Electric and magnetic measurements for characterizing superconductors

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University of Liège, Belgium

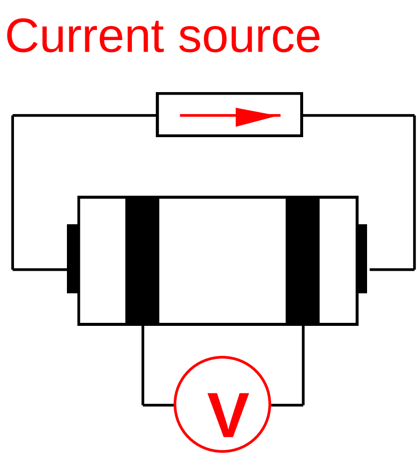
What kind of **electric and magnetic measurements** can we make to characterize superconductors ?

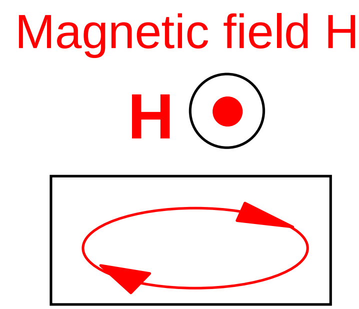
What kind of **information** can we extract from measurements ?

Purpose of this lecture

To better understand how we can characterize the electrical and magnetic propeties of materials through

ELECTRIC measurements and MAGNETIC measurements





<u>Transport</u> current
(applied externally)

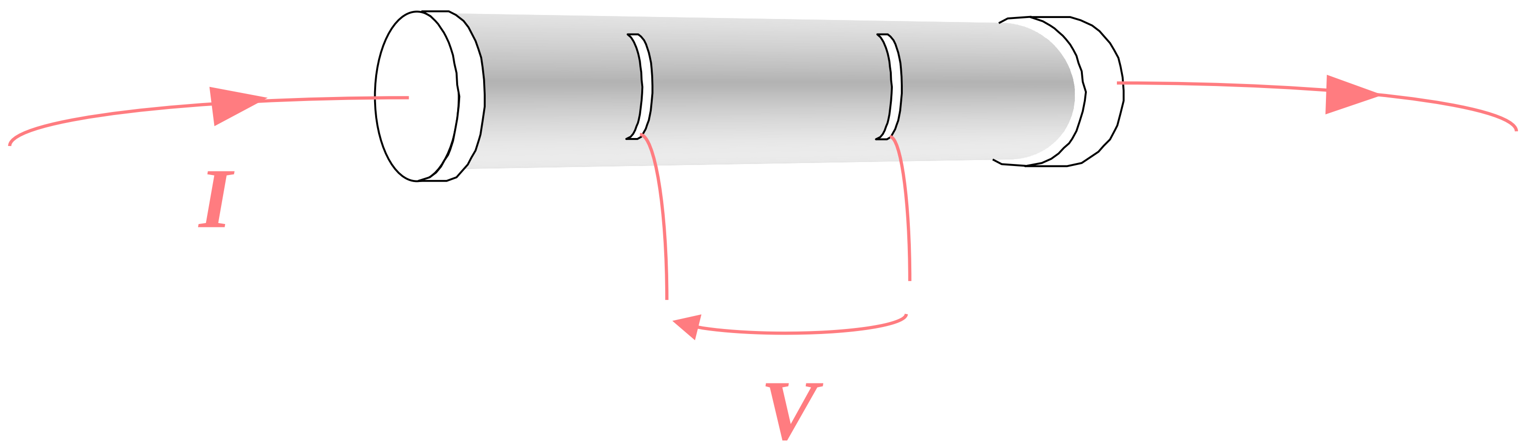
<u>Induced</u> current
(by the applied magnetic field)

Part A :
Electric (transport) measurements

Part B :
Magnetic measurements

Part A :
Electric (transport) measurements

The main difficulty for transport measurements on superconductors = ?

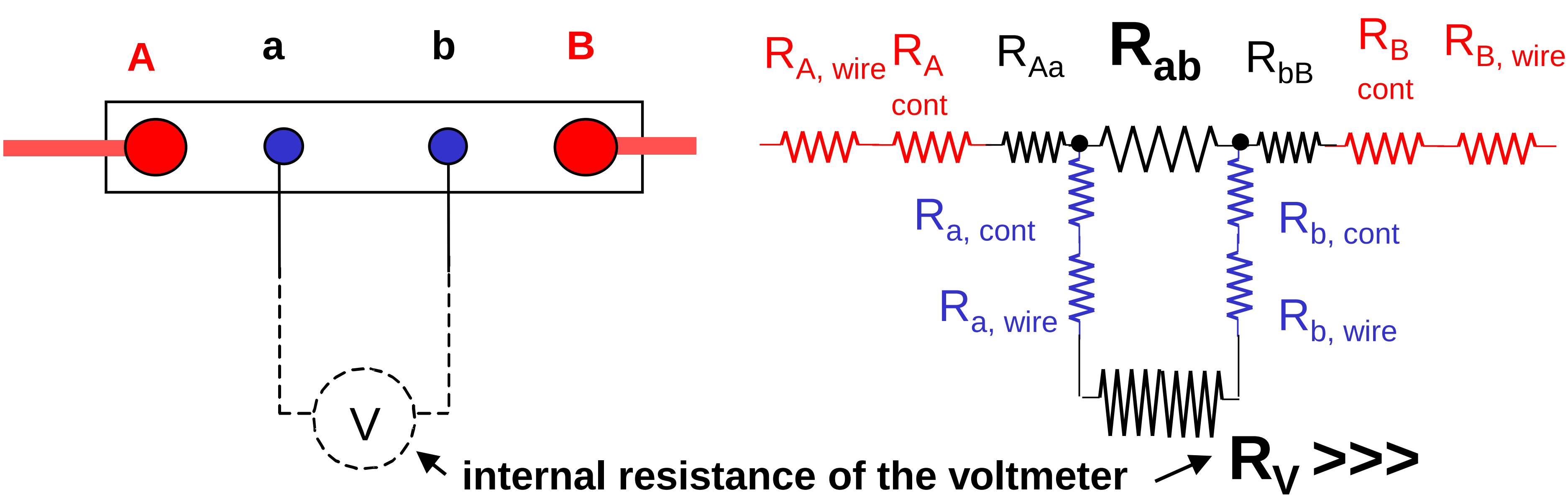


The finite resistance of electrical contacts

Influence of contact resistance & wire resistance

2-wire connexions

4-wire connexions

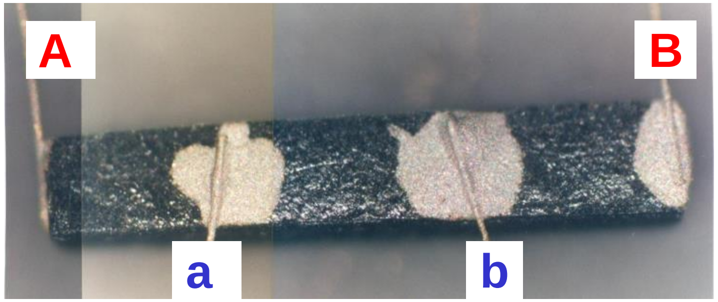


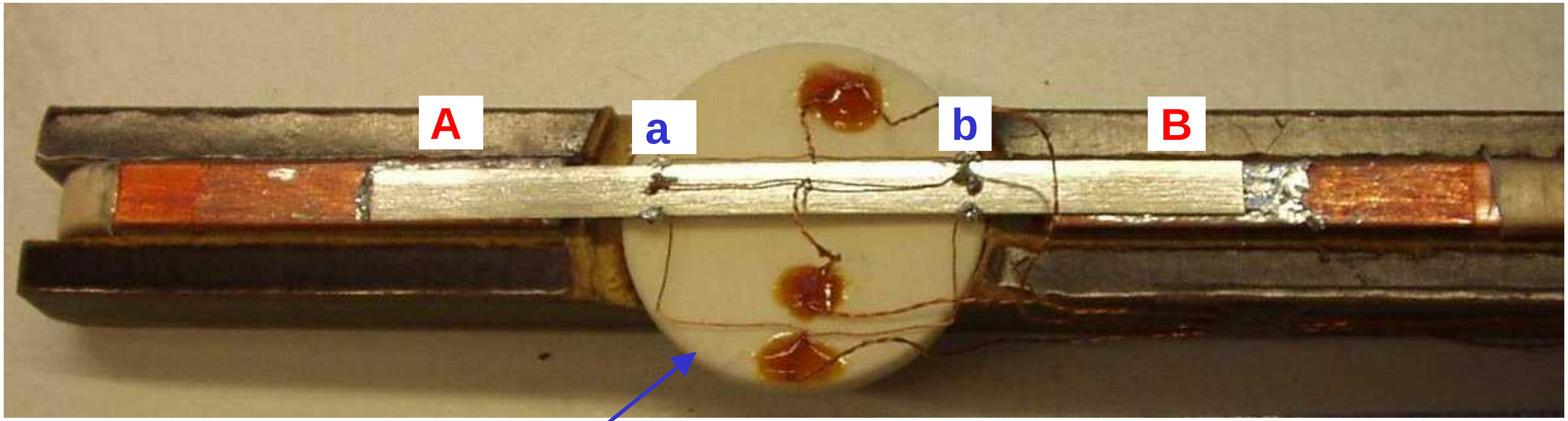
4-wire measurement (Kelvin connections)

With 4-wire connexions …
The **current** contact R and wire R are outside the measurement circuit
The **voltage** contact R and wire R can be neglected with respect to R of the voltmeter

Examples :

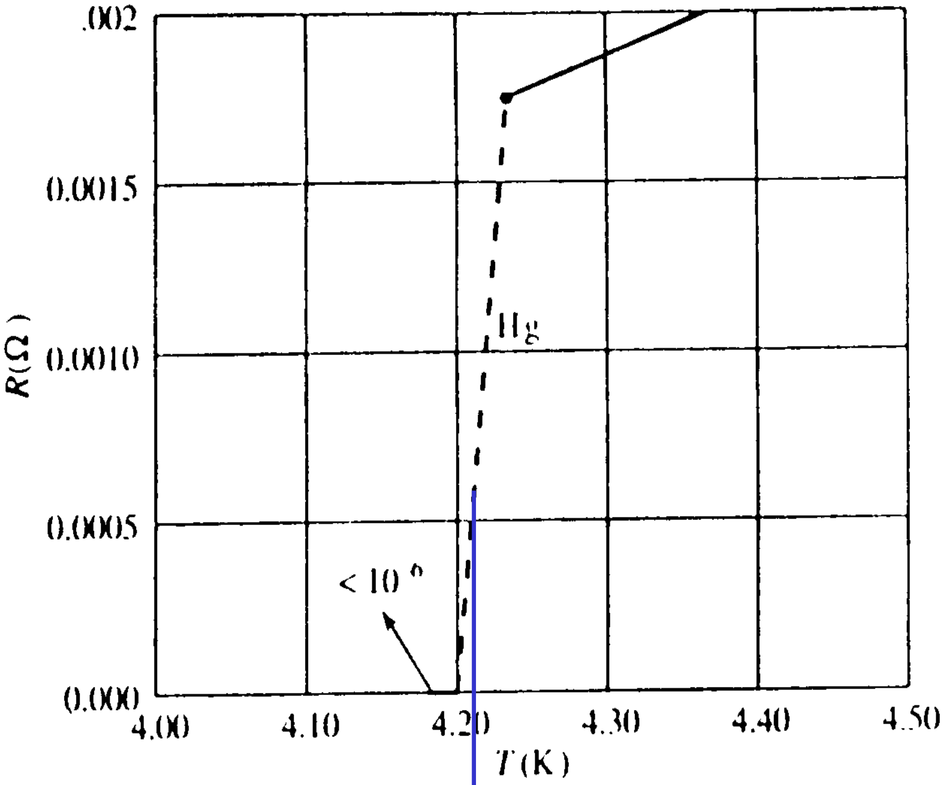
A, B = current contacts
a, b = voltage contacts





NB : for AC measurements : **twisted wires** are required to avoid inductive pick-up !

Which information can we probe with a resistance vs. temperature measurement ?



T_c

critical temperature

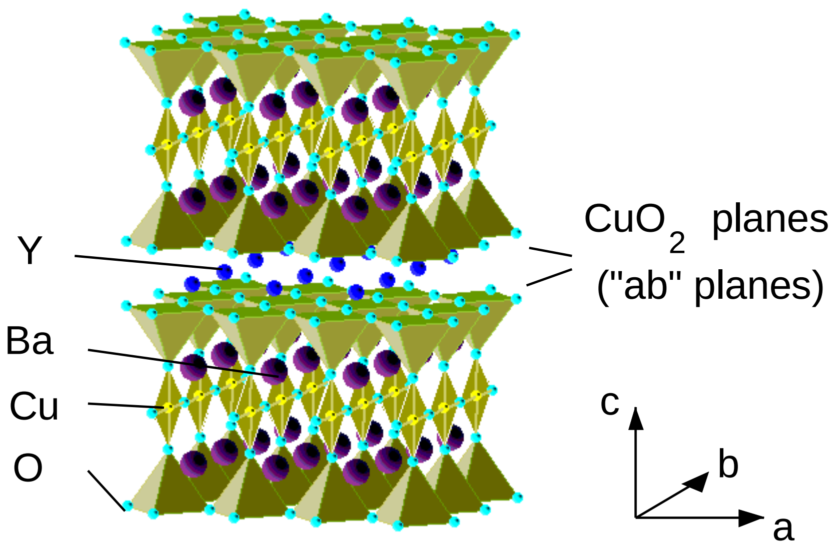
Which information can we probe with a resistance vs. temperature measurement ?

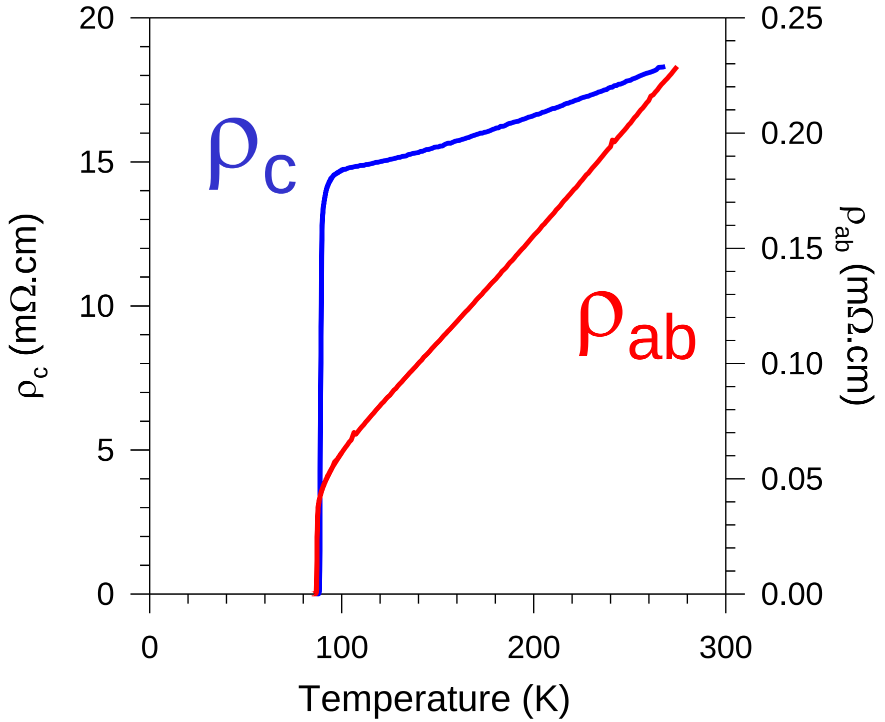
For High Temperature Superconductors (HTS) R(T) measurements allow also to investigate…

(i) Anisotropy
(ii) Granularity
(iii) Irreversibility Line (IL)

(i) Anisotropy

Example : $YBa_2Cu_3O_7$ single domain

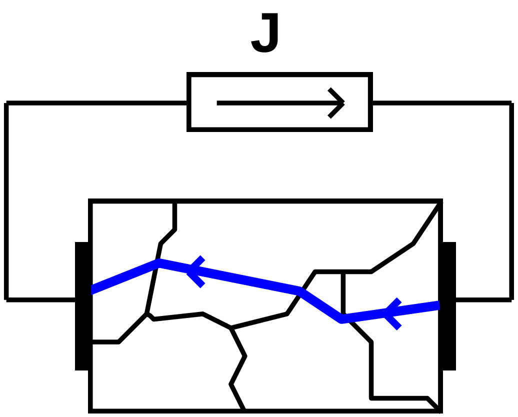




It should be also noted the **pinning of flux lines B**
is larger for **B || ab** than for **B || c**

(ii) Granularity

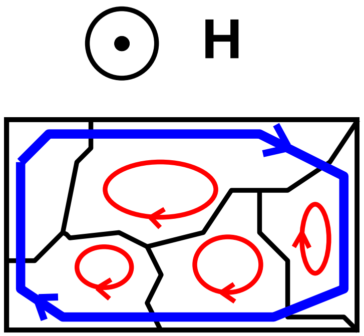
Transport current



Intergranular current J_{CJ}

Shielding currents

Applied magnetic field



Intergranular current J_{CJ}

Intragranular current J_{CG}

$$J_{CJ} < J_{CG}$$

Grain alignment - or **texturation** - is a key ingredient to improve the **intergranular** critical current density

Orientation Dependence of Grain-Boundary Critical Currents in $YBa_2Cu_3O_{7-\delta}$ Bicrystals

D. Dimos, P. Chaudhari, J. Mannhart, and F. K. LeGoues

Thomas J. Watson Research Center, IBM Research Division,
Yorktown Heights, New York, 10598
(Received 4 May 1988)

The critical current densities across grain boundaries have been measured as a function of misorientation angle in the basal plane of bicrystals of $YBa_2Cu_3O_{7-\delta}$. For small misorientation angles, the ratio of the grain-boundary critical current density to the bulk critical current density is roughly proportional to the inverse of the misorientation angle; for large angles, this ratio saturates to a value of about $\frac{1}{50}$. These results imply that achieving a high degree of texture both normal to and within the basal plane is important for the obtaining of very high critical currents in pure polycrystalline samples.

