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Defect reactions associated with divacancy elimination in silicon

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Abstract

Defect reactions associated with the elimination of divacancies (V_2) have been studied in n-type Czochralski (Cz) grown and float-zone (FZ) grown Si crystals by means of conventional deep-level transient spectroscopy and high-resolution Laplace deep-level transient spectroscopy (LDLTS). Divacancies were introduced into the crystals by irradiation with 4 MeV electrons. Temperature ranges of the divacancy disappearance were found to be 225–275 °C in Cz Si crystals and 300–350 °C in FZ Si crystals upon 30 min isochronal annealing. Simultaneously with the V_2 disappearance in Cz Si crystals a correlated appearance of two electron traps with activation energies for electron emission 0.23 eV $\{E(0.23)\}$ and 0.47 eV $\{E(0.47)\}$ was observed. It is argued that the main mechanism of the V_2 disappearance in Cz Si crystals is related to the interaction of mobile divacancies with interstitial oxygen atoms. This interaction results in the formation of V_2O centres, which are responsible for the $E(0.23)$ and $E(0.47)$ traps. Electronic properties of the V_2O complex were found to be very similar to those of V_2 but energy levels of the two defects could easily be separated using LDLTS.

In FZ Si crystals, a few electron traps appeared simultaneously with the V_2 annihilation. The small concentration of these traps compared with the V_2 concentration before annealing prevented their reliable identification.

1. Introduction

The divacancy (V_2) is one of the dominant defects induced in Si crystals by irradiation with high-energy particles (electrons, neutrons, ions, etc) [1, 2]. V_2 is formed either as a multiple displacement defect in the primary radiation damage event or as a result of the

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interaction between two monovacancies. The two vacancies in the complex are positioned at adjacent lattice sites [1]. The centre can exist in four charge states ($V_2^{=}$, V_2^{-}, V_2^{0}, V_2^{+}) and introduces three deep levels, $V_2(=/-)$, $V_2(-/0)$ and $V_2(+/0)$, into the band gap located at $E_c - 0.23$, $E_c - 0.41$ and $E_v + 0.20$ eV, respectively [1–6]. These identifications are fairly well established despite frequently observed intensity differences between the deep-level transient spectroscopy (DLTS) peaks corresponding to the single and double acceptor levels, an effect especially pronounced in Si crystals irradiated with neutrons or heavy ions. Two possible reasons are likely to account for the observed effect:

(a) the interference of the $V_2(-/0)$-related DLTS signal with signals due to other defects with close emission characteristics [7, 8] and
(b) the local charge carrier compensation in highly disordered regions that prevents electron population of the $V_2(=/-)$ level [9].

Watkins and Corbett [1] have shown that divacancies could migrate rather long distances prior to dissociation. The activation energy of the V_2 diffusion has been found to be about 1.3 eV [1, 10]. The migrating V_2 can interact with other imperfections in the Si lattice. In particular, oxygen atoms are thought to be effective traps for the V_2, and the divacancy–oxygen interaction is suggested to be the main mechanism of the V_2 elimination upon annealing of irradiated Czochralski (Cz) grown Si crystals [1, 11–14]. An EPR Si-A14 signal and an absorption line at 833.5 cm^{-1} have been assigned to a complex incorporating the divacancy and an oxygen atom (V_2O) [12, 14]. However, the electronic properties of the V_2O centre have not been established definitively.

In a recent article by Monakhov *et al* [15], the transformation of V_2-related acceptor levels to two close levels (labelled as X levels in [15]) was observed in oxygenated float-zone (FZ) grown Si upon annealing in the temperature range 220–300 °C. The levels were tentatively assigned to the V_2O complex. However, in their discussion of the possible origin of the observed new levels Monakhov *et al* [15] mentioned that in previous work on V_2 annealing the transformation of V_2 levels to X levels was not observed in Cz Si crystals. It was speculated that there might be a significant energy barrier for the $V_2 + O_i \rightarrow V_2O$ reaction and that the interaction with oxygen could not be the main mechanism of the V_2 disappearance in Cz grown silicon.

The situation in relation to V_2 annihilation upon annealing of FZ Si crystals is even less certain. The V_2 dissociation as well as interactions with impurity atoms were suggested to occur in FZ Si [1, 16].

