Advanced Laboratory
Dept. of Physics & Astronomy
Stony Brook University

(Jan. 18, 2006)

Pre-experiment and mid-experiment evaluation sheet

You must meet with a staff member for the experiment you are doing before beginning an experiment and before the lab period indicted in the middle of the experiment for an evaluation of your preparation and your progress to date. This form must be signed and dated by the staff member to indicate you have done the required work to date. It must be attached as the first page of your report. You will loose one grade point from your report grade for each section of this form that is not properly signed on time. If this is your first experiment you should also be prepared to demonstrate you have read and understood the information on log books in the course notes.

Superconductivity

Student name____

Partner name____

Pre-lab discussion:

Be prepared to demonstrate you have read the background material for this experiment by discussing the following questions with the staff:

1. What does the I-V curve of a Josephson junction look like? Identify the critical current and the gap voltage.
2. How does the gap voltage of Nb vary with temperature. Be quantitative.
3. What are 2 possible ways you might be injured by liquid helium or nitrogen.

Completed before first period for the experiment?   Yes/No

Staff comments____

Staff signature______________________________________Date____________________

Mid-lab mini-report:

Due at least one period before you begin work with the real junctions.

Use the apparatus to determine the resistance of one of the resistors in the ersatz probe. The most important part of this report is to present a clear and correct analysis to the accuracy of your measurement to demonstrate your understanding of the apparatus. Consider both random and systematic errors. You must be quantitative, stating how you determined the contribution of each error source and then calculated the final error in R.

Completed before fifth period for the experiment?   Yes/No

Staff comments:____

Staff signature______________________________________Date____________________

Include this sheet as the first page of your final report for the experiment.

Superconductivity in Niobium
(Revised Oct. 10, 2006–draft)

I. INTRODUCTION:

Superconductors can be described, phenomenologically, by the two fluid model, in which the metal contains a mixture of normal and superconducting electrons. Below a transition temperature T_c normal electrons begin condensing into superconducting pairs, this creates a gap 2Δ in the normal electron (or quasiparticle) energy spectrum much as the band gap in a semiconductor. As the temperature decreases and a greater fraction of the normal electrons condense into pairs, the energy gap widens. This gap can be observed in the I-V curves of tunnel junctions with superconducting electrodes, since the quasiparticle current is blocked by the gap for voltages $V < 2\Delta$. The microscopic theory leading to this gap will likely be beyond you unless you have a strong background in solid state physics. The results of the theory along with a phenomenological discussion of quasiparticle tunneling are presented e.g. in Van Duzer [1] sec. 2.11–2.16.

In the experiment you will measure the I–V curves of niobium tunnel junctions (see Fig. 1) for temperatures ranging from 4.2 K to over 9 K enabling you to determine the temperature dependence of Δ along with that of both the pair and quasiparticle tunnel currents. For "weak coupling" superconductors the value of the gap at $T = 0$ is related to the superconducting transition temperature T_c by

$$\Delta(0) = 1.76 k_B T_c \tag{1}$$

For Nb, some corrections to weak coupling results are required. This is discussed e.g. in Ref. [3]. The exact properties of the Nb depend on just how the films were made, but [3] should serve as a guide to what sort of corrections to expect. The ratio $\delta \equiv \Delta(T)/\Delta(0)$ of the gap at finite and zero temperatures is a universal function of $t \equiv T/T_c$. δ is given implicitly by an integral equation: Van Duzer 2.11 Eq. 3. The numerical solutions to this equation have been published [2] . Near T_c there is an analytic form for this ,

$$\delta = 1.74(1-t)^{1/2} \tag{2}$$

This result is valid only very near to T_c. An empirical formula, which fits the gap function rather well thought the whole temperature range is given in Ref. [4] as $\delta^2 = \cos(\frac{\pi}{2}t^2)$. Another, very good, approximate formula for the gap is given in ref. [6]. In addition to the quasiparticle tunnel current, the junction also carries a supercurrent of paired electrons which flows even when $V = 0$ between the electrodes of the junction. Junctions which carry supercurrents are called Josephson junctions and are discussed in Van Duzer, chapter 4. The maximum value of the this zero voltage current, I_c, is related to the energy gap by,

$$I_c = \frac{\pi\Delta(T)}{2eR_n} \tanh\left(\frac{\Delta(T)}{2k_BT}\right) \tag{3}$$

where R_n is the junction resistance for voltages well above the gap voltage.

