Design and Performance of Remote Controlled ^3He Refrigerator in Hybrid Magnet(Part II. Several Instruments and Techniques Developed in HFLSM)

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Design and Performance of Remote Controlled ^3He Refrigerator in Hybrid Magnet*

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Synopsis

A remote controlled ^3He refrigerator for use in a hybrid magnet is described. Temperatures between 1.3 K and 0.43 K can be remote controlled by ^3He vapor pressure within the temperature drift of 0.01 K in magnetic fields up to 23 T. Using this refrigerator, several kinds of physical measurements, such as electrical resistance, capacitance and ultrasonic attenuation, have been carried out without being disturbed by noise peculiar to the hybrid magnet system. Additionally, magnetic field dependence of capacitance thermometers at liquid ^3He temperatures is discussed.

I. Introduction

Some kinds of phase transitions have been observed or expected to be observed in high magnetic fields at very low temperatures, for example an electronic phase transition in graphite[1], an excitonic phase transition in bismuth[2], a field-induced superconducting transition in $Eu_{1-x}Sn_xMo_6S_8$[3]. Some electronic properties of matter are expressed as a function of $\mu_B H/k_B T$, meaning that increase of magnetic field H corresponds to decrease of temperature T. Thus a high magnetic field system combined with low temperatures is very effective in studying these physical properties of matter. Stationary high magnetic fields are produced with a hybrid magnet consisting of a superconducting magnet and a water-cooled

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normal-conducting one, as described in Part I of this special issue. On the other hand, stationary low temperatures are simply obtained with a ^3He refrigerator. However, there are several problems to be taken into consideration in combining the ^3He refrigerator with the hybrid magnet, as follows. (1) Leakage magnetic field due to the big superconducting magnet is so large that the gas handling system loaded with motors should be located away from the ^3He cryostat. Accordingly, a pumping system of large capacity is necessary to make up for the deficiency due to the long pumping lines. (2) For safety, operators cannot enter the room for the hybrid magnet in operation. Therefore, the ^3He refrigerator has to be remote controlled in the neighboring room. (3) Because of the limitation of the operation time of the hybrid magnet, low temperatures should be kept under comparatively fast sweep of the magnetic field. (4) For precise physical measurements, electromagnetic and mechanical noise peculiar to the hybrid magnet system must be reduced as much as possible.

In this paper, we report the design and performance of the remote controlled ^3He refrigerator for the hybrid magnet of HFLSM of Tohoku University. As regards the temperature measurement in high magnetic fields, magnetic field dependence of capacitance thermometers at liquid ^3He temperatures is also discussed.

II. Apparatus

1. ^3He cryostat

We have taken a method that samples for measurements are directly immersed in liquid ^3He, so as to be kept in good thermal contact with the cryogen and to minimize heating due to the fast sweep of the magnetic field in answer to the problem (3) in Chap. I. This method is also suitable for introducing a sample orienting device with respect to the magnetic field. Figure 1 shows a schematic drawing of the ^3He cryostat. An extended drawing of the sample holder part is shown in Fig. 2. This cryostat can be operated for a long time in a ^3He circulation mode. Liquid ^3He is precooled in the copper spiral tube immersed in pumped liquid ^4He in the 1 K pot, followed by introduction into the sample space from the bottom of the liquid ^3He bath. The circulation rate of ^3He is adjusted with the needle valve. Of course, this cryostat can be operated in a single cycle mode. The 1 K pot, whose volume is 0.3 ℓ, is continuously supplied with liquid ^4He through the flow impedance and/or supplied through the needle valve. The effective volume of the liquid ^4He bath is 12 ℓ, and its

