Experiment No-2

Temperature dependence of resistivity of semiconductors using four-probe method

Aim of the Experiment

To Study temperature dependence of resistivity in germanium (Ge) and to estimate the electronic band gap (E_g) energy in Germanium (Ge) using Four-point probe setup with furnace.

Introduction

The three classification of solids, namely, metals, insulators and semi-conductors is based on the energy band structure in the solid. Energy bands for solids, are the solutions of quantum many-body equation (Schrödinger-like equation) which represents electron energies (eigen values) as a function of the electron wave vector (*i.e.*, momentum).

The properties of the bulk material used for the fabrication of transistors and other semiconductor devices are essential in determining the characteristics of the completed devices. Resistivity and lifetime (of minority carriers) measurements are generally made on germanium crystals to determine their suitability. The resistivity, in particular, must be measured accurately since its value is critical in many devices. The value of some transistor parameters, like the equivalent base resistance, are at least linearly related to the resistivity.

Electronic Conduction in Solids

Electrical properties of semiconductors involve the dynamics of the charged particles within them. Therefore, we must have an understanding of the forces which control the motion of these particles. Atoms are electrically neutral in isolation. Electrons are arranged in different shells, and the closer they are to the nucleus the more strongly they are bound. If we take the particular case of silicon, a well- known semiconductor, we find that it has 14 electrons which are accommodated in the shells as $1s^2 2s^2 2p^6 3s^2 3p^2$. Since the 3p shell is not even half filled, the 4 electrons are available for chemical binding giving silicon a valency of four. (Germanium also has a chemical valency of 4, but from the fourth shell).

If we bring many atoms close to one another, interatomic forces become quite strong as electronic orbits begin to overlap. The outer shell electrons play an important role, because their orbits are the most disturbed. These electrons are no longer associated with a particular atom, the outer shell electrons may make an orbit around one atom and continue about another. In this fashion, the outer shell or valence electrons are continually traded among atoms and wander all over the solid. The continuous interchange of valence electrons between atoms holds the solid together. This is the predominant type of bonding in silicon and germanium.

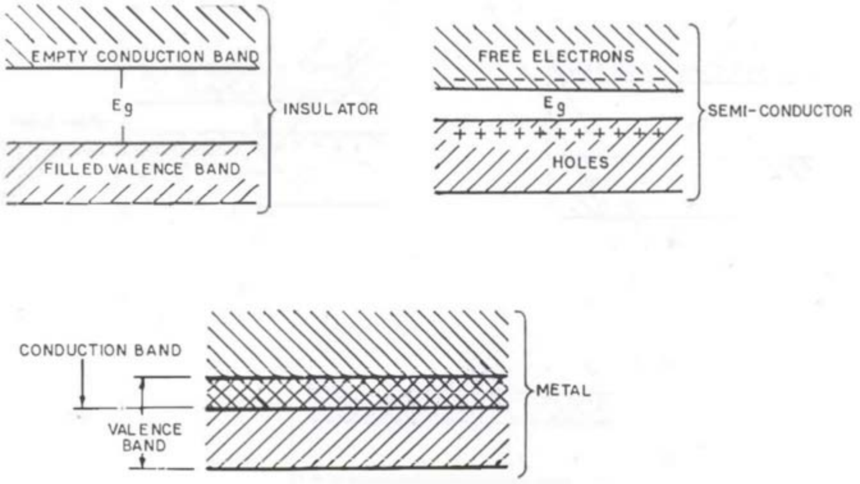


Figure 1 Band structure for solids

In solids, atoms are usually arranged in a regular ways to achieve a dense packing and thereby form a crystal. The arrangement has very desirable characteristics, *i.e.*, the transport of holes and free electrons is very cooperative in these structures. When the arrangement is not crystalline complication arise. Here we will be concerned only with the properties of perfect crystals. Silicon and germanium (and carbon) crystallize with an identical crystal structure, called diamond structure.

