Low Temperature Physics Experiment

March 2001 Revision

Objective

This experiment has two objectives 1: To measure the temperature dependence of the resistivity of a good metal either Indium (In) or Tin (Sn), between room temperature (~295 K) and the lowest temperature we can achieve in the Advanced Lab (~ 1.2 K). (Please note that this is about 2.5 times colder than the lowest naturally occurring temperature in the universe.) Part of the goal is to find the superconducting transition temperature. The rest of the temperature dependence is interesting, too. 2: To measure the heat capacity of liquid helium as a function of temperature. Thermodynamic phase changes often produce extraordinary features in the temperature dependence of the heat capacity. There is such a phase change in liquid ^{4}He between two different liquid states. The idea of two different liquid phases was so unprecedented in its day that this phase transition went undiscovered for the first 30 years of liquid helium research despite intensive research on the liquid by scores of experimentalists.

Overview

The first step is to make an In or Sn sample suitable for a resistance study. Because the resistivity of a metal is typically quite small, in the range from 10^{-6} to 10^{-5} Ω-cm at room temperature, in order to have a sample with a reasonable resistance it is necessary to make a sample with a long length and a small cross sectional area. You will do this by condensing a thin film of metal vapor onto a glass slide.

Next you will have to construct an appropriate ohmmeter. You will make what is called an ac, four probe resistance measurement using a lock-in amplifier. You will need to measure the resistance of your sample through rather long, high resistance wires. Four probe resistance measurements keep the resistance of the wires from disguising the resistance of the sample. We use an ac technique to eliminate thermoelectric voltages that occur along temperature gradients. The ac approach also allows the measurement of very small voltages which makes it possible to measure the resistance of the sample with small currents. This minimizes Joule heating and critical current effects.

Next the sample is mounted in a specialized container, called a cryostat or dewar, and cooled in stages from room temperature to a temperature near 1 K. The first stage is done by slowly cooling the sample using liquid nitrogen. This process takes overnight and leaves the sample at about 80 K. Then the sample is cooled over a period of a few minutes to 4.2 K by immersing it in boil-

ing liquid ^{4}He. It is further cooled by reducing the pressure of the liquid helium with a vacuum pump. This takes an hour or so. Once the lowest temperature is reached, the dewar is closed off from the pumping line and a constant electrical power is dissipated in a resistor near the bottom of the cryostat. The helium warms up at a rate that depends on its heat capacity. Typically this takes of order half an hour. Finally, the sample and cryostat are allowed to slowly return to room temperature over a period of a couple of days. The resistance of the sample is continuously monitored throughout all steps of the temperature cycle by a computer running a labview program. It also monitors the temperature, as measured by a silicon diode resistor, and the pressure in the dewar. Below 4.2 K and below, the most accurate temperature measurements are derived from the pressure data.

At this point, you will have all the data you need to describe the temperature dependence of the resistance of your sample from 295 K to nearly 1.2 K. In addition you will have data that you can analyze to extract the heat capacity of the liquid helium between about 1.4 K and 4.2 K.

Finish your lab report and its over.

About this Lab Manual

This lab manual provides the background material that you will need. It is incredibly wordy but it is probably best if you read it all carefully. It is long because its goal is to explain everything about the experiment, not just tell you the bare bones of what to do. In places the manual provides detailed step-by-step instructions. These are the parts of the experiment where mistakes will ruin equipment and/or the procedures are so exotic that no one could work them out for himself in the three weeks the experiment takes. In other places the manual just imparts the general idea. This leaves the responsibility of deciding exactly what to do with you. You should realize that talking to the instructor about your plans is always an option and frequently a very good idea. The hope is that at the end you will have done a successful experiment, understand why you did what you did and how all the stuff you used works. There is also a good deal of physics in both what you'd think of as the experiment, and in the procedures and the measuring devices. You should learn all of that, too.

Procedure

Superconductivity Sample Preparation

A. Overview

To make a high resistance sample of low resistivity metal one must make a long sample with a small cross sectional area. A good way to do this is to evaporate a thin film of the metal onto an

insulating substrate. Our set up, which is one typical of research laboratories, is shown in Fig. 1.

Glass Cover

Glass Slide

Mask

Film Thickness Monitor

Lid

Glass Bell Jar

Ring Stand

Current Feed Thru

Boat
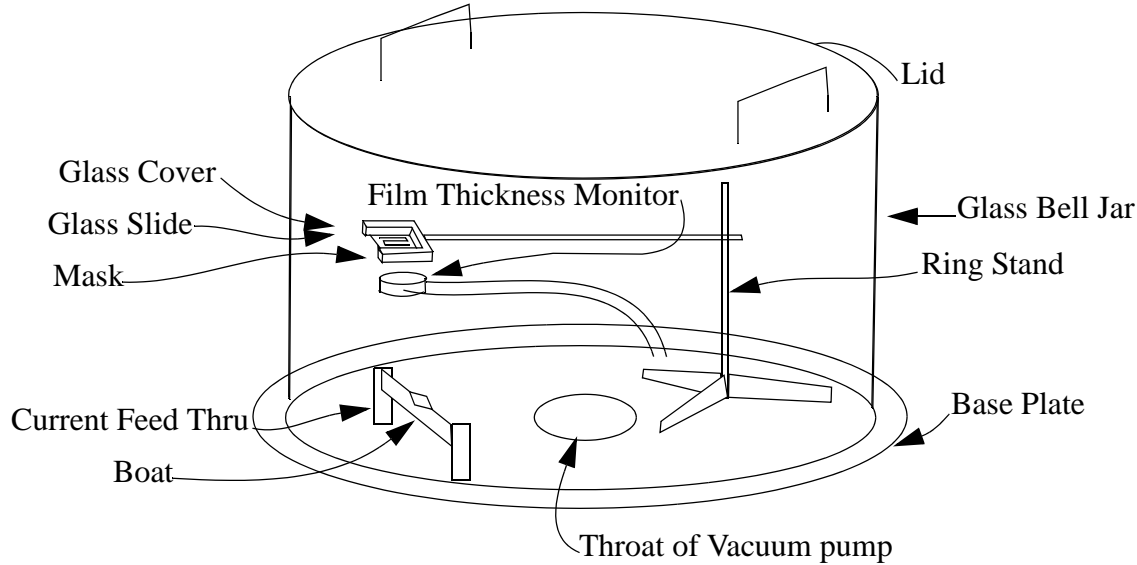
Base Plate

Throat of Vacuum pump

Figure 1. Schematic diagram of the Advanced Lab thin film evaporator. To set the scale, the overall diameter is about 20". Not shown is a glass chimney that straddles the boat and shields most of the interior of the bell jar from evaporating metal.

In order to make a thin metal film on a substrate, some of the metal is placed inside a refractory metal resistive heater, called a boat. The substrate is usually placed on a mask, a stencil made from a thin sheet of metal with holes cut in it to define the shape of the film. The substrate is placed on the stencil and the back covered up to prevent metal atoms which bounce around in the vacuum system from contaminating its back surface. The inside of the bell jar, in which the boat, mask and substrate are mounted, must be evacuated to prevent the metal from oxidizing and to allow the evaporated atoms to travel ballistically to the substrate. Once the pressure is low enough current is run through the boat until the metal in the pocket in the center of the boat melts and begins to evaporate. If the air is out of the way, evaporated atoms fly off in all directions in ballistic parabolas. The turning points of the atoms' trajectories are typically far outside the bell jar so that to a good approximation you can think of the paths of the atoms as straight lines from the boat to whichever solid surface they hit first. Because the metal goes everywhere, a glass chimney (a hollow glass cylinder with slots cut part way up opposite sides so it can straddle the boat) is used to keep metal from coating things that are hard to clean. Some of the evaporated metal atoms will fly towards the mask and some of those will go through the holes in the mask and hit the glass substrate. When they hit these cold (cold compared to molten metal anyway) surfaces the vast majority of them stick and a metal film builds up. The thickness of the metal and the rate at which the thickness grows are measured by the Film Thickness Monitor. The monitor should be placed as close to the glass slide as is practically possible. The straight lines between the pocket of the boat and the sample and the pocket and the monitor must be unobstructed. The current to the boat is turned off once the film reaches the desired thickness and the evaporation rapidly ceases as the boat cools. This process leaves a metal film on the glass substrate everywhere there is a hole in the substrate.

B. Setting up the bell jar.

1. It is impossible to say what the status of the bell jar will be when you get to it. It may already have been removed from rest of the evaporator and be sitting somewhere in the lab. If it is on top of the bell jar it may or may not have a partial vacuum in it. If it is on the evaporator, you will need to remove it and set it down out of the way. The bell jar is made out of glass. It hates everyone and is vigilantly and relentlessly looking for a chance to chip or break and ruin your experiment. Needless to say, you must not drop it. Less obviously, the edges are extremely easy to chip, even through the rubber gasket, and you must set the bell jar down very gently and place the lid on top of it very gently. It is heavy and awkward and this is hard to do, but it must be done anyway. Make sure there are no chunks of anything on the gaskets or on the surfaces you are setting it on before setting the bell jar or lid in place. Any chunks in the seals may cause a chip that will leak. There are numbered valves on the evaporator. Their function is described in detail below. To remove the bell jar, make sure Valves 4 and 6 are closed. Open Valve 11. If it hisses, wait until it stops. Then you can remove the bell jar. Often over zealous vacuum grease users put so much grease on the rubber seals that the bell jar is hard to lift. To get it off, place a hand at the lower left corner of the bell jar in Fig. 2 and push with your other hand or shoulder depending on your height on the upper right hand corner. Do not strike any sharp blows during this process (it's still made of glass after all) but smoothly ramp up the force you apply until the lower right corner lifts off. It'll be easy after that. Once it's off, you can set it on the little platform behind and to the right of the evaporator.
2. Use a molybdenum (moly) boat. Make sure it is firmly clamped at both ends. You want power to dissipate in the boat, not at the boat-clamp contact. Fill the pocket of the boat with Sn or In shot. Put them in one piece at a time until the boat is full. Don't drop any. Highly purified metals are expensive. If there is a used boat there and you know or can find out what metal is in it, you can reuse the boat. If it is less than half full, add more material. Put the chimney around the boat.
3. The glass substrate should be a ~1" square. You can make one from a microscope slide by cutting off a 1" length. Score across its width with a glass cutter and break it. Clean it with soap and water or Windex and then rinse it in water. Crud in the water winds up on the substrate if you allow water drops to evaporate. So blow the last of the rinse water off with the can of compressed gas duster in the lab. If you can't see any smudges or dust particles, the slide is clean enough.
4. Place the substrate over the mask and push it all the way against the "bottom" of the U-shaped bracket that supports the mask. Glass should cover all the openings in the mask. If it does not, your substrate is too short. Make another one. Place a second 1" piece of glass on top to keep second bounce Sn or In atoms from sticking to the back of your substrate. This is just an aesthetic point. Your sample will be ugly but usable if you don't cover it.
5. You may want to use the two nuts in the bell jar to tilt the mask holder slightly. Place them under the two feet of the ringstand closest to the boat. The evaporator shakes when the mechanical pump runs. The substrate may tend to walk off the mask if you don't tilt things this way.
6. Gently bend the Film Thickness Monitor until it is next to the substrate. Make sure that neither the mask holder or the chimney eclipse the monitor as seen from the boat.
7. Place the bell jar over the whole works and place the lid on top. Remember all of the cautions from Step 1, above. Place the metal lath blast protector around the bell jar. It prevents big

pieces of glass from flying across the room in the wildly unlikely event that the bell jar implodes under vacuum. If the blast protector doesn't stop the flying glass, something or someone else will.

