

Investigation of Electrical Properties of highly pure Nano crystalline Lead Selenide

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Abstract - Lead Selenide (PbSe) is layered transition metal chalcogenides (TMDCs) and it exhibits vast variation in electrical and physical properties. Here in the present research work Lead Selenide (PbSe) is used in highly pure nano crystalline pallet form having thickness in few millimeters. It is very interesting to know the semiconducting behavior of Lead Selenide (PbSe) at various electrical parameters. Here the nature of the current carrying conductor was measured by Hall effect. The Hall parameters such as Carrier concentration, Hall mobility, Hall Coefficient and Conductivity were measured and analyzed for PbSe. The temperature behavior of electrical parameters is studied by Vander Pauw's, Four probe and High temperature basal plane techniques. The measurement of resistivity and conductivity at room temperature is carried out by Vander Pauw's technique. The behavior of resistivity with temperature is studied at high temperature perpendicular to c-axis (along the basal plane) and parallel to c-axis(normal to the basal plane) and calculation of activation energy is carried out.

Key Words: PbSe, TMDCs, Hall Effect, Resistivity, Vander Pauw's method, Four Probe method, Activation Energy, conductivity

I: INTRODUCTION:

Semiconductors based on selenium are very important in the category of semiconducting systems which have been vastly studied due to their amazing electronic and optical properties.[1] The lead selenide semiconducting material attract attention of many researchers due to non-toxic, economically cheaper, abundant and there semiconductor properties.[2] Lead Selenide thin films are mostly used as a major material in infrared sensor, grating, photo resistor, lenses and various optoelectronic devices[3]. Previously very few researchers have worked on the Nano crystalline powder form of Lead selenide so here there is a vast study of Electrical properties of Nano crystalline Lead Selenide.

In this research work Hall Effect is used to study the nature of semiconducting material whether it is p type or n type. Also series of experiments using Hall Effect instrument carried out to calculate Carrier Concentration, Hall mobility and Conductivity of the semiconducting material. The resistivity of the sample at room temperature is studied by Vander Pauw's method and conductivity is evaluated. Highly accurate Four probe instruments is used to study the temperature behavior of resistivity of Lead Selenide (PbSe) perpendicular to c-axis (along the

basal plane) and activation energy is evaluated. The high temperature behavior of resistivity parallel to c-axis (normal to the basal plane) is also studied and activation energy is evaluated.

II: MATERIAL (SAMPLE) PREPARATION FOR EXPERIMENTATION

Highly pure (99.99%) nano crystalline powder form of Lead (Pb) and Selenium (Se) were weighted with extreme accuracy using the weighting Reptech RA-503 with following calculated amount of prescribed material. Prepared sample is collected in Glass sample collector for carrying out further electrical analysis.

II-A: SAMPLE WEIGHT CALCULATION

- (1) Atomic mass of Lead (Pb): 207.2 gm/mol, Atomic mass of Selenium (Se): 78.96 gm/mol
- (2) Atomic mass of Lead Selenide (PbSe): $207.2+78.96 = 286.16$ gm/mol

II-B: SAMPLE PREPARATION IN VERY THIN PALLET FORM

- (1) Weight of Pb (Lead) in 10 gm sample: $-\frac{207.2 \times 10}{286.16} = 7.2404$ gm.
- (2) Weight of Se (Selenium) in 10 gm sample: $-\frac{78.96 \times 10}{286.16} = 2.7592$ gm.
- (3) Total weight of prepared sample of PbSe = Weight of Pb+ Weight of Se= $7.2408+2.7592 = 10.0000$ gm

Now the sample of 10 gm PbSe is mixed well with sample mixture and the sample is precisely taken in a very accurate and sensitive pallet making machine with hydraulic pressure to make very thin pallet having thickness of very few millimeters which is then annealed at 200 °C and cooled down to room temperature.

III: MEASUREMENT OF RESISTIVITY

The present study includes the measurement of resistivity at room temperature and in both the ways perpendicular to c-axis which is along the basal plane (ρ_{\perp}) and parallel to basal plane which is normal to the basal plane (ρ_{\parallel}).

III-A: RESISTIVITY MEASUREMENTS PERPENDICULAR TO C-AXIS (ALONG THE BASAL PLANE) (ρ_{\perp})

The resistivity perpendicular to c-axis (along the basal plane) is measured by the two methods given below.

- (1) Vander Pauw's method
- (2) Four Probe Resistivity method

Here the Vander Pauw's method is used for measurement of Electrical resistivity at room temperature while the Four Probe resistivity method is used for resistivity measurement at the range of temperature from 303 K to 473 K.

