RELAXATION PROCESSES UNDER MICROINDENTATION OF InP CRYSTALS SUBJECTED TO SCRATCHING DEFORMATION

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Abstract

The elastic strain relaxation was estimated by the use of the method of acoustic emission signal registration under microindentation. The coefficient K for the estimation of relaxation degree under unloading was introduced. The preliminary strain degree of InP single crystals and accordingly the internal stresses were varied by the scratching method as well as by the testing temperature (293÷723 K) and storage of predeformed crystals at room temperature.

It was shown that the relaxation processes are determined by the stress defect structure state and are the predeformation temperature function. These processes are tightly related to the internal strain stress, characterizing the start state of InP single crystals.

1. Introduction

Action of different external factors causes the increase of internal stresses. Besides, the limit values of stresses (overstresses) in some sample regions are reached. This fact leads to formation of new defects or conversion of existing ones in crystals.

The energy part, released due to the defect structure transformation, i. e. due to the relaxation, goes into the energy of elastic waves. These oscillations are spread over the solid as a elastic waves of stresses and can be registered by means of the high sensitive sensor [1-3].

The mechanical deformation generates a great number of different type defects. Due to them the internal energy of solids, including the semiconductors, increases [4]. This leads to change of their physical properties, such as optical, electrical, structural, mechanical, etc. In turn this influences the material behavior in operation especially under different external field attack.

The internal defect and stress state of solids stipulates their capability to elastic strain relaxation too. The relaxation processes due to the structure ordering, as a rule, promote the internal energy reduction. These processes promote also the structure self-regulating and material stability, the more uniform stress distribution and the overstress removal. All effects are followed by the defect structure and stress field change. Accordingly the relaxation processes are important for stabilization of the strength and structure state of semiconductors too. The efficient use of semiconductors as devices essentially depends on their ability to accommodation.

Quite a number of articles, initiated to study the influence of the dislocations, impurity and native defects on many physical properties of semiconductor crystals, exists [4-7]. However investigations of the defect structure and internal stress influence directly on the relaxation processes in semiconductors are practically absent.

Accordingly the aim of the present paper is to study the influence of internal stress state introduced by preliminary deformation on the elastic mechanical stress relaxation of InP single crystals.

It is known that the elastic deformation relaxation is followed by irradiation of acoustic (elastic) waves, their emission essentially depending on sample internal energy, its stress and defect state [8]. The relaxation processes result in the internal (stored) energy decrease.

Method of acoustic emission (AE) is based on the registration of elastic waves, occurring under some internal processes (mechanical deformation, failure, phase transition, etc.). So, it was shown that the activation energy of relaxation processes related with structure transformation in the glassy materials can be determined by AE method using [9]. The plastic deformation heterogeneity and stress gradient are important conditions of acoustic emission existing. Such conditions are observed under the concentrated load action (microindentation). Clear-cut heterogeneity of plastic deformation and essential strength gradient occur under such deformation [10]. This deformation peculiarity occurred under microindentation promotes the relaxation process progress under both loading (indentor penetration) and unloading (indentor removal). The last events are followed by acoustic signal emission. In connection with the mentioned above the microindentation is very suitable for relaxation process study. The elastic strain relaxation was estimated by the use of the acoustic emission signal registration method under microindentation.

2. Experimental procedure

Pure InP (indium phosphorus) single crystals, grown by the Chochralsky technique, were investigated. The samples size amounted to 1×9×10 mm.

Preliminary deformation of (111) InP samples was performed by scratching along <110> directions. The indentor load for the scratches was equal to 20g. The distances between scratches (d) were varied within the interval 20-120μm. So, depending on the distance between the scratches, the crystal regions with the different strength degree (ε) were formed, and with d decreasing the deformation degree increases. So, ε decreases with the d increase, accordingly the internal stress decreases too.

For the sake of the internal stress change different temperatures of preliminary deformation (T) of samples (T=293÷723 K) as well as their annealing (storage) at room temperature during 0.5-5 months were employed too.

The elastic strain relaxation was estimated by the use of the method of acoustic emission (AE) signal registration under microindentation. Microindentation as an origin of the elastic mechanical waves, was made in the sample regions between the scratches. The Vickers diamond pyramid was used as indentor. The indentor load was equal to P=50g.

