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Optical and Interferometric Studies on Selenite Cleavages and Their Etch Patterns

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Selenite cleavages obtained from good-quality single crystals have been etched in different etchants and the etch patterns studied optically. By etching a matched pair for successive periods it is shown that the dislocation lines run right through the body of the crystal. When a thin crystal plate is etched for a sufficiently long period holes are produced at a few isolated places. It is conjectured that at these sites the attack starts from both sides. Etch patterns on the two sides of the thin plate match completely even where holes are not formed. At those sites where holes are not formed, though the attack starts from both sides, precipitation of some impurity may have inhibited the action of the etchant. By etching all the faces of three plates produced by cleavage of a single thicker plate a correlation of the etch patterns on all the faces was established. From the numerical measurements made on the corresponding pits on all six cleavage faces the inclinations of the dislocation lines were calculated and the lines were found to be parallel to each other. On some cleavages, a few pits having wings and thus an anomalous structure were observed. The wings are air wedges in the crystal and thus have no correlation on matched pairs. Stratigraphical etch patterns have been observed on some cleavages and it is shown that the pattern runs right through the body of the crystal. The implications are discussed.

Introduction

It is now well known that optical and interferometric studies of cleavages and their etch patterns throw important light on the dislocation content and the history of the growth of a crystal. Numerous investigators have reported interesting results on various crystal cleavages, chief amongst them being Gilman & Johnston (1956), Patel & Ramanathan (1962), Patel & Goswami (1964) and Patel & Desai (1965). Bright & Ridge (1961) have reported some studies of the development of etch pits and dehydration nuclei on the (010) face of selenite and the nature of the sites favourable for their formation. Sella & Sella (1962) from their electron microscope studies have reported the existence of parallel dislocation lines in selenite. The work by the above investigators on selenite was done on isolated cleavage faces of the crystal, while in the present investigation the authors have made use of matched cleavage pairs of faces for various experimental purposes and have obtained detailed information regarding the nature of the cleavage faces and the dislocations in the crystal. This work forms part of a series of such investigations undertaken in this laboratory on various cleavable mineral crystals.

Experimental

A number of high quality single crystals of selenite were purchased from R. F. D. Parkinson, England. The crystals were cleaved by inserting the edge of a sharp razor blade parallel to the (010) plane and applying slight pressure. The freshly cleaved faces were examined by multiple beam interferometry (Tolansky, 1948), after thin silver films had been deposited on them. 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. As the crystals are affected by moist air, precautions were taken to avoid the effect of moisture.

Observations

Fig. 1(*a*) represents the typical (010) cleavage face of selenite which reveals very clearly the usual cleavage lines. As the cleavage is initiated by inserting the edge of a razor blade without giving a blow, the cleavage does not form the usual river pattern. The topography of the face is revealed by the multiple beam interference fringes of Fig. 1(*b*). It is clearly seen from the nature of the fringes that the cleavage is not very flat and the fringes are more or less similar to those observed on mica cleavages, showing various hills and dales on the surface. The cleavage steps also form a sharp discontinuity on the surface as in the case of mica. Fig. 2(*a*) represents an etch pattern produced on a cleavage face after etching in analytical reagent grade nitric acid at room temperature for two minutes. The etch pattern consisting of the usual rhombohedral pits is clearly seen. The nature of the etched surface is revealed by the interferogram of Fig. 2(*b*) taken over the region of Fig. 2(*a*). It reveals that the etch attack is twofold. Along with the preferential etching at sites where pits are produced, the surface is also attacked by the etchant, as is revealed by the matted structure of the fringes. Fig. 3 is a magnified picture of the etch pattern revealing clearly the nature of the etch pits. As in the other mineral crystal cleavages, it may be noted that:

(1) The pits can be classified into two types: (i) point bottomed and (ii) flat bottomed.
(2) The pits are of different sizes all having the same rhombohedral shape.

(3) The bottoms of the pits are not quite central.
(4) Some of the pits have occurred in pairs.

