

Gamma Irradiation of Lithium-Drifted Silicon*

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Boron-doped silicon, compensated by the lithium ion drift method, was irradiated at room temperature with ^{60}Co gamma rays. Resistivity and Hall-effect measurements show the samples to be p type immediately after irradiation. Kept near room temperature the p -type conductivity of the samples spontaneously increases because lithium donors are lost by precipitation on irradiation-produced defects. Also other defects are introduced which have an electron energy level at $E_v + (0.25 \pm 0.03)$ eV in float zone and at $E_v + (0.30 \pm 0.03)$ eV in oxygen-rich silicon. The latter level is ascribed to the association of oxygen with an interstitial silicon atom.

INTRODUCTION

Research carried out in the last twenty years has established that irradiation introduces electrically active defects into semiconductors. These materials are very sensitive to irradiation since their electrical properties are determined by relatively small amounts of impurities. This sensitivity particularly applies to lithium-drifted silicon in which the close compensation of acceptors by lithium donors is easily upset by the introduction of radiation defects. For this reason an investigation of the radiation effects in this high resistivity silicon was undertaken. The purpose of the experiments was to obtain information on radiation effects in silicon in general, e.g., on the interaction between irradiation-induced defects and chemical impurities like lithium, boron, and oxygen. More specifically the experiments were aimed to contribute to the understanding of the effects of irradiation upon the lithium-drifted nuclear particle detector.

A brief description of the experiment and the results is given first. Boron-doped silicon samples, compensated by the lithium ion drift process, are irradiated at room temperature with 1.17- and 1.33-MeV photons from a ^{60}Co source. Via intermediate Compton electrons, primary defects, vacancies, and interstitial silicon atoms are created uniformly through the crystals. These mobile defects are captured by the chemical impurities present in the silicon, forming stable composite defects. The originally intrinsic silicon becomes p type immediately after the irradiation. Kept at, or near, room temperature the resistivity of the samples spontaneously decreases until a saturation is reached. At regular times during this annealing process Hall effect measurements were made over a wide temperature range to determine the concentrations and energy levels of the electrically active defects. Experiments were performed on five samples taken from three batches which differed in the concentrations of boron and oxygen. A characteristic of the experiments is the presence of highly mobile lithium ions which have the ability to form complexes with other impurities.

EXPERIMENTAL METHODS

A. Preparation of Samples

The initial material used in these experiments is p -type, boron-doped, single-crystalline silicon, supplied by Merck, Sharp and Dohme (M), Wacker Chemie (W), and Texas Instruments Inc. (TI). Silicon from Texas Instruments was sold under trademark "Lopex," and contained appreciable amounts of oxygen, detectable at 4°K by the optical absorption at a wavelength of 9 μm .^{1,2} The concentrations of boron and the compensating donors were determined by resistivity and Hall effect measurements. Dislocation densities were obtained by making counts of etch pits.^{3,4} The effective drift mobility of lithium ions, a measure for interactions between lithium ions and other defects like oxygen, was determined by measuring the lithium ion drift rate in reverse-biased p - n junctions. Resulting values for the impurity concentrations are summarized in Table I. A full account of the investigation of the basic silicon is given in previous publications,^{5,6} dealing with related experiments on silicon from the same batches.

Lithium is introduced into the samples by diffusion from a lithium-in-oil suspension and distributed through the crystals by the lithium ion drift technique.⁷⁻⁹ A lithium-compensated intrinsic layer, containing equal amounts of lithium and net boron, is drifted to a width of about 2 mm in all cases. To obtain samples well suited for resistivity and Hall effect measurements on the intrinsic region the greater part of the low-resistivity p - and n -type boundary layers is removed by etching. The final shape, adapted to the requirements of the Van der Pauw method,¹⁰ is shown in Fig. 1. Phosphor-bronze springs, clipped around the remaining low resistivity spots, provide electrical contact to the samples. Immediately after etching a low resistivity, typically 10 000 Ω cm, is always observed. This is due to an n -type surface layer and a possible contribution from a nonideal compensation. By keeping the sample in a dry oxygen atmosphere for some days, and applying a cleanup drift procedure for several weeks, the resistivity is in-

creased to a room-temperature value of at least 200 000 Ω cm.

