Quantitative Imaging of Flux Vortices in Superconductors

J.C. Loudon, C.J. Bowell and P.A. Midgley

Department of Materials Science and Metallurgy, University of Cambridge, Pembroke Street, Cambridge, CB2 3QZ.

Email: j.c.loudon@gmail.com

Abstract. Transmission electron microscopy offers the possibility of imaging flux vortices in superconductors quantitatively and at video rate. Here we use the technique to image flux vortices in MgB$_2$ and investigate their magnetic structure.

1. Introduction

Superconductors have zero electrical resistance and expel magnetic flux from their interiors (the Meissner effect). If a magnetic field is applied to an ideal (type I) superconductor no flux enters unless the field exceeds the critical field, $H_c$, at which the material ceases to be superconducting. However, in type II superconductors, the whole superconducting state is not destroyed at once but above the lower critical field, $H_{c1}$, magnetic flux penetrates the superconductor by flowing along channels called flux vortices. The vortices consist of a core where superconductivity is suppressed with a size given by the coherence length, $\xi$, surrounded by circulating supercurrents which persist over a distance called the penetration depth, $\Lambda$. The magnetic flux density associated with each vortex persists over the same distance, $\Lambda$. Each vortex carries a single quantum of magnetic flux given by $\Phi_0 = h/2e$ where $h$ is Planck’s constant and $e$ is the electron charge. Vortices repel one another and so their arrangement in the absence of pinning is a 2-dimensional hexagonal lattice called an Abrikosov lattice [1]. The performance of almost all superconducting devices is determined by the behaviour of flux vortices as energy is dissipated when they move. It is important, therefore, to characterise their movement and magnetic structure.

Flux vortices can be imaged using transmission electron microscopy [2] due to the magnetic flux density from the vortices deflecting the electron beam and appear as black-white features in an out-of-focus image (Fig. 1). This is a unique imaging technique as it gives information on the internal structure of individual vortices, not just the stray fields, and allows imaging at video rate.

MgB$_2$ was investigated in this experiment. It was discovered to be a superconductor in 2001 and has a transition temperature, $T_c = 39$ K [3]. It has a hexagonal crystal structure (space group 191: $P6/mmm$, lattice parameters: $a = b = 3.086$ Å, $c = 3.542$ Å) consisting of alternating layers of magnesium and boron.
2. Sample Preparation and Experimental Methods

MgB$_2$ single crystals were synthesised by Dr J. Karpinski as described in ref. [4] and were thinned to 250 nm in the $c$ direction using focused ion-beam (FIB) milling so they were electron-transparent. The sample needs to be tilted to a high angle ($\alpha$ in Fig. 1(a)) to maximise the electron beam deflection and so was attached to a tilted copper post as illustrated in Fig. 1(b) giving a tilt angle of $45 \pm 1^\circ$. A liquid-helium cooled specimen stage with a base temperature of 10 K was used to cool the sample and energy-filtered images were recorded with a CCD camera using a Philips CM300 transmission electron microscope equipped with a field-emission gun operated at 300 kV. A magnetic field was applied to the sample by altering the setting of the twin lens in the microscope. Further details are given in ref. [5].

3. The Magnetic Structure of Flux Vortices

Information on the magnetic structure of flux vortices can be derived from the out-of-focus images. Simulations based on the modified London equation [6] shown in Fig. 2 demonstrate that the contrast of the images (defined here as the standard deviation divided by the mean) is a unique function of the penetration depth, $\Lambda$, provided that the sample thickness is known. We note here that as the sample is tilted, two thicknesses are relevant: we denote $l$ as the thickness measured normal to the plane of the specimen and $t$ the thickness measured parallel to the electron beam. The two are related via $l = t \cos \alpha$. In principle, then, we can measure the penetration depth of individual vortices and if the magnetic structure of certain vortices is altered, say by pinning, it should be possible to identify which vortices

**Figure 1.** (a) Experimental arrangement for imaging flux vortices. The electrons are deflected by the component of the B-field normal to the electron beam so each vortex appears as a black-white feature in an out-of-focus image. (b) The specimen geometry (not to scale). The MgB$_2$ specimen was mounted to a copper post glued to a standard 3 mm diameter copper ring at an angle of $45^\circ$. An electron transparent window was then cut by focussed ion-beam milling.

