Liquid Level Indicator for Metal Dewar

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A simple liquid level indicator for a metal dewar has been constructed with the use of superconducting elemental Ta and Nb-Ti alloy as well as carbon resistor. Electrical and superconducting properties, repeatability, sensitivity, and a practical operation of the indicator have been investigated at 78°K and 4.2°K. The hysteresis or instability effect of the resistance for Nb-Ti wire was found at the magnetic field of 17 kG. Evaporation rate of liquid helium can be estimated in the dewar with a few indicators.

1 Introduction

A number of devices sensitive to the level of liquid nitrogen or helium in a dewar have been reported.[1] The principles used in these devices are as follows ; (1) change in resistance accompanying the superconducting transition, (2) flotation of material having bulk density lower than that of the liquid, (3) difference in mass flow rate through an impedance of a liquid as compared with its vapor, (4) difference in dielectric constants between a liquid and its vapor, (5) difference in heat transfer rate between a solid and a liquid and between a solid and a vapor, and (6) difference in thermal oscillation characteristics of a tube depending upon whether it is immersed in the liquid or in the vapor.

In our laboratory, a metal dewar has been used for the experimental study of electron spin resonance and electron nuclear double resonence in the V-band region (6 mm wavelength) at low temperatures.[9] To detect the level of a "liquid" in the dewar, we have constructed a simple and compact level indicator with a superconductor and solid carbon resistor. In the present paper, some of the experimental results obtained from these indicators about the sensitivity, repeatability, and other electrical properties will be shown. Finally actual operation of the liquid level indicators will be presented with a sensing element of Ta and carbon in our metal dewar.

2 Experimental

2・1 Sample

A cryostat for magnetic resonance experiment usually accomodates a waveguide, cavity, coupling and tuning rods, etc. The upper part of the cryostat is connected to a microwave circuit such as a magic tee. The lower part of it is put between the pole pieces of an electromagnet. The level indicators should, therefore, be as small as

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possible in the cryostat. At the same time, the whole assemble is required to be simple. We then decided the indicators to be of such a type that the change in the electrical resistance may be detected. There are two kinds of sensing elements, i.e., superconductor and carbon resistor.

The superconductors used are an elemental tantalum Ta of the type I and Nb-Ti alloy*) of the type II superconductor. The Ta foil of thickness $20\,\mu$ was cut into a ribbon shape of width 0.5—1 mm. That ribbon was then wound helically around a small bakelite bobbin, as shown in Fig. 1, and fixed by "araldite." Soldering Ta with a copper lead by a commercial solder is usually impossible. Prior to soldering, the Ta ribbon and the lead of 1 mm diamter were both bound firmly by winding a copper wire of 0.2 mm diameter around them. Then the bound parts were soldered by In–Sn or Pb–Sn solder. Nevertheless the electrical contacts broke down sometimes under experiment. We have also attempted to use the "spot-welding" technique, i.e., a heavy current is supplied instantaneously to the thin foil and the lead which are compressed by the electrodes. Good contact was attained, though this method also required sufficient care, because the Ta foil is very thin. On the other hand, Nb-Ti alloy wire is a copper-coated superconductor and no difficulty in making the sensor was encountered. The coated copper was first etched by a dilute HCl solution except the terminal parts to obtain the bulk Nb-Ti wire, and then the wire was wound in the same manner as shown in Fig. 1. The resistances at room temperature were in the range 3–5Ω for both superconductor-sensors.

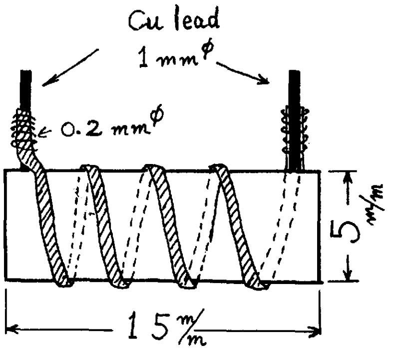

Fig. 1. Superconducting sensing element.

