

STABILIZATION OF NANO-SIZED STRUCTURES IN THE VOLUME OF SINGLE-CRYSTALLINE SILICON FOR PHOTOCONVERTERS

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The paper considers the possibilities of stabilization of microstructures created in the latent tracks in single-crystalline silicon by multicharged ions from the nuclear fragments formed as a result of heavy element photofission in the process of hydrogenating. The presence of hydrogen in the amorphous silicon structures leads to the clustering of vacancies, intrinsic interstitial and impurity atoms. For quantum structures, passivated with hydrogen atoms, the annihilation process is slowed down. In the process of annealing the silicon structures the strong $(Si - H)_n$ -bonds prevent the defect annihilation and thus stimulate the processes of precipitation and clusterization. Hydrogen, filling the irradiation-broken bonds, neutralizes their electrical activity. Optimal conditions for stabilisation of such structures are determined: irradiation doses, methods of hydrogenation and dissociation of H_2 , annealing parameters.

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1. INTRODUCTION

A traditional approach to the problem of controlling the properties of semiconductor materials, based on the process of doping them with impurities creating certain electron levels in the forbidden zone, has principal restrictions exerting the influence on the efficiency of silicon photoconverters. Lack of effective methods of interface structure formation in the (c-Si) semiconductor, providing a maximum of charge carrier extraction from the photoconverter volume, is one of main causes for a low efficiency of photo-cells fabricated from silicon materials [1]. Radiation processes allow forming in the single-crystalline matrix of a silicon structure with nano-sized amorphous-microcrystalline filaments possessing an increased electrical conductivity [2]. Application of such-type structures improves the slow charge carrier extraction from the volume of the c-Si photocell emitter structure volume (see Fig.1). The quantum structures L_n, L_p, L'_n, L'_p are formed as a result of radiation processes in the $c - Si(p, n)$ -matrices by nuclear fragments of uranium-238 photofission at the electron accelerators KUT-1 ($E_e \sim 12 MeV$), EPOS ($E_e \sim 26 MeV$) with subsequent hydrogenation possibly combined with radiation process. The structures of $c - Si(p, n)$ matrices are subjected to fragmentation by the accelerated electron beam. The $p - n$ junction width δ_{p-n} is determined by the drift lengths of nonequilibrium current carriers, L_{cn}, L_{cp} are the diffusion lengths of

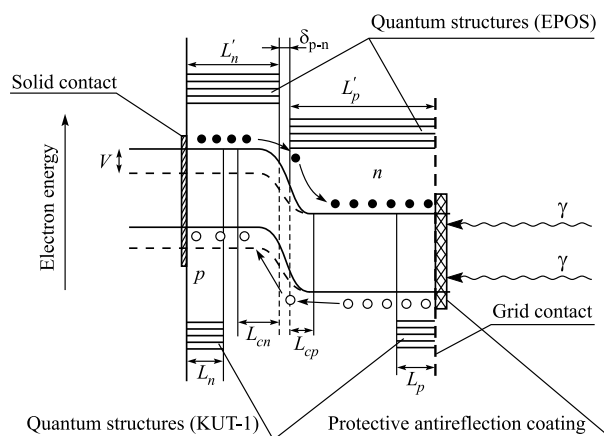


Fig.1. Structure format of a solar radiation-to-electric energy photoconverter having an increased efficiency

nonequilibrium carriers in the monocrystalline silicon. Amorphous phase stabilization and localized state density decrease in the mobility gap are provided by the technological hydrogenation process. A porous structure of disordering channels along the ^{238}U nuclear fission fragment path can promote the hydrogen migration to the amorphous phase boundaries in the $Si(p, n)$ matrix volume. The hydrogen presence in silicon leads to the clusterization of vacancies of intrinsic interstitial and impurity atoms. The main condition leading to the cluster formation consists in the simultaneous presence of hydrogen

