

The IRM Quarterly

Winter 2000-2001, Vol. 10, No. 4
Institute for Rock Magnetism

A cautionary note

On interpreting frequency-dependence
of susceptibility solely in terms of
superparamagnetism

or

Two ways to be wrong

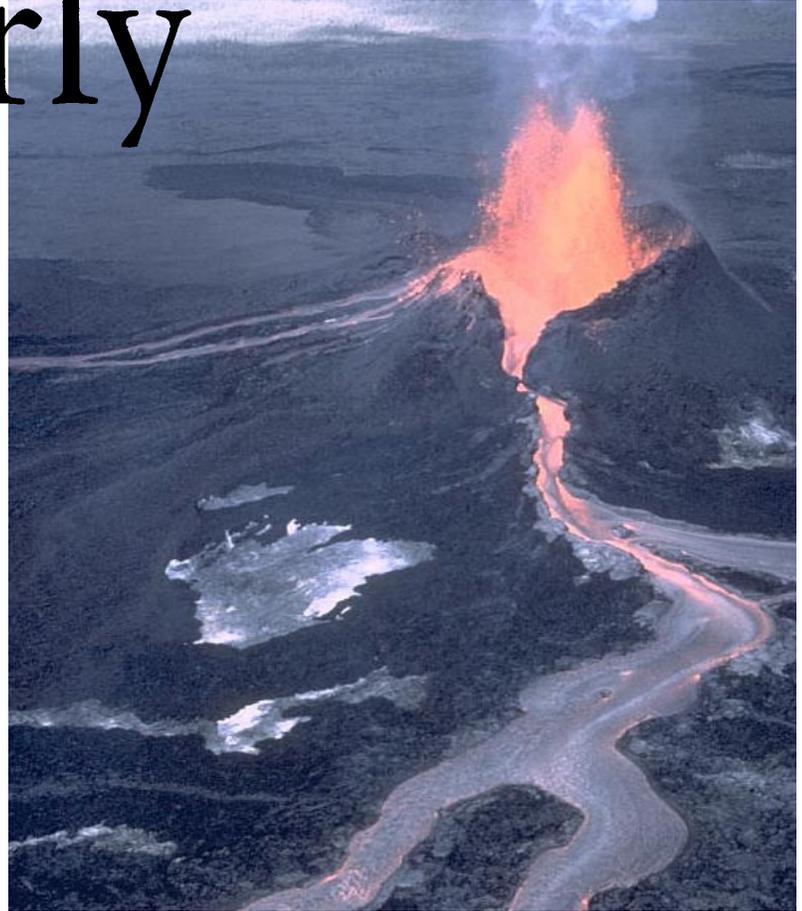
Helga de Wall
*Geologisches
Institut,
Universität
Heidelberg, Ger-
many*

**Horst-Ulrich
Worm**
*Magnon Interna-
tional, Dassel,
Germany*

Magnetic susceptibility measurements on rocks can serve a wide range of purposes: The determination of induced magnetizations for anomaly interpretations, magnetic fabric studies, simple estimates of ferrimagnetic mineral contents, and, in conjunction with other magnetic parameters, rock magnetic granulometry studies. Since susceptibility measurements are almost exclusively carried out in AC fields of various amplitudes, the frequency and field dependence of susceptibility of relevant minerals must be of concern for the respective studies.

Early studies in the 1960s and '70s dealt mostly with magnetite, and it must have been a comforting result that this very common mineral does not exhibit a noticeable change in susceptibility up to fairly high fields and frequencies up to > 1 kHz - with the well known exception of grains close to the superparamagnetic / stable single domain (SP/SSD) threshold.

It was much later that other natural ferrimagnets have been studied in some detail. For pyrrhotite a strong field dependence of susceptibility has been measured, where the onset on non-linear field dependence may be as low as 5 μT (~40 A/m), depending mainly on grain size (Worm et al., 1993). A quite similar field dependence has been measured on titanomagnetites with intermediate Ti-composition, but the effect decreases with decreasing Ti-content (Jackson et al., 1998; deWall, 2000). And hematite, among its other recently-discovered



Basalt flows from Kilauea and the rest of Hawaii contain titanomagnetites of varying compositions, which in turn contain a few surprises in their susceptibility behavior.

surprising properties, exhibits an AC field dependence very similar to those of pyrrhotite and titanomagnetite (Kletetschka, 2000).

Worm and coworkers (1993) also studied the frequency dependence of susceptibility (χ_{fd}) on pyrrhotite and magnetite samples, experimentally and theoretically. For mm-sized pyrrhotite samples the in-phase susceptibility decreases noticeably only above \gg 5 kHz due to eddy current effects. The frequency response for a mm-sized magnetite crystal is essentially flat up to 20 kHz because of its lower electrical conductivity. Because theory predicts the eddy current effects to decrease with decreasing size, it has been considered safe to conclude that 'regular' rock specimens containing sub-millimeter pyrrhotite or magnetite grains should exhibit no frequency dependence of susceptibility in the frequency domain of all commercial instruments. And because the conductivity of titanomagnetites decreases with

increasing Ti-content it also appeared safe to predict a flat frequency response of the susceptibility. However, nobody measured it.

The frequency dependence of susceptibility of basalts

It was χ_{fd} measurements on Hawaiian basalts that sparked the above mentioned study on the field dependence of susceptibility of titanomagnetites. The samples possessed a seemingly strong frequency dependence of susceptibility. However, the measurements were performed with a Sapphire instrument SI2, which employs different field strengths of 90 and 300 A/m for two different frequencies. The apparent frequency dependence has convincingly been explained to be truly a field dependence by Jackson and coworkers (1998).

During a later study on basalts from the Hawaiian Deep Drillhole HSDP II

susceptibility

continued on page 6...

Visiting Fellows' Reports

Magnetic properties of loess-palaeosol deposits from the Russian plain for particle-size estimations

Elena Virina

Moscow State University
virina@paleo.geogr.msu.su

The two main goals of my studies in IRM were: a) to improve the detection and characterization of pure magnetite in palaeosols by features in the low-temperature dependence of H_C , SIRM and especially magnetization in magnetic fields about 100 mT, which may show anomalous behavior near magnetite's isotropic point or Verwey transition; b) to reveal particle size distributions on the basis of IRM temperature dependence and also M_s temperature dependence. The last possibility is based on Langevin theory, which predicts that in strong magnetic fields at low temperatures, significant decay of magnetization may be caused only by particles in the nm size range.

