INFLUENCE OF REST ON MECHANICAL PROPERTIES OF GaP SINGLE CRYSTALS DEFORMED BY SCLEROSCOPIC METHOD AT DIFFERENT TEMPERATURES

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Abstract

The areas of the GaP (111) plane placed between the scratches parallel to [110] have been investigated. The deformation degree of these areas was varied by distance between scratches (60-180 μm) and scratching temperature (T = 20, 150, and 450°C). It was obtained that the microhardness value (H) is smaller and the acoustic emission (AE) is more intensive for the areas with higher stress state. It was established that rest (~500 h) resulted in the AE significant increase for complete cycle of microindentation and emission in unloading. It was shown that the active formation of cracks and, especially, a material break off around the indents under microindentation of the investigated GaP zones have been observed while the AE increase; however, the H value in this case changed weakly. Obviously, after rest the revealed deformation acts differently influence the AE and microhardness changes. The essential changes of the relaxation coefficient (K) dependence on deformation degree as a result of rest have been revealed: K linearly increases with deformation degree increase before rest and monotonically decreases after rest.

1. Introduction

It is known that the structure defects and internal stresses of crystals are related to each other and influence their mechanical properties. The effects of external factors (temperature, impurity, deformation, etc.) cause new defect formation and transformation of existing in crystal defects, too. These processes are followed by an increase of the internal stress and changes of the mechanical properties [1-5]. It has been established that the modification of parameters characterizing mechanical properties (microhardness, yield stress, dislocation mobility, etc.) correlates with the changes of both internal energy and material structure and reflects the modification in crystal as a whole.

Emission of acoustic signals (AE) initiated at deformation is sensitive to modification of material structure state, too [2-4, 6-11]. The literature data evidenced the interrelation between the defect state of crystal and acoustic wave origination [2, 6-16].

This point is confirmed by many authors; for example, by the results obtained for silicium crystals: AE was observed on the samples with dislocations and was absent on the dislocation free samples [11]. The AE signals arising under microindentation of solids are mainly connected with the generation and further evolution of cracks [2, 4, 6, 7].

External action on crystal is as a rule followed by the definite structure rearrangement causing changes of both strength and AE signal number. Accordingly, the study of correlation between changes of defect structure state, microhardness magnitude as well as emission of elastic waves under microindentation is important as favors the understanding of the material deformation nature [2, 6-9]. However, relation between these features is insufficiently studied. The problem is especially significant for semiconductors used as a device material and accordingly, subjected to action of the different stress fields (mechanical, thermal, electrical, etc.).

Taking into account the problem importance, the influence of preliminary deformation by scratching on microstructure, microhardness value, and AE intensity arising under microindentation of the GaP single crystals has been studied.

2. Experimental procedure

The investigations were carried out on (111) plane of GaP single crystals grown by Chochralsky method [5]. The samples of the 1x8x10mm size were studied. The preliminary deformation by scratching along [110] has been introduced. The indentor load (P) under scratching was equal to 20 g. Deformation degree and, accordingly, the value of internal stresses are varied by changing distance (**d**) between scratches over 40-180 μm. Investigations in the middle of zones between scratches were carried out. In these zones the different microstructure and deformation degree in dependence on distance between scratches were formed, and in this case the smaller **d** the higher deformation degree. The scratching temperature, i.e., deformation temperature (T_{def}) was equal to 20, 150, and 450°C.

The influence of subsequent aging of 500-hour duration at room temperature on the studied parameters has been investigated, too.

The microhardness (H) and acoustic emission (AE) arising under microindentation were used for investigation. Microhardness was measurement by means of PMT-3 device, the Vickers pyramid was used. The acoustic emission impulses were registrated using the special accommodation to PMT-3. Besides, the number of signals for the whole microindentation cycle (N), under indentor penetration (loading) (N_1) and under indentor removal from sample (unloading) (ΔN) were separately registered. Microhardness measurements are carried out under indentor load (P) 50 g, and registration of AE signals -100 g. Coefficient $\Delta N/N$ for estimation of relaxation under unloading was introduced.
The scanning and light microscopes were used.