The AE signal registration was performed under indentation of InP samples by means of the PMT device, comprising the special acoustic installation. This installation allowed the AE signal registration under both indentor penetrating (loading) (N_1) and its removal from samples (unloading) (N_2). Therefore $N_1+N_2=N$ – is number of AE signals during whole indentation process. The AE signal registration was performed at room temperature.

The coefficient $K=N_2/N$ for the estimation of relaxation degree under unloading was introduced. Besides, the introduced coefficient shows that with K increasing the elastic strain relaxation becomes more essential.

3. Results and discussion

3.1. The influence of the preliminary deformation degree on the relaxation ratio

In Fig. 1 the relaxation coefficient dependences on the preliminary strain degree(d) at different temperatures (T) are presented. It is seen that the K coefficient depends on both d and T. It will be emphasized that both parameters (d — distance between scratches and T — redeformation temperature) characterize the structural and stressed state of InP single crystals and consequently their internal energy.

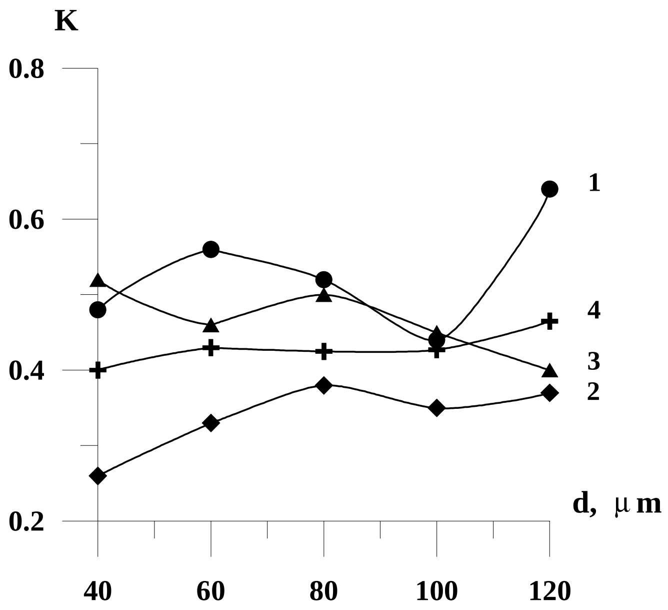

Fig.1. The relaxation coefficient dependences on the degree of preliminary strain, induced at T, K: 1-293; 2-423; 3-573 and 4-723.

Besides, the temperature elevation leads to the sample plastification and with d increase the preliminary strain degree decreases. We have therefore assigned the K(d) dependences are determined by stress state, introduced under the preliminary strain. All the K(d) dependences have pronounced non-monotonous character , however the extreme arrangement on the curves is determined by predeformation temperature. The dependences shape (curse) is also estimated by predeformation temperature. The K(d) curves for 293 and 423 K show the non-monotonous K increase with d increase, curves 1 and 2, Fig. 1. Weakly pronounced but similar behaviour is observed for 723 K, curve 4. In contrast, the K(d) for 573 K demonstrates another dependence character: coefficient K decreases, not increases, with d increase, Fig. 1, curve 3. Besides, in the latter case coefficient K achieves the minimal magnitude in the d=120 μm region, i. e. in the sample region predeformed more weakly, Fig. 1, curve 3. While the K for 293, 423 and 723 K in this region on the contrary reaches the maximal, not minimal, values, curves 1, 2 and 4.

It is also seen from Fig. 1 that relaxation processes, i. e. K magnitude, decrease with the preliminary deformation temperature elevation: curves 2, 3 and 4 are placed lower than curve 1 for practically whole d. This means that as the plastic strain contribution increases, consequently the elastic one to the total predeformation decreases, the relaxation activity under unloading becomes less.

In this case some peculiarities of the curve arrangements will be observed: curve 2 is placed not higher but much lower than curves 3 and 4. While the temperature due to curve 2 is lower than the ones due to curves 3 and 4, Fig. 1.

The revealed phenomenon is unusual, as the regularities of the K(d) curve arrangement are disturbed. Besides, this effect did not agree with the mentioned above that the plasticity contribution to the preliminary strain increases with the temperature rise that is accordingly followed by the relaxation process decrease.