The first thesis of my studies is based on previous results achieved almost 30 years ago. At that time, for one sample of the clay fraction from an ancient palaeosol from Moldova, we observed a strong magnetic peak near the magnetite isotropic point on $M(T)$ curves in magnetic fields of 980 and 8500 Oe. Starting measurements here, I very soon realized clearly that my previous results were not reproducible: they were caused by contamination or an experimental artifact. So my first result was discouraging. Yet here I saw that magnetite's Verwey transition may be observed in sediments and loess-palaeosol deposits (e.g., in the work of E.A.Oches & S.K. Banerjee).

I measured magnetic properties for almost 30 samples collected from loess and palaeosols at 4 sites located at South-West (Ukraine and Moldova), Center and South East parts of the Russian plain. Surprisingly, there is little evidence of the presence of unaltered magnetite: high-temperature thermomagnetic curves are highly irreversible, and low-temperature measurements show no indications of the magnetite isotropic point or Verwey transition. The absence of inflections on low-temperature curves is most probably caused by oxidation of magnetite.

We are more optimistic about particle-size estimations. We were most interested in observing the lower end of this distribution and estimating the minimal limit. For this purpose loss of remanence at low temperatures may be used and also loss of magnetization in strong magnetic fields and at low temperatures.

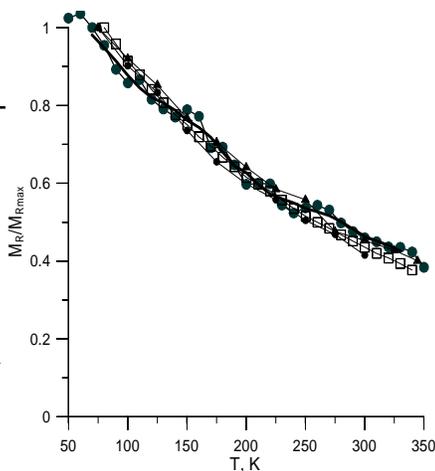


Fig. 1. Saturation remanence determined from hysteresis loops as a function of temperature. All samples show very similar behavior. Clay fractions from: recent chernozem; loess; chernozem-like paleosol; parabraunerde; and a red paleosol.

In Fig.1 loss of remanent magnetization SIRM, defined from hysteresis loops during heating, is shown for several samples. These samples are clay fractions from different palaeosols at the Novaya Etuliya section (Moldova). The SIRM behavior is similar for different samples. (We used 75 K as the minimum temperature for particle size estimations because at lower temperatures some signs of magnetic ordering in paramagnetic minerals were observed from $M_p(T)$ curves). The loss of remanence is almost linear and reaches 60-70% of $SIRM_{max}$ at 350 K. The same results, but more accurate, were defined by $SIRM(T)$ measured by MPMS. We can conclude that particle-size distribution obtained by this temperature dependence is close to uniform. Comparison with the unblocking-temperature spectra for Yucca Mountain tuff (Worm & Jackson)

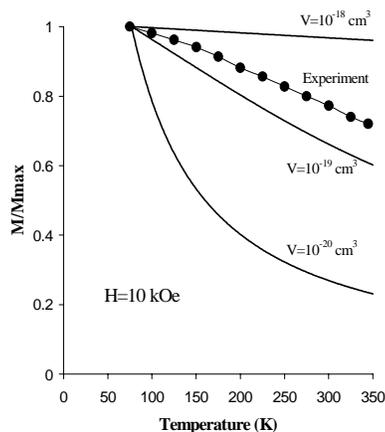


Fig. 2. Comparison of measured temperature dependence of strong-field magnetization with theoretical curves for magnetite grains of different size.

suggests that this distribution spans from $0.5-6^{-24} \text{ m}^3$, i.e. the lowest particle sizes are of nanometer range.

Comparatively large loss of saturation magnetization also indicates the presence of nanoparticles. In Fig 2 theoretical curves based on Langevin theory for assemblies of different volumes, corrected for temperature dependence of spontaneous magnetization of magnetite, are shown together with typical experimental curves. We can conclude from comparison of these curves that nanoparticles may have a significant role. (Note that the temperature range here was limited by 75 K as a minimum because of the well known paramagnetic signal problem).

At the last moment we also measured frequency-dependence of magnetic susceptibility through the low-temperature interval for a sample of palaeosol with the highest peak of magnetic susceptibility. Here for the first time we can see some signs of blocking at the lowest temperatures and also wide maxima near room temperature, where the frequency dependence of susceptibility reaches about 30%. (Frequency range was 1 – 1000 Hz) (Fig.3).

So, we can conclude, that particle-size distribution in loess-palaeosol deposits is really very wide and spans an interval at least from SSD to the smallest particles of nm range, with maxima near the room temperature SSD-SP threshold. It allows excluding the supposition about magnetotactic bacteria as a major source of magnetic enhancement in soils.

At last, I have to say, that I was very impressed with the lab, the precious instruments and accessories for sample preparation. But I was mostly impressed by the very warm and kindly people and atmosphere of the lab. I am grateful to all for their patience and help.

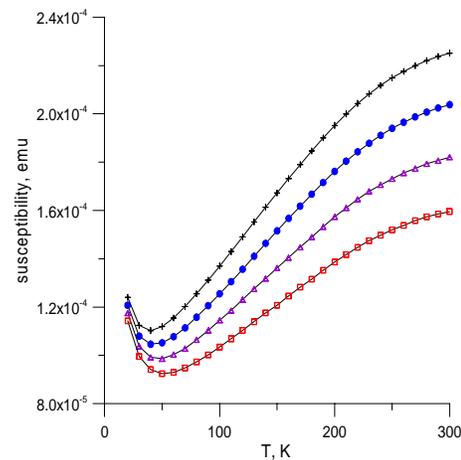
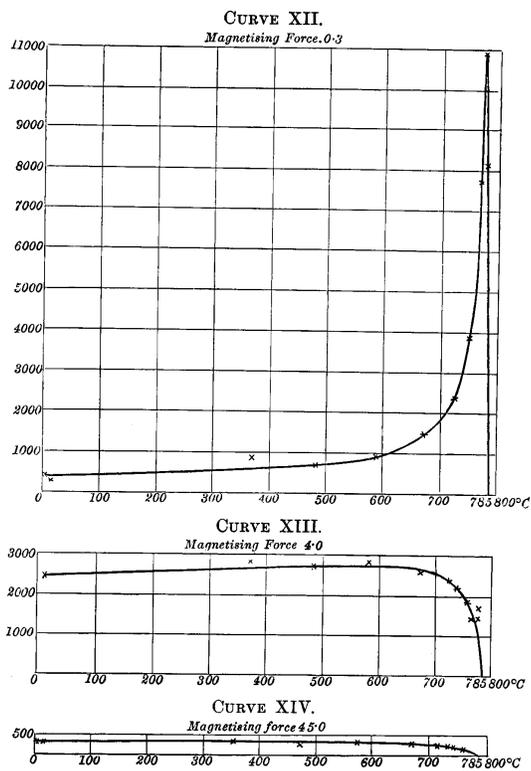


Fig. 3. Temperature dependence of low-field susceptibility at different frequencies. Clay fraction of paleosol from N. Etuliya



The first documented Hopkinson peak: "In Curves XII, XIII and XIV, the abscissæ are temperatures, and the ordinates are induction ÷ magnetising force, called by Sir William Thomson the permeability, and usually denoted by μ . These curves correspond to constant magnetising forces of 0.3, 4.0, 45.0. Looking at the curve for 0.3, we see that the permeability at the ordinary temperature is 367; that as the temperature rises the permeability rises slowly, but with an accelerated rate of increase; above 681° C. it increases with very great rapidity, until it attains a maximum of 11,000 at a temperature of 775° C. Above this point it diminishes with extreme rapidity, and is practically unity at a temperature of 786° C."

