CHAPTER IV

EXPERIMENTAL TECHNIQUES

In this chapter, a brief review of various experimental techniques employed by the author during the course of the work is presented. The Bridgeman's method and Chalmer's method of growing single crystals from melt are well-known, so also are the techniques of phase-contrast microscopy, light-profile microscopy, and multiple beam interferrometry used in the optical study of the crystal surfaces. A detailed account of various types of furnaces and the methods of growing single crystals is given by Lawson and Neilsen[1].

The discussion of the techniques involved in the optical investigation of the crystal surfaces is available in the literature[2-4]. Hence the details of the theory and construction of the various apparatus are not discussed.

The various techniques employed are discussed under three different heads:

A. Crystal growing techniques,

B. Techniques for optical studies,

C. X-ray technique.

A. CRYSTAL GROWING TECHNIQUES

Preparation of Tellurium single crystals from the vapour phase:

Tellurium single crystals have been grown from the vapour by sublimation of Tellurium in an evacuated pyrex glass tube. The tellurium metal used was the grade-I metal from Johnson Matthey Chemicals Ltd., U.K. and 99.995% purity metal supplied by M/s. Light & Co., U.K. The temperature of the furnace was recorded by Pt-Pt 10% Rh thermocouple.

The experimental set up is shown in Fig.IV.1. The crystals were grown in elbow-shaped pyrex tubes

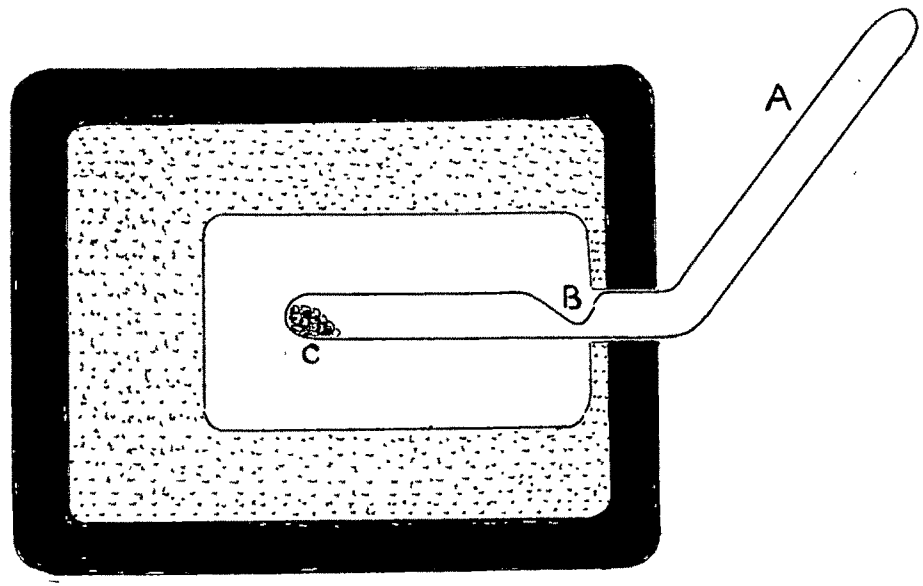

Fig. IV-1

having a diameter of 2 cm,wall-thickness of 1.5 mm and length of the arm _ 10 cms. The tube was bent at an angle of 120° and had a notch just before the bent towards the sealed end. Tellurium metal was made into bits, cleaned in HCl to remove oxide adhering to the metal, washed in distilled water, rinsed in alcohol and ether and air-dried. The glass tube was cleaned and vacuum baked at a higher temperature before filling it with the metal bits. Then the filled tube was evacuated to a pressure of 10^{-4} mm of mercury and sealed off. The temperature of the furnace was gradually raised to 520°C and kept constant for about 20 hours. It was then cooled gradually to room temperature, by reducing the current. The crystals formed on and near the notch. The tube was then removed carefully and cut at the sealed end with a glass-cutter. Hexagonal crystals got detached from the walls on gently tapping the tube.

