CHAPTER 2

EXPERIMENTAL TECHNIQUES

2.1 Introduction

In the present investigation a number of varying experimental techniques have been employed. As for example, the observations of etch patterns on alkali halides are made with the help of Vicker's Projection Microscope, used in dark and bright field illumination and PMF-17 Olympus Metallurgical Microscope used in bright field illumination as well as in phase contrast illumination. The measurements of etch features are made using the well known technique of multiple beam interferometry and light profile microscopy. Microhardness measurements are made by using the Vicker's indentation technique. For resolution in extension whenever necessary electron microscopy is also used. Only a short account of these methods is given here.

2.2 Silvering Technique

In order to enhance contrast the surfaces to be examined were coated with silver. To obtain a highly reflecting and uniform layer of silver, a condition of perfect cleanliness is of paramount importance. The cleaning process to be used depends upon the nature of the surface about to receive the silver.
Optical flats are first washed with water and soap and then cleaned with hydrogen peroxide and rubbed gently with dry cotton wool. This is continued till, on gently breathing over the surface, no breath figures are formed. In the present work, cleaning of alkali halide cleavages was done by ionic bombardment in the silvering unit by passing a high tension discharge.

For evaporating silver films, evaporating unit of the type 12EA, manufactured by Edward and Co., London, was employed (figure 3). It consists of a vacuum chamber in the form of a large pyrex bell jar, 35.6 cms. in height and 30.5 cms. in diameter resting in an annular recess upon a gasket of neoprene rubber which rests on a horizontal metal base plate. The chamber is evacuated by a three stage silicon-oil-diffusion pump backed by a rotary pump. A number of vacuum tight insulated electrodes pass through the base to which the evaporation filament and the electrodes for the high tension discharge are connected. A vacuum tight cone shutter can be swung in and out of position over the filament. The backing vacuum and the final vacuum can be read directly by the Pirani gauge and the Philip's ionisation gauge incorporated in the unit.

The chamber is first evacuated by rotary pump
and when a pressure of about 0.1 mm. of mercury is reached, the high voltage discharge is started. This is continued until the pressure on the Pirani gauge, which shows an increase due to the evolution of absorbed gases, after reaching a maximum begins to fall. At this stage the chamber is connected to the diffusion pump. Silver is generally evaporated when the pressure in the chamber, which is indicated by Philip's ionisation gauge, falls to below $10^{-5}$ cms. of mercury. When this pressure is reached the molybdenum boat is heated by a current of about 50 amperes. The filament is covered by the adjustable shutter in order to protect the substrate from receiving the vapour of the burnt impurities. Such impurities can have a serious influence in increasing film absorption. Deposition of silver is started half a minute after silver starts boiling (which could be seen from outside) by removing the shutter from over the filament for the required time. The thickness of silver film and hence the reflectivity is judged either by viewing the filament from the top of the bell jar through the specimen, if the specimen is transparent or, if the filament is calibrated it can be judged from the time of evaporation.

The dependence of reflectivity $R$, transmission $T$ and absorption $A$ for different
The thickness of silver films is shown in figure 4. To obtain high reflectivities, say 90%, the thickness of silver film should be nearly 500 Å, which is much less than the thickness of such silver films which do not contour the surface (Tolansky and Bhide, 1956). With pure silver, it is possible to get reflection coefficient of 0.94 in the green region and a reasonable transmission of one per cent. Since now a days powerful sources are available such reflectivities can be used.

2.3 Vicker's Projection Microscope

For interferometric as well as microtopographical studies, the Vicker's projection microscope (figure 5) was used. This is of the inverted metallurgical type, the specimen being placed above the objective lens. The main collimating system of the microscope is carried on a movable arm to facilitate the change over from reflection system to transmission system. A diagrammatic representation of the optical system of the microscope for reflection is shown in figure 6. It consists of a universal illuminator consisting of a semi-reflecting plate, an iris lens and the objective lens. In reflection, the objective thus acts as a condenser, and the convergence of the incident light depends on the relative positions of the back focal plane and the virtual image formed by the
field condenser. When these are coincident, a parallel beam falls on the specimen.

