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Production of vacancy-oxygen defect in electron irradiated silicon in the presence of self-interstitial-trapping impurities

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The enhancement by carbon of vacancy-oxygen (VO) defect formation in electron irradiated silicon was investigated using many samples of various carbon contents. The effect of carbon is well described by a simple analytical model of competing trapping of self-interstitials by VO and by carbon (and by emerging carbon-related defects like C_iO_i and IC_iO_i). The trapping ratio by C_s and by VO was determined to be about 0.9, and the optical calibration coefficients for C_iO_i and IC_iO_i were deduced. In crystals containing a high concentration of Ge, germanium also acts as a self-interstitial trapping impurity and, thus, enhances VO production. The trapping efficiency of Ge is 1000 times less than that of carbon. © *2011 American Institute of Physics*. [doi:10.1063/1.3657946]

I. INTRODUCTION

Irradiation-produced vacancies (V) in Czochralski-grown (CZ) silicon are known[1–3] to be quickly trapped by oxygen that is initially present as an electrically inactive interstitial species (O_i). The electrical, optical, and electronic properties of the resulting vacancy-oxygen (VO) defect (also called the A-centre) have been studied[2–10] intensively over the years. The defect has an acceptor level at $E_c - 0.17$ eV. It is optically active, with a local vibration mode (LVM) band at 830 cm^{-1} for the neutral charge state at room temperature (RT).

Since self-interstitials (I) are simultaneously created in the same amount as V, a production of VO in an appreciable concentration is possible only in the presence of some traps for I—otherwise VO would be almost completely annihilated by the $I + VO$ reaction. The carbon impurity (initially in an electrically inactive substitutional state C_s) is well known[11–16] as an efficient trap for I. The emerging interstitial carbon atoms (C_i) are trapped by oxygen and carbon producing C_iO_i and C_iC_s defects that further trap self-interstitials. In lightly doped material, carbon is most likely the major trap for I, and hence, the concentration of VO is expected to be controlled by the carbon concentration $[C_s]$ (square brackets are used to denote the concentration of a species). Indeed, it was noted[17] that, at sufficiently high irradiation dose, [VO] is higher in a sample of a higher $[C_s]$. The goal of the present work is to analyze the carbon effect quantitatively, using a database of many samples of different $[C_s]$.

Another impurity that apparently enhances VO production is germanium.[18] An additional goal of the present work is to separate the contributions of Ge and C into self-interstitial trapping.

II. EXPERIMENTAL DETAILS

The Cz-Si samples used in this study were about 2 mm thick. The oxygen concentration was deduced from the intensity of the 1107 cm^{-1} band (with a calibration coefficient of $2.45 \times 10^{17} \text{cm}^{-2}$) and the carbon concentration from the 605 cm^{-1} band (with a calibration coefficient of $1 \times 10^{17} \text{cm}^{-2}$). These concentrations—before irradiation—are listed in the third and fourth columns of Table I; the measured substitutional carbon concentration is identical to the total carbon content and denoted [C]. Due to irradiation, the concentration of substitutional carbon is significantly reduced; this value—denoted $[C_s]$—is shown in the fifth column. For oxygen, a reduction is relatively weak and not indicated. The samples Ge1 and Ge4 are doped with Ge (1×10^{17} and 4×10^{18} cm^{-3}, respectively). The effect of the Ge impurity as a trap for self-interstitials is discussed below; it is significant only at $[\text{Ge}] > 3 \times 10^{19}$ cm^{-3}. The sample Sn3.1 contains $3 \times 10^{17} \text{cm}^{-3}$ of tin—an impurity that is known[19] to compete with O_i in trapping V. Since $[\text{Sn}] < [O_i]$ in this sample, vacancy trapping is still mostly by oxygen although some reduction in [VO] may occur. This sample was included into the database, because it is the only available sample with a carbon content intermediate between high and medium. All samples were boron-doped ($[\text{B}] \leq 2.5 \times 10^{15}$ cm^{-3}) with the exception of the Sn-containing sample which was lightly doped with phosphorus ($[\text{P}] = 6 \times 10^{13}$ cm^{-3}).

