

Low Temperature Transport

Experiment LTT

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PHY4803L — Advanced Physics Laboratory

Objective

Temperature has dramatic effects on the transport properties of metals and semiconductors. The electrical resistance of pure metals can decrease by several orders of magnitude as temperature is reduced from room temperature to cryogenic temperatures. The junction voltage of a p - n junction increases in a characteristic way as temperature is lowered, allowing the Si diode to be used as a thermometer. In this lab you will study these properties at temperatures between about 10 and 325 K.

References

Charles Kittel, *Introduction to Solid State Physics*, Wiley (2004), ISBN 978-0-471-41526-8

M. Omar Ali, *Elementary Solid State Physics*, Addison-Wesley (1975) ISBN 0-201-60733-6.

Theory

The following is a very brief introduction to the subject matter involved in this experiment. You are expected to read a solid state textbook such as Chapters 6–9 of Kittel or Chapters 3–7 of Ali to gain a good foundation on the topics discussed here.

Metals physics

A metal is a solid in which one or more electrons per atom are free to move throughout the solid. In the language of band theory, the highest occupied band is partially filled, so that the Fermi surface lies in the middle of the band, and there is an infinitesimal energy between the highest occupied orbital and the lowest unoccupied one.

The simplest model of a metal is the *free-electron model*, where the atomic potential is taken to be a constant, U_0 , and the electrons are treated as non-interacting. Then the Schrodinger equation for each electron becomes

$$-\frac{\hbar^2}{2m}\nabla^2\psi(\mathbf{r}) + U_0\psi(\mathbf{r}) = E\psi(\mathbf{r}), \quad (1)$$

where \hbar is Plank's constant, m the electronic mass, and E the energy eigenvalue. Without loss of generality, we can take $U_0 = 0$ and find that there are plane wave solutions for the eigenfunctions $\psi(\mathbf{r})$:

$$\psi(\mathbf{r}) = \sqrt{\frac{1}{V}}e^{i\mathbf{k}\cdot\mathbf{r}}, \quad (2)$$

where V is the sample volume and the factor $\sqrt{1/V}$ satisfies the normalization condition $\int \psi^*\psi dV = 1$. The quantity \mathbf{k} is the wavevector of the plane wave. Its magnitude k is related to the deBroglie wavelength λ via

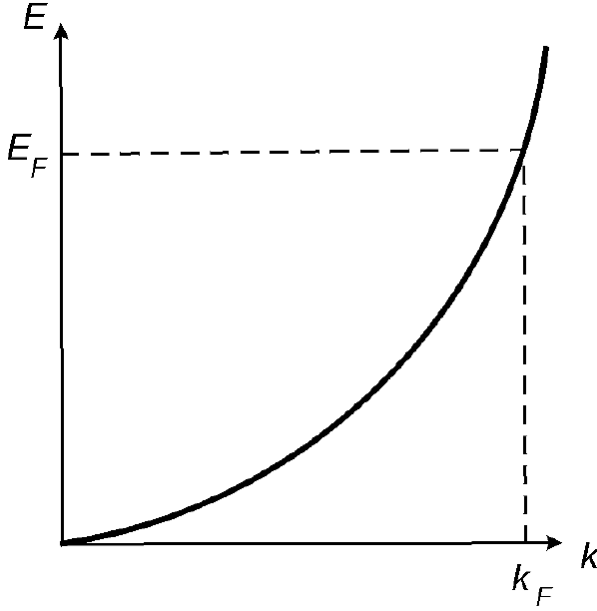


Figure 1: Dispersion relation (E as a function of k) for free electrons. The Fermi energy and Fermi wavevectors are indicated

$k = 2\pi/\lambda$ and \mathbf{k} is related to the electron momentum \mathbf{p} via $\mathbf{p} = \hbar\mathbf{k}$.

The eigenenergy E appearing in Eq. 1 (for $U_0 = 0$) is the electron kinetic energy $p^2/2m$ and thus depends only on the wavevector magnitude:

$$E(\mathbf{k}) = \frac{\hbar^2 k^2}{2m}, \quad (3)$$

The allowed values of \mathbf{k} are determined by the boundary conditions. It is conventional to use *periodic boundary conditions*, where the sample is taken to be a cube of edge L , $V = L^3$, and the wavefunction is required to be periodic:

$$\psi(x + L, y, z) = \psi(x, y, z), \quad (4)$$

and similarly in the y and z directions. This condition¹ leads to quantized values for each

¹One can come to the same conclusions by assuming fixed boundary conditions, with $\psi(x, y, z) = 0$ for $x \leq 0$ and $x \geq L$, and similarly for y and z .

component of \mathbf{k} :

$$k_i = 0, \pm\frac{2\pi}{L}, \pm\frac{4\pi}{L}, \pm\frac{6\pi}{L}, \dots \quad i = x, y, z. \quad (5)$$

In other words, quantization leads to allowed wavevectors $\mathbf{k} = (k_x, k_y, k_z)$ lying on a Cartesian grid in a three dimensional “ k -space” with the spacing between grid points in all three directions given by $2\pi/L$. Because the energy $E(\mathbf{k})$ depends only on the magnitude of \mathbf{k} , it is constant on the surface of a sphere in k -space.

Electrons are fermions and obey the Pauli exclusion principle. Hence each of the allowed wavevectors in the grid can be occupied by at most two electrons, one spin up and one spin down. The ground state for all electrons (lowest energy state occupied at zero temperature) is then found by filling up the allowed k -space grid, two electrons per point, starting from the lowest energies (smallest k) and working up to higher energies (larger k) until all the free electrons are assigned. Thus the ground state has two electrons at all k -space grid points inside a sphere—called the Fermi sphere—of some radius k_F —called the Fermi radius or Fermi wavevector.

