and from this plane C(4) is displaced by −0·005, C(5) by −0·005, and C(6) by +0·005 Å. The plane through C(5), C(6) and N is

$$-0\cdot7906X-0\cdot6096Y+0\cdot0573Z=-4\cdot0845\,,$$

which is inclined by 1·2° to the plane through C(4), C(5) and C(6), so that the hexamethylenediammonium ion may be regarded as planar within reasonable limits of error.

Consideration of the thermal parameters in Table 1 indicates that there are no particularly anisotropic vibrations; a fuller analysis of these has not been carried out.

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The Etching of Matched Fracture Faces of Quartz

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Small synthetic quartz crystals from the Bell Telephone Laboratory, U.S.A., grown in molar sodium hydroxide solution at 360–400°C and 20,000 lb.in^{-2} were cleaved along planes parallel to the rhombohedral faces. The cleavage faces were etched in different concentrations of hydrofluoric acid. When they were etched in a solution containing 40 parts of the acid in 60 parts of water, etch spirals together with the usual etch pits were produced. By etching matched cleavage pairs, one-to-one correspondence of the spirals produced was established. It is conjectured that the spirals are associated with the etching of screw dislocations in the crystal.

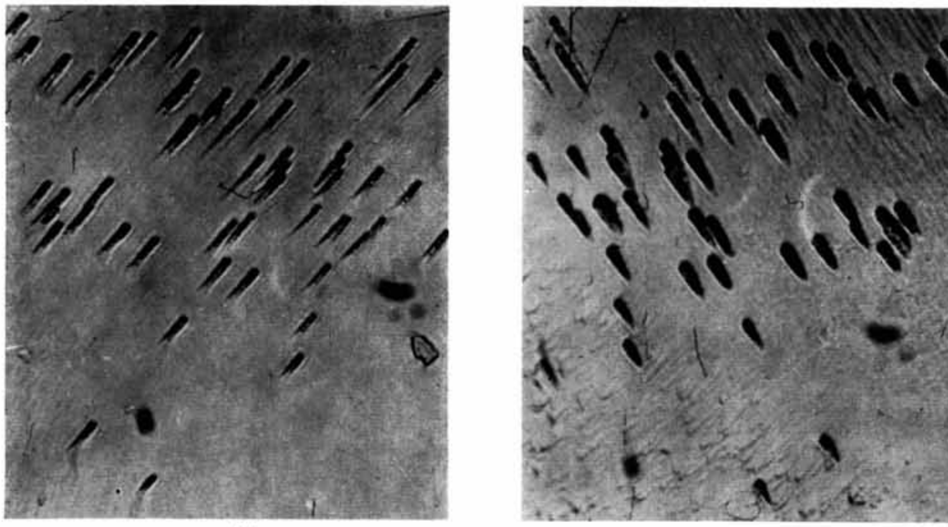
Efforts were made to cleave these crystals parallel to the prism faces along which it has no natural cleavage. Cleavage (fractured) faces more or less parallel to the prism faces were obtained. Matched pairs of the fractured faces were etched in hydrofluoric acid and one-to-one correspondence in the distribution of etch pits was established. It has thus been shown for the first time that the etch patterns on fractured faces which may not be truly crystallographic planes completely match. By etching the prism fractured faces successively for different periods, and by comparing the etch patterns on the two sides of a thin plate, it was shown that the etch pits reveal the sites of dislocations.

