CHAPTER 10

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CONCLUSIONS AND FUTURE PLAN OF WORK

The conclusions drawn from the experimental observations on growth and characterisation are :

1) Quantity of acid in gel has a dominating effect on the morphology of d-AHT crystals, whereas gel pH and acid pH do not show any significant effect.

2) For a wet and fungus free gel, the gel age does not noticeably affect crystal growth.

3) Crystal habit is independent of the feed solution concentration. However, for the penetration and growth of crystals, the minimum concentration of feed solution should be 1.5 M.

4) Irrespective of the concentration of tartaric acid, the setting time for a gel for a particular quantity of acid in gel is fixed.

5) Needle shaped crystals are obtained at 20% quantity of acid in gel, irrespective of the molar concentration of the acid and feed solution. Similarly, orthorhombic disphenoidal for 30% and 40% tartaric acid in gel, and sphenoidal crystals for 50% and 60% tartaric acid in gel.

6) For 7.5% of tartaric acid, in gel and below, the setting time for gel is zero - a precipitate is formed instantaneously.

7) Gelation time varies in inverse proportion with gel pH, at a given temperature.

8) For a particular quantity of tartaric acid in gel, the same morphology is seen at the interface, central and bottom parts of the test tube.

9) Bigger crystals were obtained in the specially modified U-tube apparatus, but there were gel inclusions in all of them.

The study of variation of diagonal length with applied load, variation of hardness number with applied load, and orientation of the longer diagonal of the Knoop indenter on as-grown and cleavage faces of d-AHT concludes that:

1) the graphical analysis of variation of the diagonal length of indentation mark with applied load suggests that there are two regions of applied load, namely low load region and high load region, where the behaviour of d-AHT appears to be different.

2) Consideration of the values of intercepts and resistance`pressure shows clearly that application of modified kicks law is highly limited.

3) The indentation does produce plastic deformation (hence workhardening) and cold working along with some elastic recovery; however, the present analysis is insufficient to explain the physics of static indentation hardness.

4) The values of intercepts, slopes and resistance/pressure are indicative of the anisotropic character of the crystal. However, the present investigation is incapable of finding their individual effects on the anisotropic character.

5) The kink observed in the straight line graphs of log P Vs log d, is a point of inflexion in the curvilinear plot of log P Vs log d or P Vs d.

6) The kink is connected with d_o, the plastic deformation for unknown load, P_o.

7) For an applied load, P, the deformation $(D-d_o)$ consists of elastic and plastic deformation which cannot be separated.

8) The treatment based on empirical laws is insufficient to unfold the actual mechanism operating in a material under hardness test. However, the expression used for the best-fit of the curve P Vs d, appears to be more reliable than empirical laws.

9) Hardness varies with load. It increases steeply initially with load for all orientations and for all the different faces, then the increase is gradual and attains almost a constant value for all higher applied loads. This behaviour reflects the varied reactions of the surfaces to the applied loads.

10) Irrespective of the indenter geometry, Meyer's law/Kick's law, modified Kick's law and hardness formula cannot be experimentally correlated with one another for synthetic d-AHT single crystals.

11) Hardness anisotropic coefficients are obtained from the graphical analysis of the observed data.

12) The analysis is extended to the observations reported in the literature for different crystals and the hardness anisotropic coefficients for these crystals are determined.

The study of controlled chemical dissolution of as-grown and cleavage faces of d-AHT yielded the following results:

1) a) for as-grown a-face aqueous solution of sodium hydroxide (0.5 N) is the best etchant for revealing characteristic etch features and

b) for a cleavage face as well as as-grown z-face, the combination of trichloroacetic acid solution and glacial acetic acid produces etch pits which retain their geometrically well-defined shaped on prolonged etching.

2) The above etchants produce etch-pits which are at the sites of dislocations ending on the surface. The strenghs of inclinations of dislocation lines are different, not only on different faces, but also on the same face. Hence point-bottomed etch-pits which are formed at preferential points during the initial stage of etching becomes flat-bottomed when the energy is reduced, indicating removal of dislocations from these points.

3) The dislocation density on a cleavage face is $7x10^{4}$ /cm2.

4) With acidic etchants, the pit shape on different faces namely, a- face, cleavage face and z-face, changes with concentration of acid, whereas the pits produced by alkaline etchants on different faces retain their shape irrespective of the etchant concentration.

5) Glacial acetic acid acts differently on different faces-heaviest on a-face and poorest on z-face indicating that z-face is the hardest among all faces. This conclusion supports the hardness studies of d-AHT faces (cf.Chapter 7).

The gel technique is indeed a very good technique to grow crystals of different materials and study their habits. However, this technique is not fully explored to grow industrially important single crystals such as semiconductors, ferroelectric crystals, piezoelectric crystals, laser crystals etc.

It is clear from the work on hardness presented in this thesis and the work carried out by earlier workers in this laboratory, that the empirical laws (Meyer's law, Kick's law, Modified Kick's law) are not in a position to reveal a detailed mechanism which produces 'hardness'. It is, therefore, proposed to develop a model theory of hardness based on the production of dislocations by indentation and their interactions with the dislocations in the grown crystal faces.

Quantitative study of etch patterns on d-AHT crystal faces by sophisticated high resolution techniques such as Transmission and Scanning Electron microscopy, electron probe analysis, is proposed to be undertaken.

RESEARCH PAPERS UNDER PREPARATION

1. Study of the quality of Ammonium Hydrogen d-tartarate single crystals grown in a silica gel medium by using different feed solutions.

2. Application of modified Kick's law to weak Piezoelectric gel-grown synthetic single crystals of Ammonium Hydrogen d-tartarate.

3. Mathematical analysis of variation of applied load with diagonal of knoop indentation mark.

4. Hardness anisotropy on cleavage faces of synthetic single crystals of Ammonium Hydrogen d-tartarate.

5. Directional hardness study on natural prism faces of synthetic crystals of Ammonium Hydrogen d-tartarate.

6. Anisotropic coefficients of natural sphenoidal faces of single crystals of Ammonium Hydrogen d-tartarate.

7. Chemical etching of gel-grown Ammonium Hydrogen d-tartarate single crystals.