CHAPTER - II

EXPERIMENTAL TECHNIQUES

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2.1 INTRODUCTION

The various experimental techniques employed during the course of this work are briefly described here. For quantitative and qualitative measurements of features on crystal surfaces, high resolution technique is used. To study the plastic deformation and other related phenomena the static indentation technique is employed. The diamond pyramidal indenters were used with Carl Zeiss Vertival microscope. Further, other techniques such as etch method, method for measuring electrical conductivity of etching solutions are also presented in this Chapter. Excellent accounts are available on optical microscopy (e.g. see Thompson, 1954), on indentation techniques (e.g. see Mott, 1956) and on electrical conductivity (e.g. see Harris, 1952).

2.2 VERTIVAL INCIDENT LIGHT MICROSCOPE : (CZ)

The vertival microscope manufactured by Carl Zeiss is one of the best and sophisticated instruments amongst the metallurgical microscopes. It can be used for different types of illuminations. Further, its utility is enhanced by providing different attachments which can be fitted to this microscope such as hardness testing unit, polarising accessories, multiple beam interference accessories etc. For efficient use of this instrument it is imperative to be familiar with various parts, arrangements for adjustment of coarse motion brake and illuminating unit incorporating bright field and dark field, for the coordination of concave mirror condensers etc. Excellent account of the above is given in the instruction manual supplied by the manufacturer. The basic unit of microscope consists of (i) the illuminating system (ii) the stage for placing the specimen (iii) body of the microscope carrying the objective and (iv) the monotube or binocular tube-arrangement (Fig. 2.3). The ray diagrams for bright and dark field illuminations are shown in Figures 2.1 and 2.2. The focussing arrangement is simple. It consists of fine focussing and coarse one. It is necessary to adjust the coarse motion brake.

(a) Adjustment of the coarse motion brake : (Fig. 2.3)

The instrument is usually supplied with the coarse motion brake released ; hence the smoothness of the coarse motion mechanism has to be adjusted by the users. This is done by holding fast one pinion head (1) and moving the other one in clockwise direction until the desired smoothness has been attained.

(b) Adjustment of illuminating equipment :

Having switched on the lamp, set switching knob (2) to bright field (see para d) and on opening

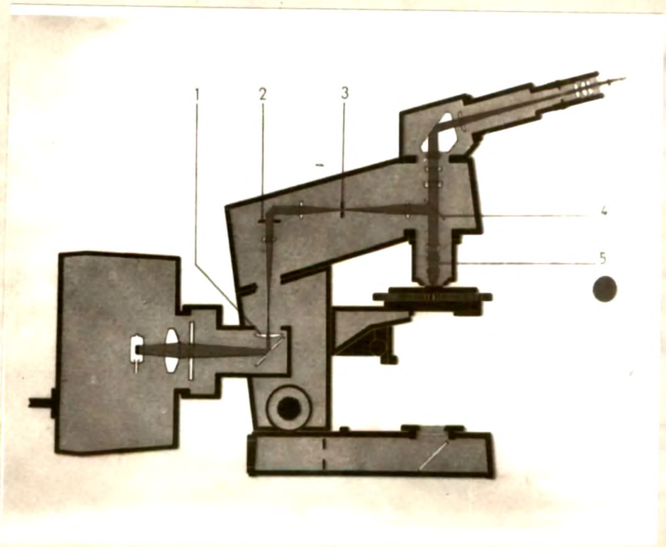

Fig. 2.1 Ray diagram for bright field

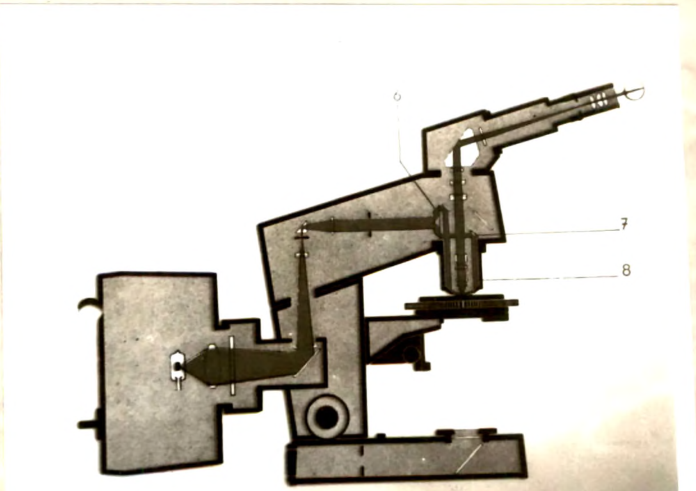

Fig. 2.2 Ray diagram for dark field

diaphragms (3 & 4), a bright circle becomes visible on the protective plate. This circle can be observed without eye-piece or - even better - after detaching angular tube. By turning the fixable so as to be loosened and moving pull-rod (not shown in Fig. 2.3) in axial direction the filament image is focussed on protective plate as critically as possible. The pull-rod is then again clamped in position and the filament image is centred by actuating centring screws (5).

(c) Coordination of concave mirror condensers :

The coordination of concave mirror condensers to the objectives is to be followed according to the instructions of the manufacturer. The concave mirror and slide especially adapted for objective 25X / 0.50 are marked with black point and those for objective 50X / 0.80 with a white point. The following magnification values refer to the equipment of carrier vertival (factor 0.63X) with angular tube (factor 1.6X) and monocular or binocular tube (factor 1X)

...... Contd.