3. Results and Discussion

The dependence of AE signal number on deformation degree is listed in Fig. 1. It is seen that with deformation degree decrease N and N_1 also decrease: by a factor of 1.5 and 2, respectively. So, in the GaP zones with more deformation degree a more intensive acoustic emission is initiated. Another character of N(d) dependence under unloading has been revealed: AE activity is practically independent of deformation magnitude, showing, however, the minimum at d = 120μm. Zone at d = 120μm coincides with the zone of change of relation between N_1 and ΔN. The $N_1(d)$ curve for region $d<120$ μm is placed above the $\Delta N(d)$ ones, in this case $N_1>\Delta N$.

However, for region $d>120$ μm, alternatively, $N_1<\Delta N$: curve 3 is placed above curve 2 (Fig. 1). This means that the mechanism of accumulation and radiation of elastic energy under microindentation is changed depending on preliminary deformation degree. It can be suggested that under loading due to the external stresses in the slightly deformed zone the dislocation pilings are formed, which as a result of stress sign change at unloading (loading-unloading) break out, increasing the AE.

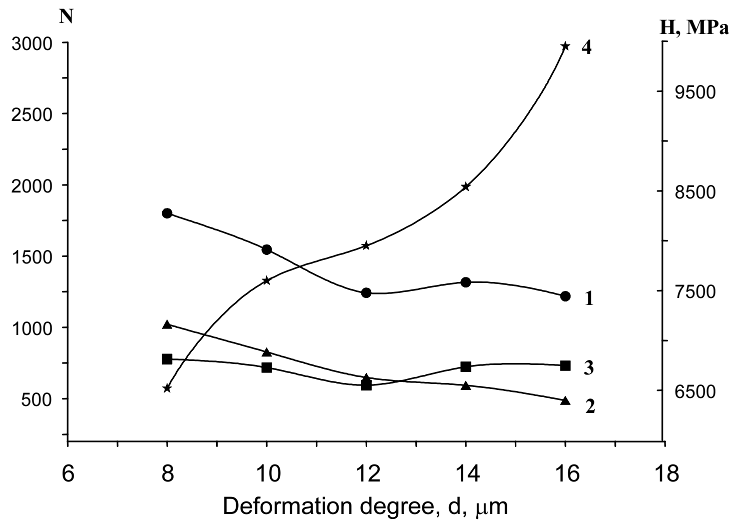

Fig. 1. Dependence of microhardness (4) and acoustic emission signal number on preliminary deformation degree: 1 - for whole cycle of microindentation; 2 - under loading (indentor penetrating); 3 - under unloading (indentor removal).

Curve 4 of Fig. 1 presents the dependence of GaP microhardness on preliminary deformation degree. It is seen that microhardness magnitude is essentially smaller for region of large deformation: with **d** increase H increases too and reaches the magnitude of the original GaP single crystals.

The results presented above correspond to the case when the preliminary deformation of GaP at T_{def} =20°C has been performed.

Besides, the preliminary deformation by scratching of GaP crystals at high temperatures (150 and 450°C) was performed. The AE signal registration at room temperature was carried out. Data for N are presented in Fig. 2. It is seen that common regularities for all deformation degree is observed: AE is smaller for T_{def}=20°C and the following relation is fulfilled – the higher T_{def} the smaller the acoustic wave irradiation. Besides, curves 1-3 are regularly placed lower with increasing of preliminary deformation temperature. This effect is especially pronounced in the temperature interval 20-150°C. In this region the temperature increase by 130°C caused the AE intensity decrease by ~40%, while in region of 150- 450°C the AE intensity decreases too, yet by ~ 20% (table).

