Magnetic tests and characterization protocols: mineralogy and grain size / domain state
Part II: isothermal weak field tests

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In the first installment of this short series of articles on magnetic tests, we discussed using strong magnetic fields to better understand the magnetic domain state and/or particle sizes of a sample. In this second installment, we will describe the use of weak field magnetizations and susceptibilities, as well as useful ratios and biplots that can help to characterize grain size or domain state. Many of the magnetizations and properties described are of extremely widespread use and have become the “bread and butter” of rock-magnetic research and some applications of rock-magnetism in particular. Other properties, on the other hand, are not as common and require instrumentation that is not as readily available, and have therefore remained more unfamiliar to some, despite the useful information they may provide.

The discipline of environmental magnetism first and foremost has incredibly benefited from advances in rock-magnetism, and many of the tests and procedures described here were devised for environmental applications. The same goes for paleointensity and other areas of research. Other disciplines, however, have sometime displayed some hysteresis (#magnetistjoke, see IRMQ 22(4)) and have maintained a more simplistic approach. As is the case, nature is complex, and so is magnetic research, so without further ado we introduce the low field tests.

Magnetic Susceptibility

Measurement of magnetic susceptibility, \(k= M / H\), in its most general (and common) form, probably involves the weakest field application that will come to mind to most paleo- and rock-magnetists. Magnetic susceptibility is most commonly measured in fields of ~200-300 A/m or less [footnote: the current generation of multifunction Kappabridges operates at AC field intensities from 2 A/m up to a maximum of 700 A/m; the Magnon VFSM range is 20 - 400 A/m; older Kappabridges had a fixed field strength of 300 A/m], which do not (generally) cause irreversible magnetizations, and therefore is a widely applied non-destructive technique to characterize a specimen’s magnetic response.

The magnetic behavior of any material is subdivided into diamagnetic, paramagnetic and ferromagnetic (ferrimagnetic and antiferromagnetic) components and therefore, when immersed in a steady field, the different minerals present will contribute positively or negatively to the bulk magnetic susceptibility of the specimen. The general assumption is that iron oxides have larger susceptibilities than other diamagnetic and paramagnetic minerals that make up the “non-magnetic matrix” and therefore, the ferrimagnetic minerals will dominate the susceptibility response. Whether the assumption is correct or not, magnetic susceptibility is a strongly concentration-dependent property, and is often referred to as “bulk” susceptibility.

Bulk susceptibility, as other magnetic properties, can be normalized by the volume or by the mass, which sometimes introduces minor confusion when dealing...
**Current Articles**

A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most are taken from ISI Web of Knowledge, after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

**Archeomagnetism**


**Biomagnetism**

**Environmental magnetism and Climate**


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"Before the breakup, and after the breakup": 1858 illustrations of Pangea by Antonio Snider-Pellegrini, from "La Création et ses mystères dévoilés" ("Creation and its Mysteries Unveiled").
with the units of measure, or at least imposes defining two “different” susceptibilities. Because of the weak fields used, it makes sense to report these in A/m, and therefore susceptibility measurements will involve magnetic moment (Am²) divided by field (A/m), which will then be equal to m³. Normalizing by volume (m³ in SI), will result in dimensionless susceptibility, referred to as $k$, which users often report as “SI unit”, or, because of the generally small values, $\mu_{SI} = 10^{-6} SI$. However, not all rocks (or specimens) have comparable densities, and volume-normalized susceptibilities often reflect variable degrees of compaction as well as differences in concentration. For this reason, and because it is generally easier to get accurate measurements of mass than of volume, susceptibility (like other concentration-dependent properties) is commonly mass-normalized. When mass is used as a normalizer (kg in SI), the bulk susceptibility is then referred to as mass susceptibility $\chi$, in m³/kg.

Assuming the susceptibility response is dominated by the ferromagnetic fraction, susceptibility is most often used as a magnetic concentration parameter, which makes it a valuable normalizer to remove the concentration-dependence from other magnetic properties (more below).

The above is valid in steady (DC) magnetic fields, for which $k$ or $\chi$ are simple real numbers. However, changing (AF or AC) fields may cause delayed responses, making the AC susceptibility in general a complex quantity:

$$k = k' - i\omega k''$$,

where $k'$ is the in-phase response of $M$ to an oscillating field $H_F$ and $k''$ is the 90° out-of-phase (or quadrature) response (Fig. 1).

