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HALL-EFFECT STUDY OF DISORDERED REGIONS IN PROTON-IRRADIATED n-Si CRYSTALS

The nature and dimensions of disordered regions emerged in n-Si single crystals irradiated with high-energy (25 MeV) protons have been studied by carrying out Hall measurements of their electrophysical parameters. Specimens fabricated with the use of the zone-melting technique and doped with phosphorus to a concentration of 6×10^{13} cm^{-3} are investigated. Irradiation was carried out at room temperature to exposure doses of $(1.8\div8.1)\times10^{12}$ cm^{-2}. Depending on the irradiation dose and the temperature of isochronous annealing, some specimens irradiated with high-energy protons revealed a drastic increase of the effective Hall mobility $\mu_{\rm eff}$, which is explained by the emergence of "metallic" inclusions in them, i.e. regions with the conductivity considerably higher in comparison with that of the semiconductor matrix. The radius of those regions was estimated to be $R_m < 80$ nm. An assumption was made that the "metallic" inclusions are nano-sized atomic clusters.

Keywords: disordered regions, proton irradiation, effective Hall mobility, silicon.

1. Introduction

The electronic properties of crystalline semiconductors are traditionally controlled by doping the latter with chemical elements that create discrete electron levels in the energy gap of a material at a required depth and with a required density of states. However, this method cannot be implemented quite often because of either a low solubility of suitable impurities in the semiconductor matrix at a high concentration of electrically active intrinsic defects or the absence of such impurities at all. This circumstance provoked the development of an alternative approach (see, e.g., work [1]), which is based on the formation of nano-sized inclusions with required properties in the semiconductor crystal matrix. Small dimensions of those inclusions give rise to a confinement of the electron wave function by a potential barrier, which results in the formation of a discrete electron spectrum quite similar with that induced by impurities. Doping the semiconductors with nano-sized inclusions allows plenty of unique instrument structures, which are widely applied in electronics, in particular, in modern semiconductor-based lasers [2], to be developed.

According to their electrophysical properties, nano-inclusions can be "dielectric" or "metallic", depending on whether their conductivity is lower or higher than that of the semiconductor matrix. Semiinsulating or true dielectric inclusions can be obtained by passivating the material with the use of the acceptor (donor) diffusion in the regions with the electron (hole) conductivity [3] or oxidizing the semiconductor locally [4]. In addition, porous silicon, a substance with inclusions that have almost zero conductivity, is widely used in photonics [5], electric [6] and solar [7] batteries, and so on. Genuine metallic nano-precipitates [8] usually form Schottky barrier transitions in a semiconductor matrix and, for this reason, are almost opaque to charge carriers. As a result, such metallic inclusions behave themselves, in effect, as "dielectric" ones.

One of the most effective ways to create nano-inclusions in semiconductors is to bombard the latter with elementary particles or ions. The radiation technology for semiconducting materials uses irradiation of different nature and in a wide range of energies. For silicon, which is the basic material of modern solid-state electronics, the treatment by proton beams turns out especially effective. In particular, irradiation with high-energy protons (with energies higher than the so-called threshold energy of about 8 MeV) gives rise to so strong displacements of sili-

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con atoms from the lattice sites that, along with separate pairs of nonequilibrium vacancies and interstitial atoms in the lattice, there emerge nano-sized disordered regions in the form of clusters of similar point defects in the crystal structure [9]. In particular, irradiation with high-energy protons makes it possible to purposefully affect the electrophysical properties of the given material [10–13].

