PART - IV

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CONCLUSIONS AND FUTURE PLAN OF WORK

CHAPTER - XII

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From the considerations of various crystal growing techniques, available in the literature, modified Kyropoulos technique was found to be the most suitable, convenient and successful technique for growing large single crystal of sodium nitrate. It is shown that solution-grown crystals are of inferior quality to the melt grown crystals of sodium nitrate. The vacancies and dislocations produced in the melt-grown crystals were removed, to a greater extent, by annealing. The hardness studies were carried out in a phenomenological manner by studying the empirical formula based on Meyer's law and the formulae for Vickers hardness number (H_v) and Knoop hardness number (H_k). The study of the variation of diagonal length of indentation mark with applied load has shown that,

(i) the graph of log d versus log P consists of two clearly recognisable straight lines having different slopes and intercepts on the axes.

(ii) the indenter load P_k corresponding to kink representing a transition from one straight line to another depends upon quenching temperature,

(iii) the slope of first part corresponding to low load region 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,

(iv) the slopes 'n_1' corresponding to low loads are more succeptible to quenching temperature. In particular for Knoop diamond pyramidal indenter 'n_1' is more susceptible to quenching temperature than for Vickers pyramidal indenter,

(v) the defect structures operate differently in low and high load regions corresponding to two parts of the graph of log d vs. log P.

The variation of microhardness, (represented by H_v or H_k depending upon the nature of the indenter) of thermally treated and untreated cleavage surfaces of synthetic single crystals of sodium nitrate has shown that,

(i) the study of hardness of untreated and thermally treated specimens (quenched from different temperatures to room temperature) indicate that the plot between hardness and load can be qualitatively divided into three portions viz. 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,

(ii) hardness depends upon quenching temperatures. A relation between hardness and quenching temperature in the high load region is given by,

(a) $H T_Q^k$ = Constant where $K = -0.17$ for $NaNO_3$ crystals

(b) $a_2 T_Q^r$ = Constant where $r = -0.07$ for $NaNO_3$ crystals,

(c) $a_2 H^s$ = Constant where $s = -0.75$ for $NaNO_3$ crystals,

(iii) Knoop hardness number has almost the same value as that of Vickers hardness number at any given temperature,

(iv) the mechanism of hardness and electrical conductivity in ionic crystals in general and sodium nitrate crystal in particular are more or less similar.

(v) the ratio of hardness (Vickers or Knoop hardness number) to electrical conductivity of an ionic crystal in general and sodium nitrate crystal in particular is constant at constant temperature, in the high load region.

The study of controlled chemical dissolution of cleavage surfaces of natural crystals of calcite in aqueous solutions of sodium hydroxide of varying concentrations has enabled the author to conclude that,

(i) plane shape of an etch pit produced by different concentrations of NaOH solutions in cleavage face of calcite is independent of concentrations. The pits at the highest concentration exhibit sharp beak ; however the shape is not changed,

(ii) etch rates are independent of time,

(iii) dislocation etch pits dimensions (lateral and tengential) attain the maximum value at certain concentration C_p (17 M) of the etchant,

(iv) the C_p value of the etchant is independent of temperature of etching,

(v) beyond C_p the etch rates decrease with increase of concentration,

(vi) the electrical conductivity of the etchant (aqueous solution of sodium hydroxide) attains a maximum value of etchant concentration which is different from the value of concentration at which etch rates become maximum,

(vii) in the study of etch phenomena, the ratio V_{tm}/V_{sm} should be considered. For all dislocation (alkaline solutions, Lactic and Formic acids) etchants the relation between V_{tm}/V_{sm} and etchant concentration and also between $_m$ and concentration are linear and independent of etching temperature,

(viii) higher viscosity and thin film formation of calcium oxide are responsible for the dissimilar values of etchant concentrations at which V_t, V_s and attain maximum values (cf. 3 and 7),

(ix) the thickness of thin film of calcium oxide formed on a cleavage face is a function of etchant concentration and etching temperature,

(x) for good visibility of etch pits, the ratio E_t/E_s should have values greater than one,

(xi) the chemical dissolution of calcite in sodium hydroxide solution is kinetically controlled.

From the quantitative study of thermal dissolution of calcite cleavages, the following conclusions are drawn,

(i) the thermal etch pits on calcite cleavage surfaces are of rhombic shape in the range of temperature 540^{o} to 605^{o}C,

(ii) the activation energies $E_{t\ th}$ and $E_{s\ th}$ of thermal dissolution are larger than the corresponding activation energies $E_{t\ ch}$ and $E_{s\ ch}$ of chemical dissolution of calcite cleavages,

(iii) thermal etch pits are produced at dislocation free placed such as kinks, steps, impurity contress etc.

The multiple beam interferometric and scanning electron microscopic studies of chemical etch patterns produced on a cleavage surface of natural crystals of calcite by etching it with a 60% glacial acetic acid has shown that,

(i) initial preferential dissolution of ledges along three non-coplanar directions taken place in such a way as to maintain the geometrical shape of an etch pit which exhibits crystallographic symmetry of a cleavage plane of calcite,

(ii) in an etch pattern resembling a growth pattern, the etch ledges (depressions) in a direction normal to cleavage plane occur in multiple of the smallest depression. However the lattice parameter along this direction is not a multiple of this depression produced by etching.

A large amount of research data on Hardness of single crystals of different materials (Barite, alkali halides - sodium chloride, chlorides and bromides of potassium triglycine sulphate, zinc, calcite, sodium nitrate and indium antimonide) is now available in the laboratory. It is therefore planned to develop a model theory of hardness to account for the observed facts (results). Further chemical and thermal dissolution of calcite cleavages is extensively studied by several workers in this laboratory. It is now possible to develop model theory of etch phenomena on calcite cleavages.