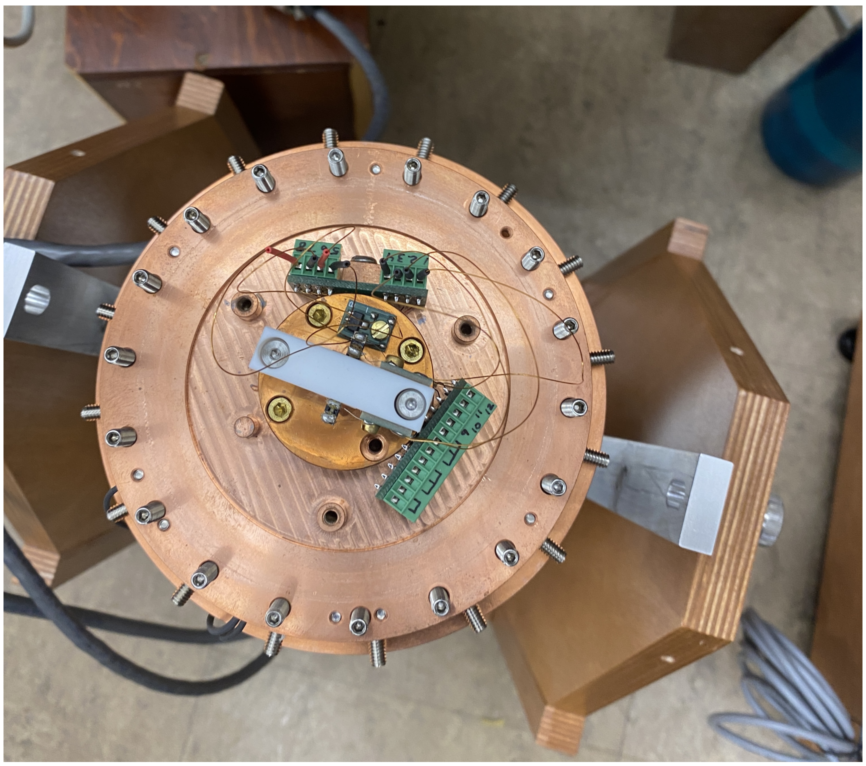
Physics
UNIVERSITY OF TORONTO

CMP
Advanced Undergraduate Laboratory

Condensed Matter Physics



Revisions

2022 Student Rhamel Roomes-Delpeache,
with revisions by David Bailey

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1 Introduction

This experiment is an introduction to cryogenic condensed matter physics in the temperature range from 77-300 K. Initially you can make measurements on high temperature superconductors, but a variety of other measurements are possible.

Initially you will study the superconducting properties of bismuth-strontium-calcium copper-oxide ($Bi_2Sr_2Ca_2Cu_3O_{10+x}$ where $0 < x < 1$), commonly known as BSCCO (pronounced "bis"-"ko"). Superconductivity is the phenomenon where some materials at low temperatures have zero electrical resistance and exclude magnetic fields [1, 2]. During this experiment you will cover many topics related to experimental condensed matter physics: cryogenics, vacuum pumps, electronics, circuit measurements, sensor calibration, ...

2 Background

Superconductivity was first discovered in 1911 by Heike Kamerlingh Onnes after his lab was the first to liquify helium (4.2 K), for which he was awarded the 1913 Nobel Prize in Physics. The field was revolutionised in 1986 when *high temperature superconductors* with transition temperatures $T_c \gg 4.2\,\text{K}$ were discovered.

Here you will be using the 4-wire method to measure the electrical resistivity in the BSCCO sample. First we will see why this method is necessary, then how it works, and finally how to implement it.

Question: Suppose we had a sample of pure copper, with a rectangular cross section 2 mm x 3 mm and length 20 mm. What is the room temperature resistance of copper, measured from end to end?

Question: Suppose the electrical connections to this sample-in-dewar were established using two phosphor bronze wires of 10 mm diameter, that were each half a meter long. What is the resistance of the wires?

As you can see the resistance is dominated by the wires when the resistivity of the sample is small (as in the case of this experiment). One might try to measure the resistance of the connecting wires and simply subtract it away from the measured resistance but this is not as simple as one would expect because the resistance of the wires are temperature dependent.

