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Tracing of dislocations in gypsum single crystals

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An attempt has been made to trace the trajectory of dislocations of isolated as well as matched pairs of (010) basal cleavages of gypsum when etched with analar grade nitric acid and 0.1 N potassium hydroxide. The dislocations are shown to be oblique, parallel, continuous lines passing through the body of the crystal. The lines are cut into smaller fragments on cleaving the crystal into thinner flakes. The implications of this are discussed.

1. Introduction

The attack of solvent or chemical reagent on a seemingly uniform solid surface is frequently localized in small depressions called "etch pits". The shape of the etch figures depends upon the nature of the solvent and the symmetry of the crystal face. Just as the regular geometry and external shape of the crystal are an orderly arrangement in which the units of construction are built up during growth, so also, when it is attacked by an etchant the etch figures thus produced are related to the internal molecular structure. The concept of lattice defects or "dislocations" satisfactorily explains the origin of etch pits and their development. Horn [1] and Gevers *et al.* [2] have demonstrated that an etch pit can be formed where a dislocation meets a crystallographic surface. They have shown in a striking way that the etch pits develop where the screw dislocations emerge at the surface of silicon carbide crystals. The study of etch patterns on both halves of a cleaved specimen of rock salt led Amelinckx [3] to the conclusion that there is a one to one correspondence between the etch pits and the dislocations. He inferred from his experiments that the cleavage is a slipless fracture.

Patel and Raju [4] have reported that when matched faces and thin flakes of synthetic quartz are etched with 40% hydrofluoric acid the triangular etch pits on the prism faces represent the sites of dislocations. The evidence of edge and screw dislocations in gypsum single crystals [5] and the history of growth of gypsum [6] have been reported when (010) cleavages of gypsum have been etched using analar grade nitric acid and 0.1 N potassium hydroxide solution as etchants.

In the present paper, an attempt is made to trace the trajectories of dislocations by etching well defined (010) basal cleavages of natural gypsum.

2. Experimental procedures

For the present work a number of high quality transparent single crystals of gypsum were purchased from RFD Parkinson, England. The crystals were cleaved by inserting the edge of a sharp razor blade parallel to the (010) plane and applying a slight pressure. For studying the etch patterns, the cleavages were etched in different etchants for the required time and the patterns were carefully studied by optical methods after depositing thin silver films to enhance contrast.

3. Observations

3.1. Successive etching of an isolated cleavage

Figs 1 and 2 show the etch patterns produced on etching a (010) basal cleavage of gypsum with analar grade nitric acid for 2 and 6 min, respectively. It is evident that individual isolated etch pits enlarge in size and depth whereas micropits disappear and appear at newer sites.

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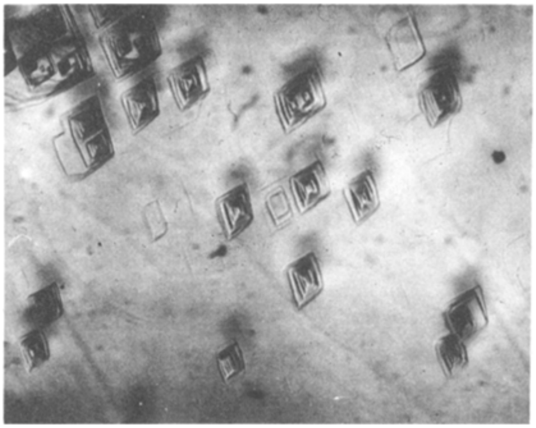


Figure 1 A cleavage face etched in analar grade nitric acid for 2 min (×72).

Figure 2 The same region as Fig. 1, etched for 4 min longer (×72)

3.2. Etching of matched pairs
Fig. 3a and b show the etch patterns produced on a matched pair etched in 0.1 N potassium hydroxide solution for 4 min. Fig. 4a and b show the etch patterns produced on a matched pair when one face is etched with 0.1 N potassium hydroxide solution and the other face with analar grade nitric acid for the required time. It is clear that there is a perfect correlation of isolated etch pits on both faces, irrespective of the etchants used.