It is known that, in the volume of semiconductors irradiated with high-energy particles, there arise complex structural damages, the so-called disordered regions (DRs), which are the origin of specific modifications in the electric and galvano-magnetic properties of semiconductors. In work [14], the influence of irradiation with 30- and 660-MeV protons on the mobility of majority charge carriers, μ_H, and the efficiency of introduction of various radiation-induced defects (RIDs) in silicon crystals of the n- and p- types of conductivity were studied with the use of Hall and photo-Hall measurements. The results of measurements showed that the Hall mobility μ_H and the electron concentration n decrease, as the integrated flux of protons increases. Infrared (IR) illumination or isochronous annealing of irradiated specimens results in the growth of μ_H and n_H, which is explained by the ionization and the annealing of secondary RIDs in the peripheral parts of DRs. The efficiency of the RID introduction and the nature of RIDs formed in n-Si at its irradiation with 660-MeV protons at various temperatures within the interval $T_{irr} = 30 \div 700$ °C were also studied [15]. The increase of T_{irr} is accompanied by the growth of μ_H, which is explained by the weakening of the secondary RID introduction in the peripheral parts of DRs and, accordingly, by the reduction in the dimensions of those clusters. It can be said that the results obtained in works [14, 15] supplement each other. Their analysis has allowed the authors to make an assumption that the DRs consist of two parts: central (core) and peripheral (shell) ones. The DR cores consist of intrinsic structural defects (vacancy or interstitial associates), whereas the peripheral part of DRs is formed by the complexes of intrinsic defects with impurity atoms, i.e. the secondary RIDs (E-centers, A-centers, oxygen + divacancy, and others). If we managed to totally free the cores of those DRs from the influence of the impurity-defect shell (with the help of IR illumination, by increasing the temperatures of isochronous annealing, T_{ann}, or irradiation, T_{irr}), the temperature dependence of mobility, $\mu_H(T)$, would shift upward, toward higher $\mu_H(T)$ values. For this purpose, it is necessary that the DR cores should be actual clusters of interstitial atoms or their associates, and that they should not be annealed before secondary RIDs in the shell ($T_{ann} \geq 600$ °C).

If there are microscopic inclusions in the crystal, which are opaque to conduction electrons, i.e. "dielectric" inclusions, one may expect a reduction of the effective mobility, μ_{eff}, for charge carriers owing to a reduction of the specimen volume accessible to them [16]. In the opposite limiting case where the matrix conductivity can be neglected in comparison with that of inclusions, i.e. the latter are "metallic", μ_{eff} is an increasing function of the volume fraction of those inclusions. In silicon, the "dielectric" inclusions are defects of the vacancy type, and the "metallic" ones are defects of the interstitial type [16, 17]. The RIDs of those two types are known to actively interact with each other in silicon crystals. Thermal treatment at a temperature of 200–300 °C removes all vacancy complexes; this process may probably occur owing to the decay of interstitial complexes and their annihilation with vacancy defects [18]. The efficiency of the RID introduction into silicon crystals mainly depends on the specimen impurity composition and the energy of bombarding particles [19].

The aim of this work was to determine the origin and the size of DRs formed in n-Si crystals in the course of their irradiation with 25-MeV protons by analyzing the results of Hall-effect measurements.

2. Experimental Technique

We studied the specimens of single-crystalline silicon with the n-type of conductivity, which were grown up, by using the zone melting technique. The concentration of phosphorus was 6×10 cm^{-3}, the density of growth dislocations did not exceed $10^3 \div 10^4$ cm^{-2}, and the concentration of oxygen determined by the IR absorption method amounted to about 2×10^{16} cm^{-3}. Specimens in the form of bars $1 \times 3 \times 10$ mm^3 in dimensions were irradiated with 25-MeV protons at room temperature (≈ 300 K) to the exposure doses within the interval of $(1.8 \div 8.1) \times 10^{12}$ cm^{-2}.

Specimens were irradiated in the pulse regime, with a pulse duration of 25 μs and a pulse repetition fre-

quency of 0.25 Hz. The distribution of protons over the beam cross-section was uniform. The flux density was determined with an accuracy of 7% at the specimen arrangement place before irradiation. After the irradiation of the whole batch of specimens, the distribution of protons over the beam cross-section was checked once more. During the accelerator operation, the particle flux density and the proton distribution over the beam cross-section practically did not change. All specimens were irradiated in the beam section where the flux density was the most uniform and maximal (about 5.0×10^{12} particle/cm^2/pulse).

Specimens irradiated to doses of 8.1×10^{12} and 5.0×10^{12} cm^{-2} were annealed at a temperature of 90 °C for 10 min. After the annealing, those specimens were exposed to the natural aging at a temperature of 300 K for 30 days. The measurements were carried out both before and after the thermal treatment with a subsequent natural aging.