The *4-wire resistivivty method* solves this problem by connecting two separate pairs of wires to the sample. One pair injects and collects current into the sample; the other pair is used to measure the potential difference across a portion of the sample (see Figure 1).

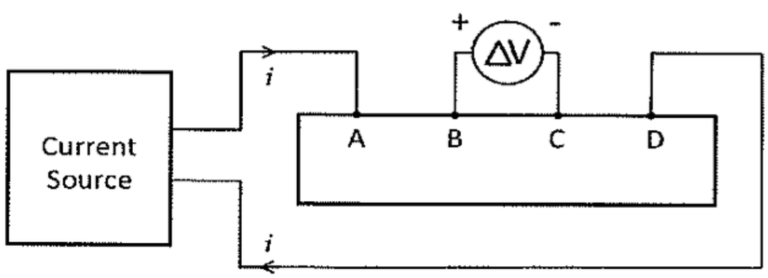

Figure 1: Schematic of a 4 wire measurement

In this experiment we will be measuring very small (microvolt) voltages which are susceptible to a lot of noise. A lock-in amplifier can reduced the affect of noise by lock onto a signal of a specific frequency and phase. (More information on lock in amplifiers is available on the APL YouYube page [3].)

To measure the resistance we take advantage of *AC ohms law*:

$$V_{rms} = ZI_{rms} \tag{1}$$

where the subscript *rms* refers to root-mean-square and Z is the impedance. Z is a complex quantity that comprising of the resistance, R, and reactance, X, of a circuit element.

$$Z = R + jX \tag{2}$$

Question: How will you measure only resistance using the lock in amplifier?

Safety Reminder

- Eye protection, gloves, and proper footwear, e.g. no sandals, must be worn when working with cryogenic liquids such as liquid nitrogen.
- Before using the cryostat, you must read and understand *Section 2: Cryogens and Safety* of the cryostat manual [4].
- When fill the cryostat with liquid nitrogen, one of the two fill tubes must be unobstructed to allow gases to escape. If this is not the case, it will be hard to fill the dewar and liquid nitrogen may dangerously bubble/splash back as the vapour tries to escape. (It would be especially dangerous for any student without eye protection, gloves, or proper foodwear.)
- Before pouring or using liquid nitrogen, always assess what might happen to you or other students if there was a spill. For example, you do not want to be in a situation where knocking over a cryostat or dewar on the table would pour liquid nitrogen onto a student's lap.
- Liquid nitrogen inside a sealed container can explode if it warms up and the gases cannot escape. Be sure never to block, impede, or otherwise tamper with the cryostat over-pressure safety valve.
- Never fill the dewar with liquid nitrogen if there is air between the inner and outer cans. If there is air, the space will slowly fill with water ice and liquid oxygen since liquid nitrogen is colder (77 K) than freezing point of water (273 K) and the condensation point of oxygen (90.19 K). Any combustable material (paper, plastic, oil, . . .) in contact with liquid oxygen is a very dangerous firebomb just waiting for a static spark to ignite.
- To avoid leaks that could be dangerous and prevent the experiment from working properly, be careful not to damage the dewar, hoses, or connectors. For example, do not put the cryostat can face down on any surface, to avoid damaging the vacuum seal.

NOTE: *This is not a complete list of all hazards; we cannot warn against every possible dangerous stupidity, e.g. opening plugged-in electrical equipment, juggling cryostats, etc. Experimenters must constantly use common sense to assess and avoid risks, e.g. if you spill liquid on the floor it will become slippery, sharp edges may cut you, etc. If you are unsure whether something is safe, ask the supervising professor, the lab technologist, or the lab coordinator. If an accident or incident happens, you must let us know. More safety information is available at* http://www.ehs.utoronto.ca/resources.htm.

3 Activities

3.1 Cryostat Exercise

Read Appendix A for information on the cryostat and complete the tasks listed.

3.2 Initial Setup

3.2.1 Calibrating Transdiodes

A *transdiode* is a transistor wired up so it acts as a perfect diode with exponential sensitivity to temperature (see Section 4a of the TeachSpin Cryostat Manual [4]). There are 3 transdiodes used as temperature transducers/sensors in this experiment:

- In contact with the LN2[1], this diode give the fastest response to any cooling due to the addition of LN2 in the system due to its proximity to the dewar.
- Attached to the copper baseplate,
- On the copper plate attached to the sample.

