EFFECT OF THE INTENSITY OF ELECTRON IRRADIATION ON THE GENERATION AND ANNEALING OF VO-CENTERS IN SILICON AT HIGH TEMPERATURES

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The kinetics of accumulation of vacancy-oxygen (VO) complexes in Czochralski-grown (Cz) n-Si is studied at different intensities of a pulsed 1-MeV electron irradiation at 360 °C. It is shown that, in the case of the irradiation accompanied by the simultaneous generation and annealing of VO complexes, the kinetics of their accumulation is nonlinear and has the form of a saturated curve due namely to the annealing. It is found out that there exists a limiting (maximum) VO concentration determined both by the irradiation intensity and by the temperature of samples under irradiation. It is also established that the intensity of high-temperature irradiation of silicon by 1-MeV electrons can essentially stimulate the annealing of the VO complexes created by them. At 360 °C, the variation of the pulse irradiation intensity from 1.25×10^{15} to 1.25×10^{16} electrons/(cm^{2}s) does not noticeably influence the efficiency of generation of VO centers in n-Si, but accelerates their annealing by approximately two orders of magnitude.

1. Introduction

Processes of formation and annealing of radiation-induced defects in semiconductors are usually investigated separately. First, one studies the processes of defect formation at some temperature. When radiation has created a sufficient concentration of radiation-induced defects in the crystal, one carries on their annealing at a temperature higher than the temperature, at which the irradiation was realized. In this case, the interconnection of these two processes, as well as the simultaneous influence of a perturbation of the crystal by ionizing radiation on these processes, remains without attention. That is why it is interesting to study the formation and the annealing of radiation defects at such a temperature, at which radiation-induced defects are effectively annealed.

From this viewpoint, it is convenient to study the VO complex in Cz n-Si crystals. The concentration of oxygen in such crystals is much higher than those of other impurities, that is why the VO concentration after the irradiation considerably exceeds those of other vacancy defects. In addition, the VO properties have been already studied during half a century, and they are known rather well now (for the irradiation temperatures, at which they are stable). From the data on measurements of the Hall effect [1–3] and those obtained by deep-level transient spectroscopy (DLTS) [4–7], it is established that a VO complex has the acceptor level $E_{\rm VO}(-/0) \approx E_{\rm c} - 0.17$eV and is an active recombination center [5]. Using the electron paramagnetic resonance (EPR), the structure of VO centers was clarified [8]. The VO complex is observed in infrared absorption spectra in two charge states: neutral and negative ones [9–12]. At room temperature, the absorption bands for the neutral and negative states are located, respectively, close to 830 and 877 cm^{-1}. At low temperatures, the positions of these bands shift to the high-frequency region, 835 and 885 cm^{-1}, respectively. The VO complex is annealed at the temperatures $T \geq 300$ °C [4, 5, 9, 10]. In Cz Si, a considerable part of VO centers can be annealed by means of the migration as a whole and the capture by oxygen, which results in the formation of the VO_2 complex [10, 13]. This defect is electrically active in the metastable VO_2^* configuration that includes a shallow acceptor level $E_{\rm c} - 0.06$ eV [14]. For today, VO centers remain an urgent object for experimental and theoretical investigations [15–18].

In the recent experiments on high-temperature irradiation [19–22] and those performed earlier by the DLTS technique, it was shown that VO centers in Si can be the main radiation defects even in the case where the temperature, at which irradiation occurs, exceeds or equals their annealing temperature ($T \geq 300$ °C). This result is interesting from the scientific viewpoint, but the kinetics of VO accumulation under high-temperature ir-

