Experiment 12

Lock-in Detection: High-T_c Superconductivity

12.1 Purpose

To introduce you to lock-in amplifiers and some topics of current interest to researchers in the department.

12.2 Introduction

Superconductivity was first observed in 1911, by Dutch physicist Heike Kammerlingh-Onnes. When he cooled mercury in liquid helium, its DC resistivity abruptly vanished around 4K. It wasn't until the 1950's that a satisfactory explanation was found — two electrons couple to a phonon (quantized lattice vibration), lowering their energy and changing even their most basic properties. By the 1970's, superconductors were fully understood, metallic compounds had been found with superconducting transition temperatures (T_c) as high as 23K, and theorists had proven that it would be impossible for anything to have a T_c much higher than 25K.

In late 1986, Swiss physicists J. Georg Bednorz and K. Alex Müller discovered superconductivity at nearly 40K in $La_{2-x}Ba_xCuO_4$, an otherwise poorly-conducting ceramic. This startling discovery sparked an intense effort to find and study more such compounds. In January 1987, the first compound was found with a T_c above the temperature of liquid nitrogen (77K) — $YBa_2Cu_3O_{7-x}$ (YBCO, T_c= 93.7K for $x = 0.08$). Today, many such compounds are known, the highest recorded ambient-pressure T_c is 138K, and there is no accepted explanation for why they superconduct. While these temperatures are still quite cold by most people's standards, they're extremely high for low-temperature physics, and are readily attainable.

Low-temperature (conventional) superconductors are in common use for high-field magnets (as used in MRI machines), but the liquid helium required to keep them cold makes the cost prohibitive for most other uses. Liquid nitrogen is far cheaper than helium, so high-T_c superconductors should eventually see far more common use. An application of particular current interest is in cellphone base stations and satellites, where the absence of electrical resistance can make bandwidth constraints less of a problem. A variety of superconductor-based quantum computing schemes have also been suggested, and are being investigated.

12.3 Theory

Aside from having zero DC resistance, superconductors are also perfect diamagnets. This well-understood property, known as the Meissner Effect, is commonly used for spectacular demonstrations — a magnet placed atop a piece of YBCO will spontaneously levitate when the YBCO goes superconducting. The ground state for this system is to have currents on the surface of the superconductor, cancelling out the magnetic field. The currents, and thus the fields, in fact decay exponentially into the sample (Figure 12.1), with a characteristic length λ_L, the magnetic (or London) penetration depth. In the high-T_c superconductors, λ_L depends on the temperature and what

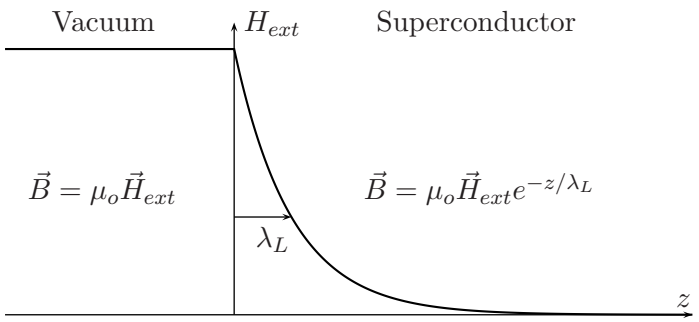

Figure 12.1: Magnetic field decaying into a superconductor.

direction the currents are travelling, and ranges from ∼0.1 to 10μm. Figures 12.2a and b show our superconducting crystal in an applied magnetic field above and below T_c.