The delayed response and corresponding out-of-phase susceptibility can arise from three different physical mechanisms (see IRMQ 13(4)): (1) magnetic viscosity with relaxation times comparable to the AC field reversal interval; (2) irreversible magnetization changes (low-field hysteresis) driven by the AC field; and (3) production of electrical eddy currents by the AC field in electrically conductive materials. Each of these mechanisms is significant only for a restricted range of materials. Viscosity on millisecond timescales is generally only important for fine SD particles, near the SP boundary. Low-field hysteresis has been documented only in multidomain pyrrhotite, hematite and intermediate composition titanomagnetics, where spontaneous magnetization is not too large and wall-pinning energies are comparable to external-field interaction (Zeeman) energies. Conductivity is only large enough to matter in metals, graphite and some sulfide minerals, in the frequency range commonly used for susceptibility measurements. Quadrature susceptibility $k''$ is therefore almost always much smaller than in-phase susceptibility $k'$. But when it is significant in magnitude, it provides important information about the magnetic (and/or conductive) mineral assemblage.

For very small SD (“viscous superparamagnetic” or VSP) grains, $k''$ and the frequency dependence of $k'$ (frequency $f = \omega/2\pi$) are closely related to viscous changes of $M$, with time $t$ (Néel, 1949; Mullins and Tite, 1973; Shcherbakov and Fabian, 2005; Egli, 2009). A useful relation for recognizing thermal relaxation is the so-called “$\pi/2$ law: $\chi''_{[\text{viscosity}]} = -(\pi/2)(\chi'/6\ln f)$, which applies to populations of VSP particles with a range of particle sizes/shapes or thermal activation energies.

These behaviors allow for grain size applications of magnetic susceptibility: at small SD grain sizes the susceptibility increases as the superparamagnetic threshold is approached, and small rotations of the grain moments in response to the field give way to complete thermal-assisted moment reversals (e.g. Stacey & Banerjee 1974). Since superparamagnetic properties are characterized by a short relaxation time $\tau$, the apparent susceptibility of grains that are superparamagnetic at DC and low AF fields will decrease strongly at frequencies, $f > 1/\tau$.

While magnetite susceptibility is independent of $H_o$ up to the maximum AC fields available in most instruments (a few hundred A/m or $\mu_T$), Clark (1984) and later Worm (1991) discovered low-field amplitude dependence for the susceptibility of pyrrhotite; this is due to low-field hysteresis, and is accompanied by an increase in quadrature susceptibility. Worm et al. (1993) also determined that pyrrhotite (depending on grain size) exhibits frequency-dependent susceptibility in sufficiently high frequencies (> 1kHz), due to induction of eddy currents. Room temperature dependence on field amplitude ($H_o$) and frequency ($f$) can thus be used as indication for coarse-grained pyrrhotite (Fig. 2).

Jackson et al. (1998) observed field amplitude-dependence for Ti-magnetite for both in-phase ($\chi'$) and out of phase ($\chi''$) susceptibility, and determined that (a) $\chi'$
decreases and (b) $\chi''/\chi'$ increases with increasing Ti-substitution (Fig. 3). With decreasing spontaneous magnetization, the susceptibility drops below the "self-demagnetization limit" of $1/N$ (where $N$ is the demagnetizing factor) that is characteristic of MD particles of high-intensity magnetic phases.

A useful parameter to quantify frequency-dependence, for example for environmental applications, is the percent frequency-dependence of magnetic susceptibility $\chi$, 

$$\chi_{fd\%} = \left(\frac{\chi_{lf} - \chi_{hf}}{\chi_{lf}}\right) \times 100$$

Where $\chi_{lf}$ and $\chi_{hf}$ are a low and high frequency values, respectively, that are typically an order of magnitude apart. The difference $\chi_{lf} - \chi_{hf}$ is zero for SD grains, and increases for SP grains (e.g. Oldfield et al., 2009).

**ARM**

Anhysteretic remanent magnetization (ARM) is acquired in the presence of a strong alternating field (AF) that decays with time, and a weak steady (DC) field. The steady field produces a bias in what would otherwise be an effective demagnetization process. The role of the randomizing AF is analogous to that of temperature in thermal demagnetization or remanence acquisition, overcoming anisotropy energy barriers in the magnetic particles, and allowing the net moment of the population to equilibrate with the ambient steady field. Thus, despite the use of strong alternating fields, ARM is considered to be a weak-field remanence, with an intensity generally proportional to the bias field, typically on the order of the Earth’s field or little higher (~50 μT - 200 μT, or equivalently, 40 - 160 A/m), and an orientation generally parallel to it (in the absence of anisotropy, or if applied along an easy-axis of magnetization). The rapidity of ARM’s (at least compared to a laboratory TRM), jointly with its magnetization effectiveness and the capability of modern magnetometers to apply these inline, makes them extremely useful.