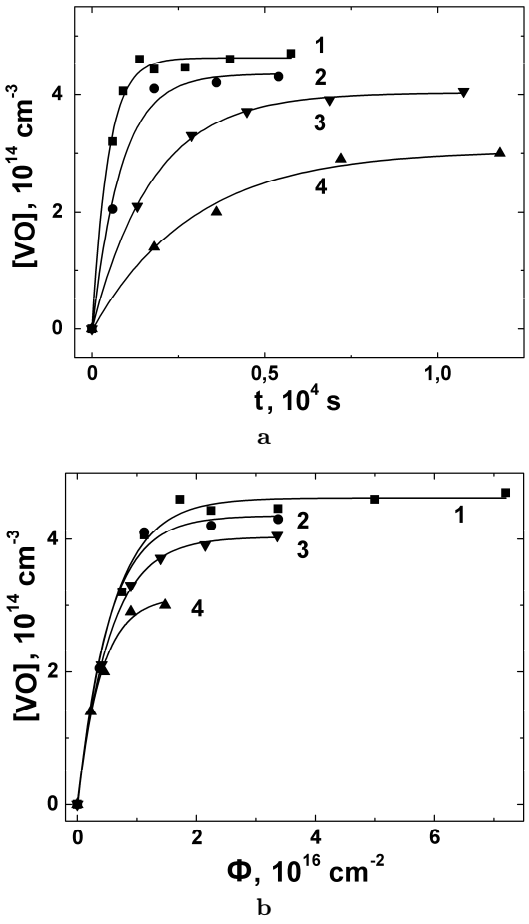

Fig. 1. Time (a) and dose (b) dependences of the VO concentration at various intensities of the 1-MeV electron irradiation at 360 °C: *1* – 2; *2* – 1; *3* – 0.45; *4* – 0.2 μA/cm^2. Points – experimental data; solid lines – the result of calculations by formula (10)

radiation, as well as the factors influencing this process, practically has not been yet investigated in detail. In addition, the understanding of the formation and the evolution of radiation-induced defects in silicon under extreme irradiation conditions can be useful for practical applications as well.

This work aimed at the study of the processes of VO accumulation in Cz n−Si at the temperature of their effective annealing depending on the electron irradiation intensity.

2. Experiment

Our measurements were performed using Cz n-Si samples with the concentrations of phosphorus [P] $\approx 1\times10^{15}$ cm^{-3}, oxygen [O$_i$] = (6÷7)$\times10^{17}$ cm^{-3}, and carbon [C$_s$] $\leq 5\times10^{16}$ cm^{-3}. As was already noted, the oxygen concentration in these samples is much higher than the concentrations of other impurities and radiation-induced defects after irradiation, so that we may consider that all vacancies generated due to the decay of Frenkel pairs contribute to the VO formation.

The samples were irradiated with the help of a pulsed electron accelerator with the following parameters: the pulse duration $t_p = 3.3$ μs, the duty cycle $s = 1000$, and the electron energy $E_e = 1$ MeV. The mean electron-flow intensities amounted to $\langle J\rangle = 0.2\div2$ μA/cm^2, which corresponded to the pulse values $J_p = 1.25\times10^{15}\div1.25\times10^{16}$ electrons/(cm^{2}s). The irradiation was performed at a temperature of 360 °C. This temperature was chosen as the optimal one, at which there take place both processes of VO formation and annealing. The samples were heated with the help of a special oven. The temperature was stabilized using a high-precision temperature regulator. After irradiation, the oven was immediately turned off. According to our estimates, the VO concentration during the cooling from 360 to 300 °C will decrease by no more than 2–3%. In order to determine the parameters of the VO thermal annealing, a part of the samples was irradiated at room temperature and after that annealed at 360 °C.

We experimentally investigated the time and dose dependences of the VO concentration at different irradiation intensities at 360 °C. Each point of these dependences corresponds to a separate irradiated sample. The VO concentration after irradiation was determined from the temperature dependences of the Hall effect. They clearly show the recharge of the level of this defect ($E_c - 0.17$ eV), which allows one to calculate its concentration to a sufficient accuracy.

