ABSTRACT

This thesis entitled "Optical Studies on Crystal Surfaces - Antimony (Growth and Etch Phenomena)" is a systematic study on the growth of antimony single crystals from the melt and vapour and the study of the dislocations by the methods of chemical and thermal etching. The thesis is divided into two parts.

Part I consists of four chapters. In chapter 1 is given a general introduction to the subject of crystal growth and etching and the reason for selecting antimony for the work. In chapter 2 is given a brief review on crystal growth which, though not quite exhaustive, includes the valuable information necessary for the subject. The results of the various authors on different metals, non-metals and semiconductors have been summarised, stressing the results on growth from the melt and vapour phase with which the present work is concerned. A review of the etch phenomenon in metals is presented in chapter 3. The results on etching of LiF are also given to keep up the continuity of the discussion. In chapter 4 the experimental techniques used to grow the crystals from the melt and vapour and the apparatus used to study the etch phenomenon are described. A brief description of the standard techniques used to study the topography has been presented.

Part II has been divided into four chapters in which the work done on antimony is discussed at length. The studies on the effect of the growth rate and temperature gradient on the preferred orientation in crystals grown from the melt by Chalmers' method under externally imposed temperature gradients are presented in chapter 5. It is observed that antimony exhibits a preferred orientation. For low velocities of growth it is observed that a linear relation exists for the orientation of the (111) planes with respect to the crystal axis. Preferred orientation could not be observed with large rates of growth. There is no dependence of the orientation on the temperature gradient and it seems that the anisotropy of thermal conductivity is the main factor influencing the orientation. This is the first work of its type on antimony.

Details of the studies on the chemical and thermal etching of antimony crystals are given in chapters 6 and 7. A systematic study of the relative merits of various etchants has been made and it is found that the purity of the metal and reagent and composition of the etchant are very sensitive parameters. No etch spirals have been observed. The etchants used could reveal dislocations on the (111) plane only. Pile-ups have been observed which

compare well with the results on copper and support the view that the pits are formed at the sites of dislocations. In some cases one-to-one correspondence between etch pits has been observed on cleavage counterparts. It is found that thermal etching can also be used for observing the dislocations by proper choice of temperature and time. The pits observed at low temperatures could not be due to dislocations. At high temperatures of the order of 380°C, it is observed that well defined pits are formed which increase in size with the time of etching, the density of pits remaining unaltered. No rotation of the pits with change of temperature has been observed in the range studied. In this case also, one-to-one correspondence on counterparts has been observed. There is a variation of pit density in the cases of chemical and thermal etching.

In chapter 8 is given a summary of the studies on the growth of antimony crystals from the vapour. Triangular and hexagonal facets have been observed. Triangular facets show oriented growth patterns consisting of triangles oriented opposite to that of the parent facet. It is suggested that they are formed at the sites of dislocations. A spiral with a large step height has been observed in the case of antimony for the first time and this has been attributed to the

interaction of a group of dislocations. Terraced and pyramidal facets have also been observed. The etching behaviour of melt grown and vapour grown crystals is the same. The general conclusions which have been arrived at are presented at the end of the thesis. Wherever the observations of others have been utilized, they are acknowledged as "references".