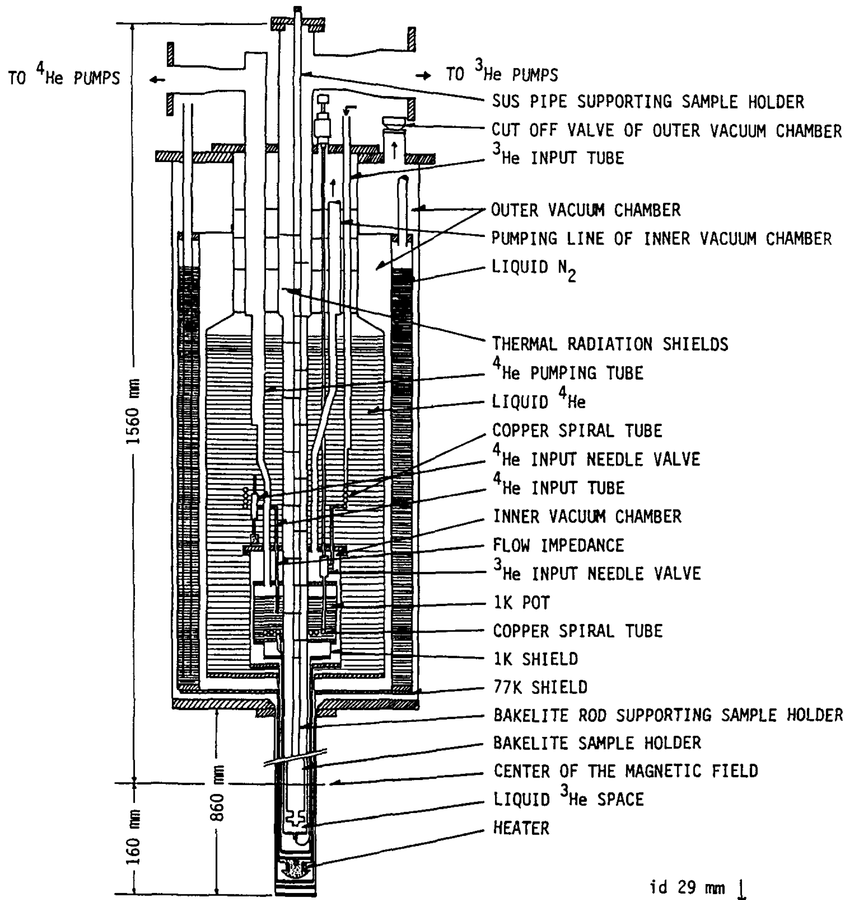

Fig. 1.
Schematic drawing of the ^{3}He cryostat.

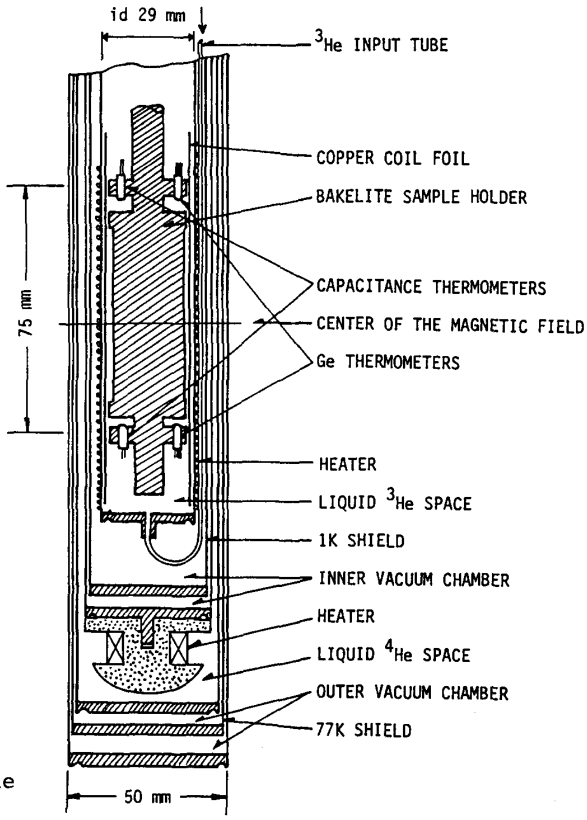

Fig. 2.
Cross-sectional view of the sample holder part of the ^{3}He cryostat.

evaporation rate is 0.65 ℓ/hr. The effective volume of the liquid N_2 bath is 8 ℓ. As shown in Fig. 2, the lower part of the cryostat is jammed with many tubes within its outer diameter of 50 mm; that is, the outer vacuum chamber, the 77 K shield, the liquid ^{4}He bath, the inner vacuum chamber, the 1 K shield and the liquid ^{3}He bath. The interval between neighboring tubes is minimized to get large sample space. These tubes are kept out of contact with each other, by using nylon string as a spacer. Consequently, the inner diameter of the sample space is as large as 29 mm. However, the outer diameter of the sample holder must be smaller than 27 mm, on account of some miss design.

