PHYSIKALISCHES PRAKTIKUM
FÜR VORGERÜCKTE DER ETH

ELECTRICAL CONDUCTIVITY

(ELEKTRISCHE LEITFÄHIGKEIT)

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N. W. Ashcroft and N. D. Merlin. Solid state physics.
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1. Theoretical Introduction[1]

According to Ohm's low, the current I flowing in a sample is proportional to the potential drop V along the sample: $V = IR$, where R is the resistance of the sample. The resistance depends on the shape and the size of the sample. To eliminate this dependence, one usually introduces a quantity characteristic only of the material of which the sample is made. The resistivity ρ is defined as the proportionality constant between the electric field E and the current density j

$$\rho = \frac{E}{j} = R\frac{A}{L}. \tag{1}$$

Here L is the length of the sample and A its cross-sectional area.

If n electrons per unit volume all move with velocity v, then the current density they will rise to can be written as

$$j = -nev, \tag{2}$$

where e is the electron charge. Minus in this equation reflects the negative charge of electrons.

At any point in a metal, electrons are always moving in a variety of directions. The net current density is thus given by the equation above where v is an average electronic velocity. In the absence of an electric field an average electronic velocity is zero and, as expected, there is no net electric current density. In the presence of a field E, however, there will be a mean electronic velocity directed opposite to the field (the electron charge being negative).

During the time t electrons, due to their acceleration by the electric field E, acquire an additional drift velocity $v_d = -\,eE\,t/m$ (m is the electron mass). This additional velocity is limited by electron scattering[2]. After the scattering, which can be a collision with impurity atom (or any other defect of the crystalline structure) or phonon, electron emerges with its velocity in a random direction. Therefore, to find an average drift velocity we should substitute t by an average time between the collisions, which is known as the relaxation time τ. It gives

$$j = \frac{ne^2\tau}{m}E. \tag{3}$$

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[1] Only very short and simple consideration of electrical conductivity is given here. Those who are interested in a more detailed and rigorous theory of the problem, can easily find many books devoted to this subject.

[2] There are different scattering processes which limit the electrical conductivity, but here we consider only scattering by static defects and phonons because these two kinds of scattering are the most important for our choice of samples and the range of temperatures.

The electrical conductivity σ then can be written

$$\sigma = \frac{1}{\rho} = \frac{ne^2\tau}{m}. \tag{4}$$

This expression for the electrical conductivity is known as Drude's formula.

The Drude's formula is about a hundred years old and it was obtained from a very simple consideration where electrons are just charged particles, which are moving inside the metal as they would do it in vacuum. However, exactly the same result can be obtain by a more rigorous approach where the Fermi distribution of electrons is taken into account. If the relaxation time τ is independent of the electrical field in the sample, it can be shown that application of an electrical field E along x-axis results in a displacement of the Fermi surface parallel to the x-direction in k-space by $eE\tau/\hbar$. In other words, in electrical field E all electrons have an additional drift velocity $v_d = -eE\tau/m$ in the direction of E.

Eq. (4) shows that the electric conductivity is limited by electron scattering. The relaxation time τ is an average time between two scattering processes. Taking into account that electrons in metallic samples are moving with the Fermi velocity $v_F \sim 10^6$ m/s, which is many orders of magnitude greater than v_d, we can find the mean free path of electrons $l = v_F\tau$.

Two scattering processes are most important for the electrical conductivity in simple metals: collisions with the sample defects and with thermal excitations (phonons) and we can introduce two different relaxation times. Let us denote by τ_{ph} the phonon scattering time. The collisions of electrons with static defects is essentially elastic and we use τ_e for the corresponding relaxation time. In this case

$$\frac{1}{\tau} = \frac{1}{\tau_e} + \frac{1}{\tau_{ph}}, \tag{5}$$

which leads to Matthiessen's rule for the total resistivity

$$\rho = \rho_e + \rho_{ph}. \tag{6}$$

This is not an exact result, but the observed deviations are usually small and it serves as a useful principle in practice. There are different kinds of defects in metallic samples: impurity atoms, defects of the crystalline lattice, etc. Important is that the number of defects does not depend on temperature and only the phonon scattering makes the resistivity temperature dependent. Since the temperature dependence is contained in ρ_{ph}, the derivative $d\rho/dT$ should be independent of the impurity concentration. This is true, however, only when amount of impurities is not too high. Increase of the impurity concentration changes the phonon spectrum of the sample and ρ_{ph}. That is why one should be careful applying the Matthiessen's rule for alloys.