From Eq. 5, the volume of each allowed state in k -space is $(2\pi/L)^3$. Thus the total number of states N in a Fermi sphere of volume $4\pi k_F^3/3$ is

$$N = 2 \cdot \frac{4\pi k_F^3/3}{(2\pi/L)^3} = \frac{V}{3\pi^2} k_F^3, \quad (6)$$

where the factor of 2 comes from the spin degeneracy. Note that N should be the same as the total number of electrons in the solid, which is related to the number density n by $N = nV$. Using this in Eq. 6 allows the Fermi radius k_F to be determined in terms of a fundamental property, the free electron density.

$$k_F = (3\pi^2 n)^{\frac{1}{3}}. \quad (7)$$

The surface of the Fermi sphere is called the Fermi surface and is where the most energetic electrons in the ground state lie. Their kinetic energy E_F is called the Fermi energy and is given by

$$E_F = \frac{\hbar^2 k_F^2}{2m}. \quad (8)$$

Exercise 1 *Copper has one free electron per atom. The density and atomic weight of copper are 8.96 g/cm³ and 63.55 g/mole, respectively. Avogadro's number is 6.02×10^{23} atoms/mole, $\hbar = 1.05 \times 10^{-34}$ Js, $m = 9.1 \times 10^{-31}$ kg, and 1 eV = 1.6×10^{-19} J. (a) Use this information to show that $n = 8.5 \times 10^{22}$ electrons/cm³, that $k_F = 1.4 \times 10^8$ cm⁻¹, that the deBroglie wavelength $\lambda = 4.5$ Å, and that $E_F = 7.0$ eV. One may also define a Fermi velocity via $v_F = \hbar k_F/m$. Show that copper has a Fermi velocity of 1.6×10^8 cm/s, about 0.5% of the speed of light. (b) Suppose an electron on this Fermi surface is moving in the x -direction, i.e. $k_x = 1.4 \times 10^8$ cm⁻¹. Calculate the difference between its energy level and the next higher energy level (one grid point over in the k_x -direction) in eV and as a fraction of the Fermi energy. Take the sample to be 1 cm in size and be careful that the subtraction of two nearly equal energies that only differ in a high order digit is performed with sufficient precision.*

As part (b) of this exercise demonstrates for energy, the very fine spacing of the quantum states relative to the highest occupied states implies that the wavevectors, momenta, and energies can often be regarded as continuous.

The free electrons in the metal have charge and will be accelerated by an external electric field, \mathbf{E} . Applying Newton's second law, we can write

$$\mathbf{F} = \frac{d\mathbf{p}}{dt} = -e\mathbf{E}. \quad (9)$$

Consequently, in the absence of collisions, the electron velocities change at a uniform rate

$d\mathbf{v}/dt = (1/m)d\mathbf{p}/dt = -(e/m)\mathbf{E}$ so that after a time t they are all shifted by the amount

$$\delta\mathbf{v} = t \frac{d\mathbf{v}}{dt} = -\frac{e}{m}\mathbf{E}t. \quad (10)$$

Collisions with impurities, surfaces, and lattice vibrations (phonons) will stop the acceleration, relaxing the electrons to equilibrium. If the mean collision time is τ , the average velocity of each electron—the *drift velocity* \mathbf{v}_d —becomes

$$\mathbf{v}_d = -\frac{e}{m}\mathbf{E}\tau \quad (11)$$

The electric current density \mathbf{j} is the charge density times the average velocity

$$\mathbf{j} = n(-e)\mathbf{v}_d = \frac{ne^2\tau}{m}\mathbf{E}. \quad (12)$$

This gives Ohm's law, $\mathbf{j} = \sigma\mathbf{E}$, with the conductivity σ given by

$$\sigma = \frac{ne^2\tau}{m}. \quad (13)$$

Exercise 2 (a) *The resistivity is the inverse of the conductivity: $\rho = 1/\sigma$ and for pure copper at room temperature $\rho = 1.68 \mu\Omega\text{-cm}$. Use this value to calculate the room temperature collision time τ for copper. (b) The power P absorbed by each accelerating electron is $\mathbf{F} \cdot \mathbf{v}$ and thus has an average value $P = |eEv_d|$ which must continuously be dissipated as Joule heat. Show that this leads to a power dissipation per unit volume (for all electrons) given by $p = E^2/\rho$. Show that the magnitude of the electric field that would require (the fairly significant) power dissipation of $p = 1$ kW/cm³ is around 5 V/m. (c) At this electric field, what would the drift velocity be? Also express v_d as a fraction the Fermi velocity. (d) The Pauli exclusion principle assures that only electrons near the Fermi surface participate in scattering events (and affect the conductivity) because electrons inside the Fermi surface have*

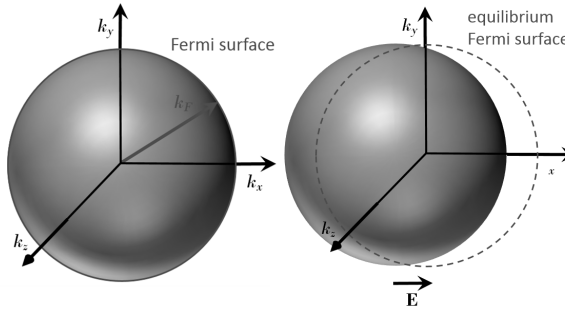


Figure 2: Fermi surface of a free-electron metal in the absence (left) and presence (right) of an electric field.

no nearby unoccupied states to scatter into. Part (c) demonstrates that the electrons acquire a very small average drift velocity in the direction of the electric field. However, during one collision time τ electrons near the Fermi surface travel through the metal without a velocity changing collision and thus the average distance they move between collisions—called the mean free path ℓ —is $\ell = v_F \tau$. Calculate ℓ for copper. Approximately how many copper atoms does an electron pass between collisions?

In the absence of an electric field, there is no current and electrons fill the Fermi sphere. For every electron with one wavevector \mathbf{k} there is another with the opposite value $-\mathbf{k}$. The two move in opposite directions and the net current due to each pair is zero. This is illustrated on the left in Fig. 2 where the Fermi sphere is centered at the origin. In an electric field \mathbf{E} in the $+x$ -direction, the electrons move with an average drift velocity \mathbf{v}_d in the $-x$ -direction and thus the Fermi sphere is maintained at a finite displacement $\delta\mathbf{k} = m\mathbf{v}_d/\hbar$. This is illustrated on the right in Fig. 2 where the Fermi sphere (solid circle) is centered off to the left. The magnitude of δk is greatly exaggerated in the figure for clarity.

For the shifted sphere, one might argue that every electron wavevector is shifted by the small amount $\delta\mathbf{k}$ and each contributes to the current producing an overall current density $\mathbf{j} = -ne\mathbf{v}_d$. Keeping in mind the magnitude of the shift $\delta k \ll k_F$, one might also argue that the large majority of electrons may still be considered to occur in pairs with opposite wavevectors. As for the unshifted sphere, each such pair would contribute nothing to the current. As the figure shows, only electrons in the shaded crescent (really just a sliver very near the original, centered Fermi surface) do not have an oppositely moving counterpart. Thus, one might then argue that only these faster moving electrons near the Fermi surface contribute to the net current. It turns out this latter view is more accurate. The number density of such electrons is much lower ($\approx nv_d/v_F$) but because they move much faster ($\approx v_F$), the current density is the same.

Scattering rates

The resistivity ρ and conductivity σ are inverses $\rho = 1/\sigma$ and thus according to Eq. 13 we can express the resistivity as

$$\rho = \frac{m}{ne^2\tau} \quad (14)$$

with the interpretation that τ is some mean collision time. This same interpretation means $1/\tau$ is an average collision or scattering rate. If $\tau = 10^{-14}$ s, then the electron undergoes $1/\tau = 10^{14}$ collisions per second, i.e., that the scattering rate is $10^{14}/\text{s}$.

