Rock-Magnetic Experimentation, Data Analysis and Sample Characterization

Thermomagnetic methods: \( M(T_{\text{meas}}, T_{\text{trt}}, t...) \) in fixed field
- \( M(T_{\text{meas}}) \) - mineral composition and concentration, independent of grain size, microstructures...
- \( k(T_{\text{meas}}, f, H_{ac},...) \) - composition, grain size...
- \( M(H_{trt}) \) for different initial remanent states - history-dependent behavior, wide range of experiments possible
- \( M(T_{\text{trt}}, H) \) - TRM acquisition, thermal demagnetization

Isothermal methods: \( M(H_{\text{meas}}, H_{trt}, t...) \) at constant \( T \)
- \( M(H_{\text{meas}}) \) - hysteresis, FORC analysis
- \( M(H_{trt}) \) - IRM, ARM acquisition, AF demagnetization, coercivity spectrum analysis

Composite methods: field- and temperature-dependent \( M \)
Thermal fluctuation tomography: \( f(V,H_k) \) for SP/SSD populations

Hysteresis, \( n. \)
Etymology: < Greek ὑστέρησις a coming short, deficiency, < ὑστερεῖν to be behind, come late, etc., < ὑστερός late.

A phenomenon observed in some physical systems, by which changes in a property (e.g. magnetization, or length) lag behind changes in an agent on which they depend (e.g. magnetizing force, or stress); any dependence of the value of a property on the past history of the system to which it pertains.
1881 Proc. Royal Soc. 33 22 The change of polarisation lags behind the change of torsion. To this action the author [J. A. Ewing] now gives the name Hysteresis.

OED

Hysteresis Loop

Hysteresis Parameters
Saturation Magnetization: \( M_s \)
Saturation Remanence: \( M_r \)
Coercivity: \( H_c \)
Coercivity of Remanence: \( H_{cr} \)
Initial Susceptibility: \( k_0 \)

For (Dilute) Pure Materials
Composition dependent: \( M_s, M_r, k_0, H_c, H_{cr} \)
Concentration dependent: \( M_s, M_r, k_0 \)
Grain size/microstructure: \( M_s/M_r, M_r/k_0, k_0/M_r, H_c, H_{cr} \)

For Magnetic Mixtures
Individual parameters and ratios all vary according to component properties and relative proportions

For (Dilute) Pure Materials
Composition dependent: \( M_s \)
Concentration dependent: \( M_s, M_r, k_0 \)
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Dunlop and Özdemir, 2007
Hysteresis in Single Domain Particles

Stoner-Wohlfarth Model (1948)

Non-interacting particles
Coherent rotation without thermal effects
Uniaxial anisotropy

Total magnetic energy of particle consists of field interaction energy ($E_H$) and the uniaxial shape anisotropy energy ($E_a$)

\[ E(\theta, \phi) = E_H + E_a \]
\[ E_H = -\mu_0 M_s H_s \cos(\phi - \theta) \]
\[ E_a = K_v \sin^2 \theta \]
\[ K_v = \frac{1}{2} \mu_0 (N_s - N_s^*) M_s^2 \]


Magnetite ($M_s=480 \text{ kA/m}$) SD needles ($H_k=240 \text{ kA/m} = 302 \text{ mT}$), $B_{pp}=160 \text{ mT}$
Angular dependence of switching fields for uniaxial SD particles (Stoner – Wohlfarth)

(a) $H_0$ aligned with easy axis ($\phi = 0$)

\[
H_c = H_K = (N_b - N_a)M_s
\]

$M/M_s = 1.0$

(b) $H_0$ perpendicular to easy axis ($\phi = \pi/2$)

Reversible magnetization

$M_r/M_s = 0, H_c = 0$

$\chi = M/M_K$

(c) for all $\phi$ (randomly oriented array of SD particles)

$M_r/M_s = 0.5$

$H_c = 0.479 H_K (\phi = 0) = 0.479 (N_b - N_a)M_s$

Dunlop and Ozdemir, 1997

Magnetization process in SD Grains
Rotation of Moments
Response of a random assemblage of uniaxial single domain (SD) particles during hysteresis cycle

Magnetization process in PSD Grains
Nonuniform Magnetization States

Dunlop and Ozdemir, 1997
Magnetization process in MD grain

Wall Energy is a function of position in a crystal due to the crystal defects

Demagnetizing field is strong enough to drive walls backs towards M=0

Nucleation, displacement, demarcation of DW with changing field

Remanent state ($M_r$) is near zero with low $H_c$

Hysteresis Parameters for MD grains

$M_r/M_s < 0.1$

$H_c(MD) < H_c(SD)$

Wall translation

Domain rotation

Domain nucleation

(a) Demagnetized state

(b) In the presence of a saturating field,

(c) Field lowered to +3 mT

(d) Remanent state, e) back field of -3 mT,

Inset shows detail of domain walls moving by small increments called Barkhausen jumps.