Using the evaporator

The vacuum system we use is an extremely common one in laboratories. There are at least two others just like it in the Advanced Lab and probably dozens of them in various labs in the School of Physical Sciences. It is a combination mechanical pump and oil diffusion pump. In Appendix A are material photocopied from John H. Moore, Christopher C. Davis and Michael A. Coplan, *Building Scientific Apparatus: A Practical Guide to Design and Construction* (Addison-Wesley Publishing Co. Inc., Reading MA, 1989) pp. 83-88 and104 -105. (This is a good book if you find yourself caring about experimental research. As of this writing, January 2001, it is still available for about $42 from Amazon.) This explains how the pumps work and how they are put together to make a vacuum system like the one on the evaporator.

Here are specific instructions for obtaining a vacuum in our lab. You must remember at all times that a diffusion pump cannot pump gas at pressures that are higher than about 1/2 Torr. Dense gas disrupts the vapor jets in the pump and that's the end of it. Exposure to air will also burn even the silicon based oil (DC 704) that we use in the pump. Burned silicon oil turns into a black tarry substance that you could use to patch holes in the parking lot. Once the pump contains this, it must all be cleaned out. You will get to do that if you screw it up and pump cleaning makes for a very dull lab report. So you must remember the golden rule of diffusion pumping at all times:

Never allow either the input or outlet of the diffusion pump to see a high pressure of any gas.

Figure 2 shows a schematic diagram of our evaporator:
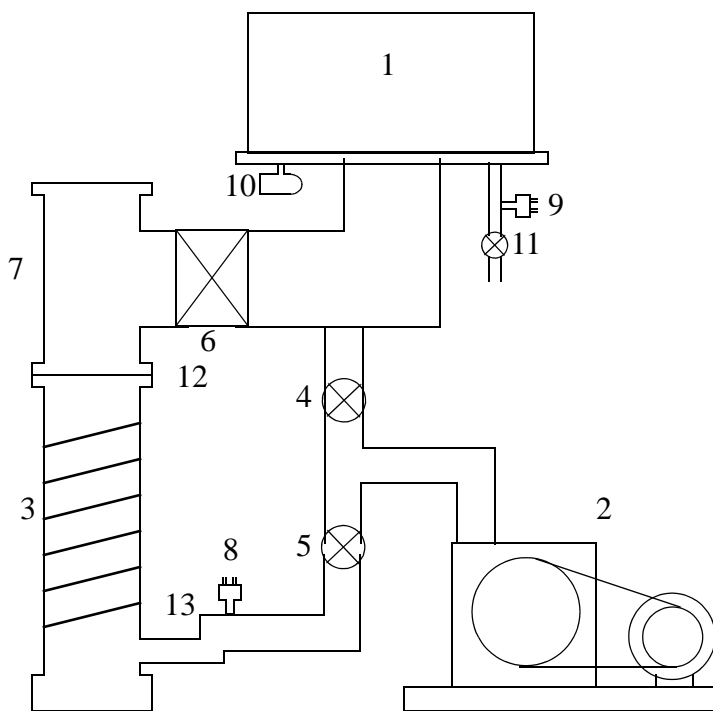

Fig. 2. The evaporator vacuum system.

1. Bell Jar. For details see Fig. 1.
2. Mechanical or fore or roughing pump.
3. Diffusion pump with chilled water lines.
4. Roughing valve.
5. Fore line valve.
6. High Vacuum valve.
7. Liquid nitrogen cold trap.
8. Fore line thermocouple pressure gauge head.
9. Bell jar thermocouple pressure gauge head.
10. Ionization pressure gauge tube.
11. Vent valve.
12. Diffusion pump inlet.
13. Diffusion pump outlet, or fore line

Notice that it is functionally identical to the system discussed in the Appendix.

C. How to pump out the bell jar.

1. When you first approach the evaporator, the mechanical pump and the diffusion pump will be off; all the valves should be closed and there will be no cooling water flowing through the coils around the diffusion pump. The bell jar may or may not be installed and there may or may not be a vacuum in it. If it is on the evaporator and you want to remove it open Valve 11 to vent the bell jar in case it is evacuated. When it stops hissing remove the bell jar and set it up according to the instructions in Part B above.
2. Make sure that valves 4, 5, 6 and 11 in Fig. 2 are closed. (Always check to see if valves are closed by trying to close them. A little thought will tell you why.) Find the right switch and turn the mechanical pump on. You'll hear it if you succeed. Open valve 5. We are about to heat up the oil in the diffusion pump. The golden rule above has a corollary: *when the diffusion pump is hot Valve 5 is **always** open except during time intervals just a few minutes long when having Valve 5 open would violate the golden rule.* After a minute or so thermocouple gauge 8, the bottom green LEDs on the gauge controller, should come on scale meaning they should light up from left to right. If this doesn't happen go for help from some cognizant authority. The units on this gauge are thousandths of millimeters of mercury, aka microns, aka millitorr or mtorr. Once thermocouple 8 is on scale, the pressure inside the diffusion pump, 3 above, is low enough so that is safe to turn on the heater. First find the valves that bring chilled water to and from the coils around the diffusion pump. Turn both of them on. The chilled

water must always be on when the pump is hot. Now turn on the switch that applies current to the diffusion pump heater. It is next to the mechanical pump switch. In 45 minutes or so the pump will be ready to go. Once the diffusion pump is hot, you are ready to evacuate the bell jar. The pumping system can be left in this state almost indefinitely. (This state is mechanical pump on, Valve 5 open, all other valves closed, water flowing in cooling coils and diffusion pump on.)

To pump out the bell jar starting at one atmosphere or any substantial fraction thereof.

3. Fill the cold trap with liquid nitrogen. Make sure the vacuum system is in the state described at the end of the last step. (See Step 2 just above.) Pour the nitrogen in through the plastic funnel. A lot of violent boiling will take place and the trap can seem full even if it isn't. So after it calms down, try to fill it some more. It is important that the trap be cold whenever the high vacuum valve (Valve 6) is open. Otherwise a film of oil will build up in the bell jar and on everything in it. As you can probably imagine this does not improve the sample quality. When pouring or handling liquid nitrogen, be careful not to let the liquid soak into your clothing. Nitrogen soaked clothing can lead to relatively nasty first or second degree frost bite. Keep adding liquid nitrogen until the trap is calm but overflows when you try to add more.
4. Rough out the bell jar. To do this, first close Valve 5, otherwise the next step will violate the golden rule and you'll be on your way to becoming a paving material supplier. Open Valve 4. A hissing noise at this point probably means Valve 11 is open. Shut it. If the hissing continues or if after 5 minutes with Valve 4 open the pressure as reported by thermocouple gauge 9 (the top row of LEDs on the pressure gauge controller) is not down to 200 mtorr, it is time to chicken out. Shut Valve 4, open Valve 5 and go for help.
5. After the bell jar thermocouple gauge, 9 in Fig. 2, reads ~ 200 mtorr, close Valve 4, open Valve 5 and open Valve 6, in that order. It requires approximately 75,000 full turns of its handle to open Valve 6, or at least it seems like this many. Be gentle and patient and just keeping turning until it is open. It will come to a hard stop. The valve is very subject to mechanical damage if net forces are applied to the valve stem. So take your time and apply torque not net force. If you don't know the difference, ask. (By the way, why do you think Valve 4 must be closed now?) Pumping much below 200 mtorr through Valve 4 with the roughing pump allows roughing pump oil vapor to diffuse into the bell jar and that's not good. A minute or so after Valve 6 is open you can turn on the ionization gauge. The filament should glow like a light bulb and remain on. If it does so, everything is ok. If it does not, wait a couple of minutes and try again. If there is still no luck, shut Valve 6 and go for help. The thermocouple pressure gauges and ionization gauges, why they work and what ranges of pressure they are good for, are described in Appendix B.
6. Now you have to decide how low a pressure to get to before making your sample. The average distance a metal atom will fly through a gas at a pressure P before it undergoes a collision is called the mean free path. The mean free path, λ, is given roughly by Equation (1). This

$$\lambda(\text{cm}) \cong \frac{5 \times 10^{-3}}{P(\text{Torr})} \qquad (1)$$

makes the mean free path at 5 x 10^{-5} Torr one meter. (What length does λ have to exceed for things to stand a chance?) A pressure of 2.5 x 10^{-5} Torr makes the mean free path 2 meters and

that is twice as good as 1 meter, but if it takes 10 hours to get there, is it worth it? Decide where diminishing returns set in and get to an adequate pressure without investing a ridiculous amount of time.

7. The pump down time is a good time to read about the film thickness monitor and set the measurement parameters it needs for the metal you are evaporating. Leave the tooling factor at 100%. Appendix C contains the things you need to know about the film thickness monitor.

It is now time to evaporate the sample. The process is described in the next section, but this is when you do it.

Assume now that the sample is made and you want to go in and get it.

D. How to get the bell jar back up to an atmosphere.

1. Turn off the ionization gauge.
2. Close Valve 6 and leave Valve 5 open.
3. Open Valve 11 for a split second and close it again. Watch the reading from thermocouple gauge 8. If it goes up scale, air is getting into the diffusion pump when it shouldn't. This may be because Valve 6 is not really shut. Shut it. Although this is highly unlikely because you probably couldn't get down to low pressures if this were the case, Valve 4 might not be completely closed so check this valve. If it was not really shut, shame on you because you did a very dumb thing in Step 5 of the previous section. Shut it. If neither of these steps succeeds in making the pressure on thermocouple 8 independent of what is going on in the bell jar, leave Valve 5 open (see the corollary above) and go for help. By far the most likely thing that will happen in this step is that thermocouple 8 will not care about the air you put into the bell jar through Valve 11. If this is the case, open Valve 11 (while taking an occasional glance at thermocouple 8's read-out just in case) and stand there until the hissing stops.
4. You can now remove the lid and/or the bell jar. The bell jar is just as easy to break or chip now as it was when you first started. So be careful with it. You can set the bell back on the little table behind the evaporator. Set it down on either of its flat ends. If you set down on its side it'll try to roll. If the bell jar is stuck to the base plate, see Step B. 1. on page 4.
5. You can now remove your sample.

Now you will need to work with your sample for a little while. Before you do you should put the evaporator into a safe state. So do the following 6 steps. These steps lead to a state that you can leave the bell jar in for quite a while. From this state it is easy to start making another sample or to shut the bell jar down entirely if your sample is ok or if you are going to leave the lab for the day (or longer).

1. Remove the chimney so someone can clean it.
2. Place the bell jar on the base plate and the lid on the bell jar. Carefully.
3. Close Valve 11.
4. Close Valve 5 and then open Valve 4.
5. Wait until the readout from thermocouple 9 (top LEDs) is on scale.
6. Close Valve 4 and Open Valve 5.
Roughing out the bell jar and then sealing it off will keep it clean between uses.