Table. Changes of AE activity at rising of temperature of GaP single crystal preliminary deformation.

d, μm	N_{20}	N_{150}	N_{450}	$N_{20} – N_{150}$	A_1, %	$N_{150} –N_{450}$	A_2, %
8	1800	927	792	873	50	135	15
10	1545	918	740	627	40	179	19
12	1242	849	698	393	32	201	24
14	1316	733	587	613	46	146	19
16	1218	692	548	526	43	144	21

Here $A_1=(N_{20}-N_{150})/N_{20}$; $A_2=(N_{150}–N_{450})/N_{150}$; A_1 and A_2 are the coefficients of AE lowering at corresponding temperature rising; $N_{20}– N_{150}$ are the changes of AE impulse number at deformation temperature rising in the interval from 20 to 150°C; $N_{150} – N_{450}$ are the changes of AE at temperature rising in the interval from 150 to 450°C.

No regularities of A_1 and A_2 change on deformation degree are observed. The AE intensity decrease for the first temperature interval is twice as much as for the second one. In this case the effect is more pronounced in region of large deformation: $A_1 > A_2 \sim 3$ times. The sensibility of GaP structure state to deformation temperature is smaller within the region of d = 120μm (table). It is also seen from the table that for all the investigated deformation zones certain regularity takes place: the AE decreases with the T_{def} increase showing, however, the highest change rate in the first temperature interval.

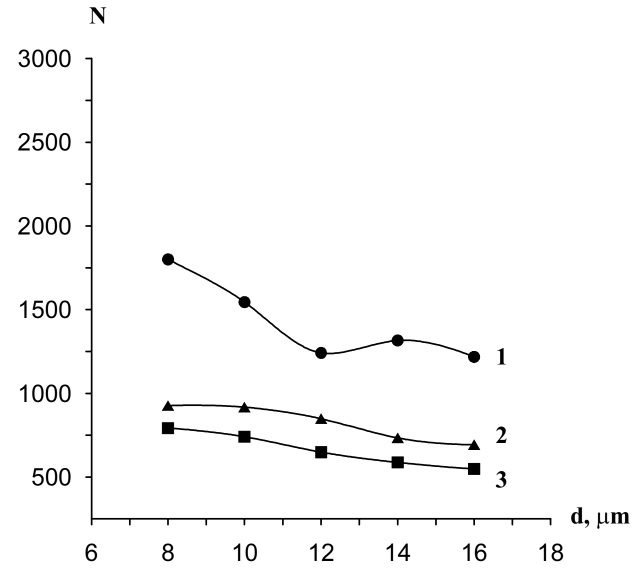

Fig. 2. Dependence of the acoustic emission impulse number for whole cycle of microindentation on preliminary deformation degree. Temperature of deformation, T°C: 20 (1), 150 (2), 450 (3).

Let us analyze this fact. Data from literature and our investigations have shown that the peculiarities of material structure are essentially determined by temperature of its formation.

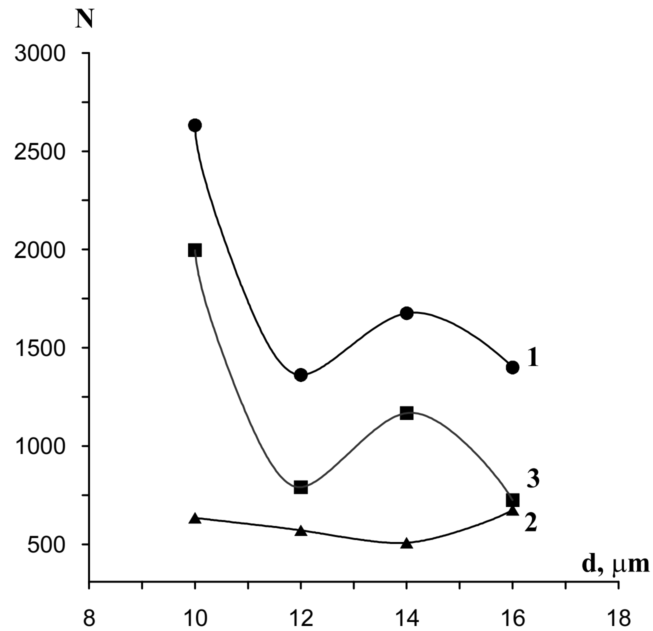

Fig. 3. Dependence of the acoustic emission impulse number on degree of preliminary deformation at 20°C for GaP single crystals after its rest: 1 - for whole cycle of microindentation; 2 - under loading (indentor penetrating); 3 - under unloading (indentor removal).