Current Abstracts

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 abstracts are culled from INSPEC (© Institution of Electrical Engineers), Geophysical Abstracts in Press (© American Geophysical Union), and The Earth and Planetary Express (© Elsevier Science Publishers, B.V.), after which they are subjected to Procrustean editing and condensation 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 5200 references, is available free of charge. Your contributions both to the list and to the Abstracts section of the IRM Quarterly are always welcome.

Anisotropy

Abelson, M., Baer, G., and Agnon, A., 2001, **Evidence from gabbro of the Troodos ophiolite for lateral magma transport along a slow-spreading mid-ocean ridge:** *Nature*, v. 409, no. 6816, p. 72-5.

Evidence for broad-scale along-axis flow has been frozen into the gabbro of the Troodos ophiolite, which spans nearly 20 km of a segment of a fossil spreading axis, near a ridge-transform intersection. The magnetic fabric at 20 sites, and the petrographic fabric at 9 sites, suggest along-axis magma flow for much of the gabbro suite. This indicates that redistribution of melt occurred towards the segment edge in a large depth range of the oceanic crust.

Biogeomagnetism

Hanzlik, M., Heunemann, C., Holtkamp-Rötzler, E., Winklhofer, M., N., P., and Fleissner, G., 2000, **Superparamagnetic magnetite in the upper beak tissue of homing pigeons:** *BioMetals*, v. 13, p. 325-331.

The upper-beak skin of homing pigeons has previously been shown to contain nerves sensitive to changes of the ambient magnetic field. We localized Fe^{3+} clusters in the subcutis and identified the material by TEM as aggregates of magnetite nanocrystals with grain sizes between 1 and 5 nm. The particles form clusters of 1-3 μm diameter. Low-temperature magnetic measurements confirm the presence of fine-grained SP particles in the tissue. Neither electron-microscopic nor magnetic measurements revealed any SSD magnetite in the upper-beak tissue.

Carriers and Origins of NRM

Katari, K., Tauxe, L., and King, J., 2000, **A reassessment of post-depositional remanent magnetism: preliminary experiments with natural sediments:** *Earth and Planetary Science Letters*, v. 183, no. 1, p. 147-60.

Two kinds of laboratory experiments were designed to investigate whether post-depositional reorientation of magnetic particles is likely to occur in nature. The first monitored changes in the magnetization of natural sediments in response to changing laboratory fields; results were inconsistent with post-depositional reorientation of magnetic particles. In the second experiment, live worms bioturbated sediments in a multi-core tube with the original sediment-water interface intact. Remagnetization was only observed in samples taken from a mound of fecal pellets formed by resuspension and redeposition at the surface. These results do not support the hypothesis that post-depositional reorientation occurs in natural, undisturbed sediments below the sediment-water interface.

Shau, Y. H., Torii, M., Horng, C. S., and Peacor, D. R., 2000, **Subsolidus evolution and alteration of titanomag-**

netite in ocean ridge basalts from Deep Sea Drilling Project/Ocean Drilling Program Hole 504B, Leg 83: implications for the timing of magnetization: *Journal of Geophysical Research*, v. 105, no. B10, p. 23635-49. Magnetite formed by two different processes: (1) oxidation-exsolution, true exsolution, and hydrothermal alteration, and (2) oxidation-exsolution, a second stage of oxidation-exsolution, and hydrothermal alteration. The primary titanomagnetite (TM60-70) that crystallized from the melt thus evolved to end-member magnetite coexisting with titanite (sphene), kassite, ulvospinel (TM-87), and ilmenite on a submicroscopic scale. The natural remanent magnetization of the basalts from the transition zone and upper sheeted dikes is characteristic of chemical remanent magnetizations that were acquired when titanomagnetite altered, in part, to magnetite during subsolidus cooling and hydrothermal alteration close to the ridge axis.

Data Analysis

Roberts, A. P., Pike, C. R., and Verosub, K. L., 2000, **First-order reversal curve diagrams: a new tool for characterizing the magnetic properties of natural samples:** *Journal of Geophysical Research*, v. 105, no. B12, p. 28461-75. A set of partial hysteresis curves known as first-order reversal curves (FORCs) are transformed into contour plots (FORC diagrams) of a two-dimensional distribution function, which provides information about switching fields and local interaction fields for the assemblage of magnetic particles in a sample. SP, SSD, and MD grains, as well as magnetostatic interactions, all produce characteristic and distinct manifestations on a FORC diagram. These diagrams provide more detailed information about the magnetic particles in a sample than standard interpretational schemes which employ hysteresis data.

Environmental Magnetism

Dearing, J. A., Hannam, J. A., Anderson, A. S., and Wellington, E. M. H., 2001, **Magnetic, geochemical and DNA properties of highly magnetic soils in England:** *Geophysical Journal International*, v. 144, no. 1, p. 183-96.

The ferrimagnetic component of highly enhanced surface soils is dominated by SP grains with a minor proportion of SSD/PSD grains that may derive from magnetosomes and magnetic inclusions. DNA screening of the soils by polymerase chain reaction (PCR) shows that the concentration of viable magnetotactic bacteria is too low (normally $<10^2$ bacteria g^{-1}) to explain the high concentrations of ferrimagnetic minerals observed. Microcosm experiments show that the destructive effects of waterlogging on secondary ferrimagnetic mineral formation are rapid and associated with significant changes in bacterial

abstracts

continued on page 4...

populations. The combined results are consistent with previous findings that ferrihydrite may be an important precursor of bacterially mediated magnetite in strongly magnetic temperate soils—a process driven by the rate of Fe flux to the biologically active surface soil.

Deng, C. L., Zhu, R. X., Verosub, K. L., Singer, M. J., and Yuan, B., 2000, **Paleoclimatic significance of the temperature-dependent susceptibility of Holocene loess along a NW-SE transect in the Chinese loess plateau:** *Geophysical Research Letters*, v. 27, no. 22, p. 3715-18.

