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Hydrogen Passivation of Double Donors in Silicon

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ABSTRACT

The interaction between hydrogen and two representative examples of double donors in silicon, i.e., sulfur and the thermal donor, has been studied by means of magnetic resonance spectroscopy. The passivation process was found to transform a double donor into a single donor which is paramagnetic in the neutral charge state. Magnetic resonance spectra associated with the passivation products consisting of the double donor and a single hydrogen atom have been identified. Implications on the microscopic structure of both centers are discussed.

INTRODUCTION

Hydrogen in silicon

The behavior of the hydrogen dopant is at present among the highlights of silicon materials science. As the silicon device technology reaches still higher levels of sophistication and miniaturization the importance of impurity reactions becomes more apparent. The hydrogen dopant and its influence on other defects present in the material is a prime example of such an impurity reaction. This is a consequence of several factors:

- Atomic hydrogen can relatively easily diffuse at moderate temperatures, with a charge-state-dependent activation energy of about 0.5 eV. The larger hydrogen aggregates, including the H_2 molecule, are believed to be immobile.
- Recently hydrogen has been recognized as one of the major contaminants in high-grade commercial silicon. It is easily introduced in a variety of ways, which include some standard techniques as used in silicon device manufacturing.
- Hydrogen is known to form stable complexes with itself - molecules, platelets - and with electrically active impurities of both shallow and deep character.

The last aspect is evident from characteristics of hydrogen diffusivity; it is very high at low concentrations but levels off for higher ones, when trapping becomes important. The reduction due to trapping is usually expressed as an effective diffusion coefficient $D_{eff} = D_H \cdot t_F/(t_B + t_F)$, where t_F and t_B represent the times that the hydrogen atom spends in isolated and bound states, respectively. The fact that the hydrogen concentration is effectively controlled by (impurity) trapping results also in a very low solubility limit of about 10^5 cm^{-3} at 300 °C. Another important consequence of trapping is most probably the so-called *super diffusion* of hydrogen, i.e. stress-induced enhancement of diffusion; in reported experiments an increase by 5 orders of magnitude has been measured [1].

Passivating action of hydrogen

Upon interaction with (atomic) hydrogen the electrical activity of various kinds of impurities can be terminated. This process, known as passivation and first reported by Pankove *et al.* [2] for shallow acceptors in silicon, is a most spectacular demonstration of an impurity reaction in semiconductors.

For shallow impurities passivation was found to be particularly effective ($\approx$ 90%) in the case of acceptors. For donors the passivation process proceeds much slower and is never very effective. The passivation mechanism can be monitored by several different experimental techniques. Among these DLTS (deep-level transient spectroscopy) and local-vibrational-mode spectroscopy have been most widely used. The most important conclusion was that the observed passivation involves close bonding between the hydrogen atom and the electrically active impurity rather than compensation, since, upon exposure to atomic hydrogen, the decreased electron density is always accompanied by a simultaneous rise of mobility [3]. Theoretical calculations followed and it is now well established that in the case of substitutional shallow acceptors passivating hydrogen takes a bond-centered position between the acceptor atom and a nearest-neighbor silicon. In case of group V donors the lowest energy configuration was found with the hydrogen antibonded on the nearest neighbor silicon atom. One should note here that the antibonding position on the donor atom itself was also found to be a local minimum-energy configuration, with the analogous passivating action.

Also hydrogen passivation of deep impurities has been reported, and among them of double donors.

Hydrogen passivation of double donors

In this paper we report on EPR (electron paramagnetic resonance) and ENDOR (electron nuclear double resonance) investigations of two double donor (DD) centers in silicon: substitutional sulfur and thermal donors (TD's). The passivation effect for both centers has been concluded earlier; the aim of the current study is to elucidate microscopic structural aspects of the impurity complexes formed during the passivation process. The EPR/ENDOR experiments were carried out in a custom-built superheterodyne spectrometer operating at 23 GHz and tuned to dispersion, at a temperature of about 9K.

