Synthesizing and Testing a Type II Superconductor

James Richardson
and
Bruce Ferguson

Undergraduate Physics Laboratory
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Abstract

Their remarkable electrical and magnetic properties make superconducting materials very desirable for experimental and commercial applications, but the limiting factor has always been the extremely low critical temperatures required. For most of this century, the critical temperatures could only be achieved using liquid Hydrogen or Helium refrigerants, and making the use of these materials very expensive and inconvenient [2]. However, in the mid 1980's, a new class of superconducting materials were discovered with high enough temperatures to permit the use of liquid Nitrogen as the refrigerant – which is much cheaper and easier to utilize. Because of this, hopes for even higher critical temperatures and increased practical applications were ignited, and superconductor research once again came to the forefront of solid-state physics research programs [1]. This included a program at the National High Magnetic Field Laboratory (NHMFL) in Tallahassee, Florida.

As part of this program, undergraduate physics students are often given the opportunity to synthesize and test a small superconductor sample, in order to give them exposure to this fascinating area [3]. The tests to which the produced sample was subjected were (1) a resistivity test, in which the critical temperature of the superconductor was determined as a function of resistance, (2) an x-ray powder diffraction test, in which the crysalline structure of the produced sample was investigated, and (3) a magnetization test, in which the magnetic properties of the material were determined as a function of temperature. A summary of the derived parameters follows:

Critical temperature from the resistivity test (reference $T_c = 92.5\ K$, [5]):

$$T_c = 91.6\ K\ \pm\ 0.9\ K\,.$$

Critical temperature from the magnetization test (reference $T_c = 92.5\ K$, [5]):

$$T_c = 93.8\ K\ \pm 2.8\ K\,.$$

From the powder diffraction test, the crystal unit cell dimensions were found to be:

$$\begin{aligned} a &= 3.8482\text{Å}\ \pm 0.0156\text{Å}\,, \\ b &= 3.8983\text{Å}\ \pm 0.0048\text{Å}\,, \\ c &= 11.7068\text{Å}\ \pm 0.0029\text{Å}\,, \end{aligned}$$

Which can be compared to the reference values from the PDF card of: $a = 3.8394$Å, $b = 3.8986$Å, and $c = 11.7057$Å. These values gave a crystal unit cell volume of (reference value, $v = 174.822\ \text{Å}^3$):

$$v = 175.62\text{Å}^3\,,$$

For the given sample volume, the expected value for the superconducting magnetic moment of the sample was:

$$m_{expected} = -0.1937\ emu\,.$$

This can be compared to the actual measured maximum value from the magnetization test of:

$$m = -0.0097712\ emu\,,$$

An effective superconducting percentage of only 5.05% was achieved with this sample. This is not entirely surprising, considering the abbreviated synthesis technique.

From the magnetization test, the determined Curie constant for the sample was:

$$C = 7.681 \, \frac{emu \, K}{mol} \, .$$

This can be compared favorably to a reference value of [2]:

$$C = 7.875 \, \frac{emu \, K}{mol} \, .$$

Finally, the determined effective magnetic moment for the molecule was found to be:

$$\mu_{eff} = 7.839 \, \mu_b \, .$$

which can also be compared favorably to the reference value of [2]:

$$\mu_{eff} = 7.937 \, \mu_b \, .$$

Contents

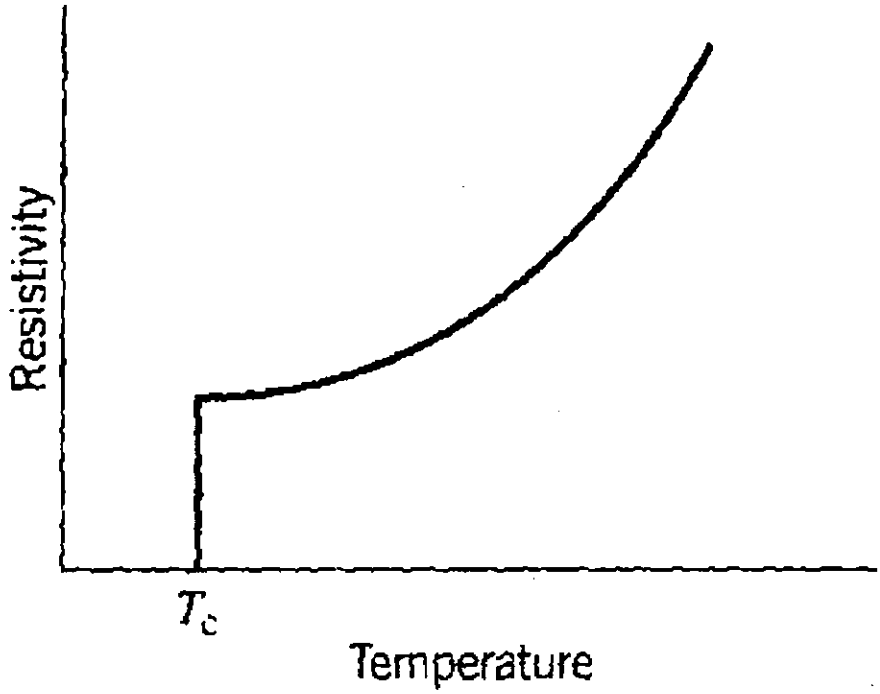

Figure 1: A typical resistivity curve for a superconductor (taken from [1]), showing the sample resistivity as a function of temperature. When the temperature reaches the critical temperature, T_c, the resistance suddenly drops to zero. This is the most basic characteristic of a superconductor.

