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Kinetics of divacancy annealing and divacancy-oxygen formation in oxygen-enriched high-purity silicon

M. Mikelsen,[1] E. V. Monakhov,[1] G. Alfieri,[1] B. S. Avset,[2] and B. G. Svensson[1]
[1]*Department of Physics, Physical Electronics, University of Oslo, P.O. Box 1048, Blindern, N-0316 Oslo, Norway*
[2]*SINTEF ICT, P.O. Box 124 Blindern, N-0314 Oslo, Norway*

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In this work the thermal kinetics of the transformation from the divacancy (V_2) to the divacancy-oxygen (V_2O) complex has been studied in detail, and activation energies, (E_a), have been obtained. Diffusion oxygenated float-zone silicon (DOFZ-Si) samples of *n*-type with a doping of 5×10^{12} cm^{-3} and oxygen content of $(2-3)\times10^{17}$ cm^{-3} have been irradiated with 15 MeV electrons. Isothermal annealing studies of electrically active defects have been performed by means of deep-level transient spectroscopy. Heat treatments at temperatures in the range 205 °C–285 °C have all shown a shift in the singly negative and doubly negative divacancy levels, due to the annealing of V_2 and the formation of V_2O. By studying the temperature-dependent rate of this process which exhibits first order kinetics, it has been found that both the annealing V_2 and the formation of V_2O have activation energies of $\approx$1.3 eV. This value is ascribed to migration of V_2, and the results favor strongly a model where V_2 is trapped by interstitial oxygen atoms during migration. In addition, the process takes place with a high efficiency since the loss of V_2 and the growth of V_2O display a close one-to-one proportionality. Finally, it has been found that the diffusivity pre-exponential factor, $D^0_{V_2}$, for V_2 is in the range $3\pm1.5\times10^{-3}$ cm^2/s, which agrees well with a simple theoretical model of V_2 diffusion in Si.

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I. INTRODUCTION

The divacancy (V_2) is one of the most fundamental point defects in Si, and it is known to occur at pronounced concentrations after irradiation with high energetic particles (electrons, neutrons, ions, etc.). V_2 can appear in four different charge states, positive (+), neutral (0), negative (−), and doubly negative (=).[1] Deep level transient spectroscopy (DLTS) measurements have established a (0/+) donor level at $\sim E_v+0.20$ eV, a (−/0) acceptor level at $\sim E_c-0.42$ eV, and a (=/−) acceptor level at $\sim E_c-0.23$ eV, where E_v and E_c are the energies of the edges of the valence and conduction band, respectively.[2–5]

V_2 is known to be immobile at temperatures up to 200 °C. In an early electron paramagnetic resonance (EPR) study by Watkins and Corbett it was found that beyond this temperature V_2 can migrate with a thermal activation energy (E_a) of $\approx$1.3 eV.[1] The study suggested that in Czochralski (Cz) Si the divacancies would anneal by trapping at other impurities during migration. In float-zone (FZ) Si the divacancy showed a higher stability under heat treatment, and dissociation was suggested to be the main annealing mechanism. Based on this suggestion the thermal activation energy for dissociation was estimated to be $\sim$1.9 eV, implying that the divacancy will diffuse large distances prior to dissociation. Interstitial oxygen atoms (O_i) are thought to be effective traps for V_2, and the divacancy-oxygen interaction has for a long time been suggested to be the main mechanism of the V_2 elimination upon annealing of Cz grown Si irradiated with high doses.[6,7]

The divacancy-oxygen, (V_2O), complex was first discovered in an EPR study on heavily electron irradiated Cz-Si by Lee and Corbett in 1975.[7] The defect was present at pronounced concentrations after high electron doses, and an EPR Si-A14 signal and an absorption line at 833.5 cm^{-1} have been assigned to it. Later, in a positron annihilation study, the annealing behavior of V_2 was investigated, and an increase in V_2O concentration similar to the decrease in V_2 concentration was observed.[8] However, the electrical properties of V_2O still remained relatively unknown. Theoretical studies on the structure and electrical states of V_2O have predicted that the defect has one donor level and two acceptor levels.[9,10] In a DLTS study on electron irradiated *p*-type Cz- and Fz-Si a shift in position of the $V_2(0/+)$ state to a new 0.24 eV level was observed.[11] The shift was faster in the oxygen-rich Cz-Si, and it was proposed that the shift was due to a transformation from V_2 to V_2O, with the 0.24 eV level being a (0/+) charge state transition of V_2O.

