

Electrically active radiation-induced defects in Czochralski-grown Si with low carbon content

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Abstract

Electrically active defects induced by electron irradiation in Czochralski (Cz)-grown Si crystals with low carbon content ($N_C \leq 2 \times 10^{15} \text{ cm}^{-3}$) have been studied by means of Hall effect measurements, deep level transient spectroscopy (DLTS) and high-resolution Laplace DLTS (LDLTS). It has been found that in n-type carbon-lean Cz-Si irradiated at room temperature a centre with an acceptor level at $E_c - 0.11 \text{ eV}$ ($E_{0.11}$) is one of the dominant radiation-induced defects. This centre is not observed after irradiation in Cz-Si crystals with $N_C > 10^{16} \text{ cm}^{-3}$. The $E_{0.11}$ trap anneals out in the temperature range 100–130 °C with the activation energy 1.35 eV.

In p-type Cz-Si crystals with low carbon content and boron ($N_B \leq 2 \times 10^{14} \text{ cm}^{-3}$) one of the dominant radiation-induced defects has been found to be a bistable centre with an energy level at $E_v + 0.255 \text{ eV}$ ($H_{0.255}$). It has been inferred from the analysis of temperature dependences of electron occupancy of this level that it is the $E(0/++)$ level of a defect with negative Hubbard correlation energy (negative U). The activation energy for hole emission from the doubly positively charged state of the $H_{0.255}$ centre has been determined as 0.358 eV from LDLTS measurements.

It is argued that the $E_{0.11}$ and $H_{0.255}$ energy levels are related to a complex incorporating an oxygen dimer and Si self-interstitial.

1. Introduction

The carbon concentration in modern large diameter silicon crystals grown by the Czochralski method is not high ($N_C \leq 2 \times 10^{15} \text{ cm}^{-3}$). The role of carbon as an effective sink for Si self-interstitials (I) is suppressed in such crystals, and interactions of self-interstitials with other traps can be expected.

It has been found that interstitial oxygen atoms (O_i) as well as oxygen-related complexes can interact with self-interstitials in Cz-Si crystals [1–10]. Experimental studies of samples irradiated at about 80 K show that O_i can trap I which results in the formation of IO_i complexes [1–5] which are stable below 270 K. This defect can trap an additional I and transform to I_2O_i [6, 7]. It is argued in a recent study [8] that the Si self-interstitial can be trapped by an oxygen dimer (O_{2i}), the concentration of which is usually $(1–2) \times 10^{15} \text{ cm}^{-3}$ in as-grown Cz-Si crystals [11, 12], with the formation of an IO_{2i} complex that is stable at room temperature. This complex gives rise to two local vibrational mode (LVM) lines at 922 and 1037 cm^{-1} . Further, the IO_{2i} centre can trap another self-interstitial, a process that results in the formation of I_2O_{2i} complexes. This has been shown to be responsible for the LVM lines at 918 and 1034 cm^{-1} [8]. It has also been suggested that oxygen trimers (O_{3i}) could interact with self-interstitials in Si [8].

Regarding the electronic properties of the I_nO_{mi} complexes, the information available is rather limited. It has been argued that the IO_i defect should be a bistable centre and could exist in three or even four charge states: from singly negatively charged to doubly positively charged [4, 9]. However, the structure of the centre in different charge states and positions of energy levels are not certain [10]. The IO_{2i} complex has been found to possess a single acceptor level at about $E_c - 0.11 \text{ eV}$ [8]. It should be noted that according to the results of theoretical calculations a complex consisting of two oxygen atoms and a self-interstitial could be a double donor in Si [9, 13]. This complex has been considered as a possible candidate for the electrically active core of oxygen-related thermal double donors in Si [9].

In order to obtain more information on the electronic properties of self-interstitial–oxygen–interstitial-related complexes, Hall effect and capacitance transient measurements have been carried out on electron-irradiated n- and p-type Czochralski-grown Si crystals with a low carbon content.

