DONOR-ACCEPTOR PAIRS IN SILICON

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ABSTRACT

Stable forms of donor-acceptor pairs in silicon arise from the interaction between negatively charged acceptors and 3d transition element donors, which, occupying interstitial sites, have high mobilities. The atomic and electronic structure of these pairs can be studied in detail by magnetic resonance. The symmetry of centers as observed in the resonance experiments gives strict constraints on the applicable atomic models. The spin of centers is related to their charge state. Hyperfine interactions frequently lead to a specific structure in the spectra, which characterizes the chemical identity of atomic components of the pair. Deep level transient spectroscopy provides a way of sensitive observation of electrically active impurities: to determine their concentrations and associated electronic levels. It allows the study of the kinetics of the pair formation process and thermally induced dissociation; binding energies can be determined. Transformation of pairs as a result of illumination was also observed. Several of the pairs can exist in geometrically different atomic configurations leading to the phenomenon of bi- or multi-stability. In the paper the donor-acceptor pair formation process is illustrated by examples involving the substitutional double acceptor zinc, for which new data became recently available.

INTRODUCTION

Donor-acceptor pairs are a familiar and important form of small impurity complexes in semiconductors. Binding of impurities in the pair is easily understood on the basis of electrostatic attraction between a positive ionized donor and a negative ionized acceptor. If at least one of the impurities has high mobility through the semiconductor crystal, as is commonly the case for impurities on interstitial sites, such pairs are readily formed. The process of pair formation can take place as a result of thermal processing at elevated temperatures but even, more slowly, at ambient temperatures. A detailed description of the pairing phenomenon is already given in an early paper on the interactions among doping defects in silicon [1]. In the more common form of pair formation between singly ionized dopants, such as B_s^- and Fe_i^+ , the binding energies of about 0.65 eV are not very large and pairs are not very stable [2]. When both impurities are doubly ionized binding energies are four times higher. In this case effective impurity gettering can be based on the pair formation process. Pairs sometimes can exist in several different site configurations leading to structural multistability.

This paper will focus on the involvement of zinc together with 3d transition metals in the pairing process in silicon. Recently new results were obtained allowing the description of atomic and electronic structure of such pairs. Zinc diffuses through silicon by a substitutional-interstitial exchange mechanism [3].

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In most papers zinc is reported to occupy the substitutional lattice position at room temperature, with a maximum solubility near 5×10^{16} cm⁻³ [4,5]. In this configuration the impurity behaves as a double acceptor with the ionization levels $2n^{0/-}$ and $2n^{-/2-}$ at about E_v + 0.30 eV and E_v + 0.60 eV, respectively. The early results were obtained by the Hall effect; they were confirmed and more detailed in the following decades by measurements of photoconductivity [6], deep level transient spectroscopy [7-14] and infrared absorption [15-18]. As an alternative structure the configuration of interstitial zinc acting as a double donor has been mentioned. This case is, however, not as well documented as observations are less numerous [4,19].

The transition elements of the iron group generally occupy interstitial sites. Given the open structure of the silicon crystal lattice, they are quite mobile. This particularly applies to the heavier elements in the series. To avoid contamination of crystals with these impurities extremely clean conditions of handling, almost never met in practice, are required. Consequently, as a rule, silicon crystals are unintentionally doped by these transition elements in a rather uncontrolled manner. While at elevated temperatures the impurities will be present as isolated species, upon lowering the temperature they will be involved in clustering processes. The transition elements are electrically active introducing deep electronic levels, of donor and/or acceptor nature, in the bandgap. Due to this activity the clustering processes, leading to precipitates of various sizes and structures, are the cause of electrical instabilities of the silicon crystals. The gettering of highly mobile transition element impurities by stable bonding to zinc acceptors could provide a way of reducing these instabilities.

reducing these instabilities. For the investigation of these phenomena the methods of electron paramagnetic resonance (EPR) and deep level transient spectroscopy (DLTS) have given a wealth of data and insight in the processes. In the next sections of this paper the results of these two methods for the pairing process between zinc as a double acceptor and the donors of transition elements in the 3d series are discussed. The positions of electrical levels of the relevant impurities are summarized in figure 1.

