Inside...

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Fe_3O_4 Smoked at 80K

Brian Carter-Stiglitz
Bruce Moskowitz
Peter Solheid
IRM

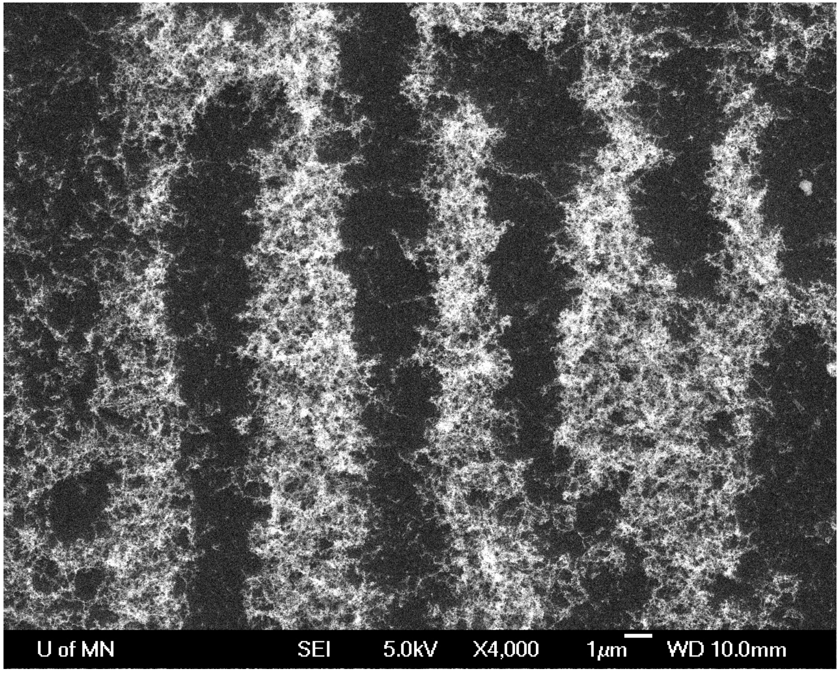

Figure 1--Uniaxial out-of-plane magnetic domains in magnetite imaged at 80 K using the "dry-Bitter" technique and a new IRM apparatus, nick-named "the smoker."

The Verwey transition has long been used by rock magnetists as a litmus test for the presence of magnetite. The rock-magnetic community's understanding of the exact details of the transition's effect on magnetite's magnetism has, however, lagged applications. One outstanding mystery is the magnetic manifestation of the Verwey transition in multidomain (MD) magnetite; this article will focus on this topic, and a new apparatus developed at IRM for making domain observations at low-temperature (Figure 1). We are just now beginning to use the new apparatus and in the following we will discuss some of the initial results and interpretations.

The Verwey Transition, the Basics

The Verwey transition is a first-order—volume changes discontinuously—phase transition occurring at ~118 K. Above the transition magnetite is its familiar cubic phase; below 118 K the low-temperature phase is monoclinic. On cooling, the orientation of the monoclinic phase is, in part, controlled by the cubic phase. The conventional paradigm is that in a zero field a random cube edge becomes the c-axis[1] of the monoclinic phase and two orthogonal (mutually and with respect to the new c-axis) face diagonals become the a and b axes of the low-temperature phase (Figure 2). In a sufficiently large field[2] the c-axis is restricted to the cube edge that is closest to the applied field. Thus, we expect that, on cooling through the transition, large single cubic crystals will form *c*-axis twins below the transition for the zero-field cooled (ZFC) case and that such twins will be absent in the field-cooled (FC) case.

The magnetic manifestation of the transition is due to the changes in the grain-scale magnetic anisotropy that it induces. Above the transition magnetite is characterized by a weak magnetocrystalline anisotropy, which is dominated by the much stronger magnetostatic or shape anisotropy. The magnetocrystalline anisotropy changes abruptly at the phase transition; the new anisotropy, while of a form with lower symmetry, is two orders of magnitude stronger than the cubic phase's anisotropy and roughly a factor of two stronger than the maximum shape anisotropy, e.g., that for an infinitely long needle. For stable single-domain (SSD) magnetite the switch of the easy axis from the (*c*-axis) long axis to the (long axis) *c*-axis on (warming) cooling through the transition is what causes the observed drop in magnetization. The easy-axis bias induced in the low-temperature phase with field cooling is the source of the elevation of FC low-temperature remanences over their ZFC counterparts for SSD grains. Essentially, at the transition magnetite switches from the familiar shape-anisotropy controlled (courtesy of its strong ferrimagnetism) magnetic mineral to one that retains its strong ferrimagnetism but becomes dominated by magnetocrystalline anisotropy. This switch should also critically affect the domain structures at low temperature.

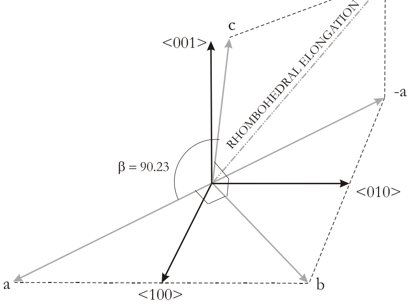

Figure 2--Crystallographic scheme for magnetite and its low-temperature monoclinic polymorph.

Empirically, we see that the remanence lost on passing through the transition and the amount recovered on cycling through it are strong functions of grain size. The former is directly related to grain size; the latter is inversely related to the same.

Behavior of Multidomain Magnetite through the Verwey Transition

Here we show ZFC and FC low-temperature saturation isothermal remanent

low-temp domains
continued on p. 8...

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[1] Actually the c-axis is tilted slightly (~0.2°) toward the -a-axis.
[2] $\mu_o H >\sim 100 mT$.

Visiting Fellows' Reports

David Krasa, University of Munich (now at University of Edinburgh)

The aim of my visit to the IRM was to study the magnetic properties of the mineral phases resulting from inversion of natural titanomaghemites in mid ocean ridge basalts (MORBs). Inversion is a process which is induced by thermal treatment and our previous studies suggest that it plays an important role in controlling the remanence properties of MORB subjected to thermal demagnetization. The proneness to inversion increases with the degree of oxidation of titanomaghemites as these become more and more cation-deficient and thus unstable. The inversion product consists of an intergrowth of Ti-poor titanomagnetite (with a composition close to magnetite) and Fe-poor hemoilmenite.

For my investigations I chose MORB samples with an age between 16 and 32 Ma and a moderate to high degree of oxidation (z = 0.45…0.85). In order to study the inversion process in detail, a succession of saturation magnetization versus temperature, $M_S(T)$, curves with progressively increasing maximum temperatures was measured for each sample using the high-temperature VSM. In between these individual thermomagnetic runs hysteresis loops, IRM acquisition and backfield curves were measured at temperatures between 310 and 10K on the low-temperature VSM.

