

Ferroelastic control of magnetic domain structure: Direct imaging by magnetic force microscopyS. D. Seddon^{1,*}, C. R. S. Haines², T. P. A. Hase³, M. R. Lees³, L. M. Eng^{1,4}, M. Alexe³, and M. A. Carpenter⁵¹*Institut für Angewandte Physik, TU Dresden, D-01069 Dresden, Germany*²*Physics, Faculty of Science, University of East Anglia, Norwich NR4 7TJ, United Kingdom*³*Department of Physics, University of Warwick, Coventry CV4 7AL, United Kingdom*⁴*ct.qmat: Dresden-Würzburg Cluster of Excellence—EXC 2147, TU Dresden, 01062 Dresden, Germany*⁵*Department of Earth Sciences, University of Cambridge, Downing Street, Cambridge CB2 3EQ, United Kingdom*

(Received 28 March 2024; accepted 1 July 2024; published 5 August 2024)

Pyrrhotite Fe_7S_8 provides an example of exceptionally strong magnetoelastic coupling through pinning of ferromagnetic domains by ferroelastic twins. Using direct imaging of magnetic domains by magnetic force microscopy (MFM), and thus elucidating the underlying ferroelastic domains, the mechanism by which this coupling controls the local magnetic switching behavior of regions on the pyrrhotite surface is revealed, and leads to quantitative fitting of field-dependent MFM phase shifts with bulk magnetometry data. It is shown that characteristic inflection points in the magnetometry data along certain directions, in particular $[\bar{1}20]_h^*$ of the hexagonal parent structure, are in fact caused by ferroelastic pinning of the magnetic moments.

DOI: [10.1103/PhysRevB.110.L060404](https://doi.org/10.1103/PhysRevB.110.L060404)

The mineral pyrrhotite Fe_7S_8 has been of interest to the paleomagnetic research community due to both its terrestrial abundance and large remanent magnetization [1], making it crucial for understanding the magnetic and tectonic history of the Earth [2]. Beyond terrestrial paleomagnetic applications, pyrrhotite is also of interest due to its presence in meteorites which originated from Mars [3–5]. Given the violent entry to Earth, where large pressure and heat changes are inevitable, specifically understanding the role that strain can have on the relevant magnetic properties is essential to interpreting meteorite samples [6–9]. Pyrrhotite has also recently been identified as a contender for improving the efficiency of Li-ion batteries [10], and as a liquid-exfoliated two-dimensional (2D) material [11]. In the context of the present study, it provides a remarkable example of how the magnetic properties of an effectively multiferroic (ferroelastic/ferrimagnetic) material can be determined by the configuration of ferroelastic domains.

There is an intimate relationship between atomic structure and magnetic structure in each of the different superstructure forms of natural pyrrhotites. Powell *et al.* [12] provides seminal results of structural refinements of the most common, 4C form, so-called because it has a superlattice repeat which is four times that of the c -dimension of the parent NiAs structure due to ordering of vacancies on the cation site. Vacancy ordering and magnetic ordering in the 4C structure combine to give potentially complex domain structures with contrasting dynamical properties. The change in point group symmetry due to vacancy ordering, $6/mmm \rightarrow 2/m$, occurs by the development of two symmetry-breaking shear strains to produce ferroelastic twin domains with up to six different orientations.

If there are six possible domain orientations for vacancy ordering, there must also be six possible orientations for the ferrimagnetic moment [13], each of which can have positive and negative directions. It follows that microstructures associated with the symmetry change $P63/mmc \rightarrow C2'/c'$ must contain both purely magnetic 180° domain walls, and a diversity of walls which are both magnetic and ferroelastic.