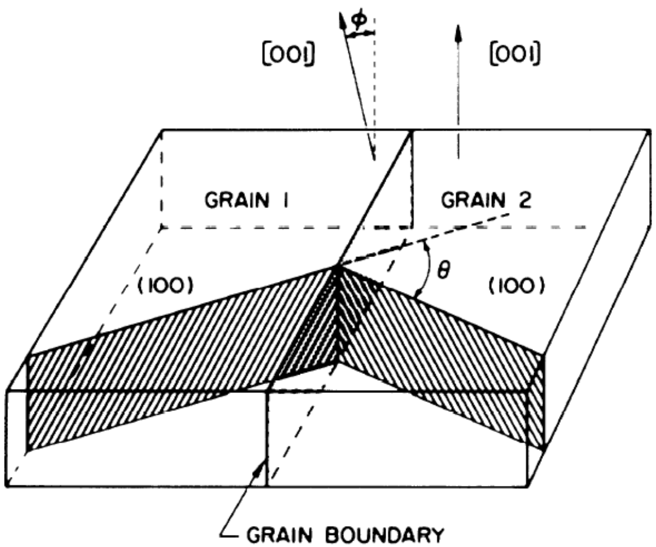
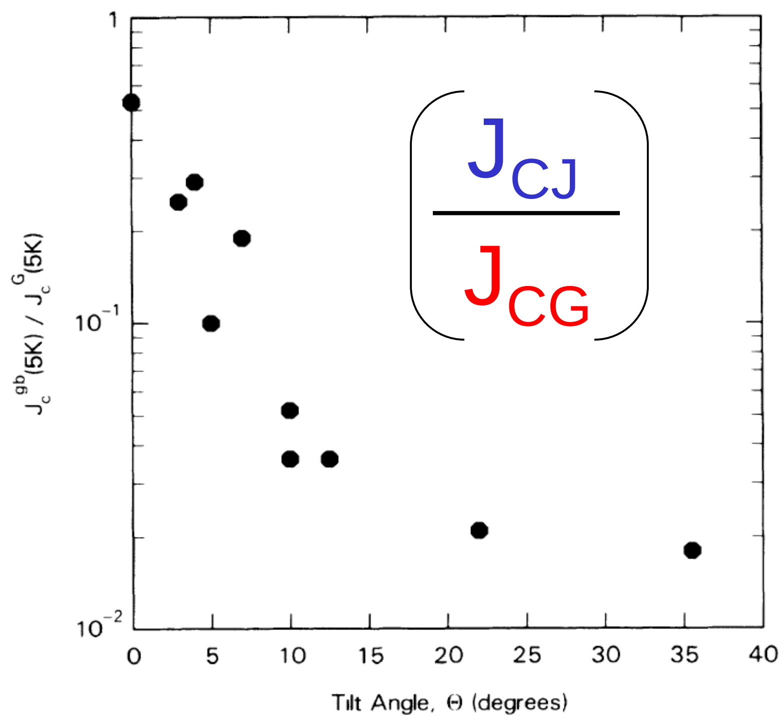
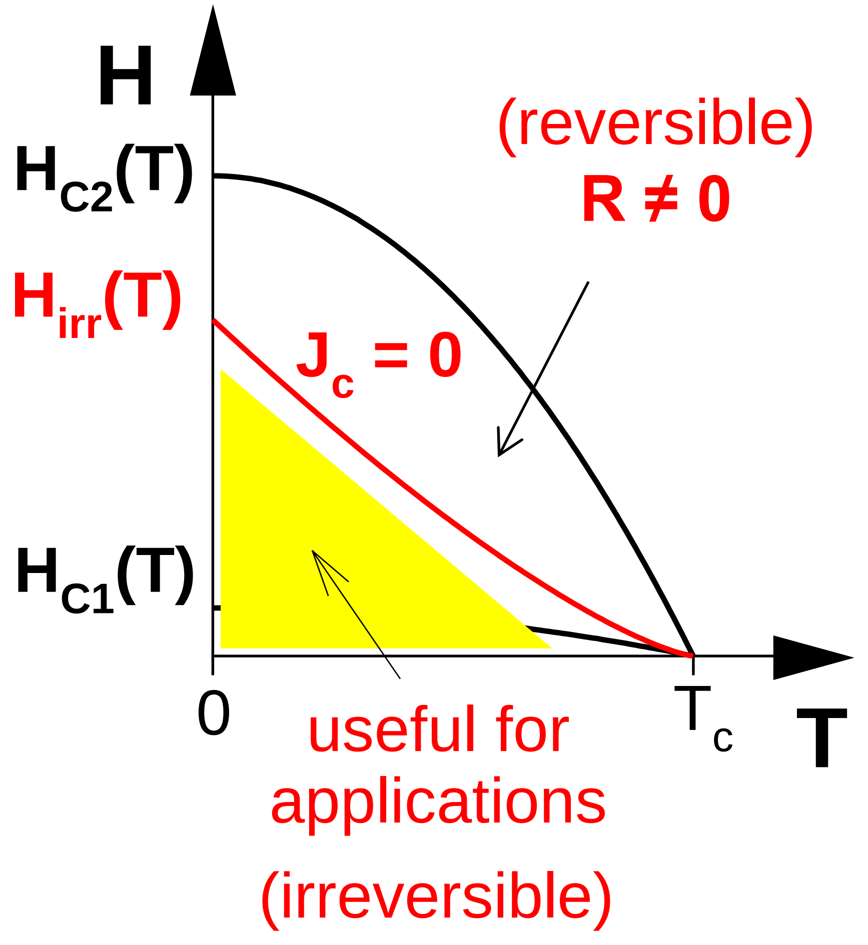


FIG. 1. Schematic diagram showing the important crystallography of the $SrTiO_3$ bicrystals which were used as substrates for the thin-film deposition.

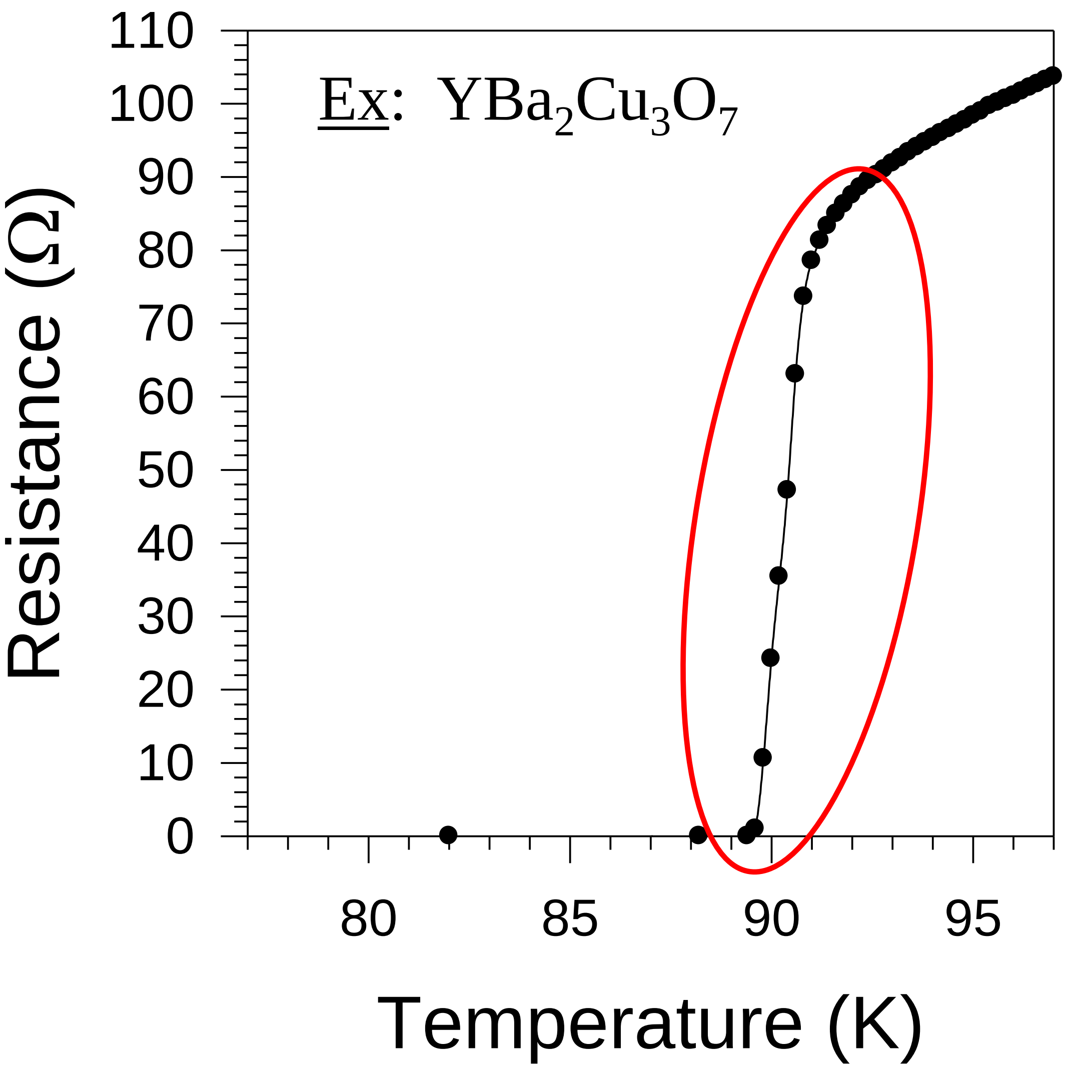


$$\left(\frac{J_{CJ}}{J_{CG}} \right)$$

(iii) Irreversibility Line



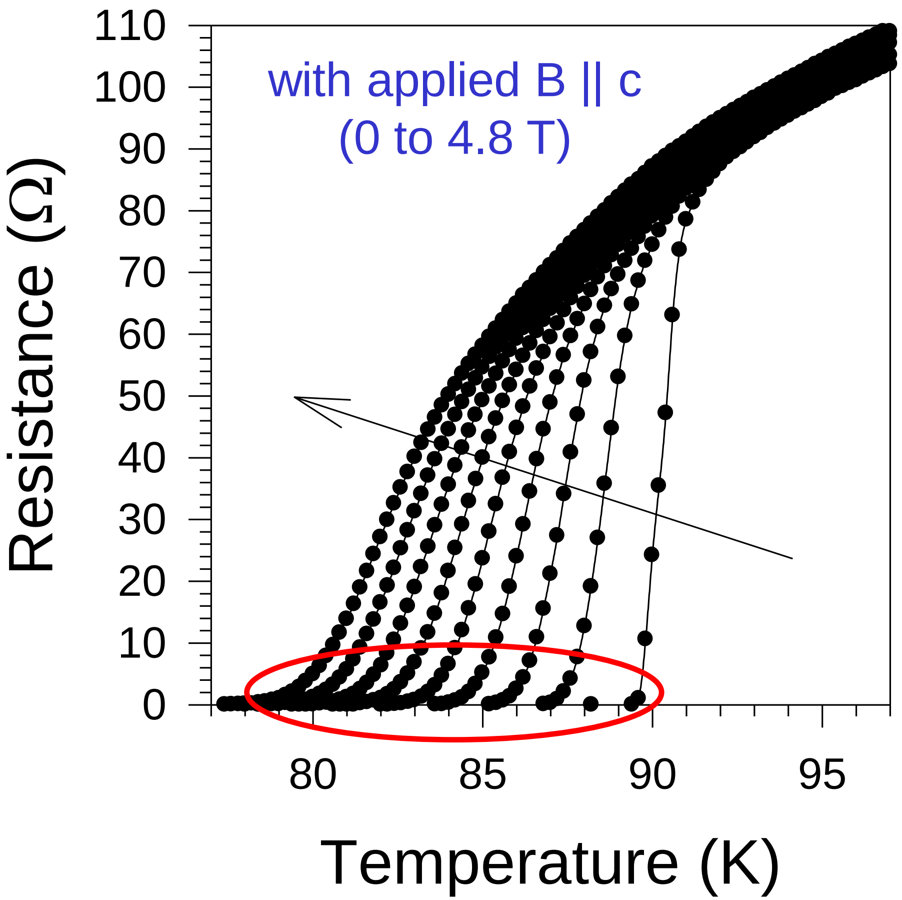
Typical R(T) curve

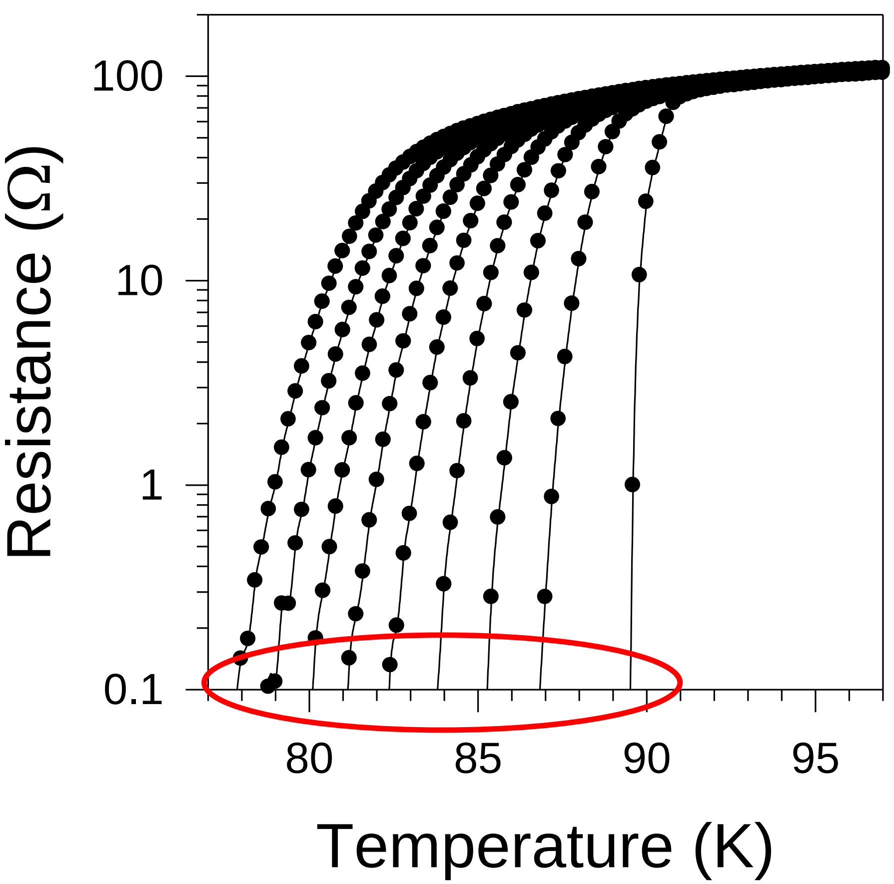


The width of the transition requires a **given criterion** to define T_c correctly

(usual criterion : inflexion point [change of curvature] but others are possible)

Typical R(T) curve





The use of a log scale can be very useful the temperature above which electrical resistance merges from the noise level (= irreversibility line)

Some (slightly more complicated) examples…

VOLUME 69, NUMBER 23 PHYSICAL REVIEW LETTERS 7 DECEMBER 1992

Vortex Lattice Melting in Untwinned and Twinned Single Crystals of $YBa_2Cu_3O_{7-\delta}$

W. K. Kwok, S. Fleshler, U. Welp, V. M. Vinokur, J. Downey, and G. W. Crabtree
Science and Technology Center for Superconductivity and Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439

M. M. Miller
Naval Research Laboratory, Washington, D.C. 20375
(Received 1 October 1992)

The melting transition in twinned and untwinned single crystals is measured resistively in fields up to 8 T as a function of the angle between the **c** axis and the **a**-**b** plane. The angular dependence follows the Lindemann criterion with $c_L = 0.15$. The suppression of melting by strong pinning by twin boundaries is demonstrated.

