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INCLINED DISLOCATIONS IN ZINC CRYSTALS

By

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INCLINED DISLOCATIONS IN ZINC CRYSTALS

THE linear dislocation defects inclined to the cleavage plane of a zinc crystal, as revealed by etching, are reported here. Single crystals of zinc are grown from metal of high purity by Bridgman's method and cleaved at the temperature of liquid air. The freshly cleaved surface was etched by 0·05% iodine in ethyl alcohol and examined under a microscope. A number of hexagonal etch pits, which grow larger and deeper, with their edges getting rounded off on etching for longer times were observed. It has already been proved that these etch pits represent sites of dislocation defects running perpendicular to the basal plane of the crystal.

One interesting feature observed by us was that in some cases a small hexagon was seen inside a larger one. Figure 1 shows such a pit with the light profile running across for the measurement of its depth. Further examination shows that the inner and outer hexagons are not concentric. This suggests that the pits may be due to the etching proceeding at an angle instead of vertically downwards due to the dislocation defects inclined to the surface. If the eccentricity a and the depth of the pit d are measured, then by applying the relation $\tan \theta = d/a$ the inclination θ of the dislocation defect can be calculated. Similar measurements have been made by Patel[1] on diamond. In the present case three such pits were observed and measurements gave a constant angle of 36° in all the three cases. Even though no conclusions can be drawn from three pits, it suggests it may have some bearing on the growth rate of the crystal. Experiments that are in progress on crystals grown at different rates may give some information on this topic. Recently, Grinburg[2] has shown that rates at which a crystal is pulled has some influence on the orientation of the crystals.

Another interesting feature observed by us is the partly developed pits along the edges of a twin lamella shown in Fig. 2. This is expected because of the heavy distortion taking place in the process of twinning which must have occurred at the time of cleavage.

FIGS. 1-2

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Some Interesting Growth Features on Antimony Crystals*

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Various growth features observed on crystals of antimony grown from the vapour phase are presented and discussed. The crystals grown in vacuum furnace develop triangular faces which are identified as the (111) planes of antimony by cleavage and etch features. These crystals show various types of growth triangles, all oppositely oriented to the edges of the main face. The density distribution of these features has been studied and interpreted on the basis of dislocations. It has been observed that each triangular hillock is the growth of the (111) facet on the cleavage plane and that these hillocks are grown at sites of dislocations. The linear arrays observed have been identified as the edge dislocations along the low angle boundaries.

IN recent years, the study of growth of metal crystals from liquid and gaseous phases has received considerable attention and a good deal of information has been published. A large number of methods of growing single crystals from melt and vapour are also well known. In this laboratory, work has recently been undertaken on growing single crystals of metals and optically studying their surfaces under different conditions of etching. The various growth features observed on single crystals of antimony grown from vapour are reported in this paper and the results on etching the surfaces so as to reveal dislocation sites are also presented.

Method of Growing Crystals

The crystals studied in the present investigation were grown in the following manner. The pure metal was kept in a graphite crucible and covered with a graphite lid The crucible was placed in a silica tube $1\frac{1}{2}$ in. diam., which formed the core of the furnace. One end of the tube was closed and the other end was connected to an exhaust pump. The measurement of the temperature was carried out by means of a CP-A thermocouple, the junction of which was kept nearest to the crucible. The system was evacuated to a pressure of 0 1 mm. Hg and the furnace was switched on. The temperature was gradually raised to 650°C. and maintained at that temperature for 3 hr The system was then cooled at a very gradual rate by regular reduction of current effected by means of a gear mechanism designed in this laboratory. On cooling down to room temperature, the tube was opened and the crystals removed carefully.

Crystals were grown on the lower surface of the graphite cover and the sides of the silica tube. The crystals exhibit shining triangular faces of 1 mm. side and show various features.

Results and Discussion

That these faces are the (111) planes of antimony crystal could be proved from the fact that it was possible to cleave the crystal after cooling it to liquid air temperature by means of a sharp blade parallel to these planes. The cleaved surface is plane and highly reflecting so that the interferograms could be taken on these surfaces. Further, the shape of the etch pits on the surfaces also proves that these are the (111) planes of the crystal. The crystals were very delicate and often broke due to high brittleness of antimony; hence, they were mounted with great care on glass plates for examination. Fig. 1 shows a typical face grown by the method described. A number of triangles, oppositely oriented to the edges of the face itself, were seen. If the process of cooling was fast, striations suggesting mosaic structure were also observed. In many cases, a number of triangles arranged in linear arrays, intersecting rows (Fig. 2) and sometimes 'Y' shaped rows were observed under high magnification. The 'Y' shaped line observed in Fig. 3, when seen under high magnification, was seen to consist of a number of small triangles and in all cases the triangular features were oppositely oriented to the face itself.

