Application of the Hall-Probe Technique for Magnetization Measurements of Superconductors.

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Abstract. A commercial Hall probe (supplied by Lake Shore Cryotronics, Inc.) is used as a sensor of the magnetic field at the surface of a magnetized sample. With the Linear Research resistance bridge LR-400, an excellent stability, linearity of response on field and an extremely small temperature dependence of the background signal is obtained, which is essential for this kind of applications of a Hall probe. At certain conditions, a resolution in field measurements of about 10 mG is reached. Examples of magnetization measurements as a function of temperature, magnetic field and time, performed on high-T_c and heavy-fermion superconductors, are discussed.

1 Introduction

Magnetization measurements of superconductors with the use of a Hall probe have been reported in several recent publications [1-5]. The critical parameters of the sensors that decide the accuracy of the magnetization measurements are determined by the stability of the Hall voltage and its weak dependence on temperature, as well by the linear field dependence of the signal. Another important parameter in this application is the temperature dependence of the background signal in zero external magnetic field. This offset signal originates presumably from an asymmetry in the positioning of the electrodes. Its value and temperature dependence are governed by the technological process for the fabrication of the sensor. Standard Hall probes are fabricated from narrow-gap semiconductors, as InSb [1] $Cd_{0.175}Hg_{0.825}Te$ [6], or from semimetal bismuth [2] (Table I). Let, for instance, the resistance of a semiconducting Hall probe change from 10 kΩ at 300 K to 40 kΩ at 5 K, while the Hall sensitivity is 1 Ω/Oe. A 1% mismatch of the position of the electrodes leads to a voltage, at changing the temperature from 300 to 5 K, which is equivalent to the application of a magnetic field of 300 Oe. Hence, such a Hall probe is useless for magnetization measurements as a function of temperature. Accurate measurements as a function of field are difficult in that case as well, since the required stability of temperature becomes too high.

TABLE I
COMPARISON OF HALL PROBES USED IN MAGNETIZATION EXPERIMENTS

Ref.	Type	R_H	$(dR_H/dT)/R_H$	$(dR_H/dH)/R_H$
[1]	InSb	50 mΩ/Oe	small	?
[2]	Bismuth	0.6 mΩ/Oe	?	?
[3]	GaAs	1 Ω/Oe	large	small
[6]	$Cd_xHg_{1-x}Te$	5 mΩ/Oe	small	very large
[7]	InAs	8 μΩ/Oe	very small	very small

2 Hall-Probe Characteristics

In this paper we summarize our experience in magnetization measurements with the Hall probe supplied by Lake Shore Cryotronics, Inc. [7]. Its sensitivity is 8 μΩ/G. This is a InAs-type of device, apparently fabricated from a strongly doped material, since it is characterized by a metallic-type of temperature dependence of the resistance. Its nominal 1 Ω resistance at 300 K drops down nearly linearly with decreasing temperature, to half of this value at a few tens of degrees and then becomes temperature independent, down to 0.4 K at least. These results are consistent with detailed studies of InAs Hall probes [8].

The sensors are encapsulated in rugged, epoxy-sealed ceramic cases, with copper leads in teflon insulation. GE varnish was used to glue the sensor to the copper plate. For measurements at temperatures above 4 K, the sample was mounted directly on the surface of the ceramic case. In this way, the sample and the Hall probe can be easily demounted after experiments, immersing the whole plate into acetone and waiting until it is disconnected. We did not observe any negative reaction of acetone on the Hall sensor. At low temperatures (below 1 K) the small heat released in the Hall sensor (the ac current amplitude is 10 mA) prevents cooling of it in a ^{3}He absorption-pomp system. To avoid this heating, the Hall sensor is thermally isolated from the variable-temperature Cu plate by a vacuum space (with a distance between the sample and a surface of the sensor of about 0.5 mm).