B. Resistivity and Hall Effect

As mentioned the method of Van der Pauw¹⁰ was used to measure resistivity and Hall effect on the irradiated specimens. Direct currents near 1 μ A, to avoid carrier injection effects, were led through the samples and the voltages developed across the other two contacts were read on a digital voltmeter with an input impedance above 10 000 M Ω . At each temperature the final Ohmic or Hall voltage was taken as the average of eight readings, obtained from all possible commutations of current and magnetic field, and permutations of contacts. The Hall effect was determined at a magnetic field of 1.5 T. Sample temperatures, in the range from 30° to 300°K, were measured by a Au(Fe)–Chromel thermocouple.

C. Gamma Irradiation

To irradiate the silicon crystals they were exposed, at room temperature or near 0°C (W samples), to a 250-Ci ⁶⁰Co source at a distance of 1.9 cm for about 24 h. The uniformity of the Compton electron flux over the sample volume is improved by enclosing the samples in an aluminum box with a wall thickness of 2 mm. The samples were irradiated in a $\langle 111 \rangle$ direction to a fluence of about 3×10^{16} γ/cm^2 , thereby introducing a low defect density near 10^{13} per cm^3 . Calibrations of the fluence, and checks on its radial uniformity, were made by lithium fluoride thermoluminescent dosimeters.

EXPERIMENTAL RESULTS

A. Preliminary Remarks

As a consequence of the irradiation the resistivity of the samples drops to a much lower value. The sign of the Hall voltage indicates p -type conduction. Carrier concentrations p are calculated from the observed Hall coefficient R_H by the equation $p = r/R_H e$. For the Hall factor r the empirical expression $r = 0.85 \times (300/T)^{0.2}$ was adopted.^{11,12} The resistivity measurements are considered as yielding information supplementary to the Hall effect, useful to check the reliability of the data. Nonuniform conduction in a sample is indicated by a temperature dependence of the

TABLE I. Impurity concentrations in the basic silicon crystals. Etch-pit density EPD, boron concentration n_B , compensating donor concentration n_d , and oxygen concentration n_o .

Batch	EPD (cm^{-2})	n_B (10^{14} cm^{-3})	n_d (10^{14} cm^{-3})	n_o (10^{14} cm^{-3})
M, 7	30 000	1.6	0.2	<10
W, 2	24 000	15.8	0.02	<10
TI, 2	1 300	7.4	0.7	620

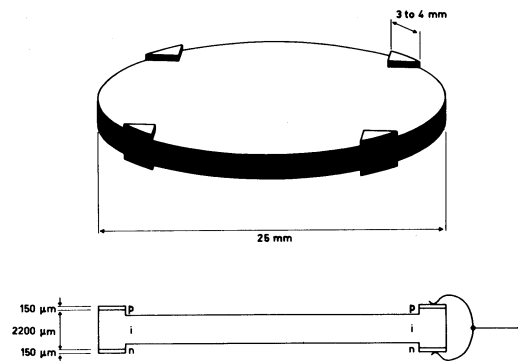


FIG. 1. Illustration of the shape and dimensions of the samples.

Van der Pauw resistance ratio,¹³ which has to be a geometrically determined constant. By combining results of Hall effect and resistivity the Hall mobility $\mu_H = R_H \sigma$ may be calculated and compared with mobilities expected on the basis of lattice and ionized impurity scattering.^{14,15} The calculated Hall mobility always showed a correct temperature dependence, except for some of the data taken at the lowest temperatures, where also the Van der Pauw resistance ratio was no longer a constant. Data of doubtful validity were rejected. Over the whole temperature range the calculated Hall mobility differs from the expected value by a constant factor of about 0.8. It was verified that the finite size of the contacts is responsible for this deviation.⁵ Appropriate correction factors were consequently applied. The calculated mobilities confirm the p -type behavior of the samples after irradiation.