The collimating system of the microscope consists of a powerful mercury lamp, a condenser and an aperture controlled by an iris diaphragm. Translational movement of the lamp permits centering of the diaphragm. An image of the source can be formed on the field iris.

For visual observations, an eye piece with a reflector is pushed into the tube below the objective. This completes the normal microscope system. For photomicrography a projection eye piece is used and the final image is formed after reflection in the projection mirror, in the plane of the screen. Slight re-focussing is necessary when the visual system is changed over to the projection system.

2.4 **Olympus Universal Metallurgical Microscope**
(Model PMF - 17)

For microtopographical studies, Olympus universal metallurgical microscope (figure 7) was used. It is an inverted (Le Chatelier) type microscope. The illuminating unit of the microscope is incorporated in the illuminating tube which carries a condenser system, a view-field diaphragm, an aperture diaphragm, a phase
contrast ring slit and a bulb centering device. The object to be seen is placed on the microscope stage which is equipped not only with a cross-wise travelling device operated by co-axial handles but also with fine motion and rotating devices. For focussing, the movement of the specimen is performed by raising and lowering the stage. Fine focussing is made by the gear mechanism and is extremely smooth with a focussing accuracy of 0.0001 cm. In order to change the objective, the microscope tube is driven up and down by constant force of spring action. The tube is pushed down into the microscope body and locked at the bottom. When the objective has been changed the tube is released. It slowly comes up and stops at the previous position. The object is seen by binocular eye-pieces. Dioptre adjustment is accomplished, after the right eye focussing is attained, by screwing in and out the left hand eye piece until exact focussing with the left eye is obtained without moving the fine focussing knob. The most advantageous part of this microscope is easy adjustment of phase contrast system. When the object exhibits too deficient contrast in an ordinary microscope, the phase plate is inserted by swinging aside the covers located on both the sides of the microscope body.

For photographing the microstructures of the
specimen, the photographic unit is built exclusively for this model. It is used for taking 36 pictures on a 35 mm. film. Photographic eye pieces are built-in inside the microscope body and are interchanged by turning the outside turret so that magnification can be changed in three steps.

2.5 Multiple Beam Interferometry

Multiple beam interferometry means the production of interference, employing a succession of coherent beams which are specifically related in phase and intensity. Ideally, the beams should be behind each other in phase in an arithmetical progression, should fall off in intensity in a geometrical progression and the series should be finite. Such beams combine and produce highly sharpened fringes.

The first theoretical investigation of multiple beam interference taking place between plane parallel surfaces was made by Airy (1831). The salient points which are of special interest in the present investigation are discussed below. A full account of this technique including the references to original papers is described by Tolansky (1948).

Multiple reflections taking place between two reflecting surfaces A and B, separated by a medium
of refractive index \( \mu \) and thickness \( t \) are shown in figure 8. For a beam of light incident at an angle \( \phi \), let the reflection and the transmission coefficients be respectively \( R \) and \( T \). Along A, there will be a series of reflected beams of intensities \( R, RT^2, R^3T^2, R^5T^2 \), etc., and along B, transmitted beams have intensities \( T^2, R^2T^2, R^4T^2, R^6T^2 \), etc. The path difference \( d' \) between any two successive beams along either of the surfaces will be \( 2\mu t \cos \phi \). The resulting phase lag \( \delta' \) between them will be \( (2\pi/\lambda) 2\mu t \cos \phi \).

When monochromatic light from an extended source is directed onto the interferometer, modified transmission Haidinger fringes are formed. If a lens is placed in the path of a system of parallel rays, Airy summation at the focus of the lens is automatically obtained.

The intensity distribution is no longer of the \( \cos^2 \) type, as in the case of two beam fringes but is modified by the multiple beam combination.

If the transmitted series of beams of geometrically decreasing intensity and the phase increasing arithmetically by \( \delta' \) is summed to infinity, the resulting intensity \( I_T \) at any point in the field corresponding to \( \delta' \) is given by
and for the reflected system

$$I_R = \frac{4R \sin^2 \delta/2}{(1 - R)^2 + 4R \sin^2 \delta/2}$$

The quantity $\sin^2 \delta/2$ can only vary from 0 to 1, at which $I$ has maximum and minimum values respectively.

