Transmission electron microscopy of exsolution lamellae in ilmenite-hematite: Implications for “lamellar magnetism”

T. Kasama[1], T. Asaka[2], R.K.K. Chong[1], R.E. Dunin-Borkowski[1], E.T. Simpson[1], R.J. Harrison[3],
U. Golla-Schindler[4], S.A. McEnroe[5], and A. Putnis[4]

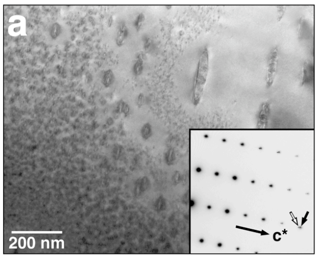
[1] Dept. of Materials Science, Univ. of Cambridge, Pembroke St., Cambridge, UK, [2] National Institute for Materials Science, Namiki, Tsukuba, Japan, [3] Dept. of Earth Sciences, Univ. of Cambridge, Downing St., Cambridge, UK, [4] Institut für Mineralogie, Universität Münster, Corrensstr. 24, Muenster, Germany, [5] Geological Survey of Norway, Trondheim, Norway

Abstract

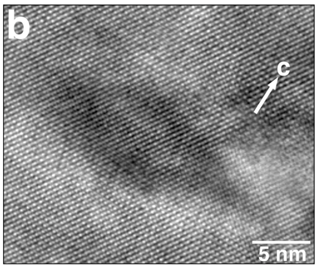
McEnroe et al. (2002) found fine exsolution lamellae of hematite (Fe_2O_3) and ilmenite ($FeTiO_3$) in rock samples with high coercivities and stable natural remanent magnetizations (NRMs). They suggested that the exsolution lamellae may be related to the acquisition of NRM. Robinson et al. (2002) used Monte Carlo simulations to suggest that the ferrimagnetic moment of an intergrowth of hematite and ilmenite could be associated with the arrangement of cations and spins at the interface between hematite and ilmenite. They described this “lamellar magnetism” as being due to “contact layers”, which are cation layers at the interface between hematite and ilmenite that do not correspond to the chemistry of either hematite or ilmenite.

Transmission electron microscopy (TEM) is powerful tool for the examination of the crystallographic and chemical structure and microstructure of rock samples at the nanometer scale. Lorentz electron microscopy can be used to observe magnetic microstructure in minerals directly at high spatial resolution. Electron tomography allows nanometer-sized minerals to be imaged in three dimensions, to provide their morphologies and distributions. Here we apply all of these TEM techniques to the characterization of fine lamellae in hematite-ilmenite, with the aim of understanding their effect on the NRM of these samples. Our results support the lamellar magnetism hypothesis. Since lower crustal rocks can contain hematite and ilmenite lamellae that exsolved during slow cooling, the formation of lamellae may be a predominant factor responsible for magnetism in the crust.

Conventional and high-resolution TEM



A bright-field (BF) TEM image of typical fine hematite lamellae in an ilmenite host and its electron diffraction pattern are shown in (a). The finer lamellae are present between coarser lamellae. These fine lamellae, which are < 50 nm in length, are observed abundantly and are surrounded by strain contrast. The <210> electron diffraction pattern reveals that the hematite lamellae and ilmenite host share (001) planes.



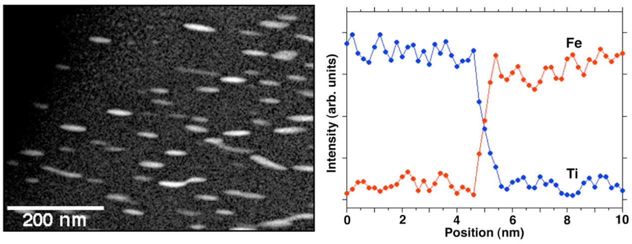
A <100> high-resolution TEM image of a fine hematite lamella in an ilmenite host is shown in (b). Such lamellae, which are < 50 nm in length and make up the majority of lamellae present in the rock sample, have no interfacial dislocations and are perfectly coherent. The strain contrast around the fine lamellae results from the absence of interfacial dislocations.