From Faraday's Law, the voltage induced in a wire loop in a time-varying magnetic field is

$$V_{loop} = -\frac{d\Phi}{dt}$$

where the magnetic flux $\Phi = \iint \vec{B} \cdot d\vec{a}$. In our setup, an external AC magnetic field

$$\vec{B}_{ext} = B_{ext} \sin(\omega t)\hat{x} = \mu_o H_{ext} \sin(\omega t)\hat{x} \tag{12.1}$$

is applied, and the voltage in a pickup coil (of length L, cross-sectional area A, and N turns per unit length) would be

$$V_{coil} = -NLA\mu_o H_{ext}\omega \cos(\omega t), \tag{12.2}$$

or

$$|V_{coil}| \propto LAH_{ext}. \tag{12.3}$$

It is customary to use $\vec{H}$, the field in matter, instead of $\vec{B}$, because $\vec{H}$ is the field we can directly control. Ampère's Law in matter reads

$$\vec{\nabla} \times \left(\frac{1}{\mu_o}\vec{B} - \vec{M} \right) = \vec{J}_f$$

or

$$\vec{\nabla} \times \vec{H} = \vec{J}_f \tag{12.4}$$

where $\vec{J}_f$ is the free current and $\vec{M}$, the "magnetization", is the magnetic dipole moment per unit volume.

However, we're not interested in measuring the applied field; we're looking for small changes to it due to the presence of a superconducting sample. The apparatus used has two identical coils a short distance apart, wound in opposite directions and connected in series. In a uniform AC field,

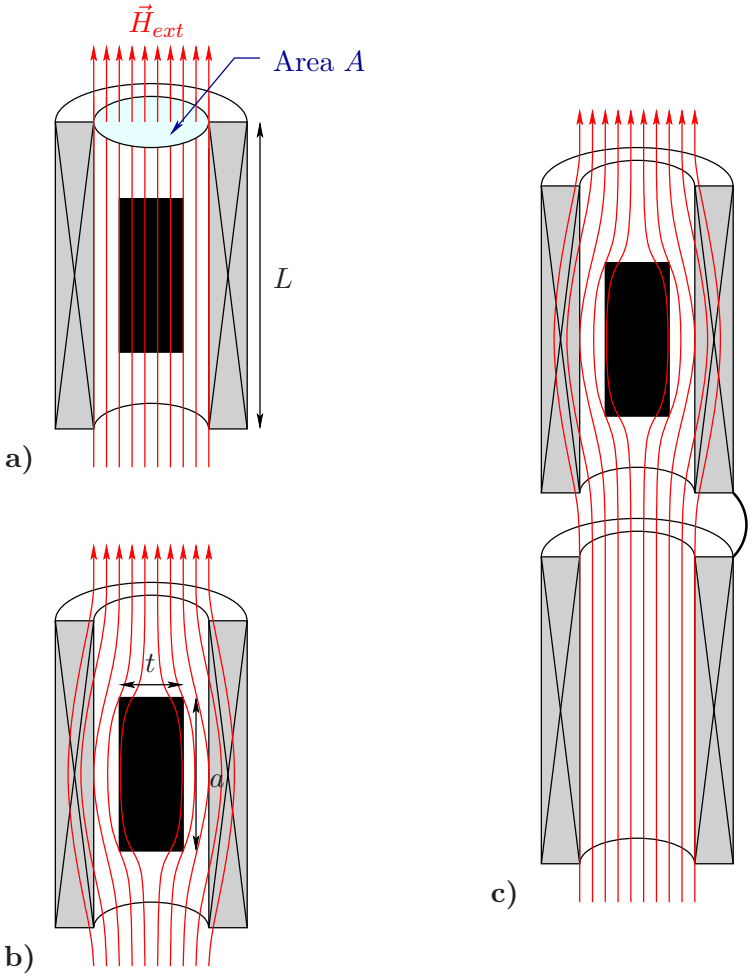

Figure 12.2: Perfect diamagnetism in a superconductor. **a)** For $T > T_c$, YBCO is slightly metallic, and does not significantly alter the magnetic field. **b)** For $T < T_c$, the superconductor expels the applied field, and the pickup coil sees a reduced field. **c)** In our apparatus, the sample is in the upper of two counterwound coils connected in series. In a uniform field (above T_c), there is no net field, but below T_c the two coils see different fields, so there is a net field detected in the circuit.