3. Results and Their Discussion

3.1. Experimental Results

Figure 1 shows the time (a) and dose (b) dependences of the VO concentration at different intensities of the electron irradiation. Being considered together, these dependences give a more complete description of the effect of the irradiation intensity J on the VO accumulation: the time ones characterize the accumulation rate ($\frac{d[\mathrm{VO}]}{dt}$) and the dose ones – its efficiency ($\frac{d[\mathrm{VO}]}{d\Phi}$). The relation between the accumulation efficiency and rate has the following form:

$$\frac{d[\mathrm{VO}]}{d\Phi} = \left(\frac{1}{J}\right)\left(\frac{d[\mathrm{VO}]}{dt}\right). \qquad (1)$$

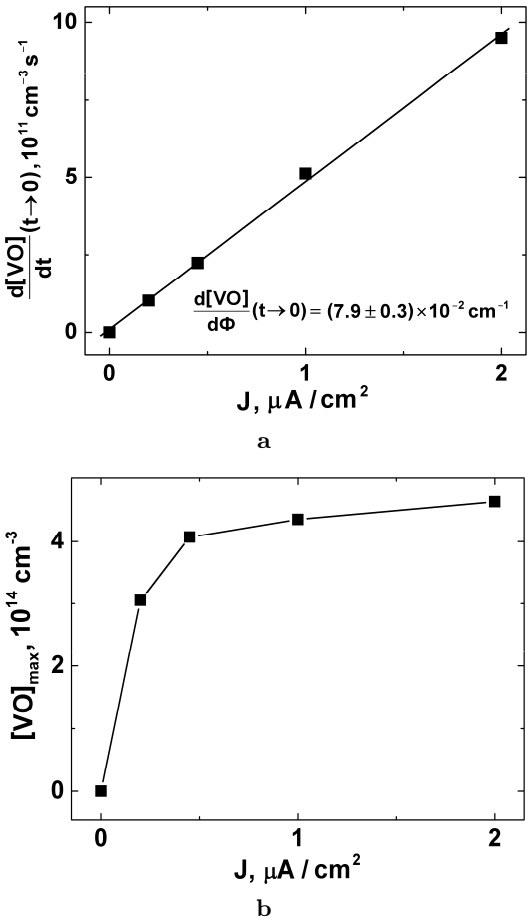

Fig. 2. Initial accumulation rate of VO centers (a) their concentration at the saturation (b) versus the intensity of a pulsed 1-MeV electron irradiation at 360 °C

The quantity $\frac{d[\mathrm{VO}]}{d\Phi}$ represents the concentration of VO centers formed by one irradiation electron and therefore can serve as an objective parameter for the quantitative comparison at different irradiation intensities.

From the experimental data given in Fig. 1, one can see that

a) the VO accumulation at 360 °C is described by a saturated curve and is qualitatively the same at all J;

b) the initial rate of accumulation of VO centers $\frac{d[\mathrm{VO}]}{dt}(t \to 0)$ is a linear function of the irradiation intensity J (see Fig. 2,a), whereas the efficiency of their formation is a constant quantity $\frac{d[\mathrm{VO}]}{d\Phi}(t \to 0) = (7.9 \pm 0.3) \times 10^{-2}$ cm^{-1}, i.e. it is independent of the irradiation intensity;

c) the VO saturation concentration ($[\mathrm{VO}]_{\max}$) grows with the irradiation intensity J, by tending to saturation, which is demonstrated in Fig. 2,b.

