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REVERSIBLE PLASTICITY OF METALLIC SINGLE CRYSTALS AT THE STAGE OF THEIR RESIDUAL TWINNING

The work studies regularities of the formation of wedge-shaped twins under growing concentrated load in single crystals of bismuth, zinc and bismuth-antimony alloy. It was established that the twinning, detwinning and stopping of the twin deformation near the stress concentrator can take place simultaneously with the load growth. Reversibility of plastic deformation during twinning in metals at the stage of residual twinning development were discovered. Various manifestations of spontaneous detwinning of wedge-shaped twins which emerge at stress concentrators when indenting Bi, Zn, Bi-Sb single crystals with increasing load are quantitatively studied. Depending on the value and the sign, local fields of elastic stress can encourage or discourage twinning, or cause detwinning.
TWINNING, DETWINNING, PLASTIC DEFORMATION, REVERSIBLE PLASTICITY, BISMUTH, ZINC.

1. Introduction

Plasticity is the property of solid bodies to be deformed irreversibly under the action of an external force. It is known that plastic deformation of real crystals occurs due to the movement of crystal lattice defects under the action of external loading. In some crystals the internal forces that appear during such movement can cause the backward motion of defects after an external load is removed. The initial shape of the crystal is restored. This process is known as reversible crystal plasticity.

Reversible plasticity is the main property of plastic deformation by twinning. It represents the first stage of the mechanical twinning of crystals, elastic twinning [1]. At this stage twinning inclusion is reversible and can completely detwin spontaneously during off-loading. Under a concentrated load, a thin interlayer in the shape of a wedge appears in the crystal; it is a wedge-shaped twin and its crystal lattice is displaced at a certain angle to the matrix. The dimensions of the twin wedge grow proportionally to the external load. If the external load decreases, detwinning of the crystal takes place: the twin decreases in dimensions thus keeping the form of a thin wedge. After unloading it disappears completely, that is, leaves the crystal. Elastic twinning is observed in all twinning crystals.

The important feature of deformation twin development at the elastic twinning stage is that its dimensions (wedge length L and its width h at the base) change proportionally to the load quantity.

After some limit value of applied stress, the twin is wedged and after unloading stays in the crystal. At the stage of residual twinning, detwinning can be observed in response to the action of an external stress of the reversed sign on the crystal [2, 3].

Numerous experimental investigations showed that the processes of twinning and detwinning determine the mechanical properties of many technically significant metals and alloys. This stimulates the interest in the study of this phenomenon. Detwinning in crystalline solids is a unique deformation mechanism partially responsible for the shape memory effect [4 – 7]. In the last years it has been revealed that twinning and detwinning are the important deformation modes in metals and alloys with various crystal structures [8 – 19]. The metals which have a hexagonal close-packed structure, such as Be, Mg, Zr, and Ti, have aroused great interest. Twinning-detwinning is an important deformation mode in these metals. Detwinning, a reverse twinning process, has been reported in some hexagonal close-packed metals and alloys during loading, unloading or cyclic deformation [20 – 27]. The detwinning characteristics in magnesium alloys obtained through a cyclic loading test have been studied in detail [28 – 35]. Detwinning describes the coalescence of a martensite twin into a single martensite crystallite [36, 37]. It is stated both experimentally and theoretically that detwinning is a unique deformation mechanism of nanotwinned metals [38 – 40]. Various theoretical deformation models of crystal twinning-detwinning have been developed [41 – 46].

Earlier we studied the development of wedge-shaped twins in bismuth single crystals under the action of an increasing concentrated load. In Ref. [47], it was shown that an imprint had several residual wedge-shaped twins after indentation of bismuth single crystals by a diamond pyramid. Their evolution with load growth progressed in different ways. The proportional length and width changing of a wedge-shaped twin was distorted with load growth. Both twins' growing and their complete stopping while the dimensions of wedge-shaped twins under load remained unchanged were possible. We revealed the cases of a spontaneous size reduction of a twinned wedge with load growth; that was a reversible twinning under load at the stage of residual twinning.

The present paper contains studies in the regularities of the formation of a wedge-shaped twinned area under a growing concentrated load in single crystals of Zn, Bi and Bi-Sb.