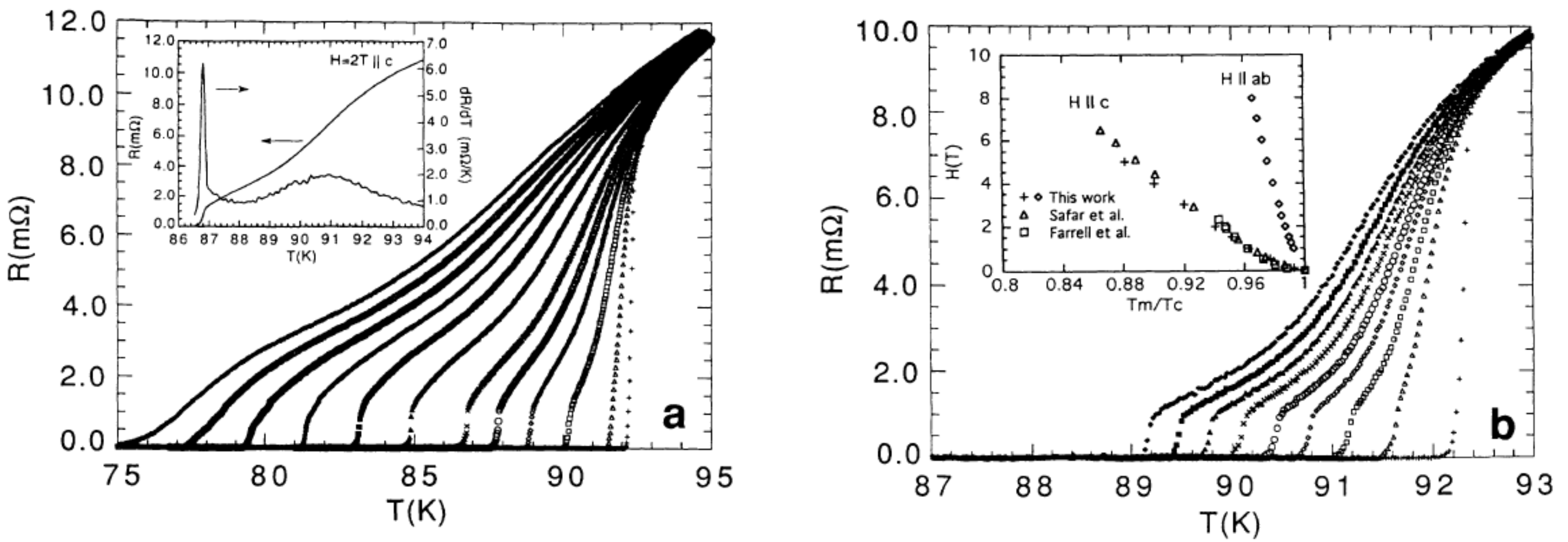


FIG. 1. (a) Resistive transition in magnetic fields of 0, 0.1, 0.5, 1, 1.5, 2, 3, 4, 5, 6, 7, and 8 T for **H**‖**c** in an untwinned $YBa_2Cu_3O_{7-\delta}$ crystal. Inset: Determination of T_m from the inflection peak of dR/dT for $H=2$ T. (b) Resistive transition in magnetic fields of 0, 1, 2, 3, 4, 5, 6, 7, and 8 T for **H**‖(**a**,**b**). Inset: Phase diagram of the melting transition for **H**‖**c** and H‖(**a**,**b**).

Granularity

Superconducting properties of natural and artificial grain boundaries in bulk melt-textured YBCO

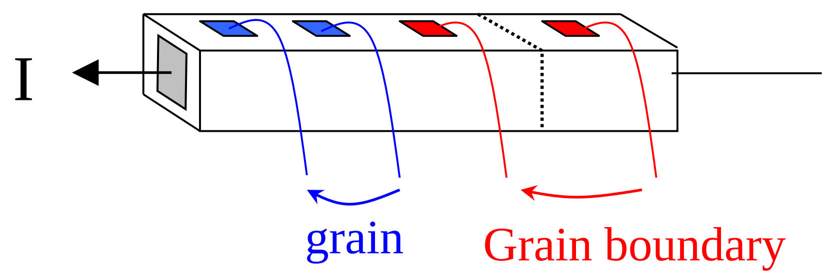
Ph. Vanderbemden [a,b,*], A.D. Bradley [b], R.A. Doyle [b], W. Lo [b], D.M. Astill [b], D.A. Cardwell [b], A.M. Campbell [b]

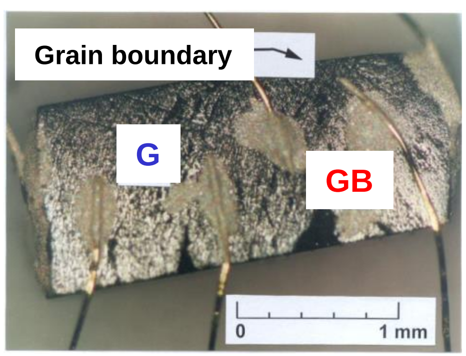
[a] *SUPRAS, Montefiore Electricity Institute B28, University of Liège, Sart-Tilman, B-4000 Liège, Belgium*
[b] *IRC in Superconductivity, University of Cambridge, Madingley Road, Cambridge CB3 0HE, UK*

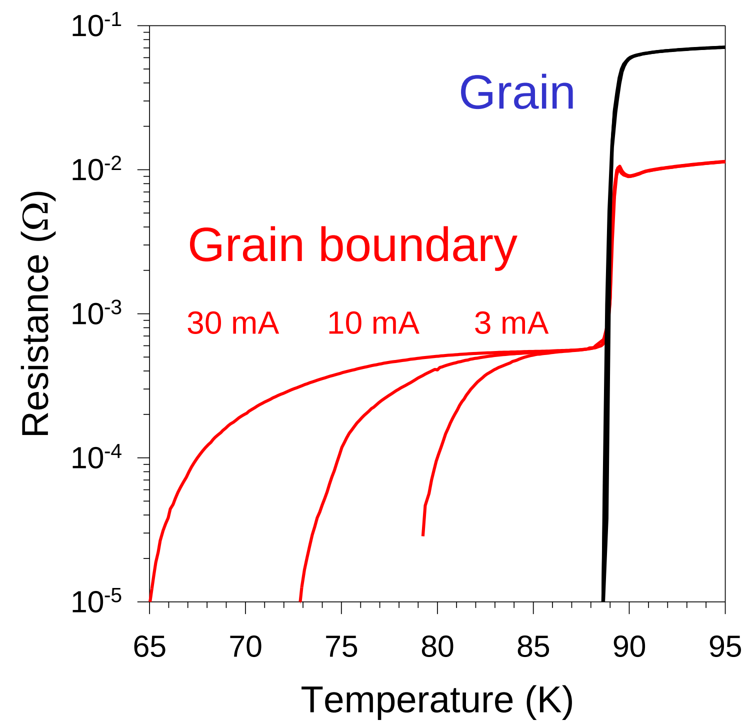
Received 29 December 1997; revised 7 March 1998; accepted 2 May 1998

PHYSICA C

Physica C 302 (1998) 257–270

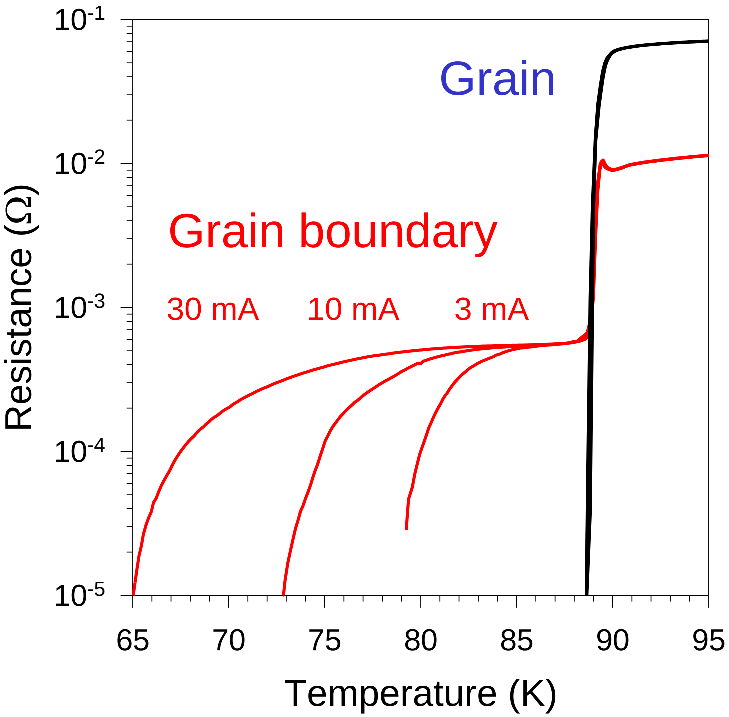




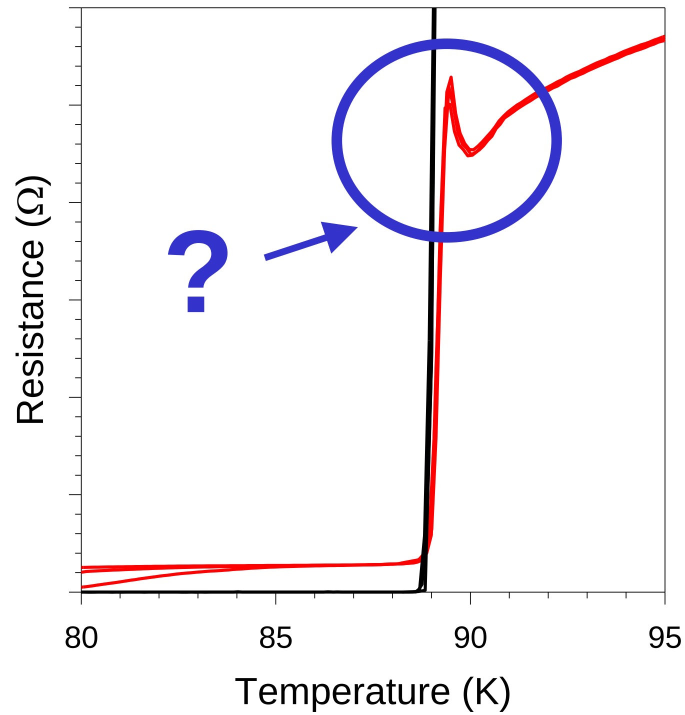


A **shoulder** in R(T) – possibly using a log scale for R
is a clear signature of the presence of **one or more grain boundaries**

Some artefacts or difficulties …



Back to
LINEAR
SCALE



The peak in R(T) just above the superconducting transition is a (relatively) common feature usually attributed to inhomogeneities and **current redistribution**

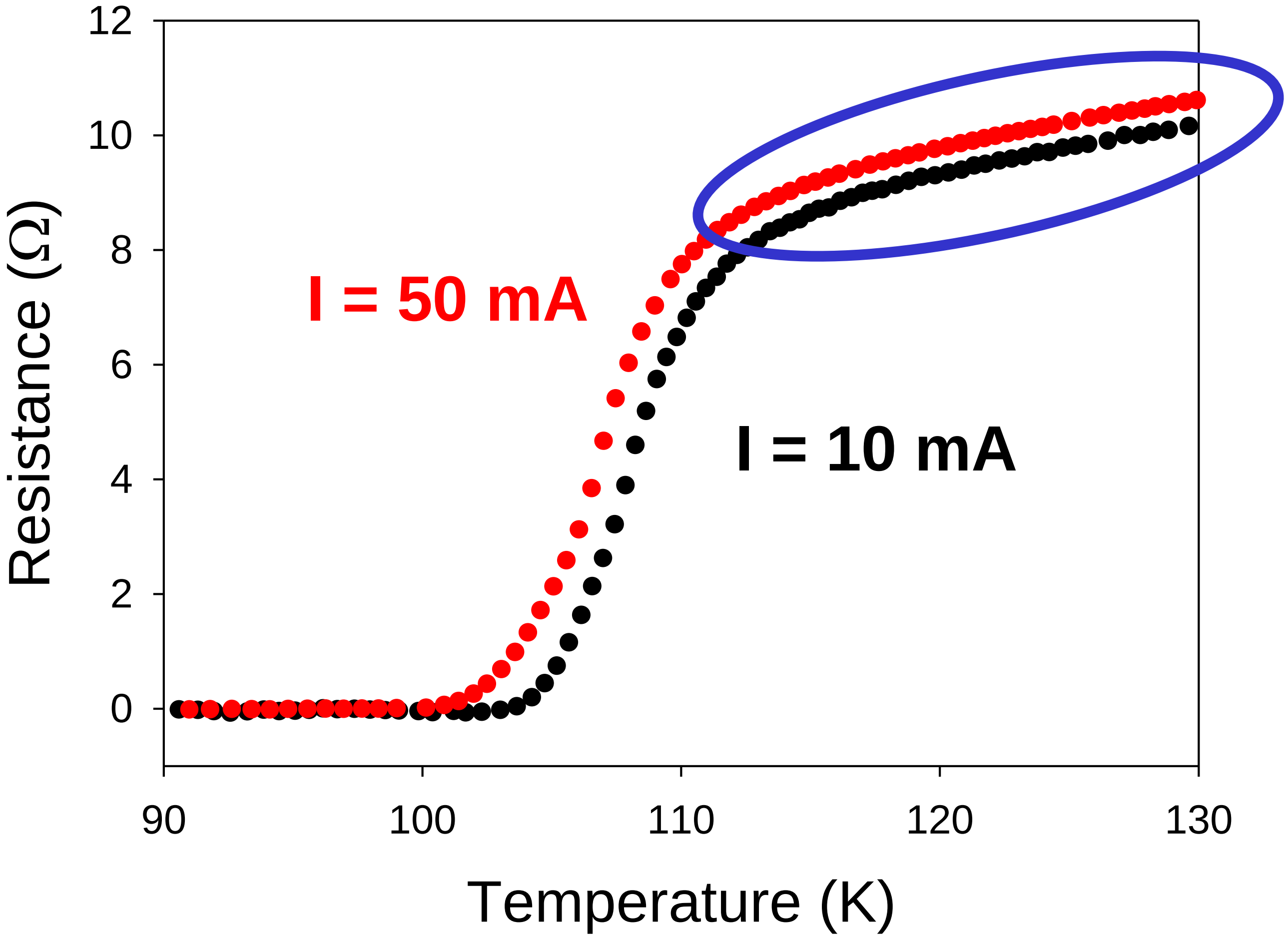
Current redistributions in superconductors with non-uniformly distributed T_c-inhomogeneities

PHYSICA C

Th. Siebold, C. Carballeira, J. Mosqueira, M.V. Ramallo and Félix Vidal

Physica C 282–287 (1997) 1181–1182

Is it superconducting physics ?

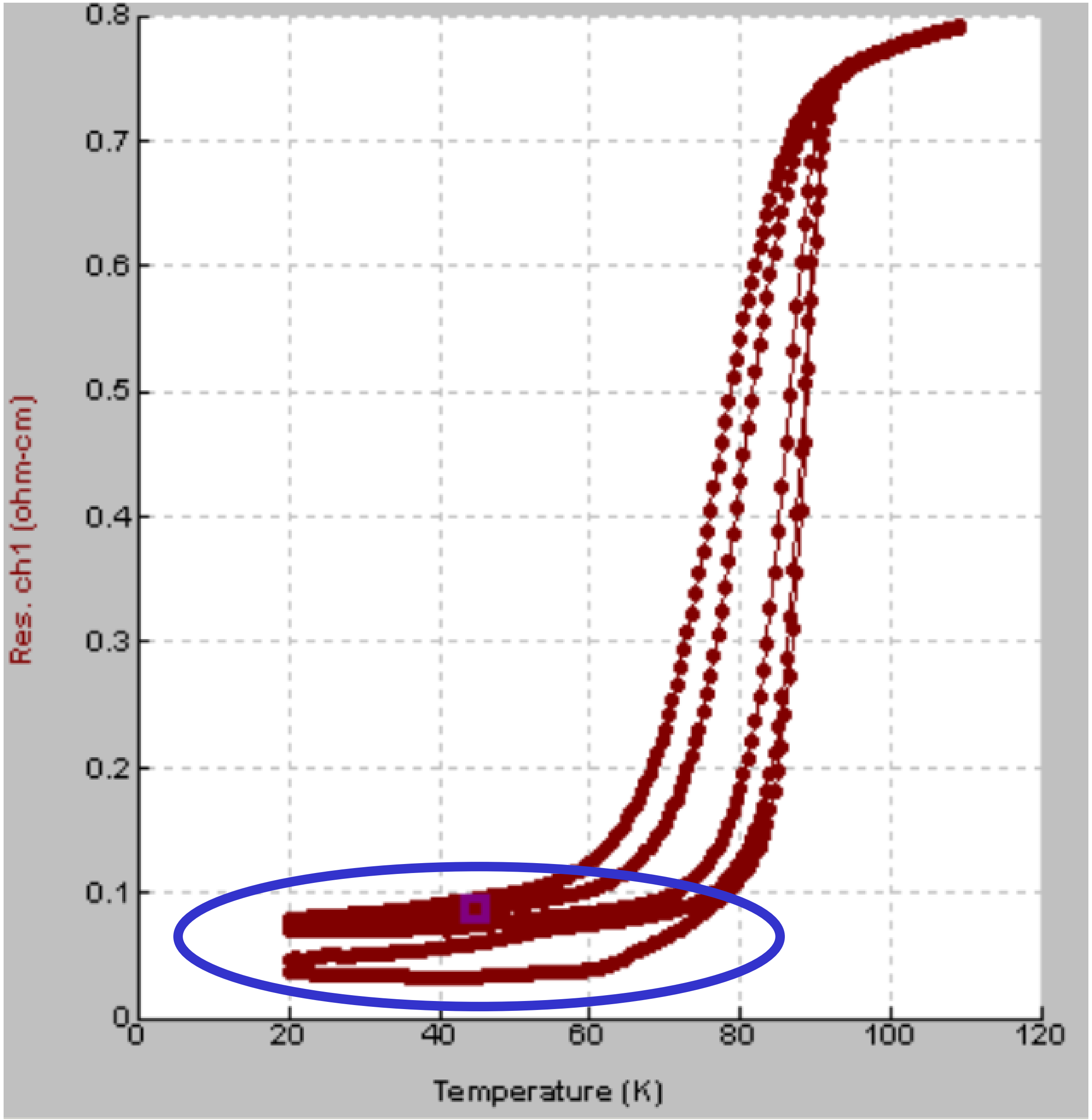


$Bi_2Sr_2Ca_2Cu_3O_{10}$
(Bi-2223)
ceramic

A larger current means also a <u>much</u> larger power dissipated in current contacts ($P = R I^2$!) and, possibly, sample heating and error in the **<u>temperature measurement</u>**

