

C O N C L U S I O N S

The present work on growth, dissolution and microhardness of synthetic single crystals of ammonium-hydrogen-d-tartrate reported useful and valuable information on some of the important and interesting aspects of growth, chemical dissolution and microhardness of non-centrosymmetric crystalline materials. The main findings of the present work are as follows :

- (i) Gel method has been successfully used to grow single crystals of d-AHT. These crystals exhibits viz. Needle shaped, orthorhombic disphenoidal and sphenoidal ^{habits} crystals. The optimum conditions for the growth of ~~the~~ these three habits are as under :

(a) For needle-shaped crystals

- (i) gel pH ≥ 3
- (ii) quantity of TA solution should be 20% of the total solution (SMS + TA).
- (iii) concentration of FS should be $\geq 2M$

(b) For orthorhombic disphenoidal crystals

- (i) $2 \leq \text{gel pH} \leq 3$
- (ii) quantity of TA solution in the total solution (SMS + TA) should be between 30% and 40%.

- (iii) concentration of FS should be between 2M and 3M.

(c) For sphenoidal crystals

- (i) gel pH \leq 2
- (ii) quantity of TA solution \geq 40% of the total solution (SMS + TA)
- (iii) concentration of FS should be between 2.5M and 3.5M.

These conditions are valid when

- (1) Specific gravity of SMS is 1.04
- (2) ambient temperature is 35°C.
- (3) Size of the glass test tubes (3.6 x 20 cm)

- (ii) The quantitative study of various parameters on nucleation and growth of d-AHT crystals has clearly shown that

- (a) Quantity of acid in gel has a dominating effect on the morphology of d-AHT crystals.
- (b) For a wet and fungusfree gel, the gel age does not noticeably affect crystal growth.
- (c) While considering the effect of concentration of FS and of gel pH, on the number of grown crystals per unit volume of gel, the

phenomenon of syneresis has to be carefully considered for obtaining useful information about FS concentration and gel pH.

- (iii) The characterization of d-AHT crystals was made by employing following well known techniques :
 - (a) Micro-chemical analysis
 - (b) X-ray powder method developed by Hesse-Lipson for determining lattice parameters of an orthorhombic crystal.
 - (c) Stereographic projection of d-AHT
 - (d) Density determination.
 - (e) Optical activity of d-AHT aqueous solution.
- (iv) Controlled chemical dissolution was employed to study crystal perfection and growth features on natural and cleavage faces of d-AHT.
 - (a) Water, unsaturated solution of d-AHT in water, formic acid and varying amounts of trichloroacetic acid dissolved in fixed quantity of glacial acetic acid preferentially act on a d-AHT surface.
 - (b) It was qualitatively established that etch pits produced by formic acid and a proper

mixture of crystalline trichloroacetic acid in glacial acetic acid were at the sites of dislocations ending on an observational plane. The average density of dislocations was $5 \times 10^5 \text{ cm}^{-2}$.

- (c) The shape cycle of etch pits on a d-AHT cleavage surface was observed by progressively increasing the quantity of trichloroacetic acid in a fixed quantity of glacial acetic acid and could be explained on the basis of anisotropic etch rates and eccentricity of etch pits.
- (d) The natural faces of d-AHT crystals namely sphenoidal and prism faces, exhibit layers and striations, which represent a mixture of growth and etch patterns. It is difficult to identify them separately.
- (e) From the geometry of striations on prism faces and layers on sphenoidal faces, a model representing growth of orthorhombic disphenoidal crystal was suggested. The model was formed from a basic unit

consisting of a needle with cross-sectional edges parallel to the sphenoidal edges.

The hardness studies were carried out in a phenomenological manner by studying the empirical formulae based on Meyer's law and the formulae for vickers hardness number (H_v) and Knoop hardness number (H_k).

- (v) The study of variation of diagonal length of indentation mark with applied load has shown that :
- (a) The graph of $\log P$ versus $\log d$ consists of two clearly recognisable straight lines having different slopes and intercepts on the axes.
 - (b) The indenter load corresponding to kink representing a transition from one straight line to another depends upon quenching temperature.
 - (c) The slope of first part corresponding to low load region (LLR) of the graph is greater than that of the second part. The intercept made by the first line has less

value than that made by second line.

- (d) The 'a' values obtained by using vickers or knoop indenter clearly indicate that of the three faces, namely rhombic prism faces $\{110\}$, sphenoidal faces $\{111\}$ and pinacoidal (cleavage) planes $\{010\}$. The sphenoidal faces are the hardest, whereas the pinacoids are the weakest faces of d-AHT.
 - (e) The defect structures operate differently in low load and high load regions corresponding to two parts of the plot of $\log P$ versus $\log d$.
 - (f) Anisotropic character is revealed by different values of diagonal lengths of indentation mark along and normal to direction $[001]$ for the same applied load.
- (vi) The hardness of natural and cleavage faces of d-AHT was carried out by using vickers hardness indenter and knoop hardness indenter at room temperature. The quench hardness studies were made for the cleavages faces. The chief

conclusions are as follows :

- (a) The study of hardness of the cleaved specimens at different quenching temperatures indicates that the plot between hardness and load can be qualitatively divided into three parts for both indenters. They are respectively low load region, corresponding to linear part, intermediate load region corresponding to nonlinear part and high load region, corresponding to linear portion of the graph.
- (b) Relation between hardness number and quenching temperature is derived

$$\bar{H}_Q^K = \text{constant},$$

where the exponent K has a value less than unity. The sign of K decides the nature of the material. The formula is valid for vickers hardness number (H_{va}) and knoop hardness number (H_k) and the value of k is - 0.5, the negative sign indicates increase of hardness number with quenching temperature.

(c) Vickers hardness number has higher value than knoop hardness number at any quenching temperature in the high load region.

(d) The sphenoidal faces are the hardest amongs all the faces.

There are a large number of non-centro-symmetric crystals of industrial importance e.g. piezoelectric, pyroelectric and ferroelectric crystals. From a survey of literature on gel-grown crystals, it is clear that the gel technique was successfully used to grow a few industrially important noncentro-symmetric crystals. This is indeed a promising field for crystal growers. It is desirable to explore it in depth.