Examination with light profiles showed that all these triangles are elevated hillocks and in no case pits. The heights, sizes and the density of these hillocks vary very largely for different specimens and also in different regions. The densities vary from 3×10^4/cm.^{2} to 7×10^4/cm.^{2}. To a great extent this depends on the rate of deposition. Furthermore, the size of the growth hillocks also depends on the rate of deposition, the larger ones being obtained at slower rates.

The nature and the distribution of these features suggest that they may be in some way connected with dislocations which play an important role in the growth of crystals. Moreover, the density distribution of these was of the same order (10^4/cm.^{2}) as that of the etch pit distribution determined by Wernick *et al* [1] on the cleavage plane of antimony. The distribution of these growth layers along the intersecting rows, shown in Figs. 2 and 3, is of particular interest.

According to Burgers[2], the boundary between two crystal planes, differing in orientation by a small rotation θ about an axis in the boundary, consists of a set of edge dislocations, parallel to the axis, of regular separation $D = b/\theta$, where b is the

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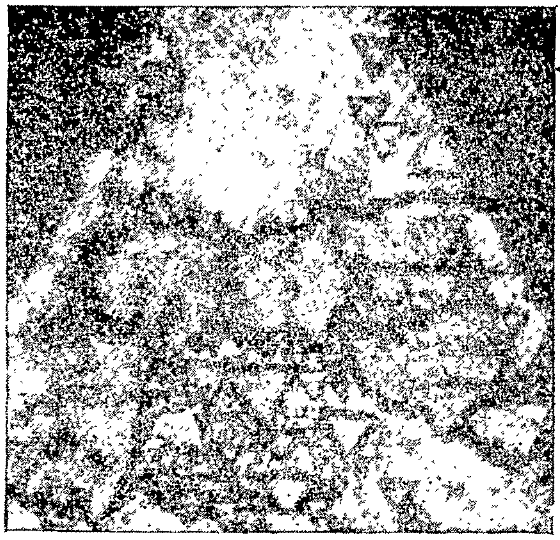
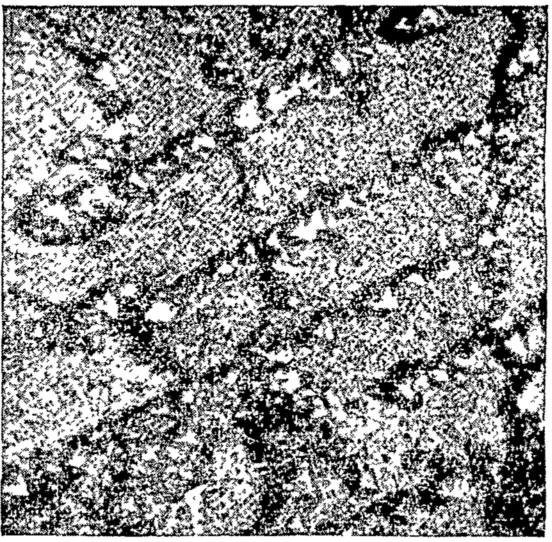


