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Capacitance-transient spectroscopy on irradiation-induced defects in Ge

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Recent studies of room-temperature irradiation-induced defects in Ge using space-charge capacitance-transient spectroscopy are reviewed. From these measurements only two defect complexes have been unambiguously identified until now: the *E*-center (the group-V impurity -vacancy pair) and the *A*-center (the interstitial oxygen-vacancy pair). However, contrary to silicon where each of these centers introduces only one energy level, in germanium the *E*-center has three energy levels corresponding to four charge states (=, –, 0, +), and the *A*-center has two levels corresponding to three charge states (=, –, 0). Another feature specific to each material is the anneal temperature. Both centers disappear below 150°C in germanium, whereas in silicon the *E*-center anneals out at ~150°C, depending on the charge state, and the *A*-center is stable up to 350°C.

Keywords: Ge-defect, deep level transient spectroscopy (DLTS), Laplace DLTS.

1. Introduction

In the beginning of the 1960s, with the invention of the planar process, silicon definitively won the battle over germanium in the microelectronics. For the next nearly 40 years almost all semiconductor research and development went into the silicon area, some went into the III-V semiconductors, and very little into germanium. Today, silicon is the dominant material for microelectronics with more than 98% of all semiconductor devices made from it, III-V compounds dominate the semiconductor -optics industry mainly due to their direct bandgaps, and Ge has a small market share as, *e.g.*, gamma-ray and infrared detectors.

The recent use of SiGe alloy layers in, *e.g.*, hetero-junction bipolar transistors [1], and the possibility of exploiting the high-mobility features of Ge to fabricate high -performance CMOS transistors [2] have, however, accentuated the need to understand irradiation-induced defects in Ge which are poorly understood compared to Si.

Most investigations in this direction date indeed 40 years back [3], before the technology clearly decided in favor of silicon. Consequently, only sparse publications have appeared since then (see [4–14] and references therein). In this respect, the present paper seeks to establish a perspective for the next years.

Recent investigations of irradiation-induced defects in Ge have mainly been based on the use of space-charge capacitance-transient spectroscopy methods such as deep level transient spectroscopy (DLTS), minority carrier transient spectroscopy (MCTS), and recently Laplace DLTS. Electron paramagnetic resonance (EPR), which has been extremely successful in studies of irradiation induced defects in silicon, for various reasons, had only limited success when applied to germanium. The transient capacitance spectroscopy methods, requiring the use of diodes, have been largely confined to *n*-type Ge due to difficulties in making good rectifying junctions in *p*-type material of resistivity ≤ 10 Ωcm.

Recently, a consistent picture of some of the dominant radiation-induced defects in room temperature irradiated germanium has appeared. This is primarily the case for the *E*-center (the phosphorus-vacancy pair, arsenic-vacancy pair, antimony-vacancy pair) and the *A*-center (the interstitial oxygen-vacancy pair), and to a lesser extent for the di-vacancy. In the following these recent investigations of irradiation-induced defects in germanium after room-temperature irradiations with light particles (electrons, protons, neutrons) at MeV energies as well as γ-rays will be reviewed.

2. Space-charge capacity transient spectroscopy

The data which will be presented in this review are based on different versions of space-charge capacitance transient spectroscopy. There are many excellent reviews and books covering these techniques, *e.g.*, the pioneering article by LANG [15], the books by BLOOD and ORTON [16] and SCHROEDER [17] and more recently the introduction and description of the Laplace transform deep-level transient spectroscopy by DOBACZEWSKI *et al.* [18]. Here, only a very brief account of the different methods encountered in the present review will be given, and the reader is referred to the above mentioned reviews and references therein for detailed information. The methods are all based on capacitance measurements of diodes, and the results from both Schottky diodes and n^+p- or p^+n-mesa diodes will be presented. A temperature-dependent capacitance transient is produced when deep energy levels, within the depletion layer of the diode, thermally emit charge carriers to the conduction or valence bands. The preceding filling of the deep energy levels with charge carriers takes place either optically in a reverse biased diode as in MCTS or by pulsing a reverse biased diode into forward bias as in DLTS. The processing of the capacitance transients to extract emission rates is central to all the different space-charge based spectroscopy methods, and is done in several different ways. In the classical DLTS and MCTS the approach is an analog signal processing in which the transient is mutiplied by a time dependent weighting function; the Laplace DLTS is based on a digital signal processing in which the transient is analyzed by an inverse Laplace transform. The resolution performance of the standard DLTS systems is such that transients with emission-rate ratios of about 12–15 can be distinguished whereas the Laplace DLTS resolution is significantly improved by reducing this ratio to a factor of ~2 [18]. The sensitivities of the different systems are similar and reach about 10^{-4}–10^{-5} times the doping level. Thus, for a typical doping level of 10^{15} cm^{-3} the detection limit is 10^{10}–10^{11} deep levels per cm^3 [16].

