MECHANISM OF ANNEALING OF VO DEFECTS IN *n*-Si UNDER PULSE ELECTRON IRRADIATION AT HIGH-TEMPERATURES

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We study the kinetics of accumulation of vacancy-oxygen (VO) complexes in Czochralski-grown (Cz) *n*-Si, at various intensities of pulse 1-MeV electron radiation at temperatures higher than the temperature of the onset of the thermal annealing of VO ($T \geq 300$ °C). It is shown that the irradiation with electrons at such temperatures causes the accelerated annealing of VO created by this radiation. The accelerated annealing of VO occurs during the action of a pulse of electrons. The maximum concentration of created VO increases with the radiation intensity and decreases, as the temperature of irradiated specimens increases. We propose a model of the process of accelerated annealing which is based on the assumption that specimen's electrons under the electron irradiation are excited in a high-energy valley. At the capture of such electrons by VO defects, the defects receive the energy which decreases essentially the energy of activation of their annealing. The high-energy threshold of the effect depends on the radiation intensity and increases with it.

1. Introduction

One of the basic radiation defects in Si is a VO complex which is created as a result of the capture of a vacancy (V) by interstitial oxygen O_i ($V + O_i \rightarrow VO$). The properties of these defects are studied for five decades and are well investigated till now (see, e.g., [1] and references therein). VO complexes are annealed at $T \geq 300$ °C. If the irradiation is carried on at a temperature which is higher than the annealing temperature of VO, two processes are running simultaneously: the creation of these defects and their annealing. If the temperature of specimens is sharply decreased after the termination of the irradiation, then the concentration of VO determined by the given conditions of the irradiation is conserved [1–7]. It was also revealed [1] that the concentration of these defects depends significantly on the radiation intensity J.

The present work is a logical sequel of our previous studies [1]. Our goal was to study the mechanism of annealing of VO in Cz $n-$Si under the simultaneous action of two factors: a high temperature of a specimen under the irradiation and the electron radiation intensity.

2. Experiment

We use specimens of Cz *n*-Si and the method to be the same as those in [1]. The specimens contain phosphorus (at a concentration of $\approx 1 \times 10^{15}$ cm^{-3}), oxygen $[O_i]((6-7) \times 10^{17}$ cm$^{-3})$, and carbon $[C_s](\leq 5 \times 10^{16}$ cm$^{-3})$. The irradiation was carried out at a pulse accelerator of electrons: the pulse duration $t_p = 3.3$ μs, the pulse on/off ratio $s = 10^3$, and the energy of electrons $E_e = 1$ MeV. In addition to the irradiation of specimens at a temperature of 360 °C (intensities per pulse were in the range $J_p = 1.25 \times 10^{15} - 1.25 \times 10^{16}$ electron/(cm^{2}s)), the specimens were irradiated at a temperature of 450 °C by a more intense flux of electrons $J_p = 5 \times 10^{16}$ electron/(cm^{2}s). The concentration of VO after the irradiation was determined from the temperature dependences of the Hall effect or by using the Deep-Level Transient Spectroscopy (DLTS).

3. Results and Their Discussion

3.1. Experimental results

In Fig. 1, we present the dependences of the concentration of VO on the irradiation duration at several intensities of the flux of electrons ($J_p = 1.25 \times 10^{15} - 1.25 \times 10^{16}$ electron/(cm^{2}s)) at a temperature of 360 °C. Figure 2 shows the DLTS spectrum of specimens irradiated at 450 °C ($J_p = 5 \times 10^{16}$ electron/(cm^{2}s)) in the temperature

