Interpretation of Low-Temperature Data Part 4:
The Low-Temperature Magnetic Transition of Monoclinic Pyrrhotite

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Use of low temperature (LT) magnetic transitions to identify magnetic minerals that carry a remanence – either natural or laboratory-induced – at room temperature, is a classic tool in rock magnetism (e.g. Nagata et al., 1964; Kosterov, 2007). This particularly applies to magnetite (Verwey transition at 118 K) and hematite (Morin transition at 265 K), transitions that are engraved in the minds of rock magnetists. A little over two decades ago, yet another LT transition has joined our toolkit: that of monoclinic pyrrhotite occurring at 32 K. This article is aimed at providing an account of the somehow complex story of this discovery (or actually its re-discovery as will become apparent), beyond what is visible in the published literature. The story will be told alternatively using first names of the three authors, or “we”, meaning Gérard and Pierre, Mark arriving on the ‘LT scene’ at a later stage.

The (re-)discovery of the pyrrhotite low temperature transition and its aftermaths

In the late eighties, both Mark Dekkers and Pierre Rochette were working on the low temperature magnetic properties of monoclinic pyrrhotite as a part of their PhD thesis, respectively in Utrecht (The Netherlands) and Grenoble (France). Mark had the advantage of working on the magnetic properties of pure phases of known grain size (he concentrated pyrrhotite grains from ores and precision-sieved them into twelve fractions). Pierre’s pyrrhotite work was part of the study of magnetic properties, including anisotropy, of deformed alpine black shales, some of them being pyrrhotite-bearing which rather obviously complicated interpretation. Pierre, however, had two decisive advantages: 1) access to the predecessor of the MPMS (SHE SQUID magnetometer with a temperature range of 2-400 K and a field range of 0-5 T) while Mark used an inhouse-built fluxgate spinner with a liquid nitrogen dewar (data reported in Dekkers, 1989), 2) working in the Néel laboratory under the close supervision of Gérard Fillion, a solid state physicist and expert on low temperature magnetic properties of all sorts of exotic compounds.

On a day in Autumn 1987, Pierre and Gérard set up the SHE instrument to measure the zero field cooling curve of room temperature (RT) saturation isothermal remanent magnetization (SIRM) of a weakly magnetic sample (NB10) in which Pierre had identified SD pyrrhotite based on thermal and AF demagnetization experiments of natural remanent magnetization (NRM) and IRM (Rochette and Lamarche, 1986). Motivation was just exploration of the unknown. The following day, we took the printout (data were often printed in those days) and plotted the curve of...
No evidence for a high coercivity NRM carrier in sediments from eastern equatorial Pacific (IODP Expedition 320/321)

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One of the major objectives of IODP Expedition 320/321 to the eastern equatorial Pacific (May-June 2009) is to improve accuracy of the Cenozoic timescale (Lyle et al., 2009; Pälike et al., 2009), and to build on the timescale improvements stemming from the past Ocean Drilling Program expeditions (ODP 138 and ODP 199) to the same region (Lyle et al., 2009; Pälike et al., 2006). As part of the post-cruise investigations, u-channel samples were collected from individual core sections (normally 2 x 2 x 150 cm³) of Site U1334, at which a thick (~170 m) section of mainly Miocene and Oligocene sediments (~5.33-33 Ma) was recovered.

The NRM of each u-channel sample was measured at 1-cm intervals before and after alternating field (AF) demagnetization at 23 steps in the 0-100 mT peak field range. U-channel measurements were performed on a 2G Enterprises SQUID pass-through cryogenic magnetometer situated in a magnetically shielded room with background field of < 500 nT at the University of Florida. The primary NRM carrier inclinations are expected to be as low as a few degrees, consistent with the paleolatitude of sampling region. This low inclination NRM carrier is well resolved during AF treatments up to 100 mT, however, for some samples, there is a weak steeply-inclined high-coercivity component that cannot be demagnetized by peak field of 100 mT. Lanci et al. (2004, 2005) also observed a high coercivity component in sediments from the same region recovered during ODP Leg 199. These authors attributed the high-coercivity component to hematite of unknown origin. As in the studies of Lanci et al. (2004, 2005), the low-coercivity component at Site U1334 appears to faithfully record the magnetic polarity stratigraphy. The magnetic polarity stratigraphy at Site U1334 will be very important to the overall stratigraphic objectives of the expedition. However, without fully understanding the origin of the high-coercivity steeply-inclined component, the magnetic stratigraphies based on the low-coercivity component will be called into question. The steeply-inclined high-coercivity component could be associated with the drilling process as drill-string magnetizations are often aligned along the drill-string. The visit to IRM was designed to determine the origin of this high-coercivity magnetization component, by testing for the presence of high-coercivity minerals such as goethite.

