High Temperature Superconductivity Advanced Lab-Magneto-transport in the normal state

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0. SYNOPSIS

<u>Introduction</u>: High temperature superconductors (HTS) have received a great deal of attention due to the unusually high critical temperature T_c , at which they become superconducting. However, the electrical behavior of HTS in the normal, non-superconducting state (T>T_c) is very unusual and continues to be a fundamental mystery despite the best efforts of experimentalists and theorists over the past 14 years.

<u>Objective</u>: This experiment will probe the electronic properties of conventional and unconventional metals. More specifically, the temperature dependence of the resistivity and Hall angle will be studied in gold (conventional metal) and the high temperature superconductor $Yba_2Cu_3O_7$ (YBCO, unconventional metal). The student will learn basic experimental techniques and gain a better understanding of the electronic properties of metals.

Goals:

- 1. Learn and use basic electrical transport measurement principles such as noise reduction, error analysis, four-probe measurement, ac vs dc techniques, and computer controlled data acquisition.
- 2. Learn to troubleshoot experimental systems; equipment and samples do not always work as they are supposed to!
- 3. Perform magneto-transport measurements on gold and YBCO thin films from room temperature to 77 K.
- 4. Determine what aspects of the YBCO results are anomalous and qualitatively discuss models to explain the anomalous behavior.

<u>Grading</u>: Though the quality and quantity of your data are important, you will be mainly evaluated on your efforts in making and analyzing your measurements. Time spent repairing or improving the measurement apparatus will be taken into account in your evaluation. Extra credit will be given if you are able to improve the measurement and/or analysis techniques.

I. INTRODUCTION

High T_c ("critical" or transition, temperature) superconductivity (HTS) was discovered in 1987 and is still not well understood. It still is not clear what the fundamental mechanism is or to what extent it is similar to the familiar low temperature superconductivity that is described by the BCS (Bardeen, Cooper Schreifer) theory.

The experiments that you will do are designed to familiarize you with the properties of the HTS materials and with some of the techniques that are used to study them.

II. THEORY

You are expected to study the BCS theory of superconductivity. (More is expected of graduate students in this respect; this theory is rather sophisticated.) The low temperature variety of superconductivity is still important and its explanation is interesting physics. And, even if one's interest is restricted the HTS case, it is necessary to know the BCS theory because much of the discussion of the new superconductors is in terms of the extent to which they might or might not follow the BCS picture. You will also be expected to familiarize yourself with the experimental properties of the low T_c materials. HTS superconductors are what is known as Type II superconductors, and you should learn what this means.

Basically, studying the BCS theory means understanding what is meant by "pairing" and the "energy gap" and what role the phonons play. You should see that superconductivity is fundamentally a many-body effect, and in this context you should note that the energy gap here is quite different than the one that appears in semiconductors.

The two most basic experimental properties of superconductors are zero electrical resistance and the Meisner effect. Look for the reason why we say that these are two separate properties and that the Meisner effect is not merely a consequence of perfect conductivity. Also, you should understand what is meant by the vortex state of type II super-conductors.

For the HTS case, you may be able to make sense of the theoretical proposals as to how pairing might be caused, but your emphasis should be on the structure of the materials and their electrical behavior. An introduction to superconductivity is provided by [1].

The field of HTS is extremely rich and has sparked intense theoretical efforts that have yet to be fully tested experimentally. A general introduction to HTS materials is provided by [2, 3]. HTS materials (e.g., YBa₂Cu₃O_{7- δ}, La₃Sr_{2-x}CuO₄) show unconventional behavior both in the normal and superconducting states. Though dc measurements in the normal state show signs of simple metallic behavior, such as a hole-like Hall effect determining a carrier density that is consistent with the oxygen doping level, there are a number of puzzles remaining. Unlike conventional metals, the resistivity ρ and the dc Hall coefficient $R_{_H}$ vary linearly with temperature T [4, 5]. In the Drude model, the cotangent of the DC Hall angle θ_H has the same temperature

dependence as ρ , but in HTSC cot θ_{H} increases as T^{2} . Furthermore, measurements on σ_{xv} show a T^{-3} behavior instead of the expected T^{-2} behavior [6-8]. These anomalies have been observed in dc measurements over a wide range of temperatures and in a wide variety of HTS materials. In order to explain the anomalous Hall effect in HTS, two basic classes of theoretical approaches have been used. The first approach argues that the system is still a Fermi liquid (FL), where excitations consist of conventional electron-like quasiparticles, but that the strong anisotropy of the Fermi surface (FS) causes quasiparticles that are on different parts of the FS to have a different character [9-11]. However, this anisotropy in the carrier behavior required to satisfy experimental measurements cannot be easily explained using microscopic arguments. The other theoretical approach is to suggest that the system has non-FL properties, where the excitations are composed of more exotic entities. One of these non-FL theories [12] argues that the excitations of HTS in the normal state separate spin and charge to produce two distinct kinds of collective excitations, spinons (fermions with spin but no charge) and holons (bosons with charge but no spin). As with FL theories, the distinct character of these two excitations can be used to model measurements, but a microscopic justification of the existence of spinons and holons in HTS is still problematic.

