

THERMAL ANNEALING OF RADIATION-INDUCED DEFECTS IN *n*-Si IRRADIATED WITH FAST REACTOR NEUTRONS

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Thermal stability of defect clusters in *n*-Si grown by the Czochralski technique (Cz) and irradiated with fast reactor neutrons has been studied. The effective concentration of carriers ($n_0 = 1.2 \times 10^{14} \text{ cm}^{-3}$) obtained in silicon after a number of isochronal annealings followed by its irradiation to a fluence of fast reactor neutrons $\Phi = 3.75 \times 10^{13} \text{ neutron/cm}^2$ has been described in the framework of the corrected defect cluster model. Three stages of the annealing of the clusters of defects with the following activation energies and frequency factors have been identified: $E_{a1} = 0.81 \text{ eV}$, $\nu_1 = 5.4 \times 10^6 \text{ s}^{-1}$; $E_{a2} = 0.4 \text{ eV}$, $\nu_2 = 1 \text{ s}^{-1}$; and $E_{a3} = 1.3 \text{ eV}$, $\nu_3 = 6 \times 10^4 \text{ s}^{-1}$. The deformation potential of defect clusters was demonstrated to reduce the activation energy of *A*-center annealing ($E_a = 1.5 \text{ eV}$) in a conducting matrix. It is established that the effective radius of the interstitial-type defect capture by defect clusters is determined by the barrier of defect capture by divacancies ($U_b = 0.41 \text{ eV}$).

1. Introduction

One of the ways to establish a relationship between the results that have been obtained making use of different experimental methods, which include an irradiation procedure and which are difficult to be compared with one another, is the annealing of radiation-induced defects. For instance, the authors of work [1] considered the level of $E_c - 0.09 \text{ eV}$ in silicon to be associated with a vacancy; this viewpoint was based on the fact that the annealing temperature for the defect observed was only 90 K. The activation energy of the *A*-center annealing was determined in the framework of both the IR-absorption and EPR methods, and became a basis for the EPR spectrum correction [2]. After holding *n*-Si specimens at 292 K, the shift of the energy position of the *A*-center from $E_c - 0.16 \text{ eV}$ to $E_c - 0.175 \text{ eV}$ was observed, and this shift was explained as due to the annealing of the level $E_c - 0.15 \text{ eV}$ belonging to a di-interstitial atom [3]. Researches of defect cluster annealing was carried out with the help of various techniques [4–8].

This work aimed at studying the isochronous annealing of clusters and point defects generated by

fast reactor neutrons in *n*-Si grown by the Czochralski method.

2. Experimental Technique

In this work, we studied *n*-Si specimens grown by the Czochralski method and characterized by the resistivity $\rho \approx 40 \text{ } \Omega \times \text{cm}$. The specimens were irradiated in a horizontal channel of a VVR-M reactor at room temperature. The conductivity and the Hall coefficient were measured taking advantage of the compensation method and making use of crosslike specimens; the measurement accuracy was about 3%. Contacts were created by depositing an Au–Si eutectic mixture at a temperature of 420 °C. The *n*-Si specimens ($n_0 = 1.2 \times 10^{14} \text{ cm}^{-3}$) irradiated at a fluence of $3.75 \times 10^{13} \text{ neutron/cm}^2$ were isochronously annealed in the temperature interval up to 600 K, the specimen being held for 30 min at each annealing temperature.

3. Measurement Results

The temperature dependences of the effective electron concentration in the *n*-Si (Cz) specimens irradiated with fast neutrons and annealed at different temperatures are depicted in Fig. 1. In Fig. 2, the dose dependence of the relative variation of electron mobility measured at 77 K is shown. Figure 3 exhibits the calculated dependence of the defect cluster concentration on the temperature of isochronous annealing. At last, Fig. 4 exposes the dependence of the calculated concentration of *A*-centers in a conducting *n*-Si matrix on the annealing temperature.