Recently, the interest in the V_2O complex has increased substantially following speculations that this defect may produce a deep level close to mid-gap that is responsible for degradation of Si-based radiation detectors.[12] In a DLTS study on irradiation induced defects in FZ-Si by Pintilie *et al.*, a level close to the middle of the band gap ($E_c-0.55$ eV) was observed and attributed to the $V_2O(-/0)$ state.[13] A correlation between the formation rate of the new center and the oxygen content was deduced. The level also showed a second order generation rate as a function of irradiation dose at room temperature, in accordance with that expected for V_2O.[14] However, no doubly negative charge state was observed in contradiction to theoretical predictions,[9,10] and the concentration of this level was only a small fraction of that of V_2.

Recent reports by Monakhov *et al.*[15] have attributed two other levels to V_2O. In a DLTS study on irradiation induced acceptor-type defects in oxygen-enriched high purity FZ-Si, a gradual shift in the positions of the $V_2(-/0)$ and $V_2(=/-)$ peaks was observed during heat treatments at

250 °C–300 °C. This shift was ascribed to the formation of a new center, labeled X, as V_2 annealed. Capture cross-section measurements supported the identification of X as a double acceptor center. The energy positions of $X(-/0)$ and $X(=/-)$ ($E_c-0.46$ eV and $E_c-0.20$ eV, respectively) are close to $V_2(-/0)$ and $V_2(=/-)$ suggesting that the electronic structure of X is similar to that of V_2, and X has been tentatively identified as V_2O. Further support for this identification was put forward in a subsequent study of irradiation induced divacancies in FZ-Si with varying oxygen content.[16] It was observed that the shift in level position occurs faster in the materials with higher oxygen content, and that the formation rate of X and annealing rate of V_2 were proportional to the oxygen content within experimental accuracy. A Laplace-DLTS study has also shown that the formation of X and the annealing of V_2 have a close one-to-one correlation.[17] In a study by Markevich *et al.*[18] this transformation was observed also in Cz-Si. These observations provide strong evidence for the identification of X as V_2O.

In a DLTS study on ion implantation induced defects in regular Cz- and FZ-Si, no shift in the V_2 position or occurrence of X were found during annealing.[19] The activation energy for V_2 annealing was ~1.3 eV in both materials. The annealing rates for V_2 in the two materials differed only by a factor 2, while the difference in O_i concentration was about two orders of magnitude. It was concluded that, in that study, O_i was not a main trap for V_2 during annealing. In this context, it should be noted that DLTS is applicable to investigations of low concentrations of defects (only a few percent of the doping concentration). In contrast, techniques such as EPR and infrared absorption are used for much higher defect concentrations, in the range of $10^{16}-10^{17}$ cm^{-3}. In the latter case, the annealing of V_2 can exhaust impurities of low concentration which affect or even dominate the annealing mechanism in DLTS studies.

A recent DLTS study on hydrogenated Si concludes that hydrogen dimers, (H_2), may play an important role in the V_2 annealing.[20] It was suggested that the H_2 may migrate and interact with V_2 at relatively low temperatures through the reaction $V_2+H_2 \rightarrow V_2H_2$, thus suppressing V_2O formation. It cannot be excluded that H_2 may play a role in the annealing of V_2 also in nonhydrogenated Si, due to residual contamination of Si with H_2. Indeed, the annealing behavior of V_2 in Ref. 19 is similar to that observed in hydrogenated Si.

In this work we have performed a detailed DLTS study of V_2 and the transformation to $V_2O(X)$ in diffusion oxygenated float-zone (DOFZ) Si samples that have been irradiated with 15 MeV electrons. Isothermal annealing has been carried out at five different temperatures in the range 205 °C–285 °C, and by extracting the temperature-dependent rate of the annealing of V_2 and the formation of V_2O, the activation energy and the frequency factor for the processes has been deduced. The results favor a model where V_2 diffuses followed by subsequent trapping by interstitial oxygen atoms. The diffusivity pre-exponential factor for V_2 has been derived and compared with a simple theoretical model of V_2 diffusion in Si.