When $\sin^2 \delta/2 = 0$; $I_{\text{max}} = \frac{T^2}{(1 - R)^2}$

When $\sin^2 \delta/2 = 1$; $I_{\text{min}} = \frac{T^2}{(1 + R)^2}$

If there is no absorption at the reflecting surfaces then $T + R = 1$ and $I_{\text{max}} = 1$, i.e. the maximum intensity of the fringe is equal to that of the incident light, no matter what the values of $A$ and $T$ are. If, however, a fraction $A$ be absorbed at each surface, then

$$T + R + A = 1$$

and hence

$$I_{\text{max}} = \left( \frac{1 - R - A}{1 - R} \right)^2$$
\[ I_{\text{min}} = \left( \frac{1 - R - A}{1 + R} \right)^2 \]

\[ \frac{I_{\text{max}}}{I_{\text{min}}} = \left( \frac{1 + R}{1 - R} \right)^2 \]

The whole fringe shape is thus quite independent of absorption.

The principal factors affecting the usefulness of multiple beam fringes are the contrast and the sharpness. The contrast is defined as \((I_{\text{max}} - I_{\text{min}})\). The sharpness is defined by the reciprocal of the fringe half width \((W)\). The fringe half width is the width at half the peak intensity.

The theory for the reflected system has been worked out by Holden (1949). For this system, the value of the \(I_{\text{max}}\) decreases while that of \(I_{\text{min}}\) increases with the increase in the absorption. The contrast \((I_{\text{max}} - I_{\text{min}})\) therefore, decreases with the increase in the absorption.

The fringe half width \((W)\) as the fraction of an order can be shown as

\[ W = \frac{1 - R}{\pi \sqrt{R}} \]

The following table no. 2.1 gives
approximate values of $W$ for a number of values of $R$.

Table No. 2.1

<table>
<thead>
<tr>
<th>$R$</th>
<th>0.04</th>
<th>0.7</th>
<th>0.8</th>
<th>0.85</th>
<th>0.9</th>
<th>0.925</th>
<th>0.94</th>
</tr>
</thead>
<tbody>
<tr>
<td>$W$</td>
<td>1/3</td>
<td>1/9</td>
<td>1/14</td>
<td>1/19</td>
<td>1/30</td>
<td>1/40</td>
<td>1/50</td>
</tr>
</tbody>
</table>

It can be seen from the table no. 2.1 that an increase in the value of $R$ decreases the value of $W$ and hence increases the sharpness of the fringes.

The relation between the reflectivities, transmission and absorption for silver films is shown in the table no. 2.2.

Table No. 2.2

<table>
<thead>
<tr>
<th>$R%$</th>
<th>70</th>
<th>75</th>
<th>80</th>
<th>85</th>
<th>90</th>
<th>94</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T%$</td>
<td>27</td>
<td>22</td>
<td>16.5</td>
<td>10.5</td>
<td>4.5</td>
<td>0.7</td>
</tr>
<tr>
<td>$A%$</td>
<td>3</td>
<td>3</td>
<td>3.5</td>
<td>4.5</td>
<td>5.5</td>
<td>5.3</td>
</tr>
</tbody>
</table>

This shows that, with the increase in the reflectivity, the absorption also increases. The increase in the absorption reduces the contrast of the reflected system. In actual practice, for the reflected system, it is found that the value of $R$ of about 80%, gives quite sharp fringes with optimum contrast.
Pebry, Perot and Buisson produced sharp Fizeau fringes, formed by a thin wedge silvered on both the sides. Tolansky (1946) was the first who investigated the critical conditions to be fulfilled by a doubly silvered wedge, so that a close approximation to the Airy summation can be achieved.