A second striking feature of superconductors, in addition to zero resistance, is the Meissner effect, that is, the expulsion of magnetic flux from bulk superconductors. A magnetic

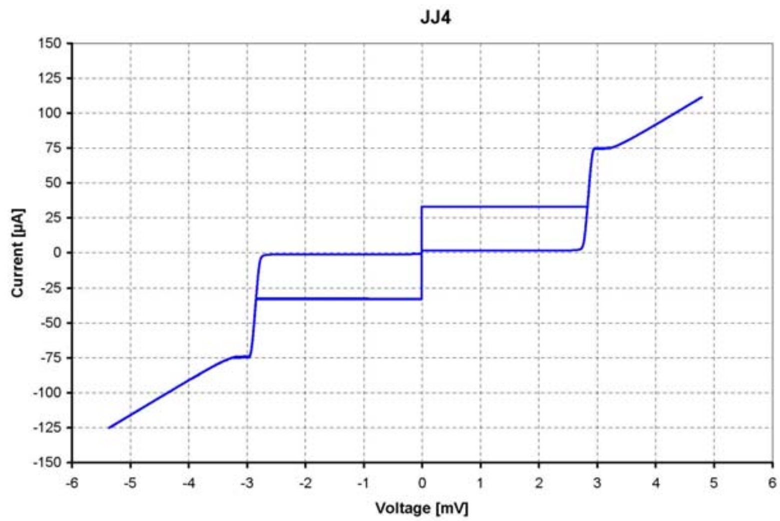


FIG. 1: I-V curve of one of the Josephson junctions available in the probe. Note the critical current I_c of about 30 μA and the current jump at the gap near $V = 2.8$ mV

field will only penetrate into a superconductor for a depth of the order of 100nm (for niobium) called the penetration depth, λ. This depth depends on the density of superconducting pairs and is therefore temperature dependent. A phenomenological discussion of this effect is presented in chapter 3 of Van Duzer. λ can be measured from the magnetic field dependence of I_c of Josephson junctions. As discussed in Van Duzer 4.05, a magnetic field applied in the plane of the junction will modulate I_c in the form of a Fraunhofer diffraction pattern. The minima of this pattern are separated by the fields needed to create one quantum of magnetic flux $\Phi_0 = 2.07 \times 10^{-15}$Wb through the junction. The flux through the junction is $\Phi = wt_{ef}B$, where w is the junction width, B is the magnetic field and t_{ef} is the effective thickness of the barrier in the junction. $t_{ef} = 2\lambda + d$ where d is the oxide thickness of about 5nm. Thus, the periodicity of I_c vs. B enables one to measure λ.

To do a reasonable job on this experiment, one must understand something of the theory of superconductors and Josephson junctions as referenced above. Since this material is not usually covered in standard courses, *you should plan to spend a fair amount of time reading the reference material if you do this experiment.*

A. Goals

Some of the properties of Nb and of Josephson junctions that you can investigate in this experiment are:

- Determination of the temperature dependence of $\Delta(T)$ and $I_c(T)$ from 4.2 K to T_c.
- Study of the variation of $I_c(T)$ with junction size and with magnetic field. This will allow you to determine $\lambda(T)$.
- Qualitatively study how temperature affects the shape of the I-V curve and understand why the things you observe happen.

B. Procedure

- Do some reading on superconductivity and the Josephson effect so you have an idea what to expect.
- Spend several lab periods mastering the electronics and the LabView program for the experiment. During this time the electronics cable from the rack will be connected to the "ersatz probe" box (see below). Do not connect it to the actual probe with the junctions until you have the approval from an instructor. *The junctions can be destroyed by improper use of the electronics.*
- Request a dewar of He from Frank once you are nearly done figuring out the electronics. It sometimes takes up to a week after your request before the He can be supplied. So plan ahead. Note that all of the circuits should be tested once the probe is cooled to 77 K to ensure that they work. Things sometimes go wrong when cooling the probe down due, mainly, to thermal contraction.
- Frank will help you cool the probe and put in into the He storage Dewar. You should read the instructions for doing this however (appendix B) so you understand what is going on and why. You need to arrange with Frank to come in for a brief period before the start of lab for some of the initial steps of the cool down. Once the probe in the He Dewar, it will most likely be left there for the rest of the experiment.
- Plan to spend the first period with He making measurements at 4.2K. Once you are finished with that and ready to take data at higher temperature, it will be necessary to pump the He gas out of the vacuum can. The staff will help you with this.
- Analyze your data as you take it as best you can. Certainly do a careful analysis between lab periods. Only after you have done this analysis will you know if you have the data you need.
- At the end of each lab period when you are measuring the junctions, be sure the shorting switch (see below) is set to "short" and the vacuum value on the top of the probe is closed.

II. APPARATUS

The main components of the apparatus are

- The cryostat probe that is immersed in liquid He. This has a vacuum can containing the junctions along with the heater and thermometer for temperature regulation.
- A pumping system with a roughing pump and a diffusion pump to evacuate the vacuum can for thermal isolation from the He bath.
- The amplifier rack, with several circuit boxes mounted on it. The box labeled "PROBE" is connected to the cable that provides the electrical leads to the probe,
- Differential amplifiers, reference resistors (mounted in boxes on the amplifier rack)

