CALIBRATIONS COEFFICIENTS FOR DETERMINATION OF CONCENTRATIONS OF VACANCY-OXYGEN-RELATED COMPLEXES AND OXYGEN DIMER IN SILICON BY MEANS OF INFRARED ABSORPTION SPECTROSCOPY

Abstract. Vacancy-oxygen complexes $V_nO_m$ ($n, m \geq 1$) in crystalline silicon are nucleation centers for oxygen precipitates, which are widely used as internal getters in modern technologies of production of silicon-based electronic devices and integrated circuits. For the controllable formation of oxygen precipitates in Si crystals in the technology processes the methods of determination of concentrations of the $V_nO_m$ complexes are required. The aim of the present work was to find values of the calibration coefficients for determination of concentrations of the $V_nO_m$ defects in Si from intensities of infrared (IR) absorption bands associated with the local vibrational modes (LVM) of these complexes. A combined electrical (Hall effect) and optical (IR absorption) study of vacancy-oxygen defects in identical silicon crystals irradiated with 6 MeV electrons was carried out. Based on the analysis of the data obtained, the values of the calibration coefficient for the determination of concentration of the vacancy-oxygen (VO) complex in silicon by the infrared absorption method were established: for measurements at room temperature (RT) – $N_{VO} = 8.5 \cdot 10^{16} \cdot \alpha_{VO-RT}$ cm$^{-3}$, in the case of low-temperature (LT, $T \equiv 10$ K) measurements – $N_{VO} = 3.5 \cdot 10^{16} \cdot \alpha_{VO-LT}$ cm$^{-3}$, where $\alpha_{VO-RT(LT)}$ are absorption coefficients in maxima of the LVM bands due to the VO complex in the spectra measured at corresponding temperatures. Calibration coefficients for the determination of concentrations of other $V_nO_m$ ($VO_2$, $VO_3$, $VO_4$, $V_2O$ and $V_3O$) complexes and the oxygen dimer ($O_2$) from an analysis of infrared absorption spectra measured at room temperature have been also determined.

Keywords: silicon, vacancy-oxygen complexes, oxygen dimers, vibrational absorption bands, calibration coefficients

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Oxygen is one of the most abundant and technologically important impurities in silicon [1]. Oxygen concentration in silicon crystals grown by the Czochralski technique (Cz-Si) is in the range of $(5–10) \cdot 10^{17}$ cm$^{-3}$, often exceeding concentrations of other impurity atoms. In as-grown Si crystals oxygen is mainly present in the form of interstitial $(O_i)$ atoms [1]. The $O_i$ concentration in crystalline Si is usually determined from room temperature (RT) measurements of the intensity of the infrared (IR) absorption band at 1107 cm$^{-1}$ [1, 2] with the use of the following equation: $[O_i] = K_{O_i} \cdot \alpha_{1107}$ cm$^{-3}$, where $K_{O_i}$ is a calibration coefficient and $\alpha_{1107}$ is an absorption coefficient at the maximum of the band at 1107 cm$^{-1}$. The most widely used value of the calibration coefficient is $K_{O_i} = 3.14 \cdot 10^{17}$ cm$^{-2}$ [2, 3].

Although interstitial oxygen atoms in Si are electrically neutral and immobile at RT, they effectively interact with intrinsic lattice defects, vacancies and self-interstitials, other impurity atoms and themselves upon radiation and thermal treatments, so contributing to the formation of a large variety of electrically and optically active oxygen-related radiation- and thermally-induced defects [1, 2]. Nearly all these oxygen-related centers give rise to local vibrational mode (LVM) lines, which can be detected by the IR absorption spectroscopy [2, 4]. Fourier-transform infrared (FT-IR) absorption spectroscopy has been successfully used in the studies of many oxygen-related aggregates including vacancy-oxygen-related complexes $V_{n}O_{m}$ ($n = 1–3$, $m = 1–4$) [2, 4–10]. Among the $V_{n}O_{m}$ defects only the VO complex (A-center) [11] and the VO$_2$ complex being in a metastable state [8] can be detected by electrical measurements in $n$-type Si crystals. On the other hand, absorption lines due to many of the $V_{n}O_{m}$ defects have been identified in the FT-IR absorption spectra [2]. For prediction of changes in electrical and optical properties of Cz-Si crystals upon radiation and thermal treatments it is necessary to know the absolute values of defect concentrations. However, the available information on calibration coefficients for the determination of concentrations of the vacancy-oxygen related complexes by IR absorption is very limited [12–14].