There are basically two scattering mechanisms. Scattering by impurities, lattice faults, the surface, or other defects is expected to be temperature-independent with a rate $1/\tau_i$ that depends on how the sample was made. This scattering rate can vary greatly between samples of the same material made in different labs

or in different ways. Scattering by lattice vibrations (phonons) is temperature dependent and the rate $1/\tau_{\text{ph}}$ depends on the intrinsic properties of the metal and does not vary significantly among different samples. The two scattering mechanisms are independent and their rates are thus additive

$$\frac{1}{\tau} = \frac{1}{\tau_i} + \frac{1}{\tau_{\text{ph}}} \quad (15)$$

Substituting this in Eq. 14 implies the resistivity can be decomposed into two terms: a temperature independent part ρ_i due to scattering by impurities, etc., and a temperature dependent part $\rho_{\text{ph}}(T)$ due to scattering by phonons:

$$\rho(T) = \rho_i + \rho_{\text{ph}}(T). \quad (16)$$

This decomposition is known as Matthiessen's rule.

At high temperatures (above the Debye temperature) the number of phonons per unit volume is proportional to the temperature and ρ_{ph} is proportional to T . At low temperatures, two effects come into play. The number of phonons falls as T^3 ; moreover, the energy and momentum of these phonons become small, so that they are ineffective in scattering electrons. Consequently the resistivity falls more quickly than the phonon density. Bloch showed that

$$\rho_{\text{ph}}(T) \propto \begin{cases} T & T \leq \Theta_D \\ T^n & T \ll \Theta_D \end{cases} \quad (17)$$

where $\Theta_D \approx 343$ K is the Debye temperature for copper and n is theoretically $n = 5$ but empirically is in the range 3–5.

At the lowest temperatures ρ_{ph} goes to zero and the overall resistivity reduces to ρ_i ; consequently ρ_i is called the residual resistivity.

Semiconductors

In pure semiconductors the highest occupied band—the valence band—is completely filled

and there is an energy gap between this and the lowest unoccupied band—the conduction band. Thus at low temperature there are almost no mobile carriers and the resistivity approaches infinite values.

Semiconductor devices are made possible because semiconductors can be *doped* with donors or acceptors to control the electrical properties. Donors are typically atoms with one more valence electron than the semiconductor whereas acceptors have one less. Examples of donors are pentavalent impurities in Ge or Si, such as P, As, and Sb; examples of acceptors are trivalent atoms, such as B, Ga, and In. In general, donor levels lie close to the edge of the conduction band and acceptor levels lie close to the edge of the valence band; the carrier density is equal to the donor or acceptor density.

Donor-doped materials conduct via electrons in the conduction band. The materials are called *n*-type because the electrons have negative charge. Acceptor-doped materials conduct via “holes” (an empty electron state) in the valence band. The materials are called *p*-type because the holes have positive charge.

The simplest semiconductor device is the *p-n* junction diode. The diode energy band diagram is shown in Fig. 10 (located at the end of this writeup) for the unbiased, forward-biased, and reverse-biased cases. (If you want to play with the parameters in a *p-n* junction take a look at www.acsu.buffalo.edu/~wie/applet/pnformation/pnformation.html.)

One side of the crystal is doped *p* type, the other *n* type. A thin (\sim few μm thick) junction separates these two sides. Away from the junction region on the *p* side are negatively-charged acceptor ions and an equal number of free holes. On the *n* side are positively-charged donor ions and an equal number of mobile electrons. In addition both sides have

a small number of thermally generated “minority” carriers of the other type (holes in the n region and electrons in the p region).

Both holes and electrons tend to diffuse through the crystal. Electrons from the n region diffuse across the junction and recombine with holes in the p region (and vice versa). These recombinations leave the n -region depleted of electron carriers and positively charged and they leave the p -region depleted of hole carriers and negatively charged. The recombinations occur over a thin *depletion layer* around the junction. This charged, double layer grows until the electric field it produces is strong enough to inhibit any further diffusion of electrons across the junction.

The inhibition is not complete (at least at finite temperatures) and the most energetic electrons and holes (those in the tails of the Boltzmann distribution function) can cross the barrier. At zero applied voltage, the current due to electrons diffusing into the p region is canceled by the minority electrons diffusing in the opposite direction. Similarly, there is a cancellation between the currents due to holes and minority holes.

A voltage applied across the junction can either increase or decrease the height of the potential barrier. If the applied voltage is such that the p region is positive (the p region is connected to the positive electrode of a battery and the n region to the negative electrode), the junction is said to be biased in the forward direction. If the voltage is applied in the opposite direction, the junction is said to be biased in the reverse direction. When the junction is biased in the forward direction, the barrier height is reduced and the current increases rapidly. In contrast, a reverse bias increases the barrier height and produces only a small reverse current up to a saturation limit $-I_0$ set by the thermal generation of minority carriers. To create a minority carrier (hole) in

the n -region, an electron must overcome the band gap energy E_g (in silicon about 1.2 eV) via its thermal energy and governing Boltzmann distribution. Consequently I_0 is very temperature dependent and given by

$$I_0 = I_{00}e^{-E_g/kT} \quad (18)$$

where k is Boltzmann’s constant and T is the temperature.

The current I as a function of applied bias voltage V is predicted by the *Shockley diode equation*.

$$I = I_0 (e^{eV/kT} - 1) \quad (19)$$

where I_0 is called the reverse saturation current because $I = -I_0$ when the diode is reverse biased ($V < 0$) sufficiently that $e^{eV/kT} \ll 1$.

This model assumes all of the current arises from recombination in the depletion region. A somewhat more detailed analysis of recombination and other conduction processes leads to a modification of the formulas for both I_0 and I placing an ideality factor η in the denominators of both exponentials

$$I_0 = I_{00}e^{-E_g/\eta kT} \quad (20)$$

$$I = I_0 (e^{eV/\eta kT} - 1) \quad (21)$$

The ideality factor depends on the details of the diode fabrication and typically varies between 1 and 2. It may also vary with voltage. Even with the ideality factor, there can be deviations from the predictions of Eqs. 20 and 21. For example, real diodes have a series resistance R_s , typically on the order of an ohm or so, which would imply that the V in Eq. 21 should be replaced by $V - IR_s$. Real diodes also have a large parallel resistance, typically over 10 k Ω , which adds a small additional ohmic current to the measurements. In addition, other details of the diode current-voltage-temperature dependences can become important, particularly at very low temperatures.

Apparatus

You will make three measurements at temperatures between about 10 and 325 K. There is an option of making a fourth measurement.

