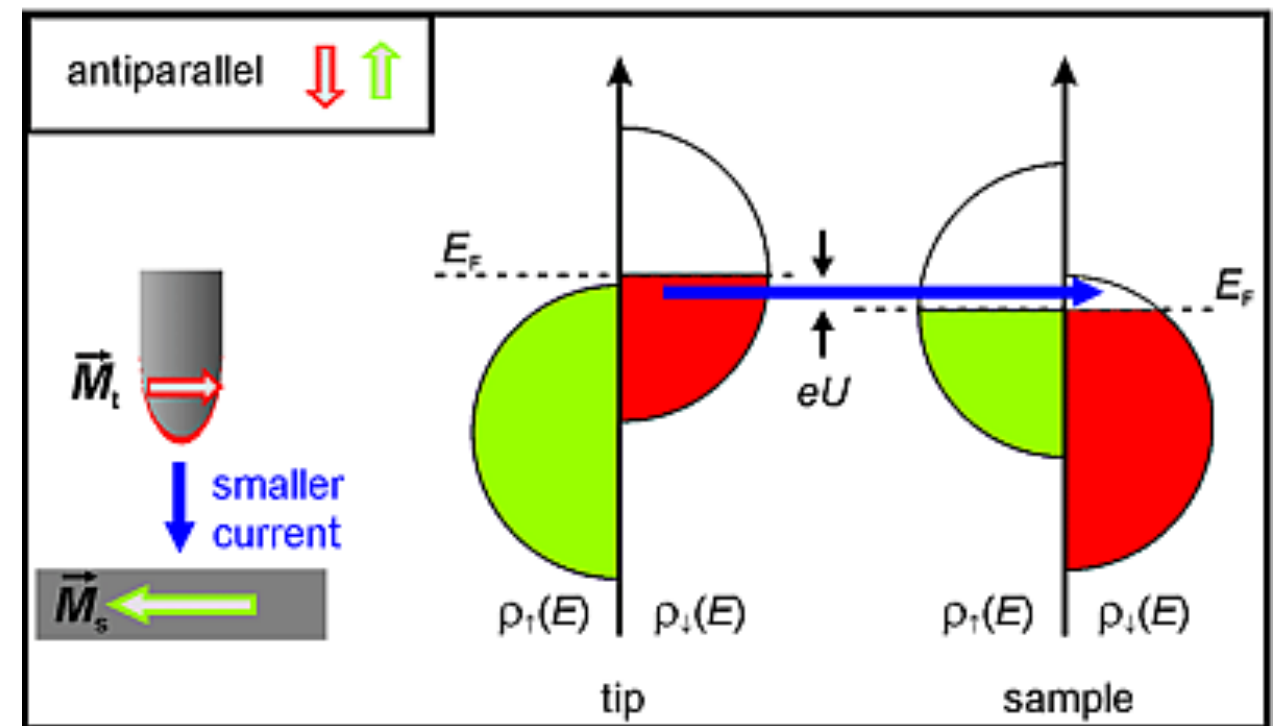
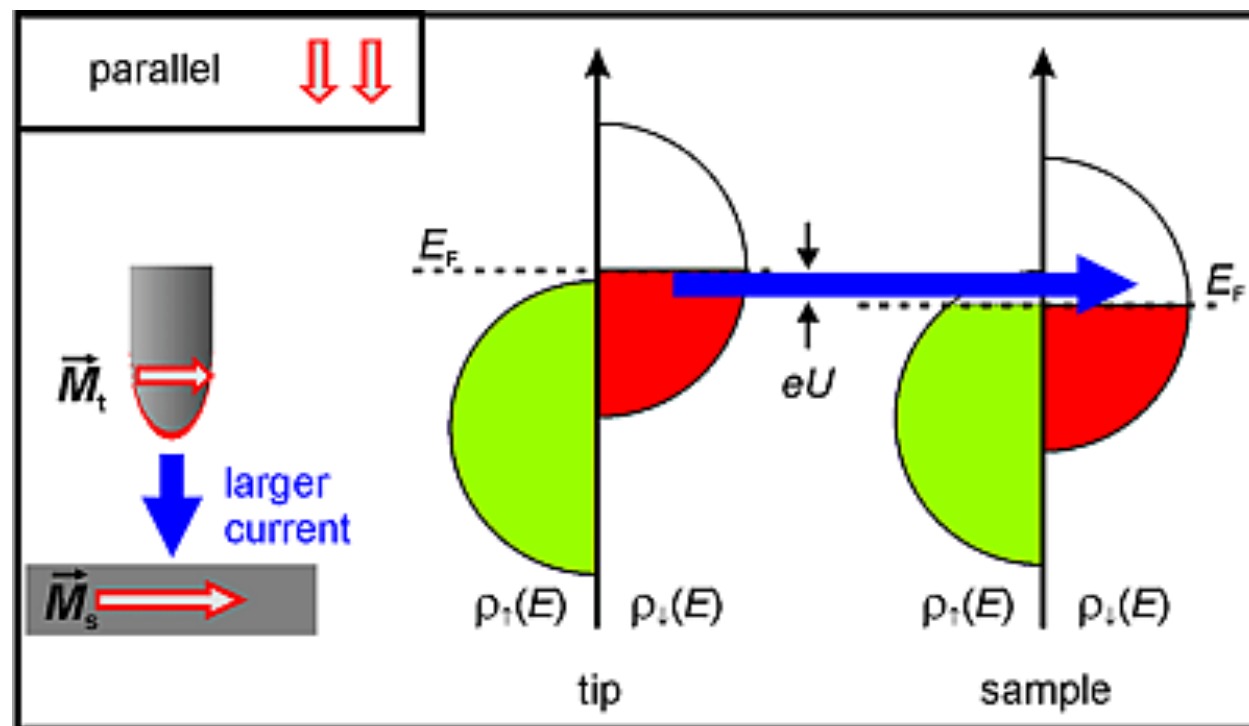
6. Electron Polarization Analysis

6.2 Spin-polarized Tunneling Microscopy

- Tunneling of spin-polarized current between tip and sample surface
- Tunneling resistance depends on relative orientation of current polarity and domain magnetization
- Extreme resolution



M. Julliere, Phys. Lett. 54A, 225 (1975):



from Wiesendanger homepage

<http://www.nanoscience.de/nanojoom/index.php/en/methods/sp-stm.html>

6. Electron Polarization Analysis

6.2 Spin-polarized Tunneling Microscopy

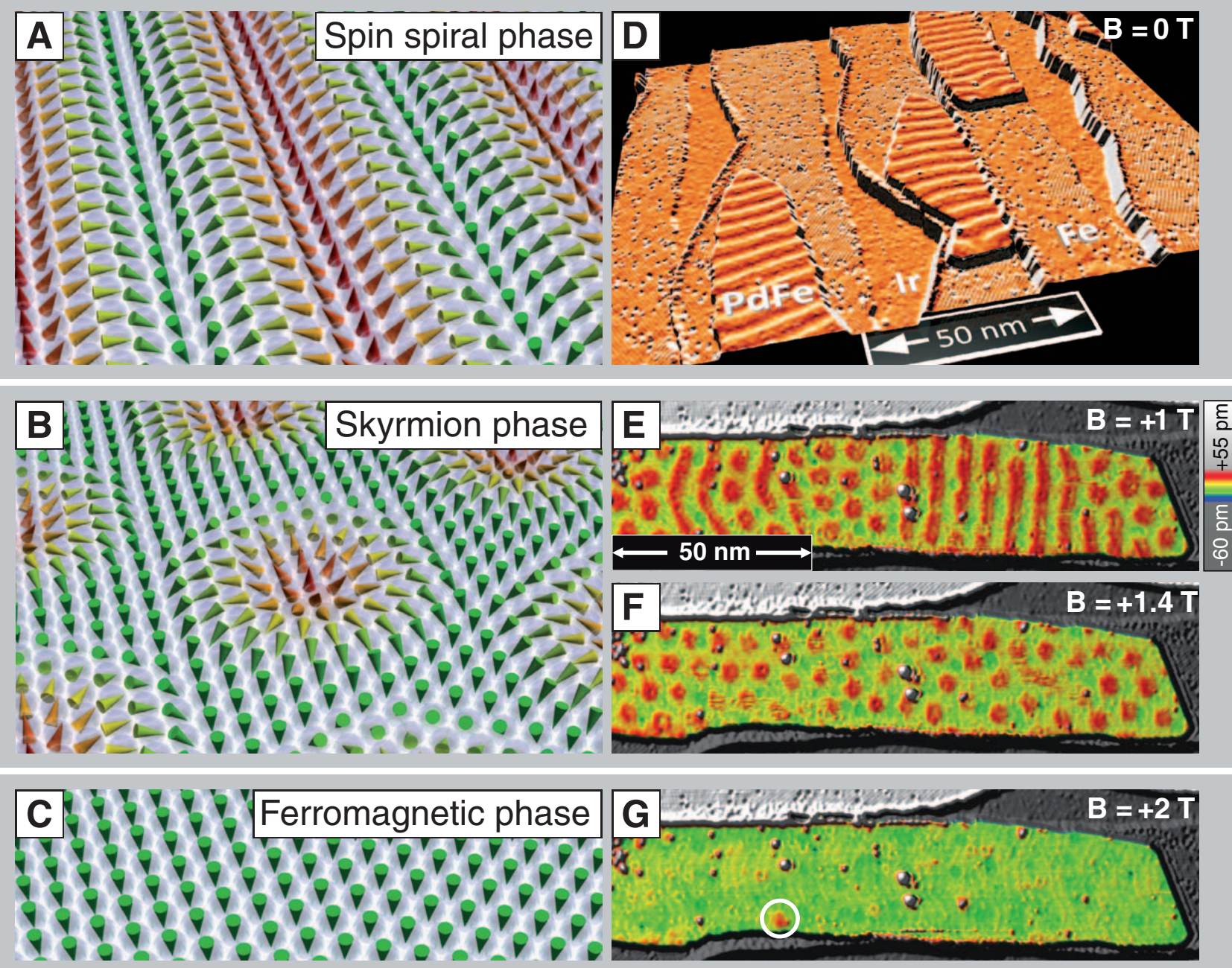


Fig. 1. Magnetic field dependence of the PdFe bilayer on the Ir(111) surface at $T = 8$ K. (A to C) Perspective sketches of the magnetic phases. (D) Overview SP-STM image, perspective view of constant-current image colorized with its derivative. (E to G) PdFe bilayer at different magnetic fields ($U = +50$ mV, $I = 0.2$ nA, magnetically out-of-plane sensitive tip). (E) Coexistence of spin spiral and skyrmion phase. (F) Pure skyrmion phase. (G) Ferromagnetic phase. A remaining skyrmion is marked by the white circle.



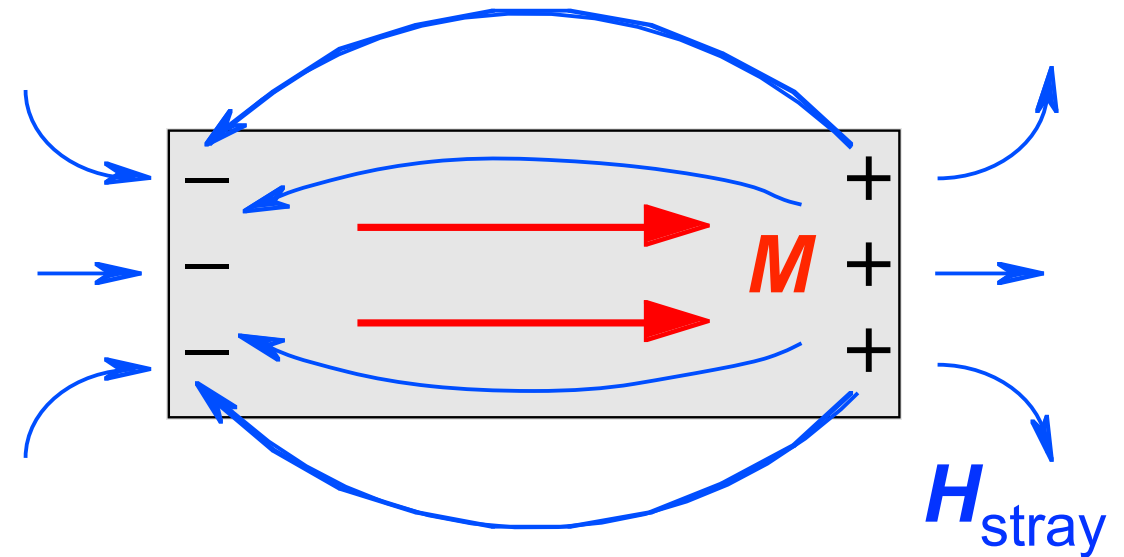
Writing and Deleting Single
Magnetic Skyrmions.
Niklas Romming et al.
Science 341, 636 (2013);
DOI: 10.1126/science.
1240573

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

$$\text{div } \mathbf{B} = 0$$

$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



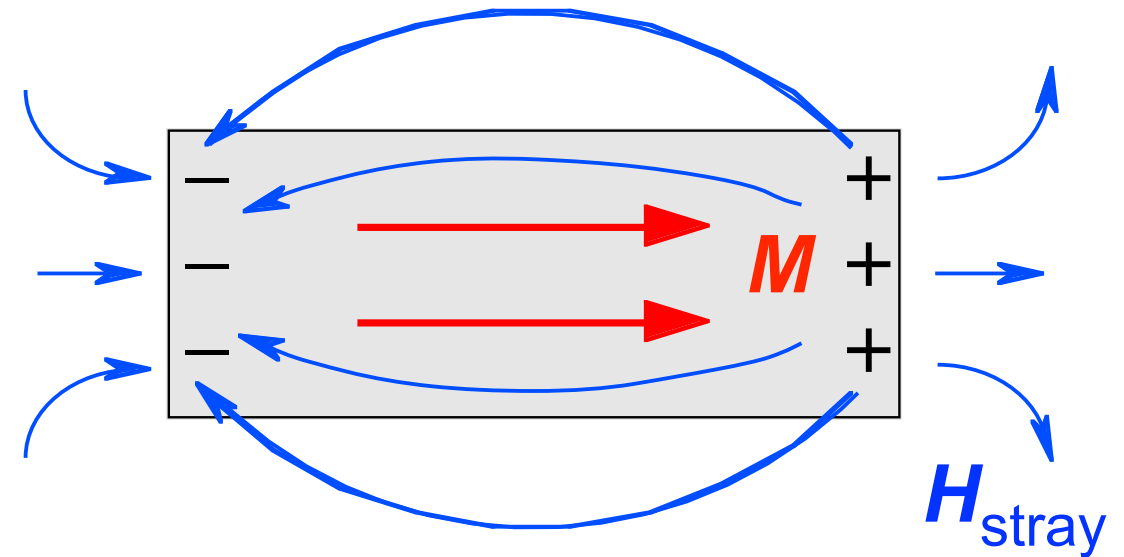
- Sensitive to $\mathbf{H}_{\text{stray}}$
 1. Bitter technique
 2. Magnetic force microscopy
 3. Hall probe microscopy
- Sensitive to \mathbf{M}
 4. Magneto-optical microscopy
 5. X-ray spectroscopy
 6. Polarized electrons (SEMPA, SPT)
- Sensitive to \mathbf{B}
 - Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

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- Sensitive to \mathbf{B}
 7. Transmission electron microscopy
- Sensitive to distortions
 - X-ray, neutron scattering

