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EXPERIMENTAL WORKSHOP ON HIGH TEMPERATURE SUPERCONDUCTORS
(11 - 22 April 1988)

PHYSICAL CHARACTERIZATION - ELECTRICAL RESISTANCE, MAGNETISATION, SUSCEPTIBILITY

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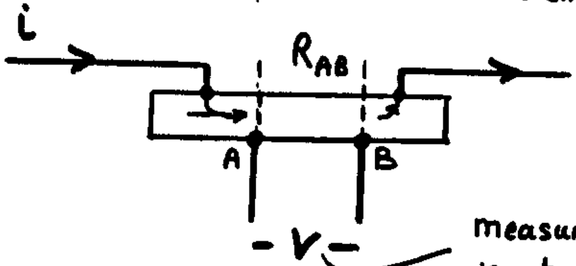
Physical characterisation - Electrical Resistance

Magnetisation, Susceptibility

Electrical resistance - 4 point measurement

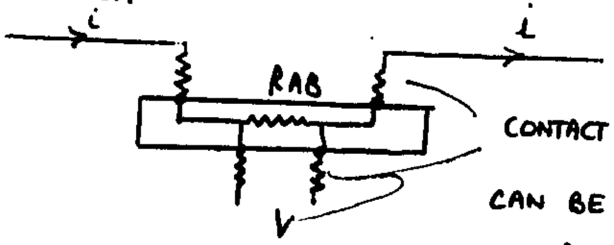
a.c. better than d.c. (no thermal emfs, high sensitivity)



measure with high input impedance amplifier $\gg R_{AB}$

$$V = R_{AB}\, i$$

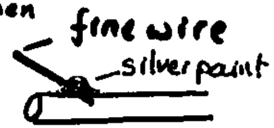
ACTUAL CIRCUIT

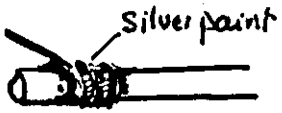


CONTACT RESISTANCES CAN BE VERY HIGH $\gg R_{AB}$

∴ MAKE GOOD ELECTRICAL CONTACTS

* Press-contacts - convenient - sometimes large contact R,
  - may crack delicate specimen
* Silver-paint - apply at RT + leave to dry



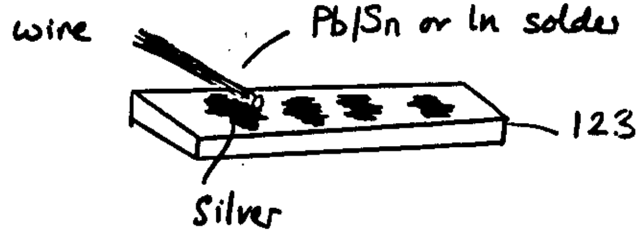


heavy currents

I_c measurements

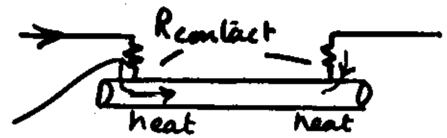
* Silver-paint - apply at RT - in green-state before final heating. or heat to ~400°C in oxygen. Direct soldering.

* Silver sintered onto surface



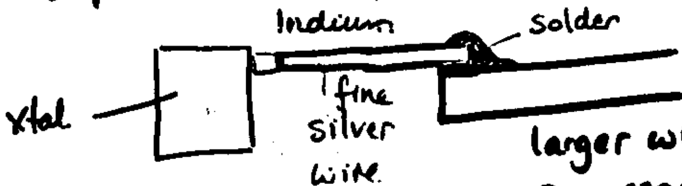
* Ultrasonic soldering of silver wires direct to surface using In, Pb/In solders

Critical current measurements must have low contact resistance leads



Say 1Ω → 1 W dissipation - heats wire + turns it normal - see LN_2 bubbles

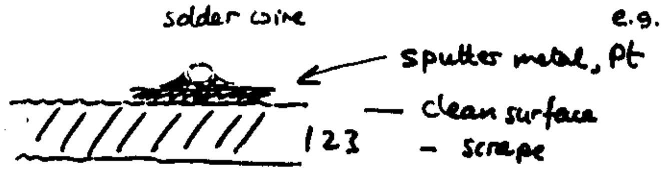
* Single crystals - Silver paint - very fine wires or solder Indium-tipped wires direct to surface ~ heat tip + press into surface



or tags } larger wires, if necessary, soldered to fine wires

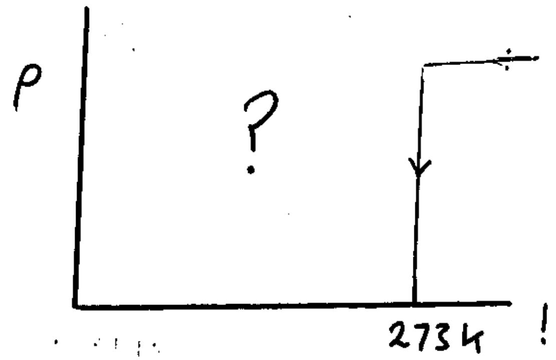
Reduce mechanical strain on connecting region

* Single xtal - or sintered material - best possible contacts
Evaporated or sputtered metal on surface then solder.

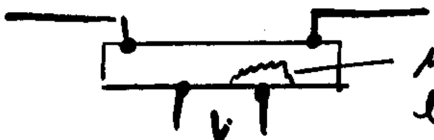


Checks to make when you discover $T_c > 273K$!

Resistance



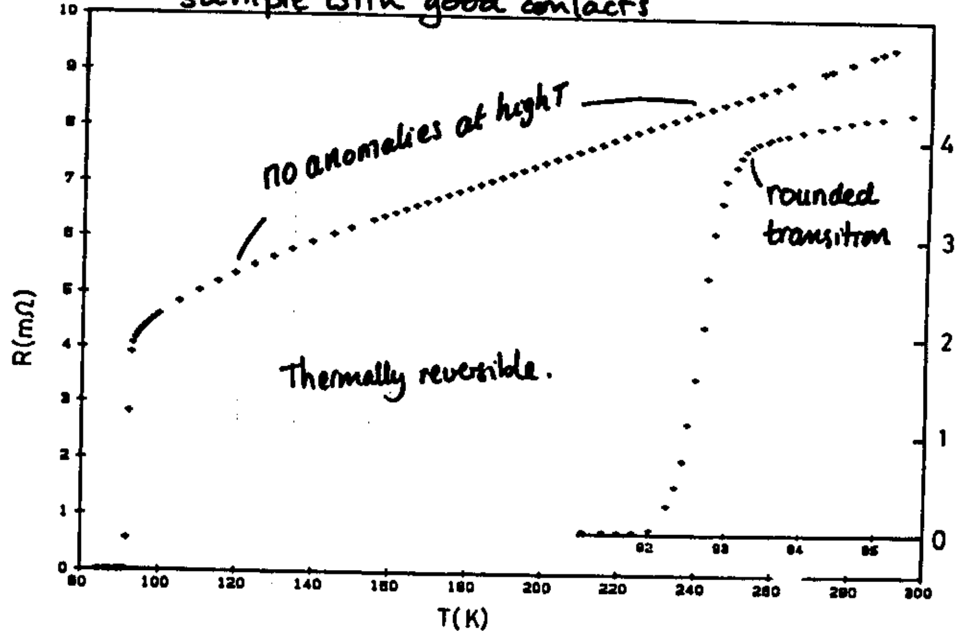
* Is it thermally reversible?
* Is it reproducible?
* Do you get same result when you interchange I/V leads? — thermal cracking can give spurious results