TABLE 2.1

Objective	Concave mirror condenser	Eye-piece			
		PK 8X	PK 10X	PK 12.5X	PK 16X
6.3X/0.12	11	50X	63X	80X	100X
12.5X/0.25	12	100X	125X	160X	200X
25X/0.50	12	200X	250X	320X	400X
50X/0.80	12	400X	500X	630X	800X
HI 100X/1.30	-	800X	1000X	1250X	1600X

(d) Bright field : (Fig. 2.3)

For carrying out examinations in bright field the switching knob (2) has to be turned until the point to be found on it does no longer face the observer. Attention should be paid to the diaphragm slide (6) with an arrangement for luminous-field stop to bring it in centre,being inserted into the carrier to reach the maximum insertion point for proper alignment. A green filter, an attenuation filter or a froasted glass may be introduced optionally or in a combined form into the filter slide (7). This slide is provided with a free passage. Filter and shutter slides have to click indistinctly. The luminous-field diaphragm is centred by actuating the two centring screws (8) and the aperture diaphragm by making use of socket wrenches to be put on to the two centring screws (9). The image of the luminous-field diaphragm is to be seen within the image of the objective sharply depicted and that of closed aperture diaphragm in the exist pupil of the objective after having removed the eye piece.

(e) Dark field : (Fig. 2.3)

For investigations in the dark field the objective corresponding to the desired magnification has to be fitted with the concave mirror condenser coordinated to it as mentioned above in (c). Switching Knob (2) has to be set in such a way that the point to be found on it faces the observer. The luminous-field and aperture diaphragm are opened completely by actuating knurled rings (3 & 4).

(f) Camera attachment : (Fig. 2.4)

For taking photomicrographs of samples, camera is attached to the microscope. The arrangement is shown in Fig. 2.4. The advantage of this method is that it is possible to observe the sample while taking photographs. Exposure times can be automatically controlled.

2.2.1 INDENTATION TECHNIQUE :

mhp 160 microhardness tester : (Fig. 2.5)

The indenting device (19) and the threaded socket

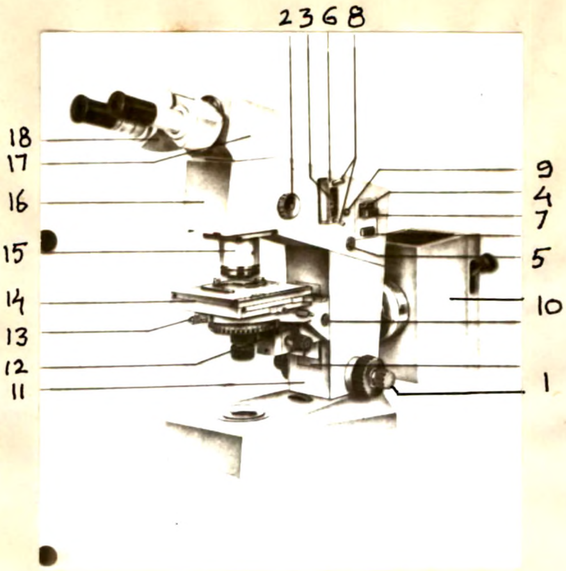

Fig. 2.3 Vertival incident light microscope

1. Pinion head
2. Switching knob
3. Diaphragm
4. Diaphragm
5. Centring screws
6. Diaphragm slide
7. Filter slide
8. Centring screws
9. Centring screws
10. 12/50 lamp with adapter
11. Basic stand for incident light
12. Stage carrier
13. Tightening screw
14. Specimen stage
15. Objective on slide with concave mirror condenser.
16. Carrier 'VERTIVAL'
17. Angular tube
18. Binocular straight tube