	V .	Cr	Mn	Fe	Zn
Е, 0.2	<u>`</u>	(Cr'Zn_)? (Cr'),	<u>Mn</u>		_
0.4	- <u>v</u>		<u>Mn'</u>	(Fe'Zn)?	-
0.6					- <u></u>
0.4	<u></u>		Mn	<u>(Fe*),</u>	
0.2	(V * Zn -)*		(Mn**Zn_*)*		Zn _

Figure 1. Energy level diagram for substitutional zinc and some elements of the 3d series, and of their donor-acceptor complexes in silicon.

STRUCTURE OF DONOR-ACCEPTOR PAIRS

Pair defects have an intrinsic axial structure. When incorporated in a silicon crystal the combination of symmetry elements of host crystal and defect allows overall trigonal symmetry. Due to distortions of the defect or to specific positions of the pair components the resulting symmetry may be lower, for instance orthorhombic or monoclinic. A defect of such symmetry, i.e. lower than the cubic symmetry of the silicon crystal itself, can be incorporated in the host in a discrete number of distinguishable orientations. The magnetic resonance experiment separately shows the differently oriented defects as their effective g values in a magnetic field of arbitrary orientation are different. The energy of a defect in a magnetic field, with its anisotropic properties taken into account, is described by a g tensor. Since each defect orientations will have its own resonance, the number of different orientations is revealed in the resonance spectrum. For directions of high symmetry, for instance parallel to a <100> direction, symmetry required degeneracies will occur. Because of this, an angular rotation pattern will have a structure with a unique relation to the symmetry of the studied center. In this paper the magnetic resonance spectra of six zinc related do.or-acceptor pairs will be discussed. For unequivocal reference the spectra are given the labels Si-NL34 to Si-NL39. For each of these the symmetry classification with the procedure as outlined before has been determined. For spectrum Si-NL35 the rotation pattern, as given in figure 2, reveals the trigonal symmetry of the associated





Figure 2. Rotation pattern of magnetic resonance fields revealing trigonal symmetry for the Si-NL35 center.

Figure 3. Rotation pattern of magnetic resonance fields revealing orthorhombic symmetry for the Si-NL36 center.

center. Spectrum Si-NL36 arises from a center with orthorhombic-I symmetry as demonstrated by the angular dependence depicted in figure 3. Full analysis of the resonances with the basic spin Hamiltonian H = $\mu_{\rm B}B$.g.S yields the complete g tensors of the defects. These parameters for the studied centers are given in table I. The parameters provide a (usually) unique spectroscopic characterization of fine structure interactions in the centers. When atoms with non-zero nuclear magnetic moments form part

When atoms with non-zero nuclear magnetic moments form part of a center, in addition to the fine structure, hyperfine interactions are present. Under suitable conditions of sufficient isotopic abundance and sufficiently strong interactions the hyperfine couplings between electronic and nuclear magnetic moments give rise to additional structure in the magnetic resonance spectra. As the structure is completely determined by the nuclear spin values and the nuclear abundances it is very characteristic for the nuclei, and consequently, in.most cases, uniquely identifies the nucleus involved. Thus, the hyperfine structure reveals the chemical identity of impurity components in a center. Examples, as relevant to the investigation of donor-zinc pairs in silicon, are given in the figures 4 to 7. Figure 4 illustrates the relatively simple case of splitting due to iron in the spectrum Si-NL38. Using the impurity enriched in its isotope ⁵⁷Fe with nuclear spin I = 1/2 the hyperfine splitting into two components corresponding to $m_{\rm I} = +1/2$ and $m_{\rm I} = -1/2$ is evoked. Remaining isotope ⁵⁰Fe with I = 0 of unavoidable natural contamination is still weakly visible. For spectrum Si-NL36 the presence of chromium was established by intentional doping with ⁵³Cr isotope with nuclear spin I = 3/2 enriched to 97.7%. In this



Figure 4. Magnetic resonance spectrum of the Si:ZnFe center, spectrum Si-NL38, showing twofold hyperfine splitting due to isotope 57 Fe, nuclear spin I = 1/2.



