Measurement of resistivity and determination of band gap using Four-Probe method

GENERAL SAFETY SUMMARY

This equipment is Class 1 equipment tested in accordance with the European Standard publication EN 61010-1.

This manual contains information and warnings that must be observed to keep the Instrument in a safe condition and ensure safe operation.

To use the Instrument correctly and safely, read and follow the precautions in Table 1 and follow all safety instructions or warnings given throughout this manual that relate to specific measurement functions. In addition, follow all generally accepted safety practices and procedures required when working with and around electricity.

SYMBOLS

The table below lists safety and electrical symbols that appear on the Instrument or in this manual.

Table: Safety and Electrical Symbols

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Description</th>
<th>Symbols</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>⚠️⚠️</td>
<td>Risk of danger. Important information. See Manual.</td>
<td>⚡</td>
<td>Earth ground</td>
</tr>
<tr>
<td>⚠️</td>
<td>Hazardous voltage. Voltage &gt;30Vdc or ac peak might be present.</td>
<td>⚡</td>
<td>Potentially hazardous voltage</td>
</tr>
<tr>
<td>⚠️</td>
<td>Static awareness. Static discharge can damage parts.</td>
<td>⚠️</td>
<td>Do not dispose of this product as unsorted municipal waste. Contact SES or a qualified recycle for disposal.</td>
</tr>
</tbody>
</table>
# Table 1. Safety Information

<table>
<thead>
<tr>
<th>Warning</th>
</tr>
</thead>
</table>

To avoid possible electric shock, personal injury, read the following before using the Instrument:

- Use the Instrument only as specified in this manual, or the protection provided by the Instrument might be impaired.
- Do not use the Instrument in wet environments.
- Inspect the Instrument before using it. Do not use the Instrument if it appears damaged.
- Inspect the connecting lead before use. Do not use them if insulation is damaged or metal is exposed. Check the connecting leads for continuity. Replace damaged connecting leads before using the Instrument.
- Whenever it is likely that safety protection has been impaired, make the Instrument inoperative and secure it against any unintended operation.
- Never remove the cover or open the case of the Instrument before without first removing it from the main power source.
- Never operate the Instrument with the cover removed or the case open.
- Use only the replacement fuses specified by the manual.
- Do not operate the Instrument around explosive gas, vapor or dust.
- The equipment can remain Switched on continuously for five hours.
- The equipment must remain Switched off for at least fifteen minutes before being switched on again.
- The equipment is only for the intended use.
- Use the equipment only as specified in this manual.
Fig. 1. PID Controlled Oven, PID-TZ

Fig. 2. Shows the front panel of the Controller Panel for identification of the various keys, indicators and displays.
OBJECTIVES:

(I) To measure resistivity of a semiconductor and a metal at room temperature
(II) To measure resistivity of a semiconductor as a function of temperature and determination of energy band gap

INTRODUCTION

Four Probe method is one of the standard & most commonly used method for the accurate measurement of resistivity. It overcomes the problem of contact resistance and also offer several other advantages. Accurate resistivity measurement in samples having a variety of shapes is possible by this method. The pressure contacts provided in the Four Point Arrangement are especially useful for quick measurement. This setup can measure samples of reasonably wide resistivity range (micro ohm to mega ohm).

BRIEF DESCRIPTION OF THE SET UP

1. PID-TZ Controlled Oven

The unit is a high quality PID (Proportional, Integral and Differential) controller wherein the temperatures can be set and controlled easily. The P, I and D parameters are factory set ( P = 1.8, I = 300, D = 80) for immediate use, however, the user may adjust these for specific applications as well as auto-tune the oven whenever required. The steps for these are given in the user manual of the controller. A common controller may be used either for our small oven, up to 200°C or a larger oven up to 600°C. The two are switch selectable and use thermocouple as temperature sensors (see Fig.1).