III. EXPERIMENT

The specific experimental techniques that you will learn are the use of a low temperature apparatus and four-probe electrical measurement.

The specimens are provided to us. A gold and a YBCO thin film sample are both mounted and wired to the sample stick. The wires leading to the two samples are labeled in Fig. 1.

This lab consists of the following measurements on gold and YBCO thin films:

- 1. Resistance (conductivity) versus temperature (and from this the transition temperature T_c in zero magnetic field.
- 2. Hall angle and Hall "constant" as a function of temperature.

Here are some questions that you should be able to answer and that you may want to discuss in your lab report:

- 1. What is anomalous about the measured transport parameters in YBCO?
- 2. What are some of the qualitative models that attempt to explain this anomalous behavior?
- 3. Why use four probes for resistivity measurements?
- 4. Why use the van der Pauw geometry?
- 5. What are the advantages of using ac measurement techniques instead of dc techniques?
- 6. What are the main sources of error in your measurements?

IV. APPARATUS AND TECHNIQUES

Some Cautions:

DO NOT get or use liquid nitrogen until your instructor shows you how. DO NOT handle specimen or attach leads until your instructor shows you how. DO NOT move or use magnet until your instructor shows you how. DO NOT use any of the electronics until your instructor shows you how.

A. Specimen probe.

Handle this with care; the wires are fragile. The gold and YBCO samples are already mounted and wired to the sample probe stick. You should not have to rewire the samples (this is a delicate and time-consuming task), but if the measurements appear faulty you will have to check the wiring. Watch out for the possibility that previous users have changed wiring connections.

The components of the specimen probe are:

- 5. The suspension tube, which slides in the cap to vary the height of the specimen.
- 5. A copper vessel around the specimen and heater, which helps to keep the temperature uniform and reduces thermal fluctuations.
- 5. A temperature sensitive diode (DT471) that is used to measure the specimen temperature.
- 5. A heater which balances the cooling by the cold helium exchange gas that will surround the can and specimen, and helps control the rate of change of temperature of the specimen.
- 5. Sample holder: provides flat mounting surfaces for the samples and temperature sensitive diode.

Figure 1 shows the back of the sample holder, to which is mounted the temperature sensitive diode (DT-471). Contacts L and M are used to send a 10 μ A current (from a LakeShore Model 102 current source) through the diode while contacts G and H measure the voltage across the diode and hence the temperature of the diode. Note the diode only works if a positive polarity current is applied (L is positive with respect to M). Figure 2 shows the electrical connections to the sample on the front side of the sample holder.



Figure 1: Back of the sample stick where the silicon temperature sensitive diode is mounted.



Figure 2: Front of the sample stick where the gold and YBCO samples are mounted. Note that the coaxial cables C1 and C2 are labeled CI and CII, respectively, at connectors at the top of the cryostat.

B. Measuring equipment

A lock-in amplifier, digital voltmeters, and current sources are supplied. These are of good quality and in fact some are quite expensive, so please be careful. Your instructor will show you how to use them.

C. The four-probe resistance method

This technique allows one to measure resistance without including contact resistances in the result. With an ohmmeter for example one measures the specimen resistance plus the resistance of the wires going to the specimen plus the contact resistance between the these wires and the specimen. The latter might be large or small, depending on whether the surfaces are oxidized or not and on how hard the contacts are pressed together. Since the contact resistance is unpredictable it must be excluded from the measurement when high accuracy is desired. This is particularly important, obviously, when one expects the measured resistance to go to zero. Figure 3 shows a block diagram of the measuring circuit. In Figure 3a, the voltage parallel to the applied current is measured (longitudinal measurement) while in Figure 3b, the voltage perpendicular to the applied current is measured (transverse measurement). Note that the specimen current is provided by a constant current source, that is, a source for which the current will not change when the specimen resistance changes. This is not an essential feature of the method but is very convenient. "Four-probe" refers to the fact that four independent contacts are made to the specimen. "Independent" means that the only current path between any two contacts lies in the specimen, not in the wires or solder. There might be a substantial resistance between each of the outer, current-supplying contacts and the specimen. Convince yourself that this will not affect the measurement of the resistance of the region between the two inner contacts. High contact resistances at the outer contacts might have other disadvantages, such as high power dissipation and resulting heating, but they will not affect the resistance reading directly. How do you determine whether or not the current is producing excessive heating? You may notice a possible problem with the four-probe circuit. The voltmeter and a section of the specimen are in parallel, so the measuring current will split and some will pass through the meter. Therefore the current in that section of the specimen is not the same as what is measured by the ammeter. Convince yourself that this will not give an error in the resistance calculation. Take into account the fact that a good voltmeter will have a very high resistance. Modern digital voltmeters may have resistances in the tens of megohms range while the typical specimen resistance will be very much less than this. Another possible problem lies in the fact that the voltmeter connections to the specimen might have a contact resistance that is not negligible compared to that of the meter. (Why would this be bad?) If you are not confident that these contact resistances are much smaller than the voltmeter resistance you have a problem. However a contact resistance nearing the megohm range is hardly a contact and normally would give strange and erratic behavior. Incidentally, this discussion shows why one of the measures of the quality of a voltmeter is the size of its internal resistance.