Figure 5. Magnetic resonance spectrum of the Si:ZnCr center, spectrum Si-NL36, showing fourfold hyperfine splitting due to isotope 53 Cr, nuclear spin I = 3/2.

Spectrum Center Symmetry	Fine structure: Electron spin Hamiltonian Parameters	Hyperfine structure: Nucleus, spin Hamiltonian Parameters	
Si-NL34 Si:ZnCu monoclinic-I	S = 1/2 $H = \mu_{B}B.g.S$ $g_{1} = 1.9980, \# [011]$ $g_{2} = 2.0872$ $g_{3} = 1.9912$ $\theta = 32.2^{\circ}$ angle(g_{3}, [100])	${}^{63}Cu$, I = 3/2 H = S.A.I A ₁ = 21.2 MHz A ₂ = 37.5 , A ₃ = 53.0 , θ = 6.0°	
Si-NL35 Si:ZnCr trigonal	S = $3/2$ H = $\mu_B B.g.S$ + $D[S_z^2 - S(S+1)/3]$ g ₁ = 1.9972 g ₁ = 2.0004 D = 70.6 GHz	${}^{53}Cr, I = 3/2$ H = S.A.I $A_{I} = 40.0 \text{ MHz}$ $A_{\perp} = 32.4 \text{ m}$ ${}^{67}Zn, I = 5/2$ H = S.A.I $+ Q[I_{2}^{2}-I(I+1)/3]$ $A_{I} = 2.9 \text{ m}$ Q = 3.2 m	
Si-NL36 Si:ZnCr orthorhombic-I	S = 1/2 $H = \mu_B B.g.S$ $g_1 = 1.9856, \# [100]$ $g_2 = 1.9903, \# [011]$ $g_3 = 2.0119$	${}^{53}Cr, I = 3/2$ H = S.A.I A ₁ = 57.5 MHz A ₂ = 28.9 . A ₃ = 59.1 .	
Si-NL37 Si:Zn?,Cr? monoclinic-I	S = 1/2 $H = \mu_B B.g.S$ $g_1 = 2.0164, \# [011]$ $g_2 = 2.0507$ $g_3 = 1.9981$ $\theta = 43.4^{\circ}$ angle(g_3,[100])		
Si-NL38 Si:Zn?,Fe trigonal	S = $3/2$ H = $\mu_B B.g.S$ + $D[S_z^2 - S(S+1)/3]$ g ₁ = 2.1520 g ₁ = 2.0328 D = 15.9 GHz	57 Fe, I = 1/2 H = S.A.I A _J = 8.6 MHz A _⊥ = 14.1 "	
Si-NL39 Si:Zn?,Cu? trigonal (monoclinic-I?)	S = 1/2 $H = \mu_B B.g.S$ $g_{I} \approx 2.12$ $g_{\perp} \approx 2.02$		

Table I. Spin Hamiltonians and parameters of the EPR spectra Si-NL34 to Si-NL39. =

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Figure 6. Magnetic resonance spectrum of the Si;ZnMn center showing sixfold hyperfine splitting due to isotope ^{55}Mn , nuclear spin I = 5/2. After Ref. 20.



