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Plastic deformation instabilities in Bi crystals under microindentation

E.I.Rogacheva, A.A.Yakovleva, S.G.Lyubchenko

National Technical University "Kharkiv Polytechnical Institute",
21 Frunze St., 61002 Kharkiv, Ukraine

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The dependences of microhardness H on the load on indentor were obtained for bismuth single- and polycrystals. The general tendency to H decreasing with P increasing up to ~0.15 N is observed. After that the microhardness practically does not change. In the range of $P \simeq 0.05$–0.15 N, in the $H(P)$ dependences the oscillations are detected whose presence is explained by qualitative changes in plastic deformation micromechanisms and in the crystal defect structure under increasing indentor load.

Получены зависимости микротвердости H моно- и поликристаллов висмута от величины нагрузки на индентор P. Наблюдается общая тенденция к снижению H с увеличением P до ~0,15 N, после чего микротвердость практически не изменяется. В интервале $P \simeq 0{,}05$–$0{,}15$ N на зависимостях $H(P)$ обнаружены осцилляции, наличие которых связывается с качественными изменениями в микромеханизмах пластической деформации и в дефектной структуре кристалла при увеличении нагрузки на индентор.

Microhardness H is an important parameter which determines the material resistance to the plastic deformation under application of a concentrated load P, creating a non-uniform stress field [1, 2]. For macrohardness the law of similarity has experimentally been established according to which H value does not depend on the indentation size d. However, in the microhardness measurements the condition of geometrical similarity usually is not fulfilled, and there is observed so-called "scale effect", which is described quantitatively by $H(P)$ or $H(d)$ dependences. The existence of scale effect complicates the comparison of microhardness values obtained by different authors, and becomes especially significant when the mechanical properties of thin films, coverings and multi-layer structures are studied.

There is a great number of works devoted to studying the peculiarities and causes of scale effect (see, for example, [1–7]). Among the main factors determining scale effect are the change in the plastic deformation mechanism in the surface layer in comparison with the volume, elastic recovery of the indentation after the load removal, and also the structural factor determined by the presence of boundaries separating defect-free areas. The majority of authors report a monotonous increase in H with decreasing P, though in some works a decrease in H with decreasing load was observed [8, 9]. In [10] for polycrystalline samples of semiconductor compound $CuInSe_2$ in a certain range of loads we observed a region of anomalous H growth in the $H(P)$ dependences with increasing P and attributed effect to qualitative changes in the crystal defect subsystem under certain levels of the plastic deformation. One should expect that such effects with a high probability will be observed in plastic materials, in which the significant deformations are achieved without the sample destruction, and will manifest themselves more distinctly in single crystals where the grain boundaries do not impede the dislocation movement. The observation of various kinds of peculiarities in the $H(P)$ dependences allows us to judge, on the one hand, about the levels of plastic deformation, at which the defect structure undergoes some changes, and, on the other hand, to choose the optimal load for the H measurement.

The goal of the present work is to establish the character of the manifestation of the scale effect under microindentation of bismuth single- and polycrystals. Bi is a soft and plastic material [11], has face-centred rhombohedral lattice close to the cubic one [12], and is easily cleaved along a perfect cleavage plane (111). The peculiarities of the crystal structure and chemical bond cause the anisotropy of properties, in particular the anisotropy of mechanical properties [11]. There is a significant variation in microhardness values for Bi reported by different authors (from 60 up to 180 MPa). One of the most probable reasons of this variation (along with the anisotropy of properties) is the scale effect, which, to the best of our knowledge, has not been investigated for Bi crystals.

In the present work the existence of the scale effect in Bi single- and polycrystals was established, the general tendency to the H growth with decreasing P was detected, and the plastic instabilities were observed in a definite range of loads.