What you do next depends on your sample.

If your sample is no good for some reason, you need to make another one. No good means electrically open. It is possible that it was born that way because of some mistake you made during the evaporation or it may have gotten scratched while you were attaching it to the sample holder in a procedure coming up. Open Valve 11 to vent the bell jar. Set the bell jar aside, make sure everything is set up as in Figure 1 and put in a new substrate. Pump it down and try it all again.

If your sample is ok and you have successfully mounted it on he sample holder, or if you are leaving for the evening, you should turn the evaporator off. To do this, make sure you've done the previous 6 steps and then

7. Turn off the diffusion pump switch.
8. Wait at least 20 minutes for the pump to cool down to some reasonable temperature.
9. Close Valve 4.
10. Turn off the mechanical pump at the switch.
11. Shut off the cooling water supply and return valves.

E. Evaporating the metal film

This is what you do when the pressure is low enough. Its place in the time sequence is indicated in the previous section.

1. Make sure that the big knob on the variac (which is an inductive voltage divider for ac voltages) is turned against the stop, all the way counterclockwise. If there is a mechanical shutter between the boat and the substrate in the evaporator, turn it out of the way by rotating the knob under the base plate shown in Fig. 2.
2. Turn on the toggle switches on the variac and the watt meter.
3. Press the open shutter button on the film thickness monitor. This doesn't open a shutter the way we have things set up, but it zeroes the monitor read out and turns the monitor on.
4. Slowly turn the knob on the variac clockwise. The variac provides ac power to the primary of the big, red voltage step-down transformer on the base of the evaporator. The evaporation boat is connected across the secondary of this transformer. The resistance of the boat is just a few ohms so it takes a good deal of current but hardly any voltage to heat it. That's why a step-down transformer does the job. You have to go slowly while turning up the current because it takes the boat temperature a few seconds to get into steady state after the current is stepped up a little. If you turn the variac up too quickly the temperature of the boat will overshoot and things will screw up.
5. While turning up the current watch the boat and the monitor. As the current goes up the boat will begin to glow, the metal in the pocket will melt, its vapor pressure will go up and the monitor will start showing a finite rate and increasing thickness. Adjust the current with the variac until you are evaporating at a rate of 10 A/sec, give or take a factor of 2. Evaporate until the film is 1500 A thick. Thicker films have higher internal stresses and unnecessarily small resistances. Remember the point of all this is to make a metal sample with a relatively large resistance. Thinner films may be discontinuous due to a tendency on the part of these soft met-

als to form island structures rather than uniform films. This is why the top surface of your film may look "milky" but the film as seen through the glass substrate is shinny bright.

6. When the film thickness is 1500 A abruptly turn the variac full counter clockwise. The boat will cool rapidly and cut off the evaporation.
7. Wait a minute or two for the boat to reach room temperature.
8. Bring the bell jar back up to an atmosphere. (See page 8.)
9. ***Evaporated films of the soft metals we use are incredibly easy to scratch.*** Once scratched there is a good chance that the sample will be ruined and you'll have to make another one. So when taking the substrate off the mask, be sure not to drag the sample along the mask. Rather rather lift the front edge straight up while using the back edge of the mask holder as a pivot. Get a forceps tip under the glass, but not near the metal, then grab the substrate and lift it up and off the mask.

Explanatory Interlude

In order to understand what we do with the sample next, you need a more detailed description of how we are going to vary the sample's temperature. To reach the lowest temperature available in our lab, we will immerse the sample in a container full of liquid ^{4}He. Except for its isotopic cousin liquid ^{3}He, liquid ^{4}He has the smallest latent heat of vaporization of any substance. It is about 1/500 of the latent heat per molecule of water and about 1/70 that of liquid nitrogen (The numbers are from. Michael de Podesta, *Understanding the Properties of Matter* (Taylor and Francis Publishing Co. Inc., Washington DC, 1996) pp. 328-329.) That is what makes it useful as a low temperature refrigerant. But the price you pay is that you must thermally isolate the liquid helium from the hot 300 K room extremely well because any but the tiniest heat leaks will make all the helium evaporate. In order to increase the thermal resistance between the container of liquid helium and the room, you must support it on long supports made of small cross sectional area (this idea should sound familiar from another context) and those supports should be made out of relatively poor thermal conductors. Any wires that go from the room into the helium should have the same characteristics. In addition, an observer standing in the liquid helium container (just imagine this; don't try it) should not be able to see any room temperature surfaces. These goals are met by specially designed containers called dewars or cryostats. The cryostat in our lab is fairly typical and all others are variations on the same theme.

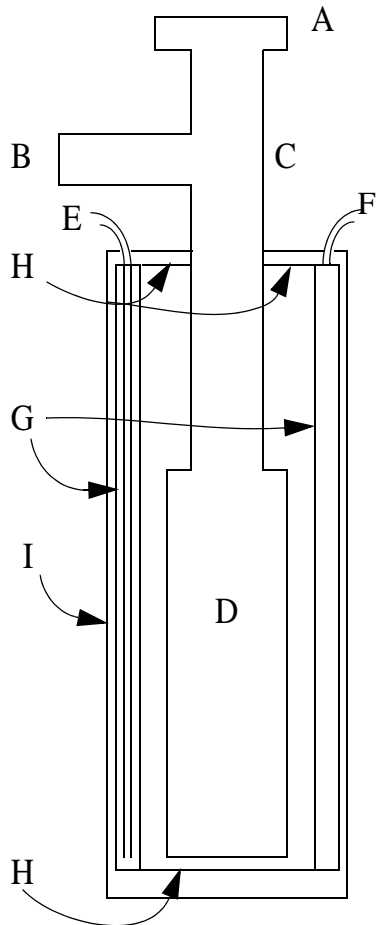A cross sectional diagram of Cryostat is shown in Fig. 3.

Figure 3. The Advanced Lab Cryofab CSM-25 Cryostat
A. Top flange
B. Pumping port
C. 2.5" diameter thin walled stainless steel neck The neck is 24" long.
D. Thin walled stainless helium well. The well is 3.5" in diameter and 18" long.
E. Liquid nitrogen fill line
F. Liquid nitrogen vent port. Only one of three is shown.
G. Annular liquid nitrogen reservoir
H. Nitrogen cooled thermal shields
I. Outer vacuum wall. Everything inside the helium well and nitrogen reservoir are thermally isolated from each other and the room by a high vacuum.

The heart of the cryostat is the liquid helium well, D. During the experiment, the superconductivity sample and the heater for the helium heat capacity measurement are located very near the bottom of the well. The well and the neck that supports it are both made of type 304 stainless steel. Stainless steel is very strong, can be easily welded and can be made into very thin-walled tubes. In addition it has a relatively poor thermal conductivity for a metal, at 1 K its thermal conductivity is about 1000 times smaller than that of copper and it is 50 times smaller at 300 K, for example. These features make it a very useful material in cryostat construction. The insert, which supports the sample in the helium well, bolts to flange A where it makes a vacuum tight seal. The helium well can be pumped on through B to lower the pressure and therefore the temperature of the liquid. (See below.) The helium well is surrounded by surfaces at liquid nitrogen temperature. The liquid nitrogen is held in an annular reservoir, G, also made from stainless steal. It is filled through a tube that goes to the bottom of the reservoir. Three vents allow the boil off nitrogen gas to escape. At the top and bottom of the nitrogen reservoir are relatively high thermal conductivity thermal shields. The shield at the top is connected to the neck of the helium well to short circuit heat conducted from the 300 K part of the neck into the nitrogen reservoir. The combined shields and reservoir mean that the helium well sees 77 K, the boiling temperature of liquid N_2, surfaces rather than 300 K surfaces. Radiation baffles in the neck region of the insert complete the shadowing of the helium in the well from the room temperature surfaces. This is important because sur-

faces radiate electromagnetic power at the rate of $\varepsilon\sigma T^4$ W/cm^2, where ε is called the emissivity and is usually near 1 and σ is the Stefan-Boltzmann constant which is numerically equal to 5.67 x 10^{-12} W/cm^2 K^4. The strong dependence on T means that the thermal radiation the helium well absorbs is reduced by a factor of 200 because of the 77 K walls. The heat load on the helium well from room temperature radiation in the absence of the shielding is about 50 W. Since a watt evaporates approximately 1 liter of liquid helium in an hour and the helium well holds about a liter, you can see that it is crucial to substantially reduce the radiative heat load by some means. Finally, the helium well and the nitrogen well are isolated from each other and from the room temperature outer surface of the dewar.

The sample itself is held in the helium well by the insert. The insert is shown schematically in Figure. 4. It has several jobs. It forms a vacuum seal to the helium well and completes the radiation
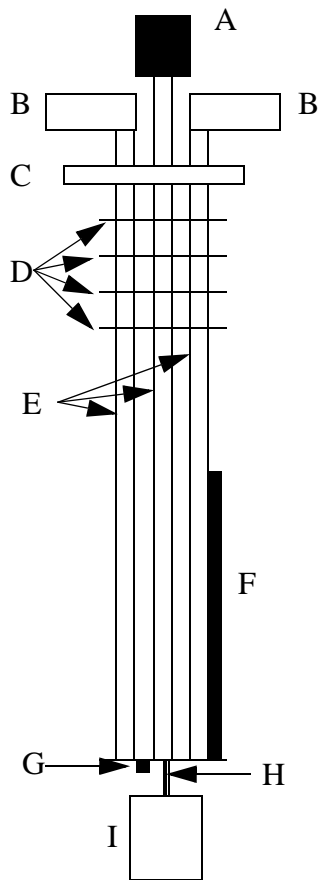

Figure 4. The cryostat insert.

A. Capacitance pressure gauge head. The gauge head is mounted through a vacuum seal. It must be removed to take the insert out of the cryostat and to fill it with liquid ^{4}He.
B. BNC junction boxes.
C. Top flange. This seals to the cryostat neck.
D. Radiation baffles
E. Support tubes. Two of these house wires (twisted pairs of Cu-Ni alloy, copper, and mini-coax) running between the BNC junction boxes and lower end of the insert.
F. Liquid He level detector
G. Diode thermometer
H. Amphenol connector
I. Sample holder

shielding. It supports the sample and provides electrical access to it and to the liquid helium level detector and the diode thermometer. It is a pretty standard example of laboratory cryogenic construction and what is where is described in the caption of Figure 4. How these things work and how to use them will be described later.

The point that you need right now to understand the ohmmeter is that because of the geometry of the cryostat and insert the sample must be attached to the electronics through several feet long wires that have non-negligible resistances. One of your goals is to detect the onset of superconductivity by measuring the electrical resistance of the sample. A fact that may have escaped your attention until now is that simple ohmmeters measure the resistance of the thing you think you are measuring plus the resistance of the wires connecting the meter to the thing. This is not a big deal if the resistance of the thing is big compared to the resistance of the wires. But you are not in this bag because you are using relatively high resistance wires and the thing you care about has a resistance that is alleged to go to 0! What must you do?