2. Experimental details

Samples for this study were prepared from n- and p-type Si crystals, which were grown by the Czochralski technique. The crystals were doped with either phosphorus or boron during growth. The phosphorus concentration was in the range 2×10^{14} – $5 \times 10^{15} \text{ cm}^{-3}$. The boron concentration was $\leq 2 \times 10^{14} \text{ cm}^{-3}$. The concentrations of interstitial oxygen and substitutional carbon atoms measured by optical absorption were found to be $(1.0–1.3) \times 10^{18}$ and less than $2 \times 10^{15} \text{ cm}^{-3}$, respectively. The samples were heat-treated in the temperature range 650–700°C for 30 min in a nitrogen atmosphere in order to annihilate the grown-in oxygen-related thermal double donors and remove any hydrogen impurity atoms, which could be introduced during growth and wafer processing. The heat-treated samples were irradiated at room temperature with 4 MeV electrons or with γ -rays from a ^{60}Co source. The sample surfaces were cleaned after the irradiation and Schottky barriers were fabricated by thermal evaporation of Au on the n-type samples and by plasma sputtering of Ti on the p-type samples. Electron and hole traps were characterized with conventional deep level transient spectroscopy (DLTS) and high-resolution Laplace DLTS (LDLTS) techniques [14]. Hall effect measurements in the temperature range 77–400 K were also used to determine the positions of energy levels of radiation-induced defects in the gap.

3. Experimental results

3.1. Cz-Si:P

A short description of results obtained by conventional DLTS on deep level traps induced by irradiation with ^{60}Co gamma-rays in carbon-lean Cz-Si:P samples was presented in [8]. It

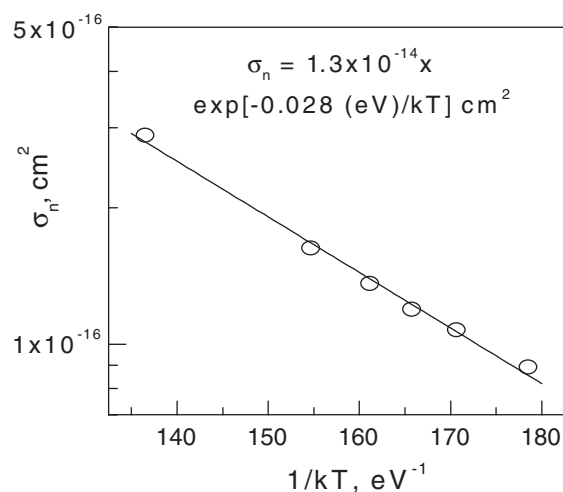


Figure 1. Temperature dependence of the electron capture cross section for the E128 trap. The solid line represents the dependence, which has been calculated on the basis of the equation $\sigma = \sigma_{\infty} \exp(-\Delta E_{\sigma}/kT)$ with parameters determined from the least-squares fit to the experimental data.

was found that the irradiation resulted in the introduction of two dominant electron traps with activation energies for electron emission (E_n) of 0.128 and 0.163 eV. A few minor electron traps were also observed. The dominant traps are referred to as E128 and E163 traps in the following text. In this labelling ‘E’ denotes an electron trap and the numbers are the trap activation energies in meV derived from Arrhenius plots of the T^2 -corrected electron emission rates. Based on the comparison of the trap parameters obtained, E_n and the directly measured capture cross section (σ_n), with those known from the literature on radiation-induced centres in Cz-Si [15, 16], the E163 trap was assigned to the vacancy–oxygen pair [17]. The E128 trap was not observed in irradiated Cz-Si crystals with carbon concentration higher than $2 \times 10^{16} \text{ cm}^{-3}$. The value of the electron capture cross section for this trap ($(1-2) \times 10^{-16} \text{ cm}^2$) and the absence of significant enhancement of electron emission by the electric field indicated that the E128 trap is likely to be an acceptor. The annealing behaviour of the trap was found to be similar to that of a defect responsible for the LVM absorption lines at 922 and 1037 cm^{-1} ; both the trap and the lines disappeared in the temperature range 100–130 °C upon a 30 min isochronal annealing. It has been argued that the defect responsible for the above absorption lines and electron emission with the activation energy of 0.128 eV could be related to a complex of a Si self-interstitial with an oxygen dimer (IO_{2i}) [8].