In order to study the nature of the dislocation lines, matched pairs of cleavage faces were etched simultaneously in the same etchant successively for four different periods and the structure of the matched pairs of pits studied by making numerical measurements. Thus Figs. 4(*a*) and (*b*) and 5(*a*) and (*b*) represent the two stages of the etch pattern produced on the corresponding regions of the matched pairs by etching them respectively for four and eight minutes. Four matched pairs of pits marked A, B, C, D and A', B', C', D' were selected from Fig. 4(*a*) and (*b*) and the eccentricities of their bottoms and their depths at each stage of etching were carefully measured. From the measured eccentricities and the depths of the pits the angles of inclination θ of the dislocation line giving rise to the pit with the cleavage surface were calculated as described by Patel & Goswami (1964). All these observations are given in Table 1.

It may be mentioned that observations similar to those given in Table 1 but taken on another crystal gave an angle of inclination which was the same for all dislocation lines in that crystal, but the magnitude of the angle was 24°. Now there are no low index planes which make angles of about 13° or 24° with the (010) cleavage and hence these dislocation lines do not fall in any such planes.

To see whether the same dislocation lines terminate on both sides of a thin crystal plate, a crystal plate of thickness 0·013 mm was selected and etched for a sufficiently long time to produce holes in some places. It may be conjectured that at the sites where the holes have been produced the attack might have started from both sides, hence producing the holes earlier at these places. Fig. 6(*a*) and (*b*) represents photomicrographs of the etch patterns on the two sides of the plate. The regions on the two sides can be identified from the pattern formed by holes. It is clearly seen in Fig. 6 that the pits on the two sides where holes are not formed have one to one correspondence in their number and position.

It is indeed very interesting to note that though the plate had nearly uniform thickness, etch attack at some isolated places produced the holes while at others it did not. This has an important bearing on the conclusions drawn regarding the nature of the dislocations.

In order to see whether the dislocations terminating on the two sides of a crystal plate run straight or zigzag through the body of the crystal, a crystal plate of thickness 265 μ was selected, such that on etching the plate a correlation in number and positioning existed in a number of pits on its two sides. This plate was then cleaved out parallel to the (010) face, producing three thin plates each having two sides (*a* and *b*, *c* and *d*, *e* and *f*) as shown schematically in Fig. 7.

All three plates were then etched simultaneously and the corresponding regions of each of them on both sides were photographed. Fig. 8(*a*)–(*f*) shows the photomicrographs of the etch patterns of the corresponding regions on the six faces *a*–*f* respectively. It is indeed interesting to note that in some regions the etch pits form the same pattern on each face and show exact correspondence in the etch pattern in all the six photographs. Out of all the pits having close correspondence in all the six pictures, some eight were selected from each face and numerical measurements were made similar to those given in Table 1. It is ob-

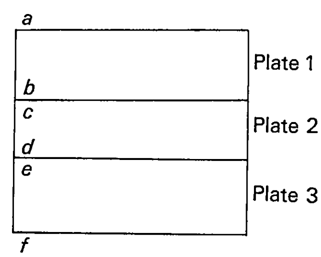
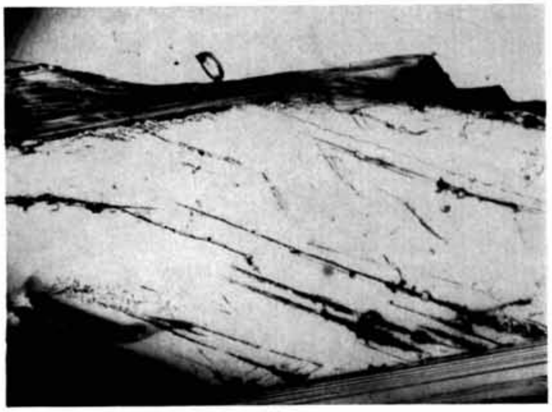


Fig. 7. Identification of faces (*a*) to (*f*) of Fig. 8 obtained by cleaving one plate into three parallel to (010).