Each transdiode takes a 10 µA current in the forward direction. A higher current may damage the diode.

In order to convert the voltage reading of each diode to the correct temperature reading each transdiode must be calibrated, i.e. the three constants in the curve

$$V(T) = c_1 - c_2 T - c_3 ln(T/1\,\mathrm{K}) \tag{3}$$

must be determined. Equation 3 is a model of the voltage-temperature dependence of the transdiode with 3-pt calibration. To do this, measure the voltage of each diode at least two (or more) known temperatures. You always have access to two known temperatures: (1) room temperature and (2) the 77 K of LN2.

3.2.2 Calibrating the transdiode attached to the cryostat.

While the dewar outer casing is not attached, use a thermometer to measure the surface temperature of the LN2 dewar and the copper baseplate. This will be your hot point. Now with the reservoir closed and at vacuum but with the sample not attached, cool the reservoir down to 77 K. You will know you have reached this temperature, when the addition of any additional LN2 does not increase the voltage. The transdiodes at 77 K should be around 990 mV. What does a transdiode read at room temperature?

3.2.3 Calibrating the transdiode attached to sample.

Figure out which wires attached to the transdiode to use to inject/eject current and which to use to measure the voltage.

[1] Liquid Nitrogen is often referred to as "LN2", where the "N2" refers to the nitrogen diatomic molecule.

3.2.4 Temperature controller

The temperature of the sample is controlled by a Small Instrumentation Module (SIM) [5] Proportional-Integral (PI) controller [6]. This controller is connected to transdiode attached to the copper baseplate of the LN2 reservoir. To understand the operation of the PI controller and determine and set its best time constant, first put the system under vacuum and cool it to 77 K. With the DC power supply *off*, set the PI controller gain to 1 and the time constant toggle to OFF. (Note: All PI controller analog signal outputs are 10x the real signal amplitude.) Rotate the *set temp* knob such that the analog output reads 9.90 V. The error LED should be green. Keeping the DC power supply off, rotate the knob such that the set temperature analog output is 9.85 V, using the oscilloscope to monitor the error and temperature analog outputs.

Now when the analog output of the set temperature diode and the temperature diode match: increase the gain dial to a value of 5 or more and decrease the set temperature by another 0.05 V. Repeat this process until you see an overshoot in the temperature analog output as the PI controller tries to match the temperature of the set point. If you do not see an overshoot you should see a gain.

Now flip the switch on the gain control to x10. Starting at a modest gain value of 1 (this is actually a x10 gain), decrease the set point temperature again by 0.05 V. Continue this process of increasing the gain then decreasing the temperature until you not only see an overshoot but an oscillation in the temperature analog output around the the set temperature. Mark down the gain setting where this is achieved. The period of the oscillation is the time constant for the system. Adjust the time constant settings accordingly and decrease the gain slightly.

With the output of the DC power supply still off set the voltage to 60.0 V and the current limit to 1.0 A, connect the DC power supply output to the back of the PI controller using banana plugs. The PI controller is now ready to be used.

3.3 Test Resistance Measurements

To ensure that you understand how to measure the resistance of the sample, complete the setup shown in figure 1 with a breadboard and resistor (ideally 0.1 Ω, why?).[2]

Using the coil driver module, inject a 100 mA AC current into circuit. The coil driver has two analog connectors on its front panel. Use a function generator to send a signal through the *osc.in* connection that controls the amplitude and frequency of the generated current. The coil driver will output a AC current from its rear panel that is 1/10 the amplitude of the voltage signal but at the same frequency and phase. Monitor the generated output by connecting *mon.out* to an oscilloscope; *mon.out* passes the current over a 1.0 Ω resistor to generate a voltage of the same frequency and amplitude as the current.

The lock-in amplifier currently used in this experiment has a built in function generator. It is best to use this generator to power the coil driver.

Use the controls on the front panel of the lock-in amplifier set the internal oscillator

[2] 0.1 Ω resistors may be found in the Electronics Lab cabinets in MP238.

frequency to 37.0 Hz. Note, lock-in amplifiers display the root-mean-square voltage (V_{rms}):

$$V_{rms} = 1/\sqrt{2}V \text{ or } V_{rms} = 1/(2\sqrt{2})V_{pp} \tag{4}$$

where V_{pp} is the peak-peak voltage. Set the oscillator amplitude such that the output current generated by the coil driver is 100 mA.