The temperature dependence of the hole emission rate $e_p(T)$ is given in [16]: $e_p(T) = \chi_p \sigma_p N_V(T) \langle v_p(T) \rangle \exp(-\Delta H_p / kT)$, where ΔH_p is the change in enthalpy as a result of the ionization, $N_V(T)$ is the effective density of valence band states, $\langle v_p(T) \rangle$ is the average of thermal velocities of holes, and $\sigma_p(T)$ is the cross-section for hole capture. The apparent capture cross-section is $\sigma_{pa} = \chi_p \sigma_p$, where χ_p is the entropy factor $\chi_p = \exp(\Delta S_p / k)$ and ΔS_p is the change in entropy. The ΔH_p and $\sigma_{pa} = \chi_p \sigma_p$ are the so-called "DLTS fingerprints"; they are extracted from an Arrhenius analysis of the emission rate as a function of temperature. ΔH_p is given relative to the valence band edge. If the cross-section for carrier capture is thermally activated, the energy barrier for carrier capture will be included in ΔH_p; for this reason ΔH_p is called the apparent enthalpy of ionization. This energy barrier for carrier capture is typically a few tenths of an eV. The change in Gibbs free energy is given by $\Delta G_p(T) = \Delta H_p - T\Delta S_p$ and is equal to $E_V + E_t$, where E_V is the valence band edge energy and E_t is the energy level of the trap. ΔS_p is often of the order of $2k$ with $T\Delta S_p$ then of the order of a few tenth of an eV at room temperature. Similar expressions apply for electron traps interacting with the conduction band. As ΔS_p and the energy barrier for carrier capture are not always available, "the level position" stated in the literature is often $E_V + E_t = \Delta H_p$ in the case of a hole trap and $E_C - E_t = \Delta H_n$ in the case of an electron trap. It appears from the above that this is justified only for very low temperatures and for traps with small or no energy barrier for carrier trapping.

The space-charge capacitance transient spectroscopy methods are not element specific. Hence in order to identify the different constituents of a defect either complementary data from an element specific technique must be obtained (*e.g.*, electron-paramagnetic resonance), or samples with different concentrations of potential constituents must be analyzed. Structural identifications of defects have been achieved in conjunction with uniaxial stress [18] .

3. Irradiation-induced defects

One of the very first unambiguous identifications of an energy level in Ge was done by NAGESH and FARMER [6]. Their DLTS spectra from *n*-type Ge Schottky diodes were rather simple after MeV γ-irradiation consisting of only two lines, but slightly more complicated after neutron irradiation, as demonstrated in Fig. 1. By comparison with EPR studies of Ge [19] they suggested to assign the line labelled

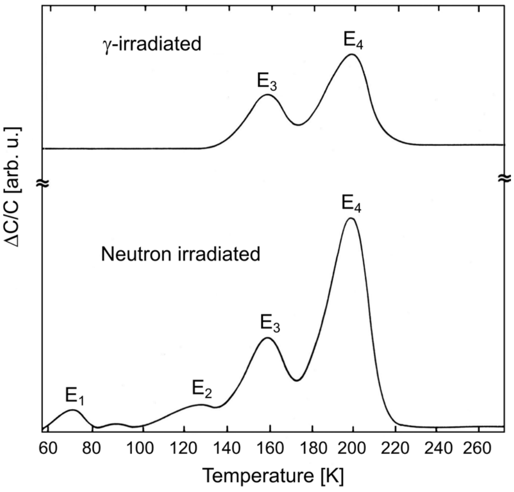

Fig. 1. DLTS Spectra of γ- and neutron irradiated n-type Ge Schottky diodes doped with phophorus. The neutron fluence was 2×10^{14} n (1 MeV equivalent/cm^2) and the γ-dose was 30 Mrad from a Co^{60} source. Reprinted with permission from V. Nagesh and J.W. Farmer, J. Appl. Phys. **63** (1988), 1549 [6]; copyright 1988, the American Institute of Physics.