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and oversaturated defect solutions in the structure. The interaction of hydrogen with impurities and defects initiates decomposition of their oversaturated solution with formation of hydrogen precipitates. As a result, there formed are passivated clusters not observed in the case of hydrogen absence. As the annealing temperature is increasing, the instable configurations become disappears while the strong $(Si : H)_n$ bounds prevent from the defect annihilation and thus stimulate the processes of precipitation and clusterization of intrinsic interstitials. Besides, hydrogen fills the broken bonds and neutralizes their electrical activity. Basing on the foregoing material, it can be concluded that cluster formation occurs in the hydrogenated silicon, containing radiation defects, in the course of the heat treatment. The electron structure of clusters reflects the internal structure and, here, the role of hydrogen is determining. Hydrogen atoms stimulate the defect formation in silicon, enter into the cluster composition and play a catalytic role in the cluster formation from finer defects. The migration process is described in terms of hydrogen atom capture and release by the energy-bound states. The hydrogen capture results in the formation of $(Si : H)_n$ cluster structures. The bound state density is changing as a function of the concentration of hydrogen, temperature and time. The average atomic displacement length is proportional to the distance between the hydrogen capture areas. The traps are the structure defects (vacancies, divacancies), grain boundaries and impurity atoms. The hydrogen diffusion occurs at the grain boundaries and between the lattice planes. In the case of

a low hydrogen concentration the cluster sizes are limited. In the case of a high hydrogen concentration the effective hydrogen diffusion is determined by the hydrogen atom capture with subsequent release from the cluster structures. If the source of hydrogen is unlimited, its migration in silicon and the profile of hydrogen concentration distribution in the material depth $C_H(x, t)$, to the region where its concentration coincides with the trap density, is described by the following equation [3]

$$C_H(x, t) = C_0 \operatorname{erfc}(x/S(t)), \quad (1)$$

where C_0 is the hydrogen concentration at the sample surface ($x = 0$), x is the diffusion depth, $S(t) = (4D_{eff}t)^{1/2}$, t is the process time; D_{eff} is the effective diffusion coefficient determined from the equation

$$D_{eff} = D_0 \exp(-E_a/k_B T). \quad (2)$$

The time of neutralization (passivation) of broken spin bonds and the formation of $Si : H$ cluster structures (see Table 2) are determined from the equation

$$t = t_0 \exp(E_a/k_B T), \quad (3)$$

where E_a is the activation energy, T is the sample temperature, respectively, $t_0 = 10^{-11}$ for $Si : H$ compounds. In the case of a high concentration of hydrogen atoms their diffusion does not depend on the degree of disordering of lattice atoms. The weak $Si - Si$ bonds are broken by hydrogen atoms and the spin-active centers are generated. Then, as a result of center passivation, additional $Si : H$ cluster structures are formed.

The time of Si:H cluster structures formation depending on the annealing temperature

	hour				min				
Time*	583	110	24	5.7	92	27	7.8	3	1.1
Temperature**	70	85	100	115	130	145	160	175	190

*- Time of Si:H compound formation; **-Annealing Temperature in °C.

Hydrogen is migrating in the material at a minimum concentration of traps determined by their energy state (0.5...1.7) eV. If hydrogen is captured by the $Si - Si$ bond centers, its diffusion into the material depth stops already in the thin near-surface layer. In the material structure the deep lying traps are first filled and then the shallow-lying ones. In the course of diffusion in the shallow-lying traps, an equilibrium hydrogen distribution is established, which, for the case of an unlimited hydrogen source is described by the Gauss distribution [3]

$$C_H(x, t) = \frac{Q}{\sqrt{\pi D_{eff} t}} \exp\left(\frac{-x^2}{4D_{eff} \cdot t}\right), \quad (4)$$

where $Q = \int N(x) dx C_0 \sqrt{D_{eff} t}$ is the total hydrogen introduced into the semiconductor through the surface unit during the diffusion time, $N(x)$ is the hydrogen concentration as a function of the distance

from the crystal surface x . According to equation 4 the surface concentration C_0 decreases with time and the diffusion layer thickness increases. To introduce hydrogen into the large depth from the crystal surface a long time is required because of its low solubility which is determined by the high value of the chemical potential, (1.2...1.3) eV. Therefore, using the existing means it is impossible to solve the problem of hydrogen migration into the silicon volume. A high hydrogen concentration at the required depth of Si material can be reached only by the forced rupture of $Si : (H)_n$ and H_2 bound states under irradiation. Besides, the hydrogen migration process is possible only in the temperature conditions given in Table 1, or at temperature breaking the bonds in complex silanes (< 550°C, but being insufficient for the $(a - Si)$ -structure crystallization. Hydrogen from Si-H bond can be released already at