**Weak/strong field tests**

**AF demagnetization spectra, the Lowrie Fuller Test**

Lowrie and Fuller (1971) devised a test that would help distinguish between remanences carried by SD or MD grains. In its original form, they compared the AF demagnetization spectra of the original NRM, or a weak field TRM, with the spectra of an IRM. Because this test removes the NRM, an alternative protocol was proposed under the assumption that an ARM is an adequate analogue to the TRM (Dunlop et al., 1973). The basic idea is that, if the weak-field TRM is more easily AF demagnetized than the strong field SIRM, the sample is in a predominantly MD state. If the opposite is true the sample is more SD (Fig. 4). Following Johnson et al. (1975), most applications of the Lowrie-Fuller test have substituted ARM for TRM as the weak-field remanence. In either case, the test has uncertain theoretical foundations (e.g. Dunlop and Özdemir 1997), and is based solely on experimental observations. The test’s va-
Figure 4. NRM/IRM AF demagnetization plots of Fuller et al. (1988). In the left-hand panel demagnetization data of altered lava carrying secondary magnetization are shown as a function of AF field: other than the first step, the IRM overall demagnetizes more readily than the NRM, suggesting SD-like behavior. On the right-hand panel Permian lavas from New Zealand show faster demagnetization of an IRM, suggesting MD-like behavior.

Magnetic Ratios and biparametric plots
Banerjee-King Plot
A useful grain-size dependent property is the anhysteretic susceptibility (χARM units of m^3/kg), and defined as the ratio of the ARM magnetization, in Am^2/kg, to the bias DC fields, in A/m, which is highest for particles in the stable SD (SSD) size range, decreasing slowly with increasing grain size and rapidly falling to zero for smaller grains (i.e. towards SP sizes, Egli and Lowrie, 002) (Fig. 6).

Similarly, the low-field susceptibility (χLF) is highest for particle sizes at the upper end of the SP range, and decreases (not necessarily monotonically) through the SSD and larger sizes (Fig. 7 a and b).

The Banerjee-King plot (Banerjee et al., 1981; King et al., 1982) tries to quantify the concentration and effective grain size of magnetic carriers, or variations in the ratio of coarse to fine grain-sizes, by constructing a biplot of anhysteretic susceptibility (χARM) versus low-field susceptibility (χLF) (e.g., Fig 8a). By plotting these two concentration- and size dependent properties against each other, it is possible to obtain an estimate of magnetite concentration and particle size (Fig 8b, King et al., 1982). The technique has been successfully used to distinguish climatic/environmental events in lake cores from Minnesota (Banerjee et al., 1981; King et al., 1982).
Grain-size dependent properties in the Tiva Canyon tuff (Till et al., 2011); magnetic particle size increases systematically upward from the base of the flow. The peak in $\chi_0$ marks the SP-SSD transition; the peaks in $\chi_{ARM}$ and in $M_r/M_s$ mark the upper end of the SSD range. The size range does not include MD particles.

**χ_{ARM}/SIRM**

Assuming a single (magnetite) remanence carrier, the $\chi_{ARM}/SIRM$ ratio, where SIRM is the saturation isothermal remanent magnetization imparted in a field of 1 T, reflects some aspect of the particle-size distribution of the remanence carriers. Because both of these are remanent magnetizations, SP particles have no influence on the ratio. $\chi_{ARM}$ decreases more rapidly than SIRM with increasing grain size, so the ratio decreases continuously from SD through the PSD and MD size ranges. In samples with a wide range of particle sizes, the $\chi_{ARM}/SIRM$ ratio is related to the proportion of finer grains in the population (Maher, 1988). Higher values sometimes exceed $2 \times 10^3$ m/A, indicating dominance by SD grains (e.g. Oldfield et al., 2003; Egli and Lowrie, 2002).