II. EXPERIMENTAL DETAILS

P^+-n^--n^+ diodes were made using high resistivity and high purity diffusion-oxygenated float-zone (DOFZ) Si wafers. In the processing, an oxide layer was grown on the FZ-wafer surface in a dry atmosphere at 1200 °C for 21 hours. A so-called oxygenation process was then performed, where O atoms from the preformed SiO_2 surface-layer diffuse into the bulk Si during annealing at 1200 °C in a N_2 atmosphere. An ordinary silicon diode process with boron and phosphorus implantation and post-annealing was then performed and aluminum was used as metal contact.

Oxygen and carbon concentrations in the samples were measured by secondary ion mass spectrometry (SIMS) and were $(2-3)\times 10^{17}$ and $(2-4)\times 10^{16}$ cm^{-3}, respectively. Capacitance-voltage measurements determined the doping concentration in the n^- layer to 5.0×10^{12} cm^{-3}.

The diodes were irradiated at room temperature with 15 MeV electrons to a dose of 6×10^{12} cm^{-2}. Before further analysis the samples were preannealed for 15 min at 225 °C in order to remove minor, unstable, defects which are not the focus in this study.[15] Isothermal annealing treatment was then performed on five samples at different temperatures (205 °C, 225 °C, 245 °C, 265 °C, 285 °C). DLTS measurements were undertaken before and after each annealing step.

The DLTS measurements were conducted using a setup described elsewhere.[5] In brief, the temperature of the sample was scanned between 77 K and 280 K, and the measured capacitance transients were averaged in intervals of a width of 1 K. The DLTS signal was extracted by using a lock-in type of weighting function, and six different spectra were obtained with rate windows in the range of $(20\text{ ms})^{-1}$ to $(640\text{ ms})^{-1}$ from a single temperature scan. Concentration, energy level position and apparent capture cross section of the traps were then estimated from the spectra.

III. RESULTS

The DLTS spectra reveal the presence of three major peaks after the preannealing at 225 °C. As reported in Ref. 15 and references therein, these peaks have been identified as the vacancy-oxygen pair VO, the singly negative divacancy $V_2(-/0)$ and the doubly negative divacancy $V_2(=/-)$.

During the isothermal annealing, a gradual shift in the position of the V_2 peaks is observed, Fig. 1. A similar behavior has also been reported in previous studies,[15,16] and the two so-called X levels occur at $\sim E_c-0.20$ and $\sim E_c-0.46$ eV, respectively. We also observe the formation of another level with an activation enthalpy of ~0.36 eV and an apparent capture cross section of $\sim 2\times 10^{-14}$ cm^2. The formation rate of this level exhibits stronger temperature dependence than that of X and it is discussed in detail elsewhere.[21]

The individual amplitudes for V_2 and X were extracted by fitting the DLTS spectra, using the values of the activation energies and capture cross sections given in Ref. 15. The amplitudes of V_2 and X and the sum of V_2 and X are plotted versus annealing time at 285 °C for both the doubly and singly charged states in Fig. 2(a) and 2(b), respectively. The sum of $V_2(=/-)$ and $X(=/-)$ remains almost constant, while that of $V_2(-/0)$ and $X(-/0)$ increases slightly with time.

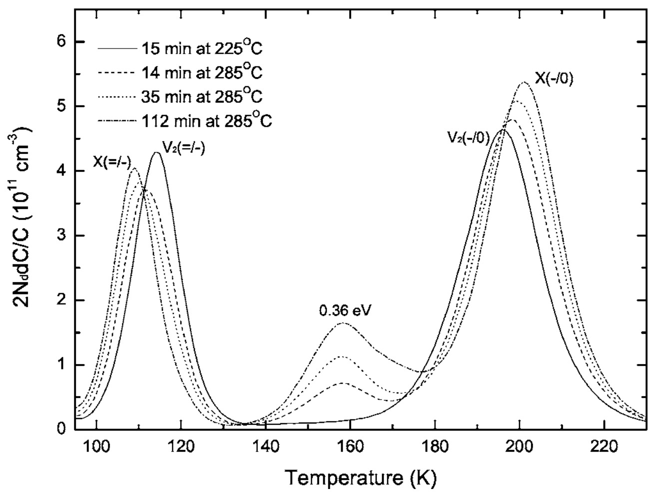

FIG. 1. DLTS spectra of an irradiated sample after isothermal annealing at 285 °C for different durations.