2. Experimental technique

Material and sample preparation. The experiments were conducted on metal single-crystal samples with hexagonal (Zn) and rhombohedral structures (Bi and Bi-Sb alloy). The crystal-growing processes and sample preparations were quite simple. These crystals possess perfect cleavage planes. The (111) cleavages in the rhombohedral crystals and the (0001) ones in the close-packed hexagonal crystals are natural metallographic sections and do not require additional treatment for microscopic examination of the surface.

In these metals, the slipping precedes the twinning and accompanies it at all stages over a wide temperature range; besides, the development of twins in them can go with brittle fracture. The crystallography of the twinning and the slipping of the above-mentioned metals was studied thoroughly. Three slip systems are implemented in the hexagonal close-packed lattice: the easy one in the basal plane (0001) $<11\bar{2}0>$; the more complex one, the pyramidal slip in the system $\{11\bar{2}2\}$ $<\bar{1}\bar{1}23>$; the prismatic slip in the system $\{10\bar{1}0\}$ $<11\bar{2}0>$. The twinning in the close-packed hexagonal structures is realized in the system $\{10\bar{1}2\}$ $<10\bar{1}1>$. Two slip systems take place simultaneously in the neighbourhood of the stress concentrator during the deformation of bismuth and bismuth-antimony alloy: the easy basal slip in the system {111} $<1\bar{1}0>$ and the secondary slip with a higher yield point in the system $\{11\bar{1}\}$ <110>, and the twinning in the system {110} <001>.

All metals under study have just one twinning system ensuring the reliability of physical interpretations of the obtained research results and simplifying considerably the dislocation analysis of twinning restructuring processes.

Similar to calcite in the case of pure twinning, crystals of bismuth, bismuth-antimony and zinc provide classical samples for studying the twinning regularities in metal crystals.

The experiment was conducted with the use of zinc single crystals with initial basal dislocation density ~ $5\cdot10^4$ cm^{-2} and pyramidal dislocation density ~ $5\cdot10^3$ cm^{-2}, as well as bismuth single crystals and bismuth-antimony alloy with dislocation density in non-basal planes ~ 10^6 cm^{-2}.

The working samples shaped as the right-angle prisms with the dimensions 10 × 10 × 5 mm were made by crystal cleavage in the cleavage plane using a sharp knife at the liquid nitrogen temperature. An impulsive force was applied to the knife in order to avoid raised waves on the sample surface; those usually appear while slowing down and stopping the cleavage crack. The high rate of crack propagation on cleavage ensured high quality of the obtained surface.

Mechanical testing. Crystal deformation was performed by indentation of the cleavage plane of a single crystal by a Vickers tetrahedral diamond pyramid. The indenter was pressed perpendicularly into the working face and the sample was kept under load for 15 s. All measurements were taken in the load range from 0.05 to 1.5 N at room temperature.

As a rule, the indenter's imprint after initial loading had several wedge-shaped twins simultaneously (Fig. 1, *a*).

As the load increased, the sizes and shapes of the initial twins changed and new ones appeared. All subsequent loadings with increasing the load were conducted by repeated crystal indentation in the same imprint and the dimensions of each twin were measured: the length L and the width h at the basis. The measurements were taken with an HWMMT-X7 microhardness tester.

The main problem of recording the intermediate stages of plastic deformation development in metals was solved using the original method of iterated sample indentation. The investigation was carried out in a sequential manner:

placing the indenter;

pointed deformation of the crystal with a controlled waiting time under load;

sample unloading;

measuring the dimensions of all twins which appeared in the indent;

changing the characteristics of the external action (load increment) and placing the indenter in the same hole;

sample unloading;

taking measurements, etc.

The deformation zone was photographed after each unloading. The special experiments proved that the repeated indentation in the same hole with the same load in magnitude and duration did not lead to the change in the shape, the dimensions and the number of primary twins. The sample unloading at various stages of twin interlayer development fixed the position of its boundaries virtually for any period of time. The load increment during the repeated placing of the indenter in the same hole lead to resuming plastic shears at twin boundaries. This simple method allowed direct observation of the influence of various factors on the formation of twins near the stress concentrator.

