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Modifying the Composition of a High Temperature Superconductor

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Modifying the Composition of a High Temperature Superconductor

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By

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Modifying the Composition of a High Temperature Superconductor

By

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ABSTRACT

The purpose of this research was to recreate a known superconductor, $YBa_2Cu_3O_x$, and modify it in an attempt to raise its transition temperature. A superconductor is any material that excludes magnetic flux from its interior when at a temperature below its transition temperature. A “high temperature” superconductor refers to material that shows superconductivity at or above the temperature of liquid nitrogen temperatures (77.15K).

Here we were successful in reproducing the original $YBa_2Cu_3O_x$ and measuring its critical temperature; However, our efforts at raising the transition temperature have, as of yet, not been successful. The processes for fabricating these superconductors are explained here.

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INTRODUCTION

The temperature at which a material becomes superconducting is called its superconducting transition temperature. A major interest in materials science is finding materials that have a superconducting transition temperature near room temperature. This would be of great significance to modern technology in that it would enable the near lossless transmission of electricity and increase the sensitivity of various sensors. At present the highest temperature superconductors that are currently available hover anywhere from 133.15K to 143.15K.

Compared to standard room temperature (293K or 20°C), these temperatures might seem extremely low, but these transition temperatures have increased greatly in the 100 plus years since the discovery of superconductivity. At the beginning of the 20th century the temperature dependence of resistivity at temperatures approaching absolute zero was unclear and being debated by theoreticians. In 1908, Kammelingh Onnes was able to liquify helium making it possible to cool materials to around 1K. Three years later in 1911, Onnes discovered that, when pure mercury was cooled to 4K, it showed no measurable signs of resistance.[1] This was unprecedented as having no resistance went against the expectation at that time in history.

The state of zero resistance was named the superconducting state, and researchers began to look at the other properties a conductor had while in this state. W. Meissner and R. Ochsenfeld found in 1933 that when in the superconducting state the material completely expels all magnetic flux including any external field.[2] This is interesting since this is different from the classical predictions of a perfect conductor. This effect (dubbed the Meissner effect) is how superconductors can be distinguished from normal conductors since this is easier to verify than having zero resistance, even in the case of high temperature superconductors.

In the early 20th century, research into superconductivity was mostly experimental dealing with observations in the lab but a theory was slowly being developed. In 1935, F. and H. London proposed the London theory which attempted to apply classical electrodynamic theory to superconductors.[3, 4] Quantum mechanics was incorporated into the theory by Pippard, Ginzburg, and Landau in the early 1950's.[5, 6] This theory was relevant due to its success in predicting type I and II superconductors. In 1957, Bardeen, Cooper, and Schrieffer were able to formulate a quantum mechanical theory that matched experimental results and completely described the key features of superconductivity.[7] This is the current working theory for metallic superconductors.

It was observed that not all superconductors exclude magnetic fields in the same way. Some, called type I superconductors, fully exclude the magnetic field from their interior until a high enough value of magnetic flux is induced, breaking the superconductivity. On the other hand, type II superconductors gradually let the magnetic field in after a lower bound of magnetic flux is reached. Type I superconductors are commonly pure elemental metals, as opposed to alloys, which is why type II characteristics were typically attributed to impurities within the material. In the 1960's, Abrikosov proposed that these were actually two distinct classes of superconductors and are characteristic of the material instead of the result of impurities.[8, 9]

Since the initial discovery of superconductors, researchers have strived to increase the transition temperature. Traditionally, Type II superconductors have higher transition temperatures than their counterpart with niobium holding the record for type I at 9.3K. For type II superconductors, the transition temperature was steadily raised from 15K to 35K until 1987 when a range of superconductors were found to have transition temperatures in the 90K range.[10, 11, 12] One would expect the process of finding higher temperature superconductors to consist of understanding the underlying structure of a superconductor and trying to fit the compounds that would create a higher temperature to that structure. However, as of now it is not possible to predict what compound will be a superconductor nor what temperature it might transition due to a lack of understanding on what structural characteristics uniquely determine a superconductor. Most research efforts have been reduced to a "guess-and-check" routine, with some research being conducted to find said structural characteristics intrinsic to superconductors.