III-A-I: VANDER PAUW’S METHOD FOR THE RESISTIVITY MEASUREMENT

The Vander Pauw’s method is invented by Leo J. Vander Pauw. This method is used to calculate resistivity of a sample having irregular shape and size. This method can accurately measure electrical properties of sample by applying the current and measuring the voltage across the sample perimeter. The Vander Pauw’s method is used to calculate resistivity of the material, the charge carrier nature of the material and mobility of the given sample. In order to use this method, the sample prepared must have thickness less than the width and the length and it has to be symmetrical. For measurement the contacts should be made on the edges of the sample as shown in Fig.1.

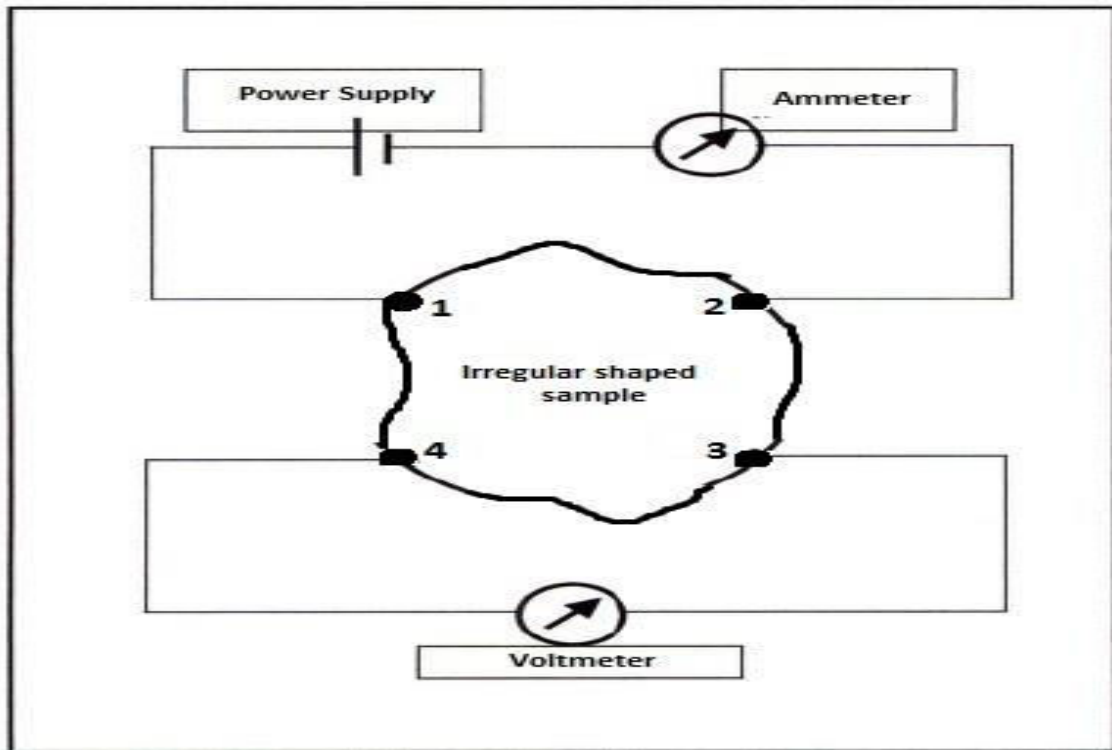


Fig 1. Vander Pauw’s method schematic Diagram

Above Fig1. show the schematic diagram/circuit diagram for the Vander Pauw’s method. As shown in above diagram the irregular shaped sample has connectors on its edges and is connected to Power supply, Ammeter and Voltmeter as per the requirement of the measurement. Now when we pass current through the terminals A and B and applying voltage to the terminals C and D then the Resistance $R' = R_{12,34}$ is defined as,

$$R' = V_{34} / I_{12} \dots\dots\dots (1)$$

Here V_{34} is the potential difference between terminals 3 and 4 and I_{12} is the current across terminal 1 and 2.

In the same way when we pass current through the terminals C and D and applying voltage to the terminals A and B then the Resistance $R'' = R_{23,14}$ is defined as,

$$R'' = V_{14} / I_{23} \dots\dots\dots (2)$$

Here V_{14} is the potential difference between terminals 1 and 4 and I_{23} is the current across point 2 and 3.

If d is the thickness of the sample, then the resistivity can be calculated by numerically from the following Vander Pauw's formula:

$$\rho = \frac{\pi d}{2 \ln 2} (R' + R'') f(R'/R'') \dots\dots\dots (3)$$

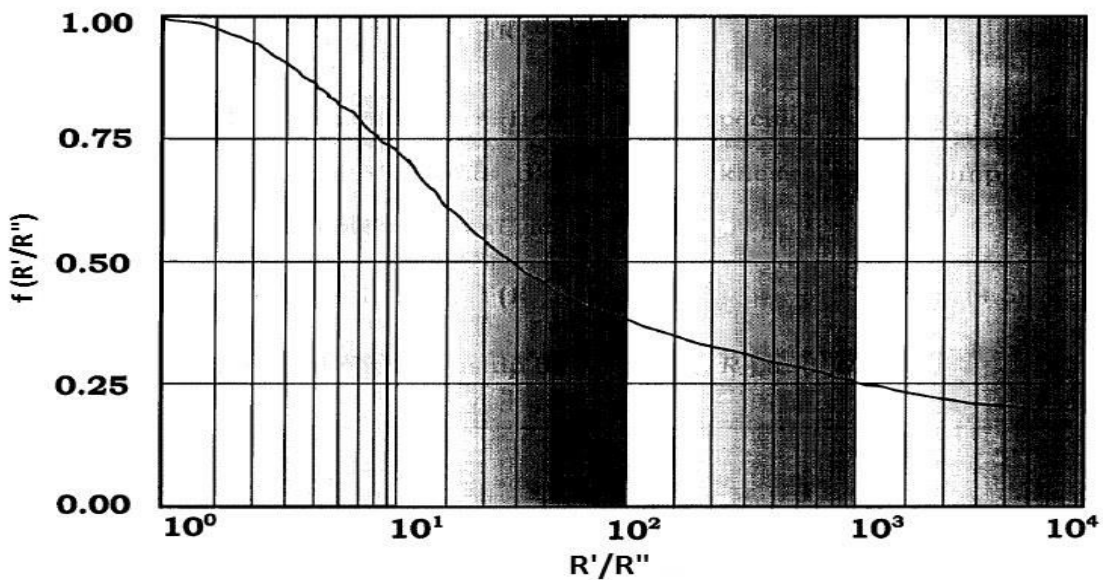


Fig.2 The relation between $f(R'/R'')$ and R'/R''

Above Fig.2 shows the relationship between $f(R'/R'')$ and R'/R'' and it is very important in order to calculate Resistivity from Vander Pauw's formula. The calculated value of Resistivity from equation (3) for the taken sample of Lead Selenide (PbSe) is 0.009060 ohm*cm and conductivity is 110.37 mho/cm.

III-A-II: HIGH TEMPERATURE RESISTIVITY PERPENDICULAR TO C-AXIS (ALONG THE BASAL PLANE) BY FOUR PROBE METHOD (ρ_{\perp}):-

In solid state physics to understand the temperature dependence of conductivity or resistivity of any semiconducting material the method of four probe resistivity is used. As its name suggest, this instrument is consist of 4 probes; 2 probes are for electric current and the 2 other ones are to measure voltage when they are applied to the material (sample). Here to study the electrical characteristics of the material the value of resistivity needs to be measured for particular area and given thickness.[7]

This method is called four-point probe because there are four points or four probes that has to be touched on the sample surface. Those four points (probes) is made lined in a straight line with the equal distance between them. A constant electric current is flown along the sample through the two outermost probes. Now If the sample has resistance, there will be a voltage drop when the current flows through the sample. The change in voltage is measured through two inner probes. Electrical quantity that shows the quality of material conductivity, such as output voltage and output current, can be determined easily using the four-point probe method.