1 Introduction

In 1911, Kamerlingh Onnes discovered a very odd class of solid materials – the superconductors [1]. The signature characteristic of these materials is that below some very low critical temperature, T_c, the electrical resistance of these materials suddenly drops to zero; that is, the material will conduct electrical current without ANY losses or I^2R heating (see Figure 1). Additionally, at below the critical temperature, the material will also become perfectly diamagnetic; that is, when exposed to an external magnetic field the material will produce an internal magnetic field which is exactly equal and opposite to the external field (called the Meissner effect [2]).

These, and other properties, make superconducting materials very desirable for experimental and commercial applications, but the limiting factor has always been the extremely low critical temperatures required. for most of this century, the critical temperatures could only be achieved using liquid Hydrogen or Helium refrigerants, and making the use of these materials very expensive and inconvenient [2]. However, in the mid 1980's, a new class of superconducting materials were discovered with high enough temperatures to permit the use of liquid Nitrogen as the refrigerant – which is much cheaper and easier to utilize. Because of this, hopes for even higher critical temperatures and increased practical applications were ignited, and superconductor research once again came to the forefront of solid-state physics research programs [1]. This included a program at the National High

Table 1: Compounds Used in Superconductor Synthesis

Compound	Molar Mass	Mass	σ	Moles	Ratio
Gd_2O_3	362.50 g/Mol	0.1803 g	0.0002 g	4.974E-4 Mol	1.000
$BaCO_3$	197.34 g/Mol	0.3925 g	0.0002 g	1.989E-3 Mol	3.999
CuO	79.545 g/Mol	0.2373 g	0.0003 g	2.983E-3 Mol	5.998
$GdBa_2Cu_3O_7$	734.54 g/Mol	$\approx$0.731 g	-	$\approx$9.95E-4 Mol	2.00

Magnetic Field Laboratory (NHMFL) in Tallahassee, Florida. As part of this program, undergraduate physics students are often given the opportunity to synthesize and test a small superconductor sample, in order to give them exposure to this fascinating area [3].

This paper describes the synthesis and testing of a Type II ceramic superconductor, composed of $GdBa_2Cu_3O_7$, performed at the National High Magnetic Field Laboratory (NHMFL), associated with Florida State University (FSU), during the Spring semester of 2000. This experiment is described in the following sections: Section 2 describes the process used to produce the ceramic superconductor sample utilized in this experiment. Section 3 describes the resistivity test used to verify that the sample did indeed achieve superconductivity at (and below) the critical temperature. Section 4 describes the X-ray Bragg diffraction test used to verify the proper crystalline structure of the sample. Section 5 describes the SQUID magnetometer test used to verify proper magnetic behavior of the sample. Finally, Section 6 summarizes the major points and primary derived parameters for this experiment.

2 Superconductor Synthesis

This section describes the synthesis of the $GdBa_2Cu_3O_7$ ceramic superconductor, beginning with the powered forms of its constituent components. This "powder sintering" process was performed under the instructions and supervision of the lab instructor, Damon Jackson.

2.1 Chemical Composition

The ceramic Type II superconductor, $GdBa_2Cu_3O_7$, was produced by the students using a powder sintering technique beginning with the powered forms of three basic compounds: Gd_2O_3, $BaCO_3$, and CuO. The three dry powders were mixed thoroughly in the proper stochiometric ratios, and then annealed repeatedly in a high temperature, O_2 environment to bring out the desired chemical reaction and crystalline structure. The desired chemical equation is:

$$2Gd_2O_3 + 8BaCO_3 + 12CuO + O_2 = 4GdBa_2Cu_3O_7 + 8CO_2\,. \tag{2.1}$$

Note the importance of the additional O_2, which will leach into the crystal during the annealing process along with the desired leaching out of CO_2. Table 1 lists the amounts, in grams, of each compound used in this experiment, along with their stochiometry:

Note that although 0.8101 g of dry chemicals went into the initial mixture, the final crystal would have a mass of only about 0.731 g due to the reactions with O_2 and CO_2, assuming no other losses. It should also be noted that the dry powders used are very susceptible to the absorption of H_2O (the are normally stored in a dry oven), and measurement inaccuracies can easily creep in due to the absorption of water while the chemicals are being weighed out. This is especially true in the humid Tallahassee environment.

2.2 Annealing Process

After these compounds had been combined, they were thoroughly ground and mixed in a stone mixing bowl. The resulting gray powder was poured into a crucible and baked in a normal atmosphere at 900C for 24 hours, and then at 1000C for another 24 hours.

During this phase, the powder had turned into a hard black pellet. It was next broken and ground again. The sample was then annealed in an Oxygen (O_2) atmosphere. The annealing was undertaken in a programmable oven. The following rates and durations of heating and cooling were scheduled into the oven's computer;

1. Heated for five hours from 20C to 1000C (+3.3C/min);
2. Then spent 24 hours at 1000C;
3. Then cooled for 15 hours to 420C (-.64C/min);
4. Then spent 48 hours at 420C;
5. Then brought to room temperature by turning the oven off;

It should be noted that the above procedure is VERY abbreviated from that used to make a high quality superconductor. The purpose of the shortened procedure is to train students in the process of superconductor production, create a testable superconductor, while at the same time minimizing the excess laboratory time required for this experiment.

