Evidence for a substitutional Mg acceptor level in silicon

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Among group-II impurities in silicon, Be and Zn result in substitutional acceptors while Mg has been known to cause only interstitial donor levels. We have diffused Mg into n^+ -p silicon diodes and found a number of hole levels in the lower half of the band gap of silicon. The dominating level in most samples, named the A level, has been studied in detail. Using space-charge techniques, thermal emission rates, the temperature dependence of capture cross sections, and photoionization cross sections have been measured. The experimental results are best explained if this level is considered to be an acceptor with an energy position of $E_v + 0.34$ eV. There are sufficient grounds to suggest that this could be the missing substitutional Mg acceptor level in silicon.

I. INTRODUCTION

Among impurities from group II of the Periodic Table in Silicon, Be (Refs. ¹ and 2), Zn (Refs. 3—5), and Mg (Refs. 6—10) have been studied in considerable detail. Be and Zn both are believed to occupy substitutional sites, when diffused thermally into Si, but Mg has been reported to occupy interstitial sites. While both Be and Zn as impurities have been reported to give rise to acceptor levels (at $E_v + 0.145$ eV and $E_v + 0.191$ eV for Be, and E_{v} + 0.32 eV and + 0.66 eV for Zn) in the band gap of silicon, Mg results in a number of donor levels lying be-
tween $E_c - 0.04$ eV and $E_c - 0.256$ eV. In Ge, all these impurities Be (Refs. 11 and 12), Zn (Refs. 12—14), and Mg (Refs. 12 and 15) give rise to substitutional acceptor levels. No magnesium acceptor level nor any Mg-related level in the lower half of the band gap of Si has ever been reported to the best of our knowledge. There is no a priori reason why Mg should not occupy substitutional sites as well, and give rise to a single or double acceptor level in silicon. Froyen and Zunger¹⁶ have recently probed theoretically the possibility of the existence of a Mg substitutional acceptor in silicon. They have come to the conclusion that, by subtle manipulation of experimental conditions during sample preparation, the substitutional Mg could be "engineered. "

The purpose of this study has been to search for the missing substitutional Mg acceptor. We report here the existence of many Mg-related levels that we have observed in the lower half of the band gap of Si. Results of detailed investigations are presented on one of these levels which could be caused by the substitutional Mg. The results of the thermal and optical space-charge measurements are best understood if this level is assumed to be an acceptor.

II. SAMPLE PREPARATION

A11 samples used in this study were made from prefabricated n^+ -p-p⁺ diodes available commercially from Rifa AB. The p region, approximately 17 μ m thick after the $n +$ diffusion, was a 1.5×10^{15} -cm⁻³ boron-doped epitaxial layer on Czochralski $p+$ [111] substrate from Wacker. The area of the n^+ region was 0.16 mm². Magnesium was introduced into the samples by means of solidstate diffusion. It was either evaporated on the front or back surface of the diodes or ion implanted onto the front surface. The purity of the magnesium used was 99.95% . The samples were cleaned in four different ultrasonic baths prior to magnesium evaporation using trichloroethylene, acetone, ethanol, and de-ionized water as solvent. Magnesium was evaporated by means of resistive heating at about 1×10^{-6} Torr using W or Mo heating elements. In some samples magnesium was ionimplanted at room temperature at 100 keV for about 10 min and a dose of 1×10^{15} cm⁻². To prevent out-diffusion, a 5000 Å SiO₂ layer was deposited on top of the samples in a chemical-vapor-deposition (CVD) reactor at 420'C. No further heat treatments were performed before diffusion.

After evaporation or ion implantation, diffusion was carried out using the following procedure. The samples were introduced into quartz ampoules which had been etched and rinsed in de-ionized water and kept in a furnace at 150'C for at least 24 h. Some of the ampoules were then evacuated to a pressure of about 1×10^{-7} Torr and sealed off using a H_2-O_2 burner. Other ampoules were, prior to the sealing procedure, filled with N_2 or Ar gas to a pressure which corresponds to about ¹ atm at the diffusion temperature. After diffusion, the $SiO₂$ layer was removed from the ionimplanted samples by etching in $HF + H₂O$ for 1 min. A large number of samples were prepared by varying the diffusion conditions and subsequent cooling procedures. Diffusion temperatures tried ranged from 950 to 1100'C and diffusion time from ¹ to 4 h. After diffusion the samples were cooled either in air, i.e., taken out from the furnace and allowed to cool to room temperature in air for about 10 min, or quenched in water. Reference samples were prepared for all batches which underwent the same treatment except magnesium deposition (or ion implantation).