3.3. Etching of a flake
Fig. 5a is a transmission photomicrograph of a gypsum flake (265 μm thick) almost parallel to the (010) face and etched with nitric acid. It is interesting to note that there is an exact one to one correspondence between the individual isolated pits on either side of the flake but that they are shifted uniformly.

In order to trace the trajectory of individual pits, the above flake was cleaved into three smaller flakes and etched simultaneously with nitric acid for the required time. Two typical etch patterns out of six recorded are represented in Fig. 5b and c. The shifts in the corresponding pits ("S" values) and the thicknesses of the original as well as the three flakes ("t" values) are measured and indicated in Fig. 6. From these measurements, the inclinations of the lines joining the corresponding pits of the original as well as its smaller fragments to the cleavage face are computed and recorded in Table I. It is clear from Fig. 6 that an individual isolated pit, say A, is shifted to A‴ (of magnitude S) for the original flake (of thickness t), whereas its corresponding shifts for the three smaller flakes of thickness t_1, t_2 and t_3 are A to A′ (S_1), A′ to A″ (S_2) and A″ to A‴ (S_3) respectively.

It may be mentioned that this set of measure-

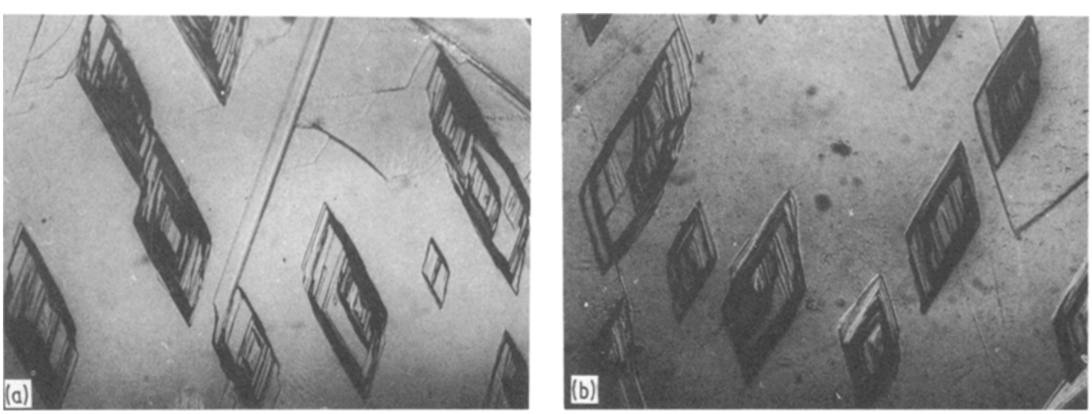


Figure 3(a) and (b) Etch patterns on matched cleavages etched in potassium hydroxide solution for 4 min (×252).