Introduction

A number of investigators have applied etch methods for gaining information regarding the history of growth of crystals and their dislocation content. Chief amongst them are Gilman & Johnston (1956), Patel & Tolansky (1957), Patel & Goswami (1962) and Patel & Ramanathan (1962). For such studies the cleavage faces are usually used. Tsinzerling & Mironova (1963) and Patel, Bahl & Vagh (1965) have studied fractured rhombohedral $(10\bar{1}1)$ faces of quartz. Patel & Bahl (1965) have reported that spiral pits can be produced at the sites of screw dislocations in graphite by selecting a suitable etchant and the right etching conditions. Since a number of investigators have reported the existence of spiral growths on the rhombohedral faces of quartz crystals, it was thought that some screw dislocations might be threading through these faces. When the crystals are cleaved parallel to these faces, the dislocations may be cut into two and will have their terminations on both the cleavage faces. Patel, Bahl & Vagh have etched such cleavages, but no spiral pits were reported. It was thought that by selecting a proper etchant and conditions of etching, the screw dislocations might be revealed by spiral etch pits developing on the crystal faces as reported by Patel & Bahl (1965). The present paper describes experiments carried out with rhombohedral and prism fractured faces of quartz.

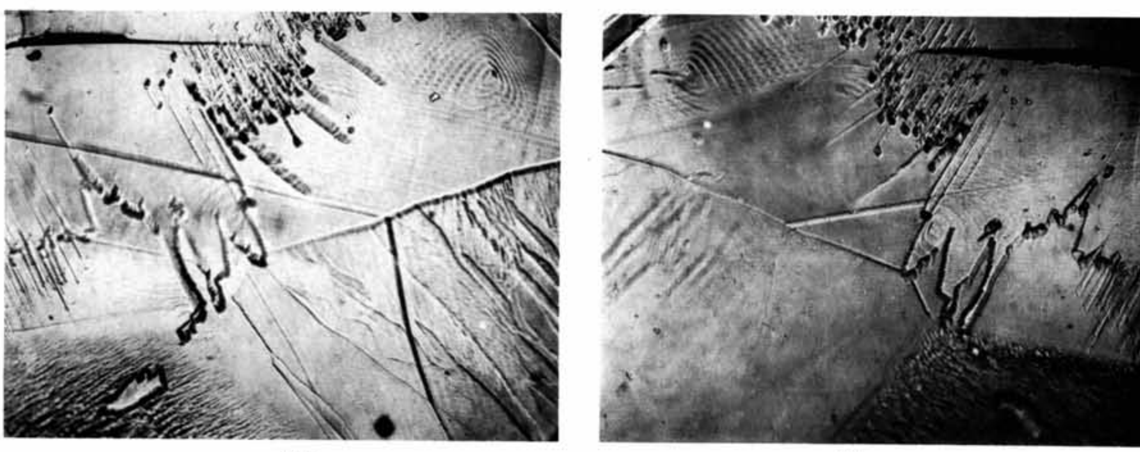
Experimental

The investigations were carried out on small synthetic quartz crystals obtained from Bell Telephone Laboratories (U.S.A.). They were grown in molar sodium hydroxide solution at a temperature of 360–400°C, and a pressure of 20,000 lb.in^{-2}. Selected crystals were cleaved parallel to the rhombohedral faces and the cleavage faces were etched in a number of solutions of



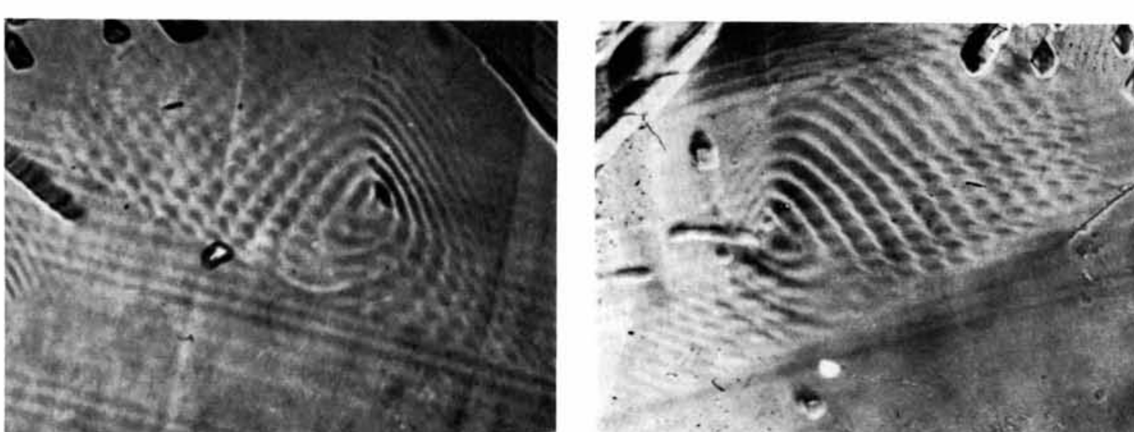
(a) (b)