During the visit to IRM, both high temperature (300°C<T<700°C) and low temperature (10 K<T<400 K) experiments were carried out on freeze-dried samples using a Vibrating Sample Magnetometer (VSM) and Magnetic Properties Measurement System (MPMS), respectively. For low-temperature remanence measurements, field cooling (FC), zero field cooling (ZFC), room temperature saturation isothermal remanent magnetization (FC-ZFC-RTSIRM) protocols were used. Verwey transition is suppressed during FC-ZFC measurements, but visible on RTSIRM curves (Fig. 1). The broad transition range (Fig.1b) and significant remanence decrease below 50 K for FC and ZFC (Fig. 1a) have been attributed to surface oxidized magnetite grains, as described by Özdemir et al. (1993).

We applied the method described by Guyodo et al. (2006) to a few selected samples in order to test for the presence of goethite. Samples were initially heated in the MPMS to 400 K, slightly above the Néel temperature of goethite, and then cooled back down to 300 K in the presence of a strong field (2.5 T). This protocol attempts to maximize any magnetization carried by goethite, acquired during cooling through its Néel temperature (~393 K). The resulting remanence was measured during

![Figure 1](image-url)

(a) Measurement of low temperature remanence acquired after field-cooling (FC) (blue) and zero-field-cooling (ZFC) (red) during warming to 300 K and (b) measurement of room temperature remanence (RTSIRM) during both cooling (blue) and warming (red).
a cooling and warming cycle between 300 K and 10 K (Fig. 2a). The sample was then removed from the MPMS and AF demagnetized using a peak field of 200 mT. Any contribution from low-coercivity components (e.g., magnetite/titanomagnetite) should be therefore removed. The remaining remanence measured using the same protocol (RTSIRM) should be largely carried by goethite, if this mineral is present. Remanence after AF demagnetization (Fig. 2b) was one to two orders of magnitude weaker than that before AF demagnetization (Fig. 2a), indicating that remanence is dominated by a low-coercivity component that has been largely removed. Subsequent heating (to 400K, above the Néel point of goethite) and cooling curves are very similar to the initial cooling curve implying no discernible contribution to remanence from goethite.

The results from IRM visit led us to look for other explanations for the high coercivity component apparent in some u-channel data. When the 2G Enterprises Model 615 controller used to apply the DC bias field for acquisition of anhysteretic remanent magnetization (ARM) was set to zero output in the computer mode, we measured an offset current of ~600 µA. This offset current was found to generate a DC bias field of ~300 nT when the ARM controller was supposedly inactive. Further investigation indicated that the zero reference voltage potentiometer (R32) in the ARM controller required adjustment. We recommend the output field be periodically checked and R32 adjusted, as necessary, to avoid this problem. We have not noticed this spurious ARM component in other samples, other than these samples from the equatorial Pacific dominated by a primary magnetization component with low inclination (due to low paleolatitudes). For some samples from this region, the occasional presence of a steeply inclined component during AF demagnetization can now be attributed to a spurious DC field emanating from the ARM-bias-field circuit that leads to a spurious ARM being acquired along the axis of the u-channel sample (the Z axis of the magnetometer) during standard AF demagnetization.

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References


The IRM Quarterly is always available as a full color pdf online at www.irm.umn.edu
The IRM at GSA
October 9-12, 2011
Minneapolis, Minnesota, USA

The Geological Society of America (GSA) will hold its annual meeting this year in Minneapolis. In conjunction with the meeting, the IRM will be participating in a number of activities. Meeting abstract deadline: July 26. Early registration deadline: September 6.