In Fig. 3 the growth of X is plotted versus the loss of V_2 for all the temperatures and times investigated. We observe that for the doubly negative charge states there is proportionality close to unity, indicating an almost exact one-to-one correlation between the annealing and formation of the two levels. For the singly negative charge states the data points fall slightly above the line; under the assumption of one-to-one transition from V_2 to X, as shown by the data for $V_2(=/-)$ and $X(=/-)$, this can be attributed to the formation of a minor level with a similar position as $X(-/0)$. The evolution of such an overlapping level with annealing time at 285 °C is depicted in Fig. 2(b).

In Fig. 4, the $V_2(=/-)$ intensity versus annealing time, t, is plotted for all the temperatures studied, and an exponential decrease holds,

$$[V_2](t) = [V_2](t=0)\exp[-c(T)t], \tag{1}$$

where $[V_2]$ is the concentration of V_2 and $c(T)$ is a temperature dependent rate constant. In Fig. 5, the relative difference between $[V_2(=/-)](t=0)$ and $[X(=/-)](t)$ versus annealing time is shown for all the annealing temperatures investigated. The evolution of $[X]$ follows the relation

$$[X](t) = [V_2](t=0) - [V_2](t=0)\exp[-c(T)t] \tag{2}$$

with a good accuracy, and also in accordance with Fig. 3.

As shown in Fig. 6, both for the annealing of V_2 and the growth of X, $c(T)$ exhibits an Arrhenius behavior,

$$c(T) = c_0 \exp(-E_a/kT), \tag{3}$$

where E_a is the activation energy and c_0 is a prefactor. For the levels $V_2(=/-)$, $V_2(-/0)$, and $X(=/-)$ the rates are accurately described by $E_a = 1.30 \pm 0.02$ eV and $c_0 = 4.4 \pm 1.9 \times 10^8$ s^{-1}. For the $X(-/0)$ peak slightly higher rates are obtained because of the contribution from the overlapping level discussed in Fig. 2. The values of E_a and c_0 extracted for the individual levels are given in Table I.

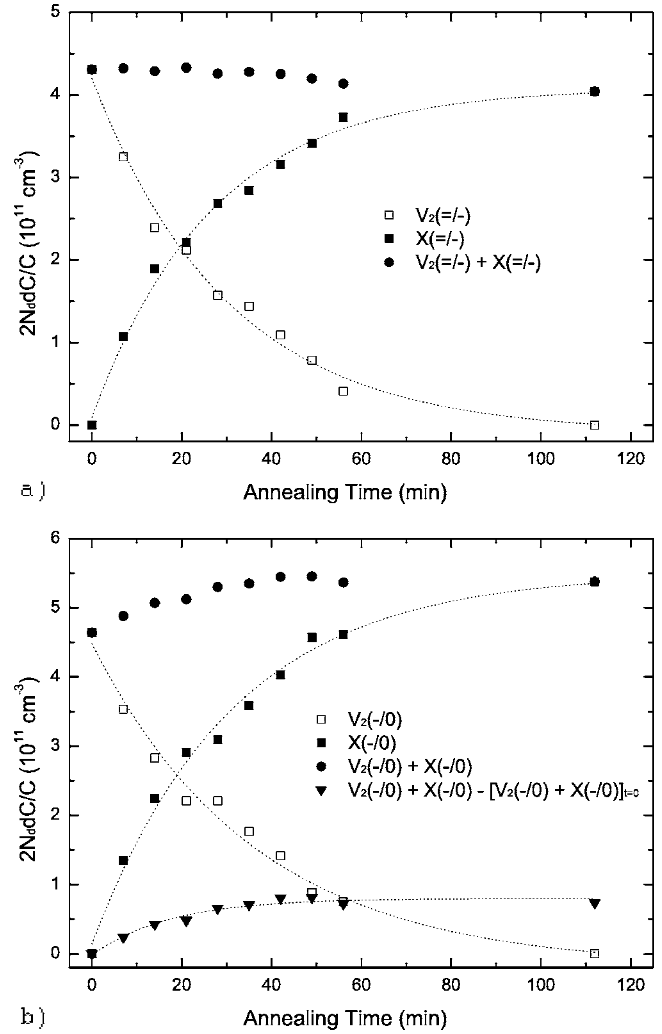

FIG. 2. (a) Intensities of the doubly negative charge states of V_2 and X and their sum versus annealing time during isothermal annealing at 285 °C. The dotted lines represent first order exponential decay fits. (b) Intensities of the singly negative charge states of V_2 and X and their sum versus annealing time during isothermal annealing at 285 °C. Also plotted is the increase in amplitude of the $[V_2(-/0)+X(-/0)]$ peak as a function of time. The dotted lines represent first order exponential decay fits.

