CHAPTER 2

EXPERIMENTAL TECHNIQUES

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2.1 Introduction

In the present investigation a number of experimental techniques such as optical microscopy, electron microscopy, X-ray diffraction, etc. have been employed. The salient features of all these techniques are described in this chapter without entering into elaborate mathematical theories for which references can be made to standard texts.

2.2 Vacuum Coating Unit

Sometimes it is necessary to enhance the contrast of the crystal surfaces under examination and as such surfaces are coated with thin uniform layer of silver which gives highly reflecting surface. For this purpose, however, a condition of perfect -ness is of paramount importance. The cleaning process to be used depends upon the nature of the surface about to receive the silver. For studying the electrical properties of crystals, silver electrodes can also be deposited on the crystal surface by this technique.

In the present investigation the vacuum coating unit was employed for evaporating the silver film on the crystal surfaces and for measuring the electrical and thermoelectrical properties of the crystals at high temperatures and at low pressures. The principle of this method is to thermally evaporate silver on the specimen surface at a low pressure. A commercial vacuum unit used for this purpose is shown in Fig. 2.1. It consists of a vacuum chamber in the form of large pyrex bell jar resting in an annular recess upon a gasket of neoprene rubber which rests on a horizontal metal base plate. The vacuum chamber is evacuated by a three stage silicon oil diffusion pump backed by an oil 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 Penning gauge incorporated in the unit.

The chamber is first evacuated by rotary pump and when a pressure of the order of 0.2 torr is reached, the chamber is connected to the diffusion pump. Silver is generally evaporated when the pressure in the chamber is about 10^{-5} torr. Ionic cleaning is done inside the chamber before the evaporation of silver. When the pressure of about 10^{-5} torr is reached, the molybdenum boat is heated by passing a low tension high current. 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, by removing the shutter from over the filament for the required time.

In order to study the electrical

and thermoelectrical properties of crystals, the vacuum coating unit described here can be used with the suitable modifications.

2.3 Incident Light Microscope - 'EPIGNOST'

Most of the preliminary examination of crystal surfaces as well as photographic recording of the observations at lower magnification was accomplished with the help of 'Epignost'. This microscope has been designed by Carl Zeiss, Jena for rapid examination of ground, polished, etched or natural surfaces of the objects and hence it affords every convenience called for this kind of examination. Being an incident light type microscope, the objective has an infinite intersectional distance, i.e. the specimen lies in the front focal plane of objective and its image is formed at infinity. The instrument contains a permanently built in tube lens which together with eye-piece forms a telescope thus resulting in a factor of 0.63 for calculating the total magnification. A 6 volts, 15 watt filament lamp serves as the source of light.

The 'Epignost' can also be used for

photomicrographic work for which it is provided with a specially suitable photomicrographic "MF" equipment in combination with a miniature camera (Fig. 2.2).

2.4 Electron Microscopes

2.4.1 Carl Zeiss EF-4

The electron optical plant (Fig. 2.3) used in the present investigation has been designed and developed for the electron optical procedures by Carl Zeiss Jena, East Germany. This electron optical plant can be used for transmission, reflection and emission electron microscopy (using bright field, dark field and stereo transmission microscope) as well as electron diffraction employing various beam paths.

Electrons emitted from the tip of a hot-tungsten filament (shaped like a hair pin) are accelerated towards the anode and focussed by a negative bias on the surrounding shield (Wehnelt cylinder). In order to control the intensity of illumination and the aperture of illumination on the object, the condenser lenses are used. The first condenser lens is to produce a reduced image of the source and the second one is adjusted to give the proper intensity of illumination.

The objects to be studied are placed in front of 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 the projective lens to produce a final image on the fluorescent screen which can be observed at a higher magnification by means of an optical microscope. The lens system of the microscope also contains the contrast and the field limiting diaphragms which can be adjusted during operation by means of a rapid swinging device. It also contains a beam diaphragm, objective diaphragm and a projective diaphragm.

The field 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 (65 mm x 90 mm size) photographic plates which can be exposed in any adjustable plate size during operation.

Besides, the highly stabilized power supply being given out in three steps of 35 kV, 50 kV and 65 kV, the plant consists of a pumping system for maintaining a vacuum of the order of 10^{-5} torr.