they will contribute equal voltages, and these will cancel, leaving no signal. However, if one half contains a piece of YBCO (Figure 12.2c), that half will give a different voltage

$$|V'_{coil}| \propto \left[LA - \ell_x \ell_y t \left(1 - \frac{2\lambda_L}{t}\right)\right] \tag{12.5}$$

$$= C\left(LAH_{ext} - |\vec{m}|\right) \tag{12.6}$$

where C is a calibration constant and $\vec{m}$ is the sample's total magnetization. The voltage detected will be

$$\left|V_{coil} - V'_{coil}\right| = C|\vec{m}| = C'\left(1 - \frac{2\lambda_L}{t}\right). \tag{12.7}$$

A measurement of the sample's magnetization, then, can be used to determine the London penetration depth.

12.4 The Apparatus

Figure 12.3 shows the key components of the apparatus.

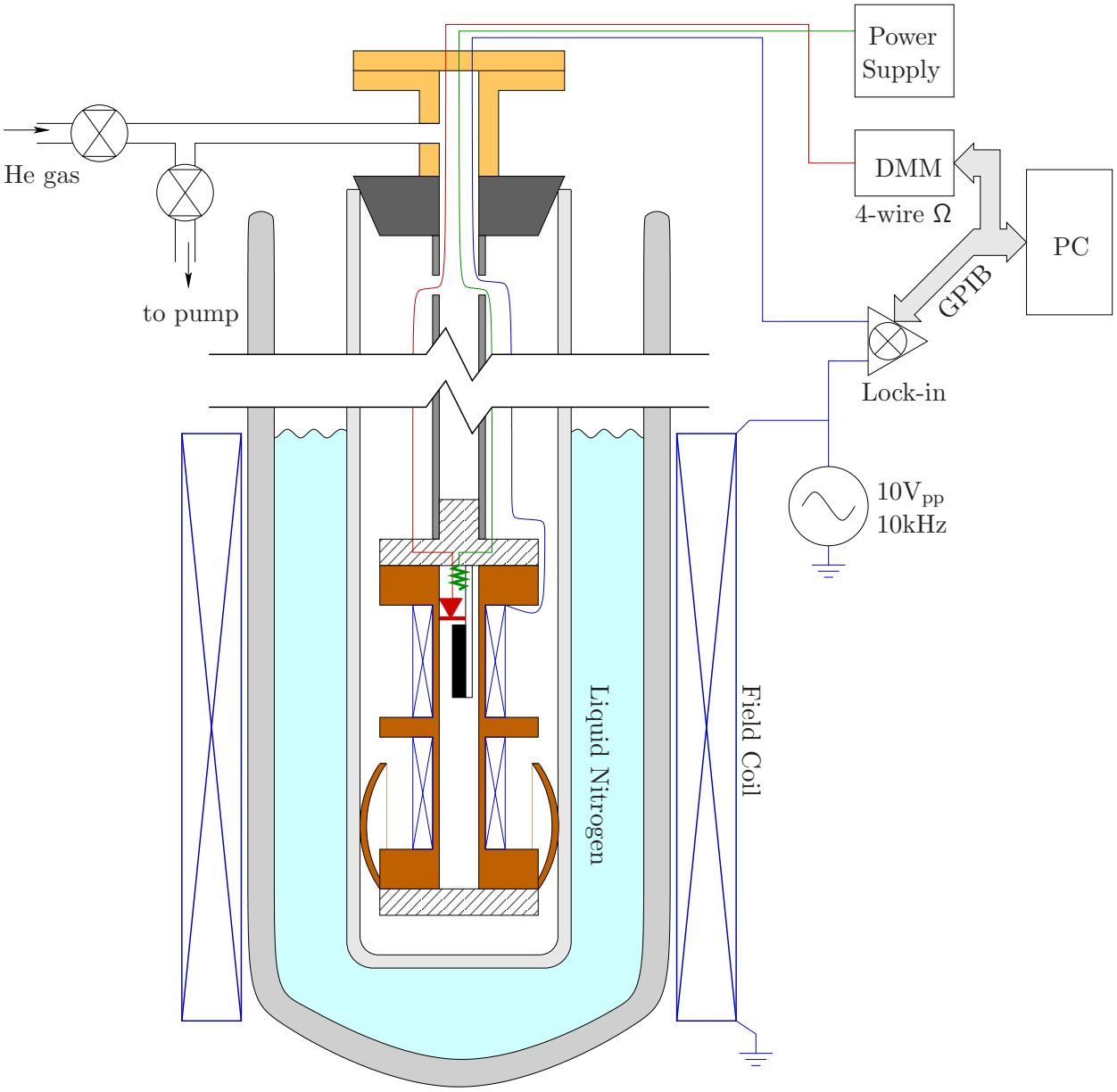

Figure 12.3: Schematic diagram of the apparatus. There are four subsystems — the vacuum/cryogenics plumbing and the magnetic detection, thermometry and heating circuitry. The computer monitors the temperature and detected signal, and runs current through the 50Ω resistor to heat the probe.

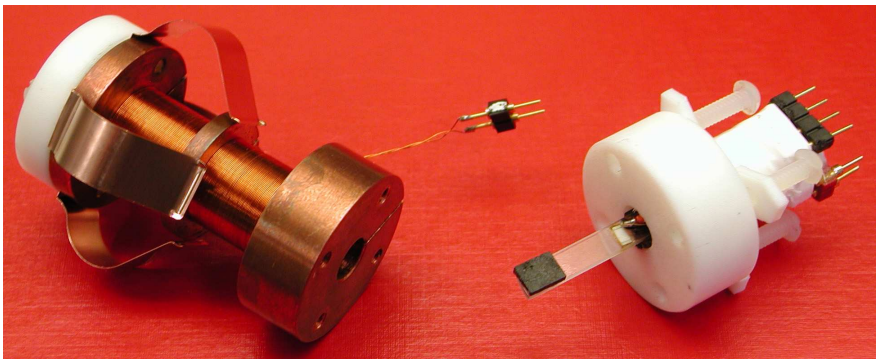

Figure 12.4: Photograph of the susceptometer.

Plumbing and Temperature Control

The probe is cooled to 77.4K in liquid nitrogen, which is held in a dewar flask. A quartz test tube separates the liquid nitrogen from the probe, which allows the probe to reach temperatures other than 77K and prevents damage from thermal shock. Beryllium-copper springs make thermal contact between the copper coil assembly and the test tube, to keep the coils cold. The sample and the thermometer and heater chips are mounted on a sapphire (Al_2O_3) plate, which is a very good thermal conductor. This is thermally insulated from the rest of the probe with teflon/quartz. Figure 12.4 shows a photo of this part of the probe.

To cool the probe, the quartz tube is filled with helium gas, which is then pumped out for thermal isolation. How does this helium business work, and why do we use helium for this purpose? To warm the sapphire plate, a current is applied to the chip resistor; the temperature reached will depend on the current applied.

Thermometry

The sapphire plate also holds a silicon diode which acts as a thermometer. Diodes thermometers are usually used by passing a constant current through them and measuring the voltage. We will use the four-wire resistance function of the multimeter to do this. (see Figure 12.5).

In a four-wire resistance measurement, a constant current is supplied to the load through two leads, and the voltage across it is measured using the remaining two leads. The resistance is then obtained through Ohm's law. Four-wire measurements are far superior to two-wire for small resistances, because the latter measures the resistance of the load *and* the wires. In a four-wire measurement, there is negligible current in the voltage leads, so only the load is measured. In the case of the diode thermometer shown in figure 12.5, the thermal gradient we apply would make it virtually impossible to account for the resistance of the leads, making 4-wire measurements crucial. The diode is also extremely non-Ohmic, so a constant bias current is required.