In 1986 Oates and Newman [12] determined the calibration coefficients for determination of the VO concentration from magnitude of the VO related absorption peak at 830 cm$^{-1}$ (measurements at RT). According to their work, $[\text{VO}] = (6.1 \cdot 10^{16}) \cdot \alpha_{830}$ cm$^{-3}$, where $\alpha_{830}$ is an amplitude of the vibrational absorption band at 830 cm$^{-1}$. Oxygen-rich Si samples doped with tin and irradiated with 2 MeV electrons at RT were studied in the work. After the irradiation the main vacancy-related defects were tin-vacancy complexes. Upon further annealing at about 160 °C these complexes started to dissociate and the released vacancies were captured by O$_i$ atoms to form VO defects. The concentration of removed O$_i$ was measured and assumed to be equal to the concentration of the generated VO complexes. Such procedure seems to be rather reliable. A very similar calibration coefficient for the determination of the VO concentration in Si was later obtained by Davies et al. [13] in their work on modeling the generation kinetics of radiation-induced defects. However, in both cases [12, 13] the IR absorption measurements were carried out with a rather low spectral resolution, the measured full width at half of maximum (FWHM) of the 830 cm$^{-1}$ band was about 9 cm$^{-1}$ in the spectra presented. The actual FWHM of this band in the spectra measured at RT is less than 6 cm$^{-1}$ (about 5.3 cm$^{-1}$ [9, 15]) and
therefore the values of the calibration coefficients determined in Refs. [12] and [13] for the 830 cm\(^{-1}\) line are questionable. IR absorption measurements with a higher resolution are required for the correct determination of an absorption coefficient for this line.

In the present work, in order to determine the calibration coefficient for calculation of concentration of the VO complex from intensity of the absorption line due to this defect, direct measurements of the concentration of A centers by the Hall effect method were carried out, and IR absorption spectra were measured on identical samples irradiated with the same doses of 6 MeV electrons. Furthermore, based on the consideration of positions and geometry of oxygen atoms in the lattice for a number of other vacancy-oxygen related centers and the oxygen dimer the calibration coefficients for determination of concentrations of these defects from infrared absorption measurements at room temperature are proposed.

**Experimental details.** We have used two similar sets of oxygen-rich Czochralski-grown phosphorus-doped n-type Si crystals with a resistivity of about 1 \(\Omega\)·cm. Both sets were irradiated with 6 MeV electrons at RT with the same doses in the range of \((1–5) \cdot 10^{16}\) cm\(^{-2}\).

The VO concentration (concentration of the defect with \(E_C – 0.18\) eV energy level in the gap) in one set of samples was determined by an analysis of the temperature dependences of free carriers, \(n\), obtained by means of the Hall effect measurements in the temperature range 77–400 K. The dependences of \(n\) were calculated taking into account the temperature dependence of the Hall factor and analyzed by the application of the conventional method based on the solution of the electrical neutrality equation [16]. Besides, to determine the parameters of radiation-induced defects in the samples used, the differential method [17] has been also used, according to which a position of the energy level \((E_D)\) and the concentration of a defect \((N)\) can be determined from the dependence of the value \(Y = kT(dn/dF)\) on \(F = E_C − E_F\), where \(E_F\) is the Fermi level position. Ionization of a defect results in appearance of a peak in the \(Y(F)\) dependence, and if the levels of various defects are located far enough from each other, then multiple peaks occur in the \(Y(F)\) dependences. For ordinary defects with positive values of electron correlation energy \((U > 0)\), the ordinate of the band maximum is \(Y_{max} = 1/4\ N\), where \(N\) is concentration of a defect, and the abscissa of the band maximum, \(F_{max}\), corresponds to the energy level position in the band gap [17].

