CHAPTER – 2

EXPERIMENTAL TECHNIQUES

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Various techniques employed in the investigations during the course of work are described briefly in this chapter together with some alternative allied techniques which may also be used. These include Bridgman-Stockbarger[1] and Zone Melting[2] crystal growth methods, X-ray diffraction, dislocation etching, preparation of thin films and measurement techniques of optical and electrical properties, surface observation etc.

I. TECHNIQUES FOR CRYSTAL GROWTH :

1.1 Synthesizing the compound :

To obtain a homogeneous mixture of the weighed proportions of the components of alloys, a melt-stirring method was used. It consists of a resistance furnace with a cylindrical core of about 45 cm in length and 5 cm in diameter. A ceramic tube of 60 cm in length and 1.5 cm in diameter is passed through the cylindrical core. A uniform temperature zone of sufficient length, depending on the maximum temperature in the furnace, is obtained inside this tube. The two ends of the tube are fitted to two brass sockets. The sockets are pivoted on frictionless bearings for smooth motion without wobbling. The tube is rotated at 10 r.p.m. by an electrical motor. A photograph of the mixing unit is shown in Figure-1. A quartz ampoule evacuated to about 10^{-4} Pa pressure and containing the charge is sealed and inserted in the ceramic tube for melting and stirring the charge. The maximum temperature inside the furnace core is kept about 100 °C above the melting point of the material. The temperature is measured and controlled (within ± 5°C) with a proportional temperature controller. The temperature is sensed with a chromel-alumel thermocouple. The rotation cum rocking of the quartz tube gives stirring effect to the molten charge. For thorough mixing and reaction of the charge, this treatment is continued for 2 to 3 days. The molten charge is then slowly cooled to room temperature.

1.2 Crystal growth :

Crystal growth is a complex process which usually takes place by a phase change and it can broadly be categorised as follows :

1. Solid state growth : Solid-Solid phase transition.
2. Melt growth : Liquid-Solid phase transition.
3. Vapour growth : Vapour-Solid phase transition.

Crystallization from melt is the most widely used method for crystal growth, mainly due to the higher growth rates obtainable and because it is easily susceptible to modifications. Also chances of chemical contamination are reduced compared to other methods. All the basic techniques are variants

Fig. 1

in the way materials can be crystallized in rods, crucibles, boats or tubes. Thus the Czochraslki technique involves controlled crystallization from a crucible by pulling the melt whereas the Kyropoulos technique does not involve pulling but crystallisation onto a seed-crystal in a crucible. Zone melting is the generic term for a series of techniques in which a molten zone (or zones) is formed and traversed along a bar so as to produce progressive melting and freezing. The horizontal Bridgman method involves the progressive crystallization of molten material in a boat or tube whilst the Stockbarger (vertical Bridgman) technique is the vertical equivalent of the lateral process[3].

(1) Bridgman - Stockbarger method :

Bridgman - Stockbarger technique is capable of producing large size crystals from thin rods to ingots of several cms in diameter. The Bridgman crucible is usually a tube of circular cross section with a tapered shape formed at one of the ends. The tapered end leads through the negative temperature gradient thereby effecting the charge transform from liquid to solid state and it provides conditions for single nucleation event required to grow subsequently as a single grain through the rest of the material as it solidifies. The Stockbarger-Bridgman vertical furnace is divided into two halves, the temperatures of which can be varied independently and hence suitable temperature gradient can be obtained. The apparatus consists of a vertical resistance furnace having a cylindrical core 55 cm in length and 5 cm in diameter, prepared in the usual way. It is properly lagged to avoid radiation losses. The temperature of the furnace is controlled (±5°C) using a chromel-alumel thermocouple and a temperature controller. The temperature gradient in the furnace in the region of interest, viz, the lower end wherethrough the molten charge is lowered for unidirectional solidification, could be varied in the range from about 35°C/cm to 45°C/cm. The ampoule containing the charge is sealed at 10^{-4} Pa pressure and is kept at the centre of the furnace. The central temperature is maintained 50° above the melting point for a sufficient time to melt the complete charge and then it was lowered down the furnace. The lowering is facilitated by a gear mechanism coupled with a 0.5 H.P. motor. The lowering speed could be varied from 0.35cm/hr to 1.0 cm/hr by changing the output shaft of the gears. A photograph of the unit is shown in Figure-2. The detailed growth of Bi_2Te_3 : Sb, Sn, Se single crystals by this method is described in chapter 3.

(2) Zone melting method :

The apparatus consists of a long quartz tube of about 100 cm in length and 2 cm in diameter. A ring or zone furnace is mounted on a trolley and the tube is passed through the furnace and clamped at its two ends. The motion of the furnace on trolley is controlled by a gear mechanism connected with a 0.5 H.P. motor. A photograph of the unit is shown in Figure-3. The vacuum

