Magnetic measurement instruments and techniques

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Acknowledgements

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Outline

- M – H loops
  - Hysteresis loop and reversal
  - Remenance curves
  - Irreversible susceptibility
  - Thermal activation
  - Time dependence
- Open vs Closed circuit measurements
- Magnetic measurements in open circuits
- Magnetometry – dc techniques
  - Flux measurement from a single turn coil (Fluxometer)
  - Vibrating sample magnetometry (VSM)
  - Superconducting Quantum Interference Device (SQUID) magnetometry
  - SQUID VSM
  - Issues with VSM and SQUID measurements
- Magnetometry – ac techniques
  - a.c. susceptibility
- Magnetometry – high frequency techniques
  - Ferromagnetic resonance
- Magnetometry – advanced techniques
  - X-ray absorption spectroscopy and XMCD
  - XFMR

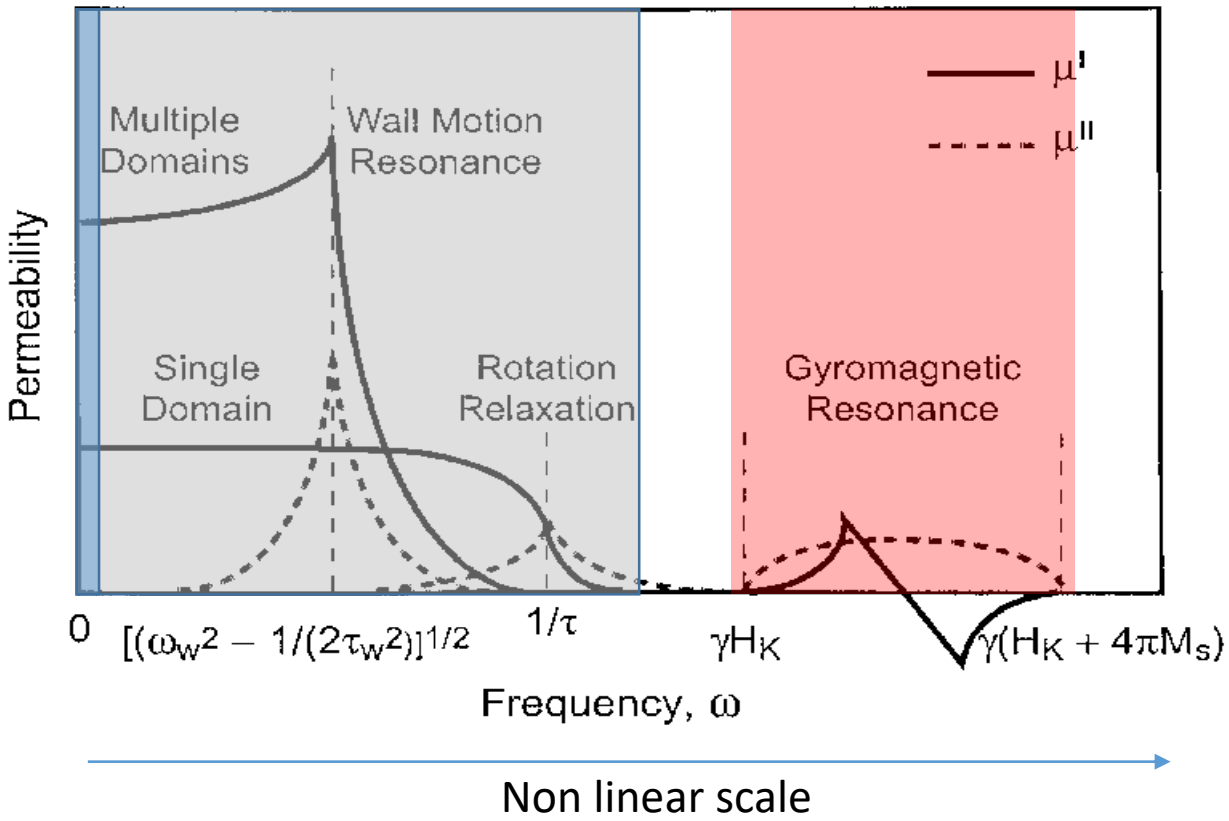
Magnetic measurement techniques

Highly dependent on the timescale of the magnetization dynamics you are interested in

dc techniques
VSM, SQUID, Kerr

ac techniques
ac susceptometry

high frequency techniques
FMR, TR-Kerr



$\mu_r = \chi + 1$

dc techniques

Hysteresis loop and reversal

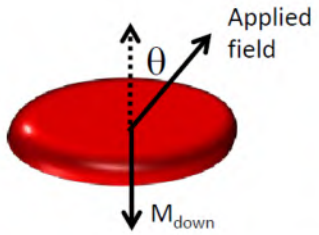
- The hysteresis loop is the most common magnetic measurement
  Apply magnetic field (H) along one axis, measure component of magnetization (M) along axis of applied field.

- It results from irreversible changes in the magnetic order due to a combination of fundamental and microstructural properties

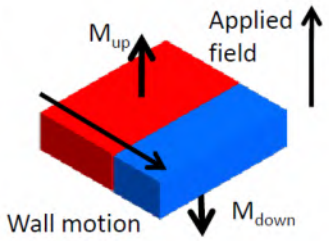
- Common parameters obtained
  - M_s – saturation magnetization (emu/cc)
  - M_r – remanent magnetization (emu/cc)
  - H_c – coercive field (Oe) ….. mean switching field
  - H_s – saturation field (Oe)

- Reversal occurs by coherent rotation, domain wall motion or a combination of both. Reversible and irreversible components present.

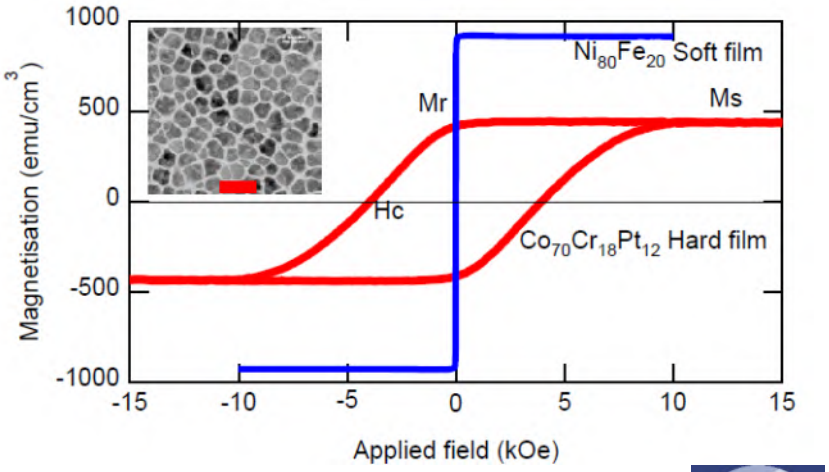
$$M_{tot} = M_{irrev} + M_{rev}$$



Coherent rotation



Domain Wall motion



Hysteresis loop and reversal

Hysteresis loop in detail

The magnetization curves and hysteresis loops of real materials have quite variable shapes.

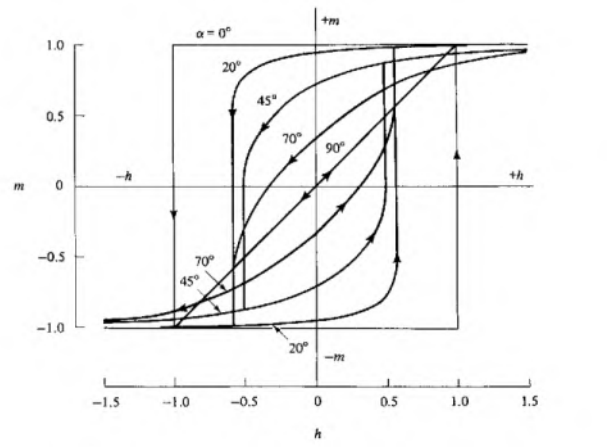
Only in three circumstances, however, do we have algebraic expressions to fit the observed curves:

1. High-field magnetization curves of single crystals

2. High-field magnetization curves of polycrystalline specimens. In both (1) and (2) the magnetization change is by domain rotation

3. Low-field magnetization curves (Rayleigh region) and hysteresis loops of polycrystalline specimens

4. Single Domain particles



E. C. Stoner and E. P. Wohlfarth, Phil. Trans. Roy. Soc., A240 (1948)

Hysteresis loop and reversal

Low-field magnetization curves (Rayleigh region)

$$\mu = \mu_i + \upsilon H$$

μ_i = initial permeability, υ Rayleigh constant

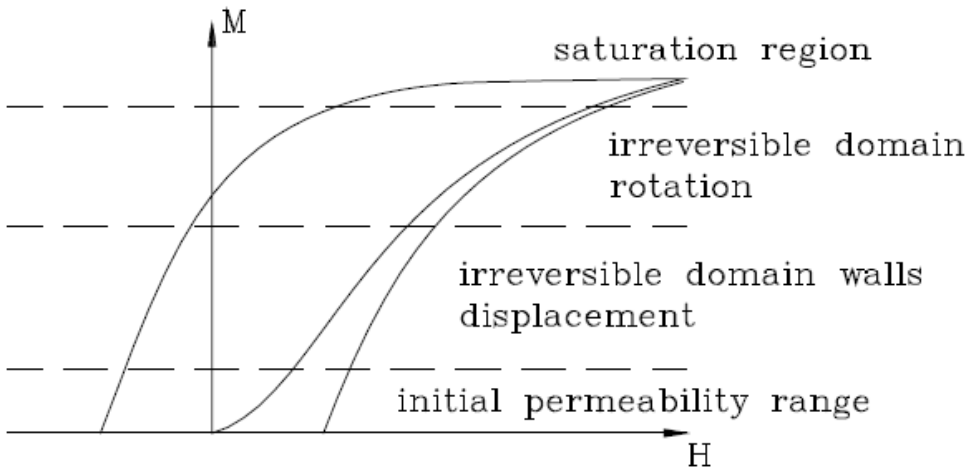
$$\mu H = \mu_i H + \upsilon H^2 = B$$

$$B = H + 4\pi M$$

$$H + 4\pi M = \mu_i H + \upsilon H^2$$

$$(\mu_i - 1)H = 4\pi M - \upsilon H^2$$

$$M = \chi_i H + \left(\frac{\upsilon}{4\pi}\right) H^2$$



The term linear in H represents the reversible part, and that quadratic in h the irreversible part

Hysteresis loop and reversal

Between the low-field Raylegh region and the high-field region near saturation there exists a large section of the magnetization curve, comprising most of the change of magnetization between zero and saturation. The main processes occurring here are large Barkhausen jumps, and the shape of this portion of the magnetization curve varies widely from one kind of specimen to another. It is not possible to express M as a simple function of H in this intermediate region.

At high fields we can use the "Law of approach"

$$M = M_s\left(1 - \frac{a}{H} - \frac{b}{H^2}\right) + \chi H$$

a – due to inclusions / microstress
b – magnetocrystal anisotropy
χH – term representing forced magnetization

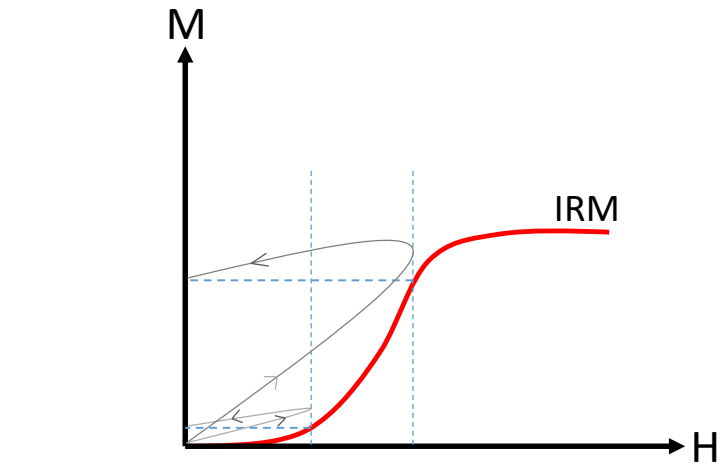
Several difficulties with this equation

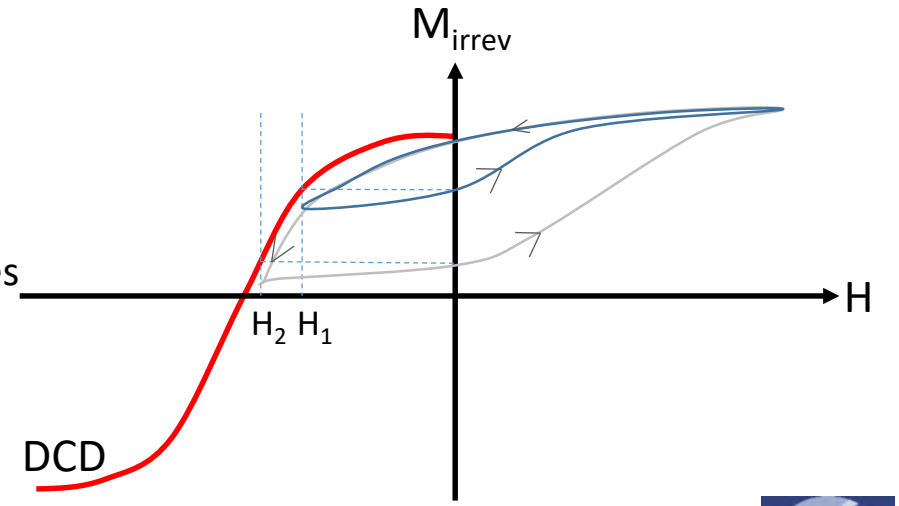
Remanence curves

- We can measure M_{irrev} from remanence curves
- The isothermal remanence curve (IRM) is measured from the demagnetised state
- DC demagnetised curves (DCD) are measured from the saturated state
- The Wohlfarth relation (in the absence of interactions) gives

$$M_{DCD}(H) = 1 - 2M_{IRM}(H)$$

- The deviation from the Wohlfarth relation indicates the type and strength of the interactions in granular systems



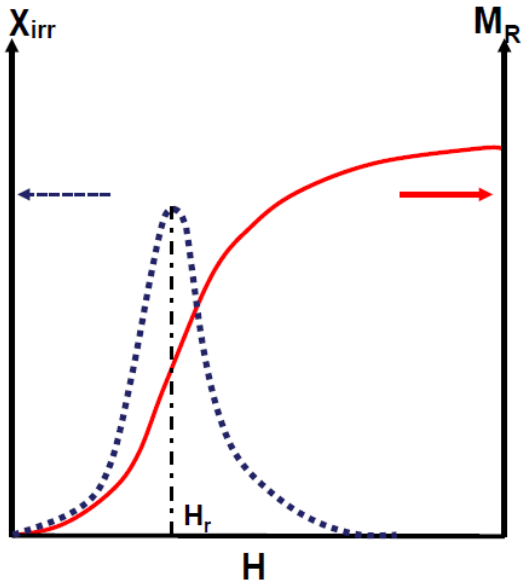


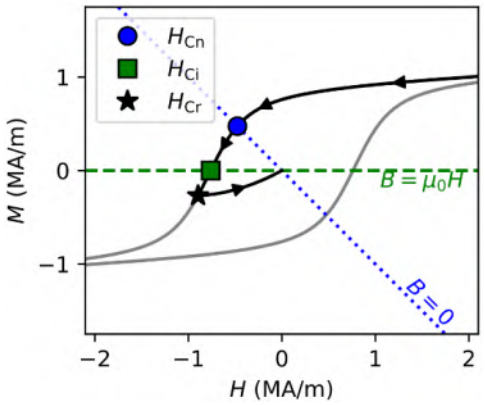
Irreversible Susceptibility

- The differential of a remanence curve (∂_H) is a measure of the irreversible susceptibility $\chi_{irrev}(H)$ which, when normalised, provides the switching field distribution.
- It provides the domain wall pinning strength distribution in continuous materials.
- It also allows a measure of M_{rev}

$$M_{rev} = M_{tot} - M_{irrev}$$

- The peak in the distribution defines the remanent coercivity (H_r) which is the mean switching field.
- The *intrinsic coercivity*, H_{Ci}, is the *H* field required to reduce the magnetization (average *M* field inside the material) to zero.
- The *remanent coercivity*, H_r, is the *H* field required to reduce the remanence to zero, meaning that when the *H* field is finally returned to zero, then both *B* and *M* also fall to zero (the material reaches the origin in the hysteresis curve).





Thermal Activation

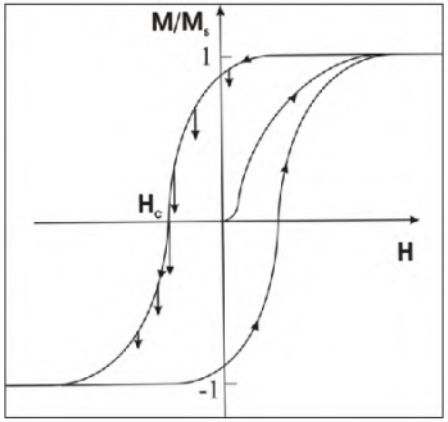
**** even more complicated as we will see

- Thermal properties affects all measurements at finite temperatures.