A double donor is characterized by a possibility to exist in three different charge states within the bandgap of silicon: neutral D^0 and two ionized ones D^+ and D^{++}. In the ground state usually the singly ionized state is paramagnetic with spin S=1/2, although also the excited paramagnetic S=1 state of a neutral donor is sometimes observed. In case of a single donor, which is paramagnetic in its neutral charge state, the paramagnetism is lost by passivation. Consequently magnetic resonance, which proved itself to be extremely powerful in structural studies of defect centers, cannot be applied to passivated single donors (or acceptors). For a double donor the situation is different: in principle it is possible to imagine that it might be passivated with one or two hydrogen atoms. This could create the situation that a passivated center would be accessible to magnetic resonance spectroscopy, since a singly passivated double donor in its neutral charge state would be paramagnetic with spin S=1/2. One should note that for double acceptors, for which a similar reasoning applies, the actual observation of such a paramagnetic state may be hindered by stress effects, in view of the presence of stress-sensitive orbital momentum components.

The effect of (hydrogen) passivation is also expected to be visible in infrared (IR) absorption. Again, for a single donor one expects that electronic transitions from the effective-mass states disappear upon passivation. For a double donor a two-stages process may be envisaged: upon passivation with one hydrogen both (helium-like) series of electronic transitions should vanish and, simultaneously, a new (hydrogen-like) series of transitions should appear. Upon eventual further passivation with a second hydrogen no electronic transitions should be observed. In addition to these the IR spectroscopy should in both cases reveal local vibrational modes of hydrogen atoms bonded within the complex.

The two centers chosen for the current study represent different categories of defects. Substitutional sulfur is a point defect and the TD is the most studied example of an extended defect of multispecies character. While substitutional sulfur is relatively simple and has been described to some detail before, the microscopic structure of the TD center, despite an enormous amount of research, presents a notorious problem and is among the most challenging issues in the materials science of silicon.

SULFUR - HYDROGEN COMPLEXES

Substitutional chalcogen impurities represent a classical example of double-donor centers in silicon. In the past these dopants have been subjected to intensive investigations and, as a result, their properties are well understood. Most of these double donors, among them the isolated S and also the S-S pair, have been identified by EPR/ENDOR and DLTS spectroscopies. In particular Pensl *et al.* [4] reported on a variety of double donor levels related to isolated chalcogens and their complexes. Upon exposure to hydrogen plasma passivation of the electrical activity of chalcogens has been established. For sulfur the most important findings [5] can be summarized as follows:

- Both double donor levels are passivated (in one step).
- All sulfur-related centers are passivated, regardless of their (original) relative concentrations.
- Upon passivation of the double donor levels no new sulfur-related levels can be detected in the gap.
- The electrical activity can be fully recovered by a 10-minutes heat treatment at 500 °C of the passivated centers.

One should note here that the above-listed conclusions do not necessarily imply that two hydrogen atoms take part in the passivation process. To decide on this issue the kinetics of the process should be studied; this was shown to involve a single step mechanism, being thus consistent with the participation of a single hydrogen atom. In line with the introductory remarks such a process is expected to lead to a simultaneous generation of new single donor levels in the gap; these were, however, not detected.

Theoretical treatment of the sulfur passivation process has been attempted by Yapsir *et al.* [6], who have examined various configurations of S-H and S-H_2 complexes. For the S-H defect the lowest-energy configuration was found with the hydrogen atom in a distorted bond-centered position of monoclinic symmetry. However, local minima were also found for both antibonding positions of hydrogen on sulfur and nearest-neighbor silicon atoms. Further, from thermodynamic considerations, it was concluded that, while S-H and S-H_2 complexes were both stable in an environment with excess of sulfur, the sulfur-hydrogen pair would be preferentially created. In this way the first-order kinetics, as reported by Pensl *et al.*, could be explained.

