showed it. They all gave normal rutile patterns, with slight increases in d-spacings.

The interesting point that emerges here is that it would appear that a considerable amount of Ta can occur in the rutile structure without affecting it in any way, whereas an appreciable amount of Nb affects it considerably and, in fact, could lead to a breakdown into a rutile (tetragonal) phase and a columbite (orthorhombic) phase, a point which, unfortunately, I have been unable to resolve as yet. If this is the case, that while Ta can tolerate a tetragonal structure, Nb cannot, we have a strong pointer, well worth further investigation, to the reason why $FeTa_2O_6$ can occur as the tetragonal form tapiolite, but that mossite, the niobium equivalent, is unknown. It would further appear, from published literature (Berry and Mason, 1959, p. 370) that a similar state of affairs also applies to Mn in the (Fe, Mn) (Ta, $Nb)_2O_6$ series.

In conclusion I wish to thank Mr. S. MacDonald, Principal Geologist (Economic Geology), for drawing my attention to this rediscovered sample, Mr. Leong Pak Cheong, Acting Assistant Director (Geochemistry), for providing the chemical analyses, and Dr. J. B. Alexander, Director, Geological Survey of the Federation of Malaya, for permission to publish this note.

References

Berry, L. G. and Mason, B. (1959) *Mineralogy*. W. H. Freeman and Co., San Francisco.

Flinter, B. H. (1959) Re-examination of "struverite" from Salak North, Malaya: *Am. Mineral.* **44**, 620-632.

THE AMERICAN MINERALOGIST, VOL 49, MAY-JUNE, 1964

## ON THE ORIGIN OF ANOMALOUS ETCH PITS IN MINERALS

R. L. Fleischer, P. B. Price and E. M. Symes, *General Electric Research Laboratory, Schenectady, New York.*

In their studies of etch figures on mineral surfaces, crystallographers have long been puzzled by so-called "anomalous" etch pits which extend deeper below the surface than is usual. Honess (1927) reviewed some of the earlier observations. Since then, Lovell (1958) has reported etched "beaks" in apatite, and Patel and Tolansky (1957) and Patel and Ramanathan (1962, 1963) have studied and suggested origins of isolated, deep etch pits in mica. It has been recently discovered that linear regions of radiation-damaged material are naturally produced and preserved in many minerals, and that these regions have an enhanced chemical reactivity (Price and Walker, 1962, 1963; Fleischer and Price,

