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Inside...

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

Figure 1. Intergrown pyrrhotite crystals from Chihuahua, Mexico. Photo by Rob Lavinsky, iRocks.com (via Wikipedia Commons).

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

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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 Pauthenet[1] 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 lacunar 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 (Fillion 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 Fillion 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.

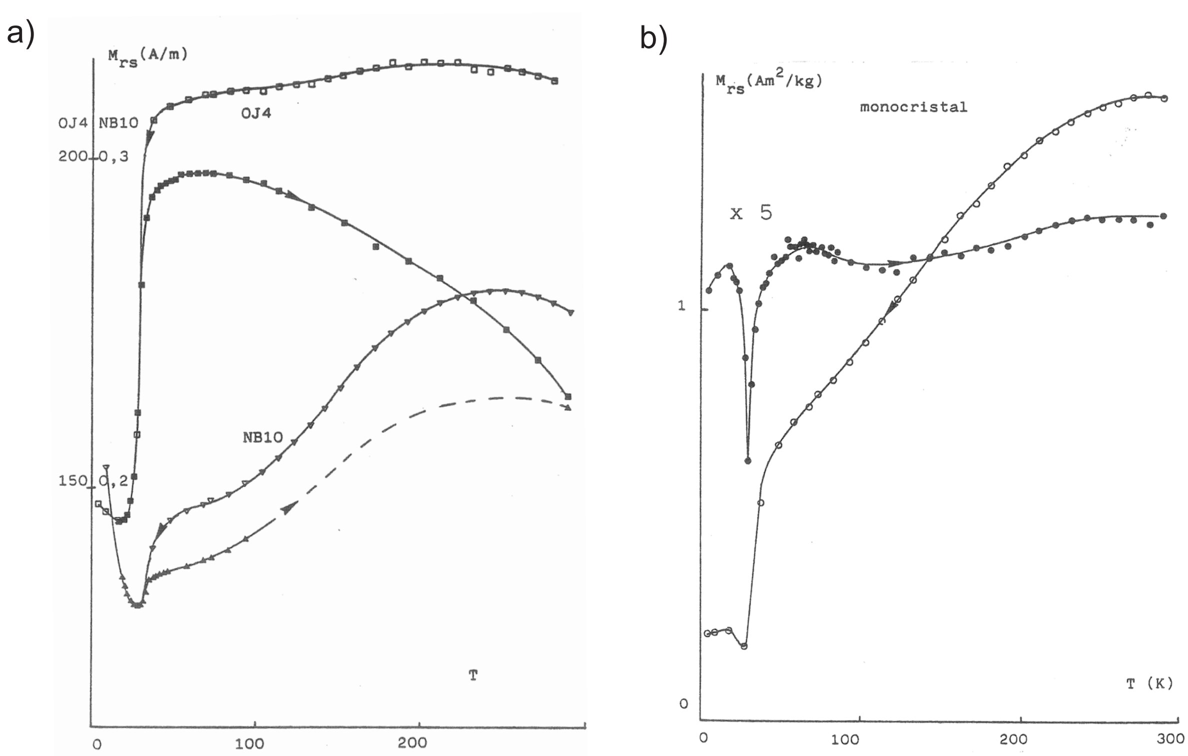

Figure 2. Absolute value of RT SIRM under zero-field cooling and subsequent heating measured with the SHE cryogenic magnetometer in Grenoble on a) two pyrrhotite bearing rocks, and b) a monocrystal (within the RT easy plane, i.e. perpendicular to the crystallographic c axis) Original curves as published in Rochette (1988).

