CHAPTER II

TECHNIQUES AND GRAPHICAL ANALYSIS

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2.1 INTRODUCTION:

The various experimental techniques employed and graphical analysis carried out during the present study are briefly described here. For the quantitative and qualitative study of features on the crystal surfaces, optical techniques were used. To study plastic deformation and other related phenomena the static indentation technique was employed. The diamond pyramidal knoop indenter was used with VERTIVAL incident light microscope. The crystals of sodium nitrate were grown from melt. Besides etch method, electrolytic conductivity bridge, semimicro-balance and heating devices used for present work are also described in this chapter.

The techniques mentioned above are well discussed in several standard text books /1,2,3,4,5/. Hence only a brief description together with their salient features will be given here.

2.2 VERTIVAL INCIDENT LIGHT MICROSCOPE (CZ):

The VERTIVAL incident light microscope manufactured by Carl Zeiss (CZ), Jena is one of the best and sophisticated instruments amongst the metallurgical microscopes. It can be used for different types of illuminations. 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. The instruction manual supplied by the manufacturer gives excellent account of this microscope and accessories. The basic unit of microscope consists of (i) the illuminating system, (ii) the stage for placing the sample, (iii) the body of the microscope carrying the objective and (iv) the monotube or binocular tube arrangement (Fig.2.1). The ray diagrams for bright and dark field illuminations are shown in figures 2.2 and 2.3. The focussing arrangement consists of fine focussing and coarse one. It is necessary to adjust the coarse motion brake.

(a) Adjustment of the coarse motion brake (Fig.2.1):

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

(b) Adjustment of the illuminating equipment:

Having switched on the lamp, set switching knob [2] to bright field (see para d) and on opening diaphragms [3 and 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 side screws so as to be loosened and moving pull-rod (not shown in Fig.2.1) in axial direction, the filament image is focussed on protective plate as much 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 (1.6x) and monocular or binocular (1x).

TABLE 2.1

Objective	Concave mirror condenser	Eyepiece			
		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.2.2):**

For carrying out examination of a specimen in bright field the switching knob [2] has to be turned until the point to be found onto 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 frosted 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 eyepiece.

(e) **Dark field (Fig.2.3):**

For investigations in the dark field the objective corresponding to 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 and 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.

2.3 INDENTATION TECHNIQUE:

mhp 160 microhardness tester: (Fig.2.5)

The indenting device [19] and the threaded socket 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 stop. 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 intermittent swings of the index line 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 inaccurate 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 pyramidal indenters may be used with mhp 160 microhardness tester. The knoop pyramidal indenter with rhombic base is employed in the present investigation. It is kept in small screw top case when not in use. It 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 a closed case when not in use to avoid dust settling on it. For optimum utilization of the tester detailed instrutions for its adjustment, etc., are given below:

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

(2) Focus microscope (without microhardness tester) onto an object having striking features or onto a centring cross. The object must be flawlessly prepared (naturally or artificially) and mounted on the object stage (use plane field achromatic objective and eyepiece 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 and 24] bring the apex of fixed (left) measuring arm of eyepiece screw micrometer to coincide with the striking feature of objeect 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 focus onto specimen.

(9) Centre the objective by turning setting screws [25 and 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 [27] in a counter-clockwise direction (Fig.2.7) to lock the indenter.

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

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

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

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

(15) Apply suitable load.

(16) Observe the upward 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 coincides with the original reference line.

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

(21) Now lower the stage considerably.

(22) Release the indenter by actuating knurled knob [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 by using necessary formula.

2.4 ETCH METHOD:

(a) Chemical etching:

This method consists of carefully preparing crystal surfaces and solutions of desired composition and concentration and 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 crystals were cleaved in the usual way, i.e., by keeping a sharp razor along a cleavage direction and then giving a sharp blow. Everytime freshly cleaved crystal surfaces were used, for etching in a freshly prepared solution.

(b) Composition of solution:

The amount of solute dissolved in a unit volume of solvent is called concentration. There are various ways of expressing the concentration of a solution. Different terms commonly used for the purpose are as follows:

(i) Percentage by volume:

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

e.g., if 10 c.c. of formic acid is poured in 90 c.c. of acetic acid to form a solution of 100 c.c., then it is 10 % formic acid solution in acetic acid.

(ii) Molarity:

The number of gram molecules of a solute dissolved in one litre of the solution is called molar concentration or molar strength. Suppose that 'X' grams of solute having molecular weight 'W' are dissolved in a solvent to make total volume 'Y' litres, then the molarity M of the solution will be,

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

e.g., 15.009 gm of L(+) tartaric acid is dissolved in 100 c.c. (= 0.1 litre) of distilled water, then molarity of solution is given by (Molecular weight of L(+) tartaric acid = 150.09 gm/mole).

$$M = \frac{15.009}{150.09} \times \frac{1}{0.1}$$

$$M = 1 \text{ Mole.}$$

(iii) Normality:

Normality of an acid is given by,

$$\text{Normality} = \frac{\text{gm/litre}}{\text{Eq. Wt.}}$$

$$= \frac{\text{gm/litre}}{\text{Mol. Wt./basicity}}$$

$$= \frac{\text{gm x basicity}}{\text{Mol. Wt. x litre}}$$

e.g., when 30 gm of L(+) tartaric acid (Mol. Wt. 150.09, basicity = 1) is dissolved in 100 c.c. (= 0.1 litre) of distilled water;

$$\text{Normality} = N = \frac{30 \times 1}{150.09 \times 0.1}$$

$$= 1.998$$

$$2\ N$$

(iv) Mole fraction (m.f.):

It is defined as,

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

m.f. x 100 = mole percent.

e.g., 50 gm of L(+) tartaric acid (T.A.) (mol. Wt. = 150.09) when dissolved in 100 c.c. of water (Mol. Wt. = 18)

$$\text{m.f.} = \frac{\text{Weight of L(+) tartaric acid/Molecular weight of T.A.}}{\text{Weight of water/Molecular Weight of water}}$$

$$= \frac{50 / 150.09}{100 / 18}$$

$$= 0.0599$$

$$\text{mole percent} = 0.0599 \times 100$$

$$= 5.99 \%$$

(v) Molality or weight molar concentration:

Molality of a solution is defined as the number of moles of solute dissolved per kilogram of a solvent. It is denoted by m. For example, 1-molal (1.0 m) aqueous solution of L(+) tartaric acid (molar mass = 150.09 gm) is obtained by dissolving 150.09 gm of L(+) tartaric acid in 1 kg of water.

$$\text{Molality} = m = \frac{1000\ W_B}{M_B\ W_A}$$

$$= \frac{1000 \times 150.09/1000}{150.09 \times 1}$$

$$= 1$$

Where,

W_B = Wt. of solute = $\frac{150.09}{1000}$ kg.

M_B = molar mass of solute = 150.09

W_A = Wt. of water in kg = 1 kg.

(vi) Weight percent (% by weight):

Weight of a solute in grams present in 100 gm of solution is termed weight percent. In a solution containing 5 gm of L(+) tartaric acid in 45 gm of water, the weight percent

$$= \frac{5}{5 + 45} \times 100 = 10\ \%\ \text{by weight.}$$

(c) Etching at different temperatures:

The etching was normally carried out in two different ways, depending upon the temperature of etching, viz., etching at

(i) room temperature,

(ii) high and low temperature.

(i) Etching at room temperature:

Since the rate of stirring of etching solution noticeably affects the etch pattern at room temperature, etching was carried out under condition of static etching in which, the motion of etchant relative to the crystal being etched is zero, i.e., no stirring.

(ii) Etching at high and low temperatures:

Chemical etching can be done at high temperature to get quick reaction between the etchant and a crystal surface. High temperature etching alters the rate of etching. In this method etchant is therefore kept in an incubator or a muffle furnace or in water bath (depending upon the etching temperature) for obtaining the required temperature.