7. Transmission Electron Microscopy

Principle

- Electrons are deflected by Lorentz force

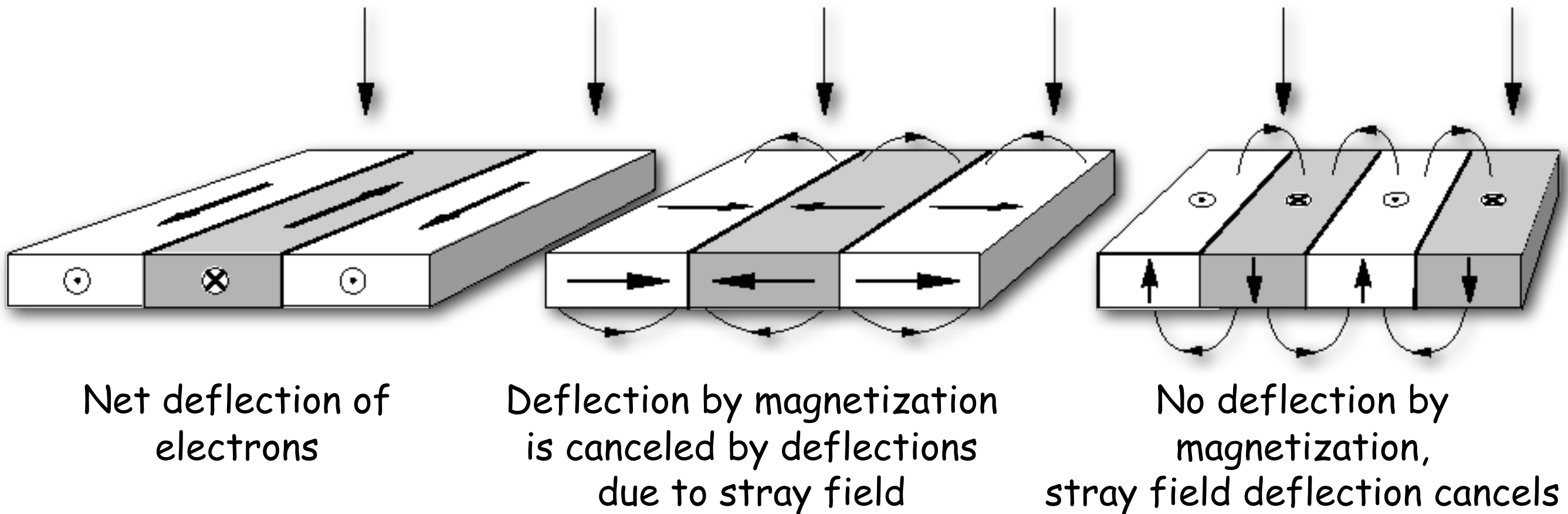
$$F_L = q_e (v_e \times B)$$

q_e : Electron charge

v_e : Electron velocity

B : Magnetic flux density

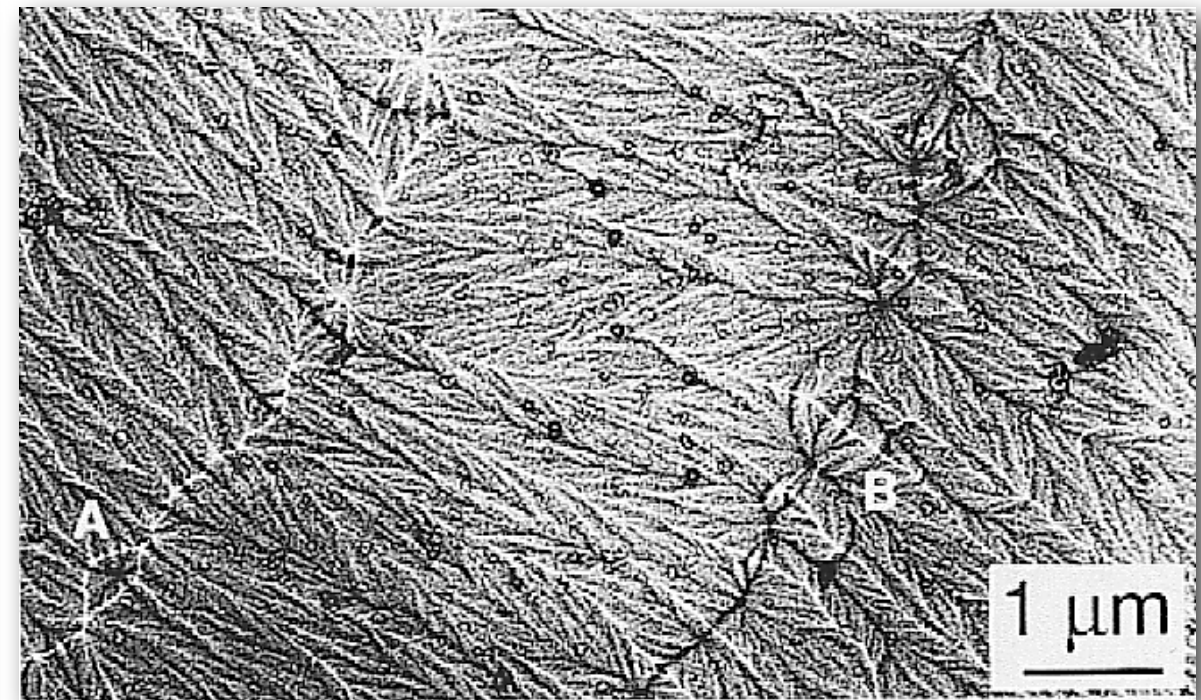
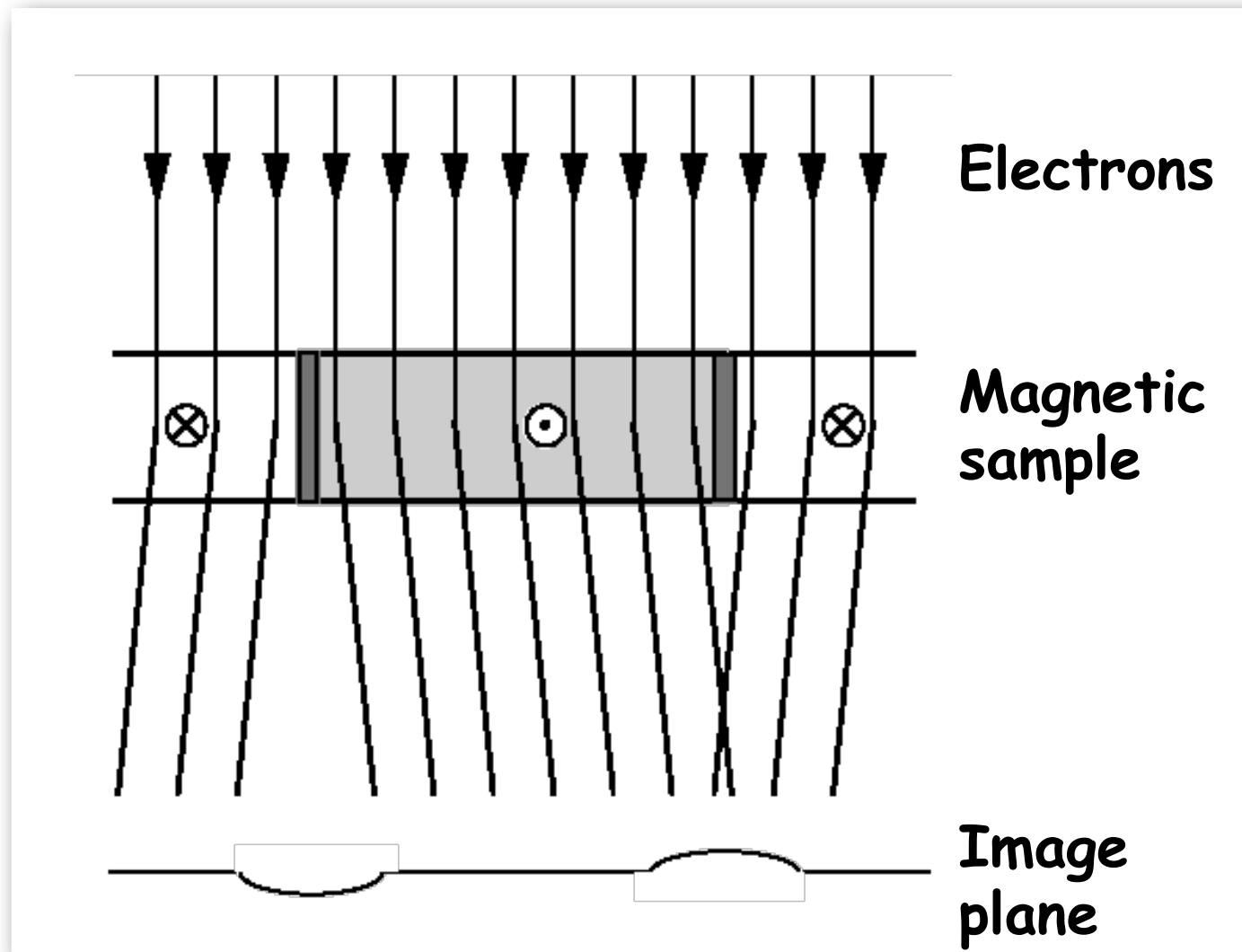
- Stray fields outside the sample contribute to contrast



- Tilting of sample may be required
- Maximum sample thickness: some 100 nm

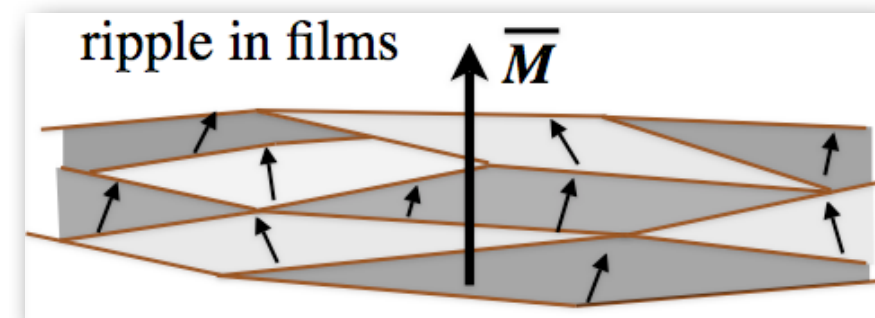
7. Transmission Electron Microscopy

7.1 Fresnel technique (defocused mode imaging)



Metallic glass, partially crystallized
(courtesy J. Chapman)

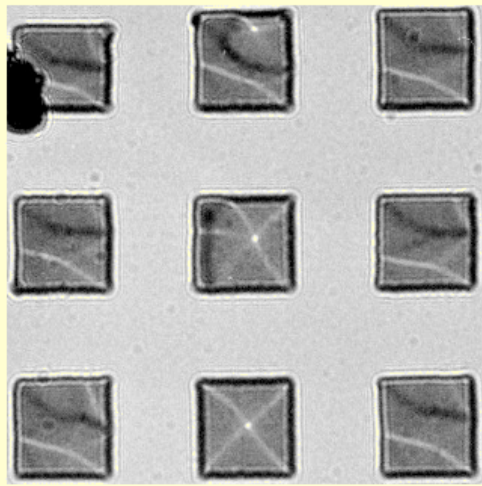
- Out-of-focus: shadow effects delineate domain boundaries
- Magnetization direction can be derived from ripple (if present)



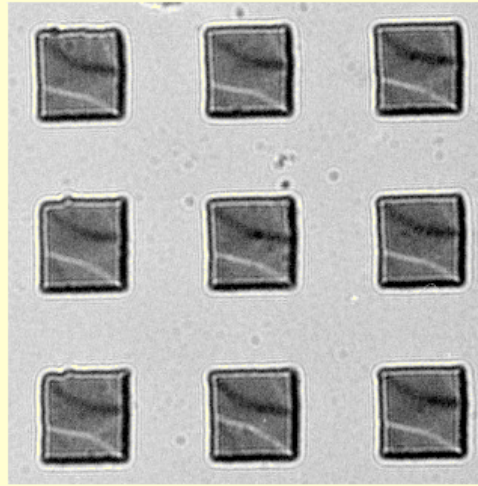
7. Transmission Electron Microscopy

7.1 Fresnel technique (defocused mode imaging)

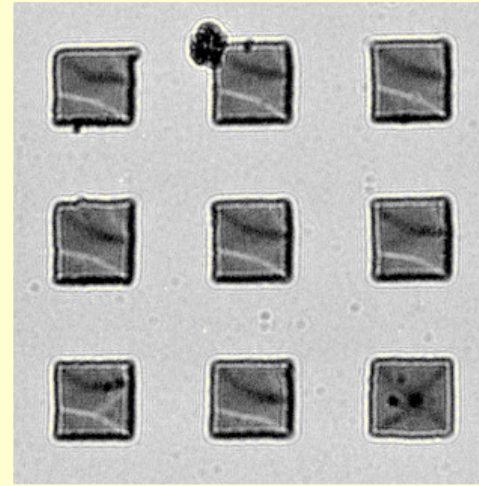
Fresnel imaging of differently sized magnetic particles (Co, 35 nm thick)



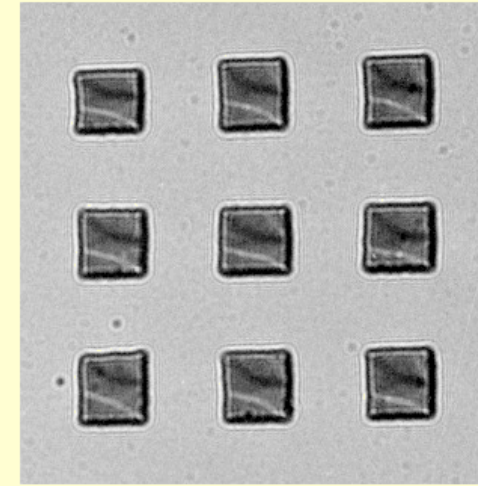
a) $1 \times 1 \mu\text{m}^2$



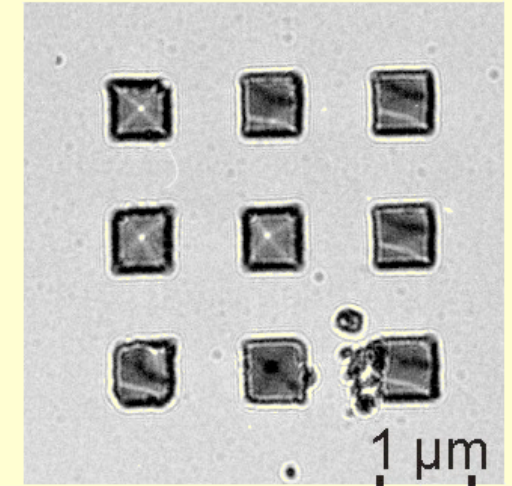
b) $900 \times 900 \text{ nm}^2$



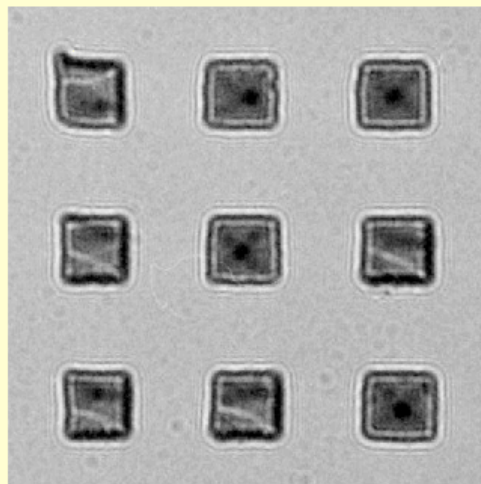
c) $800 \times 800 \text{ nm}^2$



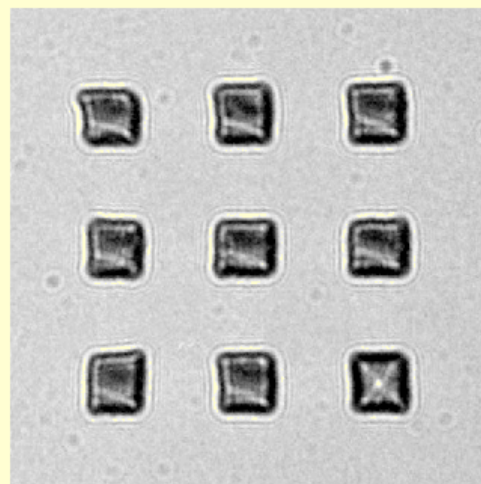
d) $700 \times 700 \text{ nm}^2$



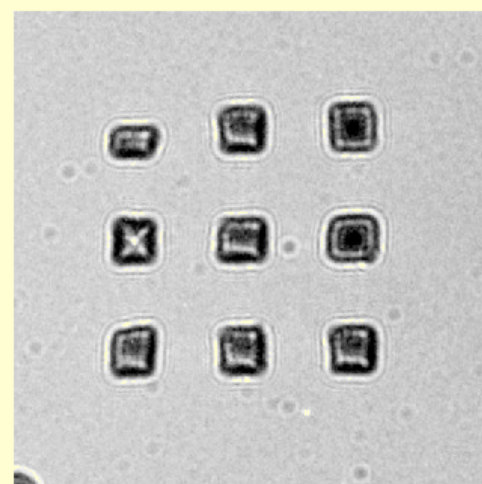
e) $600 \times 600 \text{ nm}^2$



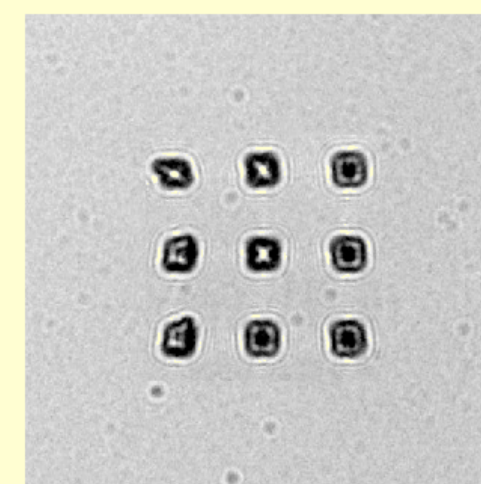
f) $500 \times 500 \text{ nm}^2$



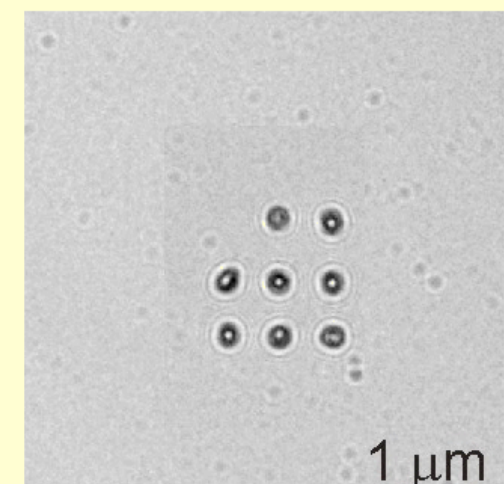
g) $400 \times 400 \text{ nm}^2$



h) $300 \times 300 \text{ nm}^2$



i) $200 \times 200 \text{ nm}^2$

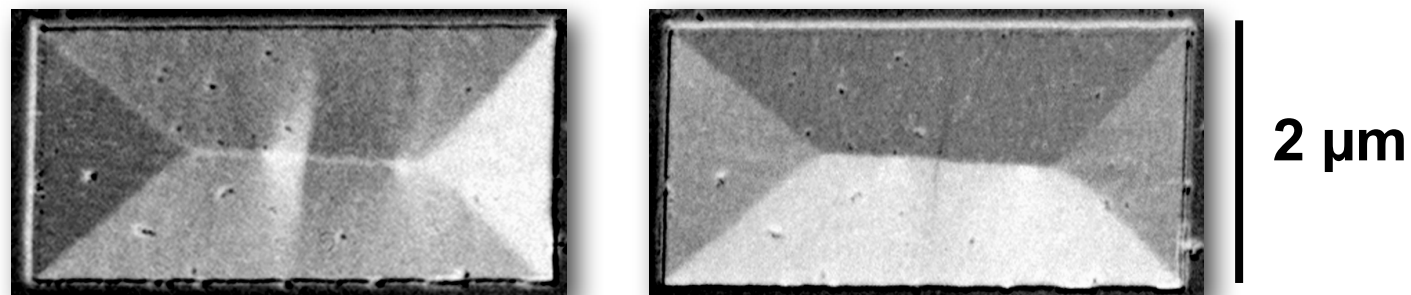
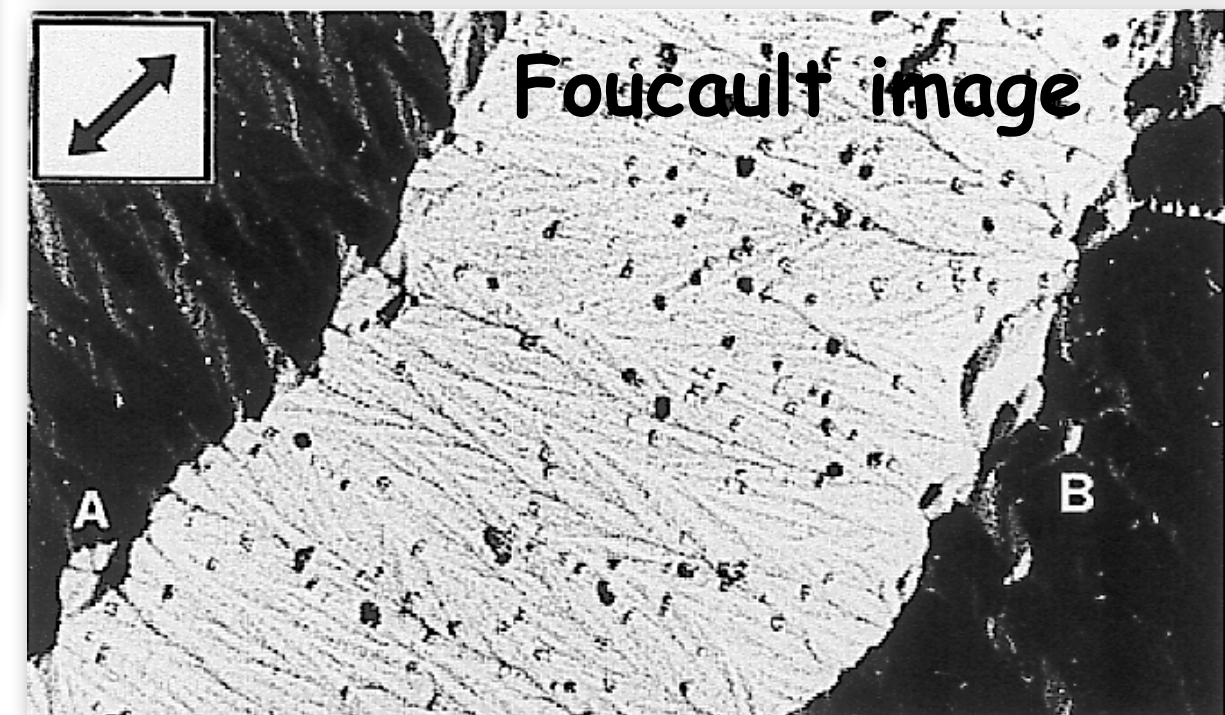
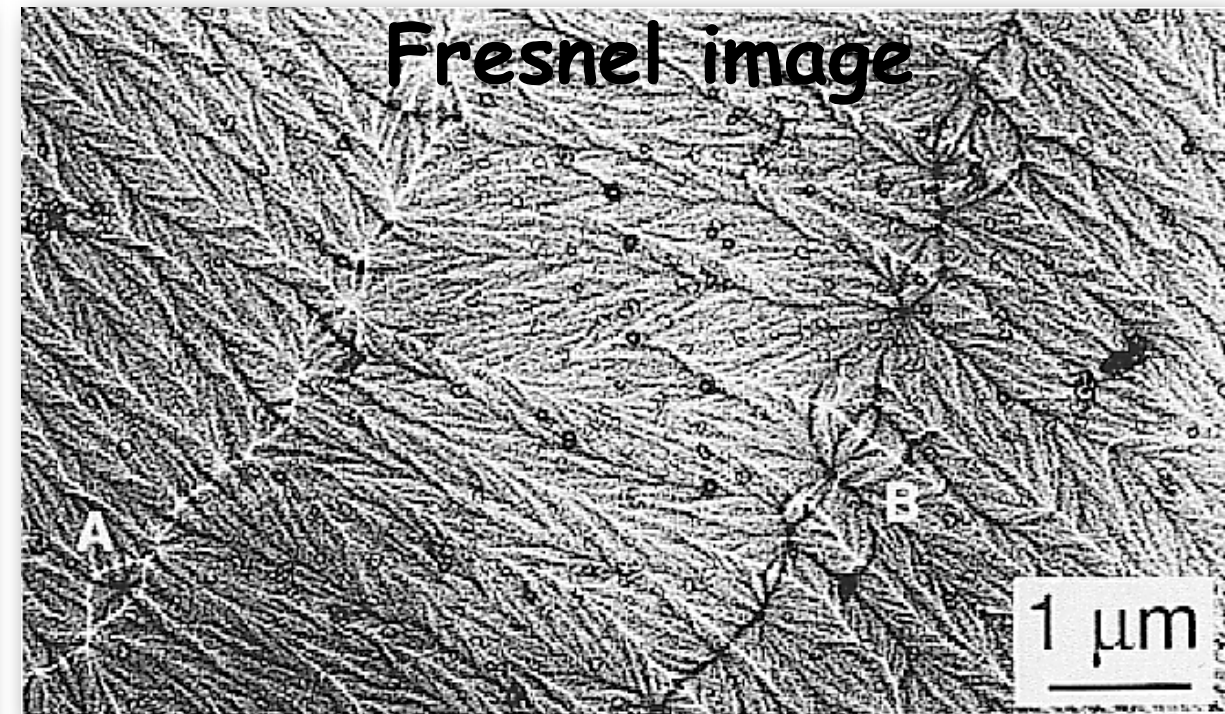
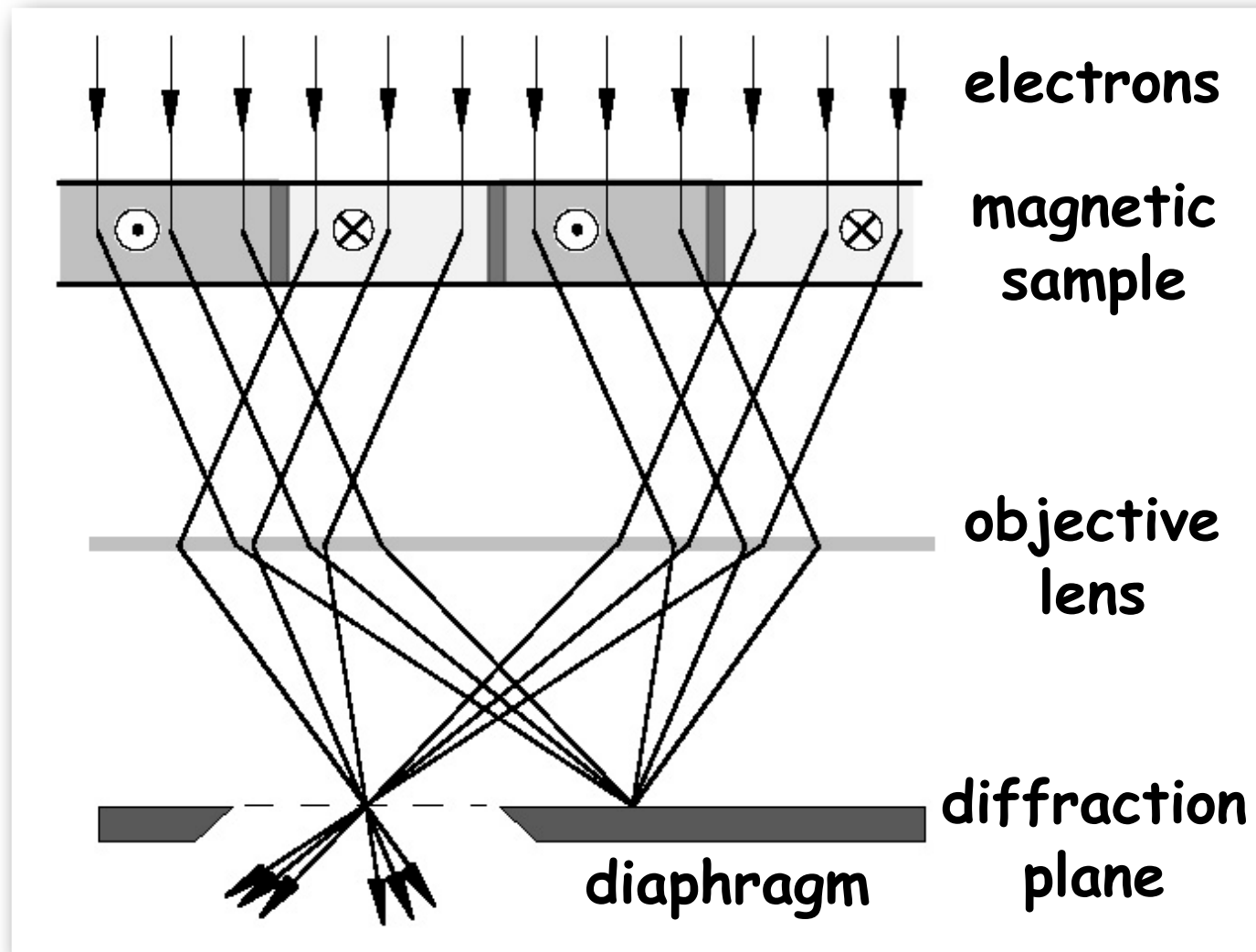