1. the resistance of a thin copper wire about 1 m long;
2. forward voltage of a 1N914 silicon diode at several fixed currents;
3. IV characteristics of the diode at several temperatures.
4. (Optional) the resistance of a thin gold film;

Refrigerator and temperature controller

The cold temperatures are achieved using a closed cycle helium refrigerator called “the cooling machine” in this writeup. The principles of operation are briefly discussed next.

The metal cylinder at the top half of the helium refrigerator is the *vacuum shroud*. At the base of the shroud and inside it, is the *expander* consisting of valves and a sealed column. The samples are mounted on a copper block attached to the top of the column. High-pressure helium expands inside the column thereby cooling the column and the samples. The helium never enters the volume between the shroud and the column. It stays inaccessible inside the column before returning to the *compressor* (blue box under the lab table) where it heats up as it is compressed back to high pressure. The hot, high-pressure helium is cooled in a heat exchanger by water from the *Cool-Pak* (white box under the lab table) before going back to the expander.

The vacuum shroud is exposed on its outside surface to the room and stays near room temperature while the very cold column is only a few centimeters away. Any gas in the volume

between the shroud and the column would create a heat conduction path between them and prevent the column from reaching the low temperatures needed. That is why the air in this volume must be pumped away with a vacuum pump. In addition, there is a separate *radiation shield* between the vacuum shroud and the column to reduce radiative heating of the column by infrared rays from the room temperature vacuum shroud. There are ports in the shroud and cutouts in the shield near the sample area to allow laser beams, for example, to illuminate the samples. We do not use this capability. In fact, the cutouts on the radiation shield should be wrapped with aluminum foil to get a small reduction in the radiative heating and thus a small reduction in the lowest achievable sample temperature.

There are no user adjustments for the cooling accomplished by the helium expansion. When the compressor switch is turned on, all components of the cooling machine (expander, compressor, and Cool-Pak) begin operating. A *heater* (a current-carrying wire wrapped around the outside of the expander column) is used to adjust the sample temperature. With the heater turned off, it takes the cooling machine about 60 minutes to reduce the temperature from room temperature to the minimum achievable temperature (around 7 K). You should plan on making measurements of the copper wire during one day of experimenting and measurements of the diode on a second day. The optional measurement of the gold film could be done on the next-to-last day or last day if time permits.

Temperature Control

A LakeShore Model 330 Temperature Controller is used to measure the temperature and control it using the heater. It has two silicon diode thermometers.

Diode A is on the sample holder and measures the “sample” temperature.

Diode B is at the top of the expander column just below the sample holder and measures the “control” temperature.

The temperature controller can be controlled remotely through a GPIB interface or locally using the keypad. The controller will automatically switch from local to remote mode any time the computer sends a command over the GPIB interface, but you must place it in local mode using the top right **Local** keypad button to use the keypad. An indicator in the upper LED display shows **REM** (remote) or **LOC** (local) to indicate the status.

Because it is easy to make mistakes using the keypad, it is recommended that you use the **Control setpoint and ramp** program to change the setpoint. (More on this in a moment.) If, using the keypad, you get into any settings you do not understand or do not want to change, hit the **Escape** key. If you think you may have changed some parameters and need to get back to the “power on settings” (the settings before you changed them), simply turn off the LakeShore 330 controller (not the cooling machine), wait a few seconds and turn it back on.

You ask for a new temperature by changing the *setpoint*. The heater power then changes and then the controller waits to see how the (control) temperature changes. The temperature can overshoot its mark at which point the heater power changes in the other direction, etc. The PID parameters (for proportional, integral, and derivative) help determine how the feedback loop between the thermometer and the heater operates. In addition to the PID parameters, there are three **Heater Range** or maximum power settings: (Low, Med, High) corresponding to (50, 5, 0.5) W of power.

Because the thermal properties change as

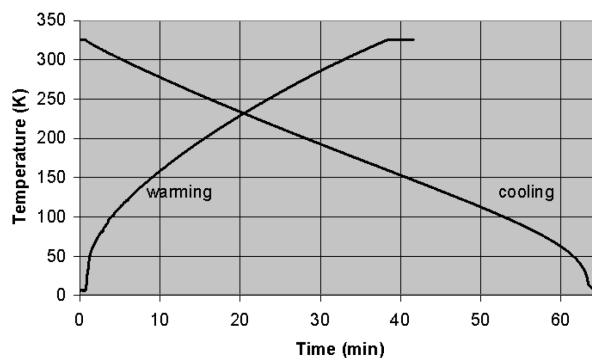


Figure 3: Temperature changes with cooling machine on and heater on at maximum power (warming) or off (cooling).

the temperature changes, the PID parameters and heater range need to be set differently in different temperature zones. These settings have been determined empirically and can be changed if you notice that any setpoint temperature causes large oscillates or takes too long to reach. Consult with the instructor on the proper procedure for doing this.

Changing the Temperature

The lowest reachable (base) temperature is around 7 K while the highest reachable temperature is around 325 K. With the heater off, it takes about one hour to get from the highest to the lowest temperature. With the heater on maximum power, it takes about 35 minutes to get from the lowest to the highest. Fig. 3 shows graphs of temperature vs. time for these two cases and demonstrates the rapid temperature changes that occur below 100 K.²

Some of the measurements will be made versus temperature at 1 K increments while the temperature is made to fall smoothly in time using the ramping feature of the controller.

²The change is largely due to a decrease in the heat capacity of the components at the lower temperatures.

The ramp causes any change in the setpoint to be performed automatically at a uniform rate that can be set from 0.1 to 99.9 K/min. Setting the ramp rate to 0.0 make the setpoint change immediately.

Keep in mind that ramping the setpoint is not necessarily the same as ramping the temperature. The controller still simply compares the setpoint and control temperature and adjusts the heater as it tries to get them equal. Thus, the temperature can still overshoot and oscillate due to the controller's feedback system. More importantly, the maximum rate of temperature change is still limited by the heating and cooling powers available, i.e., Fig. 3.

Measurements and LabVIEW programs

A LabVIEW program is called a vi (pronounce vee-eye for **v**irtual **i**nstrument). If you make changes to any of the vis used in this laboratory, you would use the **Save As** item in the File menu of that vi, and save a copy to your own area in the *My Documents* folder. All **Save** items in the File menu save the vi; **THEY WILL NOT SAVE DATA**.

Every vi that collects data in this experiment will ask you to provide a file name for storing any resulting data. Work your way to the *My Documents* folder (you might want to create a subfolder therein) click into it, and change the default file name (typically **DATA.TXT**) to something more descriptive. Record the filename in your lab notebook. If this step fails, the vi terminates without taking any data. If it succeeds, the vi will save the data to this file when data collection is complete and you click on the red-letter **STOP** button near the graph to stop the vi. Most other ways of stopping the vi will prevent the data saving step from executing and the collected data will be lost.