Resistive sensing elements under test are Allen-Bradley resistor (nominal 56Ω at room temperature), Speer resistor (100Ω), and domestic resistors (33 and 56Ω). In the case of the domestic resistors whose bulk volume is large, the outer shell (original diameter 5 mm) was removed by an abrasive paper to have good thermal contact and sensitivity. The other resistors were used as they are.

2 • 2 Measurement

Two methods were used for the electrical measurements. First, the so-called Wheatstone bridge method was used for the detection of the level indicator, as shown in Fig. 2. The one arm R_x is connected to a sensing element, a superconductor or resistor, and the variable arm R is a standard decade resistor (0.1~1000Ω). For a null detection, a dc amplifier is connected. In the case of the level indication use, a dc ammeter of 50 μA full-scale is used instead, for simplicity, to read the deflection. Second, the conventional dc potentiometric method was used for the precise measurement such as the

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*) The model SUPER-SW-25 superconducting wire, Nb-50Ti, produced by Vacuum Metallugical Co., Ltd. The nomial critical field 122 kG, critical temperature ~10°K, outer diameter 0.35 mm, core diameter 0.25 mm, and insulation Formvar.

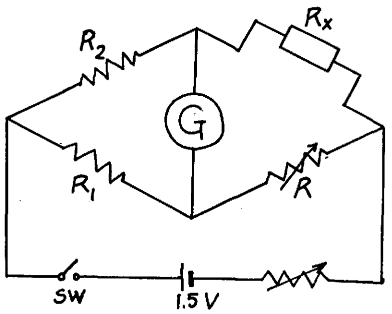

Fig. 2. Wheatstone bridge for the detection of the change in the resistance.

magnetic field dependence of the resistance. However, no current was reversed at each measurement, because we were mainly concerned with the change in the resistance with and without the magnetic field.

The electromagnet used in our laboratory is of low impedance type, 50 mm in the pole gap and max. 22 kG in the field strength. As described below, the field strength less than about 100 G is neither controlled precisely nor read accurately, because this magnet is manufactured for higher field use up to 22 kG. And the residual magnetic field of the pole piece may be comparable order of magnitude to the field to destroy the superconducting state of Ta at 4.2°K, so another magnet will be required for the detailed study of the determination of the critical field less than 100 G. No measurement was made between 78° and 4.2°K of the temperature dependence of the resistance.

3 Experimental Results and Discussion

3・1 Electrical Properties

Ta :

The temperature dependence of the resistance of Ta sensor is shown in Fig. 3, in the range 300°—78°K and at 4.2°K, where the dotted line shows the expected behavior. According to the usual transport theory of metals, the resistivity ρ depends on temperature T as $\rho \propto T$ for $T \gg \theta$, and $\rho \propto T^5$ for $T \ll \theta$, where θ is the Debye temperature. With $\theta = 245°K$[2] for Ta, the normalized resistivity ρ/ρ_θ vs. normalized temperature T/θ from the observed values, known as the Bloch-Grüneisen relation,[3] is also shown in the figure. Here in the expression of ($\rho/\rho_\theta =$) R/R_θ, R and R_θ are the resistance of the sensor at temperature T and θ, respectively. Within the experimental errors, the curve fits well with the data accomplished so far.[3]

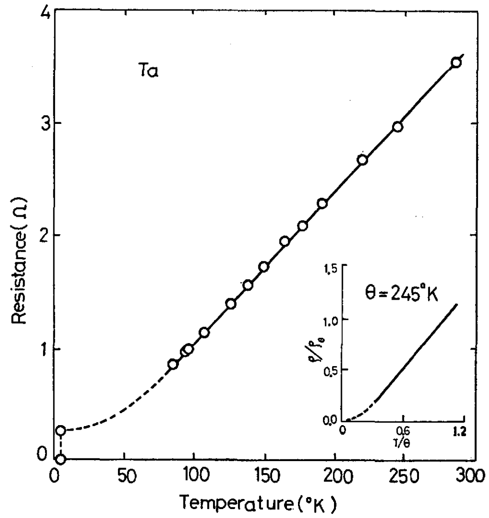

Fig. 3. Temperature dependence of the resistance of the Ta sensor.
An expected behavior is indicated by a dotted line. The Grüneisen-Bloch relation is also shown with the Debye temperature $\theta = 250°K$.[2]

