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LETTER TO THE EDITOR

Magnetoresistance fluctuations in mesoscopic aluminium structures

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Abstract. Unusual magnetoresistance behaviour has been observed in mesoscopic disordered aluminium samples. Large resistance fluctuations were found, with peaks $> 2R_N$ and troughs implying re-entrant superconductivity, occurring as a function of magnetic field, at high field and $T \ll T_c$. The results are consistent with a recently proposed phase diagram, and the apparent re-entrance is attributed to the overlap of a connected superconducting filament with all the measurement probes. The results suggest that microscopic superconducting regions persist in fields of above 1 T in disordered aluminium at mK temperatures.

There is considerable current activity in mesoscopic physics: the study of systems whose dimensions are comparable to characteristic scattering lengths within the materials from which they are composed. Recently there has been a growing interest in mesoscopic superconducting and hybrid structures, the defining criterion for such a system usually being that its size be comparable to the coherence length, ξ_0, or the London penetration depth, λ_0.

Size effects in superconductors have been investigated extensively [1–4]. Experiment and theoretical work more recently have shown and predicted several new mesoscopic phenomena, including magnetoresistance oscillations [5–10], negative magnetoresistance and a resistance anomaly around the transition temperature T_c [11–19], multistability of the superconducting state [20] and a prediction of re-entrant superconductivity in a magnetic field [21] for a disordered material at temperatures well below T_c. Many of the observations can be explained by considering the microscopic arrangement of superconducting and normal regions within the structures, and their connections with voltage and current probes.

Here, we report an investigation of the magnetoresistance behaviour of mesoscopic superconducting aluminium samples. Large resistance fluctuations are seen in the range 0–2 T, with high excess resistances of $R > 2R_N$, (R_N being the normal-state resistance of the device), together with resistance minima indicating the re-entrance of superconductivity. The results suggest that filamentary superconducting regions persist to fields above 1 T on a microscopic scale at mK temperatures in disordered aluminium.

The device structures were simple blocks of aluminium approximately 1.6 μm^2 square and 0.25 μm thick, and so all dimensions are comparable to the bulk value of the superconducting coherence length, $\xi_0 \approx 1.6$ μm, although critical field measurements in these mesoscopic disordered samples imply $\xi_0 \approx 150$ nm. Electrical contact to each block

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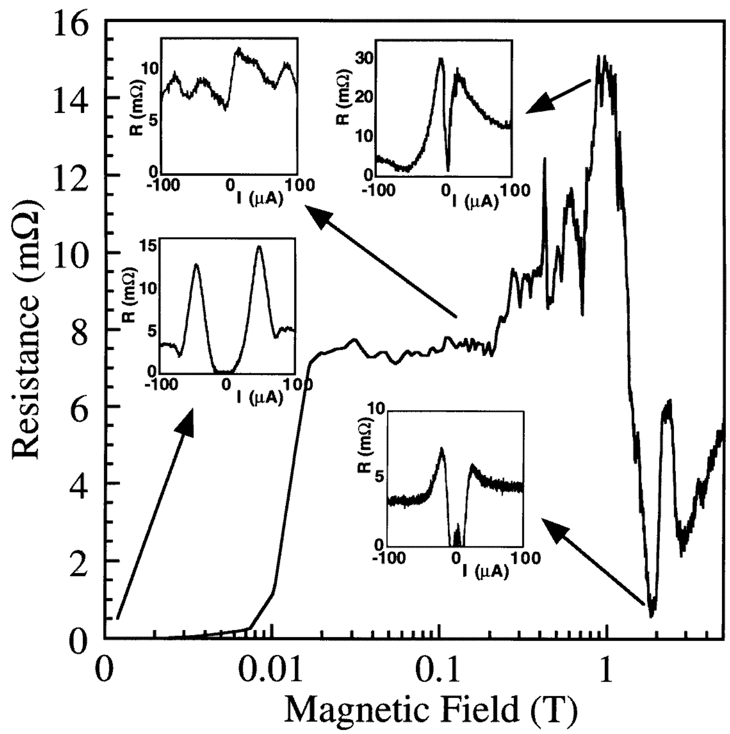

Figure 1. Magnetoresistance trace of a mesoscopic aluminium sample, showing re-entrant behaviour near 1.95 T. The resistance peak of approximately $2R_N$ at around 1 T is very much greater than has been previously reported. The insets show the response for current sweeps with the field held constant.

