APPENDIX - B

FISSION TRACKS AND LOW ANGLE TILT BOUNDARIES ON $(10\bar{1}0)$ CLEAVAGES OF NATURAL APATITE CRYSTALS

B - 1 Introduction

Lovell (1958) has reported etch beaks on (0001) basal planes of apatite and attributed them to fission fragments. She also observed low angle boundaries composed of etch pits representing the sites of edge dislocations. More recently Price, et al (1964) have described the origin of anomalous etch pits on the basal planes. Price, et al (1964) have used the etch patterns on apatite and other minerals for determining their geological ages from the density of fission tracks observed in the etch pattern.

In this appendix, a study of dislocation etch pits and fission track etch pits produced on $(10\bar{1}0)$ cleavage faces of apatite has been reported. It may well be pointed out that the earlier investigators made such studies on isolated natural $(10\bar{1}0)$ faces. Low angle tilt boundaries observed on these $(10\bar{1}0)$ cleavages are also described.

B - 2 Experimental

The prism ($10\bar{1}0$) cleavages were obtained in the manner described already in Appendix A. Etching was carried out in 50 % citric acid solution. Normally the etch patterns were examined after depositing thin silver films on the etched surfaces in incident light; when the studies were made on fission tracks, no silver film was deposited and the etch patterns were studied in transmitted light.

B - 3 Observations of fission tracks

The typical etch pattern produced on matched pairs on ($10\bar{1}0$) cleavages is shown in figures 135 (a) and 135(b) (X 350). Attention is drawn to the following :-

(1) Unlike the etch patterns on many crystals, the number and position of pits on the matched faces, do not correspond exactly although, there is considerable correspondence.

(2) Though the pits had more or less the same shape, they are not all of the same size.

(3) A close comparison of the two sets of the etch patterns reveals that correspondence exists in the larger pits

and that they are nearly of the same size on both the faces; the smaller pits have no correspondence.

(4) The attack of the etchant, produces two types of pits, individual isolated large pits, and numerous small crowded pits.

When $(10\bar{1}0)$ cleavages from other crystals were etched in the same way the patterns consisted of the pits similar to those described in figure 135 (X 350) together with some anomalous etch pits similar to those reported by Lovell (1958). Price et al (1964) have reported what were previously called anomalous etch pits originate at fission fragments in the case of apatite and many other crystals. In order to distinguish the pits nucleated at fission tracks from those nucleated at dislocation sites, an etched cleavage face showing some anomalous etch pits was successively etched for two more periods. Figures 136(a) (X 300), 136(b) (X 300) and 136(c) (X 300) represent the same region on $(10\bar{1}0)$ cleavage face of apatite etched for 20 min., 40 min. and 60 min. respectively.

A study of the etch patterns shown in above figures reveals the following features :-

(1) As mentioned earlier, two types of pits are formed.

(2) The etch pattern consists of pits, with and without trails.

(3) The trails of the pits are randomly oriented.

(4) The pits are more or less of the same shape.

(5) The pits with trails continue to have trails on further etching while a few without trails may develop trails at a later stage.

(6) The trail of the pits is slowly developed during etching, onee it is fully developed, its length remains constant. The lengths of most of the trails in the pictures are between 3 to 5 microns.

(7) Comparison of successive etch patterns reveals that a number of small new pits develop with small trails in the begining; on continued etching these pits get bigger and the trails develop fully.

For further investigation of the two types of the pits mentioned above, etch patterns were produced on matched pairs on which both the types of pits could be obtained. Thus figures 137(a) (X 300) and 137(b) (X 300) are the photomicrographs of etch patterns on corresponding regions of a matched pair. The pictures reveal that :-

(1) The pits are of both types and are of assorted sizes having more or less the same shape.

(2) All the pits without the trails have one to one correspondence on the matched pairs.

(3) Some pits having trails on one face have corresponding pits with trails on the matched face. Usually these pits are relatively larger on both faces.

(4) There also is correspondence in few pits which have trails on one face but none on the matched face.

(5) Almost all of the smaller pits having trails have no corresponding pit on the matched face.