- Probability of switching

$$P = \exp(-t/\tau), \qquad \tau^{-1} = f_0 \exp\left(-\frac{\Delta E}{k_B T}\right)$$

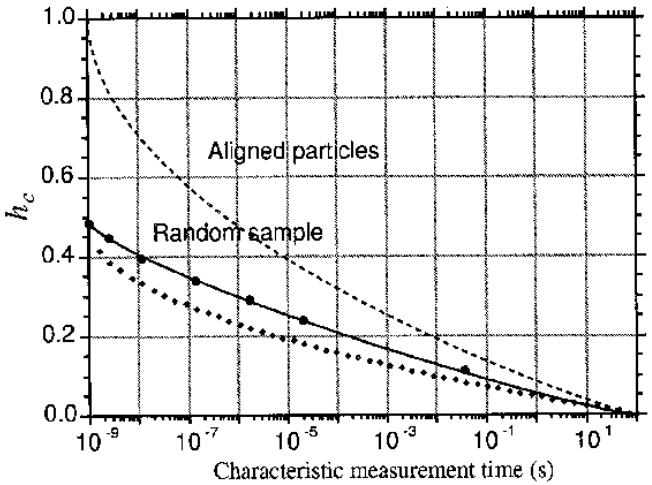
Where f_0 is an attempt frequency (~1x10^9 s^{-1}) and ΔE is an energy barrier that is due to anisotropy, domain wall pinning or nucleation.



- The 100 s criteria (P= 50%) gives ΔE = 25 $k_B T$!

- The consequence of this is that most measurements of hysteresis are time or frequency (R = dH/dt) dependent.

$$H_c(R) = H_K\left[1 - \sqrt{\frac{k_B T}{KV} ln\left(\frac{f_0 H_K k_B T}{KV\left(1 - {}^{H_c}/_{H_K}\right)}\frac{1}{R}\right)}\right]$$



Be careful when making comparisons between a series of films that the measurement conditions are the same (or check t dependence)!!!

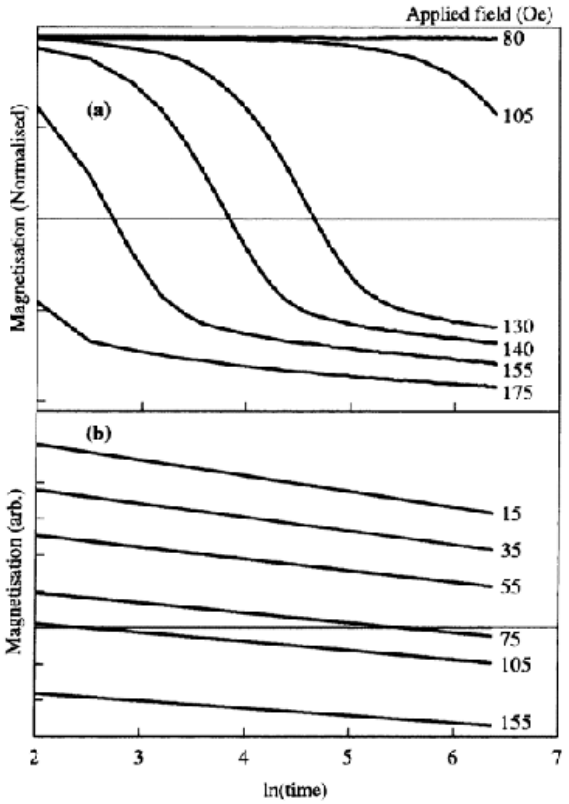
Time dependence

- For a single energy barrier the variation of M with time will be exponential but for a distribution f(ΔE) the summation gives:

$$M(t) = M(0) \pm S(H)\ln(t)$$

$$S(H) = \frac{dM(H)}{dln(H,t)}$$

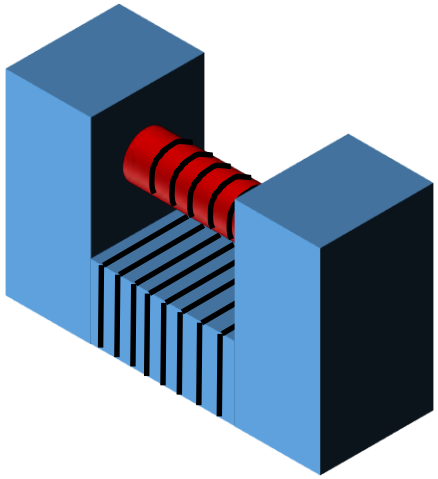
- S is field dependent with a maximum near the coercivity.
- For storage applications S needs to be zero at H = H_d



Time dependence of magnetization (Tb = 19:8 A° , Fe = 8:5 A°) multilayers: (a) 6 bilayer film, (b) 14 bilayer film.

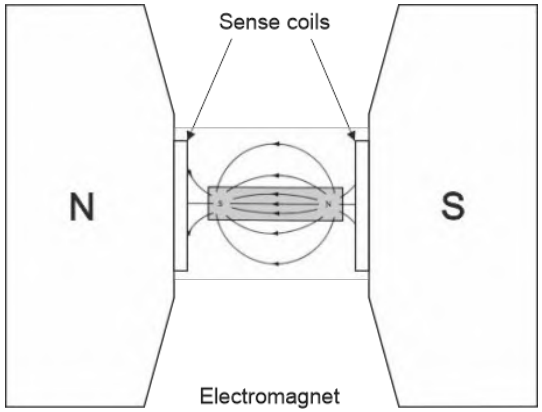
Open vs Closed circuit measurements

- Measurements on bulk magnetic materials can be made in a closed circuit using permeameters. When the flux path lies entirely within the magnetic material the circuit is said to be closed. No poles are produced. Measures B (or dB/dt) not M.



- Not possible for particulate or thin film geometries / samples which need to be measured in open circuit. However, this forms free poles on the sample surface which create a demagnetising field (H_D).

- Open circuit systems cannot measure B (or M) directly so a calibration must be made.



Some issues seen in the literature!

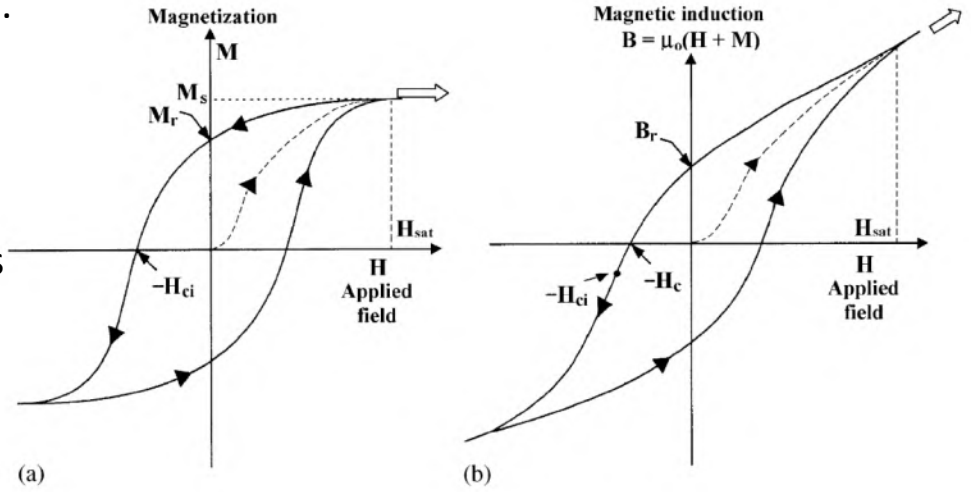
1. Definition of coercivity **** magnetization taken to be zero at H_{ci} on the B vs H curve rather than on the M – H curve. i.e. $H_{ci} \equiv H_c$. For soft materials $B \approx \mu_0 H$ so $H_{ci} \approx H_c$

2. Term "saturation induction" is sometimes used. However, no saturation of the magnetic induction can ever occur.

3. The shape of the hysteresis loop **depends** on the direction of the applied field if anisotropies are present. However the sample must always have the same saturation magnetization (M_s) independent of measurement direction. H_s may be direction dependent.

4. M-H loops are often plotted normalised to M_s without M_s being given (reader to assume bulk value!)

5. Whilst H can be measured quite accurately using a Hall probe, M is a little more tricky and therefore so is the true field with it.



**** even more complicated as we will see

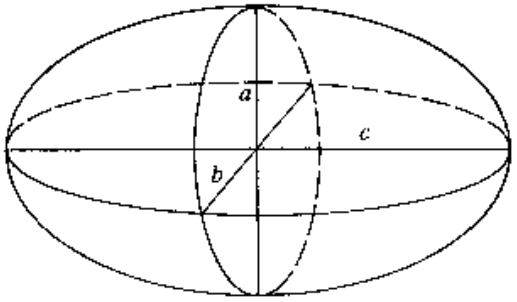
Magnetic measurements in open circuits

dc magnetic measurements (M-H loops) are typically made using a VSM, SQUID or VSM-SQUID. The total magnetic moment (μ) of the sample/ substrate + sample holder is measured as a function of applied field (H_a). We wish to plot M vs H_{tr} where

$$M = \frac{\mu_{sample}}{V_{sample}}, \qquad H_{tr} = H_a - N_d M$$

and N_d is the demagnetizing factor.

Only ellipsoid samples have a uniform H_d throughout the volume. Unless the sample is a ellipsoid there is no single demagnetizing factor that applies for all parts of the sample at all levels of magnetization.
Approximate demagnetizing factors for various samples can be found in the literature i.e. R. M. Bozorth, Ferromagnetism



Choose your sample geometry carefully!

Magnetic measurements in open circuits

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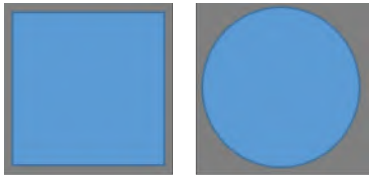
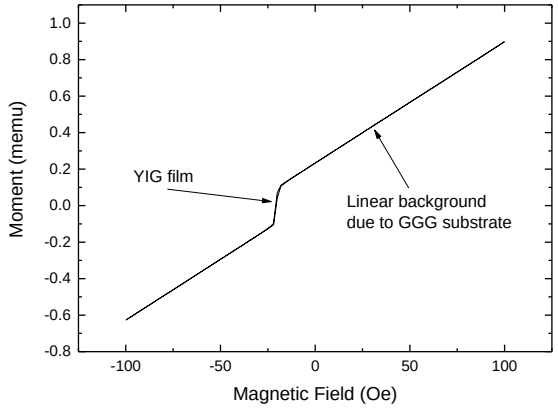
- For thin film samples, circular samples are better (no sharp corners where domains walls are easily nucleated)

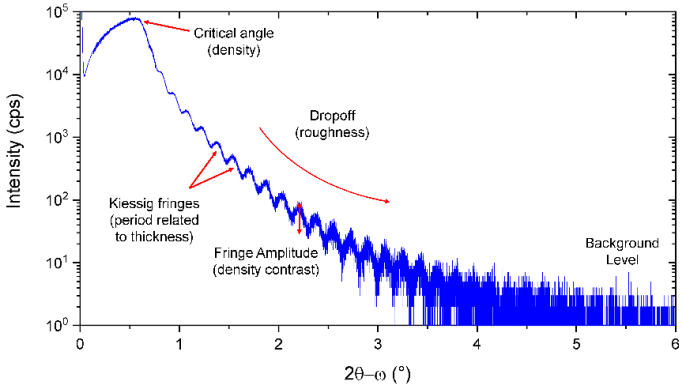


- Substrates should ideally be non-magnetic (diamagnetic) – not always possible i.e. YIG/GGG. Diamagnetic no T dependence, Curie paramagnet (1/T dependence).

- Substrate / sample holder M vs H needs to be subtracted. Often this is requires the subtraction of a linear gradient measured at H >> H_s. Check by measuring substrate before thin film deposition. Often you find surprising results!



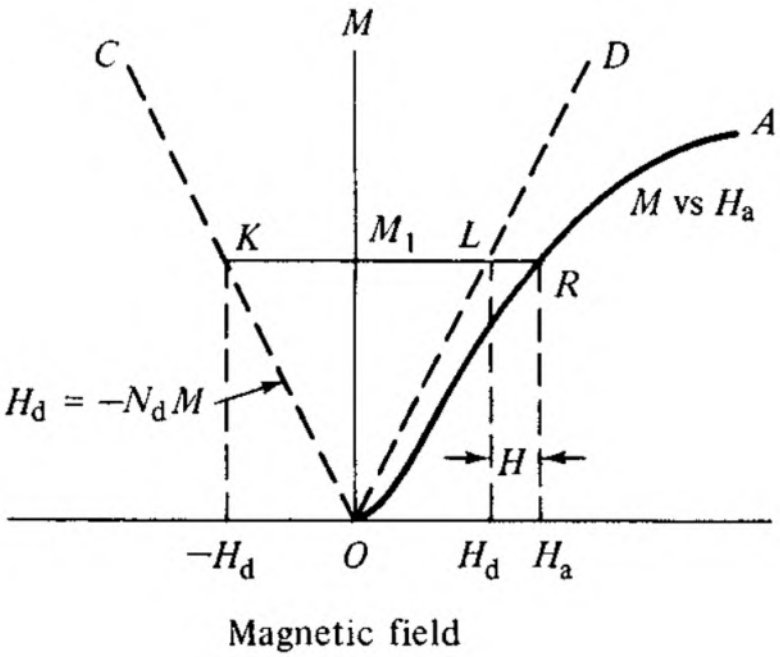
- **The problem of volume**. To obtain M (emu/cc), the volume of the (magnetic) sample is required. For thin films this can lead to significant errors in M via the error in V. Area can be obtained using a calibrated microscope. Thickness often requires XRR and the assumption that the thickness is uniform across the whole sample. Relative error in thickness is significant for ultrathin films (≈ nm). Factors into the true field (although not an issue for soft magnets).

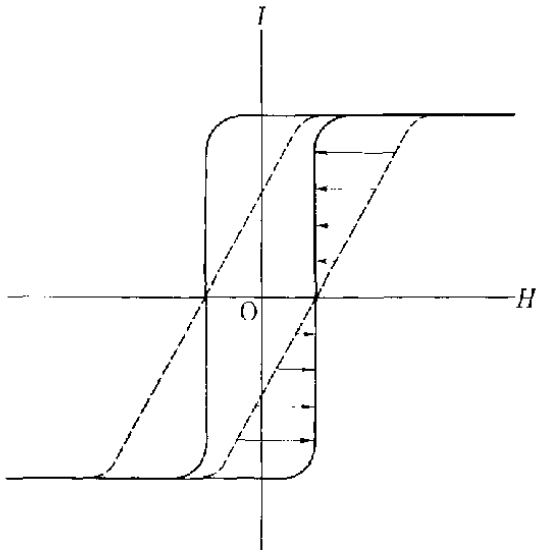


Magnetic measurements in open circuits

Shearing Correction

$$H_{tr} = H_a - N_d M$$





As a result of this correction, the magnetization curve takes on a more upright form, with increased susceptibility and remanence. Unless otherwise stated, published magnetization curves **should** have been corrected for demagnetizing effects and therefore represent the properties of the material independently of the shape of the sample used for the test.

Magnetometry

Flux measurement

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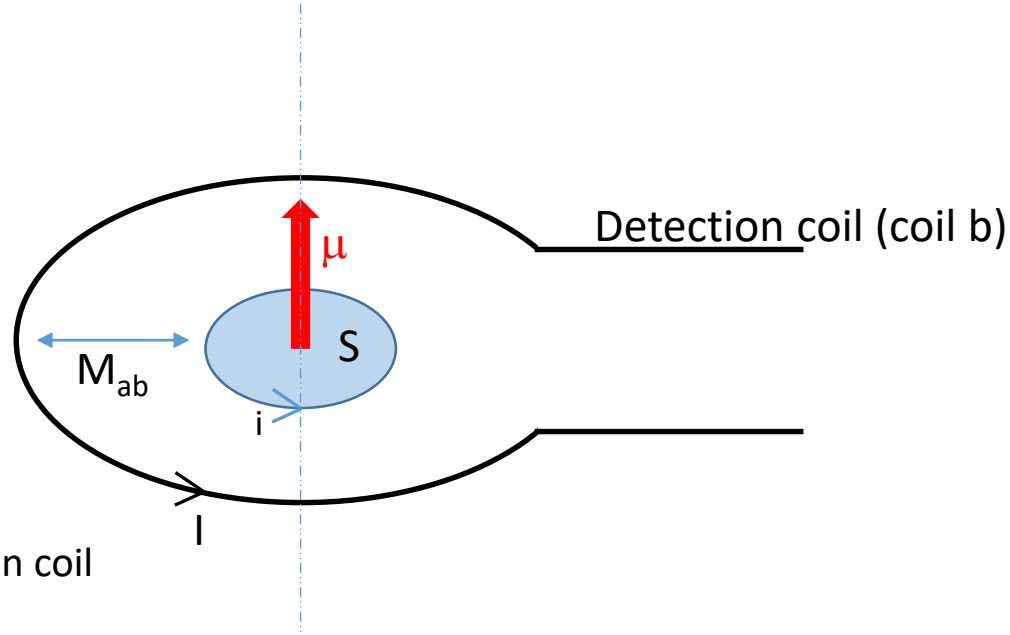
What is the flux (Φ_b) that passes through the detection coil due to a magnetic moment?