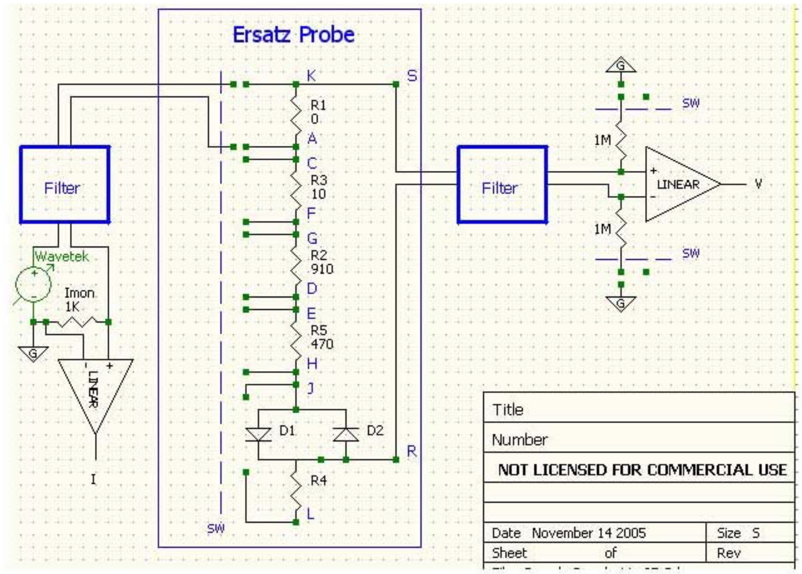


FIG. 2: Schematic of the "pseudo-sample" circuits in the "Ersatz Probe" box on the rack. The various circuits are selected using the 5 positon switch.

- The "ERSATZ PROBE", which can be used to test your measurement circuit without using the real probe
- A DC voltage source (labeled "Thermometer source" and located on amplifier rack),
- A "General Resistance" decade resistor, and 2 Keithley R195A multimeter, for temperature measurements
- Wavetek sweep generator serving as junction I-V curve source
- HP digital oscilloscope
- Two HP 34401A multimeters and computer for I-V curve measurement
- Power supplies for the heater and the magnet
- "General Radio" decade resistor and one Keithley 195A multimeter (one of those used for measuremnt of T) for magnet current measurement

Some of the cables (Twinax) used to connect the circuit elements have TWO leads in the shielding. These low noise cables and connectors look similar to the regular BNC cables and connectors, except for the blue color. When making connections with these cables, the two inner leads of the connector have to be matched carefully. *Do not twist the twinax connectors while inserting the plug since the pins can be broken.*

The main switch of the amplifier rack is on the left back side.

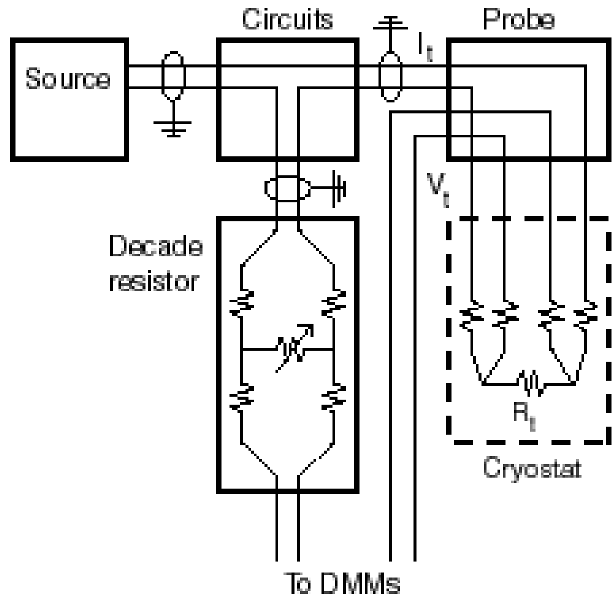


FIG. 3: Schematic of the temperature measurement circuit

A. Temperature measurements and regulation

Temperature is measured using a Lake Shore germanium resistor thermometer[9] that has a well defined relationship between resistance and temperature. Calibration tables for the thermometer are located with the equipment manuals for the experiment (in the red folder) and in the folder for this experiment on the web site. Chapter 7 of the HP DMM manual[10] has useful information about measurement techniques in general.

The thermometer resistance R_T is measured using Keithley digital multimeters (DMMs) to measure the current and voltage across the thermometer in a four wire resistance measurement. The variable voltage source provides a current I_T which is measured by using a DMM to measure the voltage drop across a known resistance provided by a decade resistor. (This General Resistance decade resistor has a resistance that corresponds accurately to its front panel settings–see data sheet. But this should still be checked with a separate four wire measurement.) V_T is measured using a DMM directly connected to the voltage terminals of R_T. Note that the voltage across the thermometer should be limited ($V_T < 3$mV) in order to avoid self heating. The tolerable voltage depends both on the temperature and on whether you want high accuracy or precision in your measurement. You can practice controlling V_T and measuring R_T using the "ersatz probe", which contains a previously measured resistor (2013 Ω) to simulate the thermometer. It also contains a 824 Ω resistor connected to the "heater" input to enable you to test the heater circuit.

Whether one or two DMMs are used for the temperature measurement depends on whether the second Keithley is being used for magnet current measurements. If only one DMM is available, then I_T and V_T measurements can be made without having to switch cables by using the rear terminals of the DMM for one measurement. There is a switch on the back to toggle which terminals are active.