The current through a diode varies as

$$I = I_o \left(e^{eV/nk_BT} - 1 \right) \tag{12.8}$$

where $I_o(T)$ is the reverse-biased saturation current, V is the diode voltage, and T is the temperature, and n is diode-dependent factor known as the 'nonideality factor'. Usually $1 < n <\sim 2.5$ I_o is also a function of temperature, and is often expressed as:

$$I_o = KT^{5/2} e^{-V_g/2k_BT}, \tag{12.9}$$

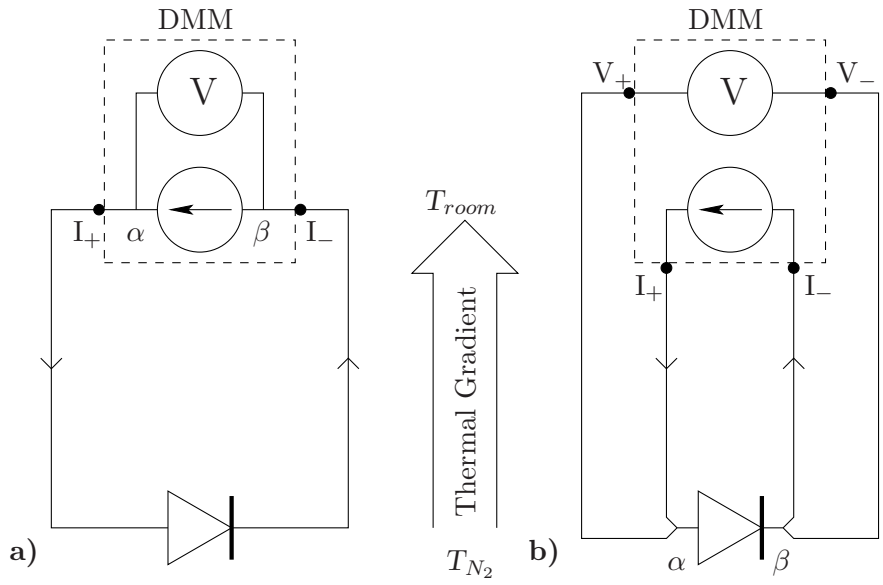

Figure 12.5: **a)** Two-wire and **b)** four-wire measurements on a diode thermometer. The DMM's current and voltage connections are marked.

in which K depends on the details of the diode and E_g is the band gap of the semiconductor. Taking the logarithm of I, we find:

$$V = \frac{n/2e}{V_g} - \frac{nk_B}{2}\left(\frac{5}{2}\ln T - \frac{\ln I_f}{K}\right). \tag{12.10}$$

The weak logarithmic dependence on T can usually be ignored, and so we find

$$V = V_0 - m(I)T. \tag{12.11}$$

The voltage measured across the diode, for fixed forward bias current is thus expected to vary linearly with temperature, and can be calibrated using measurements at 77.4 K and room temperature. It is important to note that the measurements ***must*** be performed on the same (manually set) range on the DMM, to ensure a constant bias current.

Magnetometry and Lock-in Detection

Outside the dewar, a large coil is used to apply an AC field ($\vec{H}_{ext}$) to the probe. To ensure field uniformity, it is quite useful to have the probe centred inside this coil. The probe itself has two counterwound coils, as described in the Theory section, to detect magnetization in the sample rather than just the applied field.

While the signal due to the sample is already quite small, we're looking at an even smaller signal — we want to determine how λ_L changes with temperature. Recall from Equation 12.7 that the detected signal $\propto$ (*sample size* $-$ *shell of thickness* λ_L). The λ_L term is the interesting one, but it's the smaller term by a factor $\frac{2\lambda_L}{t} \sim 10^3 - 10^5$. Any minute changes in λ_L will be completely drowned out by noise proportional to the sample size.

The advantage we have in this experiment is that we know exactly what frequency our signal should have. To separate it from broadband noise, all we need is a filter. Because the noise

is much stronger than the signal, we need a very narrow filter — our signal has a vanishingly small frequency width, so the probability of having broadband noise at exactly the same frequency also vanishes. Unfortunately, the feasability of constructing such a narrow filter for an arbitrary frequency vanishes at least as quickly.