The practical resolving power and the maximum useful magnification obtainable with the present equipment are about 20 Å and 50,000 X respectively.

2.4.2 Philips EM-400

EM-400 is the advanced transmission electron microscope based on clean high vacuum technology. It incorporates a computer memory controlled seven lens optical system and kV section between 20 and 120 kV. New standards of thermal, mechanical and electrical insulation make resolution of 1.4 Å realisable under all normal laboratory conditions.

In recent years much improvements have been made in the design of electron microscope. The design of the Philips EM-400, Holland (Fig. 2.4) is so new, with so much innovation and so many noval ideas that besides all the improvements made in the previous models, many more have been introduced in this present model. This electron optical unit is surrounded by a magnetically isolated outer wall of thick, soft iron which, while isolating the electron optics from magnetic influences, also shields the lenses from convection currents. To minimise the thermal stability, cooling water system is surrounded for all lenses.

Newly designed objective lens gives exceptionally low aberration constants. To realise the potential of this excellent lens, the optical system is designed to incorporate a total of seven lenses, of which two are condenser lenses, one objective lens, one diffraction lens, one intermediate lens and two projective lenses. Five imaging lenses ensure that a wide range (50 X to 800,00 X) of high fidelity, full field images are produced on the screen. Correct balance betwen magnifying lenses reduces radial, spiral and chromatic errors at each magnification to negligible levels.

In this microscope, vacuum seals in beam area, a major source of reactive components are drastically reduced in number by the use of stainless steel linear tubes throughout the column. The column is pumped to an operational pressure of 10^{-7} terr by an ultra clean ion getter pump. A 200 µm differential aperature in the second projector lens provides essential separation of the clean column environment from the plate camera and viewing chamber. Effective vacuum separation is achieved without loss of image area.

An electron gun, over here, produces an extremely wide range of beam intensities at all accelerating voltages. Emission current can be varied in six steps between 2.5 - 100 μA. Basic gun conditions can also be modified by varying the filament wehnelt cylinder distance and brightness upto 10^6 amp/cm^2/sterad can be achieved. Highly coherent spots down to 0.2 μm diameter can be formed on the specimen using standard hair pin filaments. Final image is formed on the large screen which is coated with phospher so that the image can be seen clearly. 12 X binoculars are attached to the microscope from which the critical focusing is done by forming the image on small screen coated with different phospher materials. This electron optical plant contains accelerating voltages of 20-120 kV (20, 40, 60, 80, 100, 120 kV) which are selected by a single switch. Lens currents are automatically adopted so that after voltage change a fully corrected image at the same magnification is produced. Microscope is having a principal tilt of specimen upto $\pm 60^\circ$ and second tilt at right angles upto $\pm 30^\circ$. The resolution of this microscope is 1.4 Å line or 3 Å point and is attainable when it is equipped with high magnification stage. The highest magnification obtainable is 18,00,000 X.

The principal advantages of this microscope are :

(i) It consists of a beam tilt for dark field which means that the beam tilts from bright field to dark field images occur without any loss of illumination. No shift of the spot occurs when switching the beam tilt device on and off.

(ii) It possesses a built in facility for taking weak beam pictures (Chapter 11).

(iii) A flat camera and a 35 mm roll film camera can be fitted so that the ideal camera can be chosen for each investigation. A complete new design of the plate camera is used which has a maximum capacity of 36 plates, 6.5 x 9.0 cms in size. Plate transport is carried out pneumatically. On each plate the following information is registered automatically :

(a) Magnification or camera length (6 digits),

(b) Exposure number (4 digits),

(c) Operator indication (2 digit code), and

(d) Accelerating voltage.

35 mm camera is mounted 32 cm higher than the plate camera. The capacity of this camera is 36 exposures, each of 28 x 28 mm in size. The greatest advantage is that in one operation, all together, 72 photographs can be taken without breaking the vacuum.

This microscope has also a built in facility for examining the specimens at different temperatures starting from liquid nitrogen temperature to about 1273° K. For this purpose specially designed (i) heating holder and (ii) cooling holder are used.

