

USE OF THIN POLYPARAXYLENE FILMS TO STUDY THE PLASTIC DEFORMATION BISMUTH SINGLE CRYSTALS

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The effect of thin polyparaxylene films on the mechanical twinning of bismuth single crystals with the (111) surface subjected to local deformation. It is found that the number of twins formed near the stress concentrator increases in the presence of the film. Possible mechanisms are proposed to explain an increase in the mobility of twin dislocations in a deformable crystal whose surface is coated with a polyparaxylene film. Spalling of bismuth is found in the regions deformed by the indenter.

Key words: *bismuth single crystals, twinning, polyparaxylene film.*

Twinning is the main mechanism of the plastic deformation of solids. At present, various energy actions [1–5] and the effect of structural changes [4–7] on the development of twinning layers have been studied. These studies are of interest not only from a practical point of view (development of a method for controlling the mechanical properties of twinning materials) but also from a theoretical point of view (investigation of the nature of interdislocation interaction for the twinning of crystals in a modified state or in a state with an excited electronic subsystem [3, 4]). The effect of the state of the surface on crystal twinning [8] is also of interest. The aim of the present work was to study the effect of thin polyparaxylene (PP) films on the twinning of bismuth single crystals.

Bismuth single crystals were grown by the Bridgman method from a raw material containing 99.999% bismuth. Samples in the form of $4 \times 5 \times 10$ mm prisms were produced by cleaving a single crystal along the (111) cleavage plane. The initial density of the basal forest dislocations was 10^5 cm^{-2} , and that of pyramidal dislocations was 10^3 cm^{-2} .

Polyparaxylene is a linear polymer with a regular chemical structure ($-\text{CH}_2-\text{C}_6\text{H}_4-\text{CH}_2-$), a molecular weight of 200,000–500,000, a density of 1.1 g/cm^3 , a tensile elastic modulus of $24,500 \text{ kgf/cm}^2$, and a tensile strength of 630 kgf/cm^2 . Polyparaxylene deposited on the substrate surface is transparent in the optical range and has a crystal structure and high dielectric characteristics. The films are conformal and unstrained and have uniform thickness. The adhesion of the PP films to smooth surfaces is insignificant if they are not pretreated (for example, using a plasmachemical method).

The samples tested were coated with PP films using the Gorham method. Monomer gas is produced by vapor pyrolysis of the starting dimer — cycloparaxylol — in a high-temperature reactor ($550\text{--}650^\circ\text{C}$). The monomer gas then passes from the reactor to a low-temperature ($0\text{--}30^\circ\text{C}$) chamber for film coating, in which it polymerizes on the surfaces at temperatures below 30°C . The production of PP films occurs in the various technological zones of the facility in a continuous-flow regime at a low pressure ($0.1\text{--}100.0 \text{ Pa}$). Bismuth single crystal with freshly cleaved (111) surfaces were placed in the vacuum chamber of the facility for PP film coating, and the gas in the chamber was evacuated to a pressure of 0.1 Pa . The film was formed on the samples at an average rate of 5 nm/min at a monomer gas pressure in the chamber of $3\text{--}5 \text{ Pa}$. The thickness of the films was $10\text{--}50 \text{ nm}$.