The microhardness was measured at room temperature on a PMT-3 apparatus using a pyramidal diamond indentor in the load range of 0.01–0.49 N. The load application, contact maintenance and the removal of load lasted for 10 sec each. The adjustment of the device was carried out using fresh chips of NaCl crystals. The error of the measurement of the indentation diagonal was ~0.2 μm. For each sample at each load at least 20 indentations were obtained. The measurements were carried out on (111) face of Bi single crystals, grown by the Bridgman technique, and also on a polycrystalline sample. The Bi purity was no less than 99.999 % of the main component. The single-crystallinity and the orientation of Bi crystallographic axes were determined using X-ray diffractometry on freshly cleaved samples. The samples with the size of $10\times8\times6$ mm^3 were cleaved from the grown single crystal along the cleavage plane. The preparation of surface for H measurements (grinding, mechanical polishing and etching by 15 % HNO_3 solution) was identical for all samples. The (111) cleavage surface of the single crystal was oriented relative to the indentor in such a way that the indentation diagonal was at the angle of 45° to the sliding direction $[1\bar{1}0]$. The indentations had the equal diagonals, but taking into account anisotropy of Bi mechanical properties the indentation sides along $[1\bar{1}0]$ direction had slightly convex shape, which was in good agreement with the results of the other works [11]. Quite often the wedge-shaped twins were observed around the indentation. The H values at each load were obtained as the mean H_{cp} of the measurement of 20 indentations, and the confidence interval ΔH was determined as $\Delta H = \beta \cdot t_n$, where β is the standard error of the mean for the series of 20 measurements, and tn is the Student coefficient, which was taken to be equal to 2 [13]. The measurements showed that with increasing load on indentor, the relative standard error of the mean value β/H_{cp} decreased from ~ 2 down to ~0.5 % which actually corresponded to an error of the indentation diagonal measurement. To establish the possibility of the influence of non-identical conditions of single crystal growth and sample preparation for H measurements, three different Bi single crystals grown by a similar technique were used. It was established that the variation of H values for different single crystals did not ex-

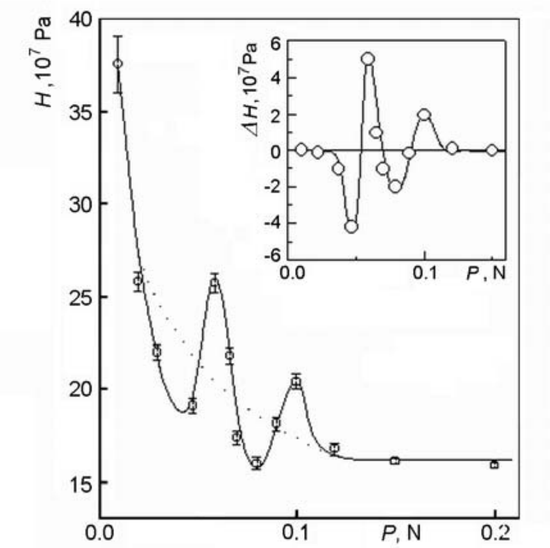

Fig. 1. The dependence of microhardness H measured on (111) face of bismuth single crystals on the load on indentor P. The dashed line corresponds to the curve constructed in the assumption of power law dependence $H = A \cdot P^{-b}$, where $b = 0.21\pm0.02$.

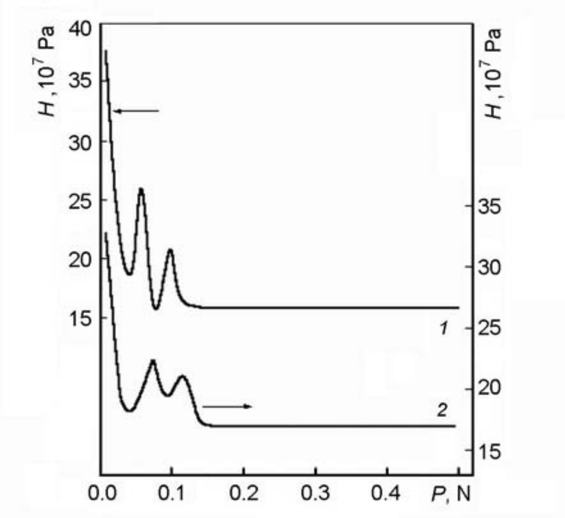

Fig. 2. The dependence of microhardness on the load on indentor for Bi single crystal on (111) face (curve *1*) and for Bi polycrystal (curve *2*).