In solids, atoms strongly interact with each other, and the valence electrons do not belong to any particular atom. That means if the solid has N no. of atoms then each atomic level must split into N sub-levels, very closely placed to each other so as to accommodate the electronic levels of the same. The spreading of energy levels depends on the degree of interaction, therefore, the inner orbits split into levels combined in a narrow energy than the outer ones. As a result of the interaction among large number of atoms in crystal ($\sim 10^{24}$ per cm^3), the energy levels found in isolated atoms will be split and form bands of allowed energies which contain almost continuum of levels in case of metals. Accordingly, electrons are located in energy bands in crystalline solids. The band which contains the valence electrons is called the valence band. The unoccupied energy levels also split up and form another band called the conduction band. The interaction between the unused shells is very large and they spread widely. In case of semiconductor, the valence and conduction bands are separated by what is called energy band gap (E_g). An electron can not assume an energy that falls with E_g.

The bands below the energy gap E_g are completely filled at absolute zero temperature and the conduction band is empty. This is a very important point and has direct consequences on the conduction properties, as we shall see soon. The fundamental theory is that current conduction is not possible in empty and filled bands. The reason, about the empty band is obvious since current is not possible without carriers. The reason about the filled band is as follows: though the valence electrons move about the crystal but they cannot be accelerated because the acceleration means gain of energy and there are no higher energy levels available to which they could rise.

Crystal band structure does not allow current conduction at T=0 in the absence any other external perturbation. If we increase the temperature, however, thermal agitation increases and some valence electron will gain energy greater than E_g and jump into the conduction band. The electron in the conduction band are free electron, and the absence of the very electron in the valence band is called a hole. Electrons in the conduction band can gain energy when a field is applied, because there are many higher energy states available. The fact that electrons left the valence band leaves some empty energy levels, this allows conduction in the valence band as well. We observe motion of holes in the direction of the field. Because of this we begin to speak of a hole as a current carrying particles.

According to the preceeding discussion, an insulator must have a large bandgap, so that at room temperature the conduction band is practically empty and the valence band is fully filled and a semiconductors must have a narrower band gap so that appreciable number of carriers are present in the valence and conduction bands at room temperature. In metals, however, the valence and conduction bands overlap and application of an electric field can, therefore, accelerate a great sea of electrons. The non-existence of a bandgap make conduction in metal almost independent of temperature, as compared to semiconductors.

FOUR PROBE METHOD

Many conventional methods for measuring resistivity are unsatisfactory for semiconductors because metal-semiconductor contacts are usually rectifying in nature. Also there is generally minority carrier injection by one of the current carrying contacts.

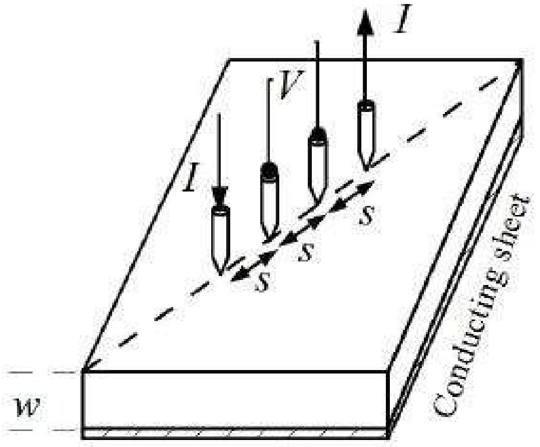


Figure 2: Typical Four-probe setup

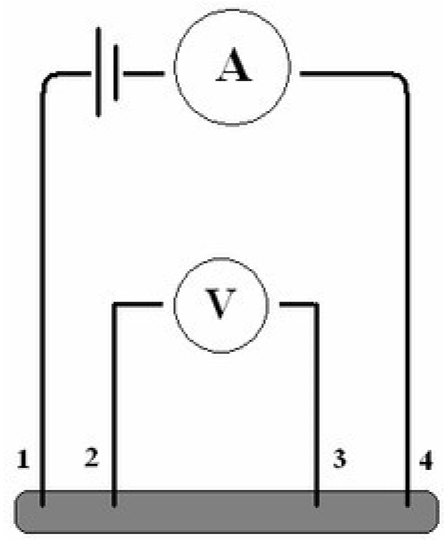


Figure 3: Four-point measurement of resistance between voltage sense connections 2 and 3. Current is supplied via force connections 1 and 4.

