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Formation kinetics of trivacancy-oxygen pairs in silicon

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The formation of donor and acceptor states of the trivacancy-oxygen (V_3O) complex in p- and n-type silicon, respectively, has been studied in detail by means of deep level transient spectroscopy. The samples were irradiated with 1.8 MeV protons, and it was found that acceptor and donor states of V_3 disappear at annealing temperatures below 200 °C, with a transition from the metastable (110) planar configuration to the stable fourfold coordinated configuration. Annealing above 200 °C unveils the formation of two levels with energy positions at $E_c - 0.34$ eV and $E_c - 0.46$ eV in n-type samples and two levels at $E_v + 0.24$ eV and $E_v + 0.11$ eV in p-type samples. (E_c and E_v denote the conduction and valence band edge, respectively.) The amplitudes of these levels show an almost one-to-one correlation with those of the V_3 -related levels occurring after irradiation. In accordance with recent reports in the literature, the emerging levels are ascribed to the V_3O complex; single and double negative charge states in n-type samples and single and double positive charge states in p-type samples. By undertaking isothermal formation of the V_3O complex in p-type samples which exhibits first-order kinetics, the diffusivity of V_3 in the neutral charge state is determined to be $(8.5 \pm 3.5) \times 10^{-2} \exp [-(1.50 \pm 0.04) \text{ eV/kT}] \text{ cm}^2/\text{s}$. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4896066>]

I. INTRODUCTION

Vacancy related defects in silicon influence the electrical and optical properties and can serve as efficient recombination centers reducing the minority charge carrier lifetime. Thus, understanding of the properties of these defects is important for Si-based devices, like, for example, solar cells. Among the multi-vacancy complexes (V_n), only the divacancy (V_2) has been thoroughly studied using several techniques,^{1–4} while V_n with $n > 2$ have been studied to a much lesser extent.⁵

Recently, there has been a growing interest towards the trivacancy (V_3) and related complexes.^{6,7} In an early electron paramagnetic resonance (EPR) study on neutron irradiated samples, a spectrum labeled A4 was assigned to V_3 in a (110) planar configuration.⁸ It was shown that A4 disappeared upon isochronal annealing in the temperature range of 250–300 °C, i.e., in the range similar to V_2 .⁹ In a Deep level transient spectroscopy (DLTS) study by Ahmed *et al.*,⁵ levels with energy positions of $\sim E_c - 0.46$ eV and $\sim E_c - 0.36$ eV were tentatively assigned to single and double acceptor states of the V_3 center, where E_c denotes the conduction band edge. However, it was found that these levels disappeared in the temperature range of 50–100 °C, which contradicted the anticipated stability of the V_3 center. Recently, in a combined DLTS and *ab-initio* study, Markevich *et al.*⁶ found that V_3 is a bi-stable defect with two configurations: fourfold coordinated (FFC) and metastable (110) planar.^{6,7} V_3 in the (110) planar configuration occurs in the charge states with energy levels at $\sim E_c - 0.36$ eV, $\sim E_c - 0.46$ eV, $\sim E_v + 0.19$ eV, and $\sim E_v + 0.105$ eV, where E_v denotes the valence band edge. These levels are attributed

to doubly negative ($=/-$), negative ($-/0$), positive ($+/0$), and doubly positive ($2+/+$) transitions of V_3 , respectively. The FFC structure is, however, the lowest in energy and has the only acceptor state at $E_c - 0.075$ eV. In n-type samples, it has been observed that the transformation from single and double acceptor states of V_3 in the planar configuration to the stable FCC structure occurs in the temperature range of 50–120 °C,⁶ consistent with the results from the previous studies of these two energy levels,^{5,10} where Bleka *et al.*¹⁰ also demonstrated a close one-to-one proportionality between their amplitudes.

