Resistivity measurement and estimation of band gap using four probe method

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The four-probe method is one of the standard & most commonly used methods for the accurate measurement of resistivities of the different materials as it overcomes the problem of contact resistance and also offers several other advantages. Accurate resistivity measurement in samples having a variety of shapes is possible with this method. The pressure contacts provided in the Four Point arrangement are especially useful for quick measurement. This setup can measure samples of a reasonably wide resistivity range ($\mu\Omega$ to $M\Omega$). This report is focused on the determination of the resistivities and band gap of the different samples provided to us (Germanium, Silicon, and Aluminium) with a non-conducting bottom surface at room temperature. Additionally, we have verified the temperature dependence of the resistivity of n-Germanium in the temperature range $(80 - 170)^{\circ}C$

I. OBJECTIVES

- To measure the resistivity of semiconductors and metal at room temperature.
- To measure the resistivity of a semiconductor as a function of temperature and determination of energy band gap.

II. THEORY

A. Electronic Conduction in Solids

The electrical properties of semiconductors involve the motion of charged particles within them. Therefore, we must have an understanding of the forces which control the motion of these particles. It is, of course, the physical structure of the solid that exerts its control.
Atoms, of which a solid is composed, consist of positively charged nuclei with electrons orbiting around them. The positive charge is compensated by negatively charged electrons, so that a complete atom is electrically neutral. Electrons are arranged in shells, and the closer they are to the nucleus the more strongly they are bound. If we take the particular case of Si, 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 third shell is not even half-filled, the 4 electrons are available for chemical binding giving Si a valency of four. (Germanium also has a chemical valency of 4, but from the fourth shell).
For solids, 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 electron may make an orbit around one atom and continue about another. In this fashion, the outer shell or valency 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 Si and Ge and is called **valence bonding**.
FIG. 1 shows an energy diagram of an individual atom.

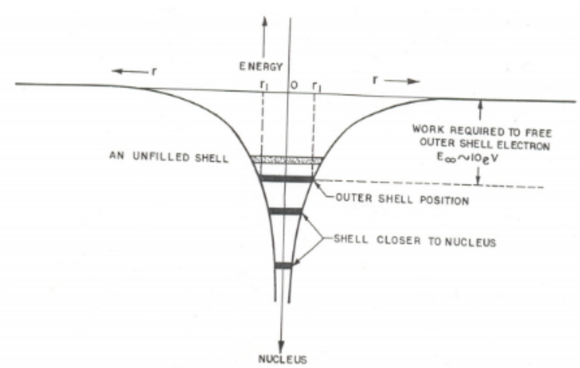


FIG. 1. Potential distribution around an individual atom

In solids, atoms are usually arranged in a regular way 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 smooth in these structures. When the arrangement is not crystalline complications arise. Here we will be concerned only with the properties of perfect crystals. Si and Ge crystallize with an identical crystal structure (diamond structure). FIG. 2 shows a potential diagram of an array of atoms.
An actual crystal is of course 3-dimensional. The most important difference between the potential plot of an isolated atom and a one-dimensional array is the splitting of energy levels. In fact, by bringing N atoms together we find 'N' times as many levels throughout the crystal. The spreading of energy levels depends on the degree of

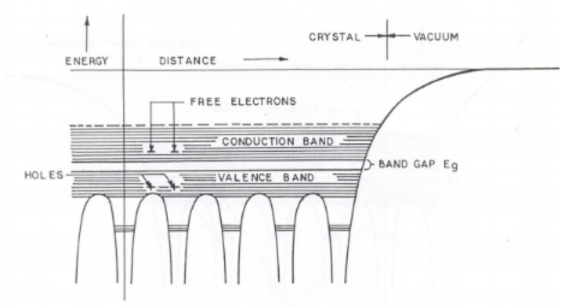


FIG. 2. Illustration of the bands in an array of atoms

interaction, therefore, the inner orbits split into levels combined in a narrow energy than the outer ones.