Figure 7. Magnetic resonance spectrum of the Si:ZnCr center, spectrum Si-NL35, showing fivefold hyperfine splitting due to isotope 67 Zn, nuclear spin I = 5/2.

fourfold the expected case splitting as shown in figure 5 is slightly obscured for the particular angle of measurement by accidental coincidences with the fine structure. The earlier reported spectrum of the ZnMn pair [20] is re-produced in figure 6. For the magnetic field parallel to the <111> direction two EPR orientations are distinguishable. For this center with electron spin S = 5/2 all transitions with $\Delta m_S = \pm 1$ between the lev-els from $m_S = \pm 5/2$ to $m_S = -5/2$ are observed. Each of these is split into a sextet by the hyperfine interaction with the manganese nucleus ⁵⁵Mn, nuclear spin I = 5/2, natural abundan-ce 100%. Finally, figure 7 illustrates for spectrum Si-NL35 of the trigonal ZnCr pair the hyperfine structure due to zinc, as a result of doping with isotope ⁶⁷Zn, nuclear spin I = 5/2, enrichment 91.9%. The spectrum is somewhat exotic as it does not show the expected splitting into 2I+1 lines. Instead a fivefold splitting

is observed. Detailed analysis in this case, to be published separately, reveals that mixing of states by quadrupole interactions, suppresses completely the normally allowed transitions with $\Delta m_I = 0$. The visible resonances correspond to "forbidden" transitions with $\Delta m_I = \pm 1$, of which five are possible. Conclusions about the impurities in the spectra based on these hyperfine interaction structures are included in table I.

Having established the atomic structure on the basis of observable resolvable hyperfine interactions, it remains to develop the model of the electronic structure. In the ionic pair model the zinc acceptor will probably assume a negative charge state, whereas the transition metal donors are likely to be positively charged. The acceptor zinc can be in the doubly ionized configuration $Zn^{2-} = [Ar]3d^{10}4s4p^3$, where the electrons in the fourth shell provide the binding of the substitutional zinc atom to the crystal. The spin of this Zn^{2-} state is $S_{Zn} = 0$. The various transition metal impurities possess a different number of electrons and, besides, they can be in different charge states as singly or doubly ionized donors. For instance, iron on an interstitial lattice position is a single donor, with level at about $E_v + 0.40$ eV (see fig. 1). In its ionized state the electronic configuration is $[Ar]3d^7$, having three holes in the d shell. Spin alignment, as prescribed by Hund's rule and experimentally verified for this ion, results in spin $S_{Fe} = 3/2$. Just adding the spin values of the components, the spin S = 3/2 is predicted for the ZnFe impurity pair. This is in agreement with the magnetic resonance parameters of the spectrum Si-NL38, which, prescribing g values near 2, is uniquely analyzed only with spin S = 3/2. The EPR spectrum Si-NL38 is thus understood to arise from a Zn^2-Fe^+ donor-acceptor pair, with an overall negative charge due to acceptor character. Additional observations confirming the identification are found in the g tensor. The parameters $g_f = 2.1520$ and $g_1 = 2.0328$, given in table I, yield for the isotropic part of the tensor $(g_f + 2g_1)/3 = 2.0725$. As evidenced by many other examples [21], the g value around 2.07 is typical for iron

A similar interpretation can be given for the ZnMn pair, reported earlier [20]. In this case again the spin on the doubly ionized zinc atom is given as $S_{Zn} = 0$. Manganese can act as a double donor following the reaction $Mn^{\circ} \neq Mn^{2+} + 2e^{-}$. The impurity favors the interstitial position requiring no bonding electrons to the crystal and the electronic configuration [Ar]3d^o with halffilled d shell. Again, according to Hund's rule and experiment, the resulting spin is $S_{Mn} = 5/2$. In the pair configuration (ZnMn)^o $= Zn_{subst}^{2-}Mn_{int}^{2+}$ the observed electron spin S = 5/2, see figure 6, finds a natural explanation. The hyperfine interaction parameters with the manganese ion, isotope ⁵⁵Mn with nuclear spin I = 5/2 and 100% natural abundance, are given in the original literature as $A_f = -154.2$ MHz and $A_{\perp} = -150$ MHz. The interaction appears to be nearly isotropic, with trace $A = (A_f + 2A_{\perp})/3 = -151.4$ MHz, indicating only a small influence of the trigonal field in the center. A comparison with the isolated manganese impurity in the silicon host can therefore be made. For interstitial Mn^{2+} , the reported value is A = -160.21 MHz; for substitutional Mn^{2-} , A =-121.5 MHz [20]. Both cases refer to electronic configuration 3d⁵ and S = 5/2. Bonding in the pairs is likely to result in some hybridization and delocalization of the d electrons and therefore to reduction of the hyperfine interaction with the transition metal ion. The value as measured for the pair is therefore consistent only with manganese on the interstitial site. The observations confirm the defect model.