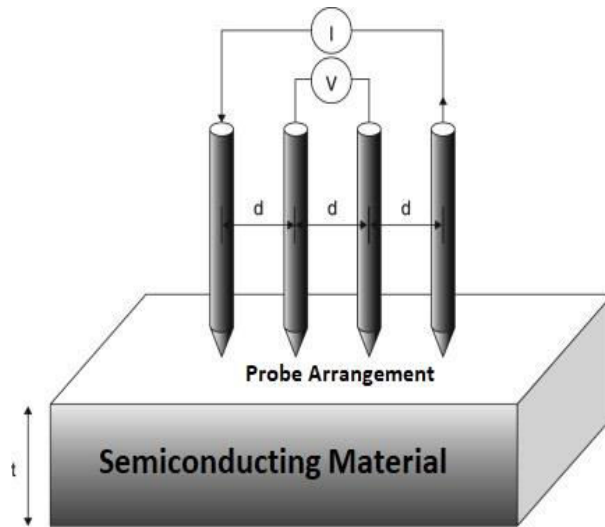


Fig.3 Schematic diagram of Four Probe Method

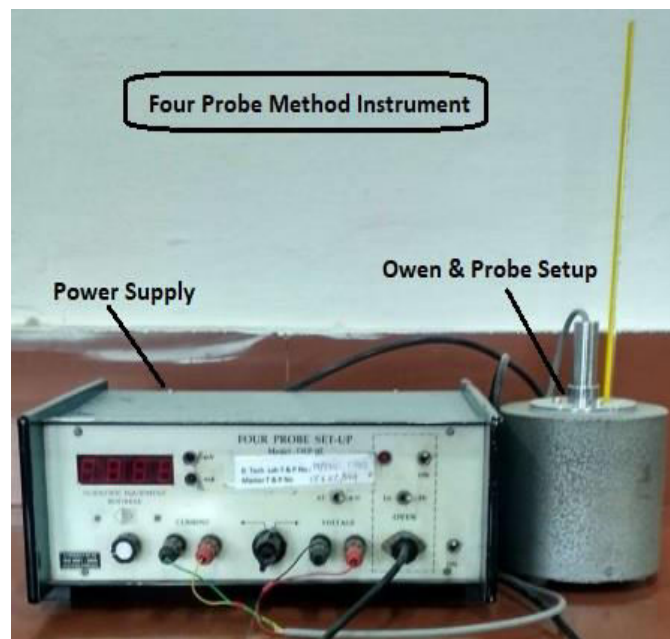


Fig.4 Instrument of Four Probe Method

III-A-II-A: EQUATION OF RESISTIVITY BY FOUR PROBE METHOD

Let's assume that a conductor of Resistance R, length L and cross section are A is given then from ohm's law theory we can write,

$$R = \frac{\rho L}{A} \dots\dots\dots (4)$$

Here ρ is the resistivity of the conductor or the semiconducting material. The Inverse of it will give the conductivity of the material.

Now as shown in above Fig-3 if t is the thickness of the material s is the spacing between the probes then the resistivity is given by,

$$\rho = \rho_0 / f(t/s) \dots\dots\dots(5)$$

Here f (t/s) is the divisor for computing resistivity and its value depends on the value of t and s.

Now if V is the voltage and I is the currents across the sample then the resistivity of the semiconducting material is given by

$$\rho_0 = 2\pi s (V / I) \dots\dots\dots(6)$$

Here the temperature dependence of the resistivity for semiconducting material we know that total conductivity of a semiconducting material is the sum of the conductivities of the all valence band and conduction band charge carriers. Now the Resistivity is reciprocal of the conductivity and its temperature dependence is given by following equation,

$$\rho = \exp (E_a / 2KT) \dots\dots\dots(7)$$

So Activation Energy

$$E_a = 2k \log \rho * T = 2k (\log \rho) / (1/T) \dots\dots\dots(8)$$

Here E_a is the Activation Energy and K is Boltzmann's Constant.

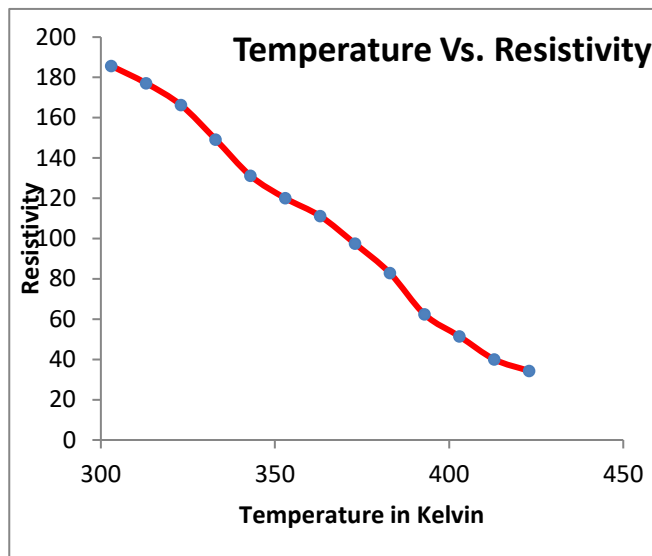


Fig.5 Temperature dependence of Resistivity

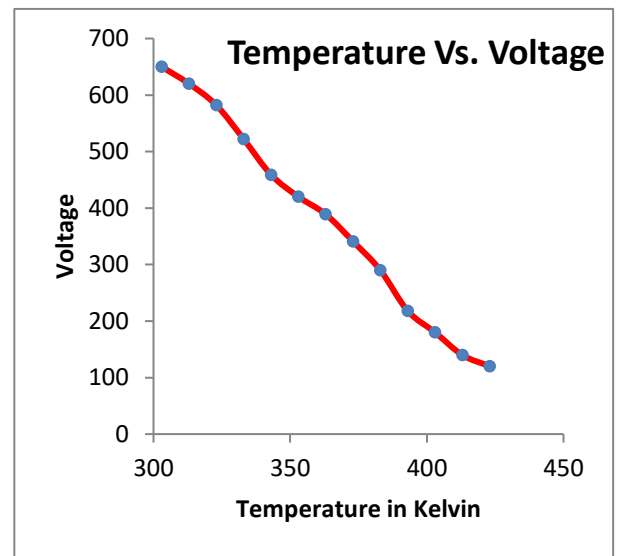


Fig.6 Temperature dependence of Voltage

Fig-5 shows the relationship between Temperature and Resistivity and it has to be noted that as the temperature increases the Resistivity is decreases for the sample of Lead Selenide (PbSe) and Fig-6 shows the relationship between Temperature and Voltage and it shows that as the temperature increase the voltage is decreases.