Individual ferroelastic twins have been observed on a scale of ~ 1 to $20 \mu\text{m}$ in natural crystals by reflected light microscopy (e.g., Bennett *et al.* [14]) and electron back scattered diffraction [15], and at a suboptical scale by transmission electron microscopy [16–18]. Although this twinning is nominally ferroelastic, it is unlikely that classical switching in response to external fields will be observed at ambient conditions because any change of orientation requires diffusion of vacancies and is expected to be negligibly slow at temperatures below $\sim 400 \text{ K}$ [15]. O'Reilly *et al.* [19] reported that lamellar magnetic domains observed in polished surfaces of polycrystalline samples using the Bitter pattern technique have a strong tendency to be aligned normal to the crystallographic c axis due to the strong magnetocrystalline anisotropy of the structure. They also found that similar 180° domain structures in other pyrrhotite samples were highly mobile under the influence of an external magnetic field.

While probing the crystallographic direction and temperature dependence of the magnetic hysteresis of 4C pyrrhotite, Haines *et al.* [13] observed that for magnetic fields applied along the $[\bar{1}20]_h^*$ direction of the parent hexagonal structure, the magnetization did not immediately reach saturation, but had several energy barriers to overcome. Such steplike behavior of the magnetization in response to an applied field is not an entirely unusual occurrence in the magnetism community, although the underlying reasons are vastly varied. For example, Ca_3CoO_6 , an Ising spin-chain system, exhibits several smooth steps on its way to complete saturation in a

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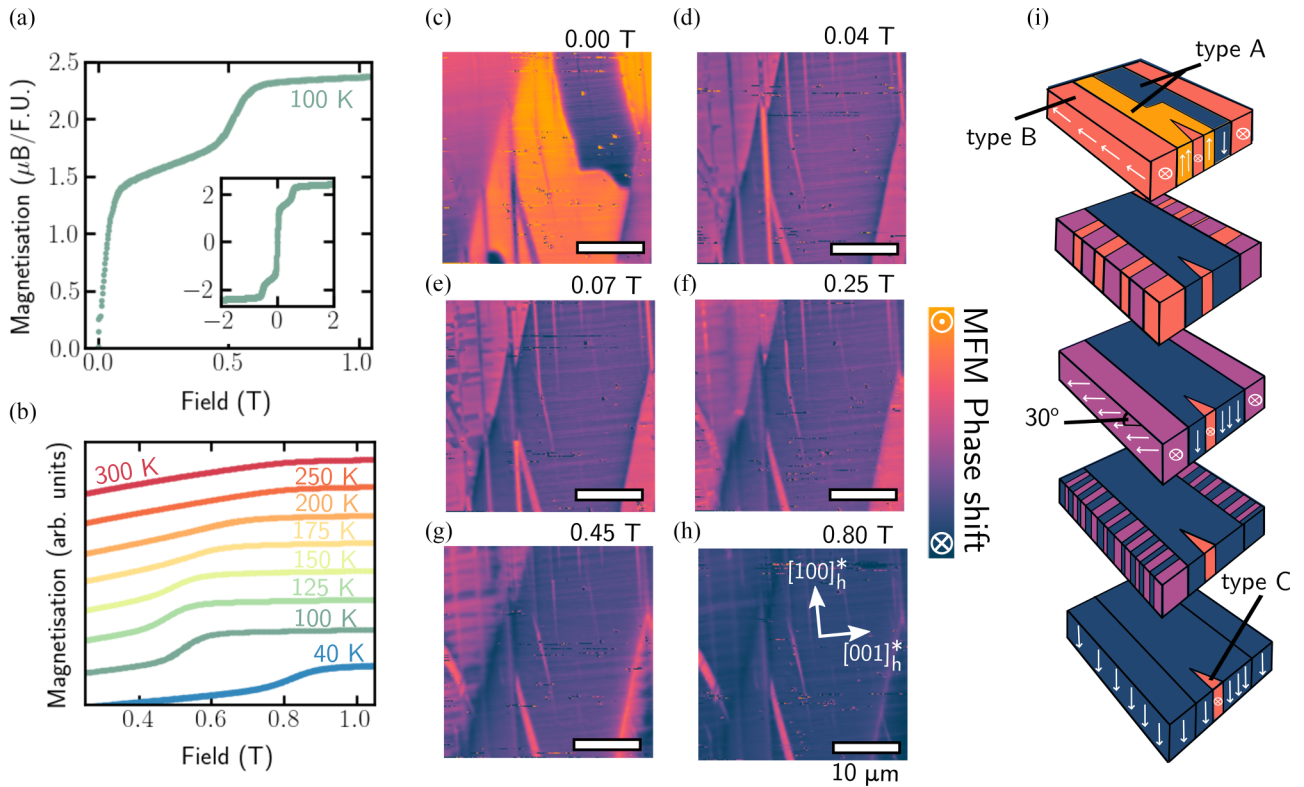