B. Time Dependence

Immediately after the irradiation the samples already show extrinsic behavior. Keeping them at room temperature, or at temperatures up to 50°C, a spontaneous further increase of p -type conductivity is observed. Finally saturation is reached. If then the temperature is raised or lowered the saturation value of the hole concentration does not change. This indicates that no equilibrium process is involved. An explanation for the increasing p -type conductivity is the loss of lithium donor atoms by precipitation on some irradiation produced defect called R^* . Since the concentration of lithium is much higher than that of the defect R^* the precipitation process obeys first-order kinetics. The time dependence of the hole concentration p is then given by

$$p(\infty) - p(t) = [p(\infty) - p(0)] \exp(-t/\tau),$$

in which τ is a time constant to be discussed below. The exponential approach of saturation is clearly observed in the Merck and Wacker samples. Figure 2 illustrates such a time dependence of the hole concentration in one of these crystals at two different recovery temperatures. For the Texas Instruments samples the above

TABLE II. Summary of the data relevant to the time constant τ .

Sample	n_{Li} (10^{14} cm^{-3})	Temp. ($^{\circ}\text{C}$)	α	τ (h)	R_{capt} (10^{-8} cm)
M, 7, 17	1.4	20	0.79	840	1.8
		30	0.86	230	2.6
		40	0.90	95	2.7
M, 7, 18	1.4	30	0.86	250	2.4
		50	0.93	39	3.0
W, 2, 12	15.8	20	0.42	78	3.1
		30	0.51	26	3.2
TI, 2, 2	6.7	40	0.081	~ 260	2.2
		50	0.12	~ 22	7.9
TI, 2, 9	6.7	30	0.044	~ 2000	1.2

picture seems to be less valid since plots of $\ln[p(\infty) - p(t)]$ versus time show some curvature. Although with less accuracy the same analysis is applied.

For the diffusion limited process of lithium precipitation the time constant τ is given by^{16,17}:

$$\tau = (4\pi\alpha n_{Li} D_{Li} R_{capt})^{-1}.$$

In this expression the lithium concentration n_{Li} equals the net acceptor concentration (Table I). Also the diffusion constant of lithium in silicon D_{Li} is known at the required temperatures.¹⁸ The fraction α of unpaired lithium ions is either calculated using the equilibrium constant for the $\text{Li}^+ - \text{B}^-$ ion pairing reaction¹⁹⁻²¹ (M and W samples) or is obtained by measuring the effective drift rate of lithium ions in a $p-n$ junction (TI samples). Once the parameters α , n_{Li} , and D_{Li} are known the observed relaxation times τ may be interpreted in terms of the capture radius R_{capt} . Data relevant to this analysis are given in Table II.

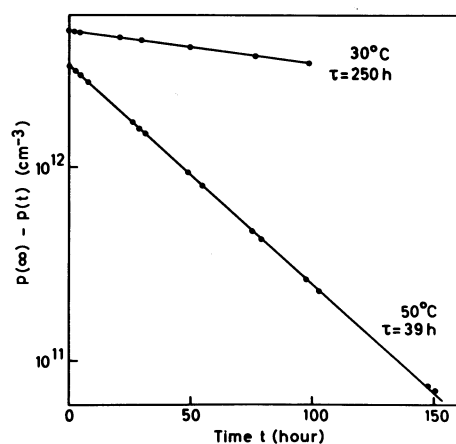
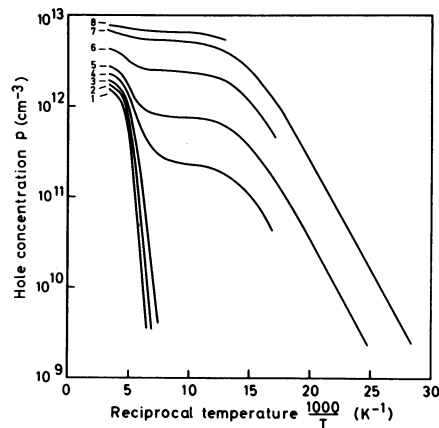
FIG. 2. Variation of hole concentration in the irradiated sample M, 7, 18 during recovery at 30° and 50°C; $p(\infty) = 7.7 \times 10^{12} \text{ cm}^{-3}$.

FIG. 3. Temperature dependence of the hole concentration in sample M, 7, 18 immediately after the irradiation (curve 1) and at various stages of the recovery process (curves 2-8).