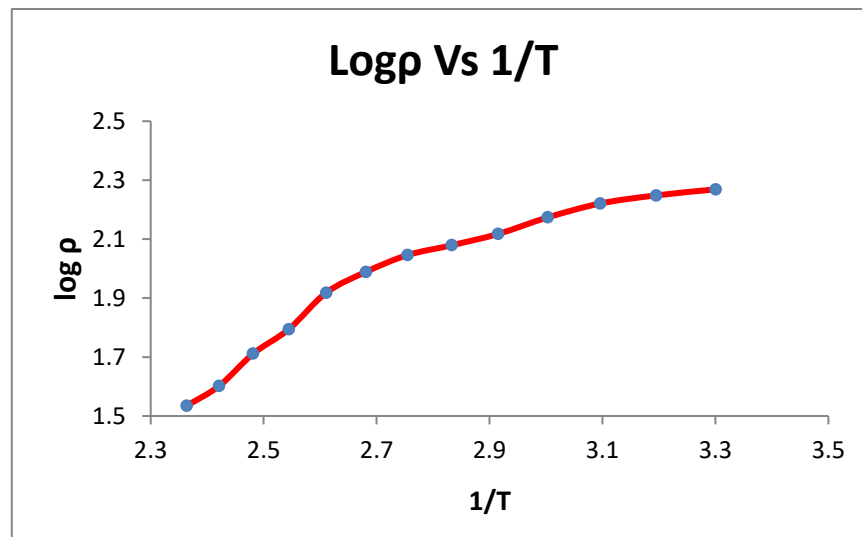


Fig.7 Graph of $\log \rho$ Vs $1/T$ for the resistivity perpendicular to c-axis

Above Fig.7 is a very important graph of $\log \rho$ Vs $1/T$ for the calculation of activation energy of given semiconducting material. Value of $f \left(\frac{t}{s} \right)$ which is the divisor for computing resistivity calculated for the instrument is 8.6112 for the taken sample of PbSe. The Activation Energy calculated for the taken sample of Lead Selenide (PbSe) is 0.31 eV.

III-A-III: TEMPERATURE RESISTIVITY PARALLEL TO C-AXIS (NORMAL TO THE BASAL PLANE) ($\rho_{||}$)

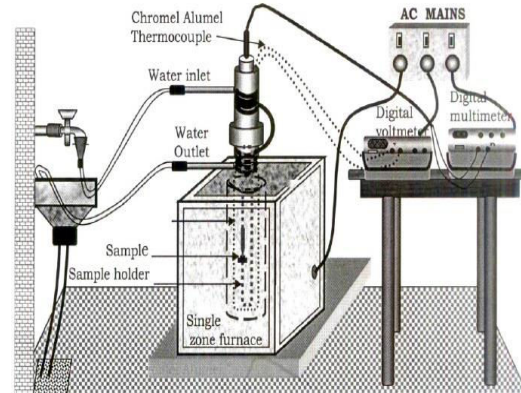


Fig.8 Instrument of Parallel to c-axis resistivity measurement Fig.9 Schematic diagram of the Instrument

Above Fig-8 shows instrument for the measurement of High temperature Resistivity parallel to c-axis which is normal to the basal plane and Fig-9 shows schematic diagram of it. Here we start from room temperature and the temperature of the sample is increased slowly in steps of 10 K and at every step the corresponding value of the resistivity of the sample is calculated. To avoid excessive heating of the sample chamber with the help of tubes it is cooled by circulating cold water around it.