At low temperatures (ice temperature or lower), the etching work was carried out in a container containing etchant in a beaker which was placed in a container or thermoflask containing ice or ice-water or dry ice or liquid air. Low temperature etching reduces the etching rate. The washed and dried crystal surfaces were optically studied by using CZ VERTIVAL microscope. The length of etch pit along [110] was measured using filar micrometer eyepiece. The rate of surface dissolution is measured using semi-microbalance described in the following section.

2.5 SEMI-MICROBALANCE: (Fig.2.8)

The balance utilizes the principle of weighing by substitution and the constancy of sensitivity for all loads. The later feature distinguishes it from ordinary beam balance where the sensitivity depends on load, i.e., it changes with the load in a way which reflects the relative positions of three knife edges, viz., left, central and right on which the beam rests. Further, in contrast to the ordinary balance, the present one is fully loaded when used.

The balance consists of three parts:

(i) Illumination system,
(ii) Optical system, and
(iii) Weighing system.

Procedure for weighing a specimen:

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

(2) Put the specimen on the pan. Close the shutters of the balance to avoid effect of vibrations due to air on the specimen/pan.

(3) Switch on A.C. mains.

(4) Adjust the approximate known weight by moving knob 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_1. Allow it to be steady.

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

(8) Note the reading in the following way:

  (a) Knob 1 indicates 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 2nd and 3rd after decimal point.

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

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

(10) Open the glass shutter.

(11) Take out the sample carefully.

(12) Switch off the A.C. mains.

Note: 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 knob slowly and gently.

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

(5) This shutter should be opened after completing the entire operation for weighing.

2.6(A)FURNACE, OVEN AND WATER-BATH:

(a) **Furnace:** (Fig.2.9)

A cylindrical crucible type furnace with vertical temperature gradient manufactured by Tempo Electric Co., Bombay was employed in the present work for growing crystals of sodium nitrate from melt and also for quenching natural crystals of calcite.

It consists of (i) heating element, a helix wound between refractory material with lead out and connections in the rear, (ii) temperature regulator, comprising of energy regulator unit, (iii) temperature indicator in the form of a thermoelectric pyrometer, and (iv) safety devices. The furnace is closed at the bottom and is opened at the top which can be covered by a thermally insulated door. Energy regulator unit (dial) is calibrated in terms of temperatures so that the desired temperature can be obtained and maintained constant. The cylindrical working space in the furnace has inner diameter 10 cm and height 17 cm. The maximum temperature which can be obtained in it is 1200°C. The accuracy of temperature with the stabilized power supply is ± 3°C for long periods.

(b) **Oven: (Fig.2.10)**

A cubical oven, manufactured by National Scientific Works, India, consists of (i) heating element at the base, covering the base area for uniform heating, (ii) temperature regulator, comprising of an energy regulator unit with indicator lamps and thermocouple connected at base, and (iii) a mercury thermometer having its bulb inside the oven at the top portion and bent stem leading out of the oven. Energy regulator unit (dial) is calibrated in terms of temperatures so that the desired temperature can be obtained and maintained constant for long periods. The maximum temperature which can be obtained is 250°C with temperature variations of ± 1°C round the desired temperature. The stabilized power is used. The dimensions of rectangular working space are 20 x 20 x 20 inch. The oven was used for annealing of grown crystals of sodium nitrate as well as for quenching them.

(c) **Water-bath:** (Fig.2.11)

Thermostatically controlled water-bath manufactured by National Scientific Works, India, was employed for etching the crystal cleavages. The water-bath consists of (i) U-shaped immersion heater of 1000 Watts, and (ii) temperature regulator comprising of energy regulator unit with indicating lamps and temperature sensing device (thermocouple) connected at the centre of the bath. Energy regulator unit (dial) is calibrated in terms of temperatures so that the desired temperature can be obtained and maintained constant. A mercury thermometer was employed to read temperature with least count 0.1°C. The accuracy of the temperature is ± 0.5°C. The water-bath has a metallic cover with six circular holes of diameter 9.0 cm and each hole could be fully covered with lids. Glass wool is used in the walls and base of the bath for obtaining thermal and electrical insulation.

2.6(b)DETERMINATION OF ELECTROLYTIC CONDUCTIVITY: (Fig.2.12)

The Philips measuring bridge PR 9500 is a compact and versatile unit (Fig.2.12). It works on the principle of wheatstone's bridge which is slightly modified by incorporating alternating source of low emf, an electronic detector and identical electrodes of fairly large size for an electrolyte. Excellent account of this is given in the 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 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 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, 3.6V. The bridge is connected to mains supply viz., 230V, 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 (Fig.2.13) with which the resistance of the solution (electrolyte) to be measured is compared are incorporated in place of R, i.e., connected to sockets Bu_1 and Bu_2 (Fig.2.12, 2.14). 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 comparisons 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%.

2.6.1 Measurement of conductivity:

An experimental set up for measuring the conductivity of a given solution (electrolyte) is shown in Fig.2.14. The position of various connection sockets and operation controls are indicated in Fig.2.12. For efficient use of the bridge, it is imperative to proceed in the following way:

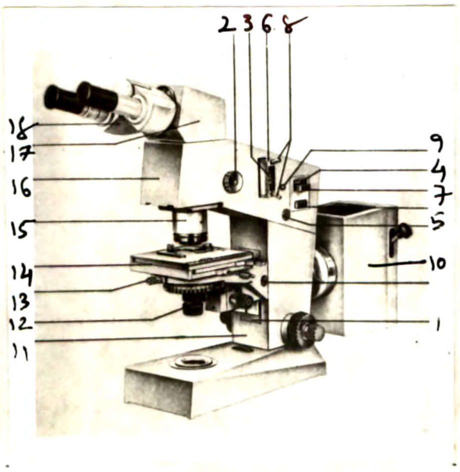

Fig.2.1 VERTIVAL incident light microscope

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

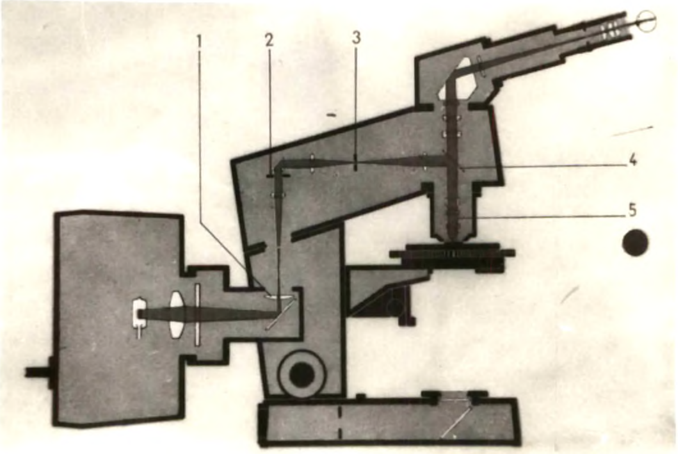

Fig.2.2 Ray diagram for bright field

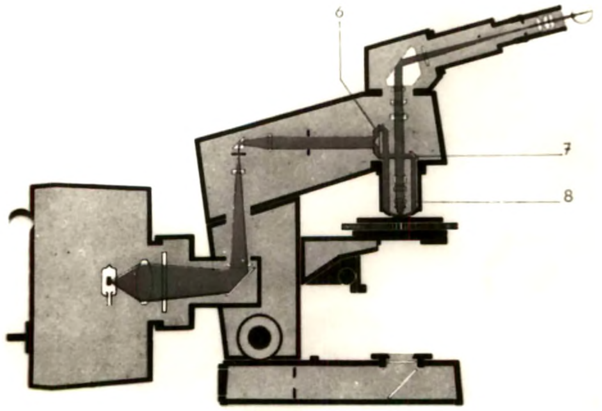

Fig.2.3 Ray diagram for dark field

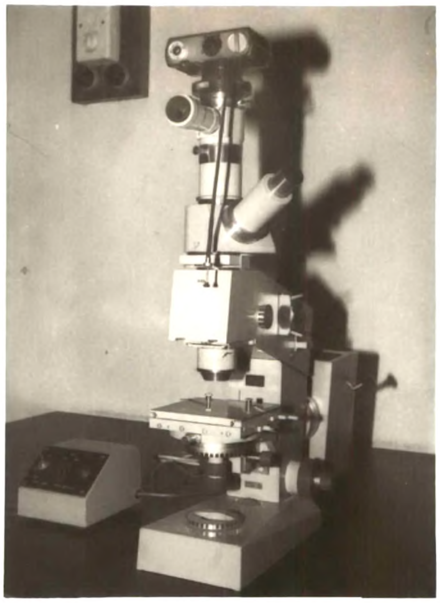

Fig.2.4 Camera attachment on microscope

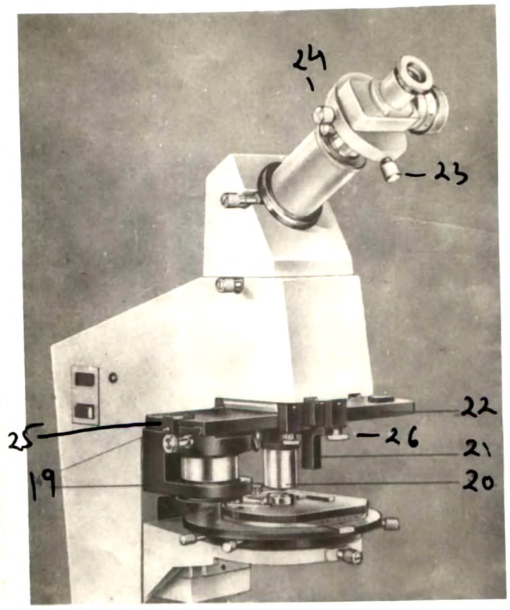

Fig.2.5 Indentation technique

19. Indentation device
20. Objective
21. Handle
22. Slide

23,24,25,26. Centring screws

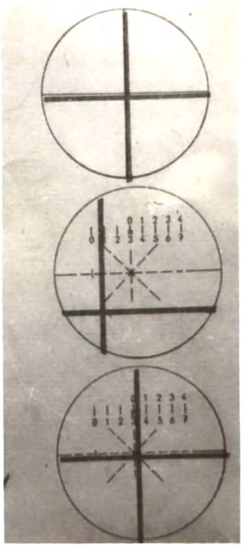

Fig.2.6 Zero position of cross wire on graticule of micrometer eyepiece

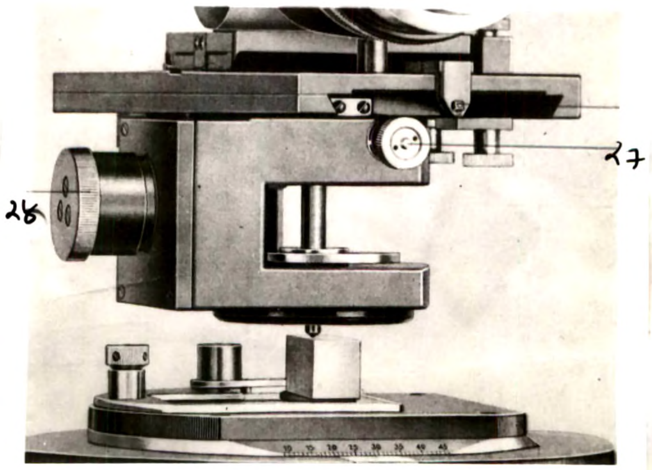

Fig.2.7 **Indentation technique**

27. Knurled knob
28. Rear knurled knob

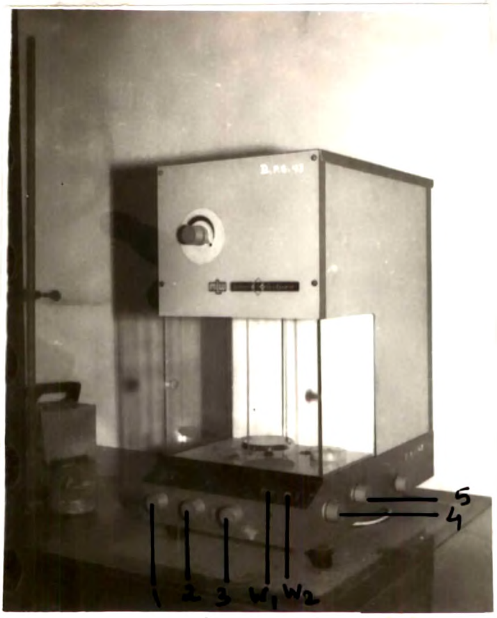

Fig.2.8 **Semi-micro balance**

1. Knob giving weights in multiples of 10 gm.
2. Knob giving weights in multiples of 1 gm.
3. Knob giving weights in multiples of 0.1 gm.
4. Knob for adjusting 4th and 5th digit after decimal point
5. Knob to make pan free;
   W_1 & W_2 - scale window.