3 Resistivity Testing

Following synthesis, the first and most basic test to be performed on the resulting sample was a resistivity test, checking for the characteristic superconductor drop of the sample resistance to zero at and below the critical temperature. This process was performed under the instructions and supervision of the lab instructor, Damon Jackson.

3.1 Resistivity Test Procedure

The resistivity test involved using a small section of the sample, to which four platinum leads were connected (using a special conducting silver glue) in a straight line arrangement. The two outermost leads were then connected to a constant current source. The two innermost leads were then connected across a very sensitive voltmeter. The resistance of the sample was then calculated by the monitoring computer by using Ohm's Law, that is, $R = E/I$.

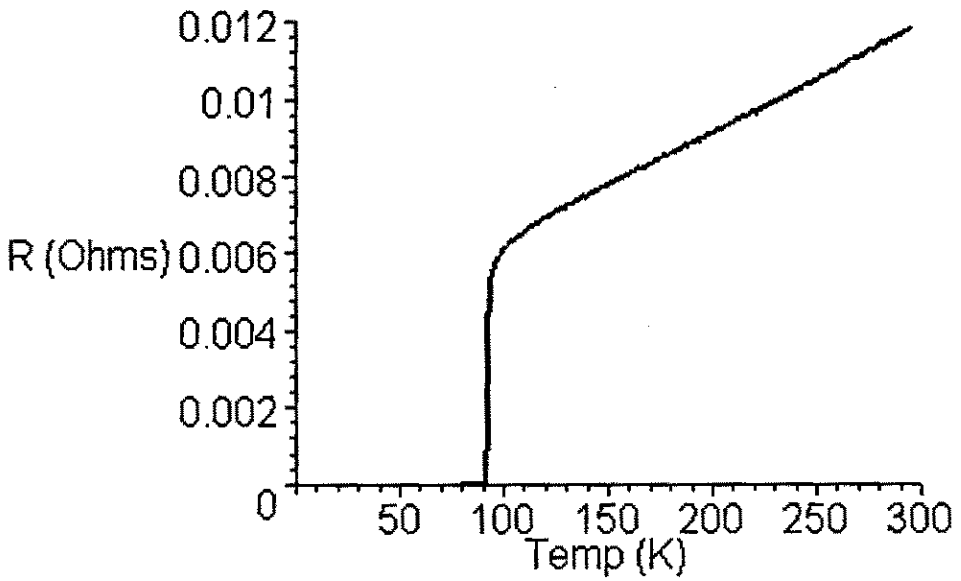

Figure 2: A plot of sample resistance (Ohms) verses sample temperature (K). When the critical temperature of $\approx 92\ K$ is reached the sample resistance drops suddenly to zero ($0\ \Omega$), as expected for this superconductor. Compare this figure to the generic curve shown in Fig. 1.

Under computer control, this sample was then slowly lowered into a tank utilizing liquid Helium as the refrigerant, such that the temperature of the sample was slowly lowered in a controlled fashion while taking periodic resistance readings. This data was stored on the monitoring computer for future analysis.

3.2 Resistivity Test Results

A plot of the results from this test is shown in Figure 2. Above the critical temperature, T_c, the resistance of the sample decreases linearly, in a manner similar to most conductors. At the critical temperature, however, the resistance of the sample drops rapidly to zero, over a transition range of about 2-4 degrees. This behavior is exactly what was expected for this Type II superconductor.

Reading directly from the recorded data, the critical temperature at which zero resistance is reached is given as (using $2 \cdot \sigma$ for a confidence interval of 95% [4]):

$$T_c = 91.6\ K\ \pm\ 0.9\ K\ .$$

This is reasonably close to the reference value of $T_c = 92.5\ K$ (from [5]).

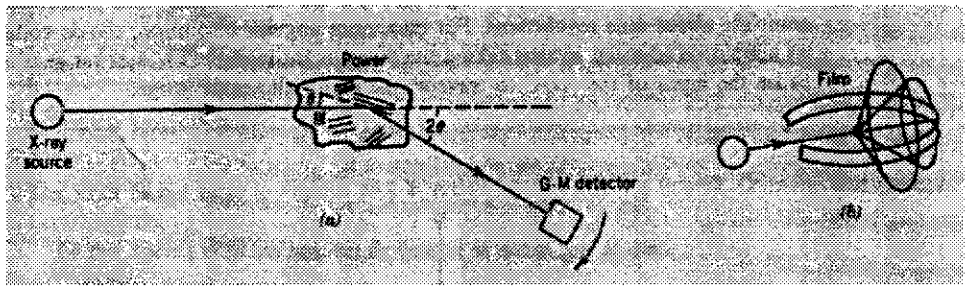

Figure 3: A diagram of the x-ray powder diffraction test setup, taken from [6]. In (a) the x-ray source is shown illuminating the powdered sample, with a x-ray detector being moved in an arc around the sample. In (b), the conical reflection pattern from two non-interfering crystalline planes is shown, along with the arc produced by the detector sweep.

4 Crystal Structure Testing

Another method for verifying that a proper ceramic crystalline structure was produced by the synthesis process is to subject the sample to a Bragg X-ray powder diffraction test [6]. This test is used to verify that the crystalline unit cells in the sample match the expected unit cell structure for a properly prepared $GdBa_2Cu_3O_7$ sample. This process was performed under the instructions and supervision of the lab instructor, Damon Jackson.