The concentration of V_3 is strongly enhanced after high energy particle irradiation and/or ion implantation. In n-type samples, it was found that V_3 starts to migrate above 200 °C with an activation energy of 1.47 ± 0.04 eV.¹¹ The study suggested that V_3 would anneal slower in epitaxial samples compared to Czochralski (Cz) ones due to the lower impurity content. One of the most abundant impurities in Cz-Si is interstitial oxygen (O_i), and trivacancy-oxygen interaction has been suggested to be the main mechanism of V_3 elimination upon the annealing.^{6,11}

It was reported in Ref. 6 that isochronal annealing of electron irradiated n-type material in the temperature range of 200–300 °C resulted in the disappearance of the V_3 related levels and concurrent formation of two new levels at $E_c - 0.34$ eV and $E_c - 0.455$ eV with an amplitude similar to those of V_3 . The new levels were assigned to doubly negative and singly negative states of the trivacancy-oxygen (V_3O) centers in (110) planar configuration. In p-type material, however, a detailed qualitative study of the transformation kinetics of V_3 to V_3O has not been undertaken. Markevich *et al.*⁷ observed a shift in the position of the $V_2(+/0)$ peak during annealing, and it was discussed that the emerging level is a combination of $V_2O(+/0)$ and $V_3O(+/0)$. In the

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same study, a level at $E_v + 0.12$ eV was observed upon the $V_3(2+/+)$ annealing and tentatively identified as $V_3O(2+/+)$. In Ref. 12, two levels at $E_v + 0.24$ eV and $E_v + 0.11$ eV levels were resolved after annealing of proton-irradiated p-type Cz samples, supporting their assignment as the single and double donor states of V_3O , respectively, made in Ref. 7.

In the present work, we report on the formation kinetics of V_3O in p- and n-type Si irradiated with 1.8 MeV protons. The kinetics is of first order and by extracting the temperature-dependent rate of the formation of V_3O , the activation energy and the frequency factor of the processes are found to be 1.50 ± 0.04 eV and $2.1 \pm 0.7 \times 10^{10} \text{ s}^{-1}$, respectively. The former is attributed to the migration energy of V_3 and applying the theory for diffusion-limited reactions; the latter yields a pre-exponential factor of $\sim 8 \times 10^{-2} \text{ cm}^2/\text{s}$ for the V_3 diffusivity.

II. EXPERIMENTAL DETAILS

$p^+n^-n^+$ and n^+p diodes were fabricated on phosphorous and boron doped wafers, respectively. The $p^+n^-n^+$ samples were prepared from a silicon wafer having a highly phosphorous-doped (n^+) substrate with a lightly n-doped (n^-) epitaxial layer grown on the front surface. These epi-wafers were subsequently processed into pads of $p^+n^-n^+$ diodes. The p^+ -layer was realized by ion implantation of boron with post-implant annealing. The thickness of the epitaxial n-layer was $\sim 20 \mu\text{m}$ and the doping concentration, N_d , in the layer was about $\sim 3 \times 10^{13} \text{ cm}^{-3}$. The n^+p diodes were prepared using Cz wafers with a boron concentration of $\sim 1 \times 10^{15} \text{ cm}^{-3}$. First, the wafers were dry oxidized at 1100°C for 3 h to grow a 250 nm thick SiO_2 layer. The n^+ layer was formed by in-diffusion of phosphorous (P) from gas phase in a quartz tube. Aluminum (Al) Ohmic contacts were prepared by thermal evaporation on the front side (n^+ or p^+ side) and silver paste was used as contact on the back-side for both sets of samples. The oxygen [O] and carbon [C] concentration in the epi-layer of the $p^+n^-n^+$ samples were $\sim (2-3) \times 10^{17}$ and $\leq 2 \times 10^{16} \text{ cm}^{-3}$, respectively, while in the n^+p samples they were $\sim 4 \times 10^{17}$ and $\leq 2 \times 10^{16} \text{ cm}^{-3}$, respectively, as determined by secondary ion mass spectrometry (SIMS).

Irradiation of the diodes was performed at room temperature (RT) with 1.8 MeV protons to a dose of $(2-5) \times 10^{12} \text{ cm}^{-2}$ and $3 \times 10^{10} \text{ cm}^{-2}$ for the n^+p and $p^+n^-n^+$ type samples, respectively. The projected range of the protons was $\sim 40 \mu\text{m}$, as estimated by simulations using the TRIM code,¹³ and far beyond the extension of the region probed in the DLTS measurements ($\sim 1-10 \mu\text{m}$). Isochronal annealing (30 min duration) was carried out for temperatures in the range of $200-300^\circ\text{C}$, while isothermal annealing was carried out at five different temperatures (200°C , 225°C , 250°C , 275°C , and 300°C). During the annealing, out-diffusion of H from the region around the projected range may be anticipated, but no indications of any H-related levels in the DLTS spectra were found.