Fig. 2.9 Vertical temperature gradient crucible furnace

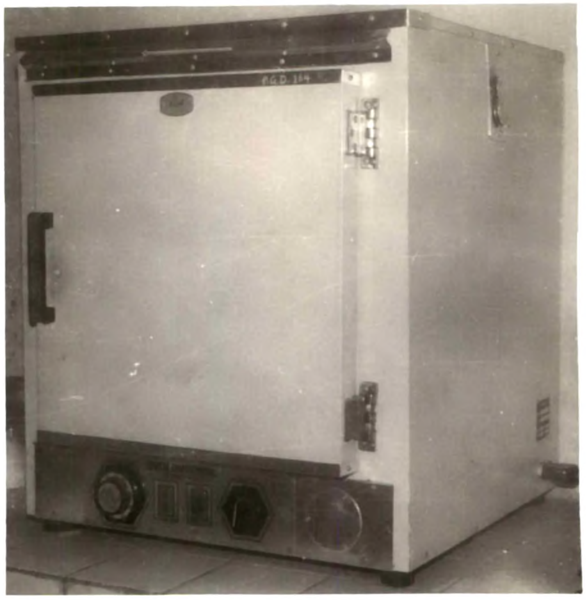

Fig. 2.10 Oven

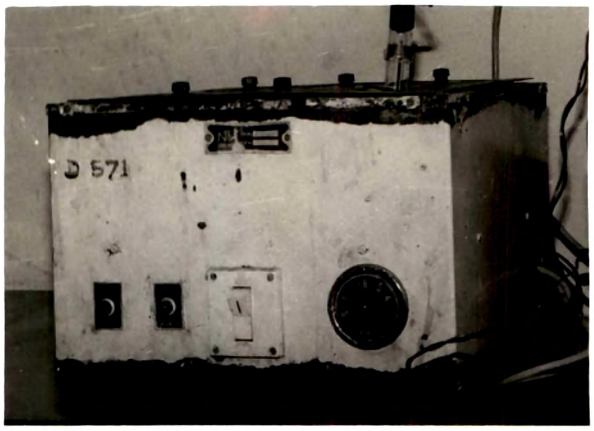

Fig. 2.11 Water-bath

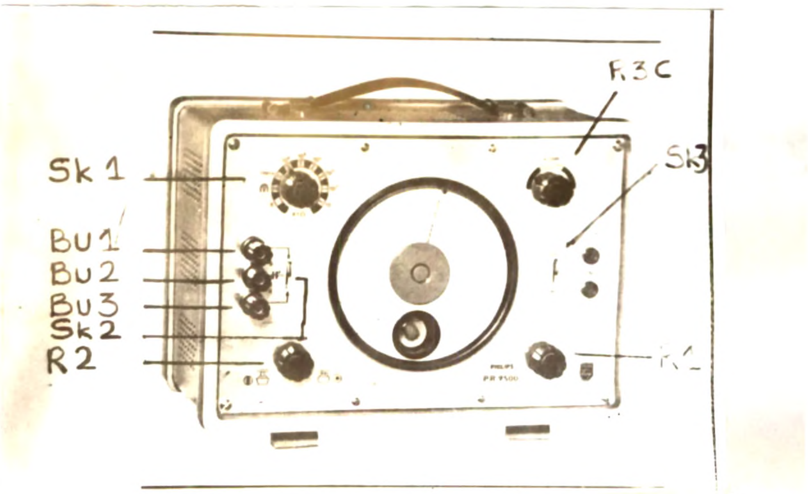

Fig. 2.12 Conductivity measuring bridge

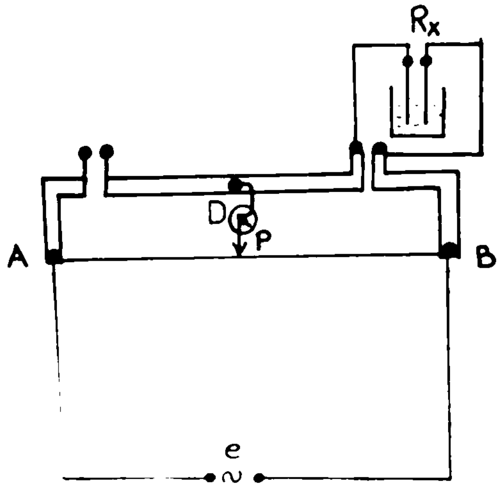

Fig. 2.13 Wheatstone net

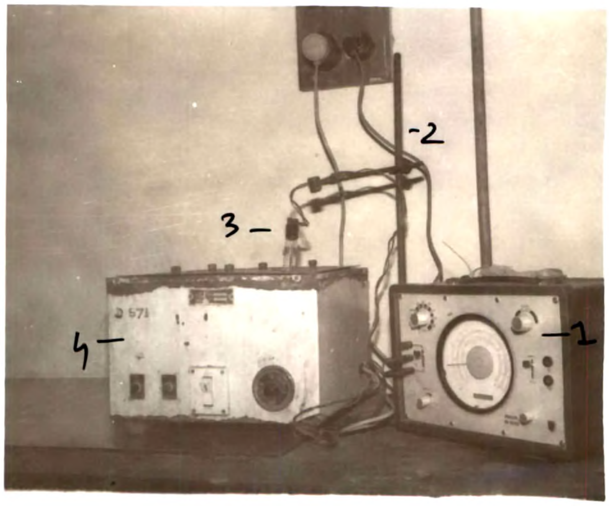

Fig.2.14 An experimental set up for measuring conductivity of a solution

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

(1) Connect the terminals of the measuring cell to sockets Bu_2 and Bu_3. 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 the 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.2.14) with the accuracy of ± 0.5°C.

(2) Set the switch Sk_3 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 R_2.

(4) Select the measuring range with the switch Sk_1, within which the resistance of the liquid is expected to be.

(5) Turn knob R_1 and move the pointer on the scale until maximum shadow length is obtained in cathode ray indicator.

(6) The value of resistance (R_x) is then found by taking the number indicated for the position of the switch Sk_1 and multiplying it by reading indicated by the pointer on the scale.

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

$$\text{Specific conductivity } (K) = \frac{1}{\tau . R_x} \text{ mho-cm}^{-1}$$

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

(8) The velocities of positive and negative ions, v^+ & v^- in the electrolyte are then determined by the formula:

$$(v^{+} + v^{-}) = \frac{1000}{C} K$$

where C is the concentration of the electrolyte in gm-equivalent per litre. The average ion velocity 'u' is then calculated as follows:

$$u = \frac{v^{+} + v^{-}}{2}$$

In the present work electrolytic solutions for measuring electrolytic conductivity are etchants employed by the author to delineate dislocations ending on a crystal surface.

2.7 SILVERING TECHNIQUE:

The crystal surface and the optical flats have to be coated with a highly reflecting layer of silver both for microscopic and interferometric studies. The principle of this method is to thermally evaporate silver onto the specimen at a very low pressure.

A commercial vacuum coating unit 'Edwards 12 EA' (Fig.2.15) was used for this purpose. The vacuum chamber was evacuated by a three stage silicon oil diffusion pump backed by an oil rotary pump. The vacuum at different stages was measured by the Pirani gauge and Philips ionisation gauge incorporated in the unit. The surfaces were thoroughly cleaned before deposition of silver. The cleaning process of surfaces depends very much on the nature of the surfaces. Optical flats were first cleaned with nitric acid, washed with water after applying soap and then with hydrogen peroxide. Thereafter they were cleaned by rubbing with cotton wool till no breath figure was formed on breathing over them. Freshly cleaved crystal surfaces did not require any cleaning. Final cleaning was done by ionic bombardment in vacuum coating unit by means of a high tension discharge.

When the pressure was about 1 x 10^{-5} torr spectroscopically pure silver was evaporated from a molybdenum boat by passing a low tension high current. In order to protect the surfaces to be coated from receiving the vapours of burnt impurities while heating the boat, it was covered with an adjustable shutter. Silver was deposited for the required time by removing the shutter from above the boat. The specimens were then optically studied.

2.8 **DETERMINATION OF VISCOSITY:** /6/ (Fig.2.16, 2.17)

In the materials kingdom, gases and liquids are considered as fluids showing several general properties, such as viscosity, density, fluidity, etc., depending on van der Waal's forces of attraction between their molecules. Viscosity is associated with the flow of a fluid. When a liquid has a streamlined flow through a pipe, all parts of it do not move at the same rate. The layer of liquid in immediate contact with a wall of the pipe is almost stationary. The velocity of flow of each successive layer increases progressively and is maximum at the centre. Since the layers are in close contact with each other, a faster moving layer tries to accelerate the slower moving one whereas the latter tries to retard the motion of the former. Thus internal friction between layers develops. This is called viscosity and the amount of friction is expressed by the coefficient of viscosity (or viscosity) of the liquid. It is expressed in dynes-seconds per cm^2 or poise. It is given by-

$$f = \eta \frac{dv}{dx}$$

where dv is the relative velocity of the faster layer with respect to the slower one, dx is the separation between these parallel layers, f is the tangential force per unit area required to maintain constant velocity gradient (dv/dx). η is therefore defined as the tangential force in dynes that must be exerted between two parallel layers, 1 sq.cm. in area and 1 cm apart, in order to maintain a velocity streaming of 1 cm per second of one layer past the other. The reciprocal of viscosity is called fluidity of the liquid.

The law controlling the flow of a liquid through a capillary tube is expressed mathematically by Poiseuille's equation-

$$\eta = \frac{\pi P r^4 t}{8 V L} \quad \ldots \quad \ldots \quad (1)$$

where,

η = the coefficient of viscosity, dyne-sec-cm^{-2}

V = the volume of liquid, cm^3

L = length of the capillary, cm.

t = time of flow, sec.

P = driving pressure, dyne/cm^2

= hdg ... ... (2)

(in case of vertical flow through a capillary in Ostwald's viscometer)

h = height of the liquid in the capillary, cm.

d = density of liquid, gm/cm^3

g = acceleration due to gravity, cm/sec^2

Substitution of (2) in (1) yields,

$$\eta = \frac{\pi r^4 h d g t}{8 V L} \quad \ldots \quad \ldots \quad (3)$$

If equal volumes of liquids having viscosities (η_1 & η_2) densities (d_1 & d_2) and times of flow (t_1 & t_2) are made to flow through the same capillary of fixed height, one by one, under identical environmental conditions (3) becomes,

$$\frac{\eta_1}{\eta_2} = \frac{\dfrac{\pi r^4 hg}{8V_1 L} \quad d_1 t_1}{\dfrac{\pi r^4 hg}{8V_2 L} \quad d_2 t_2}$$

Ratio of the above equations gives-

$$\frac{\eta_1}{\eta_2} = \frac{d_1 t_1}{d_2 t_2} \qquad \ldots \qquad \ldots \qquad (4)$$

Thus, knowing d_1, t_1, d_2, t_2 and η_2, the viscosity of the unknown liquid can be determined. Alternatively by taking a standard liquid with viscosity η_2, the relative viscosity of the liquid can be determined. It is thus clear from (3) that determination of coefficient of viscosity by Ostwald's viscometer actually involves two experiments:

(i) Determination of viscosity of the liquid,

(ii) Determination of density of the liquid.