A common solution would be to do a Fourier transform of your detected signal, so you can look at the frequency spectrum and pick off the frequency you want. The problem is getting a frequency spectrum in the first place. Fourier transforms require significant amounts of data and data analysis. The solution implemented in this experiment instead uses a special type of phase-sensitive filter and voltmeter known as a lock-in amplifier.

The lock-in is given reference and sample signals, with phases ϕ_r and ϕ_s, and the reference signal is converted to a sine wave of unit amplitude:

$$V_r = \sin(\omega_r t + \phi_r)$$
$$V_s = \mathcal{A}\sin(\omega_s t + \phi_s)$$

The reference and sample signals are then mixed to yield an output at their sum and difference frequencies.

$$V_r \times V_s = \frac{\mathcal{A}}{2}\left[\cos\left(\left(\omega_s - \omega_r\right)t + \phi_s - \phi_r\right) - \cos\left(\left(\omega_s + \omega_r\right)t + \phi_s + \phi_r\right)\right] \tag{12.12}$$

In this experiment, the output frequency will be the same as the input frequency, so the first term will be at DC and the second at a frequency of 2ω.

Next, the mixed signal passes through a low-pass filter to yield only the DC component:

$$V_r \times V_s \xrightarrow{\text{low-pass filter}} \frac{\mathcal{A}}{2}\cos(\phi_r - \phi_s)$$

Frequencies other than the reference frequency do not give DC components, and can be removed by the low-pass filter. However, while it's far easier to make a low-pass filter than a band-pass filter at arbitrary frequency, the filter still has a width — the inverse of its time constant. You may have noticed, though, that signals are maximized for $\phi_r = \phi_s$, and a phase difference of $\pi/2$ will not contribute to the output. So we're getting phase information for free (or we can take advantage of a known phase relationship to further improve our data).

This is the basis of phase- and frequency-specific detection. Lock-in amplifiers provide an immense advantage when measuring small signals if the system can be driven by an AC voltage with a very stable amplitude and phase.

Why is phase-sensitive detection important in this experiment? Because of the derivative in Equation 12.2, we're interested in the inductive response of the detector coils, which will be $-\pi/2$ out of phase with the driving field. Since the driving coil is out of phase with the function generator by $+\pi/2$, the voltage from the sample's magnetic moment will be *in phase* with the function generator. On the other hand, there will be resistive signals $\pi/2$ out of phase with the function generator — resistance both in the wires and in the superconductor (it has zero DC resistivity, but a small AC resistivity). Phase-sensitive detection allows us to distinguish between the inductive and resistive components.

In this experiment, the counterwound detector coils provide V_{sample} and we're primarily interested in the inductive response, at zero phase shift. If time permits and you feel like investigating the resistive response, then you can set the reference phase to $\pi/2$.

12.5 Procedure

Study I — Lock-in Signal Detection

This part of the experiment will introduce you to lock-in detection of small signals. Recall that resistance R is determined by resistivity ρ, length D and cross-sectional area A through

$$R = \int_0^D \frac{\rho}{A} dx.$$

1. What are the highest and lowest available time constants on the lock-in? What is the best frequency resolution you would expect from each? (This unit has a second filter stage, labelled as "Post," for steeper roll-off. You should disable it, by setting it to "None.")

2. Connect the function generator to both the reference and A inputs (use 1 kHz, ∼250 mV). Take amplitude readings for phase shifts from -π to π. Does this behave as you expected?

3. You should find a small slab of aluminum at your bench, with four female banana connectors. Use the digital multimeter's four-wire resistance function (method mentioned earlier) to find its resistance, and thus its resistivity. The voltage-sensing leads are labelled as "Ω4W Sense" and the current source (I_+) is marked with a lightning bolt (⚡). Remember, you want to measure the voltage due to a current, and you're trying to eliminate any effects from resistance in the leads or contacts. Perform a two-wire measurement for comparison.

