

## МАТЕРІАЛИ ДЛЯ СЕНСОРІВ

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## SENSOR MATERIALS

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### STRUCTURE CHANGES IN GaAs CHIPS DEFORMED BY PRESSING AT 300 K

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#### Summary

#### STRUCTURE CHANGES IN GaAs CHIPS DEFORMED BY PRESSING AT 300 K

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Structure changes around an impress of indenter on GaAs single crystals were investigated. Samples in the shape of rectangular parallelepipeds with the sizes  $2.4 \times 3.1 \times 3.2 \text{ mm}^3$  conterminous correspondingly to the crystallographic directions  $[01\bar{1}]$ ,  $[011]$  and  $[100]$  were used. The impresses were being put on side surfaces  $(01\bar{1})$  and  $(0\bar{1}1)$  with loads on indenter 0.20 N. Then the sample was subjected to pressing along the direction  $[100]$  (the greatest dimension) up to the stress  $\sigma = 83 \text{ MPa}$  and was being maintained under the loading during 120 h at  $T = 300 \text{ K}$ . After the removal of the loading two types of dislocations were revealed by chemical discriminating etching near the indenter impress. They were the prismatic loops, which have issued from the stress concentration area in the process of creeping, and dislocations sliding along the planes  $\{111\}$ . Running off of dislocations along the cleavage planes as a result of splitting off of the chip was observed.

**Keywords:** GaAs, mechanical pressure, deformation, relaxation, dislocations.

#### Анотація

#### СТРУКТУРНІ ЗМІНИ В КРИСТАЛАХ GaAs, ДЕФОРМОВАНИХ СТИСКАННЯМ ПРИ 300 K

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Досліджувалися структурні зміни навколо відбитка індентора на монокристалах GaAs. Використовувалися зразки у формі прямокутних паралелепіпедів розмірами  $2,4 \times 3,1 \times 3,2 \text{ мм}^3$ , що відповідають кристалографічним напрямкам  $[01\bar{1}]$ ,  $[011]$  і  $[100]$ . Відбитки наносилися на бічні поверхні  $(01\bar{1})$  і  $(0\bar{1}1)$  при навантаженнях на індентор 0,20 Н. Потім зразок піддавався стисковій уздовж  $[100]$  (більшого виміру) до напруження

$\sigma = 83$  МПа і витримувався під навантаженням 120 годин при  $T = 300$  К. Після зняття тиску хімічним вибіркоким травленням виявлено два типи дислокацій поблизу відбитка індентора: призматичні петлі, що вийшли з області концентрації напруження шляхом переповзання, і дислокації, що ковзають по площинах  $\{111\}$ . Виявлено розбіг дислокацій по площинах спайності в результаті сколу кристала.

**Ключові слова:** GaAs, механічне стискання, деформація, релаксація, дислокації.

#### Аннотация

#### СТРУКТУРНЫЕ ИЗМЕНЕНИЯ В КРИСТАЛЛАХ GaAs, ДЕФОРМИРОВАННЫХ СЖАТИЕМ ПРИ 300 К

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Исследовались структурные изменения вокруг отпечатка индентора на монокристаллах GaAs. Использовались образцы в форме прямоугольных параллелепипедов размерами  $2,4 \times 3,1 \times 3,2$  мм<sup>3</sup>, совпадающими соответственно с кристаллографическими направлениями  $[01\bar{1}]$ ,  $[011]$  и  $[100]$ . Отпечатки наносились на боковые поверхности  $(01\bar{1})$  и  $(0\bar{1}1)$  при нагрузках на индентор 0,20 Н. Затем образец подвергался сжатию вдоль  $[100]$  (большого измерения) до напряжения  $\sigma = 83$  МПа и выдерживался под нагрузкой 120 часов при  $T = 300$  К. После снятия давления, химическим избирательным травлением выявлено два типа дислокаций вблизи отпечатка индентора: призматические петли, вышедшие из области концентрации напряжений путем переползания, и дислокации, скользящие по плоскостям  $\{111\}$ . Обнаружен разбег дислокаций по плоскостям спайности в результате скалывания кристалла.