Figure 4: ac longitudinal a) and diagonal b) voltage measurements using the reference output of the lock-in amplifier as a voltage source.



Figure 5: Sample probe stick.

The presence of unwanted thermal volatges in a measuring produce significant errors in circuit can voltage measurements, especially when various parts of the circuit are at very different temperatures. Their presence can be detected by reversing the direction of the current because the thermal voltages will not reverse sign when you do this. In the van der Pauw geometry the electrical contacts are on the perimeter of the sample. Figure 4 shows longitudinal (a) and diagonal (b) electrical measurements using a lock-in amplifier as an ac current source. Note that in this case, the digonal measurement is not truly transverse, since it may contain both the parallel and perpendicular (to the applied current) components of the voltage drop across the sample. Also note that the anisotropy in the shape of the sample (e.g., the sample length is twice its width) is reflected more strongly in anisotropy in the four-probe resistance, as will be discussed Appendix A1.

D. A simple cryogenic system

The cryostat used for performing the temperaturedependent measurement consists of the specimen probe, a

stand to hold the probe, and a well-insulated container partly filled with liquid nitrogen.

In principle, data can be taken during cooling and during warming but students have generally had better results with warming. It seems easier to keep the rate of change of temperature slow and to maintain thermal equilibrium inside the specimen can during warming.

The specimen probe stick (see Fig. 5) is lowered into a sealed metal sample tube that is inside an outer vessel that is filled with liquid nitrogen (LN). The sample makes thermal contact to the LN through a dilute He exchange gas which is present in the sample tube. Typically, the sample tube is completely evacuated after the sample has been inserted. Then, a small amount of helium gas is allowed to enter the sample tube. This is accomplished by attaching a helium gas filled latex tube to the sample tube port, pinching the tube off 10 cm from the port, and finally opening the port valve to allow the pinched off portion of helium gas to enter the sample tube. The port valve is then closed. If it becomes difficult to warm the sample up to higher temperature (i.e., thermal contact between the sample and the surrounding LN is too great), one can pump further on the sample tube to reduce the amount of helium exchange gas, and thereby reduce the thermal connection between the sample and the cold LN. As the specimen slowly warms up, a graph of specimen voltage vs. diode (temperature sensitive) voltage can be collected by the computer. If the warming rate is too slow it can be increased using the heater. Be careful. If the rate is too fast you will not have temperature equilibrium inside the specimen can. Also, the current in the heater wire can generate magnetic fields that may disturb your measurements. Aside from looking for such signs, how can you find out whether the warming rate is too fast or not?

Examine the table to see how linear the diode voltage is with temperature. How well does such a representation show the temperature changes?

E. Vacuum pumping system

A single mechanical pump is used to alternately pump the outer vacuum space or the sample tube (sample space). Valves are provided for pumping and isolating both the sample space and the vacuum isolation space. In addition, helium exchange gas can be added independently to each space to aid in thermal contact, warming, cooling, etc.

If a vacuum is to be maintained in the sample space or in the vacuum isolation can, close the sample space valve and the vacuum space valve before shutting off the pump. After the pump has been turned off, allow a minimum of ten seconds before trying to restart the it. After the pump has been switched off, vent the input to atmosphere to prevent oil from being drawn into the pumping hose.



Figure 6: Cryostat for making low temperature measurements in an external magnetic field.

F. Cryostat

The cryostat (dewar) is used for low temperature measurements. This dewar has three independent spaces as shown in Fig. 6. The first is the vacuum space that is evacuated to provide thermal insulation. The second space consists of a reservoir that contains the liquified cryogen, in this case liquid nitrogen. The third and innermost region is the central sample tube, which contains the sample stick. Typically, one first pumps out the vacuum space for 15 minutes. After that space evacuated, the vacuum space valve is closed and the pump is used to evacuate the sample space. **AFTER** the sample space is evacuated, LN can be poured into the reservoir through the fill funnel. Be sure the sample space is well evacuated, or water may freeze on the sample. To aid in cooling the sample, a small amount of helium exchange gas can be introduced into the sample tube, as described in Section D.

