CHAPTER : 2

EXPERIMENTAL TECHNIQUES
2.1 INTRODUCTION:

For recording observations during the present investigation different experimental techniques like X-ray Diffraction, Energy Dispersive Analysis of X-rays, UV-VIS-NIR spectroscopy, Resistivity measurements, Hall effect measurements, Thermoelectric power measurements, optical microscopy and capacitance measurements, have been used. A brief account of these techniques, experimental details and apparatus used is given in this Chapter.

2.2 X-RAY POWDER DIFFRACTION (XRD):

X-ray diffraction is a very important technique that has long been used to address all issues related to the crystal structure of bulk solids, including lattice constants and geometry, identification of unknown materials, orientation of single crystals and preferred orientation of polycrystals, defects and stresses etc.

A diffractometer measures the intensity of X-rays reflected from one stack of planes at a time, so that it is of interest to examine what kind of spatial arrangements are best suited for such measurements. The intensity of a diffracted beam is measured directly by an electronic counter. There are many types of counters, but they all convert incoming X-rays into pulses of electric current in the circuit connected to the counter. This circuit counts the number of current pulses/sec and this number is directly proportional to the
intensity of the X-ray beam entering the counter or detector. A diffractometer is designed somewhat like a Debye-Scherrer camera, except that a movable counter replaces the strip of film. In both instruments, essentially monochromatic radiation is used and the X-ray detector (film or counter) is placed on the circumference of a circle centered on the powder specimen. The essential features of a diffractometer are shown in Fig. 2.1[1].

A powder specimen C, in the form of a flat plate, is supported on a table H, which can be rotated about an axis O perpendicular to the plane of the drawing. The X-ray source is S, the line focal spot on the target T of the X-ray tube; S is also normal to the plane of the drawing and therefore parallel to the diffractometer axis O. X-rays diverge from this source and are diffracted by the specimen to form a convergent diffracted beam which comes to a focus at the slit F and then enters the counter G. A and B are special slits which define and collimate the incident and diffracted beams.

The receiving slits and counter are supported on the carriage E, which may be rotated about the axis O and whose angular position 2θ may be read on the graduated scale K. The supports E and H are mechanically coupled so that a rotation of the counter through 2x degrees is automatically accompanied by rotation of the specimen through x degrees. This coupling ensures that the angle of incidence on, and reflection from, the flat specimen will always be equal to one another and equal to half the total angle of diffraction, an arrangement necessary to preserve focusing
Fig. 2.1 Schematic features of a X-ray diffractometer
conditions. The counter may be power-driven at a constant angular velocity about the diffractometer axis or moved by hand to any desired angular position.

For obtaining the diffractogram from a polycrystalline or powdered sample using the above set up, the counter is set near \( \theta = 0^\circ \), and connected to a counting-rate meter. The output of this circuit is fed to a strip-chart recorder. The counter is then driven at a constant angular velocity through increasing values of \( \theta \) until the whole angular range is "scanned". At the same time, the paper chart on the recorder moves at a constant speed, so that distances along the length of the chart are proportional to \( \theta \). This results in a diffractogram showing a record of counts per second (proportional to diffracted intensity) versus diffraction angle \( \theta \).

2.3 ENERGY DISPERSIVE ANALYSIS OF X-RAYS (EDAX):

Most scanning / transmission electron microscopes and electron microprobes are equipped with energy dispersive spectrometer (EDS) facilities. The EDS system is capable of identifying elements with atomic number \( (Z) \geq 11 \) in a few minutes. Chapter 7 of [2] has dealt with EDS system in detail. Only a brief review of element identification with EDS facilities will be given here.

