Superconductivity

1. Introduction

When cooled down below a characteristic critical temperature $T_c$, a specific phase transition of electron system can be observed in certain materials. This transition is characterized by a sharp decrease of electrical resistance to zero, and complete ejection of magnetic field from the superconductor. Both phenomena: resistance disappearance and ideal diamagnetism are the main subjects of the present experiment.

2. Literature

description of the experiment in the supplementary material


additional literature:
3. Preparation

Electrical resistance. Describe the temperature behavior resistance of typical metals, semiconducting and superconducting materials. Explain the main phenomena which lead to the temperature dependence of resistance of these materials. What is the reason for superconductivity?

Magnetic properties. What is going on with superconductor in the magnetic field (ideal diamagnetism, Meissner). Explain the difference between superconductors of the first and second type. What is flux quantization, Shubnikov-phase and Abrikosov lattice.

Magnetic susceptibility. Describe magnetization curves of diamagnetic, paramagnetic, ferromagnetic materials and ideal superconductors of the 1st and 2nd type. How high magnetic field, temperature and current density influence the superconducting phase?

Describe the transport measurements with 4-point method and the inductive measurements of AC-susceptibility. Inquire about set-up, the electrical wiring in the deep-stick and lock-in technic.

4. Experiment

a) Resistance measurements

At first, measure resistance vs. temperature $R(T)$ dependence of Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ sample by decreasing the temperature. Reach the superconducting state. Perform the same $R(T)$ measurements by increasing the temperature. Compare two measured curves. from the room temperature to superconducting state. Discuss the superconducting fluctuations on the resistance curve with your supervisor.

b) Inductive measurements

The Real (Re) and Imaginary (Im) parts of AC-susceptibility $\chi=\chi'+i\chi''$ are temperature dependent. Take both parts by cooling the sample down and warming it again up.

c) Levitation experiments
Bring the superconducting samples above the magnet and try to make it levitate. Try different magnet configurations (ring magnet, magnet film, quadrupole). In which case the superconductor can move in magnetic field and in which case possibility to move is suppressed.

Demonstrate the Meissner-Ochsenfeld-Effect.

5. Analysis

for a)

How behave the sample in the region with normal conductivity? Find $T_{c,0}$ (the temperature, at which the resistance goes to zero), $T_{c,\text{onset}}$ (the temperature, at which the transition to superconducting state starts) and $\Delta T_c=(T_{c,0} - T_{c,\text{onset}})$ for both measurements. Why there is a difference between the measured curves? Which curve is better to use for the analysis?

Explain the importance of the superconducting fluctuations in the phase transition.

for b)

Find the critical temperatures as it was done for a), with the difference that $T_{c,0}$ is the temperature, at which the superconductor is not an ideal diamagnet any more. At which temperature is the imaginary part of the susceptibility $\chi''$ maximal? Show that the imaginary part of the susceptibility is $\propto$ proportional to the energy loss ($A_H=\pi(\mu_0 H_A C)^2 \chi''$).

for c)

Explain your levitation experiments. Which forces affect the samples? What is the connection between the levitation force and the magnetization? Why can the samples made of the superconductor materials with a strong pinning levitate under the magnet?
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Superconductivity

The experiment consists of three parts:

- resistance vs. temperature measurements
- ac susceptibility vs. temperature measurements
- demonstration of the Meissner effect and vortex pinning. Levitation in magnetic field and magnetic road experiments.

1. Measurement of superconducting samples

The critical temperature $T_c$ can be found with the help of resistive and inductive methods. The critical temperature $T_c$ is characterized by different specific values:

- $T_{c,\text{on}}$ labels the beginning of transition, e.g., the temperature, at which the resistance starts clearly deviate from the resistance at normal conducting state.
- $T_{c,\text{mid}}$ labels the temperature, at which the resistance drops by 50% of its resistance at normal conducting state $R_N$.
- $T_{c,\text{zero}}$ labels the temperature, at which the resistance decreases to "zero" or, in other words, to a so small value that it cannot be measured experimentally. $T_{c,\text{zero}}$ represents the temperature value, at which the material is superconducting in the conditions of measurements.

Furthermore, the transition width $\Delta T_c$, which shows the temperature difference between 10% and 90% of $R_N$, is required to be defined.