The structure for crystal regions with equal **d** and different T_{def} is not the same. At high T_{def} the deformation structure is more plastic, the probability of crack and microcrack initiation is small and dislocation run within imprint is larger, the deformation temperature is higher [5, 8]. The deformation zones formed at lower temperature are more elastic stresses. Within the indentation region the dense dislocation pile-ups, cracks, and breaks up are observed. Acoustic emission under microindentation reflected such difference of the deformation structure.

The different deformation structures are formed depending on deformation degree, too. The plasticity properties at small deformation degree are largely observed that correlate with the AE data of figures 1 and 2. In both cases (different temperatures as well as different deformation degree) the AE intensity decreases with increasing share (degree) of plasticity of the total GaP preliminary deformation. This allows us to conclude that the larger the deformation structure plasticity of GaP crystals the lower the acoustic emission activity under microindentation.

Dependences of AE signal number on preliminary deformation degree of GaP crystals after aging (rest) during 500 hours are presented in Fig. 3. It is seen that the curves of the N(d) and ΔN(d) dependences are non-monotonous; besides, the AE impulse number decreases with the deformation degree decrease. It is interesting that aging causes the opposite effect: AE does not decrease but essentially increases after rest (cf. Figs. 3 and 1). After aging, the uneven character of impulse number change under transition from one region to another was revealed too.

For example, transition from 100 to 120 μm resulted in the N decrease by more than twice after rest, while before rest it decreased by a factor of 1.3 only. More essential changes during aging in the relation between N_1 and ΔN were found. The curve of ΔN(d) is placed above the curve of $N_1(d)$ for all investigated deformation degrees. After aging the character of these dependences is modified, too: ΔN non-monotonously decreases with **d** rise, while N_1 slightly increases opposite to that observed before rest (Fig. 1). Especially marked arrangement of curves corresponds to the large deformation zones (d = 100 μm): $N_1 > \Delta N$ before and $N_1 < \Delta N$ after aging; besides, $\Delta N/N_1$ is equal to 0.9 and 2.9 before and after rest, accordingly. Relation between N_1 and ΔN for the low deformation zones as a result of rest does practically not change (Fig. 3).

The interesting results under comparison of AE activity before and after aging of deformed GaP were obtained (Fig. 4). It is seen that N and ΔN after rest increase: curves 2 are placed above curves 1. The effect is pronounced at all deformation degree, but it is more essential in large deformation zone. At d = 100 μm both N and ΔN increase by 1.7 and 2.8 times, accordingly. As **d** increases the enhancement effect decreases. The relative modification of N_1 and ΔN has been mentioned above.

Dependence of AE intensity enhancement on preliminary deformation degree after rest of GaP single crystals evidences that preliminary deformation before rest is the reason of the more active emission after rest.

Note that the GaP microhardness slightly changes after rest, only a weak increase of H is observed in the region d = 100 μm. It follows that processes resulting in the acoustic emission modification after rest do not bear the full responsibility for the microhardness change. The obtained data allow us to conclude that elemental acts responsible for the microhardness and acoustic emission changes due to rest are not quite the same. The results of $K=\Delta N/N$ estimation confirm this conclusion (Fig. 5). It is seen that K regularly decreases with the deformation degree decrease (**d** increase) before aging, while after rest it sharply increases*). For example, at d = 100 μm after rest K increases by a factor of 1.8 (Fig. 5). For slightly deformed zones of GaP the K values before and after rest are nearly the same, even a certain K decrease after rest is observed. The difference of K(d) dependence courses before and after rest evidences that both self-correcting processes and crystal structure state created by preliminary deformation play a significant role during rest.