3. Results and discussion

It was experimentally revealed that the dimensional change of residual wedge-shaped twins of the imprint under repeated crystal indentation with the increased load followed one of the ten modes:

L and h grow simultaneously (mode 1);

a) *b*)

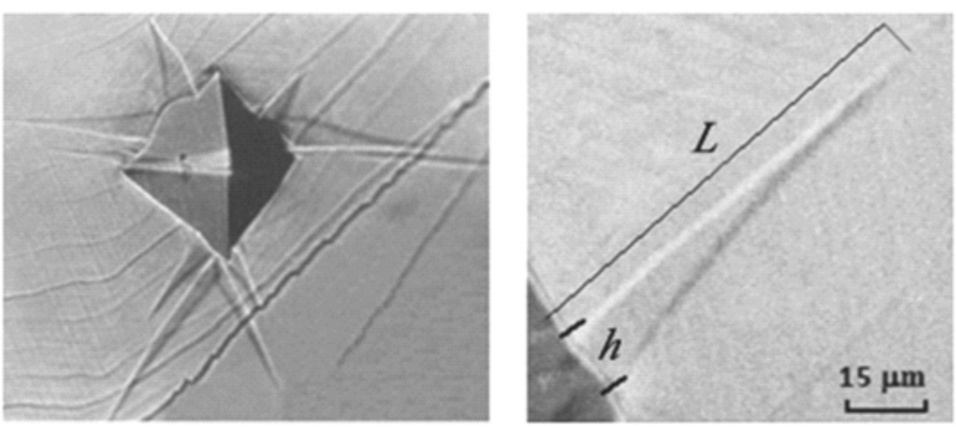

Fig. 1. Micrographs of a diamond pyramid imprint in (111) plane of a bismuth single crystal with a set of wedge-shaped twins (*a*) and a wedge-shaped twin interlayer (*b*); L, h – the twin length and its width

L grows and h remains unchanged (mode 2);

h grows and L stays unchanged (mode 3);

L grows and h decreases (mode 4);

L decreases with h growth (mode 5);

L decreases with the stable h (mode 6);

h decreases with the stable L (mode 7);

both L and h can remain unchanged with load growth (mode 8);

L and h decrease simultaneously (mode 9);

the twin disappears completely (mode 10).

All cases when some dimension of a wedge-shaped twin remains unchanged with the external load growth prove the stopping of a twin interlayer by internal causes, i. e., structural defects of various physical natures. The size reduction of a twin or its complete disappearance (the crystal detwinning) is a manifestation of twinning reversibility.

The reversibility of plastic deformation in our experiments appeared not at the elastic stage as a result of crystal unloading or under reversed sign stress but in the residual twins with an increase in the direct external mechanic stress. This is a new phenomenon which is not certain for the pure twinning, and its physical explanation should be searched for in the differences of twin wedge development conditions under concentrated load in calcite and in metal crystals.

In this situation, an equilibrium state of an isolated elastic twin under load is ensured by elastic and inelastic forces which effect on the length unit of a twinning dislocation in an assembly equal to zero, that is,

$$F_{elast} + F_{inelast} = 0 \qquad (1)$$

where F_{elast} are the forces produced by an external load and elastic fields of the dislocation assembly; $F_{inelast}$ are the braking forces conditioned by the crystal structure and its defects and also the surface tension forces acting on a twin from a mother crystal.

The growth or the attenuation of the external load increases the augend in Eq. (1) and transmits the ordered motion to twinning dislocations in the twinning plane and in the twinning direction.

The interaction of a twin with stoppers in metals slows down its growth and contributes a component influencing the dislocation assembly to the inelastic forces. The defects of various nature and power which disturb the crystal structure generate local fields of elastic stress in a crystal with their sign and intensity being impossible to take into consideration in the process of deformation development. When summarized, the mechanic stress caused by the pyramid, twins and structure defects produce a complex field of mechanic stress. Local noncompensated fields of elastic stress appear in the deformation zone with the stress rate and the sign at any point around the imprint being impossible to identify definitely. The balance of the wedge-shaped twinned interlayer under load can be described by the following equation:

$$F_{elast} + F_{inelast} + F_{local} = 0 \qquad (2)$$

where the third component F_{local} characterizes the forces acting on the twinning dislocation assembly from a summary field of the elastic stresses. Probably, it is the rate and the sign of the forces responsible for a nonsynchronous, ambiguous dimensional change of wedged twins under load.