Table 1. *Asymmetry of etch pits in selenite*

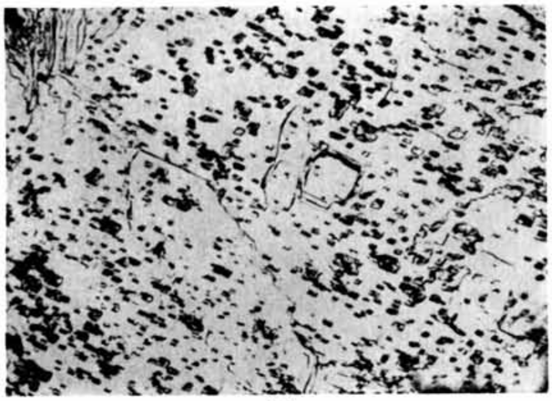
Stage of etching	Pit no.	The face Depth	Eccentricity	θ	Its counter-face Depth	Eccentricity	θ
1st	1	2·85μ	12·00μ	13°21′	2·85μ	12·00μ	13°21′
	2	2·91	12·28	13 20	2·91	12·14	13 29
	3	3·00	12·42	13 35	3·00	12·45	13 33
	4	3·00	12·42	13 35	3·10	12·58	13 50
2nd	1	3·57	14·71	13 49	3·57	14·61	13 44
	2	3·71	15·00	13 54	3·71	15·43	13 31
	3	3·84	15·71	13 44	3·88	15·73	13 51
	4	3·82	15·70	13 40	3·82	15·69	13 41
3rd	1	3·857	15·60	13 54	3·86	15·65	13 52
	2	3·90	15·85	13 29	3·90	15·80	13 37
	3	4·00	16·57	13 34	4·05	16·60	13 43
	4	4·07	16·81	13 35	4·07	16·81	13 35
4th	1	4·285	18·00	13 22	4·28	18·00	13 22
	2	4·30	18·10	13 22	4·30	18·08	13 23
	3	4·55	18·86	13 34	4·55	18·82	13 35
	4	4·58	18·89	13 38	4·58	18·89	13 38



(a)

(b)

Fig. 1. (a) A typical (010) cleavage face of selenite (× 25). (b) Multiple beam interferogram of (a).

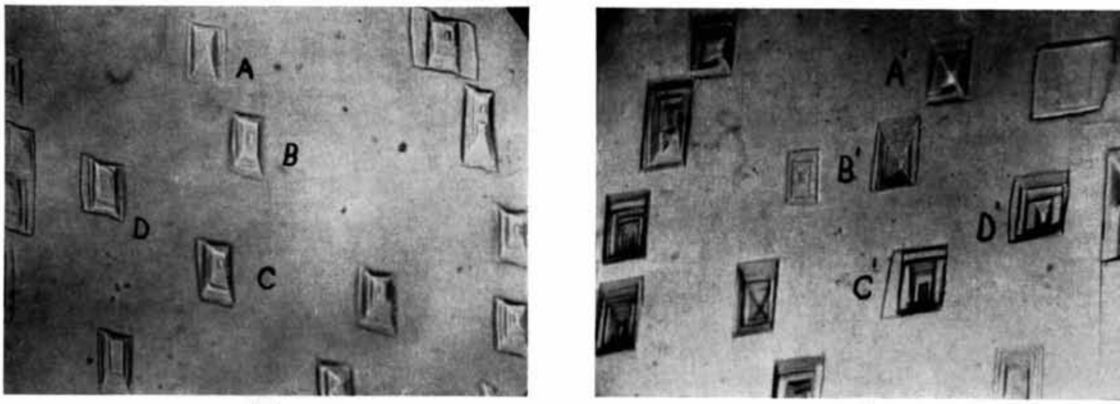


(a)

(b)