Your instructor will show you how to evacuate the dewar walls. NEVER add liquid nitrogen until this is done.

G. The large electromagnet

This magnet is used to produce static or slowly ramping magnetic fields between -7.5 and +7.5 kG. In principle, the magnet can reach 8 kG, but this maximum field requires a current

approaching 142 A, which is close to the maximum current that the magnet wire can handle. The static magnetic field is set using a crank knob, and the field can be ramped automatically +-50% about the static field. For measurements of the Hall voltage as a function of magnetic field, the magnetic field sensor is connected to a voltmeter, which is accessed by the computer during the field sweep so that the signal as a function of magnetic field can be recorded. The polarity of the magnetic field can be reversed using a knob on the magnet control panel. Please refer to the Varian magnet manual for further details on operating the electromagnet.

H. Data recording

Recording data is important, but it is often tempting to be careless about it. Make up your mind to take the time to do it carefully, fully and neatly. Use a bound notebook. Write the date at the start of a new day. Note the time of the start and the end of a series of measurements such as a warming run. Write all of the relevant information such as specimen description, current, temperature, etc. If you are making many runs, such as Hall resistance at different temperatures, it is useful to record the runs in a tabular format in your lab book. An example table is provided in Appendix A4. Write it before you start, not after. Write comments in the comments box on the LabView program's front panel while you are taking data. Be sure to also record the run and the relevant information in your lab book. COMPUTERS ARE EXTREMELY USEFUL FOR RECORDING DATA, BUT DO NOT RELY SOLEY ON THE COMPUTER TO KEEP TRACK OF YOUR MEASUREMENTS. The date and time should always be recorded by the acquisition software. You may discover later that you neglected to write something important. The time of the plot is often of great help in reconstructing exactly what you did. Do not try to decide whether the run that you are about to begin will be important enough to require writing down all the information. You may realize later that something interesting happened or that it turned out to be the only really good run in some respect. If it is worth doing it is worth taking notes about.

Recommended software:

- LabView- interface software that allows the computer to control and monitor external devices, e.g., multimeter, lockin amplifier, electromagnet, temperature sensor, etc.
- SigmaPlot- data analysis software allows one to analyze and plot data
- Mathcad- mathematical analysis software that can be used to perform analytical as well as numerical calculations.
- Mathematica- similar to Mathcad but has a more powerful for performing analytical/symbolic calculations.
- Microsoft PowerPoint-Useful for creating slides for oral presentations.
- Microsoft Word-Useful for writing up lab reports.
- Mathtype- Convenient tool for writing mathematical expressions within Microsoft Word and PowerPoint.

APPPENDIX A: TRANSPORT MEASUREMENTS

1. Resistance Measurements

An applied electric current density \vec{j} flowing through a material will give rise to an electric field \vec{E} with a constant of proportionality represented by the resisitivity ρ of the material.

$$\vec{E} = \rho \vec{j} \tag{1}$$

By multiplying both sides of Eq. (1) by the length ℓ over which the electric field is applied and replacing the current density *j* with the current *I*, one obtains an expression for the potential difference across the material:

$$E * \ell = \rho j * \ell$$

$$I = jA$$

$$\rightarrow V = E * \ell = \left(\rho \frac{\ell}{A}\right)I = RI$$

$$\rightarrow R = \rho \frac{\ell}{A}$$
(2)

where *A* is the cross sectional area through which the current flows. Here we are assuming that *j* and *E* are uniform (i.e., do not depend on position in the sample). This is just the familiar expression known as Ohm's law. Since resistance is dependent on the sample geometry, it is useful to transform resistance measurements into more fundamental quantities such as resistivity ρ , which is intrinsic to the material and independent of the measurement geometry. Consider a sample consisting of a bar of uniform material with thickness *t*, length ℓ , and width *w* (see Fig. 7). A current *I* is flowing along the length of the bar, producing a voltage V across the length of the bar. The resistance of this sample is:



Figure 7: Longitudinal resistivity measurement configuration



Figure 8: van der Pauw measurement configuration.

$$R = \rho \frac{\ell}{A} = \rho \frac{\ell}{wt} \tag{3}$$

If the bar is perfectly square ($\ell = w$), Eq. (3) simplifies to the form:

$$R = \rho \frac{\ell}{\ell t} = \frac{\rho}{t} = R_{\Box}$$
(4)

 R_{\Box} is independent of the size of the sample (as long as its shape is square), and only depends on the resistivity ρ and the thickness *t* of the sample. Though this is not as fundamental a quantity as ρ , R_{\Box} is very useful for describing thin-film materials (where $t \ll \ell \approx w$).