Fig.1

Fig.2



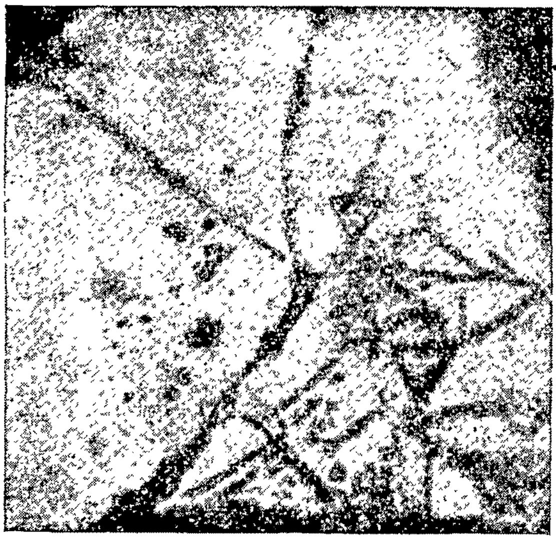


Fig.3

Fig.4

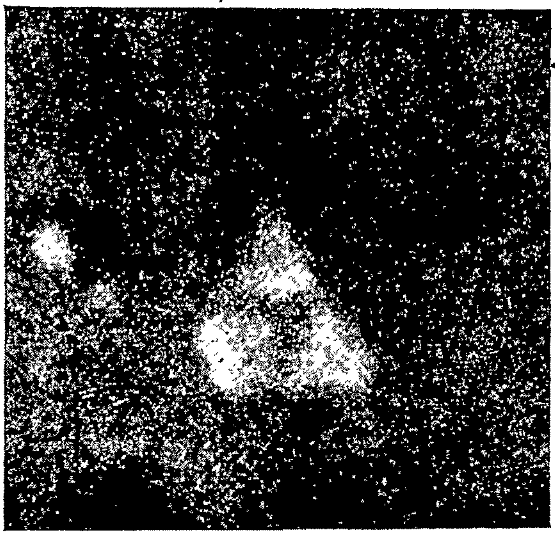


Fig.5

Fig. 1 — Naturally developed (111) face of antimony showing the growth features (×90)

Fig. 2 — Rows of growth triangles on the (111) plane of antimony (×460)

Fig. 3 — Another as-grown face showing the growth features (×170)

Fig. 4 — The face shown in Fig. 3 after etching (×170)

Fig. 5 — Development of the etch pit at the centre of a growth triangle (×1900)

appropriate lattice translation vector. If the rows of growth triangles represent the low angle boundaries, they must have a linear density in agreement with the relation derived by Wernick *et al.*[1] in the case of etch pits. That is to say that, for the three intersecting rows shown in Fig. 3, the linear density n along the three rows A, B and C should obey the equation

$$n_A = n_B + n_C \qquad \ldots\ldots \quad (1)$$

when we consider the two intersecting rows (Fig. 2), $n_B = 0$, hence

$$n_A = n_B \qquad \ldots\ldots\ldots \quad (2)$$

i e in the case of two intersecting rows the linear density should be equal. These relations were found to be true in many cases, which lends support to the idea that these rows mark the dislocation tilt boundaries normal to the (111) plane.

This conclusion has further been confirmed by etch features obtained on the faces. Various workers[1,3-5] have developed different reagents which reveal the dislocation sites in the form of etch pits. The etchant developed in this laboratory by the previous workers[5], consisting of a mixture of tartaric acid and nitric acid, was found to be suitable for the work, for the action was slow enough and longer times of etching could be tried keeping the surface bright. In general, a time of c $1\frac{1}{2}$ min. was required for the development of the pits which is characterized by the appearance of bubbles on the surface.

The various etch features are shown in Figs. 4 and 5. Fig. 4 shows a natural face showing the growth triangles, and Fig. 5 shows the same after etching It is seen that rows of etch pits have been developed at the sites of the growth triangles which resemble those observed by Wernick *et al.*[1] on antimony and those observed by Vogel *et al* on silicon, which have been proved to be the low angle boundaries. It may be observed that these rows are developed at sites where the growth triangles are seen. One-to-one correspondence between the growth and etch figures is possible in many cases, even though some etch pits are developed at certain sites where no growth triangles are seen.

As the etchant reacts on the surface, the growth layers are dissolved gradually and etch pits appear. The exact time at which the growth layers disappear and the pits appear is difficult to judge. However, it has been possible, in a few cases, to show the development of etch pits at sites of growth triangles, as can be seen from Fig. 5. This observation provides further evidence that the latter are formed at dislocation sites It is interesting to note that in all cases the etch and growth features are oppositely oriented.

The observations on etching on a cleavage plane are in agreement with those reported earlier A twin lamella does not develop any pit in its body, for it is a different plane, even though one edge is vigorously etched so as to form grooves which is due to the large free energy available at these sites due to twinning. Further, with the etchant reported here, a fine structure is seen in twin lamella similar to those observed with super-exol[1].

It is thus possible to identify the growth features observed on the antimony crystals as the oriented growth of the (111) facets on the (111) plane at the sites of the dislocations The mechanism by which the change of orientation of the growth facet takes place is not yet known. It should be pointed out, however, that no spirals are seen in these crystals. As regards the rows of triangles observed, they have been identified as sites of edge dislocations which do not have the screw component to promote the spiral growth In the case of randomly distributed triangles, which may be the sites of screw dislocation, the absence of spiral may be due to the fact that the crystals are grown under high supersaturation which is steadily increasing as the temperature is decreased.

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