General Specifications

The controller is designed around Autonics Temperature Controller Model TZN4S. Although this is a very versatile piece of equipment, below is a summary of the specifications that are relevant to the present application. For more details the reader may refer to the full catalog of the controller available at www.autonics.com

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature Range</td>
<td>Ambient to 200°C/600°C</td>
</tr>
<tr>
<td>Power Supply</td>
<td>100-240VAC; 50/60Hz</td>
</tr>
<tr>
<td>Display Method</td>
<td>7 Segment LED display</td>
</tr>
<tr>
<td></td>
<td>[Process value (PV):Red, Set value (SV):Green]</td>
</tr>
<tr>
<td>Input Sensor</td>
<td>Thermocouple (Chromel – Alumel)</td>
</tr>
<tr>
<td>Control Method</td>
<td>PID, PIDF, PIDS</td>
</tr>
<tr>
<td>Display Accuracy</td>
<td>± 0.3%</td>
</tr>
<tr>
<td>Setting Type</td>
<td>Setting by front push buttons</td>
</tr>
<tr>
<td>Proportional Band (P)</td>
<td>0 to 100.0%</td>
</tr>
<tr>
<td>Integral Time (I)</td>
<td>0 to 3600 Sec</td>
</tr>
<tr>
<td>Derivative Time (D)</td>
<td>0 to 3600 Sec</td>
</tr>
<tr>
<td>Control Time (T)</td>
<td>1 to 120 Sec</td>
</tr>
<tr>
<td>Sampling Time</td>
<td>0.5 Sec</td>
</tr>
<tr>
<td>Setting (P, I &amp; D)</td>
<td>Manual / Auto-tuned</td>
</tr>
</tbody>
</table>
Controls

1. OVEN SELECTOR Switch – to select between the smaller 200 °C or larger 600 °C ovens. Select 200°C for the small oven used in this experiment.

2. POWER CONNECTORS – a 3-pin round for small 200°C oven and two sockets for the large 600°C oven

3. SENSOR CONNECTOR – Common thermocouple input for both ovens

4. OVEN ON-OFF switches – for individual oven with its own indicator

5. PID TEMPERATURE CONTROLLER – for setting, displaying and controlling the temperature of the oven used. Details shown in Fig.2 above

6. MAINS SWITCH – for connecting the mains power to the unit

2. Constant Current Source, Model: CCS-01
(for low resistivity to medium resistivity samples)

It is an 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 preset maximum value. Variations in the current are achieved by a potentiometer included for that purpose. The supply is a highly regulated and practically ripples free d.c. source. The constant current source is suitable for the resistivity measurement of thin films of metals/ alloys and semiconductors like germanium.

Specification

- Open Circuit Voltage: 10V
- Current Range: 0-20mA, 0-200mA
- Resolution: 10µA
- Accuracy: ± 0.25% of the reading ± 1 digit
- Display: 3½ digit, 7 segment LED with auto polarity and decimal indication
- Load Regulation: 0.03% for 0 to full load
- Line Regulation: 0.05% for 10% changes
Controls

1) **Range Switch** – The current meter can be switched between 20mA and 200mA range using this switch. Keep the range switch at the desired range and set the desired current using the current control knob. In case the meter shows over ranging (sign of 1 on the left and all other digits goes blank) range switch maybe shifted to higher range.

2) **Panel Meter** – Display the current in mA.

3) **Current Control** – This is to feed the desired current in the Sample.

4) **Current Output** – Connect suitable connector from Four probe Arrangement in this connector. This will enable the unit to feed desired current in the sample

5) **ON-OFF switch** – To power the unit ON/ OFF.

---

### Fig. 4: Low Current Source, LCS-02

---

3. **Low Current Source, Model : LCS-02**  
*(for high resistivity samples)*

Low Constant Current Sources are needed when the sample resistance, either inherently or due to contact resistances, is large. These include the resistivity measurement of silicon wafers or high resistivity film deposits. Large values of the sample resistance make the measurement prone to noise pick-up from the mains and elsewhere. This is one of the most significant problems of high resistance measurement.

In the present unit the problem of pick-up has been reduced to very low levels by having a battery operated source. Since the current requirement is small and the circuit being specially designed for this purpose, the batteries should have a reasonably long life. Further, a transistor circuit has been preferred over an Op-Amp based circuit as it offers a reduction of the battery count and is also simpler. An internal voltage reference of 2.5 volt ensures reliable operation even when the battery voltage falls and a ten turn potentiometer makes the current adjustment very easy. The actual current is read on a 3½ digit LCD display. There are two current ranges, which may be selected with the help of a switch on the panel.