4. Now, connect the function generator as a current source (at ∼1 kHz) and connect the oscilloscope to the voltage posts. You will need to measure the applied current with the multimeter. Find the resistance, and thus the resistivity of the aluminum.

5. Finally, replace the oscilloscope with the lock-in, remembering to also connect the function generator to the lock-in's reference input (what phase?). Using the lock-in signal, again determine the resistance and thus the resistivity. How do the various resistivity measurements compare to each other and to the accepted value?

Study II — Magnetization of $YBa_2Cu_3O_{6.95}$

You will now determine the magnetic properties of a crystal of YBCO by AC susceptometry. Refer to Figure 12.3 for a diagram of the apparatus.

1. Before doing anything else, take a measurement of the diode voltage under bias current, using the four-wire resistance measurement function of the multimeter, with the range manually set to 20kΩ. The multimeter must remain on this resistance range for the duration of the experiment. As long as the bias current stays fixed, it is ok for you to record the diode voltage in units of ohms (these are the units that the multimeter will supply the voltage in - because it thinks its making a resistance measurement). A resistance value for 77K should already be present in the LabVIEW program, completing the temperature calibration — you don't need to adjust this point.

2. Connect the function generator (10kHz, $10V_{pp}$) to the lock-in's Reference input and the outer coil, and the detector coil to the lock-in's A input. Insert the susceptometer into the drive coil, watching the signal on the lock-in. It should reach a maximum, then decrease toward

the centre of the drive coil. Why? Find the position that minimizes the signal, and use this position for the remainder of the experiment.

3. The test tube containing the probe needs to have 1 atm He gas in it. To this end, pump out whatever gases may be in it, using the vacuum pump, then gently fill it with helium gas and close the valve. Fill the dewar with liquid nitrogen. The diode's resistance should rise rapidly, then level off as the probe approaches 77K.

4. With the probe at liquid nitrogen temperature, you now want to remove the helium. Start the pump, and, leaving the valve to the probe closed, pump the line down to $\lesssim$ 10 torr. Slowly open the valve, and allow the probe to pump down to $\lesssim$ 10 torr. Set the phase on the lock-in to zero, and ensure that the signal is not off the scale. With the helium removed, you may ***slowly*** warm the sample, using the DC power supply as necessary. Take data as you do so. You want the probe to warm at about 0.5 K/min. Continue recording the lock-in output and resistance to at least 105K.

5. You might expect your plot of lock-in voltage vs. temperature to be zero above T_c, where the field fully penetrates the superconductor, but your graph will probably show an offset and a slope in this regime. What causes can you think of for the slope and offset? To remove this background signal, fit the linear portion above T_c, and subtract this line from the *entire* data set.

6. Rescale your data so the voltage values vary from zero to one. Since $V \propto |\vec{m}(T)|$, this plot shows $\frac{|\vec{m}(T)|}{|\vec{m}(T_o)|}$ vs. T.

7. Finally, use Equation 12.16 or 12.17 to extract $\lambda_L(T)$. To do this, take $\lambda_L(77\text{K}) = 3000\text{Å}$, and the sample's dimensions as $5.50 \times 4.35 \times 1.30$ mm^3, ± 0.05 in each. Generate a log-log plot of $\lambda_L(T)$ over the range T_c – 10K $\rightarrow T_c$. The x-axis should use $\frac{T_c - T}{T_c}$ instead of T. Is there any evidence of a power law $\lambda_L(T) \propto \left(\frac{T_c - T}{T_c}\right)^n$? (Note that the assumption $\lambda_L \ll t$ is not true very close to T_c, because λ_L diverges at T_c.)

12.6 References

For E&M, any undergraduate text will work (e.g. Griffiths' *Introduction to Electrodynamics*). A high school-level (and overly optimistic and partially wrong) introduction to superconductivity may be found at `http://www.superconductors.org`, but Tinkham's 4$^{\text{th}}$ year-level text *Introduction to Superconductivity* is **much** better. Additionally, some similar research has been done at UBC — Chris Bidinosti's Ph.D. thesis, available from on-campus at
`http://www.physics.ubc.ca/~supercon/archive/bidinosti-phd.pdf` may be useful, particularly parts of its Introduction. The growth of the YBCO crystal is described in
`http://arxiv.org/abs/cond-mat/0209418`.