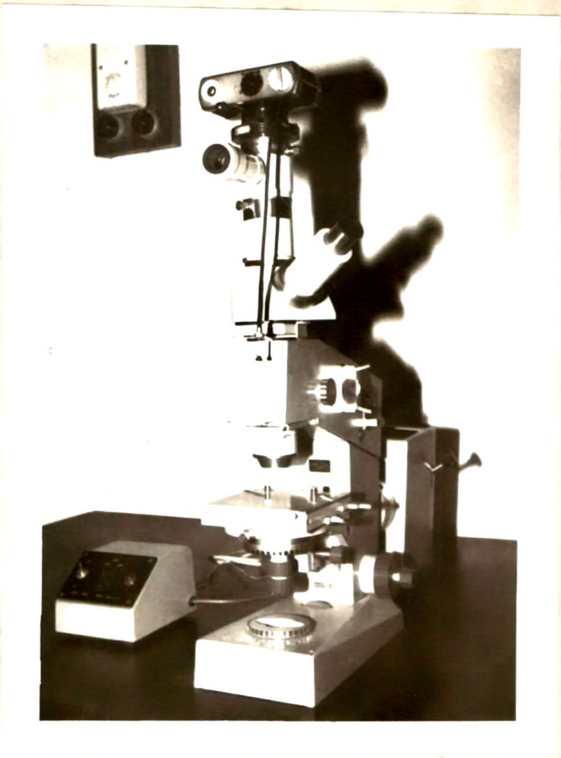

Fig. 2.4 Camera attachment on microscope

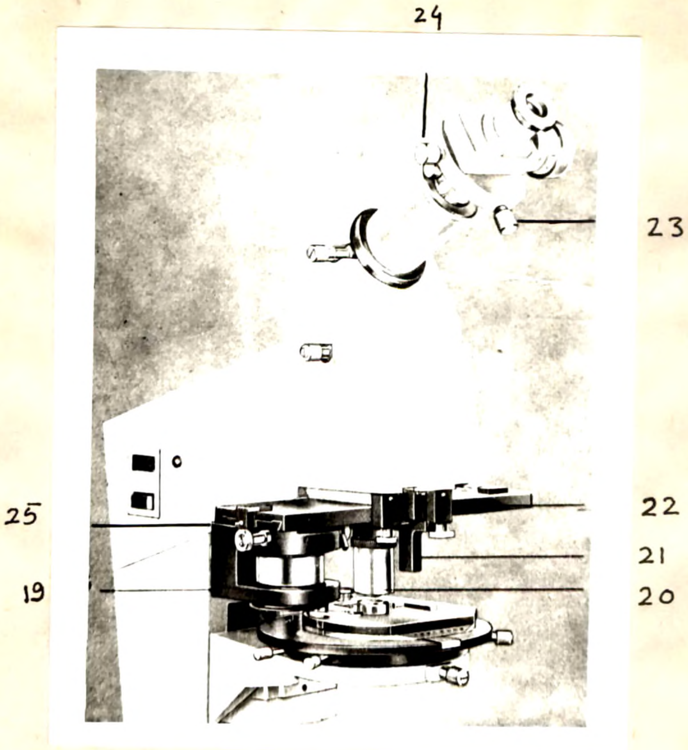

Fig. 2.5 Indentation technique

19. Indentation device
20. Objective
21. Handle
22. Slide
23. Centring screws
24. Centring screws
25. Setting screws
26. Setting screws

for objective (20) are mounted on a common carriage, which can be moved to and fro laterally by handle (21) in the slide (22) until it meets the stops. This makes it possible to place either the indenter device or the objective above the test specimen. The threaded socket also has a threaded collar for concave mirror condensers, so that the indentation can also be measured with peripheral dark field illumination.

A fitment is attached to the upper surface of the slide for mounting the microhardness tester in the corresponding mount of an upright incident light microscope. The indenter device is suspended from two springs, so that it is rather sensitive to vibrations, which are manifested by continuous or intermittment swings of the index lines on the force scale. If the vibrations exceed the tolerable level or (what is rarely the case) have a frequency that is in resonance with the natural frequency of the microhardness tester, provision must be made for absorbing the vibrations of the microscope or else the latter must be set up in a part of building subject to little vibrations. Otherwise, the necessarily inacturate application of the force, on the one hand, and the boring action of indenter on the other would result in errors that might affect the hardness readings considerably.

Various diamond indenters may be used with mhp 160 microhardness tester. Regular four-sided vickers indenter and the Knoop pyramidal indenter with a rhombic base are employed in the present investigation. They are kept in small screw top cases when not in use. One of them can be inserted into the tester mounting by means of a special clamp. A stud on the mounting and a corresponding slot in the indenter hold provide for correct alignment. The mhp 160 microhardness tester is a sensitive instrument that requires careful handling. Dropping it will certainly result in ruining its adjustment. The microhardness tester should always be kept in closed case when not in use to avoid dust settling on it. For optimum utilization of the tester, detailed instructions for its adjustment etc. are given below :-

(1) Level the stage (14) by using highly sensitive spirit level.

(2) Focus microscope (without microhardness tester) on to an object having striking features or on to a centring cross. The object must be flawlessly prepared (naturally or artificially) and mounted on the object stage (use plane field achromatic objective and eye piece with cross line or measuring rod).

(3) Move the object until striking feature or intersection of centring cross coincides with eyepiece cross.

(4) Exchange upper tube section having eyepiece for special tube with eyepiece screw micrometer and adjust.

(5) By turning the centring screws (23 & 24) bring the apex of fixed (left) measuring arm of eyepiece screw micrometer to coincide with the striking feature of object or centring cross. Both measuring arms form a cross (Zero position). (Fig. 2.6).

(6) Remove the objective with its slide.

(7) Insert microhardness tester with diamond indenter.

(8) Now screw formerly used objective into microhardness tester and place it in observation position. Critically focuss on to specimen.

(9) Centre the objective by turning setting screws (25 & 26) with socket wrench until the striking feature of the object (or centring cross) is again coinciding with apex of fixed measuring arm of dashed figure in micrometer eyepiece (Fig. 2.6).

(10) Turn the Knurled knob in a counter clockwise direction (27, Fig. 2.7) to lock the indenter.

(11) Move the change-over slide to indenter position.