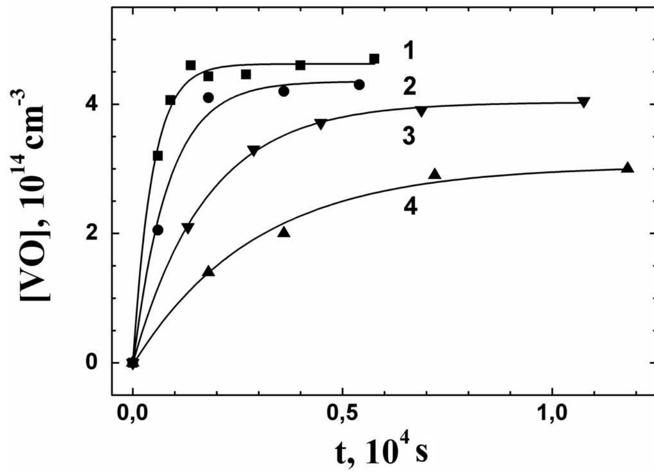


Fig. 1. Time dependences of the concentration of VO at various intensities of 1-MeV pulse electron radiation at 633 K: *1* – 12.5; *2* – 6.25; *3* – 2.81; *4* – 1.25×10^{15} electron/(cm^{2}s). Points – experiment; continuous curves – results of calculations by formula (3)

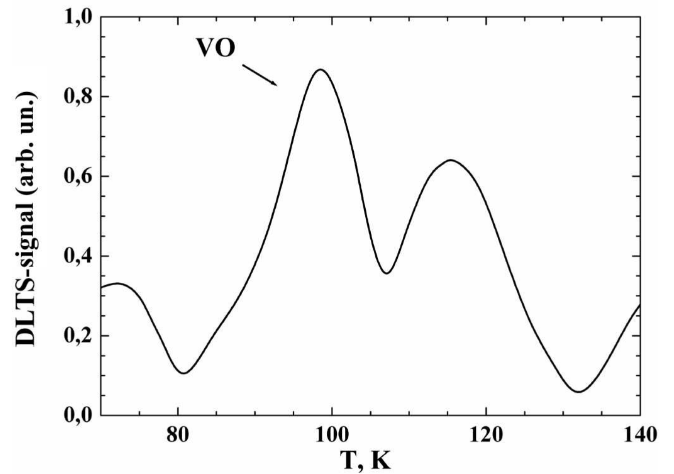


Fig. 2. DLTS spectrum of specimens irradiated at 723 K (the irradiation duration $t = 20$ min, $J_p = 5 \times 10^{16}$ electron/(cm^{2}s), $F = 6 \times 10^{16}$ cm^{-2}, [VO]= 5×10^{13} cm^{-3}). The bias pulse amplitude from –4 to 0 V; the window is 6 ms

region, where the VO complex is recharged (the peak at about 100 K).

It is seen that

a) the accumulation of VO is characterized by the curve with saturation and occurs qualitatively in the same way at all radiation intensities J. In this case, as was shown in [1], the efficiency of the creation of VO is independent of J and is equal to $\frac{d[\mathrm{VO}]}{dF} = (7.9 \pm 0.3) \times 10^{-2}$ cm^{-1} (Fig. 1);

b) the concentration of VO at the saturation ($[\mathrm{VO}]_{\max}$) increases with J and shows the tendency to the saturation (Fig. 3);

c) as the temperature of specimens under irradiation increases, the value of $[\mathrm{VO}]_{\max}$ decreases. In specimens irradiated at 450 °C ($J_p = 5 \times 10^{16}$ electron/(cm^{2}s)), $[\mathrm{VO}]_{\max} = 5 \times 10^{13}$ cm^{-3} (Fig. 2), whereas $[\mathrm{VO}]_{\max} = 4.6 \times 10^{14}$ cm^{-3} in specimens irradiated at 360 °C ($J_p = 1.25 \times 10^{16}$ electron/(cm^{2}s)), (Fig. 3).