Elemental mapping using energy-filtered TEM

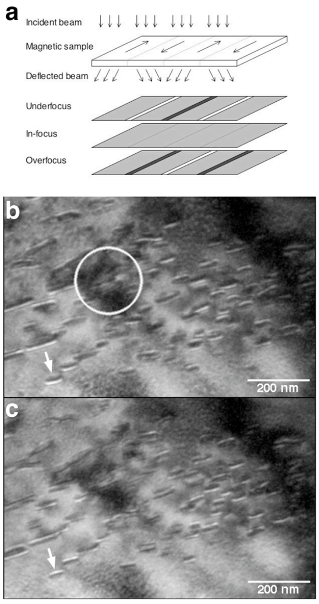
A representative Ti map of fine ilmenite lamellae (5-10 nm in thickness) within a hematite host is shown below. The ilmenite lamellae have lens-like or sometimes kinked shapes, and are usually thinner than corresponding hematite lamellae observed in ilmenite. The Ti map was used to determine the size, shape and distribution of the particles, whose strain contrast made it difficult to see them in BF images.

Iron and Ti intensity profiles were determined across one of the lamellae shown in the Ti map, in which the beam direction is close to <100>. The profiles suggest that the lamellae have a sharp (< 1 nm) compositional interface with their host.

The chemical compositions of the lamellae are $Ilm_{13-19}Hem_{87-81}$ for hematite and $Ilm_{97-99}Hem_{3-1}$ for ilmenite, as measured by energy-dispersive X-ray spectrometry (McEnroe et al. 2002).



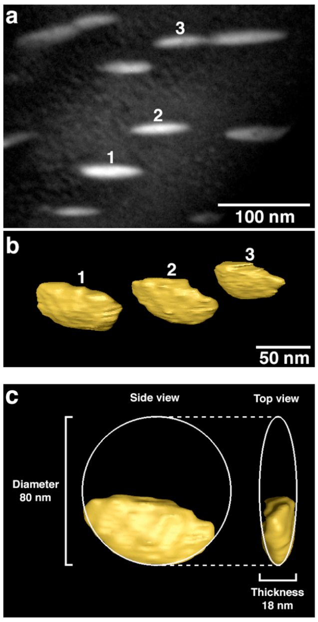
Lorentz electron microscopy



The schematic diagram in (a) shows the approach used to image magnetic domain structure by Fresnel (defocus) imaging. The Lorentz force associated with the in-plane component of the magnetization deflects the incident electron beam. Magnetic domain walls give rise to dark or bright contrast. By analyzing the contrast, the local direction of the magnetic moment can be determined.

Lorentz images of ilmenite lamellae in hematite, acquired after saturating the sample with a 5 T field in a direction parallel to the a-axis of hematite or ilmenite, are shown in (b) and (c) for overfocus and underfocus image conditions, respectively. Asymmetric contrast appearing on each lamella inverts between the images obtained overfocus and underfocus. Since the contrast appears parallel to the interfaces of the lamellae, we suggest that it is not simply due to the canted magnetization of hematite, but that it may result from the presence of additional magnetic moments at each interface. The magnetic moment in each lamella orients to its long-axis, i.e., perpendicular to the c-axis. Strong diffraction contrast seen in the region marked by a circle makes the magnetic contrast invisible in this region.

Electron tomography



A tomographic reconstruction of the three lamellae indicated in the Ti map in (a) is shown in (b). The reconstruction confirms that the shape of the lamellae is lens-like, as suggested by previous studies (e.g., McEnroe et al. 2002), although the lamellae are cut by sample preparation using Ar-ion milling. The top surface of the lamellae is rough because of damage by ion milling and electron beam exposure.

The reconstruction of lamella 2, with outlines to guide the eye, is shown in (c). There is no significant difference between the sizes that were measured in projection (73 nm diameter and 19 nm thickness) and estimated from the outlines (80 nm and 18 nm). However, if they had been larger or cut by sample preparation, then it would have been possible to misinterpret their true shape and size. Tomography then allows the “real” lamella size to be calculated directly from the observed shape and size in the reconstruction.

References

McEnroe et al. (2002) Geophysical Journal International 151, 890-912.
Robinson et al. (2002) Nature 418, 517-520.

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