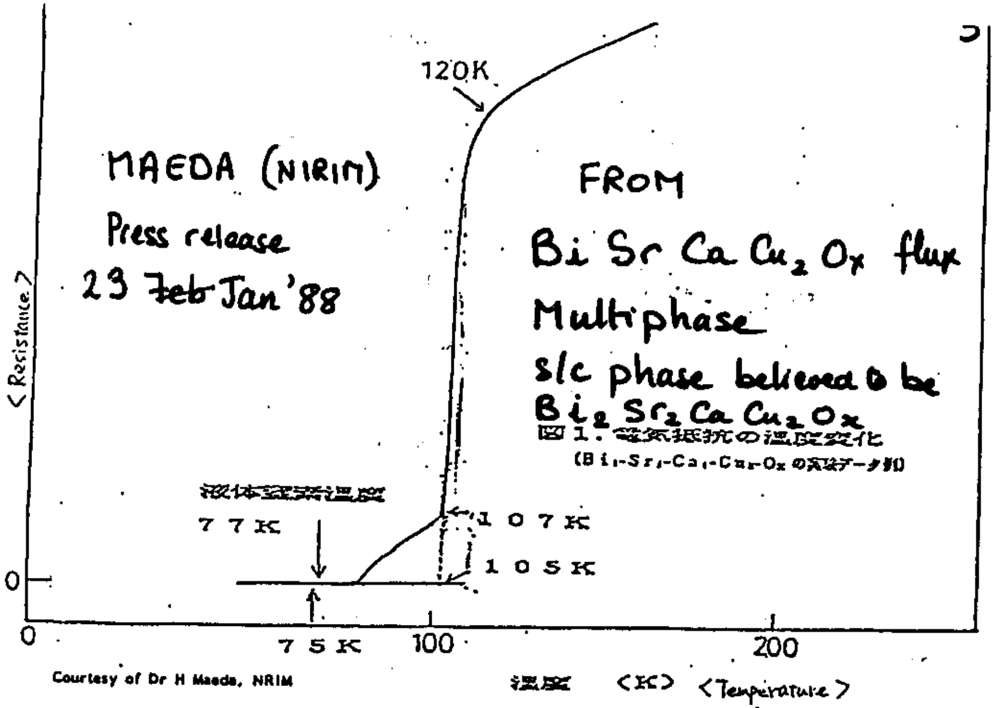


μ-crack isolates I voltage lead → V=0 BUT NOT SUPERCONDUCTIVITY

* MEASURE CONTINUITY OF LEADS.

Typical measurements for well-sintered sample with good contacts





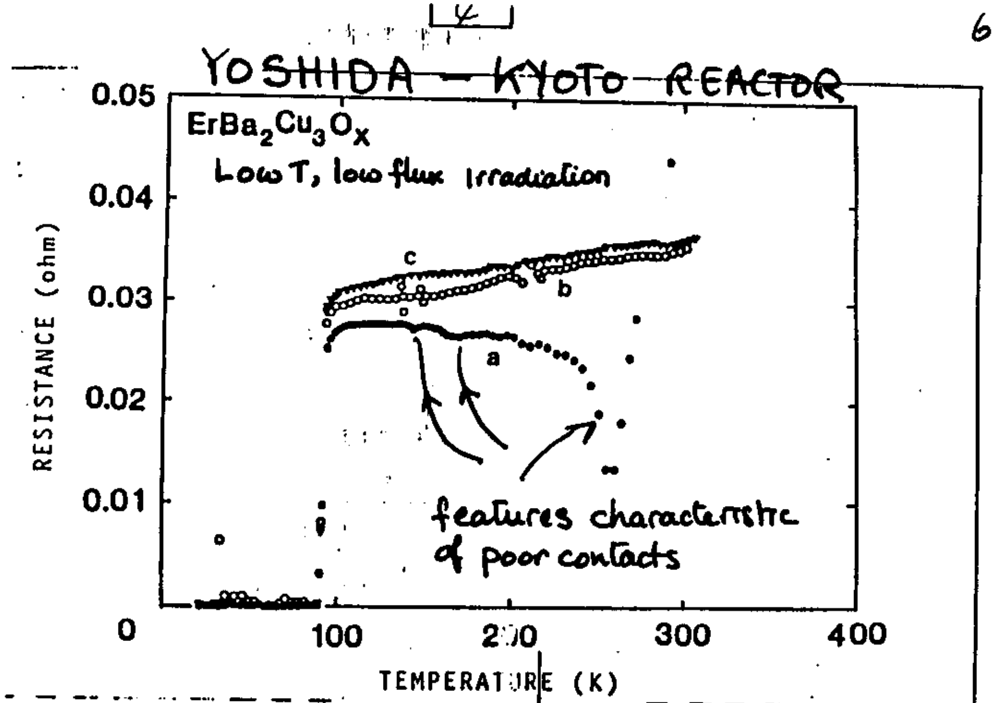
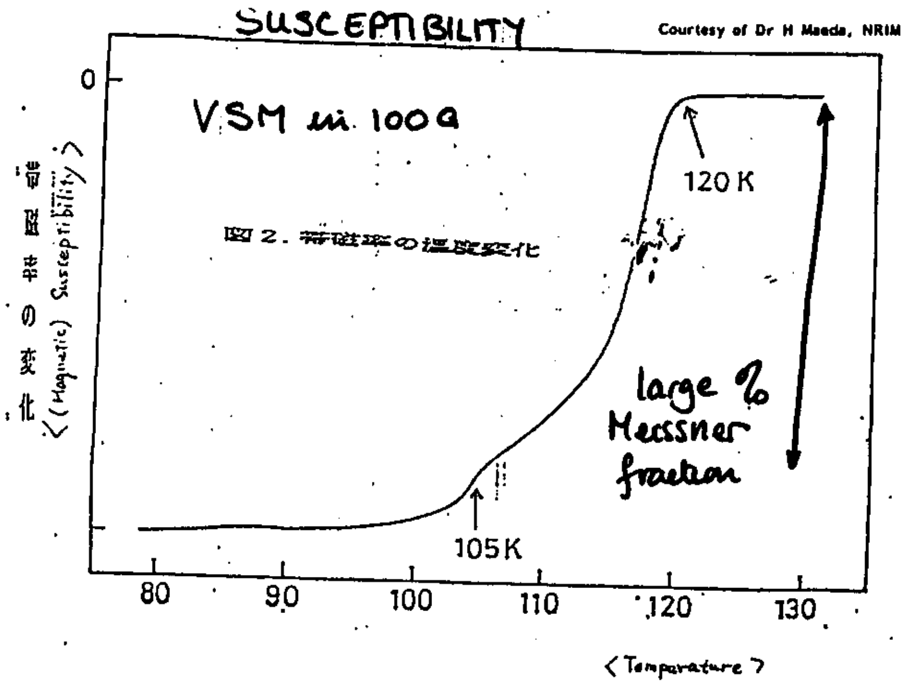
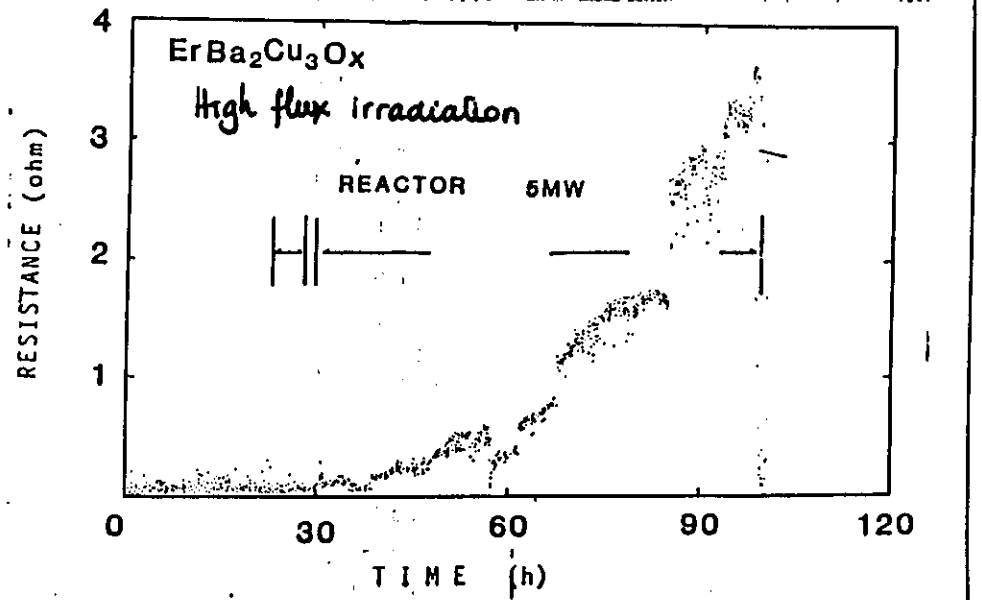


