Critical parameters of superconductors
Why do this experiment?

Superconductivity is a very interesting property from both commercial and basic scientific points of view. Superconductors are widely used in sensitive instruments for electrical and magnetic measurements, and in satellite and telecommunications circuits. Superconducting magnets for Magnetic Resonance Imaging is a great medical and commercial success as a diagnostics tool. Power circuits based on superconducting properties are available from most large companies, although the technique so far has not been a commercial success. Because superconducting components are now spreading to all parts of society a basic knowledge of superconductors and their properties should be of great value to engineers and physicists.

Aims of the experiment:

After completing this experiments you should

- understand critical currents, critical temperature, and critical fields,
- have a basic knowledge about measurement techniques,
- understand the properties of a very useful instrument, the “lock-in”.

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!!! WARNINGS !!!

PLEASE READ THIS FIRST!

**Warning 1: Contacts …**

… are one big weakness of ceramic superconductors. It is not possible to use normal methods to make strong contacts, such as soldering or welding.

The wires on the plate you will be using here have contacts consisting of silver paint. This means that
1) they are mechanically weak,
2) they cannot take more than 0.5 A without burning out.

Please do not destroy the laboratory equipment -
KEEP THE CURRENT BELOW 0.5A AT ALL TIME,
and do not pull on the wires to the sample.

**Warning 2: A superconducting material is a perfect short circuit!**

You are going to measure the resistance of a potentially superconducting material by applying a current. If you try to do this using a power supply directly you will exceed the current rating of the sample as shown above - you will have a short through the superconductor!

You must always use a (current limiting) resistor in series with the superconductor samples!

![Diagram](image-url)
EXPERIMENTAL EQUIPMENT

The superconducting plate to be studied has been encased in a metal sheath (for protection) together with a Copper-Constantan thermocouple for temperature measurements.

For cooling down the sample below the critical temperature one uses a Dewar filled with liquid nitrogen. To study the critical parameters of the superconducting sample a lock-in voltmeter, an electromagnet, power supplies and multimeters are available.

Please be careful - do not lift the sample by the electrical wires!

THEORY

Superconductivity is a low-temperature phenomenon, and the superconducting state in a material can be destroyed by applying

- a temperature $T$ higher than the **critical temperature**, $T_c$,
- an external magnetic field $B$ larger than the **critical field**, $B_c$,
- a current larger than the **critical current**, $I_c$.

In this experiment you will try three techniques to measure the three critical parameters $T_c$, $B_c$, and $I_c$, the last two on the same specimen. All measurements will be carried out above the temperature of liquid nitrogen, about 77 K, and the accuracy of some of the data will not be very good, but you should get the correct order of magnitude.

The **critical current** $I_c$ will be measured resistively, i.e. you will apply a supercurrent to the material and note the current value at which a voltage appears over the sample. This is the most basic and simple method, but also the most dangerous - it is easy to overheat the contacts or even the sample. A better accuracy is usually obtained by using an equivalent method, applying a constant current through the sample and noting at which temperature a voltage appears on heating. There is reason to believe that the supercurrent increases exponentially with decreasing temperature below $T_c$, so we expect you to find the critical current at 77 K by plotting the logarithm of $I_c$ versus $T$ and extrapolating linearly to 77 K.

The **critical magnetic field** $B_c$ will be measured by measuring $T_c$ by the resistive method with and without a small external magnetic field. $B_c$ varies with $T$ according to the empirical formula

$$B_c(T) = B_c(0) \left[1 - \left(\frac{T}{T_c}\right)^2\right],$$

which can be used to find $B_c(0)$ and $B_c(77)$ from your measured data.

In the first two tasks you measure $T_c$ by the resistive method. Very often, it is easier to measure the **critical temperature** $T_c$ of an unknown material inductively, because this method does not require electrical contacts to be formed. In this method, the sample is placed inside an inductive coil, and the inductance (and sometimes the resistance) of this coil is measured as a function of temperature. In the superconducting state a material does not allow a magnetic field to penetrate the surface, and the magnetic field from the coil is "pushed out" from the superconductor. At $T_c$, the field can take up its "normal" equilibrium position through the material studied. The measured inductance will then usually have a step change at $T_c$. 
**Task 1:**

**Measurement of the critical current, I_c**

In this experiment you need to be able to vary the temperature of the sample in a simple way, and we suggest that you suspend the sample by a string inside a Dewar flask (a silvered glass container for liquid nitrogen). By moving the Dewar mechanically up and down you can then vary the temperature of the sample.