Detailed measurements reported in this paper were performed on three batches of samples. Batch ¹ was diffused for 1 h at 1050 °C in N_2 atmosphere of about 1000 mbar at the diffusion temperature and cooled in air. Batch 2 was diffused in an evacuated ampoule for ¹ h at 1000'C and cooled in air. Batch ³ was implanted with Mg ions at 100 keV with a dose of about 10^{15} cm⁻², heat treated in an evacuated ampoule at 1010'C for ¹ h and cooled in air. Good Ohmic contacts were made by rubbing Ga-Al onto the back surface of the diodes and the samples were mounted on standard TO-5 holders with silver epoxy. The net acceptor concentration in the diodes at room temperature, after Mg diffusion, was $\sim 10^{15}$ cm⁻³ as determined from C-V measurements. All samples showed linear $1/C^2$ versus V plots in accord with abrupt junctions.

III. EXPERIMENTAL DETAILS

Thermal emission rate data were obtained using a deep-level transient spectroscopy (DLTS) (Ref. 17) setup described in Ref. 18. The capture measurements were carried out with a DLTS setup designed specially for applying short pulses in the nano-second regime. 19 To apply such short pulses the setup was almost perfectly 50 Ω matched so as to avoid reflections and unwgnted ringing. The rise and fall times of the pulse generator used were less than 250 ps and it could also apply single pulses so that single shot measuremepts could be performed which facility was used to measure capture cross sections at lower temperatures where DLTS measurements were very difficult due to smaller emission rates. The capture measurements were performed at fixed temperatures, by stabilizing the temperature at the DLTS peak for a particular rate window setting. The product of the series resistance R and the depletion layer capacitance C (RC product) of the diodes used for capture measurements were measured in situ with a current probe and sampling oscilloscope. An analysis of the equivalent circuit of a reverse biased diode (the reverse bias equal to V_R) shows that the voltage over the space charge region, i.e., over the depletion layer capacitance, is proportional to $V_R \exp(-t / RC)$ for times close to the beginning of the voltage pulse. The half-width of the current spike associated with the rise of the voltage pulse gives the RC prod $uct^{19,20}$ of the diode

For optical measurements a Zeiss MM3 double-prism monochromator with $CaF₂$ prisms or an ARC grating monochromator VM-505 were used as sources of the probe light. 1he latter was a part of an evacuated setup (including an evacuated globar as the light source) with a vacuum better than 1×10^{-2} mbar. The photocapacitance was monitored on a Boonton 72B capacitance meter with a fast analog output (option 03) and a 100-mV test signal (option 05). Relative photoionization cross sections were measured using the initial slope method. The spectrum was then calibrated to absolute values by measuring the emission rate from the full capacitance transient at one photon energy and measuring the photon flux at that energy with a calibrated thermopile. Around the photoionization cross section threshold the background thermal radiation posed a problem since the hole emission rates due to this radiation were comparable to those caused by the probe light at these energies.

A 4- μ m short-pass filter mounted on the cap of the TO-5 holder, on which the sample was mounted, reduced the hole emission due to background radiation substantially but not entirely. This residual emission was constantly monitored during the measurements and subtracted from the emission rates resulting from the incidence of the probe light.

IV. EXPERIMENTAL RESULTS

Figures ¹ and 2 show the DLTS spectra of two representative Mg diffused n^+ -p Si diodes on which Mg was vacuum evaporated, one (no. 1) with a large and the other (no. 2) with a moderate concentration of defect levels. They show a number of peaks corresponding to different hole levels. As mentioned in Sec. II, we tried different conditions for sample preparation. In most of the samples thus prepared, we found a dominating level identified as level A in Figs. 1 and 2. We tried to establish the exact sample preparing conditions for which this level appeared in large concentration but were not able to pin them down exactly. Thus, for confirmation, we introduced Mg into Si through ion implantation (and subsequent diffusion), and these samples showed the same dominating level A. The DLTS scan of such a sample (no. 3) is shown in Fig. 3. The levels seen in Figs. $1-3$ were not observed in the reference diodes.