E_3 to the oxygen-vacancy pair. The line labeled E_4 was tentatively identified as stemming from the phosphorus-vacancy pair, and a similar line was also observed in their Sb-doped samples. The two other lines which were only found in neutron irradiated samples were concluded to be of a more complicated nature. The line labeled E_2 was tentatively attributed to the di-vacancy, and the E_1 as a line from the planar tetravacancy. The level positions (ΔH_n – values), anneal temperatures and identifications from Nagesh and Farmer's investigations are given in the Table.

Table. Results from NAGESH and FARMER'S [6] investigation of γ- and neutron irradiated n-type Schottky diodes. The labels of the different lines refer to Fig. 1.

	E_1	E_2	E_3	E_4
ΔH_n [eV]	0.09	0.17	0.27	0.35
T_{ann} [°C]	–125	–100	–90	–125
Identification	Tetravacancy	Divacancy	A-centre (O_i-V)	E-centre (P-V)

Later on, a very comprehensive investigation of proton- and electron-irradiation induced defects in n-type Ge was presented by FAGE-PEDERSEN *et al.* [11]. These authors irradiated both Sb-doped samples of low carbon and oxygen concentrations, and the samples in which the n-type doping came from oxygen-related thermal donors and not from a group-V dopant. Typical DLTS spectra after electron and proton irradiations of Sb doped samples are shown in Fig. 2. A dominant line with an apparent enthalpy of ionization of $\Delta H_n = 0.37$ eV is observed. This line is absent in the sample in which the *n*-type doping stems from oxygen-related thermal donors. Instead, a dominant line of an apparent enthalpy of ionization of $\Delta H_n = 0.27$ eV is found in this sample as shown in Fig. 3. Annealing temperatures similar to those found by NAGESH and FARMER [6] were found by FAGE-PEDERSEN *et al.* [11], and their conclusion that the two dominant lines observed in their spectra are the same as the two observed by Nagesh and Farmer is well-founded. Fage-Pedersen *et al.* investigated the temperature dependence of the electron-capture process, and found that for both centers the capture cross-section are thermally activated with barriers of 0.023 eV for the *A*-center and 0.041 eV for the *E*-center. The strong line in Fig. 2 with the apparent enthalpy of ionization 0.23 eV was not identified but seemed to be both Sb and interstitial related [11]. The shoulder with an apparent enthalpy of 0.29 eV which is only observed in the proton irradiated samples was tentatively identified as related to the di-vacancy. However, the line at