An excess concentration of minority carriers will affect the potential of other contacts and modulate the resistance of the material. The method described here overcomes the difficulties mentioned above and also offers several other advantages. It permits measurements of resistivity in samples having a wide variety of shapes, including the resistivity of small volumes within bigger pieces of semiconductor. In this manner the resistivity of both sides of p-n junction can be determined with good accuracy before the material is cut into bars for making devices. This method of measurement is also applicable to silicon and other semiconductor materials.

In this setup, four sharp probes are placed on a flat surface of the material to be measured, current is passed through the two outer electrodes, and the floating potential is measured across the inner pair. If the flat surface on which the probes rest is adequately large and the crystal is big the semiconductor may be considered to be a semi-infinite volume. To prevent minority carrier injection and make good contacts, the surface on which the probes rest, maybe mechanically lapped.

In order to use this four probe method in semiconductor crystals or slides it is necessary to assume that:

1. The resistivity of the material is uniform in the area of measurement.

2. If there is minority carrier injection into the semiconductor by the current - carrying electrodes most of the carriers recombine near the electrodes so that their effect on the conductivity is negligible. (This means that the measurements should be made on surface which have a high recombination rate, such as mechanical lapped surfaces).

3. The surface on which the probes rest is flat with no surface leakage.

4. The four probes used for resistivity measurements contact the surface at points that lie in a straight line.

5. The diameter of the contact between the metallic probes and the semiconductor should be small compared to the distance between probes.

6. The boundary between the current-carrying electrodes and the bulk material is hemispherical and small in diameter.

7. The surfaces of the semiconductor crystal may be either conducting or non-conducting.

(a) A conducting boundary is one on which a material of much lower resistivity than semiconductor (such as copper) has been plated.

(b) A non-conducting boundary is produced when the surface of the crystal is in contact with an insulator.

Brief Description of the Apparatus Required

1. Probes Arrangement:

It has four individually spring loaded probes, coated with Zn at the tips. The probes are collinear and equally spaced. The Zn coating & individual spring ensure good electrical contacts with the sample. The probes are mounted in a teflon bush which ensure a good electrical insulation between the probe. A teflon spacer near the tips is also provided to keep the probes at equal distance. The whole arrangement is mounted on a suitable stand and leads are provided for current and voltage measurements.

2. Sample: Ge or Si crystal in the form of a chip/slice

3. Oven: It is a small oven for the variation of temperature of the crystal from room temperature to about 200 °C.

4. Four Probes Set-up: (Measuring Unit)

It has three subunits all enclosed in one cabinet.

(i) Multi-range Digital Voltmeter

In this unit intersil 3½ digit single chip A/D converter ICL 7107 has been used. It has high accuracy, auto zero to less than 10 μV, zero drift-less than 1 μV/°C, input bias current of 10 pA and roll over error of less than one count. Since the use of internal reference causes the degradation in performance due to internal heating, an external reference has been used.

Specification

1. **Range:** X 1 (0 - 200.0 mV) & X 10 (0 - 2.000 V)
2. **Resolution:** 100 μV at X 1 range
3. **Accuracy**: ± 0.1% of reading ± 1 digit
4. **Impedance:** 10 M ohm
5. **Display:** 3½ digit, 7 segment, LED (12.5 mm height) with auto polarity and decimal indication. Overload Indicator: Sign of 1 on the left and blanking of other digits.

(ii) Constant Current Generator

It is a IC regulated current generator to provide a constant current to the outer probes irrespective of the changing resistance of the sample due to change in temperatures.

The basic scheme is to use the feedback principle to limit the load current of the supply to pre-set maximum value. Variations in the current are achieved by a potentiometer included for that purpose. The supply is a highly regulated and practically ripple free d.c. source. The current is measured by the digital panel meter.