Any way you build a simple ohmmeter, usually called a two terminal ohmmeter, it consists of two devices in the same box. One way to do it is shown in Figure 5. This ohmmeter consists of a cur-
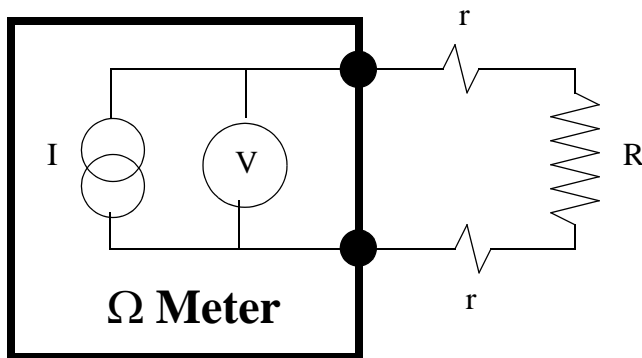

Figure 5. A two terminal ohmmeter. The ohmmeter consists of a constant current source, I, in parallel with a voltmeter. The resistance you want to measure is R and it is attached to the two terminals of the ohmmeter with wires of resistance r. The two terminals are the big black dots.

rent source, I, in parallel with a voltmeter, V. (The other way to do it is with an ammeter in series with a voltage source. The lead problems we are discussing don't go away in that case either.) A current source adjusts its output voltage so that a constant amount of current flows out of it regardless of what is connected across its terminals. Perfect current sources, like perfect anythings, don't exist, but you can get pretty close for lots of practical situations. When the ohmmeter is hooked to a resistor R through leads of resistance r, as in Fig. 5, none (well hardly any) of the current flows through the voltmeter, because voltmeters have huge input impedances. It all flows through the series combination of the leads and resistor R that you wish you were measuring. So the voltmeter measures the voltage V. Since it "knows" how much current it is supplying, it can tell you V/I. You, usually, interpret this number as R. But it is really R + 2r. It can't tell you just R because it never has any idea what the resistance of the wires you decide to use might be. Now if R >> 2r and/or you just need a rough number for R, you don't care. But if you are not in this limit or if you really want to make a precise measurement of R, the ohmmeter in Fig. 5 won't do.

The way to eliminate the effect of the wires is, ironically, to use more of them. The method is called a four terminal resistance measurement. To do it, you separate the current source and voltmeter as shown in Figure 6.
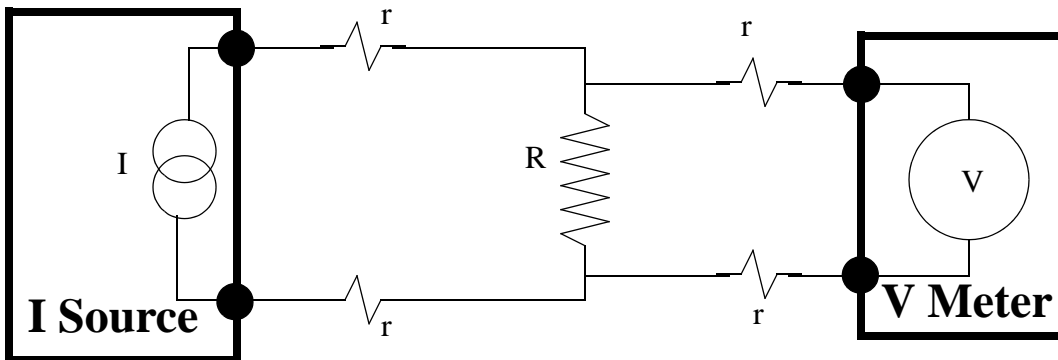

Figure 6. A four terminal resistance measurement of R. The current source and volt meter functions of the simple volt meter are split apart. The solid circles are the four terminals that give this circuit its name.

To understand how the four terminal technique suppresses the resistance of the leads consider an ideal current source and ideal voltmeter. An ideal voltmeter has an infinite input impedance so no current flows through the two lead resistances, r, in the voltmeter-R loop. Thus there is not voltage drop across them. The only two voltage drops in that loop are the drop IR across R, which really does carry the full current I, and the drop across the voltmeter. These must be equal in size. Hence the voltmeter tells you IR in this case. The current source, since it is an ideal current source, puts out I amps regardless of the size of the resistance across its output. There is a voltage drop of Ir across both lead resistances in the I source-R loop. But the volt meter does not see those voltages, so they do no harm. Thus in the ideal device limit, contamination of the R measurement by the leads is completely eliminated.

We do not have ideal I sources or V meters. If you choose the right ones they can come satisfyingly close. Not only that, you can calculate the effects of their non-ideality easily and correct the measurement for them. If you choose the equipment well, the corrections will be very small to begin with, so staggeringly high precision in the calculation of the corrections is not required.

A little further below we will describe how to actually make the actual physical realization of the ohmmeter shown schematically in Figure 6. The main point for now is to understand why you attach four wires to your sample, not just two.

F. Attaching sample to the insert and the leads to the sample.

At last you can understand why we attach wires to the sample the way we do. The sample looks like Figure 7.   The pairs of round pads in the upper left and right corners are used to attach the
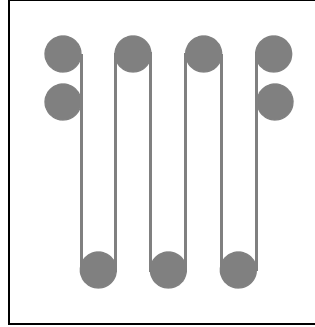

Figure 7. The superconductivity sample. The substrate is glass and the grey areas are evaporated metal. The paired round pads in the upper right and left are used to attach the four leads for the resistance measurement. The other round pads are there to make it easier for the machinist to make the metal mask you used.

wires. The current leads should be attached to the top ones and the voltage leads are attached to the bottom two. It is best if the wires that ultimately connect to the center pins of the BNCs at the top of the insert are attached to the same side of the sample. This eliminates possible grounding problems further down the line.

1. First remove the sample holder from the bottom of the insert. The sample holder is electrically connected to the bottom of the insert through an Amphenol 9 pin connector. It is secured by two 4-40 Allen head screws. (The 4 is a secret code that tells you the diameter of the screw and the 40 is the number of turns per inch. This is one of an elaborate set of standard screw sizes used in this country.) Remove the screws, put them someplace where you won't lose them and pull the sample holder out of the connector. Pull the connector straight out so you do not bend the pins in this process. They break off after just a few bending incidents.
2. Clamp the sample holder in a bench vice. Make the vice grab the sides of the green G-10 material. If you tighten the vice too much, the G-10 will buckle and then break. If you don't tighten it enough, the sample holder will slip in the vice when you push on it.
3. Somewhere on the table in the lab there should be a piece of very soft silver wire about 1/16" inch in diameter. With a clean razor blade, slice off a ~ 1/32" high disk of this metal. This metal is Indium. It is very soft and when mushed against glass or metal it sticks to it. Don't waste any In or lose the wire. Wire like this costs about $2.50 per inch (2001 dollars). So it's expensive enough that it should be treated with some care. That said, throw the first disk away and cut a second one. The end of the wire is oxidized so the first disk has only one clean indium surface. The second, and subsequent ones, will have two clean flat surfaces.
4. Pick up the second Indium disk with a tweezer and set it on the evaporated pad you want to attach a wire to. See Figure 8A. ***Remember, it is all too easy to scratch the evaporated metal right off the substrate.*** Don't touch it with anything except the In. It is ok if the In disk over-

laps the edge of the metal but keep it away from the skinny parts of the evaporated film. The In will stick to either the evaporated film or the glass.
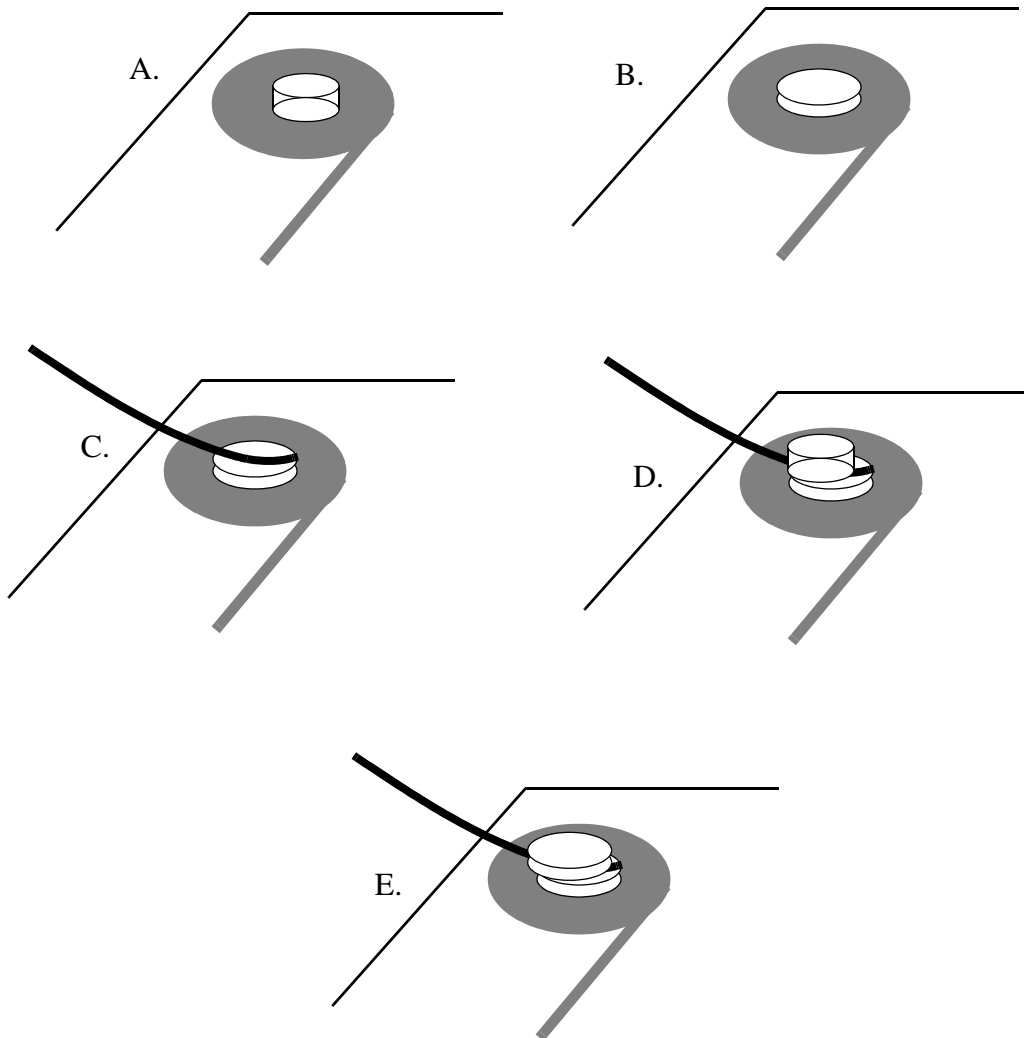

Figure 8. Making electrical contact to the superconductivity sample. See the text for details

5. Take the blunt, handle end of the tweezer and use it to mash the In disk onto the sample. This is what makes it stick and is the step at which the sample holder will slide in the vice if ratio of the mashing force to the clamping force is bigger than one. The mashed disk is shown in part B of Fig. 8.
6. Take the wire from the appropriate screw on the G-10 part of the sample holder and hold it on top of the mashed indium disk with a pair of tweezers. With the tip end of another tweezer, push the wire far enough into the mashed In so it stays there, as shown in Fig. 8C, when you let go of it.
7. Cut another In disk from the wire. And set it on top of the wire and mashed disk, Fig. 8D. Finally, mash this disk down. This completes the contact. Make three more just like it and you are there.