The samples were irradiated with 2 MeV electrons, using the Dynamitron accelerator at Takasaki-JAERI (Japan). The temperature of irradiation was about 70 °C. The samples are divided into three groups of irradiation dose. The first group, of four samples, was irradiated at a dose $\phi = 5 \times 10^{17}$ cm^{-2}, the second group, of three samples—at $\phi = 10^{18}$ cm^{-2}, and the third group, of two samples—at 2×10^{18} cm^{-2}. The IR spectra were recorded at RT, with an

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TABLE I. Characteristics of electron-irradiated samples. The concentrations [O_i] of oxygen and [C] of carbon are measured before irradiation. The other columns refer to the post-irradiation state.

Sample	Dose (10^{18} cm^{-2})	[O_i] (10^{17} cm^{-3})	[C] (10^{16} cm^{-3})	[C_s] (10^{16} cm^{-3})	[VO] (10^{16} cm^{-3})	A_{862} (cm^{-1})	A_{1020} (cm^{-1})
Ge1	0.5	7.5	2	1.5	3.4	0.14	0.07
Ge4	0.5	4.3	10	4.9	3.6	0.26	0.08
E3	0.5	8	16	9.6	3.9	0.34	0.06
E1	0.5	5.3	75	60	4.8	0.38	0.03
M4	1	7.4	5	1	5.9	0.28	0.07
H3	1	7.3	22	13.2	6.2	0.53	0.12
Sn3.1	1	7.5	47	18	7.3	0.63	0.13
M7	2	8.2	4	2.3	7.2	0.09	0.09
M5	2	7.4	8.8	3.9	9.8	0.46	0.24

FTIR spectrometer of 1 cm^{-1} resolution. The two phonon intrinsic absorption was always subtracted by using a float-zone sample of the same thickness.

III. EXPERIMENTAL RESULTS AND DISCUSSION

Fig. 1 shows RT absorption spectra of irradiated samples, in the wavenumber range of 530–1060 cm^{-1}, for three representative carbon concentrations: high, medium, and low (samples E1, E3, and M7 of Table I). The most pronounced irradiation-induced bands are those of VO and C_iO_i. There are also weaker bands due to IC_iO_i—a defect formed by $I + C_iO_i$ reaction. A band due to the di-carbon defect C_iC_s is very weak and near the detection limit. The bands due to C_i are not seen at all—since at the irradiation temperature of 70 °C the produced C_i are immediately trapped, mostly by O_i. By comparing the samples E1 and E3 (of the same dose), it appears that the VO band is stronger at higher [C_s]. The sample M7 in Fig. 1 has received the highest dose, and the VO band is strong, in spite of a low [C_s].

The VO concentration listed in Table I was calculated using a calibration coefficient[20] of 6.25×10^{16} cm^{-2} for the 830 cm^{-1} band at RT. This calibration was coupled[20] to the oxygen calibration assumed in that work which was the same as that adopted above. Were the oxygen calibration to be changed (for instance, to a frequently used value of 3.14×10^{17} cm^{-2}), then the VO calibration coefficient would be increased proportionally to 8×10^{16} cm^{-2}. The two last columns of Table I show the intensity of larger bands due to the C_i-related defects: C_iO_i (at 862 cm^{-1}) and IC_iO_i (at 1020 cm^{-1}).

A. Analytical model of VO production in the presence of carbon traps for self-interstitials

The accumulation of VO and of accompanying carbon defects C_iO_i, C_iC_s and subsequently formed clusters (such as IC_iO_i and larger ones) in dependence of the irradiation dose was numerically simulated[11,17,21–23] assuming many trapping reactions for V and I and thus using a large number of fitting parameters. This problem can be, however, solved analytically, with several reasonable simplifications:

- neglecting the di-carbon defect C_iC_s (as mentioned above, the band due to C_iC_s is always weaker than those of carbon and of carbon-oxygen species).
- assuming the same kinetic coefficient for trapping of I by all the carbon-related traps C_s, C_iO_i, IC_iO_i, etc.
- neglecting production of divacancies V_2—justified by a relatively low ratio[24] of the production rates of V_2 and V by 2 MeV electron irradiation.