**Figure 2.** Simulations showing the variation of the contrast seen in out-of-focus images as a function of penetration depth, $\Lambda$, for a superconductor with a thickness $l = 200$ nm, tilted at an angle $\alpha = 45^\circ$ for different defocus levels, $\Delta f$. 

International Conference on Strongly Correlated Electron Systems (SCES 2011) IOP Publishing
Journal of Physics: Conference Series 391 (2012) 012117 doi:10.1088/1742-6596/391/1/012117
have changed and in what way. This has been done in ref. [6] where some of the images of vortices appear stretched because the vortex cores are pinned on columnar defects. However, the changes to the magnetic structure of the vortices are inferred from a visual comparison between experimental and simulated images. Here we make a quantitative comparison.

The specimen thickness was found using energy-filtered imaging as explained in ref. [5]. This produced a thickness map of the specimen where the thickness \( t \) is given as a multiple of the inelastic mean-free path of the electrons, \( \lambda \). The inelastic mean free path was then calibrated using electron holography [5], giving \( \lambda = 152 \pm 2 \) nm.

\[
\Delta f \text{ in cm}
\]

\[
\begin{align*}
&\Delta f = -2.56, -1.80, -1.21, -0.71, -0.32, 0, 0.29, 0.57, 0.80, 0.99, 1.23 \\
&\text{Thickness: } 307\text{nm}, 312\text{nm}, 318\text{nm}, 315\text{nm}, 374\text{nm}, 366\text{nm}, 378\text{nm}, 280\text{nm}, 430\text{nm}
\end{align*}
\]

**Figure 3.** Flux vortices in MgB\(_2\) taken at 11 K. Each column shows the same vortex taken at different defocus levels. The thickness \( t \) of the specimen along the electron beam direction is given at the bottom of each series in terms of \( \lambda \), the inelastic mean-free-path and calibrated in nm using \( \lambda = 152 \pm 2 \) nm. Simulated images for thicknesses \( t \) of 280 nm and 430 nm with \( \Lambda = 110 \) nm [7] are shown on the right.

Fig. 3 shows images of vortices acquired from MgB\(_2\) at 11 K at different defocus levels and simulations for 280 nm and 430 nm thicknesses assuming \( \Lambda = 110 \) nm [7] are shown for comparison.
in the two columns on the right. As discussed in ref. [8], a visual comparison between the images and simulations is not a very sensitive method of comparison. Even so, a comparison of the length of the vortex image along the black-white interface shows that the simulations for a 280 nm thickness compare better with the images than those for 430 nm. Note that altering the penetration depth in the simulations does not affect this length but only changes the contrast.

Simulations have shown [8] that a more sensitive comparison can be made by plotting the image contrast of the vortices versus the defocus. Fig 4(a) shows such a comparison for one vortex and the best fit for this vortex was with a thickness of 280 nm (the specimen thickness was 318 nm for this vortex) and a penetration depth of 110 nm. The data in such plots were more scattered than we are presently able to account for – the errorbars show the likely variation in the contrast measurement caused by the noise in the image – and they also tend to be assymmetric with positive defoci having larger contrast values than negative defoci, an effect which is difficult to understand.

Both the visual comparison and the contrast plots show that the data are best fit by a simulation of a vortex which is shorter than the specimen thickness. This indicates that there are dead layers of non-superconducting material of around 15–30 nm either side of the superconductor. These are likely caused by the ion-beam thinning of the specimen and electron microscopy experiments on semiconductors prepared using the same technique have reported a 30 nm thick dead layer [9].