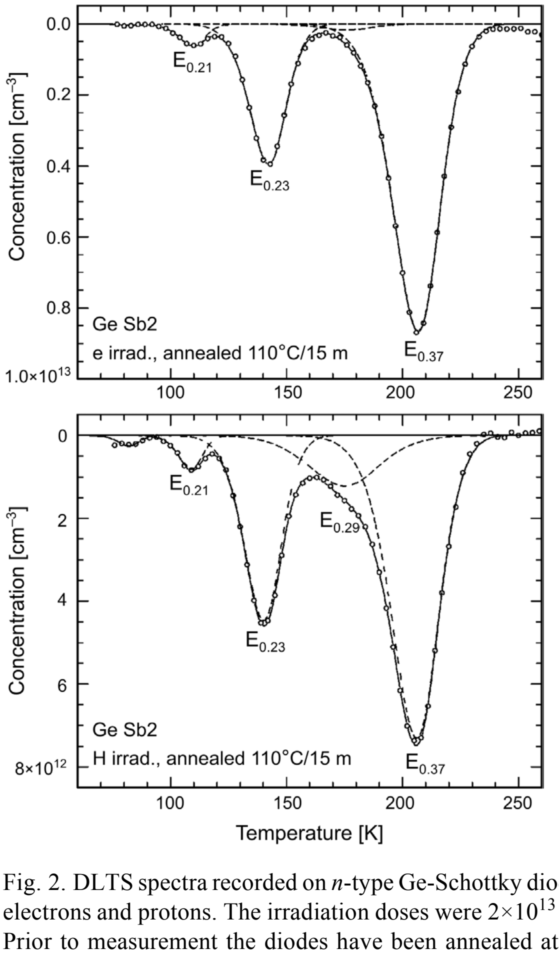

Fig. 2. DLTS spectra recorded on *n*-type Ge-Schottky diodes doped with antimony irradiated with 2-MeV electrons and protons. The irradiation doses were 2×10^{13} e^-/cm^2 and 1.7×10^{11} protons/cm^2, respectively. Prior to measurement the diodes have been annealed at 110°C for 15 min. Reprinted with permission from J. Fage-Pedersen, A. Nylandsted Larsen, A. Mesli, Phys. Rev. B **62** (2001), 10116 [11]; copyright 2001, the American Physical Society.

ΔH_n = 0.17 eV assigned by NAGESH and FARMER [6] to the di-vacancy was not observed by FAGE-PEDERSEN *et al.* [11].

In addition to the electron traps, FAGE-PEDERSEN *et al.* [11] also observed a strong hole trap with an apparent enthalpy of ionization of ΔH_p = 0.30 eV in the Sb-doped

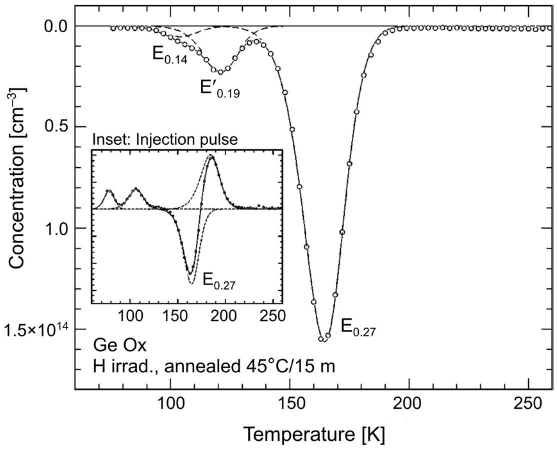

Fig. 3. DLTS spectra following 2-MeV proton irradiation of *n*-type Schottky diode doped with oxygen-related thermal donors. The irradiation dose was 6×10^{10} protons/cm^2 and the diode was annealed at 45°C for 15 min after irradiation. The inset shows a DLTS spectrum measured under injection pulse; there is no indication of the hole trap with ΔH_p = 0.30 eV which should appear at a temperature of ~135 K. Reprinted with permission from J. Fage-Pedersen, A. Nylandsted Larsen, A. Mesli, Phys. Rev. B **62** (2001), 10116 [11]; copyright 2001, the American Physical Society.

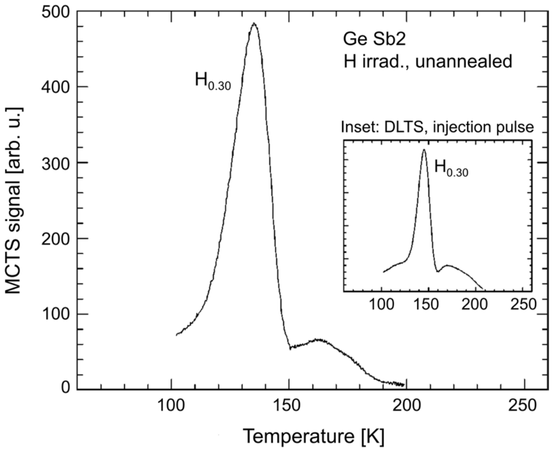

Fig. 4. DLTS and MCTS analysis following proton irradiation at 2-MeV of *n*-type Ge-Schottky diode doped with antimony. The irradiation dose was 5×10^{11} protons/cm^2. The inset shows a DLTS spectrum measured under injection. Reprinted with permission from J. Fage-Pedersen, A. Nylandsted Larsen, A. Mesli, Phys. Rev. B **62** (2001), 10116 [11]; copyright 2001, the American Physical Society.