Mutual inductance between two coils

$$M_{12} = \frac{N_2 \phi}{I_1}$$

$$\Phi_b = M_{ab} i = \frac{M_{ab} \mu}{S}$$



Flux due to fictitious current (I) in detection coil

$\Phi_a = M_{ab} I = B.S$

$M_{ab} = B.S/I$

$$\Phi_b = M_{ab} i = \frac{B \mu}{I}$$

B/I can be obtained from the Biot-Savart law

Magnetometry

Flux measurement

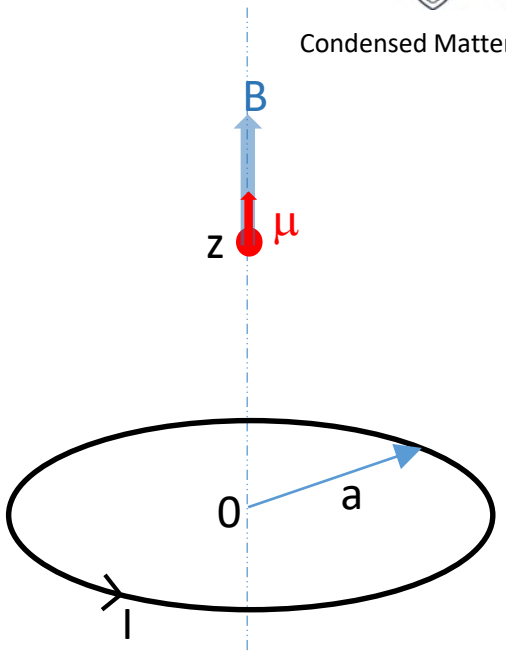
Biot – Savart law:

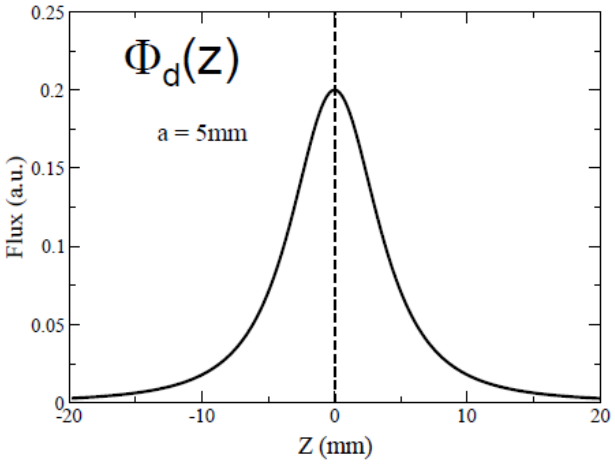
Field created at a distance z above the plane of a coil

$$B(z) = \frac{\mu_0 I}{2} \frac{a^2}{(a^2 + z^2)^{3/2}}$$

$$\Phi_d(z) = \frac{B(z)\mu}{I} = \frac{\mu_0}{2} \frac{a^2}{(a^2 + z^2)^{3/2}} \mu$$

$$\Phi(z) = G(z)\mu$$





Can measure flux directly – SQUID

or……

Can make use of Lenz's law and measure the induced voltage from the change in flux, V = - dΦ/dt – need to vary z or μ with time.

Magnetometry

Flux measurement

Vibrating sample magnetometer – z(t)

$$V(t) = -\frac{d\phi}{dt} = -\frac{d\phi}{dz}\frac{dz}{dt} = -\frac{dG}{dz}\mu\frac{dz}{dt}$$

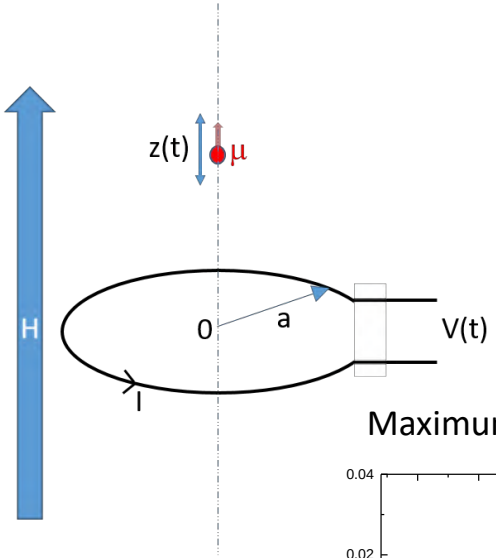
$$z(t) = Z + Ae^{-i\omega t}$$

$$\frac{dz}{dt} = -i\omega Ae^{-i\omega t}$$

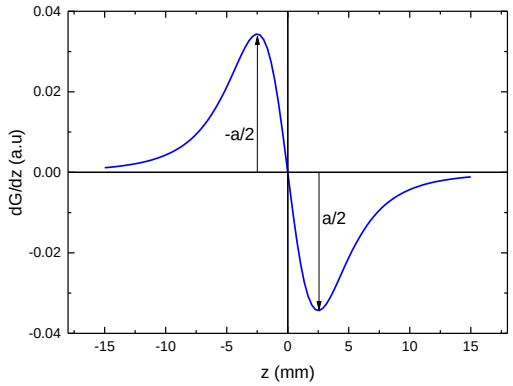
$$\frac{dG}{dz} = -\frac{3\mu_0 a^2}{2}\frac{z}{(a^2+z^2)^{5/2}}$$

$$V(t) = -\boldsymbol{\mu}\frac{i3\mu_0 a^2 z\boldsymbol{\omega A}e^{-i\omega t}}{2(a^2+z^2)^{5/2}}$$

For A ≪ Z



Maximum sensitivity at z = a/2



However – maximum in the geometric response is very sharp, centering must be precise and slow drift of the applied field is not cancelled.

Solution: multicoil configurations

1st order gradiometer: 2 coils (VSM)

2nd order gradiometer: 4 coils (SQUID)

Magnetometry

Flux measurement

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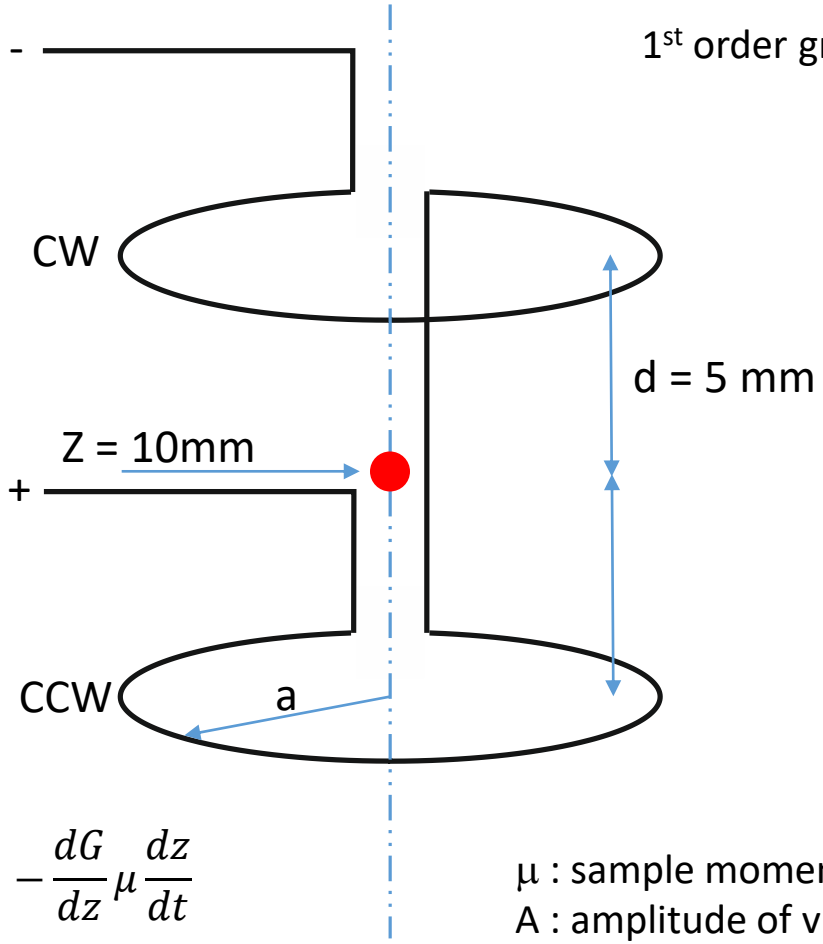
Vibrating sample magnetometer – z(t)



1st order gradiometer

A = 2 mm

$\omega/2\pi$ = 40 Hz

$$V_{coil} = -\frac{d\phi}{dt} = -\frac{d\phi}{dz}\frac{dz}{dt} = -\frac{dG}{dz}\mu\frac{dz}{dt}$$

μ : sample moment (emu)

A : amplitude of vibration

$$\frac{d\phi}{dz} = \frac{d(\phi(z-(Z+d)) - \phi(z-(Z-d)))}{dz}$$

Magnetometry

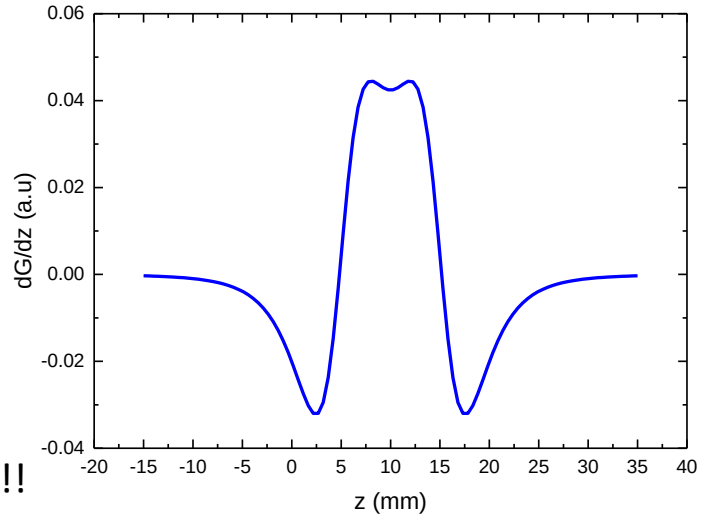
Flux measurement

Vibrating sample magnetometer – z(t)

Need to saddle (centre) the sample in the detection coils

In z-direction

$$\frac{dG}{dz} = -\frac{3\mu_0 a^2}{2}\left[\frac{z-(Z+d)}{(a^2+(z-(Z+d))^2)^{5/2}} - \frac{z-(Z-d)}{(a^2+(z-(Z-d))^2)^{5/2}}\right]$$



However, sample needs to be saddled in x and y directions as well!!

Magnetometry

Flux measurement

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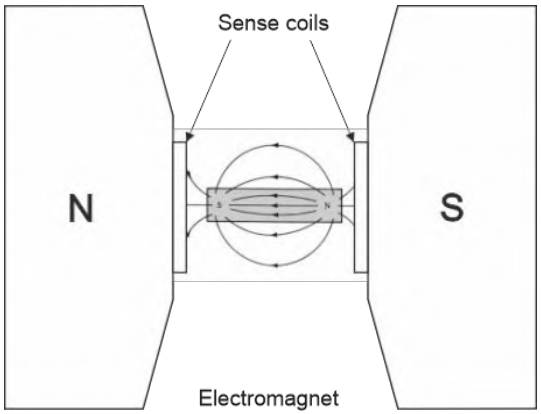
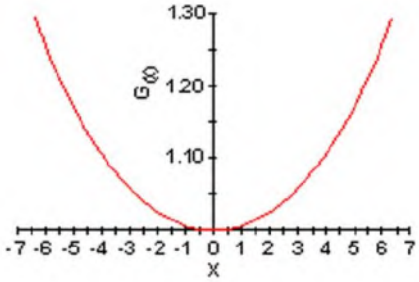
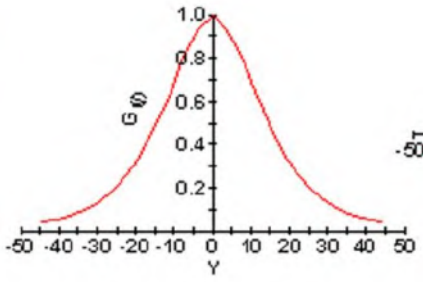
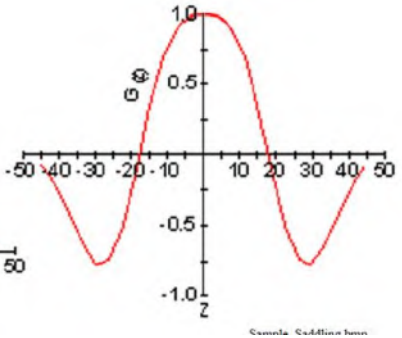
Vibrating sample magnetometer – z(t)

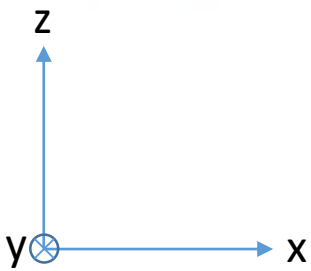
Sample Saddling

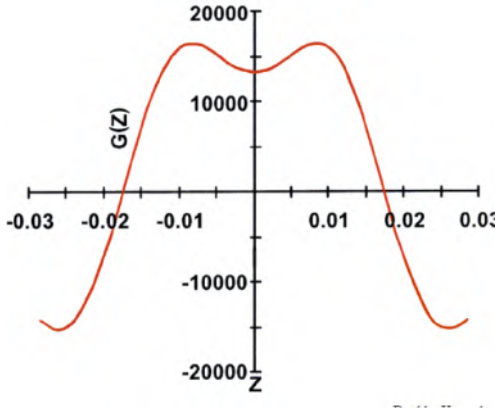












From Lakeshore 8600 series user manual……

Sample must be centred in the detection coils and on axis of rotation if angular studies are desired (or saddle required after each rotation)!

DSP Lock-in detection

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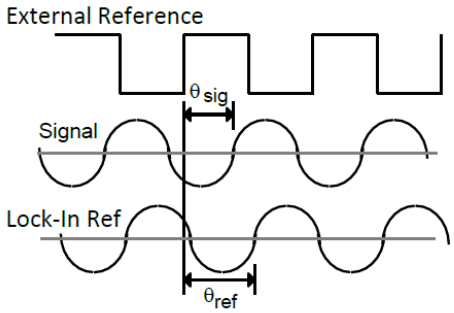
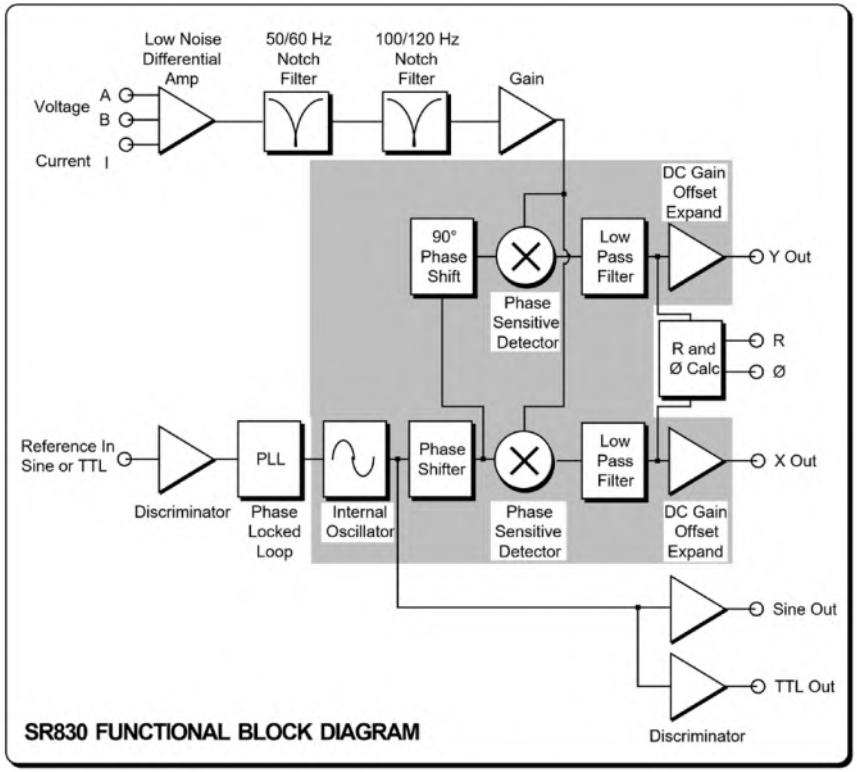
$$V(t) = -\frac{d\phi}{dt} = -\frac{d\phi}{dz}\frac{dz}{dt} = -\frac{dG}{dz}\mu\frac{dz}{dt}$$

$$V(t) \propto \boldsymbol{\mu} e^{-i\omega t}$$

Measure signal only at frequency ω





$$V_{psd} = V_{sig}\left([\sin(\omega_r t + \theta_{sig})] \times [\sin(\omega_L t + \theta_{ref})]\right)$$

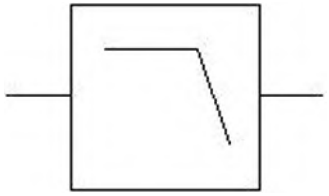
$$= 1/2\, V_{sig} \cos([\omega_r - \omega_L]t + [\theta_{sig} - \theta_{ref}]) - 1/2\, V_{sig} \cos([\omega_r + \omega_L]t + [\theta_{sig} + \theta_{ref}])$$

difference sum

DSP Lock-in detection

$V_{psd} = V_{sig}\,([\sin(\omega_r t + \theta_{sig})] \times [\sin(\omega_L t + \theta_{ref})])$

$= 1/2\ V_{sig} \cos([\omega_r - \omega_L]t + [\theta_{sig} - \theta_{ref}]) - 1/2\ V_{sig} \cos([\omega_r + \omega_L]t + [\theta_{sig} + \theta_{ref}])$



$$\rightarrow \quad \frac{1}{2}V_{sig}cos\left[(\omega_r - \omega_L)t + \left[\theta_{sig} - \theta_{ref}\right]\right]$$

If $\omega_r = \omega_L$ $\qquad V_{psd} = V_{sig}cos(\theta_{sig} - \theta_{ref})$

A dc signal proportional to the signal amplitude. The PSD and low pass filter only detect signals very close to the reference frequency – locks in to the signal at frequency ω.