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O'Reilly, 1984

Magnetite hysteresis properties: $f(d)$

Room temperature saturation remanence ($M_r$) and Coercivity ($H_c$) as a function of grain size for magnetite (Dunlop and Özdemir 2007)

Rather than the rather abrupt decrease at the SD threshold predicted theoretically, gradual decreases in properties are observed over several decades of grain diameter

Magnetite + Hematite

Large SP particles

SD/SP magnetite

Shear zone mylonite

Housen, 1992

Mixtures of Magnetic Phases

Composition and Grain Size

Distorted Loops

Hausen and Fuller, J. Geophys. Res., 86, 6505-6522, 1983

Tauxe, 1996

Housen, 1992

Tauxe et al. 1996
Unmixing Hysteresis Loops
1. \( M(H) = M_r(H) + M_p(H) + M_{af}(H) + M_{lin}(H) = M_f(H) + M_{lin}(H) \)

Additional hysteresis measurements and parameters
- Stoner-Wohlfarth Model
- randomly-oriented elongate uniaxial SD grains
- \( M_r(B) \): "remanent hysteretic magnetization" – irreversible magnetization changes
- \( B_{rh} \): median field of \( M_r \), \( \sim B_{cr} \)
- \( M_{ih}(B) \): "induced hysteretic magnetization" – reversible magnetization changes

SD magnetite
fine hematite

SSD+SP (8 nm)

SSD+SP (4 nm)
Unmixing Loops
2. Coercivity classes

Additional hysteresis measurements and parameters

M(B): initial magnetization curve from saturation remanence
Mrr: hysteresis energy loss
Mrr = Esh/Ehys: TED ratio
Mrr(B): remanent hysteretic curve
Ehys > (4MssBc), \( \sigma > 0 \)
Ehys = (4MssBc), \( \sigma = 0 \)
Ehys < (4MssBc), \( \sigma < 0 \)

Additional hysteresis measurements and parameters

Sigma=0.64 ("wasp-waisted")

Backfield remanence and $H_{CR}$ in SD Grains

Angular dependence of switching field for randomly-oriented uniaxial SD grains (Stoner-Wohlfarth)

The "Day Plot"

"Squareness-coercivity plot": Grain size / domain state, composition

"Day plot": Grain size / domain state

The "Day Plot" includes graphs showing the relationship between grain size, domain state, and composition. The "Squareness-coercivity plot" examines how squareness and coercivity vary with grain size. The "Day plot" illustrates the relationship between grain size and domain state.
coercivity ratio $H_{cr}/H_c$ becomes large for mixtures of hard and soft materials (e.g., mgt-hmt, SP-SD)

for TM60, SD-MD mixtures are quite different from discrete PSD sizes

for magnetite, SD-MD mixtures are indistinguishable from discrete PSD sizes

Theoretical Day Plots for Magnetite (Dunlop, 2002)

Day Plots: Behavior of mixtures

Day Plots: Behavior of mixtures

CS914.001
Questions

- How can we tell if M is saturated?
- What are the uncertainties in $M_s$ and $\chi_{HF}$?

Analysis of Variance and F tests

sum-squared deviations (variance) from best-fit model

= noise variance (pure error)
+ misfit variance (model error)

$$F = \frac{\text{misfit variance}}{\text{noise variance}}$$

= (total variance − noise variance)/(noise variance)

high values of $F$ -> reject model

How do we calculate noise variance?
Inversion Symmetry through origin: \( M(H) = -M(-H) \)

1. Quantify noise variance (‘pure error’):
   \[ \sigma^2 = \frac{\sum (\Delta_i)^2}{N} \]

2. Quantify signal/noise ratio:
   \[ \sigma_m^2 = \frac{\sum (M_i)^2}{N} \]
   \[ s/n = \frac{\sigma_m}{\sigma} \]
   \[ Q = \log_{10}(s/n) \]

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Real and Imaginary Susceptibility
(conductivity, viscosity, hysteresis)

- \( H(t) = H_0 \cos(\omega t) \)
- In-phase (‘real’):
  \[ M'(t) = k' H(t) = k' H_0 \cos(\omega t) \]
- Out-of-phase (‘imaginary’ or ‘quadrature’):
  \[ M''(t) = k'' H_0 \sin(\omega t) \]

AC Rayleigh Loops:
Rayleigh Magnetization Law

\[ M = H(\chi_0 + \alpha H_0) \pm \alpha (H_0^2 - H^2) / 2 \]

\[ \chi' = \chi_0 + \alpha H_0 \]

\[ \chi'' = \frac{4 \alpha H_0^2}{3 \pi} \]

\[ \frac{d \chi'}{dH} = \frac{4 \alpha H_0}{3 \pi} \]

For \( H < \chi_0 / \alpha \),
\[ dM/dH = \text{constant} \]
Low-field hysteresis (Rayleigh) loop for basalt sample

\[ H = 1000 \text{ A/m} \]
\[ B = \mu_0 H = 1.26 \text{ mT} \]

Dunlop and Özdemir, 1997

Amplitude Dependence of Susceptibility

Rayleigh Relationship

\[ \chi' = \frac{4}{3\pi} \frac{d\chi''}{d \ln H_0} \]

Synthetic TM spheres

Rayleigh Relationship

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Synthetic TM spheres
For materials that undergo hysteresis in weak fields:

1. Susceptibility increases with field amplitude
2. Out-of-phase susceptibility increases with field amplitude

MD TM above 100K, MD pyrrhotite above 34K MD hematite above 250K
Thermal fluctuation tomography

hysteresis/backfield measurements over a range of T -> distribution of V, Hk (grain size and shape)

\[ \tau(V, M_s, H_k, T, H_0) = \tau_0 \exp \left( \frac{VM_s(T)H_k(T) \times (1 - H_0 / H_k(T))^2}{2kT} \right) \]

For shape anisotropy: \( H_k(T) = (N_b - N_s)M_s(T) \)

Relaxation time is governed by:

* Intrinsic Mineral Properties
  \( M_s(T) \)

* Experimental/Natural Conditions
  \( H_0, T \)
\[ \tau(V, M_s, H_K, T, H_0) = \tau_0 \exp \left( \frac{VM_s(T)H_K(T) \times (1 - H_0 / H_K(T))^2}{2kT} \right) \]

For shape anisotropy:

\[ H_K(T) = (N_x - N_y)M_s(T) \]

Relaxation time is governed by:

1. Intrinsic Mineral Properties
   \( M_s(T) \)
2. Experimental/Natural Conditions
   \( H_0, T \)
3. Grain Characteristics
   \( V, \text{shape } (N_x-N_y) \)

For \( H_0 \neq 0 \), blocking contours are curves of constant \( VhK \)

For \( H_0 > 0 \), blocking contours shift to right

\[ \Delta M = \int_{\text{area}} f(V, H_{K0})dA \]
DC demagnetization of IRM (constant $T = T_1$)

$\Delta M = \int f(V, H_{k0}) dA$

$\mu_0 H_{k0}$ [T]

Log$_{10}(V$ [m$^3$])

DC backfield [mT]

Blocking contours (H, T$_1$)

$\Delta M = \int f(V, H_{k0}) dA$

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CS914
10-300K, $\Delta T = 10^6$
$\mu_0 \Delta H = 5$ mT

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Coercivity Component Analysis of Remanent Magnetization Curves

Different phases
Different grain sizes
Different anisotropies
Different sources

Decomposition of IRM acquisition/demagnetization curves into several components with the use of model functions (e.g., log-Gaussian coercivity distributions)
Fig. 10: Statistical distribution of switching fields in two modern soils collected in Minnesota (a) and on the Western Chinese Loess Plateau (b). The black curve in each plot is the derivative of a magnetization curve obtained by stepwise demagnetization of an ARM. This curve has been modeled with a set of statistical distributions (colored curves), each representing the distribution of switching fields of a particular set of magnetic particles with a common geohistorical origin. PD are pedogenic magnetic or maghemite grains. D and L are low coercivity minerals from the parent sediments (glacial till and loess, respectively). B is the contribution of a high-coercivity component (hematite). Notice that the properties of PD are amazingly similar in the two soils, despite the difference in climate and parent material. This observation suggests that the mechanism of magnetic enhancement (but not the amount) is similar in a wide group of soils.

Egli, 2005 (Environmental Magnetism Workshop)
http://www.irm.umn.edu/Misc/soils_case_study.pdf

Egli, 2004

Coercivity Deconvolution

Natural sediments

MTB

Cubo-octahedral

elongated

\[ \frac{\Delta M(B, B_{meas})}{dB_{meas}} \]

Magnetic Components

\[ \frac{\Delta M(B, B_{meas})}{dB_{meas}} \]

FORC model: aligned uniaxial SD grain(s), no bias, \( B_c = 100 \text{ mT} \)

\[ \frac{\Delta M(B, B_{meas})}{dB_{meas}} \]

Reversal field

Measurement field

\[ \frac{\Delta M(B, B_{meas})}{dB_{meas}} \]
FORC model: aligned uniaxial SD grain(s), 15 mT bias, $B_c=100$ mT

FORC Analysis / Preisach Theory

Magnetic hysteresis of real samples can be represented as the superposition of a large number of elemental "hysteron s" – square loops which each have a coercivity (half width) and bias field.

Each point in the FORC/Preisach space has a specific coercivity and bias, and the FORC/Preisach function represents the relative number or strength of hysteron s with those characteristics.

Positive bias
(loop shifted to right)

Negative bias
(loop shifted to left),
Same coercivity

Sum of two hysteron s
with equal and opposite bias, same coercivity

Zero bias
Low coercivity

Zero bias
Higher coercivity

Sum of two hysteron s
with zero bias, different coercivity

Sum of two hysteron s
with opposite bias, same coercivity
FORC Analysis / Preisach Theory

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Each point in the FORC/Preisach space has a specific coercivity and bias, and the FORC/Preisach function represents the relative number or strength of hysterons with those characteristics.

Magnetization process in MD grain

MD FORC distribution: lots of hysterons with low coercivity and a wide range of positive and negative bias fields

Magnetization curves from Stoner-Wohlfarth Model for various angles between the direction of magnetic field and easy axis

Stoner-Wohlfarth (ideal noninteracting uniaxial SD) FORC behavior

hysterons with small bias fields and a range of coercivity extending up to $H_k$