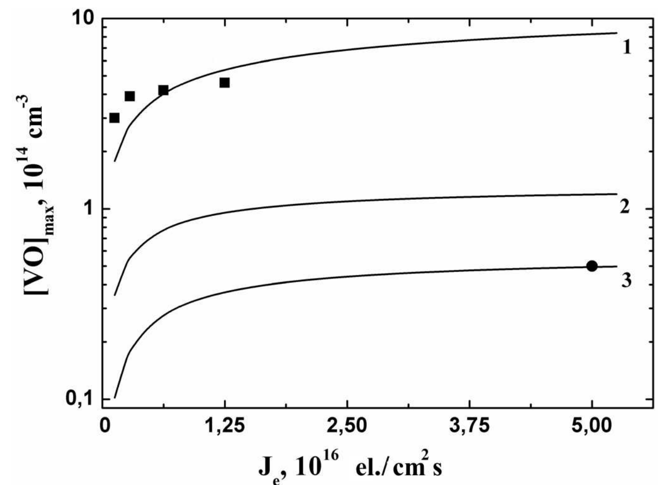


Fig. 3. Maximum concentration of VO versus the electron radiation intensity at: *1* – 633 K; *2* –680 K; *3* – 723 K. Points – experiment; continuous curves – results of calculations by formula (4), where $1/\tau_1$ is determined by formula (12), and $1/\tau_2 = 2 \times 10^8 \exp(-1.5/k_{\mathrm{B}}T)$

3.2. Kinetics of accumulation of VO at 360 °C

The accumulation of VO under pulse electron irradiation is a result of the periodic action of two alternate processes:

1) during the action of a pulse, there occur the creation of VO (the rate of generation of free vacancies λ_V) and simultaneously their annealing with the time constant τ_1. In this case, the system of kinetic equations consists of two equations which describe the creation and the annealing of vacancies V and VO:

$$\begin{cases} \frac{d[\mathrm{V}]}{dt} = \lambda_V - \chi_{\mathrm{VO}}[\mathrm{V}][\mathrm{O}_i], \\ \frac{d[\mathrm{VO}]}{dt} = \chi_{\mathrm{VO}}[\mathrm{V}][\mathrm{O}_i] - \frac{[\mathrm{VO}]}{\tau_1}, \end{cases} \quad (1)$$

where χ_{VO} is the constant characterizing the capture of a vacancy by oxygen;

2) in the time interval between successive pulses, there occurs only the thermal (without irradiation) annealing of VO which were accumulated during the action of a

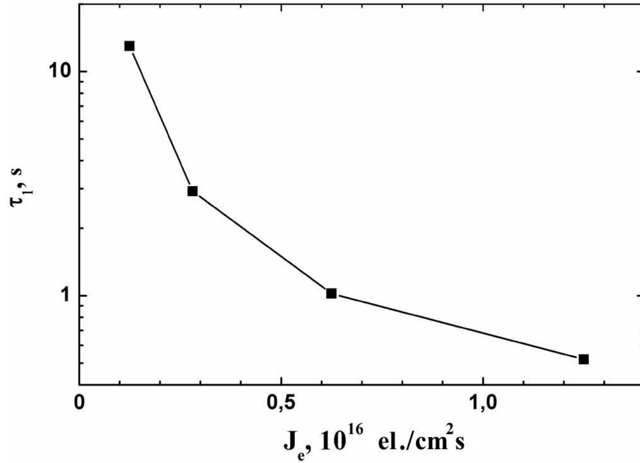


Fig. 4. Annealing constant τ_1 versus the electron radiation intensity at 633 K

pulse. This thermal annealing is characterized by the parameter τ_2:

$$\frac{d[\mathrm{VO}]}{dt} = -\frac{[\mathrm{VO}]}{\tau_2}. \tag{2}$$

The kinetic equations for all periods of the irradiation are the same as (1) and (2). Only the initial conditions will be varied due to the accumulation of defects. As a result, the dependence of the concentration of VO on the irradiation duration t reads [1]:

$$[\mathrm{VO}] = \lambda_V \tau_1 \times \frac{\exp\left(-\frac{t_{\mathrm{off}}}{\tau_2}\right)\left(1-\exp\left(-\frac{t_p}{\tau_1}\right)\right)}{1-\exp\left(-\frac{t_{\mathrm{off}}}{\tau_2}\right)\exp\left(-\frac{t_p}{\tau_1}\right)} \times$$

$$\times \left(1-\exp\left(-\left[\frac{t_{\mathrm{off}}}{\tau_2}+\frac{t_p}{\tau_1}\right]\times\frac{t}{t_{\mathrm{off}}+t_p}\right)\right), \tag{3}$$

where t_{off} is the time interval between neighboring pulses.

In formula (3), t_p and t_{off} are the given technical parameters of the pulse irradiation. We determined the constant of annealing τ_2 from another experiment. For this purpose, we irradiated a part of specimens at room temperature and then carried out the annealing at $T > 300$ °C. Hence, only λ_V and τ_1 can depend on the radiation intensity.

It was shown in experiments [1] that an increase in the radiation intensity J causes a linear increase in the rate of generation of free vacancies λ_V. This means that each electron creates the same number of free vacancies at various J ($\lambda_V/J = (7.9 \pm 0.2) \times 10^{-2}$ cm^{-1}).

The nonlinear accumulation of VO with output to the saturation (Fig. 1) is caused by the simultaneous presence of two competing processes: the creation of VO and their annealing. Whereas the creation runs with constant rate, the rate of annealing is proportional to the concentration of defects: $d[\mathrm{VO}](\mathrm{t})/dt \sim [\mathrm{VO}](\mathrm{t})$. The concentration of VO increases with the irradiation duration. Respectively, the rate of annealing and its contribution to the process of accumulation increase as well. If the rate of annealing becomes equal to the rate of creation, the concentration of VO approaches the saturation. In our case, this happens, if the following condition is satisfied in (3): $\left[\frac{t_{\mathrm{off}}}{\tau_2}+\frac{t_p}{\tau_1}\right]\times\frac{t}{t_{\mathrm{off}}+t_p} \gg 1$. Then we have

$$[VO]_{\max} = \frac{\lambda_V}{\frac{t_{\mathrm{off}}/t_p}{\tau_2}+\frac{1}{\tau_1}}. \tag{4}$$

By measuring the concentration of VO under saturation, the value of τ_1 can be determined from (4). In Fig. 4, we present the dependence of τ_1 on the radiation intensity at 360 °C. It is seen that τ_1 decreases by more than one order, as J increases by one order. This means that the electron radiation intensity can significantly stimulate the annealing of VO created by this radiation.

3.3. Models of accelerated annealing of VO

Thus, as the intensity of the irradiation of Si by high-energy electrons at high temperatures grows, the annealing of VO complexes created by this radiation is accelerated. The quantity τ_1 characterizing the rate of annealing of defects during the action of a pulse of electrons decreases, as the intensity increases. The reasons for such a change should be clarified.

As J increases, two processes are running:

1) the rate of creation of Frenkel pairs (primary defects) grows;

2) the rate of generation of nonequilibrium charge carriers increases as well.

One of these processes must stimulate the annealing of VO during the action of pulses of radiation.

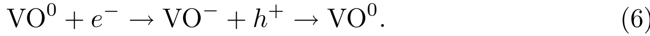
VO complexes can disappear due to the capture of vacancies, whose concentration increases with the intensity

$$\mathrm{VO} + \mathrm{V} \rightarrow \mathrm{V_2O}. \tag{5}$$

But process (5) is improbable, since the vacancies are captured by oxygen atoms, whose concentration is much greater than the concentration of VO.

