# CHAPTER 2

**EXPERIMENTAL TECHNIQUES**

<table>
<thead>
<tr>
<th>CONTENTS</th>
<th>PAGES</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1 Introduction</td>
<td>17</td>
</tr>
<tr>
<td>2.2 Silvering Technique</td>
<td>17</td>
</tr>
<tr>
<td>2.3 Replica Technique for Electron Microscopy</td>
<td>19</td>
</tr>
<tr>
<td>2.4 Various Microscopes Used</td>
<td>19</td>
</tr>
<tr>
<td>2.4.1 Incident light microscope 'Epignost'</td>
<td>19</td>
</tr>
<tr>
<td>2.4.2 Vickers projection microscope</td>
<td>20</td>
</tr>
<tr>
<td>2.4.3 Electron microscope: Carl Zeiss EF 4</td>
<td>21</td>
</tr>
<tr>
<td>2.5 Multiple Beam Interferometry</td>
<td>23</td>
</tr>
<tr>
<td>2.6 Indentation Technique</td>
<td>25</td>
</tr>
<tr>
<td>2.7 X-ray Diffraction Technique</td>
<td>27</td>
</tr>
<tr>
<td>2.8 Etching Technique</td>
<td>28</td>
</tr>
<tr>
<td>References</td>
<td>29</td>
</tr>
</tbody>
</table>
2.1 Introduction

During the present investigations, a number of experimental techniques such as silvering technique, optical and electron microscopy, multiple beam interferometry, microhardness indentation, X-ray diffraction etc. have been employed. A brief account of them is given below.

2.2 Silvering Technique

The crystal surfaces and the optical flats have to be coated with a highly reflecting layer of silver for both microscopic and interferometric studies. For this purpose silver was thermally evaporated on to the specimen using a commercial vacuum coating unit 'Edwards 12 EA'
(fig. 2.1). 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 built in the unit. The surfaces were thoroughly cleaned before the deposition of silver. The cleaning process of surfaces depended 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 $H_2O_2$. Thereafter, they were cleaned by rubbing with dry cotton wool till no breath figure was formed on breathing over them. Freshly cleaved crystal surfaces did not require any cleaning. However, the as-grown habit faces as well as the etched surfaces were usually cleaned by washing them with water after applying soap. Final cleaning was done by ionic bombardment in the vacuum coating unit by means of a high tension discharge.

When the pressure was about $1 \times 10^{-5}$ 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.
2.3 Replica Technique for Electron Microscopy

Growth features and some of the etch patterns were studied in transmission electron microscopy by preparing their carbon replicas. A single stage replica technique developed in this laboratory by Patel and Patel which provided better resolution than the earlier techniques was employed for this purpose. For the preparation of replicas an accessory coating unit of Carl Zeiss Jena (fig. 2.2) was used. A few monomolecular layers of NaCl were first deposited on the surface whose replica was to be prepared, by thermal evaporation at a pressure of $1 \times 10^{-5}$ torr. Over this coating spectroscopically pure carbon film of required thickness was deposited by striking carbon arc inside the coating unit. Finally, the carbon film was metal shadowed with chromium for contrast. The specimen with the film was then taken out and immersed in distilled water after breaking the film at the edge of the surface, when the thin film of NaCl dissolved and thus the carbon replica got separated from the crystal surface easily and safely. The floating replica was then collected over a copper grid and dried before examining in the electron microscope.

2.4 Various Microscopes Used

2.4.1 Incident light microscope 'Epignost'

Almost all preliminary examination of the
crystal surfaces, as well as photographic recording of the observations at lower magnifications, were done with the help of 'Epignost'. This microscope has been designed for the rapid examination of ground, polished, etched or natural surfaces of the objects and hence it affords every convenience called for this kind of examinations. Intended for low magnification only (upto about 285 X) it is accordingly provided with coarse adjustment. Being an incident light type microscope the objective has an infinite intersectional distance i.e. the specimen lies in the front focal plane of the objective and its image is formed at infinity. The instrument contains a permanently built-in tube lens which together with the eye-piece forms a telescope thus resulting in a factor of 0.63 for calculating the total magnification. A 6 V 15 W filament lamp serves as the source of light.