4. Annealing of Radiation-induced Point Defects

The easiest way to describe the annealing is to use equations similar to those, which are used in chemical kinetics. A characteristic feature of the annealing of the

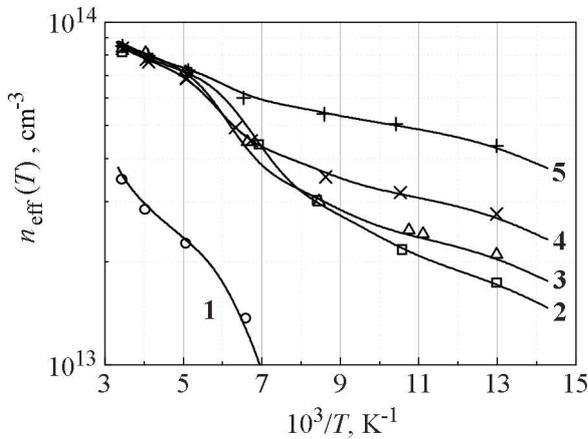


Fig. 1. Temperature dependences of the effective electron concentration in *n*-Si (Cz) specimens ($n_0 = 1.2 \times 10^{14} \text{ cm}^{-3}$) irradiated with fast reactor neutrons at a fluence of $3.75 \times 10^{13} \text{ neutron/cm}^2$ and annealed for 30 min at various temperatures of 303 (1), 475 (2), 523 (3), 578 (4), and 603 K (5)

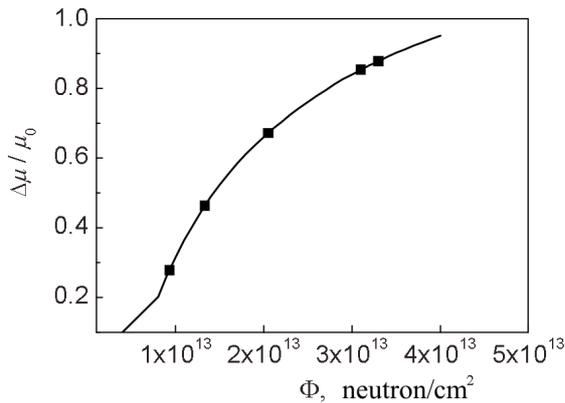


Fig. 2. Dependence of the relative variation of electron mobility $\Delta\mu/\mu_0$ in a *n*-Si (Cz) specimen ($n_0 = 1.2 \times 10^{14} \text{ cm}^{-3}$) on the fluence of fast reactor neutron irradiation Φ measured at a temperature of 77 K

first order is that either every defect becomes annealed or its concentration increases irrespective of other defects. Defects of the vacancy type become annealed at their annihilation with mobile defects of the interstitial type, the migration energy of which depends on their charge state [1]. The diminishing of a certain quantity P^i , which is governed by the defect concentration, in the course of the annealing process of the first order can be characterized in terms of a rate constant K_i :

$$\frac{dP^i}{dt} = -K_i P^i. \quad (1)$$

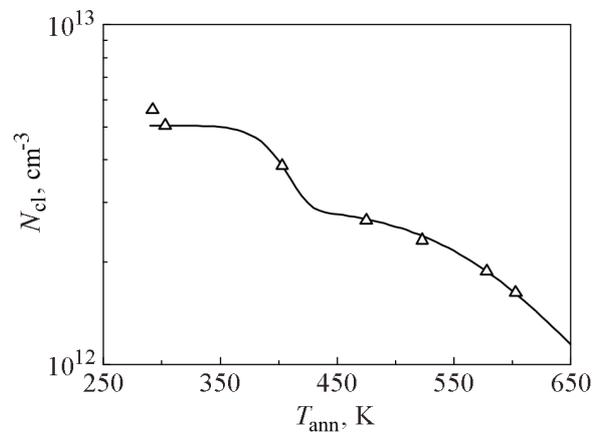


Fig. 3. Dependence of the cluster concentration N_{cl} in the *n*-Si (Cz) specimens ($n_0 = 1.2 \times 10^{14} \text{ cm}^{-3}$) irradiated with fast reactor neutrons at a fluence of $3.75 \times 10^{13} \text{ neutron/cm}^2$ and annealed for 30 min on the annealing temperature T_{ann} . Triangles correspond to experimental data, the solid curve exhibits the results of calculations