j) $100 \times 100 \text{ nm}^2$

Courtesy J. Zweck

7. Transmission Electron Microscopy

7.2 Foucault technique (in-focus)

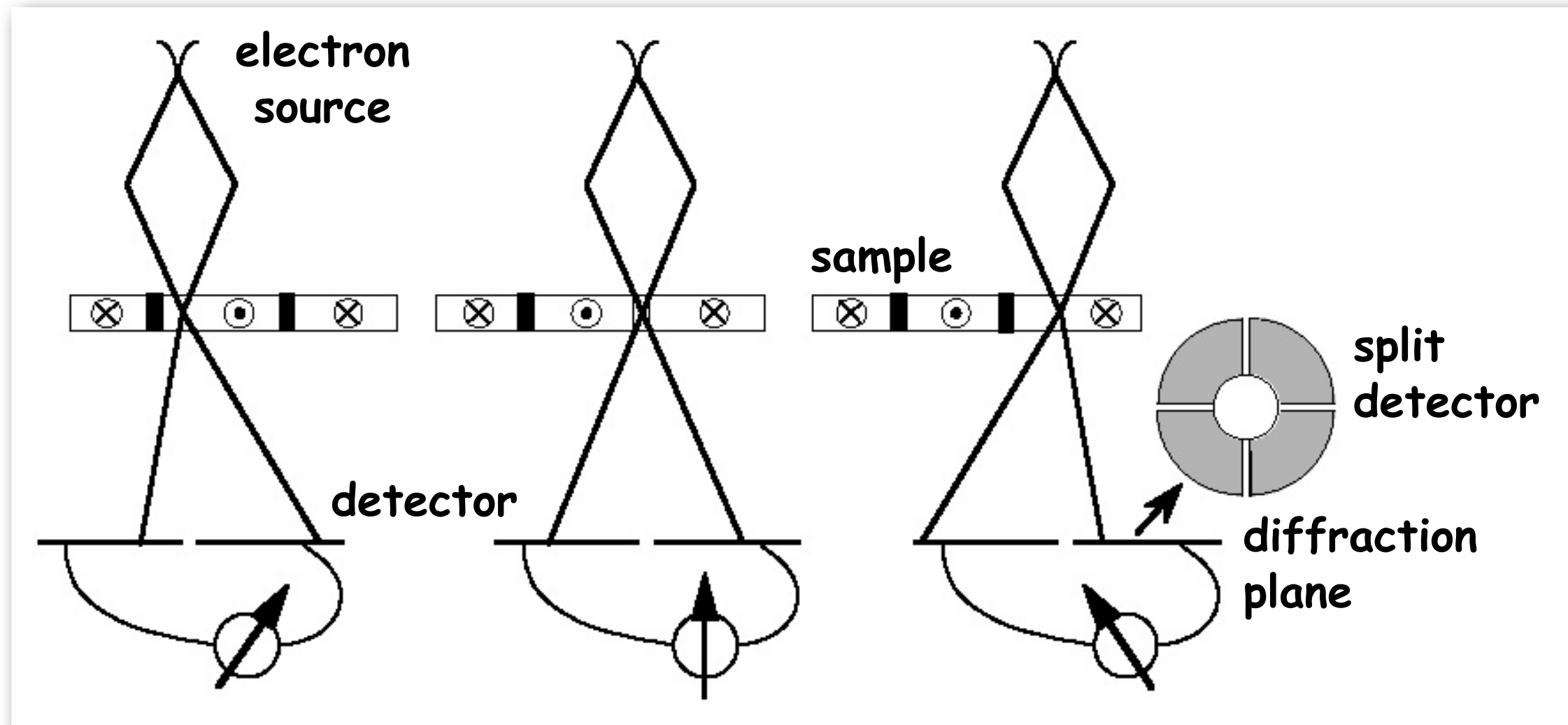


Permalloy, 24 nm thick
(courtesy J. Chapman)

Metallic glass, partially crystallized
(courtesy J. Chapman)

7. Transmission Electron Microscopy

7.3 Differential Phase Contrast (DPC) Microscopy

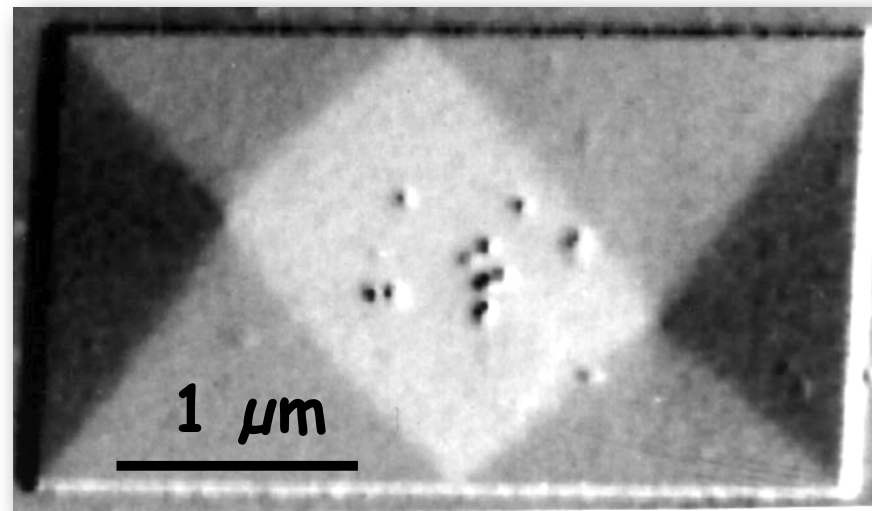
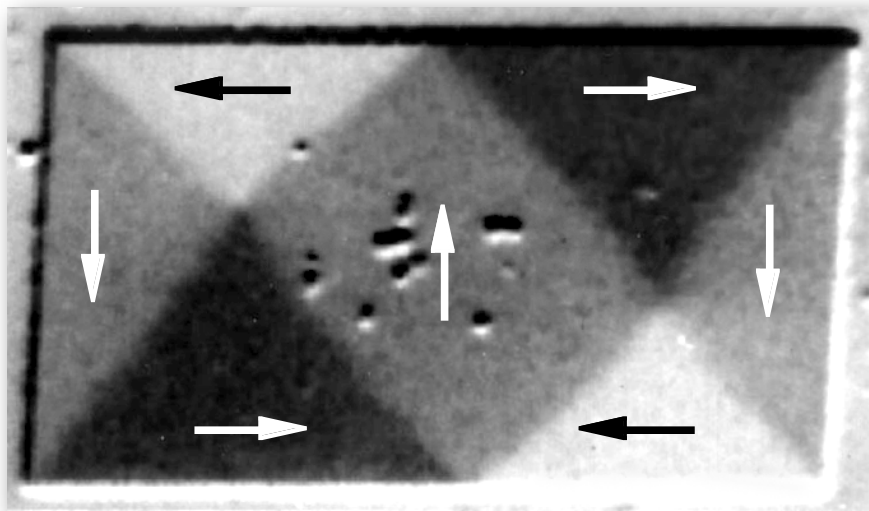


- Domain contrast like in Kerr microscopy
- Resolution better than 10 nm
- Quantitative determination of magnetization direction (by combining signals of a quadrant detector)

7. Transmission Electron Microscopy

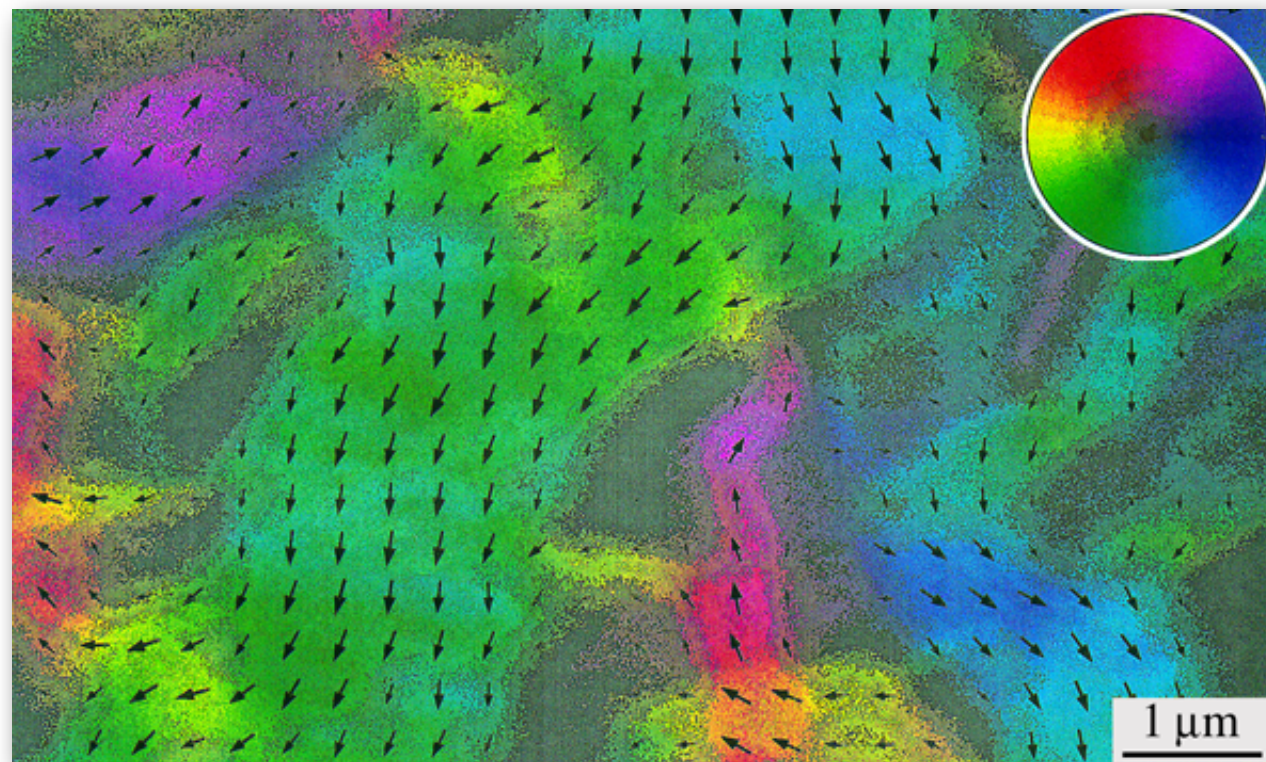
7.3 Differential Phase Contrast (DPC) Microscopy

In a scanning TEM



Permalloy, 60 nm thick
(courtesy J. Chapman)

In a conventional TEM



AFM coupled Co-Cr-Co sandwich
(courtesy J.P. Jakubovics)

Difference between Foucault images,
obtained at different angles of incidence

7. Transmission Electron Microscopy

7.4 Electron Holography

Principle

$$\text{grad } \varphi_e = (2 \pi q_e D/h) \mathbf{B}_0 \times \mathbf{s}_b$$

Diagram illustrating the principle of electron holography, showing the relationship between the phase gradient and various parameters:

- $\text{grad } \varphi_e$: phase variation
- φ_e : phase of electron wave
- D : film thickness
- \mathbf{B}_0 : magnetic flux density perpendicular to beam direction
- \mathbf{s}_b : beam direction

- Magnetization influences phase of electron wave
- Phase gradient is perpendicular to B_0
- Lines of constant phase are parallel to B_0
- Flux between two lines is equal to flux quantum h/q_e

Electron Holography:

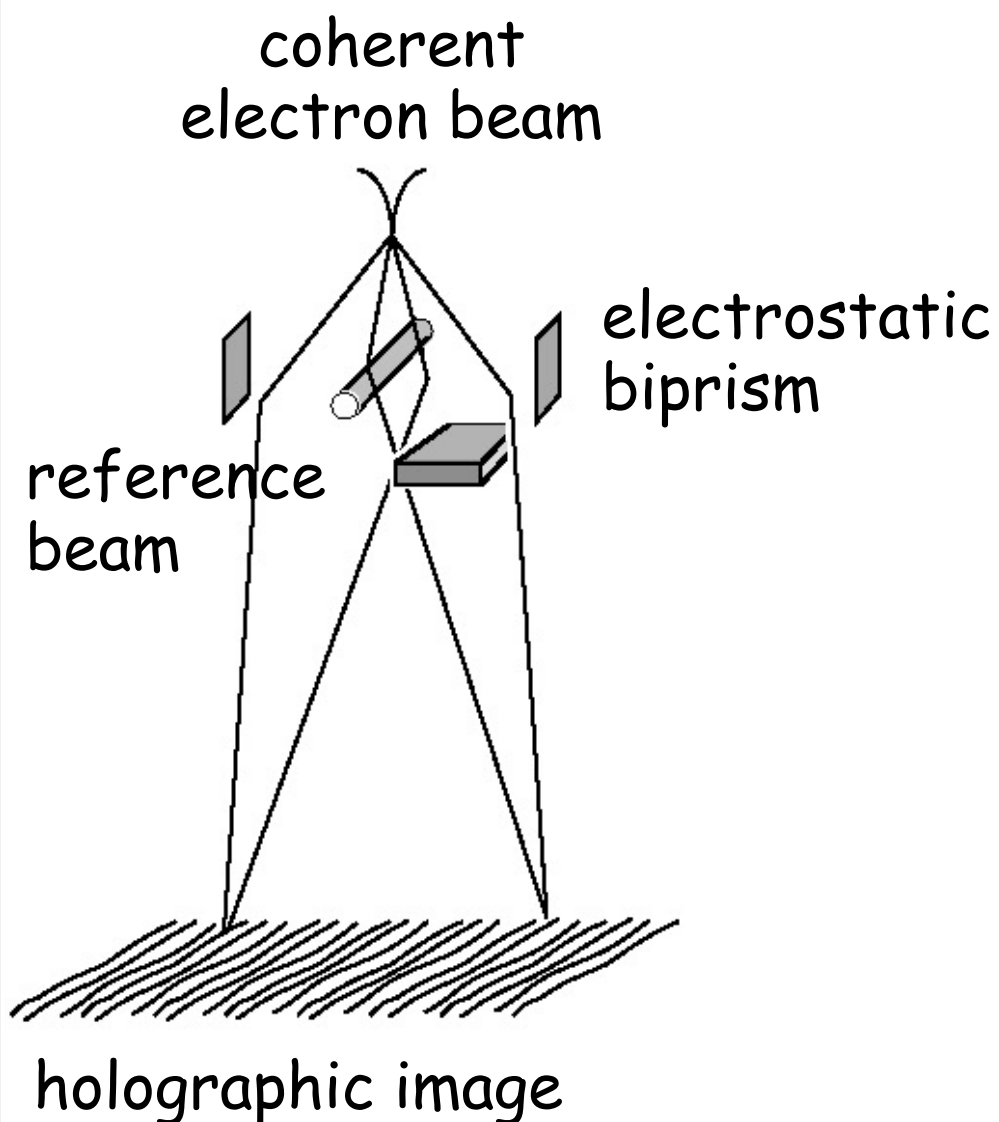
- Interference pattern of 2 electron waves shifted in phase
- Evaluation in optical interferometer

7. Transmission Electron Microscopy

7.4 Electron Holography

Off-axis holography (Tonomura et al. 1980)

Generation of hologram



Optical reconstruction

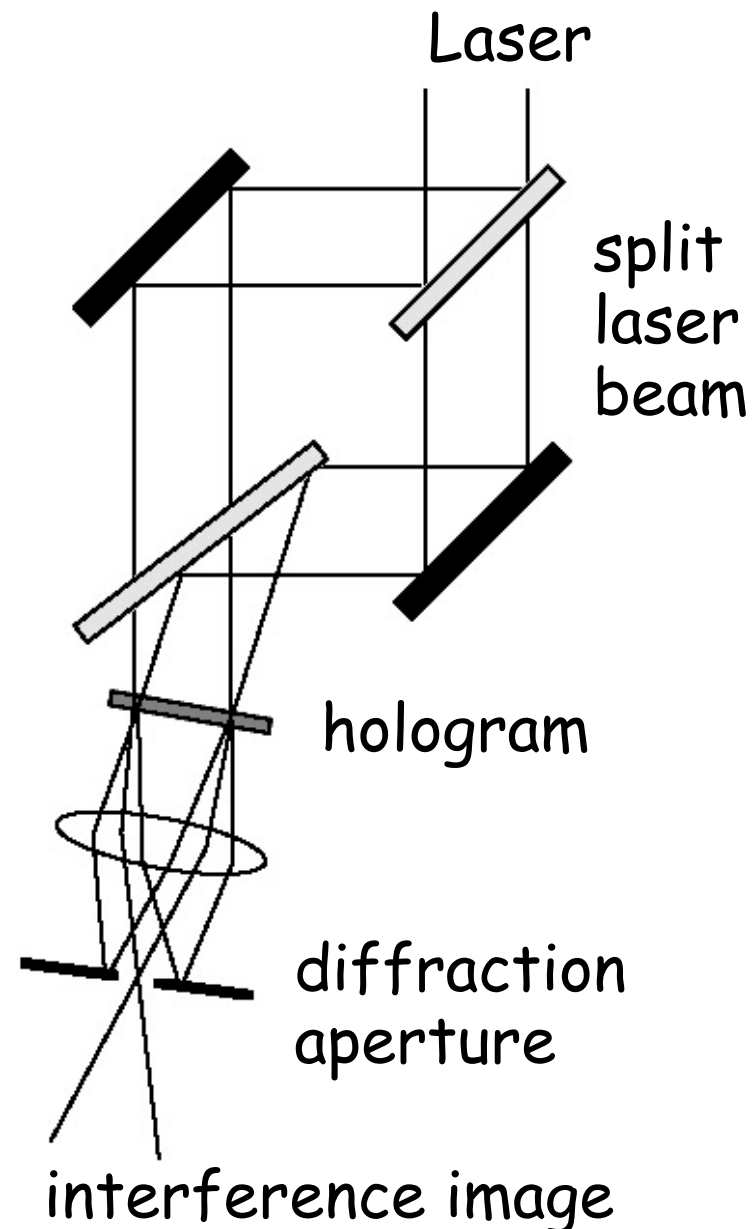
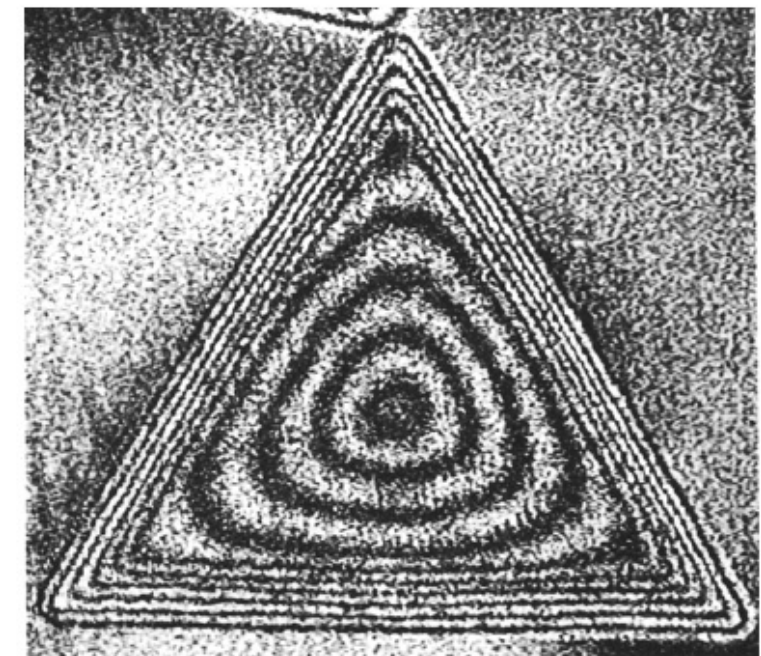
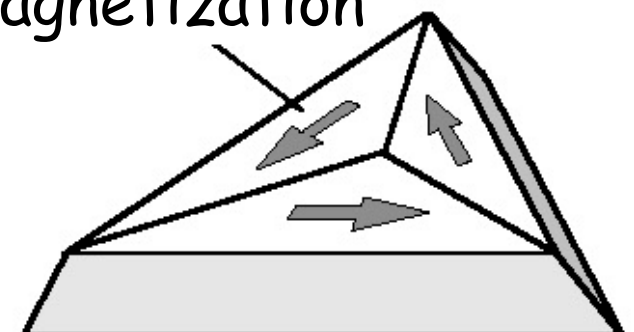


