CHAPTER IV

EXPERIMENTAL TECHNIQUES

Various techniques used in the investigations presented in this thesis are described in brief in this chapter. The Bridgman's method of crystal growth, zone-refining for purification of metals, method of growing the crystals from the vapour phase are all well-known. So are, the powerful techniques of Multiple-beam Interferometry, Phase Contrast Microscopy and Light Profile Microscopy in the optical studies of the crystal surfaces. These techniques are all described and discussed in detail in the relevant papers and reviews and a detailed description seems to be unnecessary. Only brief descriptions are presented; the constructional details, being standard ones, are omitted. The details of the theory are also avoided. The zone-refining technique is discussed in some detail.

A- BRIDGMAN'S METHOD:

Introduced by Bridgman[1] this method is one of the most suited for growing single crystals of low melting metals. Single crystals used in this work reported in this thesis were grown by this method. The metal is sealed in a pyrex glass tube with a capillary end, under vacuum as described by Bridgman. The capsule is lowered down the vertical temperature gradient furnace. Standard procedure[2] was adopted for preparing the furnace, the core of which was made of a

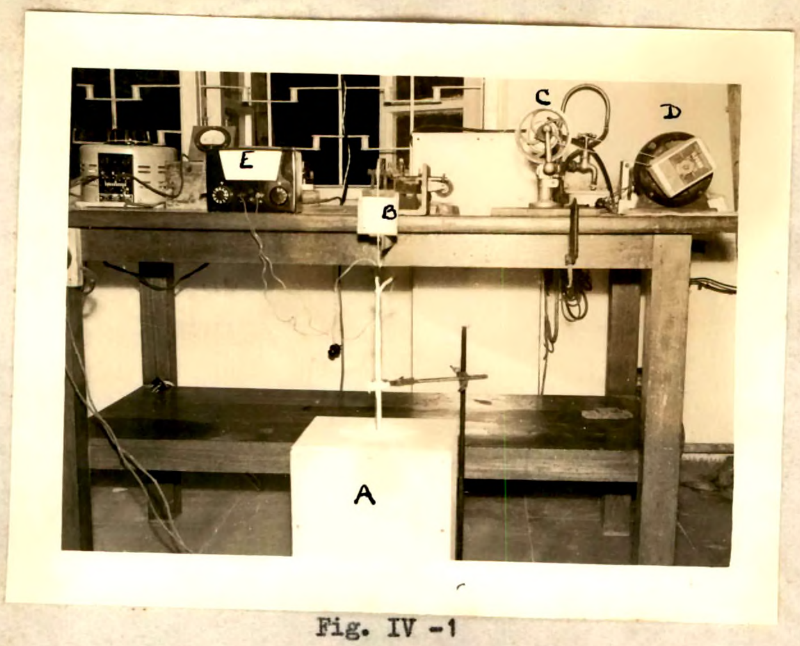

Fig. IV -1

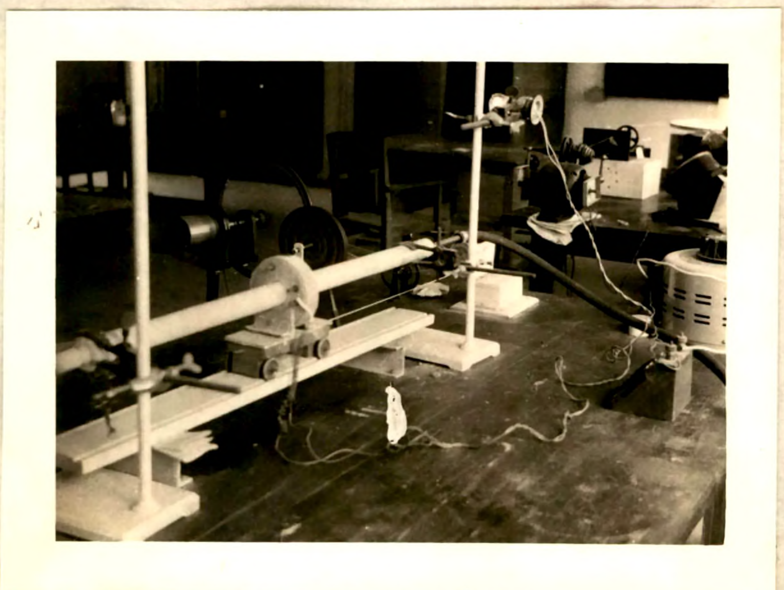

Fig. IV -2

porcelain tube. The arrangement is shown in Fig.IV-1. The gradient furnace A is kept on the floor. The lowering of capsule is facilitated by the gear mechanism B, coupled with pulley system C, which is rotated by the motor D. This arrangement can be used for a wide range of lowering rate of the capsule the lowest being 0.1 cms/hr. The temperature distribution inside the furnace is measured by means of a thermo-couple connected to a milli voltmeter E. The temperature gradient in the furnace could be adjusted between 10°C/cm and 35°C/cm. The gradient can be roughly controlled by completely or partially closing the bottom of the tube. The upper end of the furnace was closed leaving space only for the smooth lowering of the capsule and hence no convection current is set up in the furnace.

B-GROWING OF CRYSTALS FROM VAPOUR PHASE:

This technique was used for growing Antimony crystals. The technique will be discussed in detail in Part III.