Fig. 2

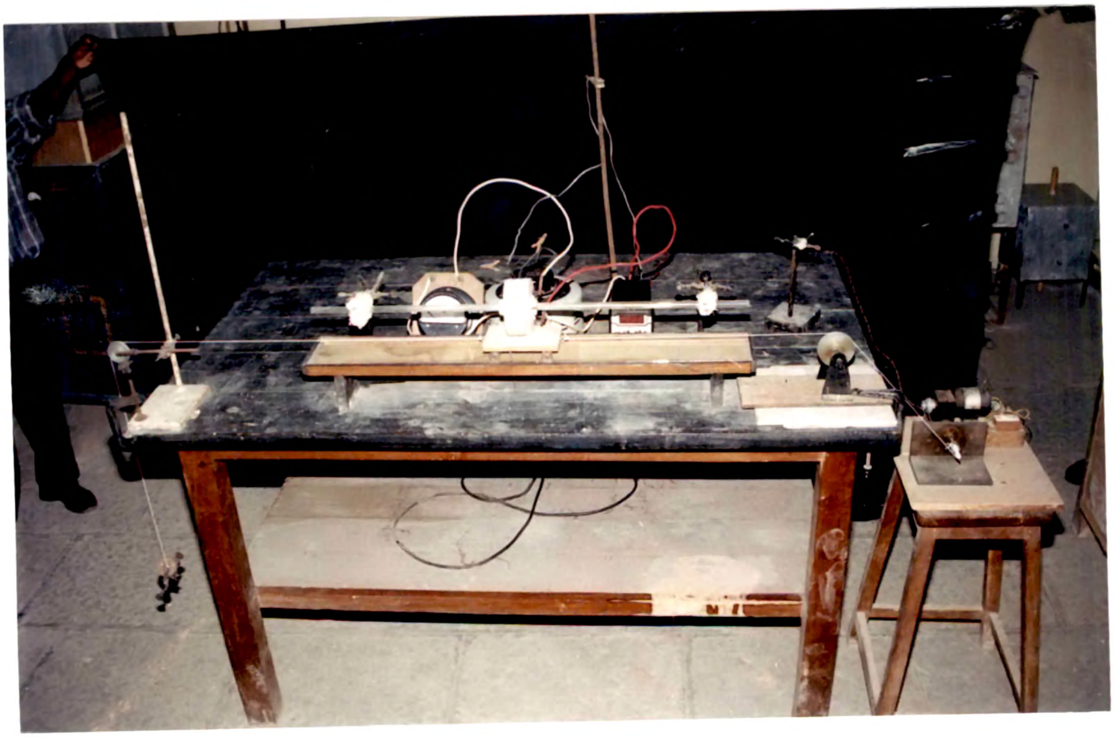

Fig. 3

sealed (10^{-4} Pa) quartz tube containing the charge was then kept inside the long quartz tube. At a maximum temperature of 750°C in the furnace, an appropriate temperature gradient, viz, of about 90°C /cm is obtainable at both the solid-liquid interfaces using this furnace. The detailed growth of Bi_2Te_3 : Sb, Sn, Se. single crystals by this apparatus is discussed in chapter 3.

The single crystalline character of the crystals thus grown was asserted by

(1) Cleavage test and

(2) Etching test .

The smoothness and hence the perfection of cleavage plane depends on the quality of the crystal grown. This can further be confirmed by etching the surface in a dislocation etchant and examining the distribution and shape of each pits.

II. THIN FILMS :

Modern day technology requires several types of thin film for a variety of applications. The films can be single or multilayer coatings on suitable substrates of different shapes and sizes.

The basic steps involved in a thin film deposition technique are :

(a) Creation of material to be deposited in an atomic, molecular or particulate form prior to deposition.

(b) Transport of material to the substrate in the form of a vapour stream, spray etc.

(c) Deposition of the material on the substrate and film growth by nucleation and growth process.

Thin film deposition techniques have been broadly classified into four main categories :

1. Physical Vapour deposition (PVD).
2. Chemical Vapour deposition (CVD).
3. Electroless or solution growth deposition.
4. Electro Chemical deposition (ECD).

By combining PVD and CVD, hybrid techniques such as reactive evaporation / sputtering and plasma deposition have also been developed. The PVD technique has been used in this investigation.

PVD techniques are further classified into :

(i) Thermal evaporation.

(ii) Electron beam evaporation.

(iii) Molecular beam epitaxy (MBE).

(iv) Activated Reactive evaportion (ARE).

(v) Ion plating.

2.1 Thermal evaporation :

It is one of the well known PVD techniques and the most widely used one. Large variety of materials (metals and semiconuctors) can be evaporated and deposited on different substrates.

In the thermal evaporation, the material is created in a vapour form by means of resistive or RF heating. On heating a material in vacuum it evaporates at a rate given by the well known Langmuir-Dushman equation. The vapour atoms are transported through vacuum and get deposited on the substrates. Only at pressures $\leq 10^{-4}$ Pa, the mean free path of molecules becomes large so that the vapour beam reaches the substrate nearly unscattered.

A variety of sources are used to evaporate materials, depending on whether the materials are in wire, foil, ingot or powdered form. The material used as a resisitive source of heat is a refractory metal like W, Mo etc. One can evaporate from two or more sources in order to deposit a multilayer film of an alloy/compound.

Direct thermal evaporation of intermetallic compounds may pose some practical difficulties. These compounds cannot be deposited without due consideration of their dissociation which may take place even below their melting points in some cases. There may arise difficulties in growing films of stoichiometric composition from alloys and complex chemical compounds. The adhesion of film to substrate depends on the level of purity of the substrate surfaces, procedure of its cleaning and film deposition condition., etc.

In order to overcome these difficulties, the technique known as flash evaporation may be used. It is generally used for deposition of materials whose constituents have vastly differing vapour pressures and which are known to decompose easily on vaporization. It is applicable to certain alloys, metal-dielectric mixtures and compounds. In most of the cases, vapours impinging on the substrate are highly supersaturated so that the film composition is not affected by condensation coefficients. In this technique, a fine powder of the compound is sprinkled over a heated sheet/ boat, which is at a high temperature as compared to the melting point of the compound, to produce an instantaneous and complete evaporation of the compound . The powder is kept in a feeder which is electromagnetically vibrated to let the powder drop slowly on the preheated boat.

2.2 ELECTRON BEAM EVAPORATION :

The temperature of the evaporant material can also be raised by electron bombardment instead of resistive heating. The thermionic electrons are accelerated through a potential of 5 KV to 10 KV and focussed on the material. The electrons lose their kinetic energy mostly as heat energy and the temperature at the focal point may be raised upto about 3000°C. At such a high temperature, most of the refractory metals and compounds can be evaporated. The temperature at the focussed point is very high, while rest of the material remains cool. The result is lesser interaction between the material and the support and reduced contamination. Evaporation rate can be varied by changing the input power.

2.3 MOLECULAR BEAM EPITAXY (MBE) :

The deposition of single crystalline (epitaxial) films by the condensation of one or more beams of atoms and/or molecules from Knudsen (effusion) source, under UHV conditions, is called molecular beam epitaxy.

Epitaxial films of compound semiconductors like GaAs, CdTe, etc. are usually deposited by this technique. The technique achieves precisely controlled properties of the film. The growth temperature is low, which minimizes the thermal effects like diffusion. The growth rate is also small (1 to 10 Å / sec.).

2.4 ACTIVATED REACTIVE EVAPORATION :

If the evaporated material (by resistive heating or electron beam evaporation) is transported through a reactive gas plasma (oxygen, acetylene etc.), the deposition technique is known as activated reactive evaporation. It is used to deposit highly adherent films of oxides and carbides.