**Ключевые слова:** GaAs, механическое давление, деформация, релаксация, дислокации.

Investigation of the thin spectrum of relations pressure–deformation and deformation–time in diamond-like semiconductors Si and Ge has allowed finding out microplastic deformation in all temperature interval 77-600 K [1-5]. It was ascertained with the help of optical and electron microscopy, that the microplasticity in these single crystals at the mentioned temperatures is conditioned by the origin and motion of dislocations exclusively in near-surface layers which have distinguishing structural and electrical properties as well as dynamics of crystal lattice. Owing to the stated causes and to a number of others [1] dislocations in Ge and Si single crystals, including those which originally had no dislocations, occur at stresses on some orders of magnitude smaller than that needed for homogeneous origin. The specific role of a surface in dislocation multiplying consists in the fact that a surface, being a potent source of vacancies, intensifies processes of creeping. Thus in Ge and Si is realized the process alternate to the thermally activated mechanism of overcoming of high Peierls barriers by slip of dislocations. As is well known, the mechanism of

Peierls requires stresses about idealized shearing strength of the chips.

The low-temperature plasticity at pressing in diamond-like semiconductors of the group  $A_3B_5$  (GaAs, GaP, InP, InSb etc.) with ionic component of bonding forces is studied less. The deformation by pressing of GaAs at low temperatures was being investigated in works [6-9]. It was shown that in the temperature range 290-420 K the deformation was created by twinning simultaneously with a slip of full dislocations, but very high tensions (0.4-0.6 GPa) were needed for activation of the plasticity. It is marked [10] that at middle and low temperatures the plasticity of GaAs is determined by the mechanism of Peierls.

While surface of semiconductor combination  $A_3B_5$  is indented, prismatic loops remain fixed [11]. At the same time it was visually shown on Ge single crystals that the dislocations near to an indenter impress can move at 300 K if the chip was subjected to a long-term axial pressing deformation [12]. It was to be expected therefore that using a similar technique of deformation it would be possible to watch scattering of dislocations on

GaAs single crystals as well. Besides it was of interest to establish whether the motion of dislocations is possible in the field of low temperatures at small and middle stresses in near-surface layers of these chips, where realization of diffusion–dislocation microplasticity is possible.

### Experimental technique

Samples of GaAs single-crystal (trade-mark AGChT-1-25a-1) in the shape of rectangular parallelepipeds with the sizes  $2.4 \times 3.1 \times 3.2 \text{ mm}^3$  conterminous correspondingly to the crystallographic directions  $[01\bar{1}]$ ,  $[011]$  and  $[100]$  were used in the experiment. Imperfections from the chip surfaces were removed by grinding with dusts ASM-3, ASM-1 with subsequent chemical polishing in solution  $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$  (solution 33,9%) = 3+1. With the help of a microhardness gauge PMT-3 the indenter impresses have being put on side surfaces  $(01\bar{1})$  and  $(0\bar{1}1)$  with loads on indenter 0.20 N. Then the chip with the impresses had been pressed through plastic gaskets along the direction  $[100]$  and was being maintained under the loading at the stress  $\sigma = 83 \text{ MPa}$

during 120 h. As it was shown in the work [4], such stress level was enough for originating a microplasticity in GaAs. After unloading samples were etched in chemical solution AB [13] for exposing of structure defects on different depth. It was established that after etching in such solution during 10 s the surface layer with thickness  $0.3 \text{ }\mu\text{m}$  was removed. Before each etching and after it the surface of the sample was photographed.

### Results and discussion

First of all it was important by creation of directional flows of point defects to realize in experiment conditions, needed for creeping of dislocations. It is known [14] that a difference of a vacancy concentration can be created in a chip by uniaxial pressing or stretching. So, application of a pressing stress (Fig. 1a) increases probability of formation of vacancies on side surfaces. Owing to it, a directed flow of vacancies to the butts is emerging, as it is shown in the same figure. If dislocations be introduced on the route of this flow, than it is possible probably to ensure their non-conservative movement, as it was observed in Ge single crystals [1].

