7.1 Resistance vs. Temperature of the YBCO SQUID

Purpose

This experiment will allow you to observe the superconducting transition of the YBCO film that forms the SQUID in Mr. SQUID[®]. By tracking the resistance of the SQUID with the V-I curve, you can watch the YBCO undergo its superconducting transition. The additional equipment needed for this experiment allows you to measure the temperature of the SQUID chip as it is cooled down to liquid nitrogen temperature.

Equipment

For this experiment you will need:

- A glass encapsulated silicon diode¹⁴ •
- 200 cm of insulated copper magnet wire of size 32 to 40 AWG¹⁵ (this corresponds to a diameter of 0.020 to 0.008 mm)
- A digital volt meter (DVM) with sub-millivolt resolution¹⁶
- A soldering iron and electronics-grade solder
- A binder clip (available from any stationary supplier)
- One or two cotton balls
- Masking tape
- An active dc constant current source capable of supplying 10 µA of current with a voltage compliance of at least 2 volts.

If you do not have a constant current source then you will also need:

- A general-purpose operational amplifier $(e.g., 74)^{17}$
- A zener diode in the range of 2.5 to 7 volts¹⁸
- A selection of resistors in the range of 1 k Ω through 100 k Ω
- A capacitor within the range of 100 pF to 100 nF
- A 9-Volt transistor battery
- A solderless breadboard¹⁹

¹⁴Such as Radio Shack[®] part number 276-1122.

¹⁵Such as Belden Beldsol Solderable Magnet Wire Types 8081 through 8087.

¹⁶Such as a Kiethley Model 197 microvolt DVM. Millivolt resolution may be acceptable but will limit the accuracy of the temperature measurements. ¹⁷Such as a LM741, Radio Shack[®] part number 276-007.

¹⁸Such as Radio Shack[®] part number 276-565.

¹⁹Such as Radio Shack[®] part numbers 276-169, 276-174, 270-175.

In this experiment, we will use a common silicon diode to measure the temperature of the SQUID in the Mr. SQUID[®] probe. Silicon diodes are a common and accurate way to measure temperature from 300 K down to liquid helium temperatures (4.2 K). They are simple to use, inexpensive, and the voltage across such a diode varies linearly with temperature. The reason a diode can be used as a linear temperature sensor is explained in a section at the end of this experiment, but this information is not required to perform the experiment.

7.1.1 Diode sensor calibration

<u>Set-up</u>: First, trim the leads of the diode to be as short as possible, then carefully solder on new leads using the copper magnet wire, each one at least 50 cm long. Be careful not to overheat the diode with the soldering iron. After the leads are attached to the diode, check the diode with an ohmmeter to verify continuity through its leads and that it still acts as a rectifier (in one direction, it should have a moderate resistance; in the opposite direction, the resistance should be extremely high).

Connect the silicon diode to the 10 μ A dc constant current source and DVM as shown in Figure 7-1. If you do not have a suitable constant current source available, a procedure to make one is outlined at the end of this experimental section. Be sure to use at least 50 cm of fine copper wire (no larger than 30 AWG) between the DVM and the diode. Turn on the constant current source and the DVM. You should get a reading across the diode of between 0.3 and 0.4 Volts. If you do not, check the wiring for shorts. If the DVM reads several volts, check to see if you have the correct polarity wired between the diode and the current source.

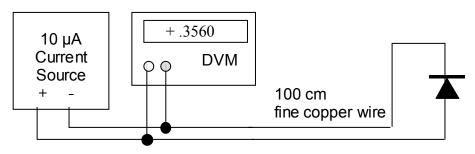


Figure 7-1 Diode setup.

<u>Temperature Calibration</u>: The next step is to calibrate the temperature response of the silicon diode. The diode voltage will increase linearly with decreasing temperature. Using an ordinary thermometer, determine the temperature of the laboratory in the vicinity of the diode (the value in Kelvin should be in the vicinity of 293 K for most rooms) and record the diode voltage at this temperature. This is our first calibration point for the sensor. Next, fill the Mr. SQUID[®] dewar with liquid nitrogen without the Mr. SQUID[®] probe in it. Slowly lower the diode into the liquid nitrogen. Once the diode is at the bottom of the dewar, push an additional 20 cm of the diodes wires into the liquid nitrogen (be careful to avoid skin contact with the liquid nitrogen). The voltage across the diode should increase to 0.9 to 1 volts. It may take several minutes for the diode voltage to reach a stable value. This is our second calibration point, at 77 K. Strictly speaking, we need only two calibration points for a linear sensor. However, if your lab has facilities to create a "dry ice and acetone" bath, you should place your diode in that bath to get a

195 K calibration point. (*Caution: These calibrations are for the diode, not the Mr. SQUID*® *probe. Do not expose the Mr. SQUID*® *probe to acetone.*)

On a piece of linear graph paper, plot the diode voltage as a function of temperature using your two (or three) data points. Draw the best-fit line to the data (you may wish to perform a least-squares-fit, if you are familiar with the technique) in order to produce a calibration curve that scales diode voltage with temperature. At this point, your diode is calibrated sufficiently to perform Mr. SQUID[®] experiments.*

<u>Mounting the diode</u>: Next, we need to attach the diode to the Mr. SQUID[®] probe in order to measure the SQUID chip temperature. Since we do not want the diode to be permanently mounted on the probe, we will use masking tape to hold the diode on the backside of the probe as shown in Figure 7-2. We want the wires to trail downward under the magnetic shield and out the bottom of the probe. Be sure to wait until you diode is at room temperature and is dry before taping it to your Mr. SQUID[®] probe. Also, try to avoid getting tape onto any part of the chip (front) side of the Mr. SQUID[®] probe. Place the Mr. SQUID[®] magnetic shield on the Mr. SQUID[®] probe so that the diode wires come out of the bottom. Stuff a small amount of cotton into the bottom of the magnetic shield to block the opening. This will improve the temperature uniformity of the region inside the magnetic shield.