In the present study detailed emission and capture measurements have been carried out on the E128 trap using the Laplace DLTS technique. Consistent with conventional DLTS measurements [8], the values of activation energy for electron emission and the pre-exponential factor have been determined from an Arrhenius plot of T^2 -corrected emission rates as $0.128 \pm 0.001 \text{ eV}$ and $3.0 \times 10^7 \text{ s}^{-1} \text{ K}^{-2}$, respectively. Determination of the enthalpy and entropy of ionization of a trap requires direct capture cross section measurements to be made. Detailed measurements of the capture cross section of the E128 trap were not carried out in [8], but these have been done in the present work. Figure 1 shows the temperature dependence of the directly measured electron capture cross section for the E128 trap. Assuming that the observed dependence can be approximated by a phonon-assisted model [18], for which $\sigma = \sigma_{\infty} \exp(-\Delta E_{\sigma}/kT)$, the values of the energy barrier for the capture, ΔE_{σ} , and capture

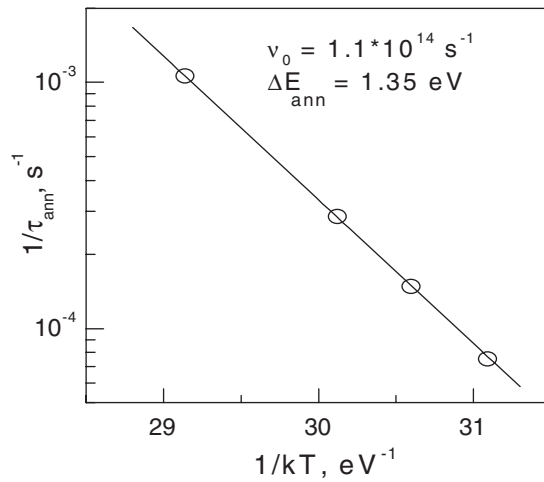


Figure 2. Arrhenius plot of annihilation rates for the E128 trap in an electron-irradiated Cz-Si sample. The solid line represents the dependence, which has been calculated on the basis of the equation $1/\tau_{\text{ann}} = v_0 \exp(-\Delta E_{\text{ann}}/kT)$ with parameters determined from the least-squares fit to the experimental data.

cross section at $1/T \rightarrow 0$, σ_α , have been determined as $0.028 \pm 0.003 \text{ eV}$ and $1.3 \times 10^{-14} \text{ cm}^2$, respectively. On the basis of the above values and the well known expression for electron emission rate, $e_n = \langle v \rangle N_c \sigma_\alpha \exp(\Delta S/k) \exp[-(\Delta H + \Delta E_\sigma)/kT]$ [19], values of the enthalpy and entropy of ionization for the E128 trap have been calculated as $\Delta H(-/0) = 0.10 \text{ eV}$ and $\Delta S(-/0) = -1.0 \text{ k eV K}^{-1}$. With the ΔH and ΔS values obtained the free energy of ionization, $\Delta G(-/0) = \Delta H(-/0) - T\Delta S(-/0)$, and the position of the defect energy level in the gap, $E(-/0) = E_c - \Delta G(-/0)$, can be determined. According to Hall effect measurements, at equilibrium conditions, ionization of the E128 trap occurs in the temperature range 80–150 K. In consequence the free energy of ionization should be in the range 0.107–0.113 eV. The $E(-/0)$ level of the defect is at about $E_c - 0.11 \text{ eV}$ and is weakly temperature dependent. This level position coincides with that derived from an analysis of temperature dependences of free electron concentration in irradiated Cz-Si samples with low carbon content.

In order to determine the activation energy of annihilation of the E128 trap, the decrease in concentration of the trap has been studied upon isothermal anneals in the temperature range 100–125 °C. It has been found that the disappearance of the trap at a constant temperature can be described by a first-order reaction. Figure 2 shows an Arrhenius plot of the annihilation rates determined for the E128 trap. The activation energy of the annihilation process and the pre-exponential factor have been determined to be $1.35 \pm 0.02 \text{ eV}$ and $1.1 \times 10^{14} \text{ s}^{-1}$. The value obtained for the pre-exponential factor is characteristic of the processes associated with reorientation/dissociation of defects in Si.