To study the transition from the superconducting to the normal state the magnetic field dependence of the resistance has been measured at 4.2°K. From the well-known law of the critical field H_c vs. temperature T, $H_c = H_0 [1 - (T/T_c)^2]$, the critical field at 4.2°K is estimated to be about 80 G for Ta, with $T_c = 4.4°K$ and $H_0 = 800$ G. The sharpness of the transition, however, depends not only on the purity and perfection of the sample but also on the relation between the direction of the field and that of the sample.4) If the field is applied parallel to the axis of a cylindrical specimen, the transition from the superconducting to the normal state is quite sharp. If the field is perpendicular to the sample, the transition is much more gradual. In fact, for an arbitrary direction of the field the critical field at which the transition occurs may be estimated by solving the phenomenological London equation, taking into account of the boundary conditions. In the present case, where the sample is formed by winding the metal helically around the bakelite bobbin, the situation seems more complicated.

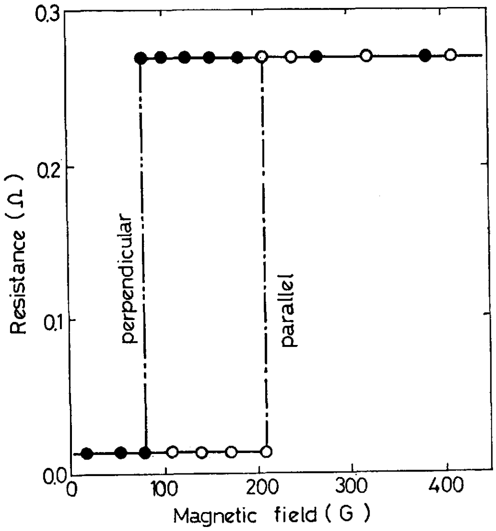

Fig. 4. The superconducting transition for the Ta sensor at 4.2°K.

Fig. 4 shows the experimental result made at 4.2°K for two directions of the bobbin, around which Ta ribbon is wound helically, one being parallel to the magnetic field and the other perpendicular. For the perpendicular direction, the resistance commences at such a field strength that the power of the electromagnet is switched on and no current controll may be made. The precise field strength is not known, as noted previously. In the figure, the estimated value of 80 G for Ta is adoped. If the field is applied parallel to the bobbin, the transition may occur at a much higher field, approximately 200 G. Once the superconducting state breaks down to the normal state, then the resistance does not increase for both case with increasing the field up to 22 kG attained. This shows no magnetoresistance effect in the present experimental conditions. To have an appreciable effect, it is necessary to increase the field much higher, of the order of magnitude of 10^2—10^3 kG, according to the reduced Kohler diagram.5) From Fig. 4 it is noted that the apparent critical field differs depending on the relative direction of the bobbin to the magnetic field.

Nb-Ti Alloy :

The temperature dependence of the resistivity of an alloy is in general not so simple as a pure metal. In Fig. 5, the