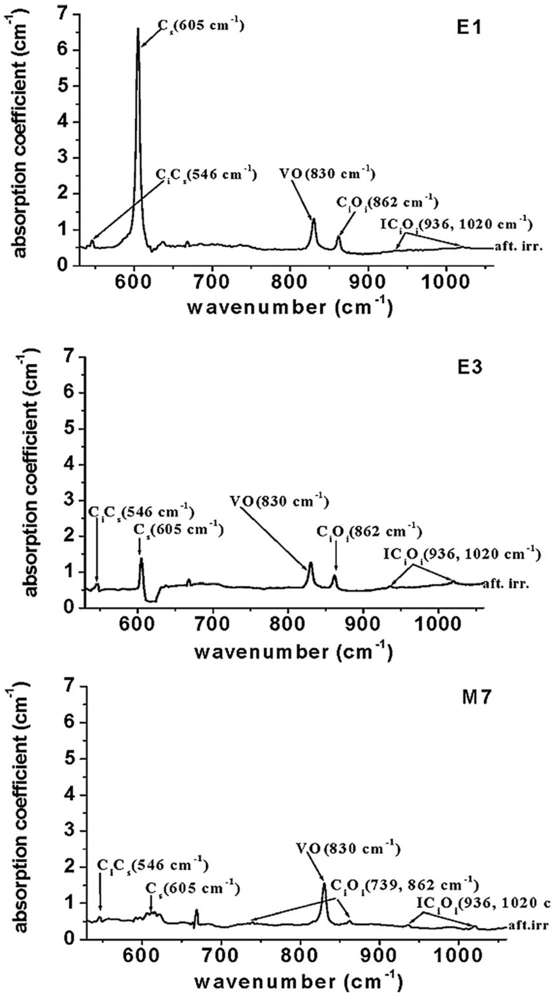

FIG. 1. Fragments of absorption spectra after electron irradiation for samples of three representative carbon concentrations: high, medium, and low (samples E1, E3, and M7 of Table I, respectively).

The production rate G of vacancies (the number of isolated vacancies produced per unit time and volume) is proportional to the dose rate $\mathrm{d}\phi/\mathrm{d}t$

$$G = \eta \ \mathrm{d}\phi/\mathrm{d}t. \tag{1}$$

The generation coefficient η is a fundamental parameter of irradiation, and yet, there is a large scatter of reported values. We accept $\eta = 0.09\,\mathrm{cm}^{-1}$. This value gives the best fit to the data processed below. Were the calibration for VO changed as mentioned above, the best-fit value of η would change proportionally, to $0.12\,\mathrm{cm}^{-1}$. Both values of η are consistent with the results of early work[24] based on an electron paramagnetic resonance study of VO at low dose.

The vacancies are immediately trapped by oxygen, and accordingly, the production rate of VO is identical to G. The VO defects, when produced, trap self-intestitials leading to a reduction in [VO]. The overall time derivative is

$$\mathrm{d}[\mathrm{VO}]/\mathrm{d}t = G - \beta_{\mathrm{vo}}[\mathrm{I}][\mathrm{VO}], \tag{2}$$

where β_{vo} is the kinetic coefficient for the I + VO annihilation reaction. The self-interstitial concentration [I] corresponds to a balance of the production (at a rate identical to G) and of the loss controlled by all the relevant traps: by VO—at a rate of β_{vo} [I] [VO]—and by the carbon-containing species $\mathrm{C_s}$, $\mathrm{C_iO_i}$, $\mathrm{IC_iO_i}$ etc—at a rate of β_{c} [I] [C]. Here, [C] is the total concentration of these species—identical to the initial concentration of $\mathrm{C_s}$—and β_{c} is the kinetic coefficient for the trapping reaction. The concentration [I] is then expressed as

$$[\mathrm{I}] = G/(\beta_{\mathrm{c}}[\mathrm{C}] + \beta_{\mathrm{vo}}[\mathrm{VO}]). \tag{3}$$

With this expression, the kinetic Equation (2) for the accumulation of VO defects takes the form

$$\mathrm{d}[\mathrm{VO}]/\mathrm{dt} = G\beta_{\mathrm{c}}[\mathrm{C}]/(\beta_{\mathrm{c}}[\mathrm{C}] + \beta_{\mathrm{vo}}[\mathrm{VO}]). \tag{4}$$