It is known that defects having dangling bonds are very effective traps for hydrogen atoms in Si [17]. Divacancies possess six dangling bonds and are expected to interact effectively with hydrogen atoms. However, the interaction of hydrogen with the divacancy and the electronic properties of the resulting complexes are not well understood. Theory predicts up to six V_2H_n ($1 \leqslant n \leqslant 6$) complexes, presumably all with electrical activity except for the fully saturated V_2H_6. Only one electrically active V_2H_n complex has been identified so far. The neutrally charged complex V_2H^0 was observed by EPR after low-temperature proton implantation followed by heating to room temperature [18]. Subsequently, the acceptor level of the V_2H complex has been associated with both an electron trap with a level at $E_c - 0.43$ eV observed by Laplace deep-level transient spectroscopy (LDLTS) [19] and an electron trap with a level at $E_c - 0.45$ eV observed by conventional DLTS [20]. Despite the close values of the activation energies, the two levels assigned to the $V_2H(-/0)$ are not identical. As hydrogen atoms are mobile at room temperature in Si crystals, the introduction of hydrogen (by wet chemical etching, H plasma treatment, etc) can alter the V_2 elimination process significantly.

Summarising, special care needs to be taken in order to identify the processes related to the disappearance of V_2 in Si crystals with different defect-impurity content. In the present work the results of our study of V_2 annihilation in n-type Si crystals with different oxygen, carbon and hydrogen content are presented.

2. Experimental details

The samples used in this study were prepared from two n-type Cz Si crystals with initial resistivities (ρ) of 5 and 30 Ω cm and from two n-type Si crystals ($\rho \sim 1$ and 60 Ω cm) which were grown by the vacuum FZ technique. The concentrations of interstitial oxygen and substitutional carbon atoms in the samples were measured by the optical absorption method at room temperature. In the Cz Si samples the oxygen concentration was about (9–10) $\times$ 10^{17} cm^{-3} and the carbon concentration was below the detection limit of 2×10^{15} cm^{-3}. In the FZ Si samples oxygen was not detectable by optical absorption measurements at room temperature and so has a concentration of $<10^{15}$ cm^{-3}; the concentration of substitutional carbon atoms was found to be about 5×10^{15} cm^{-3}. The samples were irradiated at room temperature with 4 MeV electrons. The irradiated samples were subjected to 30 min isochronal annealing in the temperature range 75–400 °C with temperature increments of 25 °C. Electronic levels were characterized with conventional DLTS and high-resolution LDLTS techniques [21]. Gold Schottky diodes were made by thermal evaporation of Au. In order to avoid hydrogen incorporation, Schottky barriers were fabricated on samples with mechanically polished surfaces. Hydrogen was introduced into some other samples by wet chemical etching in HF + HNO_3 mixtures before the Au evaporation.

3. Experimental results

3.1. Cz Si

Figure 1 shows conventional DLTS spectra for two neighbouring electron-irradiated Cz Si samples, which were measured with the Schottky diodes fabricated on mechanically polished and chemically etched surfaces. Four dominant peaks with maxima at about 75, 95, 135 and 230 K for an emission rate window of 200 s^{-1} are observed in the spectrum for the sample with the mechanically polished surface. These peaks are referred to as ECz129, ECz163, ECz239 and ECz424 peaks in the following text. In this labelling 'E' denotes an electron trap, 'Cz' is the material growing method, and the numbers are trap ionization energies in meV derived from Arrhenius plots of T^2-corrected electron emission rates. The conventional DLTS spectrum resembles those reported in the literature for electron-irradiated Cz Si crystals [4, 5, 22]. On the basis of a comparison of the measured trap parameters (activation energies for electron emission and values of electron capture cross section) with the known values for defects induced by electron irradiation [4, 5, 22], we assign the ECz129 peak to an acceptor level of the self-interstitial–dioxygen complex, the ECz163 peak to an acceptor level of the vacancy–oxygen (VO) complex, the ECz239 peak to a double acceptor level of divacancy and the ECz424 peak to a single acceptor level of divacancy with an admixture of another level with small concentration. This minor level disappeared completely after annealing at 100 °C.