samples but not in the sample doped with thermal donors (see Fig. 4 and the inset of Fig. 3). Similar to the *E*-center level discussed above, the hole trap is an acceptor level, and it seemed obvious to identify it as being another charge state of the *E*-center. Against this identification spoke, however, the annealing behavior which was complicated and different from that of the *E*-center. It was nevertheless suggested by FAGE-PEDERSEN *et al.* [11] that the two levels with $\Delta H_n = 0.37$ eV and $\Delta H_p = 0.30$ eV were two acceptor levels of the *E*-center, and, furthermore, that the 0.37 eV-level was a double-acceptor level while the 0.30 eV-level was a single-acceptor level. The double-acceptor character of the electron trap was based on its very small capture cross-section, indicative of a repulsive center, and on results from an investigation of the *E*-center in Si_xGe_{1-x} compounds [20] in which it was demonstrated that for $x \geq 0.25$ the single acceptor level changes from being an electron trap in the upper half of the band gap to being a hole trap in the lower half of the band gap. In the light of this, it was argued that it would be somewhat surprising to rediscover the single-acceptor level in Ge in the upper half of the band gap.

The problem of the different anneal temperatures was afterwards solved by MARKEVICH *et al.* [21] using γ-irradiated Sb-doped *n*-type Ge Schottky diodes. Taking advantage of the superior resolution of Laplace DLTS they were able to demonstrate that the $\Delta H_p = 0.30$ eV line of FAGE-PEDERSEN *et al.* [11] actually consists of two lines (see Fig. 5): a dominating one with $\Delta H_p = 0.307$ eV which anneals at the same temperature as the $\Delta H_n = 0.37$ eV line and a smaller line which is stable beyond 200°C. This double nature of the line explained the complicated annealing

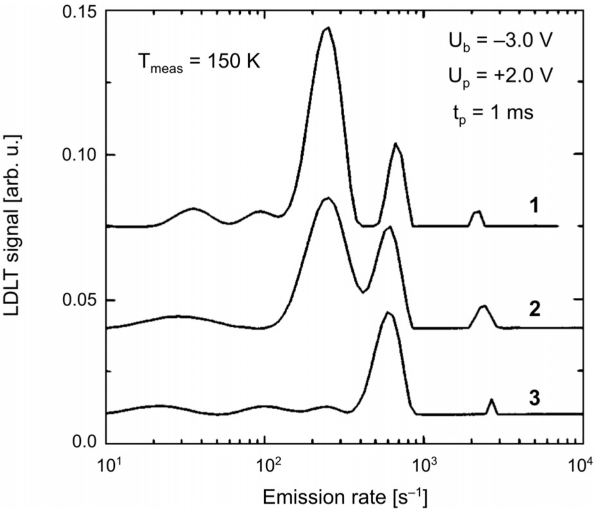

Fig. 5. Laplace DLTS investigations by MARKEVICH *et al.* [21] of a *n*-type Ge Schottky diode doped with antimony irradiated with γ-rays from a Co^{60} source (1) and after 30 min. annealing at 140°C (2) and at 200°C (3). The dominating line in the as-irradiated spectrum corresponds to a level at $\Delta H_p = 0.307$ eV. Reprinted with permission from V.P. Markevich, A.R. Peaker, V.V. Litvinov, V.V. Emtsev, L.I. Murin, J. Appl. Phys. **95** (2004), 4078 [21]; copyright 2004, the American Institute of Physics.

nature observed by FAGE-PEDERSEN *et al.* [11]. Thus, in addition to confirming the results of FAGE-PEDERSEN *et al.* [11], MARKEVICH *et al.* [21] also gave further support to the assignment of the $\Delta H_n = 0.37$ eV line and $\Delta H_p = 0.307$ eV line, to the E(=/–) and E(–/0) levels of the Sb-V complex, respectively. More recently, MARKEVICH *et al.* [14] have demonstrated that similar behaviours were found, in