If this hypothesis is correct, the reversibility phenomenon is mostly expressed in the twins situating in the places of the biggest distortion of the crystal structure with the most complex pattern of local overstress, that is, near the imprint boundaries, in the twins with branchy structure and near large twin interlayers. This is convincingly proved experimentally. It was noted that interlayers of two types disappear most often: small twins near the contour of the imprint and the twin arms originating at the curved boundary of a wedge-shaped twin (Fig. 2).

The appearance of a new larger twin near the existing twins is always accompanied by a partial detwinning of the nearest interlayer (Fig. 3).

It is apparent from Fig. 4 that twinned wedge *3* with incoherent boundaries does not only block the development of a smaller neighboring interlayer *2* by its elastic stress field but also leads to its degradation.

The appearance of new twins and the development of twins in groups unpredictably change the pattern of the heterogeneous spatial field of elastic stress near the imprint. The character of change in the twin dimensions can *a*) *b*)

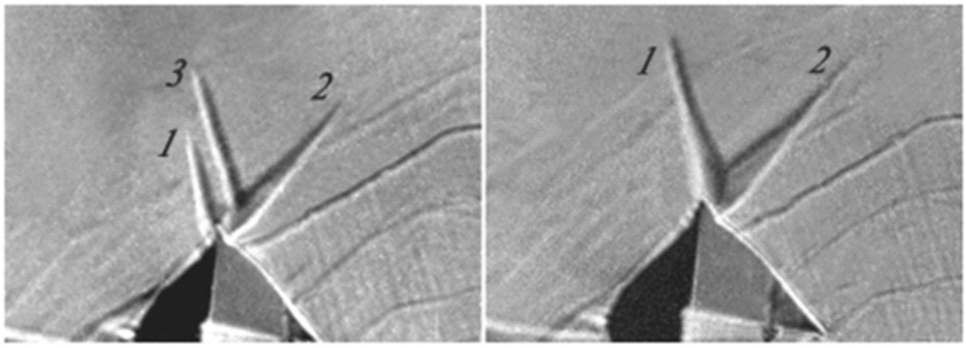

Fig. 2. The disappearance of twin arm *3* with the growth of an external load in bismuth: *P* = 0.3 N (*a*), 0.5 N (*b*); *1, 2, 3* are the numbers of the initial twins

a) *b*)

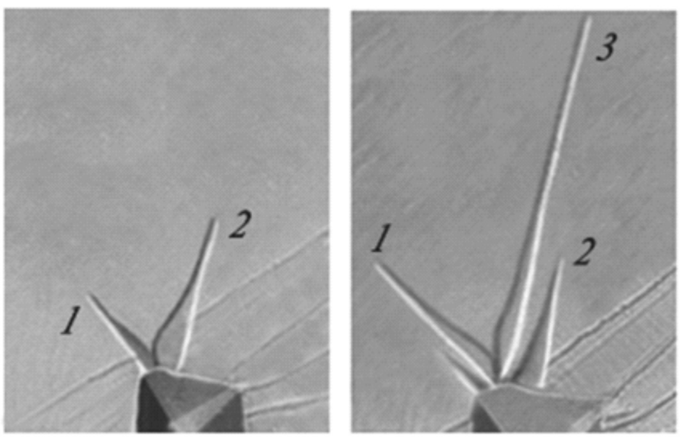

Fig. 3. The partial detwinning of interlayer *2* at new twin nucleation *3* with an increase in static load: *P* = 0.1 N (*a*), 0.3 N (*b*); *1, 2* are the numbers of the initial interlayers

a) *b*) *c*) *d*)

