Piezoresistive torque magnetometry below 1 K

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(Received 30 April 1998; accepted for publication 10 November 1998)

We have investigated the performance of piezoresistive cantilevers as magnetometers in the temperature range below 1 K. The de Haas-van Alphen effect was used to study the temperature dependence of a sample of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$, fixed on the end of a cantilever, as a function of the excitation current through the piezoresistive device. We found that by using a small thermalizing wire connected directly to the sample, large excitations were not incompatible with sample temperatures remaining low, thereby establishing the use of these devices as sensitive magnetometers well below 1 K. A large hysteretic behavior observed at low fields (below 0.01 T) and low temperature (below ~2 K) precludes their use in that regime. © *1999 American Institute of Physics.* [S0003-6951(99)01503-X]

Torque magnetometry is a powerful technique. It is a good tool for measuring magnetization in high magnetic fields because the signal increases linearly with field amplitude for a given magnetic moment ($\boldsymbol{\tau}=\mu_0\boldsymbol{\mathcal{M}}\times\mathbf{H}$). Also, since the torque depends only on the perpendicular component of the magnetization, it is a sensitive probe of anisotropy.

Small cantilever based torque magnetometers can be made using microfabrication techniques. Many detection mechanisms are possible. This can be an optical readout[1] which requires good alignment of optical fibers, or it can also be capacitance based, in which case care must be taken to eliminate parasitic capacitance. We are interested in another technique, a piezoresistive readout. This makes force measurement as simple as measuring a resistance. The drawback is that to obtain a high signal-to-noise ratio a large excitation current must be used which can change the temperature of the sample under study. We will show that this heating problem can be avoided even at temperatures as low as 0.1 K.

This type of cantilever has previously been used to perform scanning force microscopy[2] and low temperature ($T\geqslant 6$ K) magnetic force microscopy.[3] As a torque magnetometer it was used to measure the effective mass anisotropy in the mixed state of some high-temperature superconductors around 100 K.[4]

The piezoresistive cantilever[5] is microfabricated in silicon. It has a U shape and the top portion is heavily doped, thus creating a conduction channel, whose resistance is about $R=2.2\,\mathrm{k}\Omega$ at room temperature. Because the resistance changes when the cantilever bends, a transverse force applied on the end of the cantilever can be measured via changes in the resistance of the device. A relative change $\Delta R/R$ of 1 ppm corresponds to a transverse motion of the end of 2.5 Å. The spring constant of the cantilever is about 20 N/m, specified by the manufacturer, so 1 ppm also corresponds to 5 nN. Given a cantilever length of 75 μm, 1 ppm is roughly equivalent to a torque of 5×10^{-13} N m.

In order for this type of device to be useful as a sensitive magnetometer at low temperature, it is important to show that the temperature of a sample fixed to the end of the cantilever can be kept low even when sizable excitation currents are used in the resistance measurement. In order to investigate the relation between excitation current and sample temperature, the de Haas–van Alphen (dHvA) effect was measured in a suitably chosen crystal. This effect is the quantum oscillations in the magnetization of a metal as a function of magnetic field, periodic in inverse field. Its relevance in the present context is that the amplitude of the dHvA oscillations depends very strongly on temperature, the more so the higher the effective mass.

In the case of electron motion confined largely to two dimensions, the fundamental oscillatory component of the magnetization is given by[6]

$$\tilde{\mathcal{M}}\propto\frac{V}{d}\frac{F}{m}R_TR_D\sin\left[2\pi\left(\frac{F}{\mu_0H}-\frac{1}{2}\right)\right], \tag{1}$$

where d is the layer spacing, μ_0 is the magnetic permeability of vacuum, m is the cyclotron effective mass (in units of the bare electron mass m_e), F is the dHvA oscillation frequency (in Tesla), μ_0H is the magnetic field amplitude (in Tesla), and V is the sample volume. R_T and R_D are the temperature and Dingle factors, respectively, given by

$$R_T=\frac{14.69mT/\mu_0H}{\sinh(14.69mT/\mu_0H)}, \tag{2}$$

where T is the temperature (in Kelvin) and $R_D=\exp(-17.85m\Gamma/\mu_0H)$, where Γ is the scattering rate (in THz).

The torque exerted by a magnetic field on a sample is only nonzero if the magnetization vector makes an angle relative to the direction of applied field. This means that the crystal under study must have some degree of magnetocrystalline anisotropy and the field must be applied at an angle relative to the high-symmetry axes of the crystal. By choosing a material with quasi-two-dimensional electron motion, and a quasicylindrical Fermi surface, so that both the effective mass and the oscillation frequency go as $1/\cos(\theta)$, and

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by applying the magnetic field at an angle θ from the normal to the conduction plane, the torque on the cantilever end is maximized.