350° C, if the ($Si - Si$) bond is formed instead of $Si - H$; at $\sim 550^\circ C$ a fast $Si - Al$ reaction takes place [4]. So, using the sources of isostatic hydrogen pressure and the technology of material radiation processing at the charged particle accelerators it is possible to shape a required profile of hydrogen concentration distribution in the path depth of uranium nuclear fission-fragment in the lateral track. Upon the strong amorphous structure hydrogenation to the level of cluster formation there can arise boron-hydrogen bonds in the $a - Si : H(B)$ -structures reducing the effect of increased radiation conduction. A maximum conduction can be recovered by the subsequent vacuum sample annealing during several minutes at $T \sim 300^\circ C$. Infrared spectroscopy results evidence on the existence of boron-hydrogen bonds in the $a - Si : H(B)$ -structures stabilizing the structure in the process of long-continued operation. Purpose of the work is experimental definition of conditions for stabilisation of metastable nano-size amorphous-microcrystalline structures in the bulk of monocrystalline silicon.

2. EXPERIMENTAL TECHNIQUE

Two variants of the hydrogenation process are developed:

- 1) posthydrogenation - after forming the quantum structures by the uranium nuclear photofission fragments (see Fig.2) there occurs a molecular hydrogen implantation into the silicon sample in the high-pressure gasostat (see Fig.3) with subsequent hydrogen molecule dissociation in the material structure at the low-energy electron accelerator.
- 2) simultaneous hydrogenation with formation of quantum structures by annealing them in the process of crystal radiation processing at the electron accelerator "EPOS" (26 MeV) above the photonuclear reaction threshold (see Fig.4).

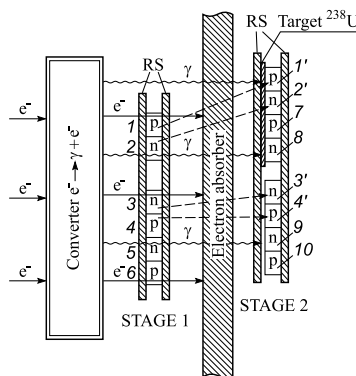


Fig.2. Two-cascade system of c -Si-crystal irradiation for the posthydrogenation process. Indexes 1-6 define position of crystals for the first stage of irradiation process, 1'-4' and 7-10 - for the second stage of irradiation behind the absorber of electrons

By combining the processes of irradiation and hydrogenation it is possible to exclude the process of hydrogenation via the high pressure gasostat with subsequent hydrogen molecular dissociation in the silicon structure with electrons from the additional accelerator. The thermostat position in the irradiation zone is shown in Figs.4. The hydrogenation of latent track disordered structures in the $c - Si$ -crystal is performed in the high-pressure gasostat with subsequent hydrogen molecule dissociation by radiation. The hydrogen atoms, segregating into the defect region, stabilize the disordered structure and play a catalytic role for cluster formation in a latent track during the heat treatment.

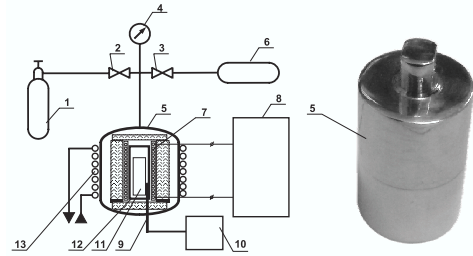


Fig.3. Installation for thermal treatment of silicon samples under pressure of 150 atm for the posthydrogenation process. 1 - cylinder with gaseous hydrogen, 2,3 - valves, 4 - 250 atm manometer; 5 - high-pressure container; 6 - hydrogen storage vessel; 7 - heater; 8 - power supply unit; 9 - thermocouple; 10 - temperature gauge; 11 - sample, 12 - capsule for placement of samples in the heater; 13 - copper pipe of the cooling system