Fig. 1. A matched pair of cleavage faces of synthetic quartz parallel to rhombohedral faces, etched in hydrofluoric acid (× 350).



(a) (b)

Fig. 2. Matched rhombohedral cleavage faces of synthetic quartz, etched in hydrofluoric acid (40:60) for 1 hr (× 140).



(a) (b)

Fig. 3. (a) and (b) show the spirals observed at the top of Fig. 2 (a) and (b) respectively (× 280).

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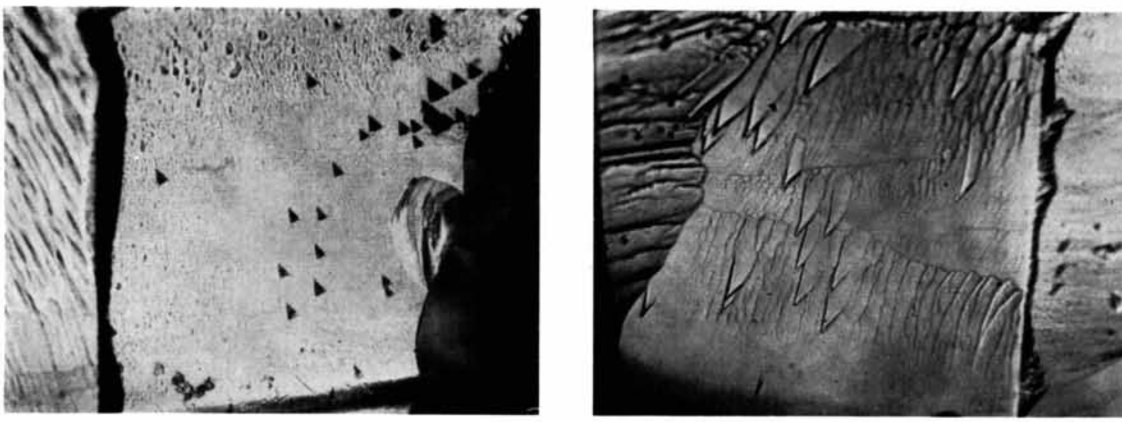
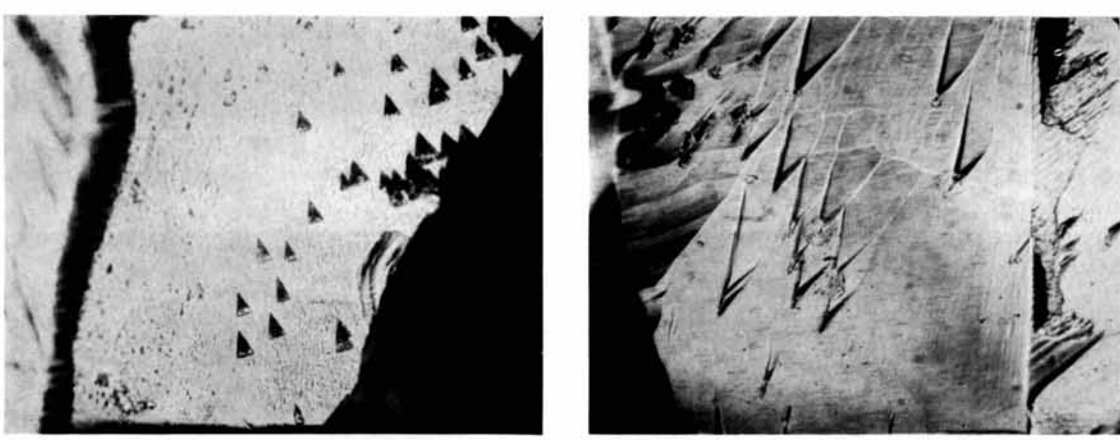