The time derivative is expressed as $(G/\eta)\ \mathrm{d}[\mathrm{VO}]/\mathrm{d}\phi$, taking into account Eq. (1). Then, Eq. (4) is integrated and gives [VO] in dependence of the dose ϕ and the carbon concentration [C]

$$[\mathrm{VO}]^2 + 2q_{\mathrm{c}}[\mathrm{VO}][\mathrm{C}] = 2\eta\ q_{\mathrm{c}}\phi[\mathrm{C}], \tag{5}$$

where q_{c} is the ratio of the two trapping coefficients: $q_{\mathrm{c}} = \beta_{\mathrm{c}}/\beta_{\mathrm{vo}}$. By Eq. (5), the normalized concentration [VO]/[C] is a function of $\phi/[\mathrm{C}]$, and this rule is well obeyed by the samples of Table I (Fig. 2). The two last points, showing some deviation, are less reliable since they refer to the lowest [C]. The solid line in Fig. 2 is the solution of the quadratic Equation (5)

$$[\mathrm{VO}]/[\mathrm{C}] = -q_{\mathrm{c}} + (q_{\mathrm{c}}^2 + 2\ q_{\mathrm{c}}\ \eta\phi/[\mathrm{C}])^{1/2}, \tag{6}$$

with the best-fit parameters: $\eta = 0.09\,\mathrm{cm}^{-1}$ and $q_{\mathrm{c}} = 0.9$. If $\phi/[\mathrm{C}] \ll q_{\mathrm{c}}/(2\ \eta)$, then $[\mathrm{VO}] = \eta\ \phi$; in this case, V and I are trapped independent of each other: vacancies—by oxygen, self-interstitials—by carbon. At $\phi/[\mathrm{C}] \gg q_{\mathrm{c}}/(2\ \eta)$, [VO] is proportional to the square root of the dose and to the square root of [C].

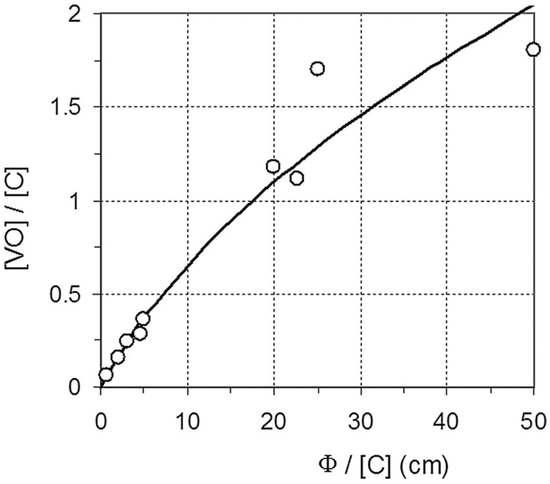

FIG. 2. Concentration ratio [VO]/[C] plotted in dependence of a normalized dose, $\phi/[\mathrm{C}]$, for samples of Table I. The solid curve is the analytical solution, Eq. (6), with the best-fit parameters.

Equation (6) allows to plot [VO] in dependence of [C], for a specified dose; in Fig. 3(a), these curves are computed for the actually used 3 doses which allows for comparison with the experimental points shown by symbols and lying reasonably close to the predicted curves. One can plot the same computed values of [VO] in dependence of the dose, for any specified carbon content, but this presentation is less convenient than Fig. 3(a) for comparison with experiment—due to many values of [C] in our sample set. To get a general idea of the dose dependence of [VO] (Fig. 3(b)), some arbitrary representative values of [C] were used.

B. Carbon loss during generation of VO

The present model also predicts a variation in the concentration of the substitutional carbon component, $[\mathrm{C_s}]$—at least for samples of not too high [C], when the irradiation-produced $\mathrm{C_i}$ species are trapped mostly by $\mathrm{O_i}$ and not by $\mathrm{C_s}$. The loss rate of $\mathrm{C_s}$ is then identical to the trapping rate of self-interstitials by substitutional carbon and equals β_{c} [I] $[\mathrm{C_s}]$ where [I] is specified by Eq. (3). Making use of Eq. (4) for [VO], one obtains $\mathrm{d}[\mathrm{C_s}]/\mathrm{d}[\mathrm{VO}] = -[\mathrm{C_s}]/[\mathrm{C}]$. Then, $[\mathrm{C_s}]$ is expressed through [VO], without using any fitting parameters,

