

Correlating magnetic flux distributions with (RbFe₂Se₂) superconductor microstructure using magnetic force microscopy J Hazi, C R M Grovenor, S C Speller

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Magnetic force microscopy of superconductors

- Magnetic force microscopy (MFM) is a probe technique used to image local magnetic field distributions by scanning a sharp tip with a ferromagnetic coating above the sample.
- The tip is oscillated at a chosen stand-off distance and the interaction force can either be measured as the change in the resonant frequency or change in the phase.



MFM at 4.5K in increasing magnetic field



The sample was cooled down in zero applied field to 4.5K. The image contrast arises from surface topography alone when no magnetic field is applied.

150 mT

Superconducting phase appears bright as magnetic flux is excluded.

2.0 deg

- Magnetic force microscopy (MFM) has previously been used to study vortex pinning by defects and grain boundaries in type II superconductors, vortex creep and vortex-shape in layered superconductors as well as flux penetration into superconductors [1, 2].
- We have used the attoAFMI together with the attoDRY1000 MFM to study single crystal RbFe₂Se₂ samples with complex mixtures of superconducting and antiferromagnetic phases.
- Constant height MFM scans at 4.5K provide a direct way to determine the micron scale distribution of the superconducting phase.

RbFe₂Se₂ single crystals

- Iron selenide compounds are of interest for studying the fundamental properties of iron-based superconductors.
- Alkali metal doped FeSe crystals exhibit unusual magnetic properties, with antiferromagnetism ($T_N \sim 500$ K) co-existing with bulk superconductivity at a relatively high temperature (T_C~30K).
- Extensive microstructural studies [3] have shown intrinsic phase separation at the micron scale in these crystals, and it is believed that different phases are responsible for superconductivity and antiferromagnetic ordering.



SQUID magnetisation measurement from the same RbFe₂Se₂ crystal used for the MFM study showing an onset T_c of ~28 K. Inset shows the



- Images were obtained at a constant height (~150nm) in the phase-locked-loop mode.
- The measured signal is the phase of the resonating MFM tip which decreases as the applied field increases.
- The bright areas thus identify regions of lower local magnetic field compared to darker areas.
- As the applied field increases, the minority features "light up" indicating the superconducting nature of this phase.

MFM: below and above T_c

Changing the measurement temperature confirms that the minority phase does not exhibit the field-shielding behaviour at 40K. This is consistent with SQUID data showing $T_c=28K$.





Frequency (kHz)

Microstructural Analysis



Secondary electron image of cleaved (001) surface showing the distinctive phase separated microstructure of a $RbFe_2Se_2$ crystal.





Energy dispersive x-ray analysis taken at 5kV in a Zeiss Merlin FEGSEM [1]. The phase is Fe-deficient with a composition of about $Rb_{0.8}Fe_{1.6}Se_2$. The minority phase is richer in Fe and deficient in Rb.

Photoemission Electron Microscopy

- In the PEEM technique, synchrotron X-rays illuminate a wide-field area of the sample surface. Images are produced from the emitted photoelectrons.
- Using x-rays linearly polarised in two perpendicular directions, the x-ray linear dichroism (XLD) produced by antiferromagnetic ordering can be imaged. • XLD spectra show that the matrix phase is antiferromagnetic and the minority phase is nonmagnetic at room temperature.





MFM: field cooling protocols

Field-cooled: 4.2 K, 200 mT



Zero-field-cooled: 4.2 K, 200 mT



(Above): XLD image of RbFe₂Se₂ taken at 706.2 eV and room temperature on the I06 beamline at Diamond Light Source.

(Left): Fe $L_{2,3}$ XLD spectra from the minority and matrix phases and a more detailed view of the Fe L₃ dichroic feature found in the *matrix indicating antiferromagnetic* ordering in this phase.

4.0

- Field-cooling results in clearer definition of the minority superconducting phase features, revealing the complex discontinuous nature of the microstructure observed in SEM.
- Differences in the field distributions in the two cases arise from flux pinning.

Conclusions

- Cryogenic MFM is a valuable contribution to the study of superconductors with complex microstructures.
- Magnetic force microscopy shows directly that the minority phase in RbFe₂Se₂ is superconducting at 4.5K but not at 40K. This is consistent with $T_c=28K$ measured by a SQUID as well as with XLD PEEM measurements identifying the matrix phase to be antiferromagnetic.

References: [1] Kirtley Reports on Progress in Physics 73, 12 (2010), [2] Volodin et al. Physica C, 369, 1, P165-170 (2002), [3] Speller et al. PRB 90 024520 (2014).

Acknowledgements: C Barker and R Gresham (Dept. of Materials, Oxford Uni.) for technical support. T Mousavi (Dept. of Materials, Oxford Uni.), P Dudin, F Maccherozzi (Diamond Light Source) for PEEM measurements, T Davies for SQUID measurement and Funding from a UK EPSRC DTA studentship.

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