2.5 ION PLATING :

In this process the substrate and film are exposed to a flux of high energy ions during the deposition. The energy of the ion is high enough to cause the changes in the interfacial region and film properties such as adhesion of the film, its morphology and density. The evaporation of material is done by resistive heating or electron beam bombardment. A plasma is created in the working gas like argon either by keeping the substrate at high negative potential or by electrons emitted from a hot filament and accelerated to an anode. The negative potential at the substrate accelerates the ions to a desired energy [4].In the present work the thermal evaporation technique has been used to grow thin films of $Sb_{0.2}Bi_{1.8}Te_3$, $Sn_{0.2}$ $Bi_{1.8}$ Te_3 and Bi_2 $Te_{2.8}$ $Se_{0.2}$.

III. EXPERIMENTAL SET UP FOR THIN FILM GROWTH :

3.1 Vacuum Coating System :

In the present work, for the thin film deposition, "Hind Hivac" vacuum coating unit, Model No. 12 A-4 (Figure-4) was used. The chamber material is polished stainless steel with vacuum sealed glass windows for visual inspection of the coating process. A pyrex glass bell-jar is also provided. The system consists of a double stage gas ballast rotary pump having a capacity of 200 lit./min. and an oil diffusion pump OD-114 having oil charge of 150 to 200 cc. Rotary pump is connected with a moisture trap mounted directly above the inlet of the pump. A tray containing the dessicant in the form of pellets (usually activated alumina) is kept inside the trap body. The gases

Fig. 4

passing through this trap come in contact with the dessicant which absorbs the water vapour present in the gas. This avoids contamination of the rotary pump oil with water and other harmful vapours.

To isolate the vacuum chamber from the pump it is provided with a solenoid valve to admit the air automatically into the rotary pump either on switching off the system or on the failure of electric power supply, thus giving a complete protection against the oil being sucked back.

To avoid the back streaming and hence contamination and loss of pump fluid, the D.P. is connected with a water-cooled baffle valve which enables a working vapour pump to be isolated while pumping system is at atmospheric pressure. a liquid nitrogen trap is also connected with the D.P. to avoid the back streaming and increase the action of Diffusion pump.

The L.T. supply for filaments or boats is obtained from a 230V input transformer by means of series or parallel connections in the secondary of the transformer. The L.T. output of the transformer is fed through a current meter and a sector switch to L.T. leads and filament holders. It is also provided with H.T. power supply for glow discharge cleaning (ion bombardment), obtained from a high reactance transformer rated at 3.5 KV, 50 mA and 5 KV AC open circuit. A solid state power pack having a DC output is provided for H.T. cleaning and cathode sputtering supply.

Fully stabilised vacuum gauges are provided : Two Pirani gauge heads one of which is mounted on the mouth of the rotary pump and the other in the chamber which can measure from 5 Pa to 10^{-2} Pa and a Penning gauge fitted with the chamber and measuring from 10^{-1} Pa to 10^{-5} Pa.

3.2 Chamber arrangement :

The chamber gadgetary comprises of a work holder ring which has a useful diameter of about 8 inches. It is supported by three pillars fitted to the base plate. A D.C. high tension discharge cleaning system consisting of a pure aluminium annular ring suitably shielded to avoid electron contamination of the work. A source shutter swinging over the source position and operated by an external lever is provided on the front of the cabinet.

A standard filament holder is fitted to the L.T. live electrodes and an earth electrode. The filament is normally positioned vertically below the centre of the work holder to give nearly uniform distribution of the vapours. For deposition of a multilayer film, it is provided with a multifilament turret designed to evaporate four different materials without breaking the vacuum. The turret is rotated by external hand wheel.

Rotary Drive : The rotary drive is useful for uniform deposition of materials on large plane surface substrates. This consists of a work holder of 6 inches in diameter and is rotated by a variable speed electric motor situated outside the chamber, without vibrations. The speed is controlled by a solid state speed control.

IV. THICKNESS MEASUREMENT :

Thickness is the most significant film parameter. It may be measured either by in-situ monitoring of the rate of deposition, or after the film is taken out of the deposition chamber. Usually the multiple beam interferrometry method[5] was used. In the latter case two reflecting surfaces are brought in close proximity such that a small wedge with a small air gap in between them is formed. If a monochromatic light is now incident on them at normal incidence, then an interference of light due to interactions of multiple reflected beams in air gap will take place resulting in a series of fringes (Fizeau) which can be observed in the back reflected light. The distance between the fringes or lines depends on the air gap as well as on the wave length of the monochromatic light. This principle is adopted and suitably modified for the multiple beam interferometic method of the measurement of film thickness[5].

A film, the thickness of which is to be determined, is deposited on a flat surface so as to leave a sharp edge between the film and the uncoated region of the substrate. An optical flat is preferred as a substrate and often a good microscopic glass slide is good enough for this purpose. The substrate with a coating of the film is then given a heavy and highly reflecting coating of a metal such as silver or aluminium so as to form a sharp step on the film edge. Another flat (optically) glass slide known as the reference plate, with a partially transparent film of the same metal, is then placed over the specimen with their metal coated surfaces in contact with each other so as to leave a small air gap at the step. A monochromatic parallel beam of light passing through a beam splitter or a glass plate inclined at 45° is then incident on the two plate assembly and reflected light is then observed through a microscope (Figure – 5a). A set of sharp fringes perpendicular to the step with equal displacements will be observed (Figure - 5b) and the thickness (t) can be determined using the relation[6]

$$\text{Thickness of thin film } t = \frac{b\lambda}{2a}$$

where,

b = Dispacement of the fringes at the step

a = Distance between consecutive fringes and

λ = Wave length of light used.

= 5893 Å (Sodium Vapour Lamp).