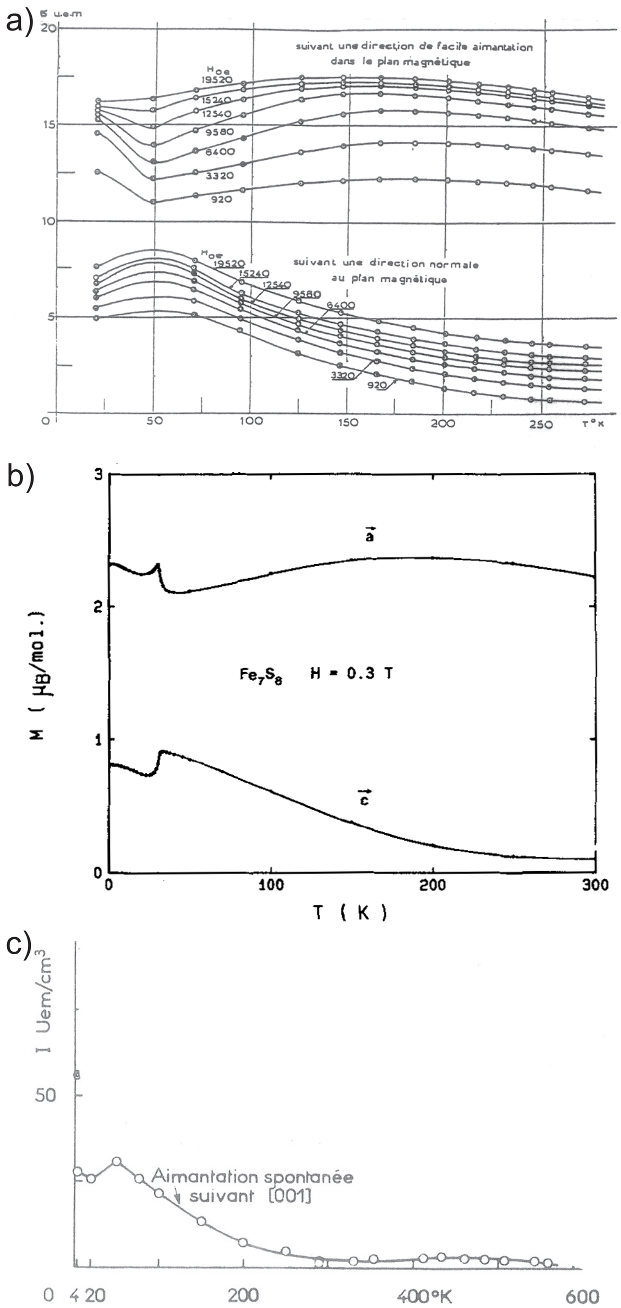

Figure 3. Induced magnetization curves parallel and perpendicular to the c axis of the same single crystal of pyrrhotite measured by (a) Pauthenet (1952) and (b) Fillion and Rochette (1988). c) Same data on another single crystal from Besnus and Meyer (1964).

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[2] 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[3]. 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 Fillion 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!

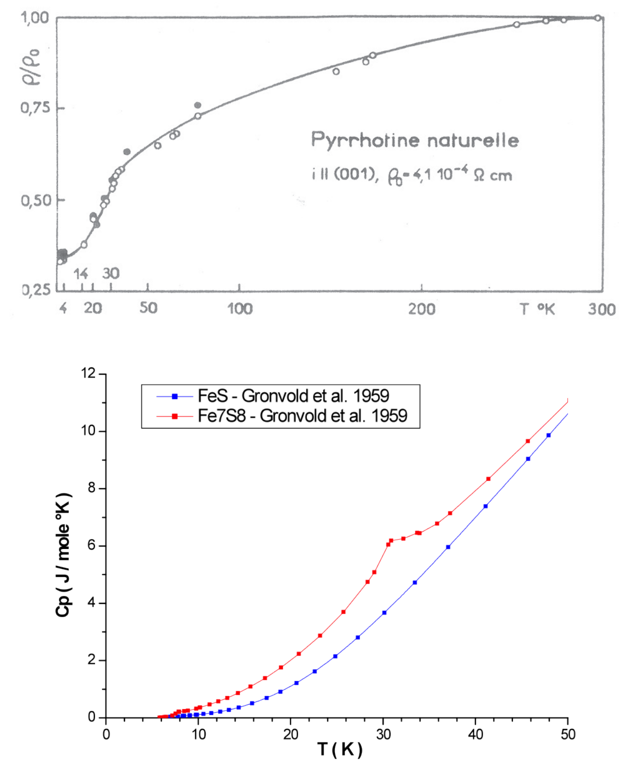

Figure 4. a) electric resistivity of pyrrhotite versus temperature, original curve of Besnus and Meyer (1964); b) heat capacity versus temperature for pyrrhotite and troilite redrawn after Gronvold et al. (1959).

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d. 2011, Strasbourg, France

Marie Jeanne BESNUS (née Swiderski) was born into a working class family in northeastern France. After a brilliant high school career in Thionville, she studied physics at the University of Strasbourg and married Yves Besnus. In 1960 she took a position at the CNRS (the French national research organization) based in Strasbourg, to prepare a PhD thesis on the magnetic properties of pyrrhotite under the supervision of André Meyer. She graduated in 1966. She spent her entire career as a CNRS researcher, working on the low temperature properties of metallic compounds, in particular those with the Kondo effect. She had close collaborations with the Louis Néel Laboratory, and the Institut Laue-Langevin, both in Grenoble. Although soon partially disabled by myopathy, she published more than 100 papers in experimental solid-state physics, before retiring in 1996. Her most cited paper (192 times in ISI) is about the compound $CeRu_2Si_2$, using magnetic, electric and thermal properties at low temperature (Besnus et al., 1985), just like in her work on pyrrhotite described here. At the end of her career, she participated in initiatives for the promotion of scientific vocations among women. We hope naming the pyrrhotite transition after her will contribute to that effort.

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