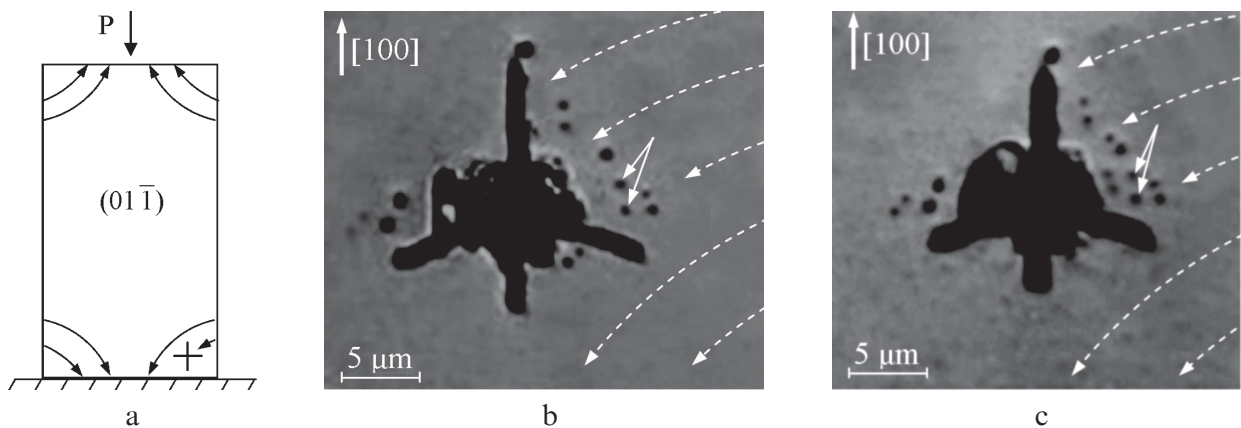


Fig. 1. a – a sample with an indenter impress (indicated by the cross) deformed by the pressure  $p = 83 \text{ MPa}$ . The arrows indicate flows of vacancies; b – dislocation structure near to the indenter impress on the depth  $3 \text{ }\mu\text{m}$  from the surface; c – the same impress after supplementary removing of surface layer  $3.3 \text{ }\mu\text{m}$  in thickness.

The indenter impresses were put on the side surface of GaAs chip on the distance  $110 \text{ }\mu\text{m}$  from the lower butt (near the right-hand edge in Fig. 1a). After exposure of the chip under loading, a dislocation structure (Fig. 1b) in the form of dislocation half-loops, having moved away from the impress (one of them is indicated on the figure by a pair of arrows), was revealed by etch-

ing. On the depth  $3.3 \text{ }\mu\text{m}$  additional loops have appeared (Fig. 1c). But after removing by the chemical etching of the layer  $5\text{--}7 \text{ }\mu\text{m}$  in thickness, dislocation were not found. It testifies that the dislocations had been moving in a near-surface layer. The sizes of loops made  $\sim 2\text{--}4 \text{ }\mu\text{m}$ . An attention must be paid to the fact that the dislocations are moving in the direction opposite to that

of vacancy flow (in Fig. 1b and 1c). The directed flow of vacancies is indicated by dotted lines. It is possible to suppose the following mechanism of the dislocation movement. Near to the indenter impress there is a supersaturation on interstitials [15]. Under the action of stresses and mentioned supersaturation, prismatic interstitial dislocation loops can arise. The flow of vacancies to the impress will promote counter movement of interstitials. Thus the loops moving away in the beginning will be extended with the interstitials and augmented in the sizes. On large distances, where the supersaturation on interstitials decreases and the vacancy concentration grows, they will decrease or will completely be dissolved. It is observed in Fig. 1b and 1c.

The relaxation of stresses near to an indenter impress, put on a side surface of the sample, hap-

pens also owing to generation of loops in definite slip planes (Fig. 2a). In Fig. 2a the dislocations, having moved away from the impress, lie in the plane  $(111)$ , and in Fig. 2b — in the cleavage plane  $(01\bar{1})$ . The observed structure can be treated as a set of dislocation loops, only one end of each loop having moved away from the impress, while the other end is fixed under the impress.

