PACS: 61.72.Ff

V.A. Nadtochiy[1], V.P. Alyokhin[2], M.M. Golodenko[1]

MICROPLASTICITY OF SUBSURFACE LAYERS OF DIAMOND-LIKE SEMICONDUCTORS UNDER MICROINDENTATION

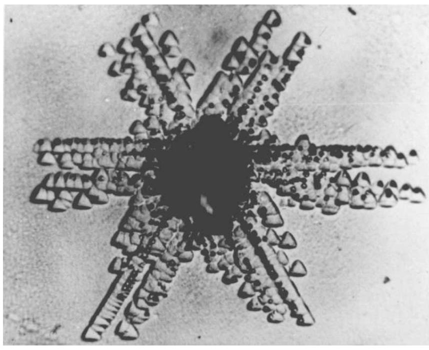
[1]Slovyansk State Pedagogical University
19 Gen. Batyuk St, Slovyansk, 84116, Donetsk Reg., Ukraine
E-mail: slavgpi@slav.dn.ua

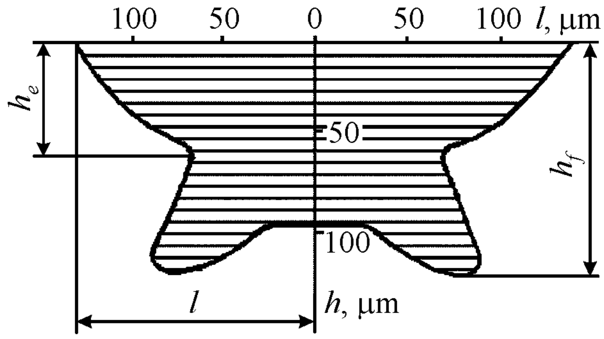
[2]Moscow State Industrial University
16 Avtozavodskaya St, Moscow, 109280, Russia
E-mail: topstaff@msiu.ru

Experimental confirmations of the influence of the free surface of a chip on processes of plastic deforming under indentation such as a decrease of the effective activation energy of dislocations with reduction of load on the indenter and a considerable decrease of temperature of the beginning of polygonization processes, when annealing microhardness rosettes in the field of small impresses, are obtained. A possibility of dislocation motion by means of creeping at temperatures lower than brittleness threshold temperature was shown on the example of GaAs.

The researches of last years have shown a possibility of plastic deformation in diamond-like chips below plasticity threshold temperature down to temperature of liquid nitrogen. However the mechanism by which the crystal form is changed is not completely clarified with reference to conditions of microindentation (the main technique of microplasticity detection in conditions of high brittleness of objects). A number of experimental data testifies to an essential influence of abnormal features of the facilitated origination and motion of dislocations in near-surface area of a chip on the general kinetics of the deforming of stuff, including the area below the brittleness threshold temperature. Considerations about possibility of non-dislocation mass transfer mechanism and about essential part of point defects in dislocation motion in covalent crystals under indentation have been suggested, though in the structural plan these problems have not been treated yet.

In this work a special role of the surface in dislocation motion facilitated, as contrasted to the motion in the volume, was investigated. By the straight structural methods on the example of GaAs the change of dislocation motion mechanism from slip to creeping at the transition into the temperature range of brittle failure has been shown. The point defects are playing the determining role in this process.





a *b*

Fig. 1. A dislocation rosette near to the indenter impress on the plane (111) of Si single crystal (test temperature 1000 K) (*a*) and the deformed area detected by the level-by-level etching (*b*)

The tests were conducted on Si single crystals. The sample was deformed with indenter on plane (111) at 1000 K by means of the device PMT-3 equipped with a heater. After processing with the selective Sirtl etchant a characteristic rosette (Fig. 1,*a*) from dislocations moving in slip planes {111} inclined to the chip surface and intersecting it along directions ⟨110⟩ was revealed near to the impress.

In Fig. 1,*b* the configuration of plastically deformed area obtained during the level-by-level analysis of dislocation structure is shown. The deformed area is shown in section along a ray of the rosette. It was important to find out how the deformed area changes at reduction of the penetration depth of the indenter into semiconductor. The ratios of the characteristic dimensions l/h_e and l/h_f were taken as criteria for estimation. Here l – the maximum ray length, h_e – bedding depth of rays, h_f – full bedding depth of dislocations.