1. Pour liquid nitrogen into the Dewar to fill it half way, then raise the Dewar such that the sample is cooled to 77 K. Connect the thermocouple to a computer and measure the temperature of the sample continuously. Refill the Dewar whenever necessary.

2. Apply a constant current of 0.1 A through the BLACK wires, and measure the voltage over the superconductor using the YELLOW wires. In the superconducting state this voltage should be zero. Lower the Dewar such that the superconductor is well above the LN₂ surface. The temperature will slowly increase. Note the temperature at which a non-zero voltage appears on the voltmeter - this is T_c. (Practical hint: choose a certain voltage level as your definition of T_c!)

3. Raise the Dewar again and cool down the sample. Increase the current to 0.2 A and repeat the experiment.

4. Continue for 0.3, 0.4, and 0.5 A.

5. Plot your experimental data for ln I_c versus T_c in a diagram and fit a straight line. Use this line to extrapolate to 77 K. What is the critical current I_c(77)?

According to the manufacturer, the voltage probes are 11 mm apart and the cross-sectional area of the sample is approximately 0.96 cm². What is the critical current density j_c at 77 K?
**Task 2:**
**Measurement of the critical field, B_c**

To measure $B_c$, you will measure the critical temperature $T_c$ with and without a small applied magnetic field, then calculate $B_c$ from the empirical formula given in the section Theory.

This is a difficult and not very accurate measurement, because you need to measure the resistance of the sample as a function of temperature while the temperature is changing quickly - the sample will be suspended in the gap of an electromagnet without much thermal insulation! To increase the accuracy you should measure $T_c$ with and without a field under as identical conditions as possible, i.e. in the gap of the magnet in both cases.

1. Make sure the magnet is switched off. Connect the thermocouple to the computer to measure temperature.

2. Apply a current of max. 100 mA through the BLACK wires and connect the YELLOW wires to the computer input to measure the resistance $R$ (or at least a voltage $V$ proportional to $R$).

3. Cool down the sample to 77 K, place the cold sample in the Teflon™ container (to get at least a little bit of thermal insulation!) and put it between the poles of the magnet. Refill the container with liquid nitrogen to ensure that the sample is cold.

4. Start the computer program. Measure $R$ as a function of $T$ until you are sure that you have passed up through $T_c$.

5. Move the sample away from the magnet a re-cool.

6. Switch on the power to the magnet coils and measure the magnetic field in the gap using a magnetometer.

7. Put the cooled sample in its Teflon™ container back between the poles of the magnet and repeat the measurement of $R$ versus $T$.

Use the data to calculate the critical magnetic field $B_c$ of the sample at 77 K and at 0 K.
**Task 3:**
**Inductive measurement of the critical temperature, $T_c$**

You have already measured $T_c$ twice by resistive techniques, and now we repeat the measurements with an inductive technique. In this experiment, a superconducting specimen in the form of a cylinder is encased in a copper coil. The coil, of course, has a resistance $R$ and an inductance $L$, and the total impedance is then $Z = R + j\omega L$.

1. Again, you can use the Dewar (see Task 1) and suspend the sample inside. Connect the thermocouple to the computer as in Task 2.

To measure the inductance and resistance you will use a lock-in amplifier, a very advanced AC voltmeter which can measure both in-phase and out-of-phase voltages simultaneously, and has a built-in signal generator. Do not fear! Read Appendix A instead!

2. Connect the coil as shown in the figure below. Make sure you understand how to read a voltage proportional to $R$ and a voltage proportional to $j\omega L$ on the voltmeter. (You can always ask the supervisor!) Connect the analog output voltage proportional to $j\omega L$ to the computer for measurement.

3. Cool down the sample by immersion in liquid nitrogen.

4. Switch on the computer program. Measure $j\omega L$ as a function of $T$ until $T$ is above 150 K.

5. If you have time, repeat the measurement measuring instead $R$ versus $T$ in the same range.

6. Plot the data and deduce the critical temperature $T_c$. 

![Diagram of the inductive measurement setup](image-url)
**APPENDIX A: Lock-in voltmeter**

The lock-in voltmeter is a highly sensitive AC voltmeter with extreme selectivity. It is able to measure a signal at one particular signal frequency in the presence of noise and interference with an amplitude at least 1000 times larger. Furthermore, it is able to measure a voltage with a specific phase angle relative to a reference signal and to reject all signals with other frequencies and/or phase angles.

