responding atoms. The K–N distance of 2·89 Å is comparable to the value of 2·78 Å (mean of three distances: 2·70, 2·79, 2·83) found in $KAu(CN)_2$ (Rosenzweig & Cromer, 1959). The C–N distance of 1·157 Å (corrected for thermal motion) is also in good agreement with the C–N distance found in other cyanides – 1·15 Å in $K_3Co(CN)_6$, 1·15 Å in $As(CN)_3$ (Emerson & Britton, 1963), 1·15 Å in $P(CN)_3$ (Emerson & Britton, 1964) and 1·17 Å in $KAu(CN)_2$.

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Optical and Interferometric Studies on Thermal Etch Patterns of Potassium Chloride Cleavages

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Freshly cleaved potassium chloride has been thermally etched at temperatures near to its melting point. It is observed that the thermal etch patterns produced consist of (1) circularly terraced and (2) spiral etch pits. The topography of the thermally etched face and of the thermal etch pits is studied by multiple-beam interference fringes. Dissociation of circular terraces of the etch pits into two branches has been observed. It is shown by a schematic diagram that this may be due to the interference of circular terraces of etch pits originating at two dislocations very near to each other. The interference of a number of circular terraced pits is recorded. It is observed that some of the etch pits have a spiral structure for a few turns and then the terraces become concentric circles. The formation of spiral pits as well as pits having closed rings has been assumed to be due to the mechanism of the etching process as reported by Lang and has nothing to do with screw dislocations.

Introduction

It is well known that when crystals are heated to a temperature very near to their melting point, thermal etch pits, similar to the etch pits produced by chemical etching, are produced at the sites of dislocations. The dislocation theory of evaporation has been given by Cabrera & Levine (1956). The correlation between the thermal etch pits and the dislocations has been reported by Hirth & Vassamillet (1958). Patel, Bahl & Vagh (1965) have established for the first time one to one correspondence in the number and position of etch pits on matched faces of sodium chloride by etching one face chemically and the other thermally, indicating thereby that the pit formations originate from the crystal defects known as dislocations. Etch spirals pro-

(a)

(b)

Fig. 1. (a) Optical micrograph of the etch pattern produced on a (100) cleavage face of potassium chloride by heating for 11 hr at 740°C. (b) Multiple beam interferogram of (a) (× 280).

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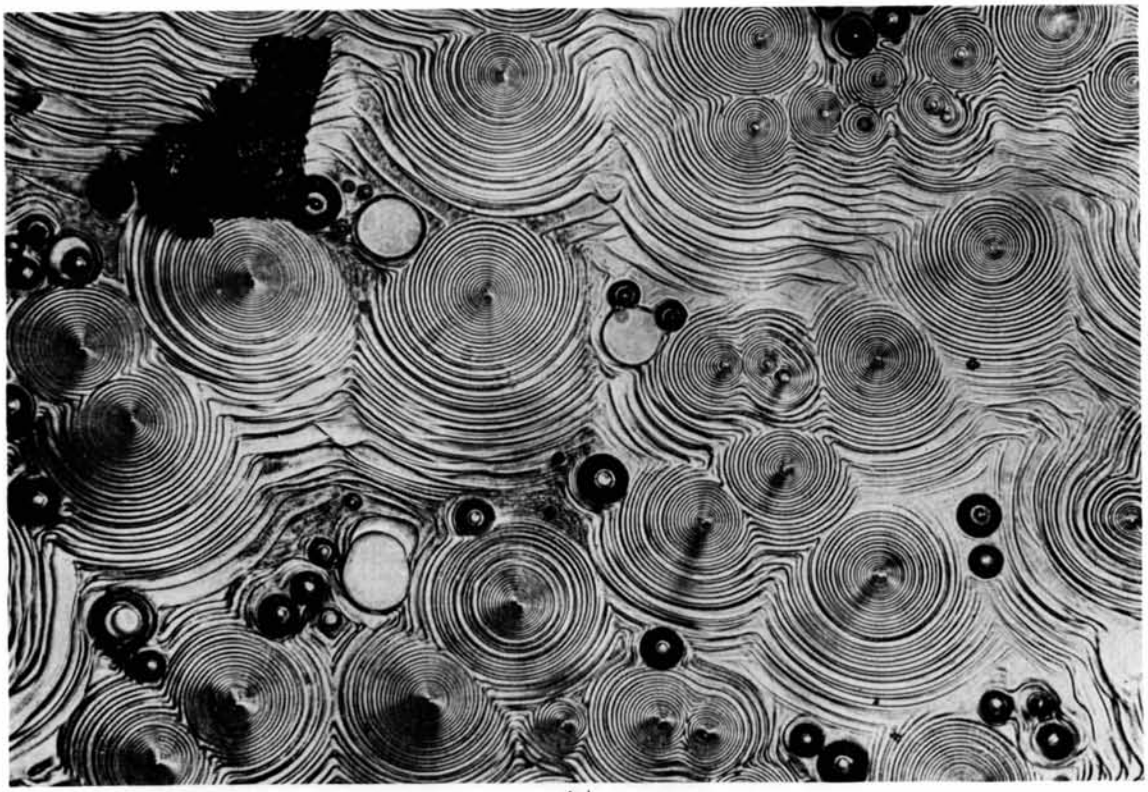