Figure 3 Resistance vs. temperature curves for the ER-2 specimen measured before and after the 25 K irradiation. a:before irradiation, b: after 25K irradiation, c: after annealing for 1 h at 320K

Figure 4.4 - Magnetic Susceptibility vs Temperature Plot for New Bi Based Material





6a 6 $\sim 10^{18}$ n/cm² { 50% fast, 50% thermal

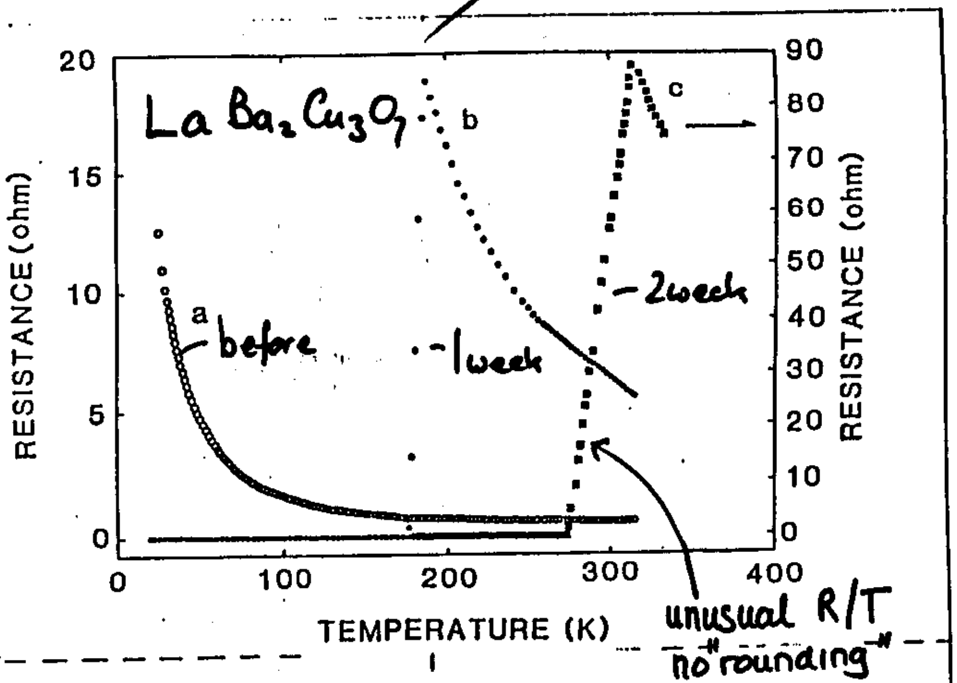
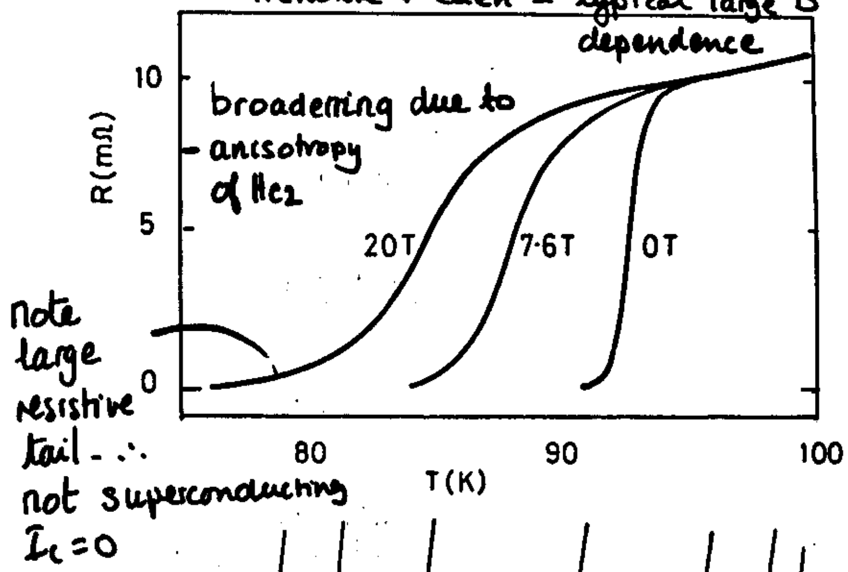
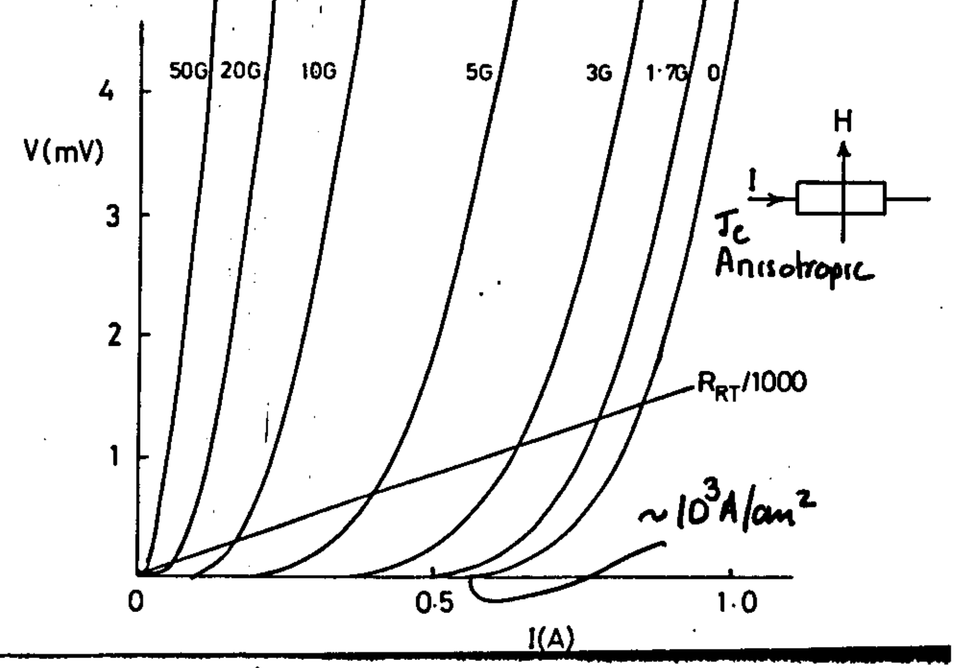


Figure 5 Temperature dependence of resistance measured during cooling down for a La-Ba-Cu oxide (LA-3). a: before irradiation, b: after the first-week irradiation at 360K, c: after the second-week irradiation at 360K

* YOSHIDA – KYOTO REACTOR
* MEASUREMENTS ALL "IN PILE"
* MORE RESISTANCE AND χ MEASUREMENTS PLANNED
* NOTE (i) LARGE RESISTANCE
  (ii) VERY SHARP TRANSITIONS