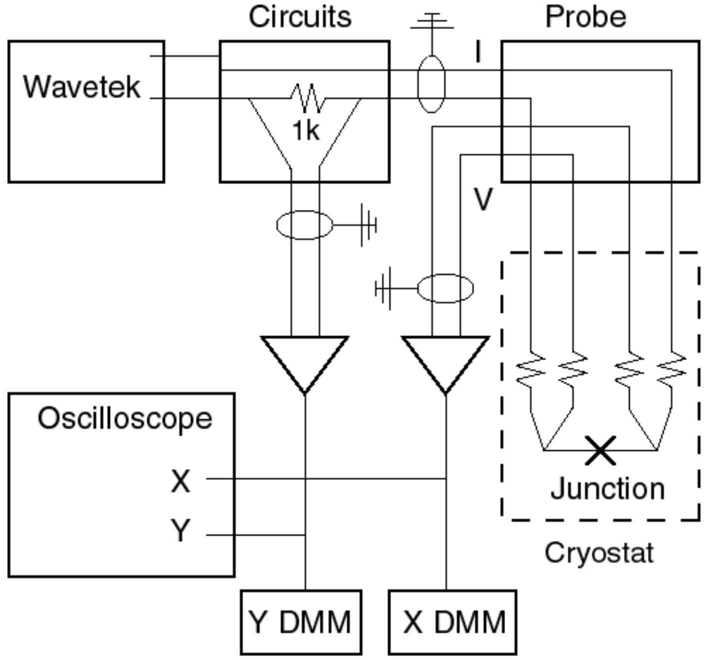


FIG. 4: Schematic of the setup for the measurement of the junction I-V curve

B. Current-voltage curve measurement

The current source for the I-V curve measurements of the junctions is a Wavetek sweep generator (function generator). The current is read by measuring the voltage drop across a 1 kΩ resistor in the "CIRCUITS" box. See Fig.4. Current and voltage signals are amplified and isolated from ground by differential amplifiers. These amplifier outputs are then displayed on an oscilloscope operating in the XY mode and monitored by two HP 34401A DMMs. These DMMs, along with the Keithley DMM reading magnet current are controlled and read by a computer through a GPIB bus using a graphical program created using LabVIEW. Section II D contains information about this program and I-V measurement techniques.

The junctions, which are located on a silicon chip in thermal contact with the temperature controlled platform, were fabricated at Stony Brook using a process that is described in Ref. [11]. It is important for you to convince yourself that the sample and the platform are at the same temperature during your measurements. One possible cause for a temperature difference is the presence of helium gas in the vacuum can. This gas is put in the can in order to provide thermal contact while cooling the probe. Its presence provides a direct thermal path from the sample to the He bath—bypassing the temperature regulated platform. It is necessary to pump on the vacuum can for at least $\frac{1}{2}$ hour with the diffusion pump in order to adequately remove this gas.

Five junctions with a range of sizes are connected. The wiring diagram for the junctions in the probe along with a micrograph of the junctions is available on the course web site. The current step at the gap voltage (see Fig.1) is proportional to the junction areas. You can use this information, along with the ratios of the current step amplitudes to figure out just which junctions are connected. A particular junction is selected though the 5 position switch on the "PROBE" box located on the amplifier relay rack. This switch simultaneously

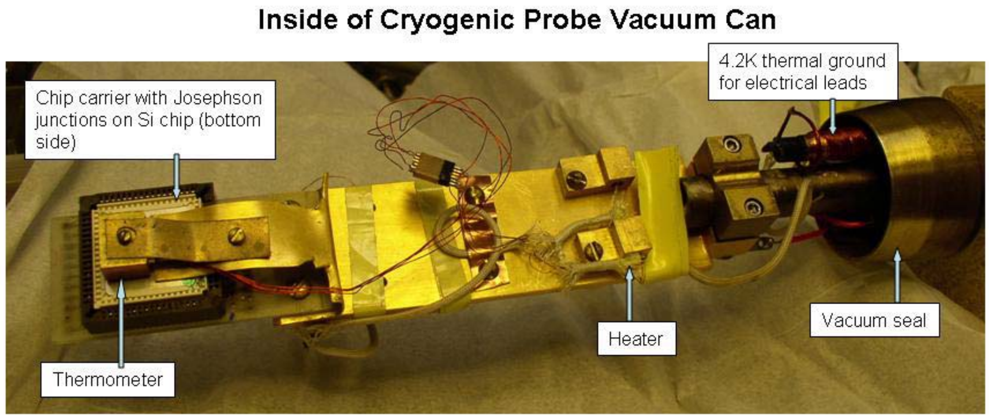


FIG. 5: Sample stage inside vacuum can

switches the current and voltage leads. *You must always place the shorting switch (just below the 5 throw switch) in the shorted position before switching junctions*