The low source resistance of the sensors, 1 Ω, gives low sensitivity to electrical interferences. Usually, the ac lock-in technique is used for measurements of the Hall voltage, which is certainly more accurate than the dc method. It is possible to improve, however, this accuracy and sensitivity by using instruments especially designed for measurements of very low signals, by matching their input impedance to the source impedance, and by operating at one fixed frequency. These features are characteristics of the Linear Research resistance bridge LR-400 [9] used by us. At certain conditions a 10 mG resolution in magnetization measurements can be achieved, similar as in the measurements performed on Hall sensors with a 10^3 larger Hall resistance [1-3,6]. This is accompanied by a higher injection current in our case. According to our estimates, the self-field produced by the Hall probe amounts to less than 10 mG, for an ac-current amplitude of 3 mA.

A very good reproducibility of the characteristics is observed. In fig. 1 the background signal is shown, measured in zero external magnetic field. Usually this signal changes from one experiment to another with a value equivalent to a few Oe, but its temperature dependence remains the same. Therefore, these changes can be easily corrected during the data analysis. Small jumps of the signal, equivalent to a few Oe of the magnetic field are observed during rapid cooling or heating, in particular at low temperatures. These can also be corrected or avoided, by slowing down the temperature changes.

3 Magnetization Measurements

To illustrate the sensitivity of measurements, fig. 2 shows the vertical component of the field-cooled magnetization, measured in Earth's field (in Amsterdam) on a disk-shaped $Bi_2Sr_2CaCu_2O_8$ single-crystalline sample (diameter of 2.4 mm and thickness in the c-direction of 0.2 mm).

The sensitive area of the Hall probe is about 1 mm in diameter and it is comparable to the sample size. The measured stray field is well related to the magnetization of the sample. The linear dependence between this field and the magnetization of the sample could be verified in low fields and at low temperatures by measuring the virgin magnetization curve in the Meissner state. In this case, the measured magnetization, $M_{exp}=H_s-H$, where H_s

is the field registered by the Hall probe and H is the externally applied field, should be found to reflect perfect diamagnetism, i.e. M=-H. For instance, for the $Bi_2Sr_2CaCu_2O_8$ sample for which the results are shown in fig. 4), we found $M_{exp}=-0.39\cdot H$, indicating that in the given geometry the sample does not screen the field perfectly. We use the result of this calibration, $M=M_{exp}/0.39=(H_s-H)/0.39$, at all fields, although its validity for higher fields depends on some assumptions. In particular, it has to be assumed that the flux distribution in the sample is similar to the one in the Meissner state, where the supercurrent flow occurs within a thin surface layer. We are able to argue that this assumption is not unreasonable in many situations. In other cases, when the magnetic induction changes throughout the sample cross-section, the calibration is only approximate (this problem has been discussed widely in literature [11,12]). The relaxation measurements shown in fig. 4 were carried out on a field-cooled sample, at a temperature of 20 K, very well stabilised (better than within ±10 mK). The external field of 6540 Oe was imposed at a temperature above T_c and the sample was field-cooled to T=20 K. In the actual relaxation measurements the process of flux penetration was studied after the applied field was decreased by 550 Oe with respect to its value during cooling. Apart from the high accuracy of the Hall probe, this method requires an excellent stability of the external field. To ensure this, we used a superconducting magnet working in the persistent mode. However, due to the large self-inductance of the magnet, shunted by a small resistance, the response time of the system is long. The sluggish change of the external field causes some problems in the interpretation of the results for times below about 30 s, as explained in details in ref. [10].

Figure 5 presents the magnetization hysteresis of a single-crystalline sample of the heavy-fermion superconductor UPt_3, doped with 11 % of boron. This compound is characterized by a low critical-current density, resulting in a low magnetization. The results shown were obtained in a geometrical configuration in which the effective screening efficiency was 0.475. For a field near to 200 Oe in fig. 5, the magnetic field created by the magnetized sample was only about 2 Oe, i.e. only about 1% of the applied field. Hence, an accurate measurement of the applied field and of the Hall-probe signal were necessary in this case.