Grenoble + Caen – typical large B dependence

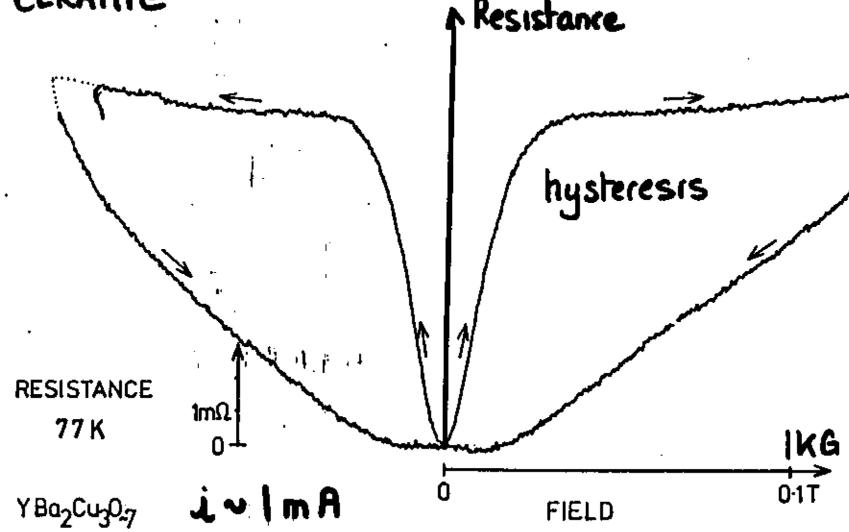




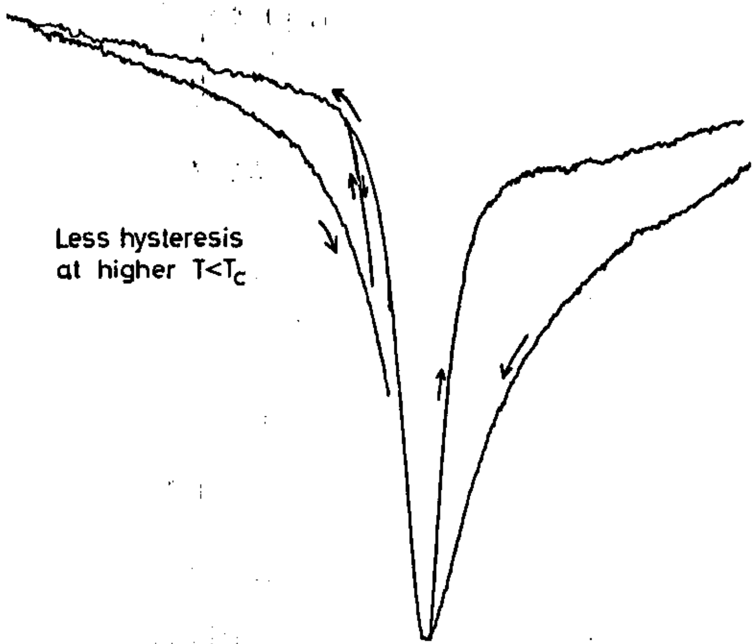
Birmingham – typical small field dependence

Magnetic resistance hysteresis

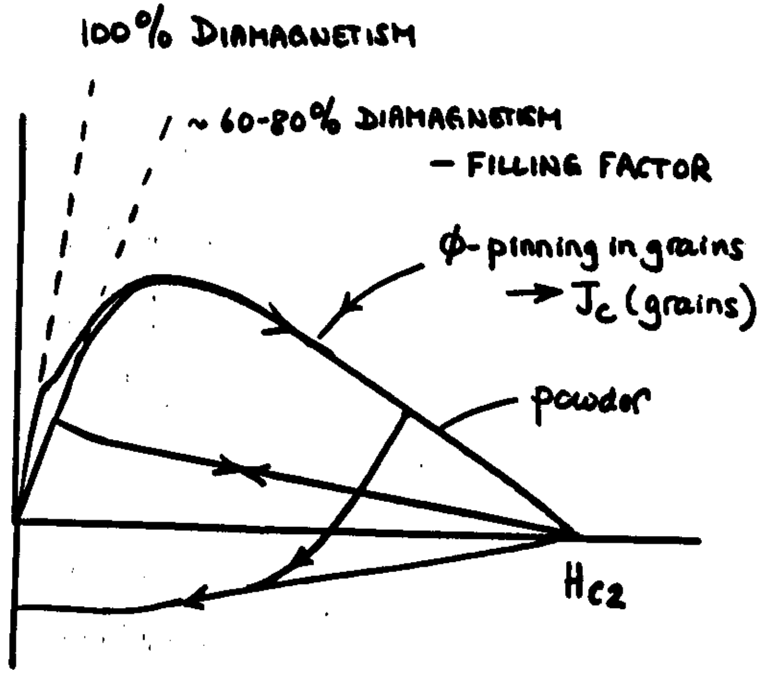
CERAMIC



Superconductivity destroyed by very small fields
~ 10s of gauss



CERAMIC MAGNETISATION

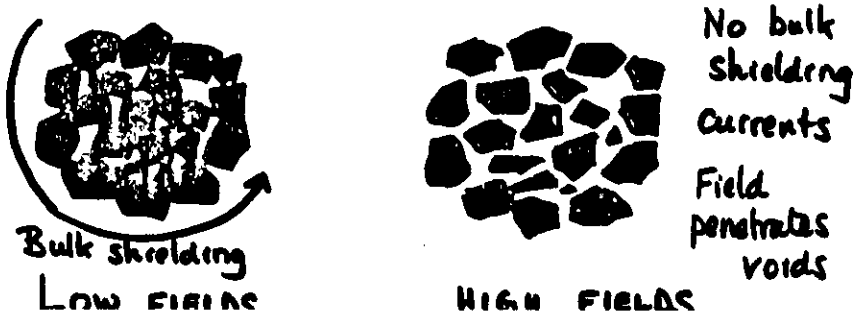


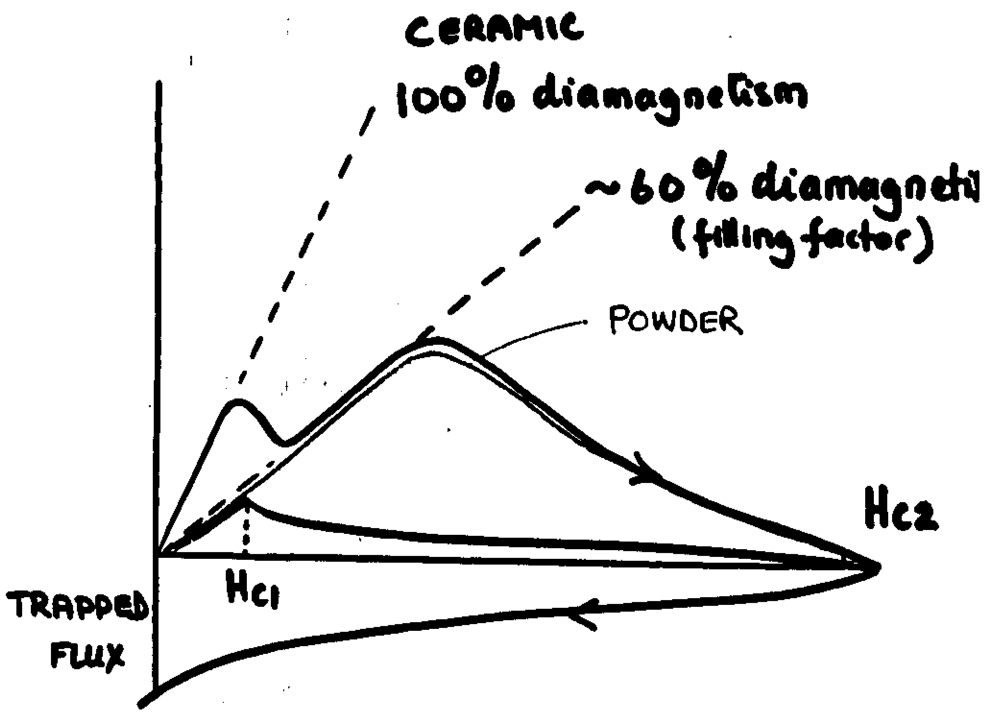
POWDER ALONE IS STRONGLY DIAMAGNETIC

— repelled by a magnet

NOT NECESSARILY AN ELECTRICAL SUPERCONDUCTOR

SINTERED SAMPLE





① Initial diamagnetism 100%

— SHIELDING CURRENT THROUGH BULK OF SAMPLE.