For diode thermometry: T. Huen, Rev. Sci. Instrum. **41**, 1368 (1970).

12.7 Appendix — Magnetic Moment of a Superconducting Platelet

Consider a superconducting platelet of thickness t and broad dimensions $\ell_x \times \ell_y$ in a uniform external magnetic field $H_{ext}\hat{x}$, as shown in Figure 12.6. Furthermore, assume that $\ell_i \gg t$, so that we can consider the platelet to be an infinite slab (λ_b becomes λ_L and λ_c plays no role).

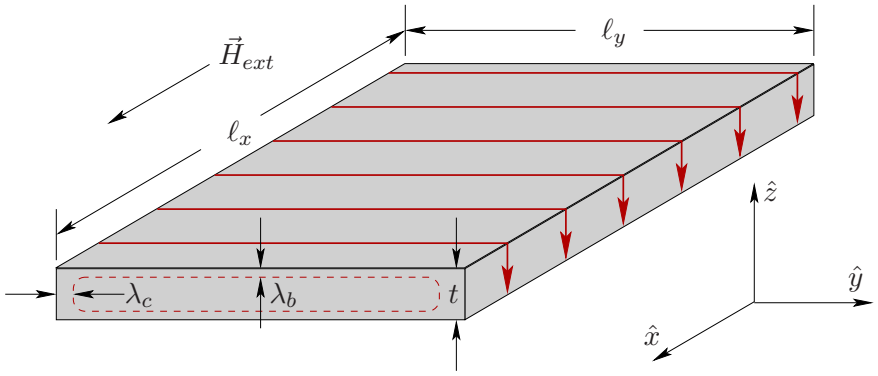

Figure 12.6: The crystal provided, with the currents and penetration depths shown. $\ell_x \approx 5.50$ mm, $\ell_y \approx 4.35$ mm, and $t \approx 1.30$ mm.

The magnetic flux density ($\vec{B}$) at any point inside the platelet will be the sum of the exponentially decaying fields from the two sides of the sample:

$$B_x(z) = D\left[e^{(z-t/2)/\lambda_L} + e^{-(z+t/2)/\lambda_L}\right] \tag{12.13}$$

where D is a constant that must be chosen to satisfy the boundary conditions $B_x(\pm t/2) = \mu_o H_{ext}$. The result is

$$B_x(z) = \mu_o H_{ext}\frac{\cosh(z/\lambda_L)}{\cosh(t/2\lambda_L)}. \tag{12.14}$$

Finally, using the general expression $\vec{B} = \mu_o\left(\vec{H} + \vec{M}\right)$, we can derive the magnetization of an infinite slab of thickness t:

$$\vec{M} = H_{ext}\left(\frac{\cosh(z/\lambda_L)}{\cosh(t/2\lambda_L)} - 1\right) \tag{12.15}$$

The total magnetic moment $\vec{m}$ is a good measure of how effectively the sample screens the field, and can be obtained experimentally. For our case,

$$\begin{aligned}\vec{m} &= \iiint \vec{M}dV \\ &= \ell_x\ell_y t\left(1 - \frac{2\lambda_L}{t}\tanh\left(t/2\lambda_L\right)\right)H_{ext}\hat{x}\end{aligned} \tag{12.16}$$

or, $|\vec{m}|$ =Volume of sample $\times$ fraction of volume screened $\times H_{ext}$.

In the limit where $\lambda_L \ll t$, this becomes

$$|\vec{m}| \approx \ell_x\ell_y t\left(1 - \frac{2\lambda_L}{t}\right)H_{ext}. \tag{12.17}$$

In this limit, the field effectively penetrates the sample to a depth of λ_L on each broad side. Either Equation 12.16 or 12.17 can be used to relate the magnetic moment to the London penetration depth.