$$[\mathrm{C_s}] = [\mathrm{C}]\exp(-[\mathrm{VO}]/[\mathrm{C}]). \tag{7}$$

The remaining fraction of substitutional carbon, $[\mathrm{C_s}]/[\mathrm{C}]$, is equal to $\exp(-X)$, with $X = [\mathrm{VO}]/[\mathrm{C}]$. This rule is, however, not well obeyed by the samples of Table I (Fig. 4), even if only the samples of moderate carbon concentration—E3, H3, Ge4, and M5 shown by circles—are taken into account (for low-carbon samples, the remaining concentration $[\mathrm{C_s}]$ is too low to be determined reliably).

The two carbon-rich samples (the rhombs in Fig. 4) show still larger deviation. In this case, the expected carbon loss, $[\mathrm{C}] - [\mathrm{C_s}]$, is close to [VO] by Eq. (7), but the actual

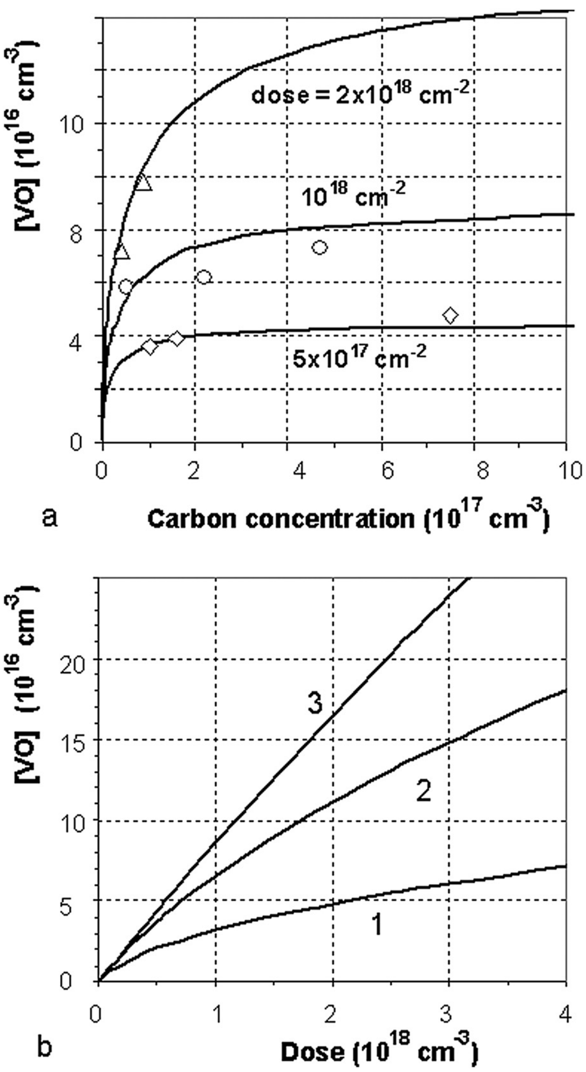

FIG. 3. The calculated concentration of the VO defect. a: in dependence of the carbon content, for three values of the dose ϕ; the symbols represent samples of Table I (rhombs: $\phi = 5\times10^{17}$, circles: $\phi = 10^{18}$, triangles: 2×10^{18} cm^{-2}). b: in dependence of the dose, for three representative carbon concentrations: 10^{16}, 10^{17}, 10^{18} (curves 1 to 3, respectively).

loss is significantly larger, probably due to a contribution of C_iC_s.

C. Concentrations of C_iO_i, IC_iO_i, and larger interstitial clusters $I_nC_iO_i$

It is also possible to calculate the concentration of C_iO_i (and of subsequently formed $I_nC_iO_i$ clusters)—if C_i is trapped mostly by O_i and not by C_s. The production rate of C_iO_i is equal to the loss rate of substitutional carbon, β_c [I] [C_s]. On the other hand, the C_iO_i species are lost by trapping self-intestitials at a rate β_c [I] [C_iO_i]. The overall kinetic equation, $d[C_iO_i]/dt = \beta_c\ [I]\ ([C_s] - [C_iO_i])$, together with Eq. (4), specifies [C_iO_i] as a function of [VO]

$$d[C_iO_i]/d[VO] = ([C_s] - [C_iO_i])/[C]. \qquad (8)$$

With new variables $X = [VO]/[C]$ and $Y_0 = [C_iO_i]/[C]$ and with the carbon concentration [C_s] substituted from Eq. (7), the kinetic equation for [C_iO_i] becomes

