Unexpected Magnetic Behavior of Natural Hematite-Bearing Rocks at Low Temperatures

Alexandra Abrajevitch, Andrew P. Roberts, Brad J. Pillans, and Rie S. Hori

Institute of Tectonics and Geophysics, Russian Academy of Sciences, Khabarovsk, Russia, Research School of Earth Sciences, Australian National University, Canberra, Australia, Department of Earth Sciences, Ehime University, Matsuyama, Japan

Abstract Hematite is a commonly occurring magnetic mineral in nature that has numerous scientific and technological applications. A characteristic property of hematite is a low-temperature spin-flop transition called the Morin transition. Above the transition temperature, hematite is a canted antiferromagnet that can carry a remanent magnetization. Below this transition, spin canting disappears and hematite becomes a true antiferromagnet although a small defect moment is usually preserved. We observe Morin transition behavior in natural samples that has not been reported before for hematite. During repeated thermal cycling of a remanent magnetization acquired at room temperature, the remanence intensity at the end of the cycle oscillates between a high remanence state at the end of odd-numbered cycles and a low remanence state (LRS) at the end of even-numbered cycles. Alternation of the high and LRSs during repeated thermal cycling points to hysteretic behavior of the spin-flop process, likely due to sublattice magnetization alignment switches along different easy magnetization axes in samples with preferred crystallographic orientations of hematite particles. We report these observations to seek to expand explanations of the magnetism of hematite.

1. Introduction

Hematite (α-Fe₂O₃) is the most stable and widespread iron oxide mineral in nature. Hematite has a corundum crystal structure with alternating iron and oxygen layers stacked along the crystallographic c-axis of the hexagonal unit cell. At low temperatures, Fe³⁺ spins order antiferromagnetically (Shull et al., 1951), so that moments in two neighboring Fe layers are exactly antiparallel and aligned along the c-axis (Figure 1). At ~260 K, hematite undergoes a spin-reorientation transition, known as the Morin transition. Upon heating through this transition, spins rotate by 90° with respect to the basal plane, within which they lie with a slight spin canting that results in a weak net magnetization (I. Dzyaloshinsky, 1958; I. E. Dzyaloshinsky, 1957; Moriya, 1960) that is related to antisymmetric exchange interactions between atomic spins. Spin flipping at the Morin transition is thought to arise from competition between the magnetic dipolar interaction and the single-ion anisotropy that arises from higher order spin-orbital effects, which have different temperature dependencies (Artman et al., 1965). Single-ion anisotropy favors spin alignment along the c axis and dominates at low temperatures. As temperature increases, single-ion anisotropy decreases faster than the magnetic dipolar interaction, which favors basal plane spin alignment so that the spins flop. The balance between the two anisotropies that control the Morin transition is affected significantly by small hematite lattice changes due to cation doping (Morin, 1950), applied pressure (Worlton & Decker, 1968), applied magnetic fields (Simkin & Bernheim, 1967), decreasing grain size (Takada et al., 1966), or nanoparticle shape variations (Sorescu et al., 1998). For stoichiometric hematite at atmospheric pressure, the transition occurs at ~262 K (Morrish, 1994). Impurity cations, structural defects, and smaller grain sizes shift the transition to lower temperatures and smear reorientation of magnetic spins over a larger temperature range (Figure 2). The transition temperature decreases with decreasing particle size; from ~250 K for 0.1 mm crystals to 190 K for 30 nm particles, until it disappears entirely in <20 nm particles (Özdemir et al., 2008).

The presence of structural defects in hematite crystals may also prevent complete magnetic moment alignment below the Morin transition, and a fraction of the remanence, the so-called “defect moment” (Dunlop, 1970, 1971), may remain at low temperatures (Figure 2). On subsequent warming, the remanence recovers partially; the recovery rate is also a function of structural defect characteristics (Özdemir & Dunlop, 2006). Usually, there is a net remanence loss on cycling through the Morin Transition due to defect unpinning (Figure 2). With repeated thermal cycling, defects gradually heal to result in almost identical cooling and heating runs (Figure 3).
In a few reported cases, thermal cycling leads to a net remanent magnetization increase (De Boer et al., 2001). The net remanence increase has been observed previously only in multi-domain magnetic particles and was attributed to domain wall configuration changes. Repeated thermal cycling in this case also leads eventually to establishment of a local energy minimum state, and to identical cooling-warming curves during repeated runs. The stability of the new magnetic state to alternating field (AF) demagnetization is low compared to untreated hematite; AFs of 30 mT can erase 90% of the remanence increase, and complete recovery of the initial remanence state is achieved after treatment at 100 mT (De Boer et al., 2001).