The sample selected for these experiments was a single crystal of the organic superconductor κ-(BEDT-TTF)$_2$Cu(NCS)$_2$, supplied by Dr K. Behnia of the CNRS at Orsay, France. It was grown by a standard electrochemical technique,[7] in the form of a platelet of regular thickness but irregular shape, with approximate dimensions $750\times450\times40\,\mu$m. The shorter dimension is perpendicular to the plane of the material, which is the plane of conduction in this highly two-dimensional electron system. This material has a superconducting T_c of 10 K and a H_{c2} of about 5 T. In previous dHvA studies, using other techniques,[8–10] one dominant oscillatory component was observed, with $m=3.2m_e$ and $F=0.601$ kT.

Our measurements were performed in a dilution refrigerator equipped with a 15 T superconducting magnet. The resistance of the device was measured using a digital lock-in amplifier (SR-850 from Stanford Research Systems). Combining it with a ratio transformer we offset the signal around 0 V which allowed us to use a more sensitive range on the lock-in to measure slight changes in resistivity. Below 1 K with a bandwidth of 1 Hz the signal was limited by an electrical noise of 10 nV (10 mΩ using 1 μA). This can be improved by better shielding, better electronics and a larger excitation current. This 4 ppm of noise corresponds to a torque of 2×10^{-12} N m. This is 2×10^{-13} A m^2 at 10 T which compares well with commercial SQUIDs which have a sensitivity of 10^{-11}–10^{-12} A m^2.

The sample was mounted on the end of the cantilever using a small drop of vacuum grease (Dow Corning high vacuum grease), and oriented with the normal to its plane at an angle of about 30° with respect to the field direction. To prevent the sample from warming up to the same extent as the cantilever, a thermalizing wire was fixed directly onto the sample. This was a 50 μm diameter copper wire of about 2 cm in length, providing good thermal conduction without being so stiff as to prevent a good torque measurement. It was stripped of its insulation to make it more flexible, without affecting heat conduction. One end of the wire was attached to the sample using silver paint and the other end was soldered to the mount. Upon cooling, because of thermal contraction, it bends the cantilever slightly, but this does not change the sensitivity.

The observed signal is shown in Fig. 1. A polynomial fit of the signal was subtracted to remove the background due to the magnetoresistance of the cantilever. The inset shows the frequency spectrum of the data with a single sharp peak at 690 T. This is in excellent agreement with the previously reported values, assuming an angle of 30°.

To determine the temperature of the sample, as a function of refrigerator temperature, field sweeps from 9.6 to 10.0 T were performed at several different refrigerator temperatures. Each sweep contained the same three oscillations. Each set of data was then fit to:

$$R=A+BH+C\sin\left(2\pi\frac{F}{\mu_0 H}+D\right), \quad (3)$$

where A and B account for the magnetoresistance of the cantilever (linear in this very short interval) and D is an arbitrary phase. The frequency F is fixed to the value of 690 T obtained from the longer sweep (between 5 and 10 T) and C is the temperature-dependent amplitude of interest. By varying the temperature, the latter should change according to R_T [see Eq. (2)]. Figure 2 shows the amplitude as a function of refrigerator temperature for different excitation currents through the piezoresistance. The solid curve is obtained from a fit of all the data represented by open symbols (0.04–1.0 μA) to the expression for R_T [Eq. (2)]. The effective mass and the 0 K amplitude are the only adjustable parameters, and the best fit gives $m=3.65m_e$. This, again, is the expected value for an angle of roughly 30°. The fact that all the data for excitations of 1.0 μA or less fall on the theoretical curve shows that no significant heating of the sample oc-

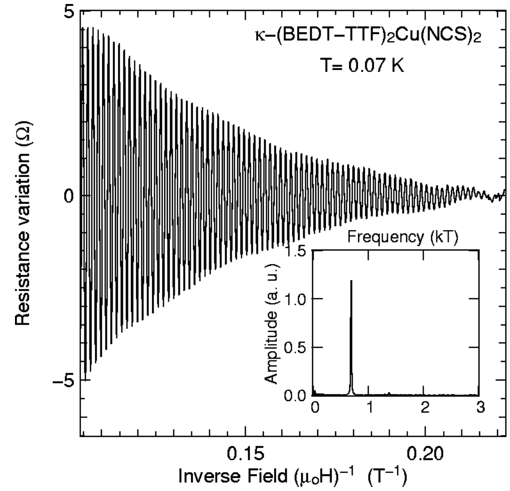

FIG. 1. dHvA signal versus inverse field for sample of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$. The inset shows the Fourier transform of that signal.

