RICE UNIVERSITY

MAGNETIZATION MEASUREMENTS NEAR THE
SUPERCONDUCTING TRANSITION TEMPERATURE

by

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ABSTRACT

The thermodynamically reversible phase transition of a metal into a superconducting state has a critical field curve, which can be represented by:

$$H_c = H_0(1 - (T/T_c)^2).$$

The difference in the specific heat of the two phases is given by

$$C_N - C_S = -(1/8\pi) T \left( \frac{d^2(H_c)^2}{dT^2} \right).$$

These equations predict a first order phase transition in a magnetic field and a second order transition in zero field. At $T_c$

$$C_N - C_S = -(1/4\pi) T_c \left( \frac{dH_c}{dT} \right)^2.$$

A singularity in the specific heat at $T_c$, similar to the singularity for liquid helium at the $\lambda$-point due to the onset of long range order, would mean that $dH_c/dT \to \infty$ at $T_c$.

A system of measuring the magnetization of a superconductor in an applied field has been constructed and utilized to determine the critical field curve near $T_c$. The system consists of a closed superconducting loop enclosing the sample, a nulling solenoid and a magnetometer probe; situated in a region of low magnetic fields.

Magnetization curves have been obtained for a sample of 99.999% pure indium over a temperature range of 3.365 K to 3.398 K. The reversibility of the curves was found to depend on the rate at which the magnetic field was applied.
The critical temperature was found to be 3.409°K. The slope \((dH_c/dT)\) for measurements made was -150 oe./°K. The agreement between values found from this experiment and previous work was reasonable. There was no indication of a singularity in the temperature range investigated.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>I. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>II. EXPERIMENTAL METHODS</td>
<td>7</td>
</tr>
<tr>
<td>A. Magnetic Fields</td>
<td>7</td>
</tr>
<tr>
<td>B. Magnetization Measurements</td>
<td>9</td>
</tr>
<tr>
<td>C. Temperature Regulation</td>
<td>14</td>
</tr>
<tr>
<td>III. RESULTS</td>
<td>16</td>
</tr>
<tr>
<td>IV. FUTURE WORK</td>
<td>20</td>
</tr>
<tr>
<td>ACKNOWLEDGMENTS</td>
<td></td>
</tr>
<tr>
<td>REFERENCES</td>
<td></td>
</tr>
</tbody>
</table>
I. INTRODUCTION

The transition of a metal into the superconducting phase was first shown to be thermodynamically reversible by Meissner and Ochsenfeld\(^1\) with the discovery that magnetic flux is expelled from the interior of a superconductor (the magnetic induction \(B\) vanishes). This behavior is to be contrasted with that of a perfect conductor, for which the magnetic induction remains at its threshold value \(B = H_a\) by virtue of the surface currents, which would die away with the irreversible production of Joule heat if the perfect conductivity were destroyed. Even before the discovery of Meissner, et al., thermodynamics had been applied to this transition. A phenomenological thermodynamics of the transition was given by Gorter and Casimir\(^2\).

For a system in thermodynamic equilibrium, it is required that the Gibbs free energy per unit volume \(G\) be a minimum. This implies that between two phases such as the normal-superconducting phases,

\[
G_N = G_S.
\]

In the normal state of a metal the Gibbs free energy is independent of any applied magnetic field, so that

\[
G_N(T,H_a) = G_N(T,0) = U - TS + pV.
\]

In the superconducting state however there is an energy dependence on the applied field \(H_a\) which gives
The Meissner effect in an infinitely long cylinder can be described by

\[ B = H_a + 4\pi M = 0, \]

or

\[ M = -H_a/4\pi. \]

In the absence of a magnetic field, \( G_S = G_S(T,0) \) where

\[ G_S(T,0) = U - TS + pV, \]

so that when a magnetic field \( H_a \) is applied

\[
G_S(T,H_a) = G_S(T,0) - \int_0^{H_a} M \, dH_a \\
= G_S(T,0) + H_a^2 / 8\pi
\]

for a constant temperature. The normal and superconducting states are in equilibrium at a value of the applied field \( H_c(T) \). Along this critical field curve the condition for equilibrium from above can be applied:

\[ G_N(T,H_c) = G_S(T,H_c). \]