Connect the *osc.out* analog channel on the lock-in to the *osc.in* on the coil driver. Monitor *mon.out* with an oscilloscope to ensure that the current is as expected.

NOTE: In order for *mon.out* to output the signal the coil driver must be driving a circuit: in this test it is the resistor on the breadboard. The output of the driver is two banana jacks on the rear panel of the coil driver module.

Now use the lock-in amplifier to measure the voltage across the resistor. Is the value what you expect?

3.4 Complete a run

With the above complete you are now ready to run your system. Make sure that you read the README.md file provided with the code for the experiment to understand how to take data.

1. Attach the sample to the nickel-plated copper baseplate.
2. Tightly screwing the brass bolts onto the baseplate will clamp the sample to the baseplate and create good thermal contact. Poor thermal contact will make it very hard for the sample to reach the desired temperature and affect
3. Close the system ensuring that all the electronics are properly working.
4. Power the PI controller using the DC power supply.
5. Use the code provided to run the system to some set temperature, and choose the sample rate.
6. Save the data as needed.

To cool the system simply turn off the heating resistors and add LN@ if needed.

3.5 Things to look for

1. The phase transition is not a sharp spike, but a slope. Find the width of the slope.
2. What are the effects of changing the sampling rate?
3. Try running the system such that the sample is at ambient pressure. Try again with it at vacuum. Are there a difference in your results?

References

[1] Michael Tinkham. *Introduction to superconductivity*. Dover Publications, Mineola, N.Y., 2nd ed edition (2004). ISBN 978-1-62198-598-3. OCLC: 853663851, URL https://app-knovel-com.myaccess.library.utoronto.ca/kn/resources/kpISE00023/toc?b-toc-cid=kpISE00023&b-toc-title=Introduction%20to%20Superconductivity%20(2nd%20Edition)&b-toc-root-slug=introduction-superconductivity&b-toc-url-slug=historical-overview.

[2] Charles P. Poole. *Superconductivity*. Elsevier, Waltham, MA, 3rd edition edition (2014). ISBN 978-0-12-409509-0. URL https://www-sciencedirect-com.myaccess.library.utoronto.ca/book/9780124095090/superconductivity#book-info.

[3] Toronto APL. "Lock-in Amplifier Quick Start" (2015). URL https://www.youtube.com/watch?v=oKmXaLlvr6c.

[4] David A. Van Baak. "CMP Variable-Temperature Cryostat Operating Manual" (2018). URL https://q.utoronto.ca/courses/278343/modules/items/4044619.

[5] TeachSpin. "PIController Manual.pdf" (2022). URL https://drive.google.com/file/d/1h_idUCgwuohgAqhnEcQWP3gW1MNY1A39/view?usp=sharing&usp=embed_facebook.

[6] TeachSpin. "SIMs" (2022). URL https://www.teachspin.com/sims.

Appendix A Cryostat and Vacuum Pump

This a brief introduction to the cryostat to get you started. For more details please find the TeachSpin Cryostat manual.