The introduction of hydrogen by wet chemical etching resulted in significant changes in the DLTS spectrum (figure 1). The most prominent features are the following:

(i) The magnitudes of the DLTS peaks due to radiation-induced defects, the ECz129, ECz163 and ECz239 traps, are significantly lower in the spectrum of the chemically etched sample compared with those for the polished sample.

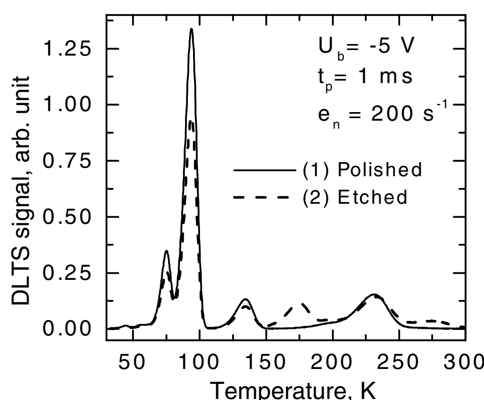

Figure 1. DLTS spectra from Cz Si crystals with an initial resistivity of 5 Ω cm after irradiation with 4 MeV electrons at room temperature. Shottky diodes were fabricated on (1) mechanically polished and (2) chemically etched surfaces. Dose of irradiation was 1×10^{15} cm^{-2}. Measurement settings were $e_n = 200$ s^{-1}, bias $-5 \rightarrow -0.2$ V, and pulse duration 1 ms.

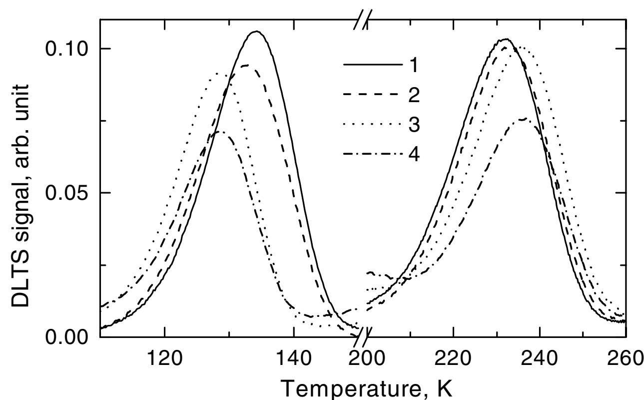

Figure 2. Development of DLTS spectra for an electron-irradiated Cz Si sample with an initial resistivity of 5 Ω cm upon 30 min isochronal annealing with temperature increments of 25 °C. The spectra were measured after anneals at: (1) 200 °C, (2) 225 °C, (3) 275 °C and (4) 300 °C. Measurement settings were as in figure 1.

(ii) A number of extra DLTS peaks are observed in the spectrum of the etched sample, the strongest peaks being those with temperatures of peak maximum at about 175 K (ECzH320) and 275 K (ECzH450).

(iii) The temperature of the peak maximum is higher for the ECz428 peak for the etched sample ($T_{\text{m-Et}} \sim 233$ K) compared with the polished one ($T_{\text{m-Pol}} \sim 231$ K).

LDLTS measurements showed that this effect is related to the appearance in the etched sample of another peak (ECzH440), which is close to the ECz428 peak. The above features are associated with the interaction of hydrogen with radiation-induced defects, particularly with the VO and V_2 [16, 19, 20, 23–26]. The ECzH320 trap can be assigned to the VOH complex [16, 19, 20, 23–26]. For reliable identification of the other H-related traps, ECzH440 and ECzH450, further work is required.

In the following we will concentrate only on the annealing behaviour of the divacancy-related levels in hydrogen-lean samples. After anneals in the temperature range 100–200 °C

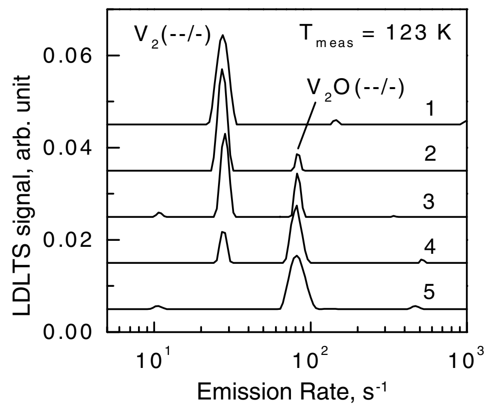

Figure 3. Development of LDLTS spectra measured at 123 K for an electron-irradiated Cz Si sample with an initial resistivity of 5 Ω cm upon 30 min isochronal annealing with temperature increments of 25 °C. The spectra were measured after anneals at: (1) 150 °C, (2) 200 °C, (3) 225 °C, (4) 250 °C, and (5) 275 °C. Measurement settings were bias $-5 \rightarrow -0.2$ V and pulse duration 1 ms.