Pre-Meeting Short Course:
Environmental Magnetism
_Saturday, October 8_

Magnetic minerals can be sensitive indicators of environmental change. Changing (paleo)climate, weather patterns, land use patterns, or air and soil pollutants can control the composition, concentration and grain size of magnetic minerals deposited in sediments or soil. This half-day short course will introduce participants to basic rock magnetic properties, the influence of environmental factors on magnetic minerals and their properties, and the standard laboratory techniques and instrumentation. Through the use of several case studies and demonstrations, we will examine the advantages and limitations of the simplest and most common magnetic methods, as well as some more advanced techniques.

Organizers: Bruce Moskowitz (IRM), Julie Bowles (IRM), Richard Reynolds (USGS)

Meeting Sessions

Geology and Mineral Deposits of the Midcontinent Rift

This session highlights the renewed interest in the tectonomagmatic evolution and metallogenesis of the Midcontinent Rift generated by recent geochronologic, geochemical, and geophysical studies and new discoveries of Cu-Ni-PGE deposits associated with the 1.1 Ga Midcontinent Rift.

Convenors: James D. Miller (U. Minnesota - Duluth), Suzanne Nicholson (USGS), R. Michael Easton (Ontario Geological Survey), Joshua Feinberg (IRM)

Mineralogy, Geochemistry, and Physical Properties of Atmospheric Mineral Dust: Influences on the Atmosphere, the Cryosphere, Ecosystems, and Humans

This session emphasizes the mineralogy, geochemistry, and physical properties of contemporary dust to understand the influences of atmospheric particulate matter on climate, weather, snow- and ice-melt, human health, landscape fertility, and ocean fertilization.

Convenors: Richard Reynolds (USGS), Josh Feinberg (IRM), Suzette Morman (USGS)

Post-Meeting Field Trip

_Date TBA: Anatomy of a Mineralized (Cu-Ni-PGE) Mafic System, the South Kawishiwi Intrusion of the Duluth Complex_

The troctolitic South Kawishiwi Intrusion (SKI) of the Duluth Complex is one of Earth’s great untapped resources of Cu-Ni-PGE and is actively being explored by four companies (Duluth Metals Limited, Twin Metals Minnesota, Franconia Minerals Corporation, and Encampment Resources). Together, these companies have qualified, through NI 43-101 resource estimates, 1.95 billion tons of indicated and inferred resources in five deposits (Nokomis, Spruce Road, Birch Lake, Maturi, and Serpentine), including 23.3 billion pounds of Cu, 7.8 billion pounds of Ni, 456 million pounds of Co, and 15.0 million ounces of Pd, 6.7 million ounces of Pt, 3.4 million ounces of Au, and 55 million ounces of Ag. Establishing the initial conditions of the magmatic system is the most essential part of analyzing and understanding the processes that formed the SKI and its related ores. The fundamental initial conditions of the SKI that will be addressed during this field trip include: (1) the geometry of the system, footwall and hangingwall rocks; (2) the composition and initial state of crystallinity of the SKI magmas; (3) the three-stage sequence of emplacement of the magmas and their relationship to the Nickel Lake Macrodike; and (4) the location of channelized magma flow zones, their thermal anomalies and resultant ores. Particular attention will be given to the use of geophysical techniques such as anisotropy of magnetic susceptibility (AMS) for ascertaining the direction of magma emplacement in the ore deposit.

Trip Leaders: Dean Peterson (Duluth Metals), Josh Feinberg (IRM), Kevin Boerst (Duluth Metals), and Evan Finnes (IRM)
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 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 10,000 references, is available free of charge. Your contributions both to the list and to the Abstracts section with more than 10,000 references, is available free of charge.

Magnetic Fabrics and Anisotropy


Archeomagnetism


Bio(geo)magnetism


Environmental Magnetism and Paleoclimate Proxies


Extraterrestrial Magnetism


Geomagnetism and Geodynamo Studies

Asari, S., V. Lesur, and M. Mandea (2010), Geomagnetic secular variation violating the frozen-flux condition at the core surface, Earth Planets Space, 62(9), 693-709.