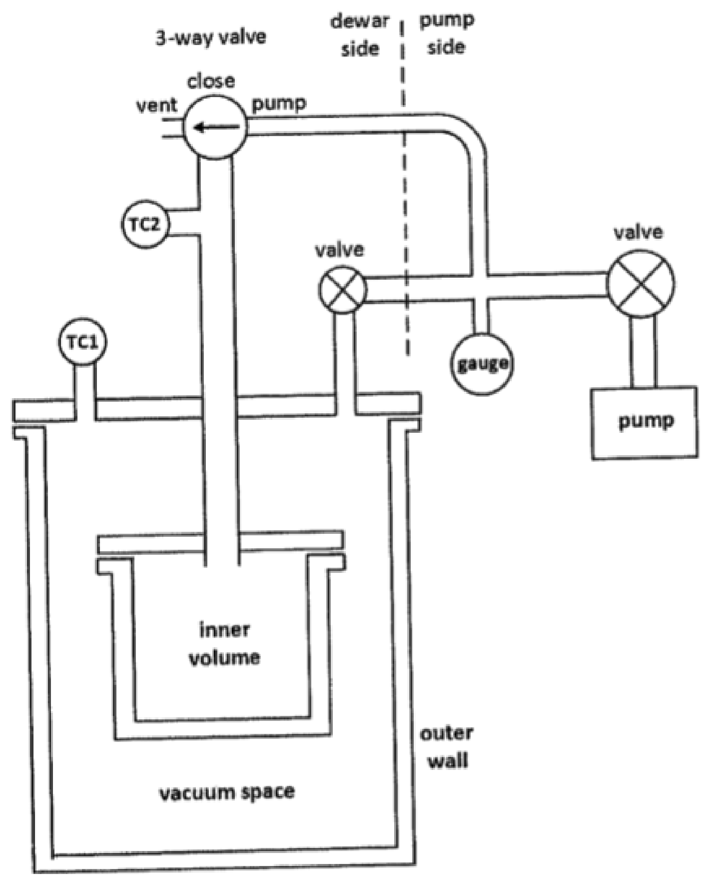

Figure 2: Schematic of cryostat system.

A.1 Parts

The apparatus has two main components (see Figure 2): the vacuum pump and the cryostat. The vacuum pump in turn includes two systems: the main pump, and a turbomolecular pump. The major cryostat components are labelled in Figure 3 and listed in Table 1

Not shown in Figure 3 are the three vacuum tight connectors which provide electrical access to the dewar. There are 4-pair, 5-pair, and 6-pair connections.

- The 4-pair connects the transdiode on the LN2 reservoir to the cryostat box. The voltage drop across the transdiode is temperature dependent, and is powered by a 10 µA forward DC current.
- The 5-pair connects to the to the back of the PI temperature controller module for control of the FET-based heating system (see Chapter 4 of the official manual for more

details). The series resistor heating system is powered by a current that you apply to 2 of the banana jack plugs in the cryostat box. These are the only two wires from the 5-pair interface that connect to the banana jack section.

- The 6-pair plug corresponds to the screw terminals on the baseplate inside the dewar.

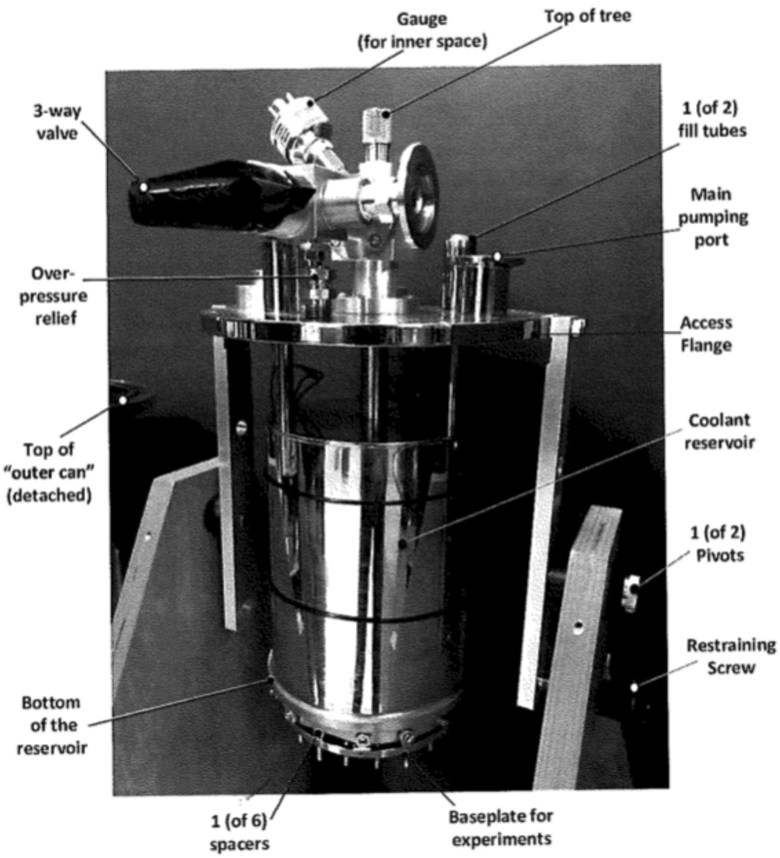

Figure 3: Main parts of the cryostat.