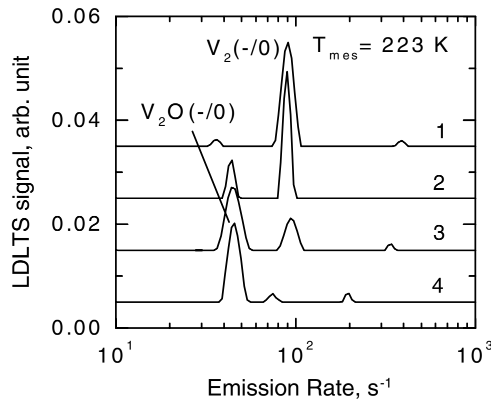

Figure 4. Development of LDLTS spectra measured at 223 K for an electron-irradiated Cz Si sample with an initial resistivity of 5 Ω cm upon 30 min isochronal annealing with temperature increments of 25 °C. The spectra were measured after anneals at: (1) 150 °C, (2) 225 °C, (3) 250 °C and (4) 275 °C. Measurement settings were bias $-5 \rightarrow -0.2$ V and pulse duration 1 ms.

a small decrease in intensity of the ECz239 and ECz424 peaks without changes in the peak positions was observed. Figure 2 shows the development of the V_2-related peaks in conventional DLTS spectra upon isochronal annealing in the temperature range 200–300 °C. Anneals in the range 225–275 °C result in changes of the temperatures of the peak maximum without significant changes in the magnitude of the peaks. The ECz239 peak moved to lower temperatures, while the ECz424 peak shifted to higher temperatures. The peaks observed subsequent to annealing are referred to as ECz233 and ECz467 in the following text. Further annealing at temperatures higher than 300 °C caused a decrease in intensity of the ECz233 and ECz467 peaks and their complete elimination at 350 °C.

Figure 3 shows the development of the LDLTS spectrum measured at 123 K after isochronal anneals. The LDLTS spectrum after annealing at 150 °C demonstrated the presence of one dominant line due to electron emission from the double acceptor level of the divacancy.

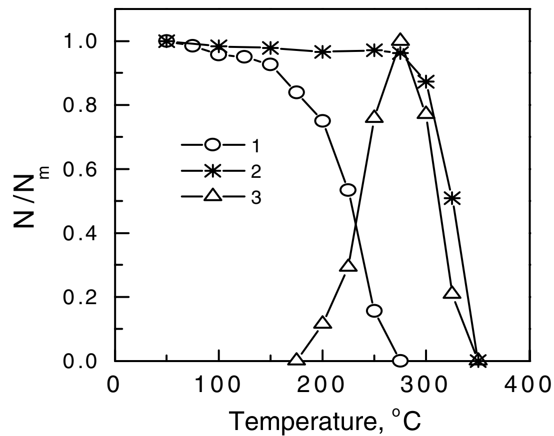

Figure 5. Changes upon 30 min isochronal annealing in the normalized concentration of divacancies in electron-irradiated (1) Cz Si and (2) FZ Si samples and (3) in the concentration of the defect related to the ECz233 trap in Cz Si samples. The irradiation dose was 1×10^{15} cm^{-2} for Cz Si and 3×10^{13} cm^{-2} for FZ Si samples.