### Specification

- **Open Circuit Voltage** : 18V
- **Current Range** : 0-2µA, 0-20µA, 0-200µA, 0-2mA
- **Resolution** : 1nA at 0-2µA range
Accuracy : ± 0.25% of the reading ± 1 digit
Display : 3½ digit, 7 segment LCD with auto polarity and decimal indication
Load Regulation : 0.05% for 0 to full load
Power : 3 x 9V batteries

Controls
(1) **Range Switch** – The current meter can be switched between 2µA, 20µA, 200µA and 2mA range using this switch. Keep the range switch at the desired range and set the desired current using the current control knob. In case the meter shows over ranging (sign of 1 on the left and all other digits goes blank) range switch maybe shifted to higher range.

(2) **Panel Meter** – Display the current in µA/ mA (as per setting of Range Switch)

(3) **Current Control** – This is to feed the desired current in the Sample.

(4) **Current Output** – Connect suitable connector from Four probe Arrangement in this connector. This will enable the unit to feed the desired current in the sample

(5) **ON-OFF Switch** – To power the unit ON/ OFF.

*Note: Please note that this unit is operated on 9V x 3 batteries. In case there is any problem in operation, please check the batteries also. Batteries are assessable after opening the Top Cover of the unit.*

**Fig. 5: Digitral Microvoltmeter, DMV-001**
1. **D.C. Microvoltmeter, Model DMV-001**

   Digital Microvoltmeter, DMV-001 is a very versatile multipurpose instrument for the measurement of low dc voltage. It has 5 decade ranges from 1mV to 10V with 100% over-ranging. For better accuracy and convenience, readings are directly obtained on 3½ digit DPM.

   This instrument uses a very well designed chopper stabilized IC amplifier. This amplifier offers exceptionally low offset voltage and input bias parameters, combined with excellent speed characteristics.

   Filter circuit is provided to reduce the line pickups of 50 Hz. All internal power supplies are IC regulated.

**Specification**

- **Range**: 1mV, 10mV, 100mV, 1V & 10V with 100% over-ranging
- **Resolution**: 1µV
- **Accuracy**: ±0.2%
- **Stability**: Within ±1 digit
- **Input Impedance**: >1000MΩ (10MΩ on 10V range)
- **Display**: 3½ digit, 7 segment LED with auto polarity and decimal indication

**Controls**

1. **Range Switch** – The voltmeter can be switched between 1mV, 10mV, 100mV, 1V & 10V range using this switch. Keep the range switch at lowest range for better accuracy. In case the meter shows over ranging (sign of 1 on the left and all other digits goes blank) range switch maybe shifted to higher range.

2. **Panel Meter** – Display the Voltage in mV/V (as per setting of Range Switch)

3. **Zero Adj. Knob** – This is to adjust Zero of Microvoltmeter before starting the experiment.

4. **Voltage Input** – Connect suitable connector from Four probe Arrangement in this connector. This will enable the unit to measure the voltage output of the sample

5. **ON-OFF switch** – To power the unit ON/OFF.

2. **Four Probes Arrangement**

   It has four individually spring loaded probes. The probes are collinear and equally spaced. The probes are mounted in a teflon bush, which ensure a good electrical insulation between the probes. A teflon spacer near the tips is also provided to keep the probes at 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 PID Controlled Oven. Proper leads are provided for current, Voltage & Temp. measurement with their universal connectors. For current measurement there is three pin connector which can be connected to the CCS-01/ LCS-02 as per requirement of sample. For voltage measurement BNC connector is used connected to DMV-001 unit. For temperature measurement, a two pin connector is provided for connection with PID- Controlled oven unit PID-200 at connector marked as Temperature Sensor.
Three levelling screws are provided in Four Probe arrangement by which we can adjust the level of platform to make it horizontal. A probe holding screw is provided at the collar of the arrangement. Initially it should be in loose position, to allow free movement of Probe Pipe. After placing the sample the Probe Pipe should be lowered so that all four pins touches the sample. Further Press the pipe very lightly so that the assured firm contact is made of all Four Pins with the sample. Tighten the Probe Holding Screw at this position. The Probe Arrangement is ready with the sample for the experiment.

**APPARATUS**

1. PID Controller with a Oven Unit, Model PID-TZ
2. Constant Current Sources:-
   a) Constant Current Source, Model CCS-01
   b) Low Current Source, Model LCS-02
3. D.C. Microvoltmeter, Model DMV-001
4. Four Probe Arrangement with Thermocouple sensor and suitable connectors for DMV and CCS/ LCS.
5. Set of test samples and emery powder.
BASIC THEORY

Four sharp probes are placed on a flat surface of the material to be measured (Fig. 7). The 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, it 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.