These fringe displacements which are in the form of parallel lines, however,

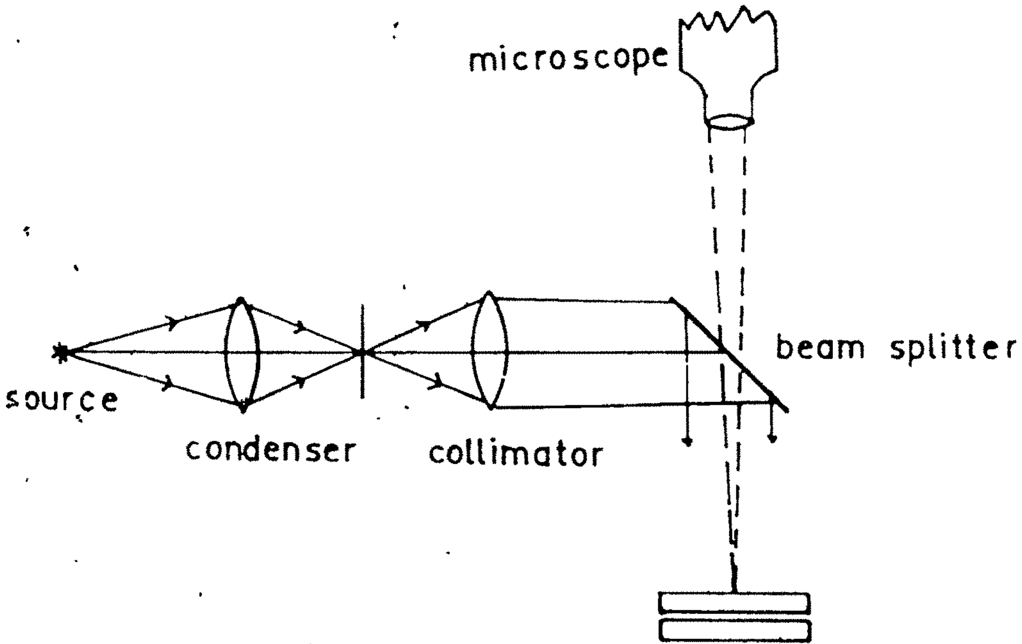
microscope
beam splitter
source
condenser
collimator

Fig. 5 (a)

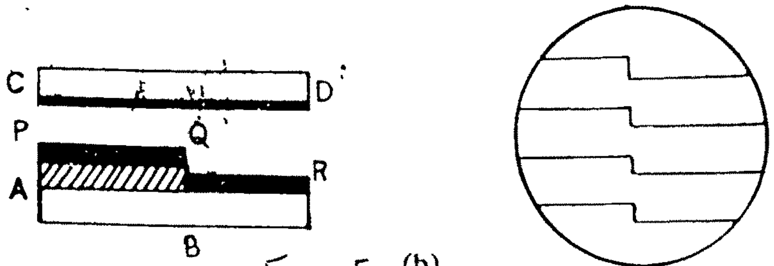
C
D
P
Q
R
A
B

Fig. 5 (b)

occur at the film edge. The sharpness of fringes depends on the reflectivity of the metal coating, the spread of the incident beam, air gap etc. It is also essential that the metal coatings on the two plates, viz., the substrate and the reference plate should be of the same material since a phase change occurs when a beam of light is incident on each of the metal coating. This technique is capable of resolution as low as about 10 Å. This is one of the simplest methods which can be adopted for measurement of thickness varying say from 30-20,000 Å. This is the only property-independent method for t measurment.

In the present work for the thin films of $Sb_{0.2}Bi_{1.8}Te_3$, $Sn_{0.2}Bi_{1.8}Te_3$ and $Bi_2Te_{2.8}Se_{0.2}$, different thicknesses were deposited on glass substrates at 10^{-4} Pa pressure .The thickneesses of these films were measured by multiple beam interferometry using the above equation .

V. OPTICAL MICROSCOPY :

VICKERS MICROSCOPE :

The microphotographical study of the crystal surface was carried out using the Vickers projection microscope.It is an inverted metallurgical type optical microscope. For examination of the crystals, this microscope carries two different systems. The present work involves optically opaque crystals and only the reflection system was used for the purpose. This equipment also provides for phase contrast and light profile techniques.

VI. X-RAY DIFFRACTION ANALYSIS :

Among the various characterization techniques, X-Ray diffractometer techniques play a very important role in structural characterization. These techniques are used for a microstructural investigation of thin films and bulk crystals. The techniques based on the use of monochromatic radiation yield quantitive structural and symmetry properties possessed by the material being investigated. In the present study the X-ray diffractometer, Philips PM 8203 with pw 1390 channel control and pw 1373 goniometer have been used for the characterization of $Sb_{0.2}Bi_{1.8}Te_3$, $Sn_{0.2}Bi_{1.8}Te_3$ and $Bi_2Te_{2.8}Se_{0.2}$ crystals. It was operated at 35 to 40 kv & 20 mA with Cu traget (λ = 1.5418 Å).

VII. MICROHARDNESS MEASUREMENT TECHNIQUE :

Hardness can be measured by various methods :

1) Scratch method
2) Abrasive method
3) Plowing method
4) Rebound method
5) Damping method
6) Cutting method
7) Erosion method and
8) Static indentation method

A brief description of these methods follows :

1. SCRATCH METHOD :

In 1822, Friedrich Mohs developed this method which is widely used by minerologists. In this method, ability of one material to scratch another is termed as the hardness of scratched material with respect to the other. If the hardness is greater than 4, Mohs method is not suitable

2. ABRASIVE METHOD :

In this method, measure of resistance to mechanical wear is taken to be the amount of material removed from the surface under specific conditions. Here the specimen is loaded against a rotating disc and the rate of wear is taken as the hardness measure.

3. PLOWING METHOD :

Here a blunt element (usually diamond) is moved across a surface under controlled conditions of load and geometry. The width of the groove produced is taken as the measure of hardness. The Bierbaum test is of this type.

4. REBOUND METHOD :

In this method usually a steel ball of standard mass and dimension is bounced from the test surface and the height of rebound is taken as the measure of hardness. The Shore scleroscope is a hardness tester of this type.

5. DAMPING METHOD :

Herein, change in amplitude of a pendulum having a hard pivot resting on the test surface is the measure of hardness.

6. CUTTING METHOD :

A sharp tool of specific geometry is made to remove a chip of standard dimension from the specimen.

7. EROSION METHOD :

In this method, abrasive grains or sand is caused to impinge on the test surface under standard conditions and loss of material in a given time is taken as the measure of hardness.

8. STATIC INDENTATION METHOD :

In this method, a steel ball, a pyramid or a cone (indentor) is forced into the surface and the load per unit area of the permanent impression formed is taken as a measure of hardness. The Brinell, Vickers, Rockwell, pin indentation and knoop tests are of this type.

The most popular and simplest method of hardness measurment is the static indentation hardness method.