In the two above presented pair models, the electron spin arises from and is localized on the transition metal ion. In agreement with this description, the hyperfine interaction with this impurity is strong and easily visible in the spectra by the induced hyperfine splitting; see figures 4 to 6. In this model, no spin density is localized on the zinc ion. In agreement with the model the hyperfine interaction with zinc for all of the pairs is observed to be small. In most cases it leads only to line broadening, not resolvable in EPR. In the case of the ZnMn center and also of the ZnCr pair, spectrum Si-NL35, the zinc interaction could be resolved, but it was indeed found to be small, with a magnitude of a few MHz only.

Also for the other pairs as represented in table I similar models can be proposed for their description. Not in all cases, however, the agreement is as straightforward as for the examples discussed above.

STABILITY OF DONOR-ACCEPTOR PAIRS

The simple basic interaction responsible for the binding in donor-acceptor pairs is the electrostatic attraction between positive donors and negative acceptors. For singly ionized impurities on the nearest neighbor position in the silicon lattice, at a distance of $r = 2.35 \times 10^{-10}$ m, the energy $e^2/4\pi\epsilon_o\epsilon_r r$ is calculated to be around 0.52 eV. As this number is smaller than the bandgap energy of silicon, one may expect that reactions can be induced related to the recombination energy of free carriers. In other words, pairs might be unstable against illumination. Light-induced



Figure 8. Light-induced transformation between Si:ZnCu pairs, spectrum Si-NL34 and spectrum Si-NL39.



Figure 9. Light-induced transformation between Si:ZnCu pair, spectrum Si-NL34, and Si:ZnCr pair, spectrum Si-NL36.

enhancement of the degree of dissociation of the boron-iron pair is indeed reported [22]. A more modest manifestation of illumination effects is the transformation of one pair into another one. Examples as observed in the recent studies of zinc related pairs are given in the figures 8 and 9. The former of the two figures gives results of the photo-EPR experiment on a sample containing Si-NL34 and Si-NL39 centers. Both centers were identified as ZnCu complexes. Hence, in this case the light-induced conversion may consist of the transformation of basically the same center between two different configurations, leading to centers of different symmetry. Alternatively, the different symmetry or lattice relaxa-tion may be related to different charge states of an impurity pair on basically the same lattice sites. By contrast, the example given in figure 9 is certainly different. Here, also by photo-EPR, the light-induced conversion is observed between centers which are definitely identified as a ZnCu pair (spectrum Si-NL34) and a ZnCr pair (spectrum Si-NL36). In this case the reaction can be under-stood as charge transfer between two different centers. As a word of caution one may add at this point that the transition metal impurities are commonly unintentionally introduced as a result of thermal treatment and doping procedures. Their presence is therefore often unknown. Interpretation of photo-EPR experiments should then be done with corresponding reservation. In principle the spectral dependencies as observed in the photo-EPR experiments give information on the electronic levels associated with the centers. However, a convincing correlation between results from photo-EPR and level determinations by other spectroscopical measurements appears particularly complex and, so far, has not been achieved.



Figure 10. Configuration coordinate diagram and microscopic models for the iron-aluminum donor-acceptor pair in silicon, in trigonal and orthorhombic configurations. After Ref. 24.