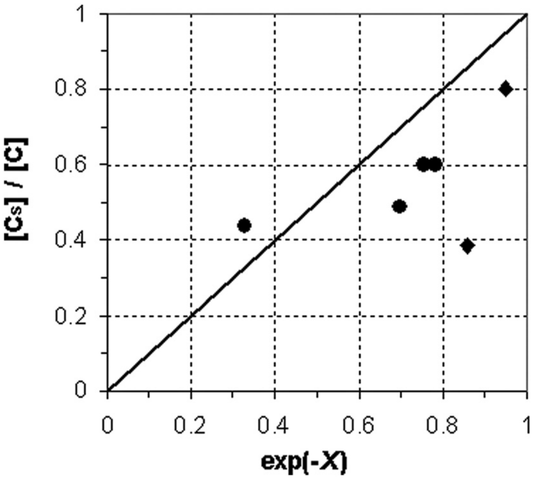

FIG. 4. The remaining fraction of substitutional carbon, $[C_s]/[C]$, expected to be equal to $\exp(-X)$, where $X = [VO]/[C]$. The samples of medium carbon content (E3, H3, Ge4, and M5) are shown by circles and the carbon-rich ones (E1 and Sn3.1) by rhombs.

$$dY_0/dX = \exp(-X) - Y_0. \qquad (9)$$

The solution is $Y_0 = X\exp(-X)$.

For the next cluster, IC_iO_i, the source species is C_iO_i instead of C_s, and the variable $Y_1 = [IC_iO_i]/[C]$ obeys the equation similar to Eq. (9) for Y_0—only $\exp(-X)$ should be replaced with $[C_iO_i]/[C] = X\exp(-X)$. The solution is $Y_1 = (X^2/2)\exp(-X)$. In a similar way, the concentrations of all the larger clusters are found one by one

$$Y_n = [I_nC_iO_i]/[C] = (X^{n+1}/(n+1)!)\exp(-X). \qquad (10)$$

The normalized concentrations Y_n are plotted in dependence of $X = [VO]/[C]$ in Fig. 5, for $n = 0$ to 6. This plot shows that the population of carbon-based interstitial defects $I_nC_iO_i$ is qualitatively different at small and large X (high and low carbon concentration):

At $X < 1$ ($[C] > [VO]$), the first species C_iO_i ($n = 0$) dominates, the second species IC_iO_i ($n = 1$) is a minor one, and larger species (n > 1) are negligible.

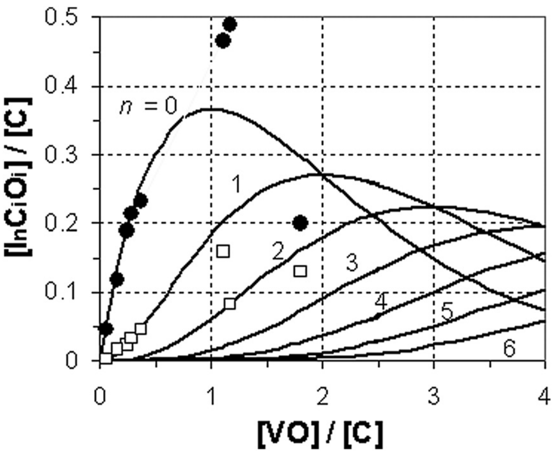

FIG. 5. Concentration of $I_nC_iO_i$ species (normalized by the carbon concentration [C]) calculated in dependence of $X = [VO]/[C]$. The experimental points—filled circles for C_iO_i (n = 0) and open squares for IC_iO_i (n = 1)—correspond to the IR band calibration discussed below.