(i) VICKERS DIAMOND INDENTER : The Vickers dimond indenter is in the form of a square based pyramid with semi apex angle = 68°.It was used to measure the microhardness of the grown single crystals by producing indentation marks on the cleaved surfaces of the specimens, under known applied loads, loading time, temperature etc. Its main characteristic is the geometrically similar impressions obtained. The Vickers hardness, H_V , was calculated using the formula,

$$H_v = \frac{1854 \times P}{d^2} \ \text{kg/mm}^2 = \frac{1854 \times P}{d^2} \times \frac{9.8}{1} \ \text{MPa} \qquad \text{-(1)}$$

In accordance with the definition given by Cooke, Toughton et al[7], where P is load in gram and d is the average diagonal length of indentation mark in microns. To measure the diagonal of the indentation mark, a micrometer eye piece with the least count 0.19 micron was used.

The Vickers Microhardness Tester (supplied by M/s. Cooke Toughton and Simms Ltd., England) was used with the Vickers projection microscope. (Figur-6). All the instructions suggested by the supplier were rigidly observed. Since there is no provision for making indentations at high

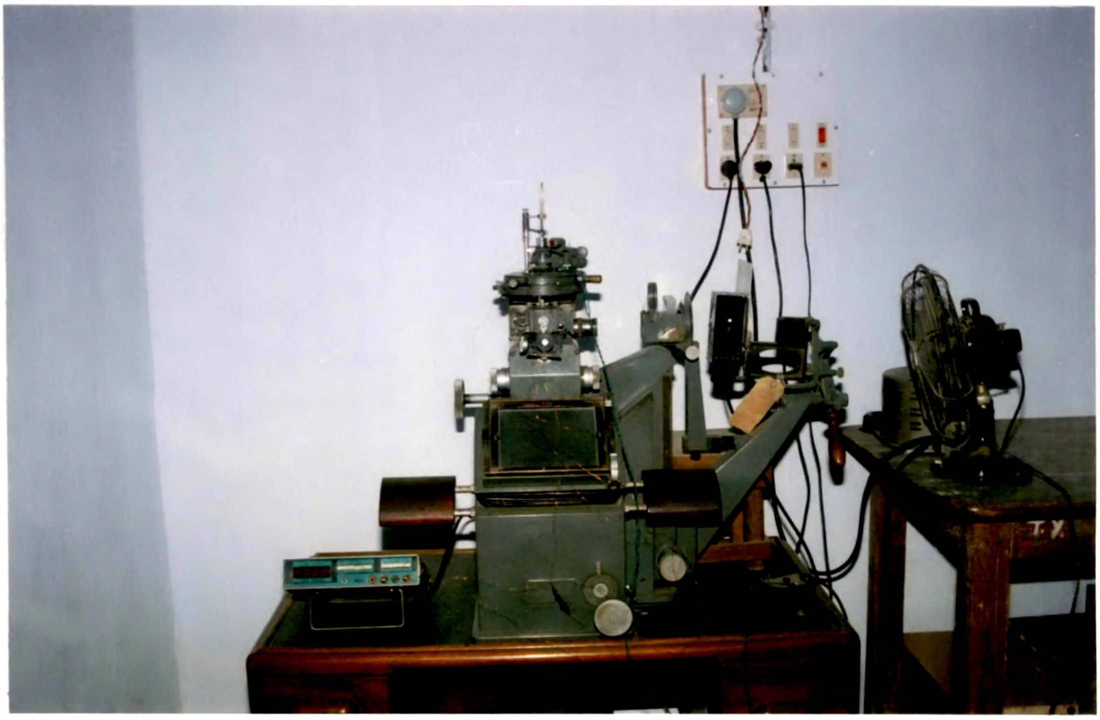

Fig.6

temperature in the above equipment, a special arrangement described below was attached to the hardness testing apparatus.

A cylindrical shape refractory block (Figure-7) was used to mount the specimen, the diameter and length of this block being such that it can easily be fitted in the collect of hardness testing unit of the microscope. A small heating element was passed through this mount. A circular brass disc of the same diameter as of the mount was fitted on top of this mount.The specimen can be fitted on the disc by a proper adhesive. A copper constantan thermocouple was placed through a groove 1mm below the top of the brass disc. Known melting points of some substances like paraffin wax, InBi, Tin etc., were checked to calibrate this heating arrangement. The error in any case did not exceed 2°C. Before indenting the specimen, care was taken to get the thermal equlibrium. The detailed study of hardness of $Sb_{0.2}Bi_{1.8}Te_3$, $Sn_{0.2}Bi_{1.8}Te_3$ and $Bi_2Te_{2.8}Se_{0.2}$ crystals at room temperature and at higher temperatures is presented in Chapter 4.

VIII. ELECTRICAL PROPERTIES :

The discovery of a great number of semiconductors among the intermetallic compounds has created an urgent need of methods to make fast accurate measurements of resistivity. Various sample geometries of films are