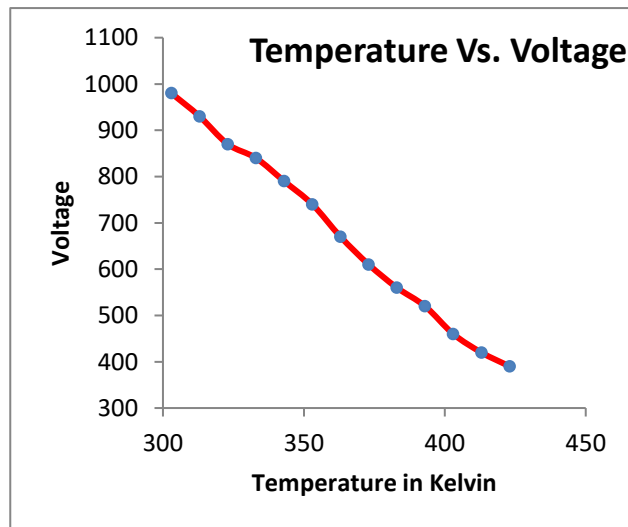


Fig.10 Temperature dependence of Voltage

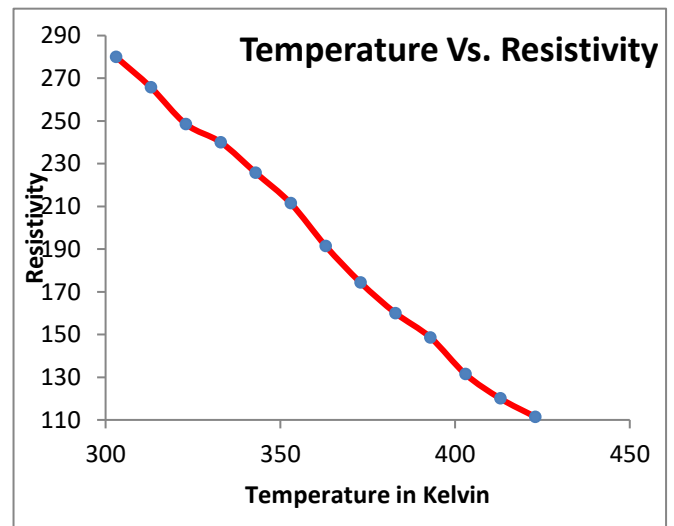


Fig.11 Temperature dependence of Resistivity

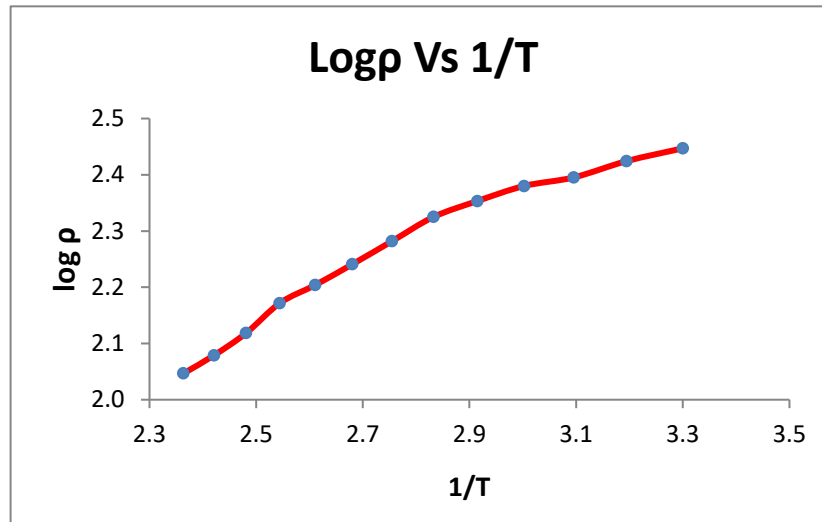


Fig.12 Graph of $\log \rho$ Vs $1/T$ for the resistivity parallel to c-axis

Now from the slope of above Fig.12 the activation Energy calculated by eqn.(8) for the taken sample of Lead Selenide (PbSe) is 0.1728 eV. Also when we carefully study Fig-5,6 & 7 and Fig-10,11 & 12 and when we compare the resistivity along the basal plane and normal to the basal plane then we clearly found that the resistivity is comparably high in the direction normal to the basal plane.

IV: HALL EFFECT

The Hall effect is very important experimentation in solid-state physics and an important diagnostic tool for the electrical characterization of materials – particularly semi-conductors. It gives us the type of charge carrier possessed by the semiconducting material either electron or hole.[4]

The Hall Effect was invented by a scientist named Edwin Hall in 1879. He has seen that electrical currents are affected by the applied magnetic fields. So, when a magnetic field is applied perpendicular to the flow of current, it will cause resistance in the current. This will result in the Lorentz force, who gives push to the negatively charged electrons in the current in a direction dictated by the left hand rule. Due to this motion of electron, there will be somehow weak but measurable potential difference (Voltage) perpendicular both to the current flow and the applied magnetic field. [5]