As a result, we obtain

\[ G_N(T,0) = G_S(T,0) + H_c^2 / 8\pi \]

and

\[ G_N(T,0) - G_S(T,0) = H_c^2 / 3\pi. \]

The Gibbs free energy is not a measurable quantity, but the entropy and specific heat which are measurable can be obtained from the above expression for \( G_N(T,0) - G_S(T,0) \). The entropy and specific heat are given by

\[
S_N - S_S = -\left( \frac{\partial (G_N - G_S)}{\partial T} \right)_p = \frac{1}{8\pi} \frac{\partial (H_c^2)}{\partial T}
\]

\[
C_N - C_S = T \left( \frac{\partial (S_N - S_S)}{\partial T} \right)_p = \frac{1}{8\pi} T \frac{\partial (H_c^2)}{\partial T}^2
\]

and

\[
C_N - C_S = -\frac{1}{4\pi} \left( \frac{\partial H_c}{\partial T} \right)^2 + H_c \left( \frac{\partial^2 H_c}{\partial T^2} \right)
\]
It has been shown both from experiment and theory that the specific heat of normal metals obeys

\[ C_N = \gamma T + AT^3 \]

where \( \gamma T \) is the contribution from the electrons and \( AT^3 \) is the lattice contribution. By assuming that the specific heat of a superconductor can be described by the expression

\[ C_S = BT^3 \]

(which has some experimental justification) we can use \( C_N - C_S \) to determine the form of the critical field curve. Integration of

\[ C_N - C_S = \gamma T + (A - B)T^3 = -(1/3\pi)(d^2H_c^2/dT^2) T \]

gives

\[ \gamma T + (A - B)T^3/3 = - (1/3\pi)(d(H_c^2)/dT) + c. \]

From Nernst's theorem, the difference in entropy as \( T \) goes to zero vanishes:

\[ S_N - S_S = 0 \rightarrow d(H_c^2)/dT = 0 \rightarrow c = 0 \]

and at \( T = T_c \), \( H_c = 0 \), which gives the condition

\[ \gamma = -(1/3)(A - B)T_c^2. \]

Integrating again gives

\[ \gamma T^2/2 + (A-B)T^4/12 = -(1/3\pi)H_c^2 + d. \]

Using the conditions at \( T = 0 \), \( H_c = H_o \) and at \( T = T_c \), \( H_c = 0 \), we obtain:

\[ d = (1/3\pi)H_o^2, \quad \gamma = H_o^2/2\pi T_c^2 \]

and

\[ H_c = H_o(1 - (T/T_c)^2) \]

which is the usual parabolic critical field equation.
The critical field curve can be represented approximately by this relationship in most metals over the whole range of temperatures $T = 0$ to $T = T_c$. The approximation is very good at very low temperatures, but it breaks down at temperatures approaching $T_c$. Experimentally determined critical field curves for many superconducting metals show terms of higher order in $(T/T_c)$ than the parabolic equation $^3$.

A discussion of phase transitions has been given by Pippard$^4$ in which he discusses Ehrenfest's classification of phase transitions. The "order" of a transition is determined from the lowest order of the differential coefficient of the Gibbs free energy which shows a discontinuity on the transition line. If the entropy is discontinuous, then there is a first order transition. In a second order transition, the entropy is continuous and the specific heat has a discontinuity. In order to investigate the "order" of the superconducting phase transition, the equation for the critical field curve can be used in the expression for the entropy:

$$S_N - S_S = -\left(\frac{1}{4\pi}\right)H_c^2 \left(\frac{dH_c}{dT}\right)$$

$$= \left(\frac{1}{2\pi}\right)(H_c^2/T_c)^2 (T/T_c) - (T/T_c)^3.$$  

This expression shows that there is a discontinuity in the entropy except at $T = T_c$. This means that in a magnetic field the normal to superconducting transition is a first order transition. At $T = T_c$, however,
\[ S_N - S_S = 0, \] so we have to investigate the specific heat:

\[ C_N - C_S = -\left(\frac{1}{4\pi}\right) T_c \left(\frac{dT_c}{dT}\right)^2 = -\left(\frac{1}{\pi}\right) \left(\frac{H_c^2}{T_c}\right). \]

This expression indicates that in zero magnetic field the transition is a second order phase transition.