C-ANNEALING FURNACE:

A tube furnace was constructed for annealing the specimens in deformation studies. The arrangement provides facilities for annealing specimens in a continuous stream of hydrogen. The gas is generated in a Kipp's Apparatus and subsequently passed thro' a column of water to remove the HCl vapour and a series of absorption towers to absorb the moisture before entering the furnace.

D-ZONE REFINING APPARATUS:

Zone-refining is the most important among the zone-melting techniques in which the impurities are moved along the charge by a series of molten zones all passed along the same direction. Developed about a decade ago, this technique has provided the semi-conductor industry with germanium and silicon of highest purity and metallurgists with a variety of metals at new levels of purity. The various ways in which this technique can be used is described by Pfan[4,5].

Basically, the method consists of moving a molten zone or a series of zones along a long charge of metal. The impurities which lower the freezing point of the metal will travel along the zone and accumulate at the end of the charge. Impurities which raise the melting point travel opposite to the zones and accumulate towards the beginning of the charge. The distribution coefficient k, defined as the ratio of solute concentration in the freezing solid to that in the liquid bulk determine the effectiveness of the process and zone-refining fails to give the desired results when k values are close to unity. A slow rate of

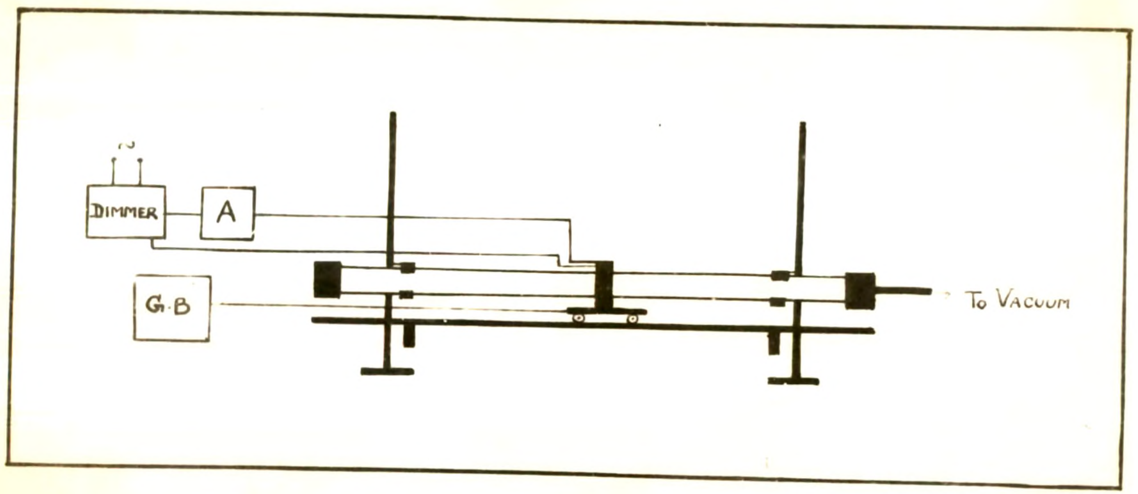

Fig IV-3

motion and a continuous stirring is required to avoid the building up of a concentration gradient in the zone.

The equipment constructed for the purpose in this laboratory consists a long quartz tube (75 cms in length) of 3 cm. diameter, over which a ring furnace mounted on a trolley moves. The motion is effected by a series of pulleys and gear mechanism run by 1/2 hp motor. The charge is placed inside the tube either in an open boat or in a vacuum sealed pyrex glass tube. The refining is done in an atmosphere of argon or nitrogen depending on the nature of the substance being refined. The furnace design is based on the charge length to provide a charge to zone ratio of about 10. The particular zone has about 1 cm. width and gives a sharp variation of temperature on either sides of the molten zone. Convection by gas stream and vibrations conveyed through the gear mechanism are the only means of stirring available on this apparatus. A photograph of the unit is presented in Fig.IV-2 with the line diagrams in Fig.IV-3.

Purification of zinc was done in this unit, using metal of the A.R. quality in a sealed pyrex glass tube about 1 cm in diameter and 20 cm long. That the refining was effective could be proved by the subsequent studies of the crystals grown using the refined metal. The unit has been used for refining of bismuth in open boats in an atmosphere of argon or nitrogen using about 30 passes.

Good quality single crystals could be grown without seed at the last pass.

E-DEFORMATION TECHNIQUES:

Deformation studies reported in this thesis comprise of elongation under tension, bending by application of load on a long crystal and indentation. The indentation is done in the micro-hardness tester attached to the Vicker's projection microscope. Other devices are described in the appropriate sections.

F-OPTICAL TECHNIQUES:

The Vickers projection microscope was used for the microscopic studies of the surface. The Crook and Thomson phase-contrast unit was attached to this instrument for phase-contrast observations. The techniques of multiple beam interferometry was used frequently to measure the step height along cleavage lines, surface distortion associated with twinning, distortion of bend planes etc. The techniques are well-established and their applications are frequently discussed in literature[6,7] and will not be dealt in detail here. The optical flats used for the studies were silvered in an Edward's coating unit. Light profile microscopy, developed by Tolansky has been used for the measurement of step heights etc. The profile insert attached to the microscope was used for the purpose and it consists of fine lines drawn on glass plate, the image of which is projected on the image of the surface.

R E F E R E N C E S

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