In heating holder the specimen is clamped into a small furnace element, which is insulated from the holder by three zirconium oxide spheres. A thermocouple is welded on to the furnace and the connection for this and the heater is carried through the four channel ceramic tube. At the other end of the holder, outside the vacuum, are electrical connections.

In the case of cooling holder the specimen is clamped into a specimen carrier, which can be cooled by conduction to a cold exchanger which itself is cooled by circulation of cold gas, e.g. liquid nitrogen. The carrier is insulated from the holder. Two thermocouples, one for temperature measurement, the other for temperature control, are soldered on to cold exchanger and are carried through the holder. This holder may also be used for limited specimen heating (393°K); in this case liquid nitrogen is not used but the thermocouple is used as a heater to heat up the specimen.

In both types of the holders anticontamination device is generally required to prevent contaminations. The temperature control and measuring unit contains the current supply for the heater and also the circuitary necessary to measure the voltage generated by the thermocouple. A digital voltmeter is used to measure the temperature of either holder. The digital voltmeter can be used to measure three parameters,

(i) the output voltage of the stabilised supply in position 'V',

(ii) the output current of the stabilised supply in position 'I', and

(iii) the voltage generated by the thermocouple in position 'mV'.

Thus in this optical system, the

improved optical design and stability make it possible to offer high resolution combined with a choice of specimen tilting and magnification ranges as per requirement. Moreover, a wide choice of holders is available giving a range of specimen orientation and treatment facilities as well as meeting the requirements of operation with accessory system such as the S(T)EM unit and electron energy dispersive analysis of X-rays. The entire unit is so designed that it is fully extendable for S(T)EM, X-ray microanalysis and energy loss analysis with both standard and field emission sources.

2.5 X-ray Diffraction Techniques

2.5.1 Philips type PW 1009 X-ray Unit

The X-ray investigations of the various specimens were made using Philips 1000 W X-ray generator type PW 1009 (Fig. 2.5). This constant potential X-ray unit provides a high quality recording of diffraction spectra with all types of cameras. The unit provides continuously adjustable high voltage, 0-55 kV and tube current, 0-40 mA. It uses a filament current stabilizer for the stabilization of voltage.

The smoothed direct voltage given out by the above generator results in shorter exposure times with consequent longer tube life. The shutters of the tube shield can be operated automatically by means of mechanical timers. There is a magnetic water valve system which automatically cuts off the water supply when the generator is switched off. In the present investigation Debye-Scherrer powder camera was used for the characterization of the grown crystals.

2.5.2 Frieberger prazisions mechanik TUR M-62

For rotation and Weissenberg photographs the X-ray unit, "Frieberger prazisions mechanik TUR M-62 (Fig. 2.6) having adjustable operating voltage 0-60 kV and tube current 0-100 mA, was employed. The equipment essentially has,

(i) a control table containing the assemblies, H.T. generator, mains voltage stabilizer, H.T. stablizer, tube current stabilizer and a safety device,

(ii) a working table consisting of synchronous motor driven automatic timer and a strong frame equipped for positioning the tube shield as well as the setting up and fixing of camera and radiation protection panels,

(iii) an efficient cooling system connected to H.T. generator and X-ray tube where a water flow of atleast 3 litres/mm is ensured.

The Weissenberg-Goniometer 61 shown in Fig. 2.6 was used for X-ray diffraction. With the aid of a specially prepared sub-structure perfect manipulation of the instrument in front of the tube is ensured. The film cylinder (effective film ø = 60 mm, length of film 150 mm) conveniently holds simultaneously three films with black paper wrappers as a protection from light.

2.6 Etching Technique

The first direct proof that dislocations can be revealed by means of etching was given by Gevers et al.[1] and Horn[2] on silicon carbide crystals. New-a-days etching has become the most widely used method for studying defects in crystals. In the present investigation, to reveal dislocations and other imperfections existing in the crystals, etching technique was employed. In this method a crystal is to be immersed 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 the different methods of etching, the method of chemical etching alone is used in the present work. The details of this technique is given in Chapter 7.