② Bulk currents suppressed by field

— SHIELDING ROUND GRAINS ALONE

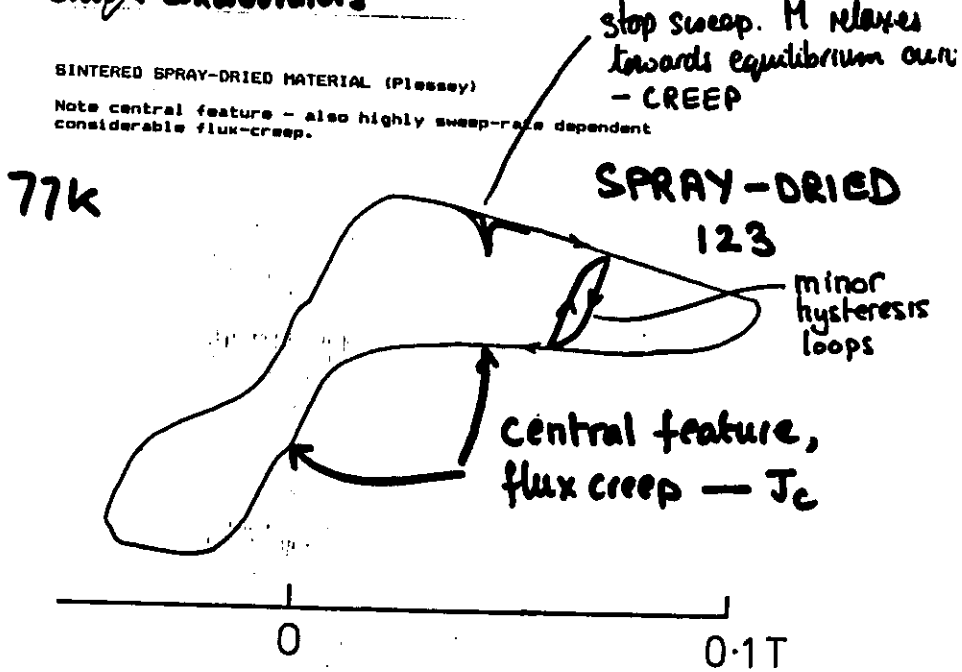
— H may effectively supress bulk s/c

DERIVE J_c (BULK) + J_c (GRAINS)

Bednorz & Muller
Chu & Collaborators

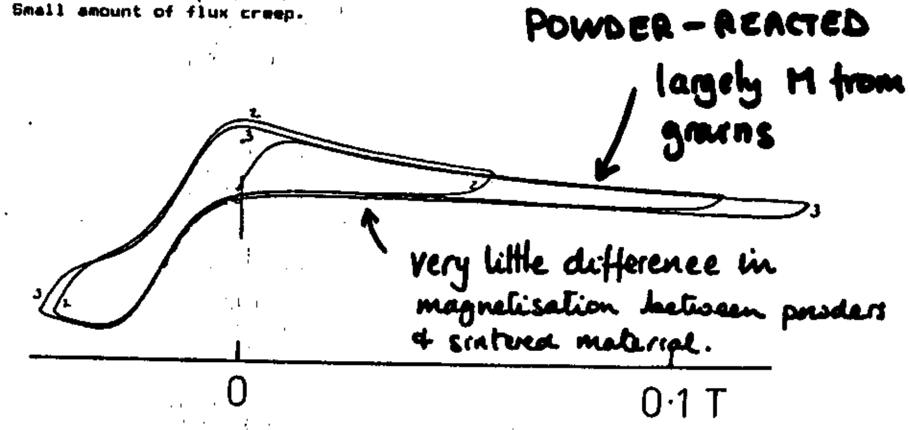
SINTERED SPRAY-DRIED MATERIAL (Plessey)

Note central feature - also highly sweep-rate dependent considerable flux-creep.



SINTERED POWDER-REACTED MATERIAL (Birmingham/Lucas)

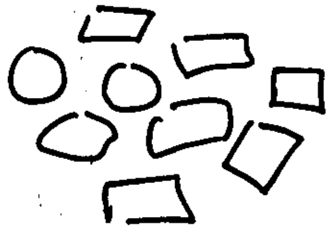
No significant central feature though observed on several other samples.
Small amount of flux creep.



Magnetisation of single-phase $YBa_2Cu_3O_7$ ceramic rings at liquid-nitrogen temperatures. Vertical scales are uncalibrated.

Nature of magnetic hysteresis

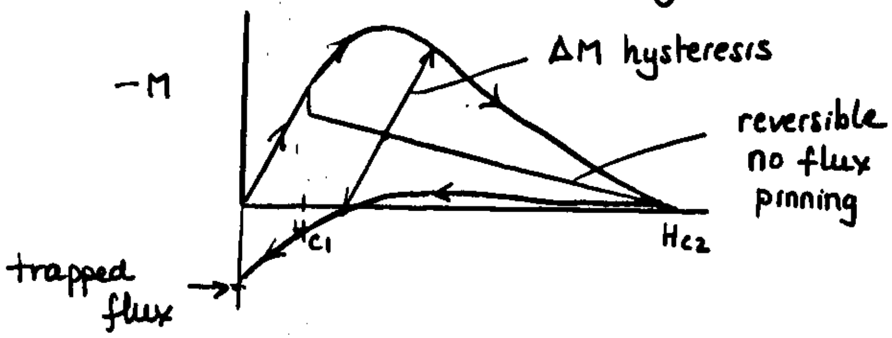
1. Consider 1st magnetic properties of powder – no connections between grains



grains

Each grain will tend to exclude magnetic flux

Magnetisation of sample = Σ magnetism of grains



Hysteresis is due to finite critical current density J

If $J_c = 0$ – perfectly reversible.

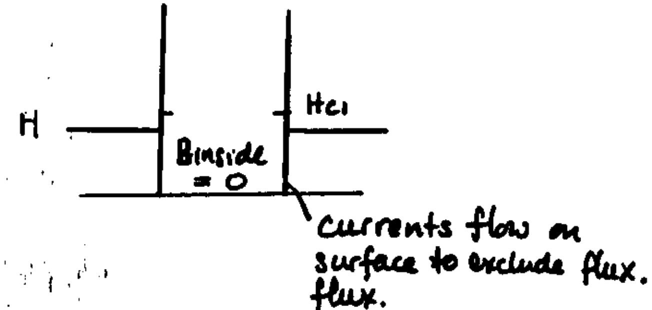
lattice imperfections, domain walls, twin-boundaries may all act as flux pinning centres – increases J_c

Also neutron irradiation damage – defects in general

Origin of magnetic hysteresis – Bean model

Consider a slab

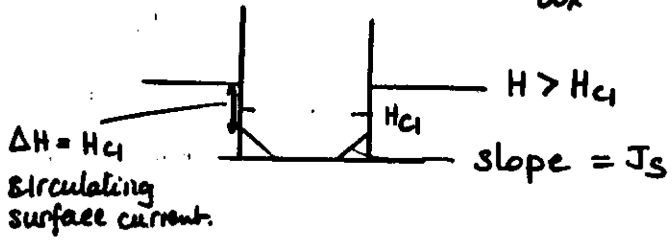
$H < H_{c1}$



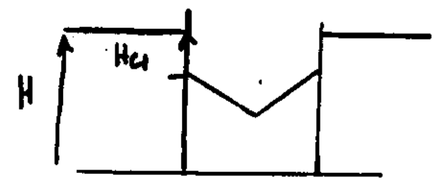
$H > H_{c1}$ flux lines penetrate from surfaces

∴ A gradient in flux is set up with

$$\vec{\nabla} \times H = j_s = \frac{dH}{dx} \quad \text{in 1-D.}$$

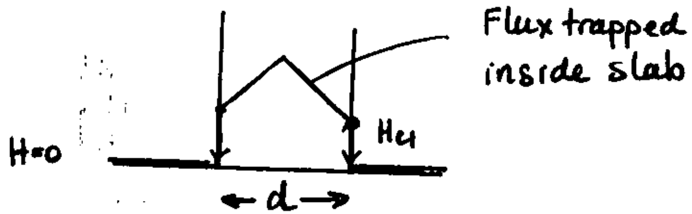


$H >> H_c$



Note, if H is now decreased there will be no change in φ inside until H changes by $2H_{c1}$.