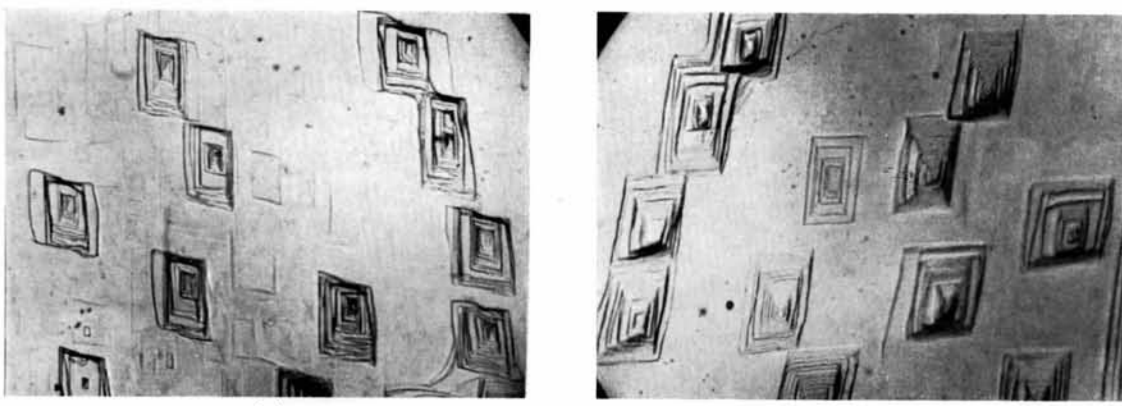
Fig. 2. (a) A cleavage face after etching in nitric acid for 2 min (× 25) (b) Interferogram of (a).

Fig. 3. Detail of part of a surface etched as in Fig. 2(a) (× 130).



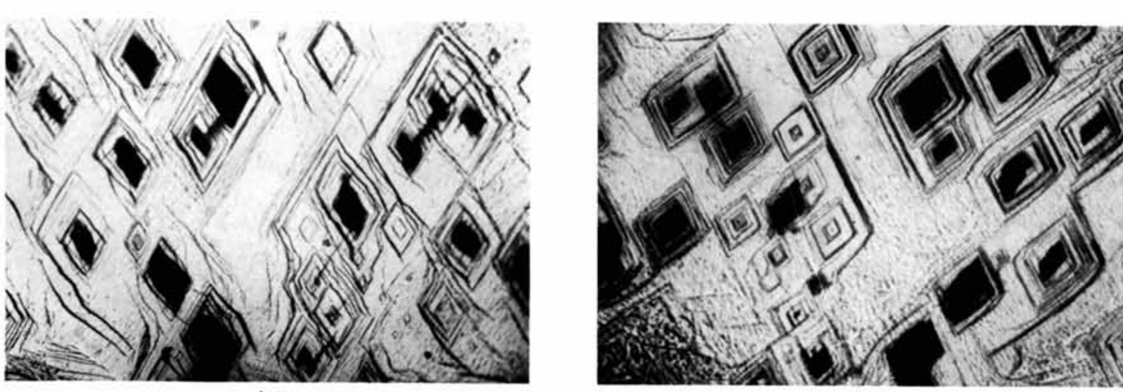
(a) (b)

Fig. 4. A matched pair of cleavage faces after etching for 4 min (× 130).



(a) (b)

Fig. 5. The same surfaces as in Fig. 4 after etching for a total of 8 min (× 130).

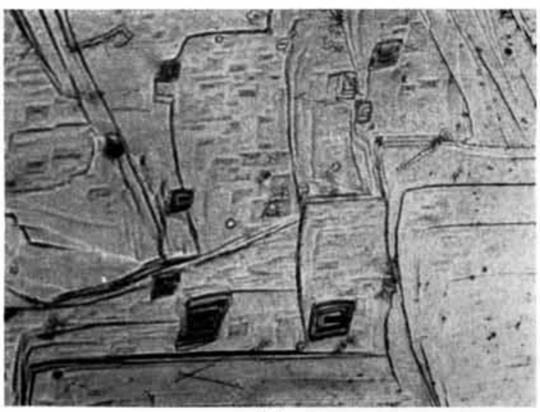


(a) (b)

Fig. 6. Two sides of a thin plate etched to produce holes (× 130).