A.2 Temperature Control and Measurements

See chapter 4 and 5 of the cryostat manual for details.

A.3 Operation

1. Assuming the dewar is at room temperature and pressure, unlatch the KF flanges[3] connecting the dewar to the vacuum pump. (There are two both connected to the access flange on the top side of the dewer.)

2. Invert the apparatus such that the access flange is towards the ground and hold the system in placing using the large knob on the side of the wooden dewar holder.

[3] KF flanges are a common standardized vacuum fitting with an O-ring between two mating surfaces.

Table 1: List of cryostat parts shown in Figure 3.

Part Name	Description
3-way valve	Controls gas access to the inner can (when installed). Once the system contains LN2 this valve can not be changed. The direction of the arrow signifies what has access to the inner space. **left → right:** inner space connected to vacuum pump. **pointing up:** inner space is at atmospheric pressure. **right → left:** inner space can be filled with some gas connected to the nipple of the valve.
fill tubes	Gives access to the LN2 reservoir. One of these tubes must always be unobstructed.
overpressure valve	Releases gas if pressure in the system is too high.
coolant resevoir	2 litre container for LN2.
top of tree	if inner can is present and the 3-way valve is turned upwards, this port gives acess to the inner space.

3. Unscrew the 4 screws attaching the outer casing to the cryostat and remove it. Place the outer casing **face up** to avoid damaging the conductive ring around its open side.

4. If the inner (silver) can is on top of the sample plate, remove it. This is done by unscrewing the brass bolts in a star pattern. Remove the inner can, being careful not to lose any bolts or washers.

5. If required, connect the sample to the nickel-plated baseplate using the screws provided. Test all electronic connections to ensure that the measurement system is working correctly. Each wire can be connected to one of the numbered screw panels on the baseplate. Through the 12-wire (6-pair) connection on the access flange, this provides electrical connections to the outside world.

6. The space between the inner and outer cans must always be at vacuum when making cryogenic measurements, but the sample can be between vacuum and ambient pressure. Because of the risk of water ice or liquid oxygen, the sample is normally kept at vacuum. If the sample is to be kept at ambient pressure, the space should be filled with dry nitrogen gas, not ambient air. Rescrew the brass bolts onto the sample plate. To have the sample in the same vacuum as the main system, simply screw on 6bolts on to the screws attached to the spacers. Use a star pattern as follows:

   (a) Place the washers such that one is concave up and the other concave down as in Figure 4,

   (b) First finger tighten all the screws.

   (c) Then use the allen (hex) wrench to tighten the brass studs as much as you can with one hand. Do not over-tighten, since this could damage the threads.

(d) Wait 5 minutes then use the allen wrench to again to tighten each stud with one hand. Do not over-tighten.

If an inner can is used this must be done for all 18 brass studs.

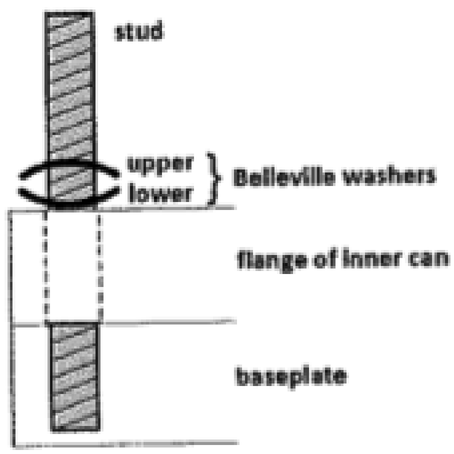

Figure 4: Sketch of the washer concave up and down.

7. Connect the KF flanges removed in the first step and turn on the vacuum pump. Use the pressure gauge up and down arrows to find the pressure between the vacuum and cryostat. The starting pressure should be $> 10^3$ hPa, but should quickly drop below 10^{-1} hPa and reach $\sim 10^{-5}$ hPa within about 30 minutes. If this does not happen and the system is stuck at a high pressure, turn off the pump and check for a leak.

8. Once the expected pressure is reached, use the funnel provided to pour LN2 into the reservoir. Ensure that one of the fill tubes in unobstructed to allow vapour to escape from the system. Pour one funnel volume at a time to cool down the system. It is best to wait a few minutes after every 2 funnels worth of fluid is added to the reservoir. It should take about 45 minutes for the system to cool to the desired temperatures.