Image shows lines of constant phase



magnetization

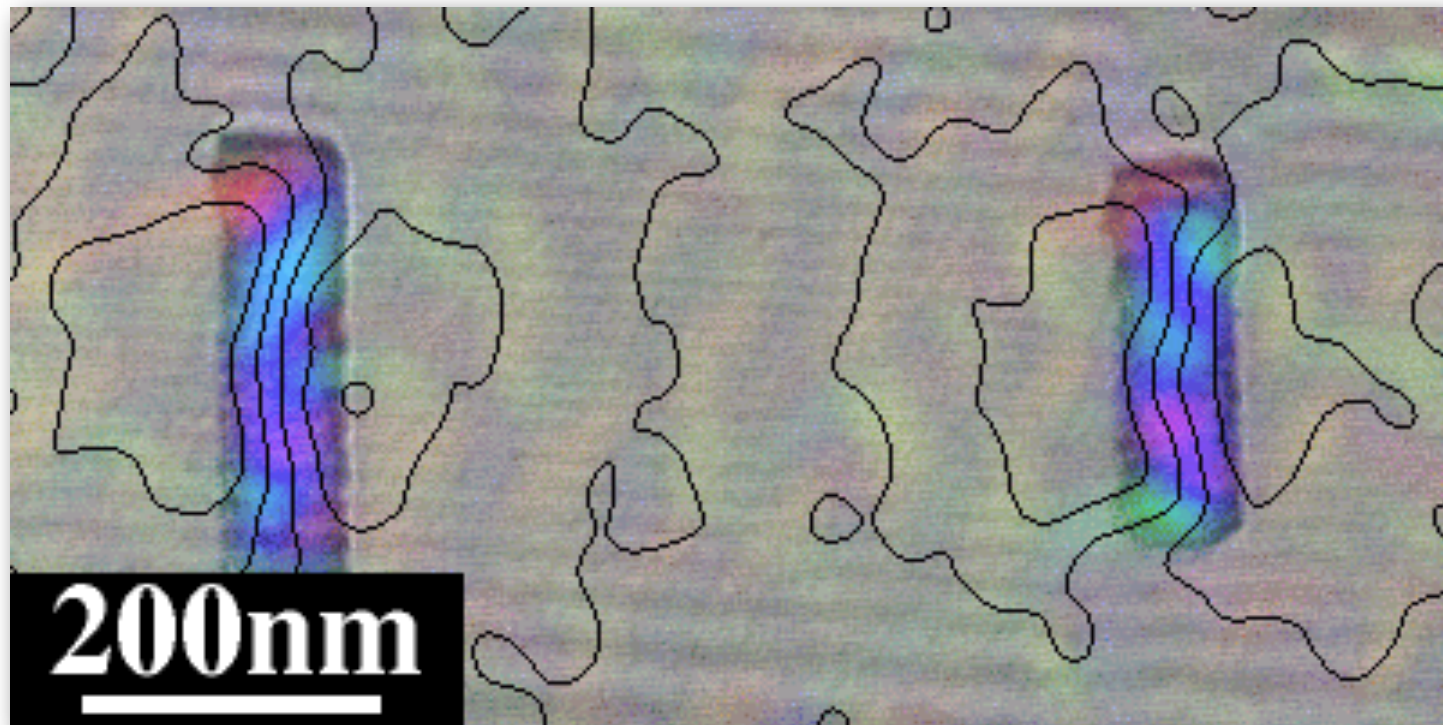


7. Transmission Electron Microscopy

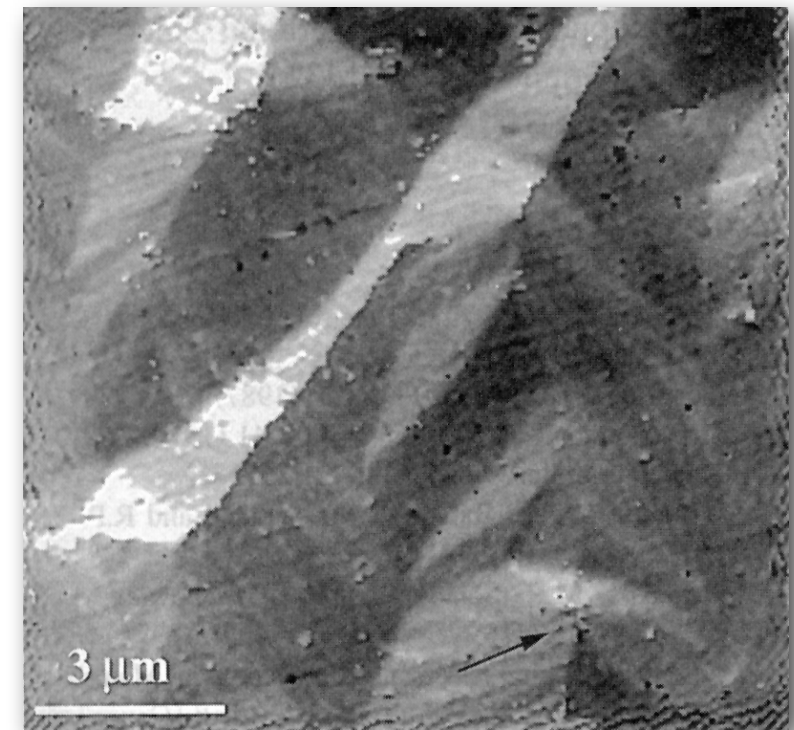
7.4 Electron Holography

Differential Holography (Mankos et al. 1994)

- Both interfering beams pass through sample along slightly different paths (distance: 10 nm)
- Reconstruction contains information about their phase difference
 - phase gradient is recorded, which is proportional to magnetization
 - “real” domain images like in Kerr microscopy
- Quantitative information about magnetization direction at high resolution



Co/Au/Ni/Al multilayer
(courtesy M. McCartney)



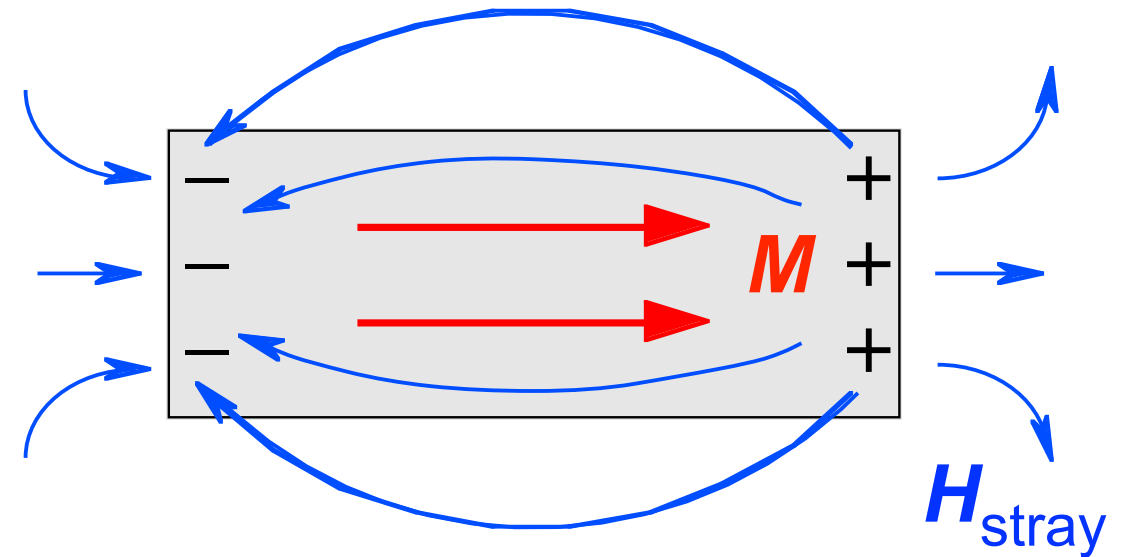
30 nm Co film
(courtesy M. Scheinfein)

Sensitivity of imaging methods

$$\mathbf{B} = \mu_0 (\mathbf{H} + \mathbf{M}) \quad (\mathbf{H} = \mathbf{H}_{\text{ext}} + \mathbf{H}_{\text{stray}})$$

$$\text{div } \mathbf{B} = 0$$

$$\downarrow$$
$$\text{div } \mathbf{H}_{\text{stray}} = -\text{div } \mathbf{M}$$



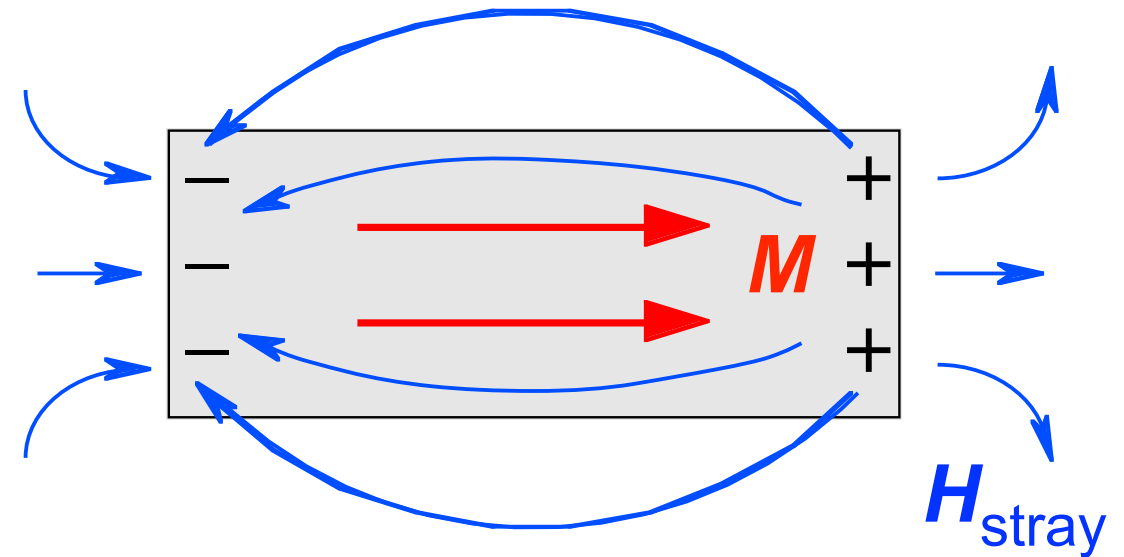
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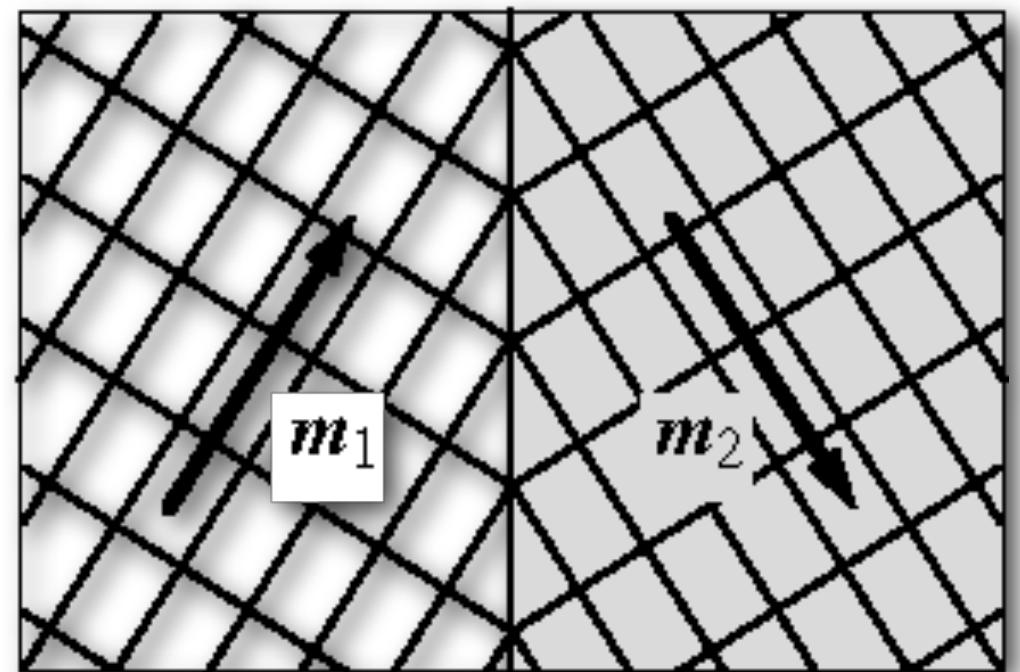
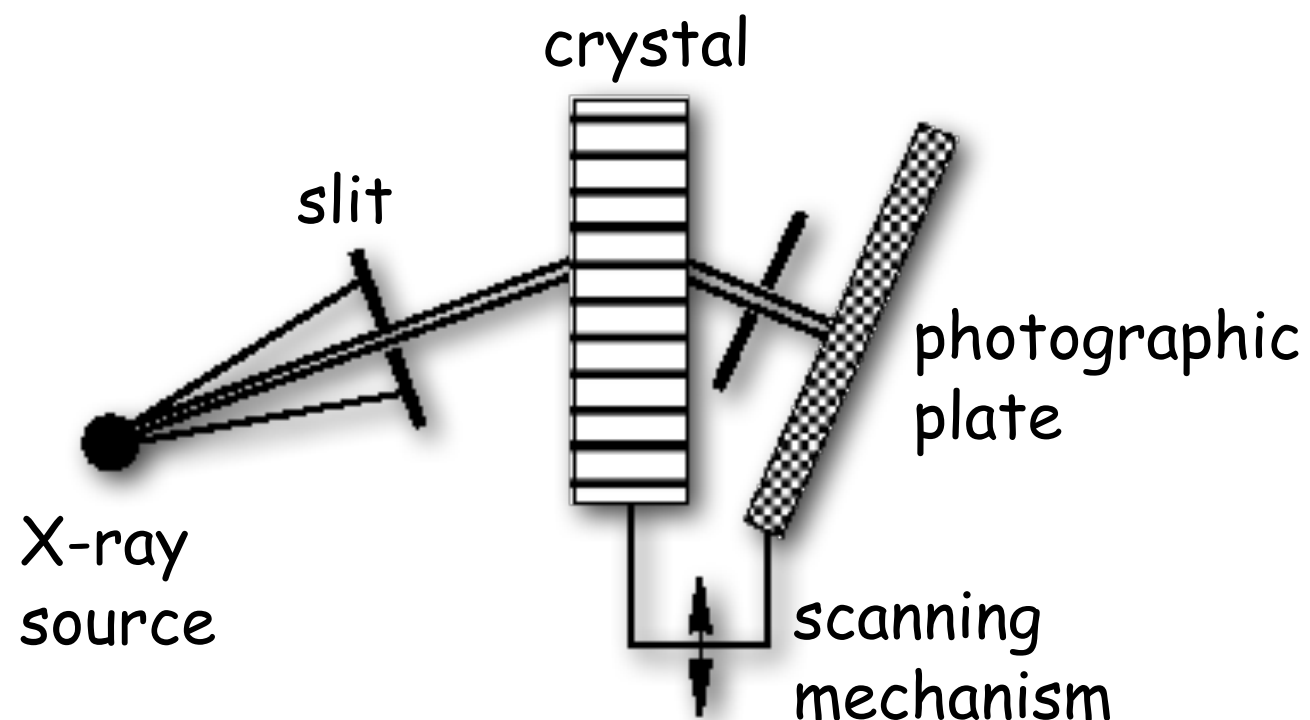
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 5. X-ray spectroscopy
 6. Polarized electrons (SEMPA, SPT)
- Sensitive to \mathbf{B}
 7. Transmission electron microscopy
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 8. X-ray, neutron scattering

8.1 X-ray Topography

- Plane-parallel X-ray beam, restricted to narrow strip
- Bragg condition fulfilled for some set of lattice planes
- Diffracted beam recorded by photographic plate
- Crystal and plate are advanced synchronously (scanning)

Contrast mechanism:

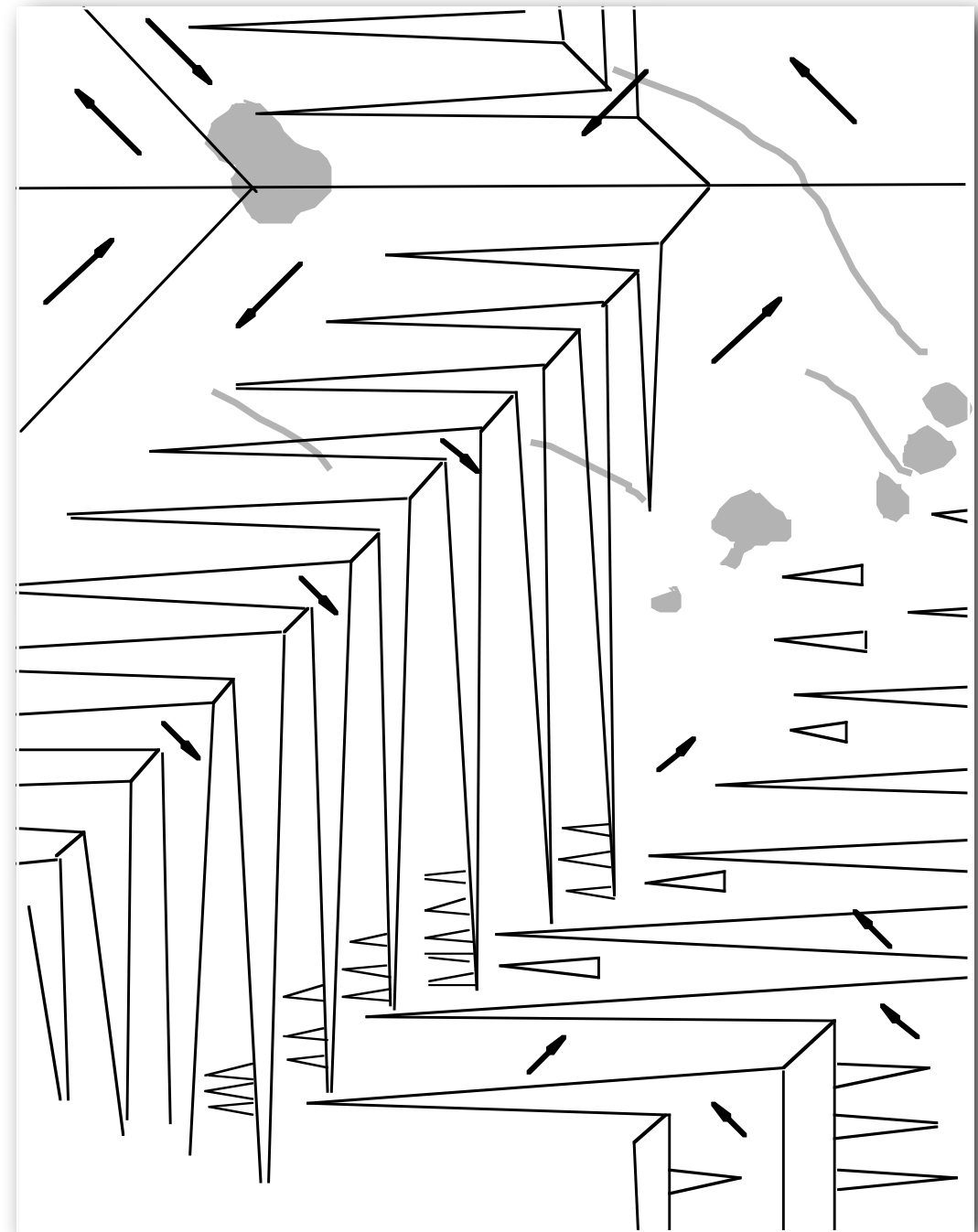
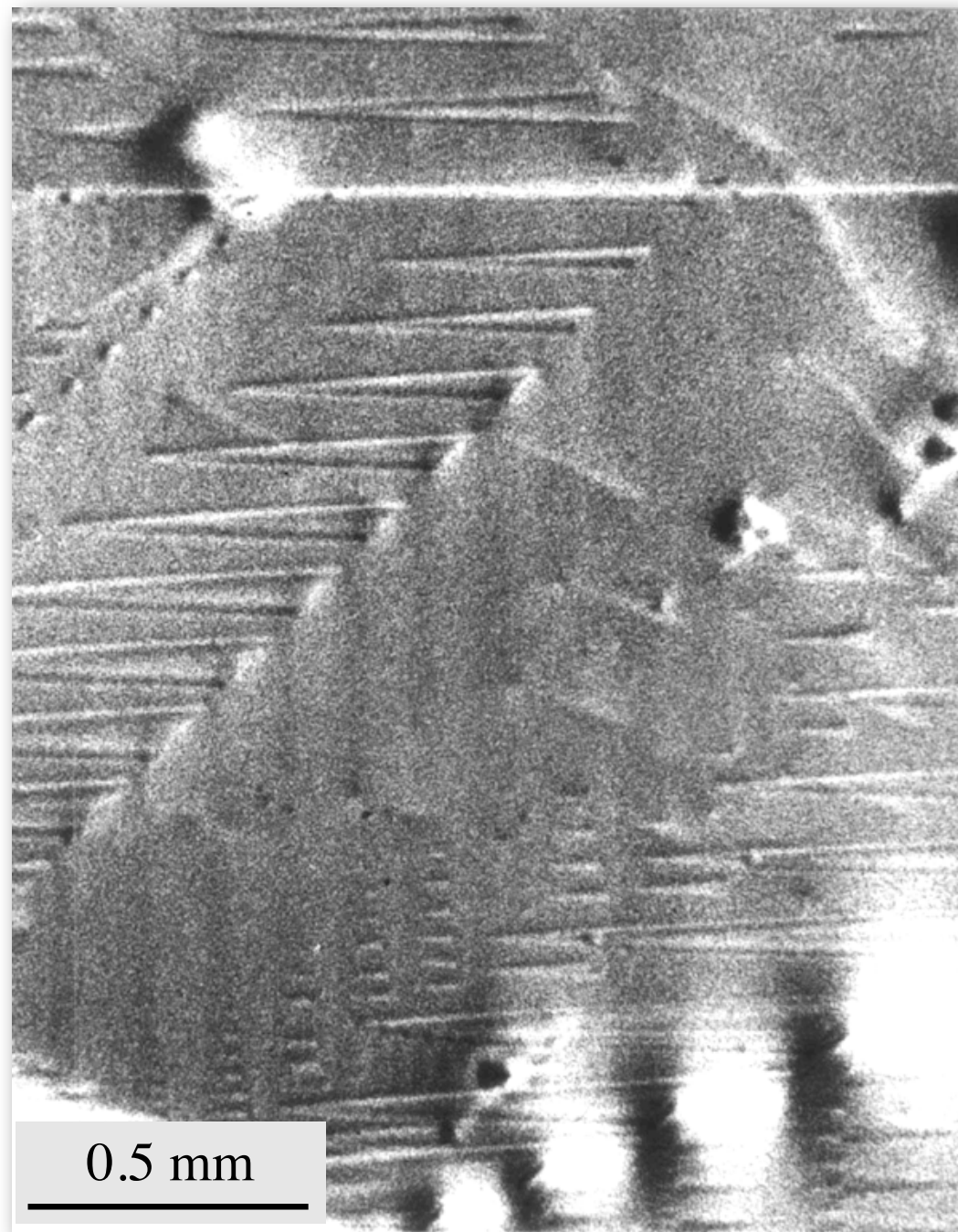
- Magnetostrictive strains disturb Bragg reflection
- Contrast at those positions, where rotation or spacing of lattice changes