C. Cryostat

The Josephson junctions are located on a vacuum isolated platform on a probe which can be inserted into a liquid He storage Dewar. There is weak thermal coupling between the platform and the He bath provided by a copper wire. The temperature of the platform is measured using a Ge resistance thermometer. You can regulate the platform temperature above the He bath temperature (4.2 K) by supplying current though a 1 kΩ heater on the platform. There is a small superconducting magnet (not shown) surrounding the vacuum can. According to our calibration, 1 mA current corresponds to 0.25 G $\pm$ 5%. This current can be measured by connecting the "General Radio" decade resistor in series with the magnet and monitoring the voltage using a Keithley 195A DMM (the DMM does not have current measurement capabilities—it needs to have an accessory installed to do this). The magnet will supply fields up to about 125 G parallel to the plane of the junctions. Do not leave the magnetic field turned up, unless you need it, since significant power is dissipated in the magnet, boiling off the helium unnecessarily. One of the staff will assist you in cooling down the probe and inserting it into the Dewar. Do not attempt to do this yourself.

D. Computer controlled measurement and techniques [12]

The LabView "virtual instrument" (VI) used to make I-V curve measurements is called "IV Measurements" and should be located on the desktop of the computer at the lab station. The VI can be loaded by double-clicking on the icon (click OK in the user name dialog box). Once the VI is loaded, execution needs to be started by clicking on the small arrow at the upper left of the screen. Make sure that all of the DMMs that it communicates with are on and functioning normally before doing this. Also, make sure that the GPIB addresses entered in the VI are the same as those of the intended DMMs themselves (the

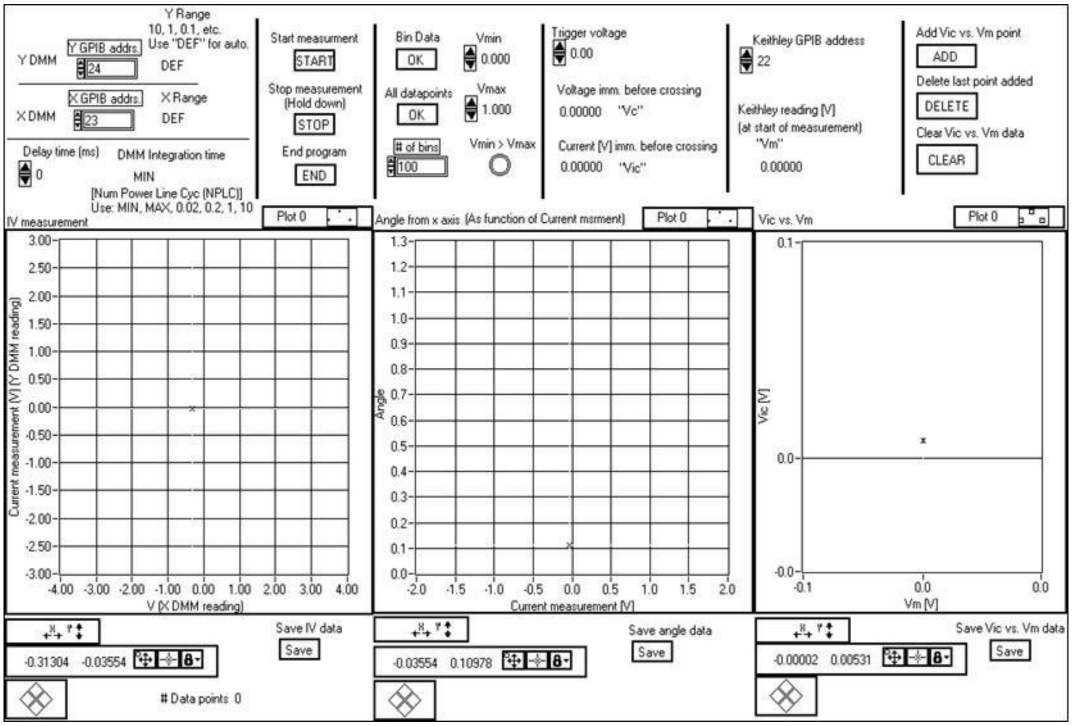


FIG. 6: Front panel display of the “IV Measurements" Labview program

current GPIB addresses of the DMMs are flashed briefly after they are turned on). Should the VI ever perform unexpectedly, execution can be stopped by clicking on the red “stop sign” box next to the arrow. Sometimes, if unexpected behavior continues on restart, it helps to exit LabView entirely and reload the VI. Also, execution of the VI should be stopped whenever not in use for long periods of time.

Before data is taken with the computer, the oscilloscope should be used to check that the I-V curve of interest is being swept out. Usually, the Wavetek should be set to a slowly varying (around or less than 0.05 Hz) triangle wave that produces a varying current with an initial amplitude of about 100 μA. To see the full I-V curve on the scope, the Wavetek frequency can be increased to tens of Hz. Make sure at all times that the low-pass filters on the amplifiers are not distorting the shape of the I-V curve.