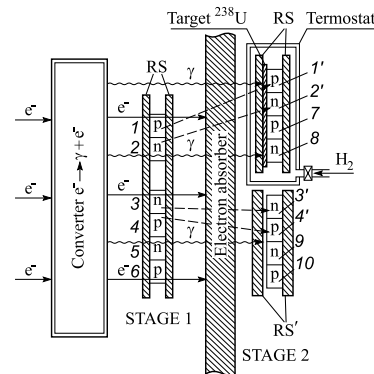


Fig.4. Two-cascade system of c -Si-crystal irradiation combined with quantum structure formation and hydrogenation. Indexes 1-6 define position of crystals for the first stage of irradiation process, 1'-4' and 7-10 - for the second stage of irradiation behind the absorber of electrons

3. IR-ABSORPTION SPECTRA OF THE IRRADIATED ATOMIC SILICON STRUCTURES

A silicon layer acts as an absorber in the visible spectrum and is transparent in the IR spectral region. IR-absorption spectra are sensitive to the oscillations

of the bonds ($Si - H$) in banding frequencies at $840...890\text{ cm}^{-1}$, modes bound with SiH_2 dihydride, and in two stretching frequencies at $1980...2030\text{ cm}^{-1}$ and $2060...2160\text{ cm}^{-1}$, connected with tension of monohydrides, dihydrides and trihydrides. The infrared radiation absorption by the free charge carriers in silicon is sensitive to the type of scattering of electrons and holes. Intraband absorption can be explained by two main mechanisms - electron scattering on the atomic oscillations and on the lattice defects. For the quantitative determination of the absorption coefficient we can use the Schmidt formula [5]

$$K = \frac{8}{9\pi} \frac{e^3 \hbar^2}{\varepsilon_0 c m^2 k^2} \left(\frac{m}{m^*} \right) \frac{N}{T^2} \frac{1}{\mu} \frac{1}{n} \text{sh} \left(\frac{\hbar\omega}{2kT} \right) K_2 \left(\frac{\hbar\omega}{2kT} \right), \quad (5)$$

where ε_0 is the dielectric vacuum constant, N is the charge carrier concentration, μ is the carrier mobility, $m_n^+ = 0.26 m$ and $m_n^* = 0.39 m$ are the average values of effective masses, T is the crystal temperature, K_2 is the modified Bessel function of the second kind. The mobility of both electrons and holes is proportional to $\sim T^{-2.5}$. The value of the temperature change in the forbidden zone width is $4.0 \cdot 10^{-4} \text{ eV}/^\circ C$, the activation energy is 1.09 eV . The value of the free-carrier absorption is proportional to their concentration, mobility and wavelength square. The value of the cross-section absorption by boron impurity centers in silicon is $15 \cdot 10^{-16} \text{ cm}^2$. The Schmidt formula for high temperatures ($\hbar\omega kT$) takes the form [6]:

$$K \approx \frac{4}{9\sqrt{\pi}} \frac{e^3 \hbar^2}{\varepsilon_0 c m^2 k^2} \left(\frac{m}{m^+} \right) \frac{N}{T^2} \frac{1}{\mu} \frac{1}{n} \left(\frac{2kT}{\hbar\omega} \right)^{3/2}. \quad (6)$$