In the irradiated crystals, the Hall constant $R_{\rm H}$ and the electron concentration n were determined, by using the Hall method; the specific conductance σ was measured following the two-probe technique. The effective mobility was calculated by the formula $\mu_{\rm eff} = \sigma R_{\rm H}$, and the electron concentration by the formula $n = r_{\rm H}/(eR_{\rm H})$, where $r_{\rm H}$ is the Hall factor. The measurements were carried out in a temperature range of $77 \div 300$ K. The specimen temperature was monitored using a copper-constantan thermocouple with an error not worse than 2 K. The measuring thermocouple junction was put in touch with the specimen surface. The error in the determination of the electron concentration was mainly associated with an error in the determination of $r_{\rm H}$, the ratio between the Hall and drift mobilities. It is known that, in silicon of the n-type at temperatures ranging from the nitrogen to room ones, the value of $r_{\rm H}$ can exceed 1 by about 10%. Therefore, if we adopt $r_{\rm H} = 1$, the maximum relative measurement error for all those quantities should be about 10%. Below, we will see that the experimentally detected relative variations of the effective Hall mobility substantially exceed the indicated measurement error.

An additional proof of the reasonable accuracy of our measurements is given by the ionization energies E_i experimentally obtained for various RID-centers, which are determined from the temperature dependences of the electron concentration: these E_i-values agree well with literature data.

3. Results of Researches and Their Discussion

The temperature dependences of the electron concentration, $n(10^3/T)$, are depicted in Fig. 1. After irradiating the crystals to an integrated proton dose of 8.1×10^{12} cm^{-2}, the corresponding curve (curve *5*) reveals an exponential section that is associated with the exhaustion of acceptor centers with the energy $E_i = E_C - 0.36 \pm 0.04$ eV. In the specimens irradiated to a dose of 5.0×10^{12} cm^{-2}, we obtain $E_i = E_C - 0.42 \pm 0.04$ eV (curve *4*); and, at doses of 1.8×10^{12} or 2.7×10^{12} cm^{-2}, the deionization of A-centers is observed in the temperature range $77 \div 300$ K (curves *2* and *3*). In the case of the specimens irradiated to a dose of 5.0×10^{12} cm^{-2},

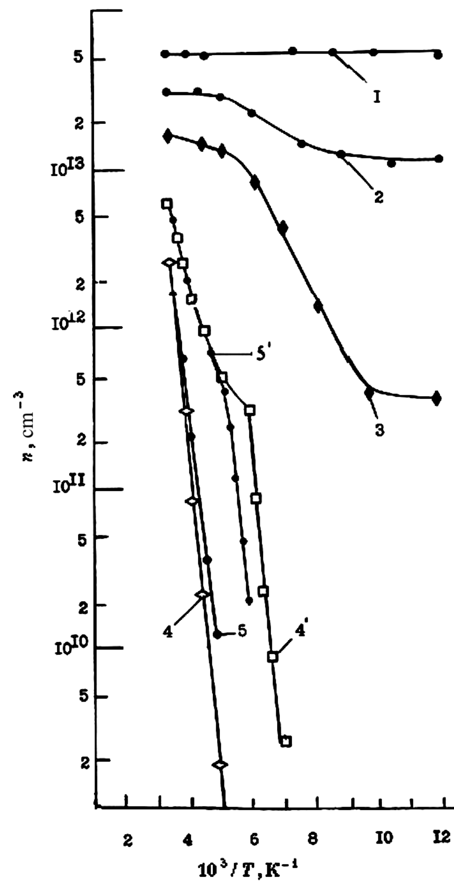

Fig. 1. Temperature dependences of the electron concentration before (*1*) and after (*2* to *5*, *4′*, and *5′*) irradiations with 25-MeV protons to doses of 1.8×10^{12} (*2*), 2.7×10^{12} (*3*), 5.0×10^{12} (*4*), and 8.1×10^{12} cm^{-2} (*5*); and after the annealing at 90 °C and the natural aging at 300 K for 30 days (*4′* and *5′*)

