A look at the critical temperature of a Yba₂Cu₃O₇ ceramic superconductor

Bradley C. Thomas

Physics Department, The College of Wooster, Wooster, Ohio 44691

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This experiment investigates the critical temperature of a high T_c , type II, $YBa_2Cu_3O_7$ superconductor. By lowering the ceramic disk into liquid nitrogen, a critical temperature is reached below which the material exhibits no electrical resistance, a phenomenon known as superconductivity. This critical temperature can be measured by passing a current through the ceramic disk while simultaneously measuring its temperature. When the resistance falls to zero, the critical temperature has been reached. Plotting this data in Igor Pro Carbon, the temperature at the point where the superconductor ceased to be zero was measured for each run and the values were averaged together. The experimental critical temperature for the superconductor was measured to be 93.3 +/-0.7K, with an error of less than a percent from the known T_c of 93K..

INTRODUCTION

In the absence of all electrical resistance, current could theoretically flow for an indefinite period of time, losing none of its potency. This phenomenon, while seemingly too good to be true, has been known to science for almost a hundred years as superconductivity and is regarded as one of the most fascinating and remarkable physical properties in our universe. While superconductivity seems to be the solution to all the problems associated with energy transportation, it does not come without a cost, namely temperature. The temperatures required to cause a superconductive material to become superconducting reside around that of liquid nitrogen, about 77K. However, while these temperatures seem uncommonly cold, cold enough to kill most living organisms, they have dramatically risen since their discovery by H. Kammerlingh-Onnes in 1911. A result of his investigations leading to the liquefaction of helium, Onnes found that simple metals such as mercury, lead, and bismuth would lose all their resistive properties when dipped in the liquid helium. At the unbelievably chilly temperatures of liquid helium (around 4K) the benefits of superconductivity were dwarfed by the immense cost of maintaining that temperature. However, in 1986, researchers at an IBM laboratory in Switzerland discovered that a specific type of ceramic from a class of materials called

perovskites became superconducting at temperatures above 35K. This remarkable adiscovery earned the Swiss scientists a Nobel Prize in 1987 and sparked new life into the prospect of a practical, cost-effective superconductor. Almost one year after the Swiss team discovered their ceramic superconductor, another perovskite ceramic material was found to be superconducting, but at 90K. The gravity of this discovery caused another great stir in the scientific community because now liquid nitrogen could be used as a refrigerant rather than liquid helium. Besides liquefying at a much higher temperature (77K as opposed to 4K for helium), nitrogen is more abundant in the atmosphere and is therefore much cheaper to gather and condense. As of today, superconductors have been fabricated with critical temperatures as high as 125K, a threshold that will no doubt be transcended with further research.

EXPERIMENT

This experiment requires the use of liquid nitrogen to cool a $YBa_2Cu_3O_7$ ceramic superconductor below its critical temperature. The superconductor itself, purchased from Colorado Superconductor Inc., is pre-equipped with a "six point probe" necessary to measure its T_c (discussed further below). A Keithley 199 system DMM/Scanner was used to measure the voltages through the superconductor while a Kepco Current Regulator CA-3 was used to provide the necessary current. The "six point probe" contained within the superconductor provided the electrical connections used to measure the critical temperature. A schematic of the connections is shown below in Figure 2.



FIG. 2. : This is a diagram of the superconductor probe. The Keithley 199 System DMM/Scanner reads three voltages, V1, V2, and V3, from the probe. V1 is the measured voltage across a 4.833Ω resistor representing the total current passing through the superconductor. V2 is the voltage used specifically to determine the resistance of the ceramic superconductor. V3 represents the voltage across the thermocouple, used to measure the temperature of the brass casing.

Once the probe reached a temperature above criticality, LabView could be initiated and the superconductor could be lowered back into the bath. After a sufficient number of data were recorded at zero resistance, the superconductor could be brought out of the liquid nitrogen and allowed to warm just above the surface. This allowed excess liquid to drop back into the dewar as well as allowing the $YBa_2Cu_3O_7$ disk and brass casing to gradually warm. After the superconductor warmed to a temperature several degrees above T_c , the data collection was stopped and the current through the probe was reduced. In all, six data runs were taken, two for each of three different currents, 161mA, 111mA, and 51mA.