The DLTS measurements were carried out by employing a refined version of the set-up described in Ref. 14. The reverse bias quiescent voltage was kept at 10 V and during

the filling pulse (50 ms duration) the bias was 0 V. The temperature was scanned between 40 and 300 K, and eight DLTS spectra with rate windows from 5 ms^{-1} to 640 ms^{-1} were extracted applying both a lock-in and a high resolution weighting function (GS4).¹⁵

III. RESULTS AND DISCUSSION

Figure 1 shows DLTS spectra of an as-irradiated $p^+n^-n^+$ sample, and after annealing at 200°C and 300°C . In the as-irradiated sample, three major peaks with energy level positions at $E_c - 0.44$ eV, $E_c - 0.23$ eV, and $E_c - 0.18$ eV are observed and identified as the single acceptor state of $V_2(-/0)$, double acceptor state of $V_2(=/-)$, and the acceptor state of the vacancy-oxygen (VO) pair. In addition, the as-irradiated sample reveals a level at $E_c - 0.36$ eV, labeled as $V_3(=/-)$ in accordance with Ref. 6.

The amplitude of the DLTS peak around $V_2(-/0)$ is larger than that of $V_2(=/-)$ which indicates the presence of other levels closely overlapping with $V_2(-/0)$. One potential candidate is the acceptor state of the vacancy-phosphorus (VP) pair. However, VP is expected to be very minute in the present samples due to the low concentration of P and high concentration of O with a difference by four orders of magnitude; O and P are the main and competing traps for migrating mono-vacancies and in the following VP is neglected.¹⁶ However, as previously reported,^{6,10} $V_3(=/-)$ and $V_3(-/0)$ are expected to have energy level positions around $E_c - 0.36$ eV and $E_c - 0.45$ eV, respectively, i.e., $V_3(-/0)$ will overlap with $V_2(-/0)$. Indeed, $V_3(=/-)$ (arising from the (110) planar configuration) disappears after the heat-treatment at 200°C for 20 min, and at the same time, a decrease in the $V_2(-/0)$ peak occurs although V_2 remains stable as illustrated by the amplitude of the $V_2(=/-)$ peak. Moreover, the reduced concentration of the $V_2(-/0)$ peak is close to the concentration of $V_3(=/-)$ in the as-irradiated sample. Hence, the deviation between $V_2(-/0)$ and $V_2(=/-)$ is mainly ascribed to the contribution of $V_3(-/0)$. Simultaneously, a new level emerges at $E_c - 0.075$ eV with the disappearance of $V_3(=/-)$ and $V_3(-/0)$, and its amplitude is almost equal to that of the V_3 levels, corroborating the identification of the $E_c - 0.075$ eV level as a single acceptor state of V_3 in the stable FFC configuration.⁶

FIG. 1. DLTS spectra of n-type samples ($p^+n^-n^+$) as-irradiated, after heat-treatment at 200°C for 20 min and 300°C for 45 min duration. The lock-in weighting function is used and the rate window is $(40 \text{ ms})^{-1}$.

After heat-treatment at 300 °C for 45 min, a shift in the V_2 peaks takes place and new levels identified as V_2O levels occur.^{3,4,17} However, the amplitude of the $V_2O(-/0)$ level is larger than that of $V_2(-/0)$, which can be attributed to the formation of an additional level with similar position as $V_2O(-/0)$. In parallel to the shift in temperature of the $V_2(-/0)$ peak, V_3 in the FFC configuration anneals out with the formation of a new level at $E_c - 0.34$ eV labeled as $V_3O(=/-)$. The difference between the amplitudes of the $V_2O(-/0)$ and $V_2O(=/-)$ peaks is almost the same as the amplitudes of the $E_c - 0.075$ eV (V_3 in FFC configuration) and $E_c - 0.34$ eV levels. These two new levels ($E_c - 0.34$ eV and the one contributing to the $V_2(-/0)$ peak) are assigned to the single and double acceptor states of V_3O , respectively, in accordance with the results in Ref. 6. The intensity of the V_3O levels is about 25%–30% of that of the V_2 and V_2O levels, and in conclusion, our results for the n-type samples show very similar behavior to those reported previously by Markevich *et al.*⁶ and support the suggestion of single and double acceptor states of V_3O at $E_c - 0.34$ eV and $E_c - 0.45$ eV, respectively.