The EDS detector collects the entire X-ray spectrum and a multichannel analyzer calibrated so that each channel corresponding to
some convenient energy, e.g. 25 or 50 eV per channel, is used to divide the spectrum into energy packets. Moseley's law relates the characteristic energy of an X-ray peak and the atomic number of the element responsible for the peak. Hence, by determining the channel numbers (energy) of peaks in the spectrum, appropriate atomic numbers can be assigned for the elements present in the electron-irradiated region. Because of X-ray absorption in the detector window (usually Beryllium sheet about 10 μm thick) and in the lithium drifted silicon chip itself, elements with atomic number Z less than ten are not usually identified or analyzed energy-dispersively. In addition, the characteristic energies of these elements are only about 100 - 150 eV apart. The vast majority of present day energy-dispersive analysis equipment cannot cleanly resolve peaks of adjacent elements in the range $4 \leq Z \leq 11$. Therefore, energy dispersive analysis is useful for identification and analysis of elements with $Z \geq 11$.

Instrumentation can be made to indicate what elements are detected in a given spectrum and to display the information., thus saving the investigator's the trouble. A way to do this is to determine the coefficients for $K_\alpha$, $K_\beta$, $L_\alpha$, $L_\beta$, $L_\gamma$ and $M_{\alpha,\beta}$ peaks for the particular calibration used in the multichannel analyzer i.e.

$$Z = \exp \left[ A \left( \ln C_n + B \right) \right]$$

where $C_n$ is the peak channel number and A and B are numerical coefficients depending on the calibration.
Above equation is a way of stating Moseley's law. A parabola can be fitted to the peaks to obtain $C_n$ using three or more data points to make the fit. Hence $Z$ can be determined automatically. Ambiguities can be reduced by checking for $\beta$ - series peaks for both the K and L lines of a given element being present in the spectrum.

Subtraction of the background intensity in energy dispersive analysis must be carried out accurately if elements present in less than 1000 ppm concentrations are to be properly identified.

In summary, elements with $Z \geq 11$ can be readily identified with energy dispersive analysis equipment, usually in a few minutes or even less. For elements present in trace amounts, longer counting times are necessary but instrument stability limits counting time to 10 or 15 minutes in practice.

2.4 UV-VIS-NIR SPECTROPHOTOMETER:

Beckman Model DK-A spectrophotometer provides a means for analyzing liquids, gases and solids through the use of radiant energy in the far and near ultraviolet, visible and near infrared regions of the electromagnetic spectrum. Analytical information can be revealed in terms of transmittance, absorbance or reflectance of energy in the wavelength range between 160 and 2500 millimicrons.
Model DK-A (Fig. 2.2) instrument utilizes a single beam of energy, which is chopped into alternate reference and sample beams to provide a double beam system within the sample compartment. Both sample and reference beams have common detection and amplification components.

Qualitative and quantitative absorption data are obtained with the Model DK-A spectrophotometer by directing radiation into a sample and determining what portion of the radiant energy is absorbed. Two radiation sources are necessary, since there is no single source that will provide energy over the entire wavelength range of the Model DK-A instrument. Radiation is provided in the wavelength range below 375 millimicron by a hydrogen lamp, the tungsten lamp is used in the 320-3500 millimicrons wavelength range.

From the source, radiation enters the optical system. Fig.2.3 shows the path followed by a single ray within the radiation beam. The beam is reflected from the condensing mirror (A) to the slit entrance mirror (B), which directs the beam to the chopper (C). The chopped beam passes through the adjustable entrance slit (D) and into the monochromator. The beam is reflected from the collimating mirror (E) in parallel rays through a reflecting quartz prism (F) which disperses the beam into its spectrum of successive wave lengths. The back surface of the prism is aluminized so that the beam is reflected back through the prism and further dispersed as it emerges. Rotation of the prism relative to collimating mirror changes the
Fig. 2.2 The model DK – 2A spectrophotometer.
Fig. 2.3 Optical diagram of spectrophotometer
angle of incidence and enables selection of a particular group of wavelengths that comprises a spectral band. This band of radiation is directed back to the collimating mirror, which focuses the entrance slit image on the exit slit (G). Upon passing from the monochromator, the radiant energy is directed by lens (H) into the double beam optical system in the sample compartment.