An investigation by Buckingham and Fairbank\(^5\) into the nature of the \(\lambda\)-transition in liquid helium showed a singularity in the specific heat instead of a finite discontinuity. The possibility of a similar singularity in the specific heat of a superconductor was considered. The singularity in the specific heat of liquid helium is explained by the long range ordering which the helium undergoes below the \(\lambda\)-point. Since in superconductors there is also a long range order associated with the correlated pairs of electrons, it has been suggested that there might be a singular term in the specific heat very near \(T_c\).

From our expression for the specific heat at the critical temperature:

\[ C_N - C_S = -\left(\frac{1}{4\pi}\right) T_c \left(\frac{dT_c}{dT}\right)^2, \]

we see that a singularity could only arise from an infinite slope \((dT_c/dT)\) at \(T = T_c\). So far all data from specific heat measurements and critical field measurements indicates that the parabolic form of \(H_c\) versus \(T\) is a good representation. The critical field curves from both types of measurements have all shown a finite
slope near $T_c$.

Measurements of the specific heat of a superconductor in a magnetic field have shown a hump in the specific heat curve around the transition region, which can be associated with the heat of transition $Q = T(S_N - S_S)^6$. However, as the field goes to zero the latent heat vanishes and the specific heat shows the predicted finite discontinuity.

One method of investigating the possible singularity is to measure the critical field as a function of temperature very near $T_c$. An infinite slope at $T = T_c$ would thus be an indication of a singularity at $T_c$.

The determination of the critical field at a given temperature in this experiment was done by measuring the magnetization of the superconductor as a function of applied field. The critical field was then determined from the value of applied field at which the superconductor made the transition to the normal state. In this experiment the transition was measured to within 10 millidegrees of $T_c$. This was not sufficiently close to $T_c$ to observe any indications of a singularity.
II. EXPERIMENTAL METHODS

In order to make magnetization measurements on a superconducting sample, there are several requirements which must be met. These include the establishment of a region of zero magnetic field in which to place the sample along with a means of applying a known magnetic field. Next, a system of detecting the magnetization of the sample is needed. Finally, some type of temperature regulator must be used to accurately control the temperature. These systems will be described below.

A. Magnetic Fields

The ambient magnetic fields in the region of the sample were cancelled by using a set of three mutually perpendicular Helmholtz coils. The field at the center of the coils could be reduced to approximately $10^{-3}$ gauss by this method.

The magnetic field applied to the sample was produced by an end corrected solenoid. Using the expression for the axial field of a thick solenoid with end coils, the magnitude of the field was calculated for different end coil dimensions and positions. The values chosen for use are listed in Table I. They were chosen because they gave the least variation of the field along the axis. This variation was less than 0.02% over a distance $R$. 
### TABLE I

**Dimensions of End Corrected Solenoid**

**Main coil**

- **Effective radius (R)**: 5.597 inches
- **Length (6R)**: 33.582 inches

**Material:**
- Aluminum pipe (O. D.) 10.75 inches
- 4-layers #10 copper wire

**End coils**

- **Distance from center (3.2R)**: 17.91 inches
- **Radius (1.1R)**: 6.157 inches

**Material:**
- Lucite rings
- 2-layers, 6 turns, #10 copper wire in series with main coil.
from the center of the solenoid.

The field was measured using a Bell "240" Incremental Gaussmeter, which uses a Hall Effect Probe. At the center of the solenoid, the field was measured to be 16.9 gauss/amp. compared to a calculated value of 17.435 gauss/amp. The deviation of the field along the axis is shown in figure 1. It was not possible to locate the sample at the center of the solenoid, so the calibration constant for the magnetic field at the center of the sample is $16.7 \pm 0.05$ gauss/amp. The position of the sample with respect to the center of the solenoid is indicated in figure 1.

B. Magnetization Measurements

The magnetization of a superconductor due to the application of a magnetic field can be measured using the conservation of the flux through a closed superconducting ring. London\textsuperscript{10} showed that the quantity

$$\phi_c = \oint \mathbf{B} \cdot d\mathbf{A} + c \oint \mathbf{J} \wedge d\mathbf{A}$$

which he called a fluxiod was conserved in a closed loop. Using this fact, J. B. Hendricks\textsuperscript{11} developed a method of measuring the magnetization utilizing the configuration of the superconducting loop and sample used in this experiment.