For understanding of this effect nature the dependence of the AE signals, arising under indentor penetrating (loading process) (N_1), on temperature and degree of the preliminary strain was investigated. It is reasonable, because the strength state, created under predeformation, is bound to affect first of all the indentor penetration processes, i. e. loading processes.

The data of the N_1 measurements are presented in Fig. 2. It is seen from the figure that the number of AE signals under loading is maximal at 423 K. On curve 1 the clear-cut maximum suitable for T=423 K is seen. This effect is especially pronounced in the InP crystal regions, offering the maximal degree of predeformation (d=40 µm), Fig. 2, curve 1. The revealed effect does not correlate with K behaviour: coefficient K reached at 423 K not maximal, but minimal values, especially for d=40 µm,, Fig. 1, curve 2.

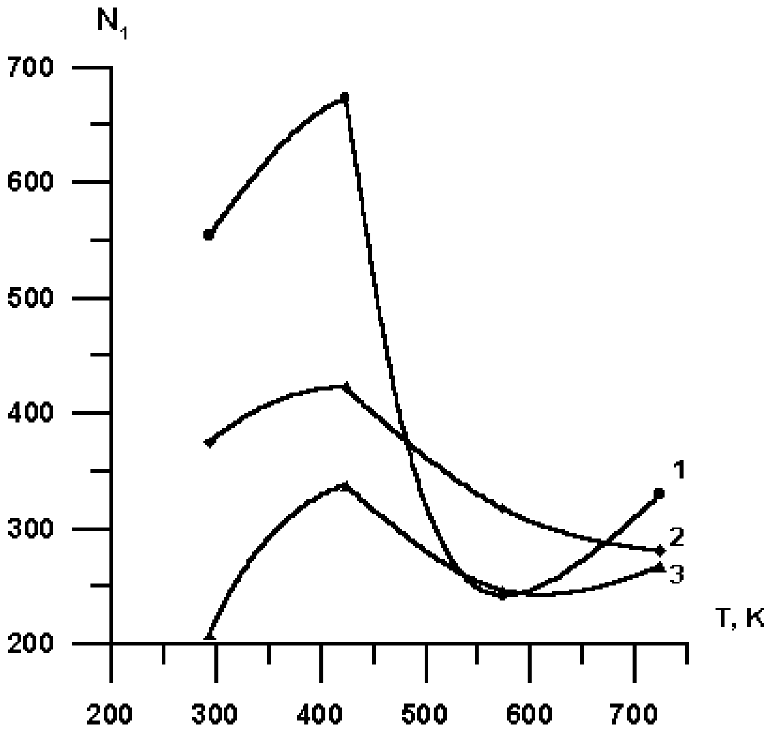

Fig. 2. The acoustic emission signal number dependence on preliminary strain temperature under indentor penetrating loading for different degree of deformation, d, µm: 1-40; 2-80; 3-120.

As $K=N_2/N$ is mainly determined by the emission of acoustic signals, emited under unloading (N_2), one needs to study the N_2 behaviour at the adequate conditions. It was experimentally shown that the N_2 (d) dependence curve for 423 K is placed lower than curves N_2(d) for 293; 573 and 723 K temperatures. This evidenced that the number of AE signals is minimal at 423 K in comparison with the one for rest temperatures. This result is opposite to the effect observed for N_1(d) dependences, Fig. 2. It is possible that during the InP crystal predeformation at 423 K, specific elemental acts of deformation are indeed formed. These ones promote AE signal intensity (activity) under loading and, on the contrary, do not assist in the AE signals under unloading. This evidences that the elemental processes under both loading and unloading are significantly different. Let us propose the following explanation.

It is known that the mechanical deformation of InP crystals is followed not only by dislocation gliding but also by microtwinning [11]. Microtwinning possesses the accommodation and relaxation character. This process promotes the dissipation of internal energy, accumulated during the plastic deformation. The latter is probably followed by the significant increase of AE signals [2]. However the microtwinning occurs not at all temperatures of deformation [12]. It was shown in [12] that there are no twins in the case of the InP single crystal deformation in the temperature interval from 300 to 700°C. Microtwinning occurs at lower deformation temperatures.