Samples of modern dune sand, pristine loess and present-day loess were studied using temperature-dependence of susceptibility (TDS) before and after CBD treatment as well as X-ray diffraction. The XRD analyses demonstrate that magnetite and hematite both exist in the Chinese loess-paleosol sequence and its modern source area, but the TDS measurements show that magnetite is the predominant contributor to magnetic susceptibility. Maghemite is present in the pristine loess and the present-day loess due to pedogenesis. The pedogenic processes that produce the maghemite are closely linked to paleoclimate and, for this transect, precipitation appears to be the most important climatic variable.

Stoner, J. S., Channell, J. E. T., Hillaire-Marcel, C., and Kissel, C., 2000, **Geomagnetic paleointensity and environmental record from Labrador Sea core MD95-2024, global marine sediment and ice core chronostratigraphy for the last 110 kyr:** *Earth and Planetary Science Letters*, v. 183, no. 1, p. 161-77. High-resolution paleointensity and O isotope records from the North Atlantic, Mediterranean, Somali Basin, sub-Antarctic South Atlantic and the relative flux of ^{10}Be in Vostok (Antarctic) ice core are used to derive a common. The resulting correlation circuit places the ice core and marine records on a common GISP2 official chronology, which indicates discrepancies as high as 5 kyr between the GISP2 and SPECMAP time scales. It further demonstrates that Laurentide ice sheet instabilities in the Hudson Strait area are synchronous with cooling in the Greenland Summit ice cores and warming in the sub-Antarctic South Atlantic and in the Vostok ice core. Geomagnetic field intensity shows common global variance at millennial time scales which cannot be attributed to climatic contamination of the paleointensity records.

Extraterrestrial Magnetism

Weiss, B. P., Kirschvink, J. L., Baudenbacher, F. J., Vali, H., Peters, N. T., Macdonald, F. A., and Wikswow, J. P., 2000, **A low temperature transfer of ALH84001 from Mars to Earth:** *Science*, v. 290, no. 5492, p. 791-5. Images of the magnetic field of martian meteorite ALH 84001 reveal a spatially heterogeneous pattern of magnetization associated with fractures and rock

fragments. Heating the meteorite to 40° C reduces the intensity of some magnetic features, indicating that the interior of the rock has not been above this temperature since before its ejection from the surface of Mars. Because this temperature cannot sterilize most bacteria or eukarya, these data support the hypothesis that meteorites could transfer life between planets in the Solar System.

Magnetic Field Records and Paleointensity Methods

Carlut, J., and Kent, D. V., 2000, **Paleointensity record in zero-age submarine basalt glasses: testing a new dating technique for recent MORBs:** *Earth and Planetary Science Letters*, v. 183, no. 3, p. 389-401.

Thellier-Thellier paleointensity experiments were conducted on a collection of glasses from three very recent submarine axial flows. One from the East Pacific Rise yields a well-defined mean value of $35.6 \pm 1 \mu\text{T}$, and comparison with published field models for the past 400 yr suggests an eruptive date estimated between 1880 and 1950 A.D. This is consistent with other evidence and thus demonstrates the use of paleointensity as a dating tool for very young MORBs. Samples from the Juan de Fuca ridge results show a more erratic pattern ($\pm 30\%$), due to large local crustal magnetic anomalies in this area. The paleointensity dating tool is anticipated to be especially efficient to investigate the volcanic cyclicity along the EPR axis during the last several hundred years.

Cottrell, R. D., and Tarduno, J. A., 2000, **In search of high-fidelity geomagnetic paleointensities: a comparison of single plagioclase crystal and whole rock Thellier-Thellier analyses:** *Journal of Geophysical Research*, v. 105, no. B10, p. 23579-94.

A Thellier-Thellier study of single plagioclase crystals from a mid-Cretaceous (113-115 Ma) basalt flow of the Rajmahal Traps (northeastern India) yields a paleofield value of $65.1 \pm 5.3 \mu\text{T}$ ($n=15$). Data from whole rock samples overlap these values but are on average lower. The authors attribute the difference in this study to the preferential growth of new magnetic minerals in the whole rock samples, resulting in an enhanced acquisition of TRM and shallowing of the slope of NRM-lost versus TRM-gained curves.

Goguitchaichvili, A., Alva-Valdivia, L. M., Urrutia-Fucugauchi, J., Morales, J., and Ferrari, L., 2000, **Absolute paleointensity results from the Trans-Mexican Volcanic Belt: implications for the late Miocene geomagnetic field strength:** *Geophysical Journal International*, v. 143, no. 3, p. 977-84.

14 samples from five individual lava flows yielded reliable paleointensity estimates, with flow-mean virtual dipole moments ranging from 5.6 to $8.5 \times 10^{22} \text{ A m}^2$. The VDM values obtained are significantly higher than those recently reported from

Miocene submarine basaltic glasses but are very similar to the mean reported from the Steens Mountain continental basaltic flows. More data are needed in order to better understand the behaviour of the Miocene geomagnetic field strength and to constrain the transition mode between the Mesozoic low and Neogene high field.

Gorgorza, C. S. G., Sinito, A. M., Vilas, J. F., Vilas, J. F., Creer, K. M., and Nunez, H., 2000, **Geomagnetic secular variations over the last 6500 years as recorded by sediments from the lakes of south Argentina:** *Geophysical Journal International*, v. 143, no. 3, p. 787-98.

Regional secular variation curves for the last 6700 calendar years for southwestern Argentina (about 41° S, 71° 30' W) have been constructed by stacking palaeomagnetic data from lake sediment cores. Spectral analysis of declination (D) and inclination (I) at the same time shows major peaks at periodicities of about 3400, 1600 and 1200 yr for both parameters. Patterns of circularity during the last 6700 yr appear to recur with a near constant interval of about 2000 yr. Both open and closed loops are associated with clockwise and counter-clockwise circularity.

Roberts, A. P., and Lewin-Harris, J. C., 2000, **Marine magnetic anomalies: evidence that 'tiny wiggles' represent short-period geomagnetic polarity intervals:** *Earth and Planetary Science Letters*, v. 183, no. 3, p. 375-88.

A new sedimentary palaeomagnetic record from the North Pacific shows two short, but clearly resolvable, polarity zones, in addition to a probable geomagnetic excursion, within Chron C5n.2n (9.92-10.95 Ma), where three 'tiny wiggles' have been reported on marine magnetic anomaly profiles. Relative palaeointensity data indicate that the field collapsed prior to and during the reversals but that it recovered to higher field intensities within the polarity intervals before collapsing to low values at the succeeding polarity transition. This indicates that some 'tiny wiggles' represent real short-period geomagnetic polarity intervals, while others may represent geomagnetic excursions. The existence of such short polarity intervals confirms the predictions of statistical analyses of geomagnetic reversal frequency and indicates that 'tiny wiggles' represent the maximum resolution of geomagnetic polarity intervals in marine magnetic anomaly records.