DSP Lock-in detection

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Fast signals

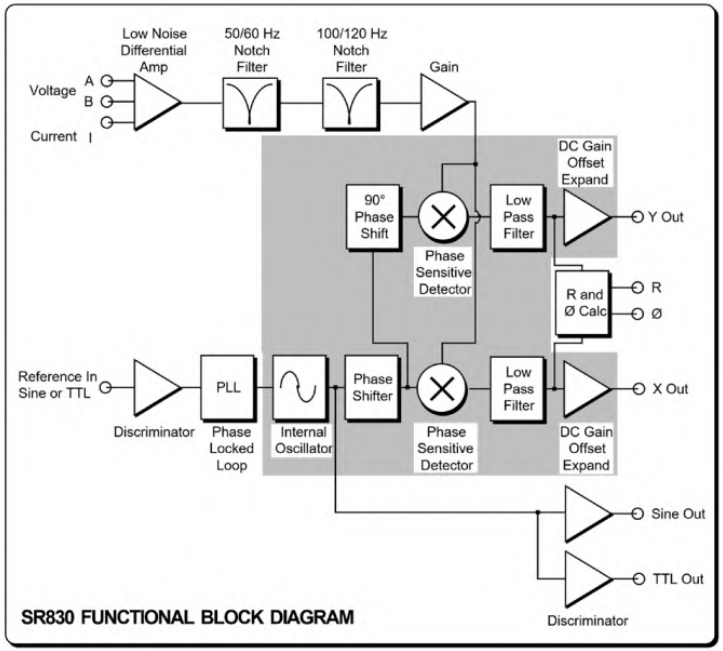
$X=V_{sig}\cos(\theta)$

$Y=V_{sig}\sin(\theta)$

Computed signals

$R = (X^2 + Y^2)^{1/2}$

$\theta = \text{atan}(Y/X)$



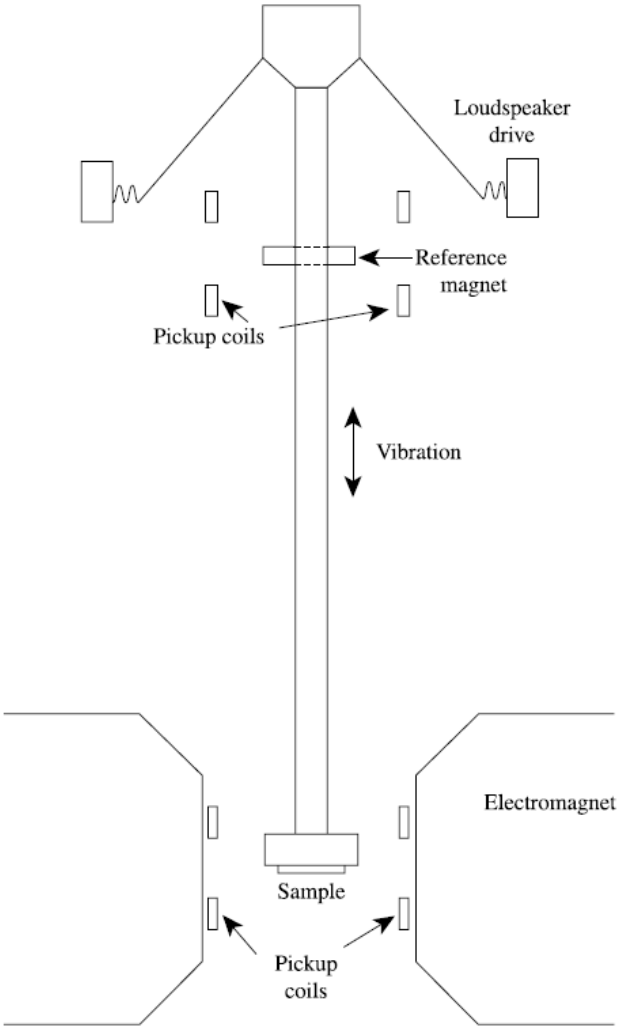
The phase sensitive detectors (PSD's) in modern lockins act as linear multipliers, that is, they multiply the signal with a reference sine wave. Analog PSD's (both square wave and linear) have many problems associated with harmonic rejection, output offsets, limited dynamic reserve and gain error. A digital PSD multiplies the digitized signal with a digitally computed reference sine wave. Because the reference sine waves are computed to 20 bits of accuracy, they have very low harmonic content. Output offset is a problem because the signal of interest is a DC output from the PSD and an output offset contributes to error and zero drift. The offset problems of analog PSD's are eliminated using the digital multiplier. There are no erroneous DC output offsets from the digital multiplication of the signal and reference. In fact, the actual multiplication is totally free from errors.

Magnetometry

Flux measurement

Vibrating sample magnetometer – z(t)

- Reference signal generated from reference magnet / coils, capacitance or optical sensor.
- Sample oscillated at 75 – 81 Hz
- Detection / pickup coils sense the signal (more recently SQUID sensor)
- AC signal analysed using DSP lock-in
- Noise base of 10^{-6} emu for coils and 10^{-9} – 10^{-8} for SQUID sensors.



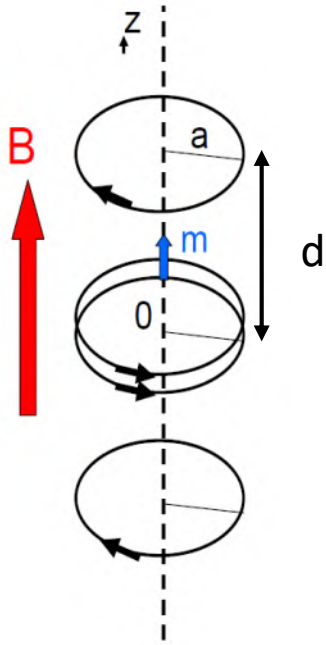
Magnetometry

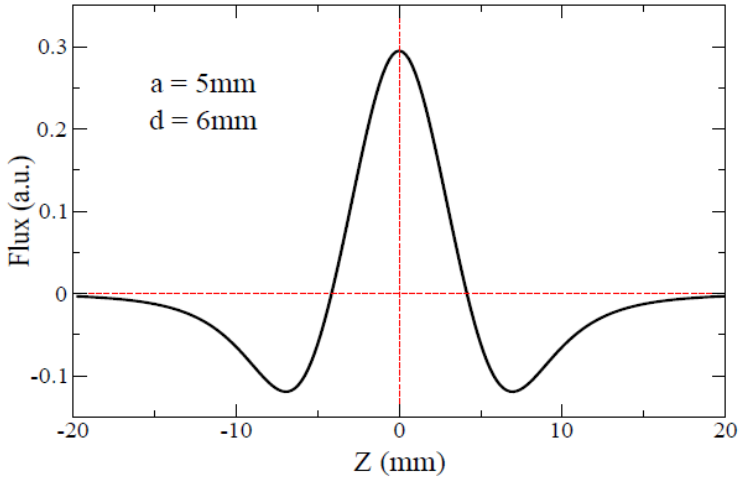
Flux measurement

Superconducting Quantum Interference Device (SQUID)

$$\Phi(z) = -\Phi(z-d) + 2\Phi(z) - \Phi(z+d)$$

- Second order gradiometer - eliminates background contribution from sample holder
- Sample moved slowly through the coils – point measurements taken with a defined no. of averages
- Typical time to complete 1 traverse is a few seconds c.f VSM 70 Hz

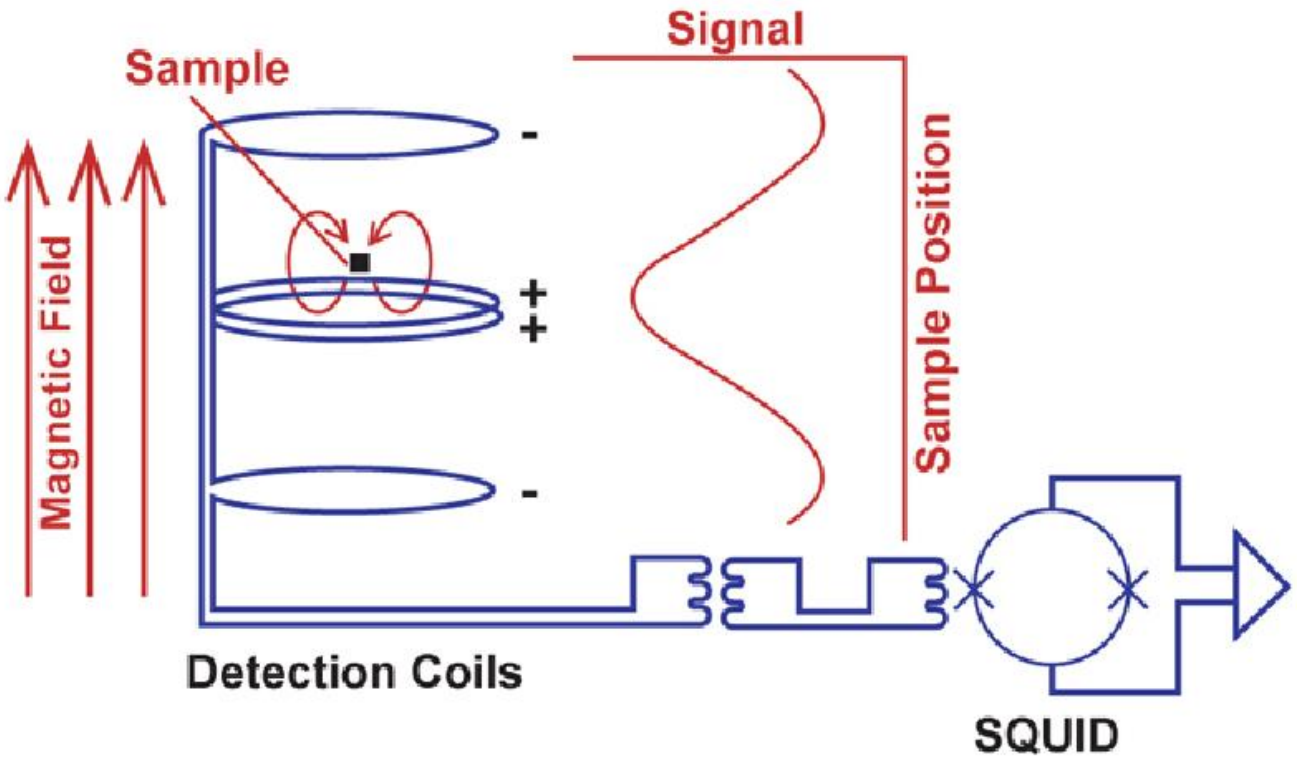




Magnetometry

Flux measurement

SQUID Detection system

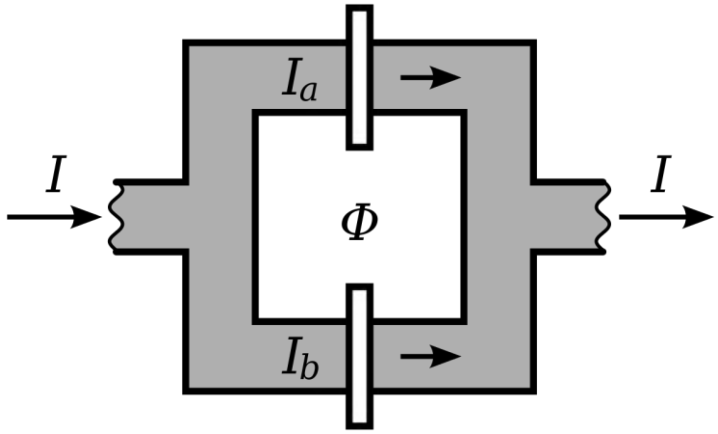


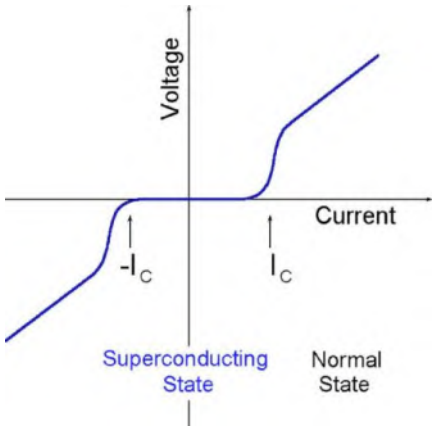
Magnetometry

Flux measurement

SQUID Device

- Based on dc Josephson effect
- Two Josephson junctions in parallel in superconducting loop
- In the absence of a magnetic flux (Φ) the current splits into two equal branches.
- It is biased with a dc current approximately equal to twice I_c and develops a dc voltage across the junctions and shunt resistors.
- A change in the magnetic flux applied through the SQUID loop induces a wave function phase change that enhances the current through one Josephson junction $I_{total}=I_b+ I_{screen} > I_c$ and reduces the current through the other $I_{total}=I_b- I_{screen} < I_c$.
- As the external flux increases or decreases, the voltage will change in a periodic manner with the period being that of the flux quantum, Φ_0.

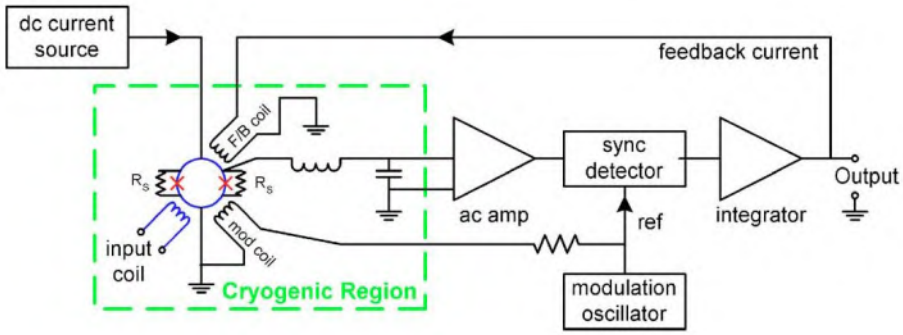




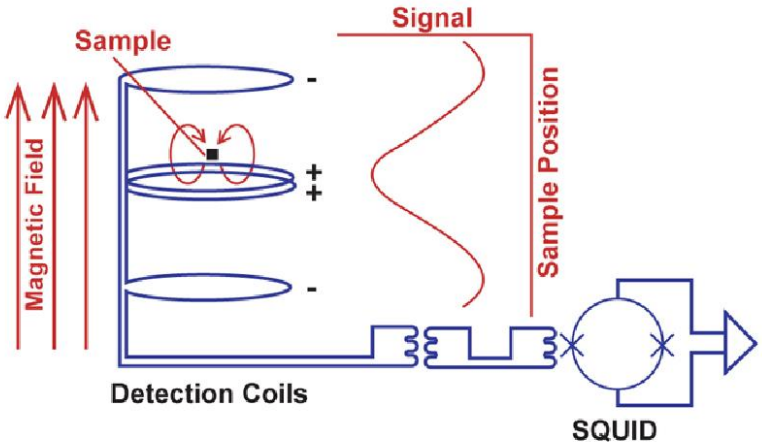
Magnetometry

Flux measurement

SQUID Detection system

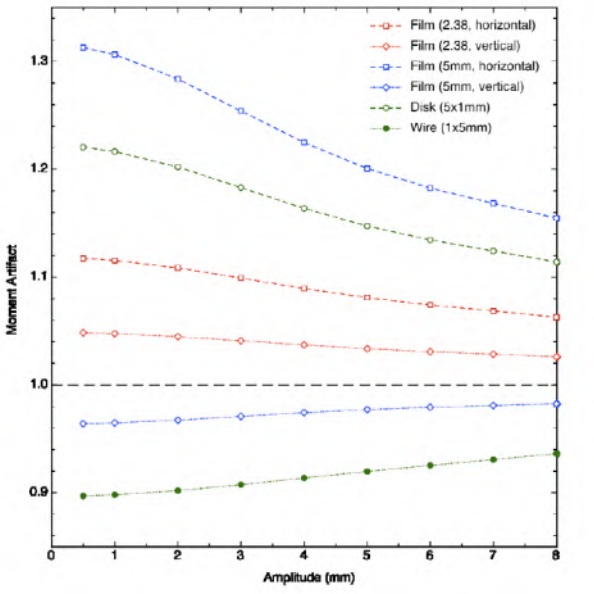


- The voltage, which is periodic in Φ_0, is used to provide a feedback current that nulls the flux penetrating the SQUID loop.
- Measure voltage as function of position
- Fit signal to dipole model of moment vs distance (inc. drift term)
- Report amplitude of fit as signal



Geometric response

Gradiometers measure the number of magnetic flux lines penetrating the plane of the detection coils. Some of the flux lines of a small sample with a moment of, say, *m* = 1.0 emu will close inside the coils, thus not being detected because there is zero net flux. However, if a sample of 1.0 emu moment completely fills the detection coils then none of these lines will close within the coils and will all be detected.