The upper part of the support of the sample holder is made of a stainless steel tube with 16 copper pieces for thermal radiation shields. The lower part below the 1 K pot and the sample holder are made of non-metallic bakelite to prevent eddy current heating due to the fast sweep of the magnetic field. The sample holder is enveloped with copper coil foil to reduce the temperature difference between the upper and lower parts of the sample holder. Two Germanium thermometers are set at the uppermost and lowest parts of the sample holder, respectively. Capacitance thermometers are also set to be used in magnetic fields. Manganin wire is wound non-inductively at the outside of the liquid ^{3}He bath as a heater in order to recover ^{3}He or to control the temperature in the sample space.

2. Gas handling system and temperature control

Figure 3 shows the gas handling system of the ^{3}He refrigerator. As mentioned as the problem (1) in Chap. I, the ^{3}He and ^{4}He pumping lines are as long as about 7 m, so that the leakage magnetic field at the position where the gas handling system is located is kept less than 0.01 T in operation of the hybrid magnet. In order to make up for the deficiency of the pumping capacity, we have used a sealed rotary vacuum pump with pumping speed of 700 ℓ/min and moreover a sealed mechanical booster pump with fast pumping speed of 500 m^3/hr for pumping out ^{3}He. A rotary vacuum pump with pumping speed of 760 ℓ/min has been used for pumping ^{4}He out of the 1 K pot. The diameters of the both pumping lines are as big as 2 inches. Both trap and purifier cooled at the liquid N_2 temperature, containing molecular sieve and active carbon respectively, have been used for purification of ^{3}He so as to prevent the ^{3}He input tube from being blocked in the circulation mode. Many pneumatic bellow valves, which can be remote controlled in the neighboring room, have been used in answer to the problem (2) in Chap. I.

In general, temperature control in magnetic fields is made by two different methods; one is a ^{3}He vapor pressure control method, and the other is a negative feedback method using a heater and a capacitance thermometer unaffected by magnetic field. The latter is simple and often used in ^{3}He cryostats of the adiabatic type, but in our case the former is considered to be better than the latter. In the lower pressure range between 10^{-3} Torr and 10 Torr, we have used a capacitance manometer (model CG-500C, UNITEC) and a pressure transducer of the capacitance type (type 120, FPI). The difference between output voltage of the manometer and set voltage is transformed into pulse current to operate a control valve (type 253A-2-2-2, MKS INSTRUMENTS) adjusting the flow conductance of the