Selkin, P. A., Gee, J. S., Tauxe, L., Meurer, W. P., and Newell, A. J., 2000, **The effect of remanence anisotropy on paleointensity estimates: a case study from the Archean Stillwater Complex:** *Earth and Planetary Science Letters*, v. 183, no. 3, p. 403-16.

Magnetic anisotropy can have particularly disastrous consequences for paleointensity experiments if the anisotropy is unrecognized and if its effects remain uncorrected. For an anorthosite sample with a well-developed magmatic foliation, the effect of the remanence fabric on paleointensity determinations is significant: paleointensities estimated by the method

of Thellier and Thellier range from 17 to 55 μT for specimens magnetized in a field of 25 μT . A correction based on the remanence anisotropy yields a paleointensity estimate of 32 μT for the Stillwater Complex (adjusted for the effects of slow cooling).

Magnetic Microscopy and Spectroscopy

Bodker, F., and Morup, S., 2000, **Size dependence of the properties of hematite nanoparticles**: *Europhysics Letters*, v. 52, no. 2, p. 217-23. Samples of nanoparticles of hematite ($\alpha\text{-Fe}_2\text{O}_3$) with average size between 6 and 27 nm have been studied by use of Mössbauer spectroscopy. The superparamagnetic relaxation was analysed on the basis of the Néel-Brown expression for the relaxation time, $\tau = \tau_0 \exp[KV/kT]$. It was found that the value of τ_0 increases with increasing particle volume, V, whereas the magnetic anisotropy energy constant, K, decreases. The electric quadrupole interaction, the isomer shift and the magnetic hyperfine field extrapolated to 0 K were found to be essentially independent of particle size.

Egli, R., and Heller, F., 2000, **High-resolution imaging using a high-Tc superconducting quantum interference device (SQUID) magnetometer**: *Journal of Geophysical Research*, v. 105, no. B11, p. 25709-27.

The stability problems of a commercially available high-Tc SQUID magnetometer have largely been solved by improving the magnetic shielding and reducing the noise due to turbulent boiling of liquid nitrogen. Magnetizations as weak as $5 \cdot 10^{-4}$ A/m can now be discriminated with a resolution of 1 mm. A software package has been developed for downward continuation of the field data and inversion for the vertical magnetization component. System performance is demonstrated for a synthetic sample and three rock samples with very different magnetic properties.

Rusanov, V., Gilson, R. G., Lougear, A., and Trautwein, A. X., 2000, **Mössbauer, magnetic, X-ray fluorescence and transmission electron microscopy study of natural magnetic materials from speleothems: haematite and the Morin transition**: *Hyperfine Interactions*, v. 128, no. 4, p. 353-73.

The NRM of a haematite-bearing stalagmite sample was substantially restored and/or conserved on rewarming after cooling below the Morin transition temperature. Mössbauer measurements indicate the presence of two types of haematite; the majority is very fine, partially superparamagnetic, and does not undergo a Morin transition above liquid nitrogen temperature. Surface mine haematite samples have also been studied. Special attention is paid to the "irreversible" Morin transition in large enough (>20 nm) haematite particles and the possible loss of NRM.

Thorpe, A. N., Senftle, F. E., Holt, M.,

Grant, J., Lowe, W., Anderson, H., Williams, E., Monkres, C., and Barkett, A., 2000, **Magnetization, micro-X-ray fluorescence, and transmission microscopy studies of low concentrations of nanoscale Fe_3O_4 particles in epoxy resin**: *Journal of Materials Research*, v. 15, no. 11, p. 2488-93. Magnetization measurements, TEM, and high-resolution $\mu\text{-XRF}$ using a synchrotron radiation source were used to examine agglomerates of 10-nm Fe_3O_4 particles in epoxy. At low concentrations (<0.5%) the magnetite particles, although closely packed in the agglomerates, did not interact magnetically. The $\mu\text{-XRF}$ results were compatible with spherical agglomerates ranging in size from 100 to several thousand nanometers, as observed in TEM measurements. At smaller step scans the resolution could be significantly improved. The synchrotron $\mu\text{-XRF}$ method could probably be used to detect particles in amounts as low as 10^{-16} g.

Wright, J. P., Bell, A. M. T., and Attfield, J. P., 2000, **Variable temperature powder neutron diffraction study of the Verwey transition in magnetite Fe_3O_4** : *Solid State Sciences*, v. 2, no. 8, p. 747-53.

High-resolution neutron diffraction data for a sample of polycrystalline Fe_3O_4 , collected at 60 K, are fitted using a simple rhombohedral distortion of the cubic unit cell. This model accounts for all of the peak splittings that occur upon cooling through the Verwey transition temperature (Tv). Lower resolution data collected between 2 and 280 K are fitted using the same model, which gives $\text{Tv} = 110 \pm 5$ K.

Magnetization Processes

Robion, P., and Borradaile, G. J., 2001, **Stress remagnetization in pyrrhotite-calcite synthetic aggregates**: *Geophysical Journal International*, v. 144, no. 1, p. 96-104.

Experimental deformation of multidomain pyrrhotite-calcite synthetic aggregates in a triaxial rig produced new components of magnetization, parallel to the direction of the pressure vessel field, in the coercivity fraction below 15 mT. The intensity of remagnetization ($M'-M_0$) increases with the applied differential stress. Bulk shortening is less than 8 per cent, thus grain rotation cannot explain selective remagnetization of the low-coercivity fraction.

Modeling and Theory

Newell, A. J., 2000, **The Lowrie-Fuller test: single-domain and micromagnetic theory**: *Earth and Planetary Science Letters*, v. 183, no. 1, p. 335-46. A new SD theory is presented that includes the effects of variations in grain shape and volume, applied stress and composition. One factor is varied at a time while the others are held fixed. If the variable is stress or grain shape, the AF demagnetization curves do not satisfy the Lowrie-Fuller

criterion for SD grains, as noted in earlier work. The same is true for variable composition if the magnetic hysteresis is controlled by shape anisotropy. Grain volume affects the AF demagnetization curve through thermal fluctuations. When the variable is volume, the sample passes the Lowrie-Fuller test. If the variable is grain orientation, the outcome depends on the distribution of orientations and the direction of the field. A highly anisotropic sample will pass the Lowrie-Fuller test in one direction and fail it in another. Thus, depending on the dominant variable, an ensemble of SD grains can pass or fail the Lowrie-Fuller test. However, a full analysis of the Lowrie-Fuller test must also take into account the critical sizes for transition from SD to non-SD properties. The SD theory will only apply to samples with grain volumes concentrated near the SP-SSD boundary. The shapes of their AF demagnetization curves will be determined mainly by the volume distribution. Therefore, they will satisfy the Lowrie-Fuller criterion for SD grains.