2. What should be done:

1. Resistance of 2 samples (pure lead and alloy) should be measured as a function of temperature down to superconducting critical temperature T_c.
2. The resistance data should be converted into values of resistivity ρ.
3. The resistivity versus temperature should be plotted in 2 separate graphs for 2 samples.
4. Using your data you should evaluate the values of ρ and $d\rho/dT$ for 0˚C ($\approx$ 273 K). For Pb you should compare your results with the table values.
5. Compare the resistivity difference $\Delta\rho=\rho(273\,\mathrm{K})-\rho(10\,\mathrm{K})$ for 2 samples.

3. Samples and Electrical Measurements

Samples are made of pure lead (Pb) and lead based alloys. Each sample is a wire of a few meters length wound on a plastic core. The composition of the alloy is written on a label attached to the core. You should make measurements for at least 2 samples, one of which is pure lead and another is an alloy.

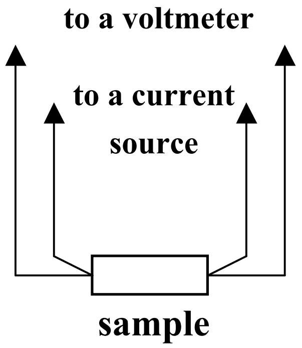

Fig. 1.

A 4-probes techniques is used to measure the sample resistance as it shown in Fig. 1. There are 2 pairs of wires soldered to the sample. One of them serves to put an electrical current I through the sample and the second one to measure a voltage V. The sample resistance in this case $R = V/I$. Important advantage of this method is that the resistance of connecting wires does not enter the result.

The current should be high enough to ensure sufficient accuracy of measurements, but, at the same time, it should not be too high not to overheat the sample by a Joule heat. You can use $I = 100$ mA for pure lead and $I = 50$ mA for alloys.

Important. The value of current produced by the current source must be checked at the beginning and at the end of each series of measurements. You should also check that the voltage on the sample is zero or negligibly small when the current is switched off.

The sample resistance should be measured at different temperatures from the room temperature down to 5-7 K. The temperature intervals between the points can be 20-25 K at higher temperatures, but they should be reduced to 1-2 K at the lowest temperatures. Note that all the samples are superconductors with superconducting critical temperatures T_c between 5 and 8 K and it is interesting to evaluate T_c for your samples.

In this experimental set-up the thermometer is fixed on the sample chamber (see description of the cryostat below) and the sample is thermally connected to this chamber only by the thermal conductivity of the helium gas. In this case establishment of the thermal equilibrium in the system takes some time. That is why you should wait a couple of minutes at each temperature before taking a point. If the sample resistance and the temperature are stable or slowly changing with time, it is an indication that you are near to the equilibrium. The resistance is a characteristic of the particular sample and you should recalculate the data into values of resistivity, which is a characteristic of the material. To do this, the length L and the diameter d of the sample should be measured (it can be done before or after the resistance measurements). The resistivity of the sample then may be found as $\rho = R\pi d^2/L$.