As a result of the interaction between the tremendous number of atoms in the crystal, the energy level found in isolated atoms will be split and form bands of allowed energies which contain almost a continuum of levels. Accordingly, electrons are located in energy bands in a crystalline solid. 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. Therefore, while there is a bandgap, E_g(or forbidden region) between the valence and conduction bands, the splitting of the higher orbit is so wide that they usually overlap.

The bands below the energy gap E_g are completely filled at absolute zero temperature and the conduction band is empty. The fundamental theory is that current conduction is not possible in empty and filled bands. The reasons for the empty band are obvious since the current is not possible without carriers. The reason for the filled band is as follows: though the valence electrons move about the crystal, they can not be accelerated because the acceleration means a gain of energy and there are no higher energy levels available to which they could rise.

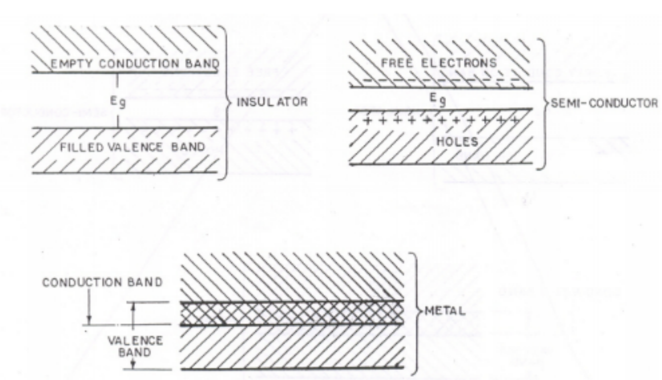


FIG. 3. Band structure of insulators, semiconductors and metals

If we increase the temperature, however, thermal agitation increases and some valence electrons will gain energy greater than E_g and jump into the conduction band. The electron in the conduction band is called a free electron, and its former place 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. Electrons can now gain energy in the valence band also, and we observe a motion of holes in the direction of the field.

B. Four-probe arrangement

It has four individually spring-loaded probes. The probes are collinear and equally spaced. The probes are mounted in a Teflon bush, which ensures good electrical insulation between the probes. A Teflon spacer near the tips is also provided to keep the probes at an equal distance. The probe arrangement is mounted in a suitable stand, which also holds the sample plate and RTD sensor. This stand also serves as the lid of the PID Controlled Oven. Proper leads are provided for current, Voltage & Temperature measurement with their universal connectors. For the current measurement, there is a three-pin connector that can be connected to the CCS-01/ LCS-02 as per the requirement of the sample. For voltage measurement BNC connector is used and connected to DMV-001 unit. For temperature measurement, a two-pin connector is provided for connection with the PID-Controlled oven unit.

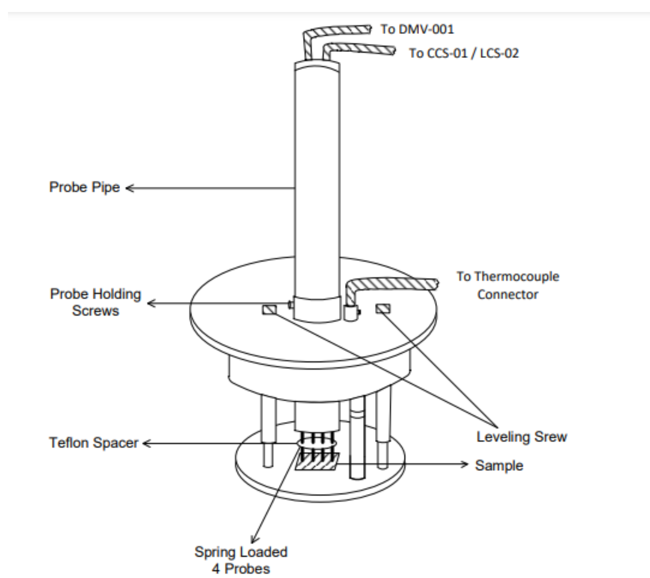


FIG. 4. Four-probe arrangement

Three leveling screws are provided in the Four Probe arrangement by which we can adjust the level of the platform to make it horizontal. A probe-holding screw is provided at the collar of the arrangement. Initially, it should be in a loose position, to allow free movement of the Probe Pipe. After placing the sample the Probe Pipe should be lowered so that all four pins touch the sample. Further, press the pipe very lightly so that assured firm contact is made with all Four pins with the sample. Tighten the Probe Holding Screw at this position.

