Etch Pits and Dislocation on Cleavage Face of Tri-Glycine Potassium Sulphate [TGPS] Crystal

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Abstract:

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. 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 Tri-Glycine Potassium Sulphate crystal [TGPS] crystal. Moreover a polishing agent consisting of methyl alcohol and distilled water was used for polishing all the surface of Tri-Glycine Potassium Sulphate crystal [TGPS].

Introduction:

Studies on dislocations were carried out by comparing various matched faces on etching in the dislocation etchant foe specific identical times and further observing them on continued etching followed by polishing and retching Generally. Dislocation studies are carried out which accounts for their truncations distinguishing of edge and screw dislocations, Low angle grain boundaries consisting of edge dislocations, twin boundaries, plastic deformation by observing the rosette around the indentation mark on different faces of the same crystal, effect of heating and radiation damages, Out of these, trajectories of dislocations have been carried out by comparing the dislocation etch pits on matched faces and by studying them at successive stage of etching on habit, cleavages and fractured faces of GPS crystal. Studies on low angle grain boundaries in organic crystals reveal the complexity of their growth as observed in succinic acid and glycine crystal, In succinic acids crystals, surprisingly, it has been noted that no low angle grain boundary is formed on habit or cleavage faces. Whereas, it has been pointed out that formation of cellular structure is not possible in glycine crystals. Further, studies on low angle grain boundaries in low transition ferroelectric organic crystals have not been attempted so far, as is clear from the literature. Therefore, so far, as is clear from the literature. Therefore, such studies have been carried out on this crystal, using dislocation etchant. In low transition ferroelectrics, The domains are reveled either by thermal shocks (1) or by mechanical stress.(2).

Dislocations are the most favorable sites to be attacked by the etchant. By studying the shape of etch pits produced at the dislocation sites on different faces of crystal either by using different etchants or by inhibitive poisoning effect, one can predict the dissolution kinetics of the crystal. In fact such studies have been reported by Barber (3) Haribabu et al (4) , Joshi and Ittyachen (5) Haribabu and Bansigir (6) . They have explained the change in shape of etch pits by considering the relative rate if removal of atomic steps in different directions Keith and Gilman (7) have accounted the observed shape of etch pits (Clacite crystals) by considering the involvement of anions of the etchants in the dissolution process. In such studies formation of kinks and thereby the dissolution has been successfully predicated by several investigations (Ghosh and Clark (8), Arizumi and Akasaki 9) Ives and Plows (10) Baronova and Nadgernyi (11) Snagwal (12) have tried to correlate the activation energies of the etchants with the dissolution of crtsyal under observation. Studies on dislocations were carried out by comparing various matched faces on etching in the dislocation etchant foe specific identical times and further observing them on continued etching followed by polishing and retching Generally. Dislocation studies are carried out which accounts for their truncations distinguishing of edge and screw dislocations, Low angle grain boundaries consisting of edge dislocations, twin boundaries, plastic deformation by observing the rosette around the indentation mark on different faces of the same crystal, effect of heating and radiation damages, Out of these, trajectories of dislocations have been carried out by comparing the dislocation etch pits on matched faces and by studying them at successive stage of etching on habit, cleavages and fractured faces of GPS crystal. Studies on low angle grain boundaries in organic crystals reveal the complexity of their growth as observed in succinic acid and glycine (13) crystal, In succinic acids crystals, surprisingly, it has been noted that no low angle grain boundary is formed on habit or cleavage faces. Whereas, it has been pointed out that formation of cellular structure is not possible in glycine crystals. Further, studies on low angle grain (14) boundaries in low transition ferroelectric organic crystals have not been attempted so far, as is clear from the literature. Therefore, so far, as is clear from the literature. Therefore, such studies have been carried out on this crystal, using dislocation etchant. In low transition ferroelectrics, The domains are reveled either by thermal shocks (15) or by mechanical stress. (16).

EXPERIMENTAL:

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 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. Hence, various etching like ethanol, methanol acetic acid (GAA) where tried. The etchant GAA+ methanol was used for studies on all face of Tri-Glycine Potassium Sulphate crystal [TGPS] crystal. Moreover a polishing agent consisting of methyl alcohol and distilled water was used for polishing all the surface of Tri-Glycine Potassium Sulphate crystal [TGPS].

