Superconductivity of In and Sn Samples

George Deamont
Sam Foreman

October 27, 2014

Abstract

Superconductivity is an important area of ongoing research with applications in many areas of science. In this experiment we attempt to observe the superconducting transition in thin films of indium (In) and tin (Sn) by measuring their electrical resistance as the they are cooled below their critical temperatures T_c. Finally, we measured the voltage across our samples as a function of the current for various values of the temperature near T_c, in order to determine how the critical current varies with temperature.

Theory & Background

Superconductivity is the quantum mechanical phenomenon in which a material has precisely zero electrical resistance, below a certain critical temperature, T_c. It was first discovered by the dutch physicist Heike Kamerlingh Onnes in 1911. There are two main types of superconductors, type 1 and type 2. Superconductors of type 1 exhibit superconducting properties only when cooled below T_c, and are well described by BCS theory [1]. Both of the metals studied in our experiment, In and Sn, are of this type. Superconductors of type 2 are alloys that are mechanically harder than those of type 1, and also exhibit various other properties.

In order to better understand the superconducting transition, it's useful to recall that electrical resistance occurs in normal metals occurs as a result of vibrations within the lattice, due to impurities and/or deviations from perfect symmetry. Also, in normal metals the resistivity decreases with decreasing temperature, yet always saturates to a finite value even as $T \to 0$; this is in contrast to superconductors, where below their critical temperature these scattering mechanisms no longer impede the motion of the current carriers. As an electron travels through the lattice of a superconductor, it slightly attracts the neighboring (positively) charged atoms, pulling a second electron in behind it.

As mentioned above, both samples studied in this experiment are of type 1, which is very well described by BCS (Bardeen-Cooper-Schrieffer) theory and provides three major insights.

1. The effective forces between electrons can sometimes be attractive in a solid rather than repulsive.
2. The "Cooper Problem," where two electrons outside of an unoccupied fermi-surface form a stable pair bound state, called a "Cooper pair."
3. The many particle wavefunction constructed by Schrieffer in which all wavefunctions near the fermi-surface are paired.

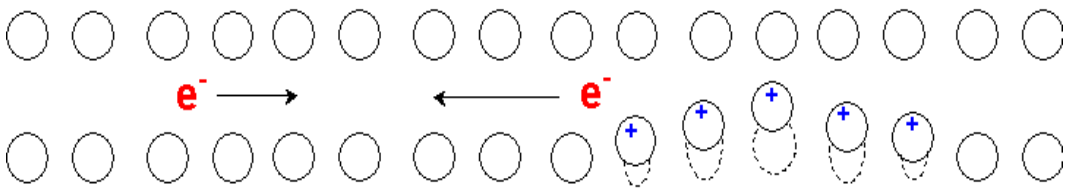

Figure 1: Illustration of electrons in a cooper pair traveling through the lattice of the metal

This thory ultimately relies on the assumption that superconductivity occurs when the attractive cooper pair interaction dominates over the repulsive coulomb force [2]. The attractive cooper pair is a weak electron-electron interaction mediated by a phonons in the lattice, as illustrated below. The work done by Cooper was a mounmental achievement in the completion of BCS theory and can be understood as follows. First, he showed that due to the fermi statistics of electrons, this paired electron-electron state can have an energy less than the fermi energy of the material ($\epsilon < \epsilon_F$), and that consequently when the temperature is low enough to ignore thermal energy,these

bound electron-electron states can form. This is one of the most important mechanisms by which the superconducting transition occurs, and it can be explained quantitatively by considering the following model.

Suppose we have two electrons interacting with the attractive cooper force at $T < T_c$. We can model this two-particle system by an anti-symmetric wave function with zero total momentum (i.e. the singlet state) of the form

$$\Psi(\boldsymbol{r}_0, \boldsymbol{r}_1) = \sum_{\boldsymbol{k}} g_{\boldsymbol{k}} e^{i\boldsymbol{k}\cdot(\boldsymbol{r}_1 - \boldsymbol{r}_2)} \left(|\uparrow\downarrow\rangle - |\downarrow\uparrow\rangle\right) \tag{1}$$