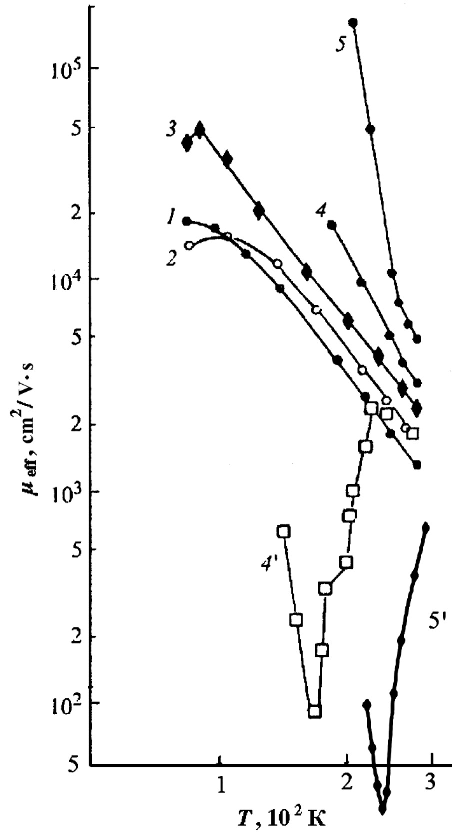

Fig. 2. Temperature dependences of the effective electron mobility before (*1*) and after (*2* to *5*, *4'*, and *5'*) irradiations with 25-MeV protons to doses of 1.8×10^{12} (*2*), 2.7×10^{12} (*3*), 5.0×10^{12} (*4*), and 8.1×10^{12} cm^{-2} (*5*); and after the annealing at 90 °C and the natural aging at 300 K for 30 days (*4'* and *5'*)

after their annealing and natural aging for 30 days at 300 K, the curve of the dependence $n(10^3/T)$ reveals an exponential section at $140 \div 170$ K, which corresponds to the exhaustion of the acceptor level with the energy $E_i = E_C - 0.36 \pm 0.04$ eV (curve *4'*). The curve of the temperature dependence of the electron concentration after the thermal treatment with a subsequent natural aging at 300 K for the specimens irradiated to a dose of 8.1×10^{12} cm^{-2} practically coincides with curve *4'* in the temperature interval from 170 to 300 K. We did not manage to carry out measurements at lower temperatures (curve *5'*).

In Fig. 1, curves *2* and *3* correspond to the exhaustion of A-centers, whereas curves *4*, *5*, *4'*, and *5'* to the exhaustion of E-centers and divacancies, although the ionization energy for those centers, owing to the electrostatic interaction between negatively charged secondary RIDs, changes [20]. One cannot exclude that the level $E_i = E_C - 0.42 \pm 0.04$ eV appears as a result of the superposition of divacancies ($E_C - 0.39$ eV) and E-centers ($E_C - 0.44$ eV). In this connection, we note that the fraction of divacancies usually amounts to about 0.9 [18].

In Fig. 2, the temperature dependences of the measured electron Hall mobility μ_{eff} are shown for specimens irradiated to various proton doses. For the initial specimen (curve *1*), the curve is described by the expression $\mu_{\text{eff}} \sim T^{-2.6}$. This fact testifies that the phonon mechanism of electron scattering dominates in the interval of measurement temperatures ($77 \div 300$ K). For the specimens irradiated to a dose of 8.1×10^{12} cm^{-2}, the dependence $\mu_{\text{eff}}(T)$ is shifted upward, and the exponent in the temperature dependence of the mobility reaches the value $\alpha \approx 13$ (curve *5*). If the exposure dose of irradiation decreases, the curve $\mu_{\text{eff}}(T)$ shifts downward, and α tends to the initial value (curves *2* to *4*). In the crystals irradiated to a dose of 5.0×10^{12} cm^{-2}, after their annealing at 90 °C and natural aging at 300 K for 30 days, one can observe an anomalously high scattering of electrons with the mobility minimum at 170 K (curve *4'*), whereas, in the specimens irradiated to a dose of 8.1×10^{12} cm^{-2}, the mobility minimum is observed at 220 K after their thermal treatment and aging (curve *5'*). The high values of mobility obtained in Hall measurements (curves *2* to *5*) are the evidence that the inclusions with rather a high conductivity and ohmic transitions at the boundaries with the semiconductor matrix are formed in the proton-irradiated n-Si specimens.

These experimental data allow us to calculate the volume fraction f_m and the average radius R_m of "metallic" inclusions in the examined n-Si specimens. According to the theory of effective medium, the Hall concentration of charge carriers in a material of this type is determined, in essence, by their concentration n in the relatively low-conducting matrix, whereas the effective Hall mobility is an increasing function of the inclusion volume fraction f_m. If, for simplicity, we adopt that the inclusions have spherical shape, then

$$\mu_{\text{eff}}/\mu_{\text{H}} \approx (1 + 3f_m)/(1 - 6f_m), \tag{1}$$

where μ_{H} is the Hall mobility in the matrix [17]. At room temperature, point-like RIDs in silicon can

only weakly affect the mobility of charge carriers; therefore, the value of electron Hall mobility in the initial material (≈1400 $cm^2/V/s$) can be taken for the parameter μ_H. The value of electron Hall mobility measured at room temperature after the irradiation to a dose of 8.1×10^{12} cm^{-2} amounts to $\mu_{eff} \approx 4500$ $cm^2/V/s$. So we obtain that $f_m \approx 0.10$. This is a reasonable estimation for the total volume fraction of clusters of interstitial atoms, which is typical of a real silicon structure subjected to irradiation with light ions and thermal treatment [19, 21].