The density of the liquid is generally determined by specific gravity bottle. In the present case, determination of coefficient of viscosity by this viscometer (Fig.2.16 with dimensions of various parts of viscometer) was made by carefully following the procedure (steps) mentioned below:

(1) Clean the viscometer with a standard cleaner such as chromic acid, acetone, alcohol, ether to remove any greasy matter sticking in it. Then thoroughly wash it with water, distilled water and dry it by blowing dry air in it.

(2) Attach a clean rubber tube with a screw pinch cock at the end A and clamp the viscometer vertically.

(3) Now add given volume of water in the viscometer through the end of the bulb E. Use the rubber pump to draw water from the end A, till the level of the water rises above the mark B.

(4) Release the pressure. Water flows vertically downward under gravity through the capillary CD of uniform bore.

(5) Start the stop watch when the meniscus just crosses the mark B. The time is noted when the meniscus just passes the point C. Let it be t_1.

(6) Repeat the experiment by taking the same volume of water number of times.

(7) Now remove water, clean the viscometer again and repeat the experiment with liquid of unknown viscosity number of times by taking the same volume of the liquid as that of water. Let the time of flow be t_2.

(8) For determining viscosity at different temperatures the entire viscometer was put in a waterbath whose temperature was thermostatically controlled. The accuracy of temperature was ± 0.5°C. The above procedure was repeated for unknown liquid and water.

(9) The calculations were made by applying (4). The observations and results and graphs were presented in the usual manner.

While performing the above experiment the following precautions were taken:

(i) Viscometer must be thoroughly cleaned.

(ii) The viscometer must be clamped again exactly at the same position.

(iii) Exactly same amount of the two liquids must be used.

(iv) The viscometer must not be disturbed during the measurement of time of flow.

Specific gravity bottle was used to determine the density of the liquid for which the viscosity was determined by Ostwald's viscometer. Following the normal procedure of weighing of the bottle with and without the liquid and repeating this for distilled water and by employing the usual formula, density of liquid was determined at room temperature. Just as viscosity at different temperatures could be found by keeping the entire viscometer in thermostatically controlled water-bath, the same procedures have been adopted for determination of density at higher temperatures. Since density of a liquid is the ratio of its mass to volume, the determination of density at high temperature involves the measurement of mass and volume at this temperature. This means that expansion of the liquid has to be determined. For this, two variants of the method are known: (1) weight dilatometer (or Pyknometer) and (2) volume dilatometer. For obvious advantages, volume dilatometer was preferred. Hence volume dilatometer with a long stem engraved (Fig.2.17) was fabricated by the author. The scale on the stem was calibrated. The figure provides complete information about the material, dimensions of bulb, stem, etc., of the volume dilatometer. The coefficients of cubical expansion of liquid are determined at various temperatures by using the formula-

$$\gamma_m = \frac{1}{v}\frac{dv}{d\theta}$$

where dv is a small change in liquid of volume v for a small change (5°C) in temperature. From these values, densities of the liquid at different temperatures were determined by using the equation,

$$d_2 = \frac{d_1}{1 + \gamma_m d\theta}$$

where d_2 is the density at $\theta°_2$C temperature and d_1 at $\theta°_2$C & $d\theta = \theta_2 - \theta_1$. It should be noted that densities of liquid are found at those temperatures at which viscosities were determined. In the present work, aqueous solution of d-tartaric acid of varying concentrations was employed as a dislocation etchant for cleavage faces of natural crystals of calcite. It was necessary to determine viscosity of d-tartaric acid solution of different concentrations at different temperatures. Ostwald's viscometer described above was used to determine the viscosity of the solution at different temperatures.

2.9 CRYSTAL GROWING TECHNIQUES:

2.9.1 Introduction:

Basically, the study of solid state physics is nothing but the study of crystals - their structure, defects, properties and growth.

There is a great demand of single crystals of greater and greater purity and perfection in every branch of research connected with the solid state and still greater demand for industrial applications. This has created a considerable interest in the study of growth of single crystals.

2.9.2 Different methods of growing crystals:

There are number of methods developed for growing single crystals of different types of materials. The classification of these methods is as shown below:

(1) Growth from solution:

   (A) Growth from water solution:

      (a) By progressively (regularly) lowering the temperature to reduce the solubility and produce crystallization under controlled conditions (e.g., inorganic salts such as alkali halides, Rochelle salt, EDT, ADP, etc.).

(b) At constant temperature-

(i) By progressively decreasing the amount of solvent by evaporating, and

(ii) by increasing the amount of solute.

(B) Growth from flux (e.g., metals and non-metals, ruby, barite, etc.)

(C) Hydrothermal growth (calcite and zincite, etc.)

(D) High pressure growth (boron nitride).

(E) Growth by gel method.

(F) Growth by electrodeposition.

(2) Growth from melt:

(a) Bridgman-Stockbarger method

(b) Zone melting method

(c) Verneuil flame fusion method

(d) Czocharalski-Kyropoulos method.

(3) Growth from vapour (Gas) phase:
(Silicon carbide, cadmium sulphide, lead sulphide, zinc telluride, lead telluride, etc.)

(4) By Chemical decomposition:
(Koref hot-wire method)

(5) By strain anneal method

(6) From high temperature solution:
(Growth of mica, tourmaline)

(7) From low temperature solution.

A survey of methods of growth suggests that almost 80% of crystals grown are from melt method. There are several standard books available on the growth of crystal by different methods /1, 7-10/.

'Growth from melt' has been found to be most successful method for growing large single crystals of semiconducting materials and alkali halide materials. All the methods of growth from a melt rely on cooling the liquid below its freezing point.

The basic condition to be satisfied for the production of single crystals from melt is that a progressive freezing of the liquid takes place in a controlled manner. In other words, the solid-liquid interface moves in a controlled manner along the growth axis. The growth rate of the crystal is the rate at which this solid-liquid interface moves in a direction perpendicular to the interface.

Generally the growth of single crystal from melt is influenced by the rate of growth, the temperature gradient along the solid and impurities. There are several factors which limit the application of this method: (i) The method is applicable only when the substance melts congruently and without irreversible decomposition. (ii) There should not be solid state phase transformation between the melting point and the temperature to which the crystal will later be cooled. (iii) The temperatures required in growing the crystals directly from pure melt are usually higher than those required by other methods. For this purpose, the furnace design and temperature control are important. The selection of a suitable material for crucible or container of the molten substance at elevated temperatures without contamination is also important.

The present author used the modified Kyropoulos method to grow single crystals of sodium nitrate from melt.

2.9.3 Kyropoulos method:

This method was developed by Czocharalski /11/ in 1918 although it is sometimes named after Kyropoulos /12/ who worked on the technique in 1926.

It is a crystal-pulling technique describing the slow pulling of the crystal from melt. Essentially a small single crystal seed is dipped into the surface of the melt and slowly withdrawn at the rate at which the crystal solidifies, while remaining in contact with the melt. If suitable precautions (e.g., avoidance of thermal and mechanical fluctuations) are taken, the material withdrawn from the melt solidifies as a large single crystal. This method is slightly modified by employing rotation mechanism of seed to avoid radial temperature gradient and impurities. The water cooling system for seed is also employed to keep the temperature of seed slightly lower than melt.

