MOTION OF DISLOCATIONS IN ANTIMONY CRYSTALS

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MOTION OF DISLOCATIONS IN ANTIMONY CRYSTALS

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1. At present two principal methods for studying the motion of dislocations in crystals have been developed and are being successfully applied: electron-microscopic and selective-etching methods. In electron-microscopic investigations, processes occurring in thin layers of metal are studied, and it is not always possible to transfer the results of these investigations directly to bulk specimens. On the other hand, the application of the selective-etching method to bulk specimens has so far not led to results unambiguously demonstrating the motion of dislocations in metals, as it has for ionic crystals ([1,2]). An explanation of this could be the suggestion, advanced by some authors ([3,4]), that the appearance of pits at dislocations during chemical etching of metal surfaces, unlike etching of ionic crystals, is due to decoration of dislocations by impurities, and not to weakening of the bonding forces between atoms in the dislocated region.

A study of the plastic properties of single crystals of antimony of very high purity, recently carried out by the authors of the present work ([5]), showed that deformation of single crystals grown from very pure antimony and having a low dislocation density led to the formation of dislocations whose density was practically the same as in deformed single crystals prepared from poorly purified antimony. This makes it possible to assume that dislocations in metals can be revealed even in the absence of decoration, and gives hope of detecting the motion of dislocations also in metallic crystals.

The motion of dislocations can be studied by the selective-etching method either during etching of a specimen under load ([1,2]), or by repeated etching of a previously etched and then deformed crystal ([6]). In this case two types of etch pits are observed: sharp-ended pits, corresponding to the places where dislocations were located at the moment of etching, and flat-bottomed pits, corresponding to places where dislocations had previously been located and from which they had moved under the action of mechanical stresses or other causes. By this method, despite its comparative simplicity, it is possible to observe fine details of the process of dislocation motion in crystals.

In the present work the selective-etching technique was applied to the study of dislocation motion in single crystals of antimony. The etching procedure for these crystals was described in ([7]). A preliminarily etched crystal was fastened

Fig. 3

Figure 1: Fig. 3

in a glass cuvette in such a way that a small gap remained between the surface under investigation and the bottom of the cuvette, into which the etching solution could enter. A stirrer was used to mix the solution during etching. A load sufficient to move dislocations was applied to the crystal. The cuvette was placed on the stage of an MIM-7 microscope, which made it possible

To the article by L. M. Soifer and V. I. Startsev, p. 1084

Fig. 1. Motion of dislocations in antimony crystals, 450×.
Inset 600×

Fig. 2. Dislocation "loops" expanding from twin interlayers, 600×

To the article by A. P. Terent' ev, I. G. Mochalina, and E. G. Rukhadze, p. 1130

Fig. 2. X-ray diffraction pattern of polyamide

continuously observe and photograph the process of motion of individual dislocations.

2. The result of the motion of dislocations can be seen in Fig. 1. The large triangular pits, corresponding to the initial position of dislocations, which had previously been pointed, have now become flat-bottomed. From each such pit there extends an intermittent, narrowing trace consisting of flat-bottomed pits (see inset to Fig. 1). Each end of a trace is characterized by the presence of a pointed pit, which gives the position of the dislocation line at the end of the motion. These traces are trajectories of motion of individual dislocations during the etching process and coincide with the directions $\langle 1\bar{1}0\rangle$.

A crystallographic consideration shows that in antimony there are three possible slip systems, which are characterized by three planes $\{11\bar{1}\}$, emerging onto the observation plane (111) in the form of three slip directions $\langle 1\bar{1}0\rangle$. All these systems are realized during deformation of antimony, as was shown earlier ([7]). In the present experiments motion of dislocations was observed along all three indicated slip directions. Thus, the existence in antimony of slip with the slip elements given above may be regarded as established.

Fig. 3. *a*—interaction of unlike dislocations, 600×; *b*—zigzag character of dislocation motion, 600×.

Measurements show that the average magnitude of a jump, i.e., the distance traversed by a dislocation line between two stops, is 0.2–10 μ (the lower limit is set by the resolution of the microscope).

Fig. 4. *a*—multiplication of dislocations, 600×; *b*—motion of a low-angle boundary during annealing, 450×.

Fig. 4

Figure 2: Fig. 4

The velocities of motion observed by us lay within the range $1 \cdot 10^{-7}$–$3 \cdot 10^{-5}$ cm/sec. With an increase in the applied stress the velocities of motion increased. At present we are carrying out experiments to determine the quantitative characteristics of dislocation motion in antimony.