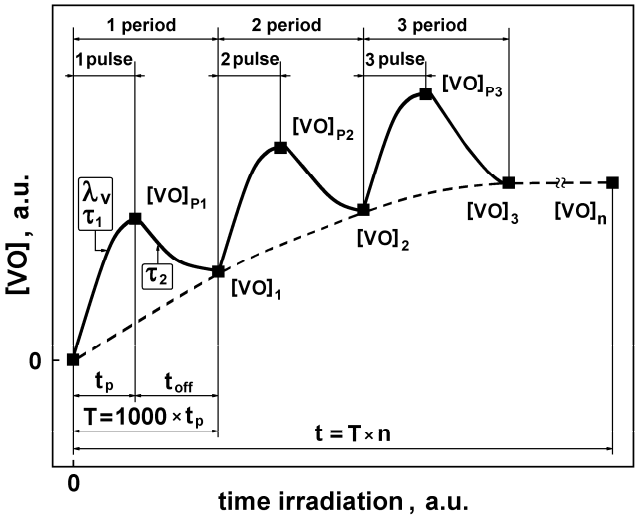

Fig. 3. Scheme of the VO accumulation at a pulsed high-temperature electron irradiation at 360 °C. Scale and proportions are arbitrary

3.2. Kinetics of the VO Accumulation

The VO accumulation under a pulsed electron irradiation at 360 °C represents a result of the periodic action of two alternate processes (Fig. 3):

a) during a pulse, the formation of VO complexes with the free-vacancy generation factor λ_{V} and their simultaneous annealing with the time constant τ_1 take place;

b) between pulses, VO complexes are thermally (without the action of radiation) annealed, which is characterized by the parameter τ_2.

As a result, the real curve of the VO accumulation is saw-toothed and the experimental curve is obtained by measuring the VO concentration in some minima of this saw-toothed dependence. However, the amplitudes of these jumps are inessential as compared to the measurement error of a defect concentration. That is why the VO concentration as a function of the irradiation time (or dose) represents a smooth curve. But the saw-toothed character of the dependence should be taken into account when considering the physics of processes.

Let us describe the kinetics of VO accumulation for the first several periods.

Period 1:

The system of kinetic equations at the action of the irradiation pulse includes two equations describing the generation and the annealing of V and VO.

$$\begin{cases} \frac{d[\mathrm{V}]}{dt} = \lambda_{\mathrm{V}} - \chi_{\mathrm{VO}}[\mathrm{V}][\mathrm{O}_i], \\ \frac{d[\mathrm{VO}]}{dt} = \chi_{\mathrm{VO}}[\mathrm{V}][\mathrm{O}_i] - \frac{[\mathrm{VO}]}{\tau_1}, \end{cases} \tag{2}$$

where λ_V is the free-vacancy generation rate and χ_{VO} is the constant of the vacancy capture by oxygen.

As follows from [25], the lifetime of vacancies in n-Si does not exceed 1 μs. This means that, during the electron pulse ($t_p = 3.3$ μs), system (2) reaches a stationary state with respect to vacancies ($d[V]/dt = 0$). With regard for the initial condition $[VO]|_{t=0} = 0$, one obtains the VO concentration at the end of the pulse:

$$[VO]_{P1} = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right). \quad (3)$$

After the end of the irradiation pulse, there starts the thermal annealing of VO complexes accumulated during the pulse. We have

$$\frac{d[VO]}{dt} = -\frac{[VO]}{\tau_2}. \quad (4)$$

The solution of (4) with the initial condition $[VO]|_{t=0} = [VO]_{P1}$ yields the resulting VO concentration after the first period of irradiation,

$$[VO]_1 = [VO]_{P1} \exp\left(-\frac{t_{off}}{\tau_2}\right),$$

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

With regard for (3),

$$[VO]_1 = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right) \exp\left(-\frac{t_{off}}{\tau_2}\right). \quad (5)$$

Period 2:

All kinetic equations are the same as those for the first irradiation period, but the initial conditions are different. The VO accumulation during the second pulse starts from the concentration remaining as of the time moment of the beginning of the second pulse, i.e. $[VO]_1$. Respectively,

$$[VO]_{P2} = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right) \times$$

$$\times \left[1 + \exp\left(-\frac{t_p}{\tau_1}\right) \exp\left(-\frac{t_{off}}{\tau_2}\right)\right], \quad (6)$$

$$[VO]_2 = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right) \times$$

$$\times \left[1 + \exp\left(-\frac{t_p}{\tau_1}\right) \exp\left(-\frac{t_{off}}{\tau_2}\right)\right] \exp\left(-\frac{t_{off}}{\tau_2}\right). \quad (7)$$