9. Measurements can be taken as the sample cools and as it warms up.

10. **To turn off the system** and revert it to a warm dry state, first close the angular valve directly on top of the vacuum pump. Next turn off the vacuum pump (flip the power switch) and turn off any heaters. The system will slowly warm up overnight and the next morning it should be at room temperature. At this point you can evacuate the gas in the inner can, then open the angular valve above the dewer itself. This will allow the system to slowly reach atmospheric pressure. After a few minutes waiting the system is again ready to be used.

Appendix B DAQ Software

The data acquisition (DAQ) software consists of 4 files that are available on the CMP experiment web page.

- event_handler.py
- lockin_7270.py
- oscilloscope.py
- temp_calibration.py

B.1 Requirements

You can use either the lab computer or your own laptop to run the experiment. Using your own computer means you won't need to copy files.

To run the data acquisition (DAQ) software from your laptop, the following python libraries must be installed:

- numpy
- pyusb
- pyvisa
- array
- threading
- time

The last three are standard Python 3 modules, but one or more of the first three may need to be installed. If you don't know how, search online for the correct method for your specific python installation. For example, for an Anaconda installation, you would use conda install -c anaconda numpy to install *numpy*.

B.2 Data Acquisition Tutorial

This tutorial shows how to make a data analysis run for a superconductivity measurement, changing the acquisition sample rate to improve the I-V curve resolution near the superconducting transition temperature.

[1]:
```
# import these libraries
import numpy as np
from event_handler import EventHandler
import time
import sys
```

Create the *event handler* for the measurements

[2]:
```
# Create an event object
cmp_sc_run = EventHandler()
```

Run the system up to a transdiode reading of 0.8 volts at a sample rate of 10 ms.

[3]:
```
cmp_sc_run.run(0.8, 10000)
```

This should produce this output.

```
Begining data aquisition...
Lock in running.
Run complete.
```

The lock-in data is stored in a dictionary, whose key value is a number corresponding to the data type and the value is a list of stored measurements.

key	**measurement type**
0	X magnitude
1	Y magnitude
3	Phase
4	Sensitivty
5	Noise

The scope data is stored in the a list. By default this is a list of voltage readings from the transdiode.

[4]:
```
lock_in_data = cmp_sc_run.lock_in.data
scope_data = cmp_sc_run.scope.data
```

[5]:
```
scope = cmp_sc_run.scope
```

[6]:
```
mag = lock_in_data[0] # x magnitude
```

[7]:
```
import matplotlib.pyplot as plt
```

[8]:
```
SEN = {1: 2e-9, 2: 5e-9, 3: 10e-9,
       4: 20e-9, 5: 50e-9, 6: 100e-9,
       7: 200e-9, 8: 500e-9, 9: 1e-6,
       10: 2e-6, 11: 5e-6, 12: 10e-6,
       13: 20e-6, 14: 50e-6, 15: 100e-6,
       16: 200e-6, 17: 500e-6, 18:1e-3,
       19: 2e-3, 20: 5e-3, 21: 10e-3,
       22: 20e-3, 23: 50e-3, 24: 100e-3,
       25: 200e-3, 26: 500e-3, 27: 1}
```

[9]:
```
import matplotlib.pyplot as plt
```

[10]:
```
# convert sensitivity output from lock in to decimals
sen = [SEN[i] for i in lock_in_data[4]]
```

[11]:
```
# convert x reading and sensitivity (float converted) to
   arrays # and complete element wie multiplications
M = np.array(mag)
S = np.array(sen)
X=M*S
```

Simply plotting the results may produce a weird looking graph.

1. The temperature of the transdiode attached to the sample is in volts with 0.99 representing 77 kelvin.
2. When converting the sensitivity to a floating point number, for some unknown reason the values must be divided by another factor of 10.
3. The sign of the voltage is negative, but only care about the absolute value.
4. There is also some aliasing that needs to be averaged over.

Now we plot the raw data

[12]:
```
plt.scatter(scope_data, np.abs(X), s=0.3)
```

Now we average over the each iteration (length of buffer is 300) and look at the absolute value of the averaged x magnitude. We see that the trend matches what we would expect. As the material decreases in temperature the resistiviity of the material decreases (ie the voltage).