There are two approaches to obtaining R_{\Box} . One can start with a perfectly square sample and define four contacts (two for injecting/collecting the current and two for measuring voltage). The measured resistance is then equivalent by definition to R_{\Box} . However, samples are rarely perfectly square and the contacts required in this geometry must cover the entire cross sectional area of the sample, which makes them more difficult to define. In the case of arbitrarily shaped samples (including oval and irregularly shaped samples) one can use the result from van der Pauw's paper [13] to obtain R_{\Box} if the following conditions are met:

- 1. The contacts are on the outer perimeter (edges) of the sample.
- 2. The contacts are sufficiently small.
- 3. The sample is homogeneous in thickness *t*.
- 4. The surface of the sample is singly connected (there are no isolated holes).

To measure R_{\Box} using the van der Pauw geometry, one simply attaches four small contacts to the edges of the sample as shown in Fig. 8. Note that the contacts do not have to be at the corners. If the van der Pauw conditions are met, R_{\Box} (which is referred to as the specific resistance and denoted by ρ in the Ref. [13]) can be obtained using the following relation [13]:

$$R_{\Box} = \frac{\pi}{\ln 2} \frac{\left(R_{AB,CD} + R_{BC,DA}\right)}{2} f\left(\frac{R_{AB,CD}}{R_{BC,DA}}\right)$$
(5)

where $R_{AB,CD}$ ($R_{BC,DA}$) is the resistance obtained by injecting/collecting a current through contacts A and B (B and D) and measuring a voltage across contacts C and D (D and A). The function f is a slowly varying function that begins at unity if the argument is 1, and slowly decreases as the argument decreases [13]. In case of a perfectly square sample (i.e., $R_{AB,CD}=R_{BC,DA}$), Eq. (5) reduces to:

$$R_{\Box} = \frac{\pi}{\ln 2} R \tag{6}$$

Note that in the van der Pauw geometry, $R_{AB,CD}$ and $R_{BC,DA}$ depend quadratically (or even more strongly) on the dimensions of the sample. As a result, if the distance between contacts A and B in a rectangular sample is twice the distance between B and C, $R_{AB,CD} \gtrsim 4 R_{BC,DA}$. It is also interesting to explore the conductivity σ of the material, which is simply the reciprocal of the resistivity ρ . In this case Eq. (1) becomes:

$$\left(\frac{1}{\rho}\right)\vec{E} = \vec{j} \to \sigma\vec{E} = \vec{j} \tag{7}$$

In general, σ and ρ are tensors (matrices), which means that \vec{E} and \vec{j} are not necessarily parallel to each other. These tensors can be written in terms of x and y components as:

$$\rho = \begin{pmatrix} \rho_{xx} & \rho_{xy} \\ -\rho_{xy} & \rho_{xx} \end{pmatrix} \qquad \sigma = \begin{pmatrix} \sigma_{xx} & \sigma_{xy} \\ -\sigma_{xy} & \sigma_{xx} \end{pmatrix} \qquad \sigma = \frac{1}{\rho}$$
(8)

One can invert the resistivity tensor to obtain conductivity in terms of resistivities. One can further simplify the expression by taking advantage of the fact that typically $\rho_{xy} \ll \rho_{xx}$.

$$\sigma_{xx} = \frac{\rho_{xx}}{\rho_{xx}^2 + \rho_{xy}^2} \approx \frac{1}{\rho_{xx}}$$

$$\sigma_{xy} = \frac{-\rho_{xy}}{\rho_{xx}^2 + \rho_{xy}^2} \approx \frac{-\rho_{xy}}{\rho_{xx}^2}$$
(9)

Using Eq. (4), one can determine the longitudinal conductivity σ_{xx} in terms of R_{\Box} in the following equation:

$$\sigma_{xx} \approx \frac{1}{\rho_{xx}} = \frac{1}{R_{\Box}t}$$
(10)

For an excellent review of van der Pauw measurements see <u>http://www.eeel.nist.gov/812/effe.htm#vand</u>.

2. Hall Measurements

If magnetic field is **B** applied perpendicular to the film along \hat{z} as shown in Fig. 9, then a current flowing through the sample along \hat{x} will not only produce voltage drop V_x along the current flow, but will also produce a Hall voltage V_{Hall} along \hat{y} that is perpendicular to the current. In simple metals, this measurement allows one to extract the sign and density of the charge carriers that are responsible for the flow of electrical current. The Hall effect is as fundamental and

important in characterizing a material as conventional zero-magnetic field resistance measurements, and can provide information that cannot be accessed with conventional resistance measurements.

The Hall effect is described by Eq. (11), where a current j_x in the x-direction produces a longitudinal electric field E_x in along the current and a tranverse electric field E_y perpendicular to the current.