The validity of the ionic model for donor-acceptor pairs has been examined closely in the well documented case of FeAl pairs in silicon. By magnetic resonance two atomic configurations of this pair atomic were identified [23]. The pair with smallest impurity disfollowing from a substitu-tional aluminum atom with an interstitial iron neighbor along a <111> direction of the crystal. It has the trigonal EPR spectrum Si-NL27 associated with it. Excitation of the iron ion to the next more distant position creates a pair of orthorhombic symmetry, as observed in the EPR spec-trum Si-NL28. Configurational bistability has been carefully studied for this complex [24]. The results are concisely and elegantly summarized in a configuration coordinate diagram. as given for the FeAl complex in figure 10. The energies for ionization of the donor pairs,



Figure 11. Deep level transient spectroscopy of silicon double-doped with zinc and manganese. After Ref. 9.



Figure 12. Equilibrium constant for the donor-acceptor pairing reaction between Zn^{2-} and V^{2+} in silicon, as a function of the temperature. After Ref. 9.

as derived from the DLTS spectra H1 and H2, are 0.20 and 0.13 eV, respectively [24,25]. The energies related to conversion from one to the other configuration are represented by the potential barriers with heights 0.50 and 0.64 eV in the positive and neutral charge states, respectively. They are derived from the temperature dependence of the kinetics of conversion reactions which re-establish thermal equilibrium populations after a perturbation. The energy difference of 0.14 eV for the ground states of the two configurations accurately matches the difference in electrostatic energy for the two interatomic configurations with the separations by 2.35×10^{-10} m and 2.73×10^{-10} m of the ions in the pair. The applicability of the model as illustrated by the configuration coordinate diagram supports the validity of the ion pair description.

In figure 1 already the electronic levels obtained from deep level transient spectroscopy (DLTS) are presented. This method also allows to determine quite accurately the concentrations of the electrically active centers. In the donor-acceptor pairing reactions all components involved show such electrical activity and can thus individually be observed. This is shown in figure 11 for the pairing reaction between zinc and manganese. Monitoring the intensities of the DLTS peaks as a function of the pair formation conditions, the kinetics of the reaction can be followed. Such an experiment was carried out in detail for the reaction between zinc and vanadium, represented by

$Zn^{2-} + V^{2+} \rightleftharpoons (ZnV)^{\circ}$. (1)

In these experiments the equilibrium of the reaction was established at the desired temperature, and then frozen by rapid thermal quenching, to allow the measurement of concentrations in a low-temperature DLTS experiment. In the mass action description the equilibrium is governed by the expression

$$[2nV]/[2n][V] = K(T) = K_{0}exp(U/kT).$$
 (2)

The temperature dependence of the reaction equilibrium constant K is exponentially dependent on the binding energy U of the pair. Results for the zinc-manganese/vanadium reactions are given in the figures 11 and 12. Figure 11 is a typical example of a DLTS spectrum from which concentrations of species are derived. Their interpretation following equation 2 is illustrated in figure 12. The slope of the straight line gives U = 2.7 eV for the binding energy of this double donor-double acceptor pair. The nearly exact fourfold increase of binding energy in comparison to the pairs with singly ionized components, FeB [2] or CrB [26], is direct confirmation of the validity of the ionic bonding model. The high binding energy of pairs derived from multiply ionizable components gives high thermal stability to these pairs. It suggests that transition element impurities can be gettered effectively by binding them to zinc acceptors.

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CONCLUSIONS

The process of donor-acceptor pair formation in silicon has been reviewed. The microscopic and electronic structure of such complexes as well as methods by which the relevant models could be established have been described. As a practical illustration be established have been described. As a practical illustration of the donor-acceptor pairing process the recently reported zinc-transition metal impurity complexes have been discussed. The existence of a vast variety of these defect centers and large binding energy as resulting from double acceptor character assumed in their structure by zinc suggests that transition element impurities, whose presence has detrimental effects in silicon device manufacturing, can effectively be gettered by intentional doping with zinc.

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