Remember we are looking at V_{rms} here to get a reading of the resistance (or more generally impedeance) because the Irms is known. To actually get the resistance value (meausrements in ohms) you have to divide by the current rms.

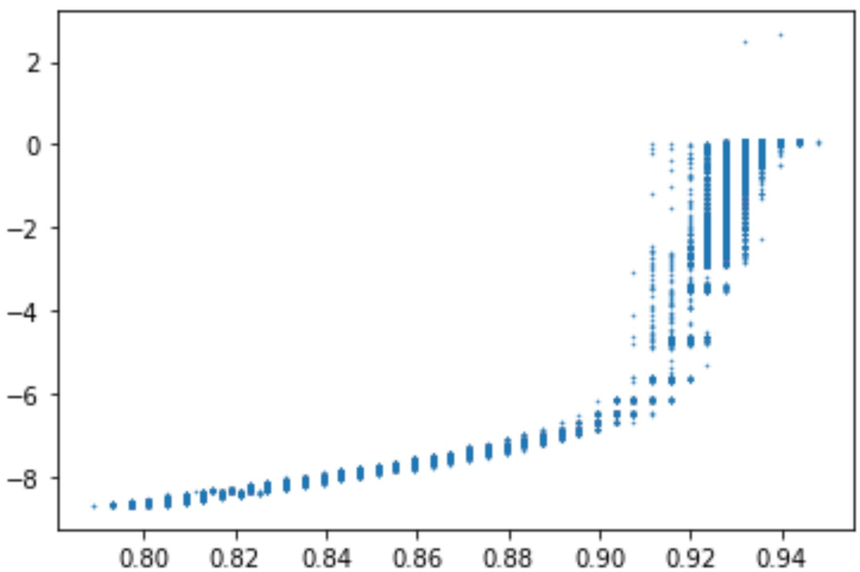

Figure 5: Raw data

[13]:
```
k = []
for i in range(0, 30900, 300):
    k.append(np.average(X[i:i+301]))
```

Plot the averaged data

[14]:
```
plt.plot(scope_data[::300], np.abs(k)*1e-1)
```

and the sensitivity,

[15]:
```
plt.plot(S)
```

and save the data:

[16]:
```
np.savetxt("lock_in_data_x.csv", lock_in_data[0])
 np.savetxt("lock_in_data_y.csv", lock_in_data[1])
np.savetxt("lock_in_data_phase.csv", lock_in_data[3])
np.savetxt("lock_in_data_sen.csv", lock_in_data[4])
np.savetxt("lock_in_data_noise.csv", lock_in_data[5])
np.savetxt("scope_data.csv", scope_data)
```

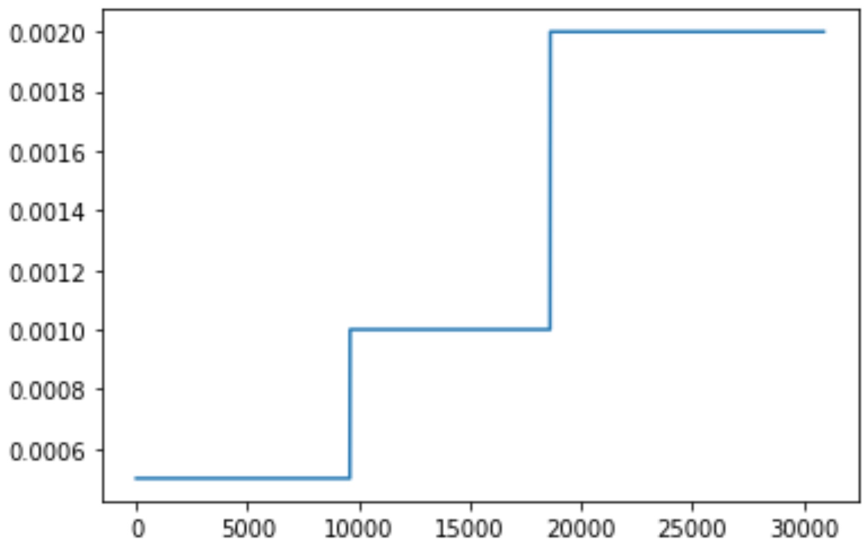

Figure 6: Sensitivity