Measured moment depends on the size, shape and orientation of the sample as well as amplitude of oscillation (beyond the predicted linear response in A)!!!!

Geometric response - saddling

$$\frac{dG}{dz} = -\frac{3\mu_0 a^2}{2}\left[\frac{z-(Z+d)}{(a^2+(z-(Z+d))^2)^{5/2}} - \frac{z-(Z-d)}{(a^2+(z-(Z-d))^2)^{5/2}}\right]$$

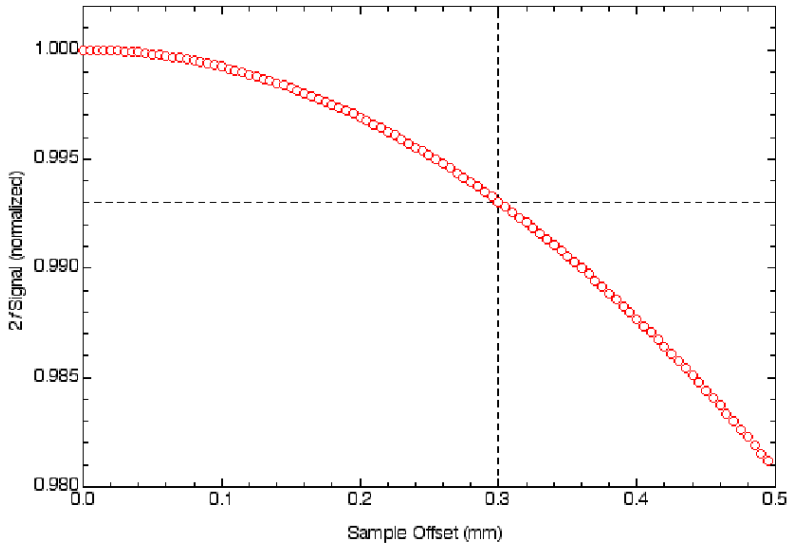


Fig. 1 Impact of axial shift on reported moment (2 mm vibration amplitude).

Coefficient of linear thermal expansion of sample rod?

M(T)?

Magnetometry
Flux measurement

Some issues with VSM and SQUID magnetometry

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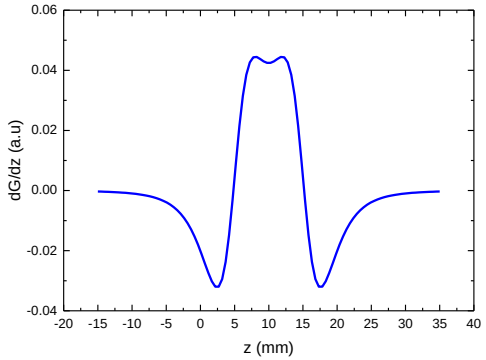
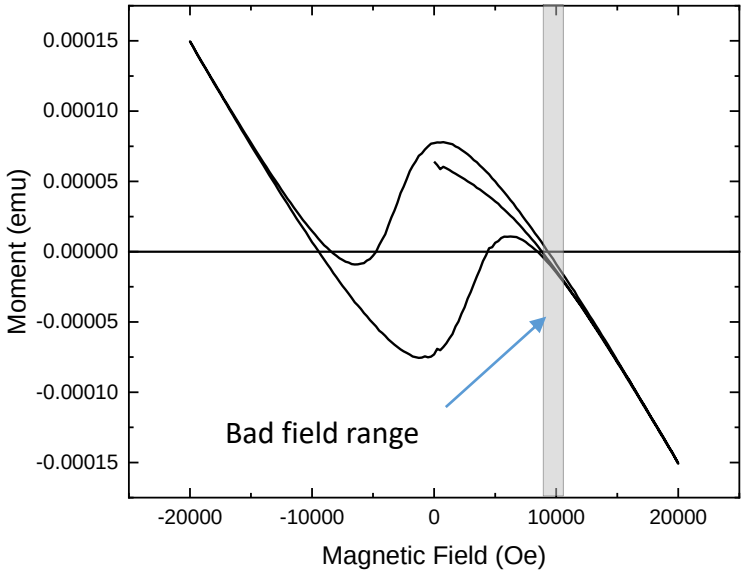
Condensed Matter and Materials Physics

Geometric response - saddling

$$\frac{dG}{dz} = -\frac{3\mu_0 a^2}{2}\left[\frac{z-(Z+d)}{(a^2+(z-(Z+d))^2)^{5/2}} - \frac{z-(Z-d)}{(a^2+(z-(Z-d))^2)^{5/2}}\right]$$





Need to choose H field for saddling carefully – can give confusing results → incorrect saddling.

Strategy: Try high field first (10 kOe) – do you get the shape above? No? Then then low field < 500 Oe.

Does the position look right by eye – centre of the poles QD-VSM – sample should be at 66 mm

Thin ferrite sample (ferrimagnet) on Al_2O_3 substrate (diamagnet)

Magnetometry

Flux measurement

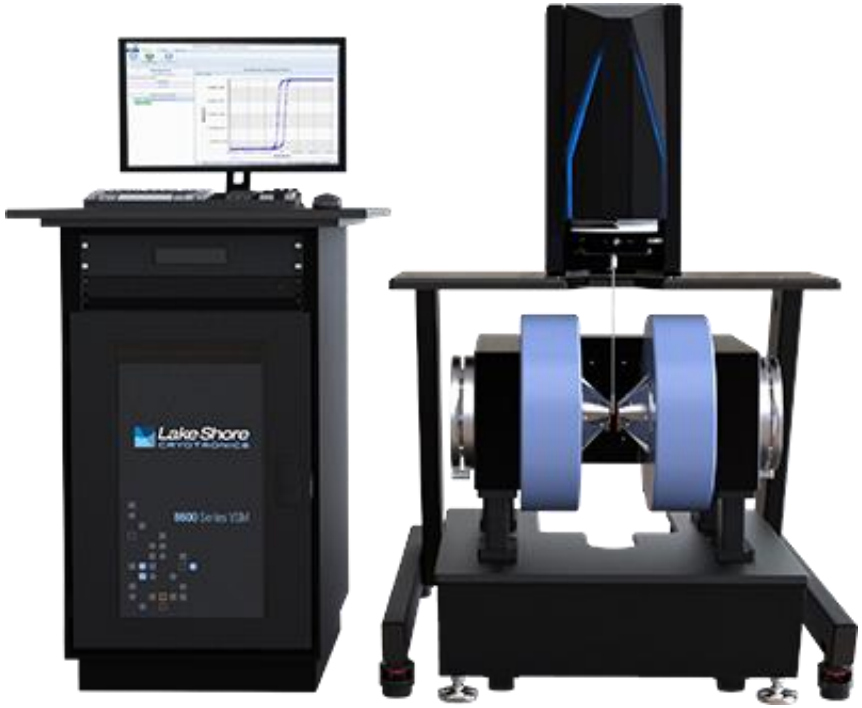
Some issues with VSM and SQUID magnetometry

UNIVERSITY of York

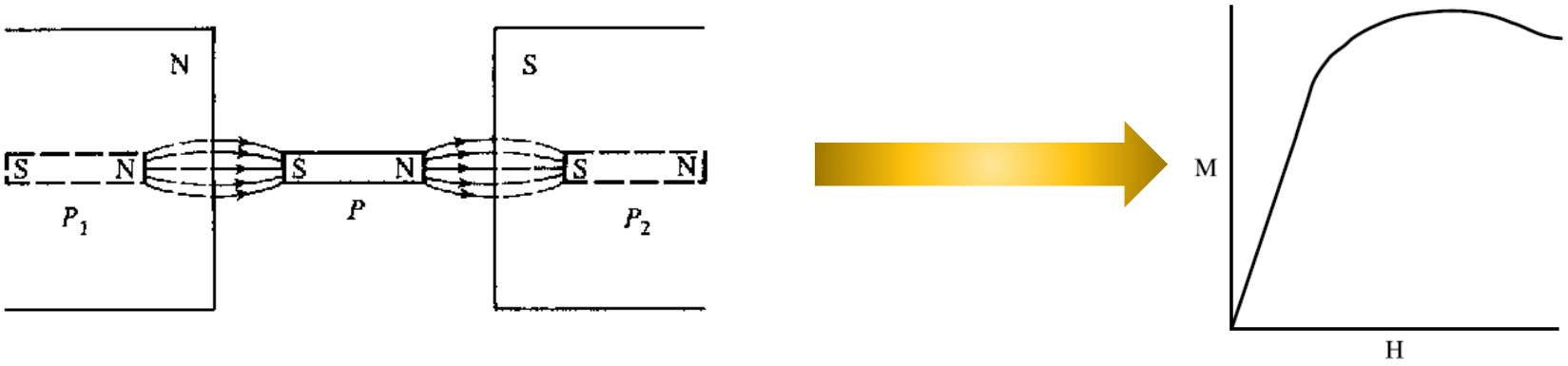
Condensed Matter and Materials Physics

$$\frac{dG}{dz} = -\frac{3\mu_0 a^2}{2}\left[\frac{z-(Z+d)}{(a^2+(z-(Z+d))^2)^{5/2}} - \frac{z-(Z-d)}{(a^2+(z-(Z-d))^2)^{5/2}}\right]$$

- On certain VSM's the magnetic pole pieces can be moved to provide a smaller / larger gap (smaller / larger fields per unit current). Pickup coils (mounted on pole pieces) therefore also move, changing d.
- Re-calibrate!!
- Always check you know the vibration amplitude.



Magnetic Image effect



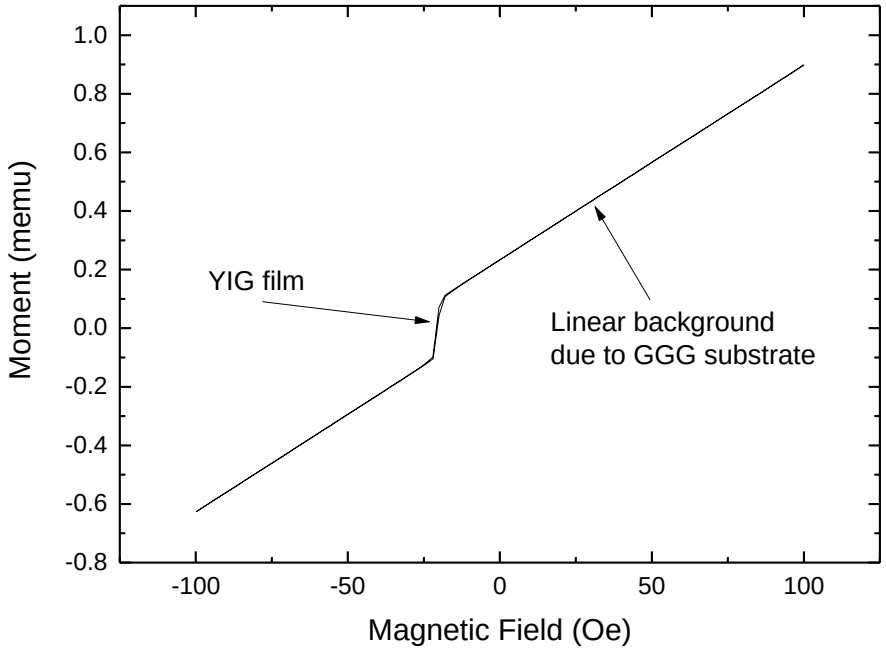
- The magnetic moment of the magnetic image exactly equals that of the specimen when the permeability μ of the pole caps is infinite, decreases as μ decreases, and vanishes when the pole caps become magnetically saturated.
- Increases the VSM signal.
- This increased signal disappears as the material of the pole caps approaches saturation.
- The image effect appears as a drop in signal at high fields, although physically it results from an increase in signal at low fields.

Magnetometry
Flux measurement

Some issues with VSM and SQUID magnetometry

UNIVERSITY of York
Condensed Matter and Materials Physics

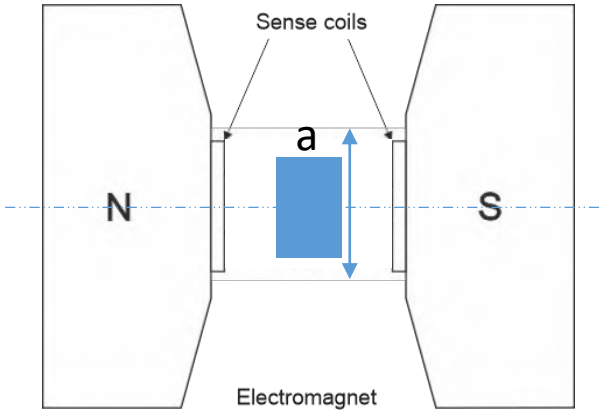
- VSM's operating in air with insulating quartz or Kel-F rods – charging due to friction with air.
- Moving charge → current → generate B field → picked up as an additional flux at the detection coils. Often seen as drift (M increasing with time) – M-H loops don't close.
- Superconducting magnet systems generally do not use close loop controllers as there is no (Hall) sensor – zero current is NOT zero field! Generally not accurate below 50 Oe - trapped flux in superconducting coils.



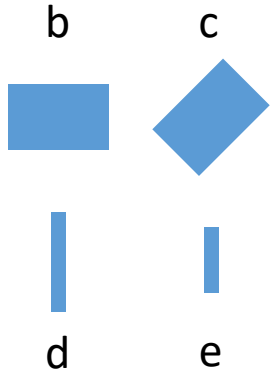
In an idealized measurement, the sample holder is made of a long, uniform material. If a sample holder does not pass completely through the detection coil set, then it creates a more local magnetic field disturbance at the top coil than at the bottom coil, and there is a net signal due to the sample holder.

This is commonly referred to as a sample holder end effect. To prevent this "end effect" from impacting measurements, the sample should be mounted at least 50 mm from both ends of the sample holder, and should be the same distance from both ends. Also, any non-uniformities in the sample holder—such as bearings, fittings, and adapters—should be duplicated symmetrically about the centre of the sample holder.