Hence, the process of excitation of the electron subsystem is responsible for a decrease in τ_1. This can occur

due to the athermic motion of defects at their recharging, which is an analog of the motion of intrinsic interstitial atoms in the model of potential inversion [8]:



$$VO^0 + e^- \rightarrow VO^- + h^+ \rightarrow VO^0. \tag{6}$$

We now write the kinetic equation for the recharging of defects,

$$\frac{d[VO^0]}{dt} = -\sigma_n^0 \upsilon_n \left(n_0 + \Delta n\right)[VO^0]+$$

$$+\sigma_n^0 \upsilon_n N_{cVO}[VO^-] + \sigma_p^- \upsilon_p \left(p_0 + \Delta p\right)[VO^-], \tag{7}$$

where $N_{cVO} = N_c \exp\left(-E_{VO}/k_B T\right)$, N_c – effective density of states in the conduction zone, E_{VO} – electron energy level of VO complexes, n_0, p_0, Δn, Δp – equilibrium and nonequilibrium concentrations of electrons and holes, respectively.

It follows from (7) that the rate of recharging (hence, and the rate of annealing) must depend on the concentrations of equilibrium and nonequilibrium charge carriers. The significant decrease in τ_1 (Fig. 4) is observed already at the intensity $J_p = 2.81 \times 10^{15}$ electron/(cm^{2}s). In this case, the evaluation of the maximum concentration of nonequilibrium carriers gives $\Delta n \approx 4 \times 10^{15}$ cm^{-3}. The equilibrium concentrations of electrons and holes at this temperature (633 K) are of the same order of magnitude. Let us increase the equilibrium concentration due to the increase in the temperature to a value such that it exceeds significantly the nonequilibrium concentration and the doping level. If the accelerated annealing of defects is determined by the process of alternate capture of electrons and holes, we must observe the accelerated annealing of formed VO complexes. Work [7] gives the temperature dependences of the rates of creation and annealing of VO under pulse electron irradiation in the range of temperatures from room temperature to 753 K. The pulse radiation intensity was 10^{15} electron/(cm^{2}s). At this intensity, the accelerated annealing of defects is not yet observed (see Fig. 4). The estimation shows that the kinetics of annealing of VO ($T = 728$ K, $n_0 = p_0 \approx 2 \times 10^{16}$ cm^{-3}) is characterized by the same parameters (the frequency factor $\nu = 2 \times 10^8$ s^{-1} and the annealing activation energy $E_{VO} = 1.5$ eV) as the isothermal annealing in the interval 570–620 K. Thus, the recharging of defects is not responsible for the accelerated annealing.

We need to seek another process which would accelerate the annealing of VO, as the radiation intensity increases.

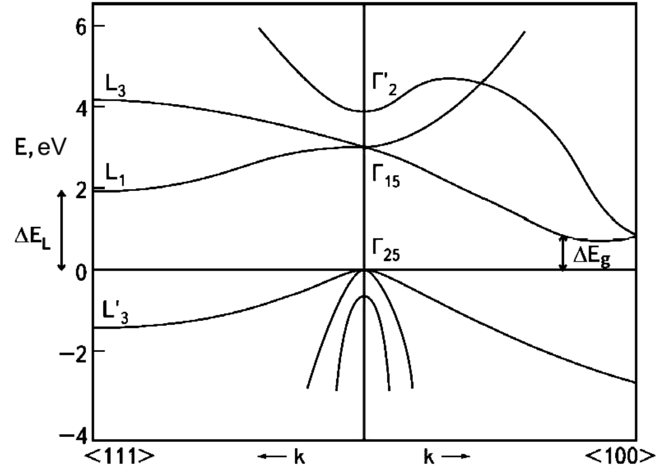


Fig. 5. Scheme of the band structure of Si

But, as we have already seen, the acceleration of the annealing of VO can be affected by exciting namely the electron subsystem of a crystal. We note that the mean energy spent on one excited electron-hole pair is equal to ~ 3.6 eV [9]. This means that electrons can fall in higher conduction subbands. In Fig. 5, we give the calculated band structure of Si [10]. It is worth noting the minimum of the conduction band at the point L_1. Different methods of calculation give somewhat different results. However, the energy at this point equals, on the average, approximately 2±0.1 eV relative to the highest point of the valence band. Hence, a part of electrons excited in the conduction band will fall in this minimum of the band. At the capture by VO, such an electron can transfer the energy to it which is close to the annealing activation energy of VO.