1.1. The critical temperature by resistance measurements.

1.1.1. Preparation and contacting of the samples

The resistive method is used for the samples made of bulk high-temperature superconductors YBa$_2$Cu$_3$O$_7$ and Bi$_2$Sr$_2$CaCu$_2$O$_{8+y}$.

For preparation of a YBa$_2$Cu$_3$O$_7$ bulk sample, Y$_2$O$_3$, BaCO$_3$ and CuO powders with a ratio $\frac{1}{2} : 2 : 3$ is used. This stoichiometric mixture is pressed to pellets (~1.5 kBar), and then calcined at 930°C in the air. By pressing and sintering at 980°C in the air, the pellets of 35 mm diameter are obtained. Then thin plates are cut from these pellets with the help of a diamond saw. The plates with the size of $l = 17 \text{ mm}$, $b = 2.7 \text{ mm}$ and $d = 1.3 \text{ mm}$ can be used for measurements. One plate is placed onto a Kapton film, which is glued with a fat (APIEZON) to a
copper holder. The Kapton film guarantees both electrical isolation and a good thermal contact between the YBa$_2$Cu$_3$O$_7$ plate and the sample holder. In order to improve the thermal contact the sample is pressed to the Kapton-film with phosphor bronze press contacts. The last are soldered to copper wires, which are connected to measuring devices. The electrical contact between the sample and the press contacts can be improved by a silver paste.

For preparation of a Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ sample a small flake (0.5 mm by 5 mm) of a Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ single crystal is glued to a sapphire substrate. Sapphire is a perfect electrical insulator and simultaneously shows a good thermal conductance. The thickness of the flake can vary around 100 nm. Four Au contacts are fabricated by thermal evaporation through a shadow mask. Schematically the sample with contacts is shown in Fig. 1.1. The substrate is glued to a copper sample holder. Electrical contact to the gold stripes are provided by thin Al wires connected by ultrasound bonding technique.

![Figure 1.1. Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}$ glued on sapphire substrate with 4 gold stripes.](image)

Four contacts must be used in order to provide 4 point geometry of resistance measurements.

### 1.1.2. Resistance vs. temperature measurements

Four point geometry of resistance measurements is shown schematically in Fig. 1.2.
The constant current $I_{tr}$ is sent through the sample and the voltage $U_{4p}$ is measured at the contacts in the middle. The voltage $U_{4p}$ is proportional to the resistance of the sample $R$ between the contacts for $U_{4p}$ measurements. The 4 point geometry allows to eliminate the resistance of the contacts and wires and is essential for superconducting samples measurements.

To determine the critical temperature of the samples the voltage $U_{4p}$ and thereby the resistance $R$ of the sample are measured as a function of the temperature. The difference of the sample and the temperature resistor positions on the sample holder and their different heat capacitances lead to uncertainty in the results of measurements. Namely, the results of measurements depend on the cooling rate. In order to obtain accurate values of the transition temperatures the data are taken by both cooling down and warming up. The cooling and warming curves differ and show hysteresis, which increases with higher temperature change rate. The errors of the critical temperatures can be found analyzed. Since the experiments are performed for high temperature superconductors a standard temperature sensor Pt100 can be used.

The transition of YBa$_2$Cu$_3$O$_7$ bulk sample turns out to be relatively wide in comparison to low temperature superconductors, e.g., Nb. It can be attributed inhomogeneity in the materials which critical temperature strongly depend on the concentration of oxygen and impurities.
Moreover, the resistive method is also used for determination of the second critical field temperature dependence $B_{c2}(T)$ of e.g., Nb film. Taken values of the resistance of Nb film are presented then as a function of the magnetic field at different temperatures. The magnetic field is provided by the superconducting 8-Tesla magnet. From the obtained $R(B)$-curves (Fig. 1.5) one can calculate the critical field $B_{c2}$, at which the superconductivity disappears, as a function of the temperature. The value at a cross-point of the extended line of the linear part of the transition $R(B)$ with the B-axis is taken for $B_{c2}$, as it is shown in Fig. 1.5 for $T = 4.2 \, K$. Fig. 6 presents the obtained values (triangles). The literature values of the critical field at $T = 0$ ($B_{c2}(0) = 0.195 \, T$) and critical temperature $T_c$ ($T_c = 9.3 \, K$) were used to find $B_{c2}(T)$ dependence with a help of the phenomenological equation