Synthesis and Properties of Magnetic Minerals

Bartkowska, J. A., Cisowski, J., Voiron, J., Heimann, J., Czaja, M., and Mazurak, Z., 2000, **Magnetization and magnetic susceptibility of kunzite**: *Journal of Magnetism and Magnetic Materials*, v. 221, no. 3, p. 273-7.

We have studied the high-field magnetization up to 14.5T and magnetic susceptibility in the temperature range 1.6-400 K of three different samples of natural crystals of kunzite, a variety of spodumene containing transition metal ions. It appears that the total magnetization and susceptibility consist of the paramagnetic contribution of magnetic ions (dominantly Mn^{2+}) and temperature-independent diamagnetic contribution of the spodumene matrix, equal to $-3.5 \cdot 10^{-7}$ emu/g.

Bonetti, E., Bianco, L. D., Signoretti, S., and Tiberto, P., 2001, **Synthesis by ball milling and characterization of nanocrystalline Fe_3O_4 and $\text{Fe}/\text{Fe}_3\text{O}_4$ composite system**: *Journal of Applied Physics*, v. 89, no. 3, p. 1806-15. Nanocrystalline Fe_3O_4 and a composite system constituted by nanocrystalline Fe and Fe_3O_4 have been synthesized by ball-milling commercial magnetite and an equimolar mixture of iron and magnetite powders. The physical parameters governing the milling process have been strictly controlled so as to achieve the nanocrystalline state of the precursor material and to avoid chemical reactions. X-ray diffraction and Mössbauer spectroscopy measurements have been carried out both on as-milled powders and on samples previously subjected to annealing treatments in the 100-600° C temperature range. The results, providing information on the structural and compositional features of the produced samples, are discussed in terms of structural disorder which is healed by subsequent annealing.

and basalts from the Riedheim dike, Hegau, Germany, Bartington bridges (in the laboratory Grubenhagen, GGA, Germany, and at Caltech, USA) have been employed by one of us (de Wall) in order to detect SP titanomagnetite grains. This instrument is specified to employ fields of 80 A/m at both frequencies of 470 Hz and 4.7 kHz, so that no field-dependent artefact should be produced. As a result, frequency dependences of susceptibility of up to 8% have been determined for some samples and it was first concluded that several of the basalt samples contain significant amounts of very fine grained titanomagnetite which were undetected by microscopy.

An unlikely correlation

Further measurements included the determination of Curie temperatures, and a surprising result was that the SP grain content is seemingly correlated with the Curie temperature and thus the Ti-content (Fig. 1). Therefore, the conspicuous SP grain presence has been further studied by time dependent isothermal remanent magnetization (TDIRM) acquisition measurements which should result in widely diverging IRM acquisition curves for single domain grain samples (Worm, 1999). However, the measured IRM curves are nearly identical for both acquisition times (Fig. 2), thus indicating multidomain rather than single domain grains.

With a modified Minikappa KLF-3 (Geofyzika, Brno), allowing susceptibility measurements at 30 and 300A/m and a constant frequency of 2 kHz, the field dependence of susceptibility of the samples has also been measured. Not unexpectedly, the field dependence (χ_{hd} = [$\chi_{300} - \chi_{30}$] / χ_{300}) also correlates with T_c and, therefore, χ_{hd} correlates with χ_{fd} (Fig. 3).

Hence the suspicion arose that the employed Bartington bridges do not exactly use identical field strengths at both frequencies. This has been confirmed by direct measurements on the

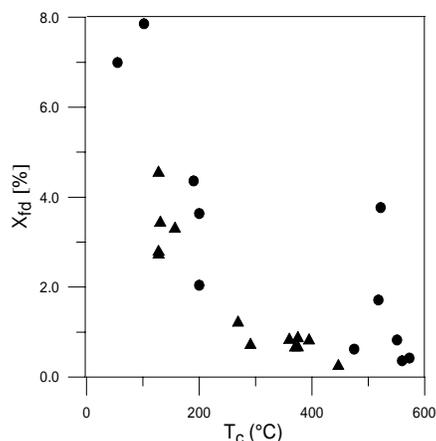


Fig. 1: Frequency-dependence (χ_{fd}) measured with Bartington instrument versus Curie temperature (T_c) of basalts from Hawaii (circles) and Riedheim, Germany (triangles).

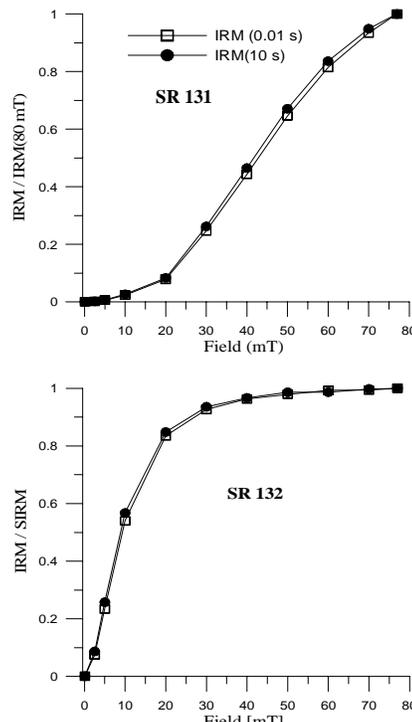


Fig. 2: Time dependent IRM acquisition of two HSDP basalts. SR131 has a Curie temperature $T_c = 551^\circ\text{C}$, and for SR132 $T_c = 102^\circ\text{C}$. Nearly identical IRM acquisition curves indicate multidomain grains.

instrument in Grubenhagen.

Instrument characteristics

The induced AC voltage in a pick-up coil depends linearly on the frequency, the strength of the (sinusoidal) field, the number of turns, and the 'flux' area. Hence, for determining the ratio of field intensities at two frequencies it is sufficient to measure the induced AC voltages, without having to know the exact number of turns and coil geometry. However, attention has to be paid to a possible frequency-dependence of the measuring voltmeter itself. Oscilloscopes generally show no frequency-dependence but have a lower resolution than digital voltmeters. We used a coil with approx. 700 turns and a diameter of 15 mm. We found for the Bartington bridge in Grubenhagen that the ratio of field intensities is $H_{lf} / H_{hf} = 1.07 (\pm 0.005)$, where $lf = 462$ Hz and $hf = 4.61$ kHz. It appeared to us that this decreased field amplitude at the higher frequency is solely responsible for the apparent frequency-dependence of susceptibility.