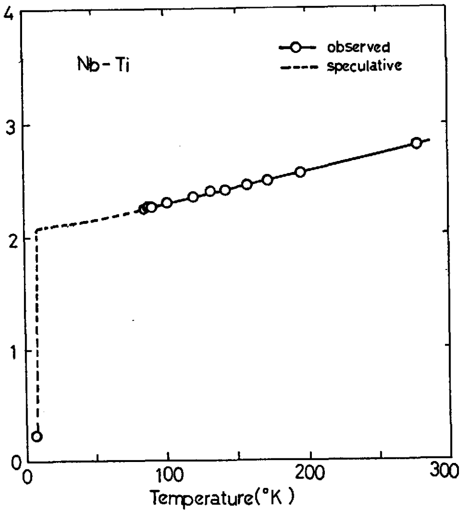

Fig. 5 Temperature dependence of the resistance of Nb-Ti sensor

observed curve for Nb-Ti alloy is shown and bellow 78°K the dotted curve is only speculative. Detailed measurement is required for these temperature ranges. If we apply the Bloch-Grüneisen relation to the linear portion of the curve, the Debye temperature is estimated to be higher than 300°K at least, though accurate calculation is not shown here.

The field dependence at 4.2°K of the resistance is given in Fig. 6 for the both directions of the bobbin, parallel and perpendicular to the field. In this figure, and even in Fig. 3, the resistance is not exactly zero at the superconducting state. This is simply due to the fact that in our experiment no current in the potentiometric circuit is reversed and the resisance is evaluated by the ratio of the potential drop of the sample to that of a standard resistor in one direction of the current, so that there may be spurious potential drops arising from the contacts at several points in the whole circuit. Now, for the parallel arrangement, the superconducting state persists up to 22 KG, while for the perpendicular case, the resistance starts to increase a little at around 5 kG and at about 17 kG it increases abruptly. For the latter case, a hysteresis effect has been found, i.e., the resistance with increasing the field is different from that with decreasing the field. It should also be noted that the transition from the superconducting to the normal state is not sharp but gradual, and that the apparent critical fields are different for both directions just like the case of Ta.

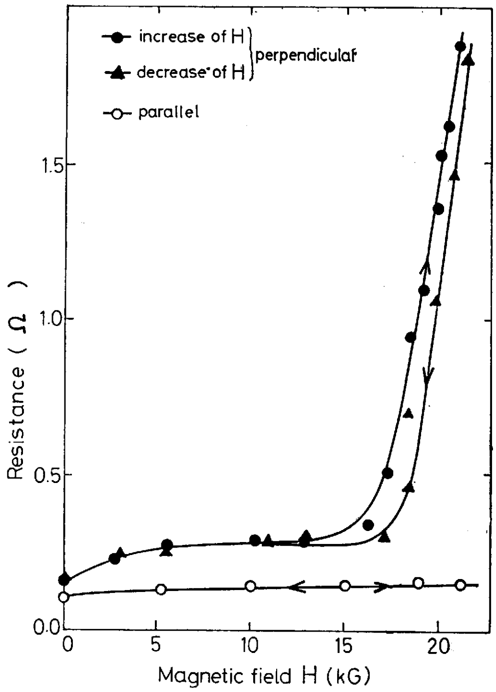

Fig. 6. Magnetic field dependence of the resistance for Nb-Ti sensor at 4.2°K.

Since the study of type II superconductors, a number of experimental evidences have been revealed of the phenomena such as "hysteresis" or "irreversible" effect and flux jump instabilities.[6] In the so-called "hard" superconductors that contain lattice defects and inhomogeneities, mostly alloys and intermetallic compounds, flux penetrates gradually over a wide range of the magnetic fields and in reversing the field most of the flux remains frozen in the specimen. It is also known that the superconducting state is unstable for the fields less than 20 kG for Nb-Ti alloy and 50 kG for Nb_3Sn due to the flux jump. This is the case for the present result, as shown in Fig. 6.