THEORY

The main characteristics of a superconductor are its ability to exclude magnetic flux throughout its interior and that it has zero resistance when superconducting. The physical features that govern these phenomena are the material’s transition temperature and value of the critical magnetic flux. The transition temperature of a superconductor is the temperature at which the material starts to superconduct. Traditionally, these values are within the 10K (-263.15°C) range and lower. The critical magnetic flux is the maximum flux that a superconductor can expel before it reverts to the normal state. This is the key difference between Type I and Type II superconductors. Type I superconductors have one critical value that, when reached, completely stops the material's superconducting state. Type II superconductors have a lower critical value and an upper critical value. When the lower critical value is reached, the material becomes semi-superconducting with pockets of the material being in the superconducting state. The number and size of these superconducting pockets exponentially decreases until the upper critical value is reached, and it stops superconducting altogether.

Both the transition temperature and critical flux value of a superconductor are of significant importance when considering modern technology. Researchers have worked towards finding what can affect these values in a specific material. This has proven to be fairly challenging since there is not a defined dependence between the transition temperature and a superconductor's composition. This effectively leaves two avenues for research, modeling, or experiments. This "guess-and-check" method is not without merit however, since liquid nitrogen temperature superconductors were found through these means. It was also discovered that the transition temperature can be raised based on quenching rate, heat treatment, and concentration of the products.

In order to fully understand the superconducting state's zero resistance, it is important to understand what resistance is on the fundamental level. Resistance comes from a break in the lattice structure's periodicity. When electrons move through a material they do so as a plane wave. This plane wave's period is closely related to the period of the lattice structure. If the lattice structure is not uniform and distance between each atom changes, the plane wave will be scattered, thus losing energy. The more chaotic a material's lattice structure, the higher resistance it will have. Thermal vibrations also play a large role in resistance since, the more thermal energy present in a material, the more the atoms are to oscillate from their lattice positions. This is the reason for conductors having lower resistances at lower temperatures. However, this does not fully account for the sharp drop in resistance when a superconductor is brought to its transition temperature.

The BCS theory is the accepted modern theory to fully explain superconductivity. As the electron moves through the lattice, it distorts the structure by attracting the positive nuclei around it. This creates areas of higher positive charge, which in turn attract an additional electron with opposite spin. This electron pair is referred to as a Cooper pair and only appears at sufficiently low temperatures since this pairing is otherwise broken by thermal vibrations of the lattice. It is important to note that a Cooper pair demonstrates boson-like characteristics and thus does not have to obey the Pauli exclusion principle. This becomes relevant when there are a large number of Cooper pairs present in a material since these form a Bose-Einstein condensate. When in this state the electron pairs will fill the lowest quantum state. The energy required to break one Cooper pair in this state is then equal to the energy required to break all pairs in the condensate. This formation of the Bose-Einstein condensate is the reason that no resistance is present in a superconductor.

As previously mentioned, superconductivity is not the same as just zero resistance. The difference between these two states is important to understand since there are interesting discrepancies between the classical interpretation of zero resistance and superconductors. Assume that there was a material that was able to obtain zero resistance, let this be called a perfect conductor. The difference between this perfect conductor and a superconductor would be in their response to a magnetic field. Magnetic flux in the interior of a superconductor is known to be zero; however, this is not the case for a perfect conductor. The magnetic flux through a material is defined as $\Phi = \int \mathbf{B} \cdot \hat{\mathbf{n}} \, da,$ with $\mathbf{B}$ being the magnetic field and $\hat{\mathbf{n}}$ being the unit normal vector to the area of the surface the field is passing through. Nature abhors change and tries to keep the magnetic flux in a material constant. If $d\Phi/dt \neq 0$ inside a material, a current I will then form in the material moving in such a direction that the magnetic field produced from this new current will oppose the change in magnetic flux.[1, 3, 5] The electromotive force produced is then equal to $-\mathbf{A} \cdot d\mathbf{B} / dt$. The total emf would then equal the voltage of the resistor plus the back emf, giving the equation:

$$-\mathbf{A} \cdot \frac{d\mathbf{B}}{dt} = RI + L\frac{dI}{dt}$$

With *R* being the resistance and *L* being the inductance of the circuit. Now for a perfect conductor $R = 0$ would give:

$$c = LI + \mathbf{A} \cdot \mathbf{B}$$

where *c* is some constant. This implies that a perfect conductor’s magnetic flux will stay constant, meaning that if a perfect conductor is in a magnetic field and it is turned off, a current will form and continuously flow in such a way that maintains the magnetic field. This also means that if a magnetic field is applied to a perfect conductor when the magnetic flux was initially at zero, the material will repel the applied field, maintaining its flux.

It has been shown that a perfect conductor’s magnetic flux depends on its initial conditions. The Meissner effect asserts that a superconductor will never have interior magnetic flux regardless of its initial conditions. This begs the question; how does this change the mathematical theory? Classically, a perfect conductor follows Maxwell's equations, the relevant one relating to flux being:

$$\frac{d\mathbf{B}}{dt} = K\nabla^2 \frac{d\mathbf{B}}{dt}.$$

The solutions to this equation give **B** as some form of exponential decay as it penetrates through the material. Far below the surface then, the magnetic flux density has not been disturbed, thus keeping it constant in time; however, this contradicts the Meissner effect since no magnetic flux is allowed inside a material that is in a superconducting state. To account for this effect, F. and H. London developed the equation for superconductors.

$$\mathbf{B} = K\nabla^2\mathbf{B}$$

$YBa_2Cu_3O_x$ is a type II superconductor that is classified as a cuprate. Cuprates are a class of high temperature superconductors that have yielded the highest temperature superconductors currently known. A cuprate's composition is alternating layers of copper and some other metal, common examples being lanthanum and barium. BCS Theory is not able to explain cuprates and how they work is still up for debate.

SAMPLE PREPARATION AND MEASUREMENT

Sample Preparation

The YBa_2Cu_3O sample was made by thoroughly mixing and grinding the dry powders together then firing the mixture in a kiln at 871℃ for 15 hours under flowing oxygen at a rate of 0.2 Liters per minute. Although the sample in question was produced with the times and temperature mentioned above, results have been seen for the same temperature for as little as 8 hours.

Table 1: The makeup for approximately one cubic centimeter of the YBa_2Cu_3O preparation before firing.

	# Moles of constituents	Molar mass (g/mol)	Mass per Mole SC (g)	Density (g/cm^3)	Volume per mole (cm^3/mole)	Mass per unit volume of SC (g/cm^3)	Melting Point (°C)	(°F)
O_3Y_2	1	225.81	112.90	5.01	22.54	0.741	2440	4420
$BaCO_3$	2	197.34	394.68	4.29	92.00	2.591	811	1,492
CuO	3	79.54	238.63	6.31	37.82	1.566	1,326	2,419
Y		88.90						
Ba		137.32						
Cu		63.54						
O		15.99						
$YBa_2Cu_3O_x$					152.35			

The Y_2O_3 was 99.99% pure, $BaCO_3$ was 99.9% pure and the CuO was 99% pure. All were from Alfa Aesar. The kiln used was an Evenheat Kingpin 88 with a Set-Pro control system. The firing temperature of the sample did not necessitate a high degree of accuracy. The kiln was modified in order to ensure an oxygen rich environment by drilling a 1/8-inch hole through the bottom of the kiln’s refractory material. A 1/8-inch diameter stainless steel tube was then inserted through the hole and connected to an oxygen tank nearby. The connection between the pipe and oxygen tank was airtight, but the end of the pipe to the kiln was open to enrich the oxygen environment. The samples were contained in an alumina crucible with a larger alumina crucible covering the sample and oxygen input port.