The asymmetric requirement forces $g_{\boldsymbol{k}} = g_{-\boldsymbol{k}}$, where $g_{\boldsymbol{k}}$ is a constant resulting from the electron-phonon interaction. Plugging this into schrodinger's equation $H\Psi = E\Psi$ gives

$$(E_{\boldsymbol{k}} - 2\epsilon_{\boldsymbol{k}}) = \sum_{\boldsymbol{k}' > \boldsymbol{k}} V_{\boldsymbol{k}\boldsymbol{k}'} g_{\boldsymbol{k}'} \tag{2}$$

If we use the assumptions from Debye's model of phonon interactions in a lattice [1], we can use the mean-field approximation

$$V_{\boldsymbol{k}\boldsymbol{k}'} = \begin{cases} -V & \text{for } \epsilon_F < \epsilon_{\boldsymbol{k}} < \epsilon_F + \hbar\omega_c \\ 0 & \text{else} \end{cases} \tag{3}$$

where ϵ_F is the fermi energy, and ω_c is a cutoff frequency characteristic of the metal in question. From this, we obtain

$$\begin{aligned} \frac{1}{V} &= \sum_{\boldsymbol{k}\boldsymbol{k}'} \frac{1}{2\epsilon_{\boldsymbol{k}} - E} \\ &\approx N_0 \int_{\epsilon_F}^{\epsilon_F + \hbar\omega_c} \frac{d\epsilon}{2\epsilon - E} \\ &= \frac{N_0}{2} \ln\left(\frac{2\epsilon_F - E + \hbar\omega_c}{2\epsilon_F - E}\right) \end{aligned} \tag{4}$$

And thus, we have

$$\frac{1}{2\hbar\omega_c}(2\epsilon_F - E) = \frac{1}{e^{2/N_0 V} - 1} \approx e^{-2/N_0 V} \text{ for } \frac{N_0}{V} \ll 1 \tag{5}$$

So, the energy of the pair satisfies $E = 2\epsilon_F - 2\hbar\omega_c e^{-2/N_0 V} < 2\epsilon_F$, as needed for the formation of the cooper pairs, which is a necessary condition for the superconducting transition.

Another important prediction of BCS theory is that there should exist a certain critical current, I_c, beyond which, the superconducting state breaks down. These theories predict that while in the superconducting state, there is precisely zero voltage across the material for any applied current of magnitude $I < I_c$. This allows us to measure the voltage across our sample as a function of applied current, from which we are able to determine the critical current. We may extract this value by determining the value of the current beyond which there is a noticable voltage increase without any increase in current, characteristic of the breakdown of the superconducting state. Further, by repeating these measurements for various temperatures near T_c we can plot I_c vs. T, and compare our results to the Bardeen equation as given below

$$I_c(T) = I_c(0)\left[1 - \left(\frac{T}{T_c}\right)^2\right]^{3/2} \tag{6}$$

From this equation we expect that the critical current should decrease monotonically with increasing temperature.

Procedure & Experiment

Thin Tin and Indium samples of various thicknesses were created to detect superconductivity, and to determine the superconductive transition dependence on thickness, as well as the critical current dependence on temperature. The following sections are detailed instructions on how to prepare samples and the cryostat, and guidelines for taking measurements.

Preparing The Substrate

The substrate refers to the glass slides that the Indium and Tin samples were resting on. A substrate was cut to a little bit longer than the size of the expected size of the metal sample. This was done using a diamond tipped pen, and a straightedge. The straightedge was used to mark the line along which the cut will be made. When cutting the substrate, it was best to run over the cutting edge numerous times with very little pressure in order to ensure a straight and clean cut.

In order to effectively observe the superconductive behavior of a sample and record reliable measurements, it was crucial that the sample be free of any contamination. This could be anything from scratches to dust on the surfaces of the metal and the substrate. Therefore, proper preparation of the sample included decontaminating the substrate. Placing the substrate in a small bottle of isopropyl alcohol, doing an ultrasonic cleaning of the substrate, and drying it using a compressed air duster achieved this. With the substrate completely submerged in the alcohol, the bottle was then placed in the ultrasonic cleaner filled with distilled water, and cleaned for about 3 minutes. The water level in the cleaner and the alcohol level in the bottle was in-line with each other in order for the cleaning process to be effective. After the cleaning, the substrate was removed from the bottle, and held in place with a pair of tweezers while air-drying it with a compressed air duster. Once

the substrate was completely cleaned (sometimes to achieve this, multiple cleanings were needed), it was placed inside a petri dish to minimize exposure to dust.