→ Keep currents **<u>as low as possible</u>** why keeping an acceptable sensitivity

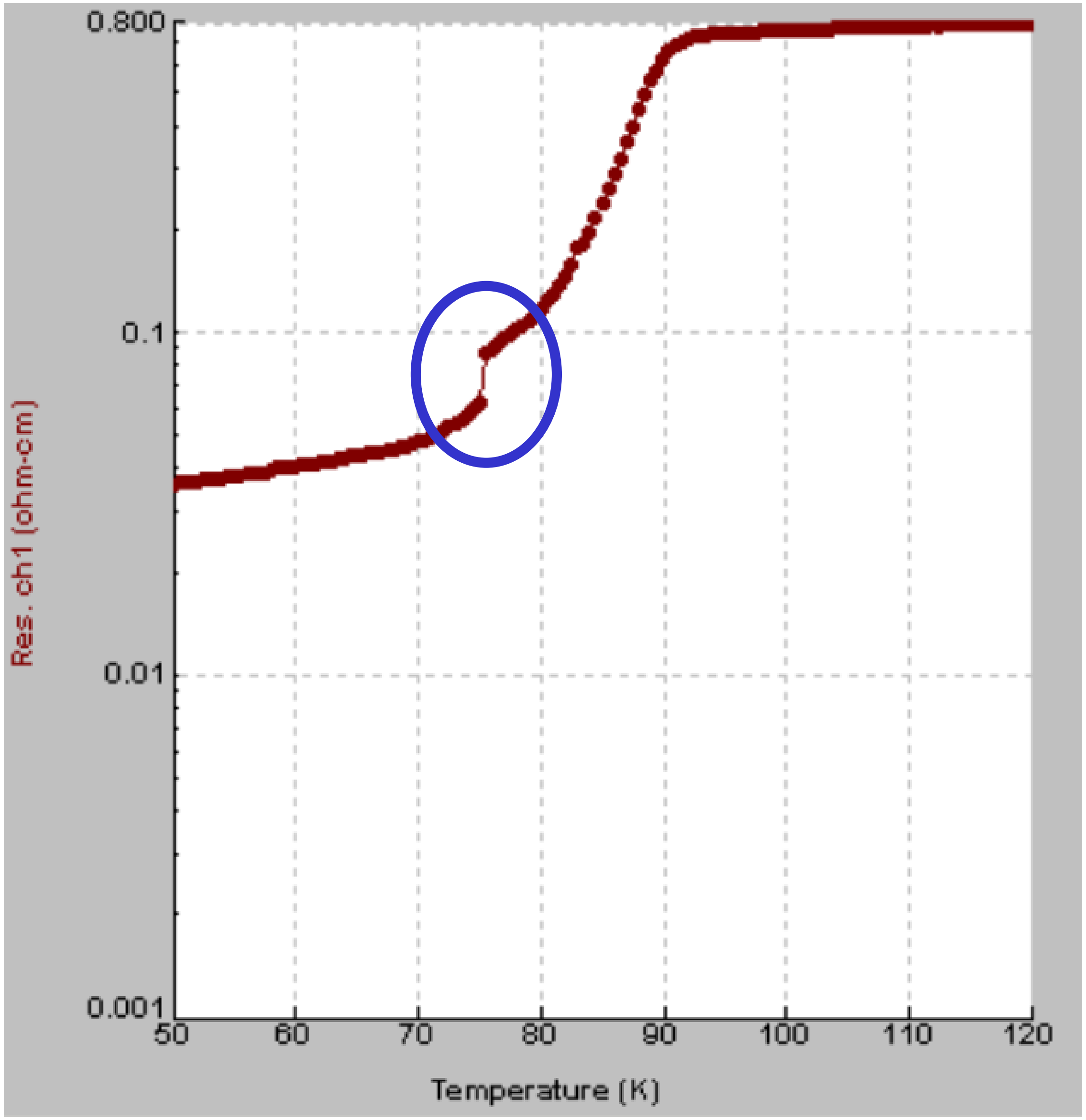
Is it superconducting physics ?



Bad sample or bad contact resistance ?

Try again with **<u>new contacts</u> !**

Is it superconducting physics ?

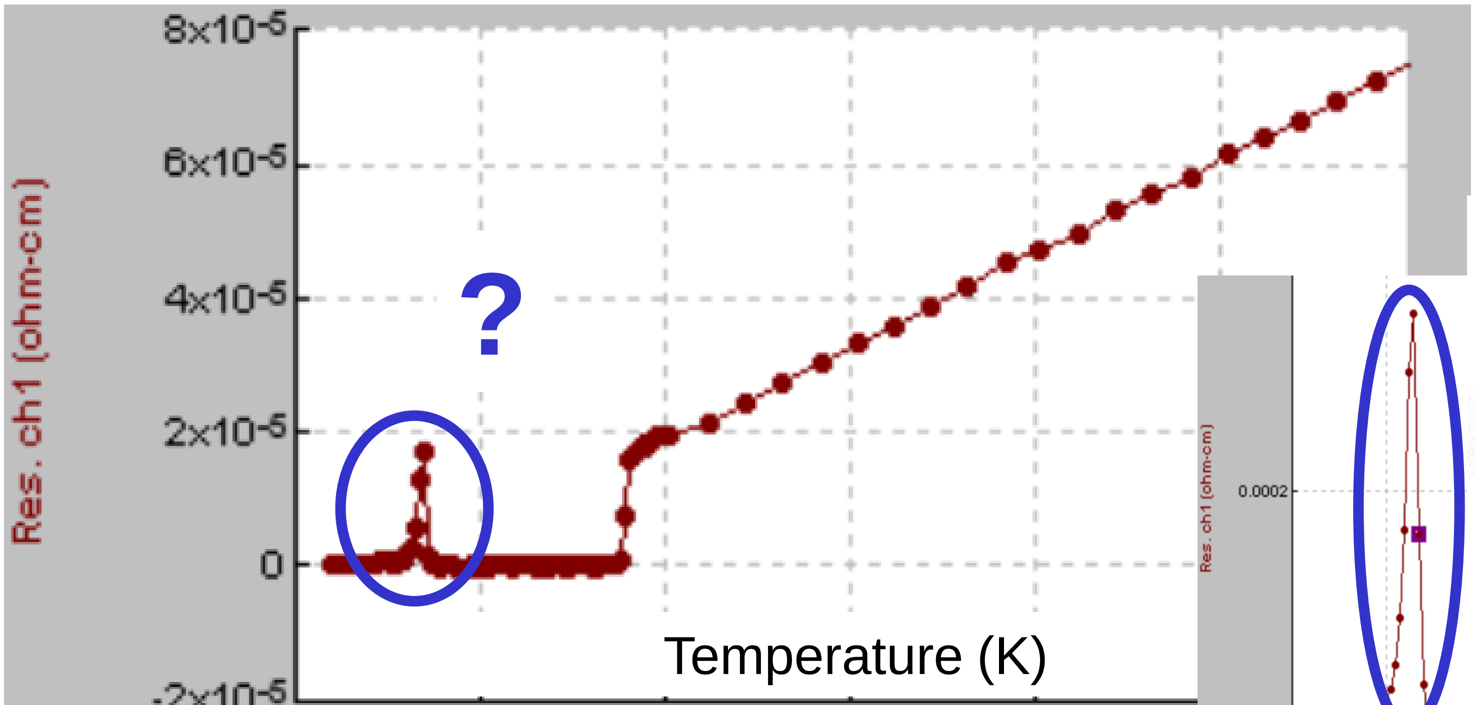


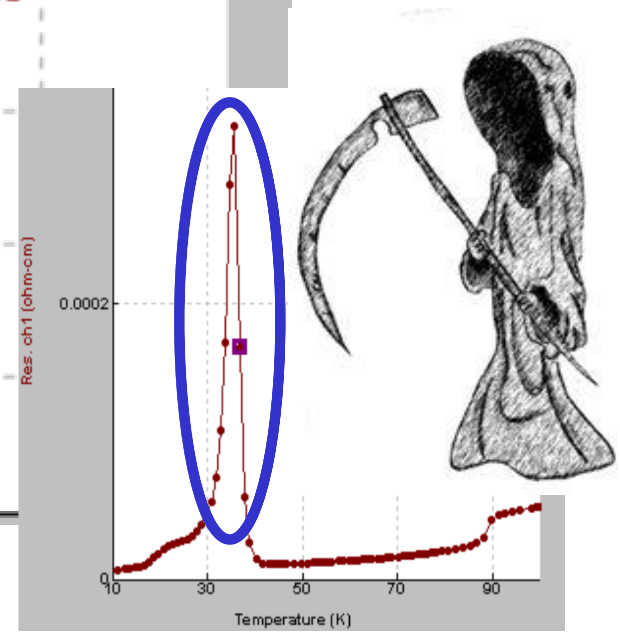
« Jumping » contact

Try again with
new contacts !

Is it superconducting physics ?

AC resistance measured in a QD Physical Property Measurement System (PPMS)





See the following application note :

Quantum Design

Distorted low-level signal readback of AC signals in the PPMS in the temperature range 25-35 K due to Inconel mitigation of inductive cross talk

Part A :
Electric (transport) measurements

Part B :
Magnetic measurements

Outline for magnetic measurements

- What are we measuring?
- How are we measuring?
- What kind of information can we extract?

Outline for magnetic measurements

- What are we measuring?
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What are we talking about ?

$$\vec{B} = \mu_0 (\vec{H} + \vec{M})$$

$\vec{H}$ = magnetic field [A / m]

$\vec{M}$ = magnetization [A / m]

$\vec{B}$ = magnetic flux density [T]

⇩

$\vec{\nabla}.\vec{B} = 0$

H and M are expressed in the same units

(in Prague too)

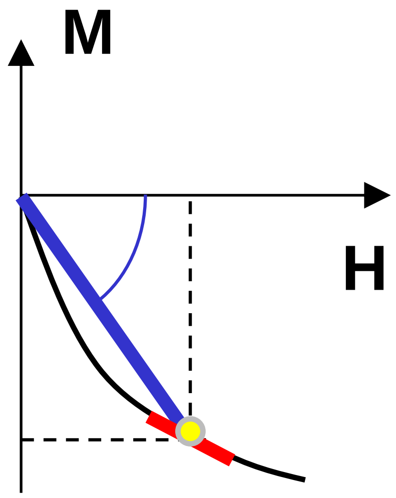
And a little bit more …

m = magnetic moment $[A.m^2]$

M = magnetization [A / m]
(= m / V)

χ_{DC} = magnetic susceptibility
(= M / H)
[DC]

χ_{AC} = magnetic susceptibility
(= dM / dH)
[AC]



And a little bit more …

m = f (physics, applied field, volume)

M = f (physics, applied field, volume)

χ_{DC}

χ_{AC}

$\left.\begin{matrix}\chi_{DC} \\ \chi_{AC}\end{matrix}\right\}$ **= f (physics, applied field, volume)**

The 'most common' (?) magnetic measurements

'DC' ?

Constant T → **M(H)**

Constant H → $\chi_{DC}(T)$

'AC' ?

Constant H → $\chi' \chi''(T)$

So: do not confuse the two m's : « M » and « m »

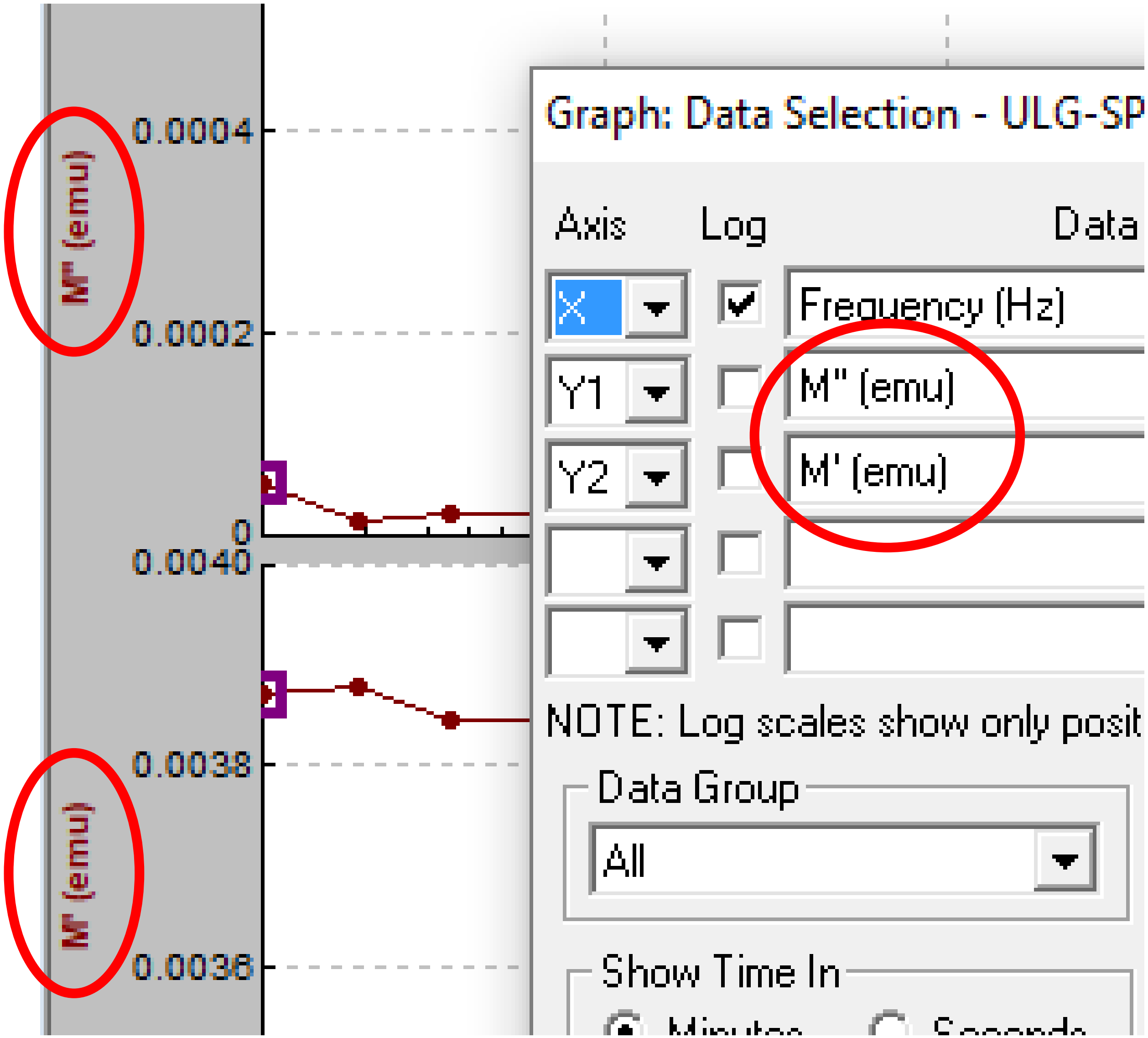
Magnetic moment [Am²]
or [emu] = [10^{-3} Am²]

$$M = \frac{m}{V}$$

Magnetisation
[A/m]

Volume [m³]

So: do not confuse the two m's : « M » and « m »



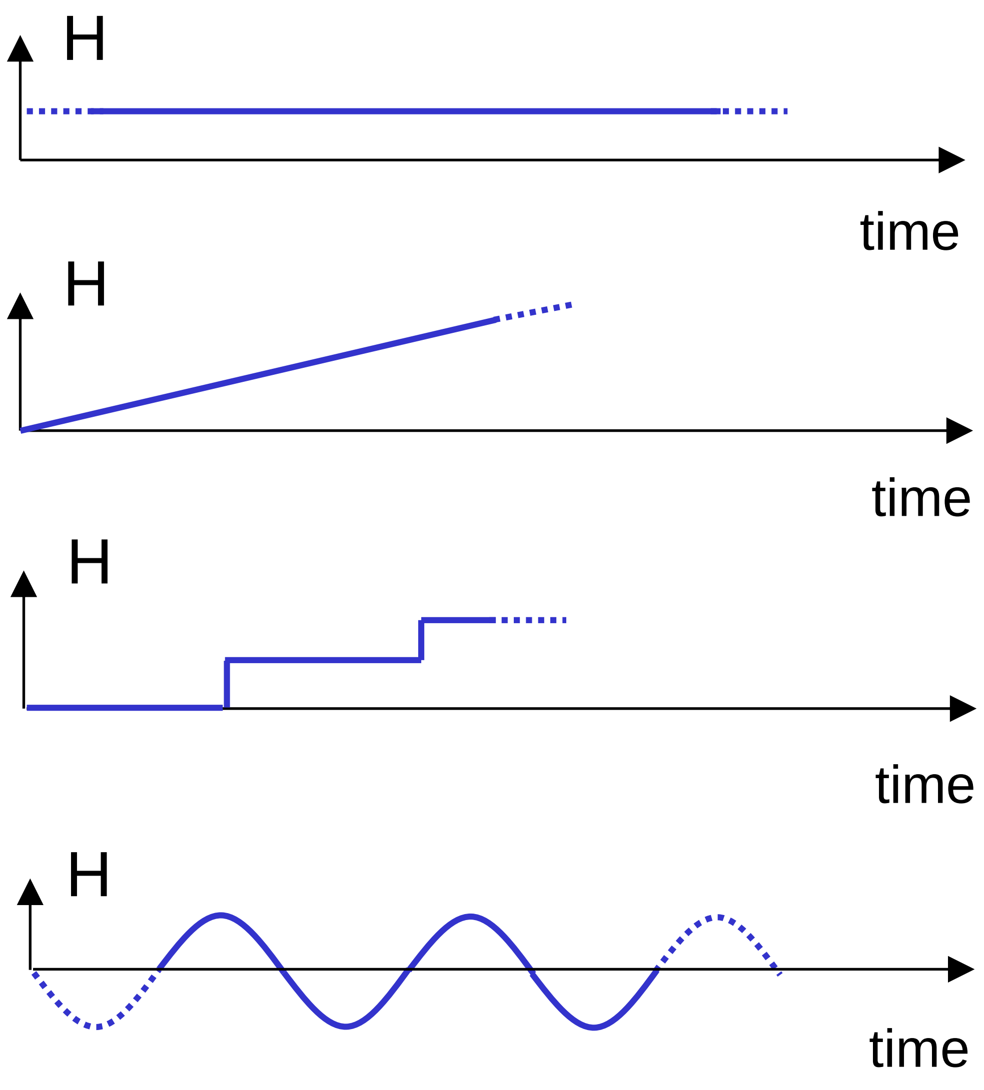
(argh!)