was by means of four underlying normal-metal probes at the corners of the square, shown schematically in figure 2(a). Further devices were also fabricated with similar thicknesses, but with sides approximately 20 μm as near-macroscopic control samples. Both the probe fingers and the aluminium block were fabricated using electron-beam lithography, and the aluminium was deposited either by RF sputter deposition in 5×10^{-4} mbar of argon, or by thermal-resistive evaporation at a pressure below 1.5×10^{-6} mbar. Differential resistance measurements were performed in a dilution refrigerator with 17 Hz a.c. excitation currents of between 10 μA and 200 μA r.m.s., used with standard lock-in techniques in four-terminal measurements. Magnetoresistance measurements were made at fields of up to 8 T, and at temperatures between 40 mK and 1.2 K. The material used had a resistivity of 2.9 $\mu\Omega$ cm. Large samples showed critical fields of 10–11 mT and $T_c = 1.2$ K. High-resolution scanning electron microscopy showed surface granularity with a characteristic scale of 40 nm.

All samples were superconducting near zero field, and saturated to a normal-state resistance of 8–12 mΩ at high field and above 1.2 K. The large samples showed no high-field fluctuations. In the smaller devices, large resistance fluctuations were observed at fields of the order of 0.5–2 T with peak excess resistances of $> 2R_N$, and minima approaching 0 Ω.

Figure 1 shows the resistance of a 1.6 μm device during an upward (slow) field sweep. As the field departs from zero, the resistance first increases sharply as the device passes through H_{c2}, remains approximately constant until about 250 mT, fluctuates strongly, then at

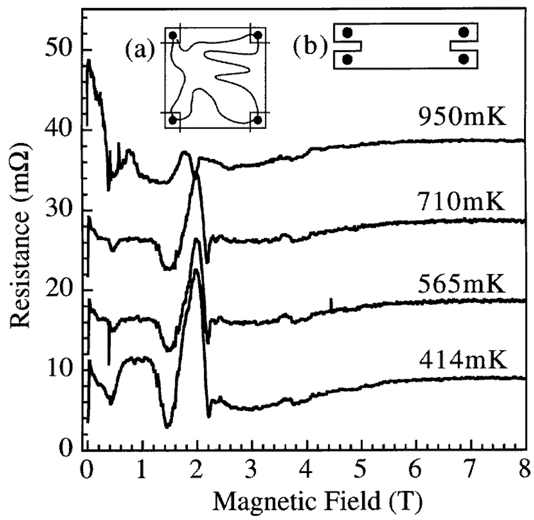

Figure 2. Magnetoresistance traces showing re-entrance and large excess resistance at various temperatures (all $< T_c$). Inset (a): a schematic picture showing the contact arrangement and a putative filamentary path connecting all contacts. Inset (b): the ribbon contact geometry of Nordström and Rapp [14].

around 1.95 T decreases to a minimum comparable to the zero-field value. The insets show d.c. current sweeps to ± 100 μA with the applied field fixed at four representative points. At zero field, the inset shows the differential resistance rise as the critical current (20 μA) is reached, then go through a peak before settling at R_N at higher currents. The inset at 1.95 T also shows a clear threshold behaviour, with a critical current of approximately 5 μA, suggestive of superconducting behaviour at this field. A small resistance is measured at zero current, and the application of a small current reduces the resistance to zero. This response, of a central resistance peak within the critical current (or field) is seen in many mesoscopic aluminium samples of this geometry, and similar properties have also been observed previously for mesoscopic superconducting aluminium wires [11].

The inset sweep taken near the resistance peak shows a large change in resistance with current, peaking at approximately $3R_N$, and troughing below 1 mΩ, showing that the measured response in this region is very sensitive to the applied field or current. This contrasts with the current response in the plateau region, which is relatively even. No fluctuations were seen at higher fields or currents than are presented here. At high field (8 T) the impedance saturated at 8–12 mΩ for all samples, as can be seen in figure 2.

Figure 2 illustrates upward field sweeps at several temperatures on one 1.6 μm structure, showing re-entrant behaviour followed by a resistance peak which is strongest at around 0.4 K. The maximum value of the peak at zero applied bias is approximately $2R_N$, which is a much larger excess resistance than has been reported in mesoscopic superconducting samples to the best of our knowledge. Some other 1.6 μm devices also exhibit a smaller excess resistance near the transition at 20 mT, in a similar manner to behaviour reported in other mesoscopic superconducting structures [9, 13].