The model DK-A instrument is double beam ratio recording spectrophotometer, i.e. radiant energies transmitted by the reference and sample beams (J and M) are compared, and the ratio of the sample energy to the reference energy is recorded as a percent transmission. The double beam optical system consists of two synchronized semi-circular rotating mirrors (I and N), and two stationary mirrors (L and K) in the sample compartment. The rotating mirrors consequently deflect and pass the radiant energy so that it is directed alternatively into sample and reference cells fifteen (or 12.5) times per second. Energy transmitted by the sample and reference cells is focused by the detector selector mirror (O) onto the detector.

Detection of transmitted radiant energy requires two detectors to cover the entire wavelength range of the instrument. One - the lead sulphide cell (P) - responds in the region between 400 to 3500 millimicron. For measurements in the wavelength range extending below 700 nm, a photomultiplier tube (Q) is used. Radiant energy that strikes the detector is converted to a proportional alternating current signal.
For obtaining the absorption spectra using UV-VIS-NIR spectrophotometer from single crystal specimens, thin flakes of approximately 0.5 mm thickness are used. These flakes are pasted on a thick black paper with a cut exposing the crystal flake to the incident light. The reference used is a replica of the black paper, having the cut in exactly the same position as the crystal flake. This arrangement is necessary because the crystal size is smaller than that of the sample compartment. Blank glass slides are also used as replica. For reflectance measurement standard aluminium coated mirror is used as reference.

2.5 RESISTIVITY MEASUREMENTS:

The resistivity measurements in the present investigation were made perpendicular to c-axis (along the basal plane) and parallel to c-axis (normal to the basal plane) by the techniques described below:

2.5.1 RESISTIVITY MEASUREMENTS PERPENDICULAR TO c-AXIS

(ALONG THE BASAL PLANE) \( (\rho_p) \):

The electrical resistivity of the samples perpendicular to c-axis (along the basal plane) can be investigated by the following techniques:

(a) van der Pauw technique

(b) Four probe resistivity method
The resistivity of the specimens can be measured at different temperatures by these methods.

(I) Low Temperature
    (a) 1 K-303 K
    (b) 77 K-303 K

(ii) High Temperature
    (a) 303 K-473 K
    (b) 303 K-973 K

2.5.1.1 van der Pauw Technique:

The crystals having irregular shape and larger size were normally selected for investigating their electrical behaviour through this method. The basic requirements of the method are:

(I) The contacts should be at the periphery of the sample,

(ii) The size of the contacts should be negligibly small as compared to that of the sample,

(iii) The contacts should be ohmic,

(iv) The sample should be very thin as compared to its area,

(v) Surface of the sample should be singly connected, i.e. the sample should be free from steps and discontinuities,

(vi) The sample should be flat so that the sample surface and contacts lie in the same plane and
(vii) The applied field should be low so that break down does not occur.

If the contacts are not taken perfectly, the observed results may be associated with some error. This error will be proportional to the dimensions of the contact and can be given as:

$$\frac{\Delta \rho}{\rho} \propto \frac{d^2}{D^2}$$

where $\rho$ is the resistivity of the sample, $\Delta \rho$ is the change in resistivity, $d$ is the size of the contact and $D$ is the size of the sample.

The extent of this error depends upon

(i) Distance of contact from circumference,

(ii) Extension of contact on the surface and

(iii) Distance between contacts.

The general circuit diagram used to measure the resistivity of the crystals (along their basal plane) is shown in Fig. 2.4.

The resistance $R_{AB,CD} = R_1$ can be defined as

$$R_1 = \frac{\text{Potential difference between contacts } D \text{ and } C}{\text{Current through contacts } A \text{ and } B}$$

or as the potential difference $V_D - V_C$ per unit current flowing between points A and B. Here, the current enters the sample through contact A and leaves it through contact B.
Fig. 2.4 Circuit arrangement for resistivity measurements through Van der Pauw method
Similarly, resistance \( R_{bc,da} = R_2 \) can be defined as,

\[
R_2 = \frac{\text{Potential difference between contacts A and D}}{\text{Current through contacts B and C}}
\]

It is observed that the equation

\[
\exp(\frac{-\pi R_1 d}{\rho}) + \exp(\frac{-\pi R_2 d}{\rho}) = 1
\]

holds good for semiconductors. Here \( \rho \) is the specific resistivity of the semiconducting material and \( d \) is thickness of the sample.