**ARM/SIRM versus DC field**

Sugiura (1979) utilized ARM acquisition (normalized to SIRM) versus DC field to quantify magnetic interactions: PSD particles (sample 6 in Fig. 10) have ARM acquisition that is more linear and plots at lower values of ARM/SIRM than SD particles (sample 1 in Fig. 10). Sugiura (1979) therefore proposed to use the shape and slope of the curves to quantify magnetic interactions. The technique was successively utilized by a number of authors on different applications (e.g. Dunlop and Ozdemir, 1997; Egli and Lowrie 2002; Egli 2006 on SD; Moskowitz et al., 1993; Till et al., 2011).

$M_r/M_s$ vs $\chi_{ARM}/M_s$

Lascu et al. (2010) proposed a method, which allows for the quantification of several magnetic parameters of a sediment at once. They use $M_s$ as a proxy for the total ferrimagnetic concentration of a sample. Further, similarly to the Day et al. (1977) plot, their approach utilizes the remanence ratio to estimate the domain state. The ratio $\chi_{ARM}/M_s$ is sensitive to both domain state (Maher, 2007; Egli 2006) and to magnetostatic interactions (Sugiura 1979). The use of $\chi_{ARM}$ as a way to quantify interactions was experimentally validated: highly interacting single domain magnetite in the teeth of chitons (marine molluscs), show low $\chi_{ARM}$, while well separated particles of similar size have much higher values. Finally, the remanence ratio is plotted against the $\chi_{ARM}/M_s$ ratio. By creating synthetic mixtures of MD, PSD, and SD magnetites Lascu et al. (2010) were able to calculate mixing lines that correspond well with the experimental data.

**Other Biplots**

Oldfield et al. (2009) describe other ratios that are useful to quantify magnetic grain-size within the SD-SP...
Figure 10. Sugiura 1979. ARM acquisition as a function of bias field (where $P_{\text{ARM}}$ denotes $\text{ARM}(H)/\text{SIRM}$). Particle interactions increase from specimens 1 (SD particles) to 6 (PSD). ARM acquisition becomes more linear as ARM decreases.

size range: $\chi_{\text{ARM}}/\chi_{\text{lf}}$ increases with magnetic grain-size, provided that the mean grain-size is within or below the stable single domain (SD) size range (i.e. grain diameters less than ~0.1 μm (Maher, 1988; Oldfield, 1994, 2007)); $\chi_{\text{ARM}}/\chi_{\text{fd}}$ increases with increased grain-size within the SP to SD size ranges (Oldfield, 1994, 2007). Plotting one quantity versus the other results in a useful bi(logarithmic) plot that helps constrain the finer magnetic grains. Figure 12 shows such a plot for loess/paleosol samples from the Chinese loess plateau, soils, and lake and marine sediments containing biogenic magnetite. Stable SD grain-sizes plot to the upper right and grain-size decreases towards the origin.

$\chi_{\text{ARM}}/\text{IRM}_{100\text{mT}}$ versus $\chi_{\text{fd}}/\%$

Maher and Thompson (1992) utilized a bivariate plot to also quantify magnetic granulometry of loess and paleosol samples. On the ordinate axis, the $\chi_{\text{ARM}}/\text{IRM}_{100\text{mT}}$ ratio increases for decreasing grain-sizes, from MD to SD. $\chi_{\text{fd}}/\%$ increases with decreasing grain-size, reaching a maximum at the SD/SP threshold. Note that the samples used contained hematite, and are therefore offset with respect to pure magnetite powders (which in turn are likely offset towards lower $\chi_{\text{ARM}}/\text{IRM}_{100\text{mT}}$ because of particles clumping …) (Fig. 13).

Magnetic Mineralogy tests

Attempts to determine the magnetic mineralogy from combinations of the magnetic properties described here and in the previous installment of this series of articles on rock-magnetic tests (IRMQ 27 (4)) have been performed by qualitatively evaluating biplots and choosing appropriate ratios. Following Thompson and Oldfield (1986), Peters and Thompson (1998) plot the ratio of SIRM/$\chi_{t}$ (units of A/m) versus a coarse estimate of coercivity of remanence $B_{c}$ (units of mT) (Fig. 14a). The plot defines somewhat overlapping distributions for different minerals, around which the authors drew irregular polygons in an attempt to define the occurrence of the different mineralogies (not shown). To better separate the distribution of pyrrhotite from those of magnetite, titanomagnetite and greigite, they plot SIRM/$\chi_{t}$ versus ARM$_{40\text{mT}}$/SARM (where the SARM was acquired in 99 mT fields, and both ARMs using a bias field of 0.1 mT). The ARM imparted over 40 mT was chosen because it was empirically determined by the authors that it best separated the two mineral groups (Fig. 14b). Likewise, they plotted a 100 mT backfield IRM over SIRM ration (IRM$_{100\text{mT}}$/SIRM) versus ARM$_{40\text{mT}}$/SARM to further distinguish greigite from magnetite, titanomagnetite and pyrrhotite (Fig. 14c).