4.1 Powder Diffraction Test Procedure

A pure solid, crystalline structure consists, at the molecular level, of an interlocking, repeating pattern of unit "cells." When bombarded with an monochromatic x-ray beam, the atoms in the crystalline structure will reflect the x-rays in such a way as to cause a diffraction, or interference pattern, the nature of which will be related to the dimensions of the crystal unit cells. This allows x-ray diffraction to be an effective method for investigating the unit cell dimensions of an unknown sample [6].

Figure 3 shows a diagram of the basic x-ray powder diffraction technique. The x-ray source used for this experiment uses an electron beam to bombard a Copper (Cu) anode, which, in turn, emits x-ray electro-magnetic radiation as the electrons are absorbed. The collimated x-ray beam is directed to the target, which consists of a portion of the experiment sample ground to a very fine powder. At those reflection angles at which Bragg's Diffraction Law is met, a reflection peak will occur, causing a peak in the detector output. The advantage of the powdered sample as opposed to the single solid unit sample is that a solid sample crystal face must be rotated through the various x-ray beam incident angles in order to detect the diffraction pattern peaks, while a fine powder sample already has "all" of these various crystal face presentation angles present randomly and simultaneously to the single x-ray beam [6].

The basic statement of Bragg's Law is given by:

$$2d\ sin\theta = N\ \lambda\ , \tag{4.1}$$

where:

$$\begin{aligned} d &= \text{distance between regularly spaced atoms,} \\ \theta &= \text{angle of x-ray incidence to crystal face normal,} \\ N &= \text{integer greater than zero: 1, 2, 3...} \\ \lambda &= \text{x-ray wavelength.} \end{aligned}$$

The parameter of interest in Eq. 4.1 is the atomic spacing distance, d. This distance is a function of the unit cell dimensions for the crystal, described by the three rectangular coordinate system distances a, b, c. The volume of the unit cell for the crystal is thus given by $a \times b \times c$. In practice, rather than using the cell dimensions a, b, c to describe the measured distance d in x-ray diffraction, it is more common to utilize the reciprocal integer values h, k, and l. The relationship between these values is given by the equation:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \,. \tag{4.2}$$

Once an x-ray diffraction scan of the sample has been completed, the task generally becomes one of matching the *theta* and d values determined from the test to known values from previously performed tabulations for various substances. In this particular experiment, the results of the x-ray diffraction test were compared to the known values for $GdBa_2Cu_3O_7$, published previously. Figure 4 shows a plot of a previously performed x-ray diffraction tests for this superconductor, annealed in an Oxygen (O_2) atmosphere.

With a known diffraction pattern peak identified in the test for the given sample, the measured value of d can then be combined with the reference values of h, k, l to form an algebraic expression having the variables a, b, and c. Combining the results of several such diffraction pattern peaks will create a system of equations from which the dimensions a, b, c can be found. With the dimensions for the unit cell known, the volume of the unit cell can also be easily found, leading to a known density and volume for given masses of the substance.

4.2 Powder Diffraction Test Results

Figure 5 shows the x-ray powder diffraction test results for the sample created in this experiment. This plot compares very well with the known plot shown in Figure 4. This indicates that although a few impurities or irregularities exist in the produced sample (shown by a few unidentified peaks), a majority of the sample appears to be of the proper Orthorhombic unit cell structure, having the correct unit cell dimensions.

Table 2 lists the diffraction peaks shown by the sample which match the diffraction pattern peaks given in the reference sheet (called a Powder Diffraction (PDF) card). The measured value for d, along with the associated values for h, k, l are also listed.

Creating a system of equations from using Eq. 4.2, the measured values for d, and the associated values of h, k, l from the PDF card yields the following values for the cell dimensions, a, b, c:

$$a = 3.8482\text{Å} \pm 0.0156\text{Å} \,,$$

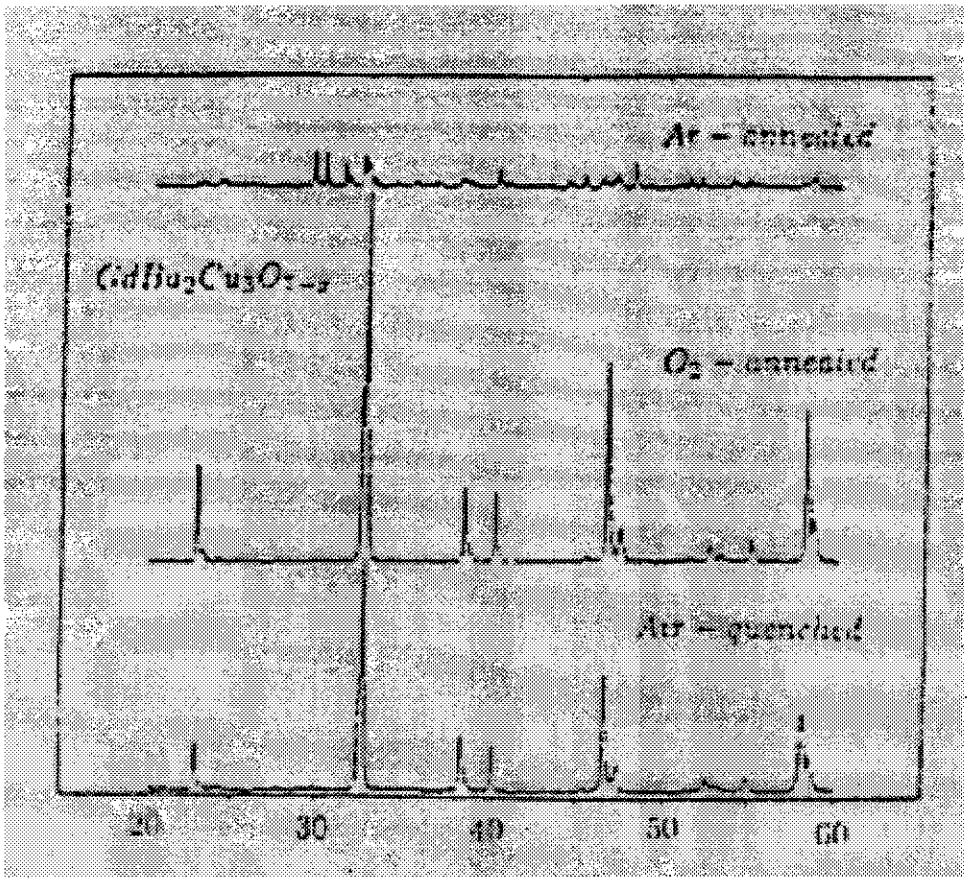

Figure 4: The expected x-ray powder diffraction pattern for the $GdBa_2Cu_3O_7$ crystal, taken from [5], with reflection amplitude shown as a function of detector angle. The sample in this current experiment was annealed in an O_2 environment and should be compared to the "O_2 Annealed" line in this figure.

$$\begin{aligned} b &= 3.8983\text{Å} \pm 0.0048\text{Å}\,, \\ c &= 11.7068\text{Å} \pm 0.0029\text{Å}\,. \end{aligned}$$

Which can be compared to the reference values from the PDF card of: $a = 3.8394$Å, $b = 3.8986$Å, and $c = 11.7057$Å. The agreement is quite good. Additionally, the volume of a unit cell for the sample was found to be:

$$\begin{aligned} v &= 175.62\text{Å}^3\,, \\ v &= 1.7562 \times 10^{-22}\, cm^3\,. \end{aligned}$$

5 Magnetization Testing

The final test to which the sample was subjected was a test of its properties when placed in an external magnetic field at various temperatures. This permitted investigations of the sample's magnetization, M; magnetic susceptibility, χ; material Curie constant, C;

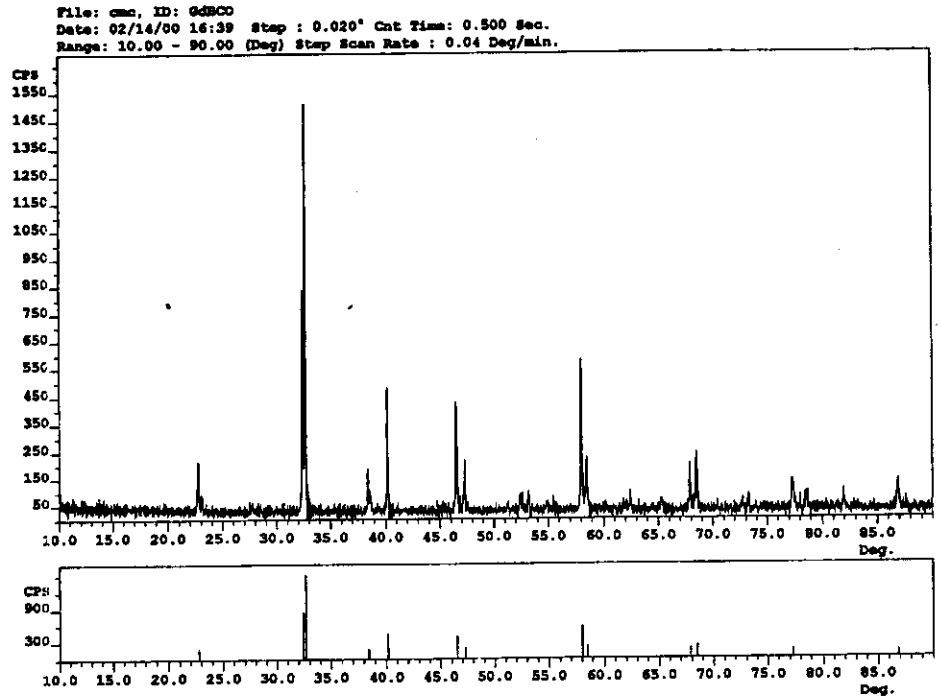

Figure 5: The actual x-ray powder diffraction pattern for the $GdBa_2Cu_3O_7$ crystal produced for this experiment, with reflection amplitude shown as a function of detector angle. This plot compares quite well with that shown in Figure 4, with the derived crystal dimensions also very close to the expected values.