The measurements indicate that inversion starts when the samples are heated above 300°C and that this is a process which is gradually proceeding with each additional heating step (Fig. 1). The most obvious features are an increasing Curie temperature and saturation magnetization and a low in coercive force for heating steps between 300 and 500°C. The series of low-temperature hysteresis measurements clearly show the gradual transition of the M_S temperature behavior from Néel's N- or P-type typical for titanomaghemites, to the Q-type typical for Ti-poor titanomagnetite. There are also characteristic changes in magnetic stability associated with inversion as can be seen from the coercivity versus temperature plots.

Another aim of my stay at the IRM was to study the compositional and magnetic microstructure of magnetic minerals in very old (>40 Ma) MORB's using the magnetic force microscope (MFM). In these older samples, titanomaghemites are often heavily altered beyond low-temperature oxidation. A typical feature is the partial replacement of the iron-oxides by titanite. My observations indicate that this secondary mineral often subdivides the titanomaghemites into smaller subvolumes thus changing the magnetic microstructure and rock magnetic properties in a characteristic manner (Fig. 2).

I would like to thank the whole IRM staff for their help and hospitality. Again, they made this visit an extremely pleasant and productive experience. Financial support by the Deutsche Forschungsgemeinschaft (DFG grant Ma2578/1-1) is gratefully acknowledged.

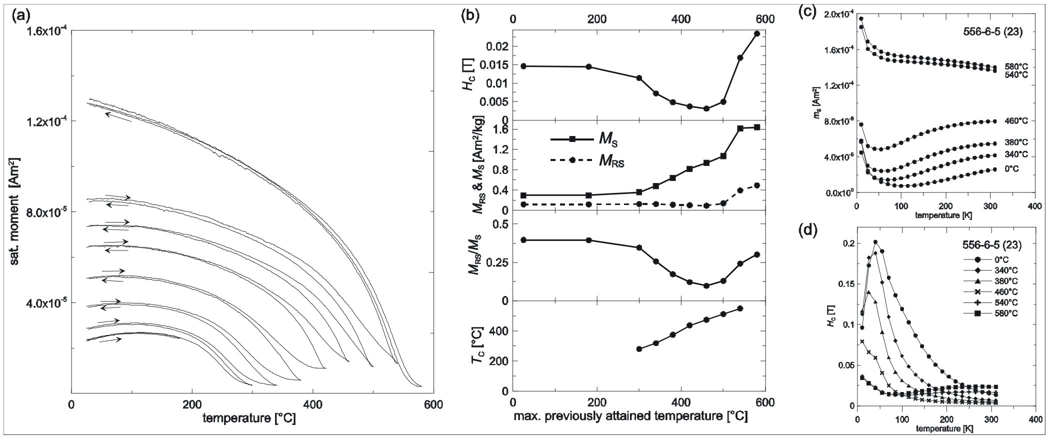


Figure 1, left, a) Evolution of saturation moment during a succession of heating cycles of a MORB sample. (b) Respective hysteresis parameters at room temperature and Curie temperatures after each heating step. (c) and (d) Saturation moment and coercive force versus temperature after successive heating steps. The maximum previously attained temperature is shown.

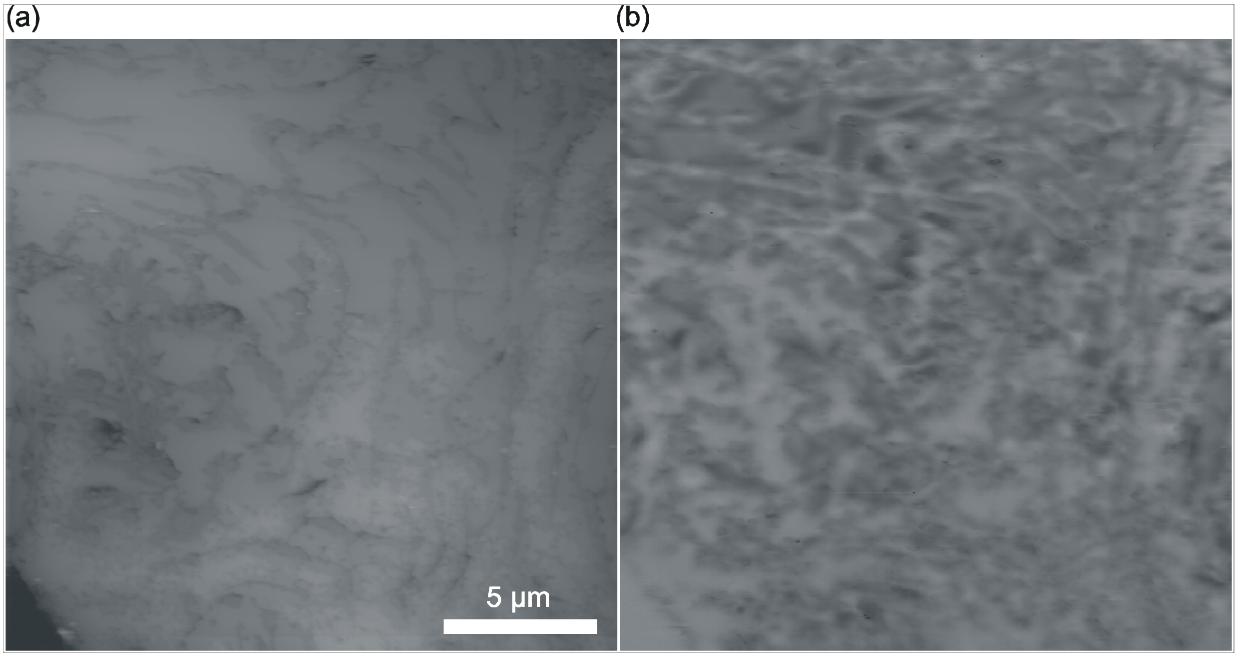


Figure 2 (a) Topographic image (AFM) of a polished section of a highly altered titanomaghemite grain. Darker channel-like structures represent titanite subdividing the titanomaghemite into smaller subvolumes. (b) MFM image of the magnetic microstructure of the particle. Titanite areas appear homogeneously grey as they have no magnetic moment and thus do not produce any MFM contrast.

The Seventh Santa Fe Conference on Rock Magnetism
St. John's College, Santa Fe NM

Thursday June 22 – Sunday June 25, 2006

The conference will feature in-depth discussions on the following themes: 1) Magnetic phases for planetary magnetic recording; 2) Techniques of measurement and modeling; 3) Paleointensity from rocks and sediments: Where do we stand now? and 4) Reconstructing paleoenvironment and paleoclimate: Special strength of magnetism. A small number of speakers (invited by the session chairs) will begin each session with critiques of particular relevant issues, to serve as starting points for discussion. All participants are encouraged to come armed with a few overhead transparencies to contribute to the discussion. No abstract submission is required.