To start and stop taking measurements, click on the appropriate buttons (not the ones that start and stop execution of the entire VI) located near the top left of the screen (the stop button might need to be held down for a few seconds if reading are being taken slowly). Be aware that the X and Y readings for the I-V curve shown are taken directly from the DMMs, which means that the Y voltage readings will have to be converted to current in later analysis of the data. The way measurements are taken is set in the upper left corner of the VI. The most important setting is the “DMM integration time”, which controls how long it takes for the DMMs to make a single measurement. The unit of this field is number of power line cycles (NPLC), so 1 should correspond to about 1/60 of a second. This does not necessarily mean that samples will be taken at a rate of 60 per second with a setting of 1 NPLC though—communication between the computer and DMM, and internal, time-consuming processes of both slow the rate of sampling significantly. An estimate of the true sampling rate can be obtained by timing a sampling run and looking at the number of samples reported in the "# of data points" field. In general, fast sampling rates (and slow sweep frequencies) are desired when one is trying to measure the critical current accurately in the regime where the crossover happens very abruptly, while longer integration times (which eliminate some noise from the signal) are better when trying to measure the slope of the I-V curve.

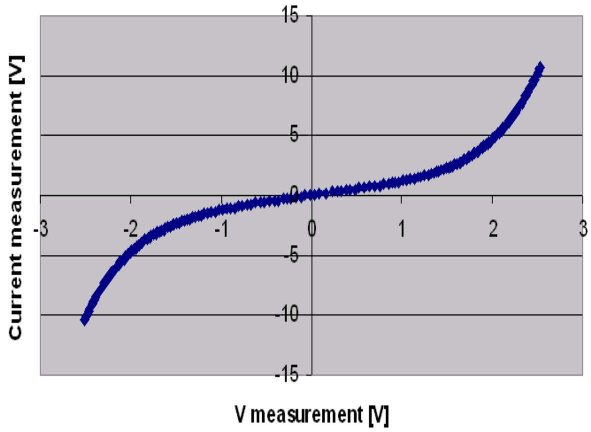


Fig. 7: I-V curve. Values shown are the ones read directly from the DMMs, so should indicate a "junction" voltage sweep with an amplitude of about 0.25 V, and a current sweep of amplitude 1 mA. Chart was made in Microsoft Excel after importing data saved in VI.

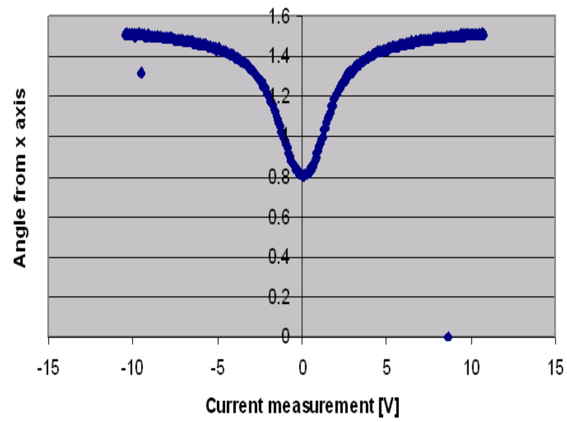


Fig. 8: Angle measurement from the data in Fig. 7

The measurement ranges of the X and Y DMMs can also be set in the upper left side area. To set the DMMs to autoranging, enter "DEF" into the boxes. Be aware that there is a slight delay when a DMM switches from one range to the next though. If this happens during a sampling run, one DMM will measure a voltage for a single data point significantly later than the other DMM, making this data point faulty. Note also that there is in fact no way to assure that any X and Y DMM measurements are taken exactly simultaneously, and there is definitely a very short delay between the X and Y measurements of each point caused by the fact that the computer can only tell one DMM to start taking a measurement at a time. This delay is detectable at high sweep rates, and should introduce a systematic error that is insignificant if the sweep rate is slow enough.

One way that the critical current can be measured is by detecting at which point the junction voltage abruptly jumps away from zero and looking at the last reading taken before this jump. This can be done by setting a trigger voltage a little above (or below) zero in the box labeled "Trigger voltage". When the "Stop measurement" button is pressed, the VI scans through the data from the X DMM to find the latest instance at which the data values cross from one side of the trigger value to the other. It then reports the x and y values of the sample point taken immediately before this crossover. To check the correctness of this report, as well as estimate the resolution and accuracy of this measurement of the critical current, zoom in on the portion of the I-V curve right before and after the voltage jump. A zoom box tool can be selected from the palette located directly below the graph. A panning tool and cursor control tool can also be selected from this palette. The two boxes below display the cursor position, and give other options for cursor control. Clicking on the box to the upper right of a graph or right-clicking on the graph itself gives more options for graph display and functionality. (Note that it is not recommended to use AutoScale when taking data because the constant rescaling slows the sampling rate.)

To the right of the start and stop buttons are controls for binning the data. The range between specified V_{min} and V_{max} is split into the number of bins specified, and the Y values of all points in each bin are averaged to create one point for that bin. This will not necessarily be useful for this experiment. Usually, specifying a longer integration time for the DMMs is the best way to reduce noise in the data. Note that the original data can be recovered after pressing the "Bin Data" button by pressing the "All datapoints" button, but that this won't work if the "Bin Data" button is pressed twice in a row.