Simplification of above equation yields

\[
\rho = \frac{\pi d}{2 \ln 2} \left( R_1 + R_2 \right) f\left(\frac{R_1}{R_2}\right)
\]

Fig. 2.5 shows the relationship between \( R_1/R_2 \) and \( f(R_1/R_2) \).

2.5.1.2 Low Temperature (Liquid Helium - 303 K) Resistivity Measurements by Four Probe Method with van der Pauw and Montgomery Configurations:

A fully automated precise electrical resistance measurement system for more than one sample has been constructed by Marhas et al [3] at the Inter-University Consortium for DAE facilities at Indore. Conventional four-probe measurements with van der Pauw and Montgomery configurations are possible with this system. Resistance measurements in the range of a few \( \mu \Omega \) to a few \( G\Omega \) are possible for six samples at a time.
Fig. 2.5 The relation between $R_1/R_2$ and $f(R_1/R_2)$
from room temperature down to liquid helium or liquid nitrogen temperatures with a temperature control accuracy of better than 10 mK. The design features of the system with special reference to the low-noise switching methods of currents and voltages are described in detail in the paper [3].

2.5.1.3 Low Temperature (77 - 303 K) Resistivity Measurements by Four Probe Method:

The method discussed below has been used for resistivity measurements in the temperature range (77 to 303 K). The cryostat LN-DP involved in this measurement is designed specially for the experiments in temperature range 77 to 303 K. It consists of a sample chamber and a sample holder as shown in Fig.2.6. The system is portable and very easy to manage. It is designed to fit directly into any of the standard liquid Nitrogen (liquid N$_2$) storage containers. The system is supplied with a standard 1/2" valve for evacuating and a 1/4" valve for introducing the exchange gas monitoring vacuum etc.

The sample holder consists of a gold-plated copper assembly, mounted with a standard platinum thermometer (Pt-100), and a heater which can be used for temperature control. Electrical leads for thermometer and the heater are brought at the top of the cryostat at a D-type 9-pin connector. The samples can be mounted on a rectangular projection as shown in Fig. 2.7. Sixteen permanent electrical points are available for the experiments. These are brought out at the top of the cryostat at the D-type
Fig. 2.6 The sample holder used in the low temperature resistivity measurement
Fig. 2.7 The cryostat LN-DP, used for low temperature resistivity measurements.
25 pin connector. At a time four resistivity samples can be mounted on the sample holder. These samples can be mounted with a low temperature conducting Apiezone-N grease.

The four probes for each of the samples are attached using proper solder / silver paste. A low power (10 Watt maximum) soldering rod is used for soldering work near the sample holder (otherwise insulation from pin to pin may break down). The samples are selected using sample changer SMC - 4.

The constant current source CCS -10 is ideal for powering wide range of transducers and also for many other purposes like four probe resistivity measurements where a stable but variable constant current is the basic requirement.

The heater power supply CCH-1000 has been specially designed for use as a heater supply for a 30-40 ohm heater. It is capable of supplying a maximum of 25 watts of power to the heater which is adequate for cryogenic purpose.

The samples are mounted on the sample holder and the electrical connections are done properly. After cleaning the 0 ring joint, the sample holder is inserted in the sample chamber and the clamp is tightened gently. The pumping valve is closed after evacuating the sample chamber to a vacuum of at least 10⁻² torr. The entire assembly is inserted and fixed in a
liquid nitrogen container. The sample holder takes about an hour to cool down to 77 K. For faster cooling it is advisable to introduce some amount of suitable exchange gas in the sample chamber via 1/4" valve. At a time four different specimens can be mounted on the sample holder. The temperature of these specimens is raised from 77 K to room temperature in steps of 10 K and the developed voltage across their inner leads is measured, for a constant current passed through the outer leads of the specimen. The block diagram of the experimental set up is shown in Fig. 2.8 and the experimental set up for the technique is shown in Fig. 2.9.