There is no conference registration fee. $205 will cover housing in the St John's dorms (double occupancy) and meals in the cafeteria. A small number of single rooms will be available for an additional $40. Thanks to the sponsorship of the National Science Foundation, we will be able to offer partial reimbursement (approximately $250 - $300) of travel costs to and from Santa Fe. For registration forms and more information see our web site.

Tentative Schedule and Session Summary

Thursday, June 22

2:00-5:30 pm: Registration and room check-in
7:00 pm: Welcome and Opening Comments (Subir Banerjee, IRM, U. of Minnesota)
7:10 pm: Evening Session: Keynote lecture on planetary magnetism: Title TBA (Lon Hood, Lunar and Planetary Laboratory, University of Arizona)

Friday, June 23

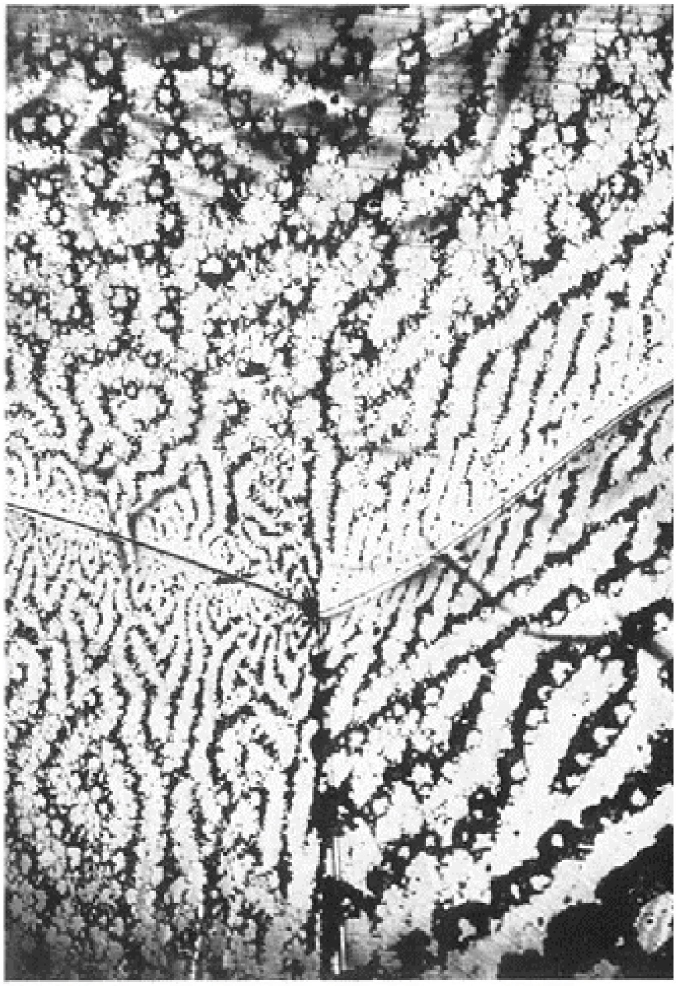
9:00 am: Morning Session: Magnetic phases for planetary magnetic recording (Özden Özdemir and Gunther Kletetschka, coordinators)
1:30 pm: Afternoon Session: Techniques of measurement and modeling (Joshua Feinberg and Andrew Newell, coordinators)
7:10 pm: Evening Session: Keynote lecture on magnetic mineral chemistry: Kinetic Paths, Metastable States & Ferrite Nucleation: The Impact of the "Semiconductor Condition" (Reid F. Cooper, Department of Geological Sciences, Brown University)

Saturday, June 24

9:00 am: Morning Session: Paleointensity from rocks and sediments: Where do we stand now? (David Dunlop and Toshi Yamazaki, coordinators)
1:30 pm: Afternoon Session: Reconstructing paleoenvironment and paleoclimate: Special strength of magnetism (Stefanie Brachfeld and Ted Evans, coordinators)
evening: free

Sunday, June 25

9:00 am: Morning Session: MagIC magnetic database: Hands on testing of current database and suggestions for future development (Lisa Tauxe and Ramon Egli, coordinators)
noon: check-out and departure



Domain image of cobalt "near a grain boundary" (Bitter, 1932).

Current Articles

Note: Due to space limitations which have required the exclusion of an ever increasing number of superb papers, abstracts will no longer be printed in the Quarterly. Instead we will print a bibliography of current research articles. The complete abstract text can be found at: www.irm.umn.edu/abstracts. It is our hope that readers check the site frequently as it will be continuously updated as articles enter into the databases. We expect that the web-page will be a useful tool for keeping on top of the magnetic literature. Finally, selected "editor's choice" abstracts will be printed in full. Please send comments or questions to the editor.

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 taken 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 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 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.

Alteration & Remagnetization

Garming, J.F.L., U. Bleil, and N. Riedinger, Alteration of magnetic mineralogy at the sulfate-methane transition: Analysis of sediments from the Argentine continental slope, Physics of the Earth and Planetary Interiors, 151, 3-4, 2005.

Torsvik, T.H., M.K. Pandit, T.F. Redfield, L.D. Ashwal, and S.J. Webb, Remagnetization of Mesozoic limestones from the Jaisalmer basin, NW India, Geophysical Journal International, 161 (1), 57-64, 2005.

Wang, D.M., R. Van der Voo, and D.R. Peacor, Low-temperature alteration and magnetic changes of variably altered pillow basalts, Geophysical Journal International, 164 (1), 25-35, 2006.

Anisotropy

Cifelli, F., M. Mattei, M. Chadima, A.M. Hirt, and A. Hansen, The origin of tectonic lineation in extensional basins: Combined neutron texture and magnetic analyses on "undeformed" clays, Earth and Planetary Science Letters, 235, 1-2, 2005.

In extensional sedimentary basins fine-grained sediments that appear undeformed at the outcrop scale can carry a magnetic fabric consistent with the regional deformation pattern. The origin of the magnetic lineation, which is often found in extensional basins, is not yet well understood. In clays from extensional basins in southern Italy, the magnetic lineation is tectonically controlled and oriented perpendicular to the main normal faults. A combined analysis of magnetic and mineral fabrics was made to gain insight into the processes that lead to a lineation in extensional settings. Low-field, high-field and low-temperature susceptibility measurements were used to distinguish the ferrimagnetic and paramagnetic contributions to the magnetic susceptibility and its anisotropy. The magnetic anisotropy of the sediments is predominantly carried by paramagnetic phyllosilicates. Neutron texture analysis was used to evaluate the spatial distribution of chlorite basal planes. Results demonstrate that the orientation of the magnetic lineation is related to the spatial distribution of chlorite, lying parallel to the common axis of differently oriented basal planes. A quantitative correlation between the magnetic and rock fabric was made comparing the low- and high-field magnetic anisotropy (ANIS, HFA) to the theoretical anisotropy calculated from the chlorite-preferred orientation. A good linear correlation is found between the degree of theoretical anisotropy and the AMS and HFA. Results show that the integrated approach of magnetic and mineral fabric investigations represents a valid alternative tool for detecting grain scale and regional deformation patterns in weakly deformed extensional basins, where macroscopic evidence of deformation is often not visible.