3.2. Cz-Si:B

3.2.1. Hall effect measurements. Figure 3 shows the temperature dependences of the carrier density, $p(T)$, in carbon-lean p-type Cz-Si samples before (curve 1) and after irradiation with 4 MeV electrons at room temperature (curves 2 and 3). It has been found that the temperature dependences of the carrier density in irradiated samples depend significantly on the measurement conditions. This is due to the introduction by irradiation of a bistable centre,

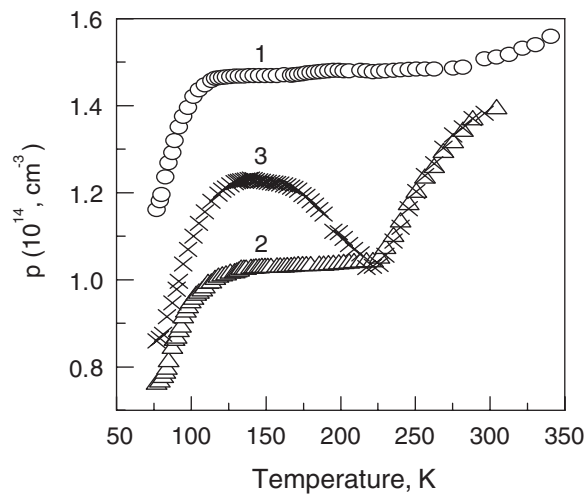


Figure 3. Temperature dependences of free carrier density for boron-doped Cz-Si samples after being (1) heated-treated at 650 °C for 30 min and then (2, 3) irradiated with 4 MeV electrons. The irradiation dose was $4 \times 10^{14} \text{ cm}^{-2}$. Measurement conditions are described in the text.

which is characterized by long time constants for the establishment of a thermodynamic equilibrium distribution between different configurations/charge states in some temperature ranges. This centre is responsible for the observed residual conductivity, which appears as a result of an abrupt change in temperature or as a result of the illumination of samples with white light. Curve 3 in figure 3 was obtained as follows: a sample was illuminated at room temperature and cooled rapidly to 77 K, then the measurements were carried out during heating up. Curve 2 was obtained when measurements were carried out during heating up after slow cooling ($\sim 0.02 \text{ K s}^{-1}$) of the sample from room temperature to 77 K. Residual conductivity is the cause of the observed decrease in the carrier density with the increase in temperature over the temperature range 150–225 K (curve 3). In the temperature range 225–275 K, where ionization of the bistable centre occurs, the experimental points correspond to equilibrium values of the carrier density.

It should be noted that the phenomenon of residual conductivity associated with the transformation of point defects in semiconductors can normally be considered as an indication of negative Hubbard electron correlation energy (U) for multi-charged centres [19, 20]. Therefore we have investigated whether the bistable centre, which has been observed in irradiated p-type Cz-Si samples, possesses the properties of a defect with negative- U . Evidence for this can be obtained from the analysis of temperature dependences of electron occupancy of the centre. It was shown earlier that the occupancy function of negative- U defects differs from the Fermi function, which describes the electron population of defects with $U > 0$ [19, 21, 22]. The analysis of the equilibrium $p(T)$ dependences in irradiated p-type Cz-Si samples shows that the changes in the carrier density in the temperature range 225–275 K are related to the changes in the hole occupation of the $E(0/++)$ energy level of a defect with negative- U properties. The $E(0/++)$ level of the defect was determined to be at $E_v + 0.255 \text{ eV}$. It should be pointed out that the phenomenon of residual conductivity and the defect with the level at $E_v + 0.255 \text{ eV}$ have not been observed in carbon-rich ($N_C \geq 2 \times 10^{16} \text{ cm}^{-3}$) p-type Cz-Si samples irradiated with electrons at room temperature.