Knowing the volume fraction of "metallic" inclusions f_m, we can determine their average radius R_m. The number of inclusions, regarded as DRs, in the whole specimen amounts to

$$N_{DR} = n_{DR} V, \quad (2)$$

where n_{DR} is the concentration of such inclusions, and V is the volume of the specimen under investigation. The volume occupied by the inclusions equals

$$V_{DR} = f_m V, \quad (3)$$

and the volume of one inclusion is

$$v_{DR} = V_{DR}/N_{DR} = f_m V/n_{DR} V = f_m/n_{DR}. \quad (4)$$

If the inclusions are spherical,

$$v_{DR} = 4\pi R_m^3/3. \quad (5)$$

From whence, we obtain

$$R_m = (3 f_m/4\pi n_{DR})^{1/3}. \quad (6)$$

According to our estimations obtained on the basis of the data of works [16, 19, 22, 23], the irradiation of Si crystals with 25-MeV protons to a dose of about 10^{13} cm^{-2} gives rise to the formation of clusters in the specimen volume with a concentration not lower than $n_{DR} \approx 5 \times 10^{13}$ cm^{-3} (it was supposed that every proton produces at least one DR). If we substitute the estimates obtained for f_m and n_{DR} into formula (6), the upper limit for the average radius of "metallic" inclusions will be equal to $R_m \approx 80$ nm.

A decrease of the proton exposure dose is accompanied by a reduction in the concentration of "metallic" inclusions, which can explain the observed decrease in the effective mobility of conduction electrons, μ_{eff}, in n-Si specimens (Fig. 2, curves *2* to *5*), because the Hall mobility is a growing function of the volume fraction f_m of such inclusions [17].

Clusters of interstitial atoms, like dislocations, create elastic stresses in the crystal. Some of nonequilibrium vacancies emitted by the DRs of the vacancy type in the course of annealing and natural aging and moving toward those clusters recombine with interstitial atoms. The others enter into a quasichemical reaction with impurity atoms in their vicinity and create a shell composed of acceptor centers [24]. It seems that the filling degree of deep centers in the shells around "metallic" inclusions tends to 1, as the temperature decreases. If so, the "metallic" inclusions become opaque to electrons, i.e. they transform into quasidielectric inclusions, which effectively scatter the conduction electrons. As a result, μ_{eff} starts to decrease, as the temperature falls down (Fig. 2, curve *4′*).

To estimate the volume fraction of "dielectric" inclusions, f_d, we may use the following approximate expression (by analogy with formula (1) for "metallic" inclusions):

$$\mu_{eff}/\mu_H \approx (1 - f_d)/(1 + f_d). \quad (7)$$

In the specimens irradiated with 25-MeV protons to a dose of 5.0×10^{12} cm^{-2} at 300 K, the "metallic" inclusions are not screened completely. Therefore, $\mu_{eff} > \mu_H$ at this temperature. As the measurement temperature decreases, the screening degree grows, and, at 170 K, the volume fraction of initially "metallic" inclusions transformed into "dielectric" ones and the impurity-defect shell calculated by formula (7) amounts to $f_d \approx 0.46$, with $n_{DR} \approx 1 \times 10^{13}$ cm^{-3} at that. Hence, the average size of inclusions should not exceed $R_d \approx 220$ nm in this case. It is quite reasonable that the dimensions of "dielectric" inclusions are larger than the dimensions of their parent "metallic" ones, because the radius growth is associated with the formation of defect shell.