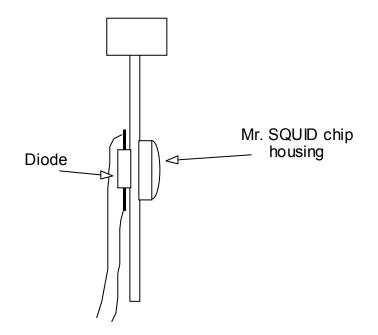


Figure 7-2 Attaching the diode sensor to the Mr. SQUID[®] probe.

Next, empty the Mr. SQUID[®] dewar until there is only 10-12 cm of liquid nitrogen left at the bottom (you can pour the unwanted nitrogen onto the floor, making sure to avoid people's feet, etc.) Place the Mr. SQUID[®] probe into the dewar as shown in Figure 7-3. You can use the binder clip in conjunction with the foam dewar cap that was shipped with your Mr. SQUID[®]

^{*}If you are performing this diode calibration for the magnetic shielding experiment (Experiment 5), you may now return to that section.

unit. If you no longer have the foam cap, a suitable replacement can be fashioned out of Styrofoam or cardboard, with a small hole large enough to fit the probe stick as shown in Figure 7-3. The binder clip is used to keep the Mr. SQUID[®] probe from sliding down into the dewar. You want to start with the Mr. SQUID[®] probe at the very top of the dewar.

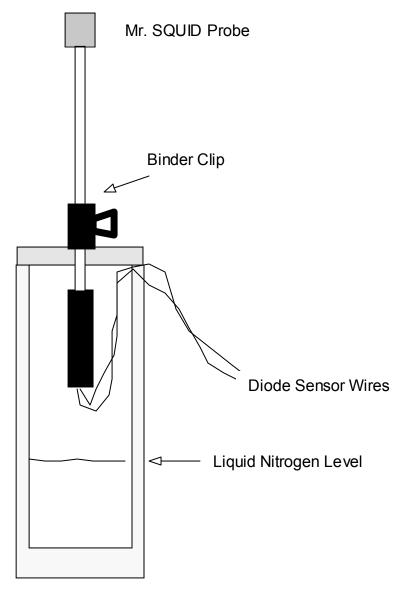


Figure 7-3 Position of the Mr. SQUID[®] probe inside the liquid nitrogen dewar.

At this point, connect the Mr. SQUID[®] probe to the Mr. SQUID[®] electronics box using the cable provided with the system, and connect the Mr. SQUID[®] electronics box to the oscilloscope with BNC co-axial connectors. Turn on the Mr. SQUID[®] electronics and set the unit to the *V-I* mode. Turn on the current source for the silicon diode and verify that it is indicating that it is near room temperature according to your calibration curve. You should see a straight line on the oscilloscope. The slope of this line is the resistance of the Mr. SQUID[®]. At room temperature this should be several hundred Ohms.

As shown in Figure 7-3, the chip end of the probe is to be suspended well above the liquid nitrogen level in the dewar. The paper binder clip is a handy way to hold the probe in a fixed position with respect to the foam cap on the dewar. Start with the end of the probe just under the level of the foam and follow the procedure below to record the temperature and resistance of the SQUID.

7.1.2 The Resistance vs. Temperature measurement procedure

STEP 1: Wait until the voltage on the diode stabilizes. This can take as long as 10 minutes.

STEP 2: Record the diode voltage in your lab notebook.

STEP 3: Record the slope of the V-I curve near the origin (V = 0 point) in your lab notebook.

STEP 4: Carefully loosen the binder clip while holding the Mr. SQUID[®] probe and lower the probe about 2 mm further down into the dewar.

This procedure is repeated until the probe has been lowered all the way into the nitrogen and has reached 77 K. The diode voltage should correspond to liquid nitrogen temperature at that point. The accuracy of the measurements and the number of data points you record is determined by your patience. If you want to spend less time, lower the probe in larger steps. You will get fewer temperature readings this way. However, if you do not wait until the diode voltage at each point is stable before recording the temperature, it will be very inaccurate.

Trouble-avoidance tip: Don't let the wires from the silicon diode extend far below the Mr. SQUID[®] magnetic shield. If they do, they might dip into the liquid nitrogen before the probe end does. If this happens, the thermal conductivity of the copper wires can cause the diode to indicate a temperature as much as 10-15 K lower than the Mr. SQUID[®] chip. This is something to watch for particularly when below 120 K.

The following data was taken using a glass encapsulated switching diode as the diode sensor. Data can be taken all the way down from room temperature, but in Figure 7-4, we only show the region around the transition temperature of the YBCO film, which will generally be near 90 K.

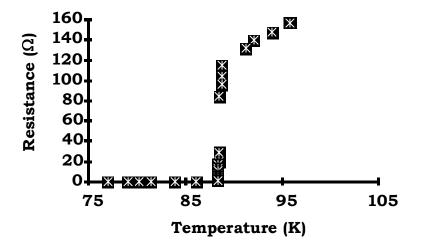


Figure 7-4 Resistance vs. temperature data for a Mr. SQUID[®] probe.