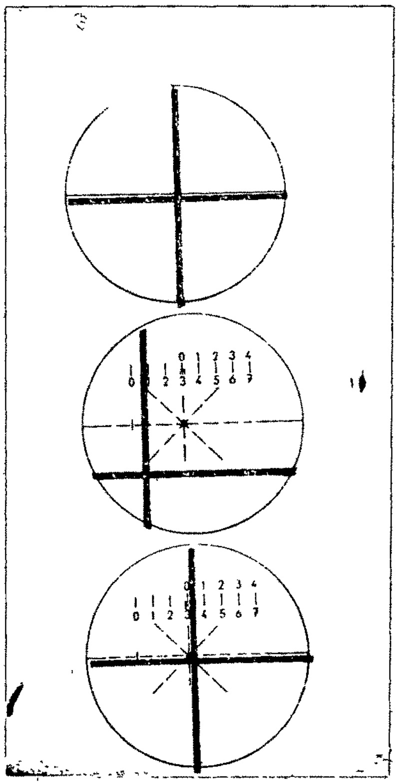

Fig. 2.6 Zero position

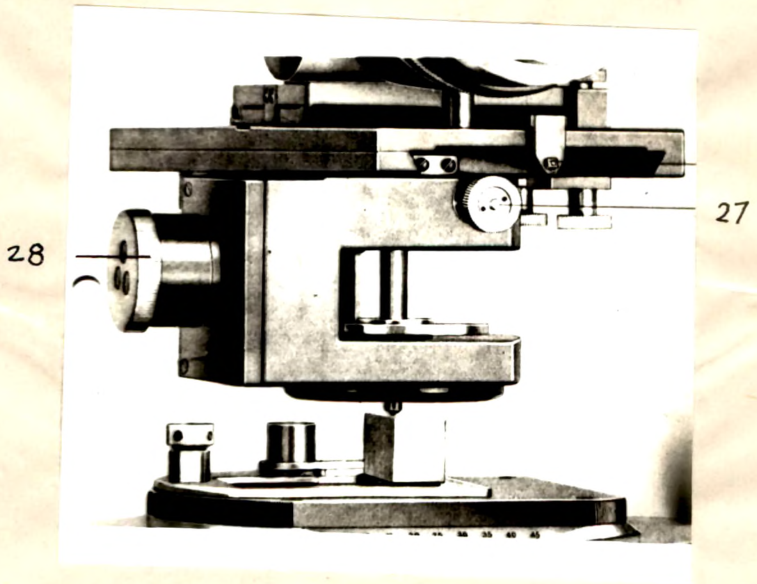

Fig. 2.7 Indentation technique

27. Knurled knob
28. Rear knurled knob

(12) Observe the horizontal bright index line of the load indicator in the eyepiece. It it is not seen, turn the rear knurled knob (28, Fig. 2.7) on microhardness tester until it is seen.

(13) Focus the line, if necessary, by adjusting micrometer eyepiece.

(14) Turn knurled knob until the index line coincides with horizontal line in the eyepiece. Should the eyepiece be oblique to the line, turn eyepiece. This fixes the initial poisition of index line.

(15) Apply suitable load.

(16) Observe the (upward or downward) motion of the index line within the image field.

(17) Observe with naked eye the tip of the indenter and simultaneously turn the fine motion knob of the microscope so that image of the tip is seen on the specimen.

(18) Now observe through eyepiece and turn fine motion knob so that the indenter just touches the specimen.

(19) When indenter touches the specimen the index line starts moving back. Turn the fine motion knob uniformly until the index line concides with original reference line.

(20) Turn fine motion knob in the reverse direction until the index line goes back to the same position (upward or downward).

(21) Now lower the stage considerably.

(22) Release the indenter by actuating knurled kbob (27) in clockwise direction.

(23) Remove load.

(24) Move the indenter device to its operating position and lock it.

(25) Turn knurled knob, if necessary, so that bright index line coincides with the horizontal line in the eyepiece.

(26) Observe the indentation mark through eyepiece and measure the length of the indentation mark with micrometer eyepiece.

(27) Compute the hardness number by using necessary formula.

2.3 ETCH METHOD :

This method consists of carefully preparing crystal surfaces and solutions of desired composition and concentration and of dipping the prepared crystal surface in the still solution for definite time at a constant temperature of etching and then gently washing it in a running solvent such as water which does not affect the crystal surface chemically. It is known that rinsing may deform the crystal if there is an appreciable change of temperature occurred at the time of rinsing. Hence adequate care in the present work was taken during rinsing of the crystal.

Big blocks of natural calcite crystal were cleaved in a usual way i.e. by keeping a sharp razor along cleavage direction and then giving a sharp blow. Every time freshly cleaved crystal surfaces were used.

2.3.1 PREPARATION OF A SOLUTION :

It is customary to express composition of a solution in following ways :

(1) Percentage by volume

Suppose X c.c. of X solution are poured in y c.c. of Y solution such that $x + y = 100$, then it is known as x% X solution.

e.g. (i) If 10 c.c. of formic acid are poured into 90 c.c. of water, then it is 10% formic acid.

(ii) If 35 c.c. of Lactic acid are poured in 65 c.c. of water, then it is 35% Lactic acid.

(2) Molarity :

The number of gram-molecules of a solute dissolved in one litre of the solution is called Molarity of the solution. This is also called molar concentration or molar strength, suppose that X grams of solute of molecular weight W are dissolved in total volume of Y litres, then the molarity M of solution will be

$$M = \frac{X}{W} \times \frac{1}{Y}$$

e.g. (i) 10 c.c. of formic acid (Mol. wt. = 46, density = 1.22) are dissolved in 90 c.c. of water, then the weight of formic acid will be

$$\begin{aligned} \text{Weight} &= \text{volume} \times \text{density} \\ &= 10 \times 1.22 \\ &= 12.2 \text{ grams.} \end{aligned}$$

Hence,

$$\begin{aligned} \text{Molarity } M &= \frac{12.2}{46} \times \frac{1}{\cancel{.09}\ 0.1} \\ &= 0.26 \times \cancel{11.11}\ 10 \\ &= 2.60 \end{aligned}$$

(ii) Suppose 35 c.c. of Lactic acid (Mol. wt. = 90, density = 1.20) are dissolved in 65 c.c. of water, then the weight of Lactic acid will be,

$$\text{Weight} = \text{volume} \times \text{density}$$
$$= 35 \times 1.20$$
$$= 42$$