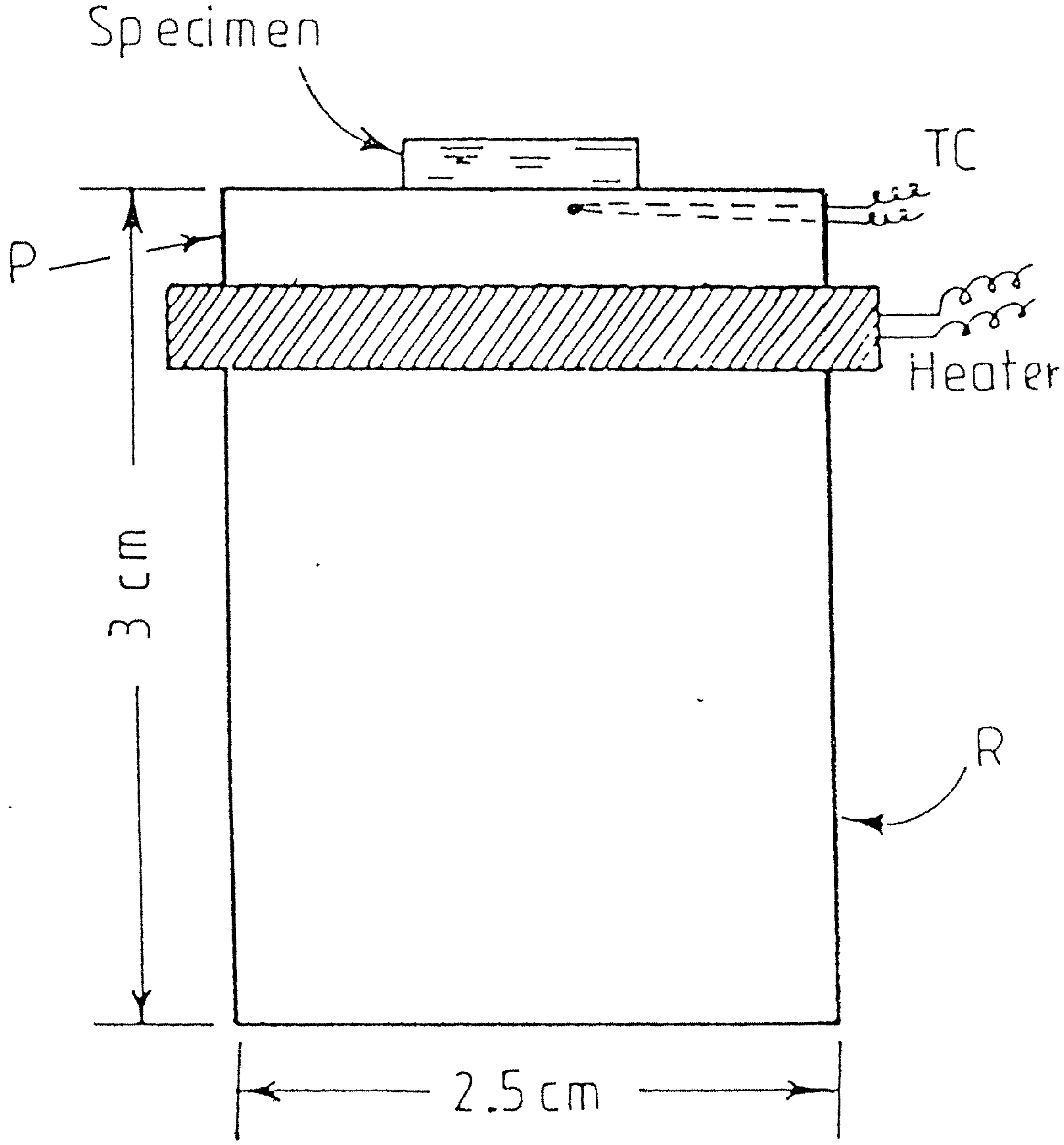

Fig.7

in use; such as clove geometry and linear four probe geometry as described by Van der Pauw[8] and Goswami[9]. For high value of resistance, linear four probe geometry is advisable. A sensitive current-meter and a stable power supply are connected in series with the sample. The voltage measurement is made by a sensitive and preferably a digital voltmeter. Change of resistivity with temperature is studied by keeping the sample in vacuum, of the order of 10^{-4} Pa, in order to prevent oxidation. For heating the substrate, radiant heater is used. The temperature is sensed by a thermocouple which is kept in similar conditions with the substrate. Externally, the thermocouple is connected to a digital millivoltmeter to measure the thermo -EMF. For resistivity measurement on crystals, the Valde's four probe geometry is used. Here, the pointed tips of closely spaced probes are contacted on the sample surface. The method does not require the probes to be ohmic [10]. This set-up is shown in Figure - 8.

For Hall measurement, linear four probe geometry is used. Charge carriers contributing the current caused by an applied electric field are deflected by a magnetic field applied perpendicular to the current. The resulting Hall voltage normal both to the current and the field can be measured. The sample is kept between the two poles of a strong electromagnet (Figure – 9) capable of magnetic field of the order of 18 kilo gauss. A sensitive current meter is connected in series with a stabilised DC power supply and the sample. Across the other two contacts, digital