Difficulties were experienced by a number of workers /13-15/ to grow large single crystals of sodium nitrate. Using modified Kyropoulos method, large single crystals of $NaNO_3$ were grown /16/. This method was followed by the author to grow these crystals in the laboratory. There are certain specific properties of sodium nitrate crystals which are required to be considered for obtaining good quality crystals.

They have pronounced thermal conductivity anisotropy, the thermal conductivity being maximum in the direction of optic axis, viz., [111]. The maximum rate of growth proceeds in this direction. As a result, if the direction of the growth does not coincide with the [111] direction, the crystal grows in blocks. The anisotropy of the heat expansion coefficient leads to thermal stresses on cooling the single crystals. The effect is enhanced by the anomalous change of thermal expansion near 275°C, where the phase transition of the second type takes place /17,18/. Hence the following requirements should be provided for obtaining crystals free from stresses.

(1) The growth must coincide with the [111] direction,

(2) The crystal must grow without any container, and

(3) The cooling after annealing must be slow.

In the present case the axis of the growth of single crystals of $NaNO_3$ on insoluble mica disc employed as a seed coincide with [111] direction. The pulling of the seed was carried out in the usual manner. The schematic diagram (Fig.2.18) indicates in detail the method of growth of $NaNO_3$ crystal. The photograph (Fig.2.19) shows the crystal pulling unit with furnace, temperature controller, power supply, rotation mechanism, etc. The crucible furnace of 80 mm in diameter and 200 mm in depth with side (1) and bottom (2) heaters was used. The bottom heater has a constant stabilized voltage supply. The power of the side heater is adjusted by the potentiometer. The thermocouple (3) placed near the coil of the side heater serves as a pick up. The furnace has a cover (4) with two windows provided for observation of crystal during its growth. In the centre of the cover there is an opening for the cooler (5), a rotating copper rod of 20 mm in diameter acts as the cooler (the water cooling is not required). The crystals are grown in aluminium crucible (6) of 70 mm in diameter, 80 mm height and 0.8 mm of wall thickness. As a seed muscovite mica disc (7) of 21 mm in diameter and 0.5 mm thickness was used. It was glued to the edge of the cooler by BF-2 agent. The crystals were grown from the raw material of G.R. quality supplied by Sara Merk, India (the contents of the assay are given in a tabular form in table 2.2).

Both heaters were switched on and when the temperature of the crucible containing powder of sodium nitrate crossed the melting point (308°C) the charge was in molten condition. The temperature of the melt was not allowed to exceed 340°C, as $NaNO_3$ decomposes at 380°C. The cooler with the seed glued on it was initially kept 2 mm away from molten surface. It was gently immersed in the heated melt so that mica came in contact with the luquid-surface. The seed was kept in this position for 30 min. Then the cooler was slowly lifted so as to maintain contact between mica and melt and almost at the same time it was rotated at a rate of 2 rpm and the temperature was lowered at the rate of 2 degrees per hour by potentiometer using only side heater. The power fed to the bottom heater was kept constant. The cooler was raised at a rate of 2 mm per hour. Thus single crystals of 15 to 20 mm height and about 60 mm in diameter could be grown in 10 hours. The grown crystal was transported with heated spincer into another furnace for annealing, where it was kept for four hours at 270°C and then the temperature of the furnace was lowered at a rate of 3 degrees per hour. The crystals thus obtained were transparent and exhibited characteristic properties such as perfect cleavage, double refraction, etc. (Fig.2.20). Single crystals of sodium nitrate can also be grown from solution, by following simple method (Holden and Singer /19/).

TABLE 2.2

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GUARANTEE CERTIFICATE

Sodium nitrate ($NaNO_3$) for analysis
Guaranteed Reagent
Mol. Wt. 84.99

Insoluble portion	Maximum
Free acid (as HNO_3)	0.003 %
Free alkali (as NaOH)	0.003 %
Ammonium salts (NH_4)	0.002 %
Arsenic (As)	0.0004 %
Calcium (Ca)	0.002 %
Total Chlorine content (Cl, ClO_3, ClO_4, as Cl)	0.004 %
Chloride (Cl)	0.0005 %

Insoluble portion		Maximum
Heavy metal (as Pb)	..	0.0005 %
Iron (Fe)	..	0.0003 %
Magnesium (Mg)	..	0.0005 %
Nitrite (NO_2)	..	0.00005%
Phosphate (PO_4)	..	0.00025%
Potassium (K)	..	0.005 %
Sulphate (SO_4)	..	0.003 %
Assay .... (mini)	..	99.5 %

2.10 ANALYSIS OF STRAIGHT LINE PLOTS:

2.10.1 Introduction:

No physical measurement is absolutely correct. It is always associated with an error. The final result in an experiment is obtained after a computation involving different physical quantities which are measured in the course of the experiment. It depends on the refinements of the experimental techniques and on the method of treatment given to different observation /20,21/. Relationships between different physical quantities in an experiment should be made as simple as possible, preferably linear and should be amenable to graphical analysis /22,23,24/.

In the present work, relations between hardness on cleavages of synthetic single crystals of $NaNO_3$ and $CaCO_3$, expressed by a hardness number and applied load or applied load and diagonal of indentation mark on a cleavage surface or a suitable combination of different physical quantities for obtaining a straight line or between etch rate and etching temperature or between etch rate and etchant concentration for studying etch phenomena on cleavage surfaces under controlled conditions of chemical or thermal etching are graphically studied. Data are plotted on a graph with carefully chosen scales along the axes so that details are not bunched, together over a small range and are commensurate with accuracy and precision of observations of the variables along the axes so as to minimize the unwanted magnification of errors associated with each observation. In the present analysis of observations, linear relationships between different physical quantities exist or are created by having suitable combinations of these quantities. Obviously the plote between variables having linear relation is straight line. It is necessary to obtain the estimation of the best straight line. For this several methods are known. They are as under:

(1) Estimation of the best straight line by eye.

(2) Estimation of $y = mx + c$ by the method of zero sum.

(3) Centroid method to estimate the best straight line ($y = mx + c$).

(4) Estimation of best straight line ($y = mx + c$) from data used in a specific manner.

(5) Statistical estimation of best straight line.

Out of these methods the author has followed the method based on statistical estimation. Hence this method of estimating best straight line is described here.

2.10.2 **Statistical estimation of best straight line:**

There are two ways of estimating statistically the best straight line:

(a) Regression of Y on X, and

(b) Regression of X on Y.

In both these methods one variable is assumed to be free from error.

(a) Regression of Y on X:

The best straight line is one for which the squares of the deviation of every point is a minimum. This is known as regression of Y on X.

Suppose there are n pairs of observations (x_1, y_1), $(x_2\ y_2)$, ... (x_n, y_n). For each known x, it is required to predict the best value of y using all observations.

$$\text{Let}\quad Y_1 = m_1x + c \qquad \ldots \qquad \ldots \qquad (1)$$

where Y_1 is not the observed value y_1 corresponding to x_1, but it is the best value considering all the observations. The difference between the actual and predicted values of y is

$$y_1 - Y_1 \quad \text{for} \quad y = y_1 \qquad \ldots \qquad \ldots \qquad (2)$$

Assuming the equation of the form

$$y = m_1x + c$$

for which values of m_1 and c are required such that,

$$(y - Y)^2 = E \qquad \ldots \qquad \ldots \qquad (3)$$

is a minimum.

$$y - Y = y - (m_1x + c)$$

$$\therefore \quad E = \sum_1^n [\ y - (m_1x + c)]^2$$

$$= \sum_1^n [\ y^2 - 2y(m_1x + c) + m_1^2x^2 + 2m_1xc + c^2] \ \ldots. \ (4)$$

For E to be minimum

$$\frac{dE}{dm} = 0, \qquad \frac{dE}{dc} = 0$$

Differentiating E w.r.t. m yields,

$$\frac{dE}{dm_1} = \sum_1^n (-2yx + 2m_1x^2 + 2xc) = 0$$

$$= \sum_1^n 2(m_1x^2 + xc - xy) = 0$$

$$\therefore \sum_1^n xy = m_1 \sum_1^n x^2 + c \sum_1^n x \quad \ldots \quad \ldots \quad (5)$$