After the experiment, the entire assembly is pulled out and the system is allowed to come to room temperature before the sample chamber is opened.

The voltage is measured at different temperatures. From the values of the voltage and current, the resistance (R) is evaluated at different temperatures. The resistivity at different temperatures is then calculated with the help of the following formula

\[ \rho = \frac{\rho_0}{G} \]

where \( \rho_0 = 2\pi s R \) and \( G = \frac{2s}{\omega} \cdot 2 \ln 2 \)

where \( s \) is the distance between the two probes. Its value is given by the manufacturers as 0.1 cm and \( \omega \) is the thickness of the sample.
Fig. 2.8 Schematic diagram of the low temperature resistivity measurements
Fig. 2.9 Experimental set-up for low temperature resistivity measurements.
2.5.1.4 High Temperature (303 - 473 K) Resistivity Measurements
by Four Probe Method:

Large size crystals having proper shape were used for the study of variation of resistivity with temperature using the four probe method described in this section.

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. An excess concentration of minority carriers will affect the potential of other contacts and modulate the resistance of the material.

The four probe method overcomes the difficulties mentioned above and also offer several other advantages. It permits measurements of resistivity in samples having a wide variety of shapes, including the resistivity of small volumes within the bigger pieces of semiconductor.

The basic model for four probe resistivity measurements is indicated in Fig. 2.10. Four sharp probes are placed on a flat surface of the material whose resistivity is to be measured. Current is passed through the two outer electrodes, and the corresponding potential is measured across the inner probes.
The four probe set up (Scientific Equipments, Roorkee, India) consists of probe arrangements, oven, constant current source, milliameter and electronic voltmeter. A simplified block diagram of the set up is shown in Fig. 2.11. Collinear and equally spaced spring loaded four probes are coated with Zinc (Zn) at the tips to ensure good electrical contact with the sample. These probes are mounted in a teflon bush to insulate them electrically. In addition, a teflon spacer near the tips is also provided to keep the probes equidistant (1.5 mm). The whole arrangement is mounted on a suitable stand from where leads are provided for voltage and current measurements. A small oven has been provided to facilitate measurements at various temperatures ranging from room temperature 303 K to 473 K. A highly regulated constant current generator, specifically designed to provide the required varying current, controllable through a potentiometer (with 100% protection against burn out) has been provided for this purpose.

The resistivity measurements on the samples carried out using this set up, assume that;

(i) The resistivity of the sample is uniform in the area of the measurements,
(ii) The surface on which probes rest is flat;
(iii) The diameter of the contact, between metallic probes and the sample, is very small compared to the distance between the two adjacent probes.
Fig. 2.10  Probe arrangement for the four probe resistivity measurements

Fig. 2.11  Block diagram and circuit arrangement for the four probe setup.
When the length of the specimen is less than 10mm only two probes are used to measure the resistance of the sample at different temperatures. The resistivity at different temperatures is then calculated by using the following formula

\[ \rho = \frac{Rbt}{l} \]

where \( R \) is the resistance, \( b \) is the width of the sample, \( t \) is the thickness of the sample and \( l \) is its length.

If the length of the sample is more than 10mm, its resistance can be measured by using all the four probes. Resistivity at different temperatures is then calculated by using the formula

\[ \rho = \rho_0 / G \]

described earlier in section 2.5.1.3.

2.5.1.5 High Temperature (303 - 973 K) Resistivity Measurements by Four Probe Method:

The four-probe set up VRTM - 408 (Scientific Solutions, Mumbai) is designed to undertake resistivity measurements on small samples from room temperature to 973 K. The system consists of following four main units (Fig. 2.12):

1. Sample holder
2. Furnace
Fig. 2.12 The high temperature four probe resistivity measurement set up VTRM – 408.
3. Furnace controller

4. Constant current source

A sample under measurement is sandwiched between two flat alumina plates. Four thin parallel wires properly positioned on the sample act as probes. Sample holder is placed inside a quartz tube which is placed inside a tubular furnace. Measurements can be conducted with sample in air, low vacuum or flowing gas. Voltage and current leads are brought out of the furnace in a 9-pin D-type connector for further connections.