4 Summary and Conclusion

The Hall-probe technique is very easy in application and is an effective tool for characterization of superconductors. We argue that it is not the value of the Hall resistance that is essential for achieving a high accuracy in magnetization measurements but the small sensitivity to variations of temperature and the linear response as a function of field. The method described can be applied for measurements of transition temperatures, lower critical fields, magnetization hysteresis and critical-current densities. In particular, it is convenient in monitoring the magnetization relaxation, where a long-time stability and a short response time are important. The sample size should be comparable or larger than the size of the sensitive element (1 mm) but the size in the third direction, parallel to the magnetic field, is not essential for accuracy of the measurements. The method should be particularly suitable for measurements of thin films. In the present version, its use is restricted to investigation of superconductors, since for a proper accuracy the field originating from the magnetized sample should have at least a value of a few percent of the applied field. It is possible to further develop this technique, using flux-transformers and placing the Hall sensor in a cryostat-space that is free of a large magnetic field, or perpendicularly to the field direction. This could increase the accuracy of magnetization-hysteresis measurements at large fields.

References

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Figure Captions

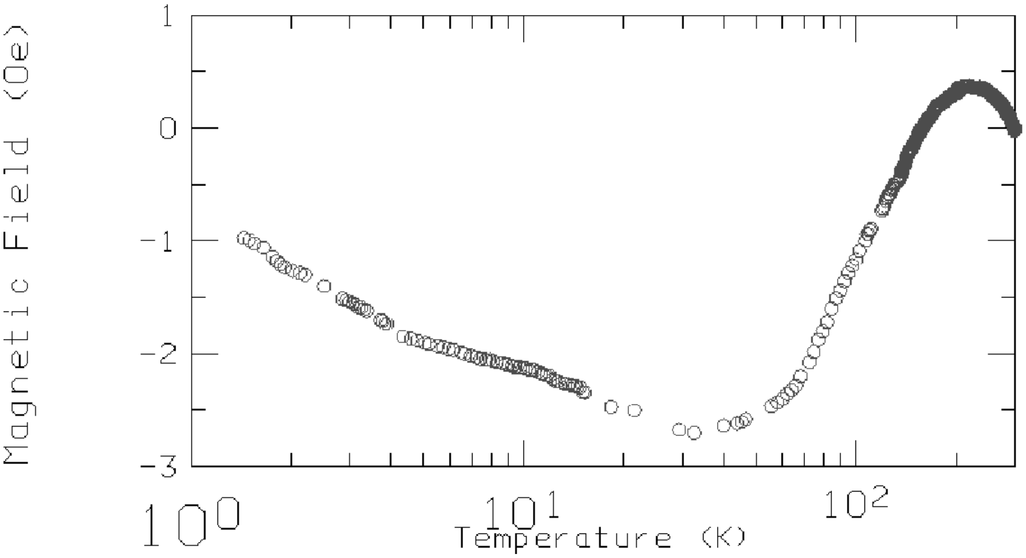

Figure 1: The background signal of the Hall probe in units of a magnetic field equivalent to this signal.

