

## The vacancy–donor pair in unstrained silicon, germanium and SiGe alloys

A R Peaker<sup>1,6</sup>, V P Markevich<sup>1,2</sup>, F D Auret<sup>1,3</sup>, L Dobaczewski<sup>4</sup> and N Abrosimov<sup>5</sup>

<sup>1</sup> Centre for Electronic Materials Devices and Nanostructures, University of Manchester, Sackville Street Building, Manchester M60 1QD, UK

<sup>2</sup> Institute of Solid State and Semiconductor Physics, Minsk, Belarus

<sup>3</sup> Department of Physics, University of Pretoria, South Africa

<sup>4</sup> Institute of Physics, Polish Academy of Sciences, Warsaw, Poland

<sup>5</sup> Institute for Crystal Growth, Berlin, Germany

E-mail: [peaker@manchester.ac.uk](mailto:peaker@manchester.ac.uk)

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### Abstract

The thermal stability and electronic properties of the vacancy–donor complexes, often referred to as the E centres, have been studied in silicon, unstrained silicon–germanium and pure germanium. The E centres have been introduced by electron irradiation or gamma rays. In silicon, Laplace deep level transient spectroscopy has been used to separate the E centre emission from the di-vacancy, thus enabling very reliable data to be obtained for the vacancy complexes with P, As and Sb. In pure Ge only the E centres associated with P and Sb are reported and in Ge rich SiGe only V–P. In all the samples measured the thermal stability of V–Sb has been found to be significantly higher than V–P. With regard to the energy levels, the activation energy of electron emission from the single-acceptor level of the E centre in silicon are for V–Sb 0.40 eV and for V–P 0.46 eV. For the pure Ge case, the single acceptor is a hole trap with emission to the valence band having energies for V–P of 0.35 eV and V–Sb of 0.31 eV. Similar values are found for Ge rich SiGe. The double-acceptor state is not seen in silicon but in germanium produces a state with an activation energy for electron emission of 0.30 eV for V–P and 0.38 eV for V–Sb. This is also reflected in the Ge rich alloys of SiGe:P that have been measured in this work.

(Some figures in this article are in colour only in the electronic version)

<sup>6</sup> Author to whom any correspondence should be addressed.

## 1. Introduction

The vacancy–donor pair or E centre is the dominant defect produced in electron- or gamma-irradiated float zone silicon, oxygen lean silicon–germanium and germanium. It is formed by the capture of a mobile vacancy at the site of a substitutional group V atom. In Czochralski (Cz) silicon the interstitial oxygen competes for vacancies, producing the vacancy–oxygen complex or A centre with a consequent reduction in the production rate of the vacancy–donor pair. However, in n-type material the free vacancy is negatively charged and the positively charged donor provides a much more attractive capture site for the vacancy than neutral interstitial oxygen by almost an order of magnitude. However, the overall branching ratio also depends on the relative concentration of  $O_i$  and the group V ions, and as  $[O_i]$  is usually many orders greater in Cz material the A centre generation dominates. Irradiation produces equal numbers of interstitials and vacancies. Interstitial reactions result in the production of interstitial-carbon-related defects ( $C_iC_s$ ,  $C_iO_i$ ,  $C_iP_s$  etc), which can be present in comparable concentration to the E centre or the A centre. In ion implanted silicon the situation is more complicated. The higher production rate of intrinsic defects results in a greater opportunity for the vacancies to react with each other, producing the di-vacancy as the dominant defect.

In silicon, E centres have an acceptor level in the gap which is between 0.4 and 0.45 eV from the conduction band, depending on the dopant species. This is very close in energy to the single-acceptor state of the di-vacancy and it is not possible using conventional DLTS to separate these two defects, except by annealing. The E centres are far less stable than the di-vacancy and so can be annealed out, without affecting the di-vacancy population. The E centre in silicon has been very extensively studied (e.g. Watkins and Corbett 1964, Brotherton and Bradley 1982, Ganchenkova *et al* 2004) and most aspects of its behaviour are now understood, including the charge state dependence of the annealing rate (Kimerling *et al* 1975). However, the detailed mechanisms of its dissociation and migration behaviour on annealing are still not clear (Markevich *et al* 2001). This issue is particularly important in heavily doped n-type silicon where vacancies are widely believed to be the precursor for reactions which result in the reduction of the achievable carrier concentration (Nylandsted Larsen *et al* 1993, Ramamoorthy and Pantelides 1996, Ranki *et al* 2002). In such material the equilibrium population of vacancies is higher than in lightly doped silicon and so it is speculated that the E centre is present in un-irradiated material.