Magnetic Field Records


Rock and Mineral Magnetism


Mineral Physics and Chemistry


Tectonics/Palaeomagnetism


Chronostratigraphy/Magnetostratigraphy


Other


Pyrrhotite “Besnus” Transition, cont’d. from pg. 1

Fig. 2a. We got immediately excited by the similarity to the Verwey transition. We decided to measure the warming curve from 4 K as well along with the measurement of other, more pyrrhotite-rich, samples. Pierre took a Swiss schist sample rich in multi-domain (MD) pyrrhotite (OJ4 from Rochette, 1987) while Gérard dug into the archives of his lab and soon came up with the spherical single crystal René Pauthenet1 used for his pyrrhotite papers (Pauthenet, 1952; Fig. 3a; Bin and Pauthenet, 1963). Both samples showed a very sharp change in magnetic behavior as function of temperature (Figs 3a and b): no doubt we had discovered a magnetic transition characteristic of pyrrhotite.

A few weeks later, Pierre’s PhD thesis (defended in January 1988) was printed with those curves included in a small last-minute chapter on this transition. An abstract was submitted to the annual EGS (European Geophysical Society, now European Geoscience Union) meeting in Bologna in March (Rochette and Fillion, 1988). Of course we planned to go further: Pierre planned various experiments that exploited the sensitivity of magnetic instruments to demonstrate the utility of this magnetic transition as a means of identifying and characterizing low concentrations of pyrrhotite in rocks. Meanwhile, Gérard took up the task of digging into the physics of the transition by working on Pauthenet’s monocrystal and looking for other synthetic samples. He measured induced magnetization curves as a function of temperature, both parallel and perpendicular to the room temperature (RT) easy plane. He

1 René Pauthenet played a major role in Grenoble, following in the footsteps of Louis Néel, as the director of SNCI (the very high field facility) and INPG (Polytechnic Institute of Grenoble). He died in 1987. Another important actor from Grenoble in the pyrrhotite story was Felix Bertaut (alias Erwin Lewy, his real prewar given name) who first proposed the lacunare crystallographic structure of pyrrhotite (Bertaut, 1953) with ordered iron vacancies in sets of iron planes alternating with fully occupied Fe planes, giving a natural explanation of the observed ferrimagnetism (see the first neutron diffraction experiments in Sidhu et al., 1959, confirming the predictions of Néel, 1953). In fact, the written comments on the 1964 Besnus and Meyer communication indicate that Bertaut was the first to suggest a comparison between the Verwey transition and the behavior of pyrrhotite around 30 K (without suggesting a real transition for pyrrhotite). Néel himself was interested in pyrrhotite (e.g. Néel, 1953) and, as a major referee (“examinateur”) of the thesis of Marie-Jeanne Besnus, played his part too.
discovered that on cooling, there is a continuous increase of the angle between the spontaneous magnetization and the RT easy plane, as well as an abrupt change of this angle at 32 K, thus explaining the remanence behavior (Fig. 3b). Pauthenet could not have identified the transition because he measured only at two temperatures: 20 and 50 K (using a liquid hydrogen dewar). Gérard submitted an abstract on these measurements to the August ICM (International Conference on Magnetism) in Paris, and a proceedings paper was published in December (Fillon and Rochette, 1988).

Soon after Pierre’s thesis defense we realized that Fig. 2b was not correct: Pierre had plotted the absolute value of magnetization because the SHE printout did not show the sign of the (remanent) magnetization. At that stage only the peak-to-peak value was recorded. To get its sign, you had to follow the signal visually to see whether the first peak (the signal consisted of a positive and negative peak) was negative or positive and relate a change in peak order to a sign change of the magnetic moment. In fact we had measured a self-reversal of RT IRM through the transition. The correct curve was published in Fillon and Rochette (1988), as well as in Rochette et al. (1990). In September 1988, Jean-Luc Mattei started his thesis on the pyrrhotite transition under the supervision of Gérard. He defended his thesis in 1990, including low temperature magnetization measurements, Mössbauer spectroscopy (Jeandey et al., 1991) and neutron diffraction (Fillion et al., 1992).