A vi is run by clicking on the run button

(right-pointing thick white arrow) in the tool bar. This arrow turns black while the vi runs and back to white when the vi stops running.

Instructions for changing the temperature

1. Open the **Control setpoint and ramp rate** vi. Click on the **Run** button in the tool bar. This vi reads and displays the sample temperature, the control temperature, the setpoint, and the ramp rate when you click on the **Read** button. It sets the setpoint and ramp rate when you click on the **Write** button.
2. **Changing the setpoint immediately:** If you just want to get to the desired temperature as fast as possible, set the **Setpoint** control to the desired value, set the **Ramp rate** control to 0.0, and click on the **Write** button.
3. **Ramping the setpoint:** First **Read** the current setpoint and if it is not where you want the temperature ramp to start (typically the current control temperature) change the setpoint immediately as per the prior instruction. If necessary, wait for the temperature to reach the starting setpoint. Then change the **Setpoint** control to where you want it to end, set the **Ramp rate** control to the desired rate, and then click on the **Write** button.
4. You can leave this vi running as it uses very little computer resources, or **STOP** it until you need it again.

Shutting down

Returning the apparatus to room temperature with all power turned off starts by shutting off the cooling machine. With no heating power applied, the column can take several hours to

return to room temperature. The time can be reduced by using the heater. The heater will work even with the cooling machine off by simply adjusting the setpoint to room temperature or slightly above. Starting from the lowest temperatures, with the heater on and the cooling machine off, the sample area and both thermistors will rise to room temperature even more quickly than the forty minutes shown in Fig. 3.

However, thermistor readings above room temperature do not mean the entire column has warmed to room temperature. Most of the column can still be very cold at this point. If exposed to room air while still cold, the column will frost over as the water in the humid room air freezes on the surface. Do not expose the column to room air until its whole length is at room temperature and it is not easy to determine when this happens.

If the column is still cold, and the heater power is then turned off, the thermistor temperature would start falling again. To turn off the heater, simply adjust the setpoint well below room temperature. If it does not fall and stays above 300 K for at least 10 minutes after shutting off the heater, the column is at room temperature and can be opened to the room air.

Measurement of resistance

The resistance is measured by a Keithley 2700 digital multimeter in Ohms-4 wire mode. This “4-wire” method allows the resistance to be measured without the parasitic influence of the resistance of the wires between the meter and the sample and of contact resistance between the wires and the sample.

The 4-wire method works as shown in Fig. 4. The film is patterned with potential “sidearms” and the resistance measured is the resistance of the part of the sample between

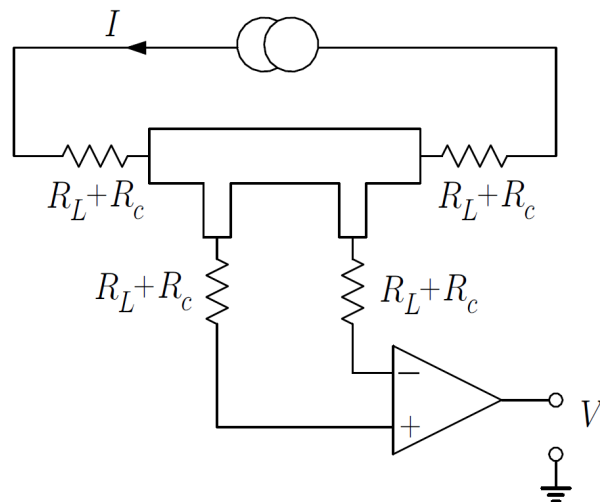


Figure 4: 4-wire method of measuring resistance. In the diagram, the “ohms-source” connection on the digital multimeter is represented as a current source while the “ohms-sense” connection is represented as a voltage amplifier. The multimeter takes the ratio of voltage to current to compute the resistance. R_L represents the resistance of the electrical leads, including the cables to the vacuum feedthrough (small) and the wires leading to the cold tip (not so small). R_c represents the contact resistance between the wires and the specimen (sometimes small, sometimes not so small).

these sidearms.

Exercise 3 Suppose for all 4 wires going to the sample, the sum of lead and contact resistance is $500\ \Omega$. Suppose further the sample resistance is $10\ \Omega$. What is the minimum input impedance of the voltage amplifier for the measured resistance to be no more than $1\ \text{m}\Omega$ from the true resistance? Hint: The measured resistance is the voltage across the amplifier inputs divided by the current I supplied by the source.

The temperature-dependent resistance is

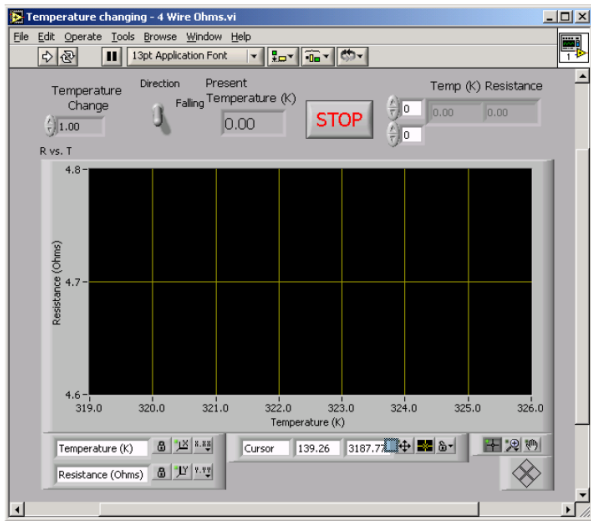


Figure 5: The Temperature changing - 4 wire ohms vi.

measured with the Temperature changing - 4 wire ohms vi. A picture of the front panel is shown in Fig. 5.

This vi uses the Keithley 2700 to measure a resistance (in Ohms-4 wire mode) every time the temperature decreases by the amount shown in Temperature change, set to 1.0 K by default.

Temperature dependence of a diode's forward junction voltage

When used as a thermometer, a constant current is passed through the silicon diodes and the voltage is measured. As described previously, the voltage required for a given current varies significantly with temperature and here, you will study this behavior for a common silicon diode (rather than those used by the temperature controller which are specially manufactured for use as a thermometer). At typical currents of 1 to 10 μA , the diode has a high resistance so that a 4-wire arrangement is not necessary. Instead the “2-wire” measurement circuit shown in Fig. 6 will be used.

The Keithley 224 current source delivers the current and the Keithley 2700 digital multi-meter measures the voltage. The Keithley 224 output is from a triax connector on the back panel, not the the banana jacks. A triax to BNC adapter cable is used to facilitate the connections.