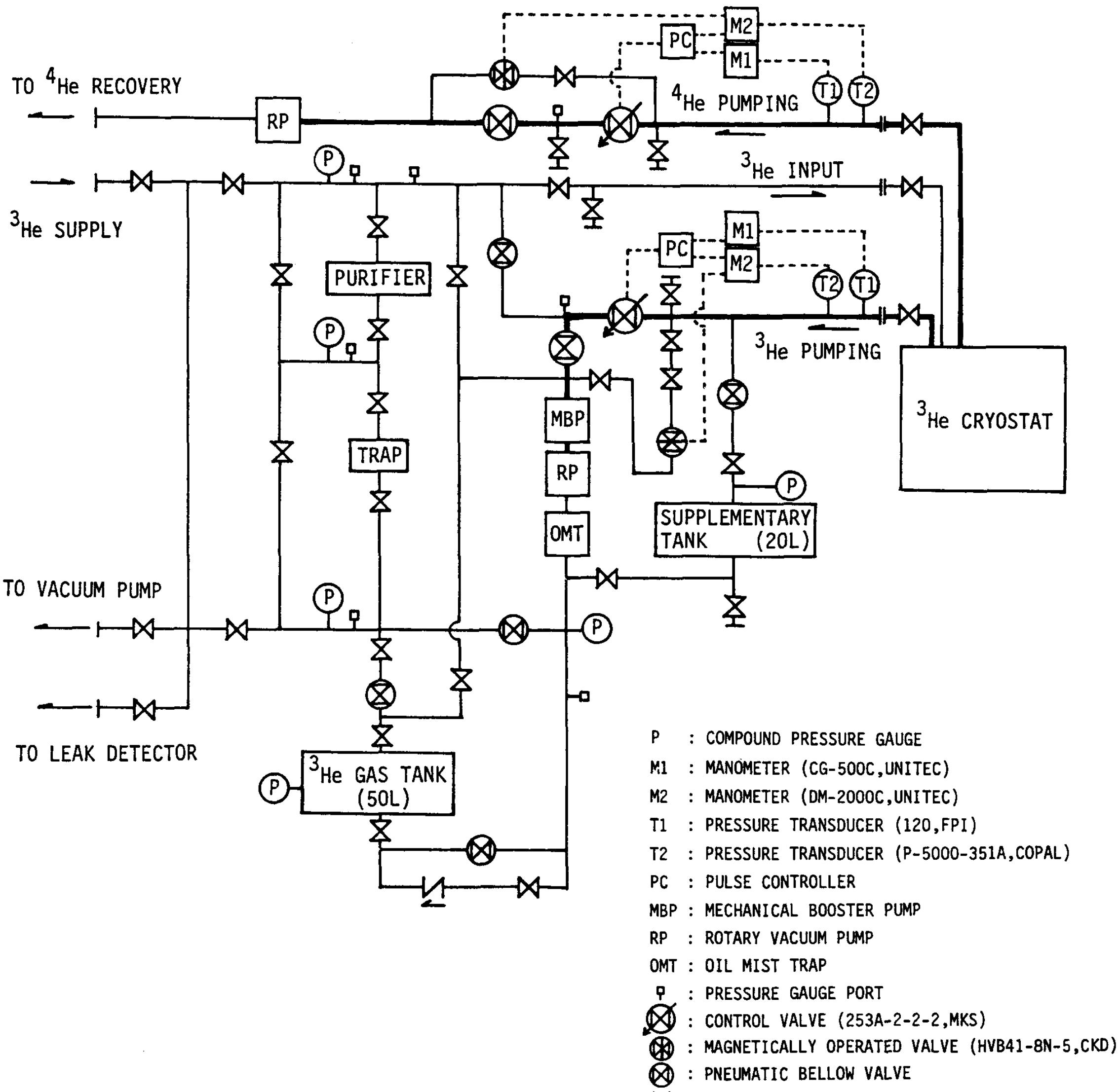

Fig. 3.
Gas handling system for the ^{3}He refrigerator.

^{3}He pumping line. In the higher pressure range between 10 Torr and 760 Torr, we have used a digital manometer (model DM-2000C, UNITEC) and another pressure transducer of the semiconductor type (type P-5000-351A, COPAL ELECTRONICS). According to the difference between output voltage of the manometer and set voltage, the bypass of the ^{3}He pumping line is opened or shut with a magnetically operated valve (type HVB41-8N-5, CKD). This apparatus for temperature control has been also applied to the ^{4}He pumping line leading to the 1 K pot.

III. Operation and performance

After the ^{3}He cryostat was cooled down to 4.2 K, the 1 K pot was pumped out down to 1.3 K in 20 minutes. Then ^{3}He gas of 20 ℓ was condensed into the sample space in less than an hour. After starting to pump out ^{3}He, the temperature of the sample space reached 0.5 K in 30 minutes. So far, the lowest temperature attained is 0.43 K.

Temperature drift in the sample space under temperature control is shown in Fig. 4. At low temperatures below 0.7 K, temperatures were controlled with the control valve in the ^{3}He pumping line. In the temperature range between 0.7 K and 1.3 K, they were controlled with the magnetically operated valve in the bypass of the ^{3}He pumping line. In both temperature ranges, temperatures were fairly well controlled within the temperature drift of 0.01 K for 2 hours under the magnetic field sweep of $H = 0\ T \rightarrow 23\ T \rightarrow 0\ T$. Additionally, temperature control at high temperatures above 1.3 K was tried. Firstly, the temperature control of the 1 K pot was made by using the similar apparatus to that for the ^{3}He pumping line, leaving the sample space in the gaseous ^{3}He atmosphere. The temperature of the 1 K pot was well controlled, but the temperature of the sample space was not stable. This seems due to long distance between the 1 K pot and the sample space. Next, the negative feedback method was tried using the heater set at the outside of the liquid ^{3}He bath and a capacitance thermometer (type CS-400, LAKE SHORE CRYOTRONICS) in the gaseous ^{3}He atmosphere. The temperature of the sample space settled down, but the temperature drift was still larger than at low temperatures below 1.3 K, as shown in Fig. 4. The temperature control at high temperatures above 1.3 K is a problem remained to be solved in future.