Carbon Resistor :

The resistance vs. temperature curves were determined by measuring the resistance at three fixed points, 300°, 78°, and 4.2°K and by applying a semi-empirical formula,

$$\log R + C/\log R = A + B/T,$$

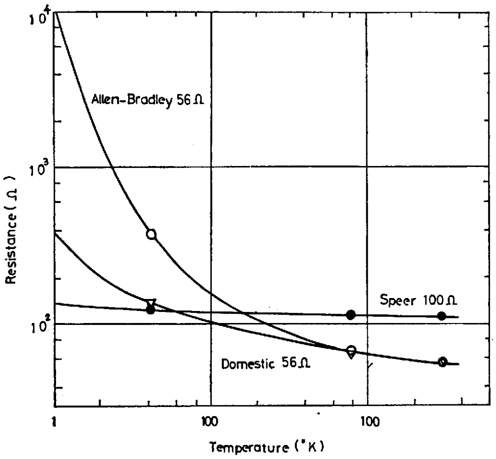

Fig. 7. Temperature dependence of the resistance for three resistors.

where A, B, and C are constants.[10] In Fig. 7 the calculated curves are shown for our samples. Carbon resistance increases with decreasing temperature. It is seen that Speer and domestic resistors are more sensitive than Allen-Bradley resistor bellow 4.2°K as Gonda's data.[7] It appears unlikely that carbon is a semiconductor in the strict sense. For an approximate expression the resistance is given in a form $R = R_0 \exp(\varepsilon/kT)$ at low temperatures, where the constant ε is like "activation-energy". From the curve the constants ε are found to be 1.1×10^{-4} and 3.7×10^{-4} eV for the domestic and Allen-Bradley resistor, respectively. Difference in the constant ε may indicate that the kind of impurity involved in carbon is different among the makers.

Repeatability or reproducibility of the resistance at liquid nitrogen temperature is given in Fig. 8, where the heat cycle was applied between 78° and 300°K. After 10 runs of the cycle, the samples were left in air for one day and then the cycle was again applied. Domestic 56Ω resistor changes by 0.38Ω corresponding to 3.5°, where $dR/dT = -0.11\Omega\ \mathrm{deg}^{-1}$. Allen-Bradley 56Ω resistor proves most excellent of all.

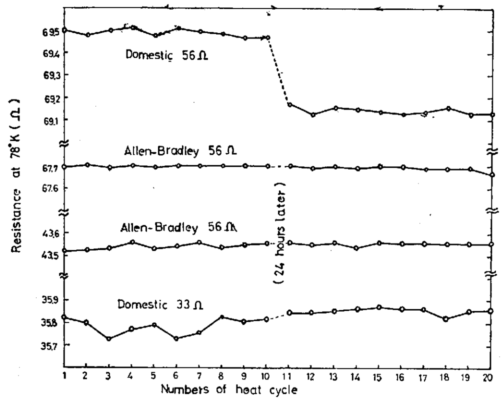

Fig. 8. Repeatability of the resistance for a few carbon resistors through the heat cycle between 300° and 78°K.

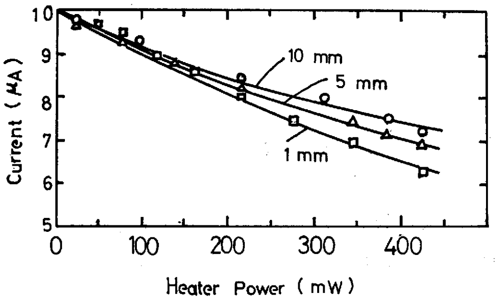

Fig. 9. Sensitivity for Allen-Bradley 56Ω resistor at 78°K. Distance of the sensor from the liquid N_2 level is taken as a parameter.

Sensitivity as a level indicator can be raised by winding a heater around the resistor. At 4.2°K a few mill-watts power is reported to be effective.[8] We have made the same experiment at 78°K, in which a nichrome heater of 240Ω was wound around Allen-Bradley 56Ω resistor. Current of an ammeter in the Wheatstone bridge, as in Fig. 2, was observed when heater current pulse of 5 sec duration was applied to the heater. Fig. 9 shows the change in current as a function of a heater power dissipated, where as a parameter we take the distance between the sensor and liquid level. At 78°K sensitivity was found to be raised for heater power more than 150 mW in the present experimental accuracy. The heater, however, was not used for actual indicator use for simplicity.