Applying the same considerations, we can write

Period 3:

$$[VO]_3 = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right) \left[1 + \exp\left(-\frac{t_p}{\tau_1}\right) \times \right.$$

$$\left. \times \exp\left(-\frac{t_{off}}{\tau_2}\right) + \left(\exp\left(-\frac{t_p}{\tau_1}\right) \exp\left(-\frac{t_{off}}{\tau_2}\right)\right)^2\right] \times$$

$$\times \exp\left(-\frac{t_{off}}{\tau_2}\right). \quad (8)$$

Period *n*:

$$[VO]_n = \lambda_V \tau_1 \left(1 - \exp\left(-\frac{t_p}{\tau_1}\right)\right) \left[1 + \exp\left(-\frac{t_p}{\tau_1}\right) \times \right.$$

$$\left. \times \exp\left(-\frac{t_{off}}{\tau_2}\right) + \ldots + \left(\exp\left(-\frac{t_p}{\tau_1}\right) \exp\left(-\frac{t_{off}}{\tau_2}\right)\right)^{n-1}\right] \times$$

$$\times \exp\left(-\frac{t_{off}}{\tau_2}\right). \quad (9)$$

The expression in the brackets of Eq.(9) represents a geometric progression and can be written down in a more compact form. We also take into account that the number of irradiation pulses is equal to the ratio of the irradiation time to the period between pulses $n = t/(t_{off} + t_p)$. As a result, the expression describing the kinetics of VO accumulation at a high-temperature pulsed electron irradiation takes the form

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

$$\times \left(1 - \exp\left(-\left[\frac{t_{off}}{\tau_2} + \frac{t_p}{\tau_1}\right] \frac{t}{t_{off} + t_p}\right)\right). \quad (10)$$

3.3.1. Description of the Experiment

Expression (10) contains five parameters. Two of them (t_p and t_{off}) are the known technical parameters of pulsed irradiation. All the other ones (λ_V, τ_1, and τ_2) characterize the processes of VO generation and annealing and are unknown. Moreover, only λ_V and τ_1 can depend on the irradiation intensity. That is why in order to decrease the number of fitting parameters, the annealing constant τ_2 was determined from an independent

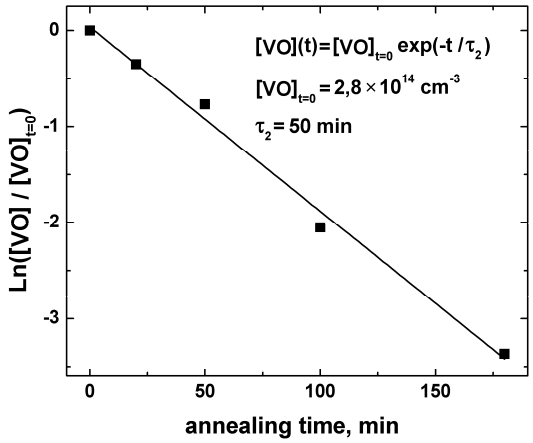

Fig. 4. Isothermal annealing of VO at 360 °C

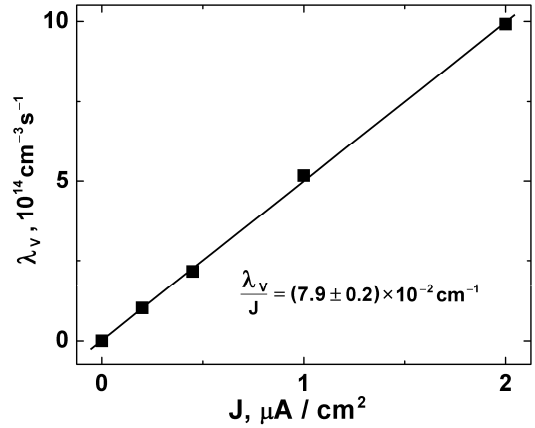

Fig. 5. Free-vacancy generation factor versus the electron irradiation intensity J at 360 °C

experiment. For this purpose, a part of the samples was irradiated at room temperature and then isothermally annealed at 360 °C. The result obtained for one of the samples is presented in Fig. 4. We obtained that τ_2=50 min.

