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SQUID Magnetometer for Magnetization Measurements

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Abstract

SQUID magnetometer for magnetization measurements. K. Gramm, L. Lundgren and O. Beckman (Institute of Technology, University of Uppsala, Box 534, S-751 21 Uppsala, Sweden).
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A magnetometer using a symmetric rf-biased SQUID (Superconducting quantum interference device) is described. The instrument has been designed for magnetization measurements of small single crystals at low magnetic fields in the temperature range 4.2–400 K. At low temperatures the sensitivity in magnetic moments is 2×10^{-12} Am^2 or 2×10^{-9} emu, which is equivalent to $\Delta\chi=10^{-7}$ SI with sample volume 3 mm^3 at 0.01 T (100 Oe).

1. Introduction

The use of magnetic flux quantization in a superconducting cylinder makes possible very high sensitivity in voltage, current and magnetization measuring devices. An early superconducting magnetometer [1] could detect susceptibility changes of 10^{-10} cm^3/g in 10 kOe equivalent to a change in a magnetic moment of approximately 10^{-6} emu or 10^{-9} Am^2. The utilization of the Josephson effect has further lead to an extensive development of new instruments of high sensitivity. A typical superconducting detector for magnetic flux is the symmetric rf-biased SQUID (Superconducting quantum interference device) described by Zimmerman, Thiene and Harding [2]. An extensive account of the operating principle is given by Silver and Zimmerman [3] and several applications to electric and magnetic measurements are given in a review paper by Giffard et al. [4]. Day [5] has described a superconducting magnetometer for both static susceptibility measurements and nuclear magnetic resonance studies. Further Cukauskas et al. [6] reports a similar instrument, where volume susceptibility changes of 10^{-10} cgs in a 1 cm^3 sample can be measured in an applied field of 100 Oe, equivalent to a change in magnetic moment of 10^{-8} emu (or 10^{-11} Am^2).

In a joint project with the Institute of Inorganic Chemistry at Uppsala on structural, electric and magnetic properties of transition element compounds we have constructed a superconducting magnetometer specially designed for milligram samples and very low applied fields. The working temperature range is 4.2–400 K. At low temperatures the sensitivity is equivalent to changes in magnetic moment of 2×10^{-12} Am^2 or 2×10^{-9} emu. At higher temperatures the sensitivity is somewhat reduced due to noise from the sample holder assembly.

2. Instrumentation

The main part of the SQUID magnetometer, i.e. the SQUID unit, is shown in Fig. 1. The unit is immersed in a He-bath at 4.2 K and contains essentially the following details: the SQUID, a magnetic flux transformer including pick-up coils, magnet coil, heatswitches and magnetic shieldings.

The SQUID with its electronic circuits (from S.H.E. Manufacturing Corp, San Diego, California) can detect about 10^{-4} of a flux quantum ($\phi_0=h/2e$). The magnetic flux transformer, made of superconducting Nb-wire (0.08 mm in diameter), consists of the pick-up coils and the SQUID coil. A satisfactory superconducting contact is achieved by pressing the wires mechanically together. During operation it is sometimes desirable to destroy the supercurrent in the magnetic flux transformer. Thus a 10 kΩ carbon resistor is mounted in thermal contact with the wire. A carbon resistor was chosen rather than a heating coil in order to minimize unwanted magnetic fields near the magnetic-flux transformer. The magnetic flux transformer has to be supported mechanically to avoid vibrations, and it must be rigorously shielded against external magnetic fields. The shielding is done by surrounding the twisted leads with superconducting lead. The whole SQUID unit is further enclosed in a superconducting lead can and the cryostat is surrounded by three layers of μ-metal. The inner μ-metal can is immersed in liquid nitrogen in order to reduce fluctuations of the magnetic domains of the μ-metal [3]. The total μ-metal shield reduces stray magnetic fields by a factor of appr. 100.

The two counterwound pick-up coils have a diameter of 3.6 mm, each containing 30 turns. The SQUID coil has 80 turns and a diameter of less than 2 mm, thus just fitting one of the SQUID's holes.With this configuration 4.2% of the flux change in a pick-up coil is transformed into the SQUID. The pick-up coils are wound on a copper cylinder, which separates the sample from the He-bath. The copper cylinder is attached to a stainless steel tube and forms a vacuum space for the sample. The sample may be heated by a small Allan-Bradley resistor attached to the sample holder (Fig. 1), which is made of pure 99.999% copper. The temperature of this copper block is measured by copper-constantan and gold (iron)-copper thermocouples attached to the upper part of the sample holder. The sample is glued to the lower part of the sample holder, which forms a 1.8 mm diameter copper pin of 18 mm length. (This arrangement is of great importance, since the heater and thermocouple otherwise might give rise to background noise in the transformer coil.) Because of the good thermal conductivity of copper, the temperature difference between the sample and the thermocouple never exceeds 0.5 K, even at 300 K.