(a)

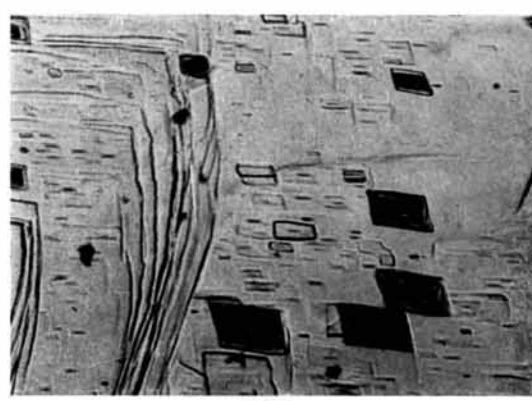
(b)



(c)

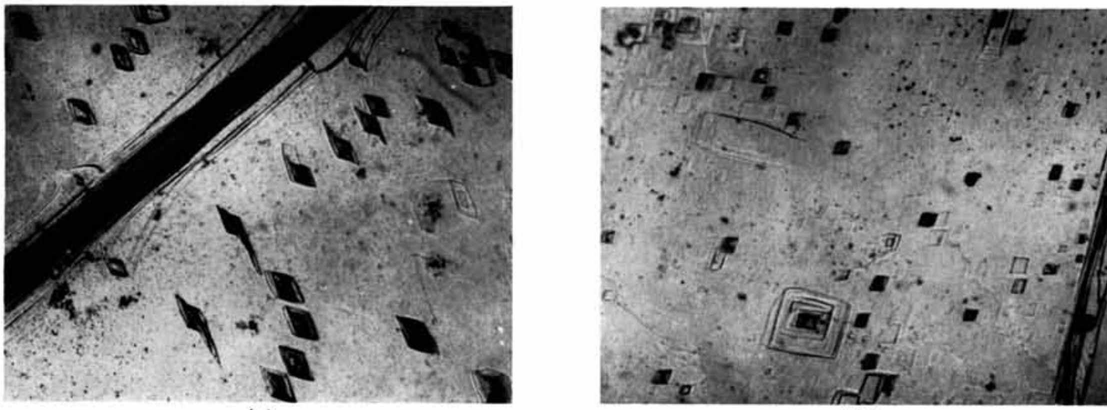
(d)

(e)



(f)

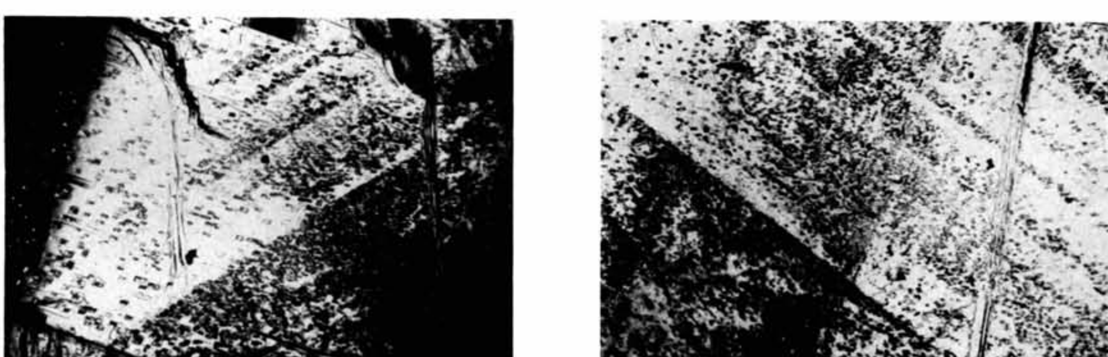
Fig. 8. Etch patterns on the six faces shown schematically in Fig. 7 ($\times$ 75).



(a) (b)

Fig. 9. (a) An etched face of selenite showing winged pits (× 130). (b) The etch pattern on the matched face corresponding to (a) (× 130).