On the rock magnetic side Pierre took advantage of the Bologna forum to gather further pyrrhotite bearing samples (e.g. from the Himalayan remagnetized limestones through E. Appel and from an Algerian granite through J.L. Bouchez) and to propose to Mark a collaboration on his well characterized suite of grain size fractionated pyrrhotite samples (Dekkers, 1988). The aim was to understand the difference between the SD and MD samples of Figure 2a and 2b and to eventually devise a grain size estimate from the low temperature behavior. Mark came to Grenoble during the summer of 1988 and measured a first set of samples. The remainder were measured by Jean-Luc in the autumn. Results showed that the reversibility of the remanence drop was a sensitive proxy of pyrrhotite grain size. An abstract was submitted to the 1988 AGU Fall meeting (Rochette et al., 1988), where the pyrrhotite transition gained a wide audience. Mark and Pierre (both present in San Francisco) decided the following publication plan: Mark would present the grain size variation of the transition on his samples in a GRL paper (taking advantage of a special issue planned for the rock magnetic session in San Francisco), and Pierre would present a longer paper reviewing all rock magnetic aspects of the transition with the aim of identifying pyrrhotite in a wide variety of rocks. The latter included weakly magnetic remagnetized limestones, the magnetic mineralogy of which was also a puzzle at that time. Mark was faster and submitted his paper in May (Dekkers et al., 1989) while Pierre submitted in October to EPSL (Rochette et al., 1990). This last paper also included for the first time LT IRM heating curves in which the IRM was imparted at 4 K. From those measurements it appeared that a RT IRM cooling curve to pinpoint the pyrrhotite transition is most appropriate.
Prehistory of the transition

In 1988 Gérard also dug into the literature and found that Besnus and Meyer (1964) published in the proceedings of the ICM conference in Nottingham (1964) a very similar (though less detailed) magnetization curve on a single crystal (cf. Fig. 3c versus 3b). Marie-Jeanne Besnus was preparing a PhD thesis on pyrrhotite in Strasbourg, using the crystals of Weiss and Forrer (1929). In addition to magnetization as function of temperature, she measured the low temperature variation of electrical resistivity and found an anomaly, again around 30 K (Fig. 4a). She also noticed that an anomaly (qualified as a “broad hump” by the original authors) of specific heat was identified near 30 K by Gronvold et al. (1959) (Fig. 4b). While Gronvold et al. did not make any comment on the origin of that “hump”, Besnus, by pointing to the coincidence of an anomaly in three independent physical properties (induced magnetization, resistivity and specific heat), implicitly suggested that a phase transition was present near 30 K. Unfortunately, she also identified a transition near 8 K and put much more attention on the latter because pyrrhotite was apparently becoming superconductive below 8 K. We use the wording ‘unfortunate’ because soon after the 1964 publication it was realized that the behavior below 8 K was an instrumental artifact, likely produced by a tin welding in the dewar (tin becomes superconductive below 11 K). The discredit of the 8 K anomaly may explain why the 30 K anomaly remained forgotten. However, in her thesis (Besnus, 1966) on pyrrhotite, M.-J. Besnus wrote of these changes near 30 K (p.75): “ces anomalies ne sont pas purement magnétocristallines... mais ...sont en relation avec une transformation soit du deuxième ordre, soit d’ordre supérieur. On pourrait envisager l’existence d’une transition ordre-désordre comme dans le cas de la magnétite” which can be translated into: “these anomalies are not purely of magnetocrystalline origin but are related to a transition of second or higher order. One can invoke an order-disorder transition as in the case of magnetite” (she is referring to the Verwey transition: she noted earlier in the text that the thermal behavior of pyrrhotite was similar to that of magnetite across the Verwey transition). It is clear in that text that M.J. Besnus has explicitly proposed a transition to explain pyrrhotite behavior around 30 K. Although she did not pursue the study of this transition, it is evident she was its original discoverer and that we only rediscovered it 25 years later. Therefore we propose to name this transition the Besnus transition. During the preparation of this article, Pierre started an email correspondence with her, in which she was delighted to see that her earliest work had gained so much attention after several decades. She was flattered to have her name proposed. Sadly, she passed away shortly afterwards.