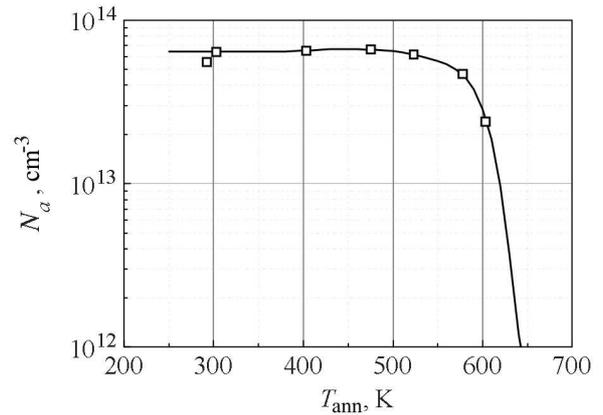


Fig. 4. The same as in Fig. 3 but for the concentration of A-centers N_a in the conducting *n*-Si (Cz) matrix. Squares correspond to experimental data, the solid curve exhibits the results of calculations

At the same time, the growth of a quantity P^j , also governed by the defect concentration, to its maximal value can be characterized in terms of a rate constant K_j :

$$\frac{dP^j}{dt} + K_j (P^j - P_m^j) = 0. \quad (2)$$

Usually, the rate constants are equal to $K_{i,j} = A^{i,j} \exp(-E_a^{i,j}/kT)$, where $A^{i,j}$ are the frequency factors, $E_a^{i,j}$ the process activation energies, k is the Boltzmann constant, T is the absolute temperature, and

i and j are the numbers of annealing and accumulation channels, respectively.

Radiation-induced defects of the VO and V_2 types are stable at room temperature, whereas those of the I and I_2 ones are mobile. At higher temperatures, VO and V_2 defects start to move towards drains (O_i , C_s).

By integrating Eqs. (1) and (2) and by summing up over various channels of isothermal annealing or accumulation of a quantity, which depends on the defect concentration, we obtain the following equation describing the variation of this quantity in the course of annealing:

$$P(t) = \sum_{i=1}^k P_0^i \exp[-A^i t \exp(-E_a^i/kT)] + \sum_{j=1}^m P_m^j [1 - \exp(-A^j t \exp(-E_a^j/kT))] - \sum_{l=1}^n P_{00}^l. \quad (3)$$

Here, P_0^i is the fraction of the quantity that is annealed in the i -th channel, P_m^j the fraction of the quantity that is accumulated in the j -th channel, and t the time of annealing. The appearance of the last term P_{00}^l on the right-hand side of Eq. (3) is associated with the fact that the annealing process does not go to the end. Such an accumulation of defects is possible, e.g., due to the dissociation of another defect with a lower annealing temperature. The annealing of defects occurs in different ways through their annihilation with interstitial atoms or di-interstitial sites, their capture by drains, the defect dissociation, or owing to the change of the energy and the frequency factor of the defect annealing stimulated by the defect reorientation.

Let us suppose that the annealing of a defect includes only its diffusion to drains. As such, there can be C_s -centers, interstitial oxygens, and defect clusters. Then, according to the diffusion-limited reaction theory [9], the rate constant can be written down in the form $K_{C_i} = 4\pi R_C D$, where R_C is the radius of interaction between a defect – for instance, a C_i -center – and O_i or C_s drains; and D is the defect diffusion coefficient. Let the drains occupy an atomic volume of the radius R_a in a specimen with the volume $V \approx 1$. Then, $R_a = (3/(4\pi N_{C_m}))^{1/3}$, where N_{C_m} is the concentration of drains (C_s , O_i) in the specimen.