How to Make a Sample

The samples were produced inside the Denton 502A High Vacuum Evaporator by melting a chunk of each metal, and allowing the melted metal to diffuse toward the substrate above. In this way, the sample thickness can be precisely monitored, the quality of the sample can be drastically improved, and the shape can also be controlled with the use of a mask. Inside the vacuum chamber, there is a filament that acts as a link between a positive and negative electrode, which provides an electrical current to flow through it. The filament also has a small reservoir that houses the metal to be melted by the current that runs through it.

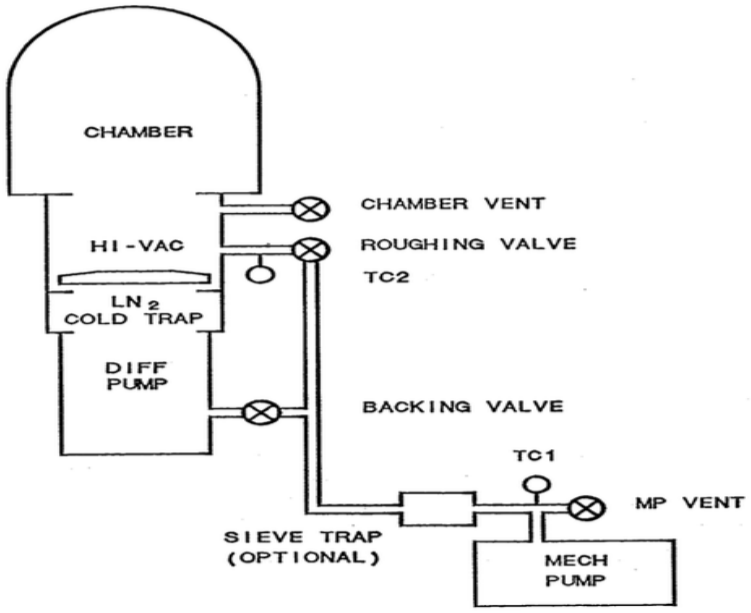

Figure 2: Diagram of the Denton 502A High Vacuum Evaporator

The following instructions are in reference to Figure 2 above. With the filament loaded inside the chamber, the vacuum was sealed using a vacuum leak sealant, and the Denton 502A was ready to pump out the air. First, a roughing stage of pumping was done. To do this, both vent valves were closed, the backing valve was closed, and the roughing valve open. Once the pressure in the chamber reaches around 100 milliTorr, high-vacuum pumping was done (the following steps are to be performed ONLY when the pressure on the thermocouple reads between 100 150 milliTorr). This was done by closing the roughing valve, opening the backing valve, and turning on the high-vacuum pump. At this point, the pressure reading on the Thermocouple Gauge cant be resolved to lower than milliTorr, so we turn to the Varian 880 Vacuum Ionization Gauge, which can resolve to Torr. In order to reach a vacuum pressure of 2-3 Torr, liquid nitrogen was poured into the cold trap, which helps to speed up the pumping process. Once the desired vacuum pressure was attained, our attention was steered towards the Inficon XTC, a device that monitors the thickness and deposition rate of the sample. Before starting the melting process, the Z-ratio of the desired samples respective element, whose value was looked up in a table, must be entered into the XTC.

The Z-ratios for Sn (Tin), and In (Indium), are 0.724 and 0.841 respectively. The filament was then turned on, after which the current source below the XTC powered on, and brought to around 120-Amps AC for starters. After a minute or two, the XTC displayed the deposition rate of the evaporation, at which point it was okay to open the shutter. A decent deposition rate is 8-10 /sec, so sometimes the current source had to be either decreased or increased accordingly. When opening the shutter, the Zero button on the XTC was also pressed at the same time to synchronize the actual thickness and the calculated thickness. Once the desired sample thickness was achieved, the shutter was closed, filament and current source turned off, and evaporator vented to bring the pressure back to ambient. This was done by closing the main and high-vacuum pump valves, and turning on the chamber vent valve. Once the Thermocouple Gauge reads atmospheric pressure, the chamber was opened, the sample carefully removed, and placed in a pastry dish to minimize exposure to the environment.

How to Prepare the Cryostat

In order to reach temperatures as low as 3-Kelvin in the cryostat, a requirement to observe superconductivity in these metals, liquid nitrogen and liquid helium where transferred into the cryostat. Figure 3 below shows the schematic of the cryostat used throughout the experiment, which we will refer to when discussing the preparation steps.