Large hysteresis was seen on field cycling (figure 3). Often the re-entrant resistance dips were not observed or did not reach such a low resistance on downward field sweeps (from several tesla to zero field), but were almost always present on upward sweeps. Downward sweeps would instead show intermediate resistances or even normal-state type behaviour for much of the high-field end of the sweep. This is similar to multistability observed in mesoscopic aluminium samples by Antonov and Petrashov [20], and we also occasionally observed a spontaneous and sudden change in the sample resistance during the sweep (figure 3).

Negative magnetoresistance and resistance fluctuation effects are well-understood in normal mesoscopic metal and semiconductor systems in terms of weak localization effects. Although many magnetoresistance effects in mesoscopic superconducting systems are understood, there has been no single explanation for the existing observations. For example, Santhanam *et al* [11, 12] reported that in a small magnetic field range and at temperatures close to T_c, negative magnetoresistance is seen in mesoscopic superconducting structures, and that this is generally accompanied by an unusual temperature dependence, with a small resistance peak ($R_{max} \approx 1.1R_N$) at temperatures just above T_c. Excess resistances have been observed in normal-metal devices with superconducting inclusions [22], as have a range of other effects: notably multistable magnetoresistance curves and a vastly extended proximity effect length [23] within the mesoscopic normal material.

A number of mechanisms for such effects have been proposed, mostly involving transport through a disordered mosaic of superconducting and normal regions within the device [13–18]. A geometry-dependent spatial variation of the order parameter, which would be expected in granular disordered material, explains a resistance increase but not an excess resistance [13, 14]. Analysis based on quasi-one-dimensional wire arrays [20] gave good quantitative agreement with experiment, as did a similar treatment based on node superconductivity [8] whereby T_c is altered slightly at branch nodes in the superconducting metal, creating normal regions when $T \approx T_c$. In this model, most current tunnelling through a normal node is via quasi-particles, so mismatches in quasi-particle conduction levels constitute a conduction tunnel barrier with $R > R_N$. Any dependence on probe lead geometry is then explained in terms of changes in constriction widths at the nodes.

The effect of probe geometry has been explored [13, 14] and has been found to be critical in the observation of excess resistances in disordered superconductors. In such a system, as the temperature(field) approaches $T_c(H_c)$, superconducting regions become discontinuous and/or filamentary, and no longer necessarily connect all four electrical probes. A ribbon geometry [14] is shown (inset in figure 2(b)), in which the largest excess resistance ($\approx 1\%$) was observed for a geometry using all four fingers as electrical probes, thus maximizing the possibility of disconnection of one or more probes from a connected superconducting path. This geometry is greatly exaggerated in the square probe arrangements reported here, to which we attribute the large excess resistances observed, and from which we infer that the sample at these fields may contain a mixture of normal and superconducting regions.

Re-entrant superconductivity has been observed previously near T_c: Lee *et al* [19] reported N–S–N re-entrant transitions in a mesoscopic aluminium wire as a small magnetic field was swept upwards near T_c, but proposed no explanation. In contrast, the results presented here are observed well below T_c and at relatively high fields. Recently, Spivak and Zhou [21] extended earlier theoretical work to present a theory of disordered mesoscopic superconductors near the upper critical magnetic field, predicting re-entrant superconductivity at low temperatures, well below the zero-field T_c, as the magnetic field is swept upwards. Their model, like those of Kim *et al* [17] and Moshchalkov *et al* [8], proposes a microstructure of multiply-connected paths in a disordered superconductor array, which depends on a mixture of disorder in scattering and fluctuations of the order parameter which have inverse field dependences. They predict re-entrant behaviour around the bulk value of H_{c2}, and a sample is thus expected to undergo multiple S–N–S–N... transitions

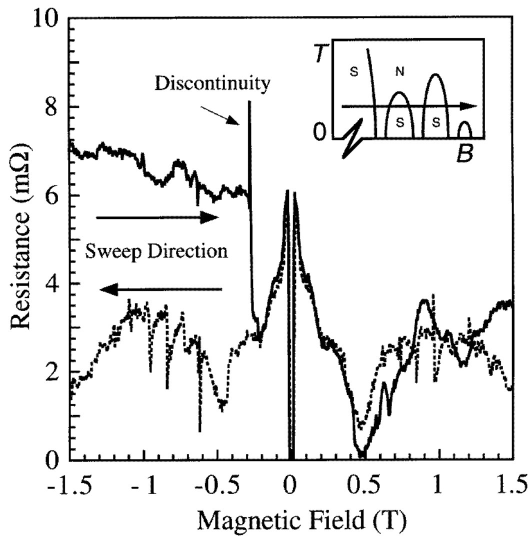

Figure 3. Hysteresis in the magnetoresistance response. The horizontal arrows show the field sweep direction. A large discontinuity at −200 mT is indicated. Inset is a proposed phase diagram for a mesoscopic superconductor (after Spivak and Zhou [21]).

at low temperatures as the magnetic field is swept upwards.