**Figure 4.** (a) A plot of contrast versus defocus for a single vortex determined experimentally is shown in black together with the contrast derived from simulated images based on the modified London equation with \( t = 280 \text{ nm} \) and \( \Lambda = 110 \text{ nm} \), shown in red. (b) An alternative method is to reconstruct the phase of the electron wavefunction from the defocussed images using the transport of intensity equation [9]. The top two panels show the defocussed images and the lower left panel, the phase reconstructed from these. A simulation of the expected phase shift is shown in the lower right panel (see text for details).

Another method of comparison is to use the defocussed images to reconstruct the phase of the electron wavefunction as it exits the specimen using the transport of intensity equation [10]. From the phase, the component of flux density normal to the electron beam can be derived. Fig. 4(b) shows two vortex images equally disposed either side of focus on which the phase reconstruction was based. The images in the lower panel show the cosine of 16 times the phase which gives a contour map of the phase. The black lines can be thought of as field lines of the component of the B-field normal to the electron beam. It should be noted that the stray field in the reconstruction re-enters the superconductor which is unphysical. This is an artefact of the reconstruction which requires that the phase be a
constant on the boundaries of the image. However, the simulation shown for comparison has been performed with the same restriction so the two images are comparable. It can be seen that the field within the vortex is very similar indicating that $\Lambda = 110$ nm provides a good fit. Strangely, the fit is best for a simulation which matches the measured specimen thickness of 374 nm although the visual comparison in Fig. 2 showed that a thickness of 280 nm was best.

4. Summary
Transmission electron microscopy is a unique technique for imaging flux vortices in superconductors as images can be acquired at video rate and the images give information on the internal magnetic structure of the vortices. This technique should help distinguish between different models of vortex structure and enable a better understanding of the effects of how vortices respond to pinning sites which is crucial for improving the performance of superconducting devices. We have acquired defocussed images from MgB$_2$ and were able to use these to obtain reasonable values for the penetration depth and the length of the vortex and to reconstruct the B-field within the specimen. Some difficulties remain: the data in plots of contrast versus defocus have more scatter than we can presently account for and show an asymmetry so that the contrast values at positive defocus are higher by some 3-5% than those for negative defocus. Further work to understand these effects is required.

Acknowledgments
The authors thank N.D. Zhigadlo and J. Karpinski, Laboratory for Solid State Physics, ETH Zurich, Switzerland for supplying the MgB$_2$ single crystals used in this experiment. This work was funded by the Royal Society and the EPSRC, grant number EP/E027903/1.

References
[1] Abrikosov AA 1957 Sov. Phys. JETP 5 1174.
[2] Harada K, Matsuda M, Bonevich J, Igarashi M, Kondo S, Pozzi G, Kawabe U and Tonomura A 1992 Nature 360 51.
[3] Nagamatsu J, Nakagawa N, Muranaka T, Zenitani Y and Akimatsu J 2001 Nature 410 63.
[4] Karpinski J, Kazakov SM, Jun J, Angst M, Puzniak R, Wisniewski A and Bordet P 2003 Physica C 285 42.
[5] Loudon JC, Bowell CJ, Zhigadlo ND, Karpinski J and Midgley PA 2012 Physica C 474 18.
[6] Beleggia M, Pozzi G, Masuko J, Osakabe N, Harada K, Yoshida T, Kamimura O, Kasai H, Matsuda T and Tonomura A 2002 Phys. Rev. B 66 174518.
[7] Manzano F, Carrington A, Hussey NE, Lee S, Yamamoto A and Tajima S 2002 Phys. Rev. Lett. 88 047002.
[8] Loudon JC and Midgely PA 2009 Ultramicroscopy 109 700.
[9] Twitchett AC, Dunin-Borkowski RE and Midgley PA 2002 Phys. Rev. Lett. 88 238302.
[10] Teague MR 1983 J Opt. Soc. Am. 73 1434.