The experimental circuit used for measurement is illustrated schematically in Fig. 8. A nominal value of probe spacing, which has been found satisfactory, is an equal distance of 2.0 mm between adjacent probes.

In order to use the four-probe method, it is assumed 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 has a high recombination rate, such as mechanical by lapped surfaces).
3. The surface on which the probes rest is flat with no surface leakage.
4. The four probes used for resistivity measurements are equally spaced and collinear.
5. The diameter of the contact between the metallic probes and the semiconductor should be small compared to the distance between probes.
6. The surfaces of the material may be either conducting or non-conducting.
   A conducting boundary (such as copper) is one on which the sample is plated or placed. A non-conducting boundary is produced when the surface of the sample is in contact with an insulator.

Fig. 7: Model for the four probe resistivity measurement
CASE 1 - RESISTIVITY MEASUREMENTS ON A LARGE SAMPLE

One added boundary condition is required to treat this case namely, the probes are far from any of the other surfaces of the sample and the sample can thus be considered a semi-infinite volume of uniform resistivity material. Fig. 7 shows the geometry of this case. Four probes are spaced $S_1$, $S_2$ and $S_3$ apart. Current $I$ is passed through the outer probes (1 and 4) and the floating potential $V$ is measured across the inner pair of probes 2 and 3.

The floating potential $V_f$ a distance $r$ from an electrode carrying a current $I$ in a material of resistivity $\rho_0$ is given by

$$V_f = \frac{\rho_0 I}{2\pi r}$$

In the model shown in Fig. 7 there are two current-carrying electrodes, numbered 1 and 4, and the floating potential $V_f$, at any Y point in the semiconductor is the difference between the potential induced by each of the electrodes, since they carry currents of equal magnitude but in opposite directions Thus:

$$V_f = \frac{\rho_0 I}{2\pi} \left( \frac{1}{r_1} - \frac{1}{r_4} \right)$$

(1)

where $r_1 = \text{distance from probe number 1}$ and $r_4 = \text{distance from probe number 4}$.

The floating potentials at probe 2, $V_{f2}$, and at probe 3, $V_{f3}$ can be calculated from (1) by substituting the proper distances as follows:

$$V_{f2} = \frac{\rho_0 I}{2\pi} \left( \frac{1}{S_1} - \frac{1}{S_2+S_3} \right)$$

$$V_{f3} = \frac{\rho_0 I}{2\pi} \left( \frac{1}{S_1+S_2} - \frac{1}{S_3} \right)$$

The potential difference $V$ between probes 2 and 3 is then

$$V = V_{f2} - V_{f3} = \frac{\rho_0 I}{2\pi} \left( \frac{1}{S_1} + \frac{1}{S_3} - \frac{1}{S_2+S_3} - \frac{1}{S_1+S_2} \right)$$

Fig. 8: Circuit used for resistivity measurement
and the resistivity \( \rho_0 \) is computable as

\[
\rho_0 = \frac{V}{I} \left( \frac{2\pi}{\frac{1}{S_1} + \frac{1}{S_2} - \frac{1}{S_1 + S_2} - \frac{1}{S_2 + S_3}} \right)
\]  \hspace{1cm} (2)

Fig. 9: Images for the case of the resistivity probes on a slice with conducting bottom surface

Fig. 10: \( G_6 \) (W/S) for probes on a thin slice with a conducting bottom surface
When the point spacings are equal, that is, $S_1 = S_2 = S_3 = S$ the above simplifies to:

$$\rho_0 = \frac{V}{I} \times 2\pi S$$  \hspace{1cm} (3)

**CASE 2- RESISTIVITY MEASUREMENTS ON A THIN SLICE-CONDUCTING BOTTOM SURFACE**

Two boundary conditions must be met in this case; the top surface of the slice must be a reflecting (non-conducting) surface and the bottom surface must be an absorbing (conducting) surface. Since the two boundaries are parallel, a solution by the method of images requires for each current source an infinite series of images along a line normal to the plane and passing through the current source.

The model for this case is shown in Fig. 9. The side surface of the slice is assumed to be far from the area of measurement and, therefore, only the effect of the bottom surface needs to be considered. In this analysis equal probe spacing $S$ shall be assumed. The width of the slice is $W$. The array of images needed is indicated in Fig. 9, where the polarity and spacing of the first few images are as shown.