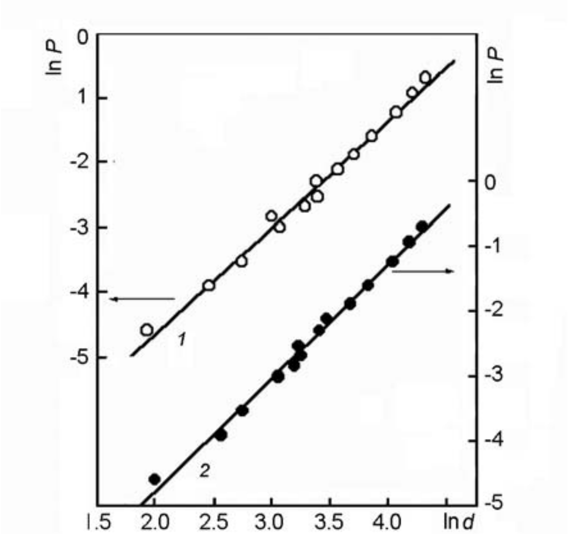

Fig. 3. The logarithmic dependences of the load on indentor P on the indentation diagonal for Bi single crystal on (111) face (curve *1*) and for Bi polycrystal (curve *2*). P is measured in newtons and d in micrometers.

ceed the variation of H values within each sample.

In Fig. 1 the microhardness dependence on the indentor load measured on (111) face of Bi single crystals is given. The distinct manifestation of scale effect is observed: up to $P \cong 0.05$ N, H decreases with the increasing load, and then the $H(P)$ dependence exhibits the oscillatory behavior, which indicates the existence of plastic instabilities, and from $P \sim 0.15$ N, the microhardness value practically does not change under further increase in P. The critical values of P, corresponding to the appearance of oscillations ($P \sim 0.05$ N), and also to the minima and maxima in the $H(P)$ dependences, are well reproduced for different samples. In polycrystalline Bi the plastic instabilities are also observed (Fig. 2), however, the corresponding range of loads is shifted towards greater P in comparison with the single crystal, which can be naturally attributed to the change in the deformation conditions in the presence of grain boundaries in crystal.

Scale effect is commonly described within the framework of Meyer's power law $P = Ad^n$, from which the power law dependence $H = BP^{-b}$ is followed, where $b = (2 - n)/n$ [1, 2]. In Fig. 3, the logarithmic dependences $P(d)$ obtained from the microhardness measurement on (111) face of Bi single crystal and on the polycrystalline sample are presented, which show that Meyer's equation is in good agreement with the experimental data (the correlation coefficient is greater than 0.99). The power law coefficients n determined from these dependences for Bi single-and polycrystals were $n = 1.65 \pm 0.03$ and $n = 1.74 \pm 0.03$ respectively.

Analyzing the obtained data, one can emphasize two principal results: 1) with increasing load on indentor up to $P \sim 0.15$ N in Bi single-and polycrystals the tendency to microhardness decreasing is observed, i.e. the scale effect takes place; 2) in the load range of $P \cong 0.05\text{–}0.15$ N, the $H(P)$ dependences have the oscillatory character. Taking into account these results, the obtained $H(P)$ dependence can be presented as imposition of the monotonous and oscillating components. The dashed line in Fig. 1 corresponds to the power law dependence $H = BP^{-b}$, where the value $b = (2 - n)/n = 0.21 \pm 0.02$ is obtained for $n = 1.65 \pm 0.03$. In the insert to Fig. 1, the $\Delta H(P)$ dependence is presented, where ΔH is the deviation of H value from the monotonous component of the $H(P)$ dependence. The relative oscillation amplitude amounts to 15–20 %.