FIG. 1. (a) Magnetization data of Fe_7S_8 acquired at 100 K with field applied parallel to $[\bar{1}20]_h^*$. There is a subsaturation inflection beginning at 0.5 T before saturation is approached at 0.7 T. The full magnetization loop is shown in the inset. (b) Temperature dependence showing how the inflection point in (a) becomes most apparent below 175 K. (c)–(h) Field-dependent MFM images of a surface of the pyrrhotite single crystal cut perpendicular to $[\bar{1}20]_h^*$. Successive images were obtained with increasing positive field, applied normal to the surface after a negative 4 T saturating field had been applied. Scale bars are 10 μm . Absolute phase shifts are discussed in Fig. 3. (i) Schematic interpretation of the sequence of images with increasing field shown in (c)–(h), including the proposed orientation of moments within the surface layer.

similar appearance to pyrrhotite. The mechanism remains in discussion, with some groups proposing a quantum tunneling of the magnetization [20] and others proposing a kind of ferrimagnetic switching where different magnetic centers on the spin-chain unit cell switch at different fields [21]—both potential explanations attributed to individual spin behaviors, and not moments acting as a larger assembly. The unusual magnetic switching behavior in pyrrhotite has also generated some discussion regarding specifically apparent differences in the behavior of the net moment [22,23]. This difference in saturated moment, reported to be an effective difference in final rotation of between 14° and 29° , has been ascribed to a “hidden anisotropy generated by defects” [22–25]. It has been recognized that twinning must influence the magnetic properties [26], but the ferroelastic nature of 4C pyrrhotite has not been directly considered and the proposed defects remain unidentified, motivating further investigation.

The 4C pyrrhotite sample used for magnetic measurements was part of the same single crystal as pieces used in previous work (see the Supplemental Material and Refs. [13,15,27]). The direction of the surface normal is the $[\bar{1}20]_h^*$, referring to the hexagonal setting of the parent structure. The plane of this surface contains the orthogonal directions $[001]_h^*$ and $[100]_h^*$. Unusual plateau-like features had previously been identified in magnetometry data for Fe_7S_8 4C samples when the magnetic

field was applied along $[\bar{1}20]_h^*$, i.e., the surface normal. A typical example of the plateau at 100 K can be seen in Fig. 1(a). The complete loop is shown in the inset, displaying a negligible hysteresis. Instead of following a typical “S”-shaped loop, there is a subsaturation kink: initial linear progression upwards of 0.1 T is followed by a second inflection at 0.5 T and trends towards an apparent saturation before finally flattening. This second inflection point is not present in magnetization curves collected at room temperature, but occurs at increasing applied magnetic field in curves collected at progressively lower temperatures below 200 K [Fig. 1(b)]. Below the 40 K Besnus transition point, the pattern of magnetization changed significantly to include opening of the hysteresis loop (see Fig. 4(c) of [15]). As described below, stabilization of the inflection point is likely due to the increasing dominance of ferroelastic pinning in the magnetic behavior. On this basis, 100 K was chosen as an appropriate temperature at which to focus the magnetic imaging investigations (for more details on the magnetic force microscopy (MFM), see the Supplemental Material [27]).