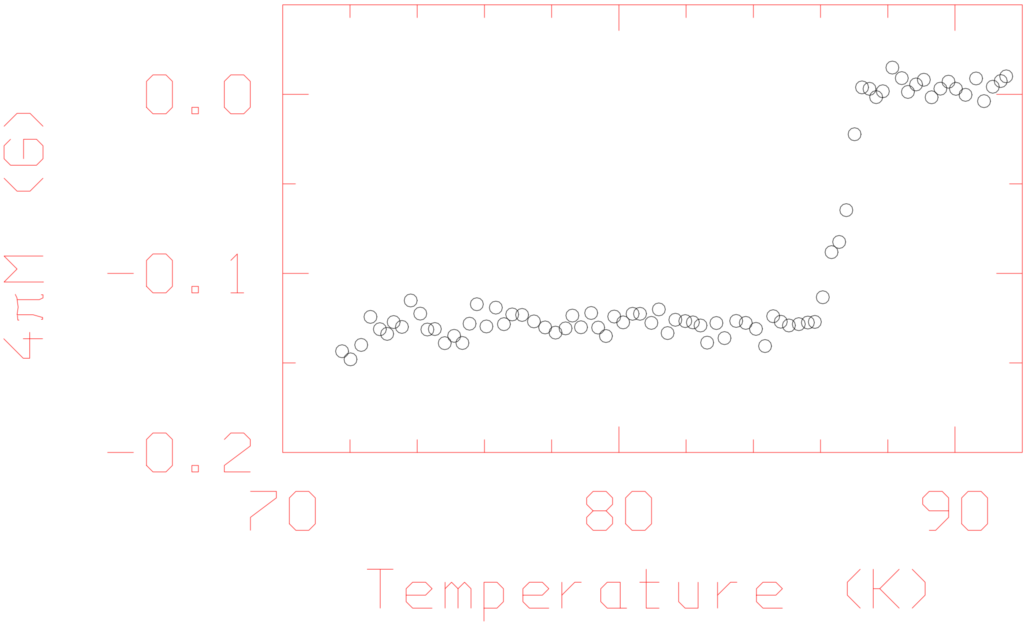

Figure 2: The field-cooled magnetization (Meissner effect) of a $Bi_2Sr_2CaCu_2O_8$ single-crystalline sample, measured in the Earth's field. The vertical component of the magnetization is determined, after subtraction of the background signal of the Hall sensor, which is shown in fig. 1.

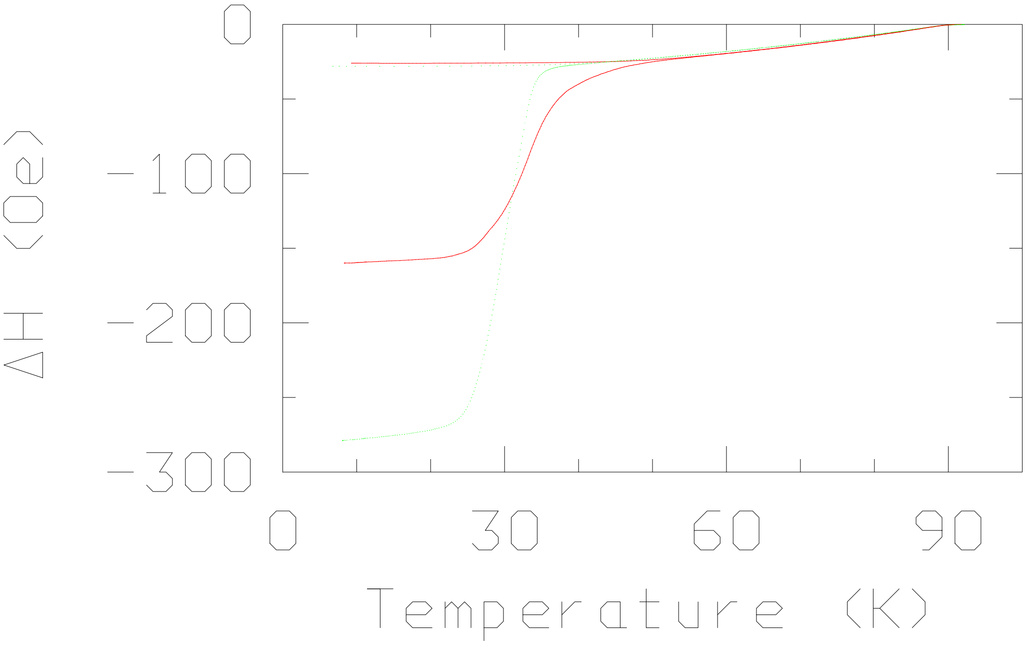

Figure 3: Typical results of the temperature-dependent magnetization (difference between the applied field and the field registered at the sample surface) of a $Bi_2Sr_2CaCu_2O_8$ single crystal, measured in the field cooling (FC) and in the zero-field cooling (ZFC) mode, in field 240 Oe (red lines) and 425 Oe (green lines). The rapid suppression of the ZFC signal at about 40 K is due to the approach of the irreversibility line, where an abrupt decrease of the critical current density occurs.