Fig. 10. An etch pattern showing a row of pits (× 130).



(a) (b)

Fig. 11. Stratigraphical etch patterns on two sides of a selenite plate.

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served that the inclinations of the dislocation lines computed from the corresponding pits on all the six faces came out to be the same within the experimental error.

Anomalous etch pits

On etching the cleavages from some crystals it was many times observed that some of the etch pits developed with wings having an anomalous appearance similar to that reported by Patel & Tolansky (1957) in the case of mica. Fig. 9(*a*) represents the etch pattern in which the anomalous etch pits are clearly seen. In order to see whether the corresponding pits on the matched face are also anomalous in nature, the corresponding region on the matched etched face shown in Fig. 9(*b*) was photographed and compared. It is clearly seen that there is no correspondence in the anomalous nature of the corresponding etch pits. It may be mentioned that, as in the case of mica, the wings of the pits are air wedges inside the body of the crystal.

Grain boundaries

Numbers of crystal cleavages when etched revealed rows of etch pits along with other etch patterns. Thus Fig. 10 represents the etch pattern in which the row of the pits is clearly seen. Assuming that the row of pits reveals the grain boundary, the spacing of the pits in it was measured and, from the lattice constant of the crystal, the angle between the two grains forming the boundary was computed to be one minute. A thin flake some 0·017 mm thick was cleaved out from the top of the crystal having the row of etch pits described above, and the fresh crystal face was etched in a different etchant. It was interesting to note that the row of etch pits was again produced. The spacing between the consecutive pits was again measured and it was found to be the same as in the previous case. It is therefore conjectured that the rows of etch pits may reveal the sites of edge dislocations in the grain boundary of the crystal.

Stratigraphical etch pattern

On the cleavages of some crystals, stratigraphical etch patterns similar to the patterns observed by Patel & Tolansky (1957) on diamond, Patel & Goswami (1962) on calcite, and Patel & Desai (1965) on calcium fluoride were observed. Fig. 11 reveals the stratigraphical patterns on the two sides of a crystal plate, 0·15 mm thick. It is interesting to note that the stratigraphical etch patterns consisting of linear rows of crowded etch pits and dividing the whole surface into regions of different densities of etch pits match completely on the two sides.

Discussion

The individual isolated pits reveal the sites of dislocations as in the case of other mineral crystals. The observations made on the inclination of the dislocation lines (Table 1), and also those made on three thin flakes cleaved out from the thick crystal plate, showing that the inclination is the same for all dislocation lines, indicate that the dislocation lines run straight through the body of the crystal and are parallel to each other in the case of selenite. That the angles of inclination of the dislocation lines in different crystals are different suggests that the inclinations may depend on the conditions under which the crystals grew. That the holes are produced only at some of the sites, even though the etch attack starts at many sites from both sides when a thin crystal plate is etched, suggests that at the sites where holes are not produced some sort of inhibitive action may be taking place. This may be due to the precipitation of some impurities at these dislocation sites during the growth of the crystal, thus inhibiting the action of the etchant. This confirms the observations reported by Patel & Goswami (1964) in the case of topaz. That the anomalous etch pits have correlation in their number and position but no correlation in their structure on matched faces, and that the wings of the pits are air wedges in the body of the crystal, suggests that the sites of these pits may indicate the usual dislocation sites.

The formation of the wings giving an anomalous character to the pits can be explained by assuming the existence of fission tracks in the body of the crystal as observed by Fleischer, Price & Symes (1964) near the dislocation line. On etching, when the pit grows larger and when its boundary touches the fission track, the etchant will etch the track and will produce an air wedge in the body of the crystal as observed. If a fission track lies near one dislocation line on one cleavage, it does not necessarily also lie near the corresponding dislocation line on the matched face so as to produce a correlation in the wing structure.

The formation of the stratigraphical etch pattern and its correlation on the two sides of a crystal plate can be explained as due to the changes in the conditions of growth during the period of growth of the crystal as explained by Patel & Tolansky (1957).

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