The average distance between O_i or C_s atoms is equal to $d = \sqrt{2}R_a$, and the mean distance, which the carbon C_i atoms have to cover in order to attain drains (C_s , O_i), is equal to $L = d/\sqrt{2}$. Hence, one can suppose

the mean path length of C_i diffusion to a drain to be equal to the atomic radius of the drain. Then, provided that the process is Markovian, $L = \sqrt{Dt}$ and

$$D = \frac{1}{t} \left(\frac{3}{4\pi N_{C_m}} \right)^{2/3}, \quad (4)$$

where t is the annealing time.

A consistent theory of diffusion-controlled reactions in solids was elaborated by Waite [10]. As a variant, he considered the case where the quasichemical interaction occurred between atoms that were characterized by different coefficients of diffusion in a solid. In our case, only one of the components of interacting pairs is mobile.

Provided that the frequency of the mobile defect hopping ν is known, the frequency factor D_0 can be evaluated by the formula $D_0 = 2a^2\nu/(3\pi)$. Here, $a = 5.43 \text{ \AA}$ is the silicon lattice constant. If the hopping frequency of a mobile defect cannot be determined, but the drain concentration is known, the frequency factor D_0 can be found making use of Eq. (4), because $D = D_0 \exp(-E_a/kT)$ either, in the case of isochronous annealing, at a temperature equal to half the defect annealing one or, in the case of isothermal annealing, at the time moment equal to half the annealing time interval. The annealing activation energy was taken equal to the activation energy of mobile defect diffusion. In so doing, the defect was supposed to cover an average distance R_a , provided that the rate of its disappearance is maximal.

In the framework of the notions developed above and making use of Eq. (3), as well as the experimental data obtained in works [11–16], we estimated the radii of capture of mobile radiation-induced defects onto such drains as an interstitial oxygen and an on-site carbon at relevant temperatures. The results of calculations are presented in Table 1.

The radii of capture of radiation-induced defects (vacancies, divacancies, A -centers, and so on) by an interstitial oxygen and an on-site carbon fall within the limits of 3 to 4 times the lattice constant of silicon. Table 1 also gives the calculated values for the activation energy of V_2 , VO , C_i , PV , I , and I_2 annealing, which are well-known from a plenty of works of other authors.

5. Annealing of Defect Clusters

The calculated temperature dependences of the effective conduction electron concentration after the specimen annealing at various temperatures are depicted in Fig. 1. The calculation parameters are quoted in Table 2. It is

T a b l e 1. Diffusion-limited annealing of intrinsic radiation-induced defects in silicon

Reaction	Capture center	Capture center concentration, cm ⁻³	D_0 , cm ² /s	E_a , eV	$R \times 10^8$, cm	T_{ann} , K	Source
$V_2 + O_i \rightarrow V_2O$	O_i	10^{18}	4.19×10^{-4}	1.3	16	500 ÷ 570	[11]
$V + O_i \rightarrow VO$	O_i	8×10^{17}	1.5×10^{-3}	0.8	15	300 ÷ 350	[12]
$VO + O_i \rightarrow VO_{2i}$	O_i	8×10^{17}	4.16×10^2	1.86	15	500 ÷ 550	[12]
$C_i + O_i \rightarrow CO$	O_i	6×10^{17}	1.17×10^{-3}	0.87	17	320 ÷ 380	[13]
$C_i + C_s \rightarrow C_i C_s$	C_s	2.9×10^{17}	2.53×10^{-3}	0.87	23	320 ÷ 380	[13]
$V^- + P^+ \rightarrow PV$	P	5×10^{14}	8.6×10^{-2}	0.8	180	300 ÷ 350	[14]
$PV^0 + O_i \rightarrow PV^0O$	O_i	9×10^{17}	3.82×10^{-1}	0.94	15	333	[15]
$PV^0 + O_i \rightarrow PV^0O$	O_i	7×10^{17}	1.06×10^{-4}	0.94	16	350 ÷ 450	[12]
$I + VO \rightarrow O_i$	VO	2.81×10^{17}	3.9×10^{-5}	0.91	22	400 ÷ 450	[16]
$I_2 + VO \rightarrow O_i + I$	VO	3.5×10^{17}	1.67×10^{-4}	0.74	20.3	290 ÷ 360	[16]
$O_{2i} + VO_i \rightarrow VO_{3i}$	VO	2.24×10^{17}	1.5	1.7	23.7	530 ÷ 570	[16]
$I + VO \rightarrow O_i$	VO	2.57×10^{17}	6.93×10^{-3}	1.3	22.5	470 ÷ 530	[16]