The most probable factors determining the decrease in H with increasing P have been discussed above. As Bi belongs to soft and plastic materials [11], elastic recovery of imprints after microindentation practically does nor occur. The influence of the structural factor connected with the presence of different types of boundaries [7] also can be excluded, if H is measured on monocrystalline samples. Thus, it follows that the principal cause of the scale effect in Bi is the difference in the properties of the surface layer and the crystal volume. From our point of view, it is necessary to take into consideration one more factor mentioned in [10] and connected with the self-organization processes in the deformation defect subsystem. At small loads the dislocation mechanism of plasticity is the most probable [14, 15]. As deformation increases, the dislocation systems start to exhibit collective properties, resulting in the formation of stable ordered structures such as rigidly connected dislocation ensembles [16]. That is why the observed decrease in H up to $P \sim 0.05$ N can be associated not only with the specificity of the surface layer properties, but also with the increasing degree of order in the dislocation structure as the total number of dislocations increases.

The sharp growth in H at $P > 0.05$ N can be caused by the fact that under some deformation, monotonically increasing strengthening of the sliding planes reaches the critical value at which the further dislocation movement along them becomes impossible and is replaced by the deformational twinning. It is known [11] that under deformation of the Bi cleavage planes (111) by the concentrated load, the plastic deformation process with twinning in one of three directions [101] is easily realized in the crystal, which is proved by the presented data. In [17] it was shown that the number of twins emerging under the indentation of Bi single crystals does not depend on P in the load range of $P = 0.05$–0.3 N, while the length of twins increases; however, the information about the twinning processes at $P < 0.05$ N is absent. If we suppose that at $P < 0.05$ N the twinning process takes place, the sharp increase in H at $P \sim 0.05$ N can be caused by the appearance of secondary twins at a certain critical value of load or by a change in the average twin length in a jump-like manner.

On the other hand, collective movement of dislocations such as plastic rotations, whose structural carriers are the defects of disclination type, can lead to the formation of the dislocation sub-boundaries dividing the crystal into fragments [16]. The microfragmentation process also can have a threshold character manifesting itself through jump-like changes in microhardness.

Thus, the obtained results show that complex processes take place in a crystal under increasing load on indentor, when the interaction between deformation defects stimulating the appearance of new types of defects and self-organization processes in the defect subsystem becomes the prevailing factor determining the H value.

From the obtained data it follows that the $H(P)$ dependences are not always monotonous functions as it is usually supposed. In plastic materials, where the significant degree of deformation without destruction of a crystal is achieved, the transition from the micro- to macrohardness can be accompanied by phenomena caused by the collective processes in the subsystem of deformation defects, leading to the appearance of the plastic instabilities.

In the Bi single- and polycrystals under the microindentation the scale effect is observed: up to $P \simeq 0.05$ N the microhardness decreases with increasing load on indentor, and in the range of $P = 0.05$–0.15 N the $H(P)$ dependences exhibit the oscillatory behavior. The existence of the plastic instabilities is attributed to the collective processes taking place in the defect subsystem and resulting in the formation of the consecution of deformation structures under load increasing.

The existence of scale effect under microindentation, the complex character of the $H(P)$ dependences, and the presence of the plastic instabilities should be taken into account when choosing the optimal load on indentor.

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Нестабільності пластичної деформації у кристалах Bi при мікроіндентуванні

О.І. Рогачова, Г.А. Яковлева, С.Г. Любченко

Одержано залежності мікротвердості H моно- та полікристалів вісмуту від величини навантаження на індентор P. Спостерігається загальна тенденція до зниження H при зростанні P до ~0,15 N, після чого мікротвердість практично не змінюється. В інтервалі $P \simeq 0{,}05–0{,}15$ N на залежностях $H(P)$ виявляються осциляції, наявність яких пов'язується з якісними змінами у мікромеханізмах пластичної деформації та у дефектній структурі кристала при зростанні навантаження на індентор.