Some work has been published recently on the Sb related E centre in pure Ge (Fage-Pedersen *et al* 2000, Markevich *et al* 2004b). This adds to the early work by Fukuoka and Saito (1982) on Sb and As related E centres and by Nagesh and Farmer (1988) on P related centres. The properties of the E centre have also been investigated in unstrained silicon rich  $Si_{1-x}Ge_x:Sb$  ( $0 \leq x \leq 0.25$ ) by Kringhøj and Nylandsted Larsen (1995) and for  $x = 0.3$  by Goubet *et al* (1992), Goubet and Stievenard (1995) and in strained  $Si_{1-x}Ge_x:P$  ( $x = 0.04$  and  $0.07$ ) by Sihto *et al* (2003) and for  $0 \leq x \leq 0.13$  by Monakhov *et al* (2001).

In this paper we have examined the P, As and Sb related E centres in Si using high resolution (Laplace) DLTS. This enables us to separate the E centre signals from the di-vacancy very reliably. Further, we have investigated the electronic properties and annealing behaviour of the P and Sb related E centres in Ge and finally have studied the behaviour of P related E centres in unstrained germanium rich  $Si_{1-x}Ge_x:P$  ( $0.938 \leq x \leq 1$ ).

## 2. Experimental procedure

A range of starting materials has been used for this study, all of which are unstrained. For the pure silicon case the comparison of P, As and Sb E centres has been carried out on (100)

5 inch epitaxial Si wafers. The n-layer was doped with P and had a free carrier concentration of  $1.1 \times 10^{16} \text{ cm}^{-3}$  and was grown by chemical vapour deposition (CVD) on an  $n^{++}$  substrate. Part of the wafer was implanted with Sb at different energies in such a way as to produce a uniform Sb concentration of  $8 \times 10^{15} \text{ cm}^{-3}$  in the first  $1.2 \mu\text{m}$  below the surface, so producing a region doped with both P and Sb. A similar procedure was followed in relation to an implantation of As in another part of the wafer. After implantation the Si samples were annealed at  $950^\circ\text{C}$  for 30 min in nitrogen to activate the dopants and to remove implantation induced disorder in the Si. The samples were then irradiated with 3.5 MeV electrons at room temperature to doses of  $1.5 \times 10^{15} \text{ cm}^{-2}$  (original slice) and  $5.0 \times 10^{15} \text{ cm}^{-2}$  (implanted). The samples were cleaned and etched in a mixture of  $\text{HNO}_3\text{:HF:GAA}$  (glacial acetic acid) in the ratio of 40:1:15 for 30 s in order to remove any surface damage. Finally, the samples were dipped in HF to remove oxide from the surface. Immediately thereafter Au Schottky contacts, 1 mm in diameter, were deposited in vacuum by evaporation on the implanted sides of the samples and then Al was deposited on their  $n^{++}$  backsides as Ohmic contacts. Two FZ slices doped with P to concentrations of  $5 \times 10^{15} \text{ cm}^{-3}$  and  $10^{14} \text{ cm}^{-3}$  and were also irradiated, in this case with 6 MeV electrons. This material had been used by us in previous studies of the E centre in Si (Markevich *et al* 2001).