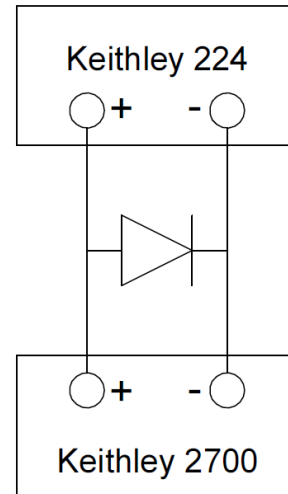


Figure 6: Circuit diagram for the diode measurements.

The temperature-dependent diode voltage is measured at fixed currents using the Tem-

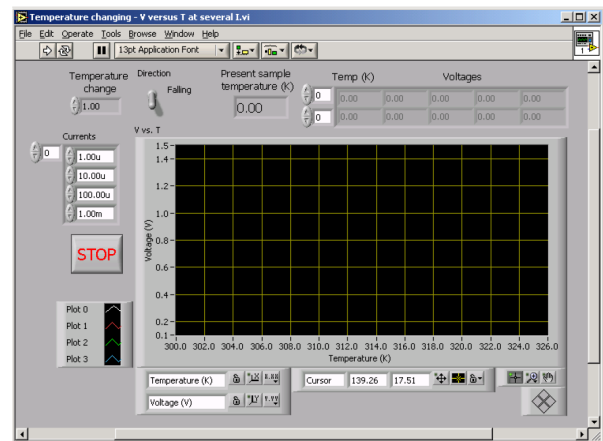


Figure 7: The Temperature changing - V versus T at several I vi.

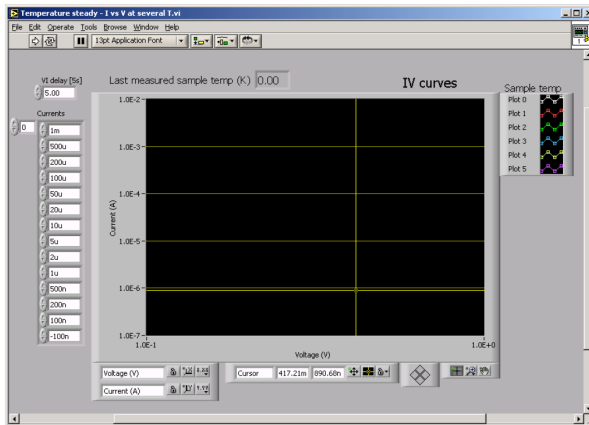


Figure 8: The Temperature steady - I versus V at several T vi.

perature changing - V versus T at several I vi (see Fig. 7). This vi also makes measurements every time the temperature decreases by the amount shown in **Temperature change**, set to 1.0 K by default. It first uses the Keithley 224 to set the current at one of the values displayed in the **Current array** control on the front panel and then uses the Keithley 2700 to measure the diode voltage. This is done sequentially for all currents in the array. Because it takes a few seconds to cycle through the measurements, the temperature will vary a few tenths of a Kelvin during the measurements, but this should not be a problem.

Diode current-voltage characteristic

More detailed current-voltage characteristics for the diode will be measured while the temperature is held steady. The front panel of the **Steady temperature - I versus V at several T** vi used to make these measurements is shown in Fig. 8. The sample temperatures are set interactively so you can't run this vi and then leave and come back later. A reasonable set of IV curves might be taken at temperatures around 10, 50, 100, 150, 200, and 300 K. If you

are at the base temperature when you start, work up from 8 K. If you start from room temperature, work down from 300 K.

As is traditional for IV curves, they are plotted as I vs. V even though the data is taken with I as the independent variable. At each temperature, V is measured by the Keithley 2700 after each of the **Current array** control values on the front panel has been applied using the Keithley 224. The default values from 0.1 to 1000 μA in a 1, 2, 5 sequence (plus one reverse bias measurement at $-0.1 \mu\text{A}$) works well.

Self heating

A power IV is delivered to any device carrying a current I through a voltage drop V . For the diode, copper wire (and gold film), the energy is released as heat and may lead to self heating and a device temperature not in equilibrium with the sample diode used to measure the temperature. While this is probably not a problem for most measurements, it may have an effect at the lowest temperatures and highest current for the diode where the power dissipation gets to about 1.5 mW or so.

A sub-vi is used to set the currents and measure the corresponding voltage for each current in an array of values. This sub-vi can be made to turn off the current for a user-selected time delay between each array element. (It always reads the voltage immediately after setting the current.) This parameter is called the **IV delay** on the front panels of the two IV data acquisition programs.

If the device temperature does not rise too quickly in response to an excitation current, this feature may successfully allow the device to re-equilibrate with the sample diode thermometer before each IV measurement. Setting the **IV delay** to zero turns it off and causes each new current in the array to be set imme-

diately after the prior array value without first setting it to zero.

Whether or not the IV delay is used, the current is always returned to zero after processing all currents in the array.

Procedure

4-wire versus 2-wire resistance

Here you will use two of the “Ohms-ranger” boxes to simulate the effects of lead and contact resistance.

1. Set one of the boxes to $R_1 = 10\ \Omega$ and the other to $R_2 = 1000\ \Omega$. Use the Keithley digital multimeter in $\Omega 2$ mode to determine the actual resistance of the boxes.
2. Connect the two boxes in series, and measure the series resistance in $\Omega 2$ mode. Convince yourself that the resistance is the sum of the two by setting R_1 to $11\ \Omega$ and $9\ \Omega$.
3. Connect the ohms sense terminals across R_1 and measure the resistance in $\Omega 4$ mode. Demonstrate that the resistance is that of R_1 by setting it to $11\ \Omega$ and $9\ \Omega$.
4. Demonstrate that the resistance reading is insensitive to R_2 by setting it to $1100\ \Omega$ and $900\ \Omega$.

C.Q. 1 *What is the maximum value of R_2 for which the resistance of $R_1 = 10\ \Omega$ is measured correctly? The minimum? Explain. For an ideal circuit, what do you expect for the maximum R_2 ? How would it depend on the actual resistance of R_1 ?*

Measurement of dc resistance

The circuit diagram of the wires to the sample area is shown in Fig. 9.