Specification

1. **Open circuit voltage:** 18V
2. **Current range:** 0-20 mA
3. **Resolution:** 10 μA
4. **Accuracy:** ± 0.25% of the reading ±1 digit
5. **Load regulation:** 0.03% for 0 to full load
6. **Line regulation:** 0.05% for 10% changes

(iii) Oven Power Supply

Suitable voltage for the oven is obtained through a step down transformer with a provision for low and high rates of heating. A glowing LED indicates, when the oven power supply is 'ON'.

Experimental Setup and Connections

1. Put the sample on the base plate of the four probe arrangement. Unscrew the pipe holding the four probes and let the four probes rest in the middle of the sample. Apply a very gentle pressure on the probes and tighten the pipe in this position. Check the continuity between the probes for proper electrical contacts.

CAUTION: The Ge crystal is very brittle. Therefore, use only the minimum pressure required for proper electrical contacts. The resistance between the Red and Black probes and Yellow and Green probe may be approximately few K-Ohms (~1-4 K-Ohms).

2. Connect the outer pair of probes (red/black) leads to the constant current power supply and the inner pair (yellow/green leads) to the probe voltage terminals.

3. Place the four probe arrangement in the oven and fix the thermometer in the oven through the hole provided.

4. Switch on the ac mains of Four Probe Set-up and put the digital panel meter in the current measuring mode through the selector switch. In this position LED facing mA would glow. Adjust the current to a desired value (Say 5 mA).

5. Now put the digital panel meter in voltage measuring mode. In this position LED facing mV would glow and the meter would read the voltage between the probes.

6. Connect the oven power supply. Rate of heating may be selected with the help of a switch - Low or High as desired. Switch on the power to the Oven. The glowing LED indicates the power to the oven is 'ON'.

Observation and tabulations

Sample: Ge Crystal **Constant Current:_____mA**

Sl. No.	Temperature T (K)	Voltage V (V)	Resistivity □ Ohm-cm	$T^{-1}x10^3$	Log$_{10}$ □
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					

Distance between the Probes (s) = 2.00 mm.

Thickness of the Crystal (w) = 0.66 mm.

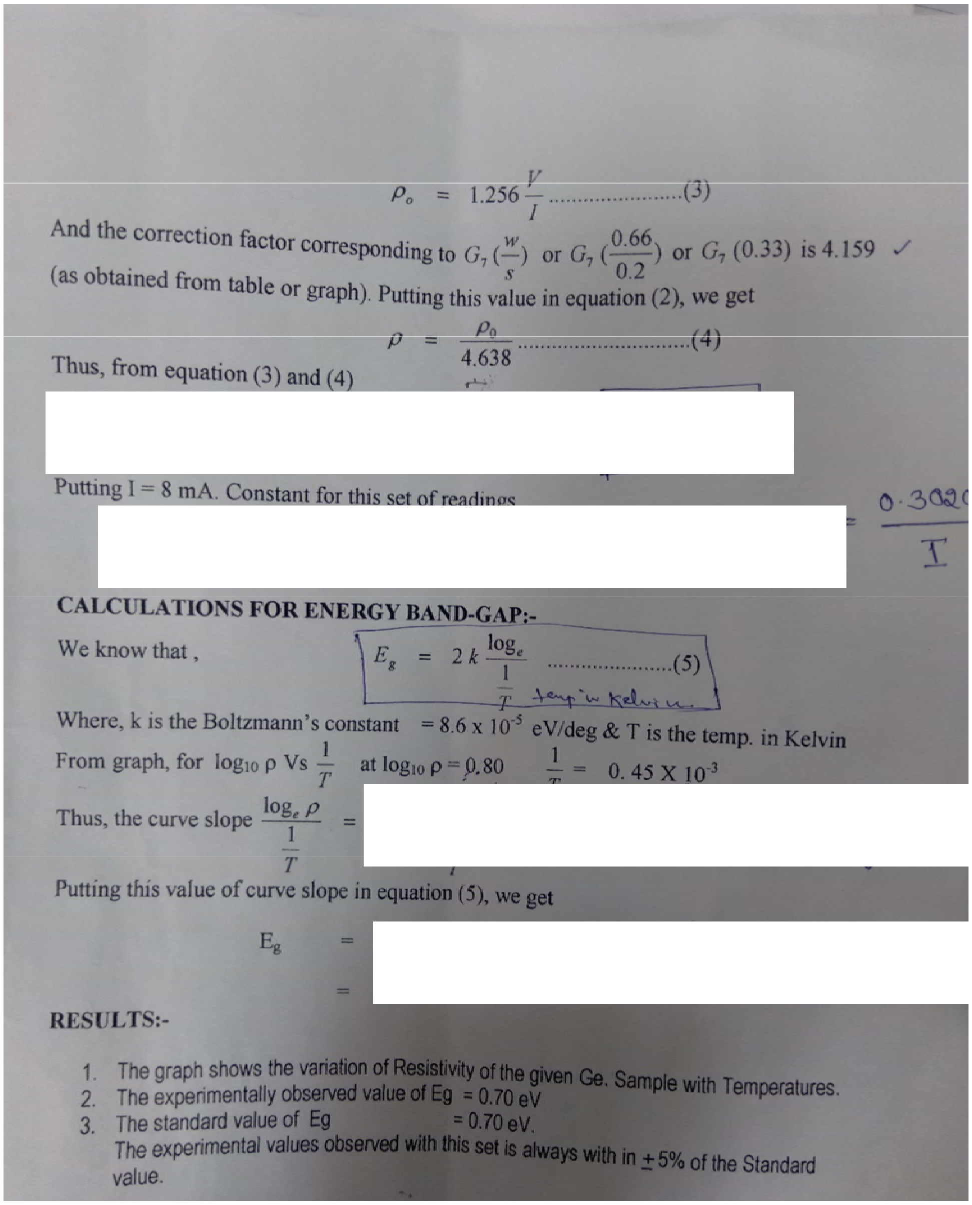
Calculations:

Resistivity Calculation (ρ): We know that, $\rho_0 = \frac{V}{I} . 2\pi s$ Eq. (1)

Since the thickness of the crystal is small compared to the probe distance a correction factor for it has to be applied. Further the bottom surface is non-conducting in the present case, so the correction factor would be

$$\rho = \frac{\rho_0}{G_7\left(\frac{W}{S}\right)} \qquad \text{Eq. (2)}$$

The function G_7 (W/S) may be obtained from Table-I or Fig. 5 for the appropriate value of (W/S). Thus ρ may be calculated for various temperature.



$$\rho_o = 1.256 \frac{V}{I} \text{ ....(3)}$$

And the correction factor corresponding to $G_7\left(\frac{w}{s}\right)$ or $G_7\left(\frac{0.66}{0.2}\right)$ or G_7 (0.33) is 4.159 ✓ (as obtained from table or graph). Putting this value in equation (2), we get

$$\rho = \frac{\rho_0}{4.638} \text{ ....(4)}$$

Thus, from equation (3) and (4)

[redacted]

Putting I = 8 mA. Constant for this set of readings

[redacted] = $\frac{0{\cdot}302}{I}$

CALCULATIONS FOR ENERGY BAND-GAP:-

We know that ,

$$\boxed{E_g = 2k \frac{\log_e}{\frac{1}{T}} \text{ ....(5)}}$$

temp in Kelvin.

Where, k is the Boltzmann's constant = 8.6×10^{-5} eV/deg & T is the temp. in Kelvin

From graph, for $\log_{10} \rho$ Vs $\frac{1}{T}$ at $\log_{10} \rho = 0.80$ $\frac{1}{T} = 0.45 \times 10^{-3}$

Thus, the curve slope $\frac{\log_e \rho}{\frac{1}{T}}$ = [redacted]

Putting this value of curve slope in equation (5), we get

E_g = [redacted]

=

RESULTS:-

1. The graph shows the variation of Resistivity of the given Ge. Sample with Temperatures.
2. The experimentally observed value of Eg = 0.70 eV
3. The standard value of Eg = 0.70 eV.
The experimental values observed with this set is always with in ±5% of the Standard value.