Figure 9: Hall measurement.

$$\begin{pmatrix} E_x \\ E_y \end{pmatrix} = \begin{pmatrix} \rho_{xx} & \rho_{xy} \\ -\rho_{xy} & \rho_{xx} \end{pmatrix} \begin{pmatrix} j_x \\ 0 \end{pmatrix}$$
(11)

In this case $j_y = 0$ since no current is flowing in the y-direction (there are no sources or sinks to maintain j_y). Note that the off-diagonal components of the resistivity tensor are responsible for the Hall effect, where a current along \hat{x} is transformed into an electric field along \hat{y} . If one expands Eq. (11) into x and y components, one obtains the following result.

$$E_x = \rho_{xx} j_x \tag{12}$$

$$E_{y} = -\rho_{xy}j_{x} \tag{13}$$

Equation (13) can be transformed into an equivalent Ohm's law form using the same techniques that were applied to Eq. (2), where both sides are multiplied by the length ℓ over which the electric field is acting and the current density *j* is replaced by the current *I*. As result, one can define a Hall voltage V_{Hall} as:

$$V_{Hall} = R_{xy}I \tag{14}$$

where R_{xy} is the Hall resistance which translates a current *I* along \hat{x} into a voltage V_{Hall} along \hat{y} . In weak magnetic fields, which is the case in most Hall measurements, R_{xy} is proportional to B and is given by:

$$R_{xy} = \frac{R_H}{t} B \tag{15}$$

where R_H is the Hall constant. For most metals R_H is truly a constant that only depends on carrier density as shown below,

$$R_{H} = -\frac{1}{nec} \tag{16}$$

where n is the density of carriers per unit volume, e is the electric charge of the carriers, and c is the speed of light.

Another important quantity in the Hall effect is the Hall angle θ_H , which is simply related to the ratio of the transverse electric field E_y and the longitudinal electric field E_x .

$$\tan \theta_{H} = \frac{E_{y}}{E_{x}} = \frac{V_{Hall}/w}{V_{x}/\ell} = \frac{\ell}{w} \frac{IR_{xy}}{IR} = \frac{\ell}{w} \frac{R_{xy}}{R}$$
(17)

In Eq. (17), we have used the fact that the magnitude of a uniform electric field is simply the voltage per unit distance (E = V/L) and used Eqs. (2) and (14) to replace voltages with resistances.

From Eqs. (3) and (4) we can convert R into R_{\Box} as follows:

$$R = \rho \frac{\ell}{wd} \quad \text{and} \quad R_{\Box} = \frac{\rho}{t}$$

$$\rightarrow R = R_{\Box} \frac{\ell}{w}$$
(18)

When the result from Eq. (18) is substituted into Eq. (17), the final expression for the Hall angle can be obtained.

$$\tan \theta_{H} = \frac{\ell}{w} \frac{R_{xy}}{R} = \frac{\ell}{w} \frac{R_{xy}}{(R_{\Box} \ell/w)} = \frac{R_{xy}}{R_{\Box}}$$
(19)

The Hall angle can also be expressed in terms of conductivity σ .

$$\sigma \vec{E} = \vec{j}$$

$$\begin{pmatrix} \sigma_{xx} & \sigma_{xy} \\ -\sigma_{xy} & \sigma_{xx} \end{pmatrix} \begin{pmatrix} E_x \\ E_y \end{pmatrix} = \begin{pmatrix} j_x \\ 0 \end{pmatrix}$$
(20)

Solving Eq. (20) for the y-component of j (which is zero in this case) one obtains:

$$-\sigma_{xy}E_{x} + \sigma_{xx}E_{y} = 0$$

$$\sigma_{xy}E_{x} = \sigma_{xx}E_{y} \rightarrow \frac{E_{y}}{E_{x}} = \frac{\sigma_{xy}}{\sigma_{xx}}$$
(21)

Substituting the result from Eq. (21) into Eq. (17) one obtains:

$$\tan \theta_{H} = \frac{E_{y}}{E_{x}} = \frac{\sigma_{xy}}{\sigma_{xx}}$$
(22)

This shows that $tan \theta_{H}$ is simply the ratio of the off-diagonal and diagonal conductivities, σ_{xy} and σ_{xx} , respectively.

For an excellent treatment on dc Hall effect measurements see <u>http://www.eeel.nist.gov/812/hall.html</u>. Note that unlike conventional the resistance *R* found in Ohm's law V=RI, where *R* depends on the sample geometry, R_{xy} is intrinsic to the film. This can be readily seen in Eq. (19). Since $\tan \theta_H$ and R_{\Box} are intrinsic properties of the film (independent of film geometry, except for its thickness), R_{xy} must also be intrinsic to the film, or else $\tan \theta_H$ or R_{\Box} would also be depend on the film geometry.