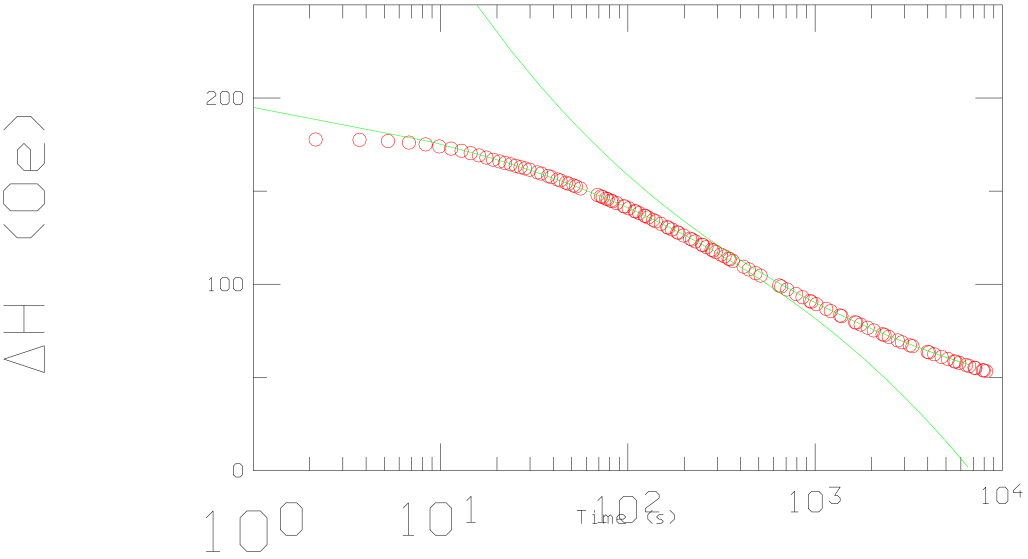

Figure 4: The magnetization relaxation in a $Bi_2Sr_2CaCu_2O_8$ single crystalline sample, with the field parallel to the c-axis. The field ΔH represents the difference between the magnetic field sensed by the Hall probe and the applied field. Measurements were performed on a field-cooled sample (cooled in a field of 6540 Oe from temperatures well above T_c) after a decrease of the field with 550 Oe. The green lines represent the fitting to the short-time and long-time relaxation functions, as described in details in [10].

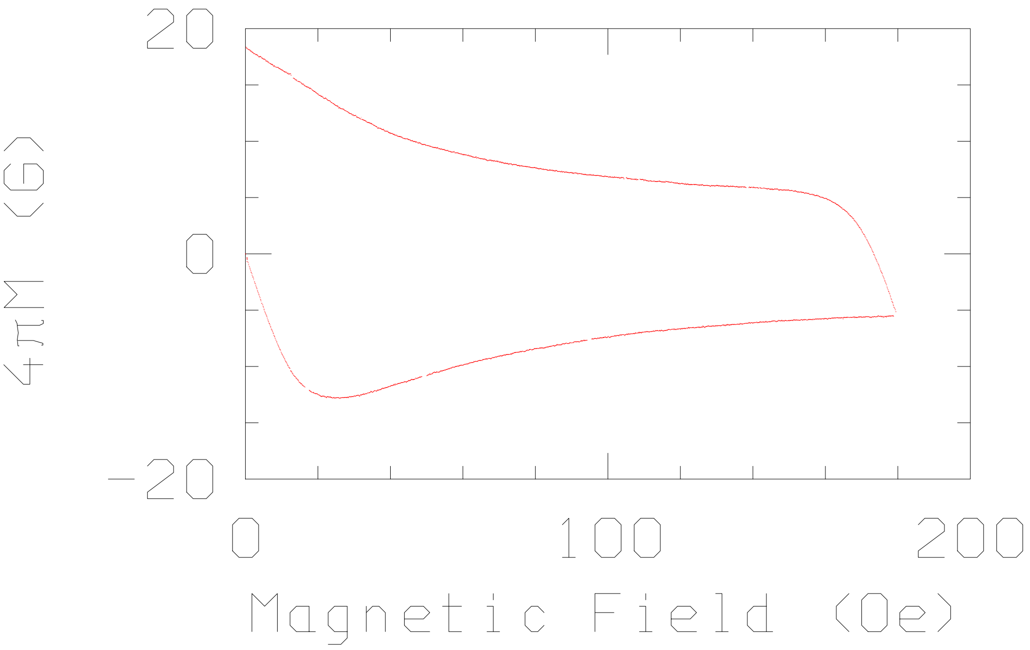

Figure 5: Magnetization hysteresis (the virgin and decreasing branches) of a UPt_3 single-crystalline sample, doped with 11% of boron, measured at T=448 mK. The critical temperature of the sample is 548 mK.