On the butt two types of dislocations were detected: sliding dislocations, lying in a plane of splitting off  $(101)$  (Fig. 3a), and half-loops (Fig. 3b), being arisen as a result of creeping. A feature of the last is the absence of their stringent orientation in the system of planes  $\{111\}$ , as it is observed at high-temperature tests by pressing. Let's mark, that the observed half-loops are deposited in a thin near-surface layer and arise at reduced stresses.

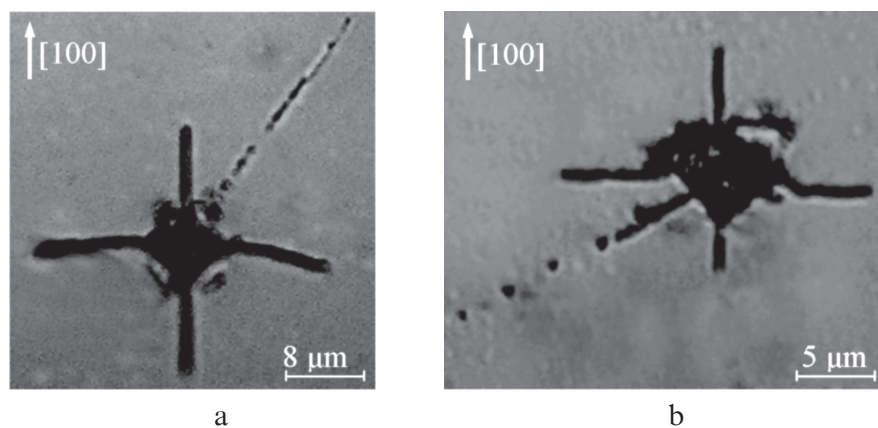


Fig. 2. a – a train of dislocation pits having moved away from the indenter impress along a line of intersection of a plane  $(01\bar{1})$  and a slip plane  $(111)$ ; the structure is photographed after removing of the layer  $0.25\ \mu\text{m}$  in thickness; b – a number of dislocation etching pits along the line of intersection of a surface  $(01\bar{1})$  and a cleavage plane  $(10\bar{1})$  on the depth  $1.5\ \mu\text{m}$ .

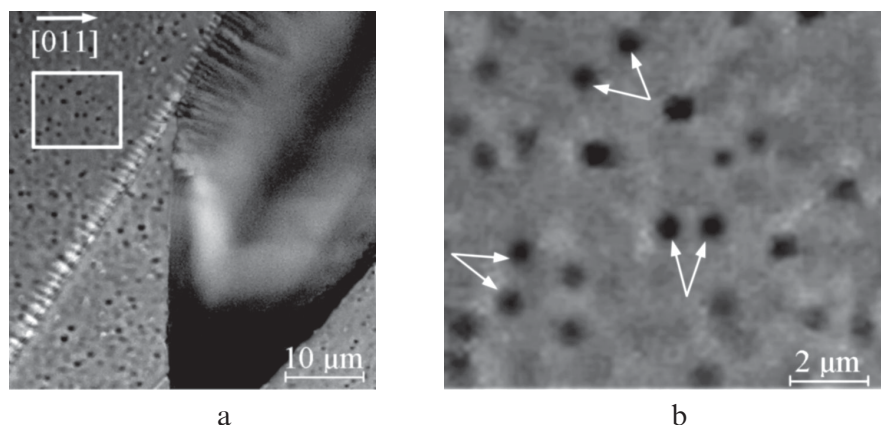


Fig. 3. a – the dislocation structure on the butt surface  $(100)$  with cracking along the plane  $(101)$  revealed by discriminating etching; b – the enlarged image of the part of the butt enclosed with the rectangle in Fig. 3a. The outlets of half-loops are indicated.



## Conclusions

Thus, the adduced results demonstrate that in semiconductor combinations GaAs, as well as in elementary semiconductors (Ge, Si), the microplastic deformation in the temperature range of brittle failure is controlled by diffusion processes. It is necessary to allow the detected features of motion of dislocations in materials with high Peierls barriers when designing sensor devices on the basis of diamond-like semiconductors.

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