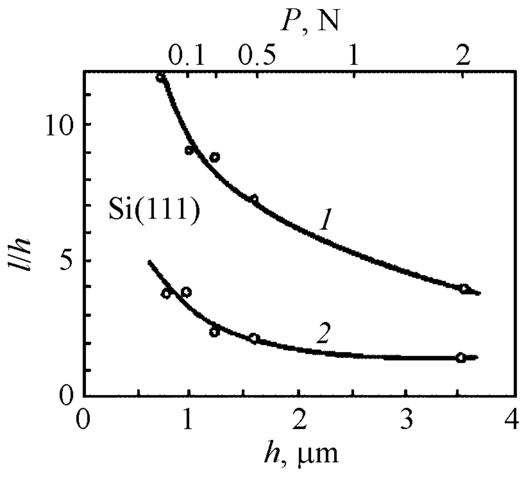


Fig. 2. The change of the geometrical parameters l/h_e (curve *1*) and l/h_f (curve *2*) of the deformed area depending on the penetration depth of the indenter. For curve *1* $h = h_e$, for curve *2* $h = h_f$

The results of researches in coordinates $l/h = f(h)$ are represented in Fig. 2. The obtained data demonstrate evident tendency to primary propagation of dislocation half-loops of the rays in a thin near-surface layer i.e. to some stretching of the deformed area along the surface, while the size of the impress decreases. The observed regularities can be explained from a position that motility of dislocations increases when deformations are localized in a thin near-surface layer [1].

It was of interest to find out, how the energy parameters of plastic flowing under indenter change when deformation is localized in a thin near-surface layer. With this purpose impresses of a standard Vickers indenter were made at temperature 300 K under loads from 25 up to 200 mN on a chemically polished surface of dislocationless mark GDG-10 Ge. Then indenter was removed and the chips were annealed in inert medium at different temperatures in the interval 700–800 K during 45 min. After detecting the dislocation structure in selective etchant the measurement of rays of maximum length were carried out (1-2 rays in each rosette). For each load the measurements were carried out on 25-30 rosettes.

A simple method of estimating the running velocity of single dislocations in chips with diamond grating from the analysis of dislocation rosettes detected near impresses and obtained at temperatures higher than the brittleness threshold or after the annealing of impresses made at room temperature was offered in [2]. The method is based on usage of an empirical equation for velocity of dislocations

$$\nu = B\tau^m \exp(-U/kT), \tag{1}$$

where U – activation energy of motion of dislocations; B, m – constants; τ – acting stresses, k – Boltzmann constant; T – absolute temperature. Having accepted stress acting on dislocation, which moves in the force field under indenter, according to [3]:

$$\tau = \frac{P(1-2\nu)y^2 - x^2}{\sqrt{6}\pi(y^2 + x^2)}, \tag{2}$$

where ν – Poisson's ratio; P – load on indenter; x, y – coordinates of an analyzed point, Kabler et al. [4] have derived the final relation for the maximum length of a rosette ray in the form

$$l \sim B^{\frac{1}{2m+1}} P^{\frac{m}{2m+1}} t^{\frac{1}{2m+1}} \exp\left(-\frac{U}{(2m+1)kT}\right), \tag{3}$$

where t – time of exposure under loading, constant $m \approx 1$.

This method allows to determine the value of activation energy of dislocation motion from the temperature relation $\lg l = f\left(T^{-1}\right)$. The values of effective activation energy of growth of dislocation rays in a rosette with temperature depending on load on the indenter are adduced in Table.

Table

Load on indenter P, mN	Activation energy U_e, eV
200	0.59 ± 0.09
100	0.51 ± 0.23
50	0.3 ± 0.2
25	0.26 ± 0.08

Adduced in Table values of U_e are much lower than that known from literature for Ge and obtained by direct measurement of the velocity of single dislocations and dislocations moving at the head of a line. Chaudhuri et al. [4] for dislocations moving at the head of a line have received $U = 1.6 \pm 0.05$ eV. The low limit of the spread corresponded to the highest tensions and upper one to the lowest. Kabler [5] has established that for single screw dislocations the activation energy $U = 1.47$ eV at all stresses and for 60° it changes from 1.49 eV at $\tau = 80$ MPa up to 2.25 eV at $\tau = 8$ MPa. According to Schäfer [6], who investigated mobility of stabilized single dislocations in the stress range 1–150 MPa, the activation energy is 1.62 ± 0.1 eV. Johnson [7] for high levels of stresses (up to 600 MPa) adduces $U = 1.4$–2.2 eV.

Our data in coordinates $l = f(T)$ are adduced in Fig. 3. The curves 5 and 6 are taken from [8]. They also are obtained while testing Ge single crystals on a plane (111), but at loads on indenter of 1 kN and 2 kN. It is known [8] that the maxima on curve $l = f(T)$ correspond to homologous temperature $t^* = T/T_m \approx 0.85$ ($T_m = 1209$ K – Ge melting temperature) at which a sharp temperature dependence of critical shearing stress begins. At the annealing temperature $t > t^*$ the process of formation of a dislocation rosette is more and more determined by the development of polygonization processes which hamper the scattering of dislocations in their slip planes. In the mentioned temperature range the rapid deceleration of growth of dislocation rays is observed. The value t^* characterizes a changing of the plastic deformation mechanism. It is intimately connected to rigidity of the crystal lattice relatively to motion of dislocations [8]. The results in Fig. 3 demonstrate that while load on the indenter is decreasing the maximum on the curves $l = f(T)$ is essentially moving into the area of lower temperatures down to $t \approx 0.55$.