Change of lattice orientation at 90° wall
(10 - 5 radian)

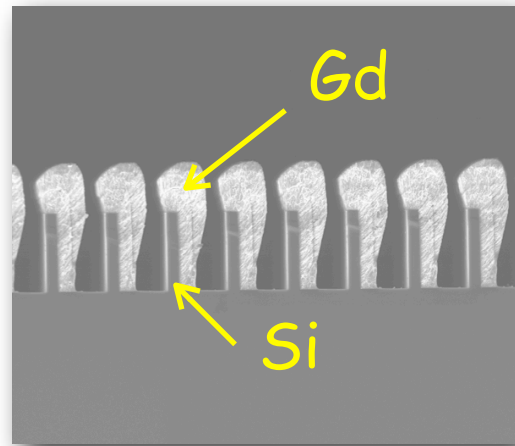
8.1 X-ray Topography

X-ray topogram of fir-tree domains on slightly misoriented (100) FeSi crystal (0.1 mm thick)



(Courtesy J. Miltat)

8.2 Neutron Dark-Field Microscopy



C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

Unpolarized neutrons



Absorption
source grating

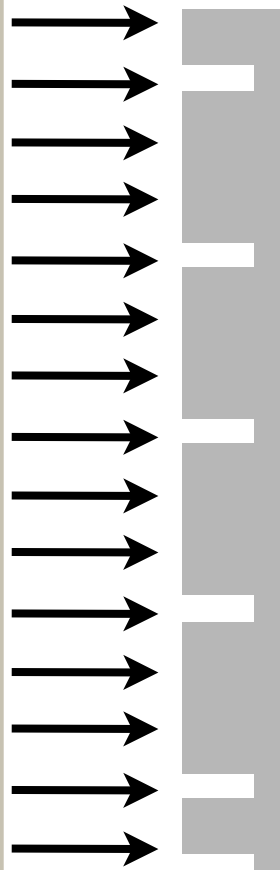
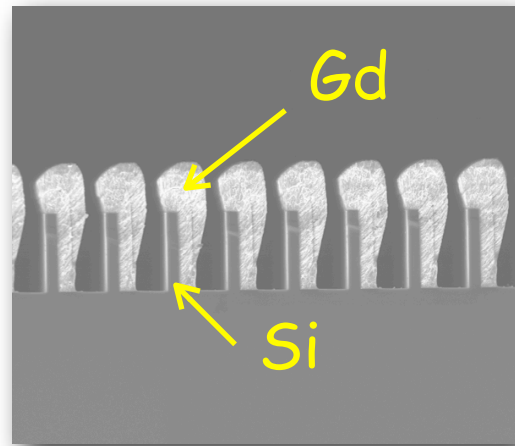


Image detector

8.2 Neutron Dark-Field Microscopy



C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

Unpolarized neutrons



Absorption
source grating

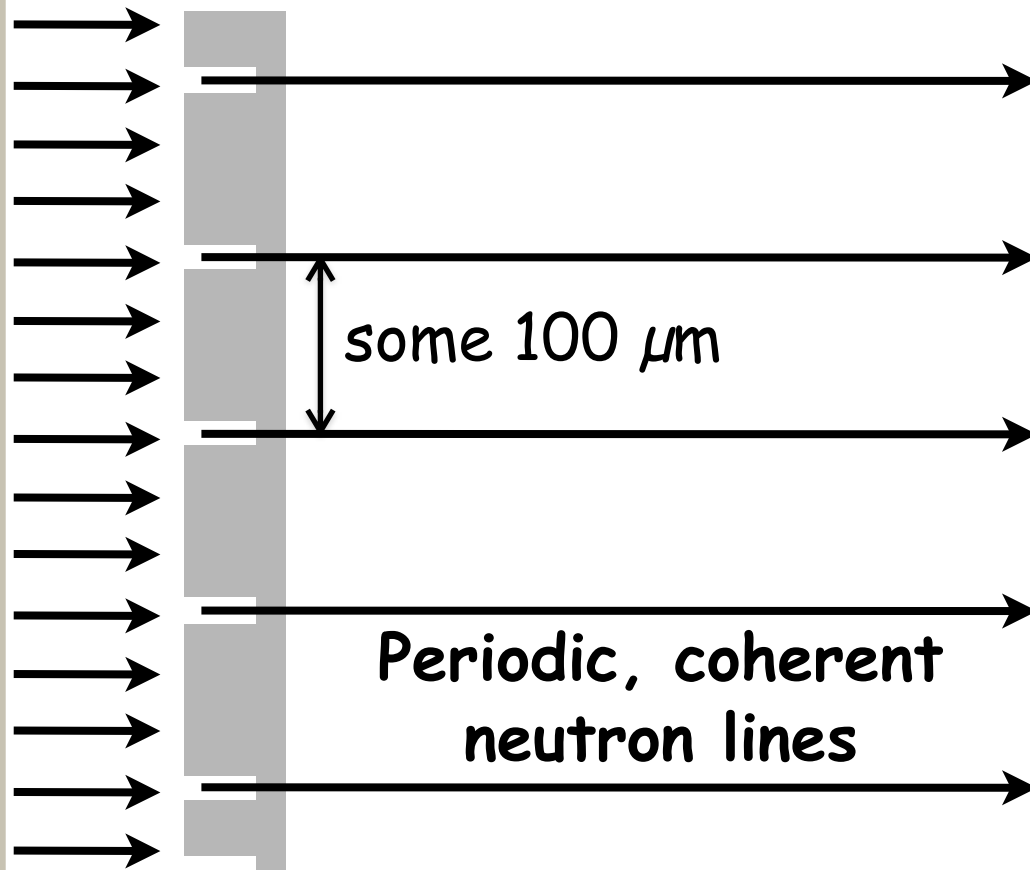
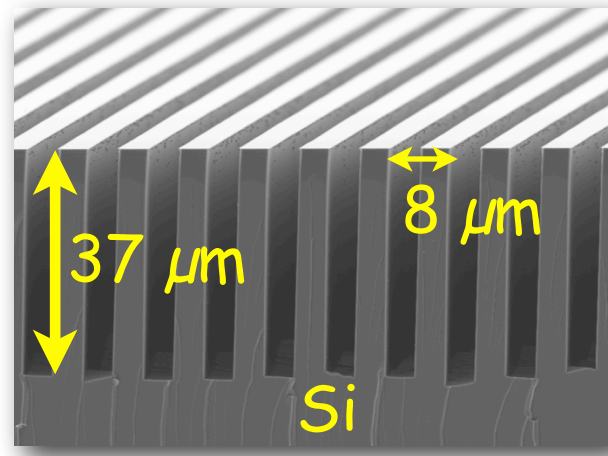
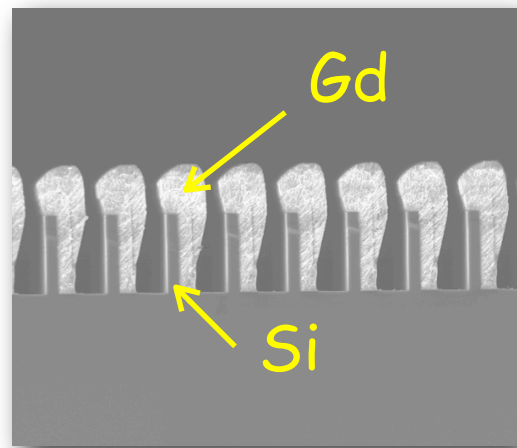


Image detector

8.2 Neutron Dark-Field Microscopy

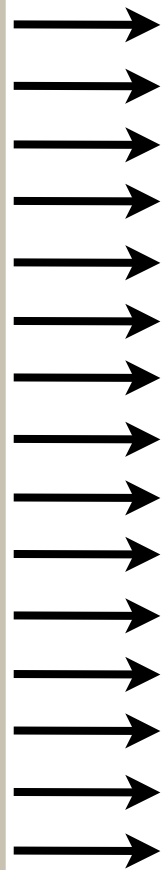


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

Unpolarized neutrons

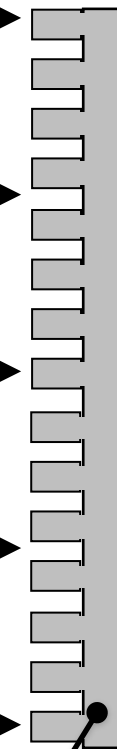


Absorption
source grating



some $100\ \mu\text{m}$

Phase
grating

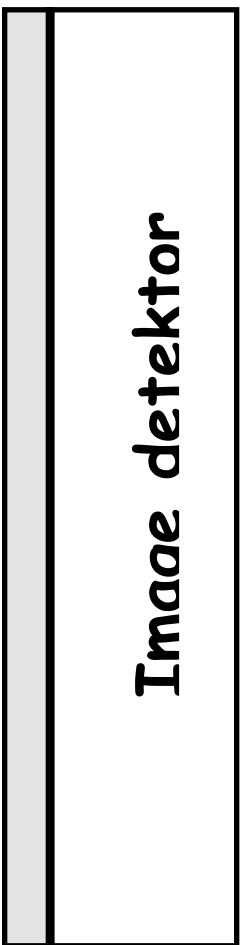


Periodic, coherent
neutron lines

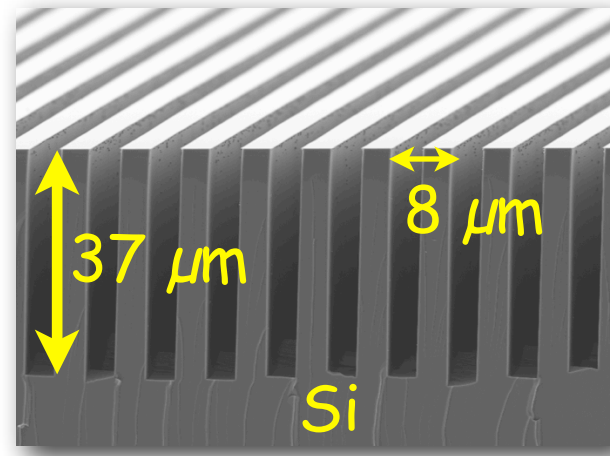
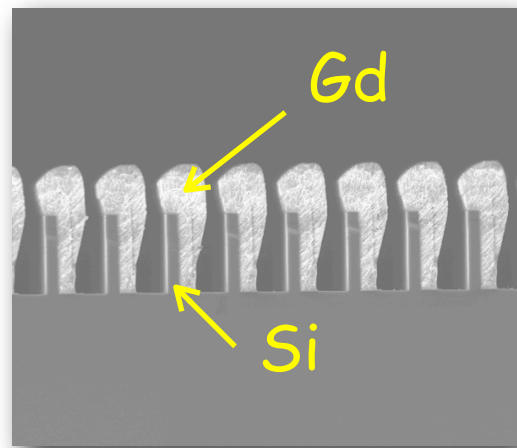
Phase shift

→ periodic phase modulation
→ Talbot effect

Image detector



8.2 Neutron Dark-Field Microscopy

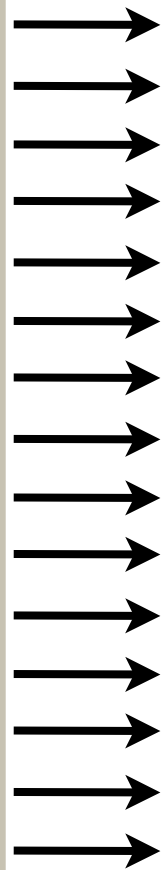


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

Unpolarized neutrons



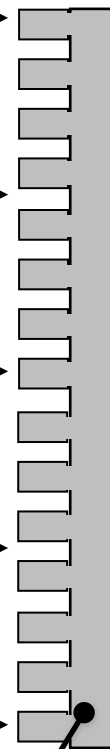
Absorption
source grating



some $100\ \mu\text{m}$

Periodic, coherent
neutron lines

Phase
grating



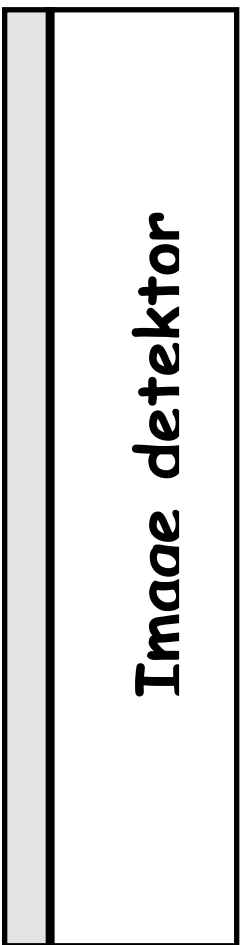
Phase shift

→ periodic phase modulation
→ Talbot effect

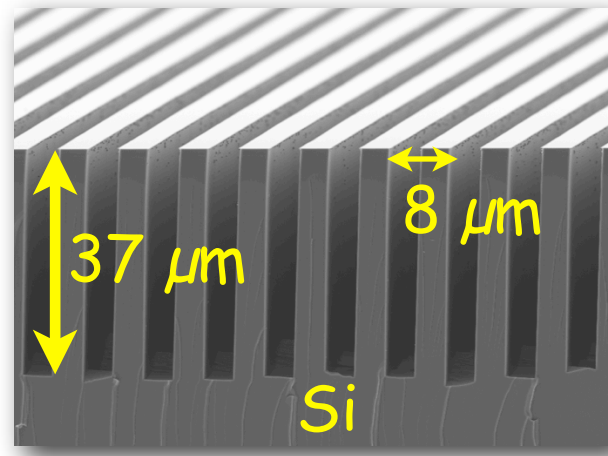
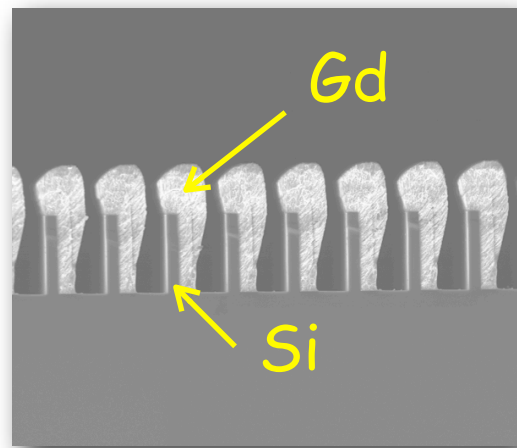


Interference pattern
(period $4\ \mu\text{m}$)

Image detector

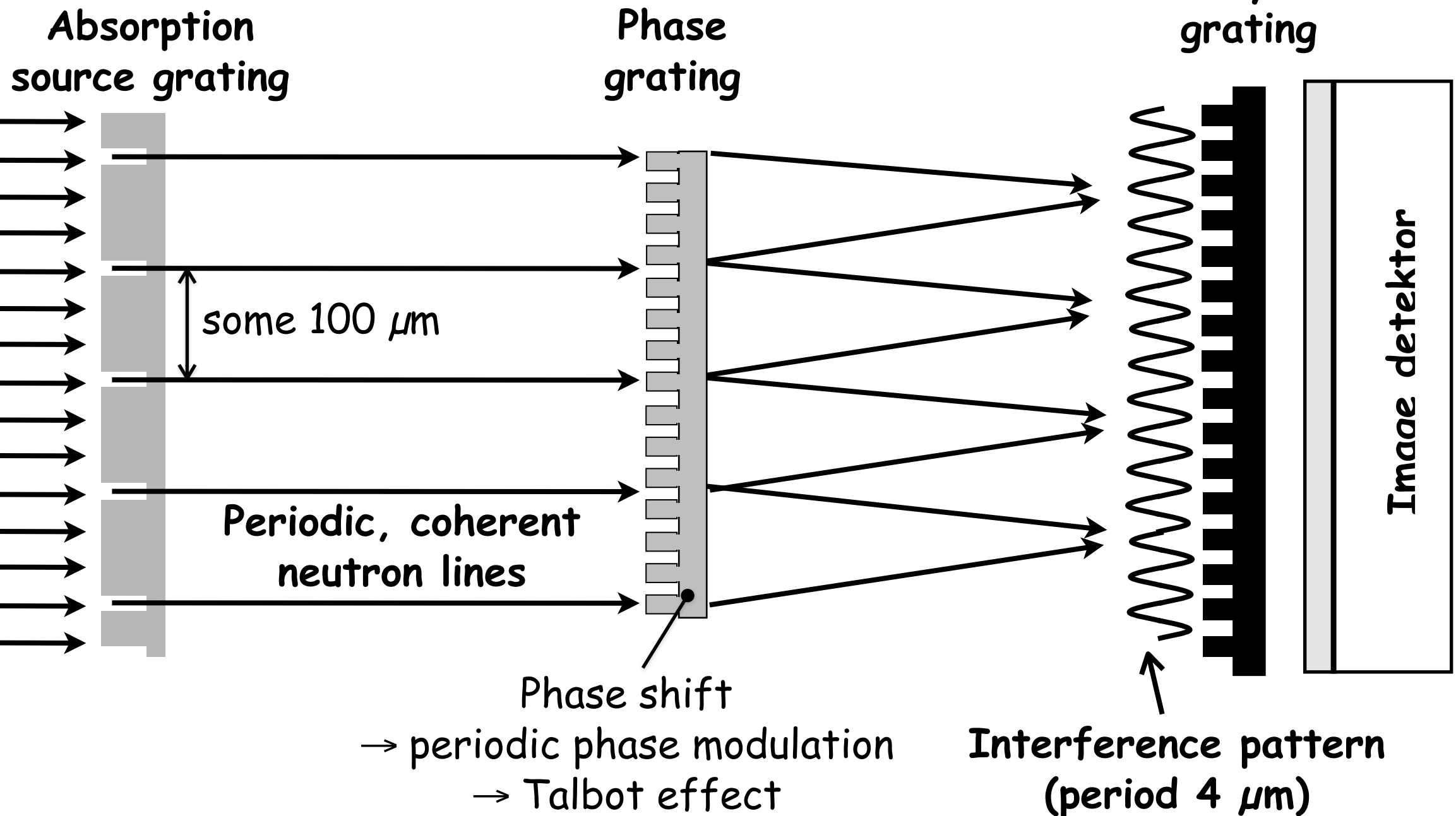


8.2 Neutron Dark-Field Microscopy

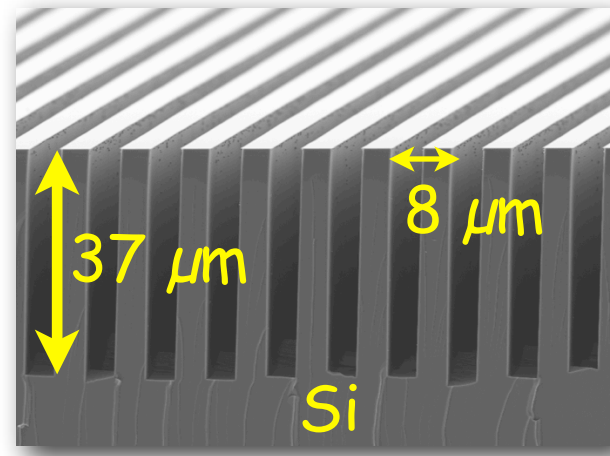
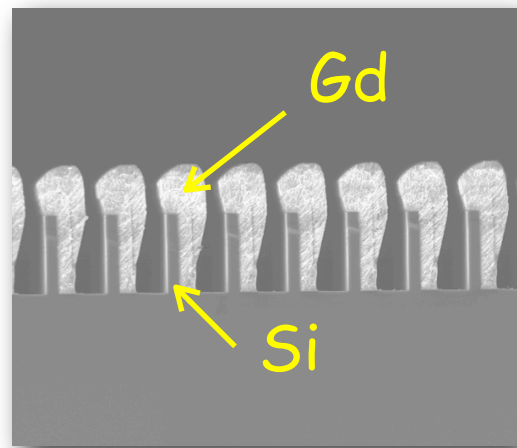


