

**Experiment title:**

Ferrimagnetic Domains and the Verwey Transition in Magnetite Fe_3O_4

Experiment number:

HC-155

Beamline:

BM5

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Report:

X-ray synchrotrons topography allows the observation of magnetic domains through the associated change along the interfaces of the lattice distortion (magnetostriction). The investigation of domains is interesting because it can give clues about the structure of the low temperature phase, which is still a matter of controversy. Magnetite is ferromagnetic at RT, with easy direction along $\langle 111 \rangle$ axis of the cubic cell. Below 120 K it changes its properties and symmetry: the easy direction is $\langle 100 \rangle$, referred to the high temperature cubic axes. The structure determination experiments indicates the monoclinic group Cc, but the existence of magnetoelectric properties suggests that the group is triclinic P 1. In addition to ferrimagnetism, the material is also ferroelectric, but the polarization direction is not well known.

We performed on BM5 white beam tomographs on two (01 1) magnetite samples produced by the Bridgman method. They are different in thickness (0.3 and 0.68 mm respectively). We used a closed-cycle refrigerator to cool down and a small electromagnet to apply magnetic fields up to 0.3 T. The films were SR films Kodak Industrex.

The thinner sample showed at RT the well known 71° and 109° walls. We have an example of misorientation contrast due to magnetostriction in fig 1. After cooling below the Verwey transition (120 K), we observed a striking domain configuration. It is different from the configuration expected for a material with easy directions along $\langle 100 \rangle$. As the visibility of the domains was not very good, we tried to solve this problem by chemically attacking the sample. The visibility did not improve too much but the domain structure changed considerably. Therefore we think that it was determined by surface effects. Under these conditions the understanding of the structure becomes complicated. A carefully repolishing of the sample is being achieved before pursuing this work.

The thicker sample shows well separated domains at the low temperature phase, after cooling without field (fig. 2). When we applied a magnetic field of 0.3 T parallel to the [100] direction on the surface, one of the domains was favored and increased with respect to the other two. The separation between images goes from 0.5 to 3 mm depending on the spot, for a film-sample distance of 165 mm. We could suggest that the domains correspond to different orientations of the c-axis (easy direction) of the low temperature phase along one of the different $\langle 100 \rangle$ directions. The magnetostatic energy is not minimum in this configuration, indicating that other terms are important (elastic, electrostatic). The magnetostatic energy term is relatively more important in the thin sample. This explain why the previous sample did not show these domains. When the sample was cooled in a 0.3 T field parallel to [100] direction no highly disoriented domains were observed, but it was possible to identify domains with a smaller separation (see sections of fig 3.). The cooling in field is known as a way to reduce the twinning of the crystal, the c-axis being along [100] direction. In addition some of the sections gave domain images not completely parallel to the surface (fig 3.). The smaller separation on the film (or not clear separation but contrast) correspond to domains where the effective misorientation comes from the smaller departures from the cubic structure. The exact nature of this domains requires further experiments to be determined.

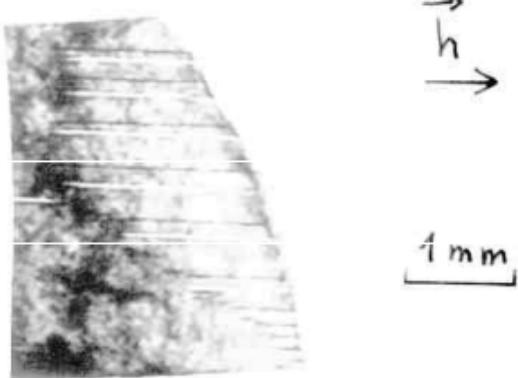


Fig 1. RT domains



Fig 2. Domains in low temperature phase (no field cooling)



Fig 3. Domains in low temperature phase (0.3 T field II [100] cooling)