3. In papers ([8,9]) it was shown that the twin boundary—matrix crystal has a dislocation nature, and the results of papers ([10,11]) indicate that internal stresses are localized at this boundary. As experiment shows, the magnitude of these stresses is sufficient for dislocations to move away from the boundary of the twin interlayer. Fig. 2 presents a typical picture of the motion of dislocations departing from twin boundaries. The two broad dark bands are ⋯

twin interlayers. To the left of each twin a developed band is visible, in which lie dislocations emerging at a small angle to the observation surface. From the twin boundaries dislocation loops diverge, expanding in their slip planes.

It should be noted that expansion of loops from the twin boundary is observed even without applying external stresses to the specimen. Apparently, the motion of dislocations is caused by stresses present in the accommodation region near the twin boundary.

As a rule, a wide twin interlayer (of thickness $\sim 30\,\mu$) has a low linear density of loops, $\sim 5 \cdot 10^1$ cm^{-1}, whereas a narrow twin interlayer (3–5 μ) has a density $\sim 3 \cdot 10^3$ cm^{-1} (the total dislocation density in the undeformed specimen was 10^3 cm^{-2}). These data apparently make it possible to conclude that the narrow twin interlayer is more highly stressed than the wide one.

The method employed makes it possible to observe the interaction of dislocations. Fig. 3a gives an example that may be interpreted as the interaction of dislocations of opposite sign. Dislocations moving in one slip plane toward one another are annihilated when they meet.

It must be noted that the motion of dislocations is not always observed exactly along the crystallographic slip directions. The trajectories of motion are often curvilinear. Apparently this can be explained as follows. Since the three slip directions $\langle 1\bar{1}0 \rangle$ in the observation plane (111) in antimony make an angle of 60° with one another, then upon application of a tangential stress in the plane (111) in an arbitrary direction there will always be components along all three slip directions. Even if the tangential stress is normal to one of the directions $\langle 1\bar{1}0 \rangle$, in the other two directions its components are always nonzero. Thus, if a dislocation, while moving in one slip plane, encounters an obstacle and the magnitude of the stress is insufficient to overcome it, it may pass into another plane and move in a new direction. As a result the motion will be zigzag-like, with segments parallel to the directions $\langle 1\bar{1}0 \rangle$. An example of such dislocation motion is seen in Fig. 3b at the place marked by an arrow. Very often the change in the direction of dislocation motion occurs over distances smaller than those that can be resolved in the microscope. As a result, a trajectory of motion of arbitrary shape is obtained.

4. According to the ideas first developed by Cottrell ([12]), dislocations in crystals are surrounded by a cloud of impurity atoms. These clouds block the dislocations, thereby reducing their mobility. However, if one takes a freshly grown crystal in which the dislocations have not yet had time to become locked by a Cottrell cloud, then, during etching under load, intensive multiplication of dislocations is observed. Fig. 4a shows a typical case of dislocation multiplication. Three pairs of loops are clearly visible, moving away from the main etch pit along the three slip directions. Such multiplication of dislocations is not observed if, before etching, the crystal is first annealed for a short time at a temperature of 300–400°, or if it is allowed to stand for several months at room temperature. In this case the mobility of dislocations also decreases sharply. This experiment, it seems to us, shows the influence of Cottrell atmospheres on the mobility of dislocations. It is interesting that the "aging" of crystals described above increases the intensity of etching at dislocations, which is also evidence of the formation of Cottrell clouds.

5. The motion of dislocations in single crystals of antimony can be demonstrated by yet another experiment. As is well known, even carefully grown single crystals have a mosaic structure. The angles of disorientation between neighboring blocks are small and equal to tens of seconds or less —

a few minutes. The boundaries between blocks (low-angle boundaries) are sites of concentration of dislocations. The dislocation density at a boundary is directly related to the magnitude of the angle of rotation of one block relative to another. If a crystal with such boundaries is annealed, displacement of the boundaries and of individual dislocations occurs. Such a case is shown in Fig. 4b. The crystal was etched before annealing, then annealed at a temperature of 460°C for 3 hours and etched again. In the photograph the dislocation nature of the boundaries and their displacement as a result of annealing are clearly visible. The flat-bottomed pits represent the positions of dislocations at the boundary before annealing, and the pointed pits—after annealing.

In conclusion we consider it our pleasant duty to express our gratitude to F. F. Lavrent' ev and V. Z. Bengus for valuable discussions.

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