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Growth and Dislocation Studies of Ferroelectric Tri-Glycine Potassium Sulphate [TGPS] Crystal

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***Abstract*— Glycine is smallest among amino acid. This paper reports the growth of glycine potassium sulphate crystal from aqueous solution through slow evaporation of water. The reported structure of pure Try-glycine compound reveals the fero-electric properties as a result of zwitterionic characteristic of Tri-glycine molecule and its chemical reactivity with the proper proportion of additive chemical compound. Crystals of glycine potassium sulphate [TGPS] have been grown by slow evaporation technique at room temperature. TGPS crystal could be obtained from non-neutral pH solution in the presence of impurities that suppress the formation of the kinetically favored TGPS crystal. Studies on dislocations and its reactivity in organic crystalline solids play an important role in many solid state phenomenons such as crystal growth, ionic conductivity, electronic conductivity, crystal strength, diffusion properties and plasticity. Etching behavior helps to understand surface characteristics and direction bandings. Dislocations provide the information for ferroelectric for the formation of domains**

Key Word:** A_1 **Growth from Solution,** A_2 **Dislocation Studies,** B_1 **Organic Compound,** B_2 **Ferroelectric GPS Crystal.

1. INTRODUCTION

Detection of dislocations employing topography of growth surface, chemical and thermal etching has been carried out on various organic crystals. These studies are further used for characterization of dislocation besides their detection in spiral etch pits, screw dislocation, mono crystallographic pit, growth and evaporation spirals corresponding to emergent screw dislocation have been observed in organic solids. Such studies have been reported on Anthracene. Tetracene, biphenyl and benzoic acid [1,2] diglycine sulphate [3,4] beta scenic acid [5] TGS [6] GASH [7] trim ethylene trinitro amine [8] P-triphenyl and anthra quinine [9], Naphthalene [10,11] betamethyl naphthalene [12,13] glycine, [14] sucrose [15,16] There are number of organic ferroelectric compounds ,their ferroelectric and electrical measurements have been carried out by a number of investigators. Out of studies including the formation and detection of domain by several investigators [17] .Meleshnia [18], Sawada and Abe [19] have applied the etching technique to study the domain pattern in TGS crystal.

2 EXPERIMENTAL

2.1. SYNTHESIS AND CRYSTAL GROWTH:

Growth of crystal from water solution is technically quite important. This method is extensively used for purifying and obtaining single crystals of organic and inorganic materials. It is well known that sugar and salt are purified by re-crystallization from the solution of these substances to grow crystals from water solution, no elaborate equipment is necessary and hence for a home experiment that is the easiest method to grow single crystals.

Application of various experimental technique employed depending on factors like basic principles involved advantages and limitation of each method. Among that the crystals with different proportion were grown from aqueous solution by slow evaporation technique at room temperature.

2.2. ETCHING TECHNIQUE: -

The etching technique is widely used to reveal the sites of dislocations. It is also used to study the ferroelectric domains. The sample under study is dipped for the known period in a beaker containing the suitable chemical agent (etchant). The crystal samples are then dried with a good quality filter paper (No.44) Care is taken to prevent impurities on the surface since well defined etch pits may not be observed in such case. The etched and dried crystal face is mounted on glass plate using clay and then it is examined under a metallurgical microscope. For thermal etching, the crystal faces

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are placed with their faces up on the crystal faces are placed with their faces up on the glass plate and heated in air atmosphere at constant temperature for a required period of time and observations are taken under the microscope

3. CHARACTERIZATION

3.1. GROWTH FROM WATER SOLUTION: -

The formation of nucleus in the absence of any defect requires a super saturation of about 25-50% Super saturation may be obtained by cooling evaporation or rarely by addition of other materials. For most of solution techniques a two stage growth process is used. i) First by allowing slow evaporation of cooling a saturated solution which will give small crystals. ii) Some of these crystals are selected and used as seeds to grow larger ones.

Thus the crystals of tirglycine potassium sulphate (TGS) crystals were grown from solution by slow evaporation technique. Glycine and potassium sulphate in molar ratio of 1:1 (called (TGPS -I) and 3:1 (TGPS -II) dissolved in water. The solution was kept undisturbed. Fine crystals were grown. The pH of the solution at super-saturation is kept at 6.0. The solution is filtered and transferred to a Petri dish and biker for crystallization. This compound is re-crystallized two times for purity. The good transparent crystals of assorted size obtain within 6 to 7 weeks. This method is extensively used for purifying and obtaining single crystals of organic & inorganic materials. The factor affecting on crystal growth are presence of impurities, changing temperature & the pH of solution to grow good crystals of appreciable size. Then the pure samples of seed crystals are taken for characterizations.