More recently the hydrogenation of sulfur donors was also investigated in IR absorption spectroscopy by Peale *et al.* [7]. They have observed electronic transitions of new sulfur-hydrogen complexes of an effective-mass single donor character; these were apparently created upon hydrogen passivation of sulfur-related double donors. The newly found centers were identified as singly passivated species and were suggested to be paramagnetic in their neutral charge state.

In the current study we have used n-type, phosphorus-doped silicon of float-zoned (FZ) and Czochralski-grown (Cz) types. The samples, of typically 1.4 x 1.4 x 10 mm^3 size, were first diffused with sulfur (1370 °C, 48 h) and subsequently with hydrogen or deuterium (1250 °C, 1/2 h). Following the diffusion step the samples were quenched to room temperature.

The EPR measurements on thus prepared samples revealed the presence of two new EPR spectra [8]. These, labeled further Si-NL54 and Si-NL55, were partly overlapping and could be resolved only by the field-scanned ENDOR (FSE) technique, as depicted in Fig.1 for the magnetic field parallel to a <100> crystallographic direction (for details of the technique see, e.g., Ref.9). The observed spectra showed splitting in two components being thus indicative of the hyperfine interaction with a 100% abundant nucleus with nuclear spin $I=1/2$; in view of the sample preparation hydrogen was an obvious candidate. This was confirmed by ENDOR measurements where spectra symmetric with respect to the Zeeman frequency of a free proton have been observed. The involvement of a single hydrogen atom in each of the newly reported centers is also consistent with the measurements on samples doped with deuterium. As expected, due to a higher nuclear spin and lower g_N value, the hyperfine structure could not be resolved for these samples. Using the hydrogen hyperfine interaction the spectra of Si-NL54 and Si-NL55 centers could be separated and their angular dependencies were measured in the FSE mode. Both spectra have the same <111> axial symmetry with a somewhat different degree of anisotropy.

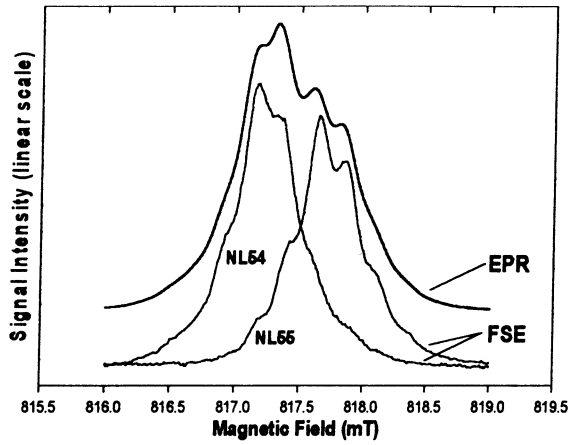

Figure 1: *EPR and FSE spectra of Si-NL54 and Si-NL55 centers for magnetic field $\vec{B}$ ||<100>, recorded at a temperature T=9 K for the sample diffused with hydrogen and natural sulfur, microwave frequency ν=22.8809 GHz.*

In order to conclusively establish the participation of sulfur in the Si-NL54 and Si-NL55 centers samples doped with isotopically enriched sulfur (25% ^{33}S, I=3/2) were also prepared. The EPR and FSE spectra recorded for these samples displayed a clearly resolved hyperfine interaction with one sulfur nucleus - see Fig.2. The fact that identical centers were generated regardless of the isotopic composition of sulfur could be fully confirmed by hydrogen ENDOR which was found to be identical in both cases.