The constituents were measured and ground to a fine powder, the mixture was fired in an alumina crucible in a range of eight to fifteen hours at 982℃. After firing, the sample was pressed in a cylindrical die at room temperature at a pressure of 41 MPa and fired again at 982℃ under flowing oxygen at a rate of 0.2 liters per minute. The sample was brought up to the firing temperature and held there for between five and eight hours and then cooled to room temperature over a span of 2 to 3 hours. If the sample was fired for too long, it would become glassy and not become superconducting. After the second firing, which is called sintering, the sample is ready for measurement. This process was done for several different compounds, as shown in Table 2. All samples listed were initially fired at 982℃ for a minimum of 8 hours. They were then sintered at 982℃ for a minimum of 5 hours. The flow of oxygen for each firing was at 0.2 liters per minute.

Since our goal was an attempt to raise the superconducting transition temperature, if the sample did not exhibit superconductivity at 77K, no other measurements were made.

Table 2: This table shows a list of compounds and the exact measurements made for each sample in grams. Measurements accurate to .0005g.

	Y_2O_3	$BaCO_3$	CuO	$BYBa_2Cu_3O_x$	La_2O_3	Pb_3O_4	CdO
$YBa_2Cu_3O_x$	0.8153	2.8496	1.7236				
$B_{.5}YBa_2Cu_3O_x$	0.8156	2.8491	1.7229	0.0269			
$B_{.05}YBa_2Cu_3O_x$	0.8154	2.8497	1.7228	0.0037			
$La_{.01}Y_{.99}Ba_2Cu_3O_x$	0.7343	2.5917	1.5727		0.013		
$Pb_{.25}YBa_2Cu_3O_x$	0.6952	2.4299	1.469			1.0557	
$CdYBa_2Cu_3O_x$	0.7388	2.5825	1.5615				0.8402
$Cd_{.01}YBa_2Cu_3O_x$	0.8143	2.8466	1.7212				0.0093
$YCd_2Cu_3O_x$	1.3519		2.8574				3.0751
$YCdCu_3O_x$	1.6318		3.4489				1.8559

Sample Measurement

To measure the transition temperature of the samples, a transformer was made with the sample as the core. Two 24-gauge copper wire coils wrapped tightly around the core with each winding having the same number of turns. One coil was the primary of the transformer and the other acted as the secondary. When the sample was in the normal state, the magnetic flux through the secondary windings were coupled to the primary but when the core became superconducting the magnetic flux was excluded from the core and the coupling between the coils became weak and the output potential of the secondary dropped dramatically.

Mounting the Sample

Samples were placed on a printed circuit board and the leads of the primary and secondary coils were soldered to the board. Already mounted on the circuit board was the thermometer, a calibrated Lakeshore model DT-670-SD-70L silicon diode. The current and potential leads of the diode were soldered to the board for a four-probe method of determining the potential across the diode. The diode was supplied with a constant 10 μA from a Keithley Model 200 programmable current source. The board and all of the leads were suspended inside of a cryostat. To ensure that the sample and thermometer were at the same temperature, the cooling and warming times were extended over an hour or two. This was accomplished by evacuating the cryostat sample chamber. In addition to decreasing the rate of heat exchange, the vacuum also prevented water vapor from condensing on the sample and the electrical contacts.

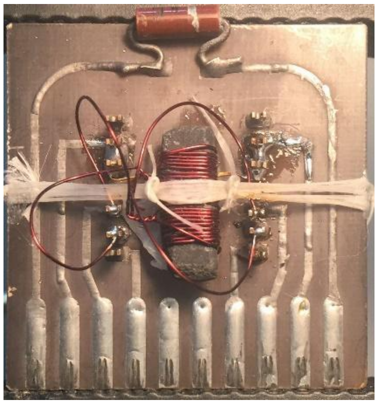

Figure 1: Sample wrapped with primary and secondary coils and mounted on circuit board.