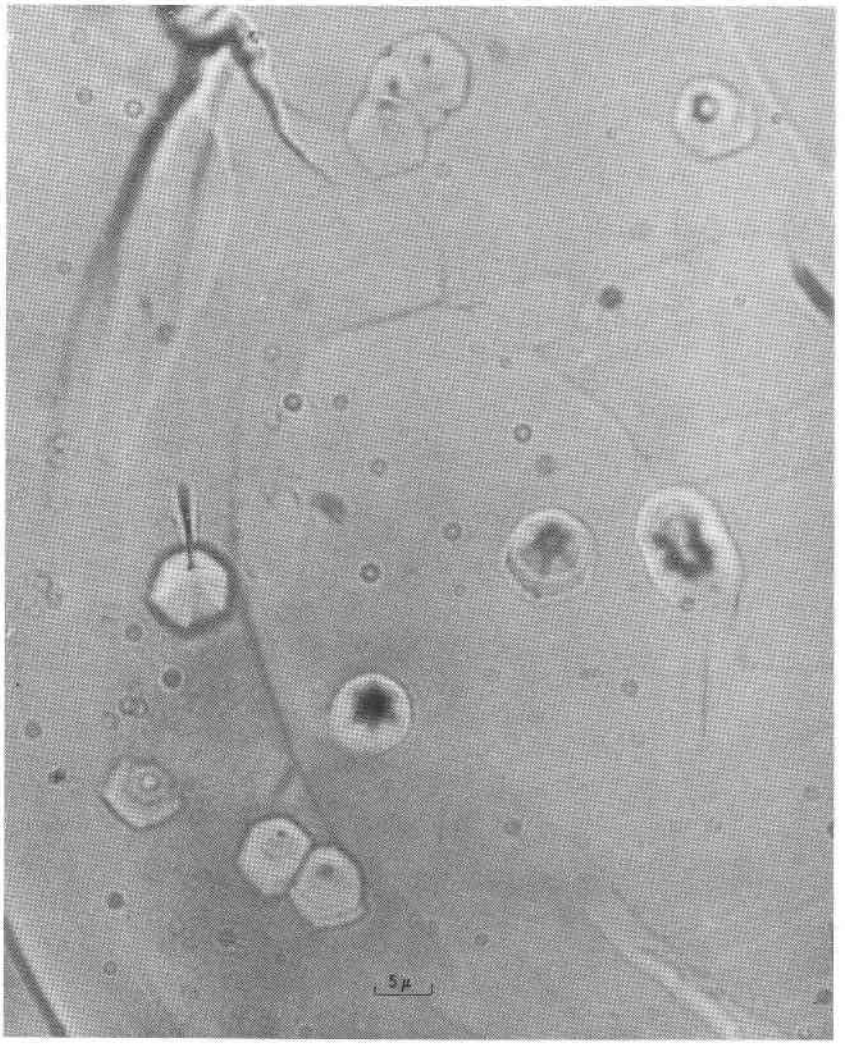


FIG. 1. Etch pits on the basal plane surface of an apatite crystal etched in concentrated nitric acid for 25 seconds at room temperature.

1963a, b). In this note we wish to point out that these damage trails are the nuclei for many of the "anomalous" pits in certain etched minerals.

Typical examples of these etch figures are shown in Figs. 1 and 2. Figure 1 is a photomicrograph of the basal plane of an apatite crystal that has been etched in concentrated nitric acid at room temperature for 25 seconds. Two types of etch pits are present. The hexagonal, pyramidal pits are attributed to dislocations as proposed by Lovell (1958), and the

pit with a beak inclined in a non-crystallographic direction is the type which Lovell calls "anomalous." Figure 2 shows a group of three isolated etch pits in muscovite mica that has been etched 7 hours in concentrated hydrofluoric acid at room temperature.

It was suggested by Patel and Tolansky (1957) that pits such as those in Fig. 2 are the result of the preferential chemical attack of impurity

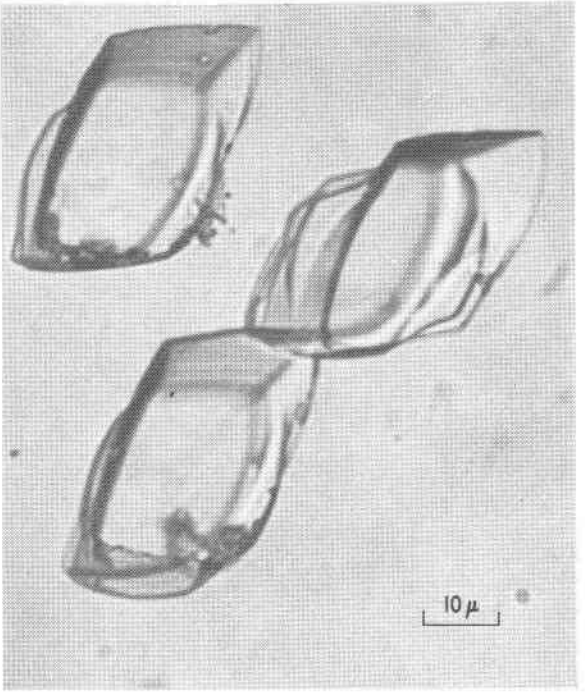


FIG. 2. Etch pits on a cleavage surface of mica that has been etched in concentrated hydrofluoric acid for 7 hours at room temperature.

centers in mica due to the localized lattice distortion around them. Patel and Ramanathan (1962) postulated that the pits originate at dislocations inclined to the basal plane. More recently, Patel and Ramanathan (1963), after studying a large number of pits with different etching characteristics, proposed that the pits form at dislocations and that impurities precipitated along the dislocation may inhibit or enhance the chemical attack. Therefore, the etching characteristics of a particular pit would be dependent on the type of impurity and the spacings between impurities.

Investigations in our laboratory show that many of the isolated pits observed in micas (Price and Walker, 1963) and many of the oblique,

beaked pits in apatite result from the preferential chemical attack of material which has been radiation-damaged by fragments from the spontaneous fission of uranium impurities occurring over geological time. Figure 3 shows three fission fragment tracks in a muscovite that has been etched 20 minutes in concentrated hydrofluoric acid. The three tracks are fine channels which, when etched for a total time of 7 hours, resulted in