Tolansky (loc. cit.) and Brossel (1947) have shown that the fringes are formed at the surface, and for a wedge angle \( \theta \), the path difference between the first and the \( n \)th beam, when \( n \) is large, to a first approximation becomes equal to 
\[
2nt - \frac{4}{3} n^3 \cdot \theta^2 \cdot t
\]
where \( t \) is the thickness of the wedge at the point of incidence of light. It can therefore be seen that the retarding path lag for the \( n \)th beam will be equal to 
\[
\frac{4}{3} n^3 \cdot \theta^2 \cdot t
\]
The retarding lag \( \frac{4}{3} n^3 \cdot \theta^2 \cdot t \) shows that the Airy sum condition will in general be secured only when \( t \) is very small, since for high reflectivities \( n \) is very large (of the order of 60) and \( \theta \) cannot be reduced indefinitely as its value determines the number of fringes per centimeter across the field of view. Taking possible values of \( n \) and \( \theta \), it can be seen that the separation between the two surfaces must be of the order of a few wavelengths of light at the most, otherwise fringe definition.
suffers severely.

Apart from the phase retardation condition, the linear displacement 'd' of the beams also depends on 't'. The linear displacement of beams for successive reflections is shown in figure 9. To a first approximation, the linear separation 'd' on the surface of the wedge between the first beam and the nth beam is $d = 2n(n+1)t\theta$.

If the values of 'n' and 'θ' are fixed, it is seen that 'd' is proportional to 't'. If 't' is of the order of a few wavelengths of light, it can be shown that for fringes 0.1 mm apart, the linear displacement of the beams is about that of the resolving power of a medium power microscope and hence the beams involved in producing a fringe do not produce any confusion.

Taking account of the phase lag variations due to errors in collimation, the linear displacement of beams etc., the experimental conditions for the production of highly sharpened multiple beam Fizeau fringes can be summarized as follows:

1. The surface must be coated with highly reflecting film having minimum absorption.
2. The film must contour the surface exactly and be highly uniform in thickness.
3. Monochromatic light, or atmost a few widely spaced monochromatic wavelengths should be used.
4. The interfering surfaces must be separated by at most a few wavelengths of light.

5. A parallel beam (within $1^\circ - 3^\circ$ tolerance) should be used.

6. The incidence should preferably be normal.

2.6 Phase Contrast Microscopy

The function of an ordinary microscope is the formation of an image in terms of brightness or colour contrast which can be converted into an image that can be observed. The change in the brightness of a light wave is caused by the change in the amplitude as a result of absorption of light and the change in the colour is produced as a result of selective absorption. This means that the changes produced by an object in the amplitude and the wavelength of a light wave can be easily detected by an ordinary microscope.

The optical path difference introduced by the microscopic specimen from the difference in the refractive index or the thickness or a combination of both cannot be detected by an ordinary microscope. The possibility of revealing those parts of the microscopic specimen, having different refractive indices, compared to the surroundings, as a change in the intensity of the image was first investigated by Zernike (1934). The method was later developed
by Jupnik, Osterberg and Pride (1946), Taylor (1949), for use with specularly reflecting objects. A comprehensive review of the subject is given by Barret, Osterberg, Jupnik and Richards (1951) in their book 'Phase Microscopy'. The detailed mathematical theory based on the use of vector diagrams has been published by Barrer (1952). The function of an ordinary microscope and the alterations required to be made to convert it into a phase microscope are shown in figure 10. In this figure, C is a condenser diaphragm. Let us consider the formation of the image of a simple transparent particle which is placed at the position shown in the figure. The rays from C are rendered parallel before passing through the specimen. According to the theory of the microscope developed by Abbe (1871), every point in the specimen is treated as a diffracting object. Consequently the incident wave does not pass without interruption through object plane but is diffracted. As shown in the figure, a portion of the diffracted wave which is shown by the solid lines continues in its original course, undeviated, while the remaining portion shown by the dotted lines gets deviated. The undeviated waves are called S (surround) waves while the deviated waves are called D waves. The S waves form an image of C at C' - the back focal plane of
the objective and after passing through C', diverge and spread over the back focal plane and are focussed by the image of the particle. The intersection of the D and S waves in the image plane forms the image.

If the particle is transparent and introduces a small path difference, it can be shown as in figure 11 that D wave lags \( \lambda/4 \) wavelength behind the S wave when the optical path of the particle exceeds that of the surround. Now \( D + S = P \) the particle wave. The amplitude and the phase of the light that enters the image of the particle, is the amplitude and phase of the P wave and the amplitude and the phase of the light that enters the image of the surround is that of the S wave. Because the light transmission of the particle and the surround is the same, the image of the particle will show no contrast as the amplitudes of the P and S waves are equal.