Peters and Dekkers (2003) subsequently extended the
work of Thompson and Oldfield (1986), and providing additional data from other magnetic minerals (goethite and maghemite) they regenerated the plot of SIRM/χ\textsubscript{LF} (which they refer to as σ\textsubscript{RS}, units of A/m) versus coercivity of remanence (B\textsubscript{cr}, units of mT). For the latter, however, they used the mean coercivity distribution derived from the unmixing of an IRM acquisition curve, which they refer to as (B\textsubscript{cr})\textsubscript{CR'}, instead of the B\textsubscript{cr} determined from a back-field curve (Fig. 15).

In a future installment we will present rock-magnetic tests performed as a function of temperature. We leave you with a summary table of the symbols and the SI units of measure for the low-field magnetic properties described in this article (on the next page).

References

Figure 13. χ\textsubscript{ARM}/IRM\textsubscript{40mT} ratio versus χ\textsubscript{fd} % (Maher and Thompson, 1992) for samples from the Chinese Loess Plateau (containing magnetite and hematite), and pure magnetite powders of different sizes.

Figure 14. a) SIRM/χ\textsubscript{LF} versus coercivity of remanence B\textsubscript{cr} for a collection of magnetic minerals; b) SIRM/χ\textsubscript{LF} versus ARM\textsubscript{40mT}/SARM empirically determined to separate the distribution of pyrrhotite from those of magnetite, titanomagnetite and greigite; c) ARM\textsubscript{40mT}/SARM to further distinguish greigite from magnetite, titanomagnetite and pyrrhotite (Peters and Thompson, 1998).

Figure 15. SIRM/χ\textsubscript{LF} versus coercivity of remanence B\textsubscript{cr} for a collection of magnetic minerals (Peters and Dekkers 2003).
<table>
<thead>
<tr>
<th>Property</th>
<th>Units</th>
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<tr>
<td>Volume susceptibility, $k$</td>
<td>Dimensionless (typically expressed as $\mu$SI = 10^{-6} SI)</td>
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<tr>
<td>Mass susceptibility, $\chi$</td>
<td>m$^3$/kg</td>
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<tr>
<td>In-phase susceptibility, $k'$ or $\chi'$</td>
<td>$\mu$SI or m$^3$/kg</td>
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<tr>
<td>Out of phase susceptibility, $k''$ or $\chi''$</td>
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<tr>
<td>Frequency dependent susceptibility, $k_f$ or $\chi_{fd}$</td>
<td>$\mu$SI or m$^3$/kg</td>
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<td>Percent frequency dependent susceptibility, $k_{f%}$ or $\chi_{f%}$</td>
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<td>Low frequency susceptibility, $k_L$ or $\chi_L$</td>
<td>$\mu$SI or m$^3$/kg</td>
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<tr>
<td>High frequency susceptibility, $k_{HF}$ or $\chi_{HF}$</td>
<td>$\mu$SI or m$^3$/kg</td>
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<tr>
<td>Low field susceptibility, $k_{lf}$ or $\chi_{lf}$</td>
<td>$\mu$SI or m$^3$/kg</td>
</tr>
<tr>
<td>High field susceptibility, $k_{HF}$ or $\chi_{HF}$</td>
<td>$\mu$SI or m$^3$/kg</td>
</tr>
<tr>
<td>Anhysteretic Remanent Magnetization, $ARM$</td>
<td>Am$^3$/kg</td>
</tr>
</tbody>
</table>

| Susceptibility of ARM, $\chi_{ARM}$                          | $\mu$SI or m$^3$/kg    |

Summary of magnetic properties, symbols and units covered in this article.

Peters, C., and M. J. Dekkders (2003), Selected room temperature magnetic parameters as a function of mineralogy, concentration and grain size, Physics and Chemistry of the Earth, Parts A/B/C, 28(16), 659-667, doi: https://doi.
Quarterly

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