Hence,

$$\text{Molarity} = \frac{42}{90} \times \frac{1}{\cancel{.065}\ 0.1}$$
$$= 0.46 \times \cancel{15.38}\ 10$$
$$\cancel{= 7.075} = 4.6$$

(iii) If 120 grams of KOH pellets are dissolved in 100 c.c. of water, then

(Mol. wt. of KOH = 56)

$$\text{Molarity M} = \frac{1200}{56} \times \frac{1}{1}$$
$$= 21.42$$

(3) Normality :

This is most widely used unit of concentration. Normality of an acid is given by,

$$\text{Normality} = \frac{\text{gms. per litre}}{\text{Eq. Wt.}}$$
$$= \frac{\text{grams per litre}}{\text{Mol.wt./basicity}}$$

$$\text{or} \quad N = \frac{\text{Volume per litre x density x basicity}}{\text{Mol. wt.}}$$

If percentage purity of an acid is given as P %, then

$$N = \frac{V \times d \times b \times P}{M \times 100}$$

where,

V = volume per litre

d = density of an acid

b = basicity of an acid

M = Molecular weight of an acid

P = Percentage purity of an acid

e.g. (i) If 10 c.c. of Formic acid ($M = 46$, $d = 1.22$, $P = 99\%$) are dissolved in 90 c.c. of water, then,

$$N = \frac{V \times d \times b \times P}{M \times 100}$$

$$= \frac{100 \times 1.22 \times 1 \times 99}{46 \times 100} \qquad (b = 1)$$

$$= 2.62$$

(ii) If 35 c.c. of Lactic acid ($M = 90$, $d = 1.2$, $P = 90\%$) are dissolved in 65 c.c. of water, then,

$$N = \frac{\cancel{60}\ V \times d \times b \times P}{M \times 100}$$

$$= \frac{350 \times 1.2 \times 1 \times 90}{90 \times 100}$$

$$= \frac{37800}{9000}$$

$$N = 4.2$$

(iii) Suppose 120 grams of KOH pellets is dissolved in 100 c.c. of water,

$$\text{Normality} = \frac{\text{grams per litre}}{\text{Eq.wt.}}$$

$$= \frac{\text{grams per litre}}{\text{Mol.wt.}}$$

$$= \frac{1200 \times 1}{56}$$

$$= 21.42$$

(4) Mole fraction :

Mole fraction is defined as

$$\text{m.f.} = \frac{\text{Weight of solute/Mol.wt. of Solute}}{\text{Weight of solvent/Mol.wt. of solvent}}$$

mole fraction multiplied by 100 is called Mole percent.

e.g. (i) If 10 c.c. of formic acid are dissolved in 90 c.c. of water, then,

Weight of formic acid = volume x density

= 10 x 1.22

= 12.2 grams

Mol. wt. of formic acid = 46

Weight of Water = 90 x 1 = 90 grams.

Mol. wt. of water = 18

Hence,

$$\text{m.f.} = \frac{12.2/46}{90/18}$$

$$= \frac{12.2 \times 18}{90 \times 46} = \frac{219.6}{4140} = .0530$$

Therefore, mole percent = 0.053 x 100 = 5.3

(ii) If 35 c.c. of Lactic acid are dissolved in 65 c.c. of water, then,

Weight of Lactic acid = Volume x density

= 35 x 1.2

= 42

Mol. wt. of Lactic acid = 90

Wt. of water = 65 x 1 = 65

Mol. wt. of water = 18

Hence,

$$\text{m.f.} \quad \frac{42/90}{65/18} = \frac{42 \times 18}{90 \times 65} = \frac{756}{5850}$$

$$= 0.1292$$

mole percent = 0.1292 x 100 = 12.92

(iii) Suppose 120 grams of KOH is dissolved in 100 c.c. of water.

$$\text{m.f.} = \frac{\text{Wt. of KOH/Mol.wt. of KOH}}{\text{Wt. of Water/Mol.wt. of Water}}$$

$$= \frac{120/56}{100/18}$$

$$= \frac{120/18}{56/100}$$

$$= 0.3857$$

Mole percent = 0.3857 x 100 = 38.57

2.3.2. ETCHING AT LOW AND HIGH TEMPERATURE :

For temperatures lower than room temperature, the beaker containing etchant of desired concentration was kept in a container such that beaker was surrounded by ice, Etching time for etchants used in the present investigation viz. formic and lactic acids is quite small. Hence the temperature could be maintained constant for a few minutes.

Chemical etching can be carried out at high temperatures to get quick reaction between etchant and the crystal surface. In this method, the beaker containing etchant is kept in a constant temperature water bath. (Bath is shown in Fig. 2110).

The washed and dried crystal surfaces were optically studied by using CZ vertival microscope. The length of etch pits along [110] was measured using filar micrometer eyepiece. The rate of surface dissolution is measured by weight-loss method using semi-micro balance described below :

2.4 SEMI MICRO BALANCE : (Fig. 2.8)

The balance utilizes the principle of weighing by substitution and the constancy of sensitiveness over all loads. The later feature distinguishes it from ordinary beam balances where the sensitivity depends on load, i.e. it changes with load in a way which reflects the relative positions of the three knife edges viz. left, central and right on which the beam rests. Procedure for weighing the specimen is as follows :-

(1) Determine the approximate weight of the given specimen by ordinary beam balance.