Figure 4 gives the emission rate data for the seven hole levels that we have observed in our Mg-doped samples seen in the spectra of Figs. 1–3. Table I gives the T^2 corrected activation energies of these levels obtained from Fig. 4. One of the levels C or G was presented in all diodes with level A but levels C and G were never observed in the same diode, so it is possible that they are actually one and the same level since their emission rate sig-

FIG. 1. DLTS spectrum of Mg-doped n^+ -p diode no. 1. $V_R = -3 \text{ V}, e_{\text{max}} = 121 \text{ s}^{-1}.$

FIG. 4. Thermal emission rate data of hole levels in n^+ -p Si diodes doped with Mg.

measured variation of the DLTS peak height $S(t_p)$ with the hole capture pulse width t_p using

FIG. 2. DLTS spectrum of Mg-doped
$$
n^+
$$
- p Si diode no. 2.
 $V_R = -1$ V, $e_{\text{max}} = 121$ s⁻¹.

natures are very similar to each other. However, detailed measurements were not undertaken for these levels. Detailed electrical and optical measurements were performed to characterize the dominant A level.

The hole capture cross section σ_p^t was determined using

$$
1/\tau = \sigma_p^t v_{\rm th} p \quad , \tag{1}
$$

where τ is the capture time constant, v_{th} the averag thermal velocity of holes, and p the bulk hole concentration. τ was obtained in the standard manner¹⁷ from the

FIG. 3. DLTS spectrum of Mg-doped n^+ -p Si diode no. 3. $V_R = -1.5$ V, $e_{\text{max}} = 121$ s⁻¹.

 S_0 being the saturated peak height. We used v_{th} $=(3kT/m^*)^{1/2}$ and $m^*=0.81m_0$ (Ref. 21). σ_p^t of the A level thus obtained at different temperatures are plotted in Fig. 5. σ_p^t decreases from 2.7×10^{-14} cm² at 127 K to 1.1×10^{-14} cm² at 170 K. The corresponding captur time constants increased from 13 to 24 ns. These measurements were performed at $a -4V$ quiescent reverse bias (V_R). The *in situ* measured RC product was ≈ 3 ns for all temperatures of measurement. The RC product of the sample is a measure of the time that is taken for the depletion region width to change from its V_R value to the pulse height bias value and hence limits the shortest meaningful pulse that can be applied to the sample. $\frac{19,20,22,23}{n}$ Thus, the shortest pulse applied was of 6-ns duration although the setup allowed pulses as short in width as 3.2 ns and although the hole emission transient magnitude did not vanish for 6-ns pulses. This was also the reason for not going to even lower temperatures for which the capture time constants became so small that for a 6-ns pulse the hole emission signal was nearly saturated.

TABLE I. The T^2 -corrected activation energies E_a of the various hole levels observed in Mg-doped n^+ -p Si diodes obtained from Fig. 4. All energies are in reference to the valenceband edge E_v .

Level	E_a (eV)	
A	0.34	
B	0.43	
\overline{C}	0.55	
D	0.21	
E	0.24	
F	0.50	
G	0.53	

FIG. 5. Variation of capture cross sections with temperature for the Mg A level.

During the DLTS measurements when a forward bias filling pulse was used to inject minority carriers across the p-n junction, the DLTS peak height of the level did not decrease from the zero bias pulse value even in high injection conditions (it actually increased since the hole filling could take place in the entire space-charge region in forward bias condition). This means that for the A level $\sigma_p^i > \sigma_n^i$.

Figure 6 shows, in absolute values, the photoionization cross section for hole emission (σ_p^0) from the A level as a function of the photon energy $h v$ of the probe light. The initial part of the spectrum (up to $h\nu=0.5$ eV) was obtained using the evacuated optical setup to avoid absorption by water vapors in air which is strong in some of the optical regions in this energy range. The average threshold energy E_i for σ_p^0 obtained from the spectra of the kind presented in Fig. 6 (the take-off energy of σ_p^0) was $E_i = 0.33$ eV.

FIG. 6. Photoionization cross sections for hole emission from the Mg A level.