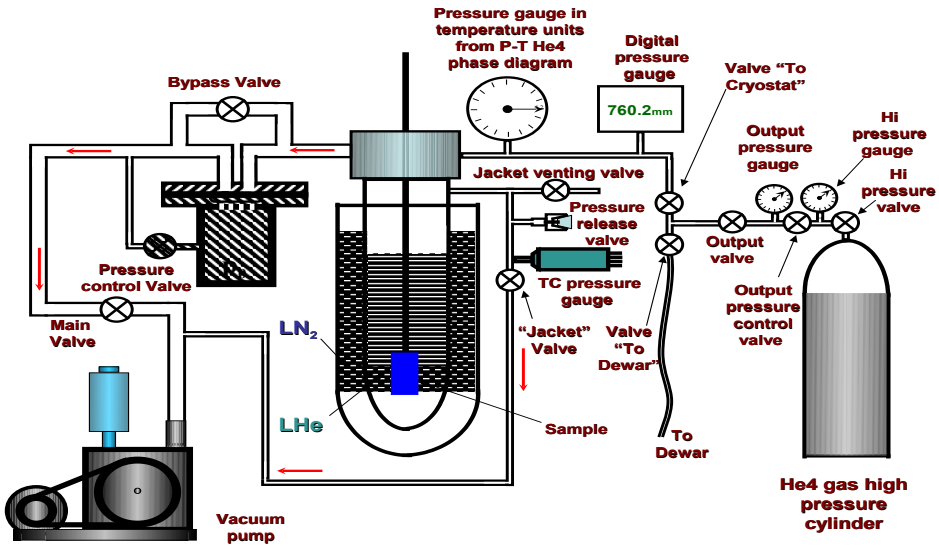

Figure 3: Schematic illustration of the glass cryostat used throughout the experiment

The liquid nitrogen barrier acted as an insulator, while the liquid helium provided the cold environment in which the sample resided in. The first step in preparing the cryostat was to pour the liquid nitrogen in the outer layer of the cryostat through the copper funnel. We poured enough liquid nitrogen to fill up the entire outer layer of the cryostat in order to provide the best insulation. The next step was to transfer the liquid helium from the Dewar to the cryostat through the use of a transfer line and He4 gas. This was done by first connecting the To Dewar hose to the He4 gas input of the transfer line, and inserting both ends of the transfer line into the cryostat and the

Dewar. With both ends connected, He4 was pumped into the Dewar at about 3 psi, which after a while forces liquid helium to transfer to the cryostat. On the computer, there is a data acquisition program with a section called Preparation of the Cryostat, which consists of two plots that monitor the pressure and temperature inside the cryostat. Once the temperature of the cryostat dropped to 4-Kelvin, we waited about 3 minutes for the liquid helium to fill up. When there was enough liquid helium inside the cryostat, the To Dewar valve was shut, which blocks He4 gas from entering the Dewar. After about a minute, the pressure from the He4 gas deteriorated, the To Dewar connection hose was disconnected, and the transfer line removed.

Preparing the Sample for Measurements

The dipstick is the main mechanism for which measurements such as voltage, current, and resistance were taken. The completed sample has a multi-level cross shape that allows multiple leads from the dipstick to be connected to the sample. For best practice, all ten possible connections to the sample were made. Figure 4 is a diagram of what a completed sample connection to the dipstick looks like.