A good contact is so strong that it takes a pretty good tug to yank it apart. You may want to practice this a little by attaching wires to a piece of bare microscope slide before you attach the real wires to the actual sample. You should be able to lift the whole slide by the wire.

Occasionally in the course of these operations, the wires break off the little screws on the sample holder that they are soldered to. If this happens to you, solder a new wire in its place and keep going. If you do not know how to do this or if you can't find the things you need, more wire for example, go for help.

8. Now replug the sample holder 9 pin connector into the socket at the bottom the insert. You still have to be careful not to bend and or break any pins. The plug goes into the socket only one way. So look at it carefully to make sure you have it right. As is almost always the case muscle is not a good substitute for care in this operation. Replace the 4-40 Allen head screws that secure the sample holder to the insert and you are through.
9. Put the insert in the dewar. Be gentle with it and in particular don't bang the sample into the walls of the cryostat neck as you put it in. Once it is in, bolt the top flange together with four 1/4-20 Allen head bolts that are, hopefully, lying around someplace near the cryostat.

G. *Making the Four-Terminal Resistance Circuit*

In our experiment we use an ac current source and an ac volt meter in place of the dc devices shown in Fig. 6. There are two reasons for this choice. First, it is possible to make very narrow band ac voltage measurements with a device called a *lock-in amplifier*, which is the backbone of the four terminal circuit you will build. Narrow band measurements (meaning they only see signals in a small range of frequencies) suppress noise and make it possible to measure signals that are otherwise invisible. The ability to measure small signals allows you to measure your sample resistance with a very small current. Small currents are important for you because a) when your sample is in the normal, resistive state large currents cause large Joule heating which will mean your sample will be hotter than you think it is and you will mismeasure the transition temperature because you really measure the temperature of the liquid helium; and b) in the superconducting state, currents destroy superconductivity, which will tend to suppress the transition temperature. Secondly dc voltage differences can arise in wires with temperature gradients along them. You will have a 300 K temperature gradient. This effect adds little batteries whose sizes you do not know into the leads attached to you sample. This of course doesn't help things. But these thermo-electric voltages are dc, and an ac voltmeter will ignore them.

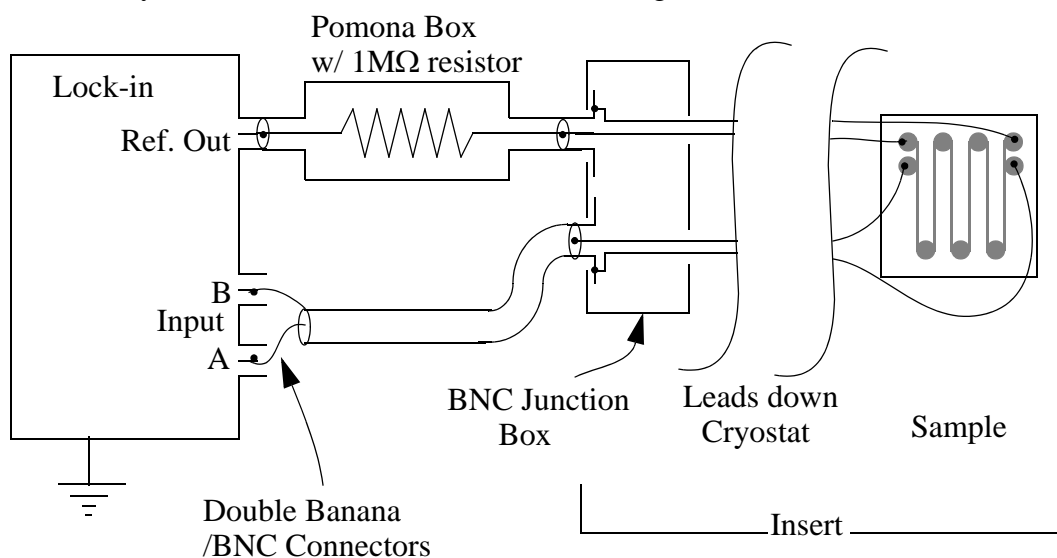You can make your ac resistance circuit as shown in Fig. 9.

Figure 9. Implementation of the ac four terminal resistance measurement. The combination of the reference oscillator in the lock-in and the 1 MW resistor at its output constitute a current source. We are using the differential inputs on the lock-in to avoid a ground loop. Notice that the BNCs on the lock-in are electrically connected and grounded. The BNCs on the insert Junction Box are electrically isolated.

Lock-in amplifiers are described in Appendix C. Briefly lock-ins are ac voltmeters that only look at signals in a very small frequency range. The center frequency of this range is called the reference frequency. Often, lock-ins contain an internal oscillator that establishes the reference frequency and that can be used to drive an experiment at that frequency. The circuit in Fig. 9 is an example. The output of the internal oscillator appears at a "Reference Out" connector on the lock-in. You can choose its amplitude and frequency to suit your aims. In our experiment we attach the sample to the reference output through a 1 MΩ resistor. The combination of a voltage source, the reference oscillator in our case, and a big resistor is, approximately, a current source. This current will oscillate at the frequency of the reference oscillator. As long as the sample has a resistance, the oscillating current will cause an oscillating voltage across the sample. The voltage will be at the same frequency as the reference oscillator because the current is. Since the lock-in measures the voltages at that frequency, you are in business.

The odd looking transition from a BNC cable to the BNC inputs of the lock-in can be constructed from BNC to double banana adaptors with the right genders in the appropriate places. Unless someone left it in place for you, putting it together is a little IQ test you'll eventually pass if you keep thinking.

You should build this circuit and try to measure the resistance of the sample with it at room temperature. You might want to practice with a variable resistance box in place of the sample first. You can check your sample resistance measurement by making a four terminal resistance measurement with one the higher quality DMM's in the lab. Good DMM's almost always have a four

terminal resistance measurement as an option. However they are dc measurements and the currents they use are in general too big for low temperature experiments.

H. The other elements on the insert.

Besides the sample and holder there are two other electrical elements on the cold end of the insert, a helium level detector and a silicon diode thermometer. In addition, there is a capacitance pressure gauge at the top of the insert.

The helium level detector. You need some means of telling when you are actually putting liquid helium in the dewar and checking how much helium is left while you are running the experiment. The liquid helium level detector does these jobs. It is the long black object on the insert labeled F in Fig. 4. It consists of a 12" long black fiber glass housing with a very thin superconducting wire running its length. Electrical leads from the level detector run up the insert to a BNC junction box. These BNC's are labeled "V+" and "I+". The are the two BNC's at the left end of the top row of BNC's on the left box as you face the cryostat. Two RG174/U coaxial cables (skinny ones) with the corresponding labels connect the level detector with its read-out box. The read-out unit drives a relatively large current through the superconducting wire. The superconducting transition temperature of the wire is somewhere above 4.2 K. When the wire is immersed in liquid, the liquid keeps it below its transition temperature and the wire's resistance is zero. But cold helium gas does not remove heat from the wire as effectively as does the liquid. So if the wire is suspended above the liquid, the current drives it normal and the resistance has its normal value. Here's the neat part. When the wire is part in and part out of the liquid, the part in the liquid superconducts but the part above the liquid is normal. So the resistance of the wire increases linearly from zero to its full normal state value as the surface of the liquid moves down its length. The read-out converts the resistance to the depth of the liquid for you and reports a number between 0 and 30 cm. 30 cm means that the entire level detector is immersed. One of the things you should learn in this experiment is that even in the normal state, the resistance of a metal is, in general, temperature dependent with the resistance increasing with increasing temperature. So when the level detector is well above 4.2 K its resistance is even bigger than when it is cold and normal. In this case, the level detector decides that the helium level is some negative number of centimeters up the detector because big resistances mean low levels as far as it's concerned.

The silicon diode thermometer. This connects to the BNC connectors labeled "I+" and "V+/I-" and are the right most BNC's in the first and second row of the same junction box that has the level detector connections.

A diode is a circuit element that to a good approximation only lets current flow through it in one direction. A common way to make one is to bond a piece of p doped semiconductor to a piece of n doped semiconductor. If you hook it up to a voltage source and measure the current that flows

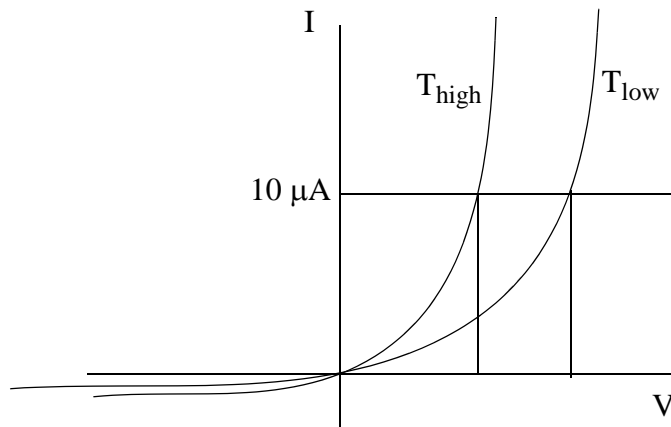through it as a function of the voltage across it, you'll get a result that looks like Figure 10. Here,

Figure 10. Current voltage characteristic of a silicon diode. For semiconducting p-n junction diodes, like our thermometer, positive voltage means that the p doped end is at a higher potential than the n doped end. The shows that the I-V characteristic is temperature dependent. The bias voltage, V, increases at fixed forward bias current, I, as the temperature decreases.

positive voltage means that the p doped end of the diode is it a higher electrostatic potential than the n doped end. When the voltage is positive, the diode is said to be "forward biased."

Diode thermometers take advantage of the fact that the curves in Fig. 10 is temperature sensitive. In fact, if you forward bias a diode with a constant current and change the temperature of the diode, you will discover that the voltage across the diode increases as the temperature decreases. Fig. 11 shows the forward bias voltage of a Si diode current biased at 10 μA as a function of the temperature. As you can see from the figure the forward bias voltage under these conditions is

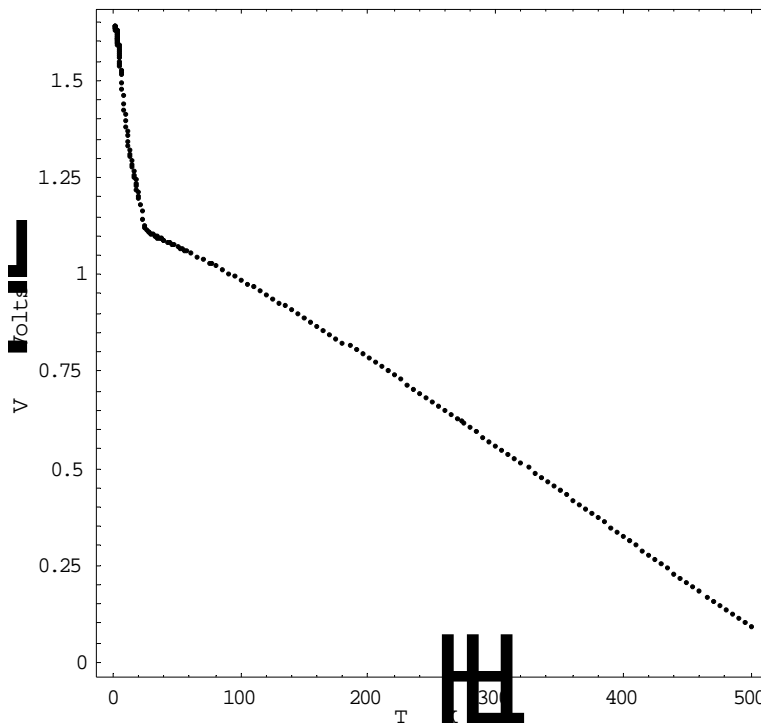
Figure 11. The forward bias voltage for a silicon diode carrying 10 μA as a function of temperature. The forward bias voltage is temperature dependent over a very wide range making these devices attractive thermometers over wide temperature ranges. Their sensitivity increases below ~ 30K.

temperature sensitive over a very wide temperature range. Thus diode thermometers are useful over the temperature range from well above room temperature to about 1.4 K. The sensitivity of

20

these devices increases dramatically below about 30 K and you need dissipate no more than ~20 μW so that self heating is not a big effect.