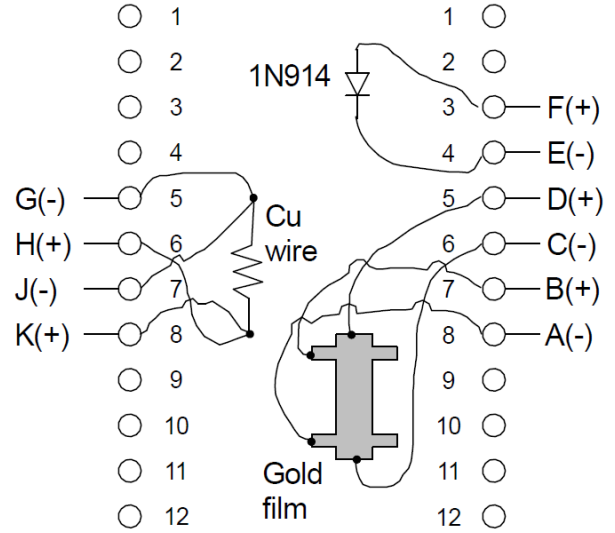


Figure 9: Connections to the samples.

5. Remove the outer vacuum shroud. (You will need to vent the vacuum system if it is not already at atmospheric pressure.) Unscrew the radiation shield. (Wear gloves to prevent fingerprints!) Note the copper wire wound around a small brass screw on the upper left. (In case you have time to measure the gold film, use a small ruler to estimate the width of the thin gold film, its overall length and the separation between its voltage sidearms. **Be careful not to touch the wires fixed to the film!!** It is only necessary to get an estimate of these geometric factors to within about 25%.)
6. Connect first the current leads (H+ G-) to the digital multimeter and next the voltage leads (K+ J-). Measure the resistance (2-wire) for each connection. Now connect both sets for 4-wire resistance and measure this quantity. These measurements will allow you to determine the room-temperature values of the (lead + contact) resistances shown in Fig. 4.

For a circular cross section, the relation between resistance R and resistivity ρ is

$$R = \frac{\rho L}{\pi r^2} \quad (22)$$

where L is the sample length and r is its radius. Thus, for a wire of a known substance, one may estimate the thickness from the resistance and measured diameter.

C.Q. 2 Assume the copper wire has the resistivity of pure copper ($1.68 \mu\Omega\text{-cm}$) and is $5.1 \times 10^{-3} \text{ cm}$ in diameter. Use your measurements of the 4-wire resistance to estimate the length of the wire.

7. Connect the digital multimeter to the 1N914 diode leads (F+ E−) and measure the resistance. Now reverse the leads and remeasure the resistance. What does this measurement tell you about the diode? About the multimeter's circuit for measuring resistance?
8. Screw on the radiation shield. (Use gloves.)
9. Check the O-rings on the expander for dirt. (Remove any you see. You may want to add a *small* amount of vacuum grease.) Gently slide the vacuum shroud over the O-rings.
10. Check that the vent valve is closed.
11. Start the vacuum pump and then open the vacuum valve. The thermocouple gauge should start to indicate vacuum after a few minutes.
12. Turn on the temperature controller, the digital multimeter, and the computer. After the pressure goes below 50 mTorr, turn on the cooling machine and do an immediate change of the setpoint to 324.9 K by following the appropriate procedure in *Changing the Temperature* starting on page 8. The LED bar on the temperature controller should show that the heater comes on. If not, ask for help.
13. Connect the current leads (H+ G−) and the voltage leads (K+ J−) to the digital multimeter as required for 4-wire resistance.
14. Do a Read from the Control setpoint and ramp vi, check that the setpoint is at 324.9 and, if necessary, wait for the control temperature to reach 324.9 K.
15. Bring up the Temperature changing - 4 wire ohms vi. Leave the switch above the graph set for Falling and the Temperature Change at 1 K. Start the vi by pressing the Run button in the toolbar.
16. The vi will ask for a file in which to store the data. Create a directory in MyDocuments, or better yet, on your personal thumb drive and save it there. Be sure to document the file name and its contents in your lab notebook.
17. Make sure the cooling machine has been running for at least a five minutes. Then ramp the temperature controller's setpoint to 4 K at 3.5 K/min,³ again following the procedure on *Changing the Temperature*. The sample temperature indicator above the graph should start to ramp down and a resistance measurement should be made and added to the graph for each degree it falls.

³The value of 3.5 K/min is near the fastest rate that can be maintained over the full range of temperatures for both increasing or decreasing ramps. The rate is limited by the smallest slopes of the curves in Fig. 3 which are both around 4 K/min.

18. If, at any point during the cool down, the shroud gets very cold (shows frost) there is either a leak or a mechanical contact between the cold section and the shroud. Stop the cooling machine and check with an instructor.
19. When $T \approx 220$ K, close the vacuum valve and turn off the vacuum pump.
20. The vi should continue to a temperature below 10 K. When the temperature stops changing, press the red-letter **STOP** button near the graph and the program will save the data to the file specified when you first started the program. The first column contains the sample temperatures and the second column contains the resistances.
21. Rather than getting one data point every degree or so as in the **Temperature changing - 4 wire ohms**, you will sometimes want to use the **Continuous - 4 wire ohms** program, which takes temperature and resistance readings as fast as it can until you tell it to stop by hitting the **Stop** button on the front panel. Remember to stop it this way to properly save your data to a spreadsheet file. The extra data is particularly important near the lowest temperatures, say from the minimum temperature achievable to around 30 K so you will get a large enough statistical sampling of temperatures and resistances for analysis. Start the program while the temperature is at its minimum and then do a slow upward ramp in temperature with the **Control setpoint and ramp** program. You will know you have good data when the graph of resistance vs. temperature shows lots of points throughout the desired temperature range and shows the scatter of the data as well as the general upward trend.

Analysis of resistance

Plot $R(T)$ —your measured resistance as a function of temperature. According to Eq. 16 (with Eqs. 17 and 22), the resistance is predicted to be

$$R_{\text{pred}}(T) = R_i + CT^\alpha \quad (23)$$

where

$$R_i = \rho_i \frac{L}{\pi r^2} \quad (24)$$

and $\alpha = 1$ at high temperatures and $\alpha = 5$ at low temperatures. The proportionality constants in Eq. 17 together with the scaling factor $L/\pi r^2$ becomes C in Eq. 23 and will be different in the two regions. Although Eq. 17 suggests that α should change from one to five right at $\Theta_D = 343$ K, these limiting power law behaviors should only be expected well above and well below $\Theta_D/5$.

Fit your data to Eq. 23 over the appropriate temperature range with R_i , C and α as the fitting parameters.⁴ For the low temperature region start by fitting only those points below a limiting temperature of 30 K. For the high temperature region start by fitting only those points above a limiting temperature of 275 K. Also plot the residuals $R(T) - R_{\text{pred}}(T)$ vs. T and report how the quality of the fit and the parameters change when you increase or decrease the limiting temperature.