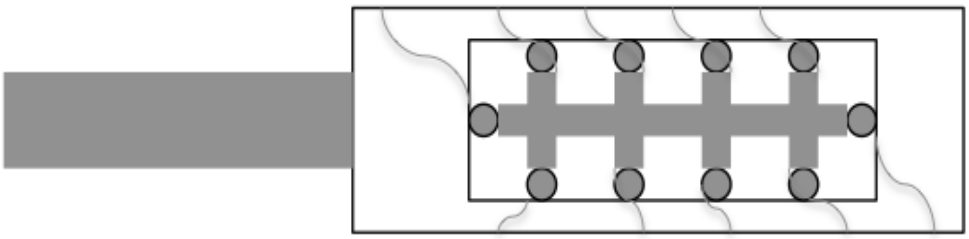

Figure 4: A completed sample connected to the dipstick

When connecting the copper leads to the sample, special fast-drying glue was used to quickly make connections with the sample, thereby minimizing contamination. Each copper lead on the dipstick has a corresponding N-value, and for each N-value, there is a corresponding R, B, and G value. These are used to make the proper connections from the dipstick to the volt and amp meters. This was a very important step because once the sample is inside the cryostat, it should not be removed for the remainder of the experimentation on the sample. Removing it can ruin the quality of the sample, therefore rendering it useless. These N and RGB-values were recorded on a separate sheet of paper to help us remember which connections went where. With the connections made, the dipstick was lowered into the liquid helium reservoir at the bottom of the cryostat, the electrical connection was made to the other end of the dipstick, and our attention was directed towards the R, G, and B nodes above the volt and amp meters. With the recorded N RGB-values at hand, the proper connections from the dipstick to the meters were made, and under the Preparation of the Cryostat program on the computer, the temperature of the cryostat was monitored until it became stable at 4-Kelvin. At this point, the temperature of the sample has reached ambient, and the sample was ready for testing.

Controlling the Temperature Inside the Cryostat

Throughout the data acquisition process, the temperature inside the cryostat was controlled using the pressure control, main, and bypass valves (see Figure 3 above), and optionally a heating unit to expedite the heating process. To decrease the temperature, we kept the main valve open so the vacuum pump can decrease the pressure, and thus the cryostat temperature. Slightly opening the pressure control valve achieved a slow temperature decrease, while opening the bypass valve achieved a coarse temperature drop. Once the desired temperature was been reached, the bypass and pressure control valves were closed to keep the temperature steady. To increase the temperature, the main and bypass valves were closed, and the pressure control valve opened. This causes the pressure, and thus the temperature, to increase. To speed up the heating, we often turned on the heater at the bottom of the cryostat. Once the desired temperature was acquired, the pressure control valve was shut.

Measurements

The first set of data measurements taken was the resistance of the sample as a function of temperature. For the purposes of observing a superconductive transition and determining the temperature at which this happens, measurements were taken over a narrow range of the expected critical temperature, usually around a 0.2 - 0.3-Kelvin range. Something that will be discussed in the next section is critical current, which is the applied current above which a superconductor will fail to transition to a superconductive state. As for the temperature sweep, it was much easier to control a temperature drop than a temperature increase. So for each resistance measurement on a sample, the temperature was first brought to a temperature above the expected critical temperature, either by increasing or decreasing the temperature depending on the initial temperature. Using the Preparation of Cryostat subprogram of the computer program can help ensure a stable temperature with minimal oscillations. Then, the Resistance vs. Temperature program was selected, which prompts the user for the current source, and how much current to use. An applied current of 2 5-mA was small enough to ensure superconductivity could be observed. Once everything was entered, the program would simply start taking resistance measurements. Using the pressure control valve, the temperature was then decreased very slowly, and data was collected until a transition was observed.

The second part of data measurements taken was the voltage of the sample at different temperatures as the applied current changed. To start off this measurement, the temperature was first decreased to below the critical temperature to keep the sample in the superconductive state. Once the temperature was steady, the V-I Curve subprogram was selected, which prompts the user once again for the current source, and then for a range of currents to apply to the sample, and the step-size. Usually the maximum and minimum currents were 100-mA and -100-mA, with a step-size of 1 2-mA. Sometimes smaller maximum and minimum currents were selected when the expected critical temperature was much smaller than 100-mA. Running the V-I Curve program simply changes the applied current while measuring the voltage at each step size iteration of the current. The resulting curve looked like a hysteresis loop with the current lagging behind on the way down from the maximum current and on the way up from the minimum current. This process was repeated for various temperatures to plot the critical current versus temperature.

Results & Analysis

For our first experiment, we attempted to measure the resistance versus temperature for the 250nm Indium sample prepared via the steps desribed above. Unfortunately, we were unable to observe a transition to precisely zero resistance, even with different current values and connection nodes. The closest transition we were able to observe for the indium sample is shown below.