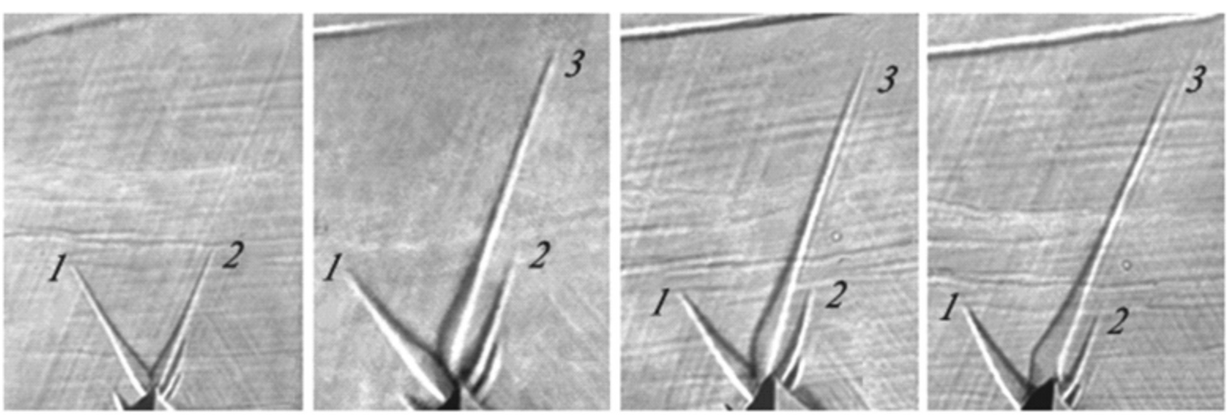

Fig. 4. Twinning and detwinning in the bismuth crystal with an increase in static load: *P* = 0.1 N(*a*), 0.3 N(*b*), 0.4 N (*c*), 0.5 N (*d*)

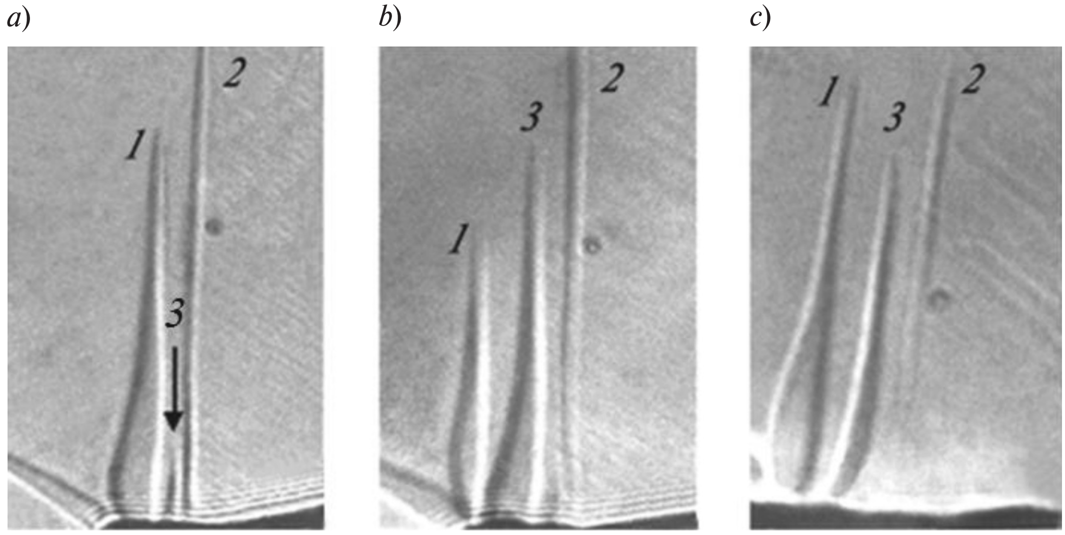

Fig. 5. Changing the twin development mode with an increase in external load:
P = 0.1 N (*a*), 0.3 N (*b*), 0.5 N (*c*); *1*, *2*, *3* are the interlayer numbers. The arrow points to a twin embryo *3*

alter in the process of a stepwise load increase (Fig. 5).

The twin embryo between interlayers *1* and *2* (Fig. 5, *a*) grows with load increment in a way that leads to a partial detwinning of both neighbouring interlayers (Fig. 5, *b*). It is noteworthy that in accordance with the considerations given above, all reversible repeated plastic shears at the boundaries take place near the boundary of the imprint and do not at the top of a twin. With further growth of the external force, twin *1* which had preferential development produces a powerful field of elastic stress with a reversed sign that leads not only to a size decrease of the neighbouring twin *3* but also to the complete disappearance of a considerably long section of a more distant twinned interlayer *2*.

The quantitative investigation of the dependence of twin amount in metals with different size changes on load rate was conducted in order to identify the physical nature of various phenomena of reversible plasticity at the boundaries of residual wedge-shaped twins.