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Wanwu, G., A regression algorithm for rock magnetic data mining, WSEAS Transactions on Information Science and Applications, 2 (6), 671-8, 2005.

Environmental Magnetism and Paleoclimate Proxies

Ahn, Y.C., and J.K. Lee, Physical, chemical, and electrical analysis of aerosol particles generated from industrial plants, Journal of Aerosol Science, 37 (2), 187-202, 2006.

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Extraterrestrial Magnetism

Boutin, D., and J. Arkani-Hamed, Pole wandering of Mars: Evidence from paleomagnetic poles, Icarus, 181 (1), 13-25, 2006.

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Magnetic Field Records and Paleointensity Methods

Gallet, Y., A. Genevey, and F. Fluteau, Does Earth's magnetic field secular variation control centennial climate change?, Earth and Planetary Science Letters, 236, 1-2, 2005.

We obtained new archeointensity data from French faience potsherds dated from the 17th to 19th century. These results further document the occurrence of sharp changes in geomagnetic field secular variation in Western Europe over the past three millennia. The intensity variation curve shows several maxima whose rising parts appear to coincide in time with the occurrence of cooling events documented in this region from natural and historical data. This coincidence suggests a causal link between enhanced secular variation of the geomagnetic field and climate change over centennial time scales, challenging the role of solar forcing as the sole factor provoking these climatic variations. We propose that the archeomagnetic jerks described by Gallet et al. [I] [Y. Gallet, A. Genevey, V. Courtillot, On the possible occurrence of archeomagnetic jerks in the geomagnetic field over the past three millennia, Earth Planet. Sci. Lett. 214 (2003) 237-242.] may engage the mechanism for centennial climate change.

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Pechersky, D.M., V.S. Zakharov, and D.M. Khvorov, Geomagnetic field of the cooling time of the Kivakka intrusion (Northern Karelia), Izvestiya, Physics of the Solid Earth, 41 (4), 292-313, 2005.

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Magnetic Microscopy and Spectroscopy

Hernanz, A., M. Mas, B. Gavilan, and B. Hernandez, Raman microscopy and IR spectroscopy of prehistoric paintings from Los Murcielagos cave (Zuheros, Cordoba, Spain), Journal of Raman Spectroscopy, 37 (4), 492-497, 2006.

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Mineral & Rock Magnetism

Qingsong, L., D. Chenglong, Y. Yu, J. Torrent, M.J. Jackson, S.K. Banerjee, and Z. Rixiang, Temperature dependence of magnetic susceptibility in an argon environment: implications for pedogenesis of Chinese loess/palaeosols, Geophysical Journal International, 161 (1), 102-12, 2005.

Williams, W., and A.R. Muxworthy, Understanding viscous magnetization of multidomain magnetite, Journal of Geophysical Research-Solid Earth, 111 (B2), 2006.

Viscous magnetization (VM) and viscous remanent magnetization (VRM) have been measured, as a function of temperature, between room temperature and the Curie temperature using a suite of well-characterized synthetic and natural multidomain (MD) magnetite samples. Particular atten-

tion was given to possible diffusion aftereffects such as dislocation creep (stress relaxation) and disaccommodation (vacancy and ionic reordering) and their contribution to viscous behavior in what has been commonly thought of as a purely thermal fluctuation process. Dislocation creep was examined by measuring viscosity before and after annealing. Annealing was found to reduce the non-log(t) behavior, where t is time. Non-log(t) behavior has been associated with diffusion aftereffects, suggesting that these are a major contributor to viscosity and (de) magnetization processes in MD samples. The positive curvature of the non-log(t) acquisition processes indicates that dislocation creep dominates over disaccommodation. This does not imply that VM and VRM are due solely to dislocation creep, but rather that VRM and VM reflect a number of unrelated temperature-dependent processes, primarily thermal fluctuations and dislocation creep. This is the first time that dislocation creep has been directly identified as contributing to viscosity at temperature. These findings will have particular implications for paleointensity determinations, as on heating a sample, its dislocation structure may relax, giving rise to demagnetizations not associated with thermal fluctuations. This will lead to incorrect intensity estimates. If no heating is performed on a geological specimen, then it is very likely that laboratory timescale stress relaxation processes will have already occurred in situ.

Mineral Physics & Chemistry

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magnetization (LTSIRM)[3] on warming for four MD samples[4]. The data are remarkably similar (figure 3) and conform with those previously reported (Brachfeld, et al., 2001; Carter-Stiglitz, et al., 2001; Kosterov, 2001; Kosterov, 2003). ZFC remanences are ~50% larger than FC remanences. The LTSIRM are fairly stable on warming to T_V, demagnetizing by only ~10%. At T_V both ZFC and FC remanences almost completely demagnetize, and hence yield delta ratios (see Moskowitz, et al., 1993) near 1.

One of the most surprising aspects of the low-temperature behavior of MD magnetite is that the FC remanence is *lower* than the ZFC LTSIRM. After all, the FC samples have a strong easy axis bias in the direction of the applied field. Phenomenologically, this behavior seems to be unique to "true" MD magnetite and is opposite to that of SSD and small PSD magnetite.

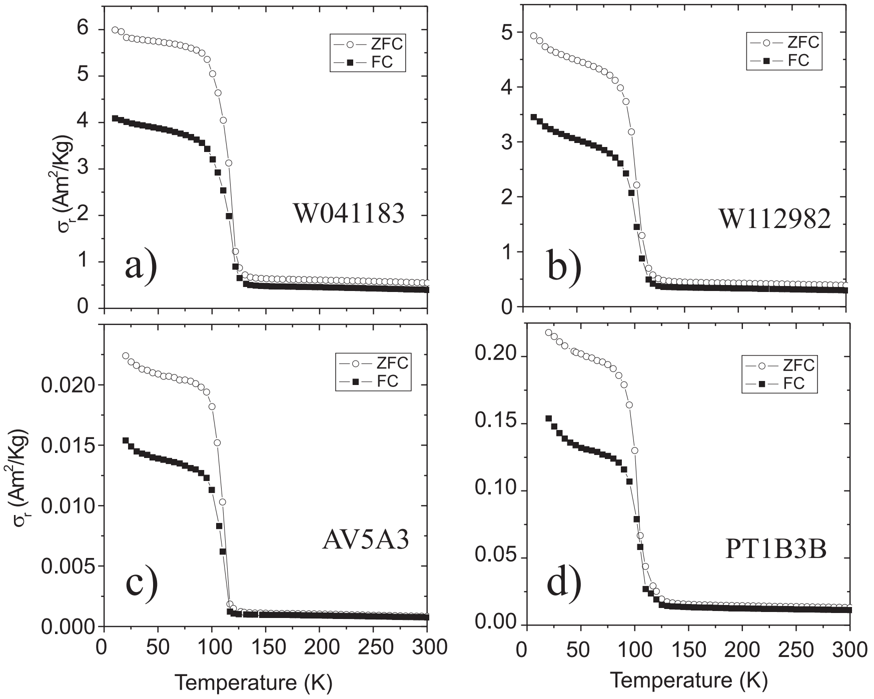

Figure 3--FC and ZFC LTSIRM's measured on warming for three samples of MD magnetite, two synthetics: a) W041183 and b) W112982; and two whole-rock samples: c) AV5A3 and d) PT1B3B.