A Stanford Research Systems Model SR830 DSP lock-in amplifier supplied a 700 *Hz* AC current for the primary coil and the leads of the secondary coil were connected to the input of a lock-in-amplifier. The two coils were in close proximity and an AC current in the primary generated a time-varying magnetic flux that was coupled to the secondary coil through the sample being measured This time varying flux generated by the primary coil induced an emf in the secondary coil. When the core became superconducting, the Meissner effect excluded the flux from the core and restricted the flux to the diameter of the wires themselves. This flux exclusion caused the output of secondary to drop. When the temperature changes were reversed and the sample allowed to warm, the temperature of the core increased and as the temperature of the sample approached the transition temperature, the flux began to thread through the secondary coil. Above the transition temperature the flux exclusion ceased, and the two coils were coupled again as indicated by the sudden rise in the output potential difference across the secondary coil. The output potential difference across the secondary coil was small, which necessitated the use of the lock-in-amplifier. The lock-in-amplifier supplied the current to the primary coil at the same frequency as the output of the secondary coil wrapped around the sample but with a possible phase shift between the two signals. The phase difference between the output signal and the input signal was adjusted to achieve a maximum DC output. This output is directly proportional to the rms value of the input signal. By using the lock-in amplifier, the small signal, possibly buried in noise, could be extracted.

The DC output of the lock-in amplifier was read with a Keithley 2110, 5 ½ digit multimeter and stored in the onboard memory for later retrieval. A second Keithley 2110 multimeter read the potential difference across the diode, stored in the onboard memory for later retrieval and conversion to a temperature. The multimeters used the same SCPI code, *Standard Commands for Programmable Instruments* code. To retrieve the data from the multimeters, a program written using Python with the specific distribution of Anaconda was written. To communicate with the multimeters, the PyVISA package from the National Instruments Visa library and USB drivers were used on a standard laptop. Since two separate multimeters were used to obtain the temperature-voltage data, the two multimeters needed to be synched. This was the main purpose of the Python code, to call both measurements simultaneously, time stamp, and pair them in an excel file. The measurements were taken every second. Since the change in temperature was slow, one second was adequate time to capture the data. The sampling rate could have been adjusted if necessary. The paired and time-stamped data was imported to an Excel file and converted to a graph of Potential vs. Temperature. The python code that was used is in the Appendix.

EXPERIMENTAL RESULTS

$YBa_2Cu_3O_x$ was the only tested superconductor that was found to be superconducting above 77K, the results for which are provided in the following table. The other produced samples, which may have been superconducting at lower temperatures, include $LaBa_2Cu_3O_x$, $La_{.25}Y_{.75}Ba_2Cu_3O_x$, $B_{.5}YBa_2Cu_3O_x$, $B_{.05}YBa_2Cu_3O_x$, $B_{.02}YBa_2Cu_3O_x$, $La_{.01}Y_{.99}Ba_2Cu_3O_x$, $Pb_{.25}YBa_2Cu_3O_x$, $CdYBa_2Cu_3O_x$, $Cd_{.01}YBa_2Cu_3O_x$, $YCd_2Cu_3O_x$, and $YCdCu_3O$