Images acquired with increasing positive field from 0 to 8 T revealed three distinct switching regimes. Exemplar images can be seen in Figs. 1(c)–1(h) with a color scale to represent the MFM phase shift in each of the many magnetic domains. The images can be broadly described in terms of three

ferroelastic domains with domain walls aligned approximately from bottom left towards top right. The central domain will be referred to below as type A and the two on each side as type B. Phase shifts in the two type-B domains, at the left- and right-hand sides of the image, are denoted by a light-pink color in zero field [Fig. 1(c)], representing regions with in-plane magnetic moments. Two regions of the central (type-A) domain show a stark contrast of yellow and deep purple, representing the maximal phase shifts measured by MFM. They are interpreted as corresponding to magnetic domains with moments lying along the positive (yellow) and negative (purple) surface normal. Application of a field as small as 0.04 T [Fig. 1(d)] was sufficient to reorient moments in the yellow region so that the phase shift color became the same as in the deep-purple region, indicating that the moments had all become aligned in the same direction. Application of the 0.04 T field did not affect the type-B domains, which remained pink [Fig. 1(d)].

Switching of some magnetic domains to align with the applied field, as seen in the progression from Figs. 1(c) and 1(d), is in good agreement with the initial rise in magnetization below 0.1 T seen in the magnetometry data [Fig. 1(a)]. In contrast with this low-field domain switching behavior, the type-B ferroelastic domains displayed an intermediate phase shift at both zero field and 0.04 T. Small downward subdomains began to nucleate in these after the initial switching of the type-A domain was complete [Fig. 1(e), 0.07 T]. The individual subdomains grew under the influence of increasing field until a new steady state was reached at 0.25 T [Fig. 1(f)]. At this stage, corresponding to a point within the intermediate plateau between 0.07 and 0.45 T of the magnetic hysteresis curve, the subdomains had alternating pink/purple colors and boundaries lying approximately east-west in the image (corresponding to the $[010]_h^*$ direction). Figure 2 shows the configuration of ferroelastic and magnetic domains, recorded by MFM in 0.07 T in more detail on a different part of the surface. Also shown are crystallographic directions within the surface. The predominant orientation of boundaries between the type-A and type-B ferroelastic domains is normal to $[100]_h^*$, suggesting that the domain walls may be parallel to (001) . (001) ferroelastic domain walls are also magnetic domain walls. Segments of these walls in some parts of the other A/B boundaries and at needle twins have different orientations, as would be expected for other ferroelastic twin walls cutting the surface obliquely. The walls between alternating pink and yellow/orange in the type-B domains tend to be oriented parallel to $[100]_h^*$, an orientation which could imply that they are ferroelastically governed as well, though purely magnetic. They are also linear, consistent with being constrained by a ferroelastic shear strain.

Thin, needle-shaped domains aligned approximately north-south in all the images are “type-C” ferroelastic domains. The magnetic moments here remained entirely constrained to the in-plane and the overall shape of the needles, like all the other ferroelastic domains present, did not move in response to the applied field, and their intermediate color (pink) in all images throughout the full sequence indicates that their moments remained aligned within the plane of the surface of the sample. Given the high longitudinal magnetic anisotropy expected of a needle-shaped ferroelastic domain, it is not unexpected that