T a b l e 2. Parameters used for calculations of the temperature dependences of the effective charge carrier concentration in *n*-Si (Cz) specimens ($n_0 = 1.2 \times 10^{14}$ cm⁻³) irradiated at a fluence of fast reactor neutrons $\Phi = 3.75 \times 10^{13}$ neutron/cm² and annealed for 30 min at various temperatures

T_{cr} , K	T_{ann} , K	E_a , eV	N_b , cm ⁻³	N_a , cm ⁻³	$\Sigma(T_{\text{cr}})$	R	$\Sigma(\Delta\mu/\mu_0)$
157	292	0.16	6.55×10^{13}	5.54×10^{13}	0.15	70	0.150
147	303*	0.162	7.95×10^{13}	6.4×10^{13}	0.135	69	0.131
74	475	0.158	1.11×10^{14}	6.6×10^{13}	0.071	72	0.074
66.5	523	0.171	1.15×10^{14}	6.15×10^{13}	0.064	80	0.065
54	578	0.178	1.16×10^{14}	4.7×10^{13}	0.052	117	0.050
46.6	603	0.17	1.165×10^{14}	2.4×10^{13}	0.045	127	0.042

N o t a t i o n s : * – 6-year annealing; E_a is the energy of the *A*-center level; N_a is the concentration of *A*-centers; N_b is the concentration of screening centers; $\Sigma(T_{\text{cr}})$ is the cross-section of cluster formation calculated on the basis of the T_{cr} -value; $\Sigma(\Delta\mu/\mu_0)$ is the cross-section of cluster formation calculated on the basis of the relative variation of electron mobility; R is the radius of defect cluster

evident that, after annealing, only the product $N_{\text{cl}}R_1$ of the concentration and the average radius of defect clusters can be determined (see works [17, 18]). Every scattered fast neutron is supposed to generate a defect cluster ($\Sigma_0 = 0.15$ cm⁻¹ is the efficiency of the defect cluster generation in silicon irradiated with fast reactor neutrons). Then, the average radius of the clusters of defects can be determined.

In work [19], it was shown that, for *n*-Si specimens irradiated with fast neutrons, the temperature value, at which electrons demonstrate maximal mobility, depends on the applied fluence Φ . Such a temperature T_{cr} was called “critical”. It became linearly shifted towards room temperatures as the fluence increased, obeying the following empirical law: $T_{\text{cr}} = \Phi(A n_0^{5/6})$, where A is the coefficient of proportionality, and n_0 is the

conduction electron concentration in the specimen before irradiation.

The amplitude of the coefficient A grows with the enhancement of the silicon radiation stability. For *n*-Si (Cz) specimens, $A \approx 0.44$ [20]. In work [21], the parameter T_{cr} was demonstrated to determine the volume fraction f occupied by defect clusters. As the exposure dose increases, the value of f linearly grows with the fluence Φ – as a result of the point defect generation in the conduction matrix of the specimen – according to the relation $f = \Sigma V \Phi$. Then, $f = T_{\text{cr}} \Sigma V A n_0^{5/6}$. Hence, by analyzing the variation of the quantity $T_{\text{cr}}(d\mu_{\text{max}}/dT = 0)$ which depends on the irradiation fluence, one can evaluate the change of the volume fraction occupied by clusters, which occurs due to their annealing, and, in such a way, determine the concentration of charge carriers removed by clusters: $n_{\text{cl}} = n_0 f$. However, if the annealing temperature is equal to or higher than 523 K, the measurements of the effective electron mobility have to be carried out at temperatures lower than the temperature of liquid nitrogen.