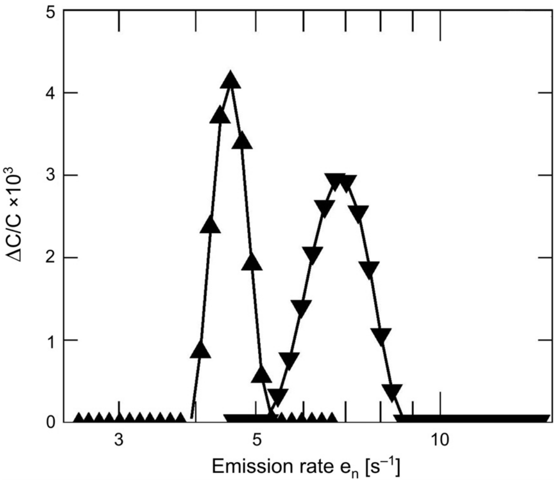

Fig. 6. Laplace investigations by LINDBERG *et al.* [23] of a p-type Ge n^+p-mesa diode doped with Ga after 2-MeV electron irradiation to a dose of 5×10^{14} e^-/cm^2. The $\Delta H_p = 0.095$ eV line (▲) is measured at 40 K and the $\Delta H_p = 0.309$ eV line (▼) is measured at 130 K. The total intensities of the lines result from summation of peak-data points, and the ratio of the total $\Delta C/C$ values of the two lines is to 1.11±0.15. Reprinted with permission from C.E. Lindberg, J. Lundsgaard Hansen, P. Bomholt, A. Mesli, K. Bonde Nielsen, A. Nylandsted Larsen, L. Dobaczewski, Appl. Phys. Lett. **87** (2005), 172103 [23]; copyright 2005, the American Institute of Physics.

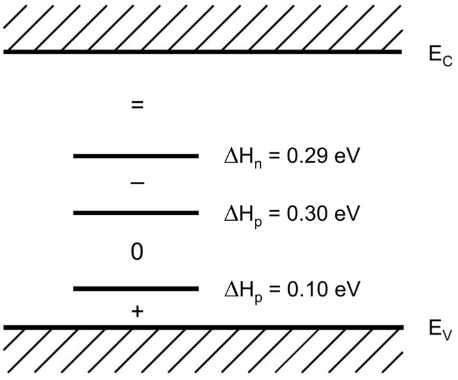

Fig. 7. Energy levels of the E center in Ge (the Sb-vacancy pair) as deduced in this review. No correction for change of entropy ΔS has been applied. The apparent enthalpies have been corrected for the capture cross-section energy barrier when available. The ΔH_n values are relative to the conduction band and the ΔH_n values relative to the valence band.

general, for the group V impurity-vacancy complex and, moreover, that the emission of an electron from the doubly negatively charged state of the centers is accompanied by large changes in the entropy ranging from $\Delta S_n = 1.95k$ for P-V to $4.2k$ for Sb-V.

While in silicon only one level of the *E*-center has been identified until now at $\Delta H_n = 0.43$ eV [22] in Ge two levels have been identified from studies of *n*-type Ge as demonstrated above. Very recently, Lindberg *et al.* [23] have succeeded in making good n^+p-mesa diodes from 2 Ωcm *p*-type Ge to study irradiation induced levels in the lower half of the band gap. Because of the way the n^+-top layer was produced (molecular-beam epitaxially grown layer doped with Sb) some Sb had diffused into the *p*-type layer rendering it practicable to study Sb related defects in the lower half of the band gap. This investigation not only confirmed the existence of the single-acceptor state with an enthalpy of ionization of $\Delta H_p = 0.309$ eV but also revealed a new Sb-related line with an apparent enthalpy of ionization of $\Delta H_p = 0.095$ eV. This new line had the same intensity as the line of the single-acceptor state (see Fig. 6) and annealed at the same temperature. Based on these observations it was concluded that this shallow level is the single donor-replica of the Sb-V complex. Thus, the Sb-V complex in Ge has four charge states as shown in Fig. 7.