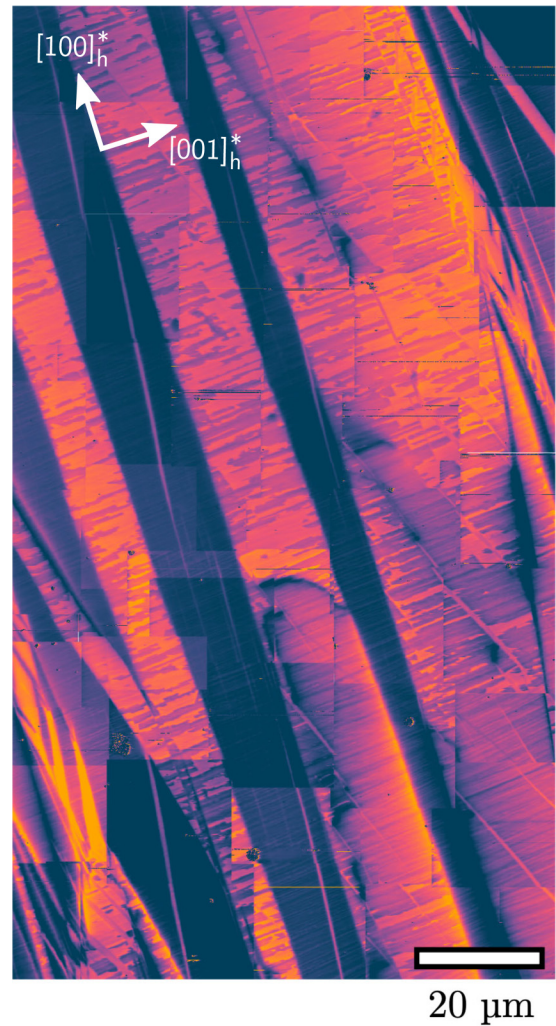


FIG. 2. Switched MFM scans of Fe_7S_8 displaying the distribution of the three types of domains revealed by applying a small magnetic out-of-plane field of 0.07 T. There is a clear preferential stripe direction of the $[100]_h^*$ (right to left diagonal), as expected.

the magnetic moment points into the tip of each needle, i.e., along its length in this section. Application of fields up to 8 T did not modify these domains in any way.

Further increases in the magnitude of the field led to the type-B domains breaking up into long, striated subdomains at 0.45 T [Fig. 1(g)]. This corresponds to the beginning of the final step up to saturation in the magnetometry data. The phase shift color of both type-A and type-B ferroelastic domains became a uniform deep-purple color by 0.8 T [Fig. 1(h)], implying that moments, apart from those in the type-C domains, had finally become aligned in a common direction either exactly parallel to or nearly parallel to the field.

A schematic interpretation of the observed pattern of domain evolution with increasing field is given in Fig. 1(i). Type-A domains are shown as initially having moments normal to the surface, while type-B and type-C domains have moments aligned in-plane. In reality, the steepest angle for moments to be inclined away from the $[100]_h^*$ - $[001]_h^*$ surface is 60° , but the essential point is that the boundary between the yellow and purple regions in type-A domains is a 180°

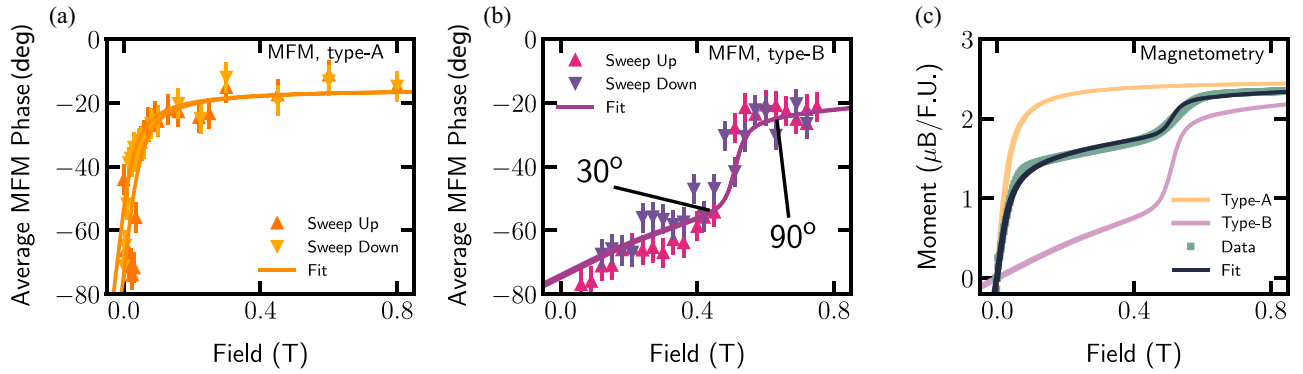


FIG. 3. Average phase in (a) and (b) are of acquired MFM images for regions selected on the Fe_7S_8 sample surface as being entirely type A and B, respectively. Each data point represents the averaged phase shift from one MFM image, with upward- and downward-facing triangles showing data points for upward and downward field sweeps, respectively. Data were fitted simultaneously with a linear combination of both fits (described in the text) alongside magnetometry data fitted in (c). The final, fitted linear combination of type-A and -B domains (in a 3 : 2 ratio) is shown in dark green overlaying the measured magnetometry data (light green). Hysteresis loops for uniform domains of either type A (yellow) or type B (purple) are shown for reference.