The most recent theory put forth to explain this phenomenon suggests that it is solely a result of the geometry of the easy-axis bias and the relationship between H_c and remanence which has been suggested for multidomain grains (Kosterov, 2003). The theory works well for ZFC and FC loops measured parallel to the cooling field direction but predicts behavior opposite to what is seen for FC loops measured perpendicular to the cooling field[5]. All of the experimental measurements are, however, wholly consistent with a previous qualitative theory that claims that the FC remanences are suppressed because of the absence of transformational twins in FC samples, whereas such twins are present in ZFC samples (Özdemir, 1999; Smirnov & Tarduno, 2002).

Clearly the best and most direct way to test and further our understanding of the low-temperature magnetic properties of MD magnetite is with domain observations at low temperature. Initial attempts at doing just this have yielded exciting results (Moloni, et al., 1996).

The Dry-Bitter Method

"[T]he old magnetic powder[6] method," was how Bitter (1932) described the not so old technique of magnetic-domain decoration that is now simply referred to as the Bitter method. As it is used today the Bitter method exploits the attraction of a magnetic colloid, or ferrofluid, to locations on a magnetic material where concentrations of magnetic flux emerge from or descend into the surface. We note here that not all walls are imaged by the Bitter technique with equal clarity; for example, Bloch[7] walls that are perpendicular to the viewing plane are easily imaged using the technique, whereas Néel[8] walls in the same orientation have much weaker stray fields and are more difficult to image.

The traditional Bitter method is not appropriate for low-temperature observations. Obviously, at temperatures below the freezing point of the colloidal suspension nothing could be observed. Moreover, if one devised a colloidal suspension that would remain liquid at the temperatures of interest, it would require optical observation at low-temperature.

Technique & Apparatus

Smith, et al., (1980) developed a decoration method that could be employed at any temperature and after decoration the deposited material would remain immobile. The technique, hereinafter referred to as the dry-Bitter method, evaporates a small amount of Fe in a He

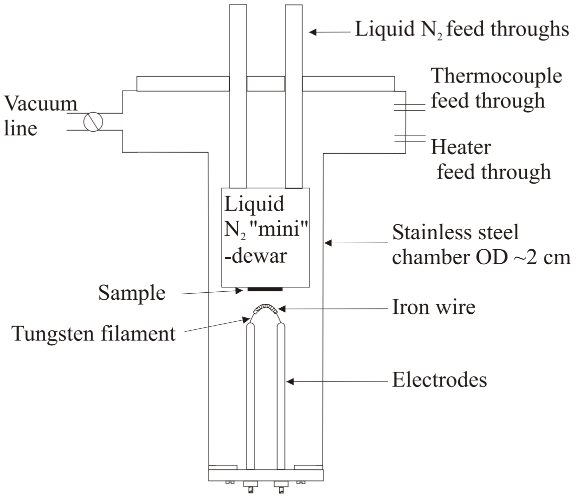

Figure 4--Schematic diagram of our low-temperature dry-Bitter domain decoration apparatus.

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[3] *Low-temperature remanence and susceptibility measurements were made using a Quantum Design Magnetic Properties Measurement System Susceptometer. Remanence was measured on warming from 10 to 300 K starting from two initial states: zero-field cooled (ZFC) from 300 K to 10 K after which a saturating field of 2.5 T was applied, and field cooled (FC) in 2.5-T field. This is the standard "ZFC/FC" measurement often conducted at IRM and elsewhere. Note that when a physicist or engineer refers to a standard "ZFC/FC" measurement they are usually referring to a similar pretreatment but the measurement of an induced magnetization.*

[4] *Four multidomain magnetite samples were characterized in this study: two synthetic samples produced by Wright industries W041183 (20.1 ± 13.4 μm [Yu, et al., 2002]) and W112982 (18.6 ± 9.6 μm [Yu, et al., 2002]), and two whole rock samples, AV5A3 and PT1B3, granite and gabbro, respectively (Brachfeld et al., 2002). All four samples show clear multidomain hysteresis behavior. The whole rock samples had Curie temperatures—deduced from saturation magnetization—consistent with magnetite. AV5A3 yielded a Curie temperature of 585 ^{o}C on warming and 580 ^{o}C on cooling, and PT1B3B yielded 587 ^{o}C and 582 ^{o}C respectively.*

[5] *We leave the empirical and logical details of this argument for another time and forum.*

[6] *Bitter (1932) did not employ today's agent of decoration, the ferrofluid, but rather a powder of hematite dispersed in alcohol.*

[7] *In a 180^{o}-Bloch wall spins rotate parallel to the plane of the domain wall. Typically such walls are observed in a configuration where the body-domain magnetization is parallel to the plane of observation and thus the wall's spins rotate such that they are roughly perpendicular to the surface in the middle of the wall.*

[8] *In a Néel wall the spins rotate perpendicular to the plane of the domain wall. Typically, for strong ferrimagnets, such a configuration is only energetically favorable in thin films; as we will see, the state of affairs is different for monoclinic magnetite.*