We consider that the captured electron stimulates the jump of the defect to the neighboring position in the crystal. As a result, VO will move till the time of its capture by an oxygen atom:

$$VO + O \rightarrow VO_2. \tag{8}$$

For the constant of accelerated annealing τ_1, we can write

$$\frac{1}{\tau_1} = \sigma_n^0 \upsilon_n \Delta n^L \chi_{VO_2}[O_i] \exp\left(-\frac{E^*_{VO}}{k_B T}\right), \tag{9}$$

where σ_n^0, υ_n, and Δn^L are the capture cross-section, thermal velocity, and the concentration of electrons in the valley L_1 of the conduction band, respectively; χ_{VO_2} is the constant of capture of VO by an oxygen atom, $E^*_{VO} = E_a - E_L$ is the difference between the activation energy of the thermal annealing E_a and the energy E_L

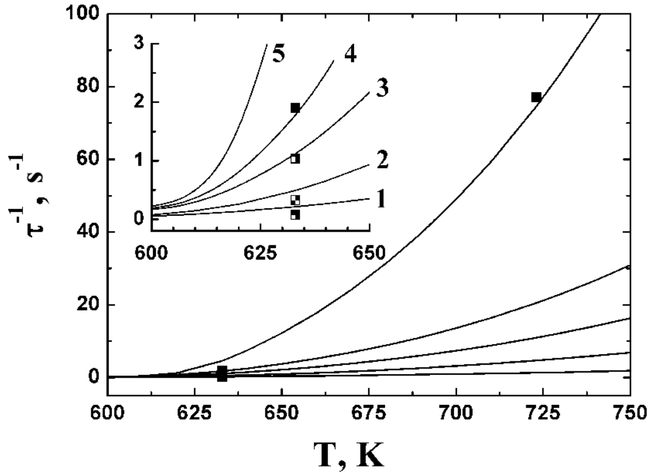


Fig. 6. Annealing constant $1/\tau_1$ versus the temperature of specimens under electron irradiation with radiation intensities: *1* – 1.25; *2* – 2.81; *3* – 6.25; *4* – 12.5; *5* – 50×10^{15} electron/(cm^{2}s). Points – experiment; continuous curves – results of calculations by formula (12)

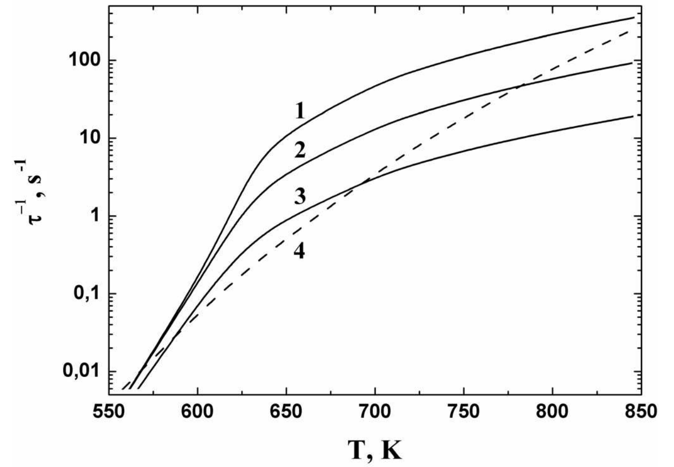


Fig. 7. Calculated annealing constants $1/\tau_1$ (curves *1–3*) and $10^3/\tau_2$ (curve *4*) versus the temperature of specimens under electron irradiation with radiation intensities: *1* – 50; *2* – 12.5; *3* – 2.81×10^{15} electron/(cm^{2}s). Curves (*1–3*) – results of calculations by formula (12); curve *4* – $10^3/\tau_2 = 2 \times 10^8 \exp(-1.5/k_{\rm B}T)$