C. Four-probe method

In order to use the four-probe method, it is assumed that:

- The resistivity of the material is uniform in the area of measurement.
- If there is a 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 a surface, which has a high recombination rate).
- The surface on which the probes rest is flat with no surface leakage.
- The four probes used for resistivity measurements are equally spaced and collinear.
- The diameter of the contact between the metallic probes and the semiconductor should be small compared to the distance between probes.
- The surfaces of the material may be either conducting or non-conducting.

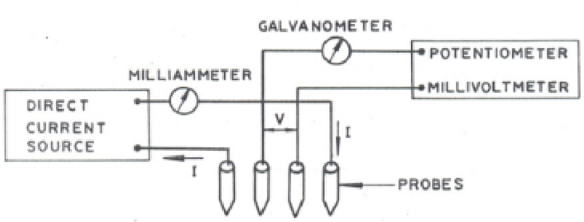


FIG. 5. Circuit for resistivity measurement using four probe method

D. Resistivity Measurements

1. *Resistivity Measurement on a large sample*

The floating potential V_f at a distance r from an electrode carrying a current I in a material of resistivity $\rho_o$is given by:

$$V_f = \frac{\rho_o I}{2\pi r} \tag{1}$$

When the point spacings are equal, the resistivity is computed as:

$$\rho_o = \left(\frac{V}{I}\right) 2\pi S \tag{2}$$

where S= point spacing.

2. *Resistivity Measurement on a thin slice conducting bottom surface*

In this case, resistivity is given as:

$$\rho = \frac{\rho_o}{G_6\left(\frac{W}{S}\right)} \tag{3}$$

where $G_6\left(\frac{W}{S}\right)$ is given by:

$$G_6\left(\tfrac{W}{S}\right) = 1 + 4\tfrac{S}{W}\sum_{n=1}^{\infty}(-1)^n\left[\frac{1}{\sqrt{(\frac{S}{W})^2+(2n)^2}} - \frac{1}{\sqrt{(2\frac{S}{W})^2+(2n)^2}}\right] \tag{4}$$

3. *Resistivity Measurement on a thin slice non-conducting bottom surface*

In this case, resistivity is given as:

$$\rho = \frac{\rho_o}{G_7\left(\frac{W}{S}\right)} \tag{5}$$

where $G_7\left(\frac{W}{S}\right)$ is given by:

$$G_7\left(\tfrac{W}{S}\right) = 1 + 4\tfrac{S}{W}\sum_{n=1}^{\infty}\left[\frac{1}{\sqrt{(\frac{S}{W})^2+(2n)^2}} - \frac{1}{\sqrt{(2\frac{S}{W})^2+(2n)^2}}\right] \tag{6}$$

For smaller values of W/S, the function $G_7\left(\frac{W}{S}\right)$ approaches the case for an infinitely thin slice as:

$$G_7\left(\frac{W}{S}\right) = \left(\frac{2S}{W}\right) ln2 \tag{7}$$

E. Temperature dependence of resistivity of semiconductors

The total electrical conductivity of the semiconductors is contributed by the conductivities of the charge carriers, electrons, and holes which is given by:

$$\sigma = e(n_e\mu_e + n_h\mu_h) \tag{8}$$

where,
n_e,μ_e= electron concentration and mobility
n_h,μ_h= hole concentration and mobility.
In the intrinsic region, the concentration of electrons and

holes are equal ($n_e = n_h = n_i$), and hence the conductivity becomes:

$$\sigma = n_i e(\mu_e + \mu_h) \tag{9}$$

For intrinsic semiconductors, the temperature dependence is given by:

$$\rho = Aexp\left(\frac{E_g}{2kT}\right) \tag{10}$$

In log_{10} terms,

$$log_{10}\rho = C + \left(\frac{1}{2.3026}\right)\left(\frac{E_g}{2kT}\right) \tag{11}$$