(a)

(b)

Fig. 2. (a) Etch pattern produced on a (100) cleavage face of potassium chloride by heating for 8 hr at 730 °C. (b) Multiple beam interferogram of (a) (× 280).

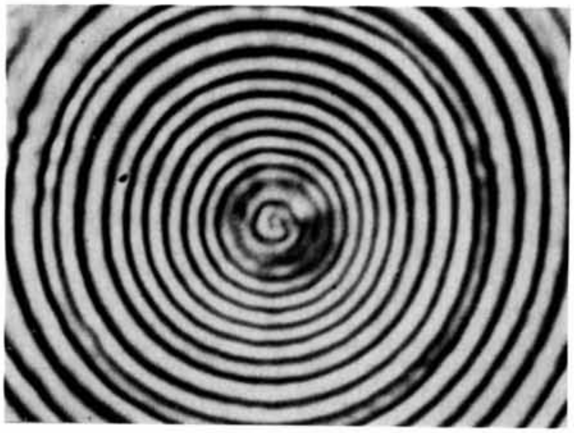

Fig. 3. One of the terraced pits of Fig. 2(*a*) (× 1310).

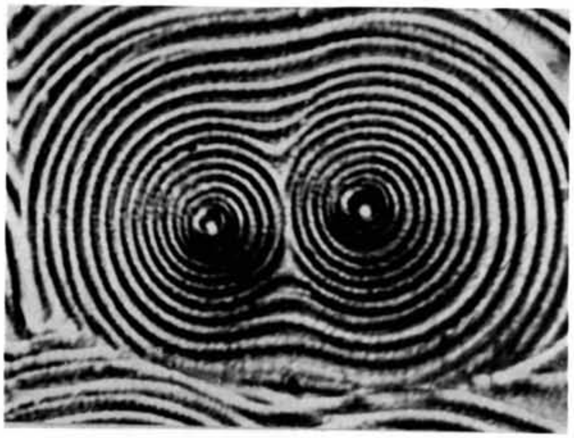

Fig. 4. Terraced pit showing a larger number of spiral turns (× 750).

Fig. 5. Terraced pit with neighbouring loops connected by branches (× 750).

Fig. 7.

Fig. 8.

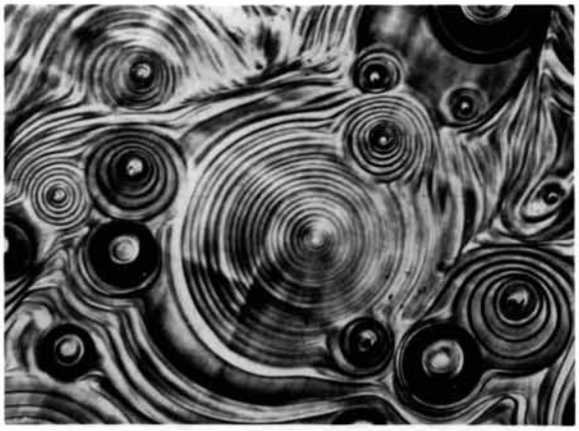

Fig. 9.