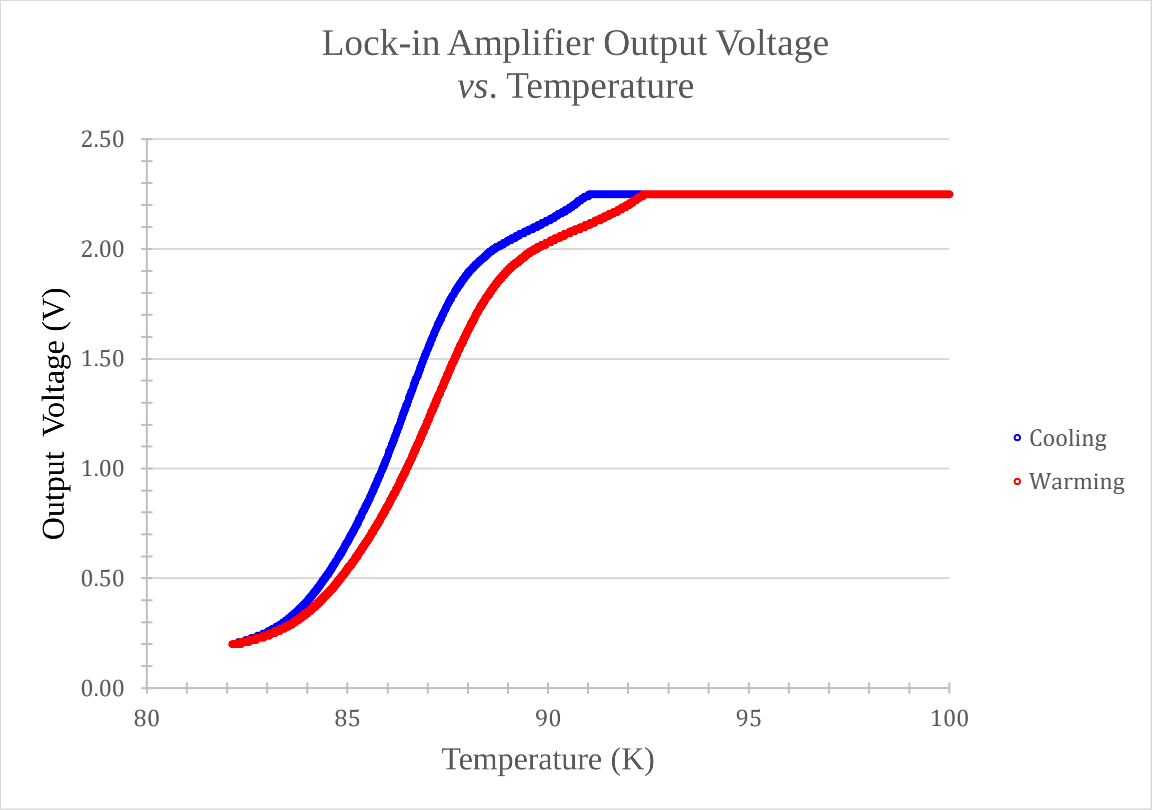

Figure 2: A graph of magnetic flux vs temperature in the successful sample of $YBa_2Cu_3O_x$.

The data clearly show a drastic drop in potential across the secondary around 87K. It can be seen from the graph that the flux is not excluded entirely at lower temperatures since the lower plateau has not fully flattened out in the presented data. This means that technically the critical temperature of the material is slightly lower than 77K since the sample has not fully excluded the flux from the primary at those temperatures.

With the two curves in Figure 2, a natural question of which one is more accurate arises. Since the temperature measured is not the direct temperature of the sample itself, but that of the silicon diode, it is important to ensure that the temperature of the diode and of the sample are as close together as possible. A thin layer of *ptfe* (Teflon) tape was placed between the diode and the sample to prevent the diode leads from contacting the sample. By slowly changing the temperature, the diode and sample are kept at relatively the same temperature, thus implying that the slower curve is the more reliable one. As shown from Figure 3, the time rate of change of the temperature is much smaller for the warm-up part of the cycle implying that the temperature of the sample and thermometer were in closer agreement.