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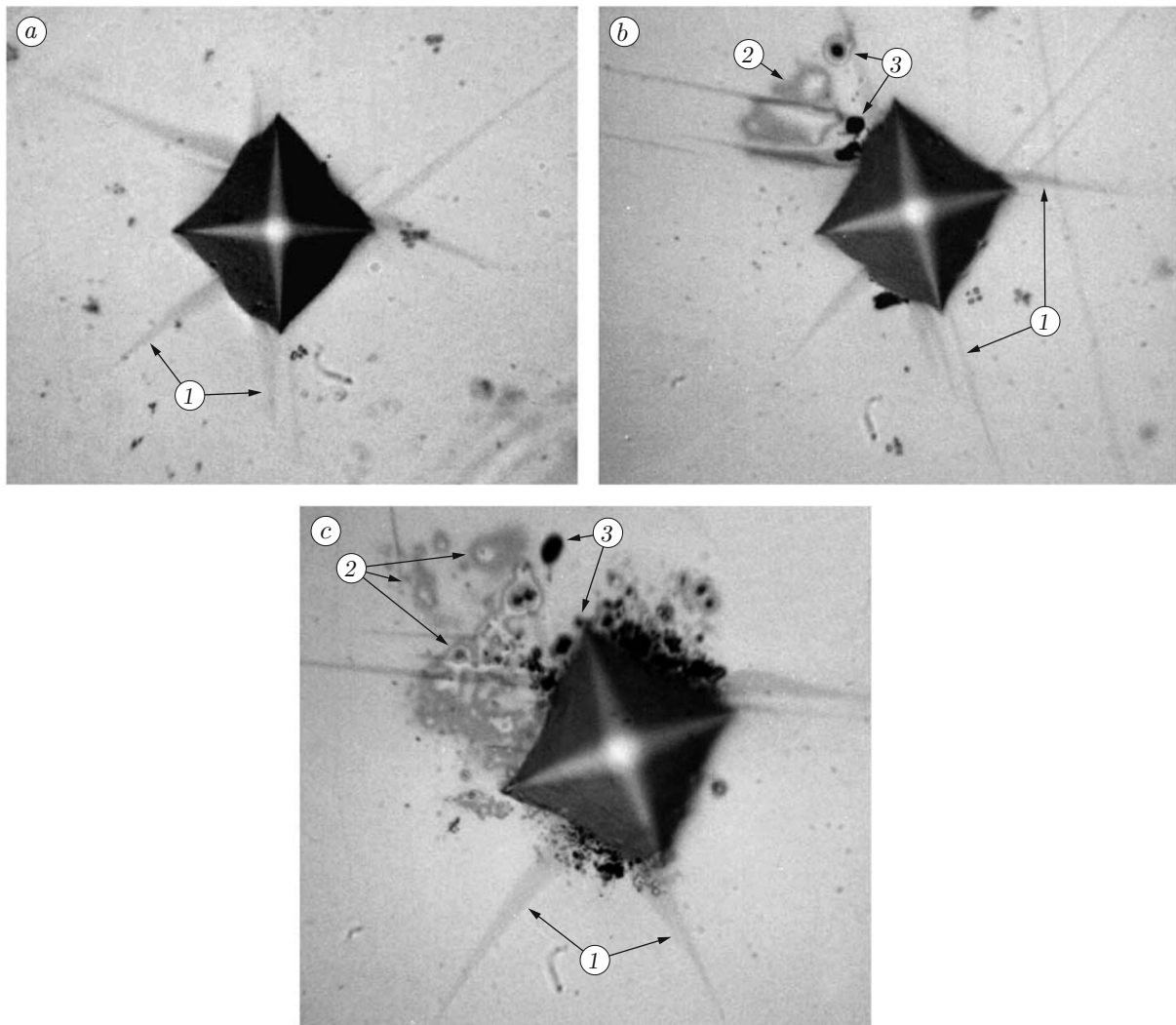


Fig. 1. Assembly of wedge-shaped twins near an indenter impression on the (111) surface of a bismuth single crystal coated with a 50 nm thick film (magnification 500) for loads on the indenter $P = 0.1$ (a), 0.2 (b), and 0.3 N (c); 1) twins; 2) region of separation of the film from the substrate; 3) spalled particles.

A complex stress state in the subsurface layer of the bismuth single crystals was produced by the Vickers diamond pyramid of a PMT-3 standard device. The geometrical parameters of the twins formed near the diamond pyramid impression were measured. The deformation pattern was recorded on the (111) surface of the bismuth single crystals coated with a PP film. The number of twins formed near the pyramid impression was determined. As in [7], the density of twin dislocations on the twin boundaries was found from the degree of noncoherence of the twin boundaries.

The deformation pattern on the (111) surface of bismuth single crystals near the Vickers pyramid impression for various indenter loads is presented in Fig. 1. It is evident that an assembly of 6–12 twins is formed around the indenter impression. The impression boundary on the surface coated with a thin PP film is rough and wavy. In addition, at an indenter load of 0.1 N, one can see darkening and small light spots in the immediate proximity of the impression, indicating a different direction of propagation of the light incident on the PP film in the region of its intense deformation (Fig. 1a). These features are likely due to the nonuniformity of the elastic properties and thickness of film. As a result, in some microregions, the material is nonuniformly pressed by the indenter, which leads to the roughness of the impression boundary, relief roughness and, as a consequence, to nonuniform reflection of the light flow.