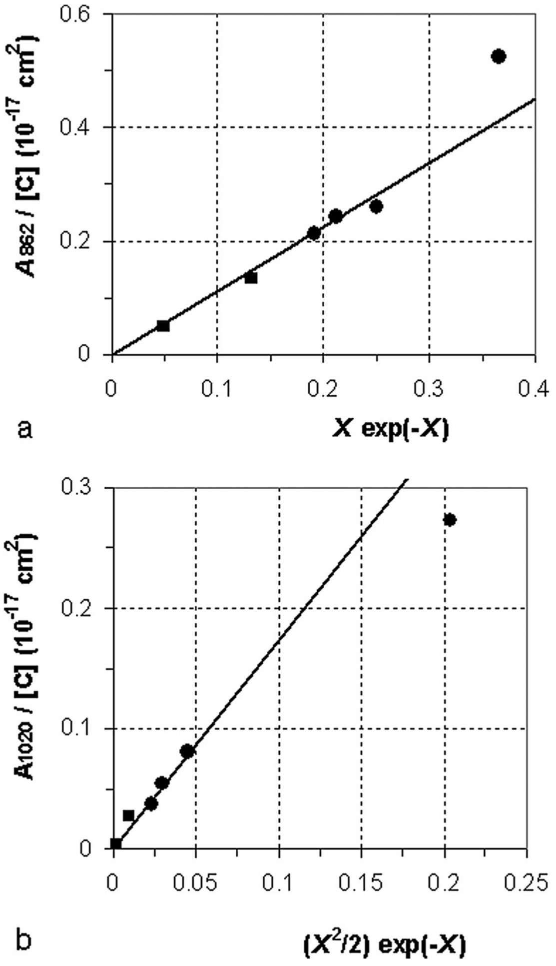

FIG. 6. Absorption intensity of: (a) C_iO_i (862 cm^{-1} band); (b) IC_iO_i (1020 cm^{-1} band) normalized by the carbon concentration [C]. The former is expected to be proportional to X exp($-X$) and the latter to ($X^2/2$) exp($-X$) where $X = [VO]/[C]$. The medium-carbon samples are shown by circles and the high-carbon ones by squares.

At $X \gg 1$ ([C] $\ll$ [VO]), there are several species—C_iO_i, IC_iO_i, $I_2C_iO_i$, etc—co-existing in comparable concentrations. An $I_nC_iO_i$ cluster contains $n+1$ stored self-interstitials (one of them is represented by C_i). The total amount of stored self-interstitials—a sum of $(n+1)$ [$I_nC_iO_i$] over n—is equal to [VO]. The total cluster concentration—a sum of [$I_nC_iO_i$]—is identical to the carbon loss, [C] $(1-\exp(-X))$, and therefore, the average size of the clusters, $<n+1>$, is equal to $X/(1-\exp(-X))$. At $X \gg 1$, all the initial carbon atoms are converted into the clusters and the average size is then simply [VO/[C]].

The experimental points in Fig. 5 correspond to the calibration coefficients for the C_iO_i and IC_iO_i related bands deduced in Sec. III D. They follow, approximately, the computed curves for both [C_iO_i]/[C] (n = 0) and [IC_iO_i]/[C] (n = 1).

D. Calibration of the bands due to C_iO_i and IC_iO_i

The concentration [C_iO_i] is proportional to the observed intensity A_{862} of the C_iO_i -related band (at 862 cm^{-1})

$$[C_iO_i] = \gamma_{862} A_{862}, \qquad (11)$$

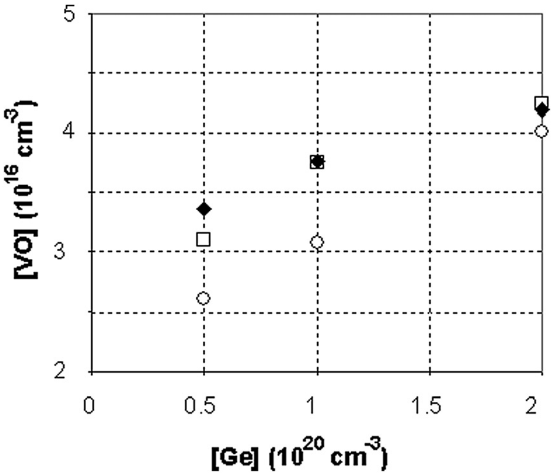

FIG. 7. The VO concentration in Ge-doped samples of Ref. 18, in dependence of [Ge] (open squares). The calculated carbon-controlled values of [VO] are shown by circles. The computed values shown by filled rhombs correspond to self-interstitial trapping by both germanium and carbon, with a proper trapping ratio $\beta_{ge}/\beta_c = 7 \times 10^{-4}$.