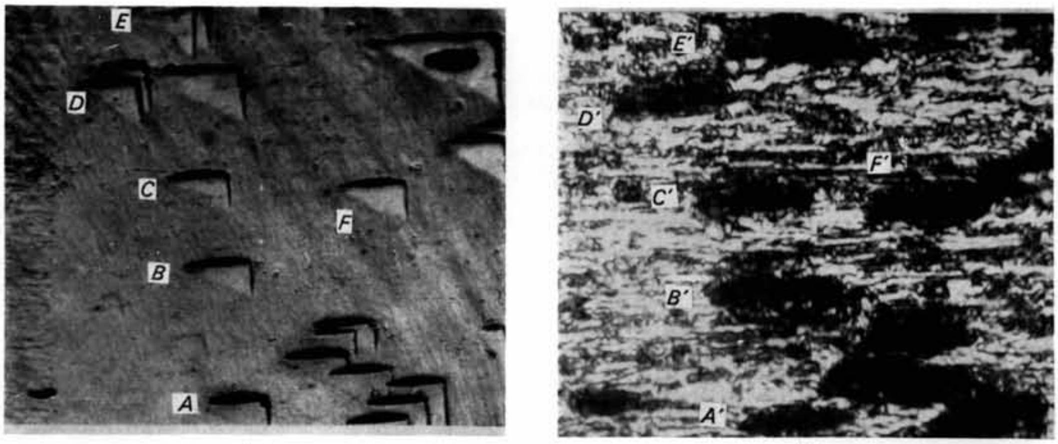
(a) (b)

Fig. 4. Matched fractured prism faces of synthetic quartz, etched in hydrofluoric acid for 8 hr (× 280).



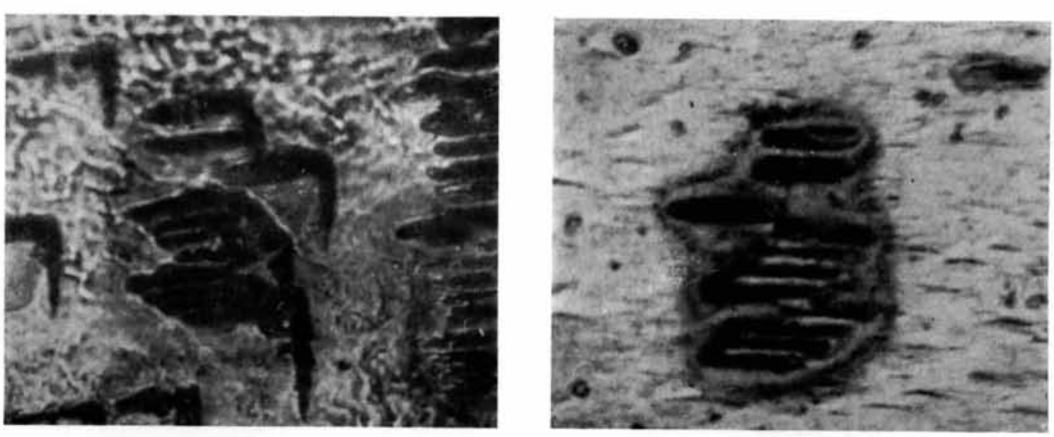
(a) (b)

Fig. 5. The same faces as in Fig. 4 after etching for a further 2 hr (× 280).