Now differentiating E w.r.t. c gives,

$$\frac{dE}{dc} = \sum_1^n (-2y + 2m_1x + 2c)$$

$$= \sum_1^n 2(m_1x + c - y)$$

For minimum E

$$\frac{dE}{dc} = 0$$

$$\therefore \sum_1^n y = \sum_1^n c + m_1 \sum_1^n x$$

$$= nc + m_1 \sum_1^n x \quad \ldots \quad \ldots \quad (6)$$

Solving the equations for c

$$c = \frac{\Sigma y - m_1 \Sigma x}{n} = \frac{\Sigma y}{n} - m_1 \frac{\Sigma x}{n}$$

$$\therefore c = \bar{y} - m_1 \bar{x} \qquad \ldots \qquad \ldots \qquad (7)$$

where $\bar{x} = \frac{\Sigma x}{n}$ and $\bar{y} = \frac{\Sigma y}{n}$

$(\bar{x}, \bar{y})$ represent the mean of all coordinates x and y. The best straight line passes through the mean of all coordinates.

Substituting value of c from (7) in equation (5) gives,

$$\begin{aligned} \Sigma xy &= m_1 \Sigma x^2 + (\bar{y} - m_1\bar{x}) \Sigma x \\ &= m_1 \Sigma x^2 + \bar{y} \Sigma x - m_1 \bar{x} \Sigma x \\ &= m_1 \Sigma x^2 + \frac{\Sigma y}{n} \Sigma x - m_1 \frac{\Sigma x}{n} \Sigma x \\ &= m_1 \Sigma x^2 + \frac{\Sigma y . \Sigma x}{n} - \frac{m_1}{n} (\Sigma x)^2 \end{aligned}$$

$$\therefore \Sigma xy - \frac{\Sigma x . \Sigma y}{n} = m_1 \left(\Sigma x^2 - \frac{(\Sigma x)^2}{n} \right)$$

$$\therefore m_1 = \frac{\Sigma xy - \frac{\Sigma x . \Sigma y}{n}}{\Sigma x^2 - \frac{(\Sigma x)^2}{n}}$$

$$\therefore m_1 = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\Sigma(x - \bar{x})^2} \quad \ldots \quad \ldots \quad (8)$$

It is well-known that two conditions determine a straight line. Having obtained the values of slope (m) and intercept (c) the best straight line for n pairs of observations is:

$$y = m_1 x + c \quad \ldots \quad \ldots \quad (9)$$

where m_1 is called regression coefficient of y on x.

(b) Regression of X on Y:

The best straight line is the one for which the squares of the deviation of every point measured parallel to the x-axis and summed for all points is a minimum. This is known as regression of X on Y. It is thus clear from the above definition of the best line that equations similar to the ones derived above in (a) can be obtained by interchanging x and y and X and Y. Thus the mean coordinates are-

$$\bar{X} = \frac{\Sigma x}{n} \quad \ldots \quad \ldots \quad (10)$$

$$\bar{Y} = \frac{\Sigma y}{n} \quad \ldots \quad \ldots \quad (11)$$

and the intercept c and slope m_2 are-

$$c = \bar{x} - m_2\bar{y} \quad \ldots \quad \ldots \quad (12)$$

and

$$m_2 = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\Sigma(y - \bar{y})^2} \quad \ldots \quad \ldots \quad (13)$$

and the best straight line is-

$$x = m_2y + c \qquad \ldots \qquad \ldots \qquad (14)$$

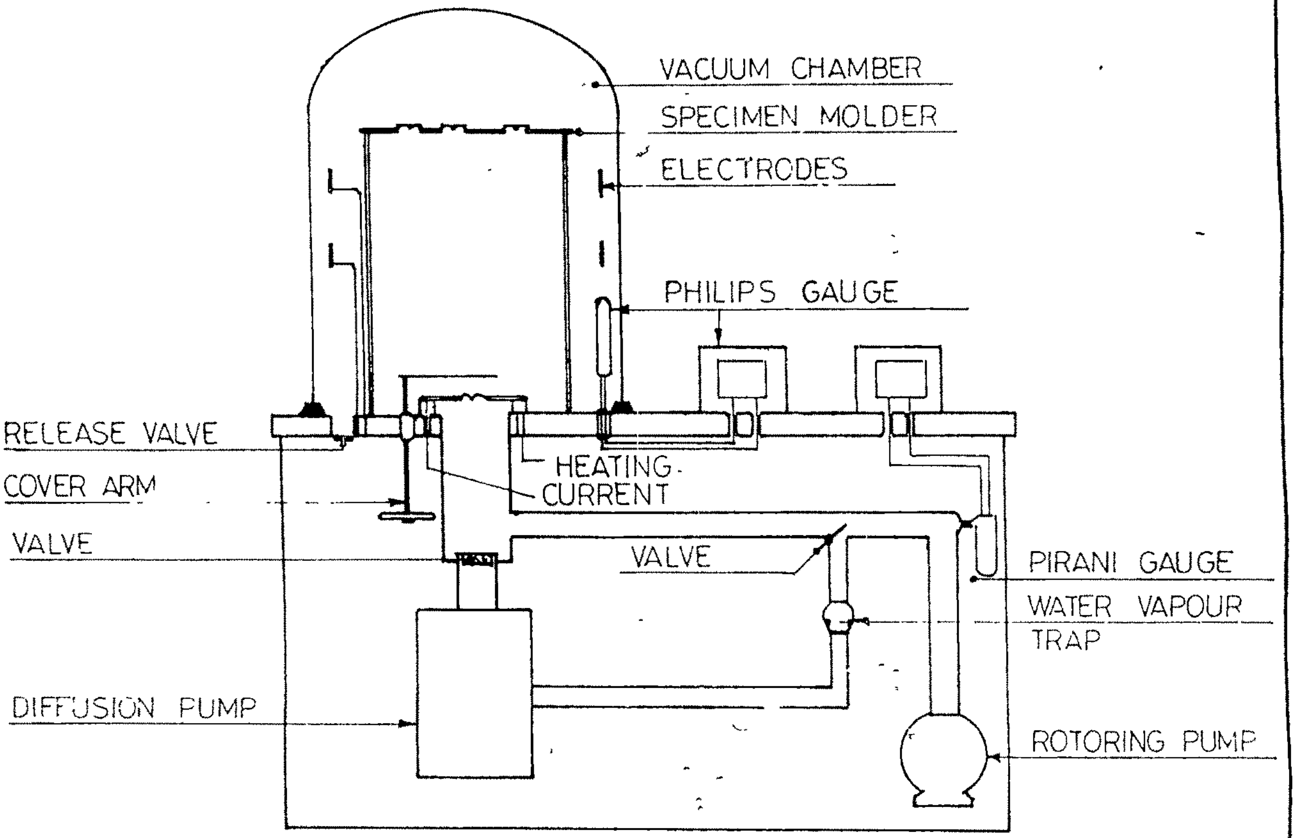
where c and m_2 are given by equations (12) and (13). m_2 is a regression coefficient of x on y.

The condition for a best straight line is that the correlation coefficient r should be nearly unity, which is defined as,

$$r = \sqrt{m_1 . m_2} \qquad \ldots \qquad \ldots \qquad (15)$$

If $r = \pm 1$, the correlation between x and y is perfectly linear. If $r = + 1$, the line has a positive slope and if $r = 1$, the line has a negative slope.

In the present work, the author has utilized, in various chapters 4, 5, 6, 9, the conditions expressed by (15) for each straight line plot in such a way that the correlation coefficient r is unity or is much nearer unity.