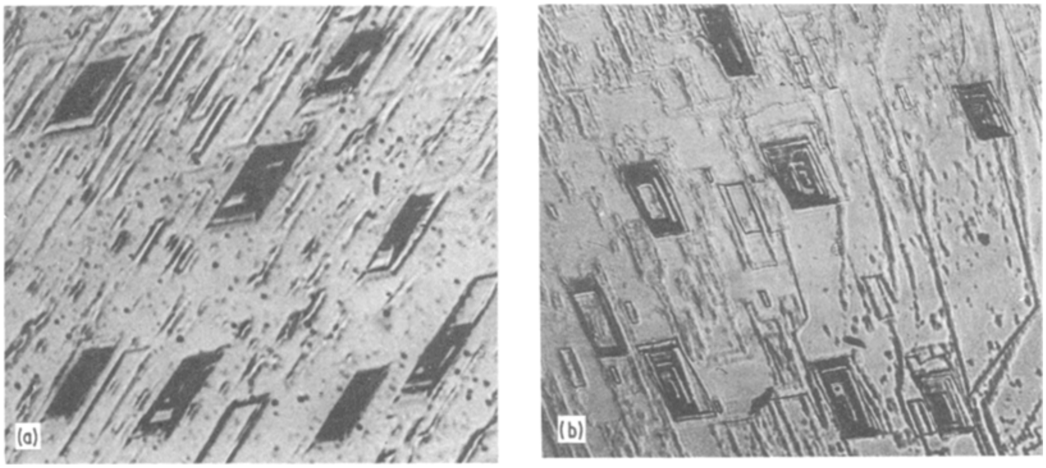
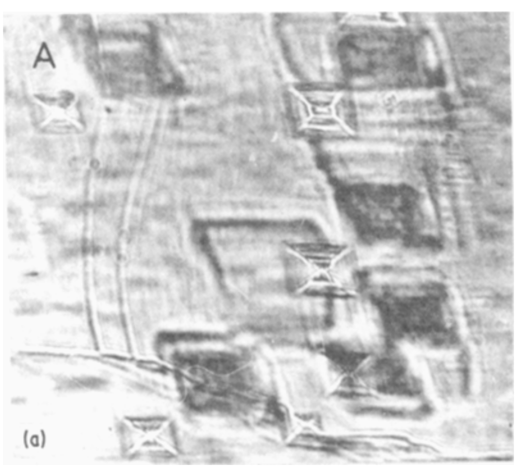


Figure 4 Etched patterns on matched cleavages (a) etched in potassium hydroxide solution and (b) etched in nitric acid (× 315).

ments has been carried out for other individual isolated pits which have their corresponding pits on the other side of the flake and the observations agree well with those for pit A marked in Fig. 5a.

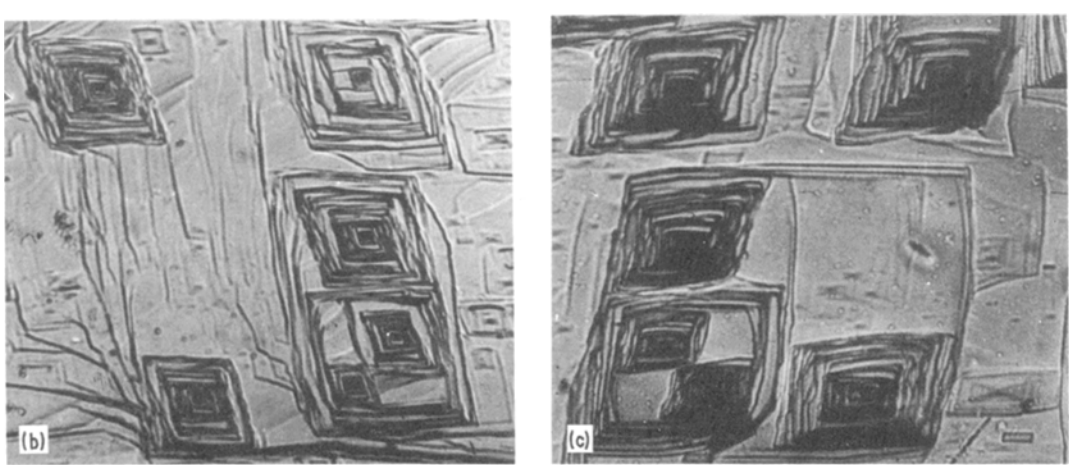


It is observed that the inclinations of the lines joining the corresponding A etch pits are the same (i.e. ~ 14°).

4. Discussion

The enlargement in size and depth of the pits on successive etching and the exact correlation of etched matched faces and flakes, irrespective of the etchants used, suggest that the sites where pits are formed represent the sites of dislocations in the crystal. The disappearance and then reappearance of micropits at new sites may be indicative of superficial defects such as point defects. On progressive etching of a flake, at each stage of

Figure 5(a) Transmission photograph showing the etch patterns on opposite sides of a flake (× 315), (b) and (c) two typical etch patterns of the region as in Fig. 5a, on smaller flakes cleaved out (× 315).