Solid curves in Fig. 1 correspond to the best coincidence of theory (10) with the experiment. The obtained values of the free-vacancy generation factor $\lambda_{\rm V}$ and the annealing constant τ_1 as functions of the irradiation intensity J are given in Figs. 5 and 6, respectively. One can see from Fig. 5 that an increase of the irradiation intensity J induces a linear rise of the free-vacancy generation rate $\lambda_{\rm V}$, which means that each irradiation electron creates the same number of free vacancies at different $J(\frac{\lambda_V}{J} = (7.9 \pm 0.2) \times 10^{-2}{\rm cm}^{-1})$. In other words, the generation efficiency of free vacancies does not depend on the irradiation intensity in the interval of values used in the experiment. At the same time, Fig. 6 demonstrates that an increase of J very strongly (by orders of magnitude) decreases the annealing constant τ_1. This decrease means that the ionizing irradiation can considerably accelerate the annealing of VO complexes.

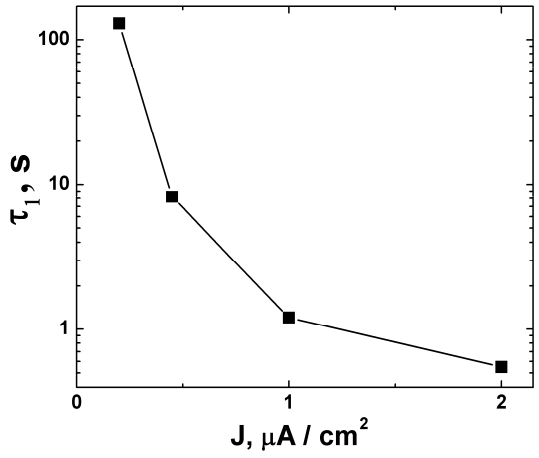

Fig. 6. Annealing constant versus the electron irradiation intensity J at 360 °C

3.3.2. Effect of Irradiation Intensity on Two Extreme Stages of VO Accumulation: Initial and Saturation Ones

A. Initial Stage

In this case, the following condition is satisfied: $\left[\frac{t_{\rm off}}{\tau_2} + \frac{t_p}{\tau_1}\right] \frac{t}{t_{\rm off}+t_p} \ll 1$. Moreover, $t_{\rm off}/\tau_2 \ll 1$ ($t_{\rm off} = 3.3 \times 10^{-3}$ s, $\tau_2 = 3 \times 10^3$ s) and $t_p/\tau_1 \ll 1$ ($t_p = 3.3 \times 10^{-6}$ s, $\tau_1 > 0.5$ s). Then expression (10) yields

$$[{\rm VO}] = \lambda_{\rm V} \frac{t_p}{t_{\rm off}+t_p} t = \frac{\lambda_{\rm V}}{s} t, \quad \frac{d[{\rm VO}]}{dt} = \frac{\lambda_{\rm V}}{s}$$

and

$$\frac{d[{\rm VO}]}{d\Phi} = \left(\frac{1}{s}\right)\left(\frac{\lambda_{\rm V}}{\langle J\rangle}\right) = \frac{\lambda_{\rm V}}{J}. \tag{11}$$

One can see from (11) that the accumulation of VO centers at the initial stages is determined only by their formation (generation of free vacancies). In this case, the rate and the efficiency of the VO accumulation are constant and will depend on the irradiation intensity only in the case where Jwill influence the free-vacancy generation factor $\lambda_{\rm V}$. Figures 2,*a* and 5 demonstrate that the