Figs. 7–9. Interference of circular etch fronts from three (Fig. 7), four (Fig. 8) and many (Fig. 9) centres (× 490).

duced by thermal etching on sodium chloride have been reported by Bethge & Keller (1960). Deo & Sharma (1964), by thermally etching sodium chloride cleavages, have shown the movement of dislocations in the crystal. Sunagawa (1960) has attributed the depression spirals observed on the natural faces of hematite to etching of screw dislocations in the crystal. Patel & Bahl (1965) have reported one to one correspondence in the number, position and nature of etch spirals produced on the matched cleavage faces of graphite by simultaneously etching them in sodium peroxide, indicating thereby that the etch spirals show the sites of screw dislocations in the crystal. Damiano & Herman (1959) and Lang (1957) have shown that spiral etch pits produced by etching should not necessarily indicate the sites of screw dislocations. While a considerable amount of work has been done on chemical etching of single crystals, it appears that the work reported in the literature on thermal etching of single crystals is very meagre. In the series of experiments carried out in this laboratory on thermal etching of alkali-halide single crystals, the authors observed some interesting etch patterns produced on potassium chloride cleavages. This paper deals with the observations on the thermal etch patterns made on these cleavages.

Experimental

Potassium chloride single crystals, obtained from Dr K. G. Bansigir, were freshly cleaved and the cleavages were heated on a platinum lid in a muffle furnace at a suitable constant temperature. After heating for the required time, the crystals were removed from the furnace, and the etched faces were silvered and examined optically as well as by multiple beam interferometry (Tolansky, 1948). As potassium chloride is hygroscopic, one has to avoid contamination with moisture, otherwise the surface features to be studied will be spoiled. In fact, in the monsoon season when the humidity was over fifty per cent, we could not work on these crystals at all.

Observations

Fig. 1(*a*) represents an optical micrograph of the etch pattern produced on a (100) cleavage of the crystal by heating it at a constant temperature of 740°C for 11 hours. It is clearly seen from the figure that the usual cleavage lines have been completely washed out and a complicated etch pattern is produced. The pattern consists of more or less circularly shaped point-bottomed etch pits of different sizes scattered all over the face. It is clearly seen that the circular layers, originating from the pits as a result of thermal etching, and receding from the point of nucleation of the pits, interfere with each other and thus form a complicated pattern. The topography of the etched face of Fig. 1(*a*) is revealed by the multiple beam interferogram [Fig. 1(*b*)] which was taken on the same region. The interferogram was obtained by keeping the optical flat, as far as possible, parallel to the surface. That this flat was so adjusted is shown by the circular fringes, which reveal the contours of the pits. The fringes show that the pits are inverted conical point bottomed pyramids. It can be seen from the interferogram that the depths of the pits differ and that they are not very deep. The deepest pit is about 3·5 microns deep; the other pits are shallower than this. A careful examination of the fringes over the pits reveals that they are circular. The fringes are not all concentric circles, but are slightly eccentric. The complicated nature of the surface, resulting from the interference of a number of pits, is beautifully revealed by the interferogram.

That the detailed information regarding the structure of the pits is not clearly revealed in Fig. 1 may be due to the fact that etching has been carried too far. A second set of experiments was therefore carried out in which the crystal cleavages were thermally etched for 8 hours at 730°C. Fig. 2(*a*) reveals the etch pattern thus produced. It is seen that circular pits more or less similar to those observed in Fig. 1, but with the structure slightly more resolved, are produced. The pits are of two different types; the small ones which appear darker are deep and the others, which are circularly terraced and comparatively large are shallower. The shallower pits appear to be all terraced, having more or less concentric circular terraces. As the pits grow larger, the terraces of neighbouring pits interfere with each other. The structure of the terraced pits is clearly revealed by the interferogram of Fig. 2(*b*). The interferogram reveals that the terraces form quite sharp steps. The region between the two concentric circles, forming the terraces, is not quite flat; this is clearly indicated by the nature of the fringes when they cross the terraces. The region appears to be curved, the middle position being at a higher level than the edges.