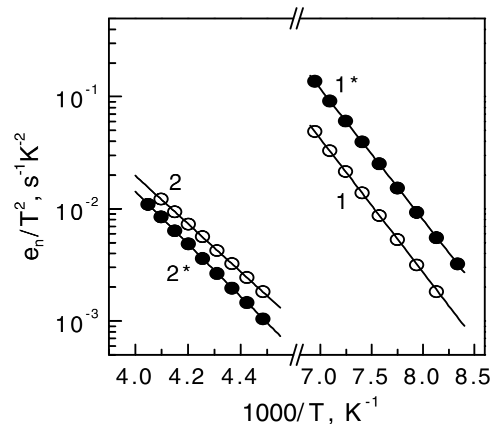

Figure 6. Arrhenius plots of T^2-corrected electron emission rate from (1) double and (2) single acceptor levels of the divacancy and from (1*) ECz233 and (2*) ECz467 traps. Emission rates were determined from LDLTS measurements on an electron-irradiated Cz Si sample with initial resistivity of 5 Ω cm after anneals at (1)–(2) 150 °C and (1*)–(2*) 275 °C.

Heat treatment at 200 °C resulted in the appearance of another line with a higher emission rate. Upon further anneals in the temperature range 225–275 °C the original $V_2(=/-)$-related peak was eliminated, while the new line developed. Changes in concentrations of the original and new traps upon annealing in the range 225–275 °C showed anti-correlation with a ratio close to 1:1. A very similar picture was observed in LDLTS spectra measured at 225–245 K after isochronal anneals (figure 4). The original $V_2(-/0)$-related LDLTS peak disappeared upon annealing in the range 225–275 °C, and the simultaneous appearance of another peak with a slightly lower emission rate was observed. The LDLTS lines that arise upon annealing correspond to the ECz233 and ECz467 traps in conventional DLTS spectra. The concentrations of the ECz233 and ECz467 traps were equal at all stages of their development. This result suggests an attribution of the traps to two different charge states of the same defect. Changes

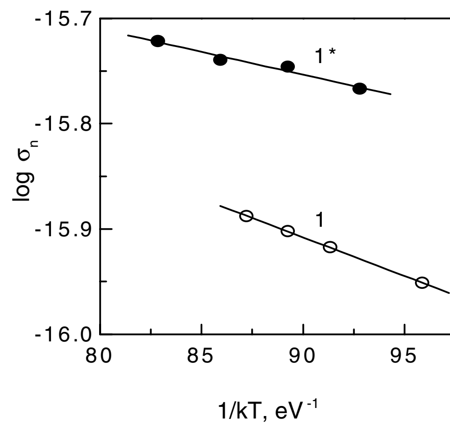

Figure 7. Temperature dependencies of electron capture cross sections for (1) the double acceptor level of divacancy and (1*) the ECz233 trap. Solid lines are calculated values on the basis of the equation $\sigma_n = \sigma_\infty \exp(-E_\sigma/kT)$ with parameters determined from least-squares fits of experimental data.

Table 1. Parameters of selected deep-level traps, which were observed in electron-irradiated Cz Si crystals. The parameters were derived from least-squares fits of experimentally determined temperature dependencies of electron emission rates (e_n) and directly measured capture cross sections (σ_n) to those described by equations $e_n/T^2 = A\exp(-E_{na}/kT)$ and $\sigma_n = \sigma_{n\infty}\exp(-E_\sigma/kT)$. The e_n and σ_n values were determined from LDLTS measurements.

Trap label	E_{na} (eV)	A (s^{-1} K^{-2})	σ_n (cm^2)	Assignment
ECz239	0.239	1.15×10^7	$5.7 \times 10^{-16} \times \exp(-0.017\ eV/kT)$	$V_2(=/-)$
ECz233	0.233	1.95×10^7	$4.3 \times 10^{-16} \times \exp(-0.01\ eV/kT)$	$V_2O(=/-)$
ECz424	0.424	6.9×10^6	$\geqslant 2 \times 10^{-15}$	$V_2(-/0)$
ECz467	0.467	3.7×10^7	$\geqslant 2 \times 10^{-15}$	$V_2O(-/0)$

upon isochronal annealing in normalized concentrations of divacancies and the defect related to the ECz233 trap are presented in figure 5.

Figure 6 shows Arrhenius plots of T^2-corrected electron emission rates for the V_2-related levels and the ECz233 and ECz467 levels. The data were obtained from LDLTS measurements. Figure 7 shows the temperature dependencies of electron capture cross sections for the double acceptor level of the divacancy and the ECz233 trap. The values of electron capture cross section for the single acceptor level of the divacancy and the ECz467 trap were estimated to be $\geqslant 2 \times 10^{-15}$ cm^2 using the filling pulse width method. Unfortunately, a limitation on the pulse width of 20 ns in our experimental set-up does not allow these values to be determined precisely with the material of the carrier density used; it is only possible to derive a lower limit. All the determined trap parameters are presented in table 1. The electronic parameters of the ECz233 and ECz467 traps are similar to those for the double and single acceptor levels of the divacancy. So, it can be suggested that the ECz233 trap represents a double acceptor level and the ECz467 trap is related to a single acceptor level.