The absorption modulation can be obtained by changing the charger carrier number (N) either due to the nuclear doping, nano-sized structure forming or due to the mobility (μ) changing (decreasing) in the case of deep atomic disordering in the crystalline lattice. The drift charge carrier mobility in the single-crystalline silicon at 300 K is $1360 \text{ cm}^2 V^{-1} \text{ sec}^{-1}$ for electrons and $\sim 500 \text{ cm}^2 V^{-1} \text{ sec}^{-1}$ for holes. The spectral region of photon absorption by free carriers in our experiment covers the wavelength interval of $(4.1...25) \mu$. In the structures with a high concentration of radiation defects the mobility value decreases and one observes a typical structure absorption by the free charge carriers that is equivalent to the value N decreasing too. Infrared absorption spectra of $c - Si$ -crystals (initial and irradiated) are shown in Fig.5 for the crystals with p -type conduction and these for the crystals with n -type conduction are shown in Fig.6 (IR absorption spectra are measured on the IR spectrophotometer UR-20 (Carl Zeiss) in a spectral range of $400...2200\text{ cm}^{-1}$). One can see that the transmission, T , increases after irradiation considerably for the p -type crystals and slightly for the n -type crystals.

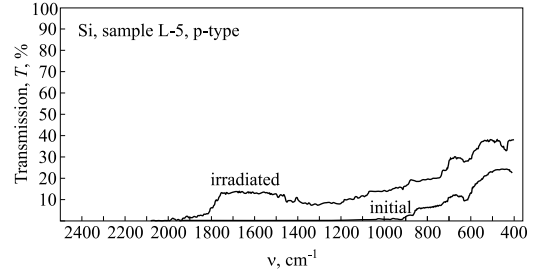


Fig.5. IR absorption spectra of the $p - c - Si$ -structure before irradiation and after radiation processing with electrons and uranium fission fragments at the accelerator "EPOS" at a dose 1800 Mrad (100 hours)

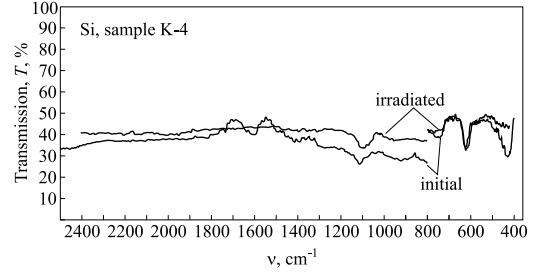


Fig.6. IR absorption spectra of the $n - c - Si$ -structure before irradiation and after radiation processing with electrons and uranium fission fragments in the accelerator "EPOS" at a dose 1800 Mrad (100 hours)

The difference in the transmission values of the p and n - structures of initial samples is explained by the difference in the values of mobility, effective masses of charge carriers and intrinsic defect concentrations. The curve of transmission in the n -structure is situated above the curve of transmission in the p -structure. For irradiated samples the values of N are decreasing because of the broken atomic bonds. Therefore, the absorption coefficients are decreasing, and, as a consequence, the transmission values are increasing. The both crystals were under similar irradiation conditions. The transmission curves of irradiated samples are situated above the transmission curves of initial samples. The absolute value of IR-radiation absorption (transmission) in the initial crystals could be measured after annealing of intrinsic defects in them. These defects determine the crystalline peak position in the region of a "halo" amorphization presence at the wide-angle X-ray spectroscopy. Therefore, the crystals must be preliminary annealed. In the process of radiation processing of crystals without preliminary annealing the concentration of intrinsic defects in them is supplemented with the concentration of created radiation defects. The structure transmission value increases with decreasing of the value of IR-radiation absorption on the disordered structures formed due to the photonuclear reactions in silicon. The contribution of amorphous structures from the uranium nuclear photo-fission fragments is not fixed as the vol-

ume, which they occupy in the crystal, is much less than the volume of disordered structures upon measuring the absorption value by the IR-spectroscopy method. The measurement is carried out throughout the crystal volume. The transmission curves of the crystals after radiation processing at the accelerators "KUT-1" and "EPOS" will be different even at an equal irradiation dose level because of the presence at the accelerator "EPOS" of a photonuclear reaction channel, creating the radiation doping factors, and in connection with the substantial disordering of the whole crystal structure. The effects of silicon structure irradiation on the IR-absorption are presented in Fig.7.