Another set of samples was used for FTIR absorption measurements. For optical measurements the samples were polished to an optical surface on two sides and the dimensions were 10×6×3 mm\(^3\). The IR absorption spectra in the wavenumber range 400–4000 cm\(^{-1}\) were measured using a Bruker IFS 113v spectrometer at temperatures of 20 and 300 K with a spectral resolution of 0.5 or 1 cm\(^{-1}\), respectively. The concentrations of interstitial oxygen ([O\(_i\)] = 1·10\(^{18}\) cm\(^{-3}\)) and substitutional carbon ([C\(_s\]) = 2·10\(^{16}\) cm\(^{-3}\)) atoms were determined from measurements of intensities of absorption bands at 1107 and 605 cm\(^{-1}\) using calibration coefficients 3.14·10\(^{17}\) cm\(^{-2}\) and 0.94·10\(^{17}\) cm\(^{-2}\), respectively [2, 3].

Absorption coefficients of the bands due to vacancy-oxygen complexes were determined by fitting the relevant absorption lines with Lorentzian components with contributions due to silicon isotopes (\(^{29}\)Si and \(^{30}\)Si) being taken into account [15, 18].

**Results and discussion.** Figure 1 shows, as an example, the temperature dependences of the free electron concentration \((n(T))\) measured for an as-grown Si sample (curve 1), and a sample irradiated at room temperature to a dose of 1.3·10\(^{16}\) cm\(^{-2}\) (curve 2) and further annealed at 150 °C for 30 min (curve 3). It is known that irradiation of moderately doped n-type Cz-Si crystals with fast electrons results in the formation of vacancy-oxygen and vacancy-phosphorus centers, divacancies (V\(_2\)) and defects with an energy level at \(E_C – 0.125\) eV, which is related to a complex consisting of the Si self-interstitial atom and two oxygen atoms, IO\(_2\) [19, 20].

A precise determination of the A-center concentration by an analysis of the \(n(T)\) dependences measured after irradiation is rather difficult because of the presence of divacancies (the second acceptor level of V\(_2\) is at \(E_C – 0.21\) eV [21–23]) and the IO\(_2\) defect. Divacancies contribute to the value of the total concentration of defects with energy levels in the range \(E_C – 0.18–0.21\) eV and because of the close positions of the VO(–/0) and V\(_2\)(–2/–) levels a careful analysis of the experimental \(n(T)\) dependences is required for the separation of contributions of the VO and V\(_2\) defects. The IO\(_2\) defect is known to annihilate upon heat-treatments in the temperature range of 100–125 °C. To eliminate the contribution of the IO\(_2\) defect into the \(n(T)\) dependences, the 30-min annealing was performed at 150 °C.
After that we have carried out an analysis of the measured \( n(T) \) dependences (Figure 1, curve 3), taking into account the well-known position of the A-center level, \( E_C - 0.18 \text{ eV} \) [21]. The concentration of A-centers determined from an analysis of the \( n(T) \) dependence was found to be \( N_A = (3.05 \pm 0.2) \times 10^{15} \text{ cm}^{-3} \).