Table 2: X-Ray Powder Diffraction Peak Data

Ref. θ	**Actual θ**	**d Å**	**(h, k, l)**	**(h, k.l)**
22.7681	22.791	3.9024	(0,0,3)	(0,1,0)
32.4319	32.438	2.7583	(0,1,3)	
32.6725	32.696	2.7385	(1,0,3)	
38.4119	38.432	2.3415	(0,0,5)	
40.2181	40.229	2.2404	(1,1,3)	
46.5256	46.553	1.9503	(0,0,6)	
47.2969	47.314	1.9203	(2,0,0)	
58.0187	58.046	1.5884	(1,1,6)	(1,2,3)
58.5119	58.537	1.5761	(2,1,3)	
67.9006	67.920	1.3793	(0,2,6)	
68.5131	68.551	1.3684	(2,2,0)	(1,0,8)
77.2400	77.329	1.2341	(0,1,9)	(0,3,3)
77.3200	77.480	1.2330	(1,3,0)	
86.8737	86.911	1.1203	(2,2,6)	(1,2,8)

superconducting critical temperature, T_c; and a measure of the percentage of the sample which behaved as a superconductor. This process was performed under the instructions and supervision of the lab instructor, Damon Jackson.

5.1 Magnetization Test Procedure

This portion of the sample testing was performed using a state-of-the-art SQUID magnetometer. The superconducting quantum interference device (SQUID) consists of two superconductors separated by thin insulating layers to form two parallel Josephson junctions. The device may be configured as a magnetometer to detect incredibly small magnetic fields.

SQUID was utilized to measure the magnetic moment, m, of the material as the sample was cooled using liquid Helium (He) in the presence of a constant external magnetic field of 1000 G. Measurements of the sample's magnetic moment were made every five to ten seconds or so while the sample cooled from 300K to 5K (about 50 measurements were made).

5.2 Critical Temperature and Magnetization Determination

Figure 5.2 shows a plot of the sample overall magnetization, m, (or magnetic moment) as a function of sample temperature (K). Note the sharp inflection point in the graph as the temperature drops below the critical temperature (about $T_c = 92-94\ K$). Above the critical temperature, the sample behaves as a paramagnet ($+m$), but below the critical temperature, the sample becomes diamagnetic ($-m$). A maximum diamagnetism is reached at $\approx 45\ K$, with a prominent "Curie Tail" (due to sample impurities) present for lower temperatures.

Reading directly from the recorded data, the critical temperature of the substance (shown by the inflection point in Fig. 5.2) is (using $2 \cdot \sigma$ for a confidence interval of 95% [4]):

$$T_c = 93.8\ K\ \pm 2.8\ K\ .$$

This compares favorably with the T_c obtained from the resistivity test (Sec. 3), and the reference value of 92.5 K.

Additionally, the sample reached a maximum diamagnetic moment of -0.0097712 *emu* at a temperature of 45.028 K. It is possible to determine an expected magnetic moment for the given sample size, assuming a perfect superconductor – allowing the determination of the total effective superconductor percentage in the produced sample. The mass of the sample used in the SQUID which produced this moment was:

$$0.0169\ g\ \pm 0.0004\ g\ .$$

Using Gaussian units, and assuming a perfectly diamanetic sample, the equation for the magnetization per unit volume of the sample would be given by [7]:

$$M = \frac{-H}{4\pi}\ , \tag{5.1}$$

and converting to a total magnetic moment:

$$m = \frac{-Hv}{4\pi}\ . \tag{5.2}$$

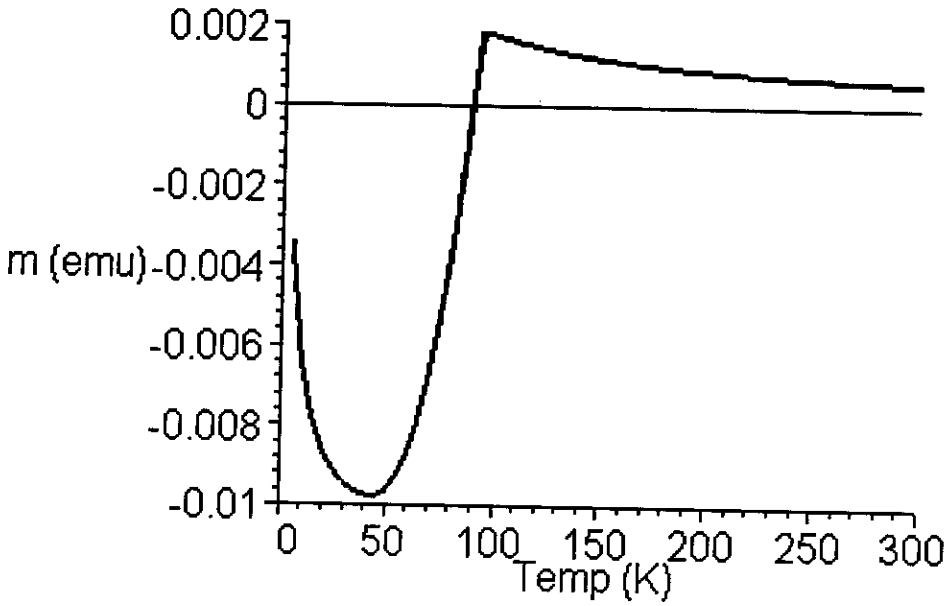

Figure 6: A plot of the sample overall magnetization, m, as a function of sample temperature. Note the sharp inflection point in the graph as the temperature drops below the critical temperature (about $T_c = 92 - 94\ K$). Above the critical temperature, the sample behaves as a paramagnet ($+m$), but below the critical temperature, the sample becomes diamagnetic ($-m$). A maximum diamagnetism is reached at $\approx 45\ K$, with a prominent "Curie Tail" (due to sample impurities) present for lower temperatures.