2. Unexpected (Novel?) Magnetic Behavior in Hematite

We report here previously undescribed magnetic behavior in several hematite-bearing rock samples. During repeated thermal cycling of an isothermal remanent magnetization (IRM) imparted at room temperature, the intensity of a residual IRM remaining after low-temperature cycles measured using a Magnetic Properties Measurement System (MPMS) oscillates between a high-intensity state at the end of odd-numbered cycles and a low-intensity state in even-numbered cycles (see Appendix A for experimental details). Such behavior is demonstrated in Marble Bar Chert samples in Figure 4. Previous studies (Kato & Nakamura, 2003; Suganuma et al., 2006) and our experiments (Figures 4a–4f) indicate that the Marble Bar Chert contains hematite, magnetite, and siderite. The Marble Bar hematite has a sharp Morin transition (Figures 4b–4f). A saturation IRM (SIRM) imparted at room temperature (300 K SIRM) follows the expected pattern during the first thermal cycle through the Morin transition (300 K => 150 K => 300 K), with remanence loss on cooling and partial recovery on subsequent warming with net remanence loss (Figure 4d). During the second cycle, SIRM decreases to a similar value at 150 K on cooling; on warming, however, it recovers to exceed the SIRM value at the start of this cycle (to produce a high remanence state, HRS). The third cycle is similar to the first, with a net SIRM decrease after cycling (low remanence state, LRS); the fourth is identical to the second with a net SIRM increase after cycling. The striking oscillating pattern between the HRS at the end of even-numbered cycles and the LRS at the end of odd-numbered cycles was observed for up to 20 repetitions. Unlike De Boer et al. (2001) who reported low stability of a domain-configuration-related HRS, both the HRS and LRS states in the Marble Bar hematite are...
stable to AF demagnetization. Demagnetization at a 170 mT peak field affects neither the HRS nor the LRS, nor does it impede the repetition order (Figure 4e). The insignificant IRM offset before and after AF demagnetization (Figure 4e) is likely due to a small sample position offset with respect to the measurement coils during repeated sample centering.

Similar behavior with regular alternation between a LRS and HRS is also observed in other natural hematite-bearing rocks, including a hematite ore (sample Hem-124) from the geological collection of Ehime University (Japan) geological collection (Hem-124); a red band of zebra rock, Australia (Abrajevitch et al., 2018); a siliceous schist pebble from the Oboke Gorge, Sanbagawa metamorphic Belt, Shikoku, Japan (Aoki et al., 2009); Kamikotan bedded chert, Hokkaido, Japan (Hori & Sakakibara, 1994; Figure 5). Compared to the Marble Bar Chert, the hematite in these samples has finer grain sizes and a higher defect density, as indicated by smearing of the Morin transition over a wide temperature interval (Figure 2). Nevertheless, after a significant net 300 K IRM loss during the first (300 K => 10 K => 300 K) thermal cycle, the samples settle into a regular alternation between the HRS at the end of even-numbered cycles and LRS at the end of odd-numbered cycles, as observed for the Marble Bar Chert, albeit with smaller differences between HRS and LRS intensities. Even a zebra rock sample (Abrajevitch et al., 2018) that appears to have normal behavior at regular resolution with identical repeated cooling-warming cycles (Figure 3) has a similar alternating trend in high-resolution (Figure 5d). Although the intensity difference between the HRS and LRS has small absolute values, it exceeds measurement errors (<13% of intensity differences).

3. Discussion

Regular alternations between high and LRSs in hematite during continuous thermal cycling through the Morin transition points to a hysteretic spin-flop behavior. Such behavior has not been documented previously. Similar behavior in rocks of different ages from widely spaced localities suggests that this feature may be a common
property of fine-grained hematite. The underlying mechanism for this behavior is difficult to ascertain because of the small number of available observations. We explore several possible explanations here. Mineral magnetic properties can be affected by magnetostatic interactions and compositional or structural defects. Magnetic interactions, as is the case with compositional defects, suppress the Morin transition; for example, samples with lamellar magnetism lack a Morin transition (e.g., Fabian et al., 2008). The Marble Bar Chert has a clear alternating pattern with the largest difference between the HRS and LRS, and contains magnetite and siderite. However, the Néel temperature of siderite (e.g., Frederichs et al., 2003; Housen et al., 1996) and the Verwey and Morin transitions, are sharp, which are typical of compositionally and structurally pure minerals and is not typical of mineral intergrowths (Dunlop & Özdemir, 2001). Other samples with similar behavior (Figure 5) contain hematite only; additional magnetic phases are not detected in IRM acquisition or low-temperature experiments. Thus, it is unlikely that such behavior results from magnetic interactions or the presence of other phases.