Our diode thermometer was calibrated by the manufacturer. The controller for it has the information in Figure 11 as measured for our very diode stored in a PROM so that its read-out is directly in temperature rather than bias voltage. The controller provides the fixed 10 μA bias current, measures the bias voltage and automatically converts it into temperature. Finally it outputs a dc voltage that is proportional to the temperature so that you can read the temperature with an analog to digital converter (ADC) and a computer. The proportionality constant depends on whether the "Expand Scale" button is pushed. Above 4.2 K the diode is the only thermometer we have. Below 4.2 K we have a second thermometer to compare the diode with and these thermometers (wouldn't you know it?) disagree.

The capacitance pressure gauge. At the top of the insert is a black cylindrical capacitance pressure gauge. It is mounted through a vacuum seal called a Wilson seal so that it measures the pressure inside the dewar. Schematically the gauge consists of two capacitor plates spaced apart by a spring. One capacitor plate is rigidly held in position and the other one can move against the spring as the pressure on the back side of the that plate changes. Increasing the pressure behind the moveable plate pushes it toward the fixed plate and decreases the capacitance accordingly. Thus the capacitance depends on the pressure and can therefore be used to measure it.

This pressure measurement is very useful because the primary, intentionally agreed upon temperature standard between 0.65 K and 5.0 K are the vapor pressures of the two stable helium isotopes. Since we will have a container full of liquid ^{4}He in thermal equilibrium with its vapor, we have the best thermometer possible built into our experiment at least at our lowest temperatures. The current world wide temperature scale is called "The International Temperate Scale of 1990" or ITS-90 for short. According to this standard the temperature scale between 1.25 K and 5.0 K is *defined* by the vapor pressure ^{4}He according to Equation 2,

$$T = A_0 + \sum_{i=1}^{9} A_i \left[\left(\frac{\ln((p) - B))}{C} \right) \right]^i, \tag{2}$$

where p is the vapor pressure of ^{4}He in Pascals (1 atm = 760 torr = 101325 Pa = 101325 N/m^2), and the A's, B and C are given in Table 1.

Table 1: Constants used in Eq. 2

	^{3}He 0.65 K - 3.2 K	^{4}He 1.25 K - 2.1768 K	^{4}He 2.1768 K - 5.0 K
A0	1.053447	1.392 408	3.146 631
A1	0.980106	0.5271 53	1.357 655
A2	0.676380	0.166 756	0.413 923
A3	0.372692	0.050 988	0.091 159
A4	0.151656	0.026 514	0.016 349
A5	-0.002263	0.001 975	0.001 826
A6	0.006596	-0.017 976	-0.00 4325
A7	0.088966	0.005 409	-0.00 4973
A8	-.004770	0.013 259	0
A9	-0.054943	0	0
B	7.3	5.6	10.3
C	4.3	2.9	1.9

(See H. Preston-Thomas, *The International Temperature Scale of 1990 (ITS-90)*, Metrolgia **27**, 3 (1990).) In case you're curious, the highest temperature ITS-90 defines is 1357.77 K, the melting temperature of pure copper at a pressure of 101325 Pa. Above this temperature and below 0.65 K you are on your own as far as ITS-90 is concerned. At any rate, the disagreement between the silicon diode and the vapor pressure temperatures in our lab means we got robbed when we paid for our diode calibration and PROM. Below 4.2 K you should use the capacitance pressure gauge and Eq. 2 to tell you the temperature.

I. The computer

If you think about it, you'll realize that the data you need for this experiment consists of measurements of the diode thermometer, the vapor pressure and the output voltage of the lock-in amplifier all measured simultaneously. This data is automatically recorded by a computer in the lab running a LabView data acquisition program called "LowTemp.vi". The analog voltage outputs of the lock-in amplifier, the silicon diode thermometer and the capacitance pressure gauge are read by a Keithley DMM. The DMM contains a switch that can connect any of 10 different inputs to its measuring circuitry. The switch is connected by a ribbon cable to a box containing 10 numbered,

floating BNC connectors. As rapidly as it can, the LabView program reads the time from the computer's internal clock, reads the voltage at BNC 1, then at BNC 2, then at BNC 3, then at BNC 3, then at BNC 2, then at BNC 1, and finally reads the time again. Then it averages the two times, the two BNC 1 readings, the two BNC 2 readings and the two BNC 3 readings. Then it writes these averages in a record of a data file in the order $\langle t \rangle$, $\langle V_1 \rangle$, $\langle V_2 \rangle$, $\langle V_3 \rangle$. It also plots the voltages on the computer screen in four graphs: each of the three voltages versus the reading number and V_1 as a function of V_2. Then the computer waits for as long as you tell it to before repeating the measurement cycle. The computer can only read one voltage at a time and you really want simultaneous readings of three voltages. The sequence of double readings just described does this at least approximately. It would be exact if the drifts during the measuring sequence were linear in time. Taylor's theorem, roughly speaking, tells you that the smaller the drifts are the closer they come to being well described as linear. Since the temperature changes that you deliberately make in the cryostat to do the experiment are responsible for the drift, change the temperature slowly when you have the choice and the averaging procedure will keep things ok. Sometimes the rate of temperature change is not up to you. In all these cases it changes slowly.

If you actually understand what you are trying to measure at each stage of the experiment, you will always know which instrument to connect to which BNC and when. The only thing this choice affects is what appears on the V_1 as a function of V_2 graph while the experiment is running. All the information you need is recorded to a data file regardless of which BNC's you hook things to. Generally you want the sample resistance on BNC 1 and whatever the most appropriate thermometer is for your temperature range on BNC 2.

To run the data collection program, double click its icon on the desktop unless it is already open. You will need to tell it the DMM's GPIB address which at the time of this writing is 16. If someone changes it, you can ask the DMM for its address by looking at its front panel and pushing buttons in some neuronally guided way. (GPIB stands for "General Purpose Interface Bus" which is the mechanism through which laboratory instruments talk to computers.) As always if you need help or just want to double check, ask. This address must be entered in a little window (called a "Control") on the grey panel that shows up when the program (called a "Virtual Instrument" or "vi" in LabViewese) is called up. To get your new address to register, mouse click the little check-mark "Enter" button in the upper left corner of the window. You will also need to tell it how many seconds to wait between measurement sequences. Then click the check-mark button again. Now the thing will run. Click the hollow white arrow button near the "Enter" button. The arrow will go from a block arrow to a cartoon character arrow with a bad case of the shakes to show you that the program is really running. A window will pop open asking you to name your data file. You do this in the usually Windows way. Click the ok box. If you picked the name of an existing file a window will open to ask if you want to over-write it. If the answer is yes, click so. If the answer is no, click "No" and then stop the program and start again. I've never figured out where the data goes in this case. Once the computer is recording data, you can pause it by clicking the pause button and start it again by reclicking it. To stop it, push the big, round stop button on the grey vi panel, not the stop sign button near the arrow and pause buttons. You will have to wait for the computer to take one or two more measurements after you press this button.

The experiment breaks up into different pieces determined by the temperature range and whether the experiment is warming or cooling. It may be a good idea to record the data from each temperature segment in separate files by stopping and restarting the program. It gets confusing, at least to me, if it is all in the same file.

J. Check things out.

At this point the following statements should be true.

1. The sample is rubber cemented to the sample holder and the sample holder is plugged into the connector on the insert. The two allen head screws that keep the sample holder from falling off are in place.
2. The insert is in the cryostat and the four bolts that hold the top flange together are in place
3. The capacitance pressure gauge is in place at the top of the insert.
4. The pressure gauge is reporting a reasonable pressure.
5. The diode cementer is connected properly to the BNC junction box on top of the insert.
6. The thermometer is reporting a reasonable temperature.
7. The lock-in amplifier ac ohmmeter is hooked up to the BNC junction boxes and reporting the actual resistance of the sample.
8. The 100 W resistor's state of health has been checked with a standard ohmmeter.
9. The helium level detector has been connected to the dewar. After several seconds it tells you the helium level is about -12 cm.
10. The level detector has been turned off again.
11. The analog voltage outputs have been connected to BNC inputs to the rear panel of the DMM.
12. You have pushed the button on the front of the DMM that tells it the signal is at the rear input.
13. When you start the computer program it starts measuring and plotting the voltages in 11.

If all 13 of these conditions are met, you can start cooling off the experiment. If any of them are not true, do whatever it takes to make them true.

K. Cooling to liquid N_2 temperature.

Since one of the goals is to measure the resistivity of a clean metal over as wide a temperature range as possible, you should measure the resistance of the superconductivity sample as the insert and sample cool from room temperature to nitrogen temperature. So you should start the program and put the data in a file you can find later. It will take about 8 hours for the temperature of the sample to near 77 K. So taking a data point every minute or two is frequently enough.

Once the computer is running you must move the liquid nitrogen storage dewar (a five foot tall, two feet in diameter stainless steel monstrosity that weighs a couple of hundred pounds when full) next to the cryostat. Take the rubber stoppers out of the nitrogen fill port and one of the nitrogen vent ports. The fill port and vent ports are clearly labeled so there should be no problem deciding which is which. Grab the big black rubber hose hanging from "Liquid" outlet on the storage dewar and slip it over the fill port tube. Open the valve on the storage dewar "Liquid" until nitro-

gen just starts to transfer over to the cryostat. If someone over tightened the valve, it may be quite hard to open. Hold onto the hose until it freezes solid and then let go. At first most of the nitrogen that comes out of the storage dewar will vaporize and come rushing out of the vent tube. After awhile, the hose and nitrogen reservoir will get cold enough so that the liquid will start to fill the reservoir. So be patient. Keep filling until liquid squirts out the vent. Then close the valve on the storage dewar. Don't over-tighten it or it will bind up as the valve returns to room temperature and be hard to open the next time. When the hose thaws, remove it from the fill line and place a rubber stopper back in the fill line. Then put a rubber stopper in the vent line. You do not want to leave the nitrogen tank open to the room, ever. Water will slowly accumulate in it and eventually destroy it if it is left open to the room.