$$B_{c2}(T) = B_{c2}(0) \left(1 - \frac{T}{T_c}\right)^2.$$

The results of calculation are shown in Fig. 6 as a solid line. It is clearly seen that the measured $B_{c2}(T)$-values for Nb film are significantly higher than the values obtained from the phenomenological equation for a pure bulk Niobium. This difference is explained with the boundary scattering effects, which make the mean free path $l$ and therefore the coherence length $\xi_{GL}$ of Nb film smaller in comparison to the pure bulk Nb.
Figure 1.4. Resistance vs. temperature dependence of Nb.

Figure 1.5. The resistance $R$ of Nb as a function of the magnetic field at different temperatures.
1.2 The inductive method of $T_c$ measurements

The inductive measurements of the critical temperature can be performed with the help of a MIB (mutual inductance bridge). It consists of a system of coils shown schematically in Fig. 1.7. There are two identical secondary coils (sample- and reference coils) with equal windings placed inside a primary coil. The parameters of the coils are listed in Table 1. The coils are wounded with a 100 $\mu$m (primary coil)- and 40 $\mu$m-thick (secondary coils) copper wire on movable teflon pieces. The inner teflon piece in the secondary sample coil is emptied inside in order to place their a superconducting sample ($R = 1.3\ mm, L = 4\ mm$) (see Fig. 1.7).
A piece of NbN bulk sample in form of a cylinder is used as a superconducting sample. NbN was obtained by glowing Nb-sheet or Nb-powder at about 1400° C in nitrogen atmosphere for several hours.

For better thermal contact the copper foil coated with a fat (APIEZON) is used. It is brought on to the sample before placing it into the inner teflon piece. The ends of the foil which are out of the teflon piece are clamped to the sample holder made out of copper.

By application the alternating current with the constant amplitude through the primary coil of the MIB, which is provided by the alternating voltage with the amplitude $U_0$ and the series resistance $R_V$ with $R_0 = R_V + R_p \gg R_p$,

$$I(t) \approx \frac{U_0}{R_0} \sin \omega t,$$

one gets inside of it alternating magnetic field

$$B(t) = \mu_0 N_p \frac{l_p}{\sqrt{l_p^2 + 4r_p^2}} I(t) =$$

$$= \mu_0 N_p \frac{U_0}{R_0} \frac{1}{\sqrt{l_p^2 + 4r_p^2}} \sin \omega t.$$  \hspace{1cm} (1.2.2)

Using the values obtained in the experiment ($U_0 = 0.4 \, V, R_0 = 1070 \, \Omega$), one can calculate the maximum value for the magnetic field of

$$B_{max} = 1.9 \cdot 10^{-5} \, T \approx 0.5 \, B_{Erde}.$$
This alternating magnetic field induces in both secondary coils voltage \( U_{\text{Probe}} \) and \( U_{\text{Ref}} \). If the sample is in normal-conducting state, it has practically no influence on the inductive voltage \( U_{\text{Probe}} \). Then

\[
-U_{\text{Probe}} \approx U_{\text{Ref}} = -\frac{d}{dt}(N_sBA) = -N_sAB_{\text{max}}\omega \cos \omega t = -2.08 \text{ mV}, \tag{1.2.3}
\]

where \( \omega = 2\pi \cdot 27 \text{ Hz} \). The sum both inductive voltages \( U_{PR} \) is almost 0 Volts. By the change to the superconducting state the magnetic field is pushed away from the inner of the sample because of its diamagnetic behavior. Its changes the inductive voltage \( U_{\text{Probe}} \) in the sample coil, while the voltage in the reference coil remains the same. Therefore \( U_{PR} \) becomes measurable with the value

\[
U_{PR} = \frac{N_s}{l_s} \omega LR^2\pi \cdot \frac{L^2}{L^2 - 4R^2 \ln \frac{L}{2R} \left( \frac{l_s}{l_s^2 + 4r_s^2} + \frac{L}{\sqrt{L^2 + 4R^2}} \right)} \cdot B(t), \tag{1.2.4}
\]

where the dipole approach is used for the field of the magnetized superconducting sample. As the signal is proportional to the suppression of the field in the range of the transition to the superconducting state, the measured value of the alternating current is equal to "-1". By putting all parameters into the Eq. 1.2.4 one gets the peak voltage of

\[
U_{PR} = 93 \mu \text{V}.
\]

