## CHAPTER 4

### EXPERIMENTAL TECHNIQUES

<table>
<thead>
<tr>
<th>Contents</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.1 Introduction</td>
<td>58</td>
</tr>
<tr>
<td>4.2 Silvering technique</td>
<td>58</td>
</tr>
<tr>
<td>4.3 Microscopes used</td>
<td>59</td>
</tr>
<tr>
<td>4.3.1 Incident light microscope &quot;Epignost&quot;</td>
<td>59</td>
</tr>
<tr>
<td>4.3.2 Vicker's projection microscope</td>
<td>60</td>
</tr>
<tr>
<td>4.3.3 Electron microscope</td>
<td>61</td>
</tr>
<tr>
<td>4.4 X-ray diffraction technique</td>
<td>62</td>
</tr>
<tr>
<td>4.5 Indentation technique</td>
<td>63</td>
</tr>
<tr>
<td>4.6 Dielectric measurements</td>
<td>66</td>
</tr>
<tr>
<td>4.7 Electrical conductivity measurements</td>
<td>67</td>
</tr>
<tr>
<td>4.8 Etching technique</td>
<td>68</td>
</tr>
<tr>
<td>References</td>
<td>69</td>
</tr>
<tr>
<td>Captions of figures</td>
<td>70</td>
</tr>
</tbody>
</table>
4.1 Introduction

A proper understanding of the experimental techniques and the instruments used is necessary before subjecting them to fruitful research. In the present work a number of experimental techniques such as optical microscopy, electron microscopy, X-ray diffraction, electrical conductivity measurements, dielectric measurements, etc. have been employed. A brief account of the materials and method is given as under.

4.2 Silvering technique

The crystal surfaces and the optical flats have to be coated with a highly reflecting layer of silver for microscopic studies. The principle of this method is to thermally evaporate silver on the specimen at low pressure. A commercial vacuum coating unit (Fig. 4.1) Edwards 12 EA (England) was used for this purpose. The vacuum chamber was evacuated by three stage silicon oil diffusion pump backed by an oil rotary pump. The vacuum at different stages was measured by 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 depend very much on the nature of 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 dry 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 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 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.

4.3 Microscopes used

4.3.1 Incident light microscope "Epignost"

Preliminary optical microscopic examination of the crystal surfaces as well as photographic recordings at relatively lower magnification are performed using Carl Zeiss Jena (West Germany) "Epignost" incident light microscope. This microscope can be conveniently used for rapid examination of surfaces of crystals. Being an
incident light type of 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. Magnification from 11 to 197 could be attained in certain steps in this microscope. A 6V, 15 W filament lamp serves as the source of light.

The Epignost with a photomicrographic "MF" equipment in combination with a miniature 35 mm camera (Fig. 4.2) can be excellently used for the photomicroscopic work.

4.3.2 Vicker's Projection Microscope

Vicker's projection microscope (England) has been used for microtopographical studies and is shown in Fig. 4.3. This is an inverted type of metallurgical microscope in which the specimen to be studied is placed in a moveable stage above the objective lens.

The flexible illuminating system which can be used for both transmission and reflection photography
consists of a powerful mercury lamp, potatalite or carbon arc lamp, a condenser and an aperture controlled 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 screen after reflection from the projection mirror. A slight refocussing is, of course, necessary when visual system is changed to the projection system.

4.3.3 Electron microscope

The Scanning Transmission Electron Microscope (STEM), Philips EM 400, with attached EDAX (Energy Dispersive Analysis of X-rays) system has been shown in Fig. 4.4.

For scanning, the electron microscope has resolution of about 5 $\mu$m. In the normal mode of operation, electrons accelerated by a potential 20 to 120 kV are directed on the sample, and the scanning coils cause the beam to move across the specimen in a square raster. The secondary electrons which are emitted from the specimen strike the collector electrode and the resulting current is amplified and used to modulate the brightness of a corresponding spot which is displayed.
on a cathode ray tube. The time associated with emission and collection of secondary electrons is negligible compared with the time of the scan, and so the number of secondary electrons collected from any point on the specimen is determined only by the "brightness" of the spot. Image contrast can arise from number of factors, particularly surface topography, atomic number, electrical conductivity, specimen orientation and electric or magnetic fields.

4.4 X-ray diffraction technique

The X-ray analysis has been carried out using the Philips X-ray generator type PW 1009 (Holland) shown in Fig. 4.5. This X-ray unit provides continuously adjustable high voltage 0-55 kV and tube current 0-40 mA.

In a powder diffractometer\(^1\), the diffracted radiation is detected by G.M. counter tubes which move through angular range of reflections. The intensities are recorded on synchronously advancing strip chart. The powder diffractometer, shown in Fig. 4.6, consists of the X-ray generator TUR M 61 made by VEB transformatoren and Rentgen Werk Dresden. The horizontal counter goniometer H203 and the linear momentum density recorder VAD 53-1 made by Freiberger Prazisions Mechanik GDR. A selection of five different goniometer angular
velocities of $1/12 \text{ min}^{-1}$ to $2 \text{ min}^{-1}$ and various recorder chart speeds are available. Determinations with the diffractometer in general, require approximately 100 times more of the substance than the conventional Debye-Scheerer method. Compared with photographic methods, the diffractometry in most cases, offers essential advantages due to the higher sensitivity, the higher resolving power, the accuracy of the intensity measurements and the elimination of the elaborate work in dark room. Above all, the diffractometric records can be obtained in a much shorter time than with photographic method.