III. EXPERIMENTAL SETUP

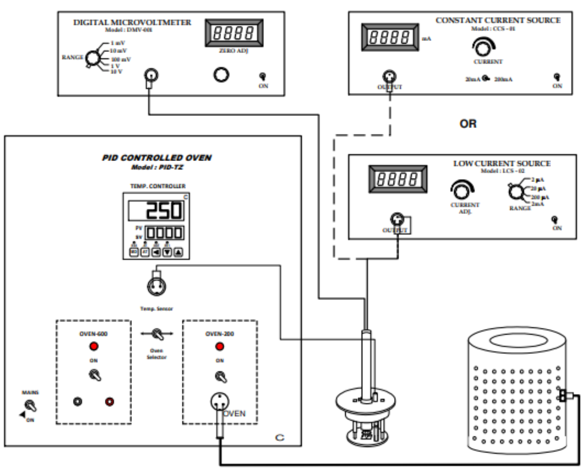


FIG. 6. Experimental setup

Components required:

- **Four probe arrangement**: Sample to be studied is kept here.
- **PID-Controlled oven**: to control the temperature on which the four probe arrangement is kept.
- **Digital voltmeter**: to measure the potential difference across the sample.
- **Low and constant current source**: to measure the current flowing through the sample.
- **Silicon, Germanium and Aluminium samples**

FIG. 7. Complete experimental setup

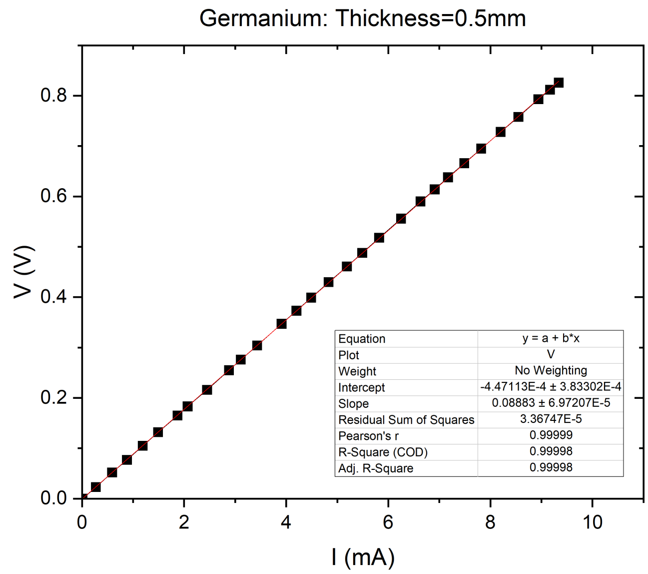
IV. OBSERVATIONS AND CALCULATIONS

Table 1: For I-V characteristics of Ge

Probe spacing, S = 0.2 cm
Thickness of sample, W = 0.5 mm
$G_7\left(\frac{W}{S}\right) = \left(\frac{2S}{W}\right) ln2 = 5.5452$

Germanium	
I (mA)	V (Volts)
0.01	0
0.27	0.023
0.59	0.052
0.88	0.077
1.19	0.105
1.49	0.132
1.87	0.165
2.07	0.183
2.45	0.216
2.88	0.255
3.11	0.276
3.43	0.304
3.91	0.347
4.2	0.373
4.49	0.399
4.83	0.43
5.19	0.461
5.49	0.488
5.82	0.518
6.25	0.556
6.63	0.59
6.91	0.614
7.17	0.638
7.49	0.666
7.82	0.695
8.2	0.728
8.55	0.758
8.94	0.793
9.17	0.812
9.34	0.826