Types of magnetic sollicitations

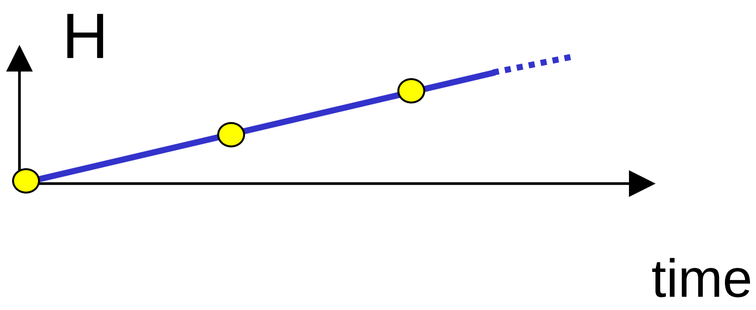
Direct current
Steady-state regime
→ « DC »

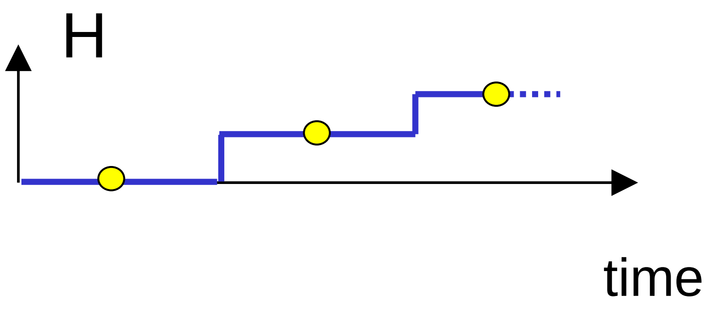
Quasi-static
Transient regime
→ « DC » (too)

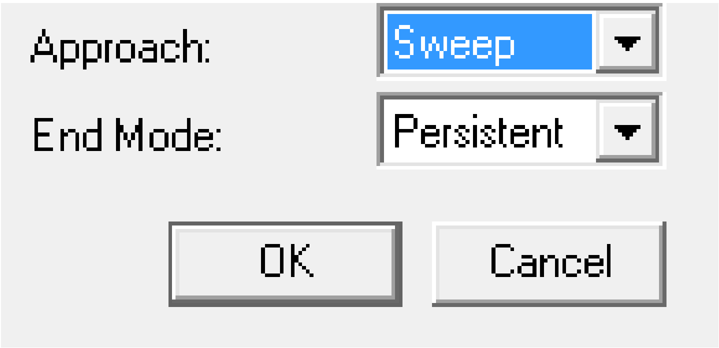
Alternating
Sinewave signal
→ « AC »

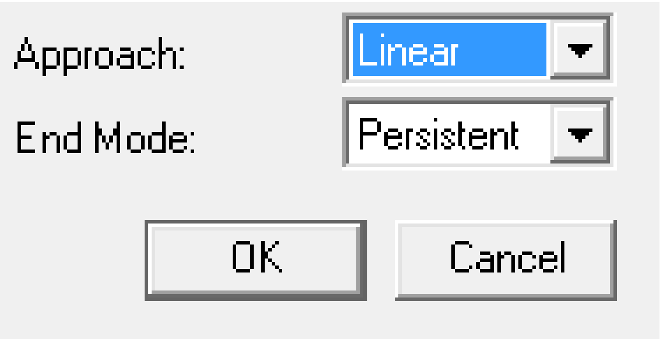


Characteristics of 'DC' measurements

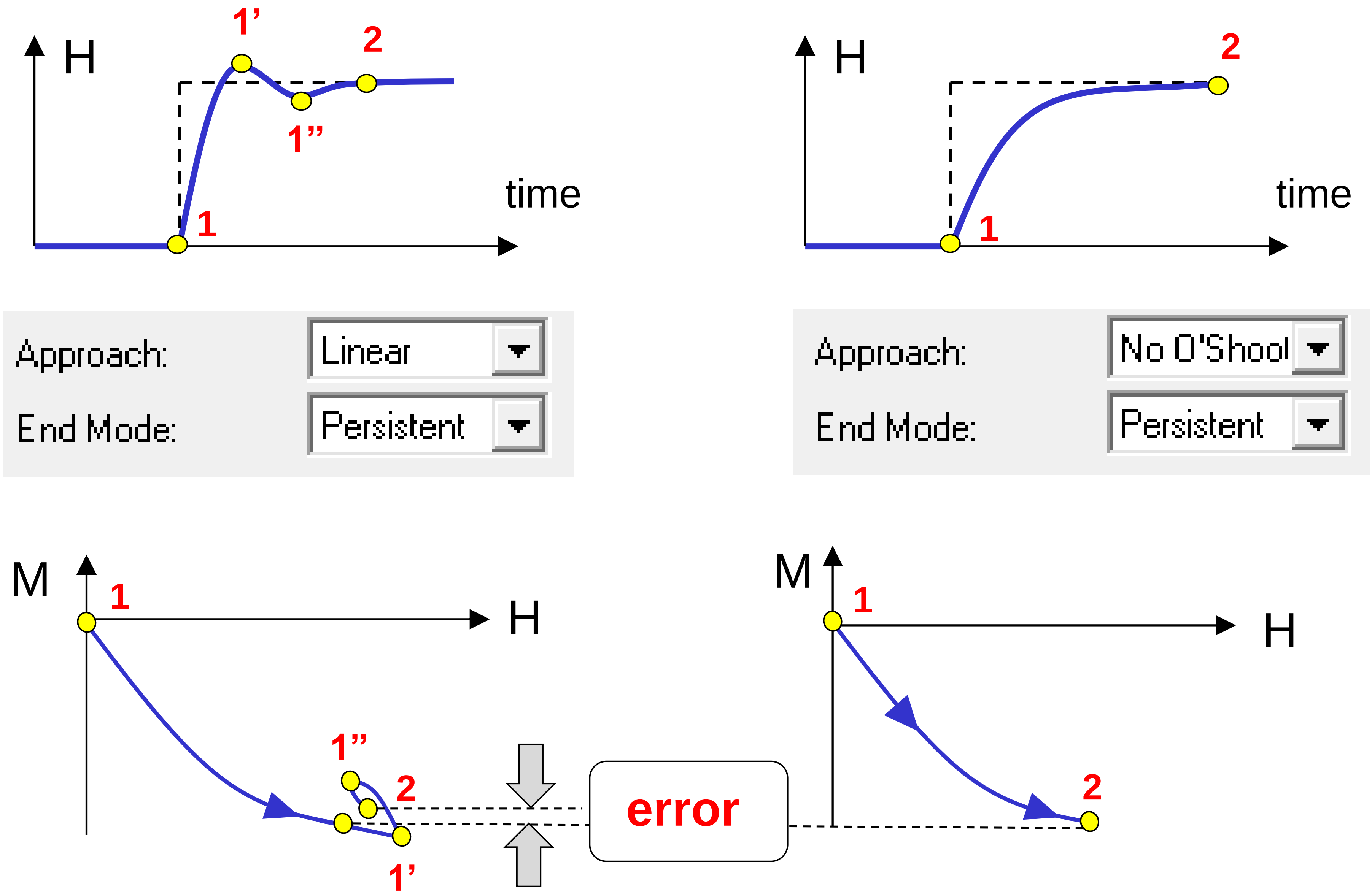








Zoom on the ‘stabilised field’ part



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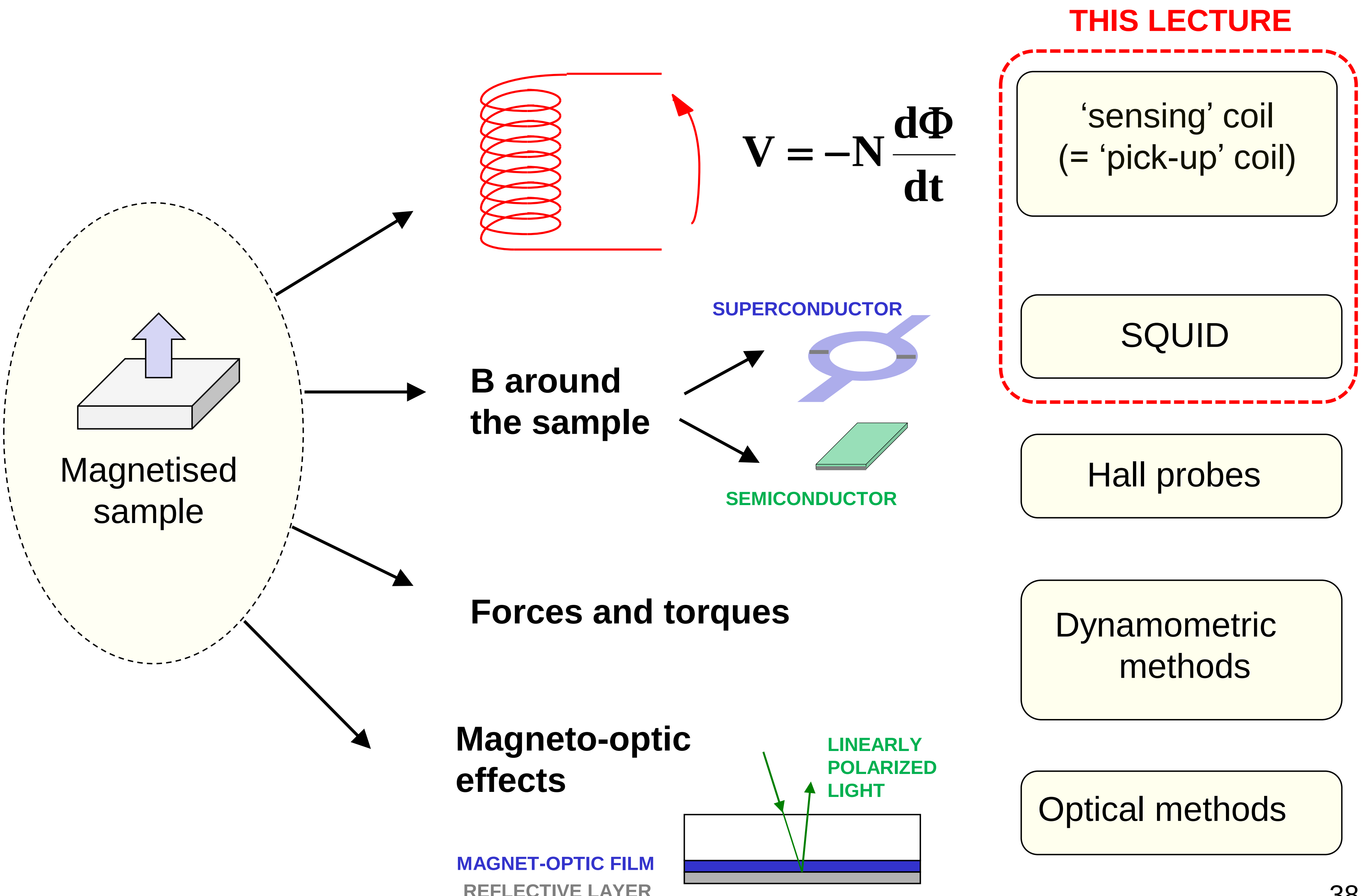
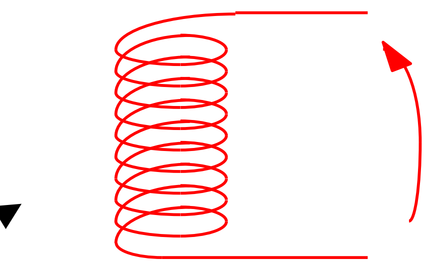
- What kind of information can we extract?

How are we measuring ?



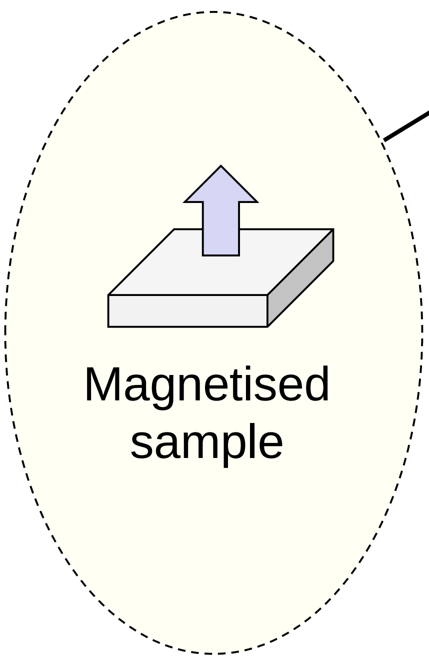
Methods based on sensing coils



$$V = -N\frac{d\Phi}{dt}$$

CONNECTED TO A VOLTMETER

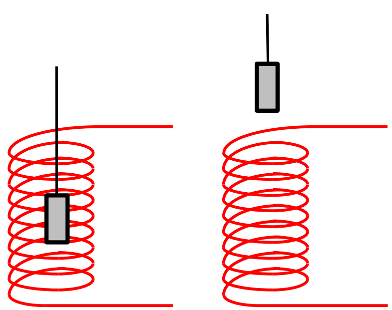
(+ INTEGRATOR)



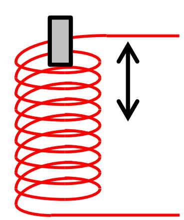
How to produce the time-varying flux $\Phi(t)$?

by **relative motion** of the sample w.r.t. the coil

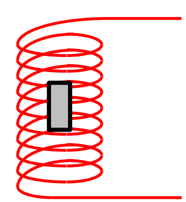
by using a **time-varying** applied field **H(t)**



EXTRACTION



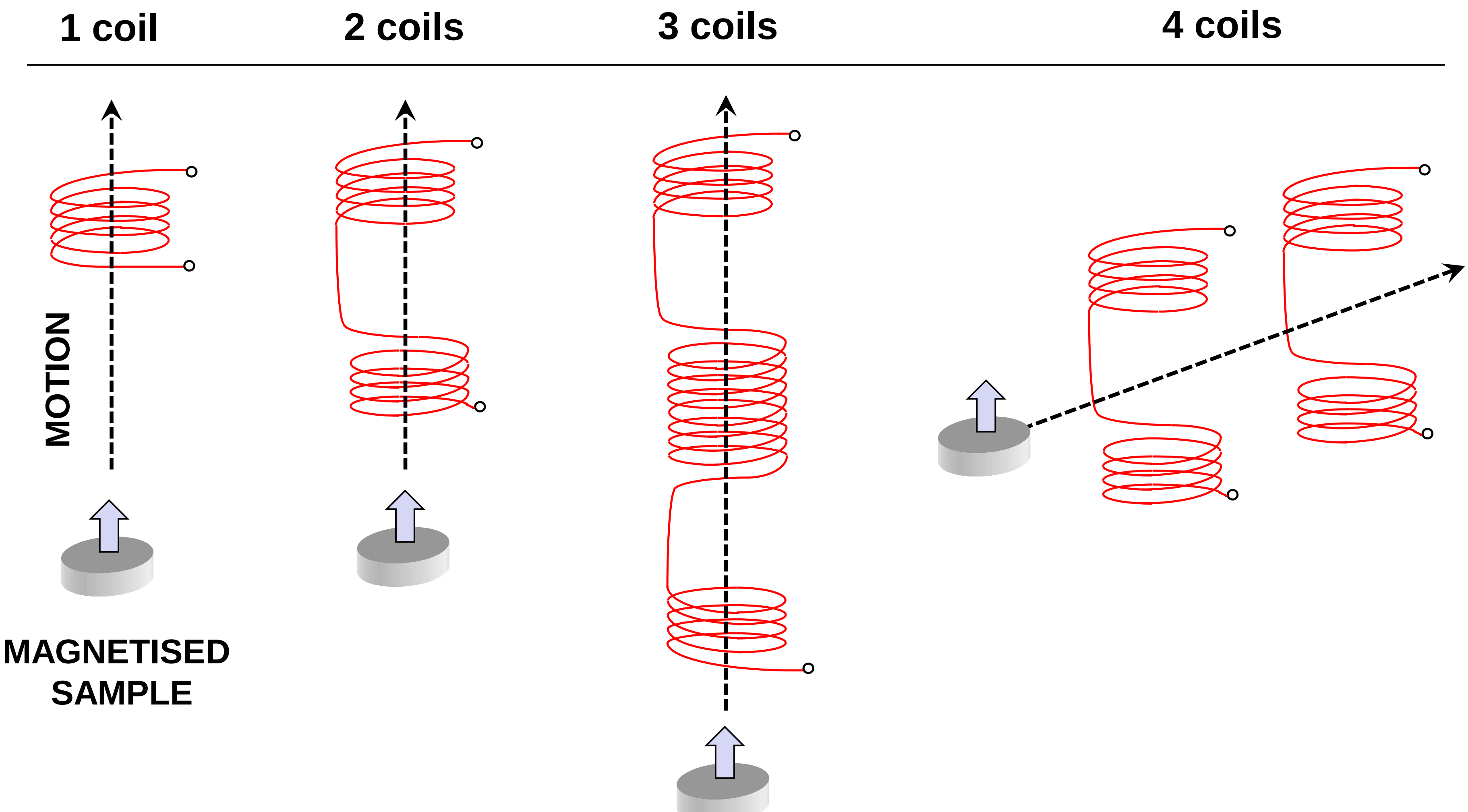
VIBRATION



QUASI-STATIC or ALTERNATING (AC)

Use of several sensing coils ?