4.5 Indentation technique

In order to study the microhardness of the crystals, the indentation technique is employed. The necessary equipment for the purpose is always to be attached to the Vicker's projection microscope and is shown in Fig. 4.7. The various components as 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. Collect 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 indenter objective.
12. Power supply for signal lamp.

The specimen to be indented is mounted on a circular aluminium disc and inserted in the collect. The beam is then balanced by the addition or removal of counter weights in such a way that the contact was just made which can be indicated by flickering of the red signal lamp. A required load is then applied. The region to be indented is scanned with help of the reading objective and then the diamond indenter is properly placed. After lowering the stage to bring the indenter near the crystal test surface, the diamond indenter is raised with the help of fine motion mechanism until it just makes a contact with the test surface. Then it is lifted sufficiently to break the electrical contact. The contact is normally maintained for, say, 30 seconds. By reversing
The motion, the indentor is removed and the indented region is examined with the reading objective through the filar eye-piece.

The diagonals of such indentation marks are measured and Vickers Hardness Number (VHN) is calculated using the formula:

\[ VHN = \frac{2p \sin \theta/2}{d^2} \text{ kg mm}^{-2} \]  \hspace{1cm} (1)

where \( p \) is the load applied on the indentor in units of kilogram weight, \( \theta \) is the angle between the opposite faces of the indentor and \( 'd' \) is the average diagonal length in mm.

For \( \theta = 136^\circ \), as is the case with the Vicker's pyramidal indentor the formula simplifies to:

\[ VHN = \frac{1.854 \times p}{d^2} \text{ kg mm}^{-2} \] \hspace{1cm} (2)

It is generally agreed that VHN is independent of the load when measured by a standard Vicker's hardness machine operating at loads greater than...
than 2.5 kg. However, several investigators found the variations in VHN with decrease in load (for operating loads less than of 1 kg) and have tried to explain their results with suitable modified formula. Specifically, the formula suggested by Onitsch\(^3\) is:

\[
VHN = 1.854 a d^{n-2}
\]  

where \(a\) and \(n\) are the constants for a given material.

4.6 **Dielectric measurements**

The Systronics LCR Bridge type 921 enables the following measurements to be made:

1. DC resistance

2. AC resistance, capacitance and inductance at frequencies of 100 Hz, 1 kHz and 10 kHz using the internal AC source at any frequency from 50 Hz to 50 kHz.

3. The loss angle and Q-factor of a capacitor or inductor.

The LCR bridge is basically a development of the well known Wheatstone bridge. One of the bridge diagonals is powered either by a DC or AC voltage according to the nature of the component to be tested. The other diagonal is a zero detector that includes an amplifier powering a meter. The bridge balance is determined by a sensitive meter preceded by the null amplifier. The amplifier voltage and the AF bridge input voltage at any frequency are available at terminals and allow the display of Lissajous figures so as to facilitate visual balancing of the bridge when the minimum null is difficult to be obtained by means of the meter.

4.7 Electrical conductivity measurements

The million megohmmeter, model 2M 160 MK III A manufactured by BPL India, has been used for the electrical conductivity measurements. The ranges of measurements possible with this meter is from 10 million ohm to 400 tera ohm and current down to 1.0 pico ampere. This self contained instrument is provided with DC test voltages of 10 V, 50 V, 100 V, 250 V, 400 V, 500 V and 1000 V. An automatic, built in delay circuit computes the charging delay time required for the test sample. Since the delay calculator indicates an over load
protection, the lamp will also flash if the test sample breaks during test or should the range selected is too sensitive.

4.8 Etching technique

The first direct proof that dislocations can be revealed by means of etching was given by Gevers et al.\(^\text{4}\) and Horn\(^\text{5}\) on SiC crystals. Now-a-days etching stands on firm footing as most widely used method for studying defects in crystals, and this has been employed in the present investigation also to reveal dislocations and other imperfections existing in the grown crystals. In this method, a crystal is to be immersed in a suitable medium, e.g. a liquid, a solution or a gaseous chemical reagent, called by the general name "Etchant". Out of the different methods of etching, the one of chemical selective etching alone is used in the present work.
References

1. B. D. Cullity : Elements of X-ray diffraction
Addison-Wesley, Inc.,

2. B. W. Mott : Microindentation Hardness
Testing
Butterworths Publication,
London (1956).


4. R. Gevers,
S. Amelinckx and
W. Dekeyser : Naturewissenschaften
32 (1952) 448.

5. F. H. Horn : Phil. Mag. 42 (1952) 1210.
Captions of figures

Fig. 4.1 "Edwards" vacuum coating unit 12 EA.

Fig. 4.2 Incident light microscope "Epignost"

Fig. 4.3 Vicker's projection microscope.

Fig. 4.4 "Philips" EM 400 with EDA

Fig. 4.5 "Philips" X-ray unit, PW 1009

Fig. 4.6 Powder diffractometer with X-ray generator TUR M 61, the horizontal counter goniometer HZG3 and the linear momentum density recorder VAD 53-1.

Fig. 4.7 Experimental set up for indentation.