In another way the concentration of A-centers can be determined from the differential \( Y(F) \) dependences [17]. The \( Y(F) \) dependence presented in Figure 2 for the Si sample irradiated with electrons and annealed at 150 °C indicates the presence of only one peak with its maximum at about \( E_C - 0.18 \text{ eV} \). The contribution of divacancy results only in a slight change of the peak shape. Based on the fact that for ordinary defects the ordinate of maximum \( Y_{\text{max}} = 1/4 N \), the value of the concentration of the radiation-induced A-centers was found to be \( N_A = (3.15 \pm 0.1) \times 10^{15} \text{ cm}^{-3} \). The average value of the A-center concentration deduced from the analysis of the data obtained by means of electrical measurements for a few Cz-Si samples irradiated with 6 MeV electrons to a dose of \( 1.3 \times 10^{16} \text{ cm}^{-2} \) is found to be \( N_A = (3.1 \pm 0.1) \times 10^{15} \text{ cm}^{-3} \).

The absorption bands related to VO in the neutral charge state (lines with their maxima at 830 and 836 cm\(^{-1}\) in the spectra measured at room temperature and low temperature, respectively) as well as in the negative charge state (lines at 877 and 885 cm\(^{-1}\) in the RT and LT spectra, respectively [2]) were detected in the absorption spectra recorded at 20 K and room temperature (Figure 3). It is found that the values of half-width at half of the maximum of the bands due to VO\(_0\) and VO\(^{-}\) are very close and are equal to 2.35 cm\(^{-1}\) and 5.3 cm\(^{-1}\) in the low-temperature and room-temperature spectra, respectively. The sum of the absorption coefficients of the lines at 836 and 885 cm\(^{-1}\) (spectrum 1 in Figure 3) has been determined as 0.089 cm\(^{-1}\), while the sum of absorption coefficients of the lines at 830 and 877 cm\(^{-1}\) (spectra 2 and 3 in Figure 3) is 0.036 cm\(^{-1}\).

The combined analysis of the obtained electrical and optical data has allowed us to deduce the following values of calibration coefficient for the determination of the VO concentration in silicon by IR absorption: RT measurements – \([\text{VO}] = 8.5 \times 10^{16} \cdot (\alpha_{830} + \alpha_{877}) \text{ cm}^{-3}\), LT measurements (≤ 20 K) – \([\text{VO}] = 3.5 \times 10^{16} \cdot (\alpha_{836} + \alpha_{885}) \text{ cm}^{-3}\).

We have also estimated the calibration coefficients for determination of concentration of the VO complex from the values of integrated absorption coefficient (IA, area under absorption bands) due to the VO related absorption lines. It has been found that values of the integrated absorption due to the VO related bands are very close in the RT and LT absorption spectra, about 0.21 cm\(^{-2}\) for the samples irradiated to a dose of \( 1.3 \times 10^{16} \text{ cm}^{-2} \). So, we can alternatively write \([\text{VO}] = 1.5 \times 10^{16} \cdot \text{IA}_{\text{VO}} \text{ cm}^{-3}\), where \( \text{IA}_{\text{VO}} \) is the integrated absorption coefficient of the bands due to the VO defect.

Figure 1. Temperature dependences of carrier concentration in Cz-Si crystal: 1 – before irradiation, 2 – after irradiation with 6 MeV electrons at \( T = 293 \text{ K} \) to a dose of \( 1.3 \times 10^{16} \text{ cm}^{-2} \), 3 – after 30-min annealing at \( T = 150 \text{ °C} \).