Sample temperatures can be controlled within ± 1°C in the specified range for time period from few minutes to days, using the matched furnace controller supplied with the system. Furnace temperature can be increased continuously at different sweep rates of 1°C per minute or slower, by selecting the RAMP mode on the furnace controller. A 3 range voltmeter is built-in the furnace controller. All the control and measurement parameters for the experiments can be acquired automatically using a personal computer via RS-232 serial interface supplied with the system.

The data stored in the computer gives the values of resistances (R) at different temperatures. The resistivity \( \rho \) can than be determined with the help of the formula

\[
\rho = \frac{\rho}{G} = \frac{2\pi s R}{\frac{2s}{\omega} X 2 \ln 2}
\]

described earlier.
2.5.2 Resistivity Measurements Parallel to C-Axis
(Normal to the Basal Plane) ($\rho_\parallel$):

The experimental set up for measurements of resistivity normal to the basal plane is shown in Fig. 2.13. The crystal is mounted on the sample holder (Fig. 2.14) which is then inserted into the sample chamber, and then closed from the top.

The temperature of the sample is raised by introducing the sample chamber assembly into a furnace. The temperature of the sample is measured with the help of Cr-AI thermocouple kept in vicinity of the sample. Starting from room temperature (303 K), the temperature of the sample is increased slowly to 703 K, in step of 10 K and at each step the corresponding value of the resistance $R$ of the sample is evaluated. From the values of resistances ($R$) at different temperatures, the resistivity $\rho_\parallel$ is determined by using the formula

$$\rho = \frac{RA}{t}$$

where $A$ is the area of the sample and $t$ is its thickness. To avoid excessive heating of the sample chamber, it is cooled by circulating cold water around it with the help of tubings as shown in the set up (Fig. 2.15)
Fig. 2.13 Experimental set-up for high temperature resistivity measurements.
Fig. 2.14 The sampleholder for the high temperature resistivity measurements.
Fig. 2.15 Cross section of the two probe set up for resistivity measurement parallel to c-axis
2.6 MEASUREMENTS OF HALL PARAMETERS:

2.6.1 HALL EFFECT MEASUREMENTS AT ROOM TEMPERATURE USING MODIFIED VAN DER PAUW TECHNIQUE:

The van der Pauw technique described earlier has been modified to determine the type of conductivity, mobility and carrier concentration of the semiconductor materials. This modification shown in Fig. 2.16 facilitates the measurement of voltage across and currents between the diagonally opposite contacts. The experimental set up for this technique is shown in Fig. 2.17. The sample for the measurement is kept in a known magnetic field produced by an electromagnet (type EMPS-5 Omega Electronic, Jaipur, India). This magnetic field modifies the path of electrons producing the Hall voltage.

Knowing the values of difference in resistance ($\Delta R$), magnetic field ($\Delta B$) and thickness of the sample $t$, the mobility of carriers is evaluated using the equation

$$\mu = \frac{t}{\Delta B} \cdot \frac{\Delta R}{\rho}$$

where $\rho$ is the room temperature resistivity of the sample.

The Hall coefficient and carrier concentration are calculated using the formula

$$R_H = \mu_H \times \rho$$
Fig. 2.16 Circuit arrangement for Hall effect measurements.
Fig. 2.17 Experimental set up for Hall effect measurement.
and for carrier concentration.

\[ n = \frac{1}{R_{\mu} e} \]

where '\( p \)' is the room temperature resistivity of the sample.

From the sign of Hall coefficient, the nature of charge carriers in the grown samples can be ascertained.

2.6.2 LOW TEMPERATURE HALL EFFECT MEASUREMENTS IN THE TEMPERATURE RANGE 77 TO 350 K:

The low temperature Hall effect measurements have been carried out with the help of cryogenic temperature controller CAT-601 developed by Scientific Solutions, Mumbai.