Loading the general text into Igor, each of the data runs were graphed, first as temperature and resistance vs. time and then combined as resistance vs. temperature (see figures below).



FIG. 3. This was the first data run taken in this experiment. Notice a "dip" in the resistance below zero corresponding to temperatures above that of liquid nitrogen (occurring while the superconductor was warming). The sharp rises in resistance signify the critical temperature of the superconductor, beyond which it is no longer superconducting. The discrepancy between the two T_c s is most likely due to inexperience in collecting data and allowing the probe to warm too quickly.



Fig. 4. This graph depicts data taken with a current of 111mA through the probe. Again the pronounced "dips" in resistance can be seen just before critical temperature. The T_c s have better agreement in this run.

The main collection of points occurring at around 85K (in both Figure 4 and 5) represents the superconductor at liquid nitrogen temperatures. However, because N_2 liquefies at 77K, this suggests that the thermocouple is off by some 10K (discussed in error section).



Fig. 5. This is a graph of data taken with a current of 51mA. Once again the "dip" in resistance is prominent and the critical temperatures agree well, possibly due to the lower current.

Surprisingly, it was found that the average resistance of the superconductor while immersed in the liquid nitrogen is negative for all runs. While close to zero, there is obviously some residual energy being picked up by the superconductor or the Keithley 199 DMM/Scanner causing the negative resistance. The standard deviation of these points is small, informing us that the error propagated by the Keithley 199 DMM/Scanner or superconductor is constant and can therefore be accounted for.

An extremely curious feature that deserves greater scrutiny is the "dip" in resistance, occurring when the superconductor begins to warm above liquid nitrogen temperatures, just before reaching T_c . After the $YBa_2Cu_3O_7$ disk is taken out of the dewar, it should still exhibit superconducting properties as it warms from 77K to its critical temperature somewhere above that. However, we see that, in all cases, as soon as the superconductor begins to warm, the resistance rises slightly above zero then abruptly falls below zero. As soon as the resistance bottomed out, it promptly rose above zero and continued until a certain temperature, at which the resistance stabilized and continued a steady incline as the superconductor warmed. The average values for the bottom of the "dip" as well as their standard deviations are given in Table 1.

Current	161mA		111mA		51mA	
	Run	Run	Run	Run	Run	Run
	1	2	1	2	1	2
$\overline{R}(m\Omega)$	-0.09	-0.23	-0.54	-0.53	-1.0	-1.1
Standard Dev.	0.01	0.03	0.01	0.04	0.08	0.04

Table 1. \overline{R} is the average resistance at trough of "dip"

From *Table 1* it can be seen that each value is well below zero resistance and each "dip" bottomed out between the temperatures of 96.5 and 102.2K.

This phenomenon is, as of yet, unexplained and proves to complicate the process of finding a critical temperature for the $YBa_2Cu_3O_7$ superconductor. Is it that the critical temperature is reached as soon as the superconductor leaves the liquid nitrogen but because of this phenomenon is delayed, or is it that this phenomenon is simply some precursor to the critical temperature, which is actually reached once the resistance passes zero? The subtle rise before the dip suggests that the critical temp. might exist before the phenomenon. However, there might also be some interaction between the $YBa_2Cu_3O_7$ disk and the brass casing which only occurs at this temperature interval.



Fig. 6. This graph shows a direct correlation between the depth of the "dip" (amount of negative resistance) and the current. Notice that at larger currents across the superconductor the effect is less prominent.