In the phase contrast microscope, a diffraction plate is introduced at C', the back focal plane of the objective as shown in the figure. On the diffraction plate the conjugate area being small as compared with the area of the diffraction plate, the deviated waves pass through complementary area, that is, through that area of the plate which is unoccupied by the image of the opening of the condenser diaphragm.
Now by depositing a suitable coating of reflecting material, such as magnesium fluoride either on conjugate or complementary area of the diffraction plate, the S wave or the P wave is artificially retarded by $\lambda/4$ wavelength. Figure 12 shows the effect of such a coating over the conjugate area. The undeviated wave $S$ is now in phase with the deviated wave $D$ and therefore the amplitude of the wave $P = D + S$ becomes maximum. Therefore the particle appears brighter than the surrounding area and this is known as a negative phase contrast. If on the other hand a similar coating is deposited on the complementary area, the particle will appear darker than the surrounding area and then this gives a positive phase contrast.

2.7 Method

In this equipment as shown in figure 13, an annular disc is placed in the position of the normal iris $D$ and the image of it is therefore formed in the back focal plane of the objective. Through this image passes all the direct light ($S$) from the object. In this back focal plane, a phase plate is introduced. It is in the form of a disc of glass in which the annulus is cut corresponding to the size of the image of the condenser annulus and is of such a thickness so as to advance the direct wave by $\lambda/4$ in relation to the
diffracted wave passing through the remainder of the plate. We therefore obtain a positive phase contrast illumination. In the apparatus used, the optical assembly, the microscope objective, the beam splitter and the phase plate are mounted in the objective. The phase plate is located between the beam splitter and the eye-piece. It is a positive phase contrast with a single phase plate having 80% absorption with a phase retardation \( \lambda/4 \) and it is found that this single plate serves moderately well for the entire range of small optical path differences.

The surface of the specimen must be adjusted perpendicular to the optic axis of the microscope so that the image of the condenser annulus is centred on the optic axis. An auxiliary microscope used in the place of an eye-piece enables the examination of the back focal plane of objective so that the image of the condenser annulus and the phase plate may be made to coincide. The ring form of the condenser diaphragm has an advantage in as much as axial symmetry is retained with no introduction of asymmetry in the image.

In the case of reflection phase contrast microscopy, the surface of the specimen should be in one plane and specularly reflecting in order to form a sharp image of the annular condenser diaphragm on the
phase plate. If the different parts of the surface of the specimen form different images of the diaphragm as happens when small areas of the specimen though highly reflecting are inclined to each other, the advantage of the phase contrast technique is lost.

2.8 Light Profile Microscopy

This technique is a modified form of Smaltz light cut method (1936) and is quite useful for the study of growth features which are rather coarse for interferometric methods. Changes in level of less than a wavelength can be easily detected by this and furthermore it can be used to decide whether a feature is a depression or an elevation. The principle of this method is based on the Smaltz light cut technique which is as follows:

The image of an illuminated slit is projected with a microscope objective at an angle of incidence of 45° on the surface under examination. The image of the surface is then viewed with a microscope, the axis of which is at right angles to the first. It is clear that if the surface under examination has corrugations then the slit will appear corrugated correspondingly. This therefore converts the changes in depth into changes in extension. This method gave magnifications in extension upto 400 and in depth upto 650.
Spaltz light cut technique has many limitations such as:

1. It does not employ the full aperture of the lens.
2. It is difficult to identify the line of the surface which has been scanned since the image of the slit appears as a bright line against a dark background.
3. Non-specular regions give a false representation.
4. A special apparatus is required to be prepared with two microscopes at right angles.

These difficulties have been overcome by Tolansky (1952), in his light profile microscope by making the following modifications:

1. Well-defined slit image should be formed.
2. Single lens should be used for both illumination and viewing.
3. It is necessary to obtain an off-centre pencil illumination in order to secure the condition of light cut.
4. Monochromatic radiation should be used to avoid the chromatic difficulties.
5. Metallurgical type microscope should be used.