In this set up four ohmic contacts are made at the periphery of the semiconductor sample mounted on the sample holder. The contacts are baked at 100° C for 24 hours. The sample holder is then inserted into the cryostat. The cryostat is filled up with the liquid nitrogen. After creating vacuum inside the sample holder with the help of a rotary pump, the connections are made. A constant current is passed through the current leads and the voltage developed at the voltage leads is measured while maintaining the temperature of the sample constant at a predecided fixed value. The value of resistance \( R_1 \) is then calculated. The current and voltage leads are now interchanged and the voltage is once again measured for a constant value of the current through the current leads. The resistance \( R_2 \) of
the sample is then determined. The resistivity $\rho$ at the given temperature is then evaluated by using the formula

$$\rho = \frac{\pi d}{2\ln 2} \left( R_1 + R_2 \right) f \left( \frac{R_1}{R_2} \right)$$

where $t$ is the thickness of the sample.

For the measurement of the Hall parameters, the sample holder with the specimen mounted on it is placed in a magnetic field and the current and magnetic field are kept constant. The voltage developed across the sample at different temperatures is measured. The Hall coefficient ($R_H$) is determined by using the formula

$$R_H = \frac{t \Delta V}{\Delta I} \times \frac{10^4}{B}$$

where $B$ is the value of the magnetic field and $t$ is the thickness of the sample.

The mobility $\mu$ of the charge carriers is obtained by using the expression

$$\mu = \frac{R_H}{\rho}$$

where $\rho$ is the value of resistivity obtained above.

The carrier concentration $n/\rho$ is determined with the help of the formula
\[
n / p = \frac{1}{R_H e}
\]

where \( e \) is the charge of the electron taken as \( 1.602 \times 10^{-19} \) Coul.

2.6.3 HALL EFFECT MEASUREMENTS AT ROOM TEMPERATURE USING LAKESHORE HALL EFFECT / ELECTRONIC TRANSPORT MEASUREMENT SYSTEM:

The Hall effect/electronic transport measurement system model 7504 shown in Fig. 2.18, manufactured by Lakeshore Cryotronics, Inc., USA has been used for the determination of the Hall parameters at room temperature.

A standard glass epoxy PCB sample holder is provided by Lakeshore onto which six contacts are so provided that shielding of each is obtained by passing them between grounded tracks. This helps in eliminating measurement errors. Central position between all six probes is provided for sample mounting, where sample is required to be fixed with the help of adhesive like quick fix etc. Indium foil is cut into a small required size and with a low watt solder gun and a low strain alloy, four wires are bonded with four corner points to provide van der Pauw geometry for measurement. For better ohmicity the sample can be baked up to moderate temperature (\( \sim 50 \) to \( 100 ^\circ \) C for about half an hour or so).

This, PCB sample holder with four ohmic contacts onto the sample is inserted into the PCB holding movable slot, which is then carefully
Fig. 2.18 Experimental set up of Hall effect / electronic transport measurement system "LAKESHORE" model 7504.
placed, between magnet pole pieces. The ideal Hall is a dedicated software provided by Lakeshore to do automated computer controlled measurement. The software is first run in variable current mode to check the ohmic nature of current transport through all the four probes by varying the combination of probes for passage of current and measurement of voltage drop across them. e.g. (13, 13), (24, 24), (12, 12), (34, 34) etc. After that, the instrument is programmed in variable magnetic field mode where all necessary selection like field reversal, current reversal, maximum field, field steps, van der Pauw geometry, six probe Hall geometry, etc. are to be provided and then the programme is allowed to run for final measurements and calculations of all required parameters: Hall coefficient, resistivity, mobility, carrier concentration etc. Simultaneously to this the nature of variation of these parameters can be observed on display by selecting the particular variable.

2.7 THERMOELECTRIC POWER MEASUREMENTS:

To measure the Seebeck coefficient of the samples used in the present investigation as a function of temperature, a differential temperature controller developed by Scientific Solutions, Mumbai [4] was used. The experimental system is shown in Fig. 2.19. It consists of two blocks.

