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INVESTIGATION OF THE MECHANICAL TWINNING OF ANTIMONY SINGLE CRYSTALS BY NANOINDENTATION

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The twinning of antimony single crystals in nanoindentation has been investigated. The role of twinning in the formation of the impression of the Berkovich pyramid on the surface (111) of antimony single crystals has been established. A new method of investigation of the mechanical twinning of single crystals has been developed. For the first time, the duration of the process of twinning has been determined experimentally by a direct technique.

As of now, the role of twinning in the formation of the indenter impression in testing of single crystals for hardness has been studied adequately [1–5]. The development of nanoindentation technology [6, 7] has opened up new possibilities of studying such a process of plastic deformation as twinning. Therefore, it is of interest to study the effect of twinning on the process of formation of the impression of the Berkovich pyramid on the surface (111) of Sb single crystals and to develop a new technique of investigation of twinning, which is based on the fixation of the penetration depth of the indenter in the case of increasing load on it.

Experimental Technique. Antimony single crystals were grown by the Bridgman method from a raw material of 99.999% purity. The samples were obtained by splitting the single crystals along the cleavage plane (111). Owing to a pronounced cleavage, the split-off plane was suitable for investigations without further treatment.

The surface (111) of the antimony single crystals was deformed on a Nano Indenter II nanohardness tester (Nano Instruments, USA). The device is intended for testing for hardness by the Berkovich trihedral indenter at small (to 20 g) loads. During the tests, the relation between the displacement h of the Berkovich indenter and the load P on it is recorded with high accuracy. The measurement accuracy of the impression depth is ±0.04 nm and the load on the indenter is ±75 nN. Deformation of the single crystals was executed at different rates, reaching 12 mN/sec.

The appearance of twins at the indenter manifested itself on the $P = f(h)$ curves in the form of discontinuities of the function $P = f(h)$. After unloading, twins in the indenter impression were observed with an optical microscope.

The dependences $\Delta h = f(t)$ were constructed in order to determine the duration of the process of twinning.

The value of the mean contact pressure P was found as a function of the depth penetration of the indenter.

Experimental Results and Their Discussion. Figure 1 gives the dependences $P = f(h)$ for different rates of loading. On the $P = f(h)$ curves, we observe discontinuities which indicate the appearance of the twins in antimony single crystals as a result of the effect of increasing concentrated load on its surface (111).

In the absence of twinning, the dependence $P = f(h)$ can be described by the known relation [8]

$$P = \frac{Hh^2}{k} . \tag{1}$$

As has been shown by the experimental results of this work (Fig. 1), the loading rate affects the character of dependence (1). In the absence of twinning, for different rates of loading dependence (1) in the general case can be described by the function

$$P = \alpha \frac{Hh^2}{k} , \tag{2}$$

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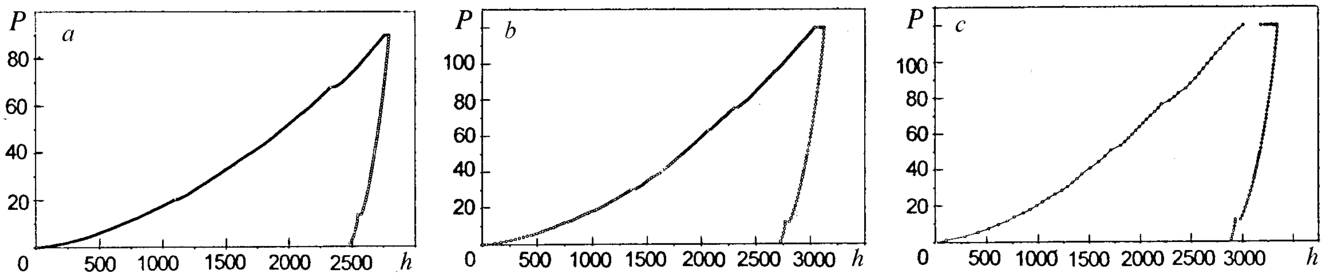


Fig. 1. Relation between the load on the indenter and the depth of its penetration at loading rates of: a) 0.25, b) 3, and c) 12 mN/sec. *P*, mN; *h*, nm.

where α is a certain dimensionless coefficient dependent on the loading rate. In (2), α is the parameter characterizing the sensitivity of the material to the deformation rate. The necessity of introducing this parameter is caused by the fact that with increase in the loading rate the $P = f(h)$ curve shifts to the region of smaller h.