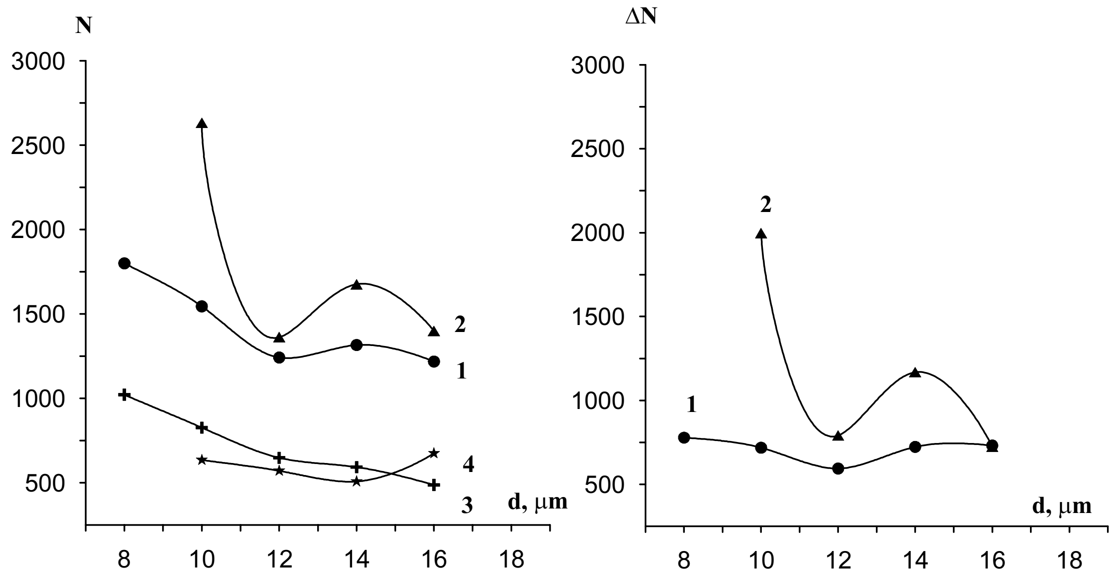

Fig. 4. Rest influence on dependence of acoustic emission on preliminary deformation degree: a) N – 1; 2, N_1 – 3; 4 b) ΔN – 1:2. Curves 1 and 3 – before rest, curves 2 and 4 – after rest.

Effects revealed in the present work for the GaP preliminary deformed by the following mode could be qualitatively explained. The structure of zones between parallel scratches is formed by the interaction of the deformation structures of individual crack.

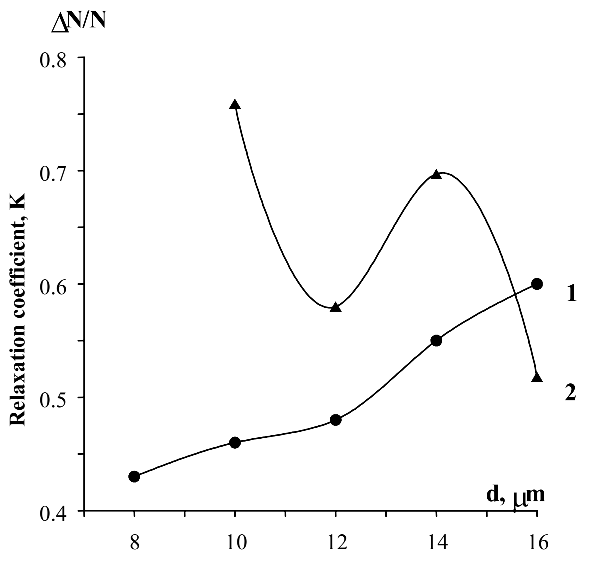

Fig. 5. Dependence of the relaxation coefficient on preliminary deformation degree of GaP single crystals during unloading: 1 – before rest and 2 – after rest.

*) The analogous courses of K(d) dependences for GaP preliminary deformed at 150 and 450°C were observed.