transferred to VO at the capture of an electron into a high-energy valley. We have

$$E_L = \Delta E_L - (\Delta E_g - E_{\rm VO}), \qquad (10)$$

where ΔE_g is the energy gap, and $E_{\rm VO} = 0.17$ eV is the electron energy level of VO. With regard for the temperature dependence of ΔE_g, we obtain that $E^*_{\rm VO}$ is in the limits 0.37–0.47 eV.

At the determination of the concentration Δn^L, we need to take into account that these electrons will disappear due to the capture by VO and phosphorus atoms (the deep level relative to L_1). We have

$$\frac{d\Delta n^L}{dt} = \lambda_n^L - \sigma_n^0 \upsilon_n \Delta n^L [{\rm VO}] - \sigma_{\rm n}^+ \upsilon_{\rm n} \Delta {\rm n}^{\rm L}[{\rm P}], \qquad (11)$$

where λ_n^L is the rate of generation of electrons into the valley L_1, and σ_n^+ is the cross-section of the capture of electrons by phosphorus atoms.

The evaluation of the lifetime of nonequilibrium carriers shows that it is less by at least one order than the duration of an electron pulse. Hence, we have the stationary state relative to Δn^L in (11). As a result, we obtain

$$\frac{1}{\tau_1} = \frac{J_p}{[{\rm VO}]_{\max} + \frac{\sigma_{\rm n}^+}{\sigma_{\rm n}^0}[{\rm P}]} B \exp\left(-\frac{E^*_{\rm VO}}{k_{\rm B}T}\right), \qquad (12)$$

where $\lambda_n^L = AJ_p$, and $B = A\chi_{{\rm VO}_2}[{\rm O}_i]$ is a constant.

In (12), the cross-sections of the capture of electrons from the valley L_1 by VO defects and phosphorus atoms are unknown. We assume that these values are slightly different from the capture cross-section for electrons from the basic band. Unfortunately, the theory of the capture of carriers at high temperatures is not available. Therefore, we make one more assumption that the temperature dependence of the capture cross-sections for sufficiently deep electron levels (when there exists a dense spectrum of excited levels) is conserved: T^{-1} for a neutral center and T^{-3} for an oppositely charged one. Thus, we can write $\sigma_n^0 = 8 \times 10^{-15}\,(300/T)$ cm^2 [11], and $\sigma_n^+ = 5 \times 10^{-15}\,(300/T)^3$ cm^2 [12].

To determine the energy E^*_{VO}, we use the data obtained within the DLTS method of measurements for specimens irradiated at 723 K. At a pulse radiation intensity of 5×10^{16} electron/(cm^{2}s), we obtain that $[{\rm VO}]_{\max} = 5 \times 10^{13}$ cm^{-3}. Substituting the data for the irradiation at 633 K ($J_p = 1.25 \times 10^{16}$ electron/(cm^{2}s), $[{\rm VO}]_{\max} = 4.6 \times 10^{14}$ cm^{-3}) and the data at 723 K in (12), we obtain that $E^*_{\rm VO} = 0.40$ eV. In other words, the value of $E^*_{\rm VO}$ falls in the interval of possible energies (0.37–0.47 eV).

Substituting the experimental values of τ_1, J_p, $[{\rm VO}]_{\max}$, and [P], as well as $E^*_{\rm VO}$, in (12), we obtain that the constant $B \approx 1.3 \times 10^2$ cm^{-1}.