The spectrum in Fig. 6 was obtained at $T=97$ K. We have measured such spectra at different temperatures ranging from 79 to 114 K and found them to be identical within the experimental error. It was not possible to extend this temperature range since at lower temperatures the shallow acceptors started to freeze out while at higher temperatures thermal emission from the level no longer remained negligible.

V. ANALYSIS OF EXPERIMENTAL DATA FOR THE A LEVEL

The change in Gibbs free energy ΔG_p needed to emit a hole from a deep level to the valence band is related to the hole capture cross section σ_p^t and the thermal emission rate of holes e_p^t at temperature T by the detailed balance relationship²⁴

$$
e_p^{\,l} = \sigma_p^{\,l} v_{\text{th}} N_v \exp(-\Delta G_p / kT) \tag{3}
$$

where N_v is the effective density of states in the valence band. ΔG_p at constant temperature T is related to the change in enthalpy ΔH_p and the change in entropy ΔS_p by the thermodynamic relationship

$$
\Delta G_p = \Delta H_p - T \Delta S_p \tag{4}
$$

so that (3) can be written as

$$
e_p^t = \sigma_p^t v_{\text{th}} N_v \exp(\Delta S_p / k) \exp(-\Delta H_p / kT) \tag{5}
$$

As seen from Fig. 5, σ_b^t for the A level has a T dependence. Since $v_{th}N_v$ has a T^2 dependence, the entire
preexponential factor in (5) has a $T^{1.1}$ dependence Therefore, the slope of the $\log(e_p^t T^{1.1})$ versus $1/T$ plot gives the enthalpy of the A levels as $\Delta H_p = 0.38 \text{ eV}$.

The decrease of σ_p^t with increase in T is characteristic of the so-called cascade capture process in which the holes are captured via a series of closely spaced excited states near the top of the valence band emitting a cascade of phonons. Since the probability of a hole being reemitted to the valence band from one of these excited states before its final capture by the ground state of the level increases with increase in temperature, the probability of final capture decreases. We compare our data with two such models of cascade capture. One model^{25,26} gives the temperature dependence of the capture cross section σ_c^t for the cascade process as

$$
\sigma_c^t = (2\sigma_0 k / m^* s^2) T^{-3} , \qquad (6)
$$

where s is the velocity of sound and σ_0 is a temperatur independent factor. It can be seen that the data in Fig. 5 lies close to a straight line with a slope of -3.1 which is in close agreement with Eq. (6). Hence the experimental data fit well with this model.

In the second model²⁷ the capture takes place in two steps. The hole is first captured by a cascade process to the energy state E_1 above the valence band. At high enough temperatures where the probability for thermal excitation of the hole back to the valence band is much larger than that for its final capture to the ground state

$$
\sigma_p^t = AT^{-2} \exp(E_1/kT) , \qquad (7)
$$

where \vec{A} is a temperature-independent factor. The subsequent steps to the ground state of the center at energy E_2 above E_1 occur at a rate v which is not thermally activated. E_2 is given by

$$
e_p^t = v \exp(-E_2/kT) \tag{8}
$$

Within this model, according to (7), $\log_{10}(\sigma_p^t T^2)$ is plotted against $1/T$ in Fig. 7. Fitting a straight line to the data $E_1 = 14 \pm 5$ meV is obtained. To get E_2 according to (8) we plotted $log(e_p^t)$ versus $1/T$ in Fig. 8 which gives $E_2=0.37$ eV. Thus, according to this model $E_1+E_2\approx 0.38$ eV, in good agreement with the enthalpy obtained from the detailed balance relationship (5). The prefactor v was determined to be between 1.2×10^{13} and 3.7×10^{13} s⁻¹.

Having obtained the temperature variation of e_p^t and σ_p^t for the A level it is straightforward to calculate ΔG_p from (3) and having obtained ΔH_p , ΔS_p is worked out using (4). It is to be noted here that the capture time constants measured in this study are 4-8 times larger than the RC product of the sample in which they were measured. It has been shown²⁰ that for such capture time constants the RC product can affect the accuracy of capture measurements. Thus, the absolute values of our measured σ_p^t are underestimated. The RC product of our samples did not change with temperature and the total observed variation in σ_p^t was small (a factor of \approx 2.5 over the entire temperature range). Thus, the effect of the RC product on the variation in σ_p^t , on which the preceding analysis of data was based, is negligible. However, the absolute values of σ_p^t , required for calculating ΔG_p and and ΔS_p , are affected by the RC product of the sample.