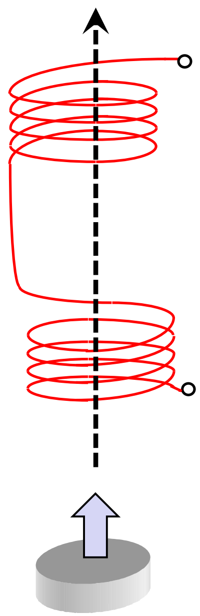
In order to increase the sensitivity and reducing inductive pick-up of unwanted AC magnetic flux, several configurations of sensing coils can be used :



Use of several sensing coils ?

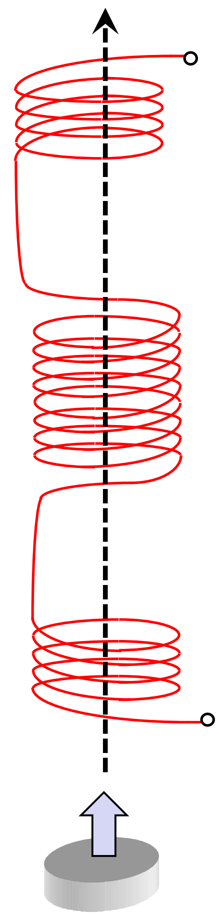
In order to increase the sensitivity and reducing inductive pick-up of unwanted AC magnetic flux, several configurations of sensing coils can be used :

2 coils	3 coils



Series – opposition
= 1st order gradiometer

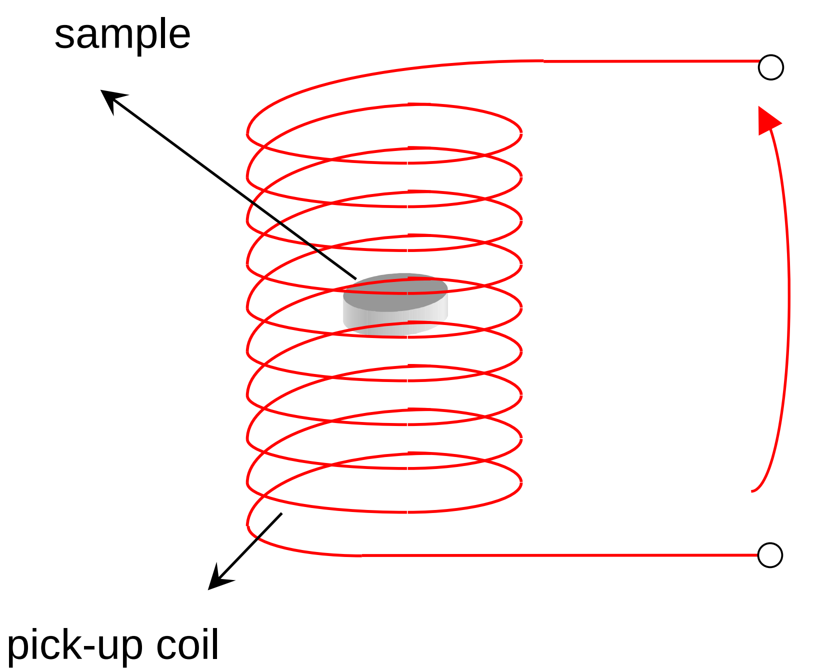
(NO OUTPUT FOR A UNIFORM AC FIELD)

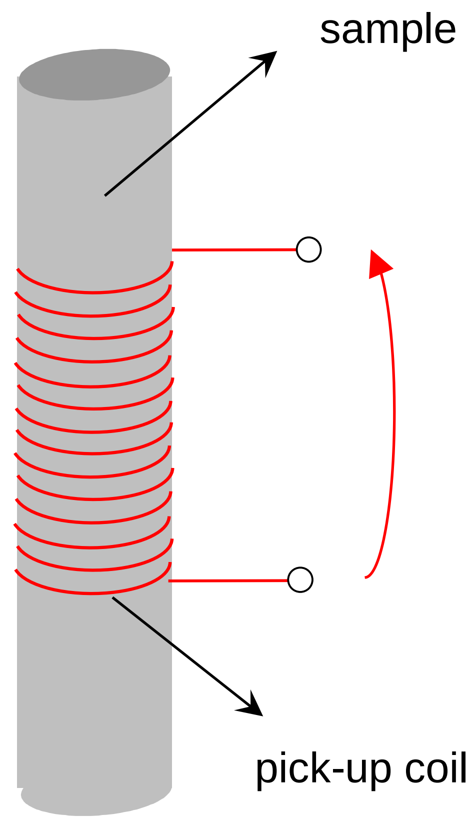


Two sets of series – opposition
= 2nd order gradiometer

(NO OUTPUT FOR A UNIFORM AC FIELD OR A UNIFORM AC FIELD GRADIENT)

Sample and pick-up coil dimensions: Two limiting cases





sample dimensions << coil dimensions

Tightly wrapped coil

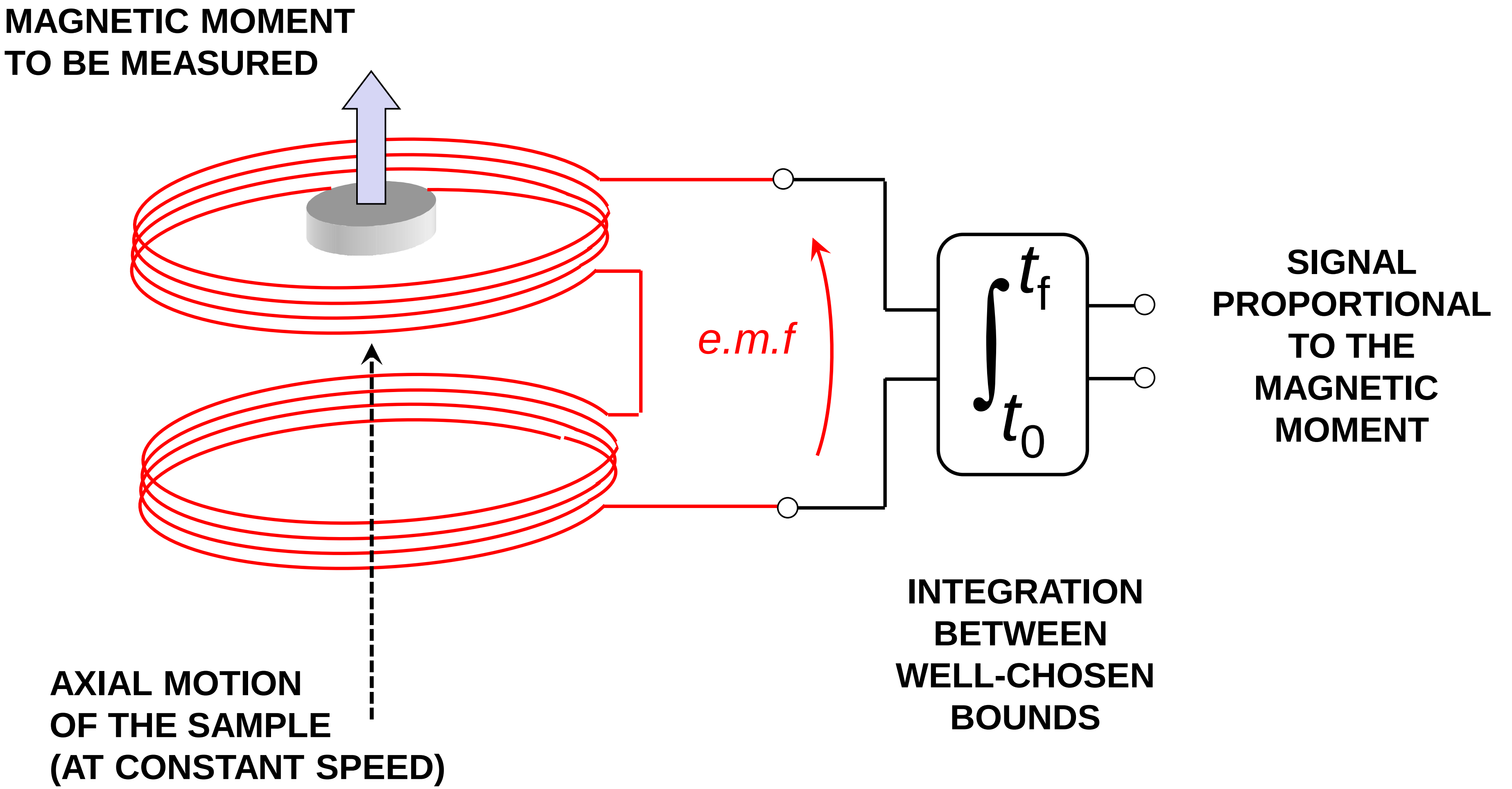
Sensitive to the magnetic moment m ∝ <M>

Sensitive to the magnetic flux ϕ ∝ <B>

Outline for magnetic measurements

- What are we measuring?
- How are we measuring?
  - Extraction method
  - Vibrating Sample magnetometer (VSM)
  - SQUID
  - Fluxmetric measurements

Extraction method



Extraction method : Key points

- Method with a very reasonable sensitivity (**10^{-7} 10^{-8} Am^2**)
- The **radius** of the sample should be **small enough** w.r.t. to that of the sensing coils

- Method used e.g. in the
  Physical Property Measurement System
  (Quantum Design)

Can be designed to make a custom system, e.g. for large samples

Egan et al
Rev Sci Instrum 86 025107 (2015)

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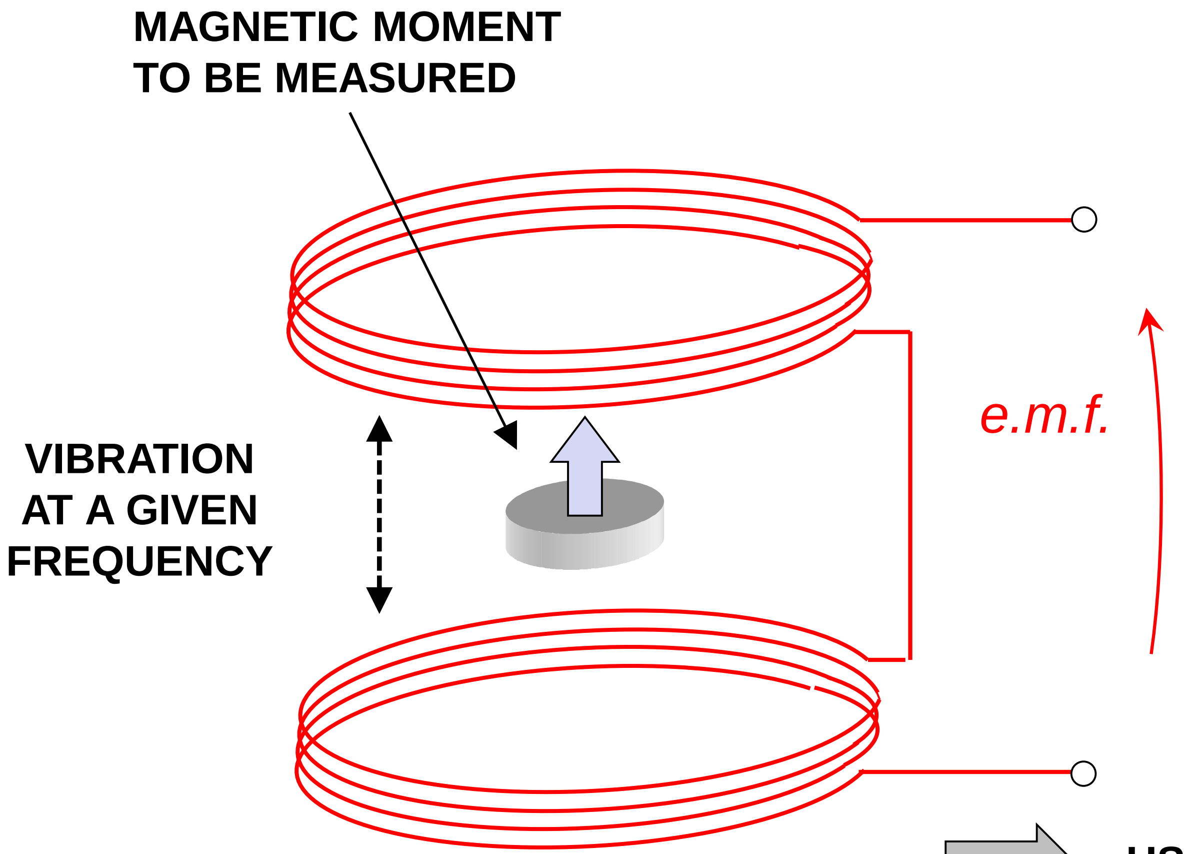
  - Fluxmetric measurements

Vibrating Sample Magnetometer



E.M.F. PROPORTIONAL TO THE MAGNETIC MOMENT

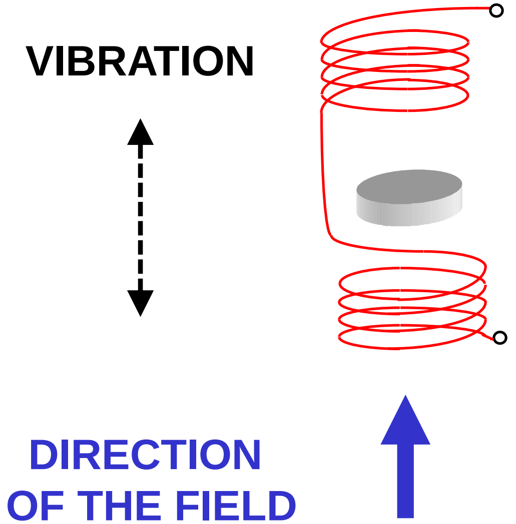
(AND AT <u>THE SAME FREQUENCY</u> AS THE VIBRATION)