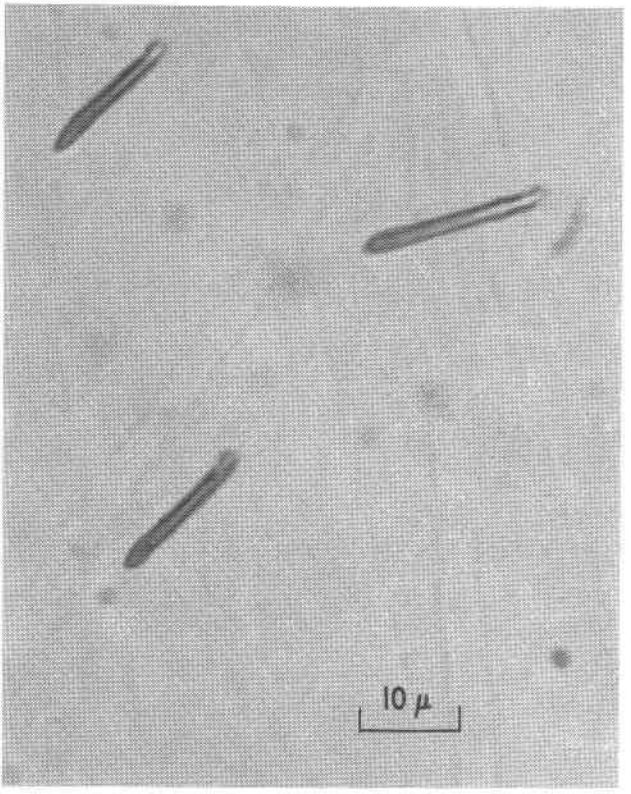


FIG. 3. Fission fragment tracks in mica. The same three pits as Fig. 2, but after an initial etch of only 20 minutes in concentrated hydrofluoric acid at room temperature.

the large pits of Fig. 2. Mica which has been deliberately exposed to a laboratory source of fission fragments contains tracks which have exactly the same appearance and enlarge into pits at exactly the same rate as the natural tracks in Fig. 3.

The general characteristics of fission fragment tracks can be compared with the observations of isolated etch pits in mica. Patel and Tolansky (1957) reported the mirror imaging of etch pits in cleavage faces of mica.

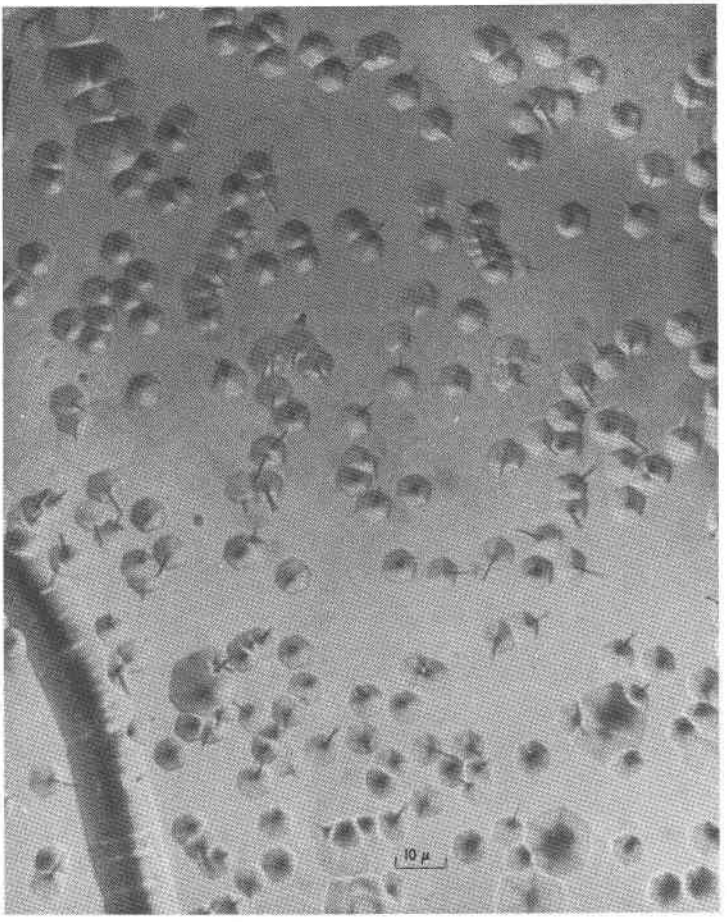


FIG. 4. Fission track etch pits on the basal plane surface of an apatite crystal that has been exposed to a laboratory source of fission fragments. Etched for 15 seconds in concentrated nitric acid at room temperature before and after irradiation.

This behavior would result from any linear defects, including fission fragment tracks. Fission fragment tracks in mica have an etchable range of about 20 microns—an average of 10 microns for each fragment from a fissioned uranium nucleus. Patel and Tolansky (1957) reported the penetration, by etched channels, of mica sheets up to 20 microns thick but not 40 microns thick; this is consistent with an origin attributed to fission fragments. Patel and Ramanathan (1963) have measured the density of etch pits and their depth in muscovites from several locations. None of the pit depths measured exceeds the fission fragment track

ranges, and the densities measured by these investigators and others are similar to spontaneous fission fragment track densities which we have measured in Indian muscovites. In another study Patel and Ramanathan (1962) have examined etched mica sheets up to 60$\mu$ thick and have found pits on the opposite faces that can be matched up. These pits are shallow and are probably due to dislocations. All of this evidence suggests that etch pits with a significant depth into mica have a spontaneous fission origin and would appear as narrow cylinders if etched for a much shorter time.