IV. DISCUSSION

A. Identification of *X*

The annealing of V_2 and the formation of X exhibit first-order kinetics with a close to one-to-one proportionality between the two processes, except for a small ($\sim 15\%$) deviation for $X(-/0)$ attributed to a minor interfering level, Figs. 2(b) and 3. The prefactor, c_0, for the transformation from V_2 to X is in the 10^8-10^9 s^{-1} range, which is substantially lower than that expected for a pure dissociation mechanism ($10^{12}-10^{13}$ s^{-1}),[22] and it favors a process involving long-distance defect migration with subsequent trapping. Indeed, the activation energy, E_a, deduced for the transformation (~ 1.30 eV) is identical with that anticipated for migration of V_2.[1]

In previous DLTS studies where X has been observed as V_2 anneals, the two X levels have tentatively been assigned

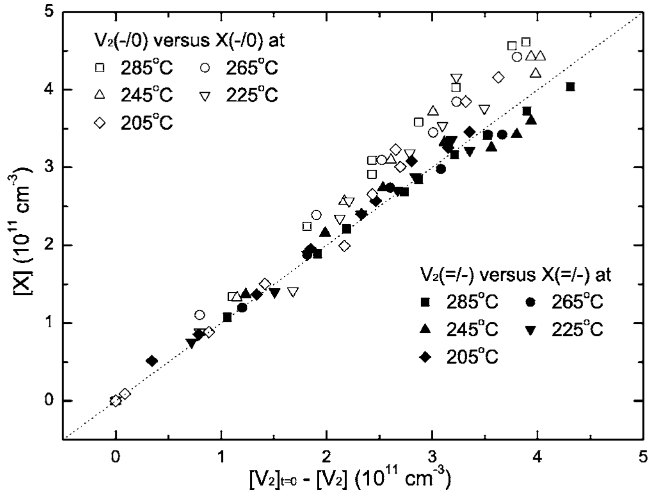

FIG. 3. Growth in X amplitude versus loss in V_2 amplitude. The dotted line represents an exact one-to-one proportionality between X growth and V_2 loss.

to the singly and doubly negative charge states of V_2O.[15–18] It has been suggested that interstitial oxygen acts as a trap for the migrating divacancy and that V_2O is formed by the reaction $V_2+O_i \rightarrow V_2O$. The findings in this study strongly support a single diffusion driven reaction, where X is formed as a product between V_2 and an immobile impurity with substantially higher concentration than V_2. Together with the previous results, in particular the fact that the reaction rate is proportional to the oxygen content,[16] there is now solid evidence for the assignment of X to V_2O.

Annealing studies of V_2 have previously been performed on Cz-Si samples with a high oxygen content ($\sim 5 \times 10^{17}$ cm^{-3}) using DLTS,[19] but no formation of X was observed. On the other hand, in a recent study by Markevich *et al.*,[18] where mechanically polished Cz-Si samples were used, formation of X was detected. The activation energies for V_2 annealing in regular FZ- and Cz-Si obtained by Pellegrino *et al.*[19] are close to those found in this study (Table I). However, as shown in Fig. 6 the rates from Ref. 19 are consistently above the ones deduced from the present study by a factor of ~ 5, which suggests a competing process with a slightly higher rate. This opens for at least two possibilities. First, there is an unknown impurity, with a concentration higher than any of the known impurities, which traps the migrating V_2 species. However, this implies a concentration in the 10^{18} cm^{-3} range and it appears unlikely. Second and more likely, one can suggest an interaction with another migrating species with a mobility higher than that of V_2, yielding a faster annealing. Since the annealing behavior of V_2 in Ref. 20 is similar to that in Ref. 19, the H_2 molecule appears as a likely candidate. On the other hand, the activation energy for migration of H_2 molecules is estimated to be