(2) Put the specimen on the pan and close the balance to avoid effect of vibrations due to air.

(3) Switch on A.C. mains.

(4) Adjust the approximate known weight by moving knobs 1, 2 and 3. (Fig. 2.8)

(5) Make the pan free by turning knob 5 anticlockwise.

(6) Scale will be seen in window W. Let it be steady.

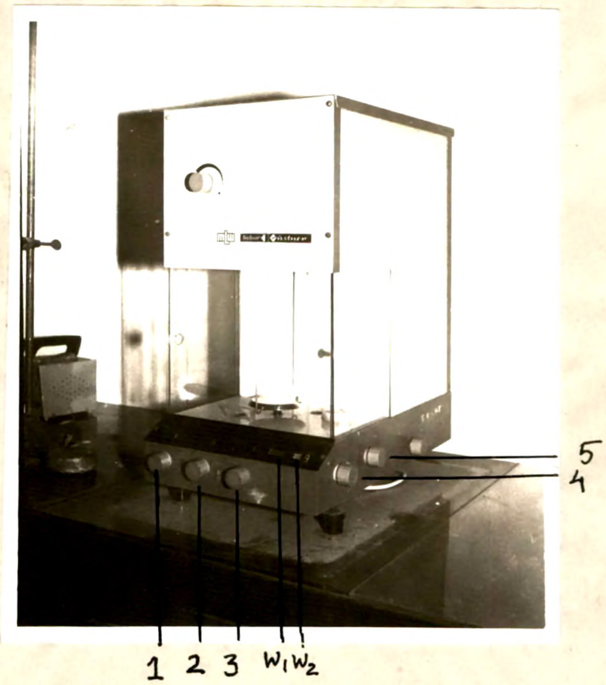

Fig. 2.8 Semimicrobalance

1. Knob giving weights in multiples of 10 gram.
2. Knob giving weights in multiples of 1 gram.
3. Knob giving weights in multiples of 0.1 gram.
4. Knob for adjusting 4th and 5th digit after decimal point.
5. Knob to make pan free.

W_1 & W_2 - Scale windows.

(7) Turn knob 4 such that marked black line coincides with scale.

(8) Note the reading in following way :-

  (a) Knob 1 indicate scale reading which corresponds to weight in multiples of 10 gm.

  (b) Knob 2 indicates scale reading which corresponds to weight in multiples of 1 gm.

  (c) Knob 3 indicates scale reading which corresponds to weight in multiples of 0.1 gm.

  (d) Scale seen in window W_1 corresponds to second and third digits after decimal point.

  (e) Scale seen in window W_2 corresponds to 4th and 5th digits after decimal point.

(9) Turn knob 5 clockwise to half-way position such that the light is not seen in window W_1. This operation locks the pan.

(10) Open the glass shutter and take out the sample carefully.

(11) Switch off the A.C. supply.

The balance is highly sensitive to jerks, shocks and vibrations. Hence adequate precautions must be taken to avoid this. Some of them are as under :

(1) Operate the balance with care.

(2) Do not touch the table carrying the balance.

(3) Rotate the knobs slowly and gently.

(4) After putting a specimen on pan, close the glass shutter.

2.5 DETERMINATION OF CONDUCTIVITY OF LIQUID (SOLUTION)

2.5.1 Introduction :

The usual classification of bodies into conductors, semiconductors and non-conductors (or insulators) is not an absolute one. A non-conductor is an ideal material. In practice it is merely a material whose conductivity is negligibly small, taking into consideration the particular purpose for which the material is to be used. An electric current in the above materials may consist of

(i) a flow of electrons, or

(ii) a flow of electrically charged molecules, or

(iii) a flow of both ions and electrons.

The first type of conduction is known as electronic whereas the second type as electrolytic or ionic. There are also materials in which the conduction is both electronic and ionic. Electrolytic conductors are electrolytes. They are distinguished from electronic conductors by the fact that the passage of an electric current is accompanied by the transfer of matter and the current is carried by ions and not by electrons. Electrolytic conductors are of two types ; (i) pure substances (liquids) and (ii) solutions. In the present work the dislocation-etchants used for delineating dislocations ending on a crystal surface are electrolytes for which the conductivities are determined for different etchant concentrations and for different temperatures.

It is necessary to define a few of the terms involved in the determination of conductivity of etchants. Electrolytes, like metallic conductors, obey ohms law, except under such abnormal conditions as very high voltages or with very high frequency currents. It follows from ohm's law that the current (C in ampere) flowing through a conductor, under the influence of a constant e.m.f., is inversely proportional to the resistance (R in ohm). The quantity 1/R is a measure of conducting power and is called conductance (G in mho). It is well known that at a constant temperature the resistance of any conductor varies directly as its length (l in cm.) and inversely as its cross sectional area (A in sq.cm.) Thus,

$$R = \frac{\rho l}{A} \qquad \ldots\ldots\ldots(2.1)$$

where ρ is a constant called specific resistance or resistivity of the conducting material. Its unit is ohm-cm. l/A is a constant of the conductor. The reciprocal of the resistivity is called conductivity (σ_c) or specific conductance. Thus

$$\sigma_c = \frac{1}{\rho}$$

$$\text{or} \quad \sigma_c = \frac{1}{AR} \qquad \ldots\ldots(2.2)$$

The specific conductance is measured in mho. cm^{-1} or $ohm^{-1}.cm^{-1}$. The conductance of an electrolyte is given by