To solve this problem, points before and after the phenomenon were examined. The resistance point directly before the "rise and dip" was recorded along with its corresponding temperature. Similarly, the point after the phenomenon that fell closest to the zero-resistance line was recorded along with its temperature. Because all points of this sort fell within $0.1 m\Omega$ of the zero line, they were taken as the point of critical temperature with an error of +/-1K (discussed in error section). Thus, two values were taken for $YBa_2Cu_3O_7$'s critical temperature. The first possible critical temperature ranged between 88.8 and 90.3 (+/-1K). While seemingly reasonable, we must remember to take into account the ten Kelvin deviation of the thermocouple. As mentioned earlier, nitrogen liquefies at 77K, but the lowest temperature measured by the thermocouple was consistently some ten degrees higher. Therefore, we can recalibrate the thermocouple with the known temperature of the liquid nitrogen by subtracting ten. Thus, the actual first possible critical temperature reduces to 78.8 to 80.3 (+/-

1K), a temperature range much less plausible. However, the second possible critical temperatures occurring after the "dip" phenomenon ranged between 98.9K and 105.4K (+/-1.5K). Subtracting the thermocouple error yields a range of 88.9 to 95.4 (+/-1.5K), which is much more likely for the T_c of this superconductor. We can average the temperature values to determine the mean T_c of this material.

$$\frac{619.7}{6} = 103.3 - 10 = 93.3 + -0.7 \text{K}$$

This value suggests that the "dip" phenomenon did occur while the superconductor was below critical temperature and that the T_c was not delayed by this. The known T_c of the $YBa_2Cu_3O_7$ ceramic is 93K¹, almost exactly what the experimental value was measured to be with an error of less than a percent. Also, the known critical temperature is well within the error range of the experimental T_c .

Kdithley 199 DMM/Scanner has a resolution to 4 significant digits, which reduces to only two when the disk becomes superconducting, though is small enough to be considered negligible for the collected data. Likewise, small discrepancies in the actual voltage as read by the Keithley 199 DMM/Scanner that occurred due to resistance in the wire connecting the probe to the superconductor were small enough not to directly affect the data and can therefore also be considered negligible.

The greatest error propagated due to thermocouple, which was calibrated to a temperature ten Kelvin above the actual temperature. While this had to be taken into account, it did not greatly affect the data because of the known temperature of liquid nitrogen, with which we could compare. Furthermore, the ambient temperature of the room was not closely monitored while the data was being collected, possibly causing some error in the measured superconductor temperature. Thus, an error of +/-1K was placed on all temperature readings to account for some slight temperature fluctuations. This error was compounded when the critical temperatures were measured for points after the "dip" phenomenon. Every point used in this examination fell (or was calculated to be within) $0.1 m\Omega$ of the zero-resistance line. Because the temperature fluctuated no more than 0.5K for +/- $0.1 m\Omega$, the error for these points was estimated to be 1.5K.

Conclusion

In this experiment the critical temperature of a $YBa_2Cu_3O_7$, high T_c , type II, ceramic superconductor was measured to be 93.3 ± -0.7 K, having an experimental error of less than a percent. The accuracy of this value was quite unexpected considering the unexplainable "dip" phenomenon where the resistance fell below zero. This phenomenon occurred just after the superconductor was taken out of the liquid nitrogen bath and began to warm. At between 96.5 and 102.2K for every run, the resistance rose slightly above zero then fell to as much as $-1.1 m\Omega$ before reaching the critical temperature. The cause of this phenomenon is, as of now, unknown, it will be referred to as the "Thomas-Merriman Dip Effect".

This experiment could be improved by devising a system in which the liquid nitrogen bath could be covered while the superconductor is cooled. An insulated cover would allow the air inside the dewar to be drastically colder, therefore allowing the superconductor to gradually cool to a lower temperature before it touched the liquid nitrogen. Likewise, it would be interesting to attempt to regulate the temperature of the superconductor between 97 and 102K to further examine the Thomas-Merriman Dip Effect and whether the resistance would be sustained below zero.

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- ¹David R. Lide, Annu. CRC Handbook of Chemistry and Physics. 77th edition **12**-84 (1997).
- ²C.P. Poole, Jr., T. Datta, and H. A. Farach, Copper Oxide Superconductors, (1988).

³Thomas P. Sheahen, Introduction to High-Temperature Superconductivity, (1994).

⁴McGraw-Hill Encyclopedia of Science and Technology: Vol. 17, p.632-648, (1992).