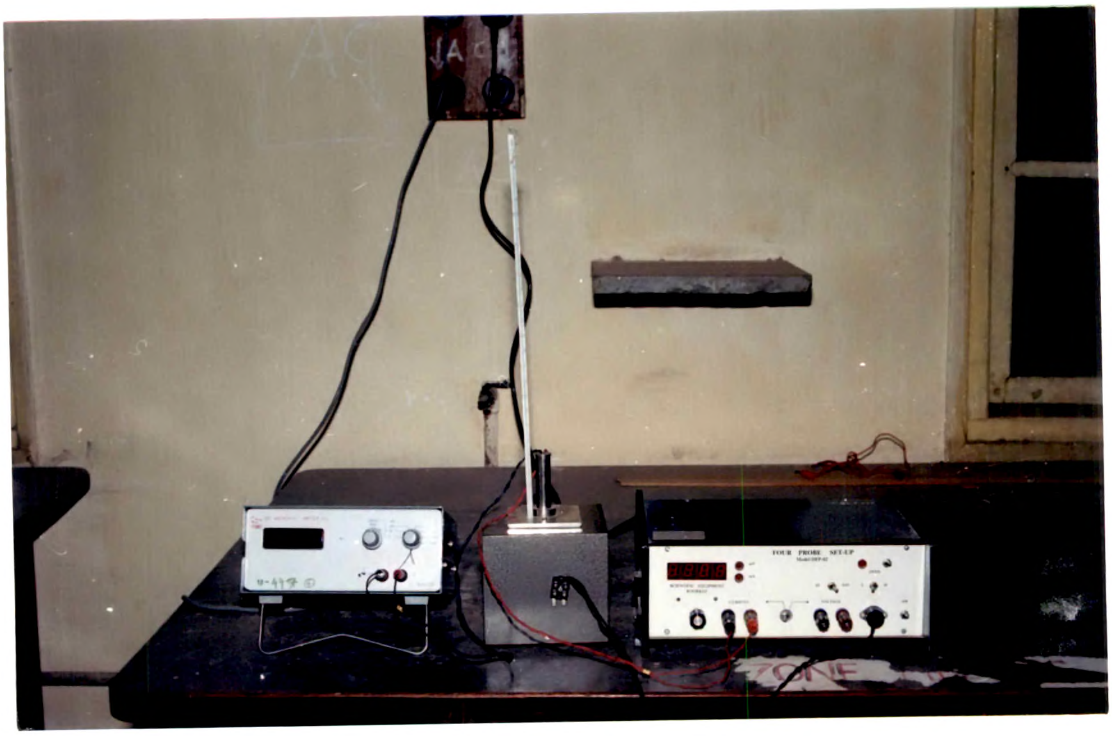

Fig. 8

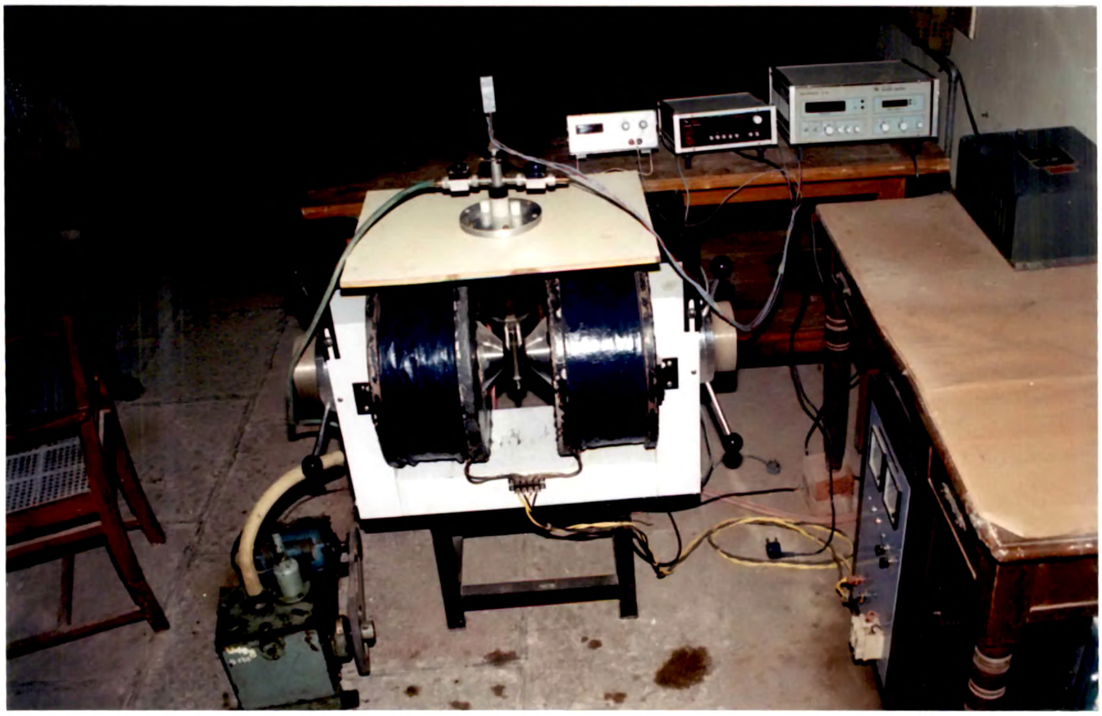

Fig. 9

microvoltmeter is connected. Current and voltage are measured in absence and presence of the magnetic field. For both the measurments, the electrodes should be ohmic.

Thermoelectric power measurments :

For the measurement of Seebeck coefficient [thermoelectric power (TEP)] as a function of temperature, a " Differential Temperature controller" developed by Scientific Solutions, Bombay was used. The thermopower measurement system, TPSS-200 is a versatile, low cost, specially developed integrated system for measurement of the thermo e.m.f. generated by the "Sample – stainless steel probe couple" in presence of a temperature gradient. The two matched main heaters mounted along the axis of the two sample holder shafts provide the controlled base temperature from 300 to 500 K. The auxiliary heaters wound on each of the sample holder shafts enable to provide the required gradient upto ± 10 K. The sign of the gradient can be changed by selecting one of the auxiliary heater coils.

The operation of the system can be understood from Figure-10 which is based on the principle of AC power control using controlled rectifiers. Using triacs as the power control devices the power to the heater and hence the temperature can be controlled. An error signal proportional to the difference of the measured and the set temperature along with external bias is

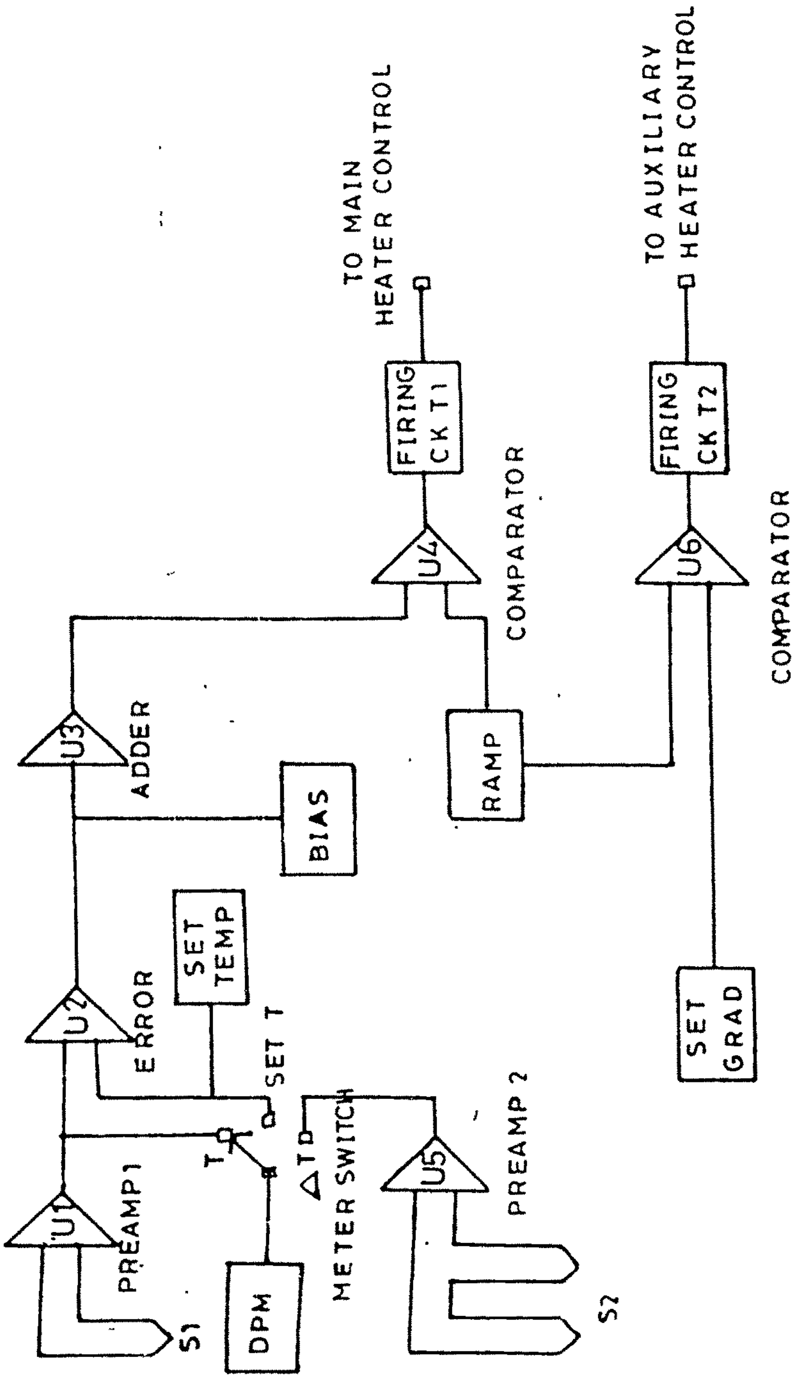
TO MAIN
HEATER CONTROL
TO AUXILIARY
HEATER CONTROL
FIRING
CKT1
FIRING
CKT2
COMPARATOR
COMPARATOR
U4
U6
U3
ADDER
RAMP
BIAS
SET
TEMP
SET
GRAD
U2
ERROR
SET T
T
ΔT D
METER SWITCH
U1
PREAMP1
U5
PREAMP 2
DPM
S1
S2