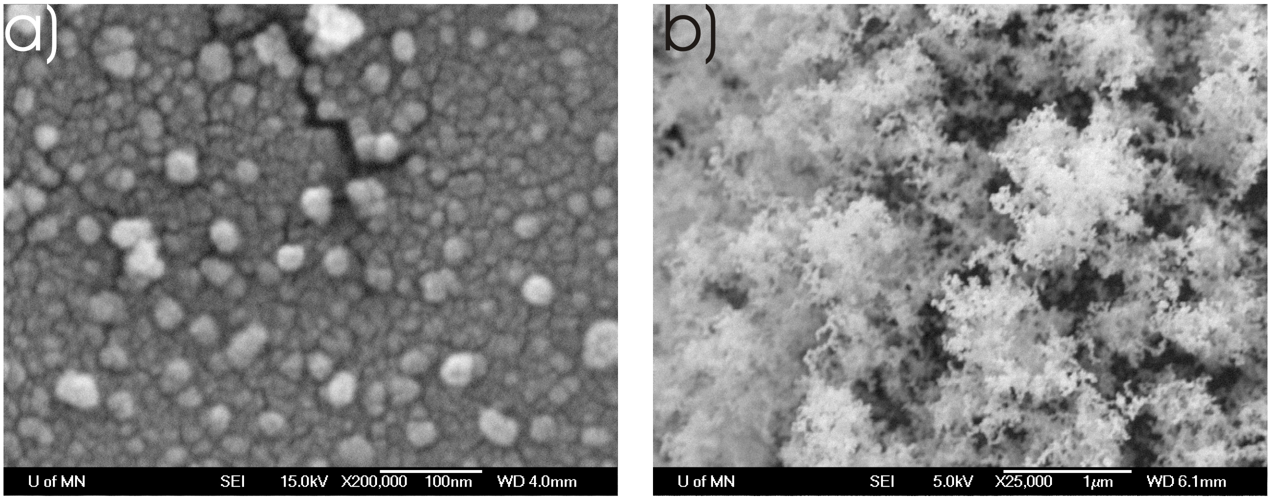

Figure 5--Images of iron nano-particles used to decorate magnetic domains.

atmosphere of ~1 Torr. As the Fe moves away from the filament it condenses into small nano-particles (~10-50 nm) of iron. The iron particles are then deposited on the surface of the sample which is some centimeters away. Given that the particles are traveling slowly enough they are, just as in the traditional Bittern method, attracted to locations where stray magnetic fields are present. Once deposited the particles remain immobile unless physically disturbed.

The apparatus that we constructed is essentially a modified version of that used by Smith, et al., (1980). The primary modification was the sample cooling system. Our system is basically a vacuum chamber; it is narrow enough to fit between the pole gap of an electromagnet; it has feed-throughs for: liquid N_2, a thermocouple, a heater, and He gas; the sample is mounted to a copper plate at the bottom of a small internal dewar which cools the sample to ~80 K; two electrode feed-throughs at the bottom of the chamber provide the current for the evaporation of the Fe wire which is wound around a tungsten filament (Figure 4).

It seems there are five essential variables that control the quality, in terms of domain observation, of a given deposition: the current in the filament; the time the current is present; the distance between the filament and the sample; the amount of Fe on the filament; and the He pressure in the chamber. If any one of these variables is maladjusted the experiment fails; four modes of failure seem to be present: a thin film may be deposited, instead of a carpet of small particles; too little material may be deposited; too much material may be deposited; and the particles may be moving too quickly and deposit on the surface little affected by the stray magnetic fields. To make things more complicated, thermal convection currents in the chamber (driven by the hot filament and the cold sample) are sufficiently different between a room-temperature deposition and a low-temperature deposition to ensure that the same set of conditions may fail in one case but be successful in the other.

Initial Results

Our first experiments were conducted at room temperature on a glass substrate. Such experiments were conducted to test whether we were successfully forming nano-particles of iron. Figure 5a[9] shows the results of such a deposition. The 10-50 nm sphericles are clearly visible on the background of the Pt-coating's grain structure. Given enough material deposited the sphericles clump together in a way that is not likely to aid in domain decoration (Figure 5b).

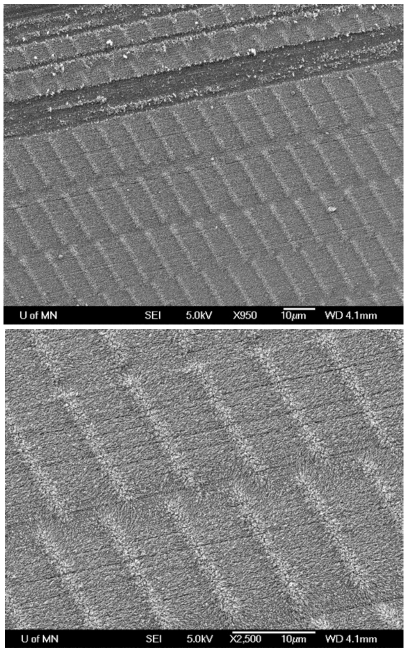

Figure 6--Dry-Bitter domain images of a magnetic hard drive.

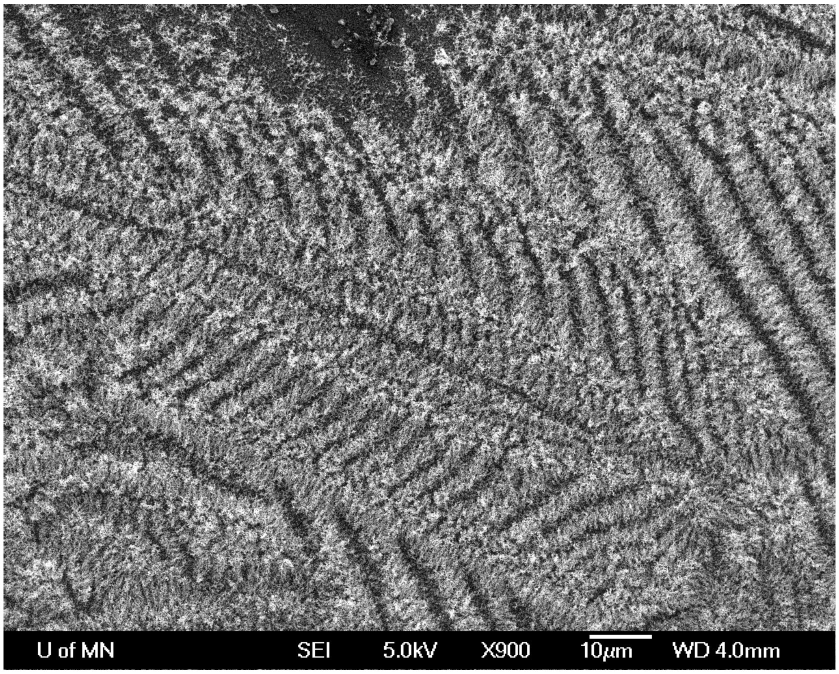

Figure 7--Dry-Bitter domain image of magnetite, (100) viewing plane.