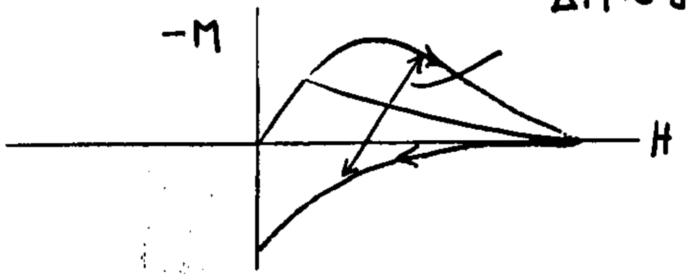
$H = 0$



Amount of flux trapped depends on d & J_c ∝ $J_c d$

For powder alone or single-xtal

$\Delta M \sim J_c\, d$



J_c within grains $\sim \frac{\Delta M}{d}$ — measured

d ← grain size measured

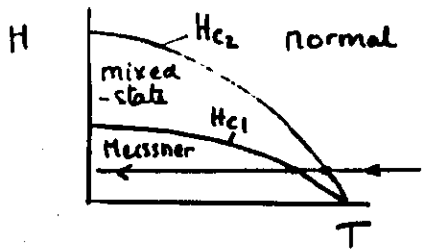
$\therefore$ J_c estimated.

Also enables J_c to be determined in a single-crystal

advantage — no contacts

* Note, initial diamagnetism is not 100% as grains do not occupy whole space.
* Cooling a grain or single-crystal in a magnetic field will tend to TRAP flux — do not expect 100% flux expulsion if PINNING exists — it always will!

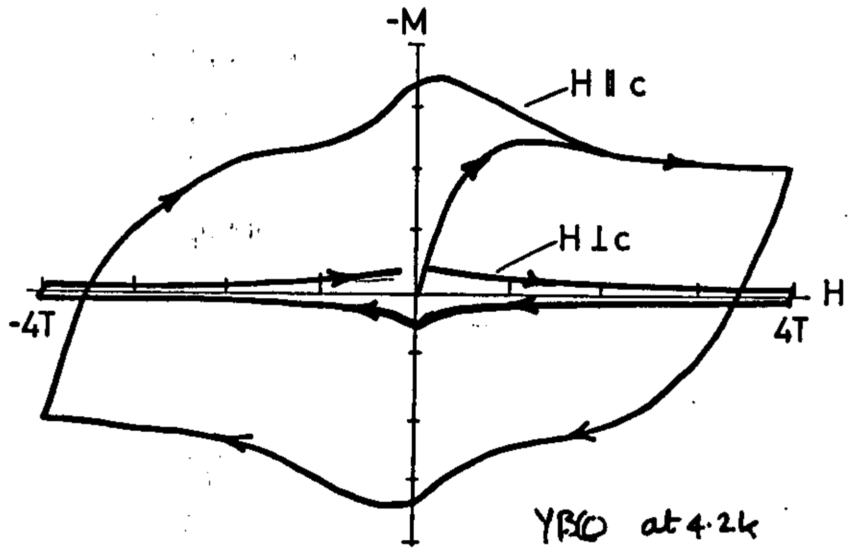
Cooling in external field



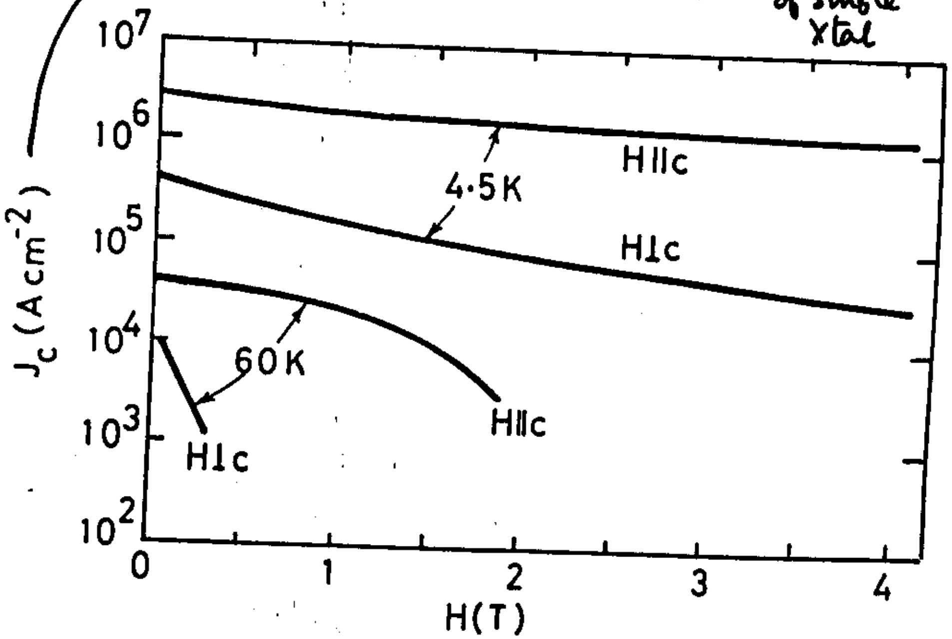
Cooling in finite H ext — pass through H_{c2} + H_{c1} — traps flux. $\therefore$ NOT complete MEISSNER effect.

SINGLE CRYSTAL

ANISOTROPY OF J_c FROM MAGNETIC MEASURES.

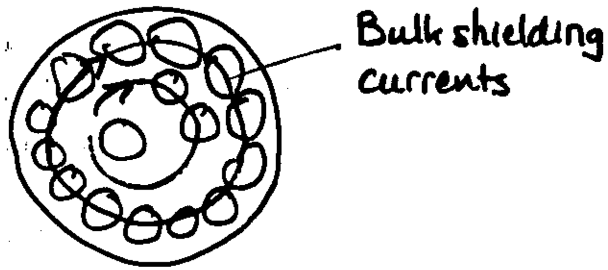


deduced from hysteresis $\Delta M \sim J_c \times d$, of single xtal



Note that a compacted powder specimen with no electrical conductivity between grains will "float" – it is strongly diamagnetic but electrically insulating.
"Floating" does <u>not</u> imply electrical superconductivity necessarily.

Sintered sample with electrical connectivity



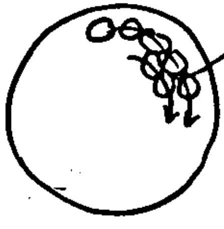
Two effects
1. Diamagnetism of individual grains
2. Diamagnetism from bulk shieldin supercurrents

1. VERY SMALL FIELDS ~ ≲ 1 gauss

   Currents ~ mA flow between outer grains to shield interior from external field. Complete MEISSNER effect.