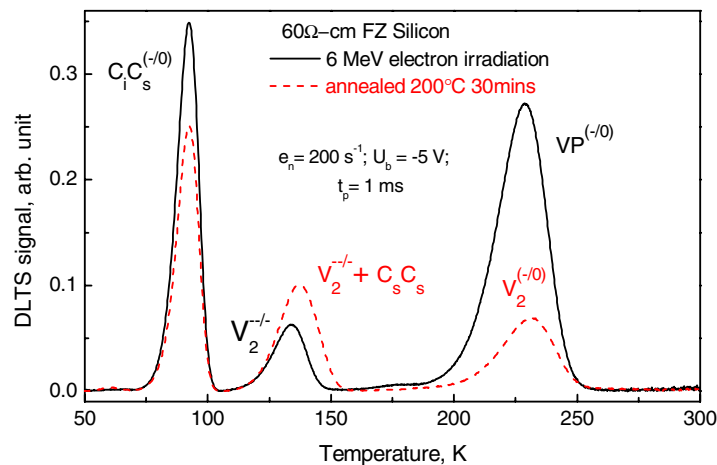
For the pure Ge case samples were prepared from n-type oxygen-lean Ge crystals doped with P or Sb. The absorption line at  $855 \text{ cm}^{-1}$  due to interstitial oxygen atoms has not been detected in the crystals, which indicates that the oxygen concentration is low ( $\leq 10^{15} \text{ cm}^{-3}$ ). Concentrations of shallow donors were in the range  $(1\text{--}2) \times 10^{14} \text{ cm}^{-3}$ . For the germanium rich SiGe, samples were prepared from oxygen lean Cz material (Abrosimov *et al* 1997). The samples were irradiated with  $\gamma$ -rays from a  $^{60}\text{Co}$  source. The irradiation temperature was about  $30^\circ\text{C}$ .

For all the samples Schottky diodes for capacitance measurements were fabricated by thermal evaporation of Au on surfaces etched in a  $1\text{HF} + 10\text{HNO}_3$  acid mixture. Current–voltage ( $I$ – $V$ ) and capacitance–voltage ( $C$ – $V$ ) measurements at different temperatures were carried out in order to check the quality of the Schottky barriers and to determine the concentration of non-compensated shallow donors. Deep electronic levels were characterized with conventional DLTS and Laplace DLTS techniques (Dobaczewski *et al* 2004). Hole traps in pure Ge and Ge rich SiGe were studied with the application of injection pulses (i.e., forward bias pulses). It has been shown in a recent study that the barrier height for the Au–Ge Schottky diodes is close to or exceeds the bandgap value (Markevich *et al* 2004b). Such a high barrier results in the appearance of an inversion layer with a high concentration of holes near the semiconductor surface. Application of forward bias to such Au–Ge diodes results in a flux of holes from the inversion layer to the semiconductor bulk, thus explaining the possibility of recharging of hole traps in the lower part of the bandgap of n-type Ge samples with Au Schottky barriers as observed previously by Fage-Pedersen *et al* (2000).

Isochronal annealing of the irradiated samples has been carried out in the temperature range  $80\text{--}300^\circ\text{C}$  with increments of  $20^\circ\text{C}$ .

### 3. The E centre in silicon

Figure 1 shows a conventional DLTS plot of electron irradiated FZ silicon. The DLTS peak at 220 K in the as-irradiated sample contains signals from the single-acceptor states of  $V_2$  and the VP pair. The  $200^\circ\text{C}$  anneal eliminates the E centre, leaving the signal from  $V_2^{-/0}$ . It has been shown (Markevich *et al* 2001) that the vacancy released from the dissociating VP centre can produce additional di-vacancies or react with  $C_iC_s$  to produce  $C_sC_s$  (which has similar electron emission characteristics to the double-acceptor state of the di-vacancy ( $V_2^{-/-}$ )) and



**Figure 1.** Conventional DLTS plot of electron emission from n-type electron irradiated float zone silicon immediately after irradiation and after a 200 °C 30 min anneal.

**Table 1.** Activation energies and pre-exponential factors of electron emission from the single-acceptor state of the E centre in electron irradiated silicon. The parameters were derived from experimentally determined temperature dependences of electron emission rates ( $e_n$ ) described by the equation  $e_n/T^2 = A_n \exp(-\Delta E_n/kT)$ . The emission rates were separated from the di-vacancy by Laplace DLTS. Calculated values of apparent capture cross section are also given.