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[9] *We used a field-emission-gun scanning electron microscope, JEOL model 6500, to produce all of the electron micrographs.*

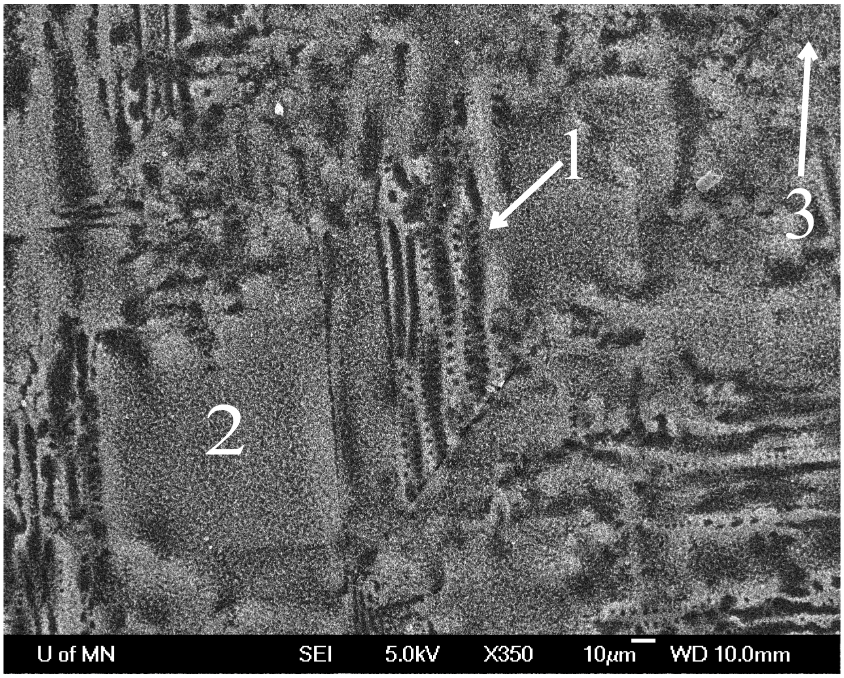

Figure 8--Dry-Bitter domain image of magnetite, (100) viewing plane, at 80 K after zero-field cooling. The numbers correspond to features described in the text. The cube edges are oriented vertically, horizontally and perpendicular to the plane of the page.

A small piece of a magnetic hard drive was our first attempt at domain observation using the dry-Bitter method. Though the deposition was too thick, the "bits" and tracks of the hard drive are clearly visible (Figure 6). The coating, while stable, can easily be wiped off; in the case of Figure 6 (top image) this was done accidentally when the surface of the sample was touched with tweezers.

We attempted imaging the domain structure of magnetite on a (100) viewing plane on a ~1cm single crystal. This is, of course, not the ideal viewing plane for magnetite at room-temperature, but below the Verwey transition it is a good choice; for the ZFC case, given the crystal is large enough to support twinning of the monoclinic phase, the sample would become polycrystalline with three c-axis twin variants, two with c-axes in the viewing plane and the third with the c-axis perpendicular to the viewing plane. In the FC case, depending on field orientation, the c-axis is restrained to just one of these configurations.

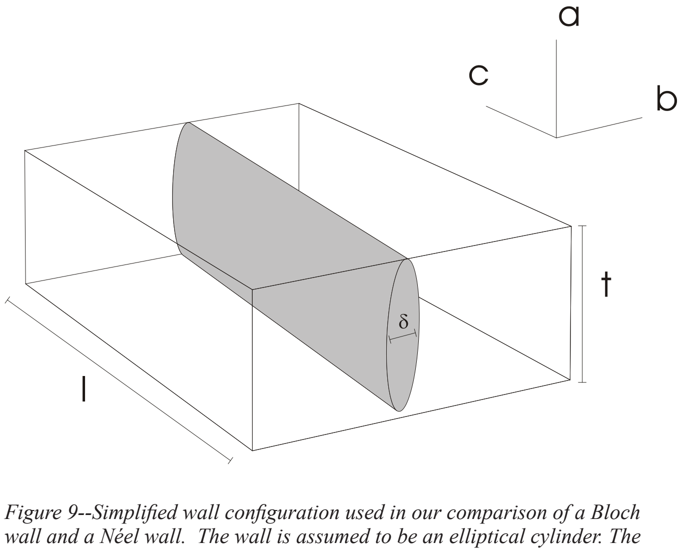

Figure 9--Simplified wall configuration used in our comparison of a Bloch wall and a Néel wall. The wall is assumed to be an elliptical cylinder. The wall plane is parallel to the long axis of the ellipse and to the a-axis of the monoclinic crystal.

The room-temperature domain pattern is complicated, due to the fact that four easy axes sharply intersect the plane; it is typified by the complicated "fir-tree" pattern shown in Figure 7. We are currently experimenting with decorating a (110) viewing-plane sample at room temperature.

We also decorated the (100) sample at 80 K after zero-field cooling from room temperature. Figures 1 & 8 give a good idea of the textures that are consistently visible. Essentially, there are three types of features: 1) areas of long lamellar domains which are interspersed with strings of circular elements that run parallel to the laths—these laths always run parallel to one of the cube edges and are clearly imaged by the dry-Bitter method (Figure 1 and 8); 2) large gray areas with little relief, whose perimeters run parallel to the cube edges and can contain a linear pattern which runs parallel to one of the cube edge directions; and 3) mottled and convoluted features of high relief that have little to no relation to the crystal's cardinal directions.

Feature 1 is entirely consistent with the type of domains observed in a material with strong uniaxial anisotropy whose easy axis is perpendicular to the viewing plane. Specifically we expect such domains for a material where $[1/2(\mu_o M_s^2)]/k_u < 1$ (see, e.g., Moskowitz, et al., 1988), where: M_s is the saturation magnetization; k_u is the uniaxial anisotropy constant—in the case of monoclinic magnetite the k_a constant from the magnetocrystalline anisotropy is the number of interest; and μ_o is the permeability of free space. Inserting the appropriate values (M_s~510 kA/m and k_u~21 x 10^4 J/m^3) we find that for monoclinic magnetite the ratio is ~0.77. The linear arrangements of circular features are, almost certainly, spike domains. Areas of the sample surface that manifest feature 1 are then twin variants where the c-axis is perpendicular to the viewing plane. The second feature is more difficult to interpret. Our initial interpretation is that these features are areas where the body domains' magnetizations lie parallel to viewing plane. We suppose that such domains and their walls are more difficult to image with dry-Bitter method since the stray field intensity would be much less than that for body domain magnetizations which are perpendicular to the viewing plane, i.e., in the case of feature 1. It is quite possible that as the Fe particles are deposited on the surface they have too much momentum to effectively reveal such features. We are presently perfecting our dry-Bitter technique in the hope of overcoming such issues. Nevertheless, one can make out dim linear features that might indicate the separations between body domains. Finally, we suspect that the convoluted feature 3 is the result of residual stresses in the surface that were not completely removed by our final amorphous silica polishing.

Performing dry-Bitter decoration at 80 K for the FC case is now a priority. We are confident that this will not only aid in the interpretation of the images—as we will be viewing a system with a single easy axis direction—but will also help answer the remaining questions discussed in the first part of this article concerning the low-temperature magnetism of MD magnetite.

Magnetite's Domain Configuration at Low-Temperature

Finally, we would like to compare the energies of various domain walls, in light of the dramatic increase in magnetocrystalline anisotropy. Our illustrating example will be that of a Néel wall and a Bloch wall parallel to (110), i.e., the plane perpendicular to the b-axis (Figure 2). Consider the situation illustrated in Figure 9, and note the specific crystal orientation indicated by the schematic.