Any metallurgical microscope, by adopting the internal metal tongue reflector, can be readily adopted for high resolution light cut. In this method,
a slit is made of small pieces of razor blade, which need not be necessarily narrow, and is placed close to the field iris. The metal tongue internal reflector, which is a metallized sector of about 30° angle, sends an off-centred pencil which is incident on the specimen at an angle of about 40°, when high power lens is used. In this method the objective serves a double purpose of bringing in an off-centred pencil and passing out to the observer E. The experimental arrangement for light profile is shown in figure 14.

Light from A, which is a monochromatic source, passes through the lens L₁ and the diaphragm S₁. It then passes through S₂, which is a field iris in the universal illuminator. The profile which consists of a mounted thin wire or a scratch on a disk of glass is placed as near to S₂ as possible. The metal tongue reflector M of an illuminator sends an off-centred pencil as shown in the figure. The procedure of investigating the profile of the surface under examination is very simple. The surface to be studied is placed at X and the microscope is focussed to get a clear image of the surface in the eye-piece E. The profile is inserted now, near to S₂, and is moved forward and backward till the sharp image of the profile is formed on the surface. The profile appears
as a dark sharp line corrugated in extension for the surface features in depth and height. From the shift in the profile, the actual depth or height can be easily calculated.

If 'm' is the linear magnification of the microscope in extension, 'i' the effective angle of incidence of the pencil and 'n' the refractive index of the medium between the work piece and the objective, the magnification of the profile will be \((2m/n) \tan i\). In this expression \((2/n) \tan i\) is a constant quantity for a particular objective which can be readily evaluated by interferometric calibration. The values of the constant \((2/n) \tan i\) for different objectives used with the microscope in this work are as under:

<table>
<thead>
<tr>
<th>Objective</th>
<th>2.2 mm</th>
<th>3 mm</th>
<th>4 mm</th>
<th>8 mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>((\frac{2}{n}) \tan i)</td>
<td>1.0</td>
<td>1.5</td>
<td>1.23</td>
<td>0.56</td>
</tr>
</tbody>
</table>

Knowing the values of the constant \((2/n) \tan i\) and the linear magnification 'm' of the microscope in extension the magnification of the profile can easily be calculated.

Magnifications up to 2000 both in extension
and in depth can be used and a full aperture is available for the information of the image. This technique can be very well used both for transparent and opaque substances. It is necessary to evaporate silver on the surfaces of all transparent substances to be studied by this technique. Opaque substances can be studied without depositing silver on them.

2.9 Indentation Technique

The equipment used for the indentation purposes and to be used with the Vicker's projection microscope (shown in figure 15) is as follows:

1. Filar micrometer eye-piece in centring mount.
2. Tube length scale used for magnification setting.
3. Base plate contact anvil.
4. Beam contact tip.
5. Collet chuck securing specimen.
6. Chemical balance weights used to apply load.
7. Load centre indicator.
8. Red signal lamp.
10. Counter-weights.
11. Diamond indenter objective.
12. Electricity supply terminals.

Pivoted beam unit is secured to the bottom
of the case by means of two finger screws. On unscrewing these, it can be removed and placed to one side.

The support block for the load position is next removed on releasing the finger screw and is secured in the pocket at the back of the microscope slide.

The socket for the vertical pillar should be to the left hand side as one faces the microscope. The vertical pillar, horizontal bar and centre pin are assembled in position. The verticality of the main bar is checked and if necessary, adjusted by the use of the screw which is afterwards locked by means of the grub screw provided.

The diamond indenter objective, assembled in the centring mount, is placed in position in the universal illuminator. Assuming that the objective is accurately centred to the optical axis of the instrument, the load position indicator pin is carefully lowered, taking care to see that its movement is truly vertical and its point centred over the diamond indenter. The horizontal bar is locked by its clamp screw, and the set screw limiting the rotation of the vertical pillar is locked by its nut.

The pin will now indicate the position of the indenter within the range of its vertical movement.
and may be clamped at any desired height just to clear off the weight placed on the beam plate.

The beam unit is now secured to the main stage plate and it will be found most convenient so to arrange things that the stage micrometers point forward and to the right. This permits an almost complete revolution of the stage in clockwise direction.

The electrical connection to the transformer or battery is then made to complete the circuit for the 4V (1.2) lamp.