Figure 5 shows the temperature difference between the upper and lower parts of the sample holder, separated by 75 mm. In the single cycle mode at low temperatures below 1.3 K, the temperature of the upper part of the sample holder was lower than that of the lower

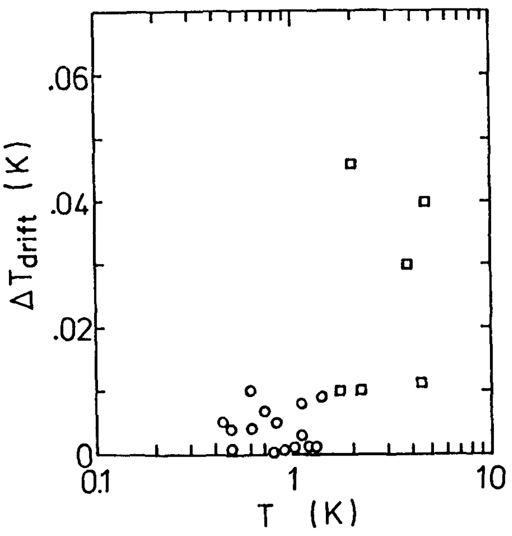

Fig. 4.
Temperature drift in the sample space under temperature control. Open circles were obtained by the ^{3}He vapor pressure control method. Squares were obtained by the negative feedback method using the heater and a capacitance thermometer (type CS-400, LAKE SHORE CRYOTRONICS) in the gaseous ^{3}He atmosphere.

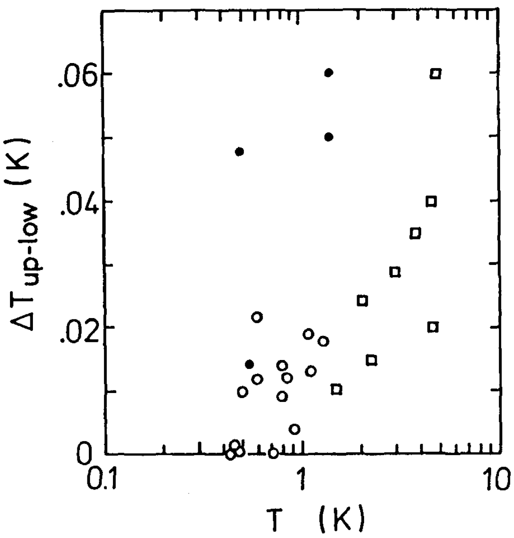

Fig. 5.
Temperature difference between the upper and lower parts of the sample holder. Open circles were obtained by the ^{3}He vapor pressure control method in the single cycle mode. Solid circles were obtained by the same method in the circulation mode. Squares were obtained by the negative feedback method using the heater and a capacitance thermometer (type CS-400, LAKE SHORE CRYOTRONICS) in the gaseous ^{3}He atmosphere.

part. This seems due to the fact that the thermal conductivity of liquid ^{3}He is not large and that liquid ^{3}He cooled in the neighborhood of the liquid surface was not circulated by convection on account of the jam in the sample space. The temperature difference was within 0.02 K, as shown by the open circles in Fig. 5. We have used copper coil foil, containing about 1000 copper wires with the diameter of 0.06 mm, but we should use more to reduce the temperature difference. In the circulation mode, the temperature difference was larger than in the single cycle mode, as shown by the solid circles in Fig. 5. This seems due to comparatively warm liquid ^{3}He supplied from the bottom of the liquid ^{3}He bath in the circulation mode. So, all physical measurements have been made in the single cycle mode, where temperatures can be well controlled for not less than 2 hours necessary for the magnetic field sweep. The temperature difference at high temperatures above 1.3 K was almost the same order of the temperature drift shown in Fig. 4.

With this ^{3}He cryostat, several kinds of physical measurements, such as electrical resistivity, capacitance and ultrasonic attenuation, have been carried out in the hybrid magnet. No counterplan has been devised in answer to the problem (4) in Chap. I, but measurements have not been so disturbed by noise peculiar to the hybrid magnet system. As mentioned in Chap. II, the sample space is enveloped with 6 tubes, including 2 copper tubes for the 77 K and 1 K thermal radiation shields. Consequently these tubes are considered to be also useful to shield the electromagnetic noise and the ripple of the magnetic field.