As the temperature falls down, the energy of electrostatic repulsion between negatively charged secondary RIDs in the cluster shell grows, and, at 170 K, its magnitude becomes comparable with the ionization energy of deep centers [20]. Electrons transit from these levels into the conduction band to become immediately captured by deep centers located in the crystal matrix, where the indicated repulsion energy is much lower than that in the shell around a "metallic" inclusion (we suppose that the RID concentration

in the impurity-defect shells around a "metallic" inclusion is considerably higher than that in the crystal matrix). Deionization of RIDs in the DR shell should bring about a reduction in the volume fraction f of inclusions and their average radius R. Really, for a temperature of 140 K, we obtain $f \approx 0.25$ and $R \approx 180$ nm. Accordingly, the degree of "metallic" inclusion screening decreases, and, below 170 K, $\mu_{\rm eff}$ starts to grow. Hence, our assumption that "metallic" inclusions are formed in n-Si crystals at their irradiation with 25-MeV protons can explain the existence of a minimum in the dependence $\mu_{\rm eff}(T)$. It seems that the non-monotonous behavior of the curve $\mu_{\rm eff}(T)$ is explained by the analogous character of the degree of screening of "metallic" inclusions in the course of measurements of $\mu_{\rm eff}$ in irradiated crystals in the temperature range $77 \div 300$ K. In the interval from 170 to 240 K, they behave as "dielectric" inclusions and, in the interval from 140 to 170 K, as "metallic" ones.

In the specimens irradiated to a dose of $8.1\times\times10^{12}$ cm^{-2} and afterward subjected to the annealing at 90 °C and the aging for 30 days at 300 K, the volume fraction of inclusions is equal to $f \approx 0.30$, for which formula (6) for the average radius of inclusions gives $R \approx 100$ nm. For those specimens, the curve $\mu_{\rm eff}(T)$ passes through a minimum at 220 K, and, below 220 K, starts to grow for the same reason as in the specimens irradiated to a dose of 5.0×10^{12} cm^{-2}. As was already mentioned [16, 17, 19], the "dielectric" inclusions stimulate a reduction, and the "metallic" ones do an increase of $\mu_{\rm eff}$. This fact explains the presence of minima at 170 and 220 K in the curves of the dependences $\mu_{\rm eff}(T)$ measured for the specimens irradiated to doses of 5.0×10^{12} cm^{-2} and 8.1×10^{12} cm^{-2}, respectively.

4. Conclusion

Hence, we found that, in n-Si specimens irradiated with high-energy protons, besides point-like RIDs and "dielectric" inclusions, "metallic" inclusions consisting of interstitial atoms can also be formed. At the interaction between such "metallic" inclusions and acceptor RIDs, the shells, which are opaque to conduction electrons, emerge around these inclusions. The charge states of defects concentrated in the shells change, by depending on the measurement temperature. Accordingly, the screening degree of "metallic" inclusions also varies, which can stimulate the observed non-monotonous change of the effective electron Hall mobility $\mu_{\rm eff}$ with the temperature. The estimation of the upper limit for the average radius of "metallic" inclusions in n-Si irradiated with 25-MeV protons to a dose of 8.1×10^{12} cm^{-2} gives the value $R_m \approx 80$ nm, with the volume fraction of those inclusions in the crystal being equal to $f_m \approx 0.10$ at that. A capability to dope silicon with highly conducting atomic clusters with the use of proton irradiation in the combination with a possibility to control their screening by means of certain annealing procedures open new prospects in the development of solid-state devices for micro- and nano-electronics.

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ДОСЛІДЖЕННЯ РОЗУПОРЯДКОВАНИХ ОБЛАСТЕЙ В ОПРОМІНЕНИХ ПРОТОННИХ КРИСТАЛАХ n-Si ЗА ДОПОМОГОЮ ХОЛЛІВСЬКИХ ВИМІРЮВАНЬ

Р е з ю м е

Метою роботи є дослідження природи і розмірів розупорядкованих областей, створюваних у монокристалах n-Si опроміненням високоенергетичними (25 МеВ) протонами, за допомогою холлівських вимірювань електрофізичних параметрів. Використано зонноплавлені зразки, леговані фосфором з концентрацією $6 \cdot 10^{13}$ см$^{-3}$. Опромінення проводилося при кімнатній температурі в інтервалі доз (1,8–8,1)$\cdot 10^{12}$ см$^{-2}$. У ряді зразків, залежно від дози опромінення і температури ізохронного відпалу, спостерігалося різке збільшення ефективної холлівської рухливості $\mu_{\rm eff}$, що пояснюється утворенням у зразках при їх опроміненні високоенергетичними протонами "металевих" включень, тобто областей з провідністю істотно вищою порівняно з провідністю напівпровідникової матриці. Радіус подібних областей оцінений як $R_m < 80$ нм. Висловлено припущення, що "металеві" включення є нанорозмірними атомними кластерами.