The simulation of twinning dislocations movement at the boundaries of residual twins in metals under load was performed with consideration for the basic properties of twinning dislocations: each crystallographic plane has only one twinning dislocation; each following twinning dislocation is at a distance of one interplanar space from the previous one; the movement of one twinning dislocation transfers a portion of matrix atoms into the twin structure.

It is easier to simulate the disappearance of a wedge-shaped twin as externally, this phenomenon is absolutely similar to elastic detwinning. It is realized by the process of reversion movement of twinning dislocations from the top of the wedge to the basis and their exit out of the crystal. It is interesting that the fraction of the disappearing twins does not virtually depend on the value of the acting load (Fig. 6, *a*).

The probability of detwinning for a certain twin increases with a decrease in the parameter h/L but its value is not defined uniquely. It is evident that the main stimulus of the inverse lattice restructuring is the energy gain due to a decrease of the internal division surfaces. Local fields of elastic stress with the opposite sign created by accumulation of dislocations in the neighbourhood of the pyramid imprint such as perfect dislocation forming slip lines and partial dislocations at the boundaries of wedge-shaped twins play a key role in the reversible twinning boundary displacement (Fig. 6, *b*).

In relation to these twins the structure defects also perform the opposite function, acting as a stopper. When meeting an impassable

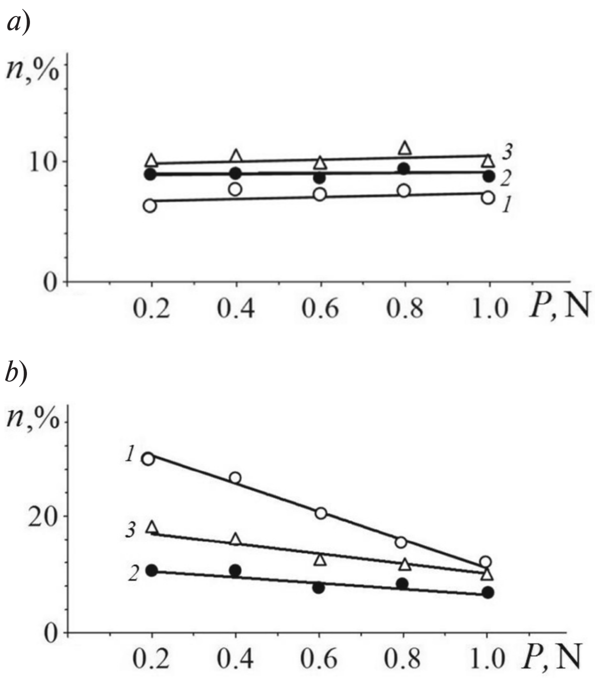

Fig. 6. The plots of the percentage *n* of disappeared twins (*a*) and the twins with a simultaneous decrease in their length *L* and width *h* (*b*) versus the load value in various crystals: Bi (*1*), Bi-Sb (*2*), Zn (*3*) (see modes 10 and 9)

stopper, the twinning dislocations halt and the detwinning process comes to an end. The fact that the amount of twins decreases with the load increase (see Fig. 6, *b*) indicates the growth of the amount of impassable stoppers with an increase in the external stress.

The twinning dislocation assembly can interact with the stoppers in the matrix which create internal stress fields different in amplitude, configuration and sign. If the local stress at the wedge top is directed against the stress from the external load, a partial decrease in the twin length with the simultaneous width growth takes place (Fig. 7, *a*). A kind of compaction of the dislocation structure can be observed at the boundaries in such interlayers.

When a group of twinning dislocations of the same sign stop near the barrier, a strong field of internal stress appears. The dislocations that appear due to the source function create the stress with the reversed sign that opposes the applied one and blocks a dislocation's generation in the source. A twinned wedge reduces at the top without changing the total number of twinning dislocations at the division boundaries (Fig. 7, *b*).

The mechanisms of the interlayer development are the same: translatory and backward motions of the twinning dislocations but in the case when the twin width *h* grows and the twin length *L* decreases (see mode 5), the motions of dislocations at the top and at the basis of the wedge occur in the opposite directions. The dislocation assembly forming a twin stops being single, it is fragmented and its development depends neither on the external load intensity nor on collective interaction of dislocations in accumulation.