2.7 References

1. Gevers, R., Amelinckx, S., and Dekeyser, W.
Naturewissenschaften 39 (1952) 448.

2. Horn, F. H.
Phil. Mag. 43 (1952) 1210.

Captions of the figures

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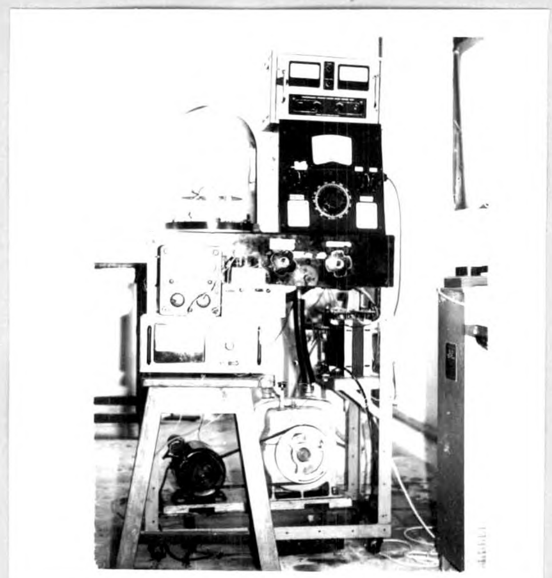

Fig. 2.1

Fig. 2.2

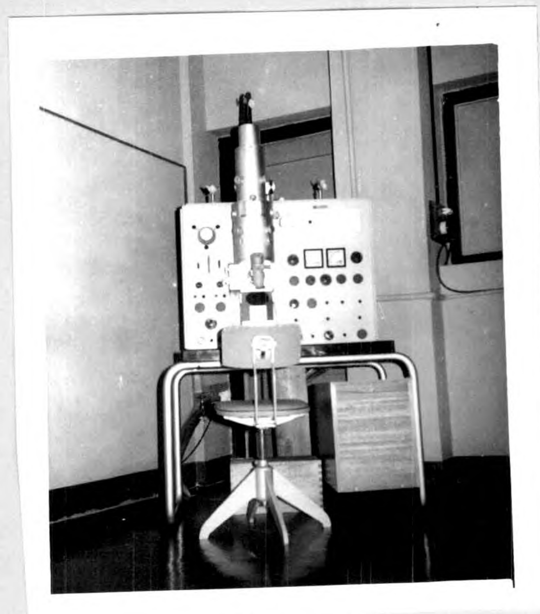

Fig. 2.3

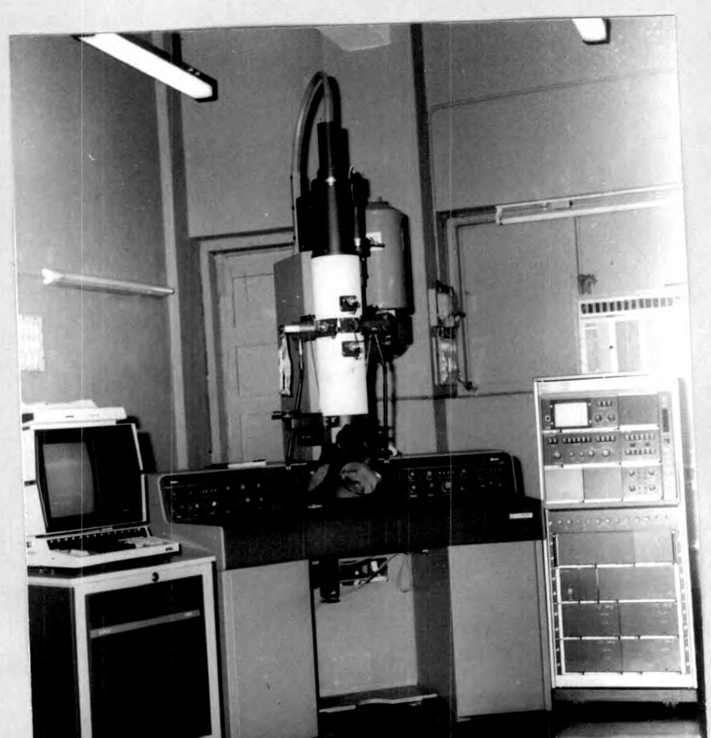

Fig. 2.4

Fig. 2.5

Fig. 2.6