Plot-1: I-V characteristics of Ge



Slope = $(0.08883 \pm 6.972 \times 10^{-5})$ V/mA
Intercept = $(-4.4711 \pm 3.833) \times 10^{-4}$ V
Resistivity,

$$\rho_o = slope \times 2\pi S$$

$$\rho_o = 1.1157\Omega.m$$

Corrected resistivity of Ge:

$$\rho = \frac{1.1157}{5.5452} = 0.2012\Omega.m$$

Error in ρ:

$$\frac{\delta\rho}{\rho} = \frac{\delta slope}{slope}$$

$$\frac{\delta\rho}{\rho} = 7.85 \times 10^{-4}$$

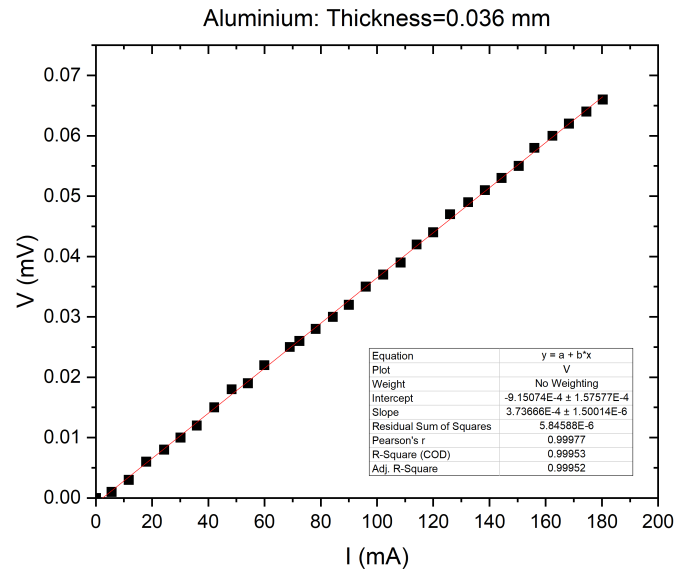
$$\delta\rho = 1.58 \times 10^{-4}\Omega.m$$

Table 2: For I-V characteristics of Al

Probe spacing, S = 0.2 cm
Thickness of sample, W = 0.036 mm
$G_7\left(\frac{W}{S}\right) = \left(\frac{2S}{W}\right) ln2 = 77.016$

Aluminium	
I (mA)	V (mV)
0.1	0
5.6	0.001
11.7	0.003
17.9	0.006
24.3	0.008
30.1	0.01
35.9	0.012
42.1	0.015
48.3	0.018
54.1	0.019
59.9	0.022
69	0.025
72.4	0.026
78.2	0.028
84.3	0.03
90	0.032
96	0.035
102.2	0.037
108.4	0.039
114.1	0.042
120	0.044
126	0.047
132.4	0.049
138.4	0.051
144.3	0.053
150.4	0.055
156	0.058
162.4	0.06
168.3	0.062
174.5	0.064
180.3	0.066

Plot-2: I-V characteristics of Al



Slope = $(3.7366 \times 10^{-4} \pm 1.5 \times 10^{-6})$ mV/mA
Intercept = $(-9.1507 \pm 1.5757) \times 10^{-4}$ mV
Resistivity,

$$\rho_o = slope \times 2\pi S$$

$$\rho_o = 0.047\Omega.m$$

Corrected resistivity of Al:

$$\rho = \frac{0.047}{77.016} = 0.00061\Omega.m$$

Error in ρ:

$$\frac{\delta\rho}{\rho} = \frac{\delta slope}{slope}$$

$$\frac{\delta\rho}{\rho} = 0.004 \times 10^{-4}$$

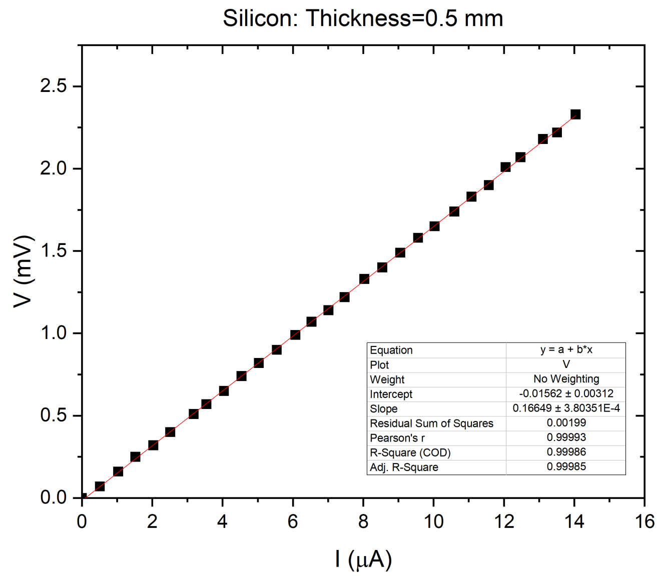
$$\delta\rho = 3.4026 \times 10^{-5}\Omega.m$$

Table 3: For I-V characteristics of Si

Probe spacing, S = 0.2 cm
Thickness of sample, W = 0.5 mm
$G_7\left(\frac{W}{S}\right) = \left(\frac{2S}{W}\right) ln2 = 5.5452$

Silicon	
I (μ A)	V (mV)
0	0
0.51	0.07
1.03	0.16
1.52	0.25
2.03	0.32
2.51	0.4
3.18	0.51
3.54	0.57
4.04	0.65
4.54	0.74
5.03	0.82
5.54	0.9
6.07	0.99
6.53	1.07
7.01	1.14
7.47	1.22
8.03	1.33
8.54	1.4
9.05	1.49
9.56	1.58
10.03	1.65
10.59	1.74
11.08	1.83
11.57	1.9
12.05	2.01
12.47	2.07
13.11	2.18
13.51	2.22
14.04	2.33

Plot-3: I-V characteristics of Si



Slope = $(0.1665 \pm 3.8035 \times 10^{-4})$ mV/μ A
Intercept = (-0.01562 ± 0.00312) mV
Resistivity,

$$\rho_o = slope \times 2\pi S$$

$$\rho_o = 2.0912\Omega.m$$

Corrected resistivity of Al:

$$\rho = \frac{2.0912}{5.5452} = 0.377\Omega.m$$

Error in ρ:

$$\frac{\delta\rho}{\rho} = \frac{\delta slope}{slope}$$

$$\frac{\delta\rho}{\rho} = 0.0023 \times 10^{-4}$$

$$\delta\rho = 0.00086\Omega.m$$

Table 4: Temperature dependence of resistivity of Ge

Constant current=5.02 mA					
Temperature (° C)	Voltage (V)	Resistivity ρ_o (ohm.m)	Resistivity ρ (ohm.m)	1/T (K^{-1})	$log_{10}\rho$
80	0.147	0.368	0.066	0.002833	-1.18046
90	0.113	0.283	0.051	0.002755	-1.29243
100	0.086	0.215	0.039	0.002681	-1.40894
110	0.064	0.16	0.029	0.002611	-1.5376
120	0.049	0.123	0.022	0.002545	-1.65758
130	0.039	0.098	0.018	0.002481	-1.74473
140	0.03	0.075	0.014	0.002421	-1.85387
150	0.024	0.06	0.011	0.002364	-1.95861
160	0.019	0.048	0.009	0.002309	-2.04576
170	0.015	0.038	0.007	0.002257	-2.1549
180	0.012	0.03	0.005	0.002208	-2.30103