The helium well and its contents will slowly cool due to thermal radiation. This is a great place to be at the end of the first lab session of the week. When you get back for the second session, things will be ready to cool further. If you must leave the experiment for more than a couple of days, refill the nitrogen every 48 hours or so.

ℓ. *Adding the liquid ^{4}He and cooling to 4.2 K.*

As mentioned in the discussion of the cryostat, liquid helium has a tiny latent heat of evaporation. If you try to just blow it in through a rubber hose, like you did the nitrogen, heat conducted through the hose will vaporize all the liquid before it got to the other end. The solution is to use a more elaborately insulated "hose" called a transfer tube. Figure 12 shows a schematic diagram of a transfer tube. The transfer tube is made of two concentric stainless steel tubes. The space
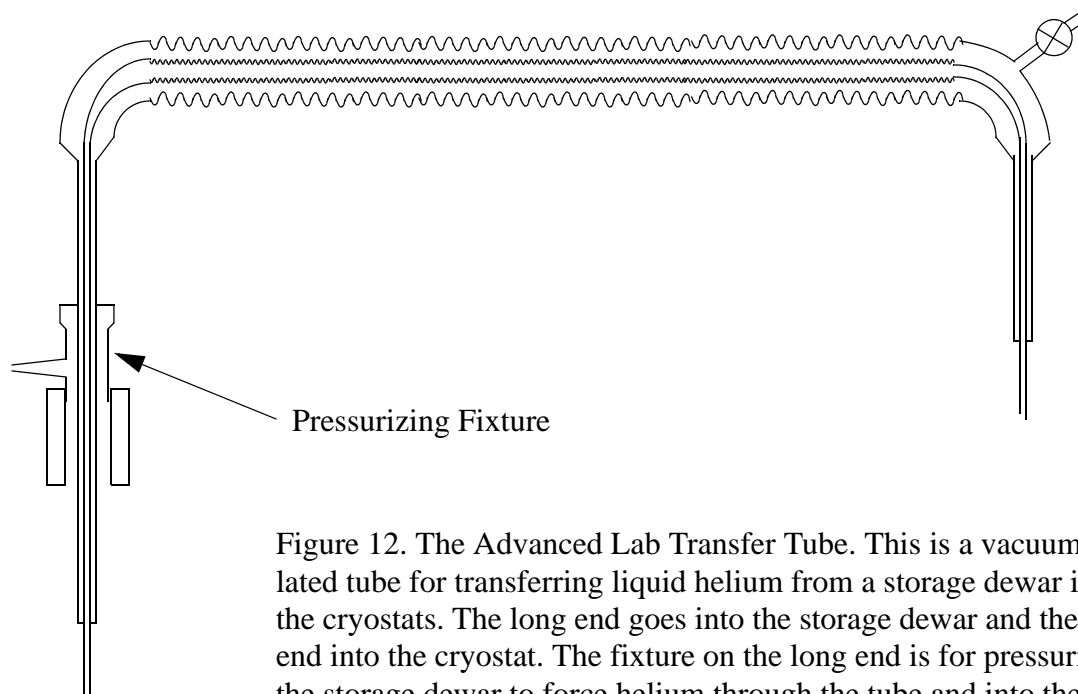

Pressurizing Fixture

Figure 12. The Advanced Lab Transfer Tube. This is a vacuum insulated tube for transferring liquid helium from a storage dewar into the cryostats. The long end goes into the storage dewar and the short end into the cryostat. The fixture on the long end is for pressurizing the storage dewar to force helium through the tube and into the cryostat.

between the tubes is evacuated to thermally insulate the inner tube from the outer tube. The tube should already have been pumped out for you so you need not pump on it yourself. Don't fiddle with the valve. The long leg of the tube goes into the storage dewar and the fixture on that end seals it to the top of the storage dewar. The short end goes into the cryostat through the Wilson seal that the capacitance pressure gauge normally occupies. A hose from a gaseous helium tank goes over the hose fitting on the fixture. On the bottom end of the fixture is a rubber hose that fits over the top 3/4" of the neck of the storage dewar. There is a hose clamp at the bottom of the rubber hose that you can tighten it if there is a leak at the neck of the storage dewar. You'll need a screw driver to do this so have one handy. The top of the tube is flexible to make it easier to use. Not shown in Fig. 12 are low thermal conductivity spacers, probably teflon, that keep the inner tube and outer tube from touching.

To Transfer Helium From the Storage Dewar to the Cryostat

1. There is a flexible stainless steel hose that attaches the neck of the cryostat to the vacuum system. Loosen the clamp that connects the flexible hose to the copper tubing of the system and offset the flexible hose. At first, when you begin to transfer liquid helium all of the liquid will boil and turn to vapor. You need to give that vapor someplace to go. Offsetting the hose from the pumping line does that.
2. Remove the capacitance manometer head from the Wilson seal on top of the insert and set it on the cryostat support. There is a cable running from the head to the read out. Don't jerk the cable out by the roots.
3. Take the tygon tube from the gaseous helium tank, located behind and to the right of the cryostat, and push it over the hose nipple on the fitting on the long end of the transfer tube.
4. You grab the long leg of the transfer tube and have your partner grab the short leg. Hold the thing so it lies in a vertical plane with the flexible part on top.
5. Loosen the Wilson seal at the top of the fixture on the long leg and slide the fixture down to within a foot or so of the bottom of the long leg. Re-tighten the Wilson seal.
6. There are three 1/4 turn ball valves on the top of the storage dewar. A ball valve is closed when its handle is perpendicular to the pipe the valve is in. It is open when the handle is parallel to the pipe. One of the ball valves is on a pipe that sticks straight up, two are on horizontal pipes set at 90 degrees to each other. One of the horizontal pipes has a one way check valve on the end. This ball valve should be open. The other two should be shut. If this is not the case, go get help.
7. Assuming the valves on the storage dewar are ok, shut the open ball valve.
8. Immediately open the ball valve on the other horizontal pipe about 1/4 of a turn. Helium gas will hiss out through the open tube. Storage dewars run a few psi over atmospheric pressure to prevent them condensing the air in the room through small leaks.
9. When the hissing stops, open the ball valve on the vertical pipe.
10. Close the ball valve on the horizontal pipe with the open tube. Now both horizontal ball valves should be closed.
11. Insert the long leg of the transfer tube into the storage dewar. Push the bottom of the rubber hose on the fixture over the neck of the storage dewar.
12. Now push the transfer tube through the Wilson seal at the top of the fitting. The short leg of the transfer tube should still be out in the room not in the cryostat. Loosen the Wilson seal a little if you need to. The hot (i.e. room temperature) tube will hit the liquid and some liquid

will evaporate. The pressure will go up in the storage dewar and gas will start hissing out of the short leg of the tube. The point of this maneuver is to cool the inside tube of the transfer tube so that you do not blow a lot of room temperature gas into the cryostat and warm it up unnecessarily.

13. If gas is escaping from the rubber hose, tighten the hose clamp just until it stops. Hopefully, you didn't forget your screw driver.

14. As the inner tube of the transfer tube cools, a white cloud will blow a foot or two out of the short end of the transfer tube. Don't do anything yet.

15. A little while later an intensely white vapor cone the diameter of the inner tube and 4 or 5 inches long will appear at the end of the short leg of the transfer tube. Now the tube is cold enough to insert into the dewar.

16. While holding the fixture on the long leg of the transfer tube in place, loosen the Wilson seal a little and slide the long leg of the transfer tube upwards until the short leg clears the top of the insert. Your partner should follow you up with the short end of the tube while you're doing this to keep the flexible part of the tube approximately straight. *Do not let the part of the transfer tube that was in the storage dewar touch your skin. It is very cold and will raise blisters.*

17. Once the tube is high enough, you should move the storage dewar around as needed so that your partner can shove the short end of the transfer tube into the Wilson seal on top of the insert. Then both of you should shove your ends of the tube through your Wilson seals. All but about 4 inches of the short leg should go into the cryostat. You should let the long leg hit the bottom of the storage dewar and then pull it up about an inch. As you push the legs of the transfer tube into the Wilson seals, be sure to push straight down and do not apply any horizontal force tending to bend them. A bent leg will probably ruin the transfer tube. The transfer tube is a $1000 (2001 dollars) hose and we don't want to buy more of them than we have to.

18. Open the gaseous helium tank so that it provides 3 or 4 psi of pressure. (If you don't know how gas cylinder valves work, ask!) This will force liquid through the transfer tube and into the cryostat. Watch the digital thermometer. It should fall rapidly to 4 K and change. Also watch the level sensor. It should become less negative, go through zero and then up to 27 or 28 cm and stop. All of this should take about 5 minutes or so.

19. A minute or two after the level monitor tops out, the sound of the gas leaving the open end of the flexible tube will change and the white vapor cloud will become denser. This means the dewar is full and it is time to quit.

20. Close the valves on the helium tank.

21. Open the ball valve on the horizontal open pipe on the storage dewar.

22. After the pressure in the dewar falls to atmospheric, remove the tygon helium hose from the hose nipple.

23. Your partner should completely unscrew the nut on his Wilson seal and you should loosen the hose clamp on the rubber hose on the fixture if you tightened it earlier.

24. Now slide the transfer tube out of the storage dewar and the cryostat. Pull the fixture off the the neck of storage dewar. The fixture and the Wilson seal nut from the insert should lift off with the transfer tube. Don't touch the part of either leg that's been in the dewar. *These are very cold and they will cause frostbite if you handle them. Wear gloves.* (Note: We're not talking gangrene and amputation style frostbite, just blisters and/or a tingly discomfort like a burn from grabbing something hot on a stove. But be careful anyway.)

25. Now place the transfer tube back where you found it.

26. Close the ball valve on the vertical pipe.
27. Open the ball valve on the horizontal pipe that ends in the check valve. (This give helium that evaporates from the storage dewar someplace to go without letting any air freeze in the dewar.)
28. Close the ball valve on the horizontal pipe that ends in an open 3/4" pipe.
29. Once the transfer tube warms up a little, remove the Wilson nut and O-ring from the short leg.
30. Replace these on the insert and remount the capacitance pressure gauge.
31. Reattach the flexible hose to the copper vacuum system. Don't forget the O-ring. Do not omit step 30 before doing this. If you do you will be trying to transfer liquid helium up the center tube of the insert

There. That wasn't too involved, was it?

m. Cooling below 4.2 K

Once there is helium in the dewar, you will want to use the pressure measurement as your primary thermometer. Be warned that there is probably a small dc voltage offset in the pressure output. You will have to deal with this in some appropriate way. There are several things you can do. Think of one or more of them and do one, or else get help.

During the initial cool down you can look for the transition temperature of the superconductor.

After you reinstall the pressure gauge head and reconnect the flexible hose, the pressure in the dewar will rise to above 800 torr. Heat leaks into the dewar at some rate and evaporates some liquid. This increases the pressure until a pressure relief valve just like the one on the storage dewar opens somewhere above 800 torr. To cool the experiment you must reduce the pressure. The reason is the same reason that you can use the vapor pressure as a thermometer. Namely, whenever two phases of a single component coexist, there is only one independent thermodynamic variable. Thus changing the pressure has to change the temperature, too. To change the pressure do the following.