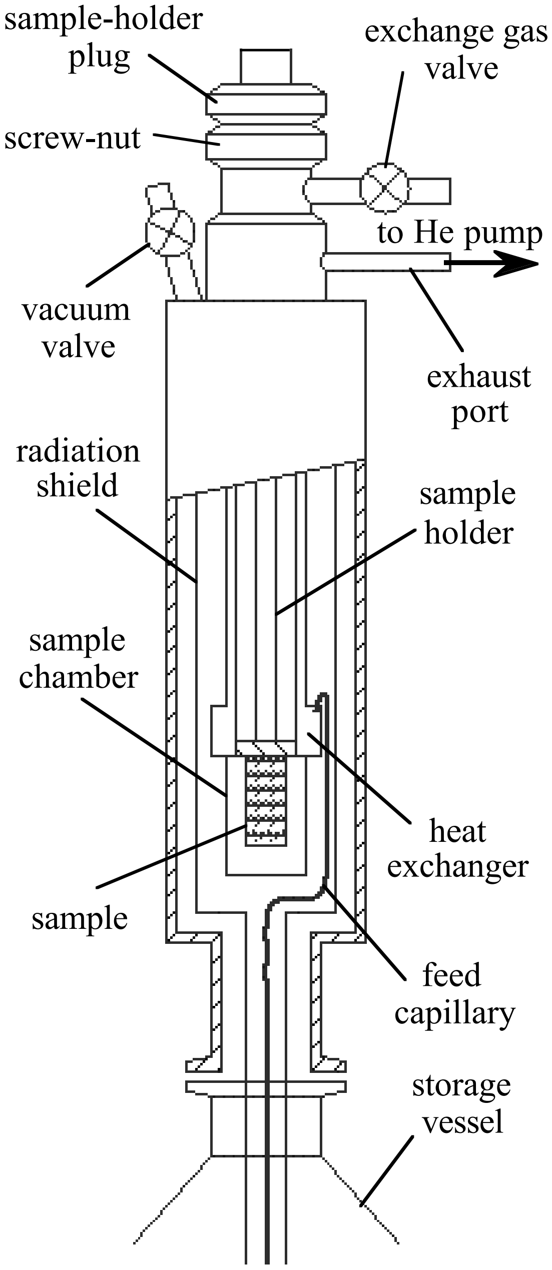

Fig. 2.

4. The Cryostat

4.1. Description of the Cryostat

A continuous flow cryostat is used to obtain temperatures between 4.2 K (temperature of liquid helium) and 300 K (room temperature). This is a simple and convenient device for this temperature range. It works on the principle of the controlled continuous transfer of a coolant (liquid helium) from a storage vessel to a vacuum insulated, radiation shielded, sample chamber. The sample temperature may be maintained at any temperature within the range of the cryostat by controlling the flow of the coolant and the power dissipated in the electrical heater attached to the sample chamber.

The main parts of the cryostat are shown in Fig. 2. The coolant flows from the storage vessel through the feed capillary and into the heat exchanger mounted on the sample chamber. It exhausts up through an annular space around the sample tube and it exits into the helium pumping line.

The coolant flow is produced by generating a pressure difference between the storage vessel and the helium exhaust line with the gas flow pump. The flow and pressure in the helium exhaust line are monitored on the flow controller (see Fig. 3). The temperature is also displayed on the same unit and is measured by a resistance thermometer attached to the sample chamber.

The sample space and outer vacuum case are evacuated through corresponding valves. You will use the exchange gas valve to evacuate the sample chamber and fill it with helium gas, but you should **never** touch the vacuum valve.

The sample is top-loaded and is cooled by a static column of helium exchange gas which thermally links the sample to the sample chamber. The sample holder is suspended by a thin walled stainless steel tube from a plug which is 'O' ring sealed to the sample access port. This 'O' ring allows simple and rapid change of the sample.

4.2. Sample Mounting

To change or install the sample you must unscrew the sample holder plug by a few turns and carefully pull it out of the cryostat. After the sample is mounted the sample holder can be fixed in the cryostat.

After this procedure the sample chamber is filled with air which should be evacuated and replaced by helium. You use a mechanical pump to pump air out via the exchange gas valve (see Fig. 1). Then you close the valve on the pumping line and fill the chamber with helium gas from a gas bottle. The helium pressure is normally adjusted to 760 torr (1 bar). Now you can close the valve and the cryostat is ready for measurements.

To change the sample after the measurements, you must first heat the sample up to at least 280 K. This step is important to avoid water condensation inside a cold sample chamber. You should **never** open the chamber if its temperature is below the freezing point. To heat up the sample chamber you should close the needle valve on the flow control unit (you also can switch off the He pump) and set the desired temperature equal to the room temperature. The heating process takes 30-40 minutes which you can use to have your lunch, for example.