Magnetic characteristics that are indicative of crystallographic defects—the Morin transition width, and the defect moment—vary widely between samples (Table 1). In our samples with atypical behavior, there is no correlation...
Figure 4. Magnetic data for a Marble Bar chert sample. (a) Isothermal remanent magnetization (IRM) coercivity distribution and (b–e) low-temperature properties. In panel (a), fitted components represent magnetite (gray) and hematite (pink). (b) Low-temperature cycling of an IRM acquired in 7 T at room temperature (300 K IRM). Arrows indicate the Morin transition ($T_M$), the Verwey transition ($T_V$) of magnetite, and the Néel temperature of siderite ($N_s$). A magnetization is induced in siderite by a small (<0.1 μT) residual field during measurement. (c) Low-temperature cycling of a 300 K IRM imparted in a 7 T field after alternating field (AF) demagnetization in a 170 mT peak field (300K IRMAF). Absence of a $T_V$ signal indicates that the magnetite contribution to the 300 K IRM is removed by this treatment; (d) consecutive cycling of the 300 K IRM. Remanence intensities at the end of each cycle alternate between a low remanence state (LRS) in even-numbered cycles and a high remanence state (HRS) in odd-numbered cycles. (e) Remanence values at 300 and 150 K for consecutive 300 K IRMAF cycles. AF demagnetization at 170 mT of the HRS (after the second cycle) and LRS (after the eleventh cycle) does not significantly affect the remanence intensity and does not change the alternations.
Figure 5. Repeated thermal cycling of other hematite-containing samples, including (a) hematite ore #124 from the Ehime University geological collection, (b) Sanbagawa siliceous schist, (c) Kamikotan chert, which is similar to the Marble Bar Chert, although with smaller amplitude, and (d) zebra rock, with regular alternations between the high and low remanence states. The high-temperature part of the zebra rock cycles are magnified in panel (d) to illustrate that they also have the observed high remanence state/low remanence state alternation (see Figure 3).
between defect density and the difference between HRS and LRS intensities (Table 1). Theoretically, systematic defect distributions, or a defect superstructure that is sensitive to temperature, can cause magnetization changes upon thermal cycling, similar to the reversible oxygen-vacancy re-arrangement induced by thermal cycling with a change in system energy detected in nonstoichiometric SrUO$_4$ crystals (Murphy et al., 2018). However, in this case, the change in system energy (or remanence intensity) could be expected to correlate with the number of defects (defect moment), which we do not observe.

More likely, the observed hysteretic spin-flop behavior is an intrinsic feature of hematite. Regular alternations between the HRS and LRS is suggested here to be accounted for by a change in sublattice magnetic moment alignment between different easy magnetic axes. This process is illustrated schematically in Figure 6a. The crystal symmetry of hematite in the basal plane has three equivalent easy magnetization axes (e.g., Fabian et al., 2011; Flanders & Schuele, 1964; Tasaki & Iida, 1963; Wohlfarth, 1955). Above the Morin transition temperature, sublattice magnetic moments lie with slight canting along one easy axis, so that the resultant moment is perpendicular to that axis. When a strong magnetic field is applied to a sample, the IRM will tend to align with the applied field direction. For an individual hematite crystal, depending on the angle between the applied field direction and crystallographic orientation, the remanent moment direction can deviate within ±30° of the applied field direction (Figure 6c). In the MPMS used here, the sample magnetic moment is approximated as a magnetic dipole aligned with the measurement coil axis, so that a single (vertical) magnetic vector component is measured. The maximum measured moment is achieved when the remanent moment is aligned with the MPMS coil axis, that is, when an easy magnetic axis is perpendicular to the applied field (Figure 6b).

The crystal structure of hematite can be regarded to result from covalent bonding interactions between atoms. The magnetic moments of individual Fe atoms depend strongly on nearest-neighbor atoms. Differences in covalent bond lengths (Blake et al., 1966) means that different Fe atoms in the crystal structure have different spin-coupling energy, so that they have different critical temperatures at which the single-ion anisotropy equals the magnetic dipolar interaction energy (the spin-flop temperature). Upon initial cooling, the spin-flop process is directed by atoms with the highest-spin-flop-temperature. The first spins to flip form a minimum energy configuration with atoms with lower spin-flop temperatures. With ongoing cooling, the next spins to flip settle into a minimum energy configuration with both spins that have already flipped and those that have not yet flipped. Such a metastable spin configuration cascade set in motion by atoms with the highest-spin-flop-temperatures results in an antiferromagnetic spin alignment configuration (configuration A in Figure 6a) at low temperatures. Upon subsequent reheating from antiferromagnetic configuration A, the spin alignment structure above the Morin transition follows the similar spin-flop spin-re-arrangement pathway governed by atoms with the lowest-spin-flop-temperatures. Spin re-arrangement beginning from antiferromagnetic state A leads to alignment of resultant sublattice moments along a different easy axis (Figure 6a). Upon cooling from the LRS, the spin flip process is again directed by atoms with the highest-spin-flop-temperature, but with a different starting spin-configuration and system energy, which results in reaching antiferromagnetic alignment B (Figure 6a). Antiferromagnetic configurations A and B have equivalent resultant magnetic moments but do not have equivalent atomic spin arrangements (Figure 6a).