In the case where twinning occurs, the function $P = f(h)$ is a piecewise-continuous function (Fig. 1) and it has the form

$$P = \begin{cases} \alpha\beta_1 \dfrac{Hh^2}{k} & \text{when} \quad 0 < h < h_1, \\ \alpha\beta_2 \dfrac{Hh^2}{k} & \text{when} \quad h_1 < h < h_2, \\ \alpha\beta_3 \dfrac{Hh^2}{k} & \text{when} \quad h_2 < h < h_3, \\ \ldots & \quad \ldots \\ \alpha\beta_n \dfrac{Hh^2}{k} & \text{when} \quad h_{n-1} < h < h_n. \end{cases} \tag{3}$$

As has already been noted, with increase in the rate of loading of antimony single crystals the dependence $P = f(h)$ shifts to the region of lower h, which is shown in Fig. 1c. Therefore, $\alpha > 1$.

As is clear from Fig. 1, the appearance of twins is accompanied not only by the discontinuity of the $P = f(h)$ curve but also by a certain slight displacement of it to the region of higher h. Therefore, $\beta_j < \alpha$ $(j \neq 1)$ and $\beta_1 = 1$, since no twinning was observed at the initial instant of loading.

An important measure of the process of twinning is the value of the density of dislocations ρ_{tw} on its boundaries. The use of nanoindentation allows one to find ρ_{tw} as follows. We write the expression

$$\dot{\varepsilon} = \frac{1}{h}\frac{dh}{dt} = \rho_{\text{tw}} bV. \tag{4}$$

It is easy to show that (4) yields

$$\frac{dh}{dt} = \rho_{\text{tw}} bdl. \tag{5}$$

Integrating (5), for a single twin we have

$$\rho_{\text{tw}} = \frac{2}{bL} \ln \frac{h_2'}{h_1'}. \tag{6}$$

The quantity L is found experimentally. Since the twinning rate is known [9], we calculate L for the twin formed from the formula

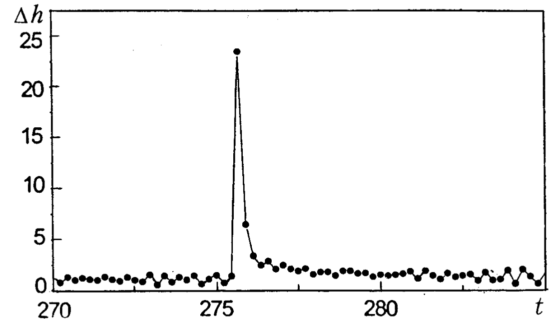
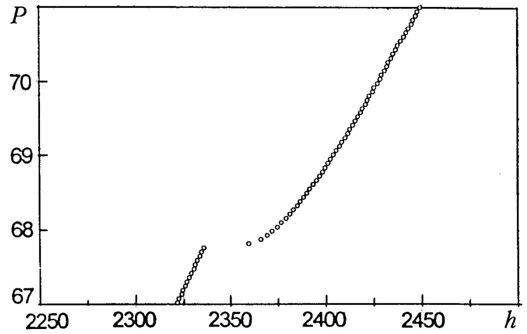


Fig. 2. Dependence of Δh on the time t. Δh, nm; t, sec.