Results:

1. The graph shows the variation of resistivity of the given sample (Ge crystal) in accordance with temperature.
2. The experimentally observed value E_g of Ge-crystal=

The experimentally values observed with this experiment is within ±……of the standard value.

Reference:

(1) Raman Scientific Instruments, Lab Manual.

(2) Introduction of Solid State Physics, by C. Kittel IVth edition.

(3) Fundamental of Semiconductor Devices by J. Lindmayer and C.Y. Wriegley,

Report

The lab report should consists of the followings:

(a) Name of the experiment; (b) Objectives; (c) A brief description of theory ; (d) Apparatus associated; (e) Observation and calculations (f) a plot of $\log_{10} \rho$ vs. $\frac{1}{T} x 10^3$ and from there calculate the band gap E_g of the sample; (g) precautions required (h) suggestions toward betterment of the experiment.

Appendix:

Table-1

Sl. No.	$\left(\frac{□}{□}\right)$	$□_□\left(\frac{□}{□}\right)$	$□_□\left(\frac{□}{□}\right)$
1.	0.100	0.0000019	13.863
2.	0.141	0.00018	9.704
3.	0.200	0.00342	6.931
4.	0.33	0.0604	4.159
5.	0.500	0.228	2.780
6.	1.000	0.683	1.504
7.	1.414	0.848	1.223
8.	2.000	0.933	1.094
9.	3.333	0.9838	1.0228
10.	5.000	0.0048	1.0070
11.	10.000	0.9993	1.00045

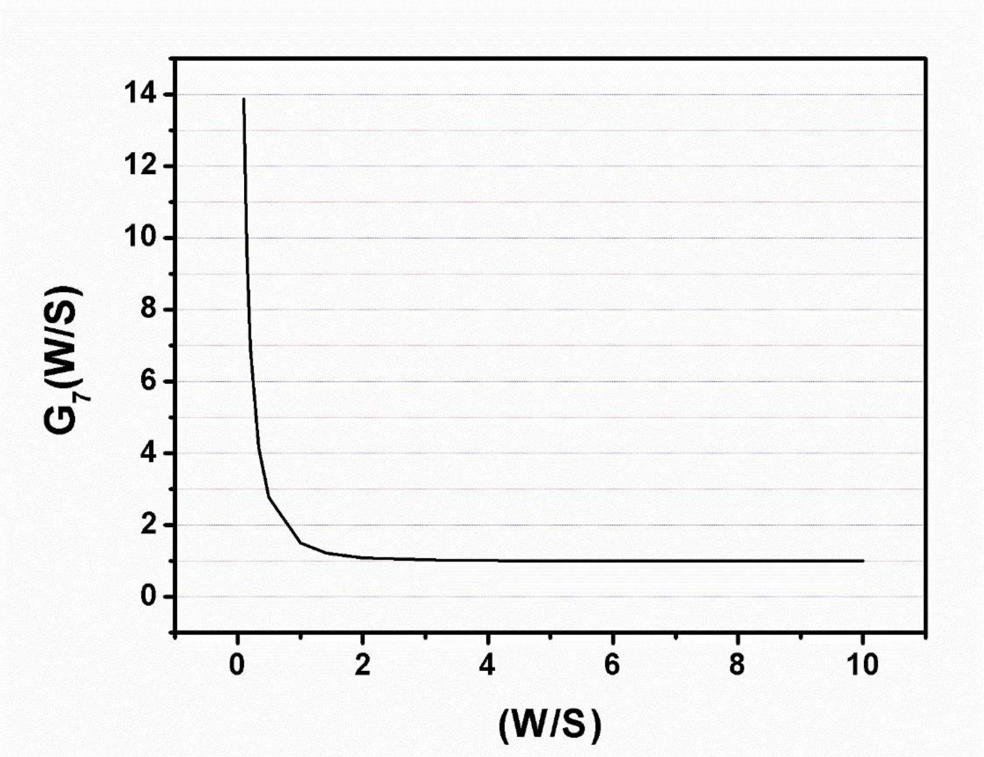


Figure: Correction division probe on a thin slice with non-conducting bottom surface.