The sample is magnetized by a superconducting magnet, operating in persistant mode. This mode of operation is essential, since a magnetic field of extreme stability is needed in order to avoid fluctuations caused by magnet current noise. The magnet coil produces a maximum field of 0.14 T, has a mean diameter of 6 mm, a length of 30 mm, and is made of 0.12 mm Nb-wire with 99.9% purity. Thus the SQUID magnetometer works in a comparatively low region of magnetic field.

Niobium was chosen as the material for both the superconducting magnet and the flux transformer circuit, since the intention

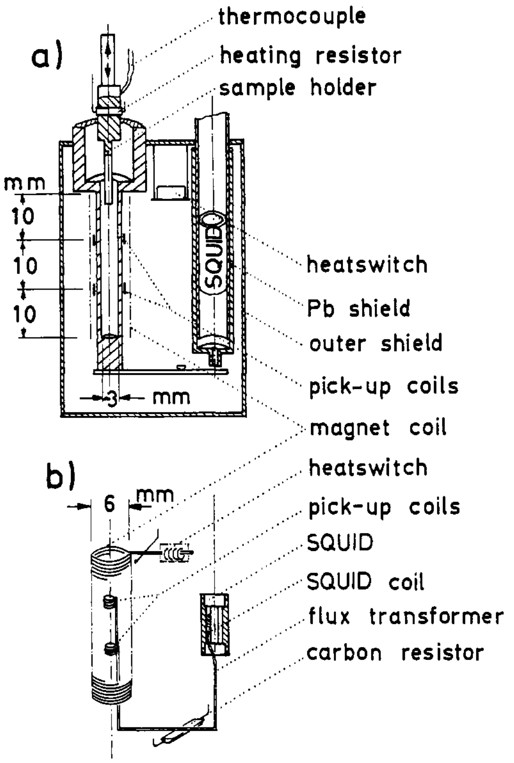

Fig. 1. (*a*) Magnetometer unit. (*b*) Magnet coil and flux transformer.

has been to measure the magnetization at very low fields. Part of our research program involves studies of critical phenomena at magnetic phase transitions, where the applied field should be as low as possible; i.e. 10^{-3} T (10 Oe) or less.

3. Measuring principle

The magnetic field B is produced by the superconducting magnet coil. Then the sample is slowly moved through the pick-up coils. This is done by means of a hydraulic system in order to avoid mechanical vibrations. The magnetic moment of the sample induces a magnetic flux change in the pick-up coils. The magnetic flux transformer exhibits a superconducting loop, thus transforming part of the total magnetic flux change from the pick-up coils into the SQUID. A typical signal curve is shown in Fig. 2.

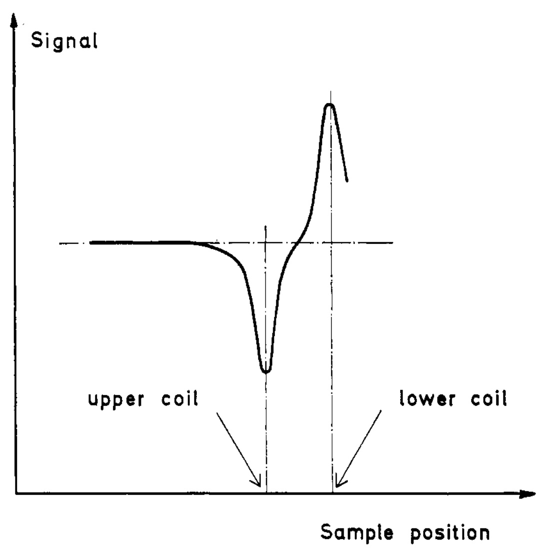

Fig. 2. XY-recorder plot, as a result of moving the sample through the pick-up coils.

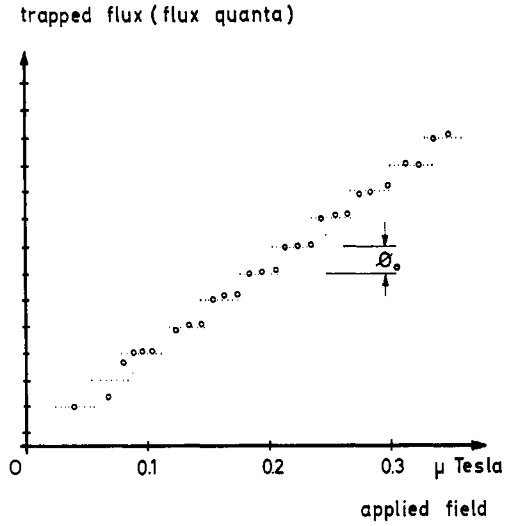

Fig. 3. Quantization of the magnetic flux within a thin superconducting ring measured by the SQUID magnetometer.

As shown, the pick-up coils are wound in opposite directions, thus compensating for external magnetic field variations.

Highly magnetized samples must be moved very slowly through the pick-up coils in order not to exceed the maximum slewing rate of the electronic system.

4. Sensitivity of the SQUID magnetometer

The rf-biased SQUID, which has a maximum slewing rate of about 250 flux quanta/s, is capable of detecting changes of order 10^{-4} of a flux quantum. The transformer conducts only a few percent of the flux from the pick-up coil into the SQUID. Thus, in connection with thermal and mechanical noise, the SQUID magnetometer in total can detect a change of about half a flux quantum ϕ_0. This is shown in Fig. 3, where the measured flux was trapped in a very thin superconducting cylinder about 0.2 mm in diameter and 2 mm in length.