The 'Epignost' can also be used for photomicrographic work. Specially suitable for that purpose is available the photomicrographic 'MF' equipment in combination with a miniature camera (fig. 2.3). For the purpose of photography the 'MF' tube is fitted to the tube carrier of the 'Epignost' in place of monocular tube.

2.4.2 Vickers projection microscope

Vickers projection microscope shown in fig. 2.4
was employed for the microtopographical as well interferometric studies. This is an inverted type metallurgical microscope in which the specimen to be studied is placed on a movable stage above the objective lens. The flexible illuminating system which can be used both for the transmission and reflection photography consists of a powerful mercury lamp, pointolite or carbon arc lamp, a condenser and aperture controlled by iris diaphragm. 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 focussed on the projection screen after reflection from the projection mirror. Slight refocussing is necessary when the visual system is changed over to the projection system.

2.4.3 **Electron microscope: Carl Zeiss EF 4**

The Carl Zeiss EF 4 electron optical plant (fig. 2.5) used in the present investigation designed and developed for the electron optical procedures consists of the following three basic units:

(a) Microscope column with control panel and the microscope

(b) Current and voltage supply cubicles
(c) Backing pump assembly.

In line with the principle of the building brick system, the tube of the three stage electron optical lens system has been secured to a stable fixture of the control panel, while the other sub-assemblies of the microscope column have been flanged on both ends of the lens tube and rigidly connected.

The electron optical system of the microscope along with the microscope column is shown in fig. 2.6. The objects to be studied are placed just below the objective lens and can be displaced in two perpendicular directions to the optic axis and can also be tilted by $\pm 10^\circ$ from the normal incident beam. The image formed by the objective lens is remagnified by the intermediate and projective lenses to produce a final image on a fluorescent screen. The lens system of the microscope also contains the control and the range diaphragm with the mechanism for shifting and disengaging them.

The final image formed on the screen can be photographed by lifting the screen from the beam path manually. The photographic plate chamber consists of a magazine holding six (60 mm x 90 mm size) photographic plates which can be exposed in any adjustable plate size during operation.
The practical resolving power and the maximum useful magnification with the present equipment are about 20 \( \frac{A}{\mu} \) and 50,000 respectively.

2.5 Multiple Beam Interferometry

Multiple beam interferometry, a very sensitive technique for the study of crystal surfaces and growth features was developed by Tolansky\(^2\) and his co-workers. Since the technique by now is quite an established one, only those points which have a special bearing on the investigation of crystal surfaces will be discussed here. When two compound surfaces (fig. 2.7) of an interferometer are parallel to each other at a distance \( t \) and have transmission and reflection coefficients \( T \) and \( R \) respectively, the summation of the transmitted series of multiple reflected beams gives the intensity distribution as

\[
I_{\text{max}} = \frac{T^2}{(1 - R^2)} \quad \text{at} \quad n\lambda = 2t \cos \theta.
\]

and

\[
I_{\text{min}} = \frac{T^2}{(1 + R^2)} \quad \text{at} \quad (n + 1/2)\lambda = 2t \cos \theta.
\]

If there is no absorption by the two component surfaces \( R + T = 1 \) by definition. The fringe peak maximum without absorption has, then, the same intensity as that of the incident light. Therefore the fringe shape is
independent of $T$ and is determined only by the reflectivity $R$. Even if we take into account of absorption, it can be seen that the absorption merely influences the total intensity and not its shape.

It should be noted that if the surface to be studied is of opaque specimen it is necessary to observe the fringes in the reflected system. In this case the silvering of the flat is more critical as any absorption in the silver reduces the visibility of the fringes.

The principal factors deciding the usefulness of the multiple beam fringes are the contrast and the sharpness. The contrast may be defined by the term $(I_{\text{max}} - I_{\text{min}})$ and the sharpness in terms of fringe half width $\delta$ which is given by the relation

$$\delta = \frac{(1 - R)}{\sqrt{R}}$$

where $R$ is the geometric mean $\sqrt{R_1 R_2}$, $R_1$ and $R_2$ being the reflectivities of the two surfaces. Hence $\delta$ is minimum when $R$ is maximum. To achieve a maximum value of $R$ it is essential to reduce the absorption coefficients as much as possible. The contrast or the visibility of the fringes also depends on absorption especially in the reflected system.
Although Fabry and Perot\textsuperscript{5}) used a thin wedge silvered on both sides to produce sharp fringes, Tolansky\textsuperscript{2}) was the first who investigated the critical conditions to be fulfilled for a doubly silvered wedge, so that a close approximation to the Airy summation\textsuperscript{6}) can be achieved. He has formulated the following conditions.