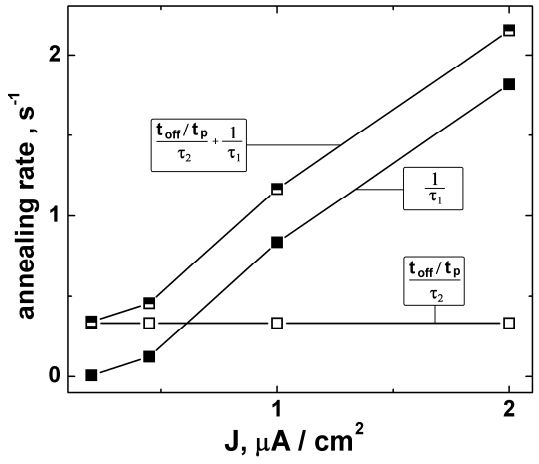

Fig. 7. Relative contribution of different mechanisms of annealing: $\frac{t_{\rm off}/t_i}{\tau_2}$ – annealing only due to the temperature (the time interval between pulses); $\frac{1}{\tau_1}$ – annealing during the action of an electron irradiation pulse, and $\frac{t_{\rm off}/t_i}{\tau_2}+\frac{1}{\tau_1}$ – their sum

efficiency of the generation of free vacancies $\lambda_{\rm V}/J$ and the initial efficiency of the VO accumulation $(d[{\rm VO}]/d\Phi)$ are equal.

B. Saturation Stage

The nonlinear accumulation of VO complexes tending to the saturation is caused by the simultaneous presence of two competitive processes: VO generation and annealing. Whereas the generation is characterized by a constant rate, the annealing rate is proportional to the defect concentration: $\frac{d[{\rm VO}](t)}{dt} \sim [{\rm VO}](t)$. At the initial stage, where the VO concentration is small, the annealing also occurs "slowly" and practically does not influence the final concentration, which is seen from expression (11). With increase in the irradiation duration, the VO concentration grows. Therefore, the annealing rate and its contribution to the accumulation rise. At the moment when the rate of annealing reaches that of generation, the VO concentration will saturate. In our case, this situation takes place under the following condition:

$$\left[\frac{t_{\rm off}}{\tau_2}+\frac{t_p}{\tau_1}\right]\frac{t}{t_{\rm off}+t_p} \gg 1.$$

According to (10), we obtain

$$[{\rm VO}]_{\max} = \frac{\lambda_{\rm V}}{\frac{t_{\rm off}/t_p}{\tau_2}+\frac{1}{\tau_1}}. \qquad (12)$$

Expression (12) shows that the annealing efficiency is determined by the partial contributions of two processes. The first one $\frac{t_{\rm off}/t_p}{\tau_2}$ describes the isothermal annealing between pulses, while the second one $\frac{1}{\tau_1}$ corresponds to the annealing during an irradiation pulse. In order to explain the experimental dependence $[{\rm VO}]_{\max}(J)$ (Fig. 2,*b*), we estimated the relative contribution of each annealing mechanism depending on the irradiation intensity with the use of the values of the annealing constants (Figs. 4 and 6). The result of the estimate is given in Fig. 7. One can see that, at $J > 0.6$ μA/cm^2, $\frac{t_{\rm off}/t_p}{\tau_2} > \frac{1}{\tau_1}$, whereas, at the intensities $J \le 0.2$ μA/cm^2, the effect of irradiation on the VO annealing is inessential, and one can consider that

$$[{\rm VO}]_{\max} = \frac{\tau_2}{t_{\rm off}/t_p}\lambda_{\rm V}. \qquad (13)$$

In (13), $\frac{\tau_2}{t_{\rm off}/t_p} = const$. Therefore, the saturated VO concentration depends on the irradiation intensity to the same extent as the free-vacancy generation factor $\lambda_{\rm V}$. As one can see from Fig. 5, $\lambda_{\rm V}$ grows linearly with J, that is why the experimental dependence $[{\rm VO}]_{\max}(J)$ at $J \le 0.2$ μA/cm^2 also changes in the same way (see Fig. 2,*b*).

At $J > 0.6$ μA/cm^2 (see Fig. 7), the contribution of the accelerated VO annealing becomes determinative $(\frac{1}{\tau_1} > \frac{t_{\rm off}/t_p}{\tau_2})$, and expression (12) yields

$$[{\rm VO}]_{\max} = \lambda_{\rm V}\tau_1. \qquad (14)$$

In this case, the saturated VO concentration is determined by the product of the free-vacancy generation rate $\lambda_{\rm V}$ and the constant of the VO annealing during irradiation τ_1. The both parameters depend on the irradiation intensity, though in different ways: $\lambda_{\rm V}$ increases (Fig. 5), and τ_1 decreases (Fig. 6). Due to this fact, the total rate of growth of $[{\rm VO}]_{\max}(J)$ decelerates, which is illustrated by Fig. 2,*b*.

4. Conclusions

The effect of the intensity of pulsed electron irradiation on the kinetics of VO accumulation in Cz *n*-Si at a temperature of 360 °C has been investigated experimentally and analyzed theoretically. It is established that, in the studied range of the irradiation intensity $J_p = 1.25\times10^{15} - 1.25\times10^{16}$ electrons/(cm^{2}s), the following phenomena take place:

a) under high-temperature irradiation resulting in the simultaneous generation and annealing of VO complexes, the kinetics of their accumulation is determined by the annealing. This provides a nonlinear (tending to the saturation) behavior of the dependence of the VO accumulation on the irradiation dose (time);

b) there exists a limiting (maximum) VO concentration determined both by the irradiation intensity and by the temperature of samples under irradiation (time constant of isothermal annealing τ_2)
c) the efficiency of the generation of vacancies $d[\mathrm{V}]/d\Phi$ (and, respectively, $d[\mathrm{VO}]/d\Phi$) does not depend on the irradiation intensity;
d) the intensity of the high-temperature irradiation by 1-MeV electrons can essentially stimulate the annealing of the created VO complexes. During an irradiation pulse, the 10-fold increase of the intensity results in the reduction of the time constant of defect annealing τ_1 by more than two orders of magnitude, which testifies to the effect of the excitation level of the electron subsystem of a crystal on the annealing of defects.

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ВПЛИВ ІНТЕНСИВНОСТІ ЕЛЕКТРОННОГО ОПРОМІНЕННЯ НА УТВОРЕННЯ І ВІДПАЛ VO-ЦЕНТРІВ У КРЕМНІЇ ПРИ ВИСОКИХ ТЕМПЕРАТУРАХ

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Р е з ю м е

Досліджено кінетику накопичення комплексу вакансія–кисень (VO) у n-Si, вирощеного методом Чохральського (Cz), при різних інтенсивностях імпульсного 1 МеВ електронного опромінення при 360 °C. Показано, щоп при опроміненні, коли одночасно іде утворення та відпал VO, кінетика їх накопичення є нелінійною і має вигляд кривої з насиченням внаслідок наявності саме відпалу. Виявлено, що існує гранична (максимальна) концентрація VO, яка визначається як інтенсивністю опромінення, так і температурою зразків при опроміненні. Виявлено також, що інтенсивність опромінення кремнію 1 МеВ електронами при високих температурах може суттєво стимулювати відпал ними ж створених комплексів VO. При 360 °C зміна інтенсивності потоку опромінення в імпульсі від $1{,}25{\cdot}10^{15}$ до $1{,}25{\cdot}10^{16}$ електрон/(см^{2}с) помітно не впливає на ефективність утворення VO в n-Si, але приблизно на два порядки прискорює їх відпал.