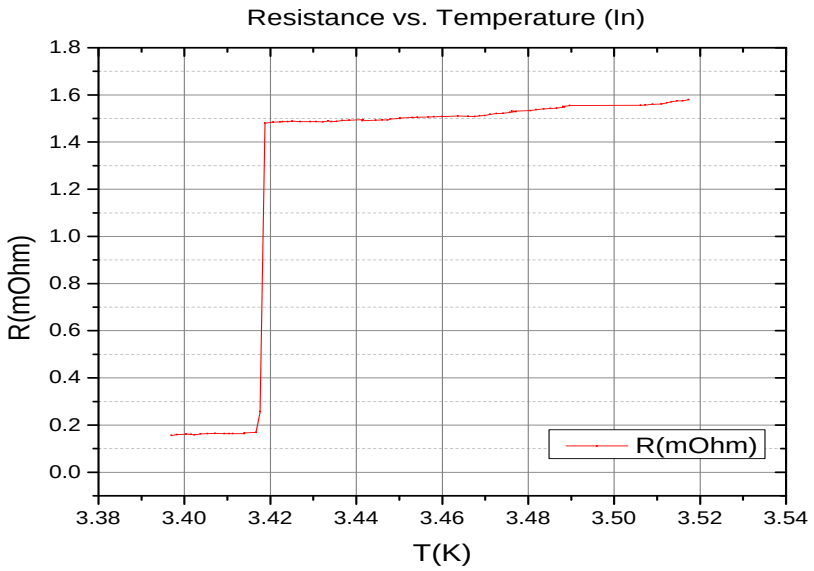

Figure 5: Resistance vs. Temperature for 250nm Indium sample

After having tried to repeat this measurement for various different configurations of node connections, even at various temperatures, we were never able to obtain the predicted superconducting transition in Indium. Because of this, we decided that we should use a tin sample instead, which is easier to work with. For this new tin (Sn) sample, of thickness 250nm, we were finally able to observe the superconducting transition. We observe this transition near the critical temperature of $T_c = 3.5$K, as shown below. In order to ensure that our results were consistent, we repreated this measurement twice with an applied current of 2A, as well as at 4A. Our results clearly indicate that this is indeed the superconducting transition, and our results are shown below.

Now, once we were able to verify that our tin sample had undergone the superconducting transition, we measured the voltage across the sample as a function of the applied current. For each value of the applied current, we see that when the current is small, the voltage is indeed precisely zero, which is indicative of the superconducting current. However, as predicted by the literature, there is a specific current called the critical current (I_c), beyond which the superconducting state fails. Once the current reaches the crtical current, we see that the voltage incereases without any increase in current. We illustrate this phenomenon for various temperatures near the superconducting temperature in the graph below.

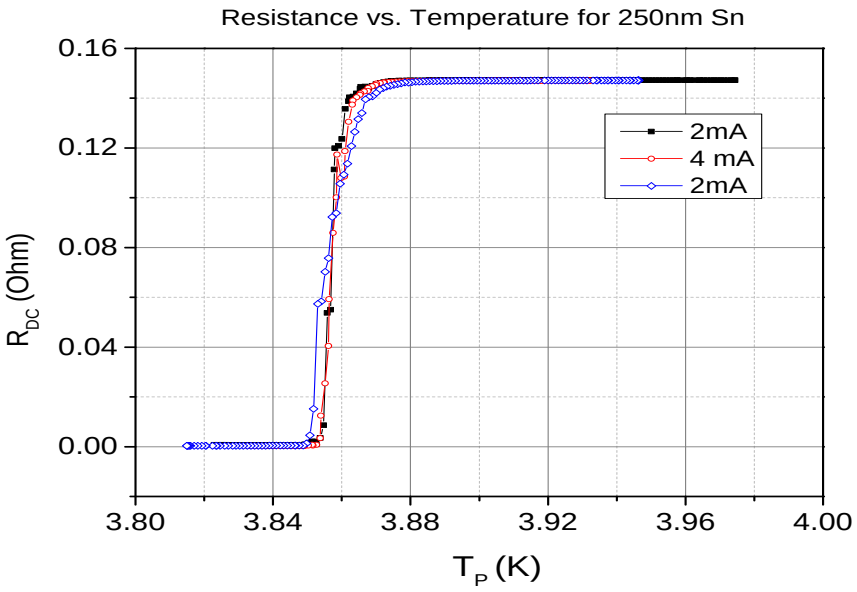

Figure 6: Resistance vs. Temperature for 250 nm sample of Sn, illustrating the superconducting transition