Dependence of the resistance on the magnetic field has been measured at 78° and 4.2°K for a few samples, where the field is parallel or perpendicular to the sample current. Magnetoresistance coefficient $\beta=[R(H)-R(O)]/R(O)$ is indicated as a function of the field strength in Fig. 10 for both direction. The values of β at 78°K are of the order of magnitude of $1\text{-}2\times10^{-3}$ at 22kG, though not shown. No remarkable difference is seen between the transverse and longitudinal arrangement. Domestic resistor shows negative magnetoresistance, while others show rather positive effect. Increase in the resistance for Allen-Bradley resistor is about 0.5% at 22 kG, which corresponds to temperature difference of 0.02°. Even such a change in the resistance may not affect the sensitivity of a level indicator.

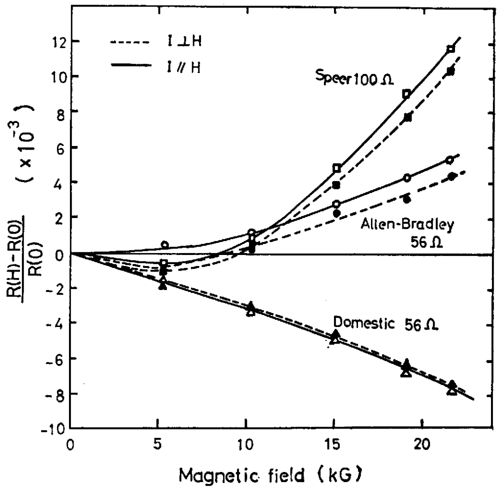

Fig. 10. Magnetoresistance effects at 4.2°K for solid carbon resistors.

3 • 2 Operation of Level Indicator

In the preceding section we have described some of the electrical and superconductive properties for Ta, Nb-Ti alloy, and carbon resistors. Finally actural operation of the liquid level indicator will be shown with a sensing element of Ta and carbon resistor in our metal dewar. As mentioned previously, the measuring system is designed to be simple, i.e., change in the resistance of the sensors is detected by the Wheatstone bridge. Positions of the sensors in the dewar are shown in Fig. 11, where Ta and resistor sensors are placed alternately, denoted in the figure by Ta-1, Ta-2, C-1, and C-2, respectively. Because the positions of Ta sensors are far apart from the center of the pole piece, the superconducting state does not break down even if the magnetic field is applied to make ESR and ENDOR measurement, where in the V-band region the field strength is up to about 16 kG.9)

Fig. 12 (a) and (b) show one example of operation. Arrows indicate the supply of liquid helium through a syphone. When the sensors are immersed in liquid, the read of the deflection of the bridge becomes constant with time. At 4.2°K the absolute value of the resistance is not always repeatable, but it remains constant with time, and thus detection of the level is possible. From the time difference between C-1 and C-2, for instance, we can estimate the rate of evaporation of liquid helium, if the volume of the liquid reservoir is known. From Fig. 12 (b) the time difference is about 11 min, and heat of vaporization of liquid helium is known to be 5.4 cal/g, so the power

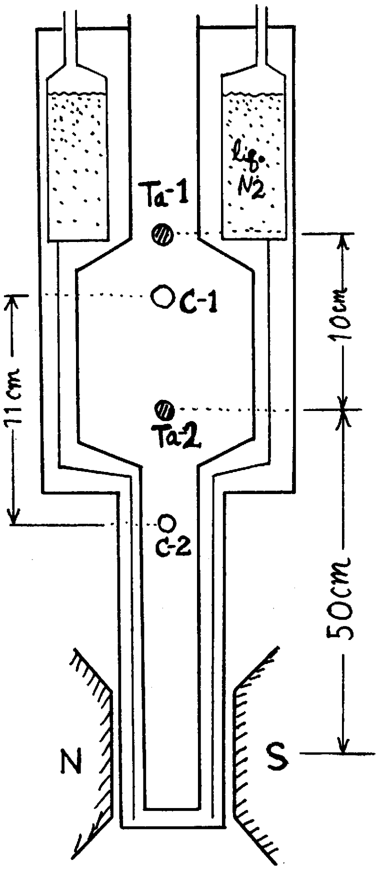

Fig. 11. Positions of the sensors in our metal dewar. Ta-1 and Ta-2 ; elemental Ta sensor, C-1 and C-2 ; Allen-Bradley resistors.