Fig. 3 is a magnified picture of one of the terraced pits of Fig. 2(*a*). It is interesting to find that the centre of the pit is of spiral nature while the periphery consists of closed circular loops. It thus appears that the pit, originating as a spiral depression for one or two turns, ultimately turns, as it grows bigger, into a pit having closed circular loops. Almost all spiral pits reveal the same characteristic. On critical examination of a large number of the pits described above, the spirals in some were traced up to a considerable number of turns before they became circular. This is shown in Fig. 4 in which the spiral can be traced up to six to seven turns. Close examination of some other pits (Fig. 5) reveals that the closed loops forming the terraces of the pits dissociated into two branches connecting the neighbouring closed loops.

As shown schematically in Fig. 6, such a pattern might be the result of the interference of circular etch fronts originating from two sources near to each other. In this figure two centres of initiation, *A* and *B*, close to each other, are selected and a few circular etch fronts are drawn with each as a centre. The resulting

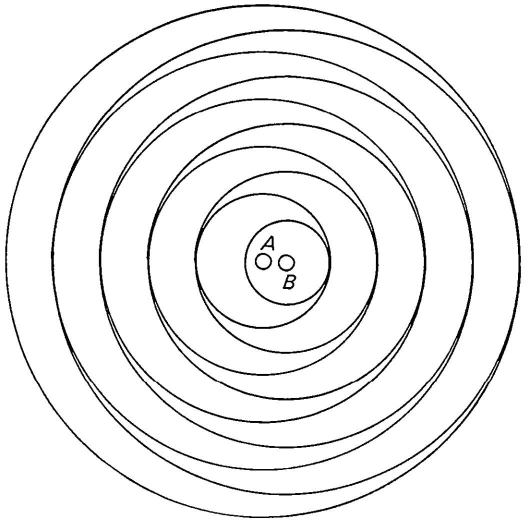

Fig. 6. Diagram of pattern due to interference of circular etch fronts from two centres.

pattern is clearly revealed in the figure, which is similar to the etch pattern shown in Fig. 5. Figs. 7, 8 and 9 reveal the patterns due to the interference of circular etch fronts from three, four and many centres respectively. It may be pointed out that when circular fronts from three sources interfere, it appears that a resultant etch front dissociates into three branches. In general it is observed that the number of branches in which a circular front dissociates is equal to the number of the centres the etch fronts of which interfere.

Discussion

Patel, Bahl & Vagh (1965) have shown that thermal etching produces pits at the dislocations in sodium chloride. It is therefore conjectured that terraced circular etch pits produced in the present work also indicate the sites of the dislocations in the crystals. Thermal etching has been shown (Patel, Bahl & Vagh, 1965) to produce circular etch pits, while in the present case circular etch pits with terraces are produced. That this may be due to the specific conditions in which the present experiments were carried out is showing by the change in the etch pattern observed on changing the conditions of etching in the present work.

Lang (1957) has explained the mechanism of the formation of spiral pattern and closed loops around an etch pit, produced by thermal or chemical etching. He has shown that no screw dislocations are necessary for producing spirals. Loops and spiral patterns have been produced by Damiano & Herman (1959) by etching the basal planes of zinc. They have explained the formation of the spirals and the loops as the result of dissolution of a step in the surface with the ends of the step pinned.

The spiral pits as well as pits having steps forming concentric rings observed in the present work may be the result of the specific process of dissolution described by Lang (1957), and it appears that it has nothing to do with the screw dislocations. The dissociation of the concentric rings into a number of branches suggests that the pattern might have been formed as a result of the interference of concentric rings from number of neighbouring sources, as indicated schematically in Fig. 6.

The authors take this opportunity to express their thanks to Dr K. G. Bansigir for supplying sufficient crystals for these investigations.

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