The floating potential $V_{f2}$ at electrodes 2 is

$$V_{f2} = \frac{rI}{2\pi} \left[ \sum_{n=-\infty}^{\infty} (-1)^n \frac{1}{\sqrt{S^2 + (2nW)^2}} - \sum_{n=-\infty}^{\infty} (-1)^n \frac{1}{\sqrt{(2S)^2 + (2nW)^2}} \right]$$ \hspace{1cm} (4)

Likewise, the floating potential at electrode (3) can be obtained and

$$V = \frac{\rho I}{2\pi} \left[ \frac{1}{S} + \sum_{n=1}^{\infty} (-1)^n \frac{4}{\sqrt{S^2 + (2nW)^2}} - \sum_{n=1}^{\infty} (-1)^n \frac{4}{\sqrt{(2S)^2 + (2nW)^2}} \right]$$ \hspace{1cm} (5)

The resistivity then becomes

$$\rho = \frac{\rho_0}{G_6 \left( \frac{W}{S} \right)}$$ \hspace{1cm} (6)

Where resistivity $\rho_0$ is computable from (2, and 3) can be used if the point spacing are different, but approximately equal. The function $G_6 \left( \frac{W}{S} \right)$ is computed from

$$G_6 \left( \frac{W}{S} \right) = 1 + 4 \frac{S}{W} \sum_{n=1}^{\infty} (-1)^n \left[ \frac{1}{\sqrt{\left( \frac{S}{W} \right)^2 + (2n)^2}} - \frac{1}{\sqrt{\left( \frac{2S}{W} \right)^2 + (2n)^2}} \right]$$ \hspace{1cm} (7)

which is tabulated in Table I and plotted in Fig. 10.
Fig. 11: $G_7$ (W/S) for probes on a thin slice with a non-conducting bottom surface
### TABLE - I

<table>
<thead>
<tr>
<th>S. No.</th>
<th>W/S</th>
<th>G₆ (W/S)</th>
<th>G₇ (W/S)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.100</td>
<td>0.0000019</td>
<td>13.863</td>
</tr>
<tr>
<td>2</td>
<td>0.141</td>
<td>0.00018</td>
<td>9.704</td>
</tr>
<tr>
<td>3</td>
<td>0.200</td>
<td>0.00342</td>
<td>6.931</td>
</tr>
<tr>
<td>4</td>
<td>0.33</td>
<td>0.0604</td>
<td>4.159</td>
</tr>
<tr>
<td>5</td>
<td>0.500</td>
<td>0.228</td>
<td>2.780</td>
</tr>
<tr>
<td>6</td>
<td>1.000</td>
<td>0.683</td>
<td>1.504</td>
</tr>
<tr>
<td>7</td>
<td>1.414</td>
<td>0.848</td>
<td>1.223</td>
</tr>
<tr>
<td>8</td>
<td>2.000</td>
<td>0.933</td>
<td>1.094</td>
</tr>
<tr>
<td>9</td>
<td>3.333</td>
<td>0.9838</td>
<td>1.0228</td>
</tr>
<tr>
<td>10</td>
<td>5.000</td>
<td>0.9948</td>
<td>1.0070</td>
</tr>
<tr>
<td>11</td>
<td>10.000</td>
<td>0.9993</td>
<td>1.00045</td>
</tr>
</tbody>
</table>

### CASE 3 RESISTIVITY MEASUREMENTS ON A THIN SLICE-NON-CONDUCTING BOTTOM SURFACE.

The model for these measurements is like the case 2, except that the bottom surface of the slice is nonconducting. This means that all the images of Fig. 3 have the same charge as the current source. Thus all the images on a row have equal charges and Eq. 5 describes the potential difference across the inner pair of probes, if $(-1)^n$ is removed from the equation. Then,

$$\rho = \frac{\rho_0}{G_7(W/S)},$$  \hspace{1cm} (8)

where

$$G_7(W/S) = 1 + 4 \sum_{n=1}^{\infty} \frac{S}{W} n \left[ \frac{1}{\sqrt{\left(\frac{S}{W}\right)^2 + (n)^2}} - \frac{1}{\sqrt{\left(\frac{2S}{W}\right)^2 + (2n)^2}} \right]$$  \hspace{1cm} (9)