C. Temperature Dependence

At several points of time during the recovery treatment the temperature dependence of the hole concentration is determined by Hall effect and resistivity measurements in the range from 30° to 300°K. During such a measurement the sample is at a low temperature and therefore the process of lithium precipitation is stopped. Results of the Hall effect for the three different kinds of samples are shown in the Figs. 3-5. Curve 1 in these figures is always measured immediately after the irradiation, curve 8 corresponds to the condition of saturation, while the other curves are measured at regular intervals in between.

To interpret the experimental curves n_R electron energy levels at a position of $E_v + E_R$ in the lower half of the gap are postulated. Taking into account also the usual levels due to lithium, boron, and the valence

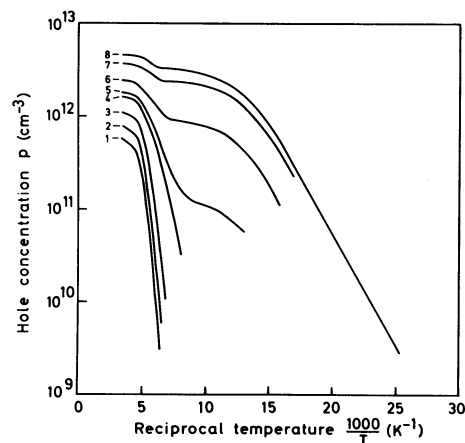


FIG. 4. Temperature dependence of the hole concentration in sample W, 2, 12 immediately after the irradiation (curve 1) and at various stages of the recovery process (curves 2-8).

band one derives an equation for the hole concentration p which reads:

$$p^3 + p^2(n_B - p_{\min} + n_B' + n_R') + p(n_B n_R' - p_{\min} n_B' - p_{\max} n_R' + n_B' n_R') - p_{\max} n_B' n_R' = 0. \quad (1)$$

In this expression the following abbreviations are used:

(a) the effective density of states in the valence band $n_v = 2(2\pi m_h kT/h^2)^{3/2}$, equal to $1.97 \times 10^{15} T^{3/2} \text{ cm}^{-3}$ for an effective hole mass $m_h = 0.55 m^{15}$;

(b) $n_B' = g_B^{-1} n_v \exp(-E_B/kT)$, E_B is the energy of the boron acceptor level above the valence band, $g_B = 4$ is the statistical weight of the level²²⁻²⁴;

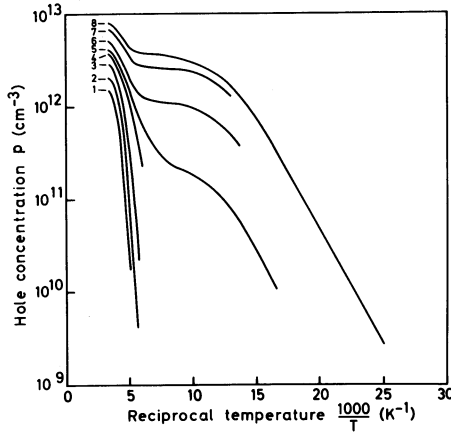


FIG. 5. Temperature dependence of the hole concentration in sample TI, 2, 2 immediately after the irradiation (curve 1) and at various stages of the recovery process (curves 2-8).

(c) $n_R' = g_R^{-1} n_v \exp(-E_R/kT)$, E_R is the energy of the level, g_R its unknown statistical weight;

(d) in case the irradiation defect is an acceptor: $p_{\max} = n_B + n_R - n_{Li}$, $p_{\min} = n_B - n_{Li}$;

(e) in case the irradiation defect is a donor: $p_{\max} = n_B - n_{Li}$, $p_{\min} = n_B - n_R - n_{Li}$.

For all of the curves 1 to 3 in the Figs. 3 to 5 it appeared that $p_{\min} < 0$, which means that at low temperatures the Fermi level gets pinned at the irradiation defect level. In the course of the recovery treatment so much lithium is lost by precipitation that $p_{\min}$ becomes positive. In that case no pinning of the Fermi level occurs and at low temperatures the boron acceptor levels are observed. Figure 6 illustrates the temperature dependence of the Fermi level in these various cases.