To estimate the influence of the RC product on σ_p^t we have used an approach²⁰ which takes into account the time dependence of the variation in depletion region

FIG. 7. Temperature dependence of the hole capture cross sections for the Mg A level assuming a two-stage capture model. The slope of the line (least-squares fit of the data points) gives the energy E_1 , above the valence band, of the first stage of capture according to Eq.(7). The given equation describes the line when T is in K and kT in eV.

FIG. 8. Thermal emission rates of holes from the Mg A level plotted against inverse of temperature. The slope of the plot gives the energy E_2 of the second stage of capture in the twostage capture model according to Eq. {8).

width, as the excitation pulse is applied to the sample, via the time dependence of the actual voltage change which is a function of the RC product of the diode being excited. Using this approach, the capture is simulated in a computer program which, doing a least squares fit for simulated and measured $S(t_n)$ values having the capture time constant τ as the only fitting parameter, works out the RC product-corrected capture cross section. From such simulations, we have estimated the actual σ_p^t for the A level to be about 2.7 times larger than the directly measured values given in Fig. 5.

Using the corrected σ_p^t values, the ΔG_p and ΔS_p obtained for five temperatures in the range in which σ_p^i and e_p^t have been measured, are summarized in Table II. For comparison, if we use the uncorrected values of σ_p^t then $\Delta G_p = 0.32$ eV and $\Delta S_p = 5.8k$ at $T = 131$ K. The optical ionization energy ΔG_{ρ}^{0} of a hole level is related with ΔG as

$$
\Delta G_p^0 = \Delta G_p + kT \ln(g) , \qquad (9)
$$

where g is the degeneracy factor. ΔG_p^0 values calculated assuming $g=4$ (Ref. 28) are also listed in Table II.

The optical data for the A level presented in Sec. IV was taken in the temperature range of 79 to 114 K. Overlap of temperature ranges for optical and thermal measurements have not been possible due to reasons already explained. The variation in ΔG_p^0 in the range of the thermal measurements is small and is within the error due to uncertainty in absolute values of σ_p^t resulting from

TABLE II. Summary of thermodynamic parameters for hole capture and emission for the Mg ^A level in Si. The σ_p^t values used here are those corrected for the influence of the RC product of the sample. The absolute values of σ_p^t are estimated to be accurate to within a factor of 2.

T (K)	e_n (s^{-1})	$10^{14}\sigma_p^t$ $\rm (cm^2)$	ΔG_p (eV)	$\Delta Sp / k$	$\Delta G_{p}^{0}(g=4)$ (eV)
170 160	303 60.5	2.8 3.4	0.32 0.32	4.3 4.6	0.34 0.34
151	12.1	4.1	0.32	4.6	0.34
141	1.51	5.1	0.33	4.7	0.34
131	0.151	6.7	0.33	4.8	0.34

the influence of the RC product of the samples. We estimate the accuracy of σ_n^t to be within a factor of 2. The average value of $\Delta G_p^0 = 0.34$ eV. This is slightly large than the optical threshold of σ_p^0 which is $E_i = 0.33$ eV.

VI. DISCUSSION

Investigations based on junction space charge techniques (e.g., DLTS) can only in rare cases give information on whether a center is a donor or an acceptor. From the usual DLTS measurements the direct information one gets about a center is whether it thermally emits electrons or holes. However, the cascade capture mechanism demands a ladder of excited states and if such a process is identified for a particular center, the existence of a series of excited states is convincingly proved as, e.g., in the cases of S (Ref. 29) and Se (Ref. 30) in silicon.

The DLTS measurements on the Mg A level show that the thermal hole emission from the ground state to the valence band by far was the dominating emission process. The hole capture cross sections show the characteristic temperature dependence for a cascade capture process. The agreement found between theory for cascade capture and experiment in this work, in addition to the fact that the A level is observed in hole emission, suggests that the hole capture involves excited hole states(s). This would imply that the A level is an acceptor.