➡ **USE OF A LOCK-IN AMPLIFIER**

➡ **EXCELLENT SIGNAL/NOISE**

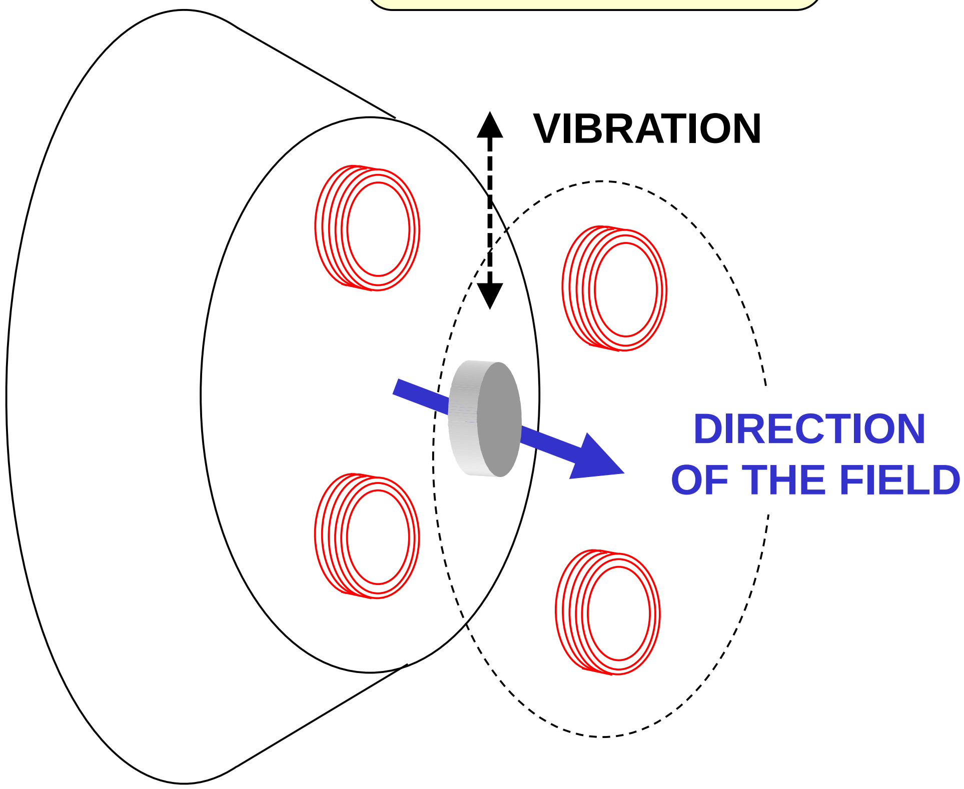
The two VSM types

AXIAL



- 2 sensing coils

RADIAL



- 4 sensing coils

The two VSM types

AXIAL

- e.g. PPMS – Quantum Design

RADIAL

- e.g. 8600 Model – Lake Shore

The two VSM types

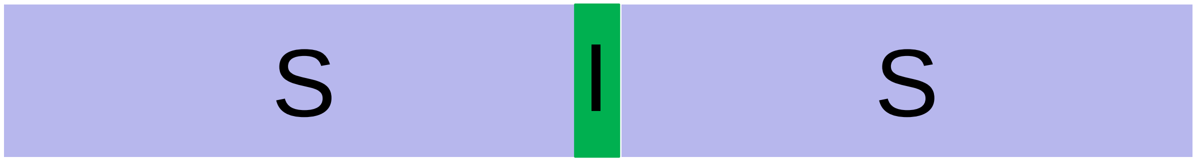
AXIAL	RADIAL
Can use a **superconducting magnet (16 T)**	Mostly **electromagnets (3 T)**
Sensitivity **~10^{-9} Am²**	Sensitivity **~10^{-11} Am²**
Accessible volume depends on the model	Large accessible volume
Requires **liquid helium**	Cryogenic fluids **only for cooling the sample**

Outline for magnetic measurements

- What are we measuring?
- How are we measuring?
  - Extraction method
  - Vibrating Sample magnetometer (VSM)
  - SQUID
  - Fluxmetric measurements

SQUID
=
Superconducting
Quantum
Interference
Device

Interference with
a ‘Josephson’ junction



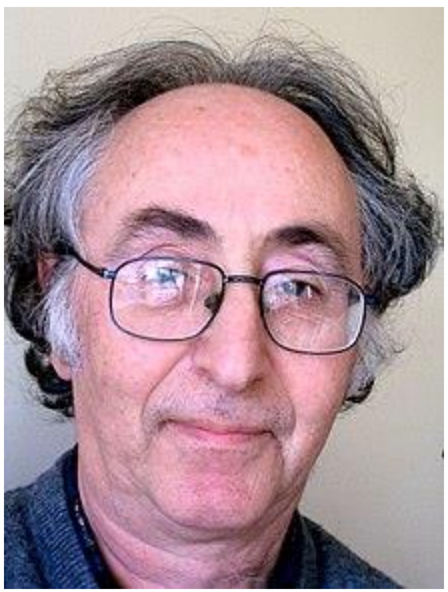
The first prediction of such effects dates from 1962

Volume 1, number 7 PHYSICS LETTERS 1 July 1962

POSSIBLE NEW EFFECTS IN SUPERCONDUCTIVE TUNNELLING *

B. D. JOSEPHSON
Cavendish Laboratory, Cambridge, England

Received 8 June 1962

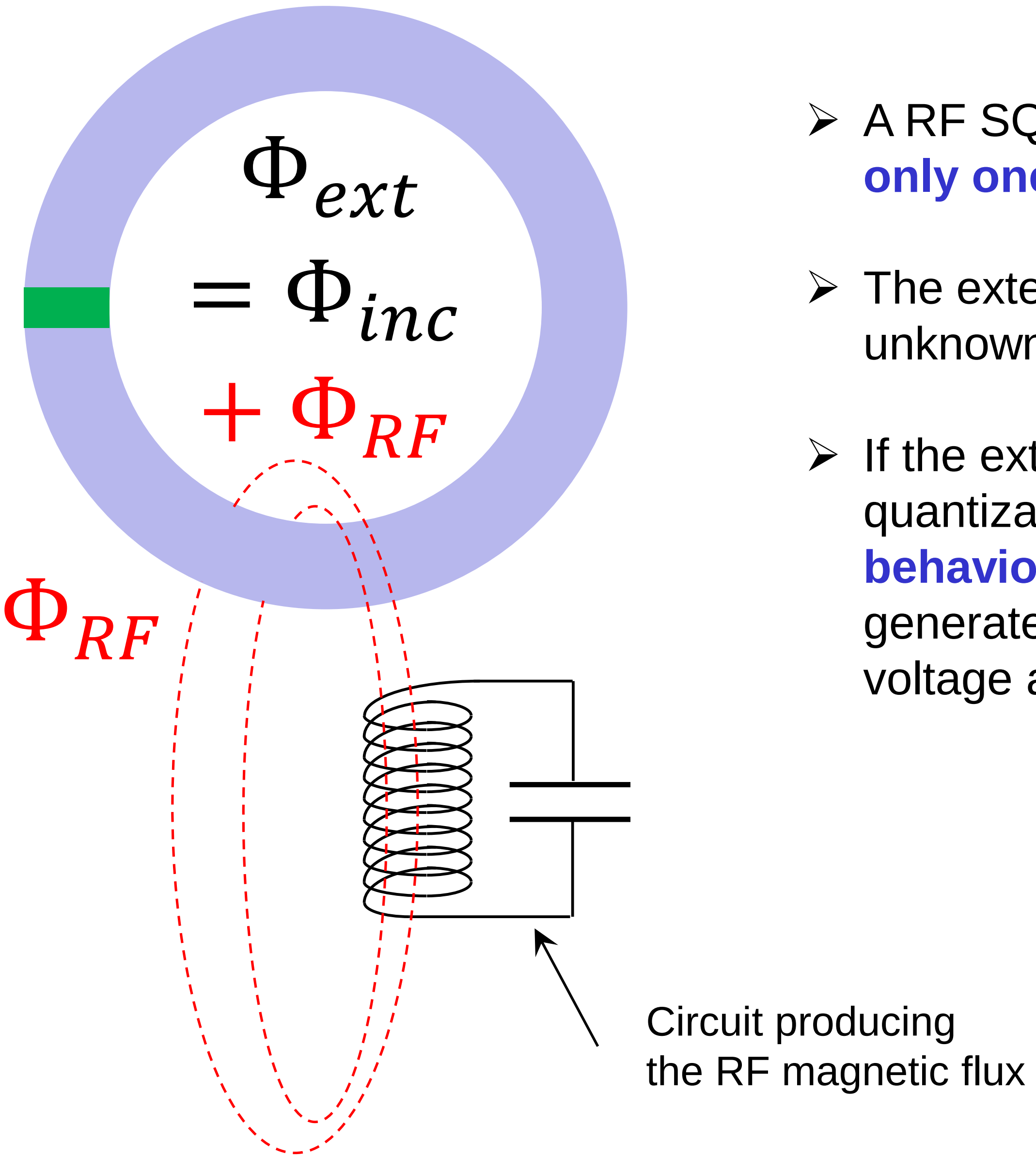


Brian Josephson
(1940 -)

(1973) Nobel prize

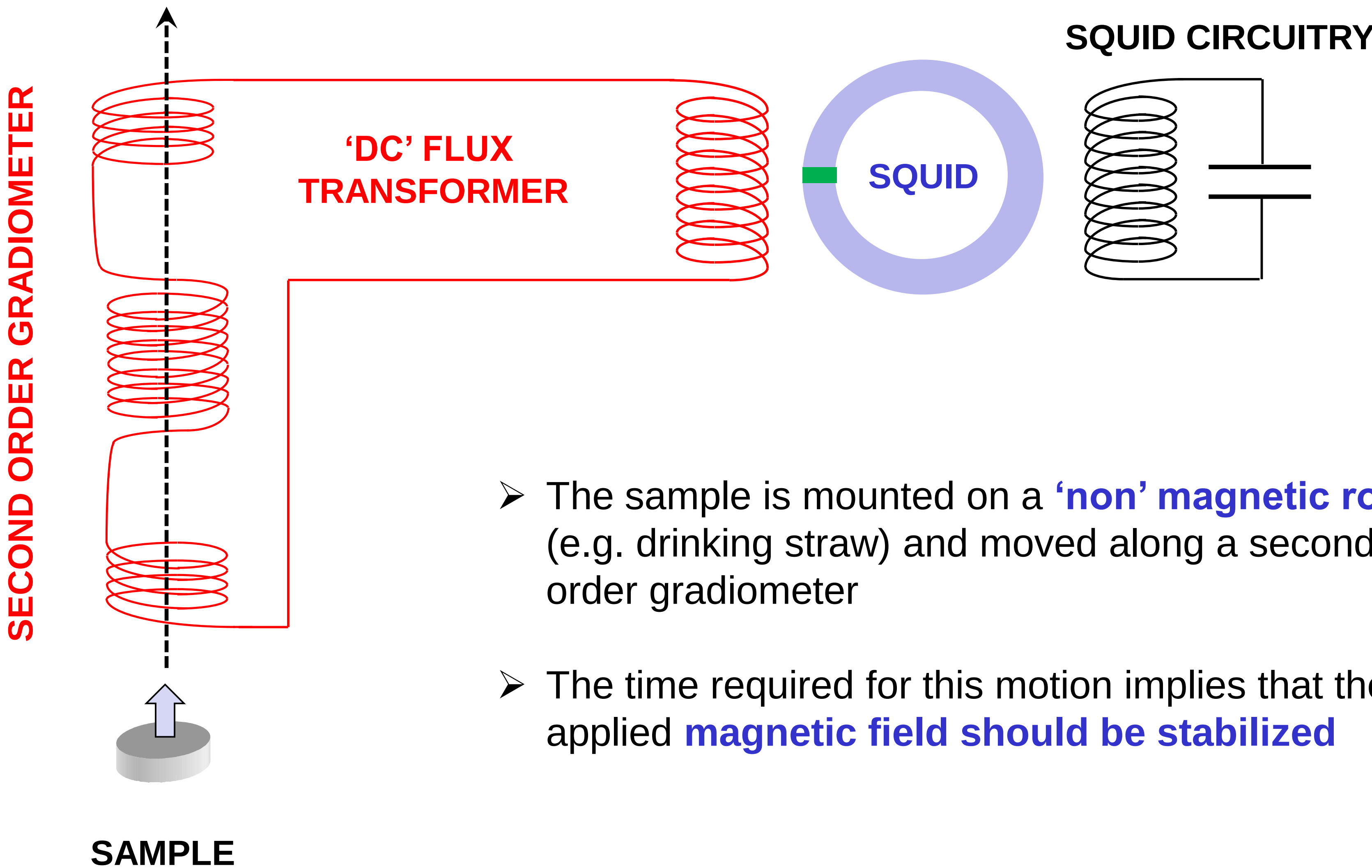
for his theoretical predictions of the properties of a supercurrent through a tunnel barrier, in particular those phenomena which are generally known as the Josephson effects.

The radio frequency (RF) SQUID



- A RF SQUID consists in **a ring containing only one JJ.** No DC current is injected.
- The external flux Φ_{ext} consists now of the unknown flux Φ_{inc} and a RF flux Φ_{RF}.
- If the external flux is changed, the fluxoid quantization creates a **hysteretic behaviour** of the SQUID loop. This generates **losses** that are reflected in the voltage across the RF circuit.

A SQUID in practice



- The sample is mounted on a **'non' magnetic rod** (e.g. drinking straw) and moved along a second order gradiometer
- The time required for this motion implies that the applied **magnetic field should be stabilized**

A SQUID is the MOST SENSITIVE magnetic flux detector.

A squid allows magnetic flux smaller than Φ_0 to be measured.

$$\Phi_0 = \frac{h}{2e} \approx 2 \,.\, 10^{-15}\ \mathrm{Tm}^2$$

- e.g. MPMS3 – Quantum Design
- Typical sensitivity

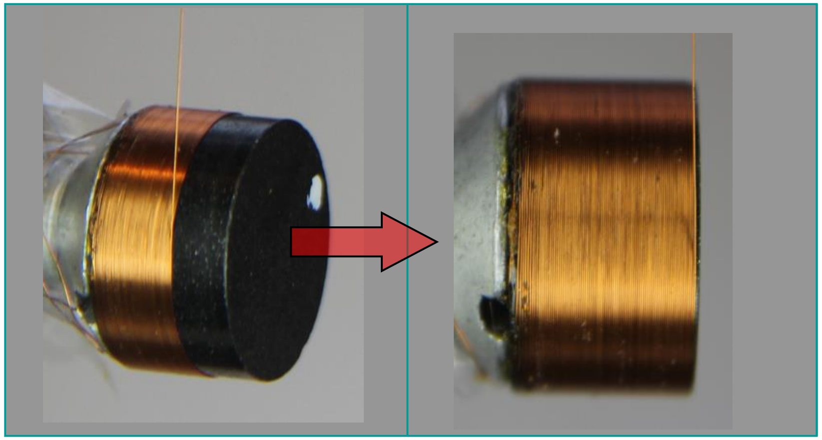
$< 10^{-11}$ Am²

Outline for magnetic measurements

- What are we measuring?
- How are we measuring?
  - Extraction method
  - Vibrating Sample magnetometer (VSM)
  - SQUID
  - Fluxmetric measurements

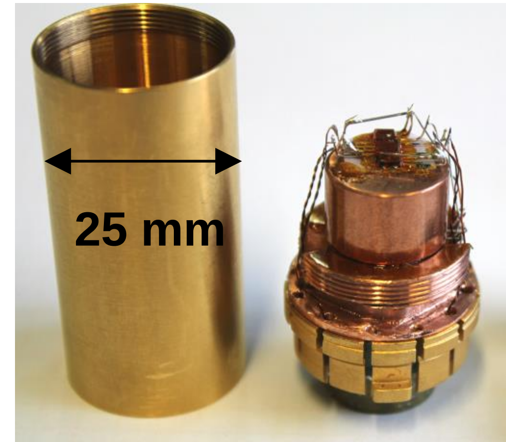
Experimental set-up for flux measurement

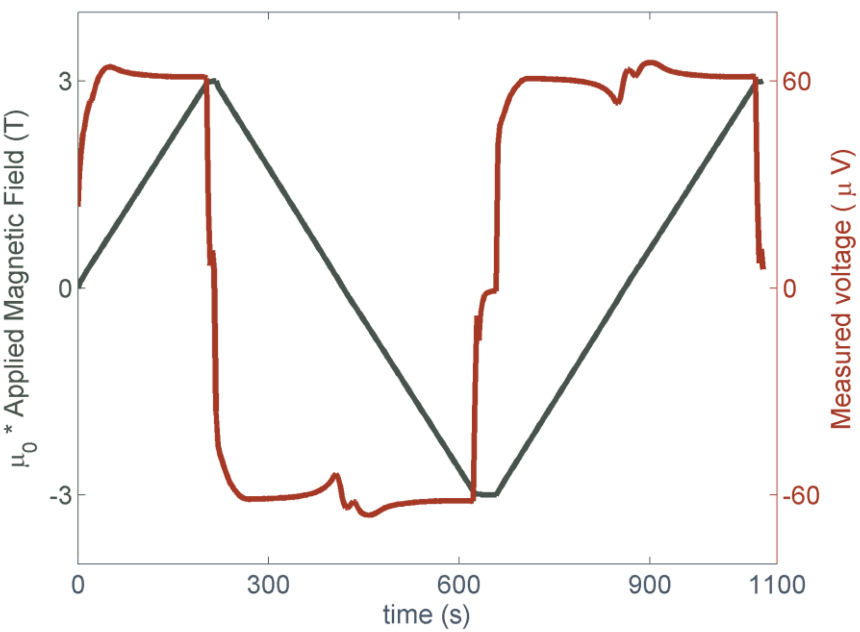
Bulk YBCO



16.5 mm diameter

6.32 mm thickness





Example of induced e.m.f. v across the coil(s) when the field is ramped slowly :

$$v(t) = -N\frac{d\phi}{dt}$$

Typical voltages ≈ **10 – 60 μV**

but requires accuracy of ≈ $\mathbf{0.01}\ \boldsymbol{\mu V}$

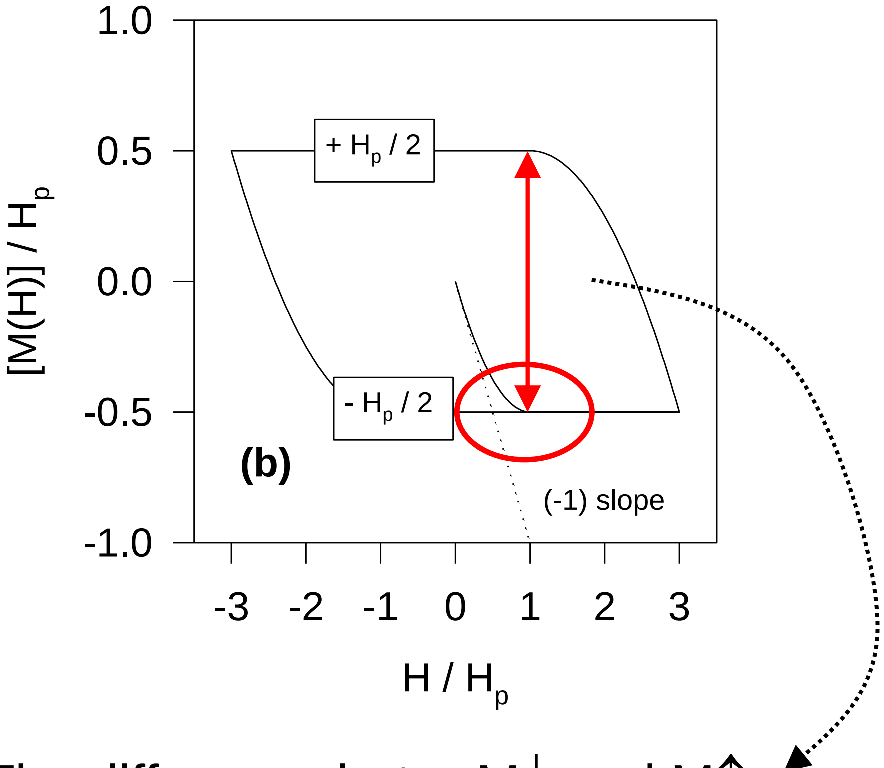
Philippe M P et al. 2014 *Physica C: Superconductivity* **502** 20-30