Figure 2 shows DLTS spectra of as-irradiated p-type samples (n^+p diodes), after annealing at 200 °C and 300 °C. In the as-irradiated sample, two major peaks appear at $E_v + 0.19$ eV and $E_v + 0.36$ eV and are identified as single donor states of V_2 and the interstitial carbon-interstitial oxygen (C_iO_i) pair, respectively.¹⁸ In addition, the spectrum of the as-irradiated sample reveals two additional levels with positions at $E_v + 0.10$ eV and $E_v + 0.09$ eV, labeled as $V_3(2+/+)$ and $H1$, respectively.

As the DLTS measurements were done 24 h after the irradiation, no levels associated with interstitial carbon (C_i) are observed, consistent with previous reports in the literature.¹⁹ Further, the C_iO_i peak is broad and contains a second level positioned at $E_v + 0.35$ eV, which completely transforms into C_iO_i after the heat-treatment at 200 °C. This observation is also consistent with previous studies on irradiated p-type samples and the $E_v + 0.35$ eV level is proposed to arise from a metastable configuration of C_iO_i ($C_iO_i^*$).^{20,21}

The $E_v + 0.10$ eV ($V_3(2+/+)$) level anneals out after the heat-treatment at 200 °C for 20 min. At the same time, a decrease in the V_2 peak takes place although $V_2(+/0)$ is

known to be stable up to temperatures above 200 °C, as previously discussed. The loss in the $E_v + 0.19$ eV level amplitude equals the amplitude of the $E_v + 0.10$ eV level in the as-irradiated sample. Recently, a similar observation was made by Markevich *et al.*⁷ and they ascribed the $E_v + 0.19$ eV and $E_v + 0.10$ eV levels to the single and double donor states of V_3 in the planar (110) configuration.⁷ Here, it should also be emphasized that the thermal evolution of the $E_v + 0.19$ eV and $E_v + 0.10$ eV levels exhibits a close resemblance with that of the acceptor states of the V_3 planar configuration in the n-type samples. A detailed study on the identification of the $H1$ ($E_v + 0.09$ eV) level is given in Ref. 22 and no further discussion is made here. This holds also for the $E_v + 0.29$ eV level, attributed to a interstitial boron-substitutional carbon (B_iC_s) center,²³ which appears after 200 °C treatment.

During isothermal annealing of the p-type samples, a gradual transformation from $V_2(+/0)$ to $V_2O(+/0)$ with a one-to-one proportionality occurs and a detailed kinetics analysis is given in Ref. 24. In parallel, defects with levels at $E_v + 0.24$ eV and $E_v + 0.11$ eV appear after the 300 °C annealing, as shown in Fig. 2; Markevich *et al.*⁷ have ascribed these levels to single and double donor states of V_3O . The amplitudes of these two levels are identical and increase gradually with annealing time, as shown in Fig. 3(a) for isothermal treatment at 300 °C. Further, the final (maximum) concentration of these levels shows a close to one-to-one correlation with the V_3 concentration directly after irradiation in non-annealed samples.

Fig. 3(b) shows the evolution of V_3O as a function of time for all the temperatures studied and the formation can be described by the relation

$$[V_3O] = [V_3] - [V_3]e^{-c(T)t}, \quad (1)$$

where $[V_3]$ is the initial concentration in the as-irradiated samples, $[V_3O]$ is the concentration of V_3O , and $c(T)$ is a temperature dependent rate constant.

Fig. 4 shows the Arrhenius plot of $c(T)$, and $c(T)$ exhibits a thermally activated behavior described by

$$c(T) = c_0 e^{-\frac{E_a}{kT}}, \quad (2)$$

where E_a is the activation energy, c_0 is the frequency factor, T is the absolute temperature, and k is Boltzmann's constant. For both the levels, $V_3O(2+/+)$ and $V_3O(+/0)$, $c(T)$ is closely described by $E_a = 1.50 \pm 0.04$ eV and $c_0 = 2.1 \pm 0.7 \times 10^{10} \text{ s}^{-1}$. The value deduced for E_a is practically identical to that obtained by Markevich *et al.*^{6,11} for V_3 migration in n-type material strongly supporting the relationship between the annealing of V_3 and the formation of the donor states of V_3O in our p-type samples. The diffusivity of V_3 , D_{V_3} can be quantitatively estimated from the results in Fig. 4. Applying the theory for diffusion limited reactions,²⁵ the following relation for V_3O holds:

$$\frac{d[V_3O(t)]}{dt} = c(T)[V_3(t)] = 4\pi R D_{V_3} [O_i][V_3(t)], \quad (3)$$

where R is the capture radius for the trapping reaction and $[O_i]$ is the concentration of interstitial oxygen. In Eq. (3), O_i

FIG. 2. DLTS spectra of p-type sample as-irradiated, after heat-treatment at 200 °C for 20 min and 300 °C for 45 min duration. The lock-in weighting function is used and the rate window is $(20 \text{ ms})^{-1}$.