Calibration – key to precise measurements



- The calibration sample must be the same shape and moment as the sample to be measured
- Often Nickel foils or even worse Nickel rods are used. These have moments of up to 10 emu.
- The moment of a thin film sample is typically 10^{-5} to 10^{-2} emu – hence as a minimum you need a very linear calibration system
- Palladium is a Pauli paramagnet with a moment of 5.28×10^{-6} emu Oe^{-1} g^{-1}. A 100 mg foil sample has a moment of 0.528×10^{-6} emu Oe^{-1}
- This gives a calibration from 0.528×10^{-6} emu to 0.533×10^{-2} emu in a field up to 10kOe
- Pd is bright and does not corrode. Pd foil can be easily cut to the correct size / shape.
- Calibration should be made for every orientation that you intend to measure your sample in.



a.c techniques

Magnetometry

Flux measurement

ac susceptibility (χ_{ac})

"Can make use of Lenz's law and measure the induced voltage from the change in flux, V = - dΦ/dt – need to vary z or μ with time."

dc magnetometry – vary z with time. $\omega/2\pi$ < 100 Hz

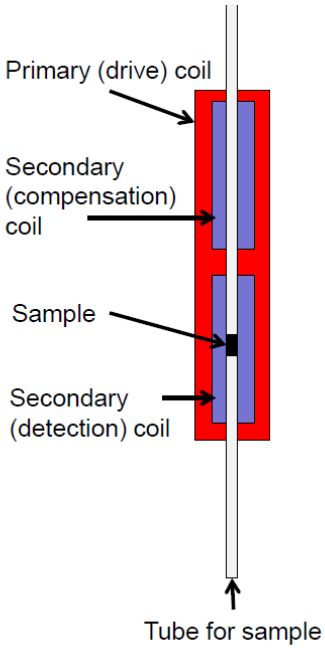
ac magnetometry (susceptometry) – vary μ with time. $\omega/2\pi$ < 100 kHz

- In ac susceptibility measurements a small ac drive magnetic field is superimposed on the DC field, causing a time dependent moment in the sample.
- The field of the time dependent moment induces a current in the pickup coils, allowing measurement via a lock-in amplifier without sample motion.
- At very low frequencies the magnetic moment follows the M(H) curve that would be measured in a dc experiment
- The induced ac magnetisation is given by $M_{ac} = \chi H_{ac} \sin(\omega t)$,
  where H_{ac} is the amplitude of the driving field, ω the drive angular frequency and χ = dM/dH is the slope of the M(H) curve

Magnetometry
Flux measurement

ac susceptibility (χ_{ac})

- Many ac susceptibility instruments are home-built, the essential requirements are:
  - Signal generator (and amplifier)
  - Lock-in amplifier
  - Computer & control system
  - Source of magnetic field
  - Ability to vary sample temperature



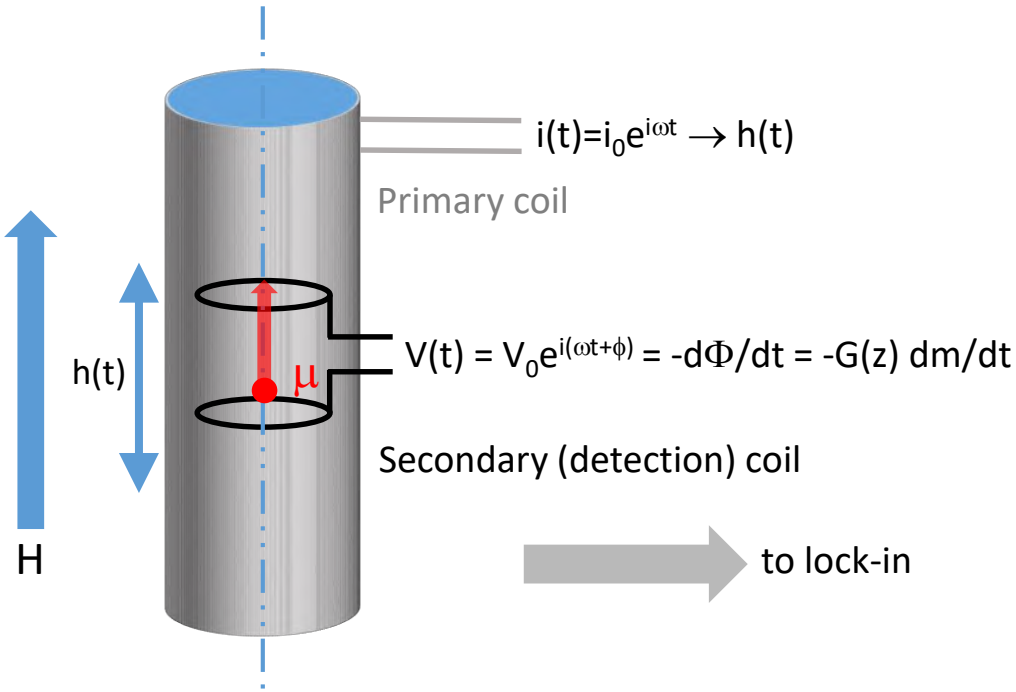
- Basic ac susceptibility measurement consists of a primary excitation coil and two oppositely wound secondary coils
- The secondary coils must be as closely matched as possible to ensure cancelation of background signals –not very easy!
- Amplitude of ac driving field –typically a few Gauss
- Frequency of ac driving field –1 kHz is common but can vary between 1 Hz –100 kHz, above 100 kHz need to consider r.f. response of system e.g. lock-in frequency range etc.

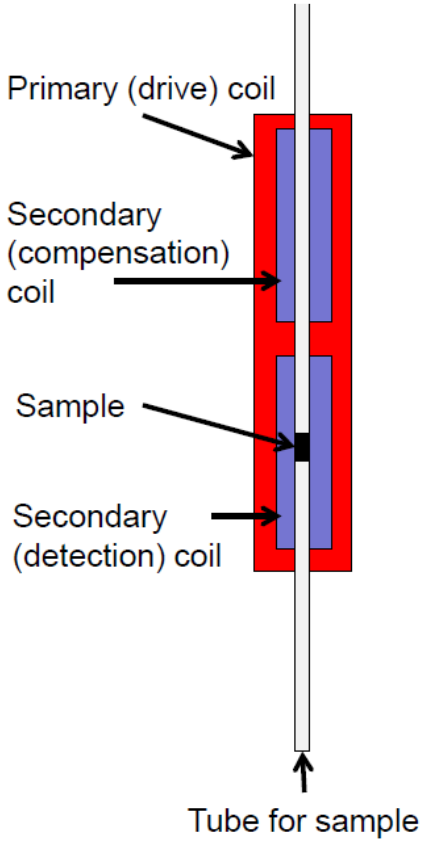
Magnetometry

Flux measurement

Condensed Matter and Materials Physics

ac susceptibility (χ_{ac})





$$G(z) = \Phi(z+d) - \Phi(z+d)$$
$$m(t) = \chi(\omega)h(t), \qquad \chi = \chi' + i\chi''$$

$$V(t) = G(z)\omega b_0(\chi' + i\chi'')e^{-i(\omega t+\phi)}$$

Magnetometry

Flux measurement

ac susceptibility (χ_{ac})

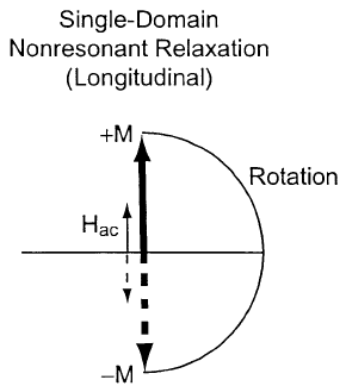
- As the frequency of the ac drive field increase the moment in the sample can no longer follow the drive field – lags.

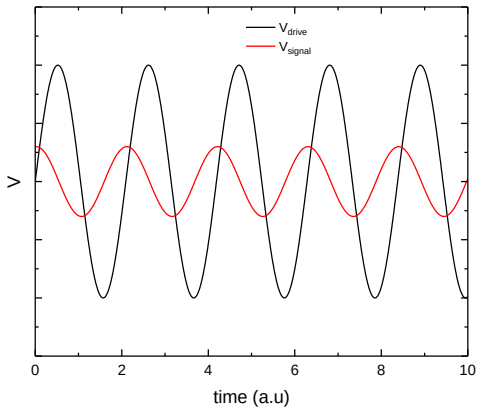
- As with all ac measurements of susceptibilities there are two components – real and imaginary.

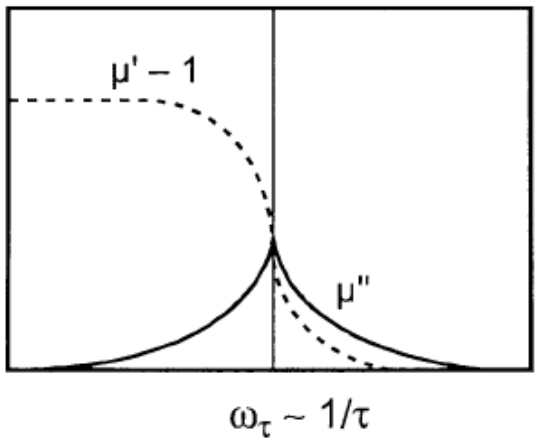
χ' – real in phase component (dispersion)

$\chi'' $ – imaginary out of phase component (absorption or dissipation processes)

$$\chi = \chi' + i\chi''$$







$$\mu_r = \chi + 1$$

Magnetometry

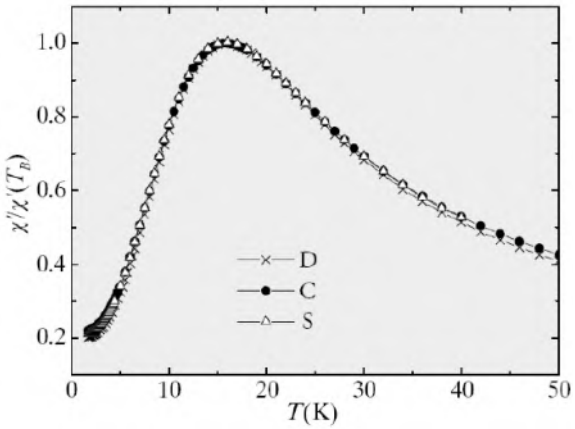
Flux measurement

ac susceptibility (χ_{ac}) - superparamagnetism

- AC susceptibility is an important tool in the characterization of small ferromagnetic particles which exhibit superparamagnetism (Néel and Brown)

- In the superparamagnetic state, the moment of each particle freely rotates, so a collection of particles acts like a paramagnet where the constituent moments are ferromagnetic particles (rather than atomic moments as in a normal paramagnet).

- Above the blocking temperature, χ'' is small and χ' follows the Curie law $\chi' \propto T^{-1}$, as expected for paramagnetic behaviour



- From the slope of 1/ χ' vs. T, one obtains the volume of the magnetic particles (assuming monodisperse particle size). In the case of noninteracting particles, χ' vs. T curves for various particle concentrations are identical when properly normalized. Deviation from this behaviour indicates that interparticle interactions are important

$$\tau^{-1} = f_0 \exp\left(-\frac{\Delta E}{k_B T}\right)$$

The utility of AC susceptibility for superparamagnetism stems from the ability to probe different values of τ by varying the measurement frequency.

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High Frequency techniques

Ferromagnetic Resonance (FMR)

The Swiss army knife for magnetism

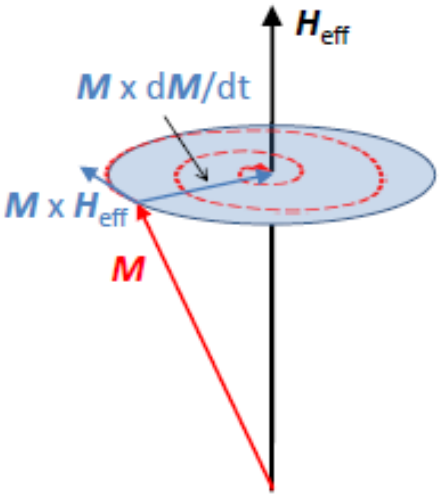
Madrid (2023)

Ferromagnetic Resonance (FMR)

Condensed Matter and Materials Physics

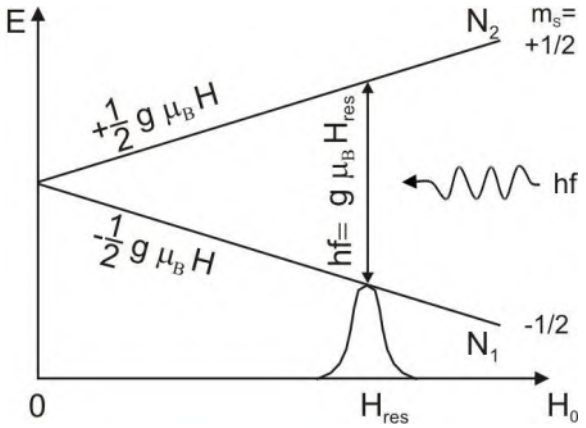
FMR is a method to measure magnetic properties by detecting the precessional motion of the magnetization in a ferromagnetic sample

Zeeman splitting in FM materials leads to Zeeman frequencies that are typically in the microwave region : $\gamma = 17.6\text{MHzOe}^{-1}$



$$\frac{dm}{dt} = -\gamma m \times H_{eff} + \alpha m \times \frac{dm}{dt}$$

$$\omega_o = \gamma H_{res}$$



Applying a magnetic field of the appropriate frequency can cause magnetic dipole transitions from the lower to higher energy levels. This resonant frequency is given by,

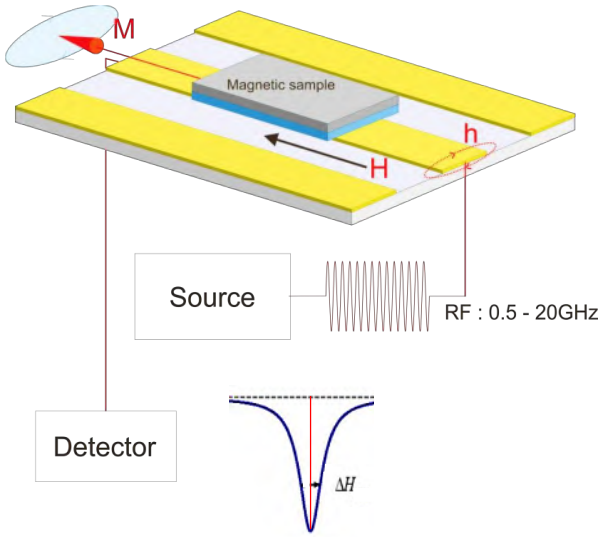
Therefore the absorption of a magnetic field of frequency ω_o can be pictured as the excitation of a precession mode of the magnetisation `gyroscope'.

Ferromagnetic Resonance (FMR)

How does this help?

$$\omega = \frac{\gamma}{M sin\theta}\left[\frac{\partial^2 F}{\partial^2 \theta}\frac{\partial^2 F}{\partial^2 \phi} - \left(\frac{\partial^2 F}{\partial \theta \partial \phi}\right)^2\right]^{1/2}$$

$$F = F_{zeeman} + F_{MCA} + F_{demag} + F_{interface} + \cdots$$



Fe_3O_4 / YSZ(111)

cubic MCA <111> easy axis, uniaxial in-plane MCA, strain induced anisotropy, shape anisotropy

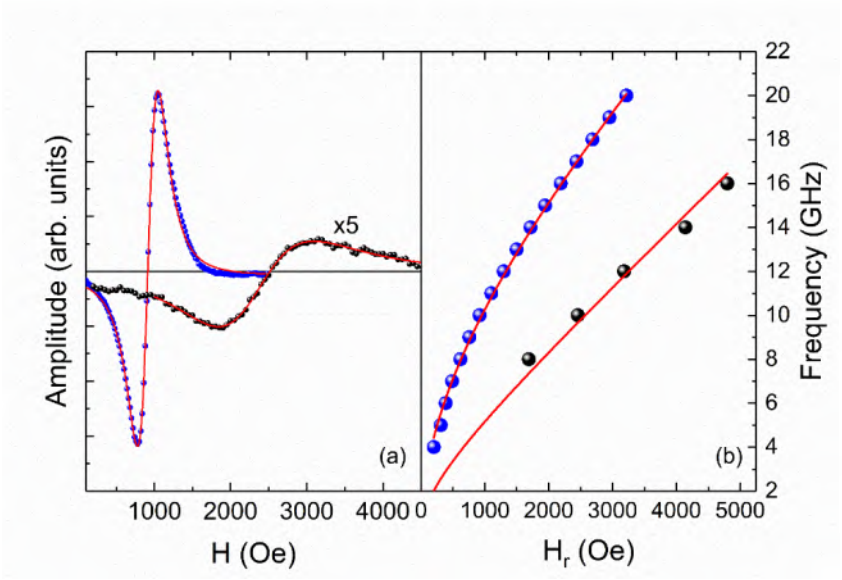
$$f = \frac{\gamma}{2\pi}\left[\left(H + 2\frac{K_{u||}}{M}\cos(2\phi)\right)\left(H - \frac{K_1}{M} + \frac{K_{u||}}{M}\left(1 + \cos(2\phi)\right) + 4\pi M_{eff}\right) - 2\frac{K_1^2}{M^2}\sin^2(3\phi)\right]^{1/2}$$

$$\gamma = \frac{g\mu_B}{\hbar} \qquad g = 2\left(1 + \frac{m_l}{m_s}\right) \qquad 4\pi M_{\text{eff}} = 4\pi \boldsymbol{M} - 2K_{u\perp}/M$$

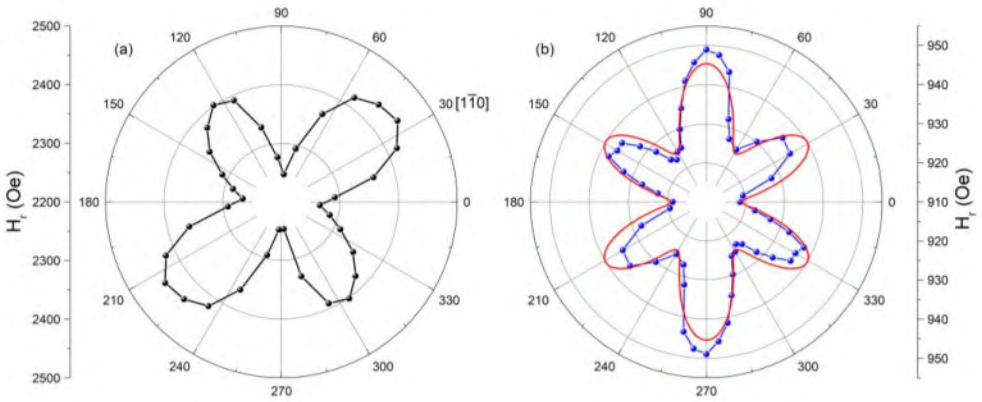
Ferromagnetic Resonance (FMR)

Condensed Matter and Materials Physics

Fe_3O_4 / YSZ(111)



Symmetry correct for the annealed sample



Large strain induced uniaxial anisotropy along surface normal – shifts the FMR to lower fields

Fits to the FMR give g = 2.19 $\pm$ 0.01 for the annealed sample (K_1 = -1.8 x 10^5 erg/cc)

$$g = 2\left(1 + \frac{m_l}{m_s}\right)$$

Orbital moment

XMCD: m_l / m_s = 0.094 $\therefore$ g = 2.18

Bulk Fe_3O_4: g = 2.12 - 2.17, Bickford *et al* Phys. Rev **78**, 449 (1950)