In the experiment could be reached only the half of this value, which is most probably due to the uncertainty in the determination of the sample geometry. To measure this voltage one requires a phase-sensitive detector (lock-in-amplifier).
2. AC-susceptibility measurements in High-Tc superconductors

In order to characterize the phase transition of a superconductor, AC-susceptometer can be used. The measurements are performed as a function of temperature and allows to find the critical temperature $T_c$ and the width of the phase transition $\Delta T_c$. Moreover, the phase purity of the sample could be found from the susceptibility vs. temperature behavior.

The concept of all susceptometers is based on measurements of the magnetic momentum $\vec{m}$. The magnetic momentum is the material property which characterizes magnetic field produced in the sample. Applied magnetic field $\vec{H}$, the temperature $T$ and other parameters, which describe the previous history of the sample, change the magnetic momentum. It is also depends on the sample volume $V$. Therefore, one defines the volume magnetization $\vec{M}$ as

$$\vec{M} = \frac{1}{V} \vec{m}.$$  (2.1)

This magnetization has the following relation to the applied magnetic field $\vec{H}$

$$\vec{B} = \mu_0 (\vec{H} + \vec{M}), \quad \mu_0 = 4\pi \cdot 10^{-7} \frac{Vs}{Am}.$$  (2.2)

The response of the magnetization to the external magnetic field $\vec{M}(\vec{H})$ can be often considered as a linear response. Then, the coefficient of proportionality is called magnetic susceptibility $\chi$:

$$\vec{M} = \chi \vec{H}.$$  (2.3)

If $\vec{M}$ and $\vec{H}$ are parallel to each other, the magnetic susceptibility $\chi$ is a scalar, otherwise, $\chi$ is a tensor. The susceptibility defined in a static magnetic field is also referred as $\chi_{DC}$. By applying an alternating magnetic field the susceptibility is defined as an alternating current susceptibility $\chi_{AC}$. Measurements of $\chi_{AC}$ will be described in detail in the next sections.

2.1 Measurements principle

The sample is fixed in a AC-susceptometer in a removable secondary coil as it is shown in Fig. 2.1. An alternating current is applied to the primary coil inducing an alternating magnetic field inside the space of the primary coil. The magnetic momentum of the sample follows the alternating magnetic field (see Fig. 2.2).
Fig. 2.1. Primary and secondary coil configuration

Figure 2.2: Illustration of the AC-susceptometer. Magnetic field and magnetic momentum of the sample are shown for two different time points.
For the exact description of the sample behavior in the alternating magnetic field, the complex susceptibility $\bar{\chi}$ has to be considered.

If the time-dependence of the applied external magnetic field $H_a$ follows the form

$$H_a = H_0 + h_{AC} \cos \omega t ,$$

then the induction voltage $U$ in the removable coil with radius $r$, per one coil loop

$$U = -\frac{d\Phi}{dt} = -\pi r^2 \frac{dB}{dt} = -\pi r^2 \mu_0 \frac{d(H_a + \bar{M})}{dt} =$$

$$= \pi r^2 \mu_0 \left( h_{AC} \omega \sin \omega t - \frac{d\bar{M}}{dt} \right) ,$$

(2.5)

where $\bar{B}$ is the average value of the magnetic induction per sample volume and $\bar{M}$ is the magnetic momentum. The term "$\sin \omega t$" describes the induction signal of the alternating field $h_{AC}$, the term "$\frac{d\bar{M}}{dt}$" describes the sample response.