1. Make sure the blue handled valve on the copper pumping system just above the flexible hose is closed.
2. Make sure that the black manostat mounted on a by-pass around the blue handled valve is also closed. Do this by turning it clockwise. A manostat is a pressure regulator. Schematically it

looks like Figure 13. As you can see from the diagram, the pressure in the cryostat can force
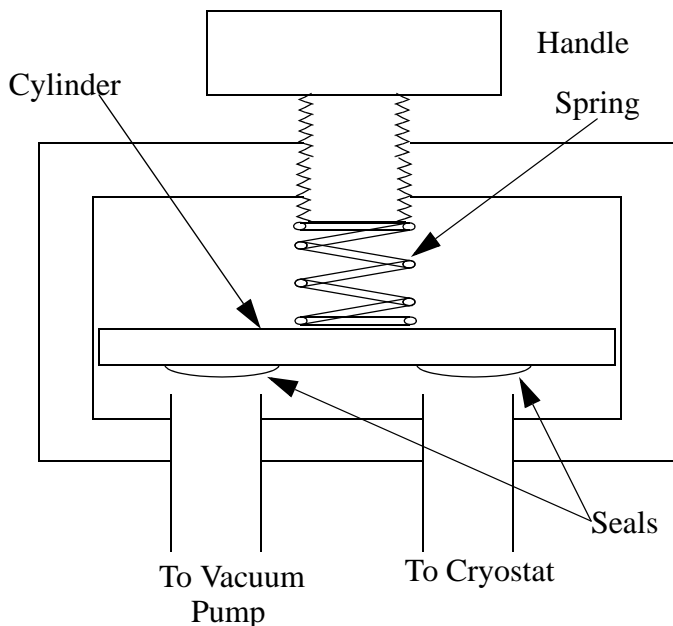

Figure 13. The manostat. A pump and the cryostat are attached to the manostat. When the pressure in the cryostat is high the spring loaded cylinder with the two seals on it is forced upward and the manostat connects the pump to the cryostat. This is the situation pictured. When the pressure in cryostat falls, the spring forces the piston down and the pump is disconnected. As the handle is screwed out, less pressure is required to connect pump and cryostat.

the cylinder with its seals upward. When this happens the pump gets connected to the cryostat and the pressure in the cryostat decreases. When it is low enough, the spring forces the cylinder downward and disconnects the pump from the cryostat. As helium evaporates the pressure goes up again and the pump gets connected until the pressure reaches the set point again. The further out the handle is turned, the lower is the set point pressure. As always, do not over torque the manostat. When it comes to a stop, don't twist anymore.

3. Turn on the big mechanical pump and Roots blower. There is a description of this type of pump in Appendix D. There is a small black switch on the control box. Turn it momentarily to "Start" and then let go. The pump will do the rest. There is a gate valve above the roots blower. Open it. It has a big black handle that actuates an elbow like mechanism. Pull with a twisting motion on the handle until the elbow locks in position with the "arm" straight.

4. When you open the valve the pressure in the cryostat will drop to about 710 torr, which is the highest pressure our manostat regulates at. This represents such a small temperature change from the temperature at atmospheric pressure that it does no harm.

5. To gradually lower the pressure, which is probably a good idea when you're looking for the superfluid onset temperature, turn the manostat handle counter-clockwise (as seen from above it) a little at a time. When you first start to turn the manostat, nothing will happen for a while. Just be patient until things start to work. Take your time. The slower you go the better will be your determination of the transition temperature. If the manostat starts to get cold you are going way to fast and are likely to destroy the manostat.

6. After the manostat is open as far as it will go, you can cool the dewar a little further by opening the blue handled valve. This will drop the temperature to the limit for our cryostat and pumping system.

n. The heat capacity experiment

Once the cryostat is as cold as it gets you can do the heat capacity measurement. To do this you want to close the manostat and blue handled valve and use the 100 Ω heater to dump a known amount of power into the helium. Three or four watts works ok. Think about what else you need to measure to turn the heat capacity into a specific heat (What's the difference?) and measure them. Close the manostat Using the small grey Hewlett-Packard power supply, apply the power, and immediately close the blue handled valve. (Why do you have to do these things and why is this the best order?) Record the vapor pressure as a function of time. This experiment will also take you through the superconducting transition again and give you another look at it.

Once the experiment gets near 4 K gas will start escaping from the cryostat through the manostat and/or the pressure relief valve. Turn off the power supply at that point and it's over.

o. Warm the cryostat up from 4 K to room temperature.

This will allow you to measure R (T) for your superconducting sample over this entire temperature range.

1. Turn the big pump and roots blower off by moving the switch to the stop or off position and letting go.
2. Take the rubber stoppers out of the nitrogen reservoir fill line and out of a vent line. Using the gaseous helium and tygon tube you used to transfer liquid helium, pressurize the vent with 4 - 5 psi of helium. Just hold it against the end of the vent tube for the few minutes emptying the nitrogen will take. When the vent is pressurized the nitrogen will be forced out the "Fill" tube. Don't stand in front of the vent tube or you'll get sprayed. When liquid stops coming out, turn off the helium.
3. Place rubber stoppers in the fill and vent tubes. This will prevent water out of the air in the room from condensing in the nitrogen reservoir and ultimately splitting it open when it contracts on freezing. *So it is important to make sure there is a rubber stopper in the fill line and both vents that do not have a relief valve on them.*
4. Two days later, the cryostat will be at 295 K. Record data appropriately. It's best not to have the "Expand" button depressed on the diode thermometer for this step, but it's not crucial.

Analyzing the Heat Capacity Data

What you'd be tempted to say at this point, is that since

$$C = \frac{dQ}{dT}, \tag{3}$$

where C is the heat capacity, dQ is a tiny increment of heat and dT is the resulting temperature rise, that you can get C from the P versus t data in part n. All you have to do is to calculate T from P and relate Q to the rate at which you dumped power into the resistor, $\dot{Q}$, as follows

$$C = \frac{dQ}{dT} = \dot{Q}\frac{dt}{dT}. \qquad (4)$$

This requires differentiating your t as a function of T data, but that can be done. (In fact, there is a routine to do it in the "MathematicaToolBox.nb" Mathematica notebook on the computer in the lab. This note book also contains all the thermodynamic functions you'll need below.) But if that's your temptation to some degree of approximation, you're right. But there are some corrections you should make.

Strictly speaking, a heat capacity is a property of a single phase. You would like to have measured how much a fixed amount of liquid helium warms up when you put in a small amount of energy. But that's not really what you did. When you did your experiment, the heat did a lot more than warm up a fixed amount of liquid helium. It also

a) warmed up the stainless steal walls of the helium well and all the materials on the insert.
b) warmed up the gas in the cryostat.
c) turned some of the liquid into gas.

How does one account for these things?

a) The helium well walls and insert.

At low temperatures, metals have higher specific heats than insulating solids. That is because metals have a specific heat contributions from the free electrons that insulating solids do not have. As a result, the stainless steel will dominate the heat capacity of the cryostat and insert at low temperatures. At low temperatures the heat capacity of a metal goes like

$$C(T) = \gamma T + \beta T^3, \qquad (5)$$

where the first term is from the electrons and the second from the lattice. Values of γ and β are hard to find for stainless steel and besides, since we didn't make the dewar we don't know exactly how much is down there. So you can use the numbers for copper to make a rough estimate of how

much the cryostat matters. This probably over-estimates the specific heat of stainless steel. For copper

$$\gamma = 0.695 \times 10^{-3} \text{ J/mole K}^2$$

$$\beta = \frac{1942}{343^3} = 4.82 \times 10^{-5} \text{ J/mole K}^4. \tag{6}$$

So estimate the mass for the insert and helium well, pretend its's all copper and decide how serious a correction to the data the cryostat and insert materials are. See the dimensions of the helium well in Fig. 3 and assume that the wall thickness of the helium well is 0.020". You've seen the insert and should be able to estimate how much "copper" it's made from. This correction is strictly additive and you can just subtract it from your data. How big a deal is it?

b) and c) Two-phase effects.

The effects of the gas and the liquid turning into gas can all be corrected for in one swell foop by applying some thermodynamics. Assuming you had the valve and manostat shut when you did the experiment to a good approximation you had a fixed number of helium atoms in the system. (The number in the room temperature plumbing is what is responsible for the fact this is an approximation, but that number is tiny compared to the number in the dewar. If the helium in the cold part of the cryostat was all gas it would be 295/T times denser than gas in the room temperature part of the plumbing. The presence of the liquid makes the relative number warm even smaller.) In that case, let V be the volume of the helium well and N be the total number of helium atoms. Let s_L, s_V, v_L, and v_V be the entropy per atom and the volume per atom in the liquid and vapor phases. Then the total volume, V, the total entropy, S , and N are given by

$$\begin{aligned} S &= n_L s_L + n_V s_V \\ N &= n_L + n_V \\ V &= n_L v_L + n_V v_V \end{aligned} \tag{7}$$

Along the liquid vapor coexistence curve, the Gibbs free energies of the liquid and the vapor must be equal. This leads to Clapeyron's equation,

$$\left.\frac{dP}{dT}\right|_{svp} = \frac{s_V - s_L}{v_V - v_L} \tag{8}$$

Combining these allows you to eliminate everything with the subscript V and write

$$S = N s_L + \left((V - N v_L) \left.\frac{dP}{dT}\right|_{svp} \right) \tag{9}$$

Now you know what the total entropy is of the helium in terms of the total amount of helium in the helium well, the specific entropy of the liquid, the volume of the well, the volume per atom of the liquid and the slope of the liquid vapor coexistence curve. Since the heat capacity you got

from Equation 4 is $C = T\dfrac{dS}{dT}\bigg)_{svp}$ differentiating (9) and multiplying by T gives,

$$ C = Nc_{svp}^{liquid} + T\frac{\partial}{\partial T}\left((V - Nv_L)\frac{dP}{dT}\bigg|_{svp}\right)\bigg|_{svp} . \qquad (10) $$

This, finally gets you the heat capacity of the liquid along the saturated vapor pressure curve. Calculating the corrections is an entertaining exercise. The stuff in the MatematicaToolBox saves you a lot of the work.

It is possible to continue on like this and calculate the principal heat capacity C_P from the result of Eq. (10). This calculation is very similar to the one you've done in your thermal physics courses that relates the heat capacities at constant volume and constant pressure. It goes like this

$$ dS = \frac{\partial S}{\partial T}\bigg)_P dT + \frac{\partial S}{\partial P}\bigg)_T dP $$

$$ T\frac{dS}{dT}\bigg)_{svp} = T\frac{\partial S}{\partial T}\bigg)_P + T\frac{\partial S}{\partial P}\bigg)_T\frac{dP}{dT}\bigg)_{svp} \qquad (11) $$

$$ C_{svp}^{liquid} = C_P^{liquid} - T\frac{\partial V}{\partial T}\bigg)_P\frac{dP}{dT}\bigg)_{svp} $$

$$ C_{svp}^{liquid} = C_P^{liquid} - TV\alpha_P\frac{dP}{dT}\bigg)_{svp} $$

In Equation 11 α_P is the thermal expansion coefficient at constant pressure. To a good approximation this is the same as the thermal expansion of the liquid along the saturated vapor pressure curve given in the MathematicaToolBox.