VACUUM COATING UNIT
(EDWARD Co LONDON)

Fig. - 2·15

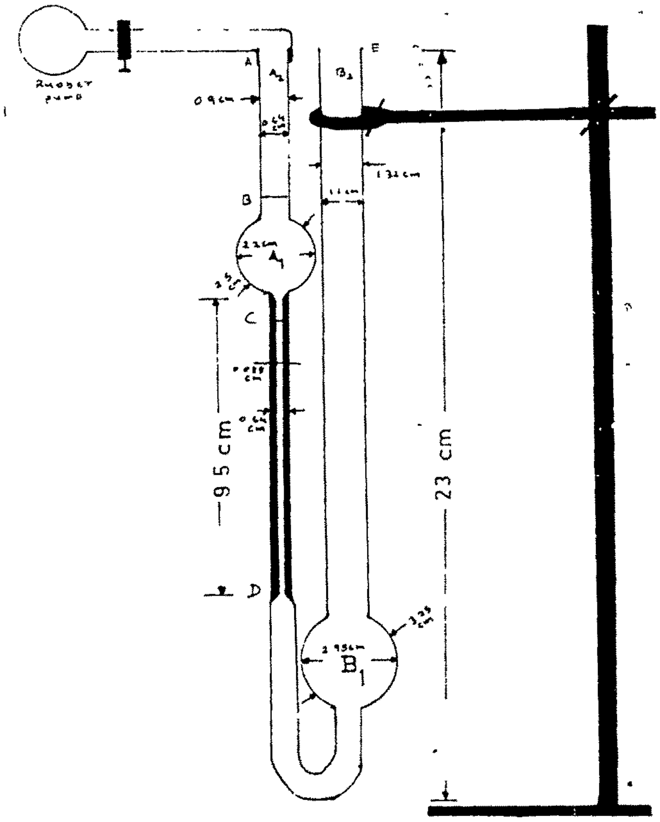


Fig.2.16 **Ostwald viscometer**

Dimensions:

1. Height of the viscometer .. : 23 cm
2. Weight of the viscometer .. : 37.194 gm
3. Total volume of viscometer .. : 46 cm^3
4. Length of the capillary .. : 9.5 cm
5. Diameter of the capillary .. : 0.088 cm
6. Outer thickness of capillary .. : 0.64 cm
7. Volume of the capillary .. : 0.057 cm^3
8. Outer diameter of bulb A_1 .. : 2.5 cm
9. Inner diameter of bulb A_1 .. : 2.2 cm
10. Volume of bulb A_1 .. : 1.151 cm^3
11. Outer diameter of bulb B_1 .. : 3.25 cm
12. Inner diameter of bulb B_1 .. : 2.95 cm
13. Volume of bulb B_1 .. : 1.545 cm^3
14. Outer diameter of tube B_2 .. : 1.32 cm
15. Inner diameter of tube B_2 .. : 1.10 cm
16. Outer diameter of tube A_2 .. : 0.90 cm
17. Inner diameter of tube A_2 .. : 0.64 cm
18. Thickness of the wall of the glass tube : 0.15 cm

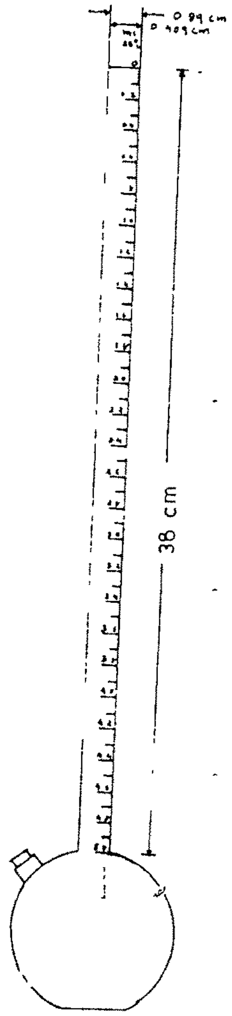


Fig.2.17 Volume dilatometer

1. Material of the dilatometer : Corning glass
2. Weight of the dilatometer : 73 gm
3. Volume of the flat-bottomed bulb : 63 cm^3
4. Dimensions of stem:
    (i) Length .. .. : 38.0 cm
    (ii) Volume .. .. : 5.0 ml
    (iii) Outer diameter .. .. : 0.89 cm
    (iv) Inner diameter .. .. : 0.409 cm
    (v) Least count of the scale on the stem : 0.02 ml

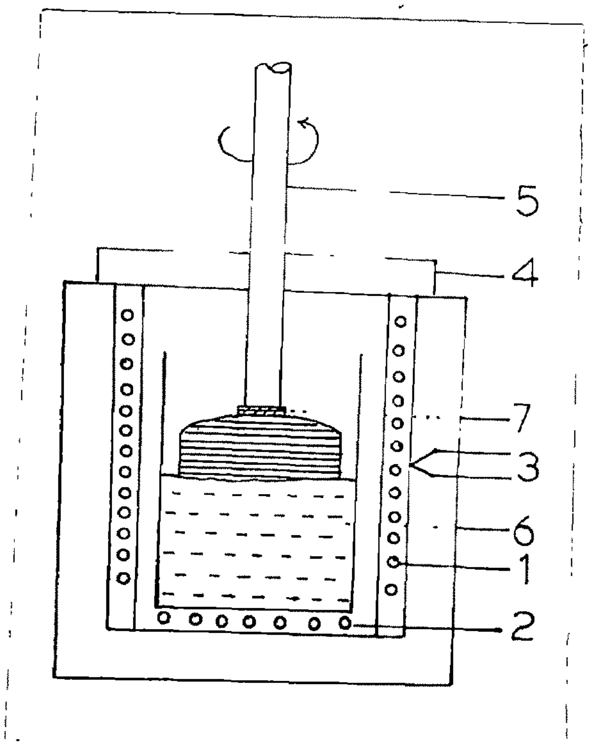


Fig.2.18 **Schematic diagram of method of growth of sodium nitrate crystals from melt**

1. Side heater
2. Bottom heater
3. Thermocouple
4. Mica window
5. Cooler
6. Crucible
7. Mica seed

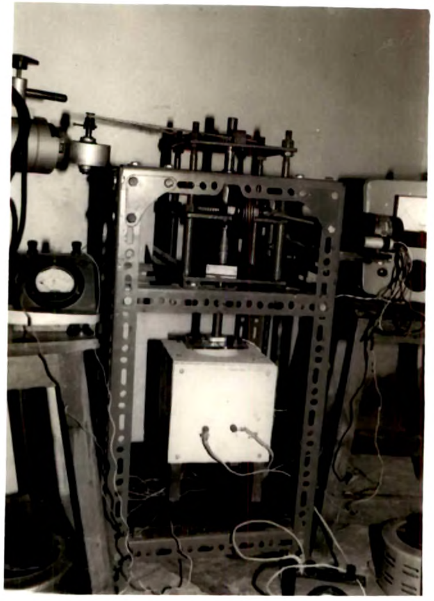


Fig.2.19 Crystal pulling unit along with furnace, temperature controller, power supply, rotation mechanism, etc.

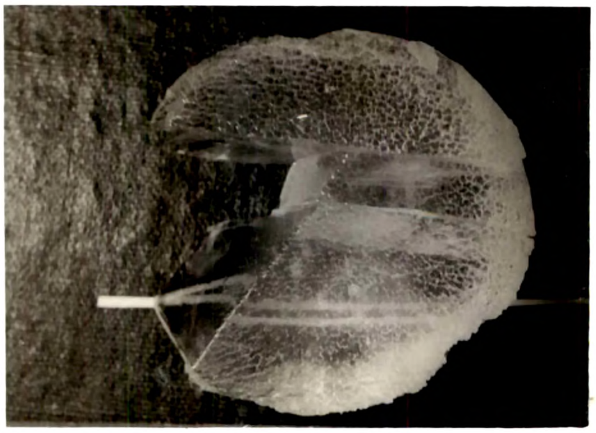


Fig.2.20 Sodium nitrate crystal grown from melt exhibits double refraction of a strip placed below the crystal

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