Characterization:

Dislocation in Organic Solids:-

: - TGS was discovered Matthias. Miller, & Rameika (17) 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 [18] investigated the first detail crystal structure. Refinement in structure and temperature dependence was brought out by subsequent neutron scattering work (Key & Kleinberg (19) with X-ray scattering studies by it. The crystal chemistry of molecule of glycine is prescribed to understand the structure

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 fig. 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

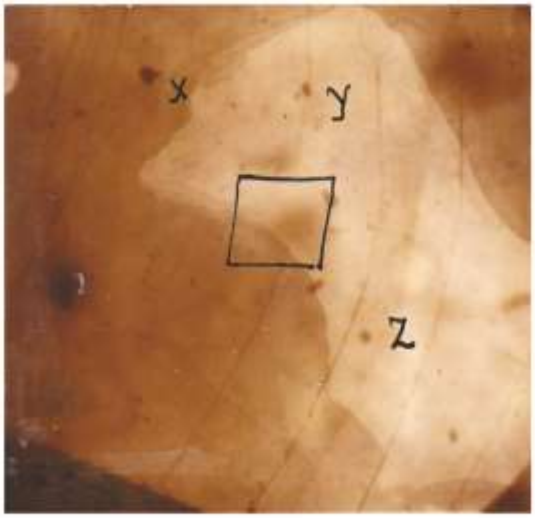

Fig. 1 (a)

Fig. 1 (b)

for two 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 Patel and Tolansky (20) 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 grater 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. Such observations are reported by finkel et.al in KCI (21) crystal.

Fig. 2 (a)

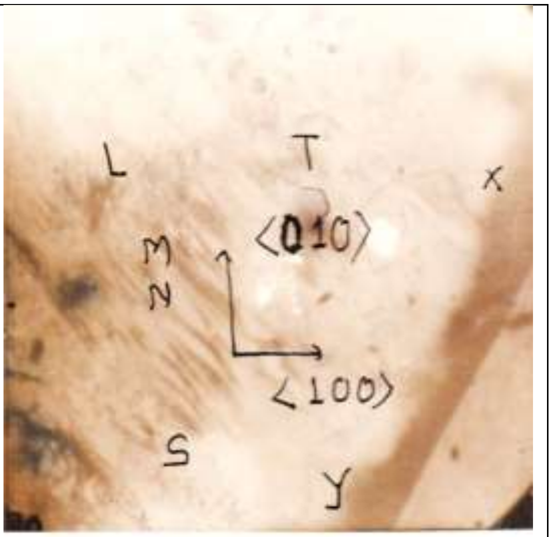

Fig. 2 (b)

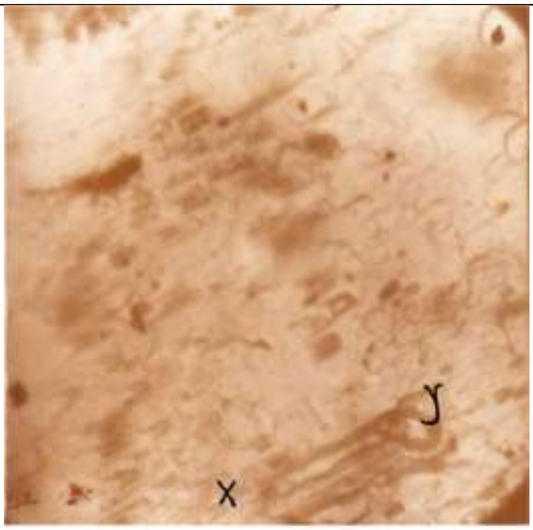

Fig. 3 (a)