The oblique beaked pits in apatite also have the same appearance as deliberately introduced fission fragment tracks. Figure 4 shows a high density of pits on a (0001) surface of an apatite crystal, each pit having a tail going at an oblique angle into the crystal. This surface has been exposed to a laboratory source of fission fragments. Since the apatite had been previously etched, the old pits due to dislocations and spontaneous fission events are larger and can be distinguished from the new pits formed after the second etching.

Some of the oblique beaked pits in apatite are due to dislocations. We have found that by etching for a prolonged time, we can distinguish sites of dislocations, which extend for long distances through the crystal, from sites of fission tracks, which have a maximum length of $\sim$20$\mu$. Pits which have a depth less than 20$\mu$ and which become flat-bottomed and lose their beaks occur at tracks, whereas pits with long tails extending many times this distance and frequently curved indicate dislocations.

The density of natural fission tracks in a mineral is related to its geological age (Price and Walker, 1963). Etched tracks have been used recently to measure ages of micas (Fleischer *et al.* and Symes, 1963; Maurette *et al.*, 1963) and natural glasses (Fleischer and Price, 1963a). Spontaneous fission tracks due to uranium impurities have now been observed in a number of minerals by etching (Fleischer and Price, 1963b), so that the dating method should have rather wide applicability.

## Acknowledgment

We are indebted to R. M. Walker for calling our attention to the paper by Patel and Ramanathan (1962) and for discussions of the origin of anomalous etch pits in mica.

## References

Fleischer, R. L., D. S. Miller, P. B. Price and E. M. Symes (1963) Fission track ages and track-annealing behavior of some micas. *Science*, **43**, 349–351.

——— and P. B. Price (1963a) Fission track evidence for the simultaneous origin of tektites and other natural glasses. *Geochim. Cosmochim. Acta* (in press).

——— and P. B. Price (1963b) To be published.

HONESS, ARTHUR P. (1927) *The Nature, Origin and Interpretation of the Etched Figures on Crystals*, John Wiley and Sons, New York.

LOVELL, L. C. (1958) Dislocation etch pits in apatite. *Acta Met.* **6,** 775–778.

MAURETTE, M., P. PELLAS AND R. M. WALKER, *Bull. Soc. Franc. Mineral.* (in press).

PATEL, A. R. AND S. RAMANATHAN (1962) Etching of mica cleavages, *Acta Cryst.* **15,** 860–862.

——— AND S. RAMANATHAN (1963) Comparative study of the etch patterns on muscovite from different sources. *Am. Mineral.* **48,** 691–698.

——— AND S. TOLANSKY (1957) The etching of crystal cleavages. *Proc. Roy. Soc.* **A-243,** 33–40.

PRICE, P. B. AND R. M. WALKER (1962) Observation of fossil particle tracks in natural micas. *Nature,* **196,** 732–734.

——— (1963) Fossil tracks of charged particles in mica and the age of minerals. *Jour. Geophys. Res.* **68,** 4847–4862.

THE AMERICAN MINERALOGIST, VOL. 49, MAY-JUNE, 1964

## ELECTROPOLISHING OF PYRITE

ERNEST G. EHLERS AND JOHN D. BIRLE, *Department of Mineralogy, The Ohio State University, Columbus, Ohio.*

Although a considerable body of literature exists on the electropolishing of metals (see references in Faust 1948 and Tegart 1959) apparently no investigations have been made on sulfides. The technique will probably prove to be a useful one for mineralogists, as crystals or aggregates with either naturally-occurring faces or artificially-produced surfaces may be used. With the proper choice of reagents, crystalline materials can be made to polish essentially uniformly on all surfaces, as is done with most metals. Alternately, using other reagents, crystalline materials may be made to polish or etch differentially on different surfaces; this would yield information on orientation, differential solubility, and distribution of defects, etc. The minerals to be examined by this technique must have a moderate to high conductivity. The electropolishing technique was developed for pyrite during a study of oxidation processes.

A simplified sketch of the electropolishing technique is shown in Fig. 1. The crystal is immersed in a solution of appropriate composition. An electrical potential is placed across the crystal and solution such that the crystal acts as an anode, and a metal plate or cylinder in the solution a cathode.

If the crystal can be electropolished in the solution, the general relationship between applied voltage and current density will somewhat resemble the curve in Fig. 2 (Tegart, 1959, p. 3). Current density is defined as the number of amperes per unit area of reactant surface.

Those portions of the curve characterized by a change in current den-