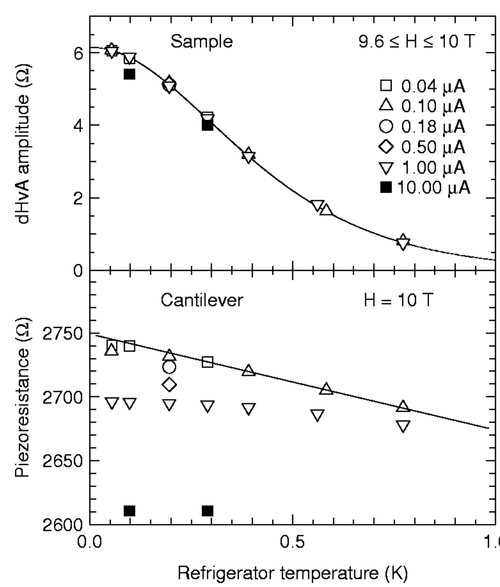

FIG. 2. Top panel: Amplitude of dHvA oscillations from crystal of κ-(BEDT-TTF)$_2$Cu(NCS)$_2$ vs refrigerator temperature, for different excitations (as indicated). The line is a fit to Eq. (2), using only data with open symbols. The filled squares are data obtained for a very large excitation and show some degree of heating. Bottom panel: Resistance of the piezoresistive cantilever at 10 T, for the same excitations. The line is a guide to the eye. At the largest excitation, the cantilever temperature is well above 1 K.

curred. This is no longer true for an excitation of 10 μA, the data for which is shown as filled squares in Fig. 2. Indeed, at a refrigerator temperature of 100 mK, the sample was warmer by roughly 60 mK (or 60%). Note, however, that this discrepancy decreases rapidly with temperature as a result of improved heat conduction, as seen for the point at 290 mK, which is only off by about 10 mK (or 3%).

This behavior is to be compared to the temperature variation of the cantilever itself, under the same measurement conditions. Given that the piezoresistance is temperature-dependent, the cantilever temperature is obtained from the constants A and B at a fixed field. Figure 2 shows the piezoresistance (determined in this way) as a function of temperature for the same set of excitation currents, at 10 T. The straight line is simply a guide to the eye passing through the points for the lowest excitation. It is interesting to look at the data at high excitations. For 1.0 μA, the sample and reference thermometer (on the mixing chamber of the dilution refrigerator) are isothermal even at 50 mK, while the cantilever never cools below 700 mK. For 10 μA, sample and reference are almost isothermal at 300 mK, but the cantilever has warmed up to 2 K or so. Hence the thermalizing wire is very effective at keeping the sample cold and enables the use of larger excitation currents than otherwise possible.

A cautionary note on the use of these cantilevers at low fields is in order. A large and abrupt hysteretic change in the resistance of the device was observed for fields below 10 mT at temperatures below 1 K, reaching a relative change of 4% at 70 mK. It was present with and without a sample fixed on the cantilever, and was reproducible. This effect is probably attributable to weak localization in the doped silicon channel. It certainly prevents the use of the device at low fields in this temperature range. At higher fields, the magnetoresistance varies smoothly (+10% for 10 T) and is not hysteretic. Given that the sensitivity of a torque magnetometer increases with field, the most promising range is obviously at high fields, where the sensitivity can become comparable to, and even exceed, that of a SQUID.

In summary, the dHvA effect was used as a measure of sample temperature to test the effect of excitation currents through a piezoresistive cantilever on the temperature of a sample fixed on the end. In order to minimize the heating effects, the sample was anchored to the refrigerator using a copper thermalizing wire attached with silver paint. From the dHvA data, we observed that this wire could be very effective in keeping the sample cold, at temperatures below 100 mK, even in the presence of excitation currents large enough to significantly warm up the cantilever itself. The observation of dHvA oscillations and the possibility of using a relatively large excitation current, hence of having high sensitivity, demonstrate that this device can be useful for magnetization measurements at very low temperature.

The authors would like to thank Rob Hill for fruitful discussions. This work was funded by NSERC of Canada and FCAR of Québec. L. T. acknowledges the support of the Canadian Institute for Advanced Research and the A. P. Sloan Foundation.

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