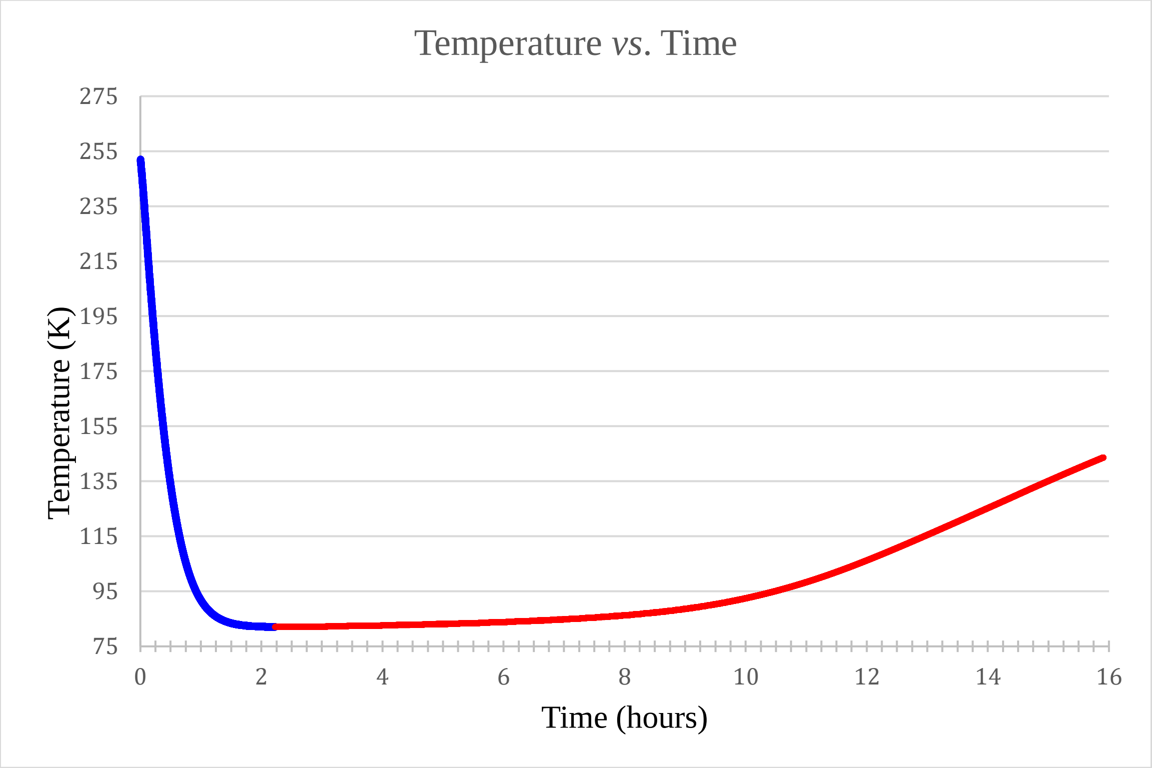

Figure 3: The temperature as a function of time for the successful sample of $YBa_2Cu_3O_x$.

Figure 4 shows a test of one of the samples which was not superconducting at or above 77K. This was the $Cd_{.01}YBa_2Cu_3O_x$ composition. The steep change in the potential at the low temperature range of the graph implies that this compound may become superconducting below 77K. When this sample was tested by being immersed in liquid nitrogen, it did not show total magnetic flux exclusion. The drastic contrast between the cooling and warming cycles of the curve is curious and too dramatic to be due to the temperature difference between the sample and the silicon diode thermometer. This composition needs to be tested again to see if this anomaly is repeatable. This particular sample crumbled after being brought up to room temperature.