When the stop button in pressed, another thing that VI does is calculate the discrete derivative of the I-V curve by calculating $\Delta Y/\Delta X$ for each point and its next neighbor. The first and last derivative measurements should, of course, not be trusted. The derivative is then converted to an angle away from the x axis (the arctangent of the derivative is taken) before being displayed. This allows one to not have to worry about very large slopes while still being able to locate local minima and maxima in the derivative. The angle is displayed as a function of the current (y) measurement.

When the start button is pressed, the VI takes a single measurement from the Keithley DMM (the one with the GPIB cord connected at the back) and displays it in the box titled "Keithley reading [V]". This allows the current through the magnet coil to be automatically recorded along with the critical current of the junction in the corresponding magnetic field. After the stop button has been pressed, the Keithley reading ("Vm"), and current before crossing reading ("Vic") can be recorded by pressing the "Add Vic vs. Vm" button. This can be done for many runs, and displays the data in the graph on the far right. Data points in this graph can be deleted one by one, or can be cleared altogether by pressing the appropriate buttons.

In order to save data, click on the appropriate buttons below the graphs. Saved files are tab delimited text files that can easily be imported into many analysis programs, including Microsoft Excel, which is present on the computer at the lab station. Just open the file, and a wizard will take care of importing the data.

III. REFERENCES

[1] Principles of Superconductive Devices and Circuits, T. Van Duzer and C.W. Turner, Elsevier (1981). This book is on reserve for this course.

[2] B. Muhlschlegel, Z. Physik, **155**, 313 (1959).

[3] Thomas P. Sheahens, Phys. Rev. B, **149**, 370 (1966).

[4] Thomas P. Sheahens, Phys. Rev. B, **149**, 368 (1966).

[5] M.B. Ketchen et al., Appl. Phys. Lett., **59**, 2609 (1991)

[6] R.F. Broom, "Some Temperature-dependent Properties of Niobium Tunnel Junctions", J. Appl. Phys. **47**, 5432 (1976)

[7] D.K. Kim, et al., "Resistive measurements of the Temperature Dependence of the Penetration Depth of Nb in Nb/AlO$_x$/Nb Josephson Junctions", J. Appl. Phys. **75**, 8163 (1994).

[8] J. Matisso, "Critical Currents and Current Disributions in Josephson Junctions", J. Appl. Phys. **40**, 1813 (1969).

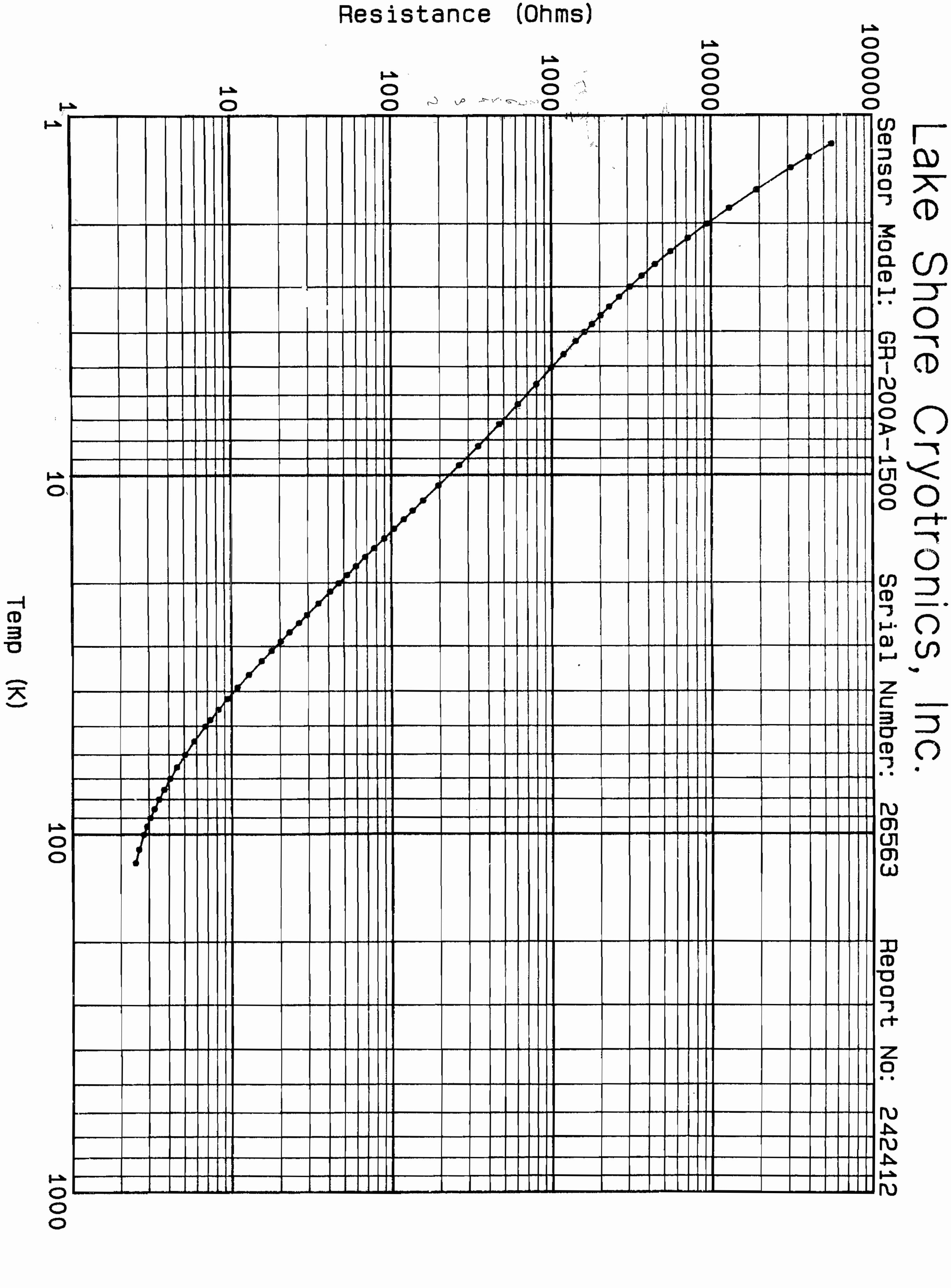
[9] Lake Shore Cryogenics germanium reisistance thermometer. The manual is on the course web site in the superconductivity folder.