**Locate the input range selector and the input connectors on your lock-in voltmeter! It is also equipped with a selector that allows you to use a differential input (A-B). Locate this.**

The price we must pay for this performance is the need for a reference signal. The frequency and phase angle of the reference signal determine the frequency and phase angle at which the voltmeter measures. The reference signal is processed in the voltmeter to have a constant amplitude which we can take as 1. Basically, the voltmeter then multiplies the input voltage

\[ U_{in} = A \sin (\omega t + \phi) , \]

with relative phase angle \( \phi \), with the reference voltage

\[ U_{ref} = \sin (\omega_{ref} t) . \]

The result will be a sum

\[ U_{out} = \frac{A}{2} \cos \left( (\omega + \phi - \omega_{ref}) t \right) + \cos \left( (\omega + \phi + \omega_{ref}) t \right) \].

All high frequencies are filtered away by a low pass filter with time constant \( \tau \), such that only the first term above remains. In practice, the results can be filtered such that only a DC signal is measured, and this signal of course corresponds to \( \omega = \omega_{ref} \), or

\[ U_{out} = \frac{A}{2} \cos \phi . \]

The voltmeter thus measures only signals with a frequency equal to the reference frequency, and the output voltage is proportional to the cosine of the phase angle.

**Locate the reference input connector!**

There may be external circuits that give a positive or negative phase shift on the signal to be measured, and it must thus always be possible to modify the phase angle of the reference signal such that we can choose any value we need between 0 and 360°.

**Locate the phase shift controls on your voltmeter. Try to understand how to do to rotate the phase by \( \pm 90° \) or \( \pm 180° \)!**

There may be many other features on a good voltmeter, such as additional filters, switchable time constants, etc., but the only feature you need here is the output voltage connector, which gives an analog output proportional to the analog output signal on the meter.

**Locate the output voltage connector - it should be connected to the computer.**
When you have located all the above details, you can start the experiment:

1. First you must decide on the reference phase. It is common practice to choose the phase angle of the \textbf{current} as the reference phase \( \phi = 0 \).
   - In your set-up, there is a resistance in series with the coil. Connect the input leads such that you measure the voltage drop over this \textbf{RESISTOR}, in which \textit{voltage and current are in phase}.
   - Change the input voltage to give a mid-scale reading on the meter. (No, not zero - half full scale!)
   - Without further changes, use the phase control to make the output voltage exactly \textit{zero}. You are then measuring the \textbf{quadrature} of the input voltage, i.e. you measure 90\(^\circ\) away from the phase angle of the current.
   - Change the phase angle by 90\(^\circ\), and you measure \textit{in phase with the current}! (If necessary, you can also add another 180\(^\circ\) to get a positive output voltage.)

You have now defined \textbf{phase angles} 0\(^\circ\) (in phase) and 90\(^\circ\) (out of phase). Note the positions of the 90 and 180\(^\circ\) switches, but do not touch the continuous phase control any further!

2. Remove the cables from the resistor and reconnect them to the \textbf{COIL}. The voltage over this is small (probably about 1000 times smaller than over the resistor), and there are potential contacts to eliminate the effects of long leads. Since you have not changed the current, you measure:
   - \textit{IR} ("resistance") at \textbf{phase angle} 0\(^\circ\)
   - \textit{IoL} ("inductance") at \textbf{phase angle} 90\(^\circ\).

You can thus choose what to measure using the phase control buttons only.
APPENDIX B: Four-Probe resistance measurements

When resistances are low it is necessary to measure them with very high accuracy, and the resistance of the connecting leads must be eliminated from the measurements. For this reason small resistors usually have four leads instead of two, connected as shown below. The resistance between the two connection points is the true resistance $R$.

In a measurement of this type a current $I$ is supplied through the two "current" leads and the resulting voltage drop $RI$ is measured using a high impedance voltmeter connected to the two "voltage" leads. Because of the high impedance, no current flows through the "voltage" leads and the measured voltage corresponds exactly to $U = RI$.

Most modern digital voltmeters are supplied with four input terminals and can measure resistances in either two-wire or four-wire mode.

Voltage source, supplying current:

- Current lead
- Actual resistance, $R$
- Current lead

No current in the voltage leads, so no voltage drop; high input impedance voltmeter measures the true resistance as $R = U/I$. 