| Sample         | Defect moment, 1st cycle (%) | $T_{\text{shmin}}$ (K) | $\Delta T_M$ WHM (K) | Thermal hysteresis (K) | LRS as a fraction of HRS (%) |
|----------------|------------------------------|------------------------|----------------------|------------------------|------------------------------|
|                | Cycle 1                      | Cycle 3                | Cycle 5              |                        | Cycle 1                      | Cycle 3                | Cycle 5              |
| Marble Bar     | 15$^a$                       | 246                    | 12                   | 1                      | 84.5                         | 88.0                    | 88.0                  |
| Hem-124        | 30                           | 194                    | 40                   | 22                     | 67.2                         | 94.0                    | 95.4                  |
| Sanbagawa      | 65                           | 150                    | 200                  | 20                     | 65.2                         | 93.2                    | 93.6                  |
| Kamikotan      | 85                           | 130                    | 185                  | 26                     | 78.8                         | 94.5                    | 95.5                  |
| Zebra          | 62                           | 180                    | 103                  | 22                     | 80.0                         | 98.4                    | 98.7                  |

$^a$Defect moment for the Marble Bar sample was estimated from the IRMAF cycle as shown in Figure 4c. The Morin transition temperature ($T_{\text{shmin}}$) is defined at the maximum remanence intensity gradient on cooling; the transition width $\Delta T_M$ is defined as a full width at half maximum (WHM) of the gradient; thermal hysteresis is defined as the difference in $T_{\text{shmin}}$ value between cooling and warming cycles. For definition of the low remanence state (LRS), high remanence state (HRS), and cycles see Figures 4 and 5.
Upon subsequent warming, the spin-flop-chain process starting from antiferromagnetic configuration B leads to the final HRS spin alignment.

A switch to another easy axis—which involves rotation of the sublattice magnetization by ±60°—changes the remanent moment orientation relative to the measurement coils, which changes the measured intensity. The maximum difference between HRS and LRS intensities is expected when the remanent moment is initially perfectly aligned with the applied field, and then switches to an alternative axis; the switch will reduce the measured magnetization component by a factor of Cos 60°, or 0.5 (Figure 6b). For crystals with initial remanent moment at the critical 30° angle to the coil axis, the magnetic alignment switch to a different easy axis will not change the measured intensity (Figure 6c). For particles with intermediate 0° to ±30° angles between the initial remanence vector and the MPMS measurement coils, the HRS can be expected to decrease by a factor of 1–0.5, depending on the initial alignment (Figure 6d).

If our explanation of the observed behavior is correct—that alternating changes between high and LRSs reflect a switch between easy axes—it also explains why the hysteretic spin-flop process has not been described before. The remanence intensity change associated with the changing sublattice moment alignment will only be detected in coherent samples with some degree of bulk crystallographic alignment, and then only in fortuitous sample orientations relative to MPMS measurement coils. Such magnetic property measurements are often conducted on large single crystals, in which domain wall structures can impose additional constraints on sublattice
moment alignments or on samples with random particle orientations, such as detrital sedimentary rocks, ground rock samples, or on assemblages of randomly oriented synthetic particles (e.g., Morrish, 1994). In contrast, we measured small rock samples in which hematite particles formed under the influence of diagenetic (Zebra Rock and Hem-124) or metamorphic (Marble Bar Chert, Sanbagawa schist) processes that can induce bulk crystallographic alignment. The median hematite size in the Kamikotan chert sample appears to be larger than in typical bedded chert sequences, in which hematite pigment is too small to record a Morin transition (Abrajevitch, 2020; Abrajevitch et al., 2011). The increased median particle size is likely due to grain growth (Ostwald ripening) during low-grade metamorphism in these rocks (Hori & Sakakibara, 1994), which also tends to create preferred crystallographic mineral alignment.