Analysis of the obtained experimental data and the accounting of the information from literature allow us to propose the following explanation of unusual behaviour of K (d) at 423 K. Probably the preliminary deformation of InP single crystals is predominantly followed by the origin of elastic twins, which under extra deformation (indentor penetration) are mainly relaxed. This process leads to the activation of the elastic wave emission, i. e. AE. In this case the internal energy of the deformed zone within the indentation decreases. Accordingly the latter results in the fall of the residual stress relaxation degree under unloading.

This effect does not take place at 573 and 723 K, as in this case the predeformation is not followed by the microtwin generation [12].

Thus, the peculiarity of K (d) dependence at 423 K is probably connected with the origin of the elastic microtwinns under predeformation and their annihilation under subsequent loading. However other factors as inadequacy of the thermoelastic stresses, the difference of the crack type and behaviour can bear responsibility for the above phenomenon too.

3.2. The storage influence of the preliminary deformed InP crystals on the relaxation ratio

The preliminary deformation at different temperatures allowed the defect and strength state of InP single crystals widely to change. The following “storage” of these samples at room temperatures resulted in the analogous effect.

The results of storage influence on the K changes are presented in Fig. 3. It is seen that the coefficient K sharply increases after storage (annealing at room temperature) for the InP

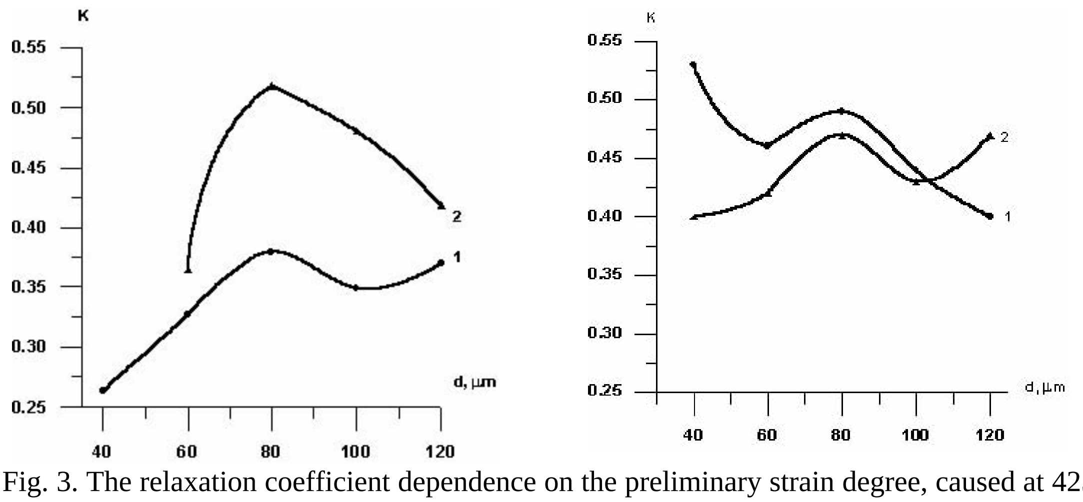

Fig. 3. The relaxation coefficient dependence on the preliminary strain degree, caused at 423 (a) and 573 (b). Curves 1 — direct after p redeformation; curves 2 — after storage during 5 months (a) and 0.5 a month (b).

crystals deformed at 423 K and, on the contrary, it decreases at 573 K: curve 2 is placed much higher than curve 1 in the first case, Fig. 3a, and it is placed lower in the second one, Fig. 3b. So, the opposite effects are observed: relaxation processes as a storage result intensify for 423 K and decrease for 573 K.

This fact confirms the assumption that the preliminary deformation at 423 K creates a special state of the defect structure and stresses in InP crystals.

The difference of relaxation effects for InP samples predeformed at 293 and 573 K (Fig. 1, curves 1 and 3) can perhaps be explained by the thermoelastic stress influence. The thermoelastic stresses are largely found in the second samples in comparison with the first ones. Probably the storage of InP samples predeformed at 573 K leads to thermoelastic stress decrease followed by the relaxation process decrease, Fig. 3b.

Thus, the obtained results show that the relaxation processes of elastic strains under unloading are highly sensitive to the internal deformation stresses and defects, characterizing the start state of InP single crystals.

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