The theory for the ideal case of an infinitely long sample and superconducting loop is considered below. The infinitely long sample allows us to consider only parallel magnetic fields and therefore simplifies the theory.
For the simple loop shown in figure 2, the magnetic flux trapped in the superconducting loop when the sample is normal is given by

$$\phi_c = B_c A_c$$

where $B_c$ is the applied field and $A_c$ is the area of the loop. The critical temperature of the sample must be lower than that of the loop in order that the sample can undergo the transition and still keep the flux trapped in the ring. Also contained in the loop is a small nulling solenoid which produces a field equal and opposite to that of the sample magnetization. The magnetometer probe is needed to measure the change in magnetic field and control the current in the nulling solenoid.

The flux $\phi_c$ can be divided into the flux through the various components: sample, solenoid and magnetometer; $\phi_s$, $\phi_t$ and $\phi_m$ respectively. The remaining flux is then:

$$\phi'_c = \phi_c - \phi_s - \phi_t - \phi_m.$$

When the sample becomes superconducting, all fluxes change. The sample expells all flux in it, so that all the initial flux is now confined to a smaller area. The fluxes inside the loop just after the sample has undergone the normal to superconducting transition become:

$$\phi_s \rightarrow \phi'_s, \quad \phi_t \rightarrow \phi'_t, \quad \phi_m \rightarrow \phi'_m \neq \phi'_c \rightarrow \phi''_c.$$

The magnetometer, which sees the change in the flux, controls the current supply to the nulling solenoid. This solenoid creates an opposing magnetic field which
tends to reduce the magnetic flux in the loop. This opposing field increases until the magnetometer reads the same field as before the transition. Thus the final fluxes are:

\[ \Phi_s' \rightarrow \Phi_s', \quad \Phi_i' \rightarrow \Phi_i''', \quad \Phi_m' \rightarrow \Phi_m + \Phi_c'' \rightarrow \Phi_c''' \]

Since the flux in the superconducting loop remains constant,

\[ \Phi_c = \Phi_c''' + \Phi_s' + \Phi_i''' + \Phi_m = \Phi_c' + \Phi_s + \Phi_i + \Phi_m. \]

Assuming \( \Phi_c'' = \Phi_c''' \), which can be obtained by making the area \( A_c' \) (area inside the loop excluding that of the sample, solenoid and magnetometer probe) vanish, we obtain

\[ \Phi_s - \Phi_s' = \Phi_c''' - \Phi_c' \quad \text{or} \quad \Delta \Phi_s = - \Delta \Phi_c'. \]

Thus we see that any change in the flux in the sample will appear as a corresponding change in the field of the solenoid. The magnetization is thus determined by measuring the current in the solenoid.

The actual configuration of the superconducting loop, solenoid and magnetometer (figure 3) was constructed by winding two coils in opposite directions. One was wound around the sample and the other around the solenoid. A third coil was then wound around the magnetometer probe. A lead superconducting shield was provided around the probe so that the magnetometer would not respond directly to any external fields. If the coils were identical, any applied field would now induce equal and opposite fluxes in the coils and the magnetometer would not respond. However, the coils were not quite identical and a current must be supplied to the solenoid to compensate for this
difference in flux linkage (and for any nonuniformities of the applied field). Because of this the plots of magnetization versus applied field have a constant slope, for a normal sample. A difference in slope for different positions of the coils could be attributed to a nonuniformity of the field.

Using the method described above, magnetization measurements have been made. When a magnetic field is applied to the sample, flux is induced around the magnetometer probe. The magnetometer output is then amplified and used to control the current to the nulling solenoid. The field produced by this solenoid induces an opposing flux in the loop, thus producing a null system. The magnetization of the sample is proportional to the current in the nulling solenoid, while the applied field is proportional to the current in the large solenoid.