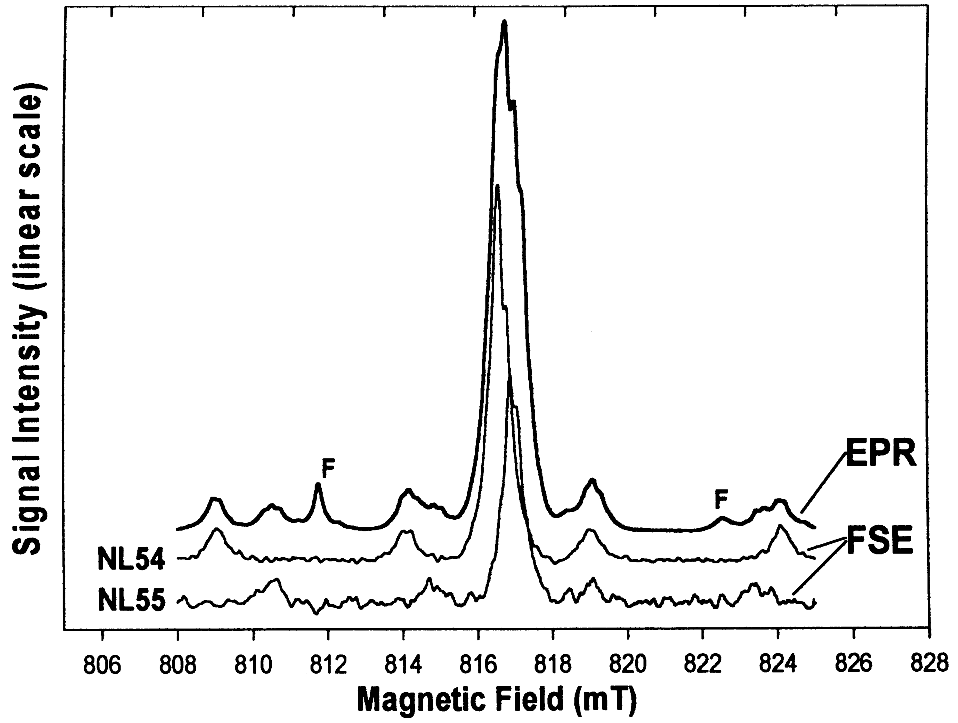

Figure 2: *EPR and FSE spectra of Si-NL54 and Si-NL55 centers for magnetic field $\vec{B}$ ||<100> in the sample diffused with hydrogen and isotopically enriched sulfur. To each central line (I=0) belongs a fourfold spectrum (I=3/2) showing the involvement of a single ^{33}S nucleus (the lines labelled F belong to a different spectrum). The spectra were recorded at a microwave frequency of ν=22.8637 GHz and at a temperature of 9 K.*

The EPR and FSE-recorded spectra could be fitted with the spin Hamiltonian

$$\mathcal{H}_S = \mu\vec{B}\cdot\mathbf{g}\cdot\vec{S} + \vec{S}\cdot\mathbf{A}\cdot\vec{I} + \vec{I}\cdot\mathbf{Q}\cdot\vec{I},$$

where **A** and **Q** represent the hyperfine and the quadrupole interactions with the sulfur nucleus, and spins are S=1/2 and I=3/2, consistent with the observed ENDOR patterns. The values of the tensor components resulting from the fitting are shown in Table I.

Table I: *Spin-Hamiltonian parameters for the Si-NL54 and Si-NL55 spectra, as obtained from computer fits of the FSE angular dependencies. Electron spin is S=1/2. Hyperfine and quadrupole tensor parameters correspond to the interactions with the ^{33}S sulfur nucleus.*

	Si-NL54	Si-NL55	unit
$g_{\parallel}$	$1.99886 \pm 4\times10^{-5}$	$1.99823 \pm 4\times10^{-5}$	
$g_{\perp}$	$2.00126 \pm 4\times10^{-5}$	$1.99974 \pm 4\times10^{-5}$	
$A_{\parallel}$	143.1 ± 2.7	124.0 ± 2.6	MHz
$A_{\perp}$	137.7 ± 2.7	117.9 ± 2.6	MHz
$Q_{\parallel}$	6.6 ± 2.7	5.0 ± 2.6	MHz
$Q_{\perp}$	-3.3 ± 2.7	-2.5 ± 2.6	MHz