We can evaluate the rate of generation of electrons into the valley L_1. The constant $\chi_{{\rm VO}_2} = (a_1)^2\, r_{{\rm VO}_2}$. Let the jump length a_1 and the radius of the capture of VO defects by oxygen atoms be of the order of 5×10^{-8} cm (lattice constant). Then, at the concentration of oxygen $\sim 7 \times 10^{17}$ cm^{-3}, we obtain that the constant $A \approx 10^6$

cm^{-1}. Hence, the rate of generation of electrons into the valley L_1 is approximately equal to the total rate of generation of nonequilibrium charge carriers under the irradiation with 1-MeV electrons. At the pulse intensity $J_p = 6.25 \times 10^{15}$ electron/(cm^{2}s), we obtain that $\lambda_n^L \approx 10^{22}$ cm^{-3}s^{-1}.

Formulas (4) and (12) allow us to obtain the analytic dependences of $[\mathrm{VO}]_{\max}$ and τ_1 on the temperature and the pulse radiation intensity. In Fig. 3, we show the dependence of $[\mathrm{VO}]_{\max}$ on J_p for several temperatures. Figure 6 presents the dependence of $1/\tau_1$ on the temperature for several radiation intensities. We see that the theoretical curves coincide satisfactorily in the error bars with experimental points.

We can determine the interval of temperatures, in which the effect of acceleration of the annealing of VO under irradiation holds. The effect should be observed only if the rate of accelerated annealing is greater than the rate of ordinary thermal annealing. To this end, we present the calculated curves of the temperature dependences of $1/\tau_1$ and $10^3/\ \tau_2$ in Fig. 7. It is seen that the effect is manifested for all intensities near 570 K. However, the temperature, at which the effect disappears, increases with the radiation intensity.

4. Conclusions

The irradiation of Cz n-Si with 1-MeV electrons at a temperature higher than the temperature of thermal annealing of VO defects leads to the accelerated annealing of defects created by this radiation. The accelerated annealing happens during the action of a pulse of electrons. The constant of accelerated annealing τ_1 decreases sharply, as the radiation intensity increases. At 633 K, the increase in the intensity by one order causes the decrease in τ_1 by more than one order. In this case, the dependence of the concentration of VO on the irradiation duration has the form of curves with saturation. The maximum concentration of VO increases with the radiation intensity and decreases, as the temperature of irradiated specimens increases.

The reason for the accelerated annealing can be the capture of electrons from the valley L_1 by VO defects. In this case, the defects receive a significant energy, which causes the decrease in the annealing activation energy for VO from 1.5 eV down to 0.4 eV.

The effect of accelerated annealing is observed in the bounded interval of temperatures which begins from 570 K (the onset of the thermal annealing of VO). The high-temperature boundary of the effect depends on the radiation intensity and increases with the intensity.

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МЕХАНІЗМ ВІДПАЛУ VO ДЕФЕКТІВ
В n-Si ПРИ ВИСОКОТЕМПЕРАТУРНОМУ
ІМПУЛЬСНОМУ ЕЛЕКТРОННОМУ ОПРОМІНЕННІ

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Р е з ю м е

Досліджено кінетику накопичення комплексу вакансія–кисень (VO) в n-Si, вирощеному методом Чохральського (Cz), при різних інтенсивностях імпульсного 1 МеВ електронного опромінення при температурах, вищих за температуру початку термічного відпалу VO ($T \geq 300$ °C). Показано, що опромінення електронами при таких температурах приводить до прискоре-

ного відпалу VO, створених цим опроміненням. Прискорений відпал VO відбувається у процесі дії імпульсу електронів. Максимальна концентрація VO, яка при цьому утворюється, зростає зі збільшенням інтенсивності опромінення і зменшується при збільшенні температури зразків при опроміненні. Запропоновано модель процесу прискореного відпалу, яка ґрунтується на тому, що при електронному опроміненні збуджуються електрони у високоенергетичну долину. При захопленні VO дефектами таких електронів дефектам передається енергія, яка суттєво зменшує енергію активації їх відпалу. Високотемпературна межа ефекту залежить від інтенсивності опромінення і зростає зі збільшенням інтенсивності.