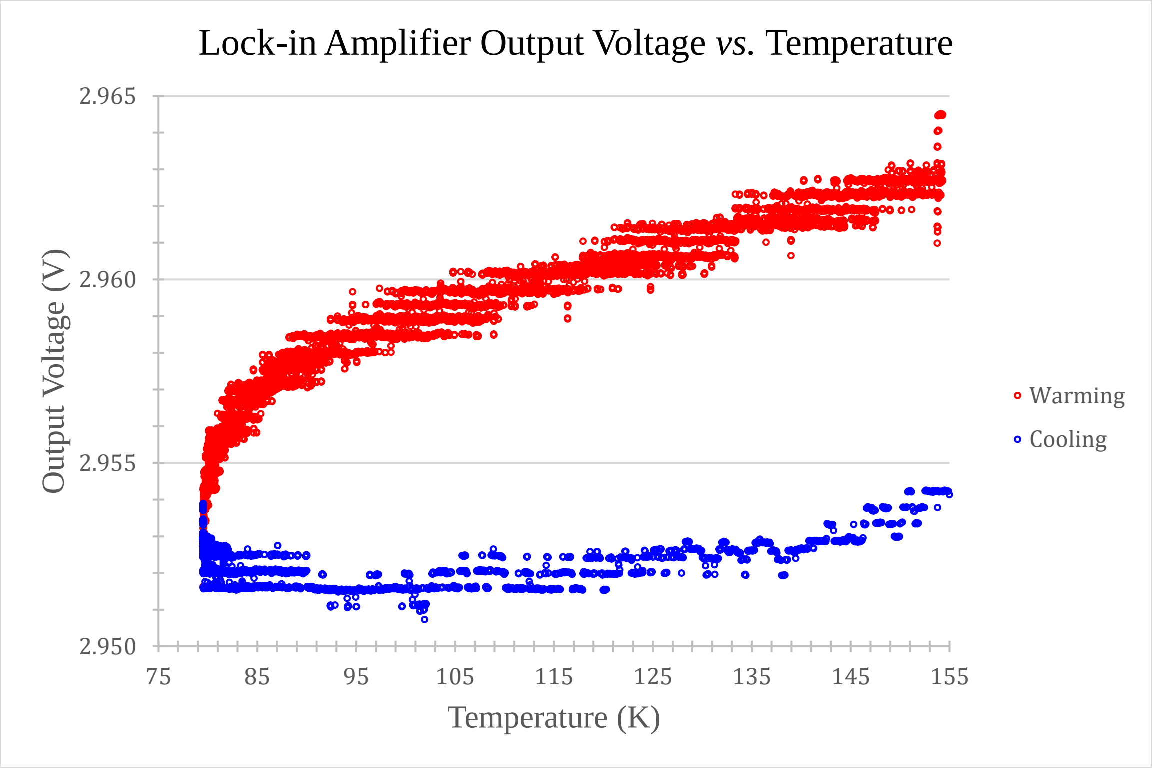

Figure 4: Temperature *vs*. time for the failed sample of $Cd_{.01}YBa_2Cu_3O_x$.

FUTURE WORKS

Considering figure 4, compositions of the $YBa_2Cu_3O_x$ family combined with cadmium show promising results. In the samples that were created, most of the cadmium variants showed signs of superconductivity at temperatures below liquid nitrogen. It’s possible that further exploration with different ratios might yield a higher critical temperature. Additionally, mercury compositions might lead to higher critical temperatures since the leading superconductor at the time of writing contains mercury.

CONCLUSIONS

The goal of this research paper was to produce a YBa_2Cu_3O superconductor and modify it in an attempt to raise the superconducting transition temperature. The transition temperature of YBa_2Cu_3O was found indirectly by obtaining a measure of the magnetic flux exclusion with temperature. However, this paper was unsuccessful in using this compound as a building block in discovering other superconductors. For our data we had some error since the critical temperature of our sample was slightly lower than the known critical temperature of this compound. This can likely be explained by the sintering process. During firing, the kiln that was being used would often shut off in the middle of the procedure and not fire for the allotted time, thus not fully sintering the sample. The sintering process also did not penetrate the interior of the sample and only affected the outside, which could have also changed the results. These results, while not being the first to discover and research them, are an important touchstone for the history of superconductors as a whole, being the first superconductor with a transition temperature above the temperature of liquid nitrogen. In addition, although the compositions used here did not reveal any interesting results, the Y-Ba-Cu-O family remains a promising potential path forward for the future of high temperature superconductors.

APPENDIX

Python Code

```
import pyvisa
import time

rm = pyvisa.ResourceManager()
f = open("Temp_vs_Volts.csv", "w")

# Sets up variables to directly talk with each meter
temprature_reading = m.open_resource('USB0::1510::8464::8016446::0::INSTR')
Lockin_reading = rm.open_resource('USB0::1510::8464::8017832::0::INSTR')

# Configure each meter into desired settings, changeable
temprature_reading.write(':CONFigure:VOLTage[:DC] 10,0.00001')
Lockin_reading.write(':CONFigure:VOLTage[:DC] 10,0.001')

# Loop is what measures the system. On one pass, it outputs the reading from
# both meters, stores it into a file then it sleeps for a time then executes
# again
#f.write(f'Timestamp, Query_Number, DC, AC')
query_number = 0
while True:

    timestamp = time.strftime("%H:%M:%S", time.gmtime())
    temp = (temprature_reading.query(':MEASure:VOLTage:DC?')).rstrip()
    volts = (Lockin_reading.query(':MEASure:VOLTage:DC?')).rstrip()

    f.write(f'{timestamp}, {query_number}, {temp}, {volts}\n')

    query_number = query_number + 1

    time.sleep(1)

f.close()
```

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VITA

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