The etched and dried crystal face is mounted on glass plate using sand clay and then it is examined under a metallurgical "METZER" universal trinocular metallurgical research microscope [METZ-780] "METAPHOT" used for the dislocation studies on crystals

3.2. DISLOCATION IN ORGANIC SOLIDS: -

TGS was discovered Matthias. Miller, & Rameika (20) TGS takes the leads now a days in application to all other ferroelectrics. Despite its complex chemical and crystallographic form it has become the object of active experimental research for two regions.

1) Among various ferroelectrics, TGPS is one of them exhibiting a second a second order phase transition and hence to offer possibility for the observation of genuine critical (i.e. fluctuation dominated) phenomenon very close to Tc.

2) Secondary TGPS is an order, disorder ferroelectric it is uniaxial and unlike KDP ferroelectrics has dielectric properties which are not grossly affected by declaration. All these suggest that in spite of its structural complexity the basic ferroelectric character is simple. TGPS has monoclinic symmetry and belongs to the polar point group 2/m above the Curie temperature Tc=49 Deg. C. and below the Curie temperature mirror plane disappeared and the crystal belongs to the polar point group 2 of the monoclinic system. The polar axis belongs to the monoclinic (two fold symmetry) 'b' axis, by using X-ray diffraction, Hoshino et. Al [21] investigated the first detail crystal structure. Refinement in structure and temperature dependence was brought out by subsequent neutron scattering work (Key & Kleinberg (22) with X-ray scattering studies by it. The crystal chemistry of molecule of glycine is prescribed to understand the structure

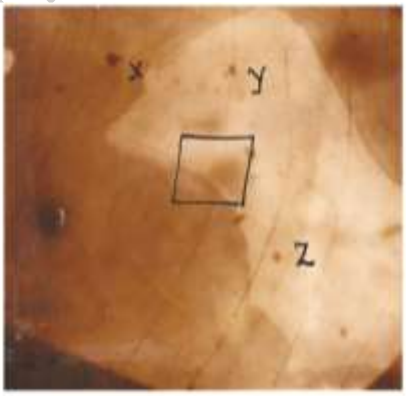

Fig. 1 (a)

Fig. 1 (b)

Figure.1 (a) & (b) Photomicrographs of showing the exact correspondance of cleavage steps on matchant cleavage faces x 70

Vol 11, Issue 4 ,April/2020
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Fig. 2 (a)

Figure 1(a). Etch pattern observed on (010) cleavage face of gps crystal (etched for 15 second) x 70

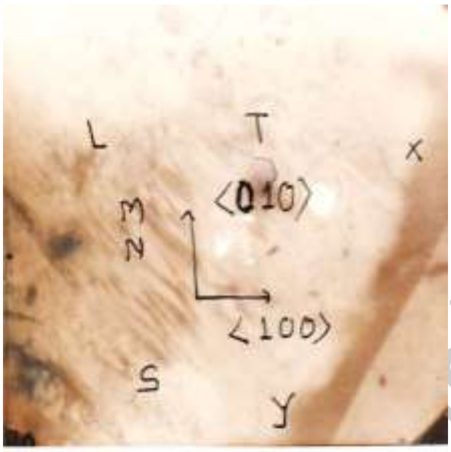

Fig. 2 (b)

Figure 2(b). Succssive stage of etching of the region of figure 2(a) (total etching time 17secind) x.70.

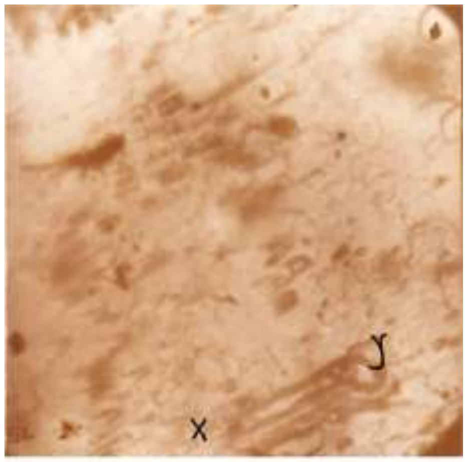

Fig. 3 (a)

Figure 3(a). Etch pattern on poloshed side of cleavage face by the dislocation etchant in 12 sec. Etch pits on matched faces x 70