The results obtained indicate that upon isochronal annealing in the temperature range 225–275 °C a transformation of the divacancy to another centre with a similar electronic structure occurs in Cz Si crystals.

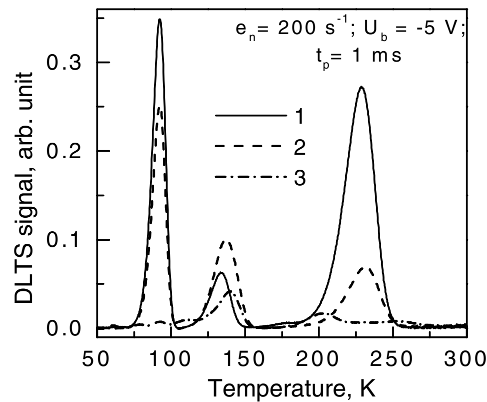

Figure 8. DLTS spectra of FZ Si samples with initial resistivity of 60 Ω cm after electron irradiation with a fluence of 3×10^{13} cm^{-2} (1) and subsequent anneal at (2) 200 °C and (3) 350 °C for 30 min. Measurement settings were $e_n = 200$ s^{-1}, bias $-5 \rightarrow -0.2$ V and pulse duration 1 ms.

3.2. FZ Si

Figure 8 shows conventional DLTS spectra for electron-irradiated FZ Si samples after an electron irradiation and at different stages following isochronal annealing. Three dominant peaks with maxima at about 92, 135 and 230 K for an emission rate window of 200 s^{-1} are observed after the irradiation. These peaks are referred to as EFZ161, EFZ240 and EFZ453 in the following text. We have assigned the EFZ161 peak to the single acceptor level of the interstitial–substitutional carbon pair (C_i–C_s) [27] and the EFZ240 peak to the double acceptor level of divacancy. The EFZ453 peak is related to electron emission from the single acceptor levels of a phosphorus–vacancy centre (E centre) and divacancy [4–6, 28]. The EFZ161 peak was found to have a small contribution ($\leqslant$5%) from the acceptor level of the VO complex. The concentration of divacancies was about 25% of the E centre concentration.

Annealing of the electron-irradiated FZ Si samples in the temperature range 125–175 °C resulted in a significant decrease in the magnitude of the EFZ453 DLTS peak. The decrease is related to the elimination of E centres upon annealing. The peak with a maximum at about 230 K (EFZ428) remaining after annealing at 200 °C (spectrum 2 in figure 8) is related to the electron emission from the singly negatively charged state of the divacancy. An examination of the spectra also shows significant changes in the magnitudes of the EFZ161 and EFZ240 peaks, the former being decreased and the latter being increased upon annealing. Application of the LDLTS technique shows that the increase in the magnitude of the peak in the range of 120–150 K is related to the appearance of another electron trap (EFZ228) with emission rates close to those for the electron emission from the doubly negatively charged state of the divacancy (EFZ240). Figure 9 shows LDLTS spectra measured at 130 K for an as-irradiated FZ Si sample and after isochronal anneals. The EFZ228 peak has been assigned to an acceptor level of a pair of substitutional carbon atoms (C_s–C_s) [29, 30]. The C_s–C_s complex is formed when a free vacancy released upon E centre annealing is captured by the C_i–C_s complex [30].

Figure 5 shows changes in the concentration of divacancies in irradiated FZ Si samples upon isochronal annealing. The changes were monitored by observation of the EFZ240 and EFZ428 traps using the LDLTS technique. After the complete disappearance of the divacancies the dominant electron trap was related to the C_s–C_s complex (EFZ228), and a few traps with small concentrations were observed (spectrum 3 in figure 8). We could not correlate the disappearance of divacancies with the appearance of other electron traps in FZ Si samples.