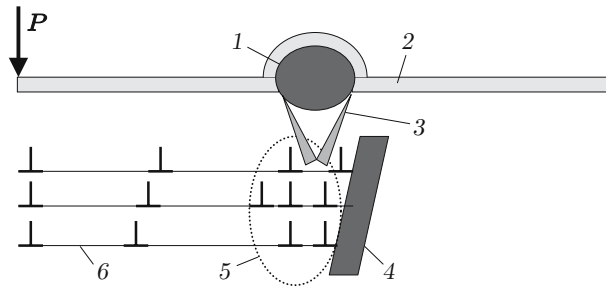


Fig. 2. Diagram of the dislocation mechanism of spalling of bismuth single crystals: 1) particle; 2) PP film; 3) crack; 4) arrester; 5) cluster of basal dislocations; 6) (111) plane.

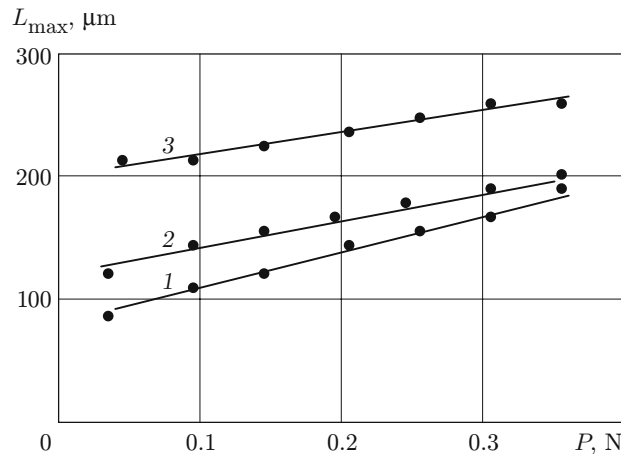


Fig. 3. Maximum length of twins versus indenter load: curve 1 refers to the bismuth single crystal without a film and curves 2 and 3 refer to the bismuth single crystal with a 12 and 50 mm thick PP film applied to the (111) surface, respectively.

As the indenter load is increased (Fig. 1b and c) the substrate material spalls in the form of black particles retained by the film with increasing number of the particles. Spalling may also occur in the case of surface deformation of bismuth single crystals in the absence of the film, where free bismuth particles are removed from the surface during the experiment or are carried away from the observation region by air flows as the stage of the PMT-3 device is moved from the indenter to the microscope objective. In the case considered, the PP film retains the spalled particles.

It is of interest to study the spalling mechanism of bismuth single crystals whose surfaces are subjected to local deformation. The concentrated load P activates not only twinning but also the basal sliding in the planes parallel to the (111) plane. Interacting with the arrester (Fig. 2), such dislocations form clusters localized near the stress arrester, and this results in curvature of the surfaces of the bismuth single crystal. In the region of stress concentration, cracks actively develop that favor the separation of bulged regions from the surface of the bismuth single crystal (Fig. 2). If the surface is coated with an elastic PP film which is weakly bonded to the surface, the spalling products are retained on the surface. If the film is transparent (as in the present work), this provides a convenient method for studying the spalling process of materials.

In addition to spalling, there is local separations of the PP films (see Fig. 1) from the substrate, primarily because of their weak adhesion. Such separations indicate the localization of stresses in these regions that do not result in spalling. These regions, however, obviously contain dislocation clusters that cannot be observed visually in the absence of the film on the surface. Therefore, the application of a film with weak adhesion to the surface of materials is a method for studying the localization of dislocations.

TABLE 1

δ , nm	$\langle N \rangle$	ρ , 10^4 cm^{-1}
0	7.1	2.9
12	7.8	2.7
50	11.2	2.3

Note. δ is the film thickness, $\langle N \rangle$ is the average number of twins arising near the indenter impression on the (111) surface of bismuth single crystals, ρ is the density of twin dislocations on the twin boundaries, $P = 0.2 \text{ N}$.

Figure 3 shows a curve of the maximum length of twins L_{\max} versus indenter load. It is evident that the length of twins increases in the presence of PP films (especially when the film thickness is 50 nm). The application of a thin PP film does not lead to a considerable reduction in the density of twin dislocations on the twin boundaries (see Table 1). This suggests that the increase in the length of the twins is due not to the stimulation of the mobility of twin dislocations but to the activation of the process of their generation.

Thus, the study of the effect of PP films of various thicknesses on the plastic deformation of subsurface layers of bismuth single crystals showed that the number and length of the twins formed near the stress concentrator increase in the case of film coating. At the same time, the density of twin dislocations on the twin boundaries does not change significantly. Spalling of the substrate material was observed at indenter loads higher than 0.2 N. Separation of the PP film was revealed on twin lamellas and particles formed during plastic deformation. The spalling mechanism of bismuth single crystals under local plastic deformation is considered.

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