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

Unpolarized neutrons



8.2 Neutron Dark-Field Microscopy

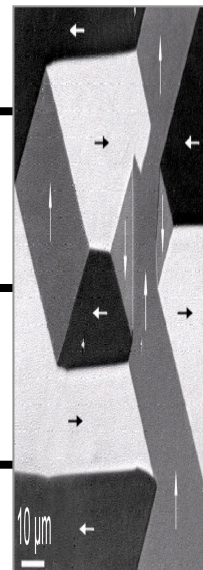
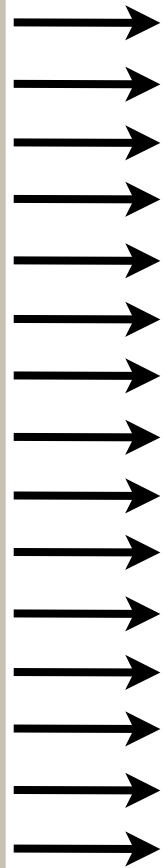


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

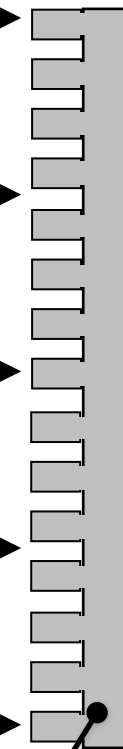
Unpolarized neutrons



Absorption
source grating



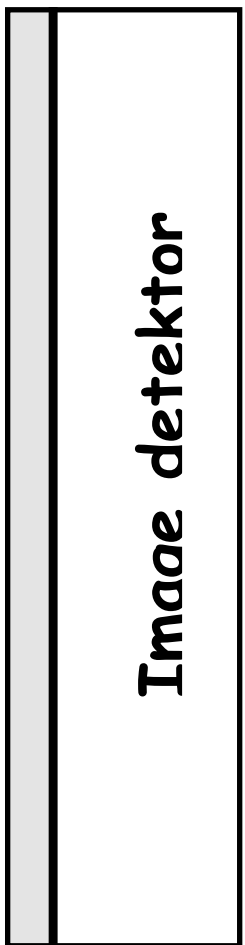
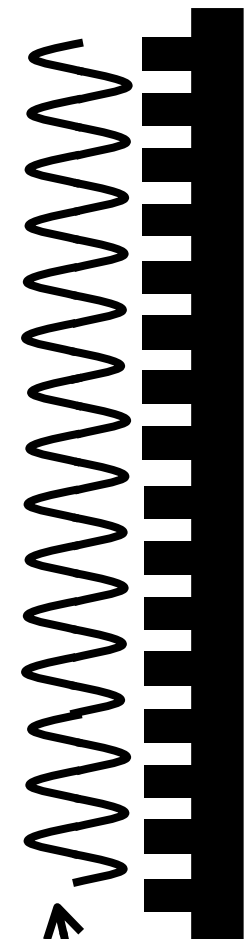
Phase
grating



Phase shift

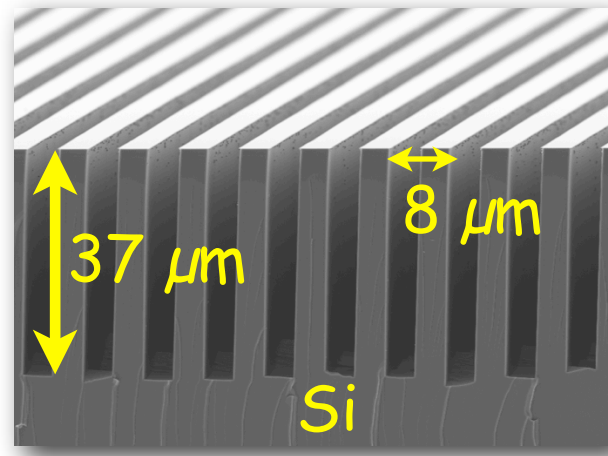
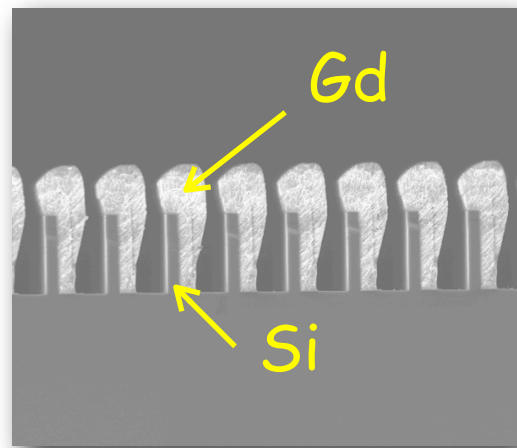
→ periodic phase modulation
→ Talbot effect

Absorption
analyser
grating



Interference pattern
(period $4\ \mu\text{m}$)

8.2 Neutron Dark-Field Microscopy

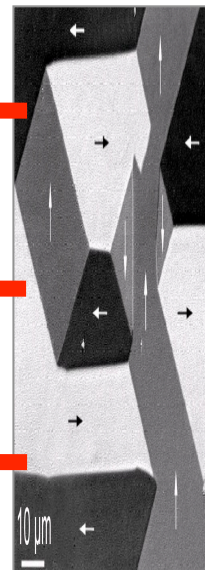
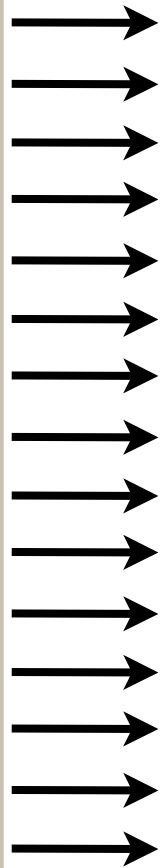


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

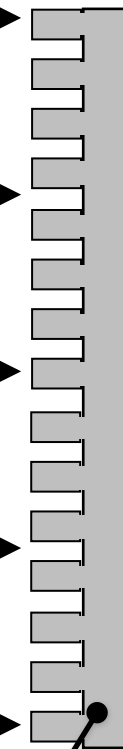
Unpolarized neutrons



Absorption
source grating



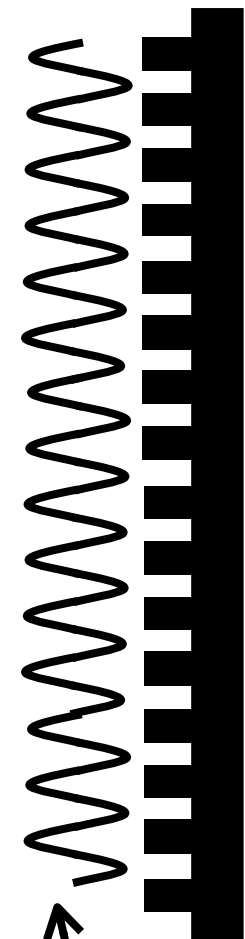
Phase
grating



Phase shift

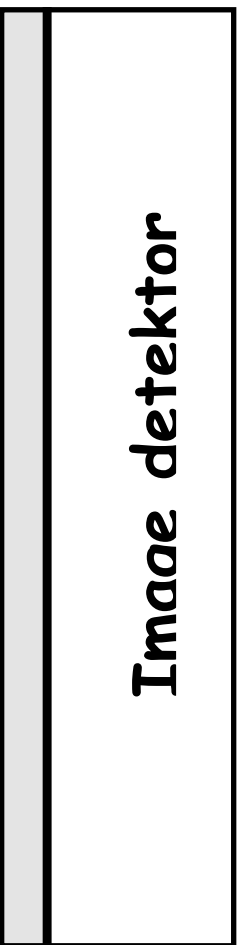
→ periodic phase modulation
→ Talbot effect

Absorption
analyser
grating

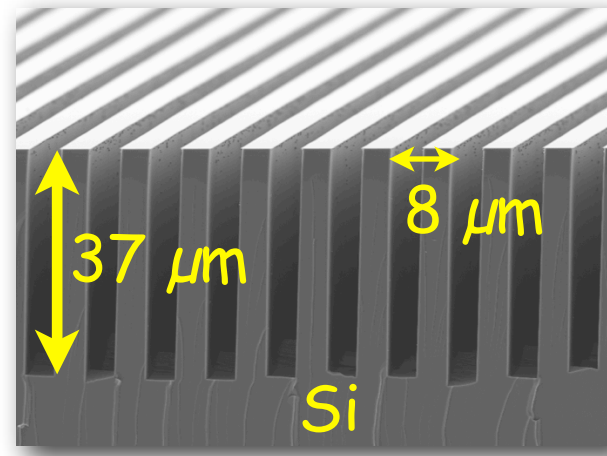
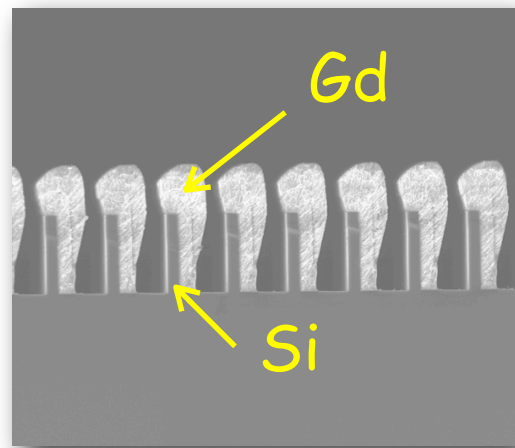


Interference pattern
(period $4\ \mu\text{m}$)

Image detector



8.2 Neutron Dark-Field Microscopy

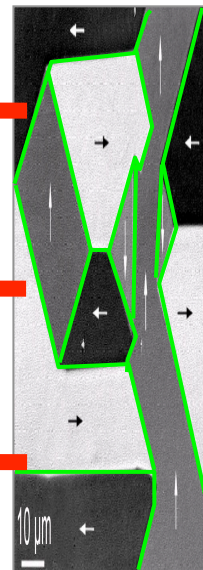
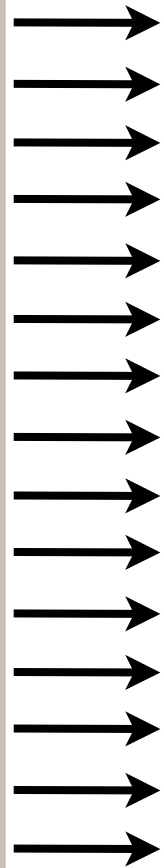


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

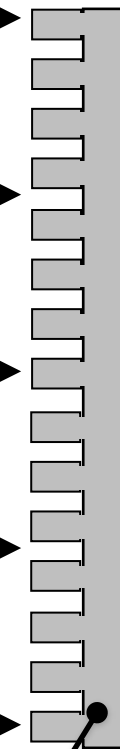
Unpolarized neutrons



Absorption
source grating



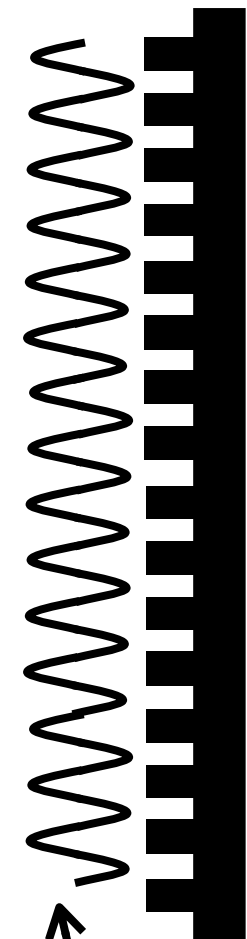
Phase
grating



Phase shift

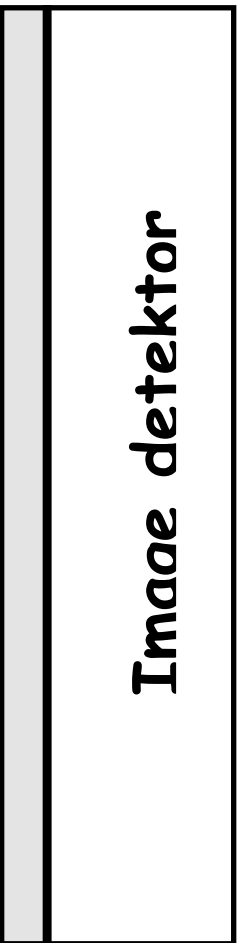
→ periodic phase modulation
→ Talbot effect

Absorption
analyser
grating

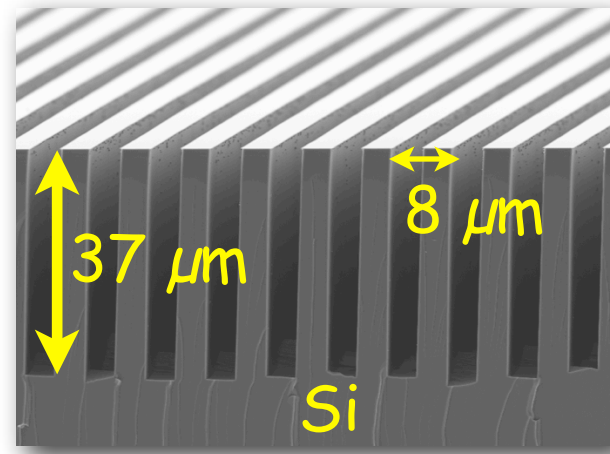
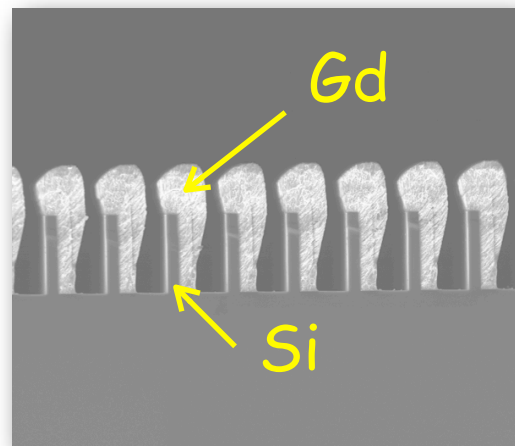


Interference pattern
(period $4\ \mu\text{m}$)

Image detector



8.2 Neutron Dark-Field Microscopy

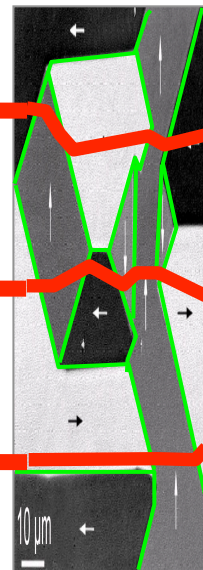
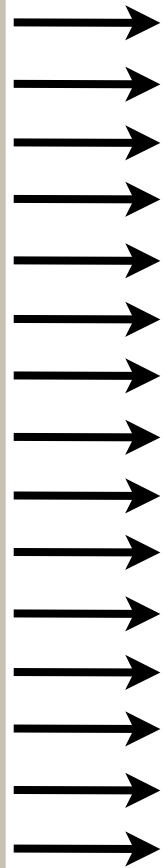


C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)

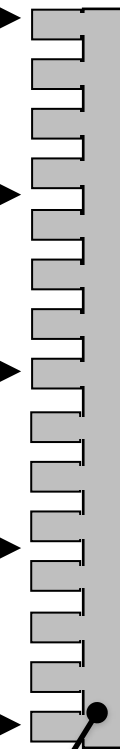
Unpolarized neutrons



Absorption
source grating



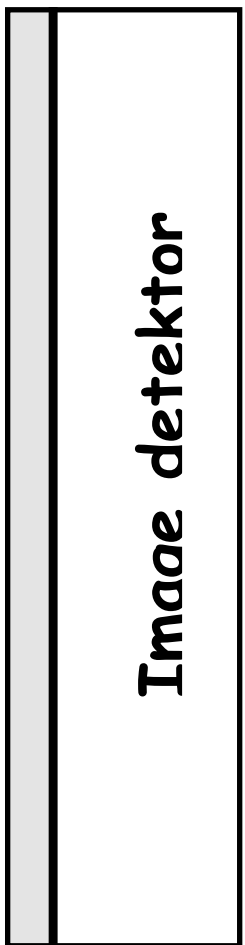
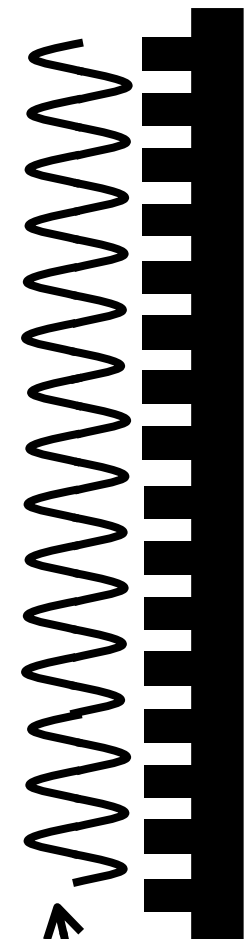
Phase
grating



Phase shift

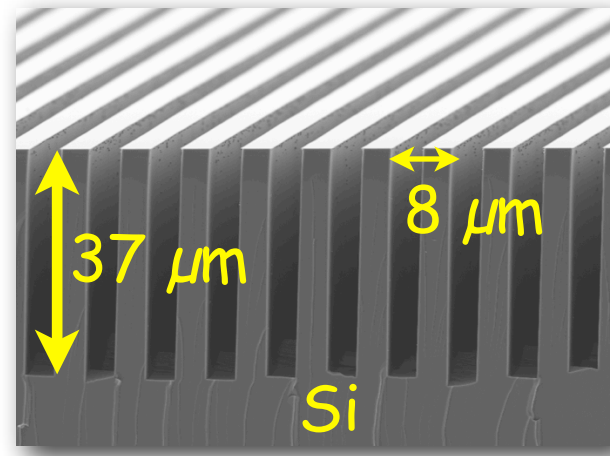
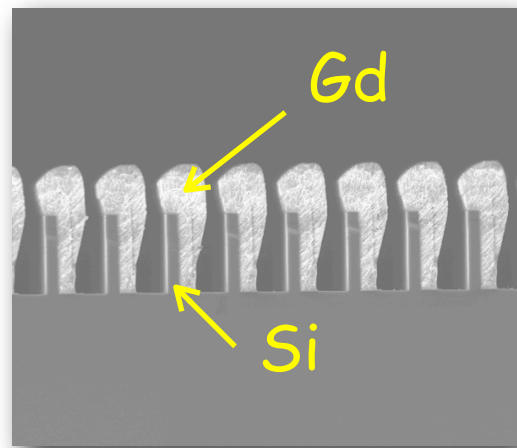
→ periodic phase modulation
→ Talbot effect