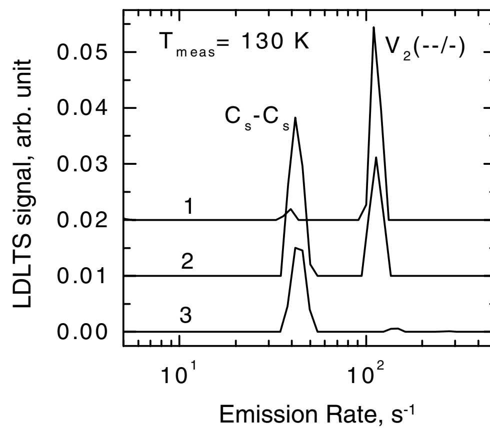

Figure 9. LDLTS spectra measured at 130 K for FZ Si samples, the conventional DLTS spectra of which are shown in figure 8.

4. Discussion

The results obtained indicate that upon isochronal annealing in the temperature range 225–275 °C a transformation of the divacancy to another centre with similar electronic structure occurs in Cz Si crystals. Comparing the results with those reported in the literature on V_2 annealing in Cz Si and FZ Si crystals [1, 11–14], one can suggest that the observed disappearance of the V_2-related levels upon annealing of irradiated Cz Si samples at 225–275 °C is associated with the interaction of mobile divacancies with interstitial oxygen atoms. This interaction results in the formation of V_2O centres, which are responsible for the ECz233 and ECz467 electron traps. The electronic properties of the V_2O complex are found to be very similar to those of V_2 but the energy levels of the two defects could easily be separated using LDLTS.

Very similar results were obtained recently by Monakhov *et al* [15] in a DLTS study of V_2 elimination in proton-implanted oxygenated FZ Si crystals. The transformation of V_2 to another defect with similar electronic properties was observed in [15] upon anneals of the implanted samples in the temperature range 220–300 °C. The new defect was tentatively assigned to the V_2O complex. However, Monakhov *et al* had some doubts about this assignment [15], because, as they mentioned, in previous DLTS works on the study of V_2 annealing out, such a transformation of V_2 was not detected in Cz Si crystals and only a small difference was observed between the V_2 elimination rates in Cz and FZ Si samples [16]. As a possible reason for the discrepancy it was suggested that there might be a significant energy barrier for the $V_2 + O_i \rightarrow V_2O$ reaction. Consequently, the interaction with oxygen was thought not to be the main mechanism for the V_2 disappearance in Cz grown silicon.

We have observed the annealing-induced transformation of the V_2-related levels to the ECz233 and ECz467 traps in both the electron-irradiated Cz Si crystals which have been studied. In addition, annealing rates (temperatures) of divacancies are found to differ significantly in Cz and FZ Si samples (figure 5) in agreement with results presented in [1], but in disagreement with the results of [16]. Upon the disappearance of V_2 in the irradiated FZ Si samples we did not observe the formation of deep-level defects with concentrations comparable to that of V_2 before annealing. A combination of these facts can be considered as confirmation of the effect of oxygen on the V_2 annealing out in Cz Si samples and of

the assignment of the ECz233 and ECz467 electron traps to the V_2O centre. Other indirect evidence for the identification of the ECz233 and ECz467 traps with energy levels of the V_2O complex comes from the similarity in annealing behaviour of these traps and the V_2O-related Si-A14 EPR signal [12]. The traps and the Si-A14 signal disappeared in the same temperature range (300–350 °C) upon 30 min isochronal annealing (figure 5 and figure 5 in [12]).

A possible reason for the discrepancy between the results presented in [16] and our results could be the difference in the type of irradiation used. Pellegrino *et al* [16] carried out their study on 5.6 MeV self-ion-implanted Si, while in our work and in [1] the Si crystals were irradiated with 1.5–4 MeV electrons, which would not produce the clusters of damage typical of high-energy ions. The effect of the type of irradiation on the annealing behaviour of V_2 was discussed earlier in [31]. It was mentioned that after neutron irradiation, which is known to produce defect clusters, divacancies disappeared upon isochronal annealing in the same temperature range in both FZ Si and Cz Si crystals, while there was a significant difference in the annealing out temperatures of V_2 in electron-irradiated Si crystals grown by different methods [31].

Acknowledgments

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