In order to utilize this last equation, the volume of the portion of the sample placed in the SQUID must be calculated. This is easily given by:

$$v = v_0\ N\ mol\,, \tag{5.3}$$

where:

$$\begin{aligned} v_0 &= \text{unit cell volume,} \\ v_0 &= 1.7562 \times 10^{-22}\ cm^3\,, \\ N &= \text{number of moles in sample,} \\ N &= 2.301 \times 10^{-5}\ mol\,, \\ mol &= \text{Avogadro's Number} \\ mol &= 6.0221 \times 10^{23}\,. \end{aligned}$$

This gives a sample value of:

$$2.433 \times 10^{-3}\ cm^3\,.$$

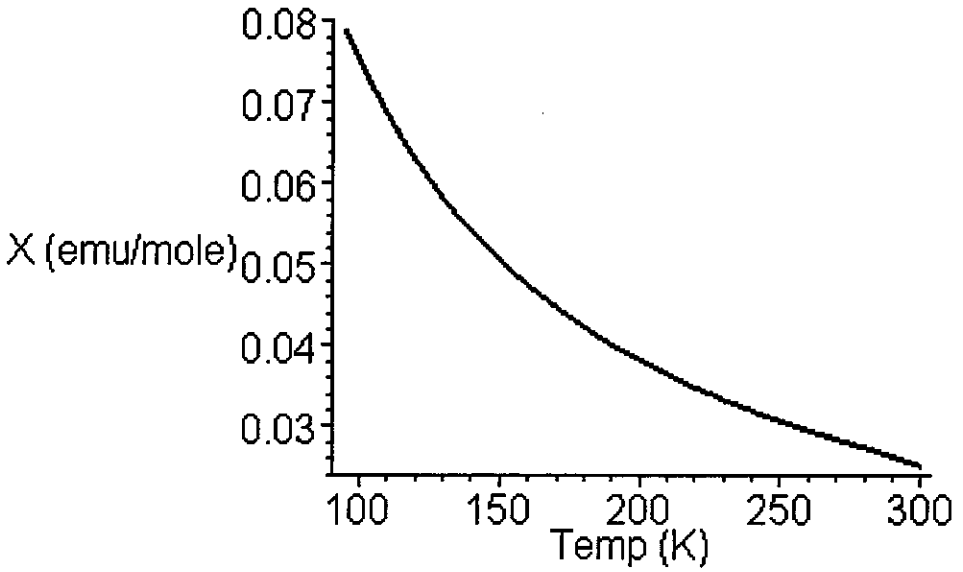

Figure 7: A plot of the sample magnetic susceptibility, χ (moles/emu), graphed as a a function of sample temperature, K, (for temperatures above the critical temperature, T_c). Note the hyperbolic behavior of χ over this range.

Plugging this value into Eq. 5.2, along with the constant external magnetic field strength of $H = 1000\ G$ gives:

$$m_{expected} = -0.1937\ emu\ .$$

Compared to the actual maximum value of $m = -0.0097712\ emu$, an effective superconducting percentage of only 5.05% was achieved with this sample. This is not entirely surprising, considering the abbreviated synthesis technique.

5.3 Magnetic Susceptibility and Curie Constant Determination

Above the critical temperature, the sample will behave as a conductor, displaying a hyperbolic paramagnetic moment with temperature (see Fig. 5.2). This hyperbolic behavior comes from the magnetic susceptibility, χ, as given in the following equation [7]:

$$M = \chi H\ , \tag{5.4}$$

where:

$$M = \text{magnetization per unit mole.}$$

Isolating the magnetic susceptibility in the magnetization data gives Figure 7. Note the positive, hyperbolic behavior of the magnetic susceptibility over this temperature range (above the critical temperature).

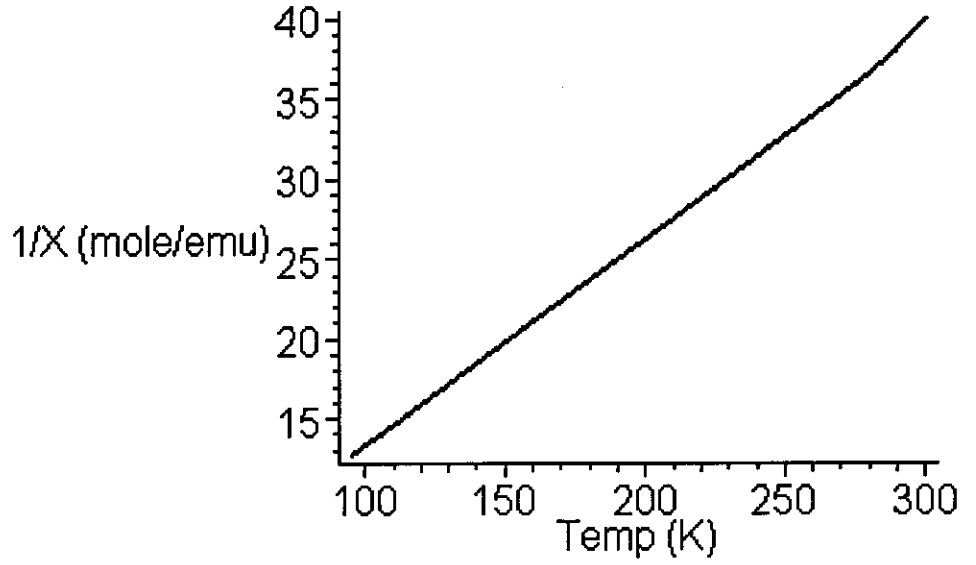

Figure 8: A plot of the sample's inverse magnetic susceptibility, $1/\chi$, as a function of sample temperature, K, for temperatures above the critical value, T_c. Note the very good linear behavior over this range, with the slope being equal to the inverse of the Curie Constant, $1/C$.