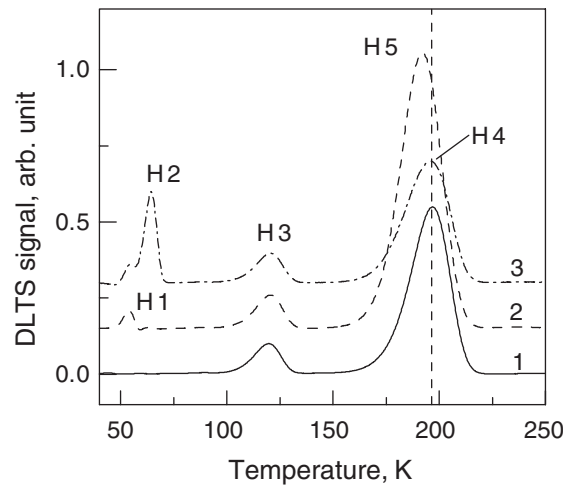


Figure 4. DLTS spectra for boron-doped Cz-Si samples with (1) moderate ($N_C \approx 5 \times 10^{16} \text{ cm}^{-3}$) and (2, 3) low ($N_C \leq 2 \times 10^{15} \text{ cm}^{-3}$) carbon content after irradiation with 4 MeV electrons. The dose of irradiation was $4 \times 10^{14} \text{ cm}^{-2}$. Measurements were carried out upon heating the sample from 35 to 300 K with the following measurement settings: $e_n = 80 \text{ s}^{-1}$, bias $-5 \rightarrow -0.2 \text{ V}$, and pulse duration 1 ms. Spectra 1 and 2 were recorded after cooling samples from room temperature to 35 K without reverse bias applied to the Schottky barriers; spectrum 3 was recorded after cooling down with the reverse bias on.

3.2.2. DLTS and Laplace DLTS measurements. Figure 4 shows conventional DLTS spectra for an electron-irradiated p-type Cz-Si sample with low carbon content (spectra 2 and 3). For a comparison a spectrum for electron-irradiated B-doped Cz-Si material with a carbon concentration of about $5 \times 10^{16} \text{ cm}^{-3}$ is also shown (spectrum 1). Samples from this material will be referred to as carbon-rich samples. The spectra were recorded upon heating the sample up from 35 to 300 K. All the DLTS peaks seen in the spectra and referred to as H1–H5 signals are related to radiation-induced defects. In addition to the H3 and H4 signals, which are observed in the spectrum of the carbon-rich sample, three more DLTS peaks have been observed in the spectra for the carbon-lean sample. It has appeared that the cooling down conditions (cooling down with reverse or zero bias applied to a diode under test) affect the DLTS spectra of electron-irradiated carbon-lean Cz-Si samples very significantly but not the spectra of the carbon-rich ones. The DLTS spectra for the electron-irradiated carbon-rich sample recorded after different cooling procedures are identical and, therefore, only one DLTS spectrum for this sample (spectrum 1) is shown in figure 4. However, changes can be observed in the DLTS spectra for samples with low carbon content recorded after different cooling procedures (spectra 2 and 3). Cooling down with the bias on resulted in a decrease of the DLTS peak with a maximum at about 195 K and the appearance of a peak with its maximum at about 65 K. It should be noted that a shift in temperature of peak maximum for the peak at about 195 K has been observed after different cooling procedures. This indicates that the peak at about 195 K in spectrum 2 consists of two traps with different emission rates.

Application of Laplace DLTS enables us to resolve emission signals from two traps contributing to the DLTS peak at about 195 K in spectrum 2. Figure 5 shows the corresponding LDLTS spectra.

Figure 6 shows Arrhenius plots of T^2 -corrected hole emission rates from the traps responsible for the DLTS peaks seen in figure 4. Trap parameters, the activation energy for

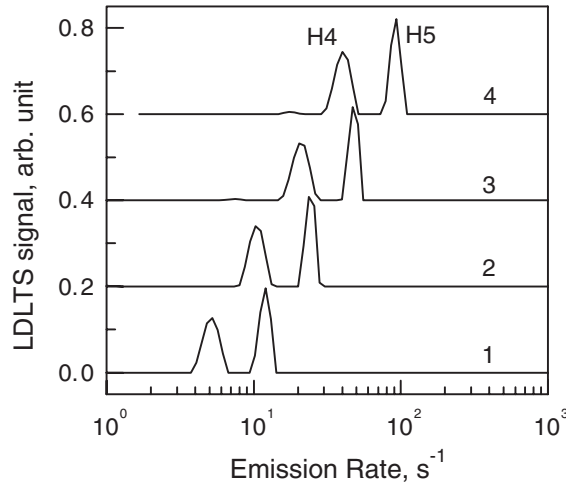


Figure 5. Laplace DLTS spectra for a boron-doped Cz-Si sample with low ($N_C \leq 2 \times 10^{15} \text{ cm}^{-3}$) carbon content after irradiation with 4 MeV electrons at room temperature. The dose of irradiation was $4 \times 10^{14} \text{ cm}^{-2}$. Measurement settings were bias $-5 \rightarrow -0.2 \text{ V}$ and pulse duration 1 ms. The sample was cooled down from room temperature to the measurement temperatures without a reverse bias applied to the Schottky barrier. The spectra were measured at: (1) 175 K, (2) 180 K, (3) 185 K and (4) 190 K.