$$\sigma_G = \frac{1}{R} = \frac{A}{l\rho} \text{ mho} \qquad \ldots.(2.3)$$

For an electrolyte of constant volume and given electrodes of fixed areas, l/A in formulae 2.1, 2.2 and 2.3 has a constant value. Thus for an electrolyte

$$\rho \propto R \quad \text{or} \quad \rho = CR \qquad \ldots\ldots.(2.4)$$

where C is called a constant of the cell (or vessel) containing electrolyte and electrodes. Thus

$$C = \frac{\rho}{R} \qquad \ldots\ldots..(2.5)$$

2.5.2 Wheatstone net :

The resistance of a conductor is measured by employing either a potentiometer method or a wheatstone bridge arrangement. The second method is normally used. The passage of a direct current through an electrolytic conductor produces polarisation. This increases its resistance. A continued passage of current through such a liquid conductor creates decomposition which causes a change in the resistance due to alteration of the concentration of the electrolyte. These effects are avoided or reduced to a negligible amount by using alternating current. This is achieved by using small, rapidly changing alternating current and by employing identical electrodes of fairly large area. Thus the conventional wheatstone net is modified by employing an alternating source of emf, say output of an induction coil giving a low emf in place of D.C. source, e.g. battery and by using a telephone (or head-phone) in place of galvanometer as a detector. Further the self inductance should be very small. This is achieved by a wire bridge instead of a post office box. The modified wheatstone bridge is shown in figure 2.9. A stretched wire AB is connected to an alternating source of emf e. T is a telephone or head-phone and R is an adjustable resistance whereas V is a vessel containing electrodes and a known quantity of electrolyte (liquid or solution) of resistance R_x under consideration. For taking measurements at different high temperatures the vessel with electrodes and electrolyte is placed in a thermostatically controlled water bath.

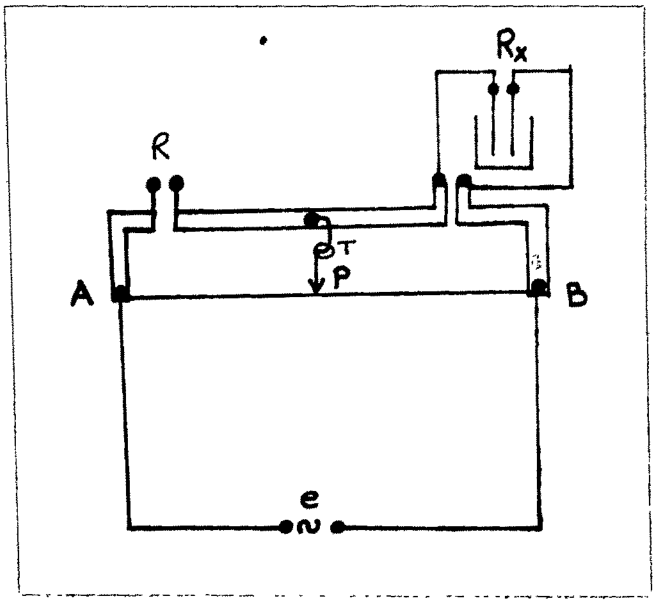

Fig. 2.9 Wheatstone net

R is adjusted so that a point P is obtained near the centre of the bridge such that the sound in the telephone is entirely cut out or reduced to a minimum. When the usual wheatsone result is applied,

$$\frac{R}{R_x} = \frac{AP}{PB}$$

$$\text{or} \quad R_x = R\,\frac{PB}{AP} \qquad \ldots\ldots.(2.6)$$

The specific resistance of the electrolyte of resistance R_x can be obtained by using the formula 2.4. It is given by

$$\rho = C\,R_x \qquad \ldots\ldots.(2.7)$$

and the electrolytic conductivity is given by

$$\sigma_c = \frac{1}{\rho}$$

$$= \frac{1}{C\,R_x} \text{ mho - cm}^{-1} \quad \ldots.(2.8)$$

$$\text{or } C = \frac{\rho}{R_x} \qquad \ldots\ldots.(2.9)$$

C is a constant depending upon dimensions of the cell (vessel). It is obtained by finding R_x for a liquid of known specific conductance.

2.5.3 Conductivity measuring bridge :

The 'Philips' measuring bridge PR 9500/90 is a compact and versatile unit (fig. 2.10). It works on the

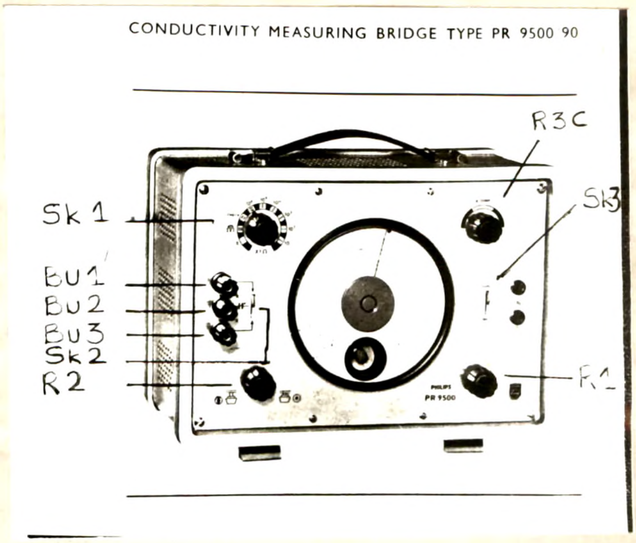

Fig. 2.10 Conductivity measuring Bridge

principle of wheatstone's bridge, which is slightly modified as mentioned above. Excellent account of this is given in ' operating manual ' of the bridge supplied by Philips, India. The characteristic features of the bridge are as follows :

(i) Electronic indicator is used as a detector instead of a telephone. ~~The reading taken by using a telephone.~~ The reading taken by using a telephone depends on the sensitivity of a human ear which is obviously not a reliable factor for accurate observations. Thus the introduction of electronic detector increases the accuracy of observation. It is a cathode-ray indicator. It is inertialess, resistant to mechanical shocks and vibrations and enables parallax-free reading.

