S.P.29. GEORGE PETER–Growth and Characterization of Diammonium Hydrogen Citrate and Citric Acid Monohydrate Single Crystals–1987–Dr. Joy George.

The thesis presents a study of the growth of Diammonium Hydrogen Citrate (DAHC) and Citric acid (CA) monohydrate single crystals from solution and their defect structures. The result of the microindentation and thermal analysis are presented. Dielectric, fractrographic, IR and UV studies of DAHC crystals are also reported.

Large and perfect crystals of size upto (60X27X5) mm^3 were grown by slow evaporation in a constant temperature bath. The faces of the grown crystals were identified by angular measurements using goniometer.

Dislocation etch studies have been carried out to asses the perfection of DAHC and CA crystals. Dislocations in DAHC crystals were delienated by propionic acid which was found to be a reliable dislocation etchant for the (OO1) face. Dislocation loops, helices, clusters impurity centers and slip lines are observed with this etchant. The etch pit density is found to be of the order of $1.2X10^2/cm^2$. Other etchants have been tried and formic acid, acetic acid and methyl alchohol were also found to be capable of bringing out dislocations on

the (001) face. The shape of the pit is found to depend on the type of the etchant used. The etch pit formation in DAHC crystals is explained on the basis of the thermodynamic theories of etching. For CA crystals also propionic acid is found to be a good dislocation etchant.

Microindentation analysis of DAHC and CA crystals was done using Vickers pyramidal indenter to study the hardness, toughness, brittleness and the nature of cracks. The hardness of DAHC was found to be 0.68 GPa. The hardness of DAHC was found to be nature of cracks. The hardness with load was analysed upto a load of 12.5 gms. The fracture toughness of DAHC is 0.17 MPa $m^{1/2}$ Loads upto 25 gms were used for the toughness evaluation. The toughness value is found to increase with increase in load in a regular manner. Brittleness of DAHC is found to be 4.04$m^{-1/2}$ Surface removal technique has shown that the threshold crack is radial and originate from the surface. From the indentation studies, hardness of CA was found to be 0.588 GPA. The variation of hardness with load is found to have a minimum around 7.5 gms. The toughness is 0.132 MPa $m^{1/2}$ and the brittleness is 4.48 $nm^{-1/2}$.

The (110) cleavage surface of DAHC exhibit typical examples of hackle structure, Wallner lines and many other interesting crystallographic markings. They are presented and discussed.

Dielectric studies were made on DAHC crystal along the three mutually perpendicular crystals axes in the region of temperature 133 K and frequency 200 to 20,000 Hz. The dielectric permittivity and the loss tangent measurements were made in a low temperature dielectric cell with heating arrangement.

The IR apectrum of DAHC was recorded in the region 4000 - 200 cm^{-1}. Absorption bands are observed at frequencies 3400, 3200, 3060, 2860, 1690, 1600, 1330, 1270, 1230 and 1090 cm^{-1} These bands are tentatively assigned to the functional groups in DAHC.

The UV spectrum was obtained in a wavelength range 195 to 500 nm. The only absorption band observed at 210 nm is discussed.

Differential scanning calorimeter was used for thermal analysis of DAHC and CA crastals. The measurements were made from room temperature upto the melting of these crystals. The calorimeter traces obtained for DAHC and CA are presented and discussed.