The superconducting loop is made of 10 mil Nb-Zr wire. The sample is indium cast in a glass cylinder with an inside diameter of 4 mm. and 13.5 cm. long. It is thought to be a 99.999% pure indium sample from United Mineral and Chemical Corporation. The nulling solenoid is made of #34 copper wire wound on a brass tube 5.65 mm. in diameter and 12.5 cm. long. The calculated calibration constant is 75.4 gauss/amp.

The magnetometer consists of a Hewlett-Packard fluxgate magnetometer probe No. 3529A, modified for use at
liquid helium temperatures \(^{12}\), used in connection with a Hewlett-Packard No. 428B Clip-on Milliammeter. On the most sensitive scale, full scale deflection corresponds to a field of \(10^{-3}\) gauss. The output of the magnetometer can be fed into a Dymec operational amplifier (2461-M2) with a gain variable between 0—11,000. The output of this amplifier programs a Harrison (855C) power supply (0—18 volts and 0—1.5 amp. output) which is connected to the nulling solenoid. The current through the solenoid is measured by determining the voltage across a 100 ohm standard resistor in series with the solenoid. This voltage is displayed on the y-axis of a Hewlett-Packard Moseley (700 AM) x-y recorder. A block diagram of this circuit is shown in figure 4.

The applied magnetic field is displayed on the x-axis of the recorder by measuring the voltage across a 0.1 ohm standard resistor in series with the large solenoid (powered by a Harrison 6265A power supply capable of a maximum 36 volts and 3 amp. output). A Texas Research and Electronics Corp. SI-100 integrator was used to provide a ramp input to the (6265A) power supply. This produced a continuously varying applied field, which could be swept at rates between 100 ma/sec. and 1 ma/sec. The 1 ma/sec. rate was used when near the transition region.
C. Temperature Regulation

The glass helium dewar had an inside diameter of 2" and a depth of 40". A run of approximately 3 hours could be obtained using 2 liters of liquid helium. The heat leak amounted to several milliwatts, the major heat sources being the magnetometer probe and the nulling solenoid which had a variable heat input.

The temperature regulating system (figure 5) was one developed by T. I. Smith\textsuperscript{13}. It uses a Philbrick Researches P65AU solid state operational amplifier and a 300 ohm resistor in place of the Dymec amplifier and 1K resistor described in reference 13. Smith describes an uncertainty of $\pm 5 \times 10^{-3}$ °K during the temperature cycle where most of the uncertainty is due to the hydrostatic head correction.

The quartz Bourdon tube gauge used in the system described by Smith measures the pressure in terms of deflection. The tube is then calibrated in terms of pressure. This deflection can also be expressed as a function of the temperature of the vapor pressure of the liquid helium by using the temperature scale formula given by Clement, et al.,\textsuperscript{14}. A table of the temperature as a function of the deflection was available for the original calibration of the tube. The experimental values of temperature used in this experiment were taken from this table. However, a recent recalibration has
been made. The values of temperature nearest the critical temperature of indium were found to be approximately $2 \times 10^{-3}$ °K lower than the values from the table. No correction was attempted since the correction was less than the uncertainty in the temperature of the helium bath.
III. RESULTS

For a type I superconductor such as indium, a completely reversible magnetization curve should be obtained for a sufficiently pure sample. However, in the S-N-S cycle shown in figure 6, there is some hysteresis. The amount of hysteresis was found to be a function of the rate at which the field was swept. Figure 7 shows the hysteresis for two different rates of sweeping the field (10 ma/sec. and 1 ma/sec.). Thus we see that there is a large effect on the width of the transition and the amount of flux trapped for different sweep rates. It can be noted that the time for the expulsion of the flux is approximately the same for both rates, indicating that the transition dynamics depend more on time than on the value of the field. Trapped flux is indicated by the fact that when the field is reduced to zero there is a negative magnetization.

Figures 6, 8 and 9 show a complete Meissner effect, an incomplete Meissner effect with trapped flux, and the expulsion of flux which was trapped in the superconductor in zero field, respectively.