In this experiment, we traverse such a phase diagram (figure 3, inset) close to $T = 0$, and one would expect to see magnetoresistance fluctuations in a sample exhibiting this type of re-entrant superconductivity. However, estimates of the spread of fluctuations, δH^*, and the width of the re-entrance, δH_c, using the approximation of Spivak and Zhou, give 20 mT and 10 mT respectively, compared with the observed values of 0.5–1 T and 20 mT. In these experiments, sample-dependent structural variations are to be expected due to variation in deposition detail, and the large difference in δH^* is attributed to the approximation which estimates the region of magnetic field where such behaviour is expected with probability close to unity; it does not correspond to a limit for a real specimen. The values of δH_c are closer, and the measured critical current implies that approximately one hundred channels are open at this point (at 44 nA/channel). This is reasonable for an experimentally accessible linkage in this geometry, and it is not surprising that the approximation of a single-channel linkage is not realized in these experiments. The large number of channels observed also suggests an explanation for the high value of δH^*, as one would expect the resistance to applied field to be higher in this case.

The key here is that the samples are mesoscopic both in terms of the superconducting order parameter and the scattering disorder due to the microstructure. The grain size of the aluminium films has been estimated from scanning electron microscopy, but a more detailed microscopic examination is needed for a better estimate of the disorder. Other experiments have measured microscopic samples [24] with most dimensions much less than ξ_0 for Al, and have not observed re-entrant behaviour. This has only been observed where the combination of size and microstructure leads to a balance between the effects of scattering and field penetration, giving a regime where mesoscopic fluctuations in the order parameter dominate the charge transport, in agreement with prediction. Probe geometry may also be an essential factor, since the apparent sample impedance will depend on how closely the current path approaches the voltage probes, which may be strongly variable, dependent on detailed sample configuration, as for the excess resistance. Whether re-entrant superconductivity is observed depends in part on whether all four measurement probes are connected by a continuous superconducting filament (figure 2(a)). This criterion will clearly depend strongly on microstructure, probe geometry, applied field and temperature, and on the device history with respect to all these parameters.

Several samples showed similar behaviour, although that illustrated was the most extreme. The experiment was repeated many times on each sample, and gave very similar but not identical results. We attribute this to a combination of flux trapping and the configuration of superconducting and normal regions in the device, which will depend greatly on the local magnetic field, and thus be strongly affected by trapped flux and by the dynamics of flux penetration and exclusion. Occasionally devices stopped showing clear fluctuations after a high-field sweep, however subsequent thermal cycling to around 1.4 K (above T_c) restored the earlier behaviour, which is strongly indicative that flux trapping was responsible for the hysteresis and bistability. (Hysteresis with magnetic field cycling which disappears when the sample is warmed above T_c is difficult to explain other than by flux-trapping by superconducting regions, given the materials present.)

The persistence of superconductivity to high applied fields in disordered materials has been observed in disordered tin alloys [25] and high values of H_c are observed in thin films of aluminium [26], which supports the picture of regions of differing order parameter in these mesoscopic structures. These results extend to sub-micron length scales the observations of filamentary behaviour in the intermediate state of thin-film superconductors [27] and recent microscopic observations of flux-quantum dynamics [28].

In conclusion, the magnetoresistance of small aluminium structures in a square geometry has been reported, and large fluctuations with a range from near-zero to $2R_N$ are seen. The observation of apparent re-entrance of a low resistance state, the hysteresis with magnetic field cycling, and the resetting of the behaviour by thermal cycling indicate that regions of superconductivity persist within the aluminium film at fields much greater than H_{c2}, at these low temperatures. We believe that this is the first observation of re-entrant superconductivity at temperatures well below the zero-field T_c, and at such high magnetic fields, and suggest that a modified Spivak–Zhou model [21] may be able to account for these results, in that the samples contain multiply-connected disordered superconducting regions, in a geometry which is field-, temperature- and sample structure-dependent, and which in the specimen shown has a superconducting filament with a weak link of approximately 100 channels. Further work on probe geometry and material disorder is needed to gain a more detailed understanding of the microscopic structure under these conditions, in order to make quantitative links with a model.

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