Preparation of Tellurium Crystals by Chalmer's Method:

The limitation of the Bridgeman's method, that it can be used only for the low melting point metals, is overcome by the horizontal moving furnace technique given by Chalmers.

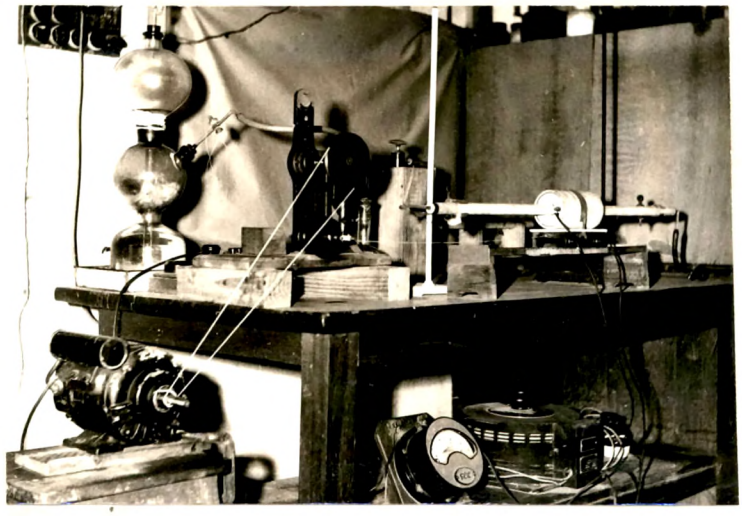

Fig. IV-2

The tellurium single crystals from melt, used for hardness studies in the present work were grown by the horizontal moving furnace technique given by Chalmers[5]. The metal was kept in a graphite boat which was pointed at one end. Because of the pointed tip, only a few seed crystals are formed in the initial stages of freezing and as the solidification proceeds, the fastest growing seed, usually, outstrips the others and a single crystal is eventually produced. The graphite boat was kept at the centre of a silica tube, 30" long and 1" in diameter. The trolley furnace which was constructed according to the standard procedure was kept on the boat containing the charge for a time sufficient enough to melt the charge. The furnace was then slowly withdrawn from the charge. The experimental set up is shown in Fig.IV.2.

The furnace is indicated by A. The stabilized power fed to the furnace was controlled by variac B. The horizontal movement of the furnace was facilitated by a gear mechanism coupled with an electric motor. A wide range of rates of motion of the furnace could be obtained by this arrangement. During the growth of the crystal, a continuous flow of hydrogen was

maintained over the charge. The gas was produced in a Kipp's apparatus and was subsequently passed through a column of water to remove the HCl vapours and through a tower containing calcium chloride to absorb the moisture. The gas was then passed over hot copper filings kept in a separate furnace, denoted by C to remove the traces of oxygen.

Preparation of Zn-Sb crystals from vapour:

These crystals were grown by sublimation of Zn-Sb alloys. The detailed growth procedure is given in part III.

B. TECHNIQUES FOR OPTICAL STUDIES

Microscopic study of the crystal surfaces were carried out by means of the Vickers Projection Microscope. Light profile microscopy developed by Tolansky was used for measuring pit-depths, while the technique of multiple beam interferometry was employed to examine qualitatively the surfaces of the as-grown crystals. The indentation studies were done in the micro-hardness tester attached to the Vickers projection microscope. These techniques are well-established and their applications are

discussed at length in literature[2,3]. Hence it is not dealt with here.

C. X-RAY TECHNIQUES

Laue back-reflection photographs and single crystal rotation photographs of single crystals of tellurium were taken, using a Phillips No.1009 type X-ray generator. The unit was operated at 35 KV and 20 mA using a tungsten target for Laue technique and a copper target for single crystal rotation technique.

REFERENCES

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5. Chalmers, B. (1953) Canad. J. Phys., 31, 132.