4.3. Operating the Cryostat

Remove a protection shield from the helium transfer tube at the bottom of the cryostat and connect a corrugated rubber tube instead. Lift up the cryostat to its top position. Remove a lid from a storage vessel of liquid helium and move it into position just under the cryostat. Lower the cryostat slowly into the storage vessel and fix the bottom end of the rubber tube on the top of the vessel. Move slowly the cryostat further down into the vessel.

Now everything is ready for measurements and you can already take your first point corresponding to room temperature.

Switch on the helium pump. Open a little bit a needle valve on the flow control unit and adjust the helium gas flow to 50-100 cc/hour with the gas flow meter. Monitor the temperature. When the temperature will be near a desired value adjust the flow rate to give a temperature just below the required value. The temperature is then raised to the required value by the heater, which can be operated either manually or automatically by the temperature controller as it is described in the next section.

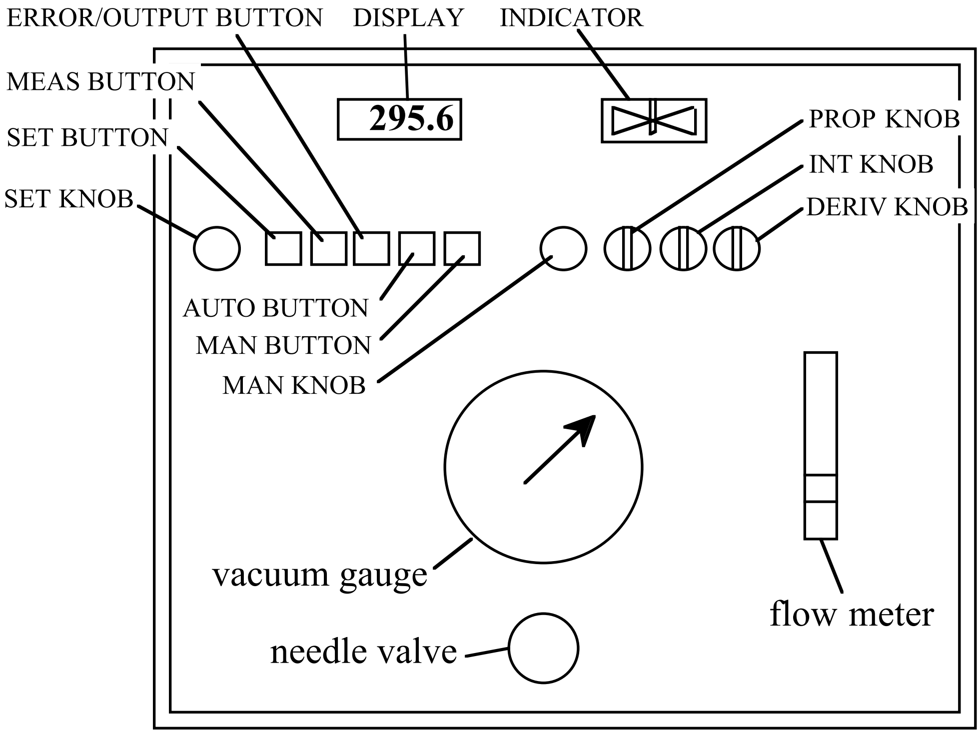

Fig. 3. Front panel of the Flow and Temperature Controller.

4.4. Temperature Controller

There is a digital temperature controller mounted together with the flow controller. This device can automatically maintain a constant temperature of the sample chamber by adjusting the power dissipation in the electrical heater.

The front panel controls are shown in Fig. 3 and below you can find their descriptions.

DISPLAY	indicates either the measured or desired temperature
MEAS. BUTTON	switches the display to indicate the measured temperature
SET BUTTON	switches the display to indicate the desired temperature
SET KNOB	sets the desired temperature
AUTO BUTTON	causes the output voltage to be controlled automatically by the controller
MAN BUTTON	enables the output voltage to be varied manually
MAN KNOB	varies the output voltage when the MAN button is depressed

INDICATOR	indicates either the temperature error or the controller output voltage
ERROR/OUTOUT BUT.	selects whether the temperature error or the output voltage is indicated
PROP KNOB	sets the controller Proportional Band
INT KNOB	sets the controller Integral Action Time
DERIV KNOB	sets the controller Derivative Action Time

Meanings of these controls are clear from their description and we discuss in more details only the last three.