Value of the deformation stresses, character and form of deformation structure near the scratches are changing while moving away from its centre. The more powerful deformation close to the scratches is observed. Therefore, in dependence on distance **d,** the interaction of different level deformation in the centre between scratches occurs. As a result, the zones of GaP with different degree and state of joint deformation in the centre between scratches are formed. Therefore, the elastic stress relaxation and, accordingly, the AE activity will be inadequate, too. It can be suggested that the H and AE changes for different **d** are connected with difference of structure formed by joint deformation.

Fact that the AE under microindentation does not decrease, but, on the contrary, increases after rest of the GaP deformed zones, especially in the zone of strong deformation, is slightly unexpected (Fig. 4). The opposite effect could be more likely expected, as one can suppose that some of superstresses in the strong deformation zone relax in the process of GaP crystal aging [16]. On the other hand, structure of GaP more regularly established during rest is more hardening and deformation of such structures runs against some difficulties. This fact is probably responsible for the active creation near imprints after rest not only of cracks but also of the material cleavages, their number being higher in the region of strong deformation (Fig. 6).

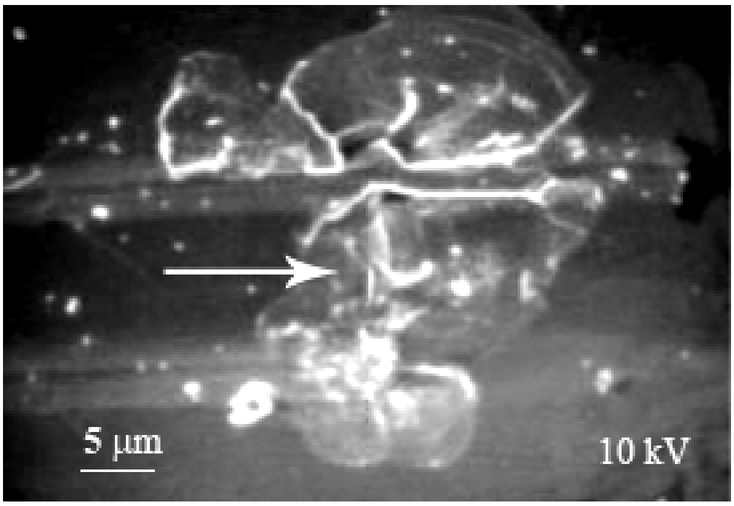

Fig. 6. Appearance of failure zone near the imprints arising under microindentation of (111) GaP between scratches after sample rest.

Initiation of the cracks and material cleavages leads to the observed AE increase after rest. The microhardness magnitude in this case is slightly changed. This means that the observed processes differently influence the AE and H modifications. Activity emission increase is pronounced at unloading, and at this stage of microindentation the well-defined cleavage appearance occurs. Sometimes the cleavages appear in 1-2 second after unloading. Correlation of these processes confirms that formation of the cleavages is one of sources of AE increase. The data also evidence that the AE summary account after rest is greatly determined by emission arising under unloading and, to a lesser extent, during loading of GaP crystals. Results given in Fig. 3 demonstrate this effect: curve 3 is placed above curve 2 after rest, the opposite arrangement was observed before rest.

Conclusions

1. There has been studied the influence of preliminary deformation injected into GaP single crystals by sclerometrical method and of their subsequent rest on microhardness and acoustic emission.
2. It was revealed that the microhardness magnitude is less and acoustic emission is more intensive for GaP regions with significantly greater stress state.
3. It was established that AE summary account of whole microindentation cycle, as well as during unloading process, strongly increases. The well-defined AE increase is probably connected with more active creation of cracks and, especially, of material cleavages near indents (prints) after rest. Microhardness magnitude in this case does not practically change. It was supposed that the revealed failure acts differently influence the change of microhardness and acoustic emission during rest.
4. There was obtained an essential modification of relaxation coefficient dependence on preliminary deformation degree as a result of GaP crystal rest. The K values after rest are greater than before rest. The dependence character also changes: K linearly increases with **d** increase before rest and non-monotonously decreases after rest. The effect is connected with difference of N_1 and ΔN modification during rest: ΔN sharply increases and N_1 weakly changes.

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