Plot-4: Temperature dependence of resistivity in Ge

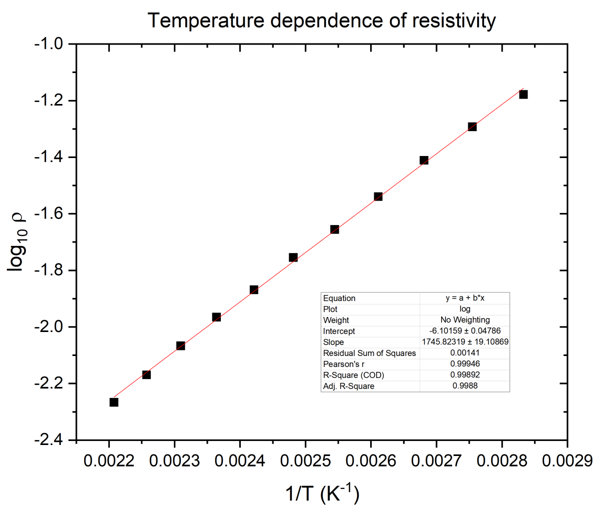


FIG. 8. $log_{10}\rho$ vs 1/T plot for Ge sample

Slope $= (1745.8232 \pm 19.1087)\Omega.m.K$ A
Intercept $= (-6.1016 \pm 0.04786)$ mV
Band gap energy (acc. to eqn. (11),

$$E_g = slope \times 2k \times 2.3026$$

$$E_g = 0.6928eV$$

Error in E_g:

$$\frac{\delta E_g}{E_g} = \frac{\delta slope}{slope}$$

$$\frac{\delta E_g}{E_g} = 0.01095$$

$$\delta E_g = 0.0076\Omega.m$$

V. RESULTS

- **Resistivity of Ge:**

$$\rho = (0.2012 \pm 0.000158)\Omega m$$

- **Resistivity of Al:**

$$\rho = (0.00061 \pm 0.000034026)\Omega m$$

- **Resistivity of Si:**

$$\rho = (0.377 \pm 0.00086)\Omega m$$

- **Band gap energy of Ge:**

$$E_g = (0.6928 \pm 0.0076)\Omega m$$

- **Variation of resistivity with temperature**

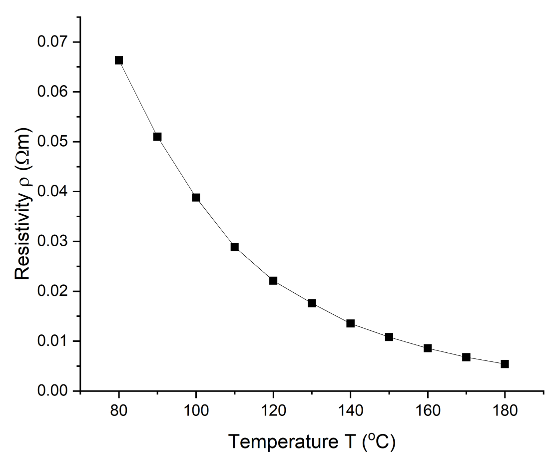


FIG. 9. Plot of resistivity(Ωm) vs temperature (^oC)

VI. CONCLUSIONS AND DISCUSSIONS

- We calculated the resistivities of Germanium, Aluminium, and Silicon using the four-probe method, and the values are specified in the RESULTS section.
- We got the resistivity value for Aluminium (a metal) to be much lower than that of the other samples, Ge and Si which are semiconductors, which is verified theoretically.
- The IV characteristics for all samples gave a linear curve as expected and followed theoretical knowledge.
- The band energy of Ge is found to be approximately 0.693 eV which suffices the theoretical value of 0.7 eV with some error bar.
- The variation of resistivity with temperature shows a very good exponential decay as shown in FIG. (9).

VII. SOURCES OF ERRORS

- Impurities in the sample.
- Fluctuations in the voltmeter readings may be due to poor probe contacts.
- Non-ideal behavior of PID-controlled oven.
- Environmental and electrical noises.

VIII. REFERENCES

- NISER Lab Manual
- `https://drpradeepatuem.files.wordpress.com/2018/07/4-probe.pdf`

- Signed data is attached on the next page.

[redacted]

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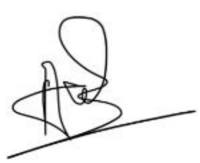

FIG. 10. Signed data