The reversibility variants in which a decrease in the twin width *h* at the wedge base is observed (Fig. 8) are the most complex for physical interpretation.

The twin growth in length and simultaneous backward motion of dislocations to the source of the twinning dislocations results from extremely specific conditions; under such conditions, powerful stress fields of the reversed sign appear near the dislocation source. The fields not only prevent the generation of new dislocations but also ensure backward motion of the existing ones.

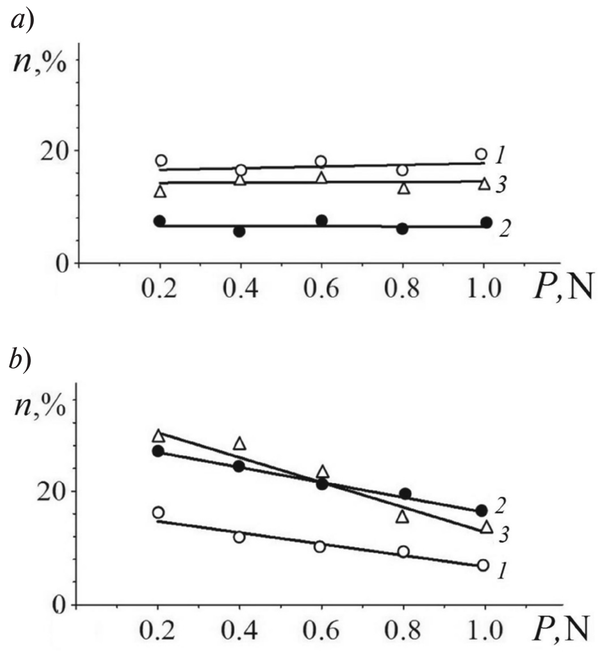

Fig. 7. The plots similar to those in Fig. 6, but for modes 5 (*a*) and 6 (*b*)

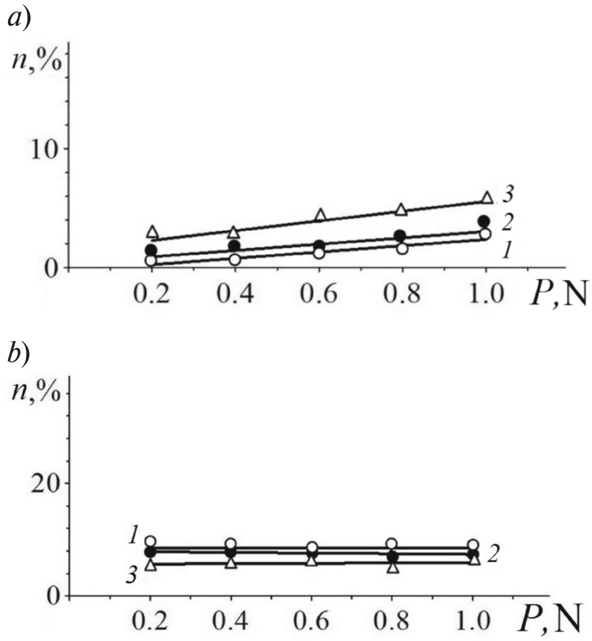

Fig. 8. The plots similar to those in Fig. 6 and 7 but for modes 7 (*a*) and 4 (*b*)

It is unlikely that such conditions can take place very often but, as the experiment showed, the fraction of twinned wedges for which *h* decreased was relatively high, that is why it can be assumed that residual wedge-shaped twin boundaries formation occurs as a result of a nucleation of not only rectilinear dislocations of the same sign in the basis but also new twinning dislocations of both signs in the shape of half-loops with subsequent recession at the boundary. Such a possibility is conditioned by the theoretical estimation of the influence of surface tension on heterogeneous buildup of twinning dislocations in a metal. In this case two branches of the same loop of a twinning dislocation which cross crystal surface have opposite signs and it makes possible the reciprocal annihilation of twinning dislocations of opposite signs from the neighbouring crystallographic planes at the twin boundaries. This means that although new dislocations in the source are still generated, the actual amount of both positive and negative dislocations reduces because of their reciprocal annihilation and twin width at the basis decreases. Another possible detwinning mechanism associated with a developed accommodative slip near an incoherent boundary is ensured by dislocation reactions during boundary crossing by perfect glide dislocations. The products of these reactions can lead to both the twin growth and its reduction, that is, to its detwinning.