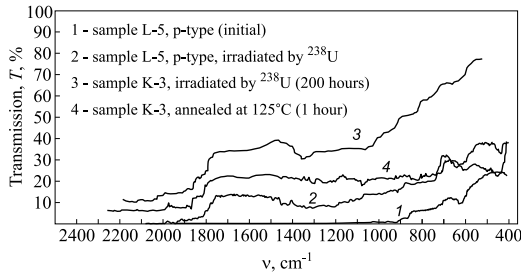


Fig.7. Dependence of the IR-spectra versus the dose of irradiation with electrons, uranium fission fragments and versus the partial annealing of the semiconductor structure (L5 – 1800 Mrad, $T = 125^{\circ}C$ and K3 – 3600 Mrad)

In the range of $2000...2200\text{ cm}^{-1}$, the free charge carriers give main contribution to the IR absorption. The irradiated sample L-5 becomes more transparent, as after irradiation the degree of crystalline structure disordering increases and, as a result, there appear many traps of charge carriers decreasing their concentration. Forming of latent tracks by the ^{238}U fragments at this stage does not lead to some change in the conduction. During the subsequent annealing the number of charge carriers is decreasing, in the region of latent tracks a nano-sized amorphous-microcrystalline structure is formed with a high conduction that leads to the integrated sample resistance lowering (see the samples K-3 before and after annealing). The hydrogen implantation causes significant changes in the absorption spectra. The lowering (recovering) of the IR transmission of disordered structures in single-crystalline silicon down to the level of transmission of $c - \text{Si}(p, n)$ -crystal with increased electrical conduction depends on the hydrogen aggregate state influence on the structure characteristics of irradiated samples. In the crystals having nano-sized structures the temperature of radiation defect annealing should not be higher than the temperature of quantum filament formation in the amorphous structures of the single-crystalline matrix. However, such a temperature process is insufficient for recovering the specific resistance value in the $c - \text{Si}$ -matrix structure between the quantum filaments. Displacement of the annealing temperature range for amorphous structures occurs due to their hydrogenation. The

hydrogenation method permits to neutralize defects requiring the high annealing temperatures that is very necessary for separation of quantum structures with an increased electrical conduction, increase of the lifetime of current carriers and their diffusion lengths in the doped single-crystalline silicon semiconductor.

4. ANALYSIS OF EXPERIMENTAL DATA

The lowering (recovering) of the transmission down to the level of transmission in the $c - \text{Si}(p, n)$ -crystal with an increased electrical conduction (quantum structures), as a function of the hydrogen aggregate state influence on the structure characteristics of unirradiated and irradiated samples, is shown in Fig.8.

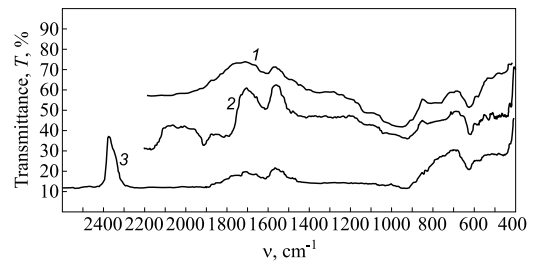


Fig.8. IR absorption spectra of the $c - \text{Si}$ - crystal after its structure radiation processing, hydrogenation and annealing 1) IR-spectrum of the $c - \text{Si} : \text{H}_2$ -crystal, irradiated with gamma-quantum by the 2500 Mrad dose at the accelerator KUT-1 and hydrogenated with molecular hydrogen in the high-pressure gasostat along the latent tracks created by uranium-238 nuclear fission fragments (molecular hydrogen pressure $P = 130\text{ atm}$, temperature $T = 125^{\circ}C$, hydrogenation time $t = 10\text{ h}$). 2) IR-spectrum of the hydrogenated silicon crystal (position 1) after irradiation with secondary electrons by the 30 Mrad dose at the accelerator KUT-1 for H_2 dissociation in the crystal structure. 3) IR-spectrum of the hydrogenated crystal after H_2 dissociation with secondary electrons (position 2) and annealing at $T = 125^{\circ}C$, annealing time $t = 2\text{ h}$ (precipitation process)