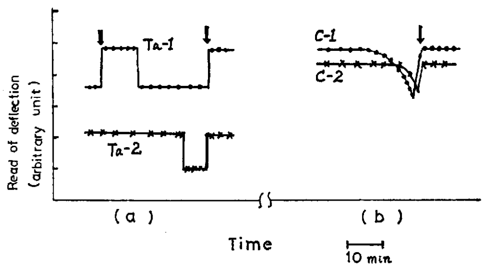

Fig. 12. An example of actual operations of our level indicator. Arrows indicate the supply of liquid helium. The ordinate shows the read of the deflection in the Wheatstone bridge.

dissipated amounts to 6.5 W in this case. Heat conduction through a solid, in the meantime, is given as[10]

$$Q=\frac{A}{L}\bar{\lambda}(T_2-T_1),$$

where A, L, and $\bar{\lambda}$ are cross-sectional area, length of a solid, and the mean heat conductivity between temperature difference T_2-T_1, respectively. Numerical calculation with this formula for the waveguide used in our experiment, made of copper and in part of stainless steel, shows heat loss of about 4.2 W. In addition, the tuning and coupling rods, and other leads may be responsible for the heat loss. This amount of loss may be too much in low temperature experiment, and we are now going to revise the whole assemble.

4 Summary

To detect a liquid level in a metal dewar for magnetic resonance experiment in the V-band region, we have constructed a simple and compact level indicator with sensing elements of elemental Ta, Nb-Ti alloy, and solid carbon resistor. The electrical or superconductive properties, repeatability, and sensitivity as well as practical operation of the indicator have been investigated at liquid nitrogen and helium temperature. In the following are these results summarized.

(Superconducting element)

(1) Ta foil (thickness 20μ) and Nb-Ti alloy (diameter 0.25 mm) were wound helically around a bakelite bobbin to have a resistance 3–5Ω at room temperature. Electrical contact of Ta with a copper lead was found difficult. (2) Temperature dependence of the resistance of Ta is well understood by the usual transport theory with the Debye

temperature 245°K, but for Nb-Ti alloy the temperature is expected to be higher than 300°K. (3) A critical magnetic field at which the transition from the superconducting to the normal state occurred, was found to differ depending on the relative direction of the bobbin to the magnetic field. In the case of Nb-Ti alloy of type II superconductor, a hysteresis effect or instability of the resistance was observed around 17 kG due to the well-known "flux jump".

(Carbon resistor)

(1) Dependence of the resistance on temperature revealed for Speer resistor (100Ω) and domestic resistor (56Ω) to be sensitive bellow 4.2°K, and these differences were considered due to the impurity involved with different activation energy-like constant. (2) Repeatability of the resistance at 78°K showed that domestic resistor was not so good. (3) At 78°K the sensitivity of the indicator was raised by winding a heater around carbon when the heater power was more than 150 mW for our measuring system. (4) No difference was observed between transverse and longitudinal magnetoresistance effect. At 4.2°K the effect was of the order of magnitude of $1\text{–}10\times10^{-3}$ at 22 kG, corresponding to temperaure difference of 0.02° for Allen-Bradley 56Ω resistor.

(Operation)

As a level indicator, however, it was found that these changes in the resistance did not give rise to any error. From the change of the liquid level in the dewar with time, rate of evaporation of liquid was estimated. It was made clear that heat conduction through a waveguide tube was responsible for the main heat loss in the present assemble.

Acknowledgement

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