It can be shown that the product $N_{\text{cl}}R_1$ is proportional to the quantity $\Delta\mu/\mu_0(\Phi)$, so that the latter determines the dependence of the former on the irradiation exposure dose; in the same way, the dependence $\Delta\mu/\mu_0(T_{\text{ann}})$ determines the dependence of $N_{\text{cl}}R_1$ on the annealing temperature. Then, $\Sigma(T_{\text{ann}}) = \Sigma_0 \Phi(\Delta\mu/\mu_0) \Phi_k^{-1}$, where $\Phi_k = 3.75 \times 10^{13}$ neutron/cm² is the irradiation fluence for specimens annealed at different temperatures. Similarly, $\Sigma(T_{\text{ann}}) = \Sigma_0 \Phi(T_{\text{cr}}) \Phi_k^{-1}$.

The dependence of the quantity $\Delta\mu/\mu_0$ on the irradiation dose measured at 77 K is shown in Fig. 2. The variations of the defect cluster concentration in a specimen, which has been stored for 6 years at room temperature, after the 30-min annealing at various

temperatures are presented in Fig. 3. The cluster concentration was determined both from the variation of T_{cr} and from the dependence $\Delta\mu/\mu_0(\Phi)$.

The change of the cluster concentration N_{cl} depends on the temperature of isochronous annealing (see Fig. 3) and, according to Eq. (1), is described by the equation

$$N_{cl}(T) = \sum_{i=1}^3 N_0^i \exp(-\nu_i t \exp(-E_a^i/kT)), \quad (5)$$

where $N_0 = \sum_{i=1}^3 N_0^i$ is the total concentration of clusters before annealing; N_0^i is the concentration of annealed clusters in the i -th channel; T and t are the temperature and the time of annealing, respectively; and ν_i is the frequency factor of annealing in the i -th channel.

Defect cluster annealing was described by Eq. (5) with the following parameters: $E_{a1} = 0.81$ eV, $\nu_1 = 5.4 \times 10^6$ s $^{-1}$; $E_{a2} = 0.4$ eV, $\nu_2 = 1$ s $^{-1}$; and $E_{a3} = 1.3$ eV, $\nu_3 = 6 \times 10^4$ s $^{-1}$. The first stage of the defect cluster annealing includes the mutual annihilation between defects of the vacancy type in clusters and interstitial silicon atoms [8]. The third annealing stage is connected with the migration of divacancies from clusters to drains. Divacancies in silicon are known to be already mobile at temperatures of 450–520 K. We may suppose that the second stage of the defect cluster annealing is connected with the clustering of divacancies, according to the reaction $V_2 + V_2 \rightarrow V_4$. Therefore, the annealing activation energy decreases by the value of the bond energy of divacancies in the V_4 -defect ($E_b = 0.9$ eV).

The data in Table 2 demonstrate that the increase of the annealing temperature brought about the growth of the experimentally determined dimensions for remaining clusters. This fact is confirmed by the results of work [4], the authors of which observed disordered regions in silicon with dimensions of about 50 Å.

The dependence of the concentration of A -centers in the conducting n -Si (Cz) matrix on the annealing temperature is presented in Fig. 4. The results of calculations by Eq. (4) testified that A -centers in the conducting n -Si (Cz) matrix are annealed with the activation energy $E_a = 1.5$ eV and the frequency factor $\nu = 1.9 \times 10^9$ s $^{-1}$. At the electron irradiation, the A -center annealing stage with $E_a = 1.5$ eV is not observed, and the annealing stage with $E_a = 1.86$ eV is connected with the migration of A -centers to drains (O_i atoms). We may suggest that the reduction of the activation energy of A -center annealing is due to the action of a strain field emerging around defect clusters, which are the main drains for A -centers.

In work [22], it was found experimentally that a phosphorus atom is characterized by the probability of vacancy capture, the value of which is $\gamma_{VP}/\gamma_{VO} = (1.8 \pm 0.9) \times 10^2$ times higher than that inherent to an oxygen atom. According to the values obtained by us (Table 1) for the vacancy capture radii of a phosphorus atom (180 Å) and an oxygen atom (15 Å), those probabilities differ by a factor of $(180/15)^2 = 144$. Therefore, our assumption that the average path length of a mobile defect at its movement to a drain is equal to the atomic radius of the drain ($L = R_a$), provided that the defect annealing rate is maximal, obtained the experimental confirmation.