Based on the above presented experimental data we identify the Si-NL54 and Si-NL55 centers as two configurations of a sulfur-hydrogen pair, i.e. a substitutional sulfur (double) donor passivated by one hydrogen atom. Consistent with the S=1/2 spin value we assign the spectra to the neutral charge state of such a complex. Such an identification is further supported by IR absorption measurements carried out on the sample used for the magnetic resonance experiment. These revealed the presence of the characteristic $1s \leftrightarrow 2p$ electronic transitions assigned earlier to the S-H donor [7]. The proposed substitutional position of sulfur follows rather naturally from the studies on isolated sulfur and also from the donor character of the observed centers. Following the experimental findings the position of the hydrogen atom has to be along a <111> direction with respect to sulfur. This leaves three possibilities: bond-centered (BC), and 2 antibonding (AB) sites on S and nearest-neighbor Si, respectively. We note here that our results do not support the conclusions of Yapsir *et al.* [6] who predicted a distorted BC site for hydrogen in the S-H complex. On the other hand they are certainly consistent with the first-order kinetics of sulfur donor passivation, as concluded from the DLTS studies [5].

THERMAL DONOR - HYDROGEN COMPLEXES

As already mentioned TD's are among the most complicated defect centers in silicon and full understanding of their microscopic picture is still missing. In addition to many other experimental techniques also magnetic resonance spectroscopy has been used to study these centers. As a result two EPR spectra, Si-NL8 and Si-NL10, both of orthorhombic-I symmetry are now associated with TD's. One of them, Si-NL8, has been identified as a singly ionized state TD^+; the microscopic identification of the second one still poses a problem, although some suggestions have been put forward [10]. Soon after the passivating effect of hydrogen was discovered also passivation of TD's has been reported [11]. The passivation degree was found to be about 70% which is somewhat less than for shallow acceptors but much more than for shallow donors. Passivation of both electrical levels of TD has been observed, but the actual number of H atoms taking part in the process (one or two) was not established. In the same study some effort has been made in order to relate the hydrogenation mechanism to the microscopic structure of the TD's; it has been suggested that the "earlier" TD's, which are generally believed to have a more simple structure, were more readily passivated.

However, the passivation is not the only effect that the presence of hydrogen has on TD's. Murray *et al.* [12] reported on enhanced TD formation for Cz-Si exposed to hydrogen plasma. The observed effect was accompanied by an increased loss of interstitial oxygen and in this way the long-assumed link between the two processes was for the first time demonstrated. The report of Murray *et al.* confirms an early work of Fuller and Logan [13], who observed an increased TD formation rate for silicon crystals grown in hydrogen atmosphere. The surprising result of Ref.12 was that by SIMS (secondary-ion mass spectrometry) measurements no presence of hydrogen has been detected in regions where the increased generation of TD's has taken place.

A possible explanation of the hydrogen-enhanced generation of TD's was offered by Estreicher [14]. The proposed mechanism involved two important steps. Firstly, the presence of a near-by interstitial hydrogen atom (T_d site) lowers the barrier for oxygen diffusion between two puckered bonded interstitial positions from the usual value of 2.56 eV to about 1.25 eV. Secondly, in the vicinity of an interstitial oxygen a hydrogen atom can easier transfer from the T_d site to a BC site; the activation energy for such a process is then lowered from about 2 eV to about 1.34 eV. The proposed explanation could account for an increased TD formation simply by enhanced oxygen diffusion. An equally important result of the mentioned study was that no preferential bonding between oxygen and hydrogen has been found.

The coexistence of both effects presents a considerable contradiction: on one hand the presence of hydrogen accelerates the formation of TD's, and, on the other, it lowers the TD concentration by terminating their electrical activity by passivation. The mechanism proposed by Estreicher offers also the possibility of a new reaction, which is not available for a "standard" passivation; it allows for a situation in which the formation of a donor center and its passivation are accomplished in a single step. As a result of such a mechanism only, or predominantly, TD's in the passivated form would be created. Yet another important consequence of hydrogen-enhanced oxygen diffusion could be a conclusion that the TD passivation process would involve a single hydrogen atom. This follows directly from the fact that the mechanism enhancing oxygen diffusion is mediated by a single hydrogen atom. Following the earlier discussion we could then expect a new paramagnetic state of the TD center to be created - a $(\mathrm{TD\text{-}H})^0$ complex. Being aware of the above we undertook to investigate a possible involvement of hydrogen in the structure of the Si-NL10 center whose particular microscopic assignment is a matter of considerable controversy.