This function $G_7(W/S)$ is tabulated in Table I. and plotted in Fig. 11. For smaller values of W/S the function $G_7(W/S)$ approaches the case for an infinitely thin slice, or

$$G_7\left(\frac{W}{S}\right) = \frac{2S}{W} \log_e 2$$  \hspace{1cm} (10)

Thus for sample $W/S < 0.25$ or sample thickness upto 0.5mm, the correction factor may be obtained from equation (10) directly.
DIGITAL MICROVOLTMETER
Model: DMV-001
ON
RANGE
1 mV
10 mV
1 V
10 V
100 mV
ZERO ADJ

CONSTANT CURRENT SOURCE
Model: CCS-01
ON
CURRENT
20 mA
200 mA

LOW CURRENT SOURCE
Model: LCS-02
ON
CURRENT
ADJ.
RANGE
2 µA
20 µA
200 µA
2 mA

PID CONTROLLED OVEN
Model: PID-TZ
Temp. Sensor
OVEN-600
ON
OVEN-800
ON

Fig. 12: Connection diagram for the set up

Fig. 13: Complete experimental set up
PROCEDURE

Before starting the experiment, make sure that the surface of your sample is clean and conducting. Particularly in case of Germanium and Silicon, an oxidation layer is formed which causes error in readings. Therefore for such samples, it is advisable to remove this oxidation layer by rubbing the sample in fine emery slurry on a glass surface for 2 – 3 minutes. The sample will be ready to use after washing it in plane water and subsequent drying.

(I) Measurement of resistivity of n-Si and Al at room temperature:
1. Put the sample (n-Si/Al) on the base plate of the four probe arrangement. Unscrew the pipe holding the four probes and slowly let the four probes rest at the middle of the sample. Apply a very gentle pressure on the probes and tighten the pipe in this position.
   **CAUTION**: Use only the minimum pressure required for proper electrical contacts particularly for semiconductor wafers and thin films.
2. Connect the outer pair of probes to the constant current source through 3-pin socket and the inner pair to the D.C. Microvoltmeter through an BNC connector.
3. Place the Four Probe Arrangement in the oven.
4. Switch ‘ON’ D.C. Microvoltmeter and allow about 5 minutes time for thermal stability. Adjust the zero of D.C. Microvoltmeter with the knob provided for it.
5. Switch ‘ON’ the Constant Current Power Supply (For Ge and Al samples)/ Low Current Power Supply (For Si sample) and check the zero reading of D.C. Microvoltmeter at zero current reading again. Adjust it, if required. In case it can not be adjusted (this may happen in highly resistive samples), note it down and treat it as zero error.
6. Increase the current gradually and note the corresponding voltage readings. Subtract the zero reading if any and check for Ohm’s Law \( \frac{V}{I} = R \). This would ensure that the system is working properly and the systematic readings could be taken.

(II) Measurement of resistivity of n-Ge at various temperatures:
7. Put n-Ge sample in the four probe arrangement following the procedure described in (I). The only difference here is that the current will be fixed (say at 5mA) and temperature is varied.
8. For different temperature settings, the PID Controlled Oven should be used. It stabilizes the temperature at the desired value while recording data.
   *The PID parameters are already set to a reasonable level \( (P = 3.0; I = 350; D = 87) \) for immediate operation of the unit in a range 80°C to 200°C. To operate the system one only needs to set the desired final temperature as described below. For other temperatures the parameters are to be set again.*

Set the PID Controlled oven PID-TZ as follows:
   (i) Connect the Thermocouple sensor’s 2-pin socket of Four Probe Arrangement and the oven socket to the PID Controller. Select the 200°C oven, switch the MAINS ON, and switch the oven ON. Now RUN the controller.
   (ii) Set a desired temperature for the first reading as described below.
Step.1  Connect the controller and the sensor, switch ON the unit. The oven should be OFF.

Step.2  The upper display would show the present oven temperature (PV or process value) and the lower display shows the current setting of the oven temperature (SV or set value).

Step.3  Set the desired temperature by using the keys <, ^, and v, taking care of the maximum temperature allowed for the oven connected.

Step.4  Press MD key to return to the RUN mode. The oven may now be switched ON and operated in the usual manner. The oven temperature at any instant would be seen on the upper display.