It happens that $E_R - E_B \gg kT$. At temperatures near 120°K, and for $p_{\min} > 0$, the Fermi level is in between and far from both levels. In this temperature range the occupation of the levels does not change. Consequently the hole concentration p equals $p_{\min}$, independent of temperature. The separation of the energy levels en-

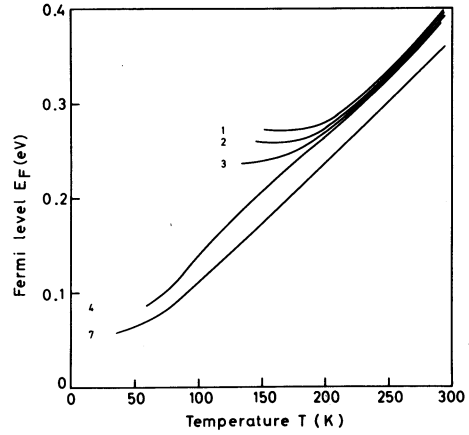


FIG. 6. Temperature dependence of the Fermi level in sample M, 7, 18. Labels to the curves correspond to those in Fig. 3.

ables one to avoid the use of the complicated expression (1) for the hole concentration. Simpler equations valid for the higher or the lower temperature regions may be used instead. An equation valid below $T = 120^\circ\text{K}$ is obtained by substituting $n_R' = 0$ in Eq. (1), yielding

$$p^2 + p(n_B - p_{\min} + n_B') - p_{\min} n_B' = 0. \quad (2)$$

To determine the parameters of Eq. (2) theoretical curves are adjusted to the experimental data for $T < 120^\circ\text{K}$ by the method of least squares.²⁵ Results of the analysis of one curve for each of the samples are summarized in Table III. The boron acceptor concentration n_B resulting from this analysis (Table III, column 4) is less than the boron concentration in the original samples (column 2), because lithium-boron ion pairing reduces the number of acceptors. Knowing the equilibrium constants of the reactions between lithium and boron,¹⁹⁻²¹ and lithium and oxygen,²⁶ the acceptor concentration to be expected (column 3) can be calculated. The discrepancy is probably partly due to a bad choice for the Hall factor r at low temperatures.

In a higher temperature range, between room temperature and about 120°K, the Fermi level is sufficiently high in the gap to ensure complete filling of the boron

TABLE III. Results concerning the boron level. Various values for the boron concentration n_B are explained in the text.

Sample	n_B original, (10^{14} cm^{-3})	n_B expected (10^{14} cm^{-3})	n_B analysis (10^{14} cm^{-3})	E_B (10^{-3} eV)
M, 7, 17	1.60	1.35	1.48	42.6
M, 7, 18	1.60	1.56	1.60	43.3
W, 2, 12	15.8	6.7	4.2	44.0
TI, 2, 2	7.4	7.2	4.8	43.7
TI, 2, 9	7.4	7.2	5.1	45.2

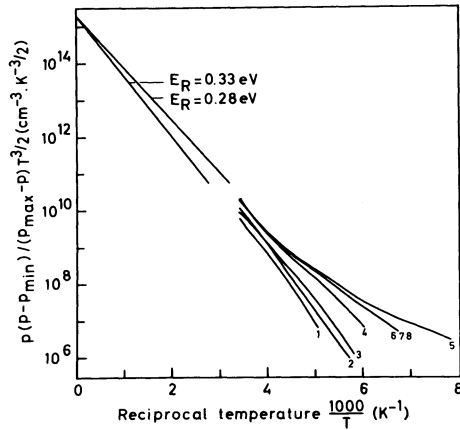


FIG. 7. Illustration of the analysis by Eq. (3) of the hole concentration in the temperature range between 120° and 300° K for sample TI, 2, 2. Curves 1–8 are calculated by the expression $p(p - p_{\min}) / (p_{\max} - p) T^{3/2}$. Straight lines represent $n_R' / T^{3/2}$ for two values of E_R ; $g_R = 1$.

levels. Under these conditions Eq. (1) simplifies to

$$p(p - p_{\min}) / (p_{\max} - p) T^{3/2} = n_R' / T^{3/2} = 1.97 \times 10^{15} g_R^{-1} \times \exp(-E_R/kT). \quad (3)$$