In a sample with a strong Si-NL10 signal but otherwise not intentionally doped with hydrogen we have indeed detected a new ENDOR spectrum, which appeared to be symmetric with respect to the Zeeman frequency of a free proton [15]. By recording the ENDOR spectra for different resonance field values and monitoring the frequency shift - see Fig.3 - we unambiguously identified hydrogen as being responsible for the detected hyperfine interaction. Having established that, we intentionally doped a series of samples with hydrogen. These were prepared from commercially available high-grade Czochralski silicon; hydrogen was introduced by sealing a sample in a quartz ampoule with a few milligrams of water and exposing it to a short high-temperature heat treatment (1250 °C, 1/2 h) followed by a quench to room temperature. Subsequently, a TD generation heat treatment at 470 °C was performed. In all thus prepared samples a hydrogen ENDOR spectrum similar to that depicted in Fig.3 was observed. Once recognized, traces of the same spectrum could also be detected in nearly

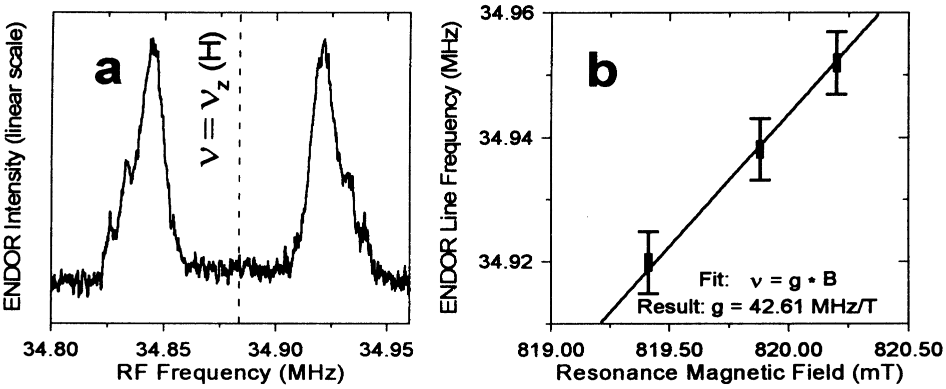

Figure 3: *The hydrogen ENDOR spectrum observed in a Cz-Si:Al sample after 470 °C, 55 h heat treatment.* (**a**) *Spectrum recorded with $\vec{B}$ ||<110> direction, B = 819.324 mT.* (**b**) *displacement of the ENDOR line as a function of the magnetic field shift fitted with a linear function. The value of the proportionality coefficient is equal within the experimental accuracy to the nuclear Zeeman frequency of a free proton.*

all the samples which did not undergo the hydrogen diffusion. This result confirms the wide-spread hydrogen contamination of commercially available high-grade silicon crystals.

The hydrogen ENDOR spectrum was found to be a superposition of independent components. By the FSE technique we were able to show that each of them was related to a different Si-NL10 species. In this way the multispecies character of the Si-NL10 centers, established before on the basis of the oxygen and aluminum ENDOR studies [16], has been confirmed. The angular dependence of the hydrogen ENDOR was studied in a hydrogen-doped Cz-Si:P sample. Preliminary analysis revealed in this case the presence of two similar hyperfine tensors which could be assigned to two different Si-NL10 species. The symmetry of both tensors was established as triclinic; their fitted values are summarized in Table II. As can be concluded, the hyperfine interaction was found to be very small explaining that its presence could not be noticed in an EPR experiment. The spin localization on the hydrogen nucleus, estimated in the framework of LCAO analysis, is minute: ≈ 0.006%.