1. The surface must be coated with a highly reflecting film with minimum absorption.

2. The film should contour the surface exactly and be highly uniform in thickness.

3. Monochromatic light or at most a few widely spaced wavelengths should be used.

4. The interfering surfaces should be separated by a few wavelengths of light only.

5. A parallel beam within 1-3° tolerance should be used.

2.6 \textbf{Indentation Technique}

To study the microhardness of the crystals grown by different methods and the propagation of dislocations in them the indentation technique was employed. The equipment attached to the Vickers projection microscope is as shown in fig. 2.8. The various components numbered in the figure are as follows:
1. Filar micrometer eye-piece in centering mount.
2. Tube length scale for magnification setting
3. Base plate contact anvil.
4. Beam contact tip
5. Collet chuck securing specimen.
6. Chemical balance weights to apply load.
7. Load centre indicator.
8. Red signal lamp.
9. Auxiliary counter weight.
10. Counter weight.
11. Diamond indentor objective.
12. Electricity supply terminals.

The specimen to be indented was mounted on a circular metal disc and the disc was inserted in the collet. The beam was then balanced by the addition or removal of counter weights in such a way that the contact was just made which could be indicated by the flickering of the indicator lamp. Required load was then applied. The region to be indented was scanned with the help of the reading objective and then the diamond indentor was placed. After lowering the stage so that the surface was sufficiently near to the indentor, the diamond indentor was raised with the help of fine motion mechanism until it made a contact with the surface of the specimen and lifted it
sufficiently to break the electrical contact. The contact was maintained for 30 seconds. By reversing the motion the indentor was removed and the indented region was examined with the reading objective through the filar eye-piece.

The diagonals of such indentations were measured whenever necessary and the Vickers Hardness Numeral (V.H.N.) was calculated using the formula:

$$V.H.N. = \frac{2p \sin \theta/2}{d^2} \text{ Kg/mm}^2$$

where \( p \) is the load on the indentor in terms of Kg. wt., \( \theta \) is the angle between the opposite faces of the indentor and \( d \) is the average diagonal length in mm.

For \( \theta = 136^\circ \) which is the case with the Vickers pyramidal diamond indentor the formula simplifies to

$$V.H.N. = \frac{1.854 \times p}{d^2} \text{ Kg/mm}^2$$

2.7 X-ray Diffraction Technique

The X-ray investigations of the various specimens were made using Philips 1000 W X-ray generator type PW 1009 (fig. 2.9). This constant potential X-ray unit provides a high quality recording of diffraction
spectra with all types of cameras. The degree of stabilization of the generator permits qualitative as well as semi-quantitative X-ray diffractometry. The shutter of the tube shield can be operated automatically by means of mechanical timers type PW 1017. Also available is a magnetic water valve type 1018 that automatically cuts off the water supply when the generator is switched off. The unit provides continuously adjustable high voltage 0-55 kV and tube current 0-40 mA. with a stabilised filament current. In the present investigations the Laue camera (Philips Holland) and the rotation camera (got prepared in the S.P.J. workshop) both in conjunction with the goniometer of type PW 1031/00 were employed. Generally tube with copper target was used.

2.8 Etching Technique

The first direct proof that dislocations can be revealed by means of etching was given by Gevers et al.\textsuperscript{8)} and Horn\textsuperscript{9)} on SiC. At present etching has become the most widely used method for studying defects in crystals. In the present investigations, to reveal dislocations and other imperfections existing in the crystals etching technique was employed. The method consists simply of immersing the crystal in a suitable medium e.g. a liquid, a solution
or a gaseous chemical reagent, these being called by the general name 'etchant'. Out of different methods of etching only one type viz. the chemical etching was used in the present work. The details of this will be given in the appropriate chapters.

In addition to the above mentioned techniques the author has also made use of the technique of chemical polishing which will be dealt with in relevant places.

References