(ii) The bridge circuit can be fed either at 50 c/s mains frequency voltage for electrolytes having low conduction or a suitable voltage at 1000 c/s for liquids of high conductivity. In both the cases the wheatstone net is supplied by alternating voltage, 3.6 v. The bridge is connected to mains supply viz. 230 V, 50 c/s by screened cables. For supply at 1000 c/s apparatus has built-in oscillator. The power consumption of the bridge is approximately 20W.

(iii) A number of standard resistances in the bridge circuit with which the resistance of the solution to be measured is compared are incorporated in place of R (fig. 2.9) i.e. connected to sockets Bu_1 and Bu_2 (fig. 2.10, 2.11). It also enables the working in the ' open bridge ' position in which the deviations, from between the standard resistance and the resistance under test, are indicated as a percentage.

(iv) It is used for determining and comparing specific resistances and conductivities of aqueous solutions with a wide range of conductivity cells. The measurement of ohmic resistances can be over a wide range, namely from 0.5 ohm to 10^7 ohm and that of specific resistances of liquids from 7 to 14 x 10^6 ohm-cm. The percentage comparisions are from - 20% to 25% of the scale. Absolute error, including reading error varies with the range and region of the scale. In the middle of the scale region it is less (about 1.5% to 2.5%) and increases at the scale ends (about 3 to 3.5%). The accuracy of measurements is not affected by fluctuations in the mains supply (230V, 50 c/s) upto ± 10%.

2.5.4 Measurement of Conductivity :

An experimental set up for measuring conductivity of a given solution is shown in fig. 2.11. The position of various connection sockets and operation controls are indicated in fig. 2.10. For efficient use of the bridge it is imperative to proceed in following way :-

(1) Connect the terminals of the measuring cell to sockets Bu2 and Bu3 marked R_X on the text plate of measuring bridge. Stir the solution to make it absolutely homogeneous. Ensure that no air bubbles adhere to the cell. During the measurements the space between platinum electrodes should be entirely filled with the liquid. Since conductivity of a liquid varies with the temperature, it is necessary to insert a thermometer in the beaker containing liquid. For measurements at various temperatures, the beaker containing liquid is kept in a thermostatically controlled water bath (Fig. 2.11) with the accuracy of $\pm 0.5^{\circ}C$.

(2) Set the switch Sk3 to the position " 1000 " when measuring well conducting liquids and to the position " 50 " when measuring very weak electrolytes.

(3) Switch on the bridge with R2.

(4) Select the measuring range with the switch Sk1, within which the resistance of the liquid is expected to be.

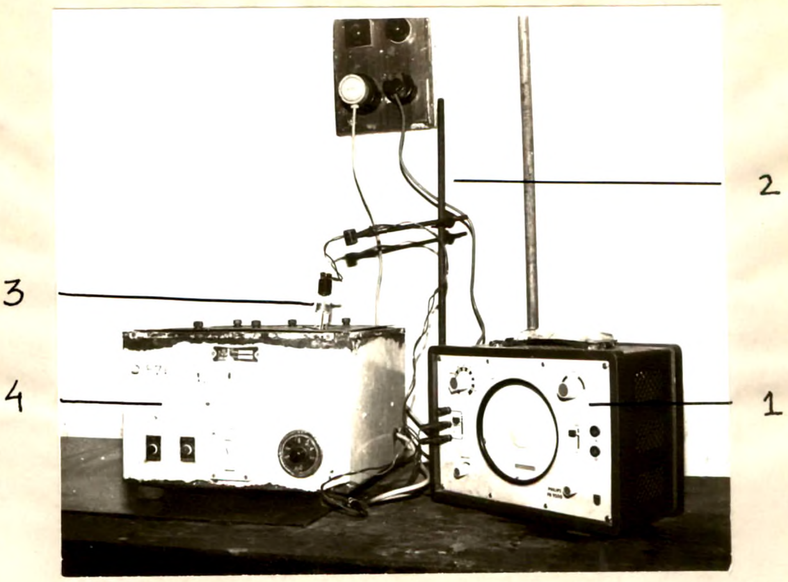

Fig. 2.11 An experimental set up for measuring conductivity of a solution

1. Conductivity bridge.
2. Retort stand.
3. Conductivity cell.
4. Constant temperature water bath.

(5) Turn knob R1 and move the pointer on the scale until minimum shadow length is obtained in cathode-ray indicator.

(6) The value of resistance Rx is then found by taking the number indicated for the position of the switch Sk1 and multiplying it by reading indicated by the pointer on the scale.

(7) Specific conductivity of the liquid is then given by the formula

$$\text{Specific conductivity } (\sigma_c) = \frac{1}{C \cdot R_x} \text{ Mho per cm.}$$

where C is the cell constant. In the present case it equals 1.41.