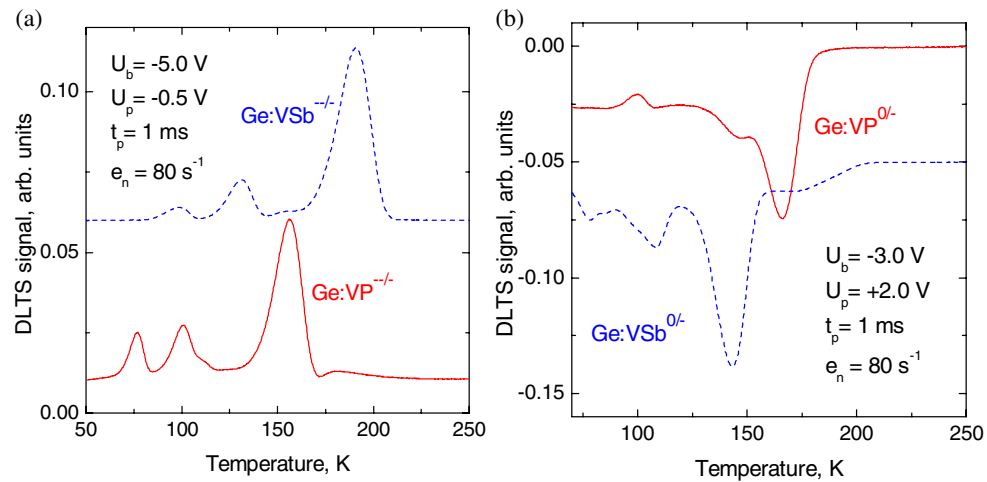
Identity	$\Delta E_n$ (eV)	$A_n$ ( $s^{-1} K^{-2}$ )	$\sigma_{na}$ ( $cm^2$ )
V-P (-/0)	0.458	$4.94 \times 10^7$	$7.6 \times 10^{-15}$
V-As (-/0)	0.435	$2.80 \times 10^7$	$4.3 \times 10^{-15}$
V-Sb (-/0)	0.401	$1.36 \times 10^7$	$2.1 \times 10^{-15}$

contributes to the magnitude of the peak at ~130 K in figure 1. It is this coincidence of emission rates which is probably responsible for the large spread of values in the literature of activation energies and significant differences in the temperatures at which the defects anneal out.

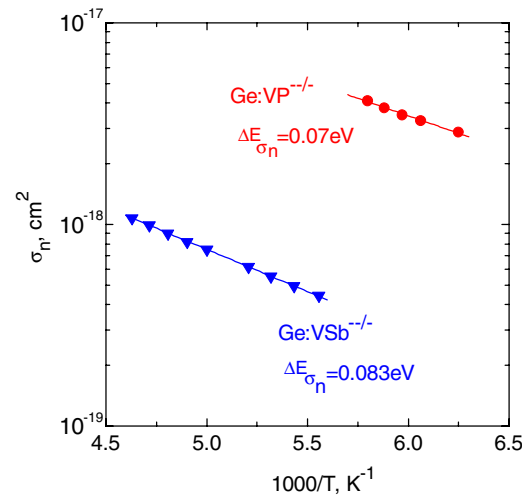
Laplace DLTS measurements enable us to separate the different species of E centre from the di-vacancy and so determine the electrical properties reliably. This is particularly difficult in relation to the phosphorus E centre. At 230 K the  $VP^{(-/0)}$  state has the same emission rate as  $V_2^{(-/0)}$ . However, at 210 K VP has a lower emission rate clearly separable in LDLTS whereas at 240 K the higher emission rate of VP can just be separated. Table 1 gives the properties of the defects studied in this way. In the P doped samples implanted with As and in those implanted with Sb two donor species are present in each slice. Using Laplace DLTS the electron emissions from the V-P and V-As(Sb) pairs are observed as clearly separated peaks and the activation parameters can be extracted unambiguously.

#### 4. The E centre in Ge:P and Ge:Sb

Figure 2(a) shows the conventional DLTS spectra of electron emission for germanium doped with phosphorus and with antimony after irradiation with  $^{60}Co$  gamma rays. Prior to irradiation there was no DLTS signal on the scale of figure 2, and no deep levels with concentrations higher than  $5 \times 10^{10} cm^{-3}$  have been detected in as-grown samples. For the antimony case,



**Figure 2.** Conventional DLTS plot of (a) electron emission and (b) hole emission from n-type Cz gamma-irradiated germanium doped with either P or Sb. In each case the dominant peak has been identified as a charge state of the E centre.



**Figure 3.** Temperature dependence of the directly measured electron capture cross sections of the double-acceptor state of the P and Sb E centres in gamma-irradiated germanium.

the dominant peak coincides precisely with a state that has been identified as the second acceptor state of the Sb–V complex (Markevich *et al* 2004b). This assignment is strongly supported by the electron capture cross-section measurements shown in figure 3. Here the absolute magnitudes of the cross-sections are indicative of capture into a repulsive state. In consequence, we are looking at electron capture by the negatively charged E centre, which then becomes doubly negatively charged.