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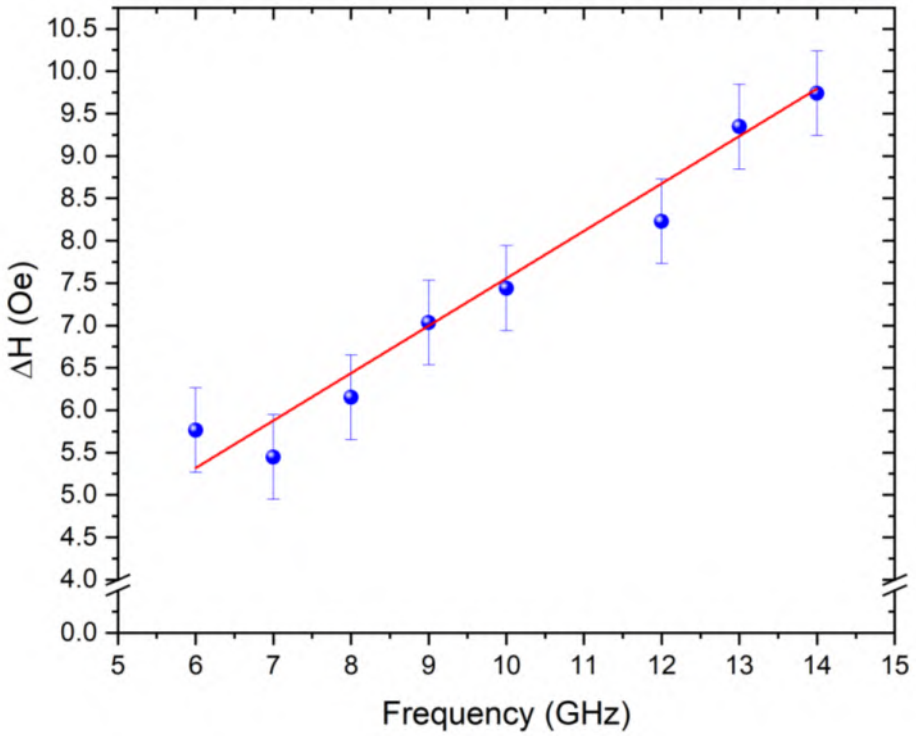
Ferromagnetic Resonance (FMR)

Condensed Matter and Materials Physics

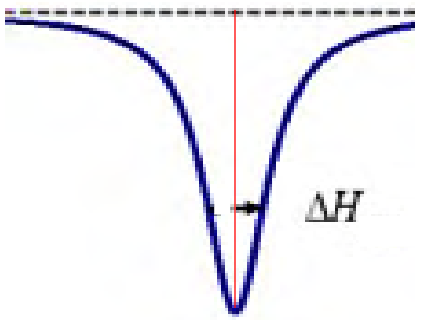
$$\frac{dm}{dt} = -\gamma m \times H_{eff} + \alpha m \times \frac{dm}{dt}$$



$$\Delta H(f) = \Delta H_0 + \frac{2\pi\alpha f}{\gamma}$$

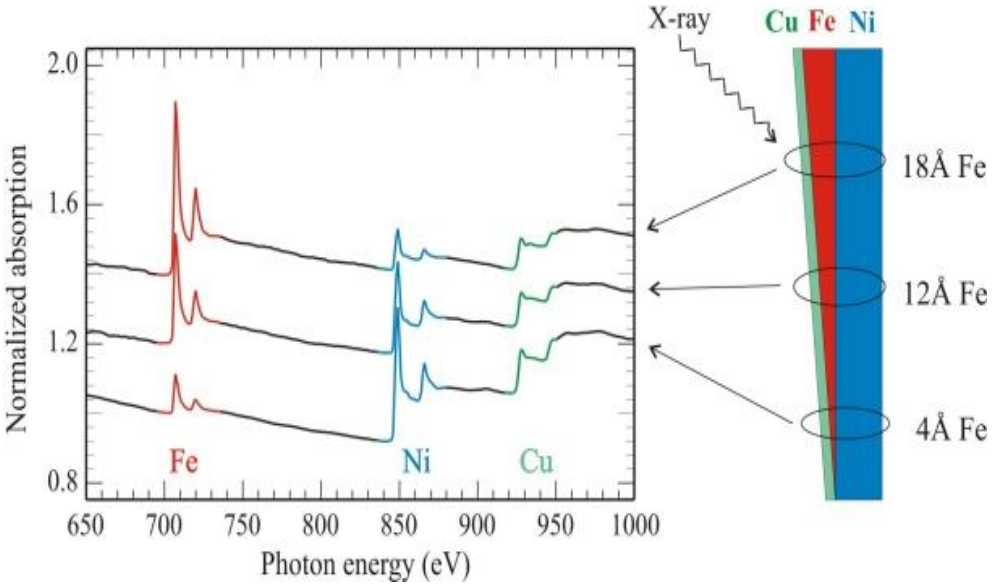
0GHz ΔH (**extrinsic** damping)

Gradient (**Gilbert** damping)



X-ray Absorption Spectroscopy

X-rays can pick materials apart: layer-by-layer



J. Stöhr and R. Nakajima, IBM J. Res. Develop. 42, 1998

X-ray Absorption Spectroscopy

- element specificity
- chemical state

Linearly Polarized X-rays

- orientation of molecules
- spin - orbit interaction
- magnetic anisotropy

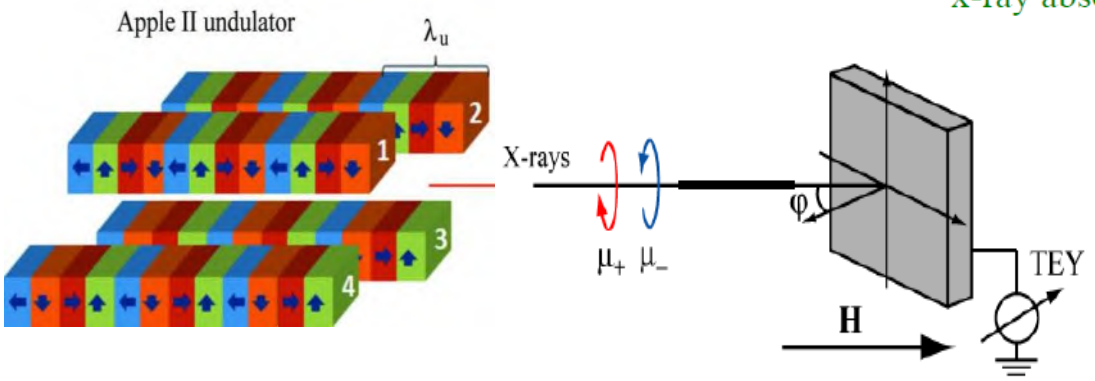
Circularly Polarized X-rays

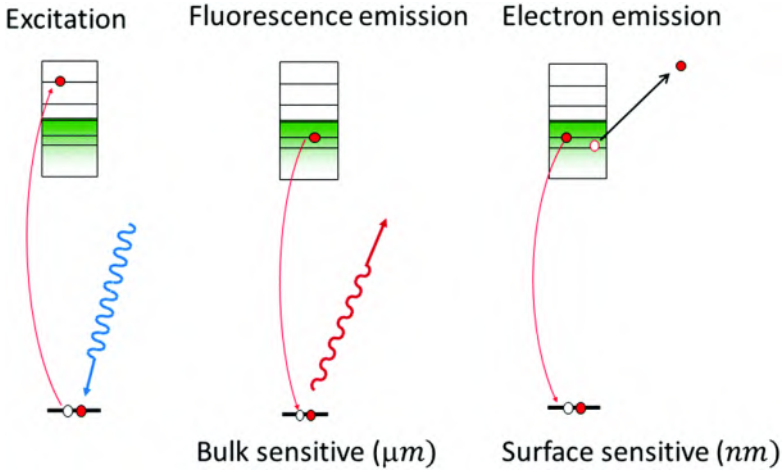
- orientation and size of the magnetic moments
- separate determination of orbital and spin moments

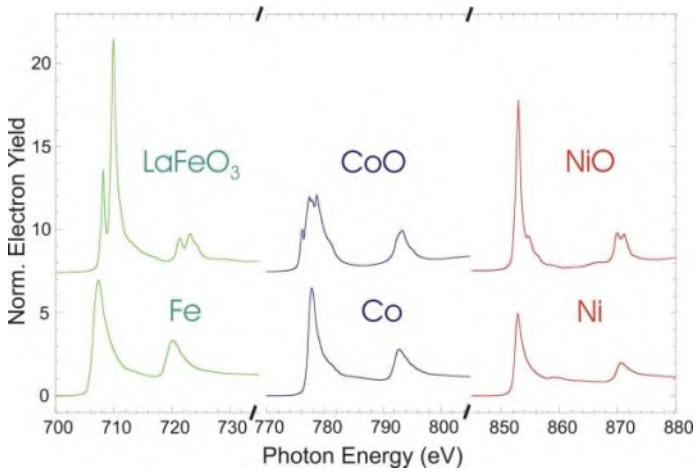
X-ray Absorption Spectroscopy

$I_s \alpha \mu$

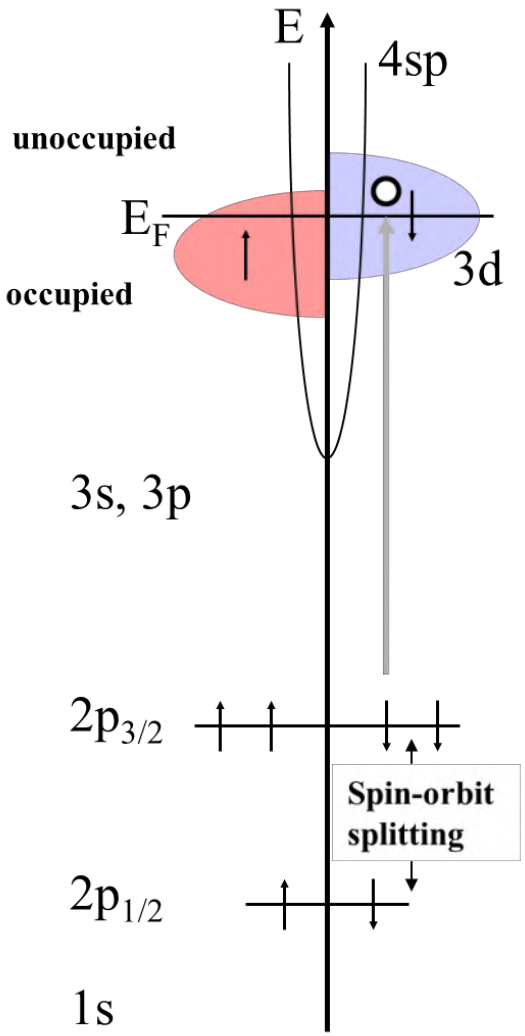
x-ray absorption cross-section







X-ray Absorption Spectroscopy



Fermi's golden rule :

$$\mu^{\pm} \propto \left|\langle f|e\cdot p|i\rangle\right|^2 \rho^{\pm}(E), \qquad \text{dipole transition}$$

Dipole selection rules:

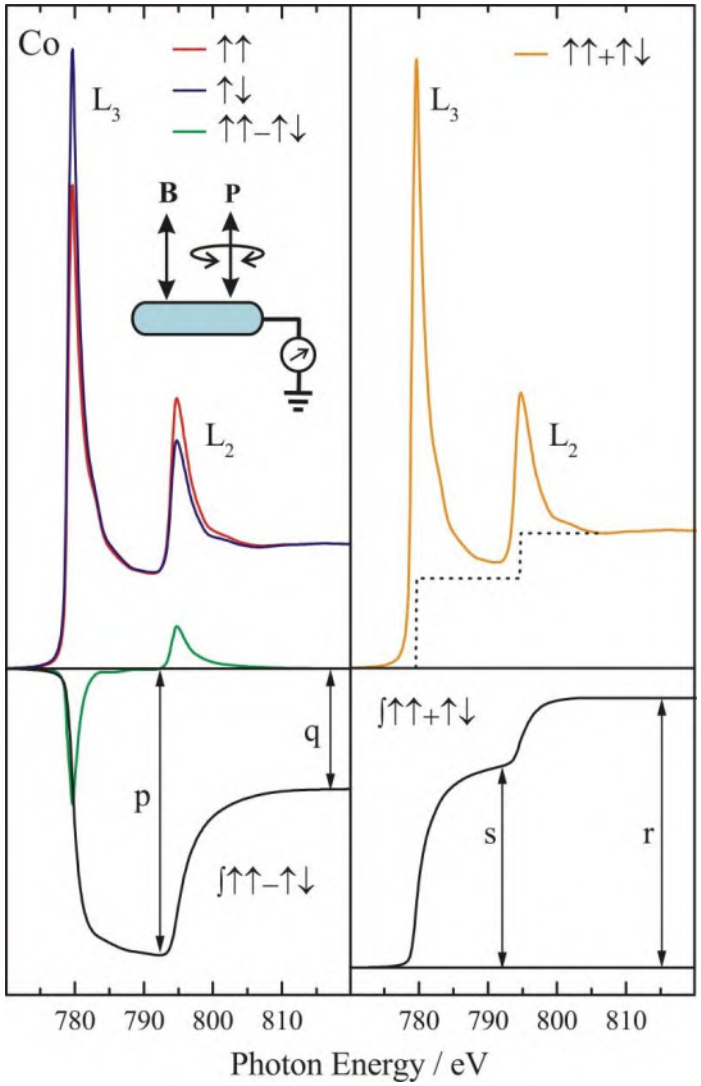
ΔL	=	-1, +1	(p $\rightarrow$ **s,d)**
ΔJ	=	-1, 0, +1	
ΔM_J	=	-1, 0, +1	
Polarization q		+1, 0, -1	(s^+, p, s^-)

L_2: $2p_{1/2} \rightarrow 4s, 3d_{3/2}$

L_3: $2p_{3/2} \rightarrow 4s, 3d_{3/2}, 3d_{5/2}$

X-ray Magnetic Circular Dichroism

Condensed Matter and Materials Physics



Sum Rules

Number of *d*-holes $n_h \propto r$

Branching ratio $\langle \boldsymbol{l}\cdot\boldsymbol{s}\rangle / n_h = 2\text{-}3s/r$

Orbital moment $\langle \boldsymbol{L}\rangle = -4\,q\,n_h/3r$

Spin moment $\langle \boldsymbol{S}\rangle + {}^{7}/_{2}\langle \boldsymbol{T}\rangle = -(3p\text{-}2q)\,n_h/r$

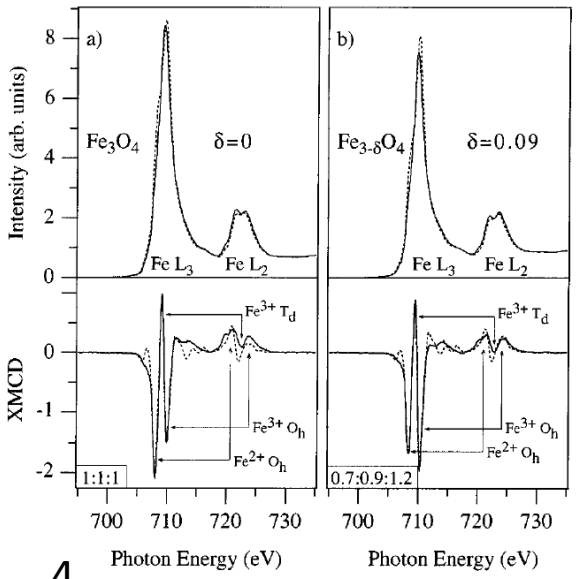
Ratio $m_{orb}/m_{spin} = 2q/(9p-6q)$

$$XMCD \propto M \cdot \sigma$$

X-ray Magnetic Circular Dichroism

Condensed Matter and Materials Physics

1



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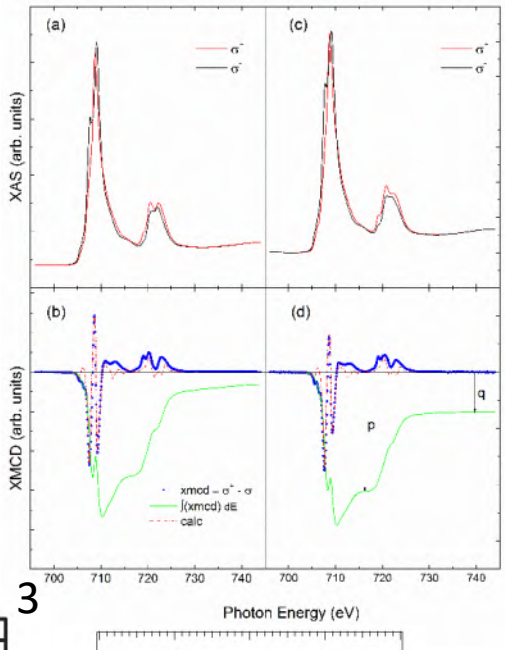
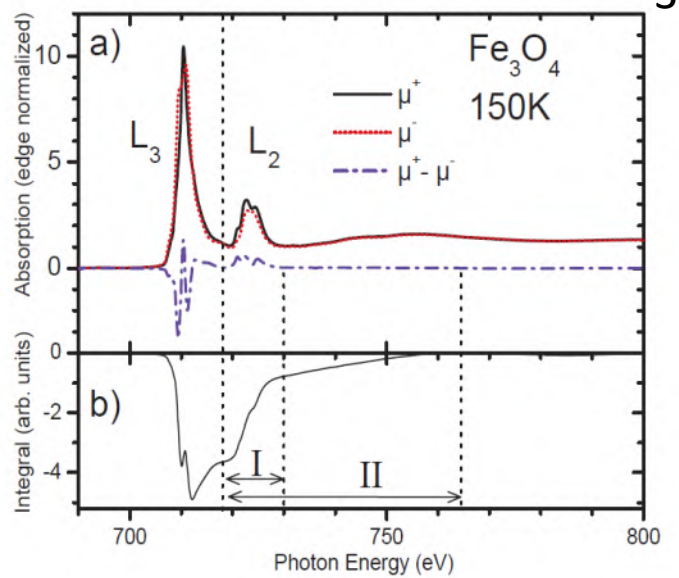
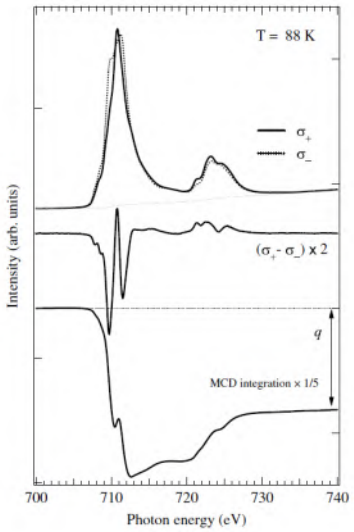