Epilogue

Over the decades following our “rediscovery” of the pyrrhotite LT transition we have been repeatedly faced with three difficult questions: 1) Why don’t we name this transition, as we do for magnetite and hematite? 2) What references should be cited for it? and 3) What’s happening at the atomic level near 32 K in pyrrhotite? We have already answered the first question. If one excludes thesis manuscripts and congress abstracts without proceedings, the following are our suggestions for references. Citing Besnus and Meyer (1964) should account for the naming

2 assuming that transformation and transition are synonyms (note by present authors)
3 The note by Ferrow et al. (2006) “Pauthenet (1952) was the first to recognize the existence of magnetic transitions in pyrrhotite at temperatures between 30 and 34 K” is not correct as Pauthenet did not write anything suggesting he was suspecting a transition from his data. In fact, the precision of his curves (Fig. 3a) does not allow identification of discontinuous behavior.
of the Besnus transition (although the real first appearance of the transition in writing is in her 1966 thesis). Depending on the focus of the quotation, one should also cite Dekkers and Rochette (1988) for a more solid state physics aspect, Dekkers et al. (1989) for the grain-size variation of RT SIRM curves, and Rochette et al. (1990) for a general overview of the rock magnetic use of the Besnus transition (although an earlier quote is in Rochette’s 1988 thesis). The number of citations presently found in the ISI database can show how past publications have tackled this question (indicated in reference list). Finally, until now, the intricacies of the transition have remained not clearly resolved, aside from the analogy with the Verwey transition (Besnus, 1966) and powder neutron diffraction and Mössbauer spectroscopy work indicating the transition has a crystallographic rather than magnetic origin (Jeandey et al., 1991; Fillion et al., 1992; see also the discussion in Ferrow et al., 2006). However, based on highly detailed single crystal neutron diffraction studies through the transition and anisotropy measurements below 32 K, Wolfers et al. (2011) have just proposed that the transition corresponds to the transformation of the RT monoclinic structure into a LT triclinic structure, most probably due to temperature dependence of the Jahn-Teller effect (a crystallographic effect that slightly distorts the shape of the Fe octahedra). This transformation generates new magnetic domains with easy axes at angles of approximately ± π/4 from the RT domains. New more precise measurements are currently done in Grenoble especially in specific heat, torque and magnetization, resistivity and magnetoresistance, along with band calculations. Stay tuned for the next publication!

References
(Number of ISI citations in parentheses)


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**Visiting Fellows**

**January - June, 2011**

**Stefanie Brachfeld**
Montclair State University
*Geomagnetic assisted chronologies in the Antarctic Peninsula Region*

**Ada Dominguez**
University of Michigan
*Testing the Current Geomagnetic Field Model with Flows from the Aral Formation, and the Columbia River Basalts*

**Myriam Kars**
Ecole Normale Supérieure
*Investigating nanoparticles and 24 K transition*

**Sophie-Charlotte Lappe**
University of Cambridge
*Investigation of the magnetic properties of synthetic dusty olivines*

**Ursina Liebke**
University of Tübingen
*Reverse polarity directions in subvolcanic, basaltic andesitic dykes - reversal record or a self-reversal magnetization?*

**Stella Lucifora**
RomaTRE University
*Magnetic mineralogy analyses on sediments from Adana basin (southern Turkey)*

**Christoph Mang**
Karlsruher Institut für Technologie
*Rock magnetic properties of the Chesapeake Bay impact structure, Virginia, USA*

**Johanna Salminen**
Yale University
*Early Mesoproterozoic supercontinent Nuna*

**Ron Shaar**
The Hebrew University of Jerusalem
*Magnetic Imaging of TRM in MD Synthetic Dendrites: Implication for Absolute Paleointensity Studies*

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*AMS Fabrics and Deformation of the San Andreas Fault Borderlands*

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