If the transition width is defined as the change in the applied field during the transition from the superconducting state to the normal state (S-N) or in the reverse direction (N-S), then the transition width can be
measured. For this experiment the N-S transition width was approximately five times that of the S-N transition width. The actual width of the S-N transition ranged from 0.05 to 0.11 oersted. It was smaller near the critical temperature because there was less magnetization. The difference in the S-N and N-S transitions indicates that it takes longer for the flux to leave the sample than it does to enter the sample. Faber and Pippard\textsuperscript{15} explain this phenomenon in their discussion on the kinetics of the phase transition. There is still some doubt as to how much of the observed width is due to the measuring circuit.

In the region within a few millidegrees of $T_c$, the magnetization curves show some oscillations (figure 10) which are probably caused by the temperature variation of the sample through the transition temperature. During the actual experiment, the sample appeared to be undergoing transitions between the normal and superconducting states at about the same rate as the temperature regulator indicated a temperature above or below the regulation value. Increasing the temperature in increments of roughly 0.5 millidegrees showed that the Meissner effect was not visible above 3.409\degree K, which compares with the value $T_c = 3.408\degree K$ given by Shaw, et al.,\textsuperscript{16}. Quoting a value of 3.409\degree K for the critical temperature has questionable meaning when the uncertainty in temperature is $5 \times 10^{-3}$\degree K.

The values for the critical field as a function of
temperature were obtained by using the value of applied field from the magnetization curve at which the S–N transition began. The values obtained are listed in Table II along with a comparison of points from previous work. A plot of the data from this experiment is shown in figure 11. The approximate slope of these points was found to be \( \frac{dH_c}{dT} = -150 \text{ oe/°K} \). Other values which have been published include 156 oe/°K\(^{18}\) and 145.9 oe/°K\(^{19}\).
<table>
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<th>This Experiment</th>
<th>Previous Work</th>
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<td>$T ,(^\circ K)$</td>
<td>$H_c ,(\text{oersted})$</td>
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<tr>
<td>3.398</td>
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<td>5.83</td>
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<td>6.70</td>
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</table>

**TABLE II**

Critical Field Data for Indium
IV. FUTURE WORK

The finite slope of the critical field curve found from the data in Table II indicates a finite discontinuity in the specific heat. However, these measurements were made only up to within about 0.01 °K of $T_c$. The full potentialities of this system have not been realized as yet. In order to make measurements closer to the critical temperature several improvements need to be made. The value of the ambient magnetic field must be lowered and the temperature regulation improved.

A method of reducing the field to the order of $10^{-5}$ gauss has been described by Hendricks$^{20}$. A high permeability magnetic shield is available and a method of letting the trapped flux out of the lead superconducting shield should not be difficult to provide. It will also be necessary to build a solenoid to go inside the magnetic shield to provide the applied field. This solenoid should have a very uniform field over the whole sample.

Some improvement in the temperature control can be achieved by using a temperature regulator built from a design of Blake and Chase$^{21}$. Their regulator achieved a regulation of $\pm$ 200 microdegrees above the $\lambda$-point of liquid helium and $\pm$ 10 microdegrees below the $\lambda$-point. Along with the temperature regulator, an accurate
thermometer will be needed. Buckingham and Fairbank used resistors and measured temperatures of the order of microdegrees. A resistance thermometer would eliminate the pressure corrections due to the hydrostatic head above the liquid helium.

Since neither theoretical nor experimental evidence of a singularity has been presented, it is impossible to estimate the precision to which measurements will have to be made to prove or disprove the existence of a singularity. The field reduction and temperature regulation mentioned here as being possible may not be sufficient.
ACKNOWLEDGMENTS

I would like to express my appreciation to Dr. H. E. Rorschach for presenting this problem and for his assistance in carrying it through. I am also indebted to Mr. Clarence Belcher for his help and to Mr. Daltro Pinatti for his many helpful suggestions and discussions.
REFERENCES

Deviation of Axial Field of Solenoid (End Corrected)

\[ B = 50.7 \text{ Gauss at center} \]

Sample Position

Distance along axis from center (inches)
Figure 2

Superconducting Loop for Magnetization Measurements
Figure 3

Actual Configuration of Magnetization Measurement System
Block Diagram of Measurement System

Figure 4
Figure 5

Temperature Regulation System
Figure 7

Magnetization for Different Sweep Rates
Figure 8
Trapped Flux in Superconductor
Flux Trapped in Zero Field
Figure 10

Magnetization as $T \rightarrow T_c$