4



3



Peak ratio at L_3

1: PRB **67**, 214408 (2004) **0.45:0.21:0.33**

2: PRB **107**, 064414 (2023) **0.46:0.22:0.32**

3: PRL **93**, 077204 (2004) **0.53:0.13:0.34**

4: EPL **73**, 97 (2005) **0.49:0.21:0.30**

Orbital Moment

2: **annealed 0.27 μ_B**

3: 0.65 μ_B

4: 0 μ_B

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X-ray detected FMR: XFMR

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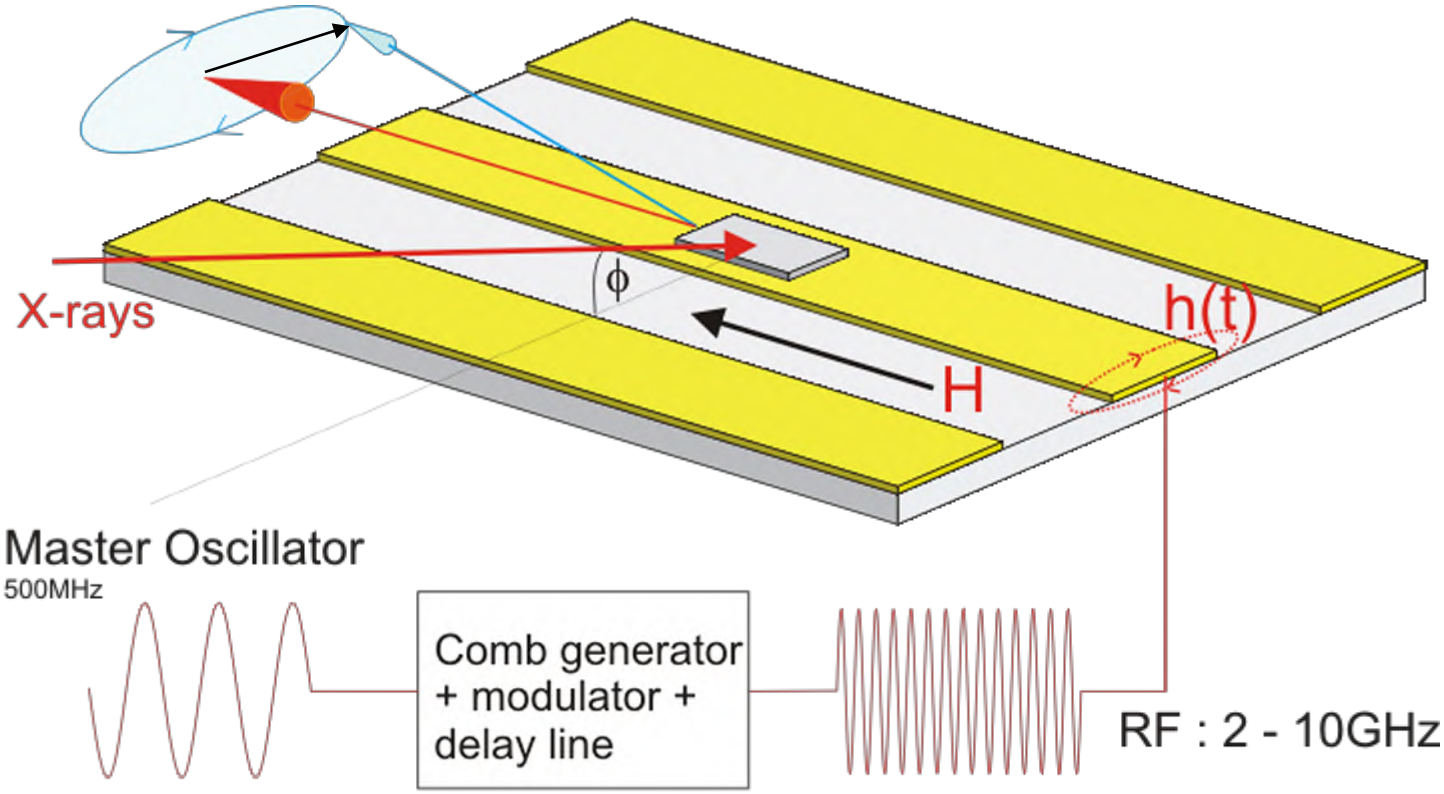
FMR has been widely used to study magnetization dynamics of multilayer systems.
Can only detect the net response of the multilayer sample. Response of individual layers inferred.

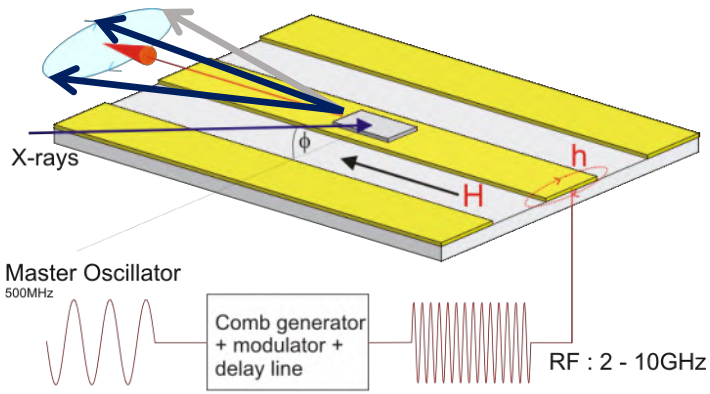


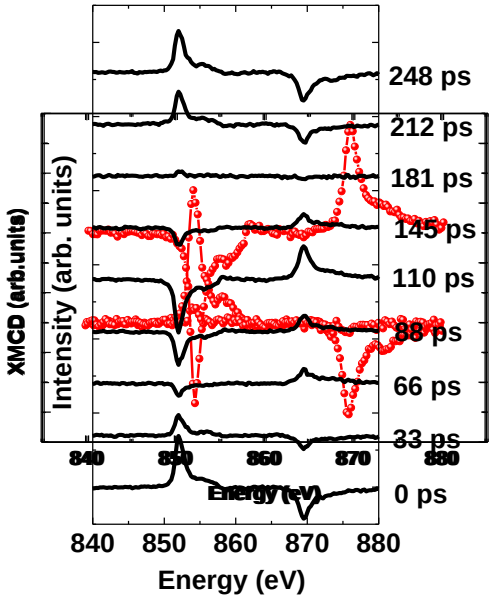
M. Marcham et. al. J. Appl. Phys, **109**, 07D353 (2011) , G. Boero *et al*., Appl. Phys. Lett. **87**, 152503 (2005), D. A. Arena *et al.*, Phys. Rev. B **74**, 064409 (2006)

X-ray detected FMR: XFMR

Condensed Matter and Materials Physics

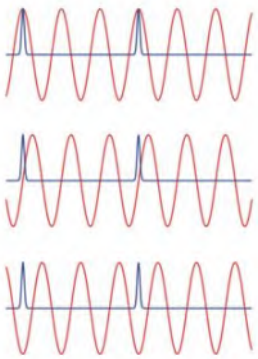
Permalloy (NiFe)





Stroboscopic Measurement

Able to make us of all bunches – no special fill pattern needed



M. Marcham *et. al*. J. Appl. Phys, **109**, 07D353 (2011)

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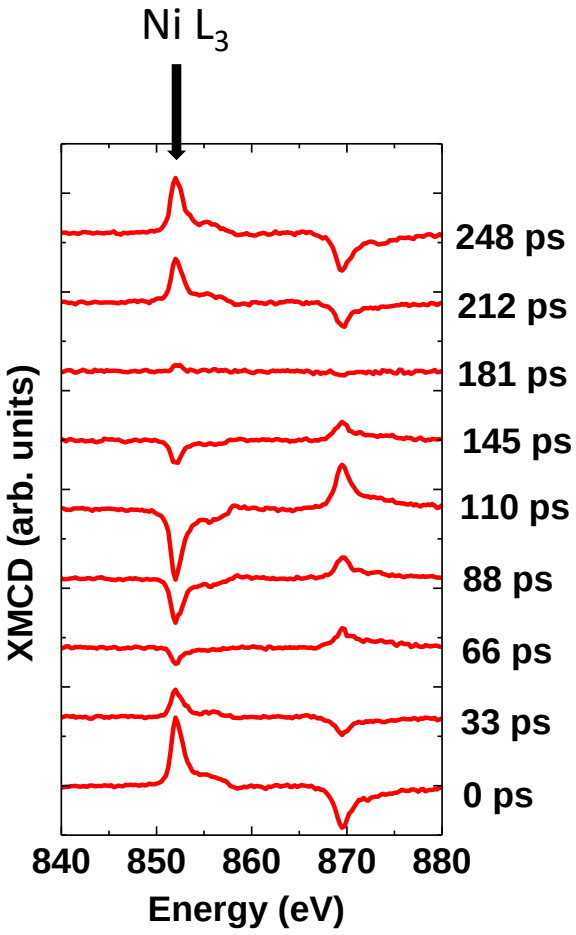
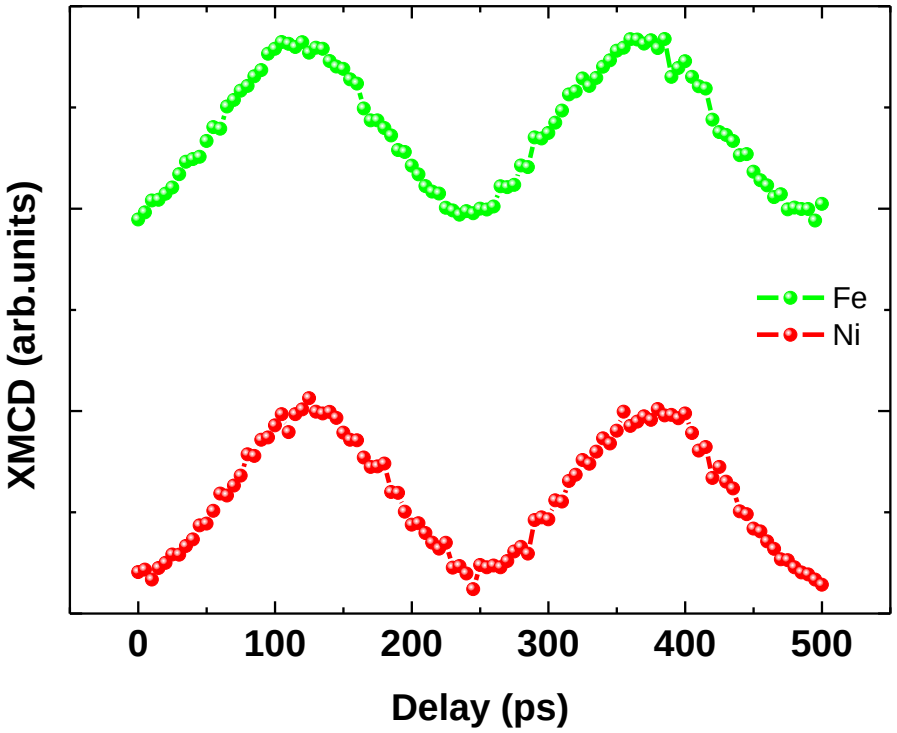
X-ray detected FMR: XFMR

Condensed Matter and Materials Physics

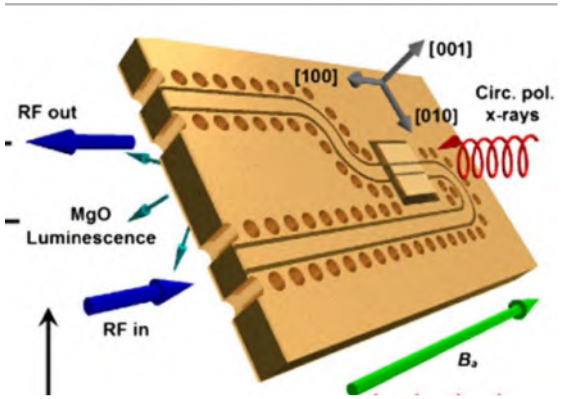


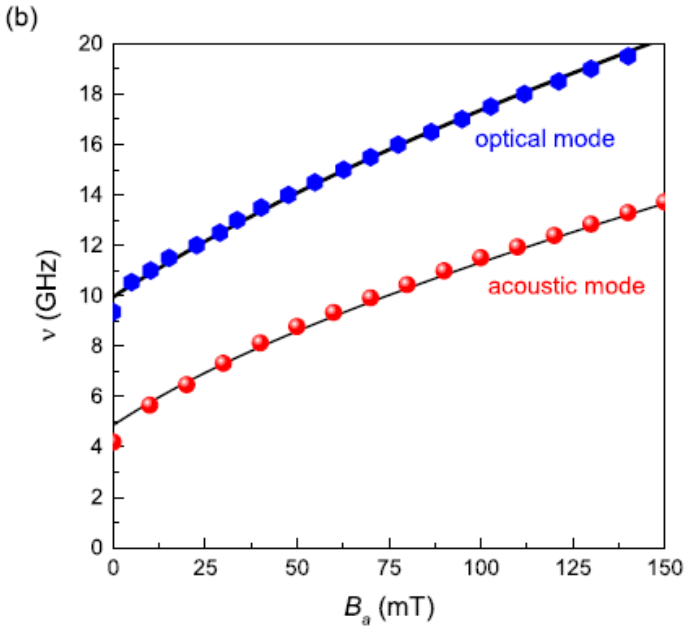


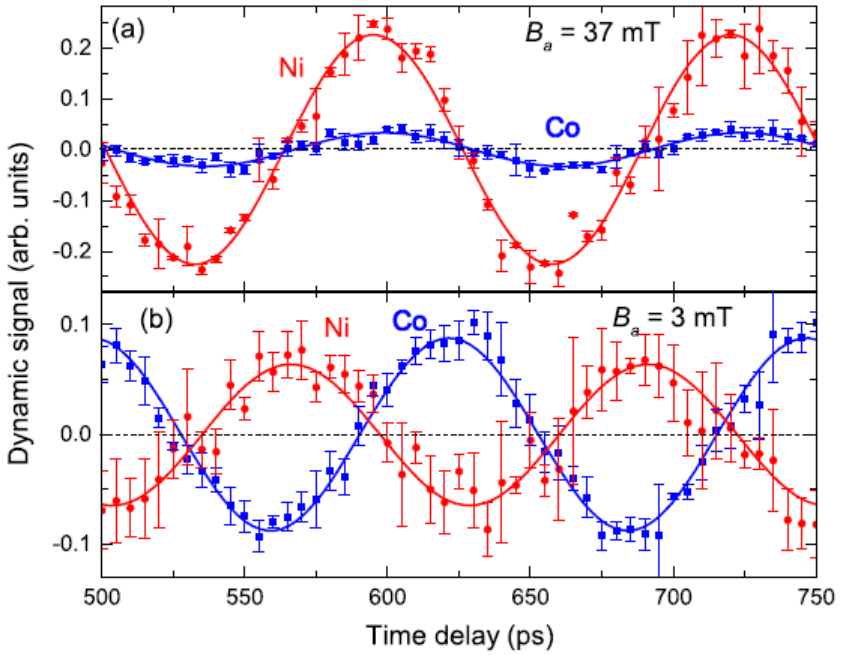
M. Marcham *et. al*. J. Appl. Phys, **109**, 07D353 (2011)

X-ray detected FMR: XFMR

Exchange coupled bilayer: CoFe/Py







New J. Phys. 17 (2015) 013019

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Thank you for your attention