It is important to note that the measured capture cross-sections are thermally activated, which must be taken into account when calculating the energy depth of the level from the conduction band. This issue and the determination of the entropy and the Gibbs free energy are dealt with in detail elsewhere (Markevich *et al* 2004a). Figure 2(b) shows the hole emission

**Table 2.** Activation energies and pre-exponential factors of hole emission from the single-acceptor state of the E centre and electron emission from the double-acceptor state in gamma-irradiated germanium. The parameters were derived from least-squares fits of experimentally determined temperature dependences of electron emission rates ( $e_n$ ) to those described by the equation  $e_n/T^2 = A_n \exp(-\Delta E_n/kT)$ . Calculated values of apparent capture cross section are also given.

Material	Hole emission acceptor state			Electron emission double acceptor		
	$\Delta E_p$ (eV)	$A_p$ ( $s^{-1} K^{-2}$ )	$\sigma_{pa}$ ( $cm^2$ )	$\Delta E_n$ (eV)	$A_n$ ( $s^{-1} K^{-2}$ )	$\sigma_{na}$ ( $cm^2$ )
Ge:P	0.348	$1.1 \times 10^8$	$9.2 \times 10^{-14}$	0.293	$9.6 \times 10^6$	$2.7 \times 10^{-15}$
Ge:Sb	0.307	$2.2 \times 10^8$	$1.8 \times 10^{-13}$	0.377	$2.1 \times 10^7$	$5.9 \times 10^{-15}$

**Table 3.** Annealing temperatures required to reduce the concentration of the E centre in Si and Ge to half its original concentration in a 30 min anneal in the negative charge state.

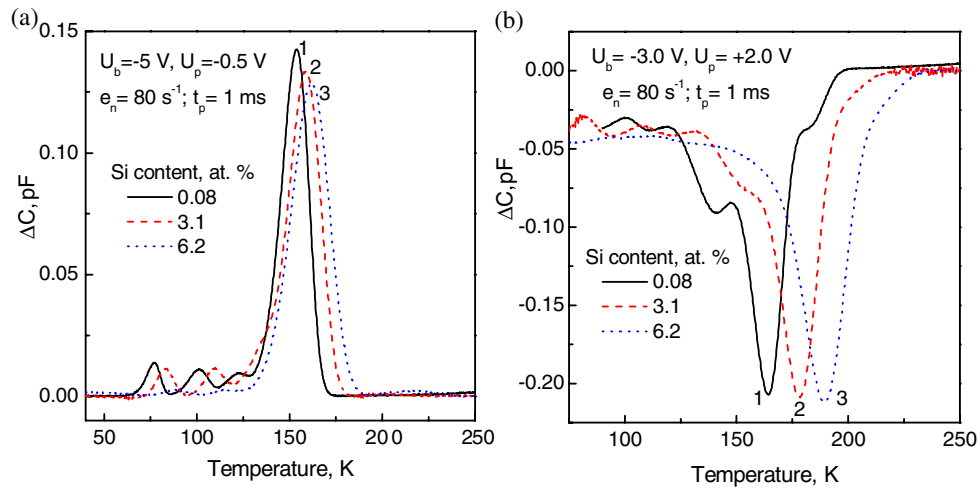
Identity	Covalent radius of dopant ( $\text{\AA}$ )	Anneal (Si) ( $^{\circ}C$ )	Anneal (Ge) ( $^{\circ}C$ )
V-P (—)	1.06	135	119
V-As (—)	1.2	180	125
V-Sb (—)	1.4	200	160

from the same samples achieved by using different excitation conditions to the electron case. As discussed previously, hole injection is possible because of the high Au on Ge barrier height. We see in each spectrum one dominant peak. This peak exhibits the same annealing behaviour as the dominant peak in figure 2(a) and hence we assign it to the single acceptor of the E centre. This assignment is supported by the hole capture process under these injection conditions. The hole concentration in the material will always be less than the electron concentration and so it would not be expected that the state would become significantly hole occupied unless it was negatively charged. This would mean that the state must be an acceptor. The activation energies and pre-exponential factors are presented in table 2.