Absorption
analyser
grating

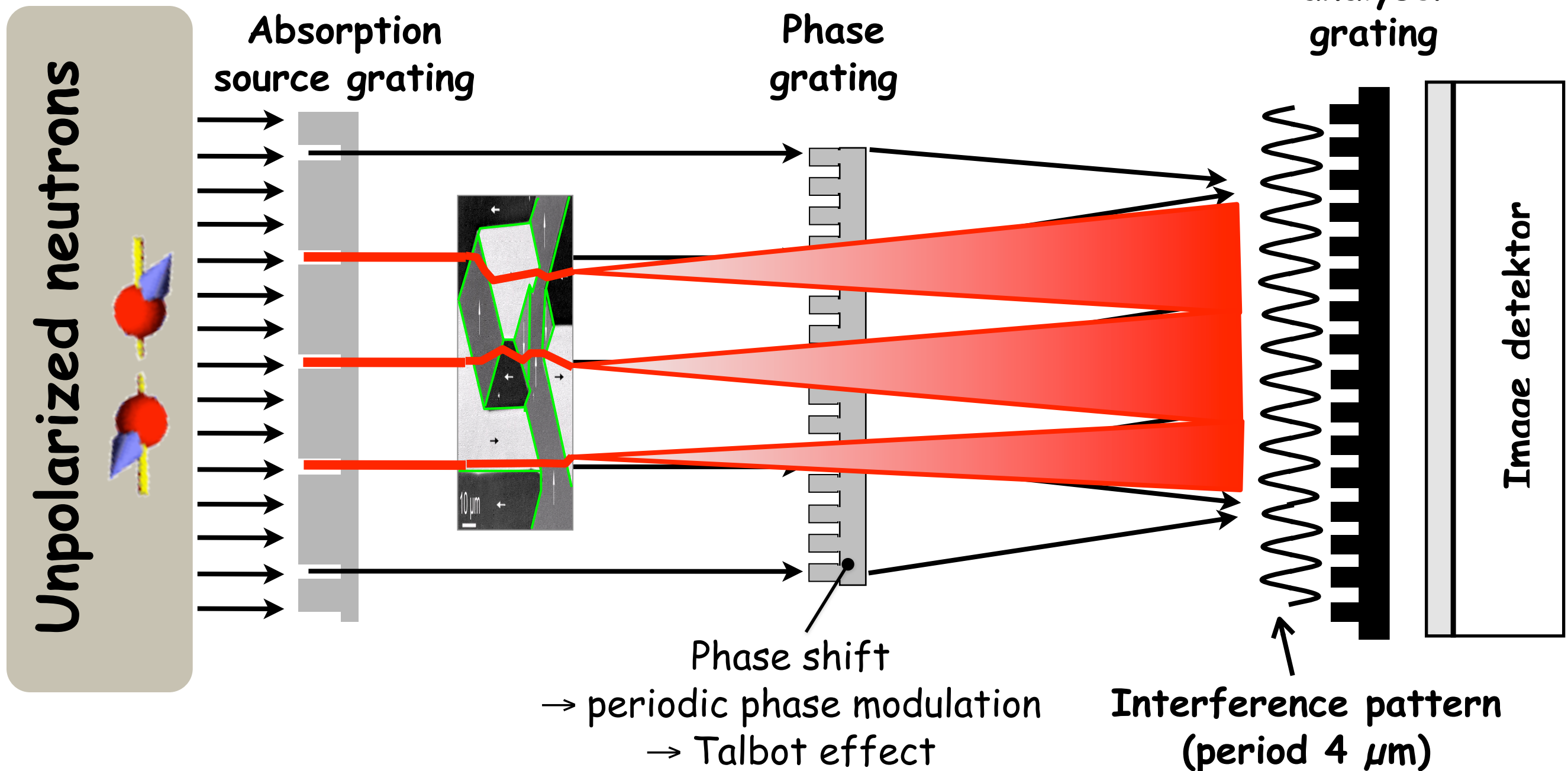


Interference pattern
(period $4\ \mu\text{m}$)

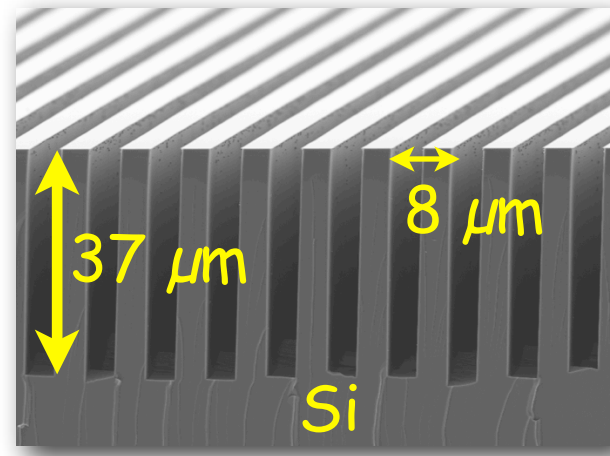
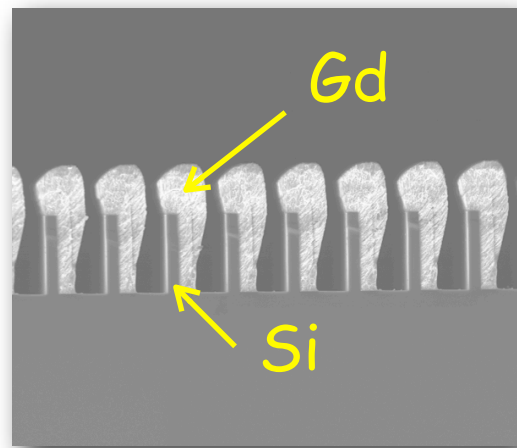
8.2 Neutron Dark-Field Microscopy



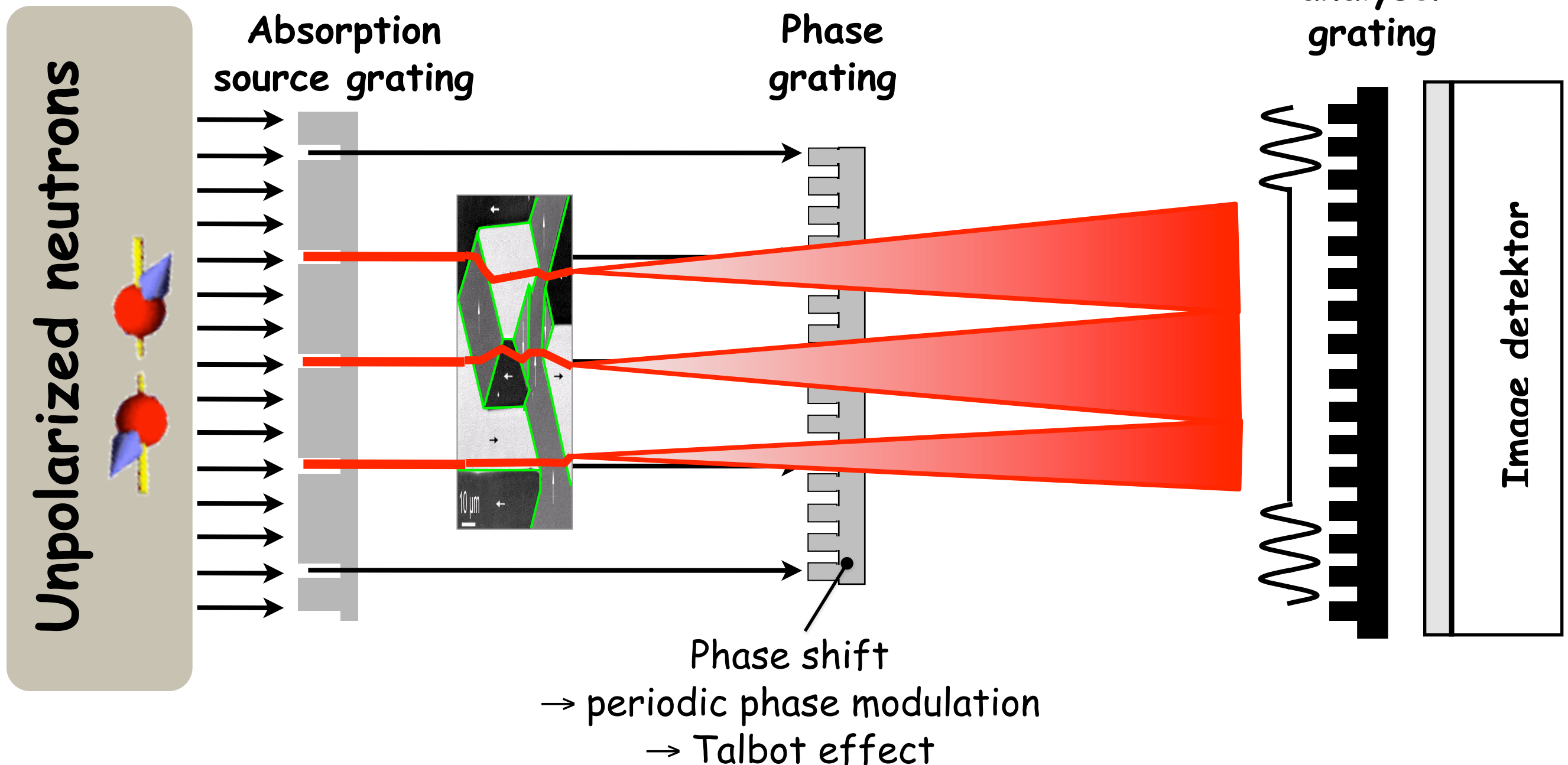
C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)



8.2 Neutron Dark-Field Microscopy



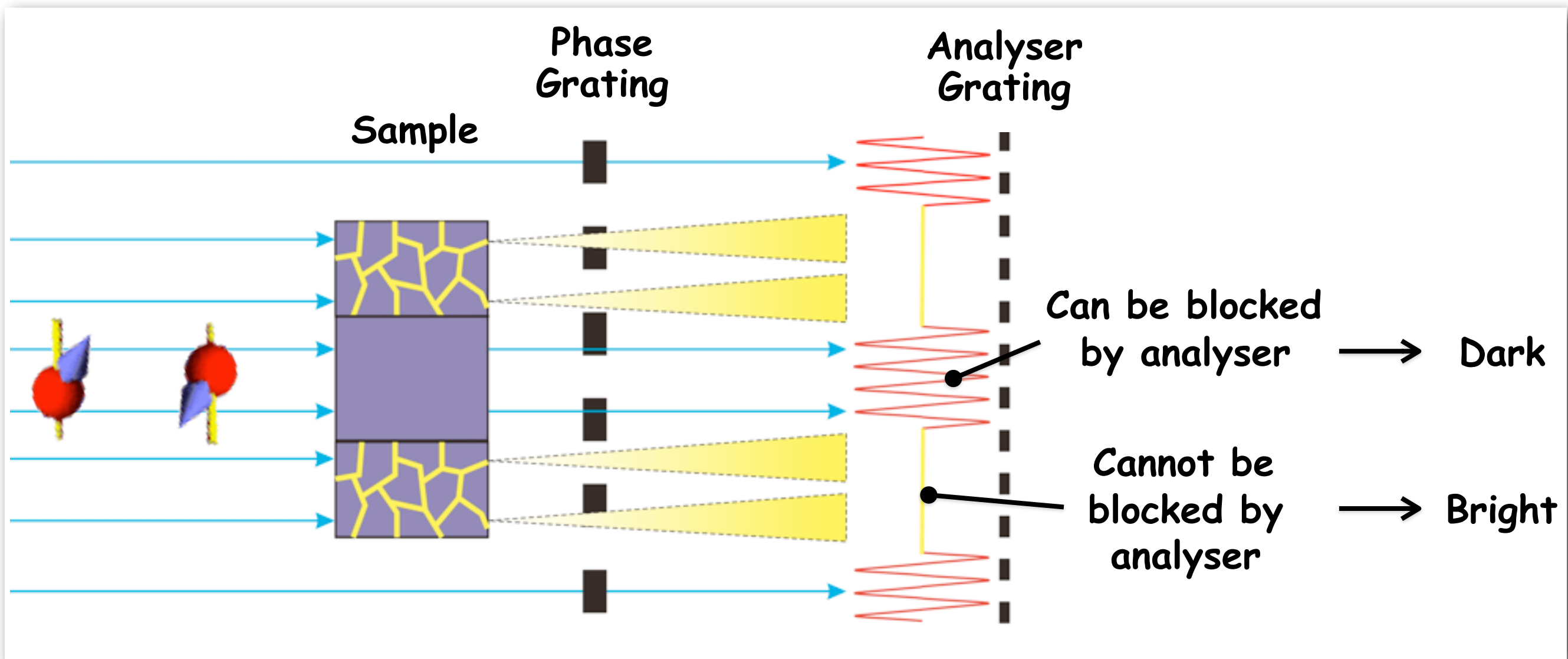
C. Grünzweig, et al.,
Phys. Rev. Lett. 101,
025504 (2008)



8.2 Neutron Dark-Field Microscopy

Principle:

Unpolarized neutrons, refracted at domain walls, locally destroy the interference pattern



→ Bright contrast is caused by refraction

→ Dark field imaging

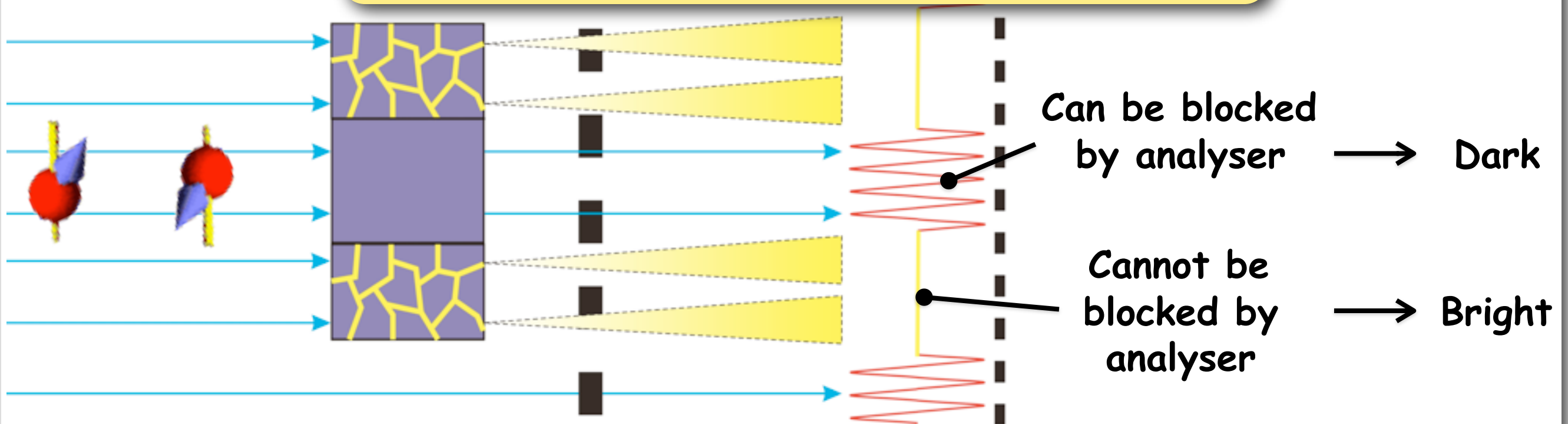
8.2 Neutron Dark-Field Microscopy

Unpolarized
local

Resolution:
Determined by pixel size of detector and scattering angle

walls,
rn

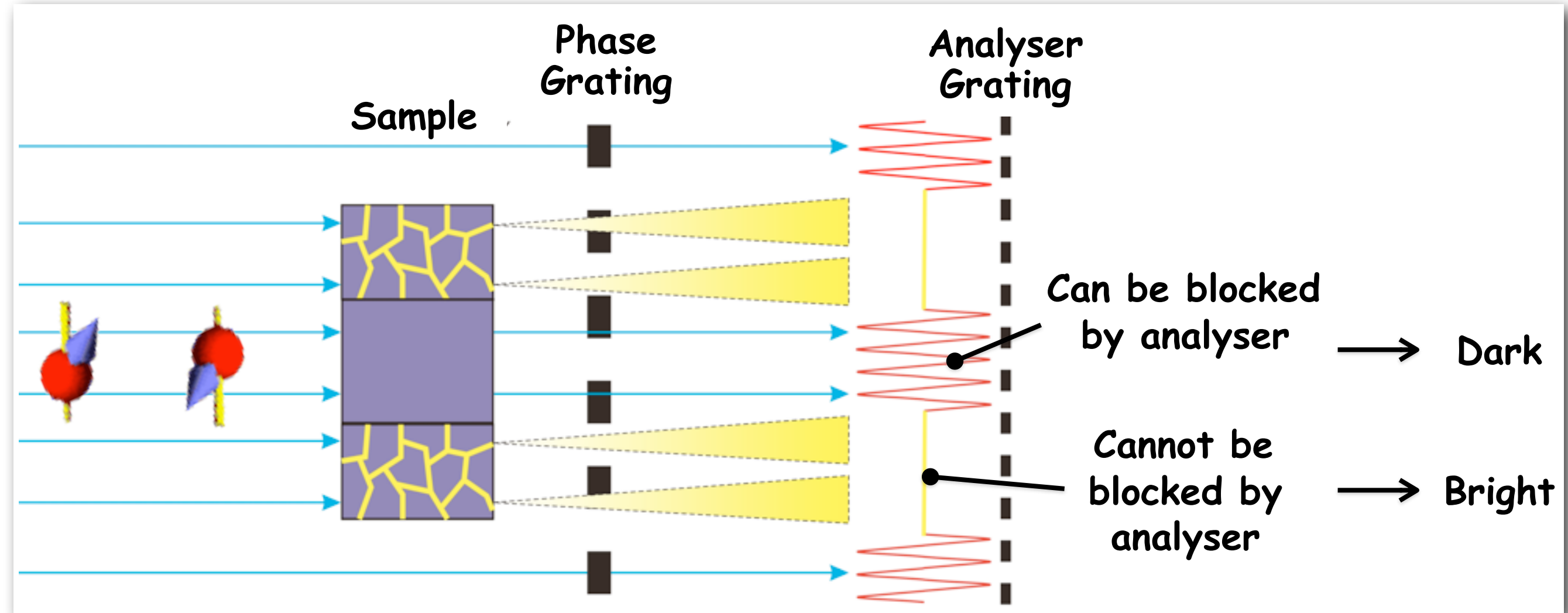
some 10 μm



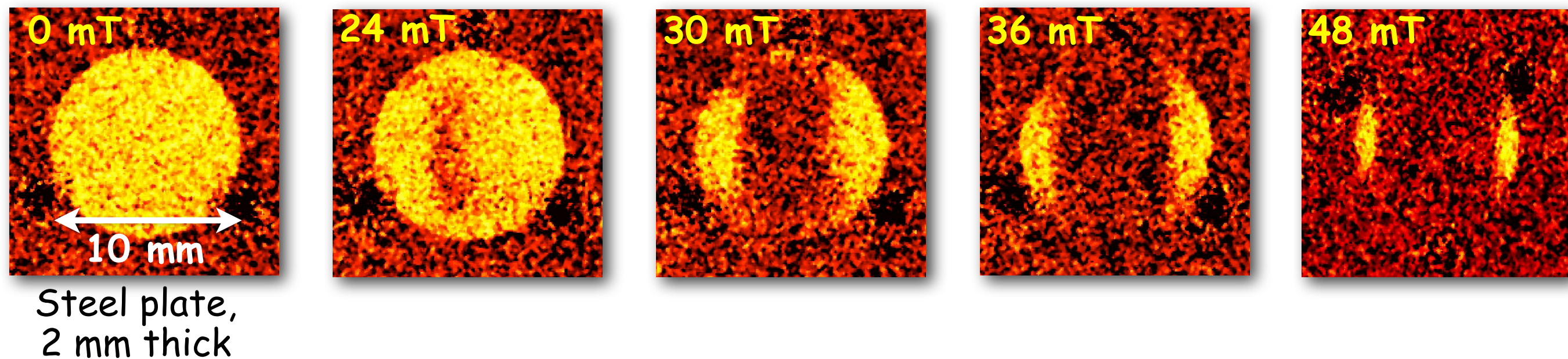
→ Bright contrast is caused by refraction