The finding of different field strengths should not be generalized for all Bartington instruments without further testing. However, at least the Bartington instrument at Caltech gave the same apparent χ_{fd} results. Bartington Ltd. itself did not have an answer to the question of different field strengths.

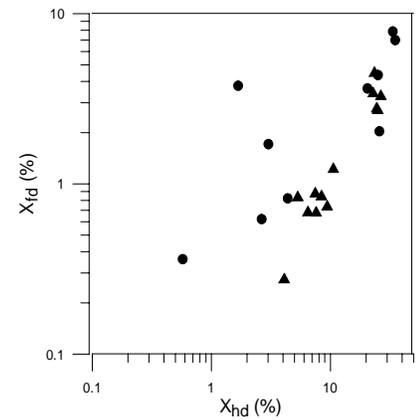


Fig. 3: Cross-plot of field dependence (χ_{hd}) versus frequency dependence (χ_{fd}) of susceptibility for basalts from Hawaii (triangles) and Hegau (circles). Measured with Bartington instrument (χ_{fd}) and modified Kappabridge (χ_{hd}).

An unexpected twist

Just a little while ago, our conclusion would have been: Blame Bartington for the apparent frequency dependence, the titanomagnetites are innocent. However, after careful measurements on the newly renovated and well-calibrated (thanks to Jim Marvin) Lakeshore susceptometer, we detected the unexpected: A noticeable gap between susceptibility values measured at 625 and 6 kHz, respectively (Fig. 4). Therefore there is a real frequency dependence, even without a significant SP presence. Notice that the susceptibility is also field dependent at around 80 A/m (the field strength of the Bartington instrument). The frequency dependence is approximately constant in low fields and it possibly increases towards higher fields.

And finally, it is true that the frequency dependence increases with increasing Ti-content (Fig. 5). For near-magnetite samples $\chi_{fd} \leq 0.5\%$, and the value approaches 6% for more Ti-rich samples; Bartington values are somewhat higher for low T_c samples because of the additional field-dependent effect.

Conclusions

As to the why – we don't know exactly. The decreased susceptibility at the higher frequency is certainly a result of viscous damping (\propto to velocity) of domain wall movement, and it is not eddy-current damping, because of the decreased damping in magnetite despite its higher electrical conductivity. It is rather intrinsic or relaxation damping, i.e. a retardation of the rotation of electron spins (Cullity, 1972). And Cullity wrote further 'The theories of intrinsic damping are difficult...' and therefore we won't

Susceptibility

continued on page 7...

Hopkinson, John

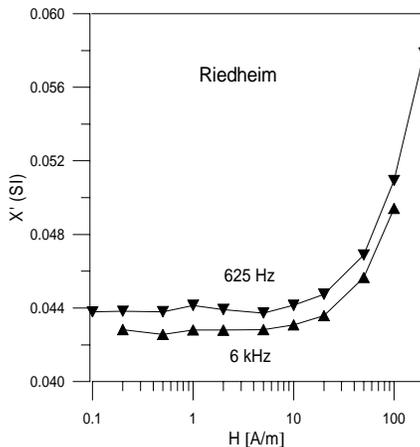
b. July 27, 1849, Manchester, England
d. Aug. 27, 1898, Petite Dent de Veisivi,
Switzerland

An electrical engineer by profession, Hopkinson carried out fundamental magnetic research in connection with his efforts to produce superior electrical generators. He was the first to precisely define the term "coercive force," and in 1885 discovered the Hopkinson Effect, the increase in susceptibility at temperatures just below the Curie point. The direct association of that magnetic transition with a change in heat capacity was also first noted by Hopkinson. He is further remembered for inventing the three-wire system for electricity distribution and for his work on lighthouse design. An avid mountaineer, Hopkinson perished while climbing in the Alps.

...susceptibility

continued from page 6

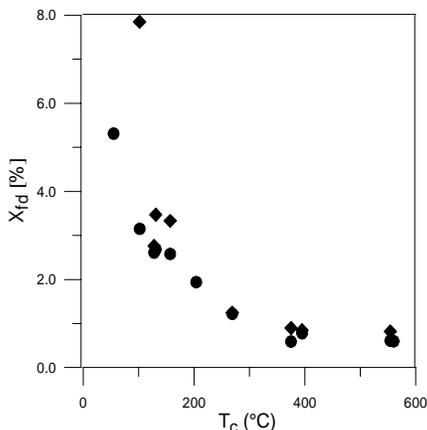
Fig. 4: Field dependence of susceptibility of a Riedheim basalt ($T_c = 128^\circ\text{C}$) measured at 625 Hz (upper curve) and 6 kHz (lower curve) with the Lakeshore susceptometer.



elaborate on this topic here further. But you may imagine that one of the suspect parameters is magnetostriction, and you can further imagine interesting sets of experiments that explore the compositional and grain size dependence of χ_{fd} and also its dependence on stress by measuring ball milled and annealed synthetic samples.

It is again amazing to notice how many rather basic rock magnetic

Fig. 5: The frequency dependence (χ_{fd}) of susceptibility of Riedheim and Hawaiian basalt samples versus Curie temperature (T_c) measured with Lakeshore susceptometer (circles) and Bartington instrument (diamonds).



properties are still waiting to be determined.

As to the common Bartington χ_{fd} measurements, make sure you are not dealing with titanomagnetites (or coarse-grained pyrrhotites or hematites) before interpreting χ_{fd} values to be caused by SP grains. We favor the TDIRM approach for estimating SP content (who would have guessed?) - simple, inexpensive, and more sensitive than χ_{fd} determinations.

References

de Wall, H., The field-dependence of AC susceptibility in titanomagnetites: implications for the anisotropy of magnetic susceptibility, *Geophysical Research Letters*, 27 (16), 2409-11, 2000.

Jackson, M., B. Moskowitz, J. Rosenbaum, and C. Kissel, Field-dependence of AC susceptibility in titanomagnetites, *Earth and Planetary Science Letters*, 157 (3-4), 129-39, 1998.

Kletetschka, G., Grain-size-dependent magnetic susceptibility of hematite, *IRM Quarterly*, 9 (4), 7-8, 2000.

Worm, H.-U., Time-dependent IRM: a new technique for magnetic granulometry, *Geophysical Research Letters*, 26 (16), 2557-60, 1999.

Worm, H.-U., D. Clark, and M.J. Dekkers, Magnetic susceptibility of pyrrhotite: grain size, field and frequency dependence, *Geophysical Journal International*, 114, 127-137, 1993.