magnetic domain. The earliest stage of increasing moment with increasing field involves elimination of these. The second stage involves a development of ferroelastic/magnetic domains within the type-B domains such that half the domains have moments lying in-plane and half have moments aligned at an angle of 30° to the surface. In other words, rotation of the moments towards the alignment of the field in the type-B domains does not occur by a continuous rotation. The intermediate plateau of moment vs field is thus a metastable state with a particular configuration of new ferroelastic/magnetic domain walls. Further increasing the field eventually causes all the moments to align with the field and accounts for the second steep increase in moment towards saturation. The in-plane magnetic structure of the type-C, needle domains remained unchanged even up to 8 T.

The plateau in the evolution of the magnetic moment with increasing positive field shown in Fig. 1(a) matches the pattern of evolution of domains in the images. The plateau also appears in each of the four quadrants of the full hysteresis cycle of increasing, decreasing, and changing direction of the field [inset in Fig. 1(a)], indicating that overall, the domains evolved in a manner that was fully reversible. None of the primary ferroelastic domain walls moved, leaving the underlying ferroelastic domain structure as a template which constrained how the magnetic domains returned when the field was removed. It follows that the precise pattern of magnetization for any given crystal must substantially depend on the configuration of ferroelastic domain walls that developed during crystallization or during cooling from high temperatures. In this context, failure of type-C domains to saturate, due to the high shape anisotropy of the needles, is likely to have been the cause of the different reports of magnetometry-derived saturation magnetization values in the literature [22–25].

In order to quantify the behavior of the three types of domains, an additional series of images was acquired. By mapping a relatively large area of around $10\,000\ \mu\text{m}^2$, a clear prevalence for stripes of alternating type-A and type-B domains with boundaries generally aligned along $[100]_h^*$ was confirmed. Magnetic domains in this orientation have been previously identified by use of the Bitter pattern

technique [15,19], but the microstructure reported on here is generally well below the resolution of ferrofluids. At 0.07 T, all of the type-A domains had saturated and so appeared as a (dark-purple) low phase shift. The unsaturated type-B domains, with a lower phase shift in the images, had begun their own, stepped rotation. Two large areas were selected as having predominantly either type-A or type-B domains.

MFM data were also acquired in specific areas of type-A and type-B domains as in successive increments of increasing and decreasing field shown; see Fig. 3. In order to ensure each image was directly comparable, the stability of type-C domains was exploited. Every individual image was phase adjusted to ensure that the type-C phase shift was the same. Each data point in Figs. 3(a) and 3(b) then represents the average phase recorded for type-A and type-B domains, respectively, where each point is an MFM image of the same area.

The data in Figs. 3(a) and 3(b) confirm the qualitative descriptions provided above. Type-A domains saturated with very low fields and followed a traditional “S”-like loop. Conversely, type-B domains started from a low phase shift, but required much higher applied fields to approach saturation. Their reasonably linear contribution to the total magnetization is punctuated by a sharp transition to a stable point where magnetic saturation was finally reached.

In order to directly compare the effect of having two alternative magnetic switching behaviors present in the material, data were fitted with a modified Langevin function (as in [28–30]) of the form

$$M = M_s \left(\frac{1}{\tanh\left(\frac{H+H_c}{S}\right)} - \frac{1}{S} \right), \quad (1)$$

where M is the moment of the phase being fitted, M_s is the saturated moment, H is the applied field, H_c is the coercive field, and S is the “shape.” The shape parameter effectively controls the squareness of the loops but, as this fit is phenomenological, no physical meaning may be derived from it. Directly relating the phase shift of an MFM cantilever to the position or rotation of the magnetic moments is analytically challenging. However, by reducing each image to its average

phase-shifted value, self-consistent with each other in an incremented field, fits of these data can be linearly combined (see the Supplemental Material [27]), alongside a conversion factor (to scale to the vibrating-sample magnetometer (VSM) data) and thus the MFM data can be fitted according to Eq. (1) to capture the behavior of the VSM data, as shown in Fig. 3(c).