The value of 14 ± 5 meV for the bonding energy of the lowest excited state (Fig. 8) participating in the cascade capture is reasonable. If it is assumed that the excited Coulomb states of the ^A level approximately follow the effective-mass theory (EMT), as observed to be the case for several deep acceptors in silicon, 31 the energy of 14 ± 5 meV should be of the order of the energy of the lowest excited hole state observed for shallow acceptors in silicon. The $2P_{3/2}$ state, with its binding energy of 15.5 meV (Ref. 32), is that excited p -like state so that the value of 14 ± 5 meV is satisfactory. The binding energies of the excited Coulomb states scale as Z^2 , where Z is the charge of the center when ionized. This implies that the binding energy of the lowest p -like state of a singly negatively charged acceptor should be about 60 meV. This indicates that the hole emission at the A level would be between neutral and the singly negatively charged state of an acceptor.

The optical ionization energy $\Delta G_p^0 = 0.34$ eV inferred from thermal measurements is slightly larger than the op-

tical threshold energy $E_i = 0.33$ eV for σ_p^0 . This is to be expected if the A level is an acceptor because of the hole transitions via the excited states (compare Fig. 6 with, e.g., the photoionization cross section spectra of Si:S and Si:Se in Ref. 33). Thus the optical measurements agree with the thermal measurements if the A level is an acceptor.

The results of our diffusion experiments show that the A level is the dominant Mg-related level in silicon and if there were doubts that the A level might have been related to some impurities in the Mg source, they were removed by the ion implanted samples. The thermal measurements on the A level can only be explained if it is an acceptor level. Mg on a single substitutional lattice site in silicon is likely to form a double acceptor. Since no Mg acceptor level has hitherto been found, the A level is a good candidate for the missing substitutional acceptor. We expect the Mg A level to be the neutral acceptor. In that case the level C/G could be a possible candidate for thesingly positively charged acceptor state.

In germanium, the group-II elements Be, Zn, and Mg In germanium, the group-II elements Be, Zn, and Mg all give rise to substitutional acceptors. $11-15$ It is interesting to note that the binding energies of the neutral Zn and Mg acceptors in Ge are similar (35.83 and 32.98 meV, respectively) and such similarity between Mg and Zn could be expected in Si as well. From this point of view the similarity of the activation energy for the A level (0.33 eV), and the corresponding energy for the substitutional neutral Zn acceptor which is about 0.32 eV (Refs. 4 and 5) is notable.

It is interesting to note here the Froyen and Zunger, 16 in a recent theoretical treatment, came to the conclusion that Mg can occupy interstitial or substitutional position depending upon the sample preparing conditions. Their treatment suggests that in intrinsic or p -type silicon the stable configuration of Mg should be predominantly interstitial while in n-type silicon it ought to be predominantly substitutional and that a rapid shift of Fermi level during cooling from the diffusion temperature could result in metastability. The A level is experimentally observed in p-type material. However, it is difficult to exactly predict what happens at the high diffusion temperatures and particularly during quenching in a real situation. The conductivity and the conduction type may be drastically altered during these processes. This possibility together with the fact that it has been very difficult to pin down the exact sample preparation conditions for large concentration of the ^A level imply that the experimental results do not necessarily contradict the theoretical predictions of Ref. 16.

To sum up the discussion, there is substantial experimental evidence to prove that the A level is a Mg-related acceptor level and there are sufficient grounds to suggest that it might be the missing substitutional Mg acceptor.

VII. CONCLUSION

We have diffused Mg thermally into n^+ -p silicon diodes and as a result have observed a number of hole levels in the lower half of the band gap of Si. One of these, the A level, has been found to be the dominating Mg level in most samples. Measurements of thermal emission rates, temperature dependence of the capture cross sections, and photoionization cross sections have been carried out for this level. The capture cross-section data fits well with cascade capture models suggesting the A level to be an acceptor. From thermal measurements, the enthalpy of the A-level $\Delta H_p = 0.38$ eV and the energy position $\Delta G_p^0 = 0.34$ eV which is in agreement with the optical threshold energy for hole photoionization cross section, $E_i = 0.33$ eV if the level is assumed to be an acceptor. All the experimental evidence favors the A level to be a good candidate for the substitutional Mg acceptor that had not been observed before.

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