In the case of the diffusion-limited recombination of particles and long-range forces between them, the reaction rate is determined by a certain effective radius R_{eff} dependent on the interaction potential [23], $R_{eff}^{-1} = \int_{r_0}^{\infty} \exp(U_b/kT) dr/r^2$. Then, provided that strongly correlated Frenkel pairs are absent, $R_{eff} = R_0 \exp(-U_b/kT)$, where U_b is the barrier of interaction between a mobile defect and a drain, so that $R_0 = R_{eff}$ if $U_b = 0$.

The analysis of the isothermal defect cluster annealing in n -Si(Ge) irradiated with fast reactor neutrons [8] showed that the effective capture radius of interstitial silicon atoms (I and I_2) into clusters is equal to $R_{eff} = 0.726 \exp(-\frac{0.41}{kT})$ in centimeter units, where $U_b = 0.41$ eV [24]. The preexponential factors in the expressions for the diffusion coefficient of interstitial and di-interstitial silicon atoms in n -Si(Ge) were also evaluated to be $D_0^I = 4.17 \times 10^{-9}$ cm 2 /s and $D_0^{I_2} = 2.09 \times 10^{-9}$ cm 2 /s, respectively.

Hence, the energy needed to activate the motion of a di-interstitial atom in silicon is $E_a^{I_2} = 0.33$ eV; for an interstitial atom, it is $E_a^I = 0.5$ eV ($U_b = 0.41$ eV). One may assert that I_2 's are in a neutral charge state, whereas interstitial silicon atoms, which participated in the annealing of vacancy defects of the cluster, are mainly in a negative charge state.

6. Conclusions

The main results of the work are as follows. The mean path length of the defect diffusion to drains is equal to the atomic radius of drains, and this estimate is valid for 50% of the defects annealed. The radius of the mobile radiation-induced defect (V , C_i , V_2 , A -center) capture by an interstitial oxygen has been calculated to lie within 3 to 4 silicon lattice constants. The temperature dependences of the effective conduction electron concentration in n -Si specimens grown by

the Czochralski method and isochronously annealed at different temperatures have been described. The strain fields of defect clusters were supposed to reduce the annealing energy of *A*-centers in the conducting *n*-Si matrix irradiated with fast reactor neutrons.

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ТЕРМІЧНИЙ ВІДПАЛ РАДІАЦІЙНИХ ДЕФЕКТІВ В *n*-Si, ОПРОМІНЕНОМУ ШВИДКИМИ НЕЙТРОНАМИ РЕАКТОРА

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Резюме

Термічну стабільність кластерів дефектів в *n*-Si, вирощеному методом Чохральського (Cz), досліджено після опромінення швидкими нейтронами реактора. Ефективну концентрацію носіїв після ряду ізохронних відпалів кремнію ($n_0 = 1,2 \times 10^{14} \text{ см}^{-3}$), опроміненого флюенсом швидких нейтронів $\Phi = 3,75 \cdot 10^{13} \text{ нейтр./см}^{-2}$, описано в рамках уточненої моделі кластерів дефектів. Визначено такі стадії відпалу кластерів дефектів з енергіями активації та частотними факторами: $E_{a1} = 0,81 \text{ eV}$, $\nu_1 = 5,4 \cdot 10^6 \text{ с}^{-1}$; $E_{a2} = 0,4 \text{ eV}$, $\nu_2 = 1 \text{ с}^{-1}$; $E_{a3} = 1,3 \text{ eV}$, $\nu_3 = 6 \cdot 10^4 \text{ с}^{-1}$. Показано, що деформаційні поля кластерів дефектів зменшують енергію активації відпалу *A*-центрів ($E_a = 1,5 \text{ eV}$) у провідній матриці. Одержано, що ефективний радіус захоплення в кластери дефектів міжвузловинного типу визначається бар'єром їх захоплення дивакансіями ($U_6 = 0,41 \text{ eV}$).