As $\bar{M}$ is a periodic function with $T = \frac{2\pi}{\omega}$, one can present $\bar{M}$ as a Fourier series:

$$\bar{M} = \langle M \rangle_T + h_{AC} \sum_{n=1}^{\infty} \left( \chi_n' \cos n \omega t + \chi_n'' \sin n \omega t \right) ,$$

(2.6)

where $\langle M \rangle_T$ is equal to

$$\langle M \rangle_T = \frac{1}{T} \int_0^T dt \bar{M}$$

(2.7)

The real and imaginary parts of susceptibility are given by the following expressions:

$$\chi_n' = \frac{\omega}{\pi h_{AC}} \int_0^T dt \bar{M} \cos n \omega t$$

(2.8)

$$\chi_n'' = \frac{\omega}{\pi h_{AC}} \int_0^T dt \bar{M} \sin n \omega t .$$

(2.9)

Using 2.5, the induction voltage can be rewritten as

$$U = U_0 \left[ \sin \omega t + \sum_{n=1}^{\infty} n(\chi_n' \sin n \omega t - \chi_n'' \cos n \omega t) \right] ,$$

(2.10)

where $U_0 = \pi r^2 \mu_0 h_{AC} \omega$ is the amplitude of the voltage signal without sample.

With the help of lock-in technique only $U_1$ (corresponds to $n=1$ in 2.10) is measured. The frequency dependence of $U_1$ is given by

$$U_1 = U_0 (\sin \omega t + \chi_1' \sin \omega t - \chi_1'' \cos \omega t) .$$

(2.11)
Due to the fact that in practice the sample is often smaller than the coil, the term in Eq. (2.11) \( \sin \omega t \) induced by alternating field \( h_{AC} \) would dominate the induction signal.

Therefore, an empty reference coil (called balance coil in Fig. 3.2) with the same parameters as the detection coil with the sample (see Fig. 3.2) but with an opposite windings is used in series. In this way a self-induced flux change due to the alternating magnetic field is eliminated and the measured voltage is equal to

\[
U_1^* = U_0 (\chi_1' \sin \omega t - \chi_1'' \cos \omega t). 
\]

In case of a complex susceptibility,

\[
\tilde{\chi} = \chi' + i\chi'' = \chi_1' + i\chi_1'' ,
\]

one can write \( H_a \) and \( U_1^* \) as:

\[
H_a = H_0 + Re(h_{AC} e^{-i\omega t}) \quad \text{(3.14)}
\]

\[
U_1^* = U_0 Re(i\tilde{\chi} e^{-i\omega t}) \quad \text{(3.15)}
\]

The real part \( \chi' \) is the inductive component (with a phase shifting of \( \frac{\pi}{2} \) to \( H_a \)). The imaginary part \( \chi'' \) is the resistive or lossy component.

Between \( \chi'' \) and the area \( A_H \), which is enclose by a hysteretic curve \( B_i = f(B_a) \),

\[
A_H = \Phi \mu_0 H_a d\tilde{B} = \int_0^T \mu_0 (H_0 + h_{AC} \cos \omega t) \frac{d\tilde{B}}{dt} dt, \quad \text{(2.16)}
\]

the following relation can be shown

\[
A_H = \pi(\mu_0 h_{AC})^2 \chi'' . \quad \text{(2.17)}
\]

One can get it by taking into account only the first Fourier component in Eq. (2.16) with \( n = 1 \).

The area \( A_H \) is proportional to the energy loss \( W_V \) per volume unit in the sample, after becoming the closed loop on the hysteretic curve:

\[
W_V = \frac{A_H}{\mu_0}. \quad \text{(3.18)}
\]

If the field amplitude \( h_{AC} \) is constant, the imaginary part \( \chi'' \) is proportional to energy loss \( W_V \) in the sample.

In the normal-conducting sample \( \chi' \approx 0 \) and \( \chi'' \approx 0 \), so that \( U_1^* \approx 0 \). In a superconducting sample in Meissner-phase \( \chi' \approx -1 \) and \( \chi'' \approx 0 \), so there is no hysteretic behavior in a
magnetization loop. The induction voltage is then \( U_1^* = -U_0 \sin \omega t \). In the range of the phase transition of type II superconductors the real part of susceptibility \( \chi' \) changes and \( \chi'' \neq 0 \). This leads to the fact that in one closed loop of \( H_a \) magnetic flow penetrates into the sample and goes out from the sample again and, therefore, \( B_i(B_a) \) shows hysteretic behavior.

In type II superconductors there are two basic loss mechanisms. The first one is due to the moving of the flow lines, analogy to the turbulent flow loss in the normal-conducting state, and the second one is because of the vortices pinning.

**Literature**