The vacancy-oxygen complex for which Nagesh and Farmer [6] identified a level with an apparent enthalpy of ionization of $\Delta H_n = 0.27$ eV, and which was later on

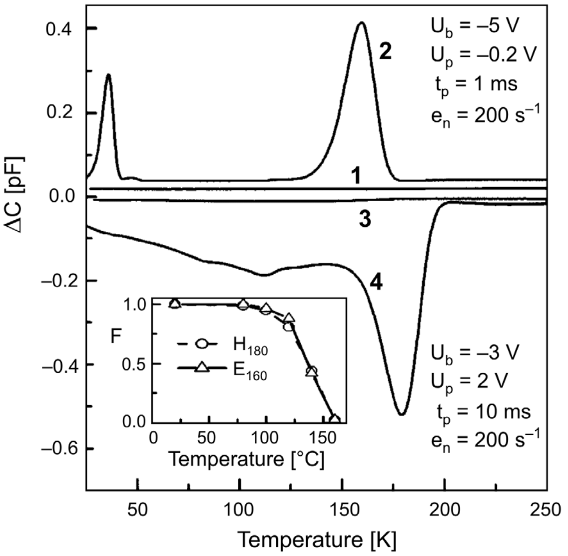

Fig. 8. DLTS investigations by Markevich *et al.* [13] of *n*-type Ge Schottky diodes doped with Sb and with a oxygen concentration of 5–10×10^{16} cm^{-3} before (spectra 1, 3) and after gamma irradiation using a Co^{60} source (spectra 2, 4). Line 2 corresponds to a level at $\Delta H_n = 0.27$ eV and line 4 to a level at $\Delta H_p = 0.325$ eV. The inset shows normalized changes in concentration of traps responsible for line 2 (called E_{180} by Markevich *et al.* [13]) and line 4 (called H_{180}). Reprinted with permission from V.P. Markevich, I.D. Hawkins, A.R. Peaker, V.V. Litvinov, L.I. Murin, L. Dobaczewski, J.L. Lindström, Appl. Phys. Lett. **81** (2002), 1821 [13]; copyright 2002, the American Institute of Physics.

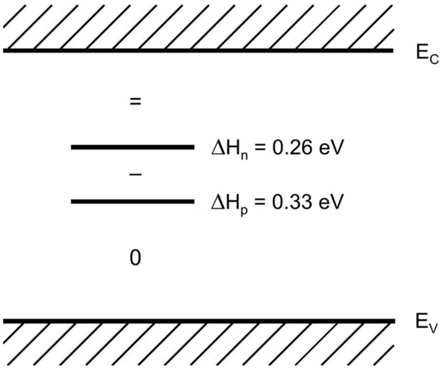

Fig. 9. Energy levels of the A center in Ge (the O-vacancy pair) as deduced in this review. No correction for change of entropy ΔS has been applied. The apparent enthalpies have been corrected for the capture cross-section energy barrier when available. The ΔH_n values are relative to the conduction band and the ΔH_p values relative to the valence band.

confirmed by FAGE-PEDERSEN *et al.* [11], has been further studied by MARKEVICH *et al.* [13, 24]. In addition to the level with $\Delta H_n = 0.27$ eV they also found a level in the lower half of the band gap with an apparent enthalpy of ionization of $\Delta H_p = 0.325$ eV (see Fig. 8). Both lines anneal at the same temperature. By comparison to the results from infrared-absorption measurements it was concluded that both these levels are related to the O-V complex, and that the $\Delta H_n = 0.27$ eV level is the double-acceptor level whereas the $\Delta H_p = 0.325$ eV level the single-acceptor level. For the double -acceptor level it has been established that the carrier capture is thermally activated with an energy barrier of 0.023 eV and an entropy of ionization of $\Delta S = 2.2k$. Thus, the *A*-center in Ge has three different charge states as illustrated in Fig. 9.

4. Summary

It is now established that the *E*-center (the group-V impurity-vacancy pair) has four charge states in Ge corresponding to three energy levels: a double-acceptor level in the upper half of the band gap, and a single acceptor and a single donor level in the lower half of the band gap. Surprisingly large values of the entropy of ionization are found for the double acceptor state (which is the only state of the *E*-center for which the entropy has been determined); the highest being found for the Sb-V pair and equals 4.2*k*. Thus, the emission from the double acceptor level is accompanied with fairly large relaxation. The *A*-center (the interstitial oxygen-vacancy pair) has three charge states in Ge: a double acceptor in the upper half of the band gap and a single acceptor state in the lower half. Both the *A*- and *E*-centers anneal below 150°C. The di-vacancy has not been unambiguously identified in Ge as different authors have reported tentative identifications of different levels in the band gap.

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