Outline for magnetic measurements

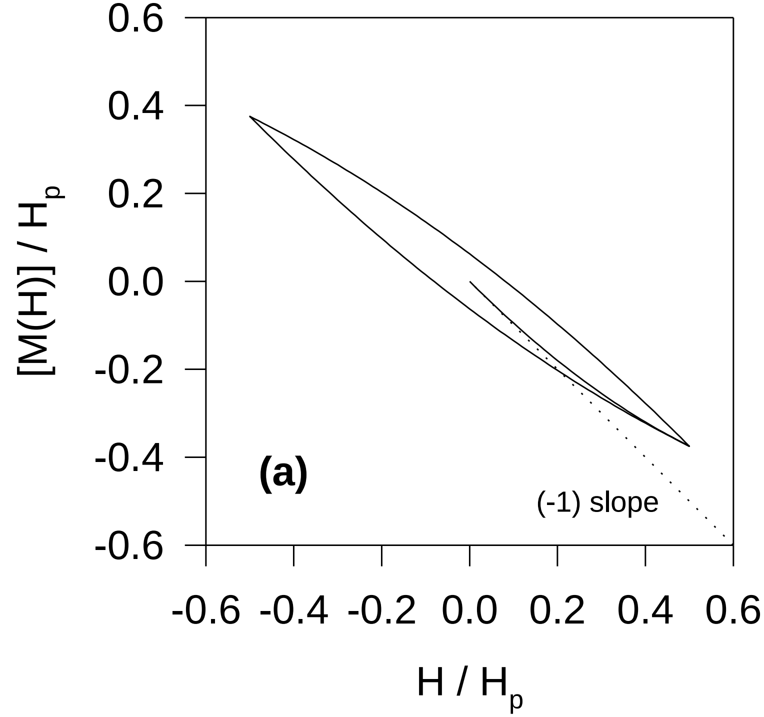
- What are we measuring?
- How are we measuring?
- What kind of information can we extract?

Different "M(H)" curves for type II (hard) superconductor as a function of H_{max}

$H_{max} = 3\ H_p$



$H_{max} = 0.5\ H_p$

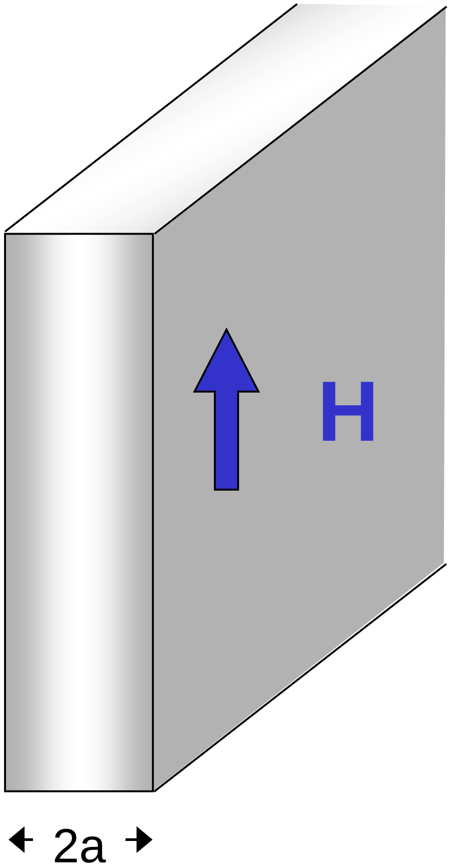


The difference betw. M↓ and M↑
is H_p (= J_c.a) in the case of an infinite slab

BUT… **this is only true <u>when the maximum field</u> H_{max} is large enough !**

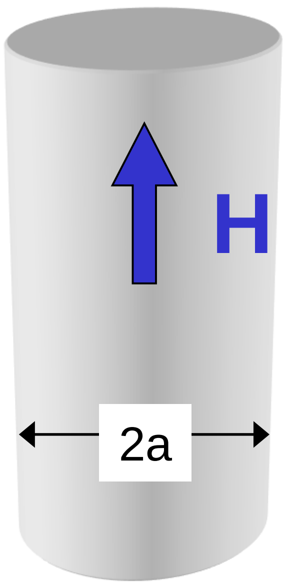
The relation between ΔM and J_c depends on the geometry of the sample

Infinite slab



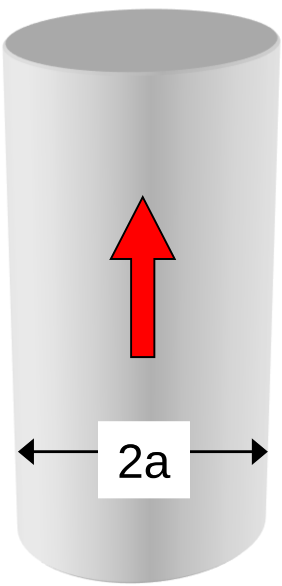
$$J_c = \frac{\Delta M}{a}$$

Infinite cylinder



$$J_c = \frac{3\Delta M}{2a}$$

And what happens if we consider an E-J curve instead of the Bean model ?



H(t) → B(t)

Do NOT forget Faraday's law

$$E = \left(\frac{a}{2}\right)\frac{dB}{dt}$$

There is always an **electric field** in magnetic experiments !
The amplitude of this field is **much smaller than in transport experiments**

Do not forget to consider these 3 quantities…

Current density :	**J (A/m²)**
Magnetic flux density :	**B (T)**
Electric field :	**E (V/m)**

Supercond. Sci. Technol. 7 (1994) 412–422. Printed in the UK

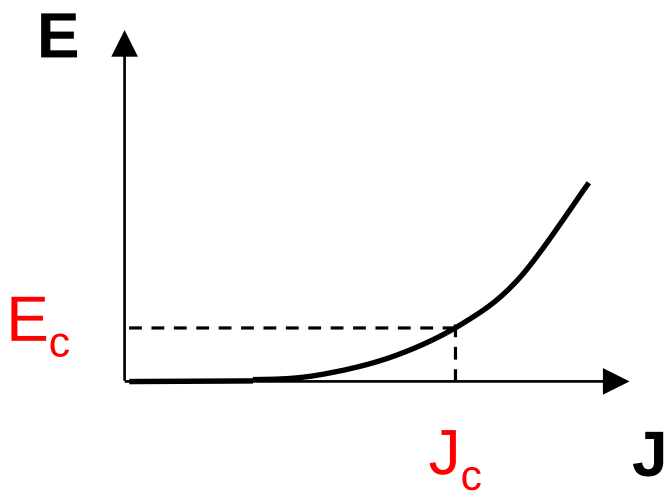
The electric field within high-temperature superconductors: mapping the *E*–*J*–*B* surface

A D Caplin, L F Cohen, G K Perkins and A A Zhukov†

Centre for High Temperature Superconductivity, Blackett Laboratory, Imperial College, London SW7 2BZ, UK

Received 13 January 1994

Consequence …



The amplitude of induced currents increases for large dB/dt !

Always specify dB/dt in 'DC' experiments !

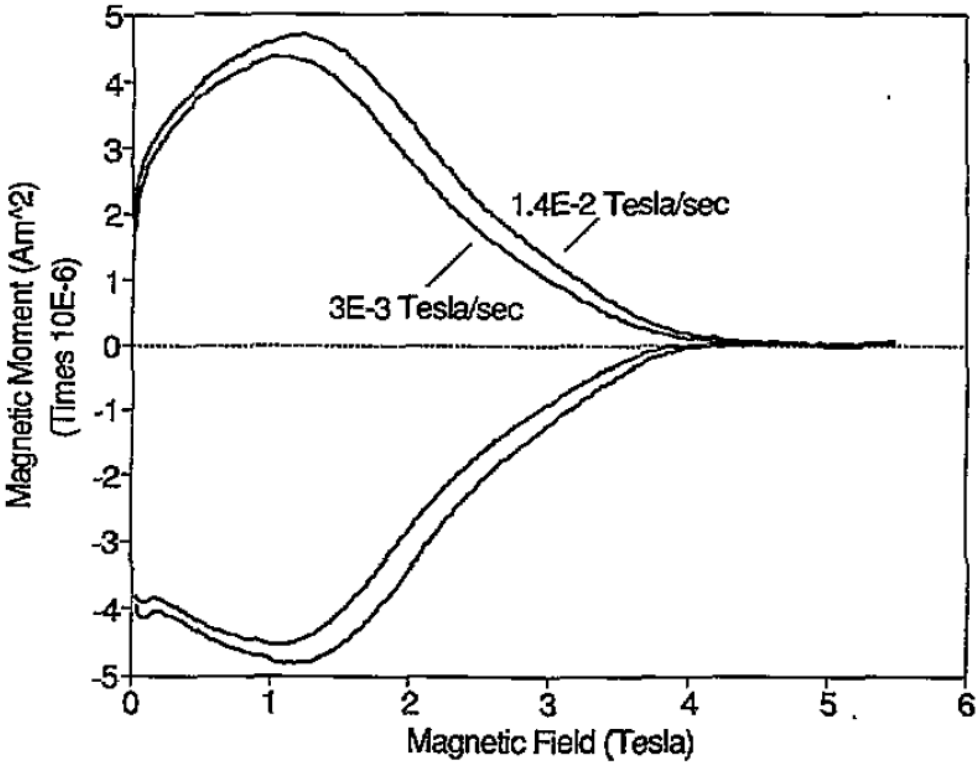
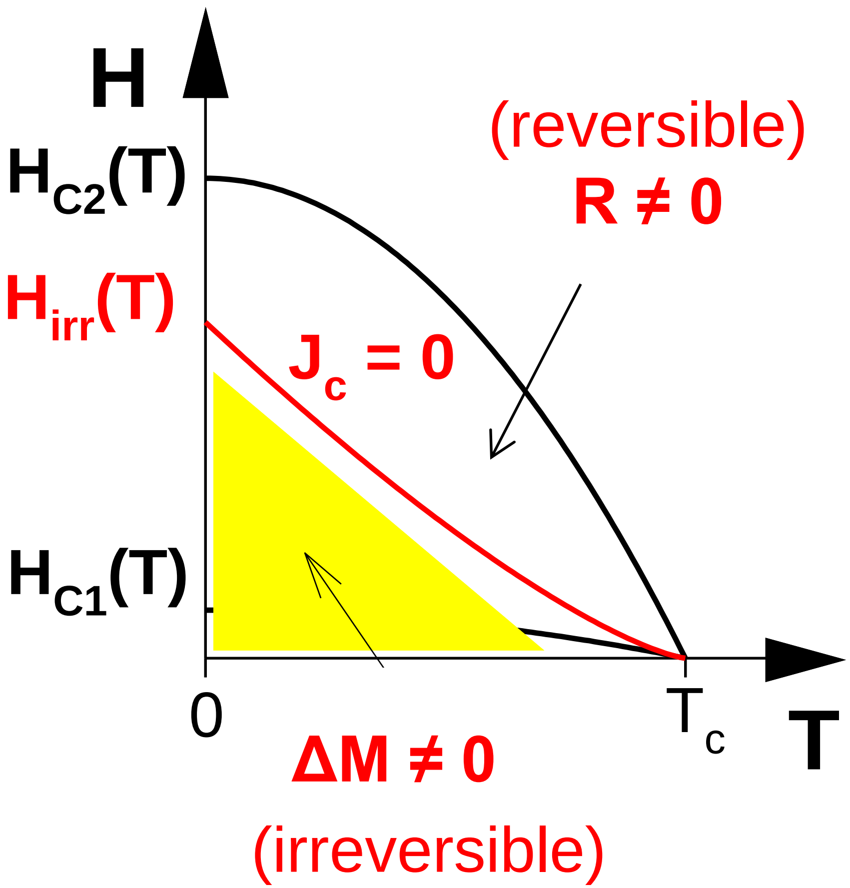
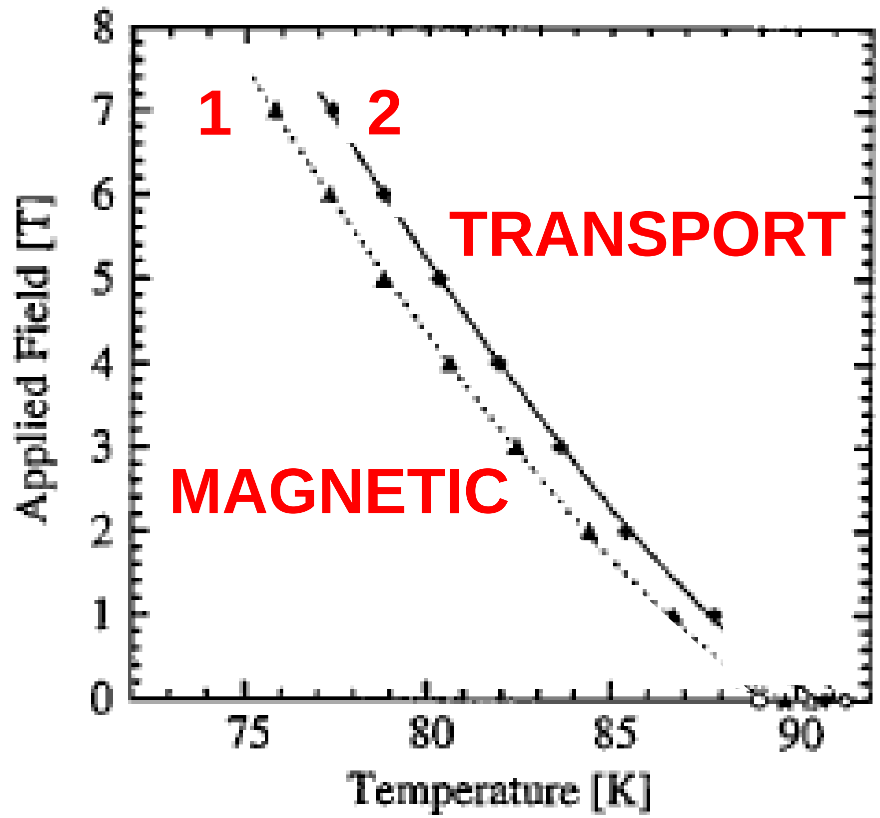


Figure 1. Typical magnetization loops of a high-quality $YBa_2Cu_3O_7$ single crystal at 84 K. Two loops are shown, the outer one having a field sweep rate $\dot{H}_{app}$ of about five times the inner one. H_{app} is parallel to the c-axis. Note the maximum (the 'fishtail' feature) in the magnetic moment at

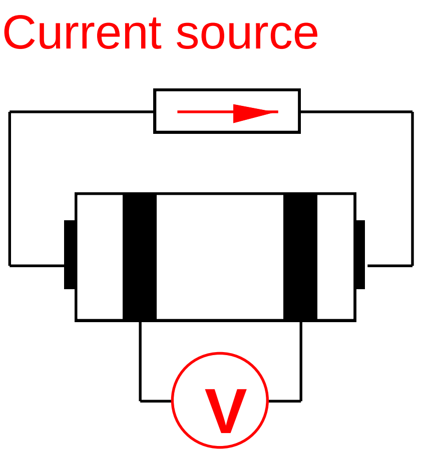
Irreversibility field from TRANSPORT and MAGNETIC

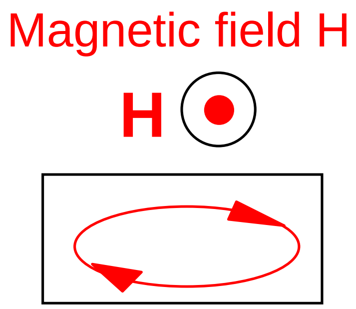




Doyle et al., APL 73, 117 (1998)

Conclusion





Transport current
(applied externally)

Induced current
(by the applied magnetic field)

Both kind of measurements are very useful and can provide invaluable information on the material properties

BUT … Be always careful when interpreting the results !

Thank you for your attention

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