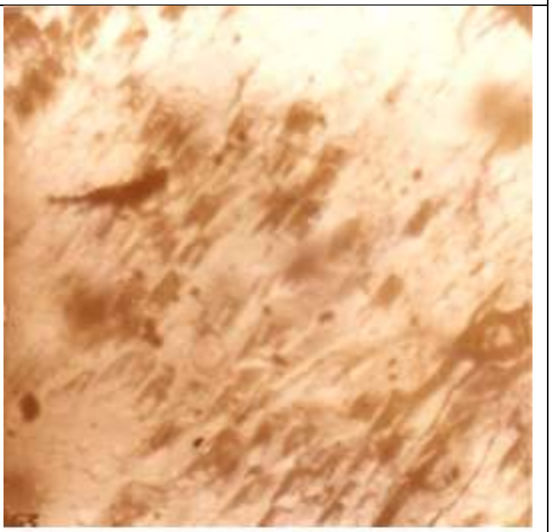

Fig. 3 (b)

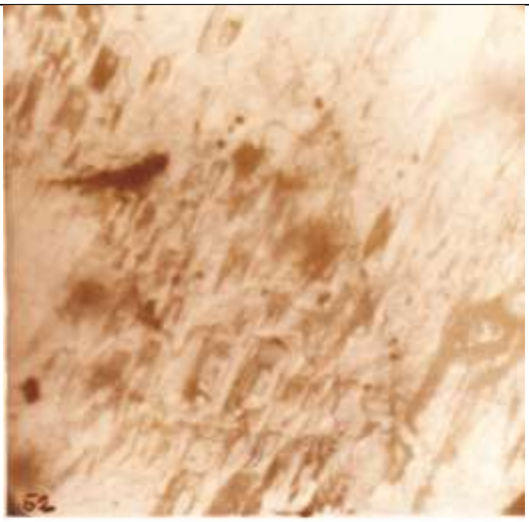

Fig. 3 (c)

Fig.4

Result and Discussion:

From the observation that (1) the etch pits increase in size at the same site by maintaining their geometry on prolonged etching. (ii) the etch pits have one to one correspondence on matched cleavage faces. It is observed that the etch pits grow in size at the same sites maintaining their geometry on its faces. The boat shaped etch pits are also observed. It is noted that the boat shaped etch pits grow in size at the same sites on prolonged etching and become flatter than those observed at the first stage of etching is the photomicrograph of dislocation region. Upper portion is with dislocation region and lower portion is without dislocation region. The structure of pits grow along <110> direction in upper region while in the lower region the circular structure of assorted size like x,y,z, are the inclusions observed on (010) cleavage face of these crystals.

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Caption of Figure:-

FIG.1 (A) AND 1(B) PHOTOMICROGRAPHS OF SHOWING THE EXACT CORRESPONDANCE OF CLEAVAGE STEPS ON MATCHANT CLEAVAGE FACES X 70

FIG.2 (A) ETCH PATTERN OBSERVED ON (010) CLEAVAGE FACE OF GPS CRYSTAL (ETCHED FOR 15 SECOND) X 70

FIG.2(B) SUCCSSIVE STAGE OF ETCHING OF THE REGION OF FIGURE 2(A) (TOTAL ETCHING TIME 17SECIND) X.70.

FIG.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) ETCH PATTERN ON ITS FACED ETCHED FOR 12 SDECOND X70.

FIG.3(C) ETCH PATTERN ON BOTTOM FACE ETCHED FOR 8 SECIND X 70.

FIG. 4 POHTOMICROGRAPH SHOWING THE ETCH PATTERN ON A CLEAVAGE PLANE PRODUCED BY ETCHANT (GAAX WATER) X70.

22) FIG.3 (A) ETCH PATTERN ON POLOSHED SIDE OF CLEAVAGE FACE BY THE DISLOCATION ETCHANT IN 12 SEC. ETCH PITS ON MATCHED FACES X 70
23) FIG.3(B) ETCH PATTERN ON ITS FACED ETCHED FOR 12 SDECOND X70.
24) FIG.3(C) ETCH PATTERN ON BOTTOM FACE ETCHED FOR 8 SECIND X 70.
25) FIG. 4 POHTOMICROGRAPH SHOWING THE ETCH PATTERN ON A CLEAVAGE PLANE PRODUCED BY ETCHANT (GAAX WATER) X70.