FIG. 3. (a) The amplitudes of the $V_3O(+/0)$ and $V_3O(2+/+)$ levels versus time during the isothermal annealing at 300 °C. (b) The difference between the initial concentration of $V_3(2+/+)$ and the growing concentration of $V_3O(2+/+)$ during isothermal annealing at 200, 225, 250, 275, and 300 °C. The values have been normalized to the initial amplitude of V_3 . The dotted lines represent least squares exponential fits to the experimental data.

FIG. 4. Arrhenius plot for the formation rate of V_3O . The dotted line represents an exponential fit of the rate values.

is assumed to be immobile exhibiting a diffusivity of $10^{-23} \text{ cm}^2/\text{s}$ in the studied temperature range.²⁶ By combining Eqs. (2) and (3) with $D_{V_3} = D_{V_3}^0 e^{-\frac{E_a}{kT}}$, one obtains

$$D_{V_3}^0 = \frac{c_0}{4\pi R[O_i]}. \quad (4)$$

Putting R equal to $5 \pm 1 \text{ \AA}$ (geometrical value) and $[O_i] = 4 \pm 1 \times 10^{17} \text{ cm}^{-3}$ (measured value) and using $c_0 = (2.1 \pm 0.7) \times 10^{10} \text{ s}^{-1}$, one arrives at $D_{V_3}^0 = (8.5 \pm 3.5) \times 10^{-2} \text{ cm}^2/\text{s}$.

V_3 is, similarly to V_2 , an intrinsic defect generated by high-energy particle irradiation, and this could account for its consistent appearance with a ratio about 30% to the V_2 concentration in samples irradiated with 1.8 MeV protons. V_3 is presumably generated directly during the proton irradiation, i.e., the generation does not involve long-distance migration and pairing of V and V_2 .²⁷ V_2 is estimated to have a threshold displacement energy of $\sim 80 \text{ eV}$ (Ref. 27) and assuming a threshold $\sim 180 \text{ eV}$ for V_3 , a ratio of 30% is obtained between the generation rates of V_3 and V_2 for 1.8 MeV protons according to TRIM simulations. Such a rather high threshold for the generation of V_3 is also corroborated by that electron energies above $\sim 5 \text{ MeV}$ are required to detect an appreciable concentration of V_3 .⁶

Moreover, the activation energy deduced for V_3 migration represents the neutral charge state (the Fermi level is close to midgap at the annealing temperatures) and a value $\sim 0.2 \text{ eV}$ higher than for migration of the neutral V_2 ²⁴ (~ 1.5 versus $\sim 1.3 \text{ eV}$) appears conceivable considering the larger size of V_3 . It is also interesting to note that $D_{V_3}^0$ is substantially larger than $D_{V_2}^0$ ($\sim 8 \times 10^{-2}$ versus $\sim 1.5 \times 10^{-3} \text{ cm}^2/\text{s}$), possibly associated with a larger geometrical factor for V_3 diffusion but further studies need to be pursued to confirm this speculation.

IV. CONCLUSIONS

Annealing studies of V_3 and V_3O complexes in proton irradiated p- and n-type Si samples have been performed using DLTS. In both types of materials, V_3 in the (110) planar configurations disappears at temperatures below 200 °C, and a transformation to the stable FFC structure takes place. At temperatures above 200 °C, formation of V_3O is demonstrated and detailed isothermal studies of the p-type samples reveal a process with first-order kinetics having with an activation energy of $1.50 \pm 0.04 \text{ eV}$. These results provide firm evidence in favor of a process where migrating V_3 's are trapped by immobile oxygen atoms resulting in the formation of V_3O pairs. In both the p- and n-type materials, V_3 appears in the neutral charge state during the transformation to V_3O . Applying the theory for diffusion limited reactions, the diffusivity of V_3 in the studied p-type samples is determined to be $(8.5 \pm 3.5) \times 10^{-2} \exp [-(1.50 \pm 0.04) \text{ eV}/kT] \text{ cm}^2/\text{s}$, which represents the neutral charge state of V_3 .

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