To form the output voltage of the temperature controller, the measured temperature is compared with a desired temperature and an error signal is generated proportional to the temperature difference. This signal is used as the input to a Three-Term Controller, which regulates the power fed to a heater. The controller output voltage at given time depends on:

a) The error at that instant (proportional action).
b) The integral with respect to time of the error (integral action).
c) The derivative with respect to time of the error (derivative action).

These constitute the three control terms. Their coefficients may be individually varied with PROP, INT and DERIV knobs, enabling very accurate temperature control. Such a complicated system is absolutely necessary to ensure stable and accurate control of temperature.

Usually it is not necessary to alter these controls. However, if the system shows the temperature oscillations or the temperature stabilization time is too long (longer than 5-8 minutes) these knobs must be adjusted. In the case of oscillations the DERIV knob should be rotated clockwise. To speed up the stabilization time, the DERIV as well as INT knobs should be rotated anticlockwise. More detailed description of the regulation procedure as well as a short theory of three-term (P.I.D.) control can be found in the Appendix.

5. Appendix

5.1. Setting of the Temperature Controller

Initially, set the DERIV control fully anticlockwise, the INT control fully clockwise and the PROP control at about the middle of its travel. Set a desired temperature. Note that the controller output varies in such a way that the measured temperature is brought nearer to the desired temperature. After a time, the measured temperature will reach a steady value close to the desired temperature or will oscillate above and below this temperature.

If oscillations were obtained in the stage described above; measure the time for one complete cycle. This is an indication of the system response time. The INT control should be set to approximately twice the system response time and the DERIV control to approximately half the system response time. The PROP control should then be increased until stable control is achieved without oscillation. In general, the best setting for the proportional band is the smallest value that does not cause oscillation.

If no oscillations were obtained initially, try reducing the PROP control. If oscillations are still not obtained, the system is inherently very stable and the minimum proportional band setting may be used. In this case, an estimate of the system response time may be made by observing the response to a sudden change in the desired temperature. This may then be used as a guide to setting the integral and derivative controls.

The above procedure only provides a guide to the control term settings. To obtain the best performance from a given system, it is worthwhile experimenting with the controls using the above settings as a starting point. In many cases, it is not necessary to use derivative action at all and the DERIV control may be left at zero. A short description of the theory behind three-term (P. I.D.) control can be found in the next section.

Once the P.I.D. controls have been set, it should not normally be necessary to alter them unless the system response time varies for some reason. (This can for example occur in low temperature systems where, as the temperature falls, metals lose their specific heat and thus respond more rapidly to changes in heater power).

5.2. Theory of Control

5.2.1. General

The aim of a controller is to maintain the temperature of a system as close as possible to some desired temperature (the set paint) and as far as possible to eliminate the effect of changes in the heat loss from the system. When a steady state is established, the heating provided by the controller will exactly balance the heat lost by the system to its surroundings. A further function of the controller is to follow any changes in the set point as rapidly as possible. Thus the criteria for good control are:

CONTROL ACCURACY	The mean temperature of the system should be as close as possible to the desired temperature.
CONTROL STABILITY	The fluctuations above and below the mean temperature should be small.
CONTROL RESPONSE	The system should follow changes in the set point as rapidly as possible.

In the following sections a number of possible control systems of increasing complexity are described, culminating in the sophisticated Three-Term (P. I.D.) control employed by our temperature controller.

5.2.2. Open Loop Operation

In an open loop system a fixed heater power is applied and the system is allowed to come to equilibrium. There is no control as such, since the heater power can only be changed by the intervention of a human operator. The system takes a long time to reach equilibrium and only changes in the heat loss from the system produce corresponding changes in the system temperature.