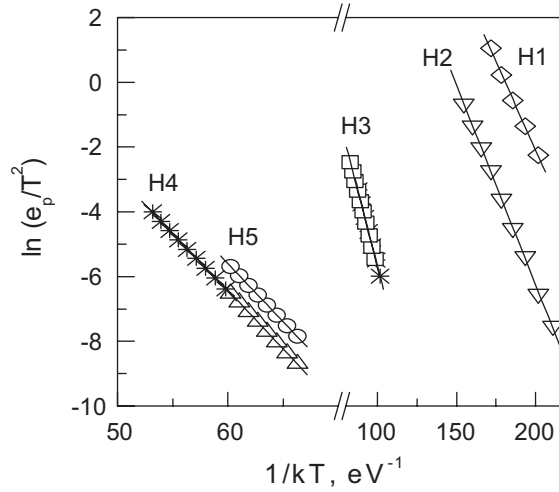


Figure 6. Arrhenius plots of T^2 -corrected hole emission rates from radiation-induced defects in electron-irradiated boron-doped Cz-Si samples. Open symbols represent the data for a carbon-lean Cz-Si sample and stars those for a Cz-Si sample with $N_C \approx 5 \times 10^{16} \text{ cm}^{-3}$. Solid lines represent the dependences calculated on the basis of the equation $e_p/T^2 = A_p \exp(-E_p/kT)$ with parameters determined from least-squares fits to experimental data.

hole emission and pre-exponential factor, determined from the Arrhenius plots, are presented in table 1. On the basis of comparison of the trap parameters with those known from the literature of electron-radiation-induced defects in Cz-Si [15, 23, 24] the H3 and H4 traps have been assigned to the first donor level of the divacancy [25] and interstitial carbon–interstitial oxygen pair [26, 27], respectively.

Table 1. Activation energies and pre-exponential factors of hole emission for radiation-induced defects in p-type Cz-Si crystals with low carbon content. The parameters were derived from least-squares fits of experimentally determined temperature dependences of hole emission rates (e_p) to the equation $e_p/T^2 = A_p \exp(-\Delta E_p/kT)$. The e_p values were determined from Laplace DLTS measurements.

Trap label	ΔE_p (eV)	A_p (s ⁻¹ K ⁻²)	Assignment
H1	0.109	3.8×10^8	?
H2	0.123	9.3×10^7	IO ₂ (0/+)
H3	0.190	6.0×10^5	V ₂ (0/+)
H4	0.360	4.0×10^6	C _i O _i (0/+)
H5	0.358	7.8×10^6	IO ₂ (+/++)

The LDLTS spectra in figure 5 have been recorded after cooling the sample from room temperature to 180–200 K with reverse bias off. There have been no significant changes in the magnitudes of the lines seen in the spectra in figure 5 after long storage of the sample in the temperature range 180–210 K with reverse bias on. Only when the sample has been heated to 225 K and kept at this temperature with the bias on is a decrease in the magnitude of the H5 line observed. Only the H4 line has been observed in LDLTS spectra after cooling the sample from room temperature to 180–200 K with reverse bias on. Instead of the H5 line an LDLTS signal from the H2 trap has appeared when the sample has been further cooled down to 60–75 K with the bias on. It should be noted also that the intensity of the H5 LDLTS signal is about twice that due to the H2 signal. So, it is inferred from the analysis of the DLTS and LDLTS spectra that the H2 and H5 DLTS signals are related to hole emission from two different configurations of the same centre. Further, it appears that the H5 signal consists of emission of two holes from the centre, a type of behaviour that is characteristic of DLTS signals from defects with negative- U properties [28, 29].

3.2.3. Combined analysis of Hall effect and DLTS measurements. The H2/H5 traps disappear in the temperature range 90–120 °C upon 30 min isochronal annealing. The annealing behaviour of the H2/H5 traps has been found to be very similar to that of the negative- U centre with the energy level at $E_v + 0.255$ eV, which has been observed in Hall effect measurements on electron-irradiated carbon-lean Cz-Si samples.