The sensitivity of the SQUID-magnetometer can also be expressed by the detectable number N of "aligned" spins. Starting with the magnetic flux $\phi = B \cdot A = \mu_0 \cdot M \cdot A$, where M is the magnetization (corresponding to $\mu_B N/l \cdot A$), the following relation between the number of spins N and the flux quantum ϕ_0 is derived:

$$N = \frac{l}{\mu_0 \mu_B} \cdot \frac{\phi_0}{2}$$

where l is the "length" of the sample with cross-section A. This gives a number N of detectable spins of 2×10^{11} for a sample length of 2 mm, which corresponds to a sensitivity in magnetic moment of about 2×10^{-12} Am^2 or 2×10^{-9} emu.

The sensitivity of magnetometers may also be expressed in terms of the susceptibility χ. Then the sample volume and applied field have to be specified. As an example our magnetometer can detect a change $\Delta\chi = 10^{-7}$ SI with a sample volume of 3 mm^3 and an applied field of 0.01 T (100 Oe).

We have shown that a SQUID magnetometer can be readily used for measurements on small samples and low magnetic fields. An excellent illustration is given in Fig. 4. The figure shows the ferromagnetic phase transition in Fe_2P [7]. The first order character of this transition (which is of magnetoelastic origin) could only be revealed by measurements on single crystals of microgram size in very low fields. (In large crystals the effects of grain boundaries broaden the transition.)

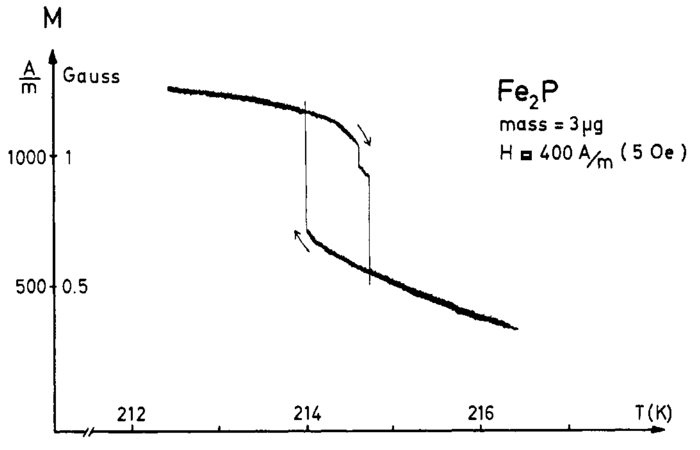

Fig. 4. A temperature sweep in the SQUID magnetometer showing the first order magnet phase transition in Fe_2P.

5. Concluding remarks

The magnetic moment of a sample is measured by passing it through the superconducting pick-up coil. The Meissner effect in the pick-up coil gives rise to some inhomogeneity in the applied field along the coil axis. For small samples this only implies a correction to the magnetometer calibration. For samples, which are longer than the pick-up coil there will be a variation of the magnetic field of about 1% along the sample. For diamagnetic and paramagnetic samples with field independent susceptibility, this effect of inhomogeneity can be neglected. In any case the magnetometer calibration is slightly dependent on sample geometry.

The superconducting wire in the magnet is made of Nb, which is a type II superconductor with a lower critical field H_{c1} of approx. 0.04 T (400 Oe). If the field is raised above H_{c1}, pinned flux in the superconductor will cause an inhomogeneous remanence field. In our instrument such a contribution to the field will never exceed 1 mT (10 Oe); and this becomes of significant importance only when low field measurements are carried out after using higher fields.

High field superconducting magnets must be supplied with a superconducting shield surrounding the pick-up coil and sample [5]. The Lorentz force between field and current in the superconducting magnet gives rise to flux creep, which will cause a serious noise when the magnet is operating in the persistant mode. Such noise is greatly reduced by the superconducting shield. The shield is kept normal until the magnet is in the persistant mode at the operating field. When the shield is then cooled below the transition temperature there are no currents in the shield and thus no flux creep affecting the field at the sample. In low field magnets such as our instrument, the flux creep is never serious and the differential shield was omitted.

In order to simplify the construction of the magnetometer pure copper was used as material for the support of pick-up coils and sample. Although this material is of very high purity, traces of iron or oxygen will introduce a temperature dependent paramagnetic signal. This is especially the case for the copper pin supporting the sample at very low temperature measurements. Copper further has a considerable nuclear paramagnetism, which also contributes to a temperature dependent background signal. The nuclear paramagnetism may be reduced by about two orders of magnitude by using silver or gold instead of copper. However, Johnson noise currents in metallic materials will give an extra contribution to signal noise. In order to eliminate the Johnson noise an insulator might be used. The pick-up coils could be wound on pure quartz, but the pin supporting the sample would have to be made of a material of good thermal conductivity, e.g. sapphire, crystalline quartz or silicon.

Acknowledgements

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