Fig. 10

used to decide the firing angle of the triac hence controlling the temperature.

The output of the sensor mounted on the main heater is amplified by the preamplifier U1 to get the voltage proportional to the temperature reading which is displayed as T. The same signal is also fed to the error amplifier U2 which compares this signal with the set temperature signal obtained from a stable reference source. The gain of the error amplifier controls the proportional band of the control and is presetted to about 30% of the full scale.

The error amplifier output is then summed with a bias signal from the bias control in the summing amplifier U3, the output of which is the voltage proportional to the temperature difference. The dc control signal is then compared to a linear ramp of 0-3 V (0 to full power) in the comparator U4. The output of the comparator which controls the firing angle of the triac is then given to the firing circuits. A similar low power control circuit is used to control the power to auxiliary circuit. Preamplifier U5 amplifies the output of the differential thermocouple for the indication of T which is the temperature gradient.

IX. MESAUREMENT OF OPTICAL PROPERTIES :

The I.R.Spectrophotometer (BOMEM, Canada, MB100 Figure - 11) was used for measurement of optical absorption or transmittance of the samples, in the wave number range 510 cm^{-1} to 6,000 cm^{-1}. In the case of

Fig. 11

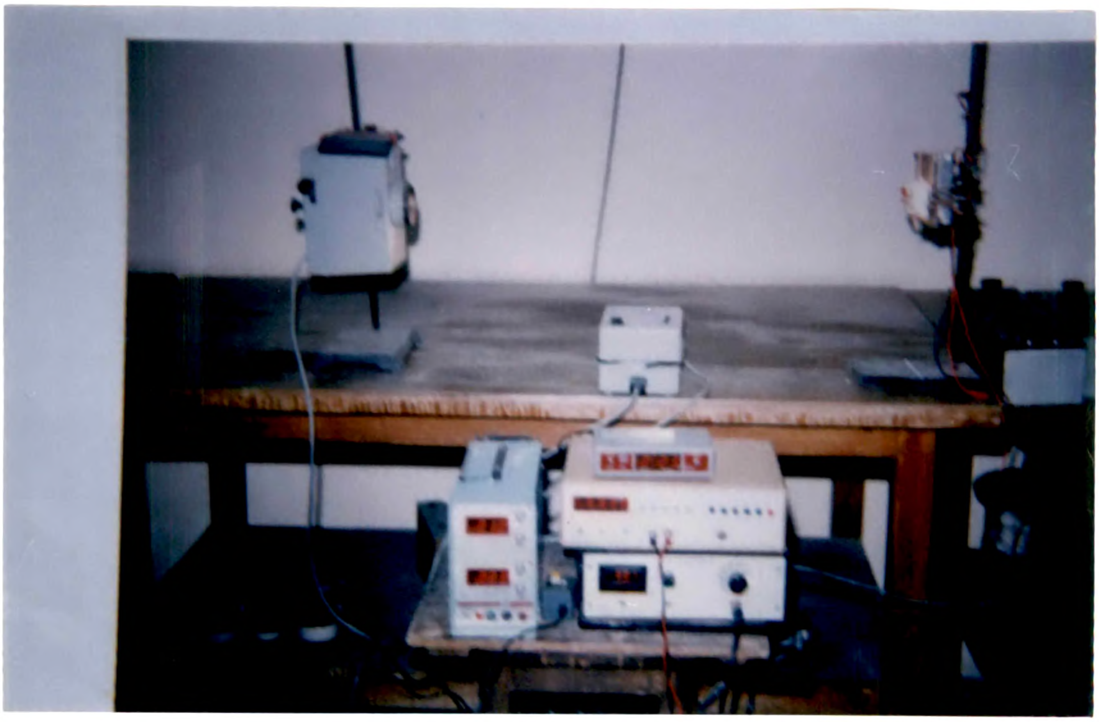

Fig. 12

crystals, fine crystalline powder was throughly mixed with spectroscopic grade dry KBr powder at about less than 5% concentration and the mixture was formed into a palette of about 1 cm. diameter using a vacuum palettizer. Whereas, in the case of thin films, the films deposited on KBr crystal substrates were used. The instrument acquires an interferogram and transforms it into a spectrum in terms of wave number. The wave number resoulution of the instrument is 4 cm^{-1}. It uses glowbar IR source and DTGS detector[11].

X. PHOTOCONDUCTIVITY MEASURMENTS :

Photoconductivity measurment is usually carried out in dark. For illuminating the sample a light source (like a Halogen lamp) is used. The sample geometry is rectangular. Finger shaped ohmic electrodes are deposited on the top of the film. The distance between the fingers is as small as practically possible. A digital voltmeter, microammeter and a regulated D.C. power supply are used for the measurements. The microammeter and regulated power supply are connected in series with the sample and the voltmeter is connected across it. A digital watch is used for the time measurement. Intensity of the light source is measured by a lux meter. The experimental set-up is shown in Figure-12.

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