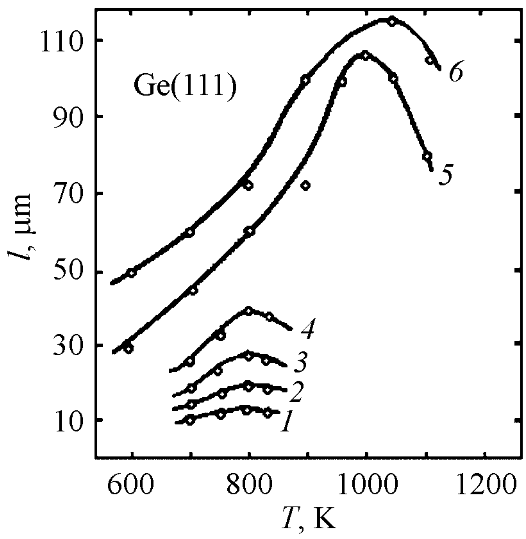


Fig. 3. Dependence of ray length of a dislocation rosette on the annealing temperature for the samples tested at room temperature by microindenting with different loads on the indenter P, 10^{-3} N: *1* – 25, *2* – 50, *3* – 100, *4* – 200, *5* – 1000, *6* – 2000

The results of the first and second series of experiments testify to changes of plastic deformation conditions at indenting thin near-surface layers of diamond-like semiconductors.

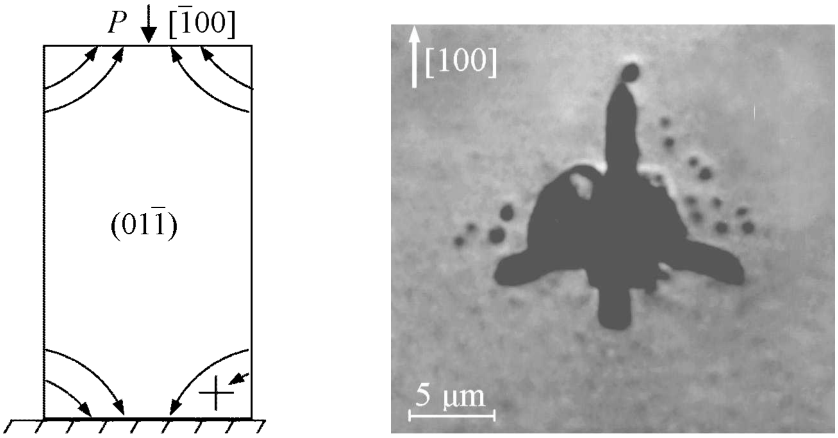
It was shown [9,10] that conditions of slip of dislocation loops resting on the surface of the chip do not correspond directly to any model [10–12] owing to essential influencing of the surface on motion of dislocations. Shown on the example of Ge and Si [1] the capability of the surface to act as a source and sink for vacancies comes to the accelerated movement of steps as a result of channel diffusion of point defects that eases the motion of bends. It is marked [13] that shape and size of dislocation loops at their thermally actuated slip in Ge and Si are largely determined by vacancy equilibrium concentration and rate of their diffusion, while in combinations A_3B_5, where differences in motility of adjacent 60°, α and β dislocations are very great, these parameters are determined mainly by concentration and rate of diffusion of vacancies of a definite type.

The determining role of point defects in the facilitated motion of dislocation loops near to the chip surface is demonstrated in our experiments on microindentation. The experiments were conducted in the temperature range of brittle failure of diamond-like semiconductors. The scattering of dislocations near to an indenter impress in the mentioned semiconductors at $T \approx 300$ K is not detected by optical methods [14] because of availability and stringent directivity of covalent bonds. At the same time experiments on the uniaxial pressing of Ge [15–18] as well as our experiments on microindentation of GaAs demonstrate a capability of scattering of dislocations if a chip with the marked impresses is subjected to a long-term deformation by uniaxial pressing.

GaAs (AGChT-1-25a-1) single crystals in the shape of rectangular parallelepipeds with dimensions of ribs 2.4 × 3.1 × 3.2 mm oriented in the stated succession accordingly to crystallographic directions $[01\bar{1}]$, [011] and [100] (Fig. 4,*a*) were used. The impresses were put on the lateral surfaces $(01\bar{1})$ and $(0\bar{1}1)$ at loads on the indenter of 200 mN. Then the sample was pressed along the direction [100] up to the stress σ = 83 MPa and was maintained under loading during 120 h at 300 K. After the removing of loading the dislocation structure shown in Fig. 4,*b* was detected near to the impress by means of selective chemical etching. Each couple of pits introduces apparently outlets of prismatic half-loop having stepped aside from the area of stress concentration by creeping. In Fig. 4,*a* the flow of vacancies from the right-hand side to the indenter impress is represented with the broken lines.

