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# DISLOCATION IN MECHANICALLY TWINNED CALCITE CRYSTALS

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**Abstract**

**Full Text**

## **CRYSTALLOGRAPHY**

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# **DISLOCATION IN MECHANICALLY TWINNED CALCITE CRYSTALS**

*(Presented by Academician A. V. Shubnikov, 10 VI 1957)*

It is usually considered that, during plastic deformation of crystals by twinning—especially crystals such as calcite and sodium nitrate—the deformed part of the crystal differs from the undeformed part only in the orientation of the atomic planes.

It seems to us that natural growth twins should indeed differ in no way from the parent part of the crystal except by the symmetrical orientation of the atomic planes of the crystal lattice. As for twins formed mechanically under the action of external forces, the portion of energy remaining in the deformed crystal must lead to substantial changes in its state.

Let us make use of the investigation of one of the structure-sensitive properties, which, as is known, include mechanical strength, creep, internal friction, solubility, and others. We chose to consider solubility and the formation of etch figures, and selected calcite as the object of study, since it is a crystal that twins readily.

1. A calcite crystal was twinned mechanically and immersed for an instant, by a side parallel to the principal section of the twinned crystal (the shear plane), in a weak solution of hydrochloric acid. This etched face was observed and photographed in an MIM-6 metallurgical microscope. In Fig. 1 it can be seen that, in the twinned part of the crystal, the etch spots are larger and the entire region of deformation is darker than the undeformed part of the crystal. This indicates that part of the energy absorbed by the crystal that has undergone plastic deformation is distributed uniformly throughout the whole volume of the deformed part of the crystal. The deformed part of the crystal is, as it were, in an elastically stressed state. An analogous phenomenon had earlier been found by Straumanis <sup>(1)</sup> in studying the mechanism and rate of dissolution of zinc single crystals. He found that twins on a face dissolve most intensely.
2. A twinned calcite crystal was lowered into a solution of hydrochloric acid of higher concentration. In Fig. 2 a photograph is presented of the etched face (the shear plane) of this crystal. In this photograph it is evident that the whole face has become covered with small etch figures arranged symmetrically with respect to the boundary of separation of the twin, while

the intermediate region <sup>(2)</sup> has been etched especially strongly. It seems to us that these etch figures resemble “hillocks” more than pits. In considering Fig. 2, special attention should be paid to the linear dark etch spots in the intermediate region, which are directed, as it were, along crystallographic planes in the original and twinned parts of the crystal. We believe that these linear dark spots and the entire dark line along the boundary of separation of the twinned crystal represent etch spots associated with the emergence of dislocations onto the surface of the twinned crystal.

For many years there has existed the supposition that a correlation exists between the density of dislocations in a crystal and etch figures. Vogel and co-workers <sup>(3)</sup> were the first to show most convincingly that the number of etch figures at a small-angle grain boundary in germanium almost coincides with the calculated number. The photograph given in their work is, to some extent, similar to Fig. 2. At present there are already a number of works <sup>(4–6)</sup> devoted to the formation of etch figures in deformed metallic and ionic crystals. The authors suppose that in all cases the etch figures were located at dislocations.

It is known that various imperfections in the structure of crystals include vacant sites in the crystal lattice, foreign atoms in the lattice, disturbances in the stacking of close-packed planes of crystals, and, in particular, dislocations. During plastic deformation of crystals, new imperfections are produced, which should be concentrated at the boundaries of the deformed and undeformed parts of the crystal.

It seems to us that Fig. 2 confirms this supposition.

3. R. I. Garber <sup>(7)</sup> showed that in the process of plastic deformation by twinning of calcite there are four stages, namely: a) the stage of elastic deformation: the deformations are small and obey Hooke’s law; b) the stage of formation of “elastic twins”: the wedge-shaped form of the twinned part and the proportionality of its dimensions to the applied force; c) the stage of formation of a thin interlayer of a stable twin; d) the stage of thickening of the twin at the expense of the main part of the crystal, the interface between the twinned and undeformed part of the crystal moving parallel to itself.

It was shown above that during plastic deformation by twinning the dislocations are concentrated in the intermediate region, especially at the interface. It was of interest to determine whether the boundary, in its motion, captures all the dislocations or whether some of them remain in the deformed part of the crystal. For this purpose, a twinned calcite crystal, etched in a solution of hydrochloric acid, was carefully examined under a microscope at various magnifications. In different places of the twinned part of the crystal, groups of etch spots were found, always arranged parallel to the interface. In some places the spots are arranged discontinuously over the whole width of the specimen (10 mm), in others—as separate groups along lines running parallel to the interface.

Figure 3 presents a group of continuous spots, the axes of which are directed

parallel to the interface of the twinned crystal. In addition, the axes of the spots are intersected by a set of linear parallel spots at an angle to the axes.

In some cases, in the twinned region after etching of the crystal surface, groups of etch figures were found in the form of so-called negative crystals of rhombohedral shape of different sizes, also arranged parallel to the interface of the twinned crystal (Fig. 4).

From our observations the following conclusions may be drawn:

1. During plastic deformation of calcite single crystals by twinning, part of the absorbed energy is distributed uniformly throughout the entire volume of the deformed part of the crystal, while the greater part of the absorbed energy is concentrated at the boundaries of the twins.
2. The greater part of the dislocations is generated in the process of plastic deformation and is collected mainly at the interfaces of the twinned crystal, and also in the deformed part of the crystal along the lines of displacement of the interface, parallel to it.
3. In some cases, dislocations in the deformed part of the crystal appear after etching in the form of so-called negative crystals; in other cases—in the form of continuous etch spots, the axes of which are directed parallel to the interface.

Fig. 1. Twinned calcite crystal etched in a weak HCl solution. is the twinned part of the crystal. 135×

Fig. 2. Twinned calcite crystal etched in a strong HCl solution. 200×

Fig. 3. Group of continuous etch pits in the twinned part of the crystal, their axes directed parallel to the boundary of separation. 200×

Fig. 4. Groups of etch figures in the form of so-called negative crystals in the twinned part of the crystal. 660×

4. The displacement of the interface during plastic deformation by twinning occurs not continuously, but discretely, according to the scheme displacement—pause. Evidence for such a mechanism of interface displacement is provided by the series of positions of the interface recorded in Figs. 3 and 4 in the form of a group of etch spots arranged parallel to it. This fact is also confirmed by the fact that, as is known, twinning is accompanied by jumps in the shear stress; on the load–elongation curve there are peaks associated with the discreteness of twinning.

The obtained traces of the interface in the form of a series of spots of etch figures indicate that during the pause some of the dislocations become fixed at temporary “positions” of advance of the boundary front and remain there. Continuing deformation (twinning) generates new dislocations, which partly move together with the interface, and partly become fixed at intermediate “positions.”

5. The two types of spots—discrete and continuous—are possibly associated with two types of dislocations, simple and spiral.
6. The distance between etch figures is  $\sim 10^{-4}$  cm, which agrees with the value given in work <sup>(3)</sup>.

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*Note: Figure translations are in progress. See original paper for figures.*

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