To test for a relationship between the consistent hematite particle orientation and the HRS/LRS alternations, we compared magnetic measurements for a coherent rock and a ground rock with randomized particle orientations using sub-samples of the Hem-124 ore. The Marble Bar Chert sample is the obvious choice for such a test because of the largest HRS/LRS difference, but it is hard and contains metastable siderite that transforms to other magnetic phases during grinding (e.g., Hillebrand, 1908), which could complicate magnetic experiments. In contrast, the Hem-124 ore sample lacks metastable phases and disintegrates easily at low applied pressures, so it has the lowest chemical and structural alteration potential of our studied samples. A sub-sample of gently crushed Hem-124 ore was measured following a similar protocol as for the rock chip measurements (see Methods in Appendix A). In contrast to the rock chip (Figure 5), alternations between the HRS and LRS are not evident for the ground sample; instead, gradual remanent intensity loss is observed in subsequent cooling cycles, which is typical of slow defect annealing (Figure 7). This difference is consistent with our explanation that the observed rhythmic remanence changes in hematite at room temperature upon cycling through the Morin Transition reflect regular switching of sublattice magnetization alignments between easy magnetization axes in hematite samples with preferred crystal alignments.

The more complex than previously described spin-flop behavior of single domain hematite, as reported here, requires comprehensive quantum mechanical explanation. Using an electron’s spin, instead of its charge, to transmit information promises advantages of higher speed and lower power consumption in electronics (Wolf et al., 2001). A better understanding of spin-flop processes in hematite may help in developing novel materials capable of controlling and transferring electron spins.

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**Figure 7.** Repeated thermal cycling for ore sample Hem-124 after grinding. Unlike the coherent rock chip (Figure 5a), there is no alternation between high remanence state and low remanence state for this sample.
4. Conclusions

We observe novel low temperature behavior in several hematite-containing rocks. On repeated cycling through the Morin transition, the intensity of an IRM imparted at room temperature oscillates between a high-intensity state at the end of odd-numbered cycles and a low-intensity state at the end of even-numbered cycles during repeated thermal cycling. We propose that alternating remanence intensities is an intrinsic property of hematite that reflects a hysteretic switch in sublattice magnetization alignments between different easy magnetization axes in samples with preferred crystallographic orientations of hematite particles.

Appendix A: Materials and Methods

The magnetic properties of a Marble Bar Chert sample and a hematite ore sample (Hem-O) from Tom Price mine, Australia, were measured with a Quantum Design Magnetic Property Measurement System (MPMS; model XL7) at the Australian National University. An isothermal remanent magnetization (IRM) was imparted in a 7 T field at 300 K (300 K IRM). The field was applied for 1 min, switched off, and the MPMS magnet was reset. The remanence was measured during cooling from room temperature (300 K) to 10 K and then during warming back to 300 K in zero field. Measurements were made using the Reciprocating Sample Option in Center mode with 3 cm amplitude and with continuous temperature sweep mode at 5 K/min. A 2-G Enterprises superconducting rock magnetometer equipped with in-line AF demagnetization coils was used for AF demagnetization experiments. The Marble Bar Chert sample was removed from and then reinstalled into the MPMS after AF treatment. The insignificant IRM offset before and after AF demagnetization (Figure 4e) is likely due to a small sample position offset with respect to the measurement coils during repeated sample centering.

The low-temperature magnetic properties of a hematite ore sample (Hem-124) from the Ehime University geological collection, Zebra Rock, Sanbagawa schist, and Kamikotan chert samples were measured at the Center for Marine Core Research, Kochi University, Japan. An IRM was imparted in a 7 T field at room temperature with a MMPM-10 pulse magnetizer (Magnetic Measurements Ltd.). Changes in IRM intensity during low temperature cycling were measured with a Quantum Design MPMS (model XL5). The measurement protocol was similar to that for the Marble Bar Chert sample; the IRM was measured in zero field at a heating rate of 5 K/min.

Coercivity spectra of samples were evaluated with IRM acquisition experiments. In the Hem_TP, Zebra (Figure 2), and Marble Bar (Figure 4a) samples, IRMs were imparted with 26 logarithmically spaced field steps to a maximum field of 7 T using the MPMS at the Australian National University. In the Hem-124, Sanbagawa schist, and Kamikotan chert samples (Figure 2), IRMs were imparted with 75 logarithmically spaced steps to a maximum field of 1.5 T using a Princeton Measurements Corporation vibrating sample magnetometer at the Center for Marine Core Research, Kochi University, Japan.

Data Availability Statement

The underlying magnetic measurements data is archived in the Zenodo data repository and is freely and publicly available online, and may be accessed directly as http://doi.org/10.5281/zenodo.5568867.

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