where γ_{862} is the optical calibration coefficient. Equation (10), at $n = 0$, means that the ratio A_{862}/[C] is expected to be proportional to X exp($-X$), with a slope equal to $1/\gamma_{862}$. This plot is presented in Fig. 6(a) for the samples of moderate and high [C]. The expected proportionality is well fulfilled excepting a less reliable point of the highest X (the lowest carbon content), and the deduced calibration coefficient is $\gamma_{862} = 8.9 \times 10^{16}$ cm^{-2}. This is not far from a previously reported[21] value of 1.1×10^{17} cm^{-2}.

Similarly, the intensity A_{1020} of the band due to IC_iO_i, if normalized by [C], is predicted to be proportional to ($X^2/2$) exp($-X$)—by Eq. (10) at $n = 1$. This rule is consistent with the data (Fig. 6(b), for the same samples), and the deduced calibration for this band is $\gamma_{1020} = 5.8 \times 10^{16}$ cm^{-3}—somewhat larger than the previously reported[21] value of 3.8×10^{16} cm^{-2}.

E. Germanium as a self-interstitial trap in Si

The Ge impurity is known[25] to trap vacancies but only at low irradiation temperature (<220 K), implying a very small binding energy of V and Ge_s. For our irradiation temperature (70 °C), no V trapping by Ge_s occurs. The reported concentration [VO] in Ge-doped samples[18] irradiated by 2 MeV electrons at a dose of 5×10^{17} cm^{-2} is shown in Fig. 7 by open squares. These samples additionally contained[18] carbon. A separation of the two contributions—by C and by Ge—is now possible since the trapping parameter q_c for carbon was specified above. The carbon-controlled values of [VO]—computed by Eq. (6)—are shown by circles. These are systematically lower than the actual values of [VO]—indicating some Ge contribution into self-interstitial trapping. With two self-interstitial-trapping impurities, C and Ge, in all the above equations, the trapping rate by carbon—proportional to β_c [C]—should be modified by adding a term due to germanium, β_{ge} [Ge]. Equivalently, [C] should be replaced with [C] + [Ge] β_{ge}/β_c. The VO concentrations calculated by Eq. (6) with this replacement (filled rhombs in Fig. 7) are close to the actual values with a proper choice of the trapping ratio: $\beta_{ge}/\beta_c = 7\times10^{-4}$. Germanium can compete with carbon for trapping self-interstitials only if the concentration ratio [Ge]/[C] is about 1000 or larger. The concentrations of produced interstitial species, Ge_i and C_i, are then comparable. Similar to C_i, the Ge_i species are likely to be highly mobile and to be immediately trapped by O_i into Ge_iO_i. The presence of Ge_iO_i in irradiated Ge-doped samples may account for the peculiarities[26] of isochronal annealing: a significant rise in [O_i] was found, started at 220 °C and accompanied by a reduction in [VO]. In the absence of Ge, no oxygen rise occurs, and VO starts to decay only at $T \geq 300\,^\circ C$. These effects are straightforwardly explained by dissociation of Ge_iO_i. The liberated Ge_i atoms then release self-interstitials ($Ge_i \rightarrow Ge_s + I$) which annihilate VO defects, thus leading to an additional rise in [O_i].

IV. CONCLUSIONS

An impact of the carbon impurity on the production of the VO defect is clearly demonstrated by comparing samples of a wide range of the carbon content, [C]. An analytical model for VO production controlled by self-interstitial trapping by carbon (or generally also by other impurities) is developed. The VO concentration, [VO], is obtained as a function of the dose and of [C]. It is concluded that carbon is only slightly less efficient a trap than VO. The concentrations of carbon-related interstitial species (C_iO_i, IC_iO_i, etc) are expressed as functions of [VO]/[C] ratio, and the optical calibration coefficients for the C_iO_i and IC_iO_i defects are determined.

In samples containing both carbon and germanium, [VO] is systematically higher than the calculated value controlled by carbon—showing that also Ge is a trap for self-interstitials, although 1000 times less efficient than carbon.

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