3. The relationship between σ_{xx} and $tan \theta_{H}$

Though σ_{xx} and $tan\theta_{H}$ do not appear to be related, one can show that for a simple metal that they are proportional to each other. If one were to divide these two parameters one would obtain:

$$\frac{\tan\theta_{H}}{\sigma_{xx}} = \frac{\frac{R_{xy}}{R_{\Box}}}{\frac{1}{R_{\Box}d}} = dR_{xy} = BR_{H} = \text{constant}$$
(23)

Since the B and R_H are constant and independent of temperature, one would expect the ratio of σ_{xx} and $tan \theta_H$ to also be temperature independent. The usually strong temperature dependence of R_{\Box} is cancelled in this ratio since R_{\Box} appears in both σ_{xx} and $tan \theta_H$ in exactly the same way. In HTS, σ_{xx} and $tan \theta_H$ do not share the same temperature dependence and hence \mathbf{R}_H is not constant, but depends on temperature.

4. Measurement Procedure

The previous sections provide a general explanation of resistance and Hall measurements. In this section, the procedure for making these measurements will be mapped out in greater detail.

There are a number of procedures that can be used to measure the temperature dependence of θ_{H} . Here are three possible routes that one could take.

- Constant T, varying B: The temperature is kept at a constant value while the magnetic field is swept from -B to +B (or vice versa). It is critical to keep the temperature as constant as possible, otherwise the change in signal due to temperature changes may overcome the magnetic field-induced changes.
- 2) Constant T, discrete B: instead sweeping magnetic field form –B to +B as in route 1), one could simply measure the diagonal voltage at +B, reverse the magnet polarity and measure the diagonal voltage at –B.
- 3) Constant B, varying T: The magnetic field is kept at a constant value of +B while the while the temperature is swept from room temperature to 77 K (or vice versa). The temperature sweep is repeated with the magnetic field at -B.

In either case, the measurements can be performed using a dc current and measuring a resulting dc Hall voltage, or using an ac current and measuring the resulting ac Hall voltage. A simple table should be kept in your lab book to keep track of the data during the measurements. An example of such a table is provided below.

| Time | Sample | Temperature (K) At start At end of run | $R_{AB,DC} = V_{AB}/I_{DC}$ | $R_{AD,BC} = V_{AD} / I_{BC}$ | Slope of V_{Hall} vs B $(\Delta V_{AC}/\Delta B)$ | I _{BD} Hall | R _{XY} | $tan \theta_H$ |
|------|--------|--|-----------------------------|-------------------------------|---|-------------------------|-----------------|----------------|
| | | | | | | | | |
| | | | | | | | | |



The sample and its four contacts may not be perfectly uniform, therefore one should average all the possible measurement configurations for both resistivity and Hall measurements. The table above is incomplete. For example, one should also measure $V_{Hall}=\Delta V_{BD}$ for current I_{AC} going though contacts A and C. Note that unlike dc measurements, where the signal must be measured for both forward and reverse current directions, ac measurements automatically measure the signal for both forward and reverse polarities.

Figure 10: Sample with four electrical contacts A-D at the corners.

The measured diagonal voltage may not have the simple linear magnetic field dependence that one expects for the

Hall effect. This is the case because the diagonal voltage includes both the longitudinal voltage drop (since one contact may be closer to the current source contact and the other contact may be



Figure 8: The diagonal voltage as a function of magnetic field is shown in (a). The Hall (transverse) contribution to the diagonal voltage is shown by subtracting the signal at negative B from the signal at positive B, and is shown in (b). Note that only the signal that is linear in B remains after the subtraction in (b).

closer to the current drain contact) as well as transverse voltage drop (i.e., Hall voltage). Therefore, the magnetoresistance (change in longitudinal resistance with applied magnetic field) will also be included in diagonal voltage signal. Fortunately, symmetry requires that the magnetoresistance is an even function of magnetic field¹ (e.g., B^0 , B^2 , B^4 ,...) whereas the Hall effect is linear in magnetic field. The diagonal voltage can therefore be represented by an expansion in powers of B as follows:

$$V_{BD}(B) = C_0 + C_1 B + C_2 B^2 + \cdots$$
(24)

The even terms can be readily removed by subtracting the diagonal voltage when the magnetic field direction is reversed,

¹ Magnetoresistance is an even function of B because if one looks at the resistance along the direction of a 1D wire, no change should be observed when the direction of B is reversed.

$$V_{BD}(B) - V_{BD}(-B) = C_0 + C_1 B + C_2 B^2 + \dots - (C_0 - C_1 B + C_2 B^2 + \dots)$$

= 2C_1 B + higher odd powers of B (25)

Note that the slope from the subtracted Hall voltage is twice the slope given in Eqs. (14) and (15). This must be taken into account when calculating R_{xy} .