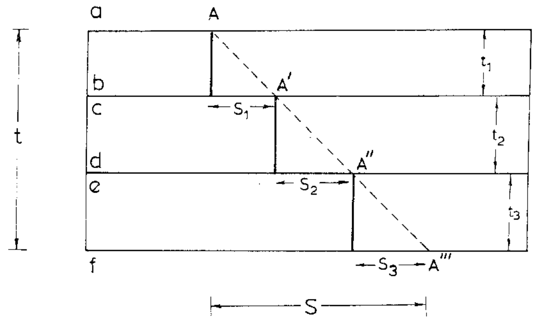


Figure 6 Schematic diagram of the flakes.

etching, the pointed bottom of the pit forms a point on the locus of a trajectory of dislocation (Fig. 7). For two stages of etching, the two points at the bottom of the pit, either X and X′ in the case of linear dislocation LL′ or Y and Y′ in the case of curvilinear dislocation CC′ represent only two points on the trajectory of dislocations, which may be either linear or curved, and not the intermediate points between them.

The exact one-to-one correspondence of the matched pairs, irrespective of the etchants used, can be explained on the basis of dislocations being cut into fragments on cleaving and etching; each dislocation fragment intersecting the cleavage initiates pit formation, thereby giving rise to an exact correlation in the etch patterns as in the present case (Figs. 3a and b and 4a and b). This will be true for dislocations which are either linear (LL′) or curved (CC′) and which are schematically represented in Fig. 7.

From Table I and Fig. 6 it is clear that the inclination of AA‴ with the cleavage face is practically the same within experimental error as the inclinations of AA′, A′A″ and A″A‴. This strongly suggests that the dislocation corresponding to AA‴ is cut into smaller fragments AA′, A′A″ and A″A‴ on cleaving a flake of thickness t into three smaller flakes of thicknesses t_1, t_2 and t_3, respectively. Since there is a correlation between the inclinations of dislocation lines computed from other pits on either side of the flake, this strongly suggests that the dislocations are parallel to each other. This is quite in agreement with the findings of Patel and Raju [7] who reported that the dislocations are inclined and parallel from measurements made on eccentricities and depths of corresponding etch pits when matched (010) faces of gypsum were successively etched.

5. Conclusions

It may be concluded that, in gypsum, (1) well-defined etch pits traceable on etching of isolated cleavages, matched pairs and flakes reveal the sites of dislocations, whereas micropits represent superficial defects such as point defects; (2) almost all the dislocations studied are linear, inclined and parallel; and (3) the dislocations are cut into smaller fragments during cleavage, whose magnitudes depend directly on the thicknesses of the cleaved out smaller flakes.

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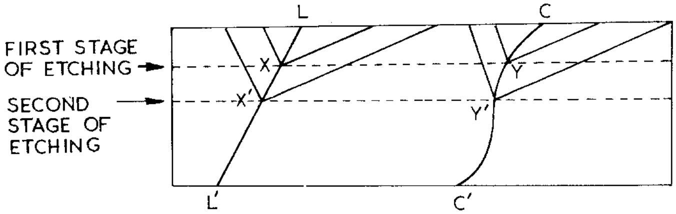

Figure 7 Schematic diagram for successive etching.

TABLE I

Flakes as represented in Fig. 6	Thickness (μm)	Shift (μm)	Inclination, θ	Dislocation line segment
1. Flake af	t = 265	S = 1047	14° 21′	AA‴
2. Flake ab	t_1 = 103	S_1 = 402	14° 22′	AA′
3. Flake cd	t_2 = 88	S_2 = 362	13° 40′	A′A″
4. Flake ef	t_3 = 67	S_3 = 277	13° 36′	A″A‴

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