If the etched faces are silvered and then examined in incident light, trails are not observed suggesting thereby that the trails are air channels in the body of the crystal. Figures 138(a) (X 300) and 138(b) (X 300) represent the same regions of figures 137(b) and 137(a) (X 300) taken in incident light after silvering the faces. It is clearly seen that the trails have all disappeared.

B – 4 Low angle tilt boundaries

During the investigations described above, it was frequently observed that the etch patterns consisted of long chains of more or less equally spaced etch pits similar to those observed by Vogel, et al (1953) in germanium. A description of such boundaries on apatite is given below.

(a) Rows of etch pits

Figure 139 (X 400) shows a row of etch pits produced by etching a $(10\bar{1}0)$ cleavage surface of apatite with 0.5 N nitric acid for about 40 minutes. From the photograph, the distance between successive pits was calculated and was found to be 0.57 microns. Assuming that the row of pits represents the edge dislocations in a small angle tilt boundary, the angle of the tilt [redacted] calculated from the spacing is found to be nearly 4 minutes. In the present investigations the spacing between the pits has been calculated actually by counting the number of pits in 5 cm. length at a magnification of about X 1000. Since the tilt is small, the orientation of the triangular pits on the two sides of the boundary appears to be the same. The author could not verify the tilt as was done by Vogel, et al (1953) in germanium, with the help of X-rays.

(b) <u>Intersecting boundaries</u>

In order to ascertain whether or not the rows of pits, described above represent the tilt boundaries in apatite, the author looked for intersection of such boundaries. Wagner and Chalmers (1960) in germanium, Wernick et al (1958) in antimony, Patel and Desai (1965) in calcium fluoride have reported that at the junction of three boundaries the relation $n_a = n_b + n_c$ holds good. Here n_a, n_b and n_c represent the number of dislocations per micron in the three boundaries. Thus figures 140(a) (X 350) and 140(b) (X 350) represent intersecting boundaries in which the pits in all the three boundaries are fully resolved. Observations were made on two such boundaries and they are given in Table No. B - 1.

TABLE NO. B - 1

PIT DENSITIES IN PITS/MICRON OF A BOUNDARY
FOR INTERSECTING BOUNDARIES

Obs. No.	Boundary n_a	Boundary n_b	Boundary n_c	$n_b + n_c$
Figure 140(a)	0.180	0.062	0.118	0.180
Figure 140(b)	0.130	0.053	0.077	0.130

It is seen that in both the cases the pit count in one branch equals the sum of those of the other two.

B - 5 Discussions and Conclusions

All our observations made in the section B - 3 can be explained by assuming that the pits nucleate at the sites of (1) dislocations or (2) fission fragments, both [redacted] of which exist in the crystal. When the crystal is cleaved some of them may be cut into two, so that their ends terminate on the two cleavage faces. When the matched cleavage pairs are etched, pits will nucleate at the terminations of both fission tracks and dislocations. The pits nucleated at the dislocation site will be without the trails; those nucleated at the fission tracks will have trails. Thus matched pits with and without trails are observed. Now the etch attack, producing the crowded micro-pits produce general dissolution of the surface. On continued etching, new layers of the crystal faces are exposed to the etchant. It is likely that some of the fission tracks are not cut by the cleavage because they lie somewhat below the cleavage surface. On further dissolution these fission tracks are exposed to the etchant, thus producing pits with trails. Since these pits develop at a later stage they will in general be smaller than those nucleated earlier. Since these fission tracks exist on only one side of the cleavage surface the pits can develop only on one of the matched faces as observed. In fact the two matched pairs cease to resemble one another after general dissolution of both faces. The correspondence in the pits having trails on one face and no trails on the matched face can be explained as follows :-

Pairs of pits without trails nucleate at dislocations cut by the cleavage. As these grow larger and deeper, one of their surface boundaries may touch an existing fission track in the body of the crystal and will thus appear to have a trail.

In general the dislocation etch pits have one to one correspondence on matched pairs, and at least one of them has no trail. The fission track pits invariably have the trails; they may or may not have correspondence and whenever they do both the pits have trails.

In the case of intersecting rows of etch pits described in section B - 4 it is seen that the pit density in one row equals the sum of densities in the other two. It is therefore suggested that the rows of etch pits represent low angle grain boundaries in the crystal.