IV. Magnetic field dependence of capacitance thermometers

As regards the temperature measurement in high magnetic fields, capacitance thermometers are widely used as sensors unaffected by magnetic field, besides a method using the relation between saturated vapor pressure of liquid ^{3}He or ^{4}He and temperature. We investigated the magnetic field dependence of the capacitance of some commercial capacitance thermometers at liquid ^{3}He temperatures, though they are guaranteed to be independent of magnetic field at temperatures above

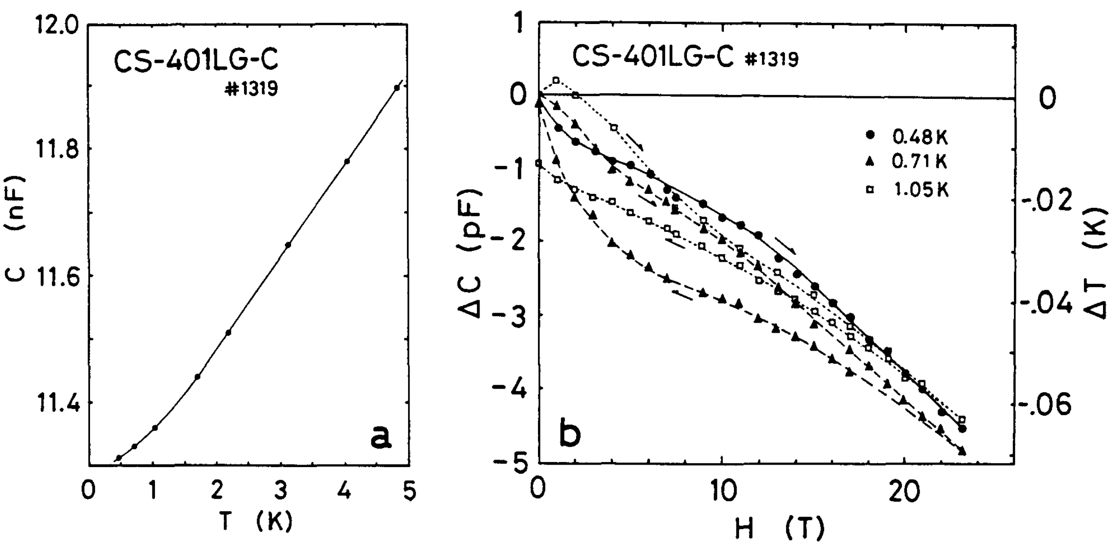

Fig. 6.

(a) Temperature dependence of the capacitance of a recent capacitance thermometer (type CS-401LG-C, LAKE SHORE CRYOTRONICS) in the absence of magnetic field.

(b) Magnetic field dependence of the capacitance of this thermometer. The right scale shows the temperature change corresponding to the capacitance change.

1.5 K. Capacitance measurements were made using a precision capacitance measurement system (model 1621, GENERAL RADIO) with a frequency of 3.33 kHz and an excitation voltage of 0.1 V. In fact, an old capacitance thermometer (type CS-400, LAKE SHORE CRYOTRONICS) shows no magnetic field dependence within the experimental accouracy at low temperatures down to 0.5 K, but a recent type (type CS-401LG-C, LAKE SHORE CRYOTRONICS) has rather strong magnetic field dependence, as shown in Fig. 6(b). The capacitance decreases with increasing magnetic field strength. Its change in magnetic fields up to 23 T is estimated to correspond to the temperature decrease of about 0.07 K, from the temperature dependence of the capacitance in the absence of magnetic field as shown in Fig. 6(a). It is clear that this capacitance thermometer cannot be used as a thermometer in magnetic fields at least in the low temperature region below 1 K. In principle, capacitance should be independent of magnetic field. The origin of this magnetic field dependence is not clear. Hysteresis with respect to the magnetic field sweep is also observed as shown by the arrows in Fig. 6(b). The origin is also not clarified. At any rate, we desire capacitance thermometers unaffected by magnetic field, such as an old type (type CS-400, LAKE SHORE CRYOTRONICS).

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