Apart from the above, more complex cases of reversibility at twin boundaries were discovered which are difficult to classify in accordance with basic features. For example, they are combined detwinnings in twin pairs with a common basis and with twinning planes intersecting at 60° angle. An alternate growth of such twins was observed at stepwise crystal loading; the growth of one twin at each stage of loading was accompanied by slowing down or detwinning of the other, on the next stage the signs of twin transformation in twins were changed into the opposite ones. In the pair of associated twins a stepwise change of twin boundaries activity was observed in one twin. The twins developed alternately by matrix lattice rearrangement on one of the boundaries of each twin. The opposite boundaries were straight-line and fixed. After another stage of loading in one of the twins the former straight-line boundary appeared curved and the boundary that had a visible profile associated with twinning dislocation generation and movement appeared straight. Such behaviour of twins cannot be explained only by elementary dislocation processes of generation of straight-line twinning dislocations in the basis and their movement in a twinning plane.

4. Summary

The experimental study showed that the size change of wedge-shaped twins in Bi, Zn and Bi-Sb single crystals is nonsynchronous and ambiguous with the growth of external concentrated load. The analysis of the twins formed around concentrators showed that their size evolution with an increase in the load occurred in different ways. The applied load was not the governing factor in the development of plastic deformation via twinning in metal crystals. Various types of plastic deformation reversibility during twinning in metals at the stage of residual twinning development were discovered:

twin length grows and its width decreases;

(vice versa) twin wedge length decreases with the growth of its width;

twin length decreases with stable twin width;

twin width decreases with stable twin length;

twin length and width decrease simultaneously;

the twin disappears completely.

All possible ways of development of wedge-shaped twins under concentrated load in single crystals under study can take place simultaneously in one act of indentation with the load growth.

The development of a residual wedge-shaped twin in metal is governed by the character of the stress condition near its boundaries. Depending on the value and the sign, local fields of elastic stress can encourage or discourage twinning, or cause detwinning. A collective mechanism of twinning dislocation movement which is crucial at the stage of growth or the reduction of an elastic twin is destroyed in metal in the process of residual interlayer development. The assembly of twinning dislocations is fragmented into separate parts which can move independently from each other and sometimes in opposite directions.

The reversible plasticity during twinning can be regulated by modification of external deforming conditions, the load intensity in particular. Understanding the mechanisms of reversible plasticity in residual twinning reveals the potential for improving both physical and mechanical properties of the twinning metals.

The discovered phenomenon of spontaneous detwinning of wedge-shaped twins in metals with the growth of external mechanical stress proves that the reversibility of plastic deformation in twinning is a fundamental property of such deformation and can be observed not only in elastic twinning but also at the stage of residual twinning.

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Чикова Т.С., Башмаков В.И. ОБРАТИМАЯ ПЛАСТИЧНОСТЬ МЕТАЛЛИЧЕСКИХ МОНОКРИСТАЛЛОВ НА СТАДИИ ИХ ОСТАТОЧНОГО ДВОЙНИКОВАНИЯ.

В работе изучены закономерности формирования клиновидной сдвойникованной области в монокристаллах Bi, Zn и сплава Bi-Sb под действием возрастающей сосредоточенной нагрузки. Установлено, что изменение размеров остаточного клиновидного двойника с ростом нагрузки может происходить по одному из десяти вариантов, в которых длина двойника и его ширина у устья либо увеличиваются, либо уменьшаются, либо остаются неизменными в разных сочетаниях. Локальные поля упругих напряжений в зависимости от величины и знака могут стимулировать двойникование, препятствовать ему или вызывать раздвойникование. Обнаруженное явление самопроизвольного раздвойникования клиновидных двойников в металлах при увеличении внешних механических напряжений свидетельствует о том, что обратимость пластической деформации при двойниковании есть фундаментальное свойство этого вида деформации и проявляется не только при упругом двойниковании, но и на стадии остаточного двойникования.

ДВОЙНИКОВАНИЕ, РАЗДВОЙНИКОВАНИЕ, ПЛАСТИЧЕСКАЯ ДЕФОРМАЦИЯ, ОБРАТИМАЯ ПЛАСТИЧНОСТЬ, ВИСМУТ, ЦИНК.

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