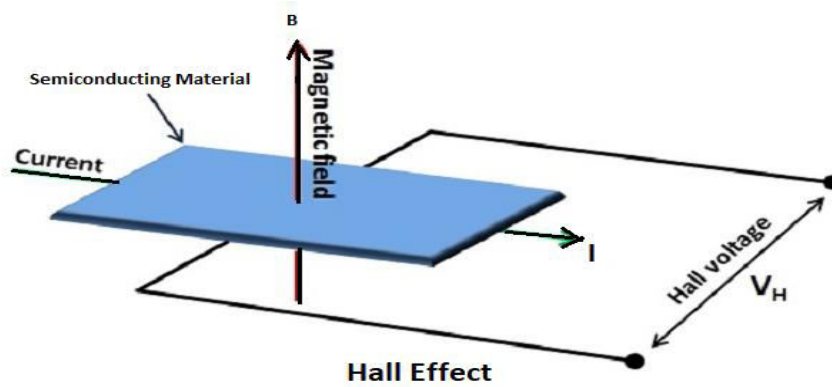


Fig.13 Hall effect schematic Diagram

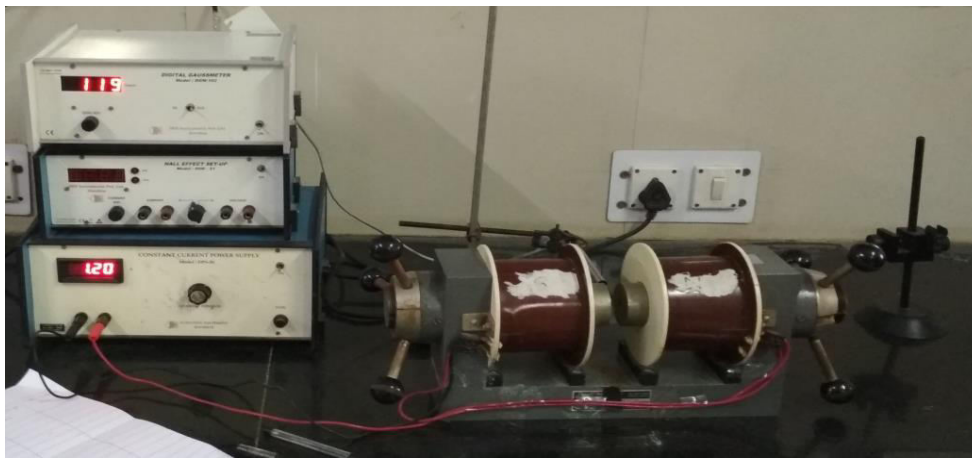


Fig.14 Instrument for the measurement of Hall Effect

Above shown Fig-13 gives us the schematic diagram for the Hall effect and as it is seen that when magnetic field B is applied to the Current(I) carrying conductor then there will be production of measurable Hall Voltage V_H perpendicular to both Current I and Magnetic field B . Fig-14 shows the instrument setup for the measurement of Hall effect which consist of Constant current supply, Gauss meter, two adjustable magnetic probes, Hall Probe and stand. A Hall effect is observed because the paths of charged particles are being bent in a magnetic field due to the Lorentz force.[6]

IV-A: EQUATION OF HALL EFFECT

As shown in Fig-13 current I is flowing through the conductor and the uniform magnetic field is B . Now if the thickness of conductor is d , charge passes through the conductor is q and n is the charge density then produced Voltage across the conductor which is Hall Voltage is given by,

$$V_H = \frac{I B}{q n d}$$

The Hall coefficient R_H is given by,

$$\text{Hall Coefficient, } R_H = \frac{1}{q n}$$

The carrier concentration is noted by,
Carrier Concentration,

$$n \text{ or } p = 1 / (R_H * q)$$

If σ is conductivity of the conductor then the Hall mobility μ_n or μ_p is calculated by,