(a) (b)

Fig. 6. (a) An etched fractured prism face of synthetic quartz (b) The same face after polishing and re-etching (× 140).



(a) (b)

Fig. 7. Etch patterns on two sides of a thin cleavage plate parallel to the prism face of synthetic quartz (× 280).

hydrofluoric acid of varying concentrations. Out of many experiments some were successful in producing spiral etch pits on the cleavage faces when etched in the solution containing 40 parts of hydrofluoric acid and 60 parts of water.

Attempts were also made to part the crystal parallel to the prism faces, and pairs of fractured faces more or less parallel to the prism faces were obtained. They were also etched and the etch patterns studied optically.

The investigations are described in two stages. Firstly, the studies on the etch patterns on the rhombohedral cleavages are recorded, and secondly, the etch patterns produced on the cleavage fractured prism faces of these crystals are described.

Observations

Etching of rhombohedral cleavages

Matched pairs of rhombohedral cleavages were simultaneously etched in hydrofluoric acid solution for a suitable period. Fig. 1 represents the etch patterns produced on a matched pair. The patterns are similar in shape and orientation to those produced on natural rhombohedral faces of quartz. A number of such rhombohedral cleavage faces were then etched in solutions of different concentrations. Fig. 2 represents the etch patterns obtained when matched faces were etched in a solution containing 40 parts of hydrogen fluoride to 60 parts of water for one hour. It is indeed very interesting to find the etch spirals along with the other etch pits on both the cleavage faces. The following points in Fig. 2(*a*) and (*b*) merit discussion:

1. That they are cleavage faces is revealed by the cleavage lines.
2. The etch patterns consist of the usual etch pits observed on natural rhombohedral faces and some spiral depressions.
3. Nearly one-to-one correspondence can be established in the number and position of both the etch pits as well as the spiral pattern.
4. Three spirals can be seen on each face out of which two are indistinct.
5. The etch spirals appear to be similar to the growth spirals on natural rhombohedral faces.
6. The nature of the spirals on both the faces is the same.

Fig. 3(*a*) and (*b*) represents magnified pictures of distinct spirals observed in Fig. 2(*a*) and (*b*) respectively. The profile of these spirals could not be investigated by multiple beam interferometry (Tolansky, 1948) as these were fractured faces and not perfect cleavage faces.

Etch patterns on the fractured prism faces

As reported in the literature, quartz has not even an imperfect cleavage along the prism faces. However after a number of trials, the authors were successful in obtaining fractured faces nearly flat and more or less parallel to the prism faces.

Fig. 4 shows the photomicrographs of the matched fractured prism faces etched in the hydrofluoric acid solution for eight hours. The shape and orientation of the pits in Fig. 4(*a*) are the same as those observed on natural prism faces, revealing thereby that the fracture has taken place more or less parallel to the prism faces. The shape of the pits in Fig. 4(*b*) differs from that of the pits in Fig. 4(*a*), which shows its matched face.

Attention is drawn to the following

1. The etch patterns on the matched fractured faces have one-to-one correlation in number and positioning of the pits.
2. The shapes of the pits differ on the two faces.
3. The faces appear quite flat as if they are natural prism faces, though in fact they are not. This is revealed by the shape of the etch pits produced.
4. Interferometric examination revealed that the faces have slight curvature, one being slightly convex and the other slightly concave.

These faces were further etched for two hours and the etch pattern on the same regions of Fig. 4(*a*) and (*b*) are given in Fig. 5(*a*) and (*b*) respectively. It is seen that the pits increased in size, but no new pits are developed, suggesting that the pits reveal the sites of dislocation in the crystal. This was further confirmed as follows.

