# PHYSICS

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## Abstract

## Full Text

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# ON SOME PHENOMENA OBSERVED DURING THE DEFORMATION OF ANTIMONY SINGLE CRYSTALS

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Studying the process of twinning in antimony single crystals during their mechanical deformation, we used a method of selective etching that makes it possible to reveal dislocations in the crystal. In this way it is possible to observe subtle effects accompanying plastic deformation of the crystal, the study of which is at present inaccessible by other methods of investigation.

1. It is well known that a twin lamella produced mechanically can be removed if a force is applied to the crystal in the direction opposite to that of the force which produced this lamella. The process of twinning–detwinning can be repeated many times, and it is considered that when the twin lamella is removed the crystal lattice at this place is completely restored and does not differ from the crystal lattice of the parent crystal.

On the other hand, R. I. Garber, carrying out repeated twinning of calcite, came to the conclusion that each subsequent formation of a twin lamella requires an increase in the applied force, i.e., the process of twinning–detwinning takes place with strengthening. In this case, however carefully the formation or removal of the twin lamella is carried out, in the end destruction of the crystal occurs (^1). This makes it possible to suppose that during detwinning complete restoration of the crystal lattice does not occur. However, the remaining defects must not be very large, since they are not detected by the X-ray method.

To detect these small defects we used the method of selective etching. Single crystals were obtained from antimony having an initial purity of 99.5% and then subjected to 120-fold zone purification. The specimens were cleaved from the single crystal along the cleavage plane (111) and had the form of disks 2–3 mm thick and 10–15 mm in diameter. Twin lamellae were produced by bending such a plate. Detwinning was carried out by bending the plate in the opposite direction. Observation was made on the cleavage plane. To reveal dislocations an etchant of the following composition was used: 9 parts by volume of concentrated nitric acid and 4 parts of distilled water.

Experiments on etching crystals for the purpose of revealing dislocations must be

Fig. 1

Figure 1: Fig. 1

approached very cautiously, since often, in addition to the places where dislocations emerge on the crystal surface, the etchant also reveals various surface and bulk defects having nothing in common with dislocations. Therefore, each time it is necessary to make sure that the etchant used is indeed a selective etchant and that all etch pits are due to dislocations. For this purpose we carried out a series of experiments (successive etching, splitting of crystals and etching of two cleavage planes, etc.) which allowed us to establish unambiguously that the etch pits formed are due to linear defects, i.e., dislocations.

Figure 1 shows photographs of the etching pattern of two surfaces formed when the crystal was cleaved. It is clearly seen that the photographs

**Fig. 1.** Etching pattern on two cleavage surfaces of an antimony single crystal. 450×

are identical, and that each etch pit in the left photograph corresponds to an etch pit in the right photograph. Upon successive etching of the crystal, the shape of the pits and their number do not change; only their size increases. This experiment, as well as others, shows unambiguously that the etch pits are due to dislocations. (Etching was carried out by immersing the crystal in the etchant for a time ranging from several seconds to several minutes, followed by washing the specimen in distilled water.)

Fig. 2 Fig. 3

**Fig. 2.** Dislocations that appeared at the place where there had previously been a twin interlayer. In the lower part of the figure, the continuous band is the remaining twin interlayer. 600×

**Fig. 3.** Intersection of the boundary between mosaic blocks by a twin interlayer. 600×

The results of an experiment on the detwinning of a stable twin interlayer are shown in Fig. 2. The thin twin interlayer was partially removed by mechanical means. The part of the interlayer remaining after detwinning is visible in Fig. 2 as a continuous band. Along the continuation of the twin interlayer, i.e., in the places where it had been before detwinning, there are etch pits—dislocations. Complete removal of the twin interlayer does not remove the dislocations; they also appear in all places where the twin interlayer had been. Thus, detwinning does not lead to complete restoration of the crystal lattice, but leaves traces in it in the form of lattice defects. These defects must hinder further twinning and cause hardening of the crystal.

It should be noted that V. M. Kosevich ($^2$) also observed the formation of dislocations upon removal of a twin interlayer. However, in his work it is asserted

Fig. 4. Dislocations arranged along slip lines. 340×

Figure 2: Fig. 4. Dislocations arranged along slip lines. 340×

that this formation is associated with defective places in the crystal, for example with places where twin interlayers intersect, with inclusions, etc. Our experiments show that this phenomenon has a more general character, and that dislocations form in all places of the crystal where there had been a twin interlayer, regardless of whether macrodefects were present there or not.

2. As theoretical calculation ($^3$), as well as experiment ($^4$), shows, elastic twins are not sensitive to the misorientation of two blocks if the angle between them is not more than 2–3′, and pass freely through the boundary between them. Residual twin interlayers are even less sensitive to the misorientation of neighboring blocks; they easily cross the boundary between blocks when the angle between them is several degrees ($^5$). However, if the etching method is used to study this phenomenon, it can be found that in reality the residual twin interlayers sense the boundary between blocks, even if the angle between them is less than 1/2°. Figure 3 shows a photograph of such a boundary, which is crossed by a twin interlayer.

**Fig. 4.** Dislocations arranged along slip lines. 340×

Having measured the density of dislocations at the boundary and used the well-known formula ($^6$)

$$\frac{b}{D} = 2\sin\frac{\theta}{2},$$

where $D$ is the distance between two neighboring dislocations at the boundary, $b$ is the Burgers vector, and $\theta$ is the angle of misorientation between the blocks, we obtain that $\theta = 30'$. It is clearly seen that the twin interlayer, passing through the boundary and not changing its shape or width at it, strongly damages the boundary, forming a large number of new dislocations near it.

This phenomenon proves to be general, and it testifies that twin restructuring of the crystal lattice is very sensitive even to small imperfections in it.

3. The generally accepted point of view ($^7$) is that antimony is a brittle crystal, that slip is absent in it, and that all plastic deformation is carried out only by twinning. However, single crystals of antimony of very high purity, as in our case (we believe that the purity of the antimony that we had after 120-fold zone refining was greater than 99.999%), possess quite different mechanical properties than single crystals prepared from antimony of 99.98% purity or similar,

with which one has usually dealt in practice up to now. Single crystals prepared from high-purity antimony possess greater plasticity, bend easily without fracture, and in their mechanical properties resemble zinc single crystals. This gives grounds for thinking that in such pure single crystals, in addition to twinning, slip occurs.

To clarify this question, a single crystal strongly predeformed by bending was etched with the etchant indicated above. Figure 4 shows photographs of the etched cleavage plane of such a crystal. Three systems of slip lines are clearly visible, along which dislocations are located. The slip lines coincide with the $\langle 1\bar{1}0 \rangle$ directions.

In conclusion, we consider it our pleasant duty to express our gratitude to V. G. Bengus and F. F. Lavrent' ev for discussion of the results.

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