Under the uniaxial loading by pressure $p = \sigma_{xx}$ the equilibrium vacancy concentration on end faces reduces to [10,19]:

$$C = C_0 \exp(-\sigma_{xx} V_a / kT), \tag{4}$$



a *b*

Fig. 4. The deformed sample of GaAs with the marked indenter impress (labeled with a dagger). The arrows indicate flows of vacancies (*a*). The dislocation structure near to the indenter impress (*b*)

where C_0 –equilibrium vacancy concentration in the unloaded chip, V_a – atomic volume. For lateral surfaces $p = 1/3\sigma_{xx}$ and $C = C_0 \exp(-\sigma_{xx}V_a/3kT)$. The flow of vacancies from the lateral surfaces to the end faces (and to the indenter impress) appears owing to the arising difference of concentrations. Simultaneously the flow of atoms in opposite direction appears. Near to an impress there is a supersaturation on interstitial sites [20]. Prismatic interstitial dislocation loops can arise under the action of stresses and mentioned supersaturation. The flow of vacancies will promote their movement and partial stress relief near to the impress.

Thus in near-surface layers of diamond-like semiconductors the process alternate to the thermally actuated mechanism of overcoming high Peierls barriers by the slip of dislocations is realized. The thermally actuated mechanism requires, in the brittle failure field, stresses of the order of idealized shear strength of chips. It was shown that at high temperatures the motion of dislocations near to the surface can occur owing to simultaneous slipping and creeping [1]. That conditions high mobility of dislocations.

1. *L.S. Milevskiy, I.L. Smolskiy*, Fizika Tvyordogo Tela **16**, 1028 (1974).
2. *I.V. Gridneva*, Ph. D. Thesis, Kiev Polytechnic Institute, Kiev (1970).
3. *B. Rösner*, Acta Phys. Acad. Sci. Hungarical **20**, 289 (1966).
4. *A.R. Chaudhuri, J.R. Patel, L.G. Rubin*, J. Appl. Phys. **33**, 2736 (1962).
5. *M.N. Kabler*, Phys. Rev. **131**, 54 (1963).
6. *C. Schäfer*, Phys. Status Solidi **19**, 297 (1967).
7. *O.W. Johnson*, J. Appl. Phys. **36**, 3247 (1965).
8. *V.I. Trefilov, L.I. Datsenko, Yu.V. Milman, M.Ya. Skorokhod, I.V. Gridneva*, Metallofizika № 24, 187 (1968).
9. *L.S. Milevskiy, I.L. Smolskiy*, Doklady Akademii Nauk SSSR **212**, 591 (1973).
10. *V.P. Alyokhin*, Physics of Strength and Plasticity of Surface Layers of Stuffs, Nauka, Moscow (1983) (in Russian).
11. *V.V. Rybin, A.N. Orlov*, Fizika Tvyordogo Tela **11**, 3251 (1969).
12. *B.V. Petukhov*, Fizika Tvyordogo Tela **13**, 3679 (1971).
13. *L.S. Milevskiy, I.L. Smolskiy*, Fizika Tvyordogo Tela **17**, 1333 (1975).
14. *Yu.S. Boyarskaya, D.Z. Grabko, M.S. Kats*, Physics of microindenting processes, Shtiintsa, Kishinev (1986) (in Russian).
15. *V.P. Alyokhin, V.A. Nadtochiy, N.K. Nechvolod, M.Kh. Shorshorov*, Structure Defects in Semiconductors, Institute of Physics of Semiconductors, Novosibirsk (1973) (in Russian).
16. *V.P. Alyokhin, V.A. Nadtochiy, N.K. Nechvolod, M.Kh. Shorshorov*, Single Crystals of High-melting, Rare Metals, Alloys and Combinations, Science, Moscow (1977) (in Russian).
17. *V. Nadtochiy, N. Nechvolod, N. Golodenko*, Functional Materials **10**, 702 (2003).
18. *M.K. Nechvolod, M.M. Golodenko, V.O. Nadtochiy, Yu.M. Gritsenko, D.G. Sushchenko*, Zhurnal Fizychnykh doslidzhen' **4**, 298 (2000).
19. *A.M. Kosevich*, Uspekhi Fizychnykh Nauk **114**, 509 (1974).
20. *Yu.I. Golovin, A.I. Tyurin*, Fizika Tvyordogo Tela **42**, 1818 (2000).