A schermatic of the wiring used to perform the transport measurement is shown below. In this case, lines A and B are used to inject/collect the ac current from the lock-in's oscillator output into the sample, while the resulting ac voltage is measured between lines E and F.



Figure 11: Example of a longitudinal four-probe ac resistance measurement. AC current is injected/collected through lines A and B, and the resulting ac voltage across sample is measured between lines E and F. RMS current is measured through ammeter (A).

5. First Measurement Assignment

Perform the following measurements on one of the samples to compare various techniques for measuring the resistance of the sample:

- 1) Use an ohmmeter to measure the dc 2-contact resistance
- 2) Use a dc current source through 2 contacts and a voltmeter across the other 2 contacts (longitudinal 4-probe configuration) to determine the dc 4-probe resistance of the sample. How does this compare with the 2-contact resistance? Does this make sense?
- Repeat Step 2) with the measurement contacts rotated by 90 degrees; instead of measuring R_{AB,CD} measure R_{BC,DA}. Is this result consistent with the shape of the sample?
- 4) Use an ac current source (oscillator/reference output from the lockin amplifier) through 2 contacts and the lockin inputs (A-B) across the other 2 contacts (longitudinal 4-probe configuration) to determine the ac 4-probe resistance of the sample. How does this compare with the dc 4-contact resistance?

APPENDIX B: AC MAGNETIC SUSCEPTIBILITY MEASUREMENTS

This lab is not currently configured for ac susceptibility measurements. For completeness, this type of measurement is discussed here, and may be added to the lab in the future. In this section of the laboratory, you will learn the basics of ac susceptometry using the mutual inductance coil method. You will also use a lock-in amplifier, which is essential for this measurement. There are several publications included in the instruction set to familiarize yourself with ac susceptometry and lock-in techniques.

You will either wind your own set of coils, or use a set that has previously been wound by other students. Depending on what kind of samples are available, you will then measure the Meissner—Oschenfeld effect in either powder, ceramic, or crystalline samples of the oxide



Figure 12: AC susceptibility measurement setup.

superconductors. As discussed, this flux expulsion leading to zero internal magnetic field is the most fundamental property of the superconducting state.

The coil arrangement will in all likelihood be imprecise, in that there will be some finite mismatch between the secondary coils. Therefore, you should determine the temperature dependence of this mismatch with no sample inside; then proceed to mount a sample into one of the secondary coils, and repeat the measurement. The difference between these two results is related to the magnetic susceptibility of the sample material. Use the #4 gelatin capsules to house the samples so that no material spills out near the coils.

Once the Meissner effect has been observed and recorded, you should estimate the amount of flux expulsion in the sample. That is, determine the volume fraction of superconductivity. This requires knowledge of the mass and density of the sample under study. You can use theoretical values as the upper limit for the density, then estimate or comment on the realistic situation (i.e. the ceramics are not 1007. dense, so the actual volume of material is not the measured mass divided by the theoretical density). If you have time, you should investigate the frequency dependence of this determination. Also, should discuss you the shape of the

superconducting—normal transition with regard to sample purity and minority phases, if any.

APPENDIX C: RESISTANCE MEASUREMENT WORKSHEET



Figure 13: Sample with four electrical contacts A-D at the corners.

Consider a sample with four contacts (one at each corner as shown in Fig. 13). The following measurements and questions will help you test the experimental system and should help clarify the various kinds of resistivity measurements that can be made on such a sample. You should think about where the current is flowing in the sample and how the lines of euipootential are distributed across the sample.

- 1. **Two probe dc resistance measurement-** Using an ohmmeter measure the following resistances: R_{AB} (resistance between contacts A and B), R_{AD}, R_{BA}, and R_{AC}. Note that the sample may not be square as shown in Fig. 13. How do the values of these resistances compare with each other? Does this make sense given the geometry of the sample?
- 2. Four probe dc resistance measurement- Using a dc current source (Keithley Programmable Current Source) to inject 1 mA current through two contacts and a voltmeter (HP digital multimeter) measure the following resistances: R_{ABDC} (current through contacts A and B, voltage across contacts D and C), R_{ABAB}, R_{ADBC}, R_{ACBD}. Recall that R=V/I. Do the relative values of these resistances make sense given the geometry of the sample? How do these resistances compare with the two probe resistances that you measured in part 1?
- 3. Four probe ac resistance measurement- Using the reference output of the locking amplifier as an ac current source to inject 1 mA (RMS) current through two contacts and inputs A/B on the lockin amplifier to measure the following resistances: R_{ABDC} (current through contacts A and B, voltage across contacts D and C), R_{ABAB}, R_{ADBC}, R_{ACBD}. Recall that R=V/I. Do the relative values of these resistances make sense given the geometry of the sample? How do these resistances compare with the four probe dc resistances that you measured in part 2?

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