In most cases the values of $p_{\max}$ and $p_{\min}$ can be read directly from the curves. If however the Fermi level pins on E_R the negative value of $p_{\min}$ is determined using the relation $p_{\min} = p_{\max} - n_R$ and a suitable extrapolation of n_R backwards in time. A graphical representation of the relationship expressed by Eq. (3) is given in Fig. 7. The curves 1 to 8 are calculated from the left-hand side expression of Eq. (3). According to the right-hand side of the equation one expects straight lines with slope $-E_R/k$, intersecting the ordinate axis at a value determined by g_R . Examination of the results shows that a reliable determination of g_R , by extrapolating the curves 1 to 8 back to $1/T = 0$, is impossible. Therefore the usual choice $g_R = 1$ was made. The value of E_R was obtained either by fitting theory to experiment using Eq. (3), or by applying the criterion $E_F = E_R$ at half filling of the level. Results of the analysis relevant to the observed irradiation defect, its concentration n_R and the energy level position E_R , are summarized in Table IV. Data with respect to the lithium precipitation are shown in Table V. The concentration of precipitation centers is given by $\Delta p_{\max} = p_{\max}(\text{curve 8}) - p_{\max}(\text{curve 1})$, where $p_{\max}$ is the constant value the hole concentration assumes at about room temperature.

Since Fig. 7 evidently shows some discrepancies between the experimental results and the theoretical predictions of the one defect level model a more detailed model was considered. It turned out that the agreement between theory and experiment could be improved substantially by introducing a small amount,

in the order of $0.1n_R$, of a second defect with a level somewhat lower but close to E_R . However a second irradiation defect level introduces three new parameters in the analysis, i.e., its concentration, energy level, and statistical weight. Since a determination of these parameters could not convincingly be made the analyses were restricted to the one defect level model, which is considered to be a good approximation.

DISCUSSION

A. Lithium Precipitation

It is seen in Table II that under widely varying conditions of temperature and impurity concentrations the capture radius R_{capt} assumes a rather constant value between 2 and 3×10^{-8} cm. This seems to verify the model of lithium precipitation on an irradiation defect in a process lacking long-range Coulomb attraction. Since the lithium ion carries unit positive charge the precipitation center has to be neutral under the conditions of this experiment, i.e., when the Fermi level is close to the middle of the gap. It is known that in oxygen-rich silicon the oxygen-vacancy complex, OV, is produced predominantly.^{27,28} Precipitation of lithium

TABLE IV. Results concerning the irradiation defect level. Values for n_R marked by an asterisk were obtained by extrapolation. The samples not shown in the table give almost identical results as those of the same batch listed below.

Sample	Curve	n_R (10^{12} cm^{-3})	E_R (eV)
M, 7, 18	1	2.2*	0.28
	2	2.2*	0.28
	3	2.2*	0.27
	4	2.1	0.26
	5	2.0	0.26
	6	1.9	0.26
	7	1.4	0.26
	8	1.0	0.25
W, 2, 12	1	1.9*	0.27
	2	1.9*	0.26
	3	1.9*	0.25
	4	1.7*	0.24
	5	1.7	0.24
	6	1.6	0.24
	7	1.4	0.23
	8	1.3	0.23
TI, 2, 2	1	3.5*	0.33
	2	3.5*	0.32
	3	3.5*	0.31
	4	3.9	0.29
	5	4.1	0.29
	6	4.2	0.29
	7	4.5	0.28
	8	4.5	0.28

on these centers has also been reported.²⁹⁻³¹ So the reaction $\text{Li}^+ + \text{OV} + e \rightarrow \text{LiOV}$ provides an obvious explanation for the observations made on the TI samples. For the M and W samples the situation is less clear. It is still possible that oxygen-vacancy complexes are introduced to appreciable concentrations in these float-zone materials.^{32,33} Probably other processes, e.g., the precipitation of lithium on lithium-vacancy complexes,^{34,35} are more important in this silicon. The amount of lithium precipitation in the oxygen-rich and in the oxygen-lean samples is almost equal. This suggests that the introduction of precipitation centers is not limited by the impurities present in the crystals, but rather by the generation of primary defects. No levels due to precipitation centers are observed in the lower half of the gap. The OV center therefore has only the one known acceptor level at $E_c - 0.17$ eV.