[10] HP DMM users manual in the wall case behind the rack.

[11] Vijay Patel, Wei Chen, Shawn Pottorf, and James E. Lukens, "A Fast Turn-Around Time Process for Fabrication of Qubit Circuits", IEEE Trans. Appl. Supercond. **15**, 117 (2005).

[12] This settup, along with the initial writeup, for this section are the work of Daniel Flickinger, who did this as a course project in the spring of 2004.

Appendix A


Lake Shore Cryotronics, Inc.
Sensor Model: GR-200A-1500
Serial Number: 26563
Report No: 242412
Resistance (Ohms)
Temp (K)
1
10
100
1000
10000
100000
1
10
100
1000

Appendix B: Instructions for cooling probe

(The staff will help you with this. Do not do it on your own.)

Preparation:

- Seal probe and evacuate to 100 mT using roughing pump.
- Shut value to roughing pump on pumping station (leave probe value open).
- Wait 5 minutes. The pressure should not increase to more than 150 mT.
- While you wait, connect the cable to the probe (remember to have the shorting switch set to "short") and measure the resistance of the thermometer. Also check that the heater and magnet circuits are working.

Cooling to 77K:

- Backfill probe with "six inches" of nitrogen gas (from 50l LN dewar). The pressure on the roughing gage should be about 1000 mT.
- Put the probe into 5l LN dewar and let it cool for about ½ hour. Monitor the pressure and thermometer. If the pressure increases there is a leak and you have to start over.
- Set the output attenuator on the Wavetek to -30db. Set the shorting switch to "connect" and measure the I-V curves of the junctions (which should look ohmic) to be sure they are connected. Recheck the heater and magnet circuits.

Cooling to 4.2K

- Pump the nitrogen out of the probe (pressure < 100 mT) and fill to 1000 mT with He gas.
- Shut the valve on the top of the probe and disconnect the pumping line. Note: The roughing value on the pumping station should also be shut.
- Slide the "Triclover" flange on the probe shaft down to about 4" above the top of the vacuum can. Be sure the shaft coupling is tight so the flange will not slip.
- Be sure the He dewar in ready. Then rapidly take the probe out of the LN and insert it in the He dewar. Note: Be sure to follow the precautions for LHe use.
- Watch the ping pong ball to decide when and how fast to lower the probe into the He dewar. The ball should not rise above ½ height. How fast you lower the probe depends on the He level in the dewar. It should take about 10-15

minute to lower the probe to the top of the liquid. Then it can be lowered to the bottom on the dewar without further delay.

- Monitor the temperature and the junction I-V curve as you lower the probe. The resistance of the I-V curve will drop rapidly at the T_c of Nb (about 9K). Try to cool the probe slowly through this region (by controlling its height) so you can get a reasonable measurement of T_c.
- As soon as the pumping line will reach the probe, reconnect it. Evacuate it to 50 mT. Then close the roughing valve and monitor the pressure for several minutes to be sure the connectors do not leak.
- Open the value on the probe. The pressure should be about 100 mT. If not, there is a leak and you not be able to take data for $T > 4.2$K until it is fixed.
- If the probe is not yet inserted all the way to bottom of the dewar, do so now. You are now ready to start measuring.
- After you have completed your measurements at 4.2 K, you must use the diffusion pump to evacuate the remaining He gas in the vacuum can so that the only thermal contact between the temperature-regulated platform and the He bath is the wire at the top of the platform. The staff will help you with this. It takes about ½ hour for the diffusion pump to warm up and them about ½ hour to pump the gas out.