(iii)  Allow about 5 minutes time to stabilize the circuit. The Process Value (PV) will slowly rise and become steady.

(iv)   Record the voltage readings for the set temperature.

(v)    Set the temperature for the next reading and repeat (ii) and (iii) above. Continue till the highest temperature needed for the experiment is reached. This must be <200°C for the safety of the four probe arrangement as well as the oven.

(vi)   Plot a graph for Log_{10} \rho \sim (1/T) and calculate the energy band gap.

(vii)  When completed switch the oven and MAINS OFF. Allow the oven to cool down for the next experiment.

OBSERVATIONS

Table 1: Data for resistivity of Al foil

<table>
<thead>
<tr>
<th>No. of commercial Al foils:</th>
<th>16</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness of one foil:</td>
<td>0.001 cm</td>
</tr>
<tr>
<td>Thickness of Stack:</td>
<td>0.16 ± 0.01 mm</td>
</tr>
<tr>
<td>Probe distance (S):</td>
<td>0.200 ± 2% cm (fixed)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Current (I)</th>
<th>Voltage (volts)</th>
<th>V/I</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Data for resistivity of n-Si

| Thickness (W): | 0.50 ± 2% mm |

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Current (I)</th>
<th>Voltage (volts)</th>
<th>V/I</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 2: Data for resistivity ~ Temperature of n-Ge

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Temp (T in K)</th>
<th>Voltage (volts)</th>
<th>$\rho$ ((\Omega\cdot\text{cm.}))</th>
<th>$T^{-1} \times 10^{-3}$ (K(^{-1}))</th>
<th>Log(_{10}\rho$</th>
</tr>
</thead>
</table>

**CALCULATIONS AND GRAPH**

From Eq. (3)

$$\rho_0 = \frac{V}{I} \times 2\pi S$$

**Correction Factor:**

Since the thickness of the samples are 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, Eq. (9) will be applied.

$$\rho = \frac{\rho_0}{G_\gamma(W/S)}$$

The function $G_\gamma(W/S)$ may be obtained from Table-I or Fig. 5 for the appropriate value of (W/S). For sample W/S<0.25, correction factor may be obtained directly from Eqn. 10. Thus $\rho$ may be calculated for various temperatures.

**Energy Band Gap**

We know, $E_g = 2k \frac{\log_e \rho}{1/T}$

Where $k$ is the Boltzamann's constant = $8.6 \times 10^{-5}$ eV/deg & $T$ is temperature in kelvin. Use the graph to determine $E_g$.  

**Thickness (W):** 0.50 ± 2% mm

**Probe current (I) =** 5 mA (fixed)
Determination of the band gap of a Ge Sample

Fig. 14: Sample Graph of $\log_{10} \rho \sim (1/T)$ for n-Ge sample
Typical values of samples:

Standard value for pure Aluminium = $2.8 \times 10^{-6}$ ohm.cm.

Typical value for n-Si (as supplied by manufacturer) = $(6\pm1)$ ohm.cm.

Typical value for n-Ge at 303K (as per datasheet of manufacturer) = 18.09 ohm.cm.

Typical value of band gap for n-Ge at 303K (as per datasheet of manufacturer) = 0.68eV.

The results given above are only for reference, exact values depend upon the sample and may not replicate.

SOME USEFUL POINTS

1. Very high resistance (> 1MΩ) may cause high off-set in D.C. Microvoltmeter which cannot be adjusted with the knob provided - Note it down and treat it zero error.

2. Unstable voltage reading may be due to improper contacts of probes with the sample.
   - (a) This may be due to insulating layer on the sample - Clean it.
   - (b) It may be due to loose contacts of probes with the sample - tighten the springs of 4-probes (1/2 thread only)
   - (c) Check the earth point and see that the whole system, constant current power supply, D.C. Microvoltmeter and Four Probe Arrangement are properly grounded.

3. In cases of semiconductor, like Si though the resistivity of sample may not be high but because of contact resistance, its effective resistance is usually very high.

Sources of Error

1. Material (Al) used in the foil is commercial grade, while standard resistance is for pure Al.
2. The thickness of Al foil is very small and there could be error of ±7% in measurement of thickness.
3. The formula for $\rho$ is valid for semi-infinitely large /very large surface in comparison with the probe distance.
4. Variation of doping in the sample

REFERENCES