New (&Recent) Visiting Fellows

The last two application deadlines (June and December, 2000) generated very large numbers of very good proposals, making it very difficult to narrow the list to the

following group of new Visiting Fellows, to whom we offer our congratulations. The next deadline will be June 1, 2001, for visits during the fall and winter; see our web site or contact the lab manager.

Spring & Summer, 2001

- Andreas Brandau** (*University of Cologne*): Rock-magnetic properties of hydrothermally altered sediments
- Helga de Wall** (*Ruprecht-Karls-Universität Heidelberg*): Field dependence and low-temperature magnetic susceptibility of titanomagnetites
- Bernie Housen** (*Western Washington University*): Remagnetized(?) red-beds, unstable(?) turbidites, magnetic bacteria, and climate-change proxies: or, 2001 hysteresis loops, a rock magnetic odyssey
- BangYeon Kim** (*Lehigh University*): Environmental magnetic study of Lake Ely, PA
- Neil Linford** (*English Heritage*): Characterising the magnetic mineralogy from two archaeological sites formed under different environmental conditions
- Roberto Molina Garza** (*Universidad Nacional Autónoma de México*): Rock magnetism of cool water carbonate sediments, ODP Leg 182 The Great Australian Bight
- Özden Özdemir and David Dunlop** (*University of Toronto - Erindale Campus*): TRM and low-temperature magnetic properties of hematite
- Yongxin Pan** (*The University of Liverpool*): Further checks on alteration of titanomagnetites in basalts: Implications for palaeointensity studies
- Alexander Zwing** (*Universität München*): Rock magnetic signatures of Late Paleozoic remagnetizations in central Europe

Fall & Winter, 2000-2001

- Ramon Egli** (*ETH Zürich*): Identification and origin of ferrimagnetic minerals in lake sediments
- Yohan Guyodo** (*University of Florida*): Detection of magnetic nanoparticles in deep-sea limestones and brain cells
- Fatima Hernandez** (*ETH Zürich*): Magnetic anisotropy study on phyllosilicate crystals
- Gunther Kletetschka** (*NASA-Goddard Space Flight Center*): Temperature dependence of exchange magnetic anisotropy in natural and artificial materials
- Christopher Pike** (*UC-Davis*): Acquisition and analysis of low-temperature first-order reversal-curve (FORC) data
- Alexei Smirnov** (*University of Rochester*): Study of low-temperature magnetic properties of pelagic sediments as indicators of magnetic mineral diagenesis.
- Basil Tikoff** (*University of Wisconsin*) and **Paul Kelso** (*Lake Superior State University*): AMS analysis of the paramagnetic component of ferromagnetic-dominated samples
- Elena Virina** (*Moscow State University*): Particle-size and composition of ferrimagnetic minerals in loess-paleosol deposits from different areas of the Russian Plain on basis of hysteresis parameters at low temperatures.

The *Institute for Rock Magnetism* is dedicated to providing state-of-the-art facilities and technical expertise free of charge to any interested researcher who applies and is accepted as a Visiting Fellow. Short proposals are accepted semi-annually in spring and fall for work to be done in a 10-day period during the following half year. Shorter, less formal visits are arranged on an individual basis through the Facilities Manager.

The *IRM* staff consists of **Subir Banerjee**, Professor/Director; **Bruce Moskowitz**, Associate Professor/Associate Director; **Jim Marvin**, Senior Scientist; **Mike Jackson**, Senior Scientist and Facility Manager, and **Peat Solheid**, Scientist.

Funding for the *IRM* is provided by the **National Science Foundation**, the **W. M. Keck Foundation**, and the **University of Minnesota**.

The *IRM Quarterly* is published four times a year by the staff of the *IRM*. If you or someone you know would like to be on our mailing list, if you have something you would like to contribute (e.g., titles plus abstracts of papers in

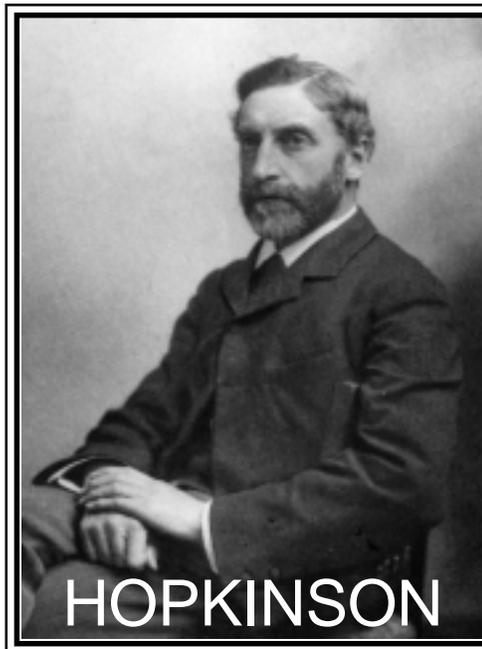
press), or if you have any suggestions to improve the newsletter, please notify the editor:

Mike Jackson
 Institute for Rock Magnetism
 University of Minnesota
 291 Shepherd Laboratories
 100 Union Street S. E.
 Minneapolis, MN 55455-0128
 phone: (612) 624-5274
 fax: (612) 625-7502
 e-mail: irm@geolab.geo.umn.edu
 www.geo.umn.edu/orgs/irm/irm.html

I R M

Institute for Rock Magnetism

The U of M is committed to the policy that all people shall have equal access to its programs, facilities, and employment without regard to race, religion, color, sex, national origin, handicap, age, veteran status, or sexual orientation.



source: Original papers by the late John Hopkinson, D.Sc., F.R.S., edited by B. Hopkinson, 1901, Cambridge University Press.

Collector's Series #20
Hopkinson

RAC Members Rotate

Following completion of their scheduled six-year terms on *IRM's* Review and Advisory Committee, Sue Halgedahl, Dan Dahl berg and RAC Chair John King are

stepping down, with our sincere thanks for their vital efforts and their insight. New RAC members Roy Roshko (*U. of Manitoba Physics Dept.*), Ron Merrill (*U. of Washington*), Rob Coe (*U.C. Santa Cruz*) will join current members Ken Kodama

(*Lehigh U.*), Friedrich Heller (*ETH-Zurich*), Lisa Tauxe (*Scripps*), and Jim Channell (*U. of Florida*) on the reformulated RAC. Ken Kodama has taken on the responsibility of chairing the RAC.

The *IRM* Quarterly

University of Minnesota
 291 Shepherd Laboratories
 100 Union Street S. E.
 Minneapolis, MN 55455-0128
 phone: (612) 624-5274
 fax: (612) 625-7502
 e-mail: irm@geolab.geo.umn.edu
 www.geo.umn.edu/orgs/irm/irm.html

Nonprofit Org.
 U.S Postage
 PAID
 Mpls., MN
 Permit No. 155