The resistivity of $1.68 \mu\Omega\text{-cm}$ —given in Exercise 2 and used again in C.Q. 2—is actually the value for pure copper. That is, it is the intrinsic resistivity at room temperature and does not include any contribution from the residual resistivity ρ_i . Let $\rho_{\text{ph}}(297 \text{ K})$ ($= 1.68 \mu\Omega\text{-cm}$) represent this known room temperature intrinsic resistivity and let $R(297 \text{ K})$ represent your measured wire resistance at

⁴You may need to include a fixed multiplier with $C = \beta C'$ so that the fit is working with a value of C' near one.

room temperature. Show that the scale factor between resistance and resistivity $\kappa = L/wt$ can then be considered experimentally determined as

$$\kappa = \frac{R(297\text{ K}) - R_i}{\rho_{\text{ph}}(297\text{ K})} \quad (25)$$

Determine κ and use it with the wire diameter d to get an improved determination of the wire length. How much does it change from the value determined in C.Q. 2?

The ratio of the resistivity at the ice point (273 K) to the residual resistivity ρ_i is called the residual resistivity ratio. Keeping in mind that ρ_i arises from impurities and other crystal defects, higher residual resistivity ratios imply higher film quality. This ratio for a high quality copper single crystal can exceed 1000. Use your measurements to determine the ratio for your wire.

According to Eq. 23, a plot of $\log(R(T) - R_i)$ versus $\log(T)$ should be a straight line with a slope α . Make such a plot using the R_i determined by the low temperature fit. Due to experimental error, you may find some negative values for $R(T) - R_i$ at the lowest temperatures for which the log function will return an error. Just ignore these points. Can you see the two power law regions in your graph? Add a line through the high temperature data having a slope of one and two other lines through the low temperature data points with a slope of three and a slope of five. Change the value of R_i in very small increments watching how the results change in the low temperature region. What does this step together with the low temperature fits suggest about this experiment's ability to check the validity of the hypothetical T^5 behavior?

Diode measurements

22. Start the vacuum pump and then open the vacuum valve. The thermocouple

gauge should start to indicate vacuum after a few minutes.

23. Turn on the temperature controller, the digital multimeter, the current source, and the computer.
24. After the pressure goes below 50 mTorr, turn on the cooling machine and do an immediate change of the setpoint to 324.9 K by following the appropriate procedure in *Changing the Temperature* starting on page 8. The LED bar on the temperature controller should show that the heater comes on. If not, ask for help.
25. Connect the diode leads (F+ E-) to the current source and the digital multimeter. Manually adjust the current to 1 mA⁵ and check that the voltage is around 0.6 V. If it is not, check your connections; you may need to reverse the leads to the diode.
26. Do a Read from the Control setpoint and ramp program, check that the setpoint is at 324.9 and, if necessary, wait for the control temperature to reach 324.9 K.
27. Open the Temperature Changing- V versus T at several I program. If you want to change the currents at which the voltages are measured, you need to do this before starting the program. Leave the switch above the graph set for Falling and the Temperature change at 1 K. Start it by pressing the Run button in the toolbar.
28. The vi will ask for a file in which to store the data. Rename the file and save it

⁵Turn on the Keithley 224, push the SOURCE key, enter the desired current with the keypad, and push the ENTER key. Push the OPERATE key if its indicator LED is not lit. If the V-LIMIT led flashes, there is a problem—the 224 could not reach the desired current within the set voltage limit; check the circuit and/or ask for help.

to MyDocuments or a USB drive directory created earlier. Again, record the file name and its contents in your lab notebook.

29. Make sure the cooling machine has been running for at least a five minutes. Ramp the temperature controller's setpoint to 4 K at 3.5 K/min, again following the procedure on *Changing the Temperature*. The sample temperature (indicated above the graph) should start to ramp down and for each degree it falls, the program takes measurements of the diode voltage at each of the currents and adds them to the graph. Note in your lab notebook the temperature change during a single set of measurements at "one" temperature.
30. If, at any point during the cool down, the shroud gets very cold (shows frost) there is either a leak or a mechanical contact between the cold section and the shroud. Stop the cooling machine and check with an instructor.
31. When $T \approx 220$ K, close the vacuum valve and turn off the vacuum pump.
32. The vi should continue to a temperature below 10 K. When the temperature stops changing, press the red-letter STOP button near the graph and the program will save the data to the file specified when you first started the program. The first column contains the sample temperatures and each subsequent column contains the measured voltages for one of the chosen currents.
33. Start the Temperature steady - V versus I at several T program. The program will wait for you to set the temperature. Do an immediate change of the setpoint to the desired temperature according to

the *Changing the temperature* procedure and then wait for the temperature to stabilize before proceeding by pressing the READY button. The program then measures the IV characteristics as described previously. Due to a bug in the LabVIEW program, which makes the first current/voltage measurement unreliable, this first measurement is performed twice. Use the second one.

34. Repeat for all desired temperatures (10, 50, 100, 150, 200, and 300 K are recommended). Write down the control and sample temperatures in your lab notebook.
35. After the last set of IV measurements, stop the program using the red-letter QUIT button on top of the graph and the program will save your data to the file specified when you first started the program. The first column contains the set of currents used. Each additional column contains the voltages measured for these currents at one particular sample temperature which temperature will be at the head of the column.

CHECKPOINT: Complete all measurements of resistance and diode IV characteristics.

Analysis of diode measurements

The voltage-current relation is predicted to be

$$I = I_0(e^{eV/\eta kT} - 1). \quad (26)$$

where I_0 is temperature dependent and the ideality factor η is of order unity. Show that the -1 is negligible compared to $e^{eV/\eta kT}$ for all forward biased data points so that a plot of $\ln I$ vs. V at each temperature should be a

straight line over the region where the theory is applicable.

From your IV measurements at constant T , make a series of such plots on a single graph and discuss the agreement with the prediction. Fit a straight line to each plot over the appropriate region (where the plot is linear). Make a table and graph to show how the fitted slope and the fitted value of the intercept ($\ln(I_0)$) change with temperature. Discuss how (and why) the slope and $\ln(I_0)$ depend on temperature. Plot the slope vs. inverse temperature, show that it is linear and use the slope of this graph to determine e/k and compare with predictions. Should your fit be forced through the origin? Why or why not? Plot the intercept vs. $1/T$ to determine the band gap energy E_g .

From the V vs. T data at several I , make plots V vs. T for each current on a single graph. Fit each plot over the appropriate region to find the silicon band gap energy E_g and compare with predictions from the literature. Make a plot of the slope of the V vs. T graph vs. $\ln(I)$ and use its slope to again determine the ideality factor. Why do you have to exclude data at the lowest temperatures? Comment on the usefulness of the 1N914 as a thermometer. How does it compare with a DT-470 commercial diode thermometer used in our cryostat? (Data from the LakeShore manual is included with the auxiliary material for this experiment.) Is there measurable voltage noise at any current? How accurately should the current be controlled to achieve a temperature accuracy of $\pm 1\%$ at 100 K? at 10 K?