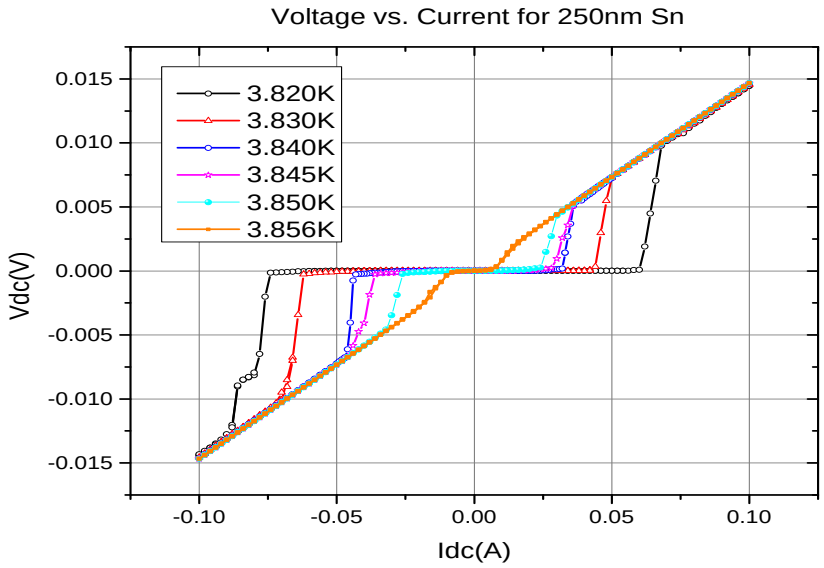

Figure 7: VI curve for 250nm sample of Sn for various temperatures near T_c

From this, we are able to obtain the critical current for each of the temperatures by manually observing the point at which we observe a sharp increase in voltage for infinitesimally small changes in the current. These values for the critical current as a function of temperature are included below. We can attempt to fit this plot via the Bardeen equation, which predicts that the critical current, I_c, should behave according to the equation given below. We can see that our results for the tin sample closely match those predicted by the literature, as indicated by the fit in the graph below.

$$I_c = I_c(0)\left[1 - \left(\frac{T}{T_C}^2\right)\right]^{3/2} \tag{7}$$

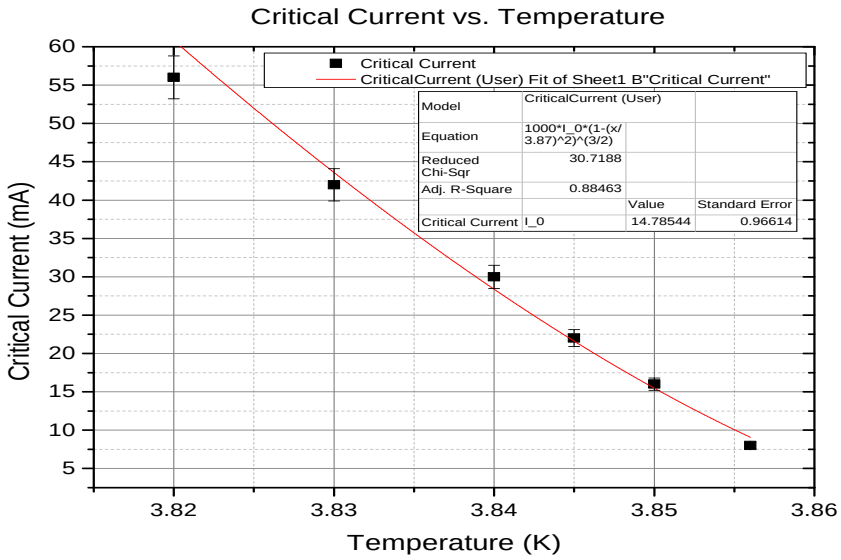

Figure 8: Resistance vs. Temperature for 250nm Indium sample

Temperature (K)	Critical Current (mA)
3.856	8
3.850	10
3.845	16
3.840	21
3.830	40
3.820	56

Table 1: Values of the critical current for various temperatures in a 250nm Sn sample

Conclusion

Samples of Tin and Indium were studied to realize superconductive behavior in these metals. A superconductive transition was observed for each metal, although the Indium sample resistance dropped a substantial amount, but never to the full superconductive state. Our findings suggest that the critical temperatures for Tin and Indium, and critical currents for Tin, are in close agreement with the theory presented in the literature. Using the same measurement techniques involved in this experiment, super-cooling to cryogenic temperatures using liquid helium can in principle provide a way to probe superconductive behavior in other superconductors.

References

1. J. Bardeen, L. N. Cooper, and J. R. Schrieffer, "Theory of Superconductivity", Phys. Rev. 108, 1175 (1957).

2. Cooper, Leon (November 1956). "Bound Electron Pairs in a Degenerate Fermi Gas". Physical Review 104 (4): 11891190.