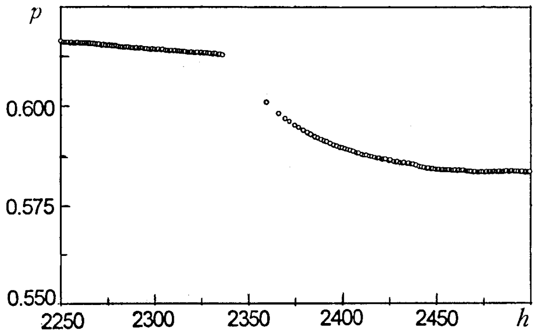


Fig. 3. Magnified picture of the portion of discontinuity of the $P = f(h)$ curve. P, mN; h, nm.

Fig. 4. Mean contact pressure p as a function of the penetration depth of the indenter. P, GPa; h, nm.

$$L = 2V(t_2 - t_1)\,. \tag{7}$$

For the case of twinning given in Fig. 2, the time of the process has a value of the order of 0.8 sec. From the boundaries of the discontinuity region of the $P = f(h)$ curve (Fig. 3) we find that $h_1 = 2320$ nm and $h_2 = 2330$ nm. Taking $V = 10^{-4}$ m/sec and $b = 2 \cdot 10^{10}$ m [2], for the twin considered we obtain from (6) and (7) that $L \approx 160$ μm and $\rho_{tw} \approx 0.25 \cdot 10^{-8}$ m^{-2}, which corresponds to the experimental data of [2, 9, 10] and indicates the legitimacy of the technique suggested for studying the main parameters of twinning.

Figure 4 gives the dependence of the mean contact pressure p produced in the indenter impression on the penetration depth of the indenter. The appearance of twins on the $p = f(h)$ curve manifests itself as a sharp decrease.

Using the data of Figs. 1 and 4, one can describe the process of deformation of the surface (111) of antimony single crystals by the Berkovich indenter in the case where the deformation is accompanied by twinning. The following stages can be distinguished in the process indicated:

1) a relatively slight increase in the mean contact pressure before the appearance of a twin;

2) a sharp decrease in p accompanied by acceleration of the penetration of the indenter into the depth of the material upon a slight change in the load;

3) further penetration of the indenter into the investigated material follows law (3).

Thus, twinning facilitates the relaxation of internal stresses in the indenter impression, thus decreasing the mean contact pressure and increasing the velocity of indenter penetration into the depth of the material.

As a result of the investigation of twinning by the method of nanoindentation, we have established that the appearance of twins on the $P = f(h)$ and $p = f(h)$ curves is revealed by the discontinuities of the indicated functions. The method of nanoindentation allowed us to determine the duration of the process of twinning. A technique for determining the density of twinning dislocations in the twins appearing at the indenter has been suggested.

NOTATION

h, penetration depth of the indenter (displacement of the indenter), nm; *P*, load on the indenter, N; *p*, mean contact pressure, Pa; h_i and h_{i+1}, preceding and subsequent displacements of the indenter, nm; *t*, time, sec; *H*, microhardness of the material, Pa; *k*, geometric parameters determined by the shape of the indenter; β_j, coefficient indicating a deviation of the function $P = f(h)$ from (2) upon the appearance of the *j*th twin; h_j, value of the displacement of the indenter which determines the limits of continuity of the function $P = f(h)$ between the instants of the appearance of twins, nm; $\dot{\varepsilon}$, rate of deformation of the material by the indenter, sec^{-1}; *b*, Burgers vector of a twinning dislocation, m; *V*, mean velocity of twinning dislocations, m/sec; *l*, mean free path of twinning dislocations determined as $l = L/2$ [2] (here *L* is the length of a twin, μm), μm; h'_1 and h'_2, penetration depths of the indenter at the instants before the twinning and after it respectively (determined experimentally from Fig. 3), nm; t_2 and t_1, instants of time before the twinning and after it (determined from Fig. 2), sec. Subscript: tw, twin.

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