From the combined analysis of the Hall effect and DLTS data the conclusion has been drawn that the $E(0/++)$ energy level at $E_v + 0.255$ eV and H5/H2 DLTS signals are related to the same bistable defect. The H5 DLTS signal consists of the emission of two holes: the first hole is emitted from the doubly positively charged state of the defect configuration A, which is stable at low (<200 K) temperatures, and the second hole emission from the singly positively charged state of the defect follows the emission from the A^{++} state practically immediately, as the activation energy for the emission of the second hole (0.123 eV) is lower than the ΔE_p value for the emission of the first hole (0.358 eV). The H2 DLTS signal is related to hole emission from the metastable positively charged state of another defect configuration, B. The neutral B^0 state is the minimum energy state of the defect at higher (>250 K) temperatures. The energy difference between A^{++} and B^0 is about 0.51 eV (2×0.255 eV).

4. Discussion and conclusions

Carbon and group III impurity atoms are known to interact effectively with self-interstitial atoms in crystalline silicon at room temperature [30]. The interaction is believed to occur via

the Watkins replacement mechanism when the group III impurities or substitutional carbon are ejected into interstitial sites [30, 31]. In Si crystals with low concentration of these impurities, interactions of self-interstitials with other traps can be expected. Indeed, it has been found in the present work that there is a significant difference between the spectra of energy levels due to defects introduced by electron irradiation at room temperature into Cz-Si crystals with low ($\leq 2 \times 10^{15} \text{ cm}^{-3}$) and high ($> 10^{16} \text{ cm}^{-3}$) carbon content.

In n-type carbon-lean Cz-Si irradiated at room temperature, a centre with an acceptor level at $E_C - 0.11 \text{ eV}$ is one of the dominant radiation-induced defects. This centre has not been observed after irradiation in Cz-Si crystals with $N_C > 2 \times 10^{16} \text{ cm}^{-3}$. The centre anneals out in the temperature range 100–130 °C with an activation energy of 1.35 eV.

In p-type Cz-Si crystals with low carbon content and boron ($N_B \leq 2 \times 10^{14} \text{ cm}^{-3}$) one of the dominant radiation-induced defects has been found to be a bistable centre with an energy level $E(0/++) = E_v + 0.255 \text{ eV}$ ($H_{0.255}$). This defect anneals out in the temperature range 90–120 °C upon 30 min isochronal annealing.

As has been mentioned in the introduction, there is some evidence that interstitial-oxygen-related defects (O_i , O_{2i} , O_{3i} , etc) can interact with self-interstitials, and a number of I_nO_m ($n \leq 2$, $m \leq 3$) complexes have been observed by infrared absorption technique. It is reasonable to suggest that one or more of these complexes can be related to the $E(-/0) = E_C - 0.11 \text{ eV}$ and $E(0/++) = E_v + 0.255 \text{ eV}$ energy levels. We believe that the complex consisting of one self-interstitial atom and the oxygen dimer (IO_{2i}) is responsible for the above energy levels. The IO_i cannot be responsible for the above levels, as it anneals out below 270 K [1–5]. The concentration of oxygen trimers is rather low ($\leq 10^{13} \text{ cm}^{-3}$) in Cz-Si samples which have been pre-annealed at 650–700 °C before irradiation [32]; therefore, the probability of formation of the IO_{3i} complex should be rather low. On the other hand, the concentration of oxygen dimers is about $(1-2) \times 10^{15} \text{ cm}^{-3}$ in the Cz-Si crystals used [11, 12], and the dimers can compete with substitutional carbon atoms in capturing self-interstitials created by electron irradiation. The temperature range of thermal stability of the $E(-/0) = E_C - 0.11 \text{ eV}$ and $E(0/++) = E_v + 0.255 \text{ eV}$ energy levels coincides with that for the centre with LVM lines at 922 and 1037 cm^{-1} , which was identified earlier as the IO_{2i} complex [8].

In summary, it is argued in the present work that oxygen dimers are effective traps for Si self-interstitials in Cz-Si crystals with low carbon content upon electron irradiation at room temperature. The resulting IO_{2i} complex can exist in four charge states (IO_{2i}^- , IO_{2i}^0 , IO_{2i}^+ , and IO_{2i}^{++}) and possess the $E(-/0) = E_C - 0.11 \text{ eV}$ and $E(0/++) = E_v + 0.255 \text{ eV}$ energy levels. Preliminary *ab initio* calculations [13] showed that the electronic characteristics of a complex incorporating a staggered oxygen dimer and $\langle 110 \rangle$ -split silicon self-interstitial are consistent with those for a defect possessing the above-mentioned energy levels.

Acknowledgments

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