2. H exceeding very small value to support i_c

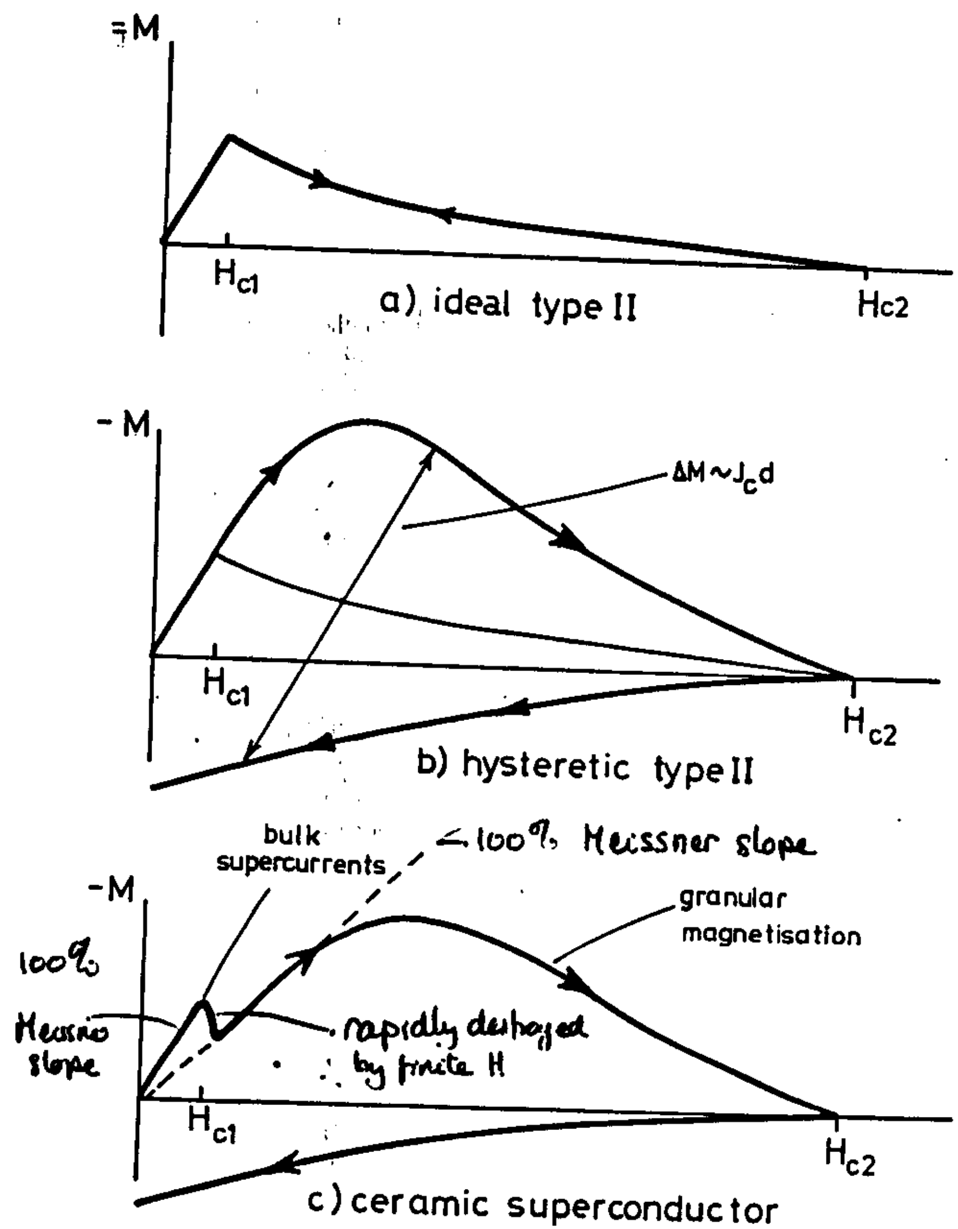
   Field starts to penetrate – determined by J_c flowing through bulk material



field penetrates between grains

$$\frac{dH}{dr} \sim J_c \text{ intergrain}$$

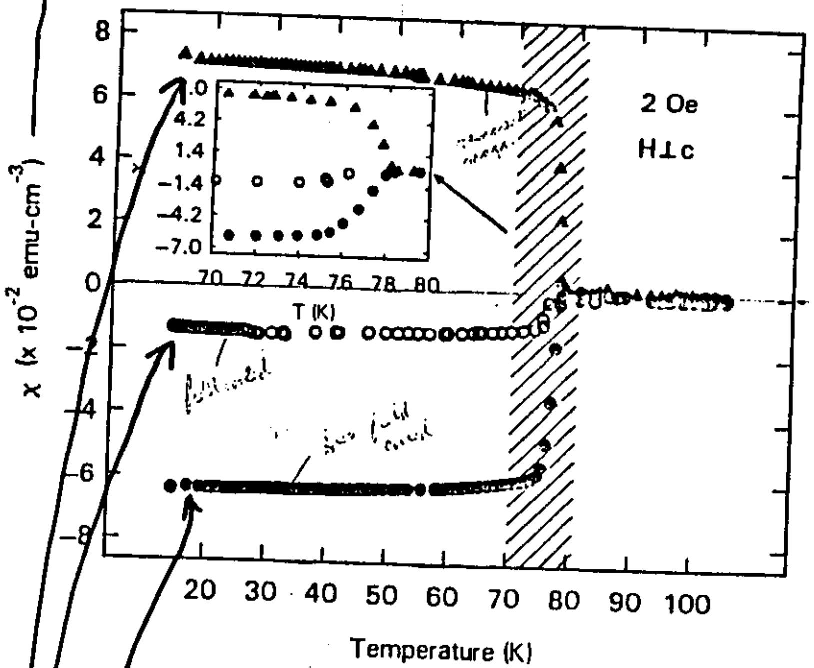
SUMMARY



Magnetisation at 77k

IBM SINGLE XTAL DATA

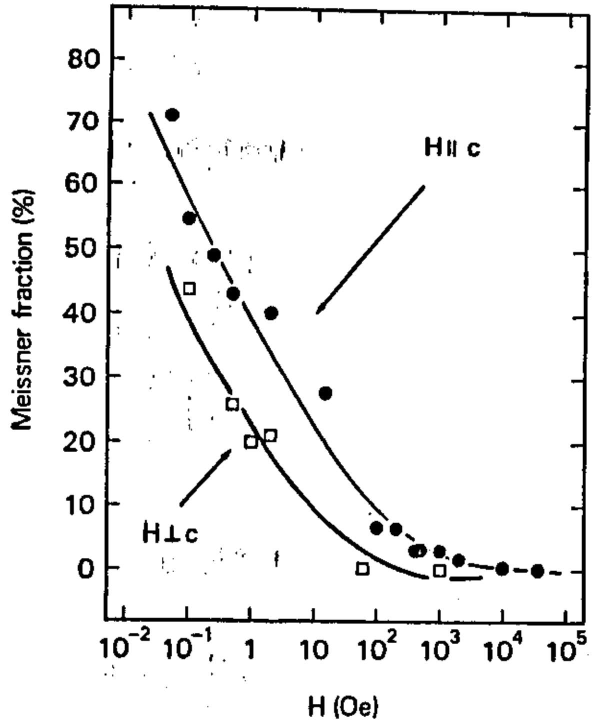
→ = ΔM/H



1. Cool in O-field, apply 2 Oe measure flux expulsion → ΔM
Repeat cool from > T_c
2. Cool in 2 Oe, measure flux expulsion – note NOT 100% of method 1 – only fractional expulsion
3. Remove 2 Oe field, measure trapped flux → +ΔM trapped

SINGLE-CRYSTAL – TRAPPED FLUX ON COOLING

Krusin-Elbaum, Malozemoff & Yeshurun, IBM (Dec '87)



↑ Cooling field

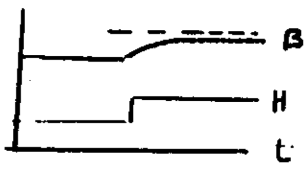
NOTE LESS THAN 100%

∴ EXPECT SIMILAR RESULTS FOR POWDER GRAINS

Fig 3

Magnetisation complicated by flux CREEP – it takes a long time to approach equilibrium state