1. A fractured prism face was etched and the etch pattern recorded as shown in Fig. 6(*a*). The etch pattern was removed completely by polishing the face. The polished face was then re-etched and the pattern recorded as shown in Fig. 6(*b*). The pits in Fig. 6(*b*) are located at the same sites as those on Fig. 6(*a*) revealing that they are dislocation pits.
2. A thin cleavage plate of thickness 0·02 mm was obtained by cleaving parallel to the prism face. It was then etched; the etch patterns recorded on the two sides of the plate are as shown in Fig. 7. One-to-one correspondence in the position of the pits can be traced. This supports the conclusion that the pits are dislocation etch pits.

The etch patterns described above are not very clear as they are on fractured faces which are not very flat.

It may also be mentioned that this is the first instance in the literature of a report of work on the etch patterns on the matched fractured prism faces of synthetic quartz which have no natural cleavage.

Discussion and conclusion

That the spiral etch pattern revealing the sites of the screw dislocations can be produced only when a suitable etchant is used is quite in agreement with the findings of Patel & Bahl (1965). That there is one-to-one correspondence in the position and also in the nature of the spirals produced on both the matched faces suggests that the production of these spirals is not due to a typical dissolution process as reported by Damiano & Herman (1959) and Lang (1957) but that they are produced at the terminations of screw dislocation lines as reported by Patel & Bahl (1965). The centres of the spirals indicate the sites of screw dislocations. The formation of the spiral patterns therefore indicates that the crystals may have grown according to the dislocation theory of Frank (1949).

The observations on (i) the matched fracture prism faces, (ii) the prism fractured faces before and after polishing, and (iii) the two sides of a thin flake having prism faces indicate that dislocation lines which exist in the crystal are cut into two by fracture along any surface of the crystal, even though the surface may not be a true low index plane as in the case of perfectly cleavable crystals. The slight difference in the shapes of the etch pits produced on the matched faces indicates that they are fractured faces. These faces had a slight curvature, one being slightly convex and the other concave. The pits produced on these matched faces would not be exactly of the same shape, because of opposite curvatures of the two faces, and this is what is observed. Thus it is clear that if two matched fractured faces, even though not quite flat, are etched simultaneously in the same medium, one-to-one correspondence in the number and position of the pits is produced, even though the shapes may differ.

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Irradiation Effects in Beryllia and Zinc Oxide

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Electron-diffraction and electron-microscope observations have been made on the effects of neutron irradiation and electron irradiation in an electron microscope on beryllia flakes and zinc oxide smoke particles. It is shown that the defects or excitations created in beryllia by electron irradiation interact strongly with neutron-induced defects to the extent that electron micrographs obtained with normal electron irradiation give a false impression of the form of the defect aggregates present. It is shown that high-intensity irradiation with electrons can transform normal α-BeO to the high-temperature, tetragonal β-BeO phase. The remnants of crystals partially evaporated by the electron beam may consist of fibres of α-BeO, fibres of β-BeO or plates of β-BeO. From single-crystal and oblique-texture electron diffraction patterns the structure proposed for β-BeO and its orientational relationships with α-BeO are confirmed. The significance of the formation of the β phase by electron irradiation is discussed in relationship to the possible nature of the defects in α-BeO responsible for the anisotropic expansion induced by neutron irradiation.

The erosion of thin near-perfect crystal plates of zinc oxide by neutron irradiation and the corrugation of the surface of such plates by electron irradiation are described.

Introduction

Electron microscope observations of the defects produced in beryllia, BeO, by neutron irradiation have been reported by Chute & Walker (1964), Wilks & Clarke (1964), Elston, Frisby & Labbe (1964) and others. These authors also reported in greater or lesser detail that electron irradiation in the electron microscope could produce visible defect aggregates in electron microscope images and could result in an evaporation of the beryllia crystals, leaving fibrous remnants with the fibres parallel to the *c* axis. These latter effects were described in more detail by Bisson (1963). The present paper reports some additional observations on these irradiation effects which suggest that some previous results may have been misinterpreted and provide further evidence on the nature of the defects involved. Also some further results are reported on the production by electron irradiation of the high-temperature β-BeO phase, recently described by Smith, Cline