The presence of a hydrogen component poses again the question of the microscopic structure of the Si-NL10 center and its relation to TD's. We begin here by noting that although the spin localization on a hydrogen nucleus is very small, its magnitude is nevertheless comparable to that found earlier for oxygen [16]. We recall further that similarly low localizations were found also for all the other thus far detected constituents of the Si-NL10 center. At the same time the lack of resolved hyperfine structure in the Si-NL10 EPR spectrum excludes the existence of such a (magnetic) site. We conclude therefore that hydrogen plays an important role in the Si-NL10 defect structure.

Table II: *Hyperfine parameters for the fitted hydrogen tensors of the Si-NL10 center.*

Tensor	i	A_i, kHz	$\mathbf{n}_i$
H-$G1$	1	55.0	(0.727, 0.606, 0.322)
	2	114.7	(-0.280, 0.690, -0.667)
	3	84.3	(-0.627, 0.395, 0.672)
H-$G2$	1	54.3	(0.735, 0.616, 0.286)
	2	103.4	(-0.392, 0.729, -0.562)
	3	84.3	(-0.554, 0.301, 0.777)

Another crucial point for the microscopic identification of the Si-NL10 center is the number of hydrogen atoms involved in the structure of the defect. Such information can be often obtained from the intensity ratio of the components of the EPR spectrum split by the hyperfine interaction. In this particular case, however, in view of the small magnitude of this interaction and also the notorious multispecies character of the center, this standard method cannot be used. Nevertheless, the number of the H atoms can deduced from the symmetry considerations of EPR and ENDOR spectra [16]. As already mentioned, the symmetry of the hydrogen ENDOR shell has been established as triclinic. In order to find out the number of atoms constituting the shell it is crucial to determine (with the same precision) the real symmetry of the corresponding EPR spectrum. This could be accomplished in an FSE experiment which has shown that the maximum intensities of the individual orientations of the triclinic ENDOR tensor were attained for different values of magnetic field. Such a result indicates the presence of unresolved splitting of the EPR spectrum and proves its true triclinic symmetry. Based on this result we conclude that only a single hydrogen atom is present in the structure of the defect. As to the actual position of this atom we restrict ourselves to note that it must be off the 2-fold axis of the original structure of the thermal donor, and then either in the symmetry plane perpendicular to the one containing oxygen atoms [16], or out of both mirror planes. While the above-presented conclusion needs to be further confirmed by a more detailed analysis of the hydrogen (and possibly deuterium) ENDOR data we point out that such a position of the hydrogen atom is not in agreement with a structural model for a TD-H complex as proposed by Deák *et al.* [17].

The current findings allow us to propose a new microscopic identification of the Si-NL10 center as a neutral charge state of a singly passivated TD. Such an identification is consistent also with all the characteristic features of the Si-NL10 structure and its similarities with the Si-NL8 [10]. At the same time it accounts for an evidently more shallow character with respect to Si-NL8.

As an interesting side remark we note that the formation of a TD-H pair is not expected in germanium. Indeed, in line with that, only a singly ionized charge state TD^+ (S=1/2) and an excited neutral state $(TD^0)^*$ were detected by EPR [18].

CONCLUSIONS

We have shown that high-temperature heat treatment in water vapor of monocrystalline silicon samples leads to an effective passivation of double donors. The passivation process involves the formation of DD-H pairs which have single donor character and are therefore paramagnetic in their neutral charge state. Two examples of such complexes, S-H and TD-H pairs, were detected and studied by EPR/ENDOR spectroscopy. We note that, although similar by nature, both complexes must differ significantly in the detailed bonding configuration of hydrogen. This is evident by their different formation/dissociation characteristics; while TD-H complexes are generated by heat treatment at about 470 °C, the S-H pairs are dissociated in this temperature range. Following the presented experimental results the theoretical models developed thus far for both centers will have to be reconsidered.

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