While not leading to a completely perfect fit, it is worth pointing out that the type-A and type-B MFM data sets were taken from small specific areas of the surface, unlike the VSM data set which is a global sample measurement. Even with the obvious challenge of a small area measured for MFM, visually, the fit is good—reflecting the key features and plateaus in the VSM data. Also plotted in Fig. 3(c) are the type-A and type-B fits were the sample to only contain type-A or type-B domains, respectively. This effectively compares the extrema where the sample only consists of one type of ferroelastic/ferromagnetic domain. It was found from the fitting procedure that more type A than type B were present in a ratio of 3 : 2, i.e., 60% of the sample was made up of type-A domains. This number does not include the type-C domains whose moment lies entirely in the plane and did not undergo reversal. This is a particularly interesting result when considering the seeming abundance of “type-B domains” in Fig. 2. Naturally, scanning only a specific area of the sample is not going to yield bulk results; however, by combining local results simultaneously with alternative bulk measurements reveals further information about the local microstructure.

The combination of MFM observations and VSM magnetic data indicate that although new ferroelastic/magnetic domain walls may be created in some domains when a magnetic field is applied, the original ferroelastic domain walls do not move. In a geological context, palaeomagnetic moments recorded in multidomain 4C crystals should therefore provide a robust source of palaeomagnetic remanence, and needle-shaped domains should be the most robust. However, any process which disrupts the original distribution of ferroelastic domains should then have a dramatic impact on the orientation and magnitude of the bulk magnetic moment. This would be a factor in relation to experimental findings that both shock and static pressure can cause demagnetization of pyrrhotite, and the proposal that loss of magnetization of the Martian crust was due to impact of meteorite bodies [8,31,32]. It would be worth examining the distribution of ferroelastic domains in

4C grains before and after such experiments to test whether redistribution of the domain walls by shearing provides a significant contribution to the loss. For example, Mang *et al.* [33] observed abundant deformation twins on a scale of 50 nm in crystals that had been subjected to shock up to a pressure of 30 GPa.

Magnetic imaging has allowed direct observations of the interaction of a particular class of ferroelastic domains, i.e., arising from cation/vacancy ordering, with magnetic domains and the response of both to an external magnetic field. The two predominant twins have been labeled as type A, with an easy axis along or slightly inclined to the surface normal of the crystal, and type B, with moments free to rotate within in-plane sample directions but with easy axes at 30° increments out of the plane. It is this rotational behavior that is responsible for the unusual inflections in the hysteresis along this specific field direction. Finally, type-C domains do not reach saturation in an out-of-plane field, due to the high structural anisotropy arising from their needlelike shape which pins the magnetic moments into the plane of the sample, along the length of the needle. These are identified as the likely cause of the different saturation magnetization that is recorded between different natural samples, due to a difference in abundance of this specific type of domain. It is clear that ferroelastic domains in pyrrhotite have a fundamental influence on the magnetic response to an external field; however, there is no sign that the twin walls between any of these domains can be moved by fields up to 8 T at 100 K, as expected [13,15,34]. In a wider context, simultaneous imaging of both ferroelastic and magnetic domains by MFM, together with the particular properties of pyrrhotite, provide a window into how magnetic properties of functional materials with multiple instabilities might be engineered through the choice of microstructure. Furthermore, the extrapolation of MFM, a primarily locally resolved technique with the measured bulk properties, highlights that with appropriate treatment or large data volumes, scanning probe microscopy techniques can be used to identify and correlate bulk behaviors.

The work was partly supported by the Engineering and Physical Sciences Research Council (EPSRC, UK) through Grants No. EP/M022706/1 (M.A.) and No. EP/T027207/1 (M.A.).

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