

CHAPTER 14

REVIEW OF THE PRESENT WORK AND THE SCOPE FOR
FUTURE WORK

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14.1 Introduction

The studies on growth, characterization and properties of RHT single crystals are systematically and elaborately carried out in the preceding chapters (5 to 13) of this thesis. The highlights of the entire present work can be summed up briefly and bright scope for further research work can be given as follows to add the existing knowledge of the materials and their properties in general and crystal growth in gel in particular.

14.2 Crystal Growth and Characterization

Gel technique yields growth of single crystals of RHT under a controlled reaction between rubidium chloride and tartaric acid. Gel is set with 1.0 M tartaric acid and rubidium chloride of varying strength is used as supernatant solution. The low concentration of rubidium chloride solution yields good quality transparent RHT crystals while higher concentration results in dendritic and hollow crystals. Gel ageing considerably reduces the number of nucleation site and gel density 1.04 gm cm^{-3} is

found most suitable for the growth of good quality crystals.

From the study of the effect of gel parameters on nucleation and growth kinetics of RHT crystals showed that these crystals grow by one-dimensional diffusion process. The effect of concentration and replenishment programme showed that the size and quality of these crystals can be improved by these methods. The period of crystallization is considerably reduced by applying an externally uniform electric field, the quality of the crystals is unaffected. Growth at elevated temperature does not favour these crystals.

Various methods such as chemical analysis, EDAX, X-ray diffraction, DSC, TGA, IR, magnetic susceptibility and density measurements are used for characterizing the grown crystals. RHT crystals belong to orthorhombic system with space group $P2_1 2_1 2_1$. Morphology of grown crystals are studied in order to facilitate the further study of directional properties. DSC and TGA confirm that the

this crystal decomposes before melting point. Magnetic susceptibility measurements revealed that it is a weak paramagnetic material due to the presence of rubidium in it.

14.3 Electrical and Dielectric Properties

The temperature dependence of dc electrical conductivity in the form of the pellets and single crystals are investigated in the temperature range 300 to 470 K. The samples show abrupt changes in conductivity and this is accounted as due to the mode of conduction (extrinsic and intrinsic) for RHT. The activation energies for the processes are calculated for both pellet and single crystals from slopes of the plots of $\ln \sigma$ versus $1/T$.

From the nature of variation of dielectric constant with frequency and temperature, it is deduced that different polarizations contribute to the dielectric constant and losses. The larger values of dielectric constant at low frequencies showed the lower concentration of impurities and defects in this crystal. Different pelletizing

pressures has no effect on dielectric constant and losses.

14.4 Hardness, Etching and Microstructures

For determining the Young's modulus and the hardness of RHT crystals Instron compression testing and Vicker's diamond indenter units are used respectively. RHT crystal is highly brittle because even at low indentation loads fracture occurs at stress concentration points. Hardness (H_V) is found to be dependent on load in lower range of applied load and found to be independent of the load at the higher applied loads. Quenching increases the hardness whereas annealing decreases the hardness of these crystals. The crystals grown at different gel column are found to have different H_V . The value of Young's modulus of RHT crystal is found to be 1.289×10^9 dyne cm^{-2} .

RHT possesses a perfect (010) cleavage planes. The perfection of this crystals are studied by chemical etching technique using formic acid solutions as etchants and the dislocation

densities are evaluated. One-to-one correspondence of etch pits on matched cleavages and successive etching experiments confirm that the etch pits nucleate at dislocation sites. Low angle grain boundaries are also observed on (010) face.

The normal and lateral rate of dissolution of dislocations are found to be independent of duration of etching, indicates a weak adsorption of the reaction product of etching of the dissolution ledges of the parent crystal. The temperature dependence of the dissolution rates are of activation character and the activation energies of dissolution along different directions are different. No change in orientation of etch pits are observed with change in temperature of the etchant.

Microtopographical studies made on as-grown (010) faces of RHT crystals revealed that the crystals grow by layer growth mechanism.

14.5 Scope for Future Research Work

The development of newer and better

methods of nucleation control remains a field open to research. A greater deal of work remains to be done to document and to explain the detailed nature of the gel structure which will be displaced by the growing crystals as in the case of RHT or incorporation of gel into the new solid by crystal growth in the gel interstices as in the case of calcite. Many important crystals, viz. ZnS, CdS, PbS, ZnTe, PbSe, CdSe, $MgHPO_4$, $CaHPO_4$, $BaHPO_4$, etc. can be grown in various types of gels, with various acid set gels and to grow various shapes of highly perfect single crystals. It is worthwhile to make an attempt of comparative studies on the growth of crystals like SnI_2 , AgI, PbI_2 , HgI_2 , $PbDPO_4$, $PbHASO_4$, $PbDAsO_4$, etc. by reaction method and complex dilution method. The growth of various metal crystals such as Ag, Sn, Cu, Sm, etc are to be tried by using chemical reducing agent. It is also possible to produce the naturally occurring habits of the crystals by controlling the experimental parameters in the laboratory and hence gel growth can be used as a tool in understanding the geological conditions.

Out of a large number of gel grown single crystals, properties of only a few are studied. Comparative studies of gel grown single crystals with those grown by other methods are worth attempting in order to probe into and take advantage of the characteristics and peculiarities of gel grown crystals. The further development of the gel method will certainly depend on the extent to which its mechanism can be understood.

With challenging and more effective method available, lot of work on growth of both doped and mixed crystals can be done. Mixed compounds such as $\text{CdI}_2\text{-SnI}_2$, $\text{PbI}_2\text{-SnI}_2$ etc. can be crystallized from gel medium. Mixed crystals can be grown by using mixed reagents and crystals can be doped with trace amount of additives either in gel itself or in the top reagent. An elaborate study, made in this direction is likely to prove helpful in understanding the mechanism of mixed crystal growth. Crystal formation and growth characteristics substantially change by absence or presence of light or change in environment.

There is a sufficient scope to study structural transformations due to phase transitions and dehydration. Further work on mechanical and electrical properties using wider range of operating frequencies, temperatures, dc-biasing, different atmospheres, different pressures in the vicinity of paraelectric to ferroelectric transition temperature, studies of ferroelectric hysteresis, ferroelectric domains and piezoelectric behaviour are of great use in devising solid state devices. The perfection properties by using X-ray topography make a fascinating field for further research work.

A Word :

Research work on these aspects should definitely add something to be existing knowledge of the most fascinating branch of Materials Science, namely crystal growth and its characterization