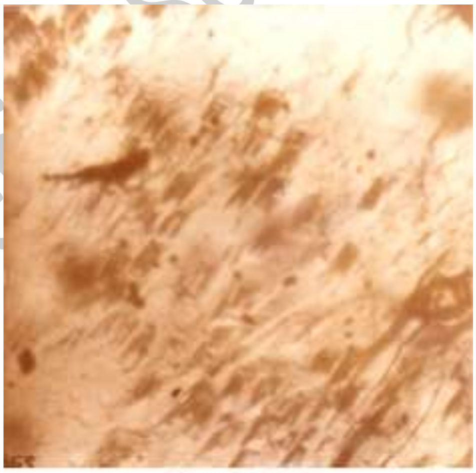

Fig. 3 (b)

Figure 3(b). Etch pattern on its faced etched for 12 sdecond x70.

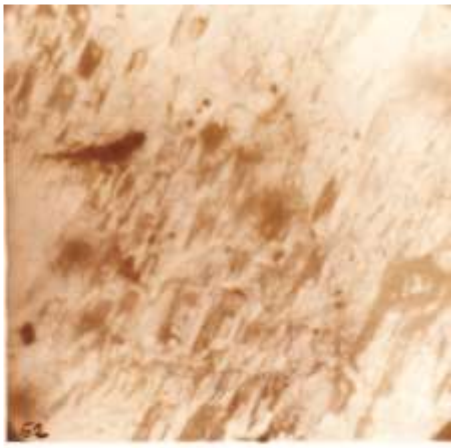

Fig. 3 (c)

Figure 3(c). Etch pattern on bottom face etched for 8 secind x 70.

Fig.4

Figure 4. Pohtomicrograph showing the etch pattern on a cleavage plane produced by etchant (gaax water) x70.

4. RESULT AND DISCUSSION

To study the etch patterns on various faces of the crystal beings with the development of a suitable etchant by trial and error method. Hence, various etching like ethanol, methanol acetic acid (GAA) produced extended elongated pits even on etching for a very short time along with the circular microstructure like feature. To improve the geometry of the pit, the length of elongations was found to be checked by addition of methanol as a poison. It was seen that glacial acetic acid and methanol in volume ratio 3:1 produced well defined etch pits on habit cleavage and fractured faces. During the development of etchant, it is also worth mentioning that when the crystal cleavage was rubbed with water wet cloth very shallow etches. Patterns were revealed as also reported on TGS single crystals. Therefore the etchant GAA+ methanol was used for studies on all face of GPS crystal. Moreover a polishing agent consisting of methyl alcohol and distilled water was used for polishing all the surface of the crystal. Fig. 1 (a) and fig. 1(b) represented matched cleavage faces of GPS crystal. It is observed that cleaved face are quite smooth and show the exact correspondence of cleavage steps, The circular structures of assorted sizes like x, y, z. in this figure are the inclusions observed on (010) cleavage faces of these crystals. Their correspondence and non correspondence has been observed in matched faces. The region between the cleavage lines in quite flat and the cleavage lines have sharp steps. An etch pattern produced by the dislocation etchant for 15 seconds on a (010) cleavage plane is shown in the photomicrograph of figure 2(a). In this figure X, Y, is the cleavage step and ST is a row of pits. Some isolated pits are marked by L.M.N. etc. The structure of etch pits reveals no definite boundary along (110) direction. The same crystal face is further etched for 2 second and the resulting etch pattern of the corresponding region is shown in figure 2(b). It is observed that (1) the isolated pits have increased in size at the same sites, suggesting that cleavage step has moved on prolonged etching and (ii) the dissolution of surface along <110> direction is faster than that along, <100> directions. Figure 3(a) shows etc pattern on the polished cleavage face etched for 12 second in the dislocation etchant, Elongated pits lying along <110> planes all facing along a single crystallographic direction is observed. The length of the pits vary on the surface, Figure 3(b) shows etch pattern on face laying parallel to the <110> cleavage etched for 12 sec. showing again a similar nature of elongated pits. Figure 3(C) shows the bottom face that opposite to the top face of figure 3(b) etched for 8 second. The dislocation of growth layers upon etching is clear from this figure 3(c). All these figure have one property in common i.e. the size of the elongated pits varies and further that all are along <110> family. Still more information regarding the dislocations can be obtained from the structure of etch pits produced by etchant (GAA+WATER) as revealed from figure 4. In this case the density of pits is greater than that seen in fig. 3(a). Along with the point bottomed etch pits. Some flat bottomed terraced and small pits are also observed. Passage of low angle grain boundary through the glide band and its blocking by the glide bend is commonly observed in this crystal.

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