The results of experiments on the silicon single-crystal sample saturation with hydrogen in the high-pressure gasostat and the numeration of IR-absorption spectra are given in the order of carrying out the radiation technologies for $c - \text{Si}$ -crystal processing. As it follows from Fig.8, the transmission level decreasing in the $c - \text{Si}$ -crystal for the IR-radiation in the range from $2000...2200\text{ cm}^{-1}$ with $\sim 60\%$ (position 1) to $\sim 10\%$ (position 3) is related with the production in the single-crystalline matrix of quantum (amorphous-crystalline) structures having an increased specific electrical conductivity. After hydrogenation and annealing at $T = 125^{\circ}C$ a new phase is precipitated, the transmission level continues to decrease from $\sim 40\%$ (position 2) to $\sim 10\%$ (position 3) and the conducting microstructures in the $c - \text{Si}$ -matrix are recovered. In the process of hydrogenation

tion the hydrogen is implanted only into the latent tracks and forms $a-Si : B, P(H)$ compounds, whose crystallization temperature significantly exceeds the temperature of annealing the point defects in the $c-Si(B, P)$ -matrix volume. So, the technological process under consideration permits to neutralize the point radiation defects in the matrix structure and to separate only the amorphous phases with quantum conducting structures in the $c-Si(p, n)$ -crystals.

5. THE CONCLUSION

The presence of hydrogen in the amorphous silicon structures leads to the clustering of vacancies, intrinsic interstitial and impurity atoms. For quantum structures, passivated with hydrogen atoms, the annihilation process is slowed down. In the process of annealing the silicon structures the strong $(Si-H)_n$ -bonds prevent the defect annihilation and thus stimulate the processes of precipitation and clusterization. Hydrogen, filling the irradiation-broken bonds, neutralizes their electrical activity. Optimal conditions for stabilisation of such structures are determined: irradiation doses, methods of hydrogenation and dissociation of H_2 , annealing temperatures.

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СТАБИЛІЗАЦІЯ НАНОРАЗМЕРНИХ СТРУКТУР В ОБ'ЄМІ МОНОКРИСТАЛІЧЕСЬКОГО КРЕМНІЯ ДЛІЯ ФОТОПРЕОБРАЗОВАТЕЛІЙ

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Рассматриваются возможности стабилизации микроструктур, создаваемых в скрытых треках в монокристаллическом кремнии многозарядными ионами из осколков ядер тяжелых элементов при их фотоделинии, в процессе гидрирования. Присутствие водорода в аморфных структурах кремния приводит к кластеризации вакансий, собственных межузельных и примесных атомов. Для квантовых структур, пассивированных атомами водорода, процесс аннигиляции заторможен. При отжиге кремниевых структур прочные $(Si-H)_n$ -связи препятствуют аннигиляции дефектов и тем самым стимулируют процессы преципитации и кластеризации. Водород, заполняя оборванные в результате облучения связи, нейтрализует их электрическую активность. Определены оптимальные условия создания таких структур: дозы облучения, методы гидрирования и диссоциации H_2 , режимы отжига.

СТАБІЛІЗАЦІЯ НАНОРОЗМІРНИХ СТРУКТУР В ОБ'ЄМІ МОНОКРИСТАЛІЧНОГО КРЕМНІЮ ДЛІЯ ФОТОПЕРЕТВОРЮВАЧІВ

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Розглядаються можливості стабілізації микроструктур, створюваних в прихованих треках в монокристалічному кремнії багатозарядними іонами з осколків ядер важких елементів при їх фотоділенні, в процесі гідрування. Присутність водню в аморфних структурах кремнію приводить до кластеризації вакансій, власних міжвузельних і домішкових атомів. Для квантових структур, пасивованих атомами водню, процес анігіляції загальмований. При відпалі кремнієвих структур міцні $(Si-H)_n$ -зв'язки перешкоджають анігіляції дефектів і тим самим стимулюють процеси преципітації і кластеризації. Водень, заповнюючи обірвані в результаті опромінювання зв'язки, нейтралізує їх електричну активність. Визначено оптимальні умови створення таких структур: дози опромінювання, методи гідрування та дисоціації H_2 , режими відпалу.