The specimen to be tested is mounted with collodion on a bakelite circular disc (1 inch in diameter) to fit freely to the collect provided. The surface to be tested should, of course, be normal to the axis of the cylindrical mount. The mounted specimen is then inserted into the collect and the milled ring tightened with the aid of the double pin key until it is firmly gripped into the holder. The collect is then registered in the 'V' slideway and locked by means of the clamp screw. The filar micrometer (reading to 0.01 mm on the micrometer drum) in its centring mount is assembled and clamped to the end of the instrument eye-piece tube. The hinged lock screw should now be released and when electrical constant is made, the
lamp will illuminate the red window. The beam must now be balanced and this is accomplished by the removal or addition of counter-weights in the pocket provided and the adjustment of the counter-weight of the screwed spindle. The flickering of the indicator lamp is of great assistance while balancing and it is advisable so to adjust the counter-weight that contact can just be maintained. Under these conditions, the contact will break on gently tapping the main casing of the instrument with the fingers.

Care is taken to see that the weights are placed just above the diamond indenter with the help of pre-set vertical pin of the load position indicator.

The selection of load is largely a matter of experience. Very light loads are used for soft materials or for minute crystalline structure. Generally, a load selected must give an impression of atleast 10 μ.

The region to be indented is scanned with the help of the reading objective, and then the diamond indenter is placed properly in the centring mount. After ensuring that the run of the fine motion mechanism is near the lower limit, the stage is lowered by coarse motion slide until the surface is believed to be approximately in focus. Clamping the slide, the fine motion mechanism is used to raise the indenter diamond
objective until the diamond makes contact with the specimen and lifts it sufficiently to break the contact between the conductors as denoted by the extinction of the red light. Strict count of the revolutions should be kept as fine motion is advanced as directed by the record on the label in the lid of the storage box. The contact of the diamond indenter and the specimen should be maintained for 15 seconds. On reversing the motion, the indented region is examined by the reading objective through the filar eye-piece (total magnification X 80).

Vickers hardness numeral (V.H.N.) is calculated by using the formula

\[ V.H.N. = \frac{1854 \times P}{d^2} \text{ (Kg/mm}^2\text{)} \]

Where 'P' is the load in grams and 'd' is the average length of the diagonal in microns of the indented impressions.

Microhardness of the specimens is also calculated in terms of Meyer number 'n' in order to avoid normal dependence of the hardness number on the force used. The Meyer hardness number is defined as the force applied divided by the area of the indent projected on to the plane of the specimen. Meyer
hardness number 'n' is calculated from the slope of the curve \( \log p \) against \( \log d \).

### 2.10 Replica Technique for Electron Microscopy

For an electron microscopic study of the crystal surfaces, electron optical plant EF-4, as shown in figure 16, and manufactured by Carl Zeiss Jena, was used.

For the electron microscopic examination single stage carbon replicas of the surfaces were made. For this purpose, carbon was deposited on to the surface, by thermal evaporation in a vacuum coating unit, shown in figure 17, at a pressure of \( 10^{-5} \) torr. The contrast in the electron micrograph was improved by shadowing the replica with chromium.

The replicas were detached from the surfaces by carefully immersing the crystal in distilled water. Alkali halides being soluble in water helped the easy detachment of the replicas from the crystal surfaces. The replicas were then mounted on the specimen grid for examination.

### 2.11 Etching Technique

Freshly cleaved crystal cleavages of alkali halides (KCl, NaCl and NaF) were dipped for a known period in a beaker containing the suitable etchant.
The cleavages to be etched were placed with their faces up in the beaker. After the required time of etch, the crystals were taken out and rinsed in suitable agents, and dried on filter paper using air blower. Care was taken to see that the cleaved surface of the crystal did not get contaminated with outside impurities otherwise well defined pits would not be obtained.

For thermal etching, the crystal cleavages were placed with their faces up on a platinum lid, and were heated in a muffle furnace kept at the required constant temperature for known period.

Chemically and thermally etched crystal cleavages were first mounted (after preliminary observation) on glass flats with collodion and then examined optically after the deposition of thin silver films on them to enhance contrast. Whenever it was necessary to re-etch the same surface, they were examined unsilvered.