$\Delta M \propto \log t$ Anderson thermal activation model



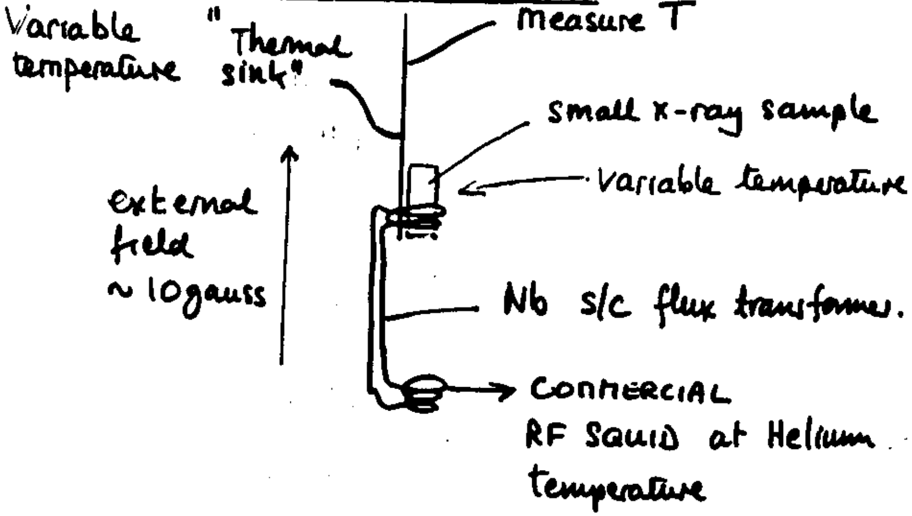
+ ALSO SUPERCONDUCTING GLASS MODELS NOT WELL UNDERSTOOD YET

VIBRATION OR SQUID magnetometers to cover full field range, say, 0 – 12T, are very expensive. > 10^5 \$

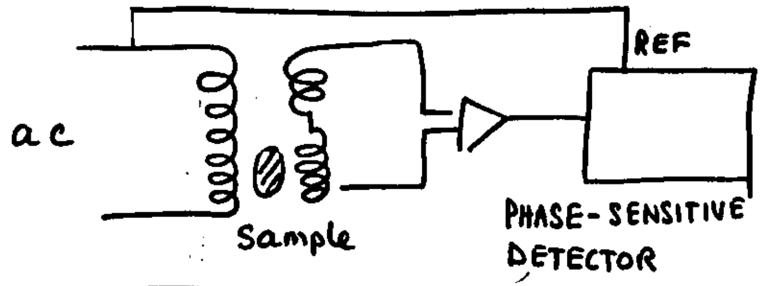
But measurement of flux ~~ex~~ expulsion on small samples is vitally important, if s/c properties of small samples for x-ray determination are to be confirmed.

A simple commercial SQUID with a magnetic field of ~ 10 gauss or so would be sufficient. $\lesssim 10^4$ \$

CONSTRUCT YOUR OWN CRYOSTAT



Susceptibility measurements



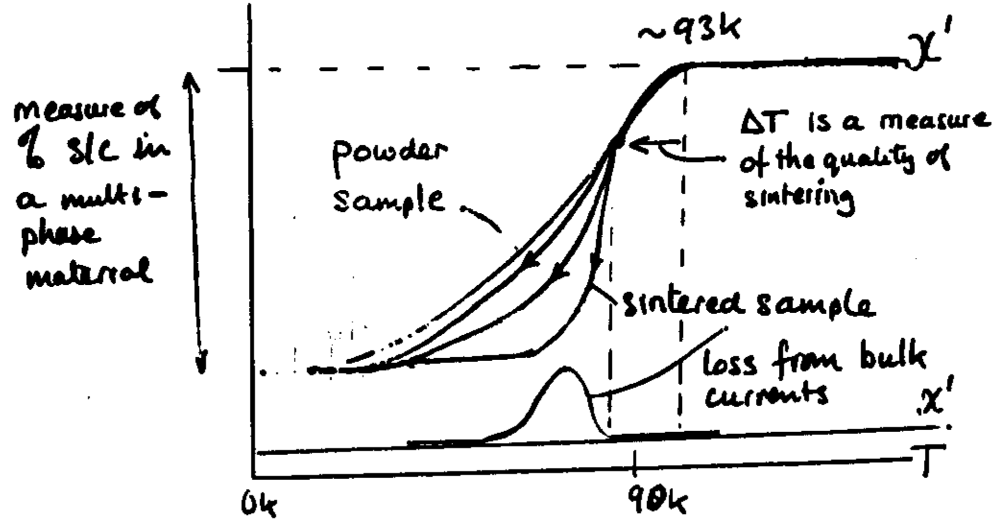
Real + Imaginary components of χ

$\chi' + i\chi''$

χ': magnetic properties.

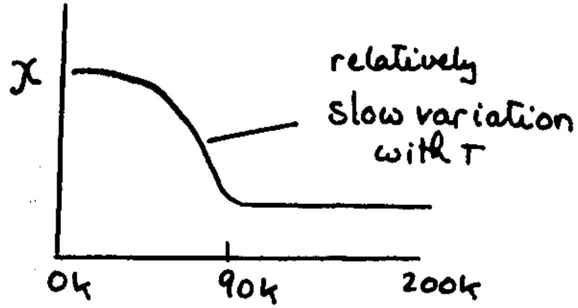
χ'': loss from magnetic hysteresis

Typical measuring ac field 1mgauss – 10gauss



PROBLEM

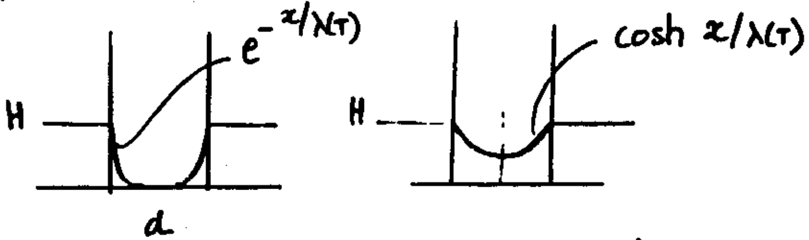
* How can we account for temperature dependence of χ for powders?



Is it due to temperature dependence of magnetic penetration depth in grains

$$\lambda = \lambda(0) / \left(1 - \frac{T}{T_c}\right)^{1/2}$$

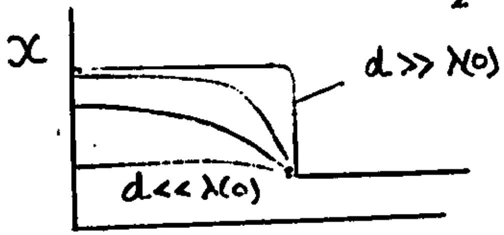
If grain size, d, comparable to $\lambda(0)$, would expect a temperature dependent Meissner effect.



T = 0, near complete Meissner effect

$T \to T_c$ λ increases

INCOMPLETE MEISSNER EFFECT $\sim \frac{1}{2}$ when $\lambda(T) \sim d$



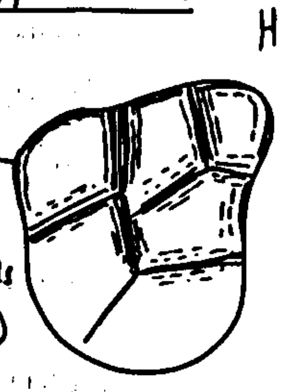
Would give a measure of $\lambda(0)$ from known d

Powder measurements would give $\lambda(0) \sim 1 - 10\,\mu$ BUT WRONG

$\lambda(T)$ estimated from powder χ assuming $\chi(T)$ determined by partial magnetic penetration when $d \sim \lambda(T)$ gives

1. Wrong temperature dependence
2. A value of $\lambda(T) \gg$ measured from other methods ~ 1000 Å (but anisotropic)

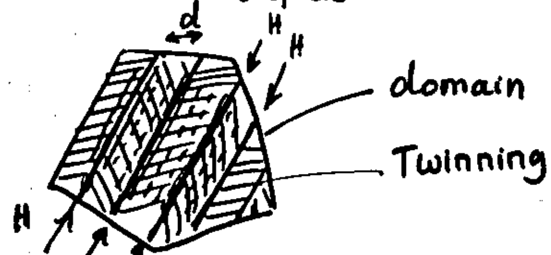
Possible explanations



Flux lines enter along domain walls

∴ Effective $d \sim \lambda(0)$ is domain size NOT size of X-tal

AND / OR within a domain flux enters along twin boundaries or other defects



Flux enters along twin boundaries ∴ $d \sim 10^3$ Å $\sim \lambda(0)$

Distribution of twin boundary spacing → observed T dependence