B. Variation of E_R

A gradual variation of the defect level energy E_R is apparent from the results given in Table IV. The analysis based on the two-level model did not indicate the simultaneous presence of two defects in comparable concentrations. This makes an explanation in terms of one defect growing at the cost of some other defect with a nearby level less probable. The change of E_R is therefore interpreted as being due to a real variation of the energy level associated with one defect. Slight variations of defect levels have been reported earlier.³⁵⁻³⁷ Moreover level energies belonging to probably identical defects are found over a rather wide range. This is evident from the discussion in the next two sections where some data taken from literature are quoted.

C. Level at $E_v + 0.30$ eV

The level at $E_R = E_v + (0.30 \pm 0.03)$ eV, observed in TI samples is clearly distinct from the level generated in the M and W samples. Oxygen therefore seems to be involved in its formation. Levels due to the same defect, observed in pulled silicon, are reported by Malovetskaya³⁸ at 0.27 eV, by Smirnova³⁰ at 0.28 eV, by Matsui³⁹ at 0.3 eV, by Nakashima⁴⁰ at 0.31 eV, and by Sonder³⁶ at 0.35 eV. These values encompass all our

results. The defect, although being dependent on the presence of oxygen, is not the well-known oxygen-vacancy complex. First there is no decrease of n_R observed in connection with the lithium precipitation on these centers. Second the quantity of lithium precipitating on defects is larger than the amount of R defects available. The level at $E_v + (0.30 \pm 0.03)$ eV is therefore ascribed to an association of oxygen with an interstitial silicon atom. This is in accord with some previous assignments.^{38,39}

D. Level at $E_v + 0.25$ eV

The behavior of the level detected in the M and W samples is similar, so that they probably belong to identical defects. Levels at about the same energy were observed by Vavilov⁴¹ at 0.21 eV or at 0.25 eV, by Sonder³⁶ at 0.21 eV, by Malovetskaya³⁸ at 0.21 eV, by Persin⁴² at 0.23 eV, and by Vitovskii⁴³ at 0.23 eV. The fact that the level is also observed in float-zone silicon free from lithium as an impurity favors the assumption that boron is involved in the defect formation. The instability of the defect, as appears from the decreasing values of n_R in Table IV, was also reported by Sonder³⁶ and Malovetskaya.³⁸

E. Introduction Rates

According to the models of Wertheim⁴⁴ and MacKay-Klontz⁴⁵ the production of primary defects by the dissociation of close pairs is dependent on temperature and Fermi level. Meaningful comparisons therefore can only be made with those experiments carried out under similar conditions. Equating the rate of vacancy formation to that of OV-precipitation centers in the TI samples the present result $\sigma = 2 \times 10^{-4} \text{ cm}^{-1}$ is comparable to results of Djerassi³⁷ and Konozenko.⁴⁶ The introduction rate of the 0.30 eV centers is $1.2 \times 10^{-4} \text{ cm}^{-1}$ whereas Sonder³⁶ found $1.0 \times 10^{-4} \text{ cm}^{-1}$. Finally the 0.25 eV center is introduced at the rate of $0.8 \times 10^{-4} \text{ cm}^{-1}$ compared to $0.6 \times 10^{-4} \text{ cm}^{-1}$ reported by Vitovskii.⁴³

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TABLE V. Data relevant to the lithium precipitation and irradiation dose Φ .

Sample	$p_{\max}(1)$ (10^{12} cm^{-3})	$p_{\max}(8)$ (10^{12} cm^{-3})	$\Delta p_{\max}$ (10^{12} cm^{-3})	Φ ($10^{16} \text{ } \gamma/\text{cm}^2$)
M, 7, 17	2.0	9.7	7.7	3.4
M, 7, 18	1.6	7.7	6.1	2.5
W, 2, 12	0.6	4.6	4.0	2.9
TI, 2, 2	1.6	8.3	6.7	2.9
TI, 2, 9	0.6	5.1	4.5	2.7

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