5.2.3. On-Off Control

In an on-off control system the heater power is either full on, if the temperature is below the set point, or off - if it is above. The control accuracy and response can be made very good with this form of control and the system can be made largely immune to changes in heat loss. However, the control stability can never be very good since the system temperature must always cycle above and below set point. The magnitude of the temperature fluctuations depends on the thermal properties of the system. For some systems, where temperature fluctuations are not important, this is a perfectly satisfactory and simple system of control (e.g. the domestic electric oven).

5.2.4. Proportional Control

A proportional control system overcomes the problem of temperature cycling by allowing the heater power to be continuously varied. The heater voltage at any instant is proportional to the error between the measured and desired temperatures. Thus a large negative error (measured temperature below desired temperature) will produce a large heater voltage in order to correct that error.

If the output voltage was proportional to the error over the whole range of the instrument, a negative error equal to half the total span of the instrument would be necessary to generate the full output voltage and a similar positive error would be necessary to reduce the output voltage to zero. Thus, although the controller would not suffer from temperature fluctuations, the accuracy would be very poor.

5.2. 5. Proportional Band

The Proportional Band of a controller is defined as the band of input signals over which the output is proportional to the error. It is normally expressed as a percentage of the total input span of the instrument and is centered about the set point.

In the situation described above, where the output is proportional to the error over the whole span, the proportional band is 100%. By reducing the proportional band, the accuracy of the controller may be improved since a smaller error will then be necessary to produce a given change in output. The proportional band thus provides a convenient method of defining the gain of the controller in a way which is independent of the type of sensor or heater voltage in use. A small value of proportional band represents a high value of controller gain.

This would seem to imply that, by sufficiently reducing the proportional band, any required control accuracy could be obtained. Unfortunately, as the proportional bond is progressively reduced, there will come a point at which temperature oscillations reappear.

The reduction in proportional band, which can be achieved before the onset of oscillations, will depend largely on the design of the system being controlled. In some systems, it may be possible to achieve the required control accuracy without oscillations but, in most cases, this will not be so.

5.2.6. Integral Action

To overcome this problem, Integral Action is introduced. Consider a system controlled by proportional action as described above, with the proportion band sufficient to prevent oscillation. The result will be stable control, but with a residual error between the measured and desired temperatures. Suppose this error signal is fed to an integrator, the output of which is added to the existing controller output. The effect of this will be to vary the overall output until control is achieved with no residual error. At this point, the input to the integrator will be zero and this will therefore maintain a constant output. Integral Action has thus served to reduce the residual error associated with a Proportional only control system. Provided the contribution from the integrator is only allowed to vary slowly, the Proportional Action will prevent the occurrence of oscillations. The response of the integrator is characterized by the Integral Action Time. This is defined as the time taken for the output to vary from zero to full output in the presence of a steady error of 1 Proportional Band.

To ensure that the integrator itself does not give rise to oscillations, it is usual to employ an Integral Action Time of at least twice the response time of the system.

5.2.7. Derivative Action

The combination of Proportional and Integral Action will suffice to ensure that accurate and stable control can be achieved at a fixed temperature. However, it is possible by the use of Derivative Action to improve the time response of the system to changes in the set point. Without derivative action, many systems will tend to overshoot the desired temperature. Derivative Action monitors the rate at which the measured temperature is changing and modifies the control output such as to reduce this rate of change. In this way, overshoot can be reduced and in many systems completely eliminated. (Derivative Action is exactly analogous to the use of velocity feedback in servo systems and serves the some function).

Like Integral Action, Derivative Action is characterized by an Action Time. If the measured temperature is changing at a rate of 1 Proportional Band per Derivative Action Time, Derivative Action will contribute a signal sufficient to reduce a full output to zero or vice verso.

5.2.8. Conclusion

The consideration above shows that only the combination of Proportional Action, Integral Action and Derivative Action in the temperature controller (P.I.D. controllers) permits accurate and stable control of temperature. Proper regulation of P.I.D. controls gives a possibility to use the same controller for any system independently of its size, thermal capacity and thermal connection to surrounding media. Accuracy of temperature stabilization for such a controller is usually limited only by the sensitivity of a thermometer.