(a) Sample holder with heaters and pick up probes and
(b) Electronic circuits controlling temperature and temperature gradient across the sample.
Fig. 2.19 The thermoelectric power measurement system TPSS-200.
The sample holder consists of two low power heaters A and B (15 W each). Temperature $T$ of the heater A is measured by thermocouple ($TC_1$) and the temperature gradient $\Delta T$ between A and B is measured by differential temperature sensor ($TC_2$). Both the thermocouples are of K type. The sample under investigation is mounted directly on the heaters, and is held by two pick up probes which are of copper (or stainless steel). These probes also measure the Seebeck voltage developed across the two ends of the sample.

Block 2 consists of temperature indicator, proportional controller and two heater control circuits which drive the two heaters A and B. With the help of these electronic control circuits it is possible to generate a stable temperature gradient between the two heaters. It is possible to control temperature from 298 K to 573 K and $\Delta T$ to ± 10 K simultaneously with better than ±1 K stability. The problem usually encountered in making thermoelectric power measurements are stray thermal emfs. In the present instrument these have been eliminated by providing choice of selection of temperature gradient in the range between 1 K to 10 K.

The sample and the electronic circuits have been incorporated into one unit. Use of low power heaters and electronic controllers makes the operation very easy and Seebeck coefficient of the samples can be very conveniently measured.
2.8 AXIOTECH REFLECTED LIGHT MICROSCOPE FOR STUDY OF MICROSTRUCTURES:

The microstructural examination of crystal surfaces as well as photographic recording of observations was accomplished with the help of Axiotech 100 reflected light microscope, manufactured by Carl Zeiss Jena, Germany. An overall view of this microscope is given in Fig. 2.20. The operation and function controls of this microscope are elegantly shown in the schematic diagram (Fig. 2.21).

The sample for microstructural examination is placed on the cover glass slip. The cover glass slip, with sample placed on it is mounted on the mechanical stage with the specimen holder. The instrument is switched on with the help of on/off switch (12). Using the Reflected-light illuminance control (11), the lamp voltage is set at (5) or (6) using the lamp voltage display (10).

From eyepiece (1) and objective lens(17), the surface structure of the sample is viewed. The specimen holder can be moved front-back and left-right using the co-axial stage drive (15) to bring the desired area in field of view. With the help of co-axial coarse and fine drive (13), the specimen holder can be moved up and down and the surface structure can be very accurately focussed.
Fig. 2.20 The set up of optical microscope "Carl Zeiss Jena" for study of microstructures.
Fig. 2.21  Schematic diagram of “Carl Zeiss Jena” optical microscope.
The optical microscope is finally attached to the CCD camera with a resolution of 0.5 X. The whole picture of the surface microstructure can be viewed live on the computer screen and stored in its memory. Finally a print out of the well focussed surface microstructure of the specimen is taken out.

2.9 CAPACITANCE MEASUREMENTS WITH MULTI-FREQUENCY LCR METER:

Multifrequency LCR meter (shown in Fig. 2.22) is a high performance, fully automatic test instrument designed to measure various component parameters values of an impedance element in the relatively low frequency region. It can measure inductance (L), capacitance (C), resistance (R), dissipation factor (D), reactance (X) and in addition, the absolute value of the vector impedance (Z) and phase angle (θ) over a wide range of frequencies with high accuracy and speed.

The test signal level can be flexibly set at the desired amplitude within the range of 1 mV to 5 mV by front panel controls. The obvious advantage of the variable test signal capability is that it becomes easy to measure non-linear impedance of elements whose parameters are strongly dependent on their operating conditions, such as inductors and other semiconductor devices. The measuring range for capacitance is from 0.01 pF to 1999.0 mF, resistance and impedance from 0.001 mΩ to 19.999 mΩ which is measured with a basic accuracy of 0.1 % to 10% depending on
Fig. 2.22 Photograph of LCR meter.
the test signal level and frequency at a typical measuring speed of 140 to 210 m/s. The wide range of capability of the LCR meter enables a measurement range from that for small capacitance and semiconductor junction capacitors to that for high capacitance, such as measurements of electrolytic capacitors to be covered. The high resolution measurement capability enables the measurement of any extremely low dissipation factor such as that of a polystyrene capacitor.
REFERENCES:


