Calibration of SQUID magnetometer for dilute alloy studies

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A SQUID magnetometer used in the study of dilute magnetic alloys or other weak magnetic systems can be calibrated by using the nuclear paramagnetism of aluminum metal. This material is useful as a calibration standard since magnetic impurities such as Fe and Mn form no local moment. This calibration is compared to others obtained with CMN powder and indium metal.

INTRODUCTION

The SQUID magnetometer with its extremely high sensitivity makes a very useful instrument for the study of weakly magnetic systems, particularly at low temperatures. Although high sensitivities can be achieved with vibrating sample magnetometers in relatively high fields,[1] the SQUID magnetometer is most useful at low fields. It has been used successfully, for instance, in the study of dilute magnetic alloys,[2] where single-impurity effects are of interest. With magnetic impurity levels of approximately 1 ppm, high sensitivity is needed. In temperature-dependence studies of these alloys, it is important to know the absolute value of the measured moment. This requires an accurate calibration which, because it is somewhat different from conventional methods, will be presented here.

In principle one can calculate from the geometry of the system an absolute measure of the magnetic moment. The accuracy of such a calculation, however, may not be good as an experimental comparison. Often the saturation moment of a small sphere of nickel is used for a standard or, alternately, one can use a small direct current loop of known area and number of turns. The absolute accuracy of the instrument depends on a knowledge of magnetic properties of the calibration standard and reproducibility of sample position. A value of ±1% is usually quoted as resulting from estimates of the uncertainty in the magnetic moment of nickel.[3]

Because of technical difficulties associated with using a nickel standard with a SQUID magnetometer, we propose the nuclear magnetism of metallic aluminum as a calibration standard for studies of weakly magnetic systems. We compare the aluminum magnetization with that of indium, cerium, magnesium nitrate, and a calibration coil of known turns and area.

I. EXPERIMENTAL DETAILS

A conventional SQUID magnetometer using SHE model 201 electronics provides the main part of the equipment. To measure small magnetizations it is important to reduce background effects in the magnetometer as much as possible.[4] A superconducting flux transformer is used to couple the magnetic flux from the sample to the SQUID sensor, allowing the flexibility of varying sample temperature and magnetic field without affecting the SQUID sensor. Figure 1 shows the basic arrangement. The two pickup coils of the magnetometer are wound in an astatic configuration to cancel stray signals. The magnetization of a sample is measured in a steady magnetic field H trapped while cooling in a superconducting niobium tube, 2.79 cm long and 7.8 mm in diameter. The SQUID is electronically locked in feedback mode on one flux quantum[5] and is capable of following flux changes without unlocking, provided such changes do not exceed the slewing rate of the amplifiers in the SQUID electronics. A change in flux from the sample gives a voltage output, zero voltage being arbitrary.

The pickup coil of the magnetometer is located inside the mixing chamber of a $^3He-^4He$ dilution refrigerator. Such an arrangement makes it inconvenient to move a magnetic body in and out of the magnetometer for calibration. In order to find the proportionality between the output voltage of the magnetometer and the magnetization of the sample, the following procedure has been used. The SQUID responds to a flux change, caused by a change in sample magnetization, ΔM,

$$\Delta\phi = 4\pi f A \Delta M, \qquad (1)$$

where f is the flux transfer ratio relating the flux change

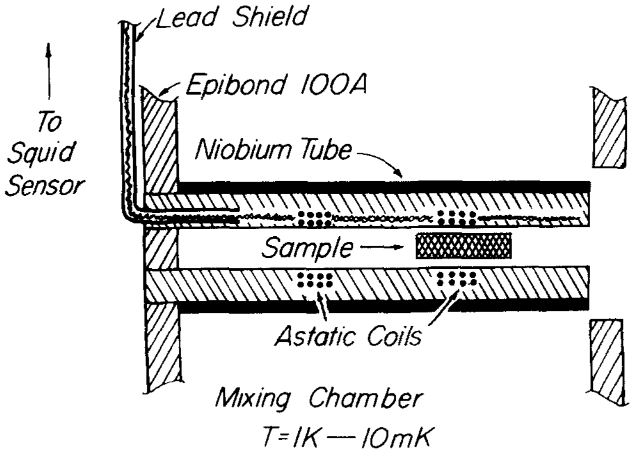

FIG. 1. SQUID magnetometer with pickup section inside mixing chamber of a $^3He-^4He$ dilution refrigerator.

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at the SQUID to that of the sample, and A is the cross-sectional area of the pickup coil surrounding the sample. The output voltage of the magnetometer is proportional to the change of flux at the SQUID; thus

$$\Delta V = k\Delta M, \tag{2}$$

and the calibration determines the constant k. If the magnetization is measured for a paramagnetic system known to obey Curie's law over the temperature range of interest, the output voltage of the magnetometer will vary as

$$V - V_0 = k(C/T)H. \tag{3}$$

C is Curie's constant, and V_0 is the extrapolated voltage at $1/T = 0$.

II. CALIBRATION

With a magnetometer designed for measurements of weakly magnetic spins, it is practical to calibrate against a standard whose magnetization is comparable. Here we chose a nuclear paramagnet whose magnetization follows Curie's law

$$M = N\frac{g^2\mu_N^2 I(I+1)}{3kT}H \tag{4}$$

down to very low temperature; the nuclear ordering temperatures in most metals are of the order of 10^{-6} K or less. From NMR data, Lande g factors and spin values I are well known; hence, Curie constants can be accurately calculated for calibration of the magnetometer.

In practice it is difficult to obtain materials of such high purity that magnetic impurities do not contribute to the total sample magnetization. Fortunately the common magnetic impurities found in most metals do not form local moments in certain metals, usually trivalent ones, and will not alter the nuclear Curie law behavior. Such is the case for Al as shown in studies of Al–Mn and Al–Fe alloys.[6] For this reason, and the fact its moment is relatively large, we chose Al for calibrating the SQUID magnetometer. [The sample flux relative to the flux quantum ϕ_0 is $\phi = (35.6/T)\,\phi_0$, which becomes substantial for low T.]

Two different Al samples were used, one of 6N (6 nines) purity and the other of 5N. They were machined into cylinders 2.2 mm in diameter and 6.2 mm long and etched in a solution of NaOH. The magnetization of each sample was measured in a field of 150 Oe for temperatures between 1.5 K and 9 mK. A field of 150 Oe is necessary to keep the sample in the normal state. Thermometry used a second magnetometer with CMN sample. Background magnetization although minimized was further corrected by taking two sets of measurements, one with the sample on each side of the astatic coil pair of Fig. 1. From a plot (Fig. 2) based on Eqs. (2)–(4) the calibration constant was determined. This number is constant for the system and samples of the same size. A factor dependent on variations of sample size can be obtained to good approximation by including a term proportional to the sample cross section, i.e., the diameter squared.

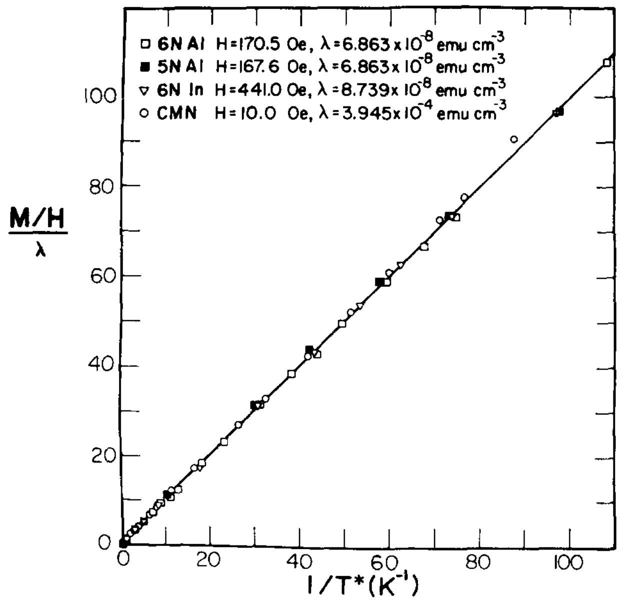

FIG. 2. Normalized magnetization for various samples used in calibration. λ is the Curie constant of each sample.

To check the calibration, the magnetization of CMN powder and the nuclear magnetization of indium were also measured as a function of temperature, for samples of the same size. Figure 2 shows these results as well, the data being normalized to their respective fields and Curie constants. The largest uncertainty in these results is due to the trapped field H which is trapped reproducibly to within ±5%. This value is smaller than that quoted in Ref. 7 and it may be due to the fact that the thickness of the niobium tube is only 0.4 mm. The data from Fig. 2 yield a calibration constant in our system of $k = 1.58 \times 10^4 d^2$ V G^{-1}, where d is the diameter of the sample in cm. Such information allows a direct comparison of any dilute alloy sample using the k obtained here for samples approximately the same size.

As an independent check, a small coil roughly the same size as the sample was placed in the magnetometer. From the coil geometry, the calibration constant k was determined at a fixed T by observing the voltage change at the output of the SQUID for a flux change in the coil. Agreement with the above calibration was good although corrections had to be introduced for the induced fields in the niobium cylinder. Finally, the calibration was checked by observing the total flux change as the aluminum sample becomes superconducting. This measurement is tricky because the signal is large and changes rapidly tending to unlock the SQUID.

The nuclear magnetism of aluminum lends itself naturally as a calibration standard for a SQUID magnetometer. The signal is reasonably large and magnetic impurities do not affect the nuclear magnetization. In a somewhat different application of a SQUID magnetometer, a calibration has been obtained using

^{3}HE[8]. Although CMN is also useful and often used for calibration purposes, it has a signal very large for our application. It furthermore is anisotropic and tends to dehydrate. CMN could not be used to calibrate an ultralow temperature experiment since it orders at millidegree temperatures; aluminum is potentially useful to temperatures much below 1 mK.

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