For the Bloch wall the spins rotate from the c-axis direction to the a-axis direction and back to the c-axis in the plane of the domain wall. For such a wall the energy per unit volume is:

$$e^B = e_{xstal} + e_d + e_e , \qquad (1)$$

where e_{xstal} is the magnetocrystalline anisotropy energy, e_d is the magnetostatic energy, and e_e is the exchange energy. The anisotropy constant for the magne-

Lev Landau

b. January 22, 1908
d. April 1, 1968

Born in Baku the son of an engineer and a physicist, Landau's prolific work covered all aspects of theoretical physics. He is, perhaps, most well known for his work concerning super-fluidity, for which he was awarded the Nobel Prize in Physics in 1962. Here, we are obligated to mention his theoretical treatment and accurate predictions concerning magnetic domains.

tostatic energy term is (approximating the wall as an infinitely-long elliptical cylinder):

$$k_d^B = \frac{1}{2}\mu_0 \frac{\delta M_s^2}{t+\delta}, \qquad (2)$$

where t is the height of the wall, and δ is its width (Néel, 1955). For a massive sample we take the following limit:

$$\lim_{\frac{t}{\delta}\to\infty} \frac{1}{2}\mu_0 \frac{\delta M_s^2}{t+\delta} = 0. \qquad (3)$$

Ignoring the slight rhombohedral distortion, e_{xstal} is:

$$\frac{1}{\pi}\int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \left(k_a\alpha_a^2 + k_b\alpha_b^2 + k_{aa}\alpha_a^4 + k_{bb}\alpha_b^4 + k_{ab}\alpha_a^2\alpha_b^2\right)d\theta, \qquad (4)$$

$$= \frac{k_a}{2} + \frac{3k_{aa}}{8}$$

where k_a, k_b, k_{aa}, k_{bb}, and k_{ab} are constants; α_a and α_b are the direction cosines with respect to the crystal axes of the monoclinic phase; and θ is the angle between the individual spins of the wall and the a axis of the crystal.

For the Néel wall, we have, again:

$$e^N = e_{xstal} + e_d + e_e. \qquad (5)$$

In the case of the Néel wall, however (Néel, 1955):

$$k_d^N = \frac{1}{2}\mu_0 \frac{tM_s^2}{t+\delta}, \qquad (6)$$

and for a massive sample:

$$k_d^N = \lim_{\frac{t}{\delta}\to\infty} \frac{1}{2}\mu_0 \frac{tM_s^2}{t+\delta} = \frac{1}{2}\mu_0 M_s^2. \qquad (7)$$

e_d is thus:

$$\frac{k_d^N}{\pi}\int_0^{\pi} \sin^2(\theta)\,d\theta = \frac{1}{4}\mu_0 M_s^2; \qquad (8)$$

here θ is the angle between the wall-spins and the axis parallel to the l dimension of the wall. e_{xstal} for the Néel wall is simply:

$$\frac{k_b}{2} + \frac{3k_{bb}}{8}. \qquad (9)$$

Comparing the energy of the two walls, assuming the exchange energy is equal for both we get:

$$e_N - e_B = \left(\frac{1}{4}\mu_0 M_s^2 + \frac{k_b}{2} + \frac{3k_{bb}}{8}\right) - \left(\frac{k_a}{2} + \frac{3k_{aa}}{8}\right) \approx -0.2\times 10^5 \frac{J}{cm^3} < 0.$$

Interestingly, the Néel wall is energetically favorable. But how common is this wall configuration? Table 1 shows the energies (less the exchange term) for various domain walls. The $(1\bar{1}0)$ Bloch wall is the least-energy configuration, followed by a Bloch wall that bisects the a and b axes, e.g., (100); refer to Figure 2 to relate the wall-plane directions to the monoclinic crystal. Thus we expect that both of these walls to be energetically favorable over the ones analyzed just above. We note that our simple calculation does not take into account complicated circumstances, such as grain shape, which may prohibit one type of wall. Pokhil and Moskowitz (1997) observed Néel walls in magnetite due to special circumstances involving spike domains, for example. The feature highlighted in Figure 8, indicated with a "1," is probably another such example. Given our initial interpretation of the feature, the walls must be of the (100) type. We suppose that such a configuration is preferable over the $(1\bar{1}0)$ Bloch wall due to the grain shape. Finally, we add the following caveat: the omission of the magnetostriction energy term from our calculations could change the overall picture.

Conclusions

In spite of the wealth of applications exploiting the Verwey transition and basic studies concerning it, numerous questions remain concerning the exact details of its effect on the magnetism of magnetite. The curious elevation of ZFC moments over FC ones is a prime example. Though we have not yet perfected it, the dry-Bitter technique has yielded valuable domain images at ambient and low temperatures. For a ZFC single crystal, our initial results delineate in-viewing-plane c-axis twin variants from their counterparts with c-axes perpendicular to the viewing plane. Imaging of the FC case will follow soon. This summer we also plan on experimentation involving other magnetic phases.

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Table 1

Wall Type	Wall Plane (see Figure 2)	Energy (J/m^3)
Bloch	(110)	13.4 x 10^4
Néel	(110)	12.1 x 10^4
Bloch	(100)	9.35 x 10^4
Bloch	$(1\bar{1}0)$	2.9 x 10^4

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Brian Carter-Stiglitz
Institute for Rock Magnetism
University of Minnesota
289 Shepherd Laboratories
100 Union Street S. E.
Minneapolis, MN 55455-0128
phone: (612) 624-5049
fax: (612) 625-7502
e-mail: cart0196@umn.edu
www.irm.umn.edu

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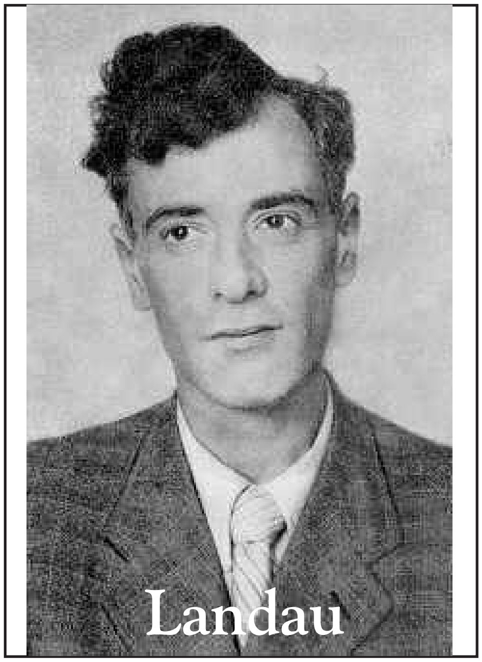

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291 Shepherd Laboratories
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