$$\text{Hall mobility, } \mu_n \text{ or } \mu_p = \sigma R_H$$

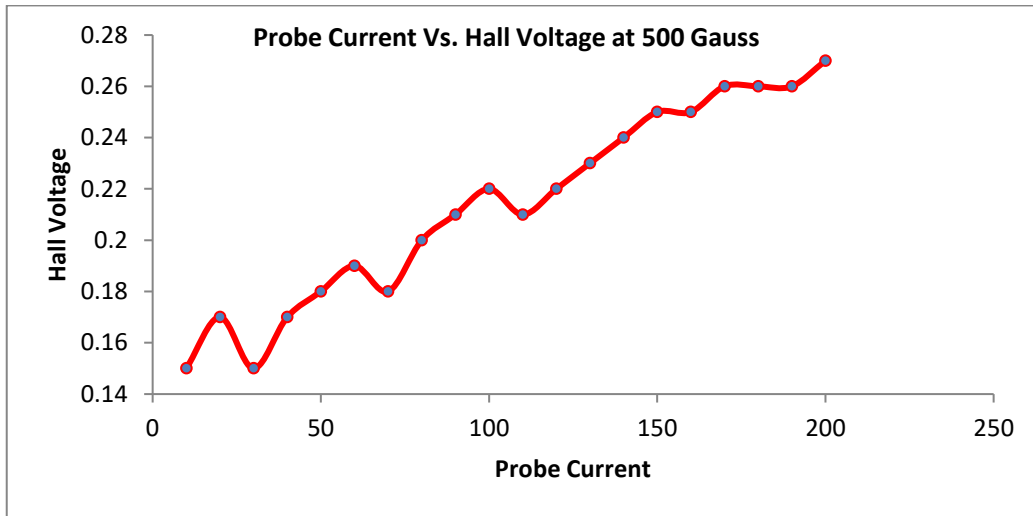


Fig-15 Probe Current Vs Hall Voltage at 500 Gauss

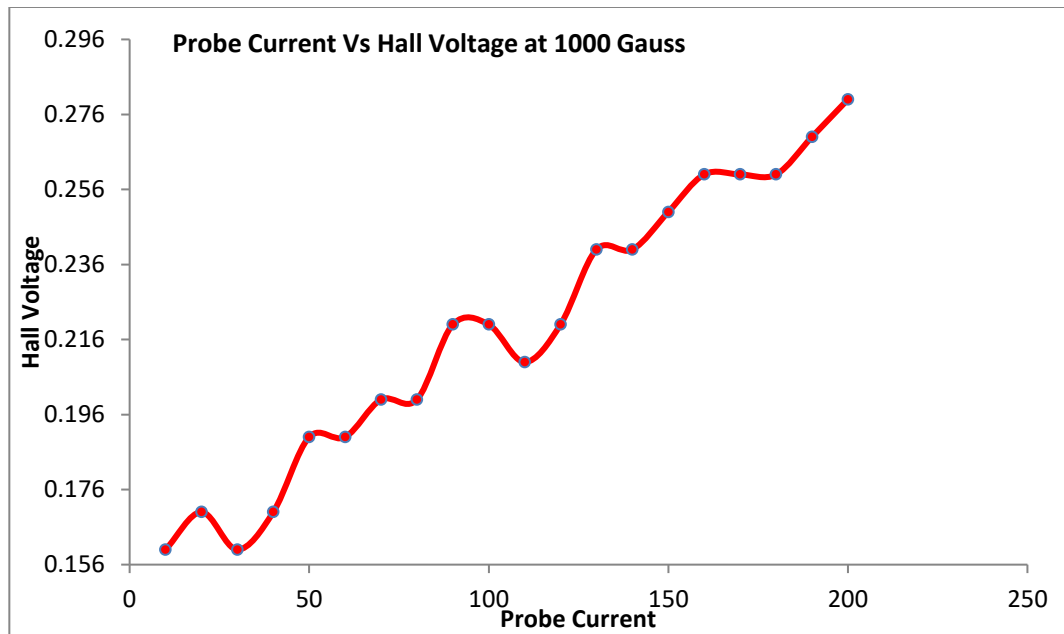


Fig-16 Probe Current Vs Hall Voltage at 1000 Gauss

Above Fig-15 and Fig-16 shows the relationship between Hall Voltage and Probe Current at Magnetic field 500 Gauss and 1000 Gauss and it is seen that the as Probe Current is increasing Hall Voltage is also linearly increased. This denotes that the taken semiconducting material has good conductivity.

Now from the above-mentioned equations we have made calculations for Lead Selenide (PbSe) and obtained the value of Hall Coefficient $3.77 \text{ cm}^3/\text{Coulomb}$ at 500 Gauss and $2.09 \text{ cm}^3/\text{Coulomb}$ at 1000 Gauss. So here the value of Hall Coefficient is positive in both the cases hence we can say that the taken sample of Lead Selenide (PbSe) is p type in nature. So holes are the majority charge carriers in the taken sample. The calculated value of Hall mobility is $416.11 \text{ cm}^2/\text{V}\cdot\text{sec}$ at 500 Gauss and $230.68 \text{ cm}^2/\text{V}\cdot\text{sec}$ at 1000 Gauss. Carrier concentration is 2.65×10^{18} at 500 gauss and 4.78×10^{18} at 1000 Gauss.

CONCLUSION

Here in present research work electrical properties of Lead Selenide successfully investigated through Hall Effect, four probe and High temperature resistivity method. From the Hall effect it is found that the conductivity of the taken sample of Lead Selenide is good and it increases linearly with the increase of current. The calculated value in both the cases of magnetic field is positive which indicates that holes are the majority charge carriers in the taken sample and the taken sample of Lead Selenide is p type semiconducting material. So we have found following facts from Hall effect.

Sample	Magnetic Field	Carrier Concentration	Hall Coefficient (cm ³ /coulomb)	Type	Mobility (cm ² /V sec)
PbSe	5000 gauss	2.65210 ¹⁸	3.77	P	416.11
	1000 gauss	4.78210 ¹⁸	2.09		230.68

Now the resistivity at room temperature is studied through the Vander Pauw's method and we found following facts.

Sample	Resistivity	Conductivity
PbSe	0.009060 ohm*cm	110.37 mho/cm

Here the temperature behavior of resistivity is studied perpendicular to c-axis (along the basal plane) and parallel to c-axis (normal to the basal plane) and it is observed that the resistivity is comparably high in the direction normal to the basal plane. The activation energy calculated in both the cases is as follows.

Sample	Technique	Activation Energy
PbSe	Perpendicular to c-axis (along the basal plane)	0.31 eV
	Parallel to c-axis (normal to the basal plane)	0.1728 eV

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