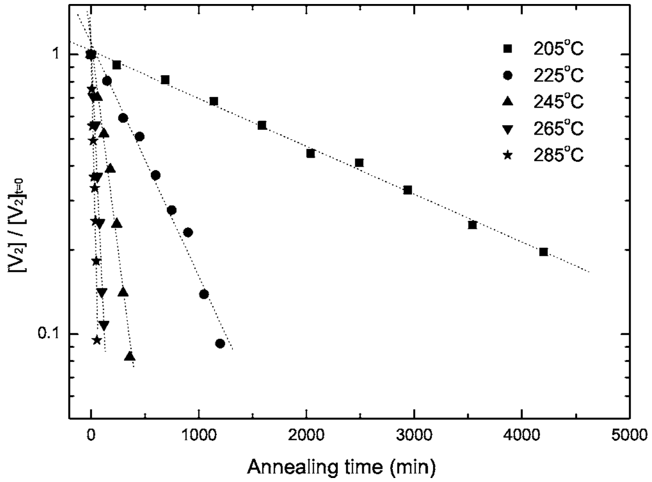

FIG. 4. Intensity of $V_2(=/-)$ during isothermal annealing in the temperature range 205 °C–285 °C as a function of time. The intensity has been normalized to that in as-irradiated samples. The dotted lines represent least-squares fits of the experimental data.

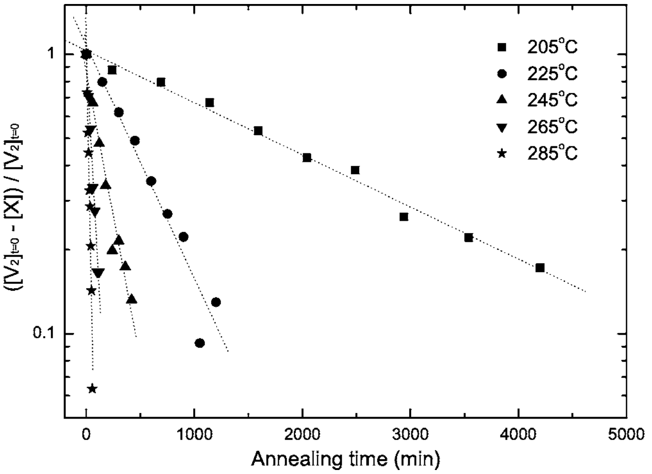

FIG. 5. Difference between the intensity of $V_2(=/-)$ in the as-irradiated samples and that of $X(=/-)$ versus time during isothermal annealing in the temperature range 205 °C–285 °C. The intensity has been normalized to that of $V_2(=/-)$ in as-irradiated samples. The dotted lines represent least-squares fits of the experimental data.

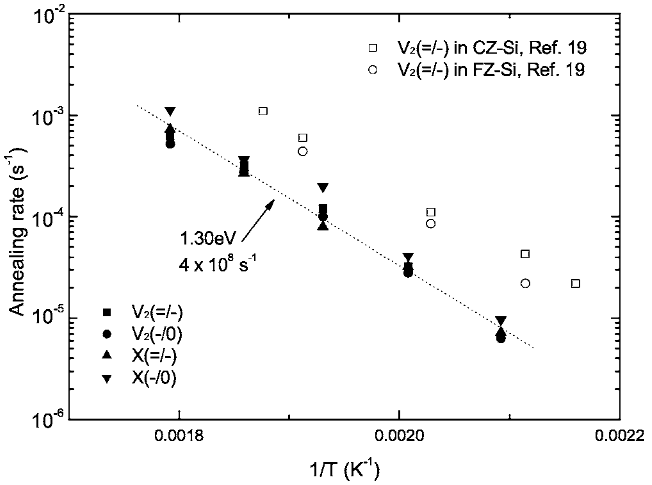

FIG. 6. Arrhenius plot for the annealing rates and formation rates of V_2 and X, respectively. Included are also data for annealing of V_2 in regular CZ- and FZ-Si taken from Ref. 19. The dotted line is a fit to the rates for $V_2(=/-)$, $V_2(-/0)$, and $X(=/-)$.

TABLE I. Frequency factor, c_0, and the activation energy, E_a, for both charge states of V_2 and V_2O. It is believed that the values for the doubly charged states, $V_2(=/-)$ and $V_2O(=/-)$, are the most reliable ones. Values for $V_2(=/-)$ in normal FZ- and Cz-Si from Ref. 19 are also included for comparison.

	c_0 (s^{-1})	E_a (eV)
$V_2(-/0)$	2.5×10^8	1.28
$V_2(=/-)$	6.2×10^8	1.32
$X(-/0)$	1.9×10^9	1.35
$X(=/-)$	4.4×10^8	1.31
$V_2(=/-)$ in Cz-Si from Ref. 19	1×10^9	1.27
$V_2(=/-)$ in FZ-Si from Ref. 19	5.4×10^8	1.25

only ~0.8 eV,[23] i.e., substantially lower than for the V_2 annealing in Ref. 19. However, at moderate temperatures up to 300 °C hydrogen is known to be trapped at different complexes, and if the reaction between V_2 and H_2 is limited by trap release, and not by migration of H_2, an activation energy above 0.8 eV would be expected. Several studies have reported activation energies for trap-limited diffusion of hydrogen, and indeed the results are in the region 1.1–1.5 eV.[23–26]

B. Migration of V_2

The results for the V_2 annealing obtained in this study can be used to give a quantitative estimate of the diffusivity of V_2, D_{V_2}. Applying the theory of diffusion-limited reactions the following relation holds:[27]

$$\frac{d[V_2(t)]}{dt}=-c(T)[V_2(t)]=-4\pi R D_{V_2}[O_i][V_2(t)], \quad (4)$$

where R is the capture radius for the trapping reaction and $[O_i]$ is the interstitial oxygen concentration. Combining Eq. (4) with $D_{V_2}=D^0_{V_2}\exp[-E_a/kT]$ one gets

$$D^0_{V_2}=\frac{c_0}{4\pi R[O_i]}. \quad (5)$$

Setting R equal to 5 Å, $[O_i]$ to 2.5×10^{17} cm^{-3}, and using $c_0=4.4\times10^8$ s^{-1} one obtains $D^0_{V_2}\approx3\times10^{-3}$ cm^2/s. Taking into account the variation between the $V_2(=/-)$ and V_2O $(=/-)$ data, plus other experimental uncertainties, $D^0_{V_2}$ is estimated to be in the range $3\pm1.5\times10^{-3}$ cm^2/s.

For a divacancy in Si, an expression for diffusivity in a given direction, labeled x, can be written as

$$D_x=\frac{1}{2}\sum_{1}^{\xi=6}\Gamma_s x_s^2, \quad (6)$$

where ξ is the coordination number, which equals the number of jumping-sites available, Γ_s is the jump frequency to a site labeled S and x_s is the x projection of the jump length to the S site. For two of the six possible sites $x_s=0$, while for the other four, $x_s=\frac{1}{4}a$, where a is the lattice constant. Assuming that Γ_s is equal for all the possible jump sites (isotropic process), Eq. (6) yields

$$D_{V_2}=\frac{1}{8}\Gamma_s a^2=\frac{1}{8}a^2\nu\exp\left(\frac{\Delta S^m}{k}\right)\exp\left(\frac{-E_a}{kT}\right) \quad (7)$$

and

$$D^0_{V_2}=\frac{1}{8}a^2\nu\exp\left(\frac{\Delta S^m}{k}\right). \quad (8)$$

In silicon, the Debye frequency, ν, is 1.3×10^{13} Hz (Ref. 28) and the lattice constant is 5.4 Å. The entropy factor $\exp(\Delta S^m/k_B)$ reduces to W_1/W_2 where W_1 and W_2 are multiplicity of states before and after a diffusion step. In first approximation, the migration is regarded as a simple process; thus W does not change significantly and $\exp(\Delta S^m/k_B)\approx1$. As a result, the theoretical estimate of $D^0_{V_2}$ becomes 4×10^{-3} cm^2/s, in excellent agreement with the experimental value.

V. SUMMARY AND CONCLUSIONS

Isothermal annealing studies of V_2 in electron irradiated DOFZ-Si samples have been performed at five different temperatures ranging from 205 °C to 285 °C. The process exhibits first order kinetics with a corresponding growth of the X center. The transformation from V_2 to X displays a close one-to-one proportionality and the rate is thermally activated with an energy of 1.30 ± 0.02 eV. These results provide firm evidence in favor of the previously proposed assignment of X to V_2O and a process where migrating V_2's are trapped by (immobile) interstitial oxygen atoms resulting in the formation of V_2O pairs. Applying the theory of diffusion-limited reactions for this process, the experimental data yield a diffusivity of V_2 given by $(3\pm1.5)\times10^{-3}\times\exp[-(1.30\pm0.02)\text{ eV}/kT]$ cm^2/s. Furthermore, using a simple model for the diffusion of V_2, a theoretical value of 4×10^{-3} cm^2/s is obtained for the pre-exponential factor, in excellent agreement with the experimental value.

ACKNOWLEDGMENTS

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