→ Dark field imaging

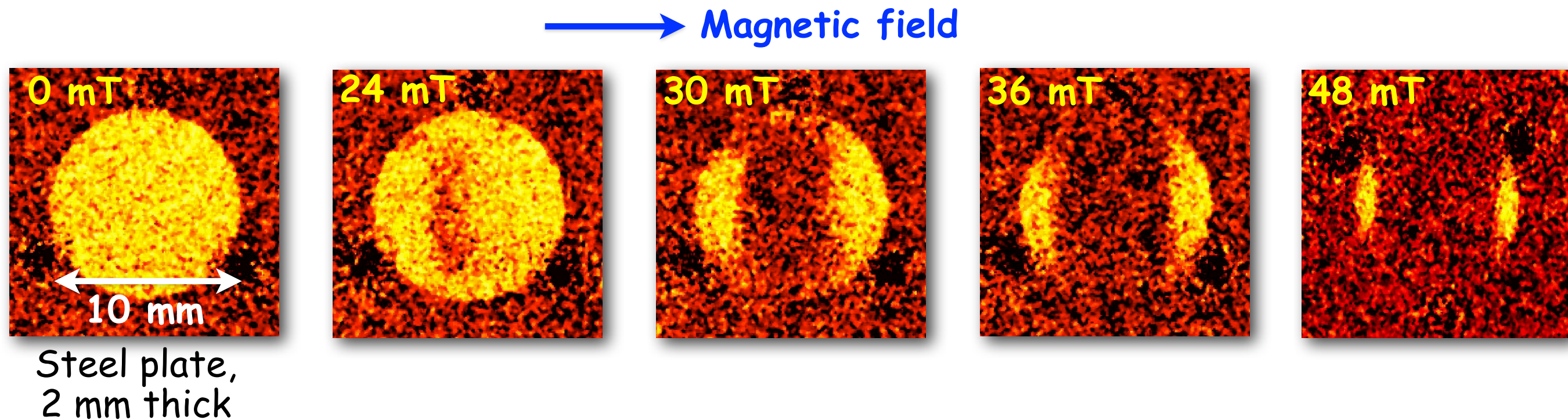
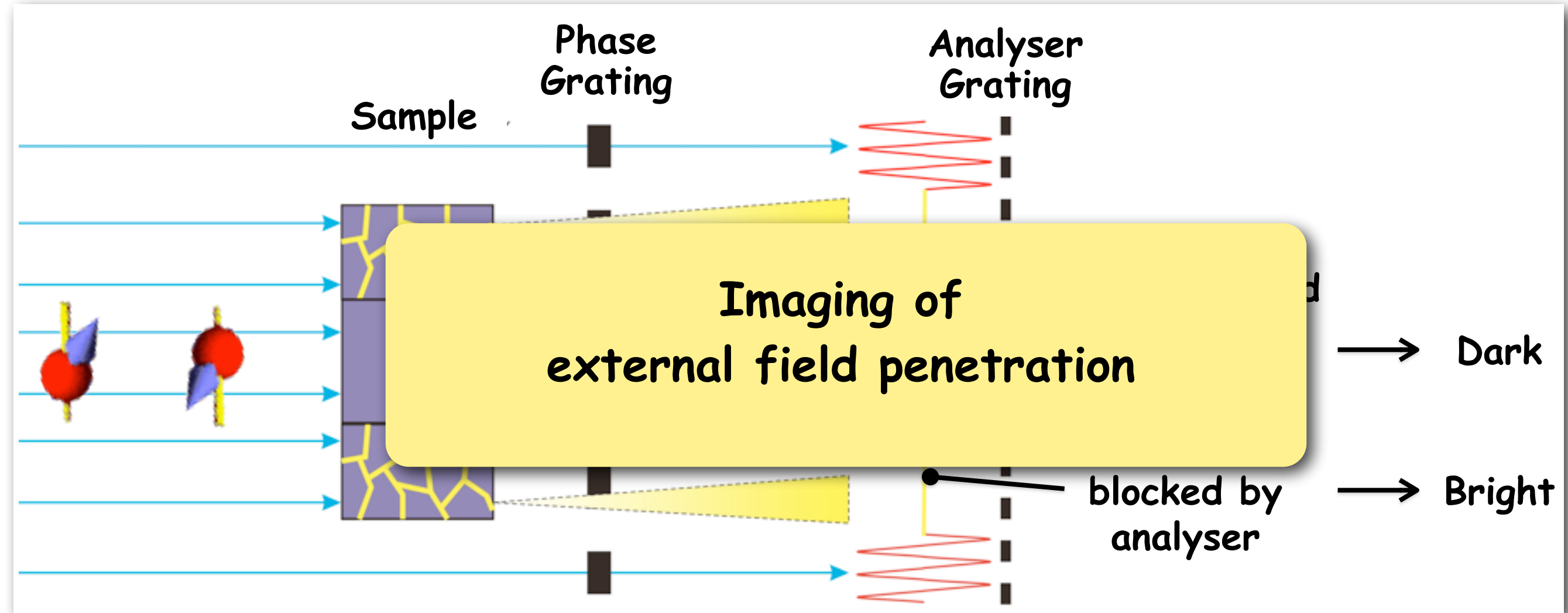
8.2 Neutron Dark-Field Microscopy



→ Magnetic field



8.2 Neutron Dark-Field Microscopy

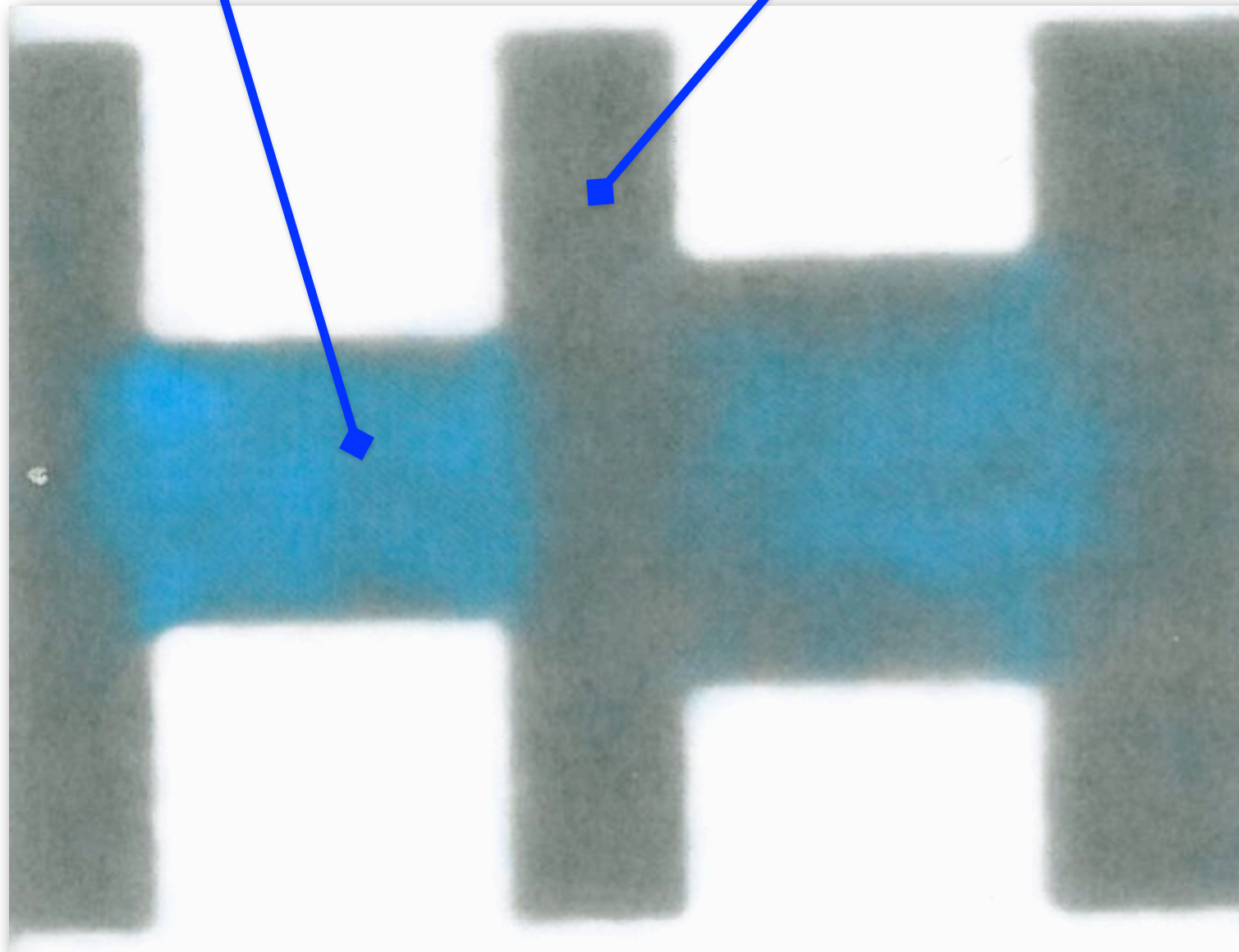


8.2 Neutron Dark-Field Microscopy

No walls:
higher flux density

Many walls:
lower flux density

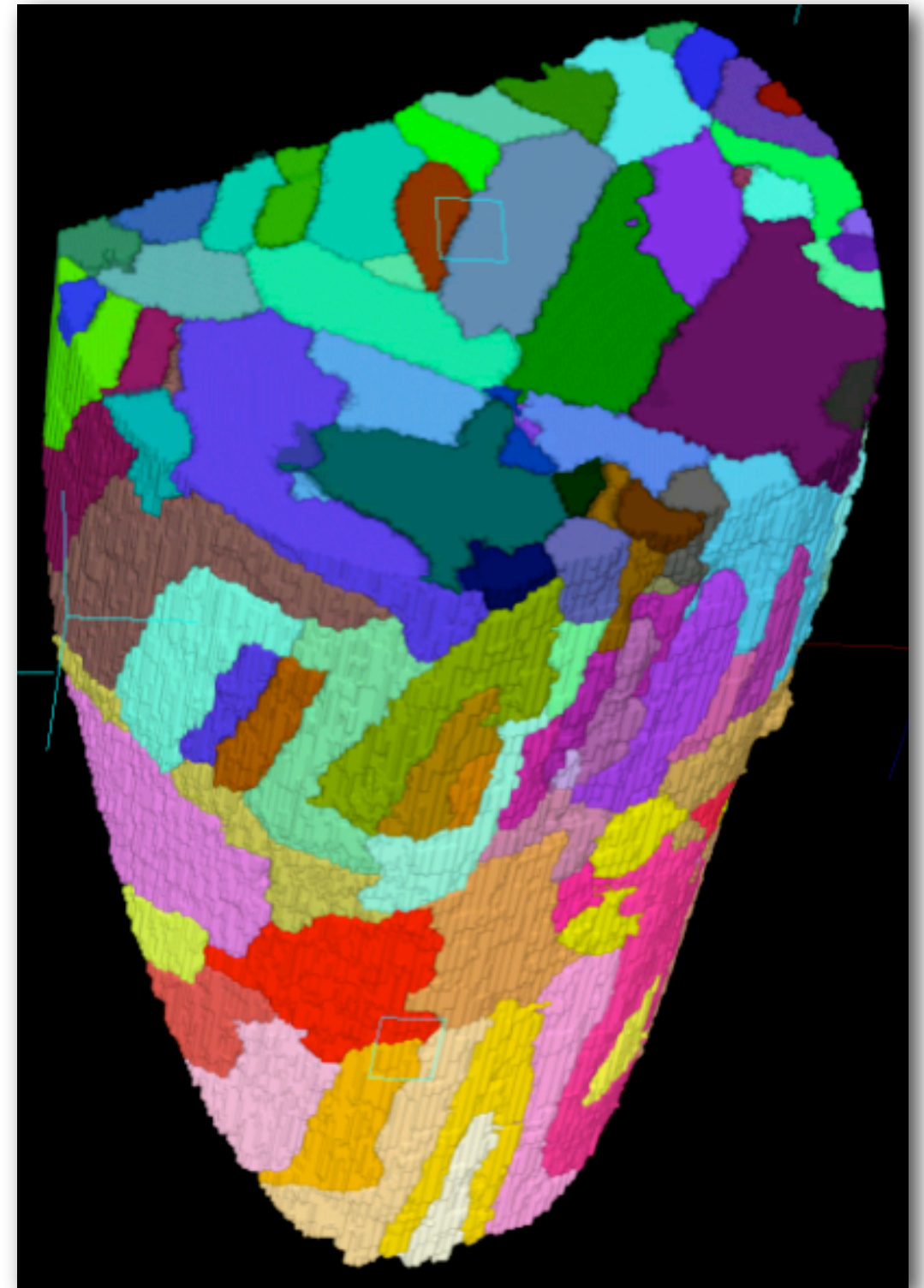
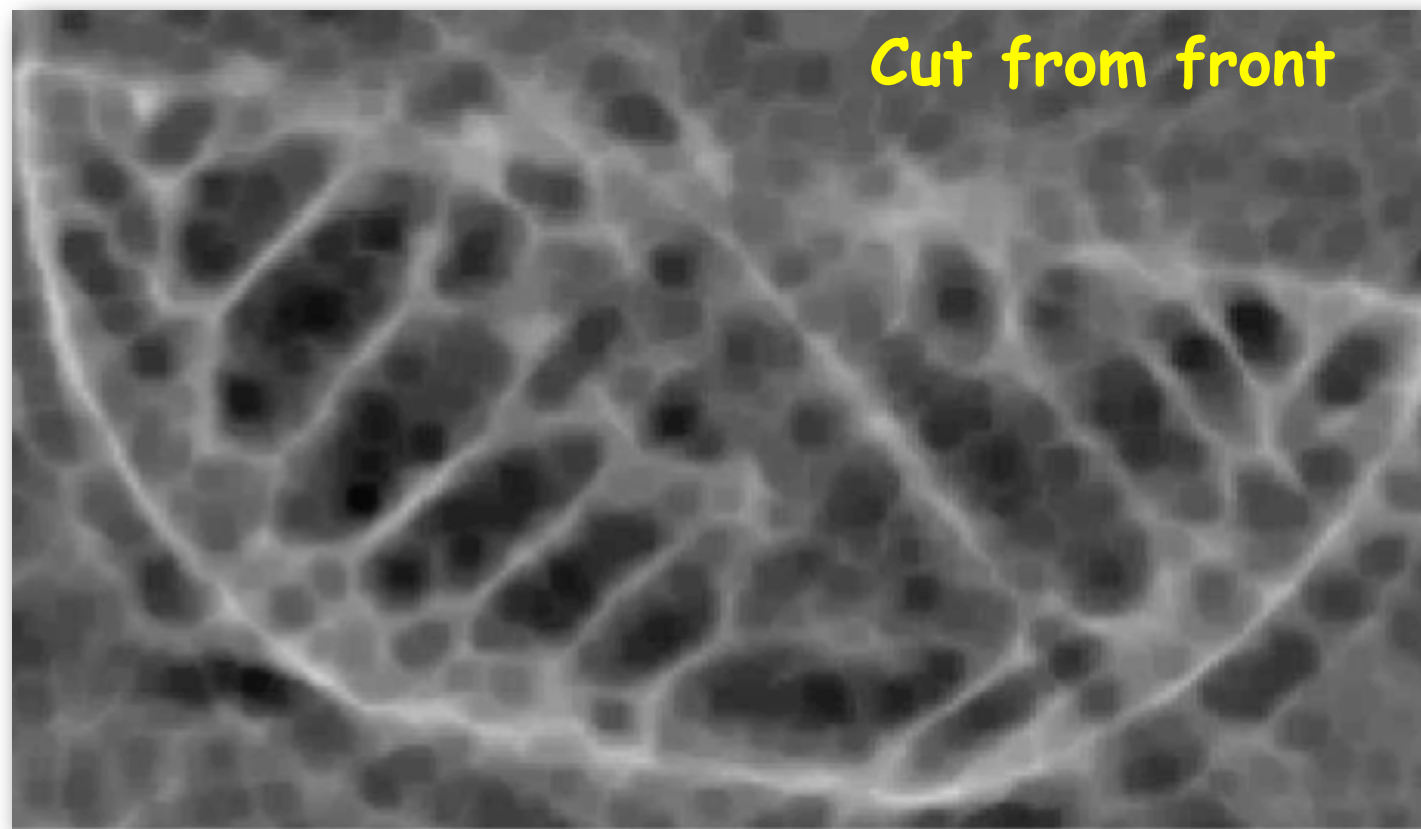
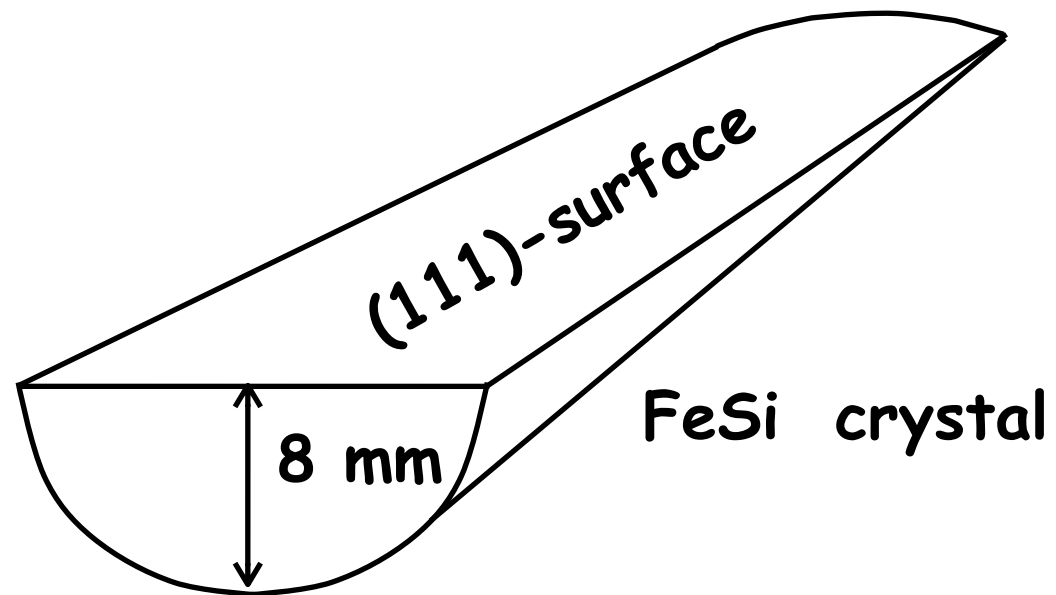
Magnetic field
→



Non-oriented
FeSi steel

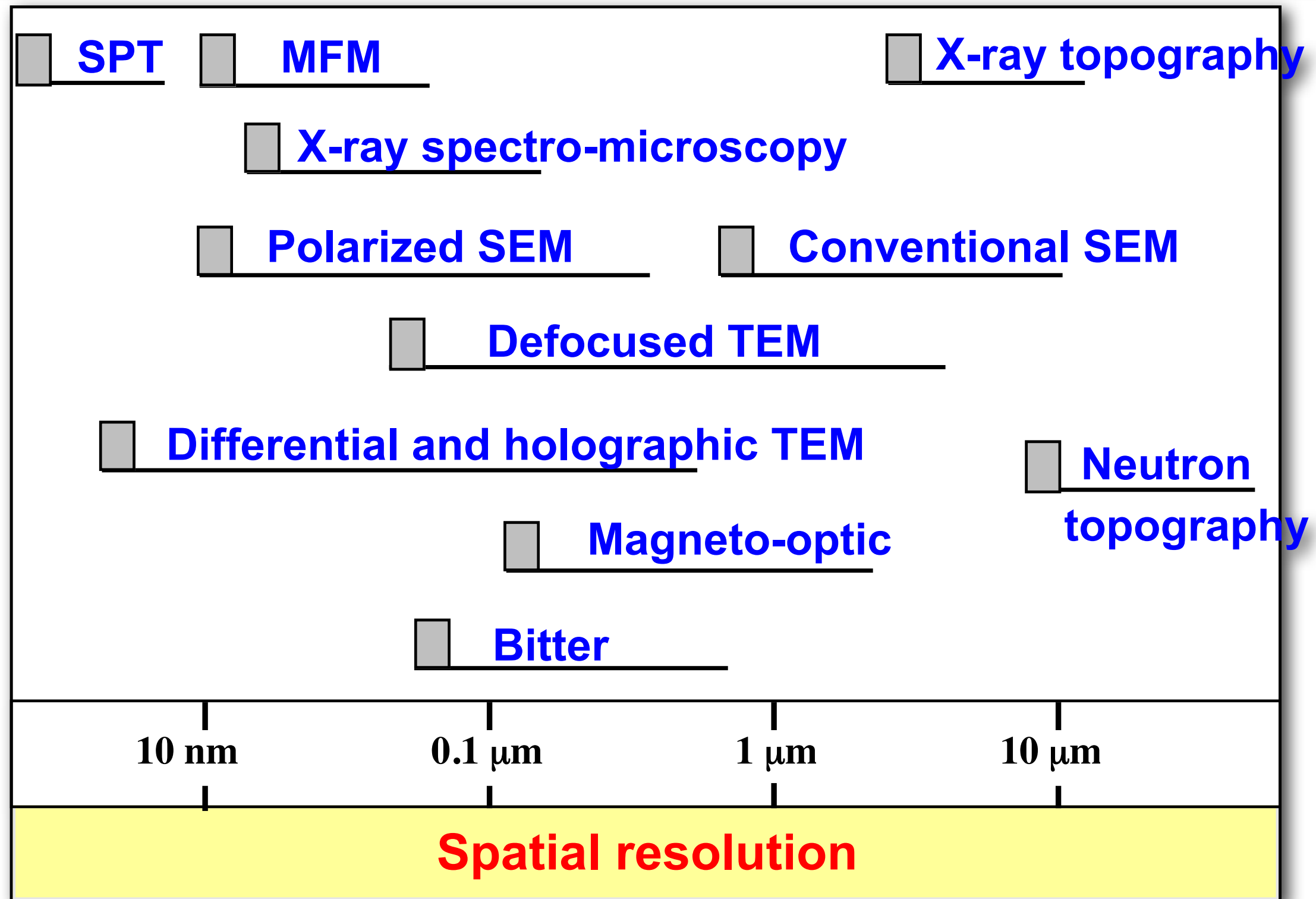
Together with B. Betz and C. Grünzweig (PSI Villigen),
R. Siebert (Fraunhofer IWS Dresden)
unpublished

8.2 Neutron Dark-Field Microscopy



I. Manke, et al.: Three-dimensional imaging of magnetic domains.
Nature Communications, 1:125 doi: 10.1038/ncomms1125 (2010)

Comparison of Domain Observation Techniques



MFM: Magnetic Force Microscopy
SPT: Spin-Polarized Tunneling

SEM: Scanning (reflection) Electron Microscopy
TEM: Transmission Electron Microscopy