Another expression for the magnetic susceptibility, χ, is given in [2] as:

$$\chi = = \frac{C}{T - T_c}\,, \tag{5.5}$$

where:

$$\begin{aligned} C &= \text{Curie constant,} \\ T &= \text{temperature (K).} \end{aligned}$$

Combining Eq. 5.4 and Eq. 5.5, converting to measured units and rearranging into a linear equation yields:

$$\frac{1}{\chi} = \frac{HN}{m} = \frac{T}{C} - \frac{T_c}{C}\,. \tag{5.6}$$

This is a linear equation in the temperature, T, having $1/C$ as the slope. This relationship is demonstrated nicely in the data when $1/\chi$ is plotted as a function of temperature, as shown in Figure 8.

Performing a linear least-squares fit to this data yields the following linear equation:

$$\frac{1}{\chi} = 0.13019 \cdot T + .24210\,,$$

which, in turn, leads to a value for C of:

$$C = 7.681 \, \frac{emu \, K}{mol} \, .$$

This can be compared to a reference value of [2]:

$$C = 7.875 \, \frac{emu \, K}{mol} \, .$$

5.4 Effective Magnetic Moment Determination

As a final derivation, the effective magnetic moment for the $GdBa_2Cu_3O_7$ molecule (as a function of the Gadolinium atom) canb be determined. [2] gives two useful equations:

$$\mu_{eff} = g_J \sqrt{J(j+1)} \, \mu_b \, , \tag{5.7}$$

where:

$$\begin{aligned} \mu_{eff} &= \text{effective atomic magnetic moment,} \\ g_J &= \text{Lande g-factor,} \\ J &= \text{total atomic spin,} \\ \mu_b &= \text{Bohr magneton.} \end{aligned}$$

Using a $g_J = 2.000$ and for the Gadolinium atom a value of $J = 7/2$, will give:

$$\mu_{eff} = 7.937 \, \mu_b \, .$$

for comparison, another equation in [2] gives an expected value for μ_{eff} based upon the determined value of the Curie constant:

$$\mu_{eff} = \sqrt{8C} \, \mu_b \, , \tag{5.8}$$

which gives:

$$\mu_{eff} = 7.839 \, \mu_b \, .$$

Which is reasonably close to the value determined from the quantum mechanics equation.

6 Conclusion

The following sections summarize the results of the testing regime to which the produced sample was subjected. Overall, the sample performed rather well, considering the abbreviated preparation technique which was utilized. If a more extensive powder sintering and annealing technique had been utilized, the resulting crystal would have had a much higher percentage of superconductivity than the 5.05% which was achieved in this case.

6.1 Critical Temperature Determinations

From the resistivity test (reference $T_c = 92.5\ K$, [5]):

$$T_c = 91.6\ K\ \pm\ 0.9\ K\,.$$

From the magnetization test (reference $T_c = 92.5\ K$, [5]):

$$T_c = 93.8\ K\ \pm 2.8\ K\,.$$

Both of these compare well with the expected transition temperature.

6.2 Crystal Structure Determinations

The crystal unit cell dimensions were found to be:

$$\begin{aligned} a &= 3.8482\text{Å}\ \pm 0.0156\text{Å}\,, \\ b &= 3.8983\text{Å}\ \pm 0.0048\text{Å}\,, \\ c &= 11.7068\text{Å}\ \pm 0.0029\text{Å}\,, \end{aligned}$$

Which can be compared to the reference values from the PDF card of: $a = 3.8394$Å, $b = 3.8986$Å, and $c = 11.7057$Å.

These values gave a crystal unit cell volume of (reference value, $v = 174.822\ \text{Å}^3$):

$$\begin{aligned} v &= 175.62\text{Å}^3\,, \\ v &= 1.7562 \times 10^{-22}\ cm^3\,. \end{aligned}$$

Demonstrating that the powder diffraction test is a powerful technique for analizing the molecular crystal structure of a given solid, and that much of our produced crystal met the desired makeup.

6.3 Magnetization Test Determinations

For the given sample volume, the expected value for the superconducting magnetic moment of the sample was:

$$m_{expected} = -0.1937\ emu\,.$$

Compared to the actual measured maximum value of

$$m = -0.0097712\ emu\,,$$

an effective superconducting percentage of only 5.05% was achieved with this sample. This is not entirely surprising, considering the abbreviated synthesis technique.

The determined Curie constant for the sample was:

$$C = 7.681\ \frac{emu\ K}{mol}\,.$$

This can be compared favorably to a reference value of [2]:

$$C = 7.875 \frac{emu\ K}{mol}.$$

Finally, the determined effective magnetic moment for the molecule was found to be:

$$\mu_{eff} = 7.839\ \mu_b .$$

which can also be compared favorably to the reference value of [2]:

$$\mu_{eff} = 7.937\ \mu_b .$$

Demonstrating that except for the percentage of the crystal which was actually superconducting, the magnetic behavior of the crystal fell quite close to the expected values.

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