Figure 2. Dependence of the quantity \( Y = kT(dn/dF) \) on \( F = E_C - E_F \), obtained by a numerical differentiation of the dependence 3, shown in Figure 1.
Based on the calibration data obtained for the VO center and assuming that the oscillator strength of the oxygen atom vibrations as well as the shape of absorption bands are the same for the oxygen atoms located in the Si vacancy with different environments we have also estimated calibration coefficients for the determination of concentrations of the VO$_2$ (LVM related band with its maximum at 889 cm$^{-1}$ in the RT absorption spectra [2, 7]), VO$_3$ (bands at 905 and 969 cm$^{-1}$ [7]), VO$_4$ (band at 985 cm$^{-1}$ [7]), V$_2$O (band at 826 cm$^{-1}$ [15]) and V$_3$O (band at 839 cm$^{-1}$ [15]) complexes in silicon by infrared absorption measurements at room temperature. The concentrations of the above mentioned complexes can be determined as: [VO$_2$] = 4.25 · 10$^{16}$·$\alpha_{889}$ cm$^{-3}$, [VO$_3$] = 8.5 · 10$^{16}$·($\alpha_{905} + \alpha_{969}$)/2 cm$^{-3}$, [VO$_4$] = 4.25 · 10$^{16}$·$\alpha_{985}$ cm$^{-3}$, [V$_2$O] = 8.5 · 10$^{16}$·$\alpha_{826}$ cm$^{-3}$, [V$_3$O] = 8.5 · 10$^{16}$·$\alpha_{839}$ cm$^{-3}$.

The oxygen dimer (O$_2$) is expected to play a key role in a number of oxygen diffusion and precipitation phenomena [1, 4, 24], including the formation of the center responsible for the light-induced degradation of silicon-based solar cells [25–28]. The control of concentration of the dimer is required for prediction of changes in electrical and optical properties of Si:O crystals upon different radiation- and heat-treatments. In the most stable “staggered” configuration [29, 30] the oxygen dimer gives rise to two LVM-related absorption bands with their maxima at 1012 and 1060 cm$^{-1}$ in the spectra measured at LT and at 1013 and 1062 cm$^{-1}$ in the RT spectra [2, 24, 31]. The configurations of two oxygen atoms in the staggered configuration resemble those for the single interstitial oxygen atoms [29, 30], so, it can be expected that the oscillator strengths for vibrations of each oxygen atom involved in O$_2$ are close to that for the O$_i$ atom. However, the calibration coefficient $K_{\alpha} = 3.14 · 10^{17}$ cm$^{-3}$ cannot be used for calculations of the dimer concentration in the same way as for single interstitial oxygen atoms, because of different HWHM values for the bands due to O$_i$ and O$_2$. In the absorption spectra measured at room temperature the HWHM value of the band at 1107 cm$^{-1}$ due to O$_i$ is 34 cm$^{-1}$ [2], while that of the band at 1013 cm$^{-1}$ due to O$_2$ is 7.8 cm$^{-1}$ [2, 24]. For calculation of the dimer concentration it is necessary to use the calibration coefficient for integrated absorption of the O$_2$-related band at 1107 cm$^{-1}$. This coefficient has been determined as $K_{1A-O_2} = 0.94 · 10^{16}$ cm$^{-1}$ [32]. Otherwise, it is possible to use the value of absorption coefficient of the O$_2$-related band at 1013 cm$^{-1}$ and $K_{\alpha-O_2} = 3.14 · 10^{17}$ cm$^{-3}$, but the obtained value should be corrected by the ratio of HWHM values of the 1013 and 1107 cm$^{-1}$ absorption bands. This approach gives [O$_2$] = 3.14 · 10$^{17}$·(7.8/34)·$\alpha_{1013} = 7.2 · 10^{16}$·$\alpha_{1013}$ cm$^{-3}$.

Conclusions. From a comparison of the data obtained by means of electrical and optical measurements the following expressions for determining the concentration of A-centers in silicon by IR absorption have been derived: for measurements at room temperature $N_{VO} = 8.5 · 10^{16}$·($\alpha_{830} + \alpha_{877}$) cm$^{-3}$, in the case of low-temperature measurements, $N_{VO} = 3.5 · 10^{16}$·($\alpha_{836} + \alpha_{885}$) cm$^{-3}$. The calibrations coefficients for determination of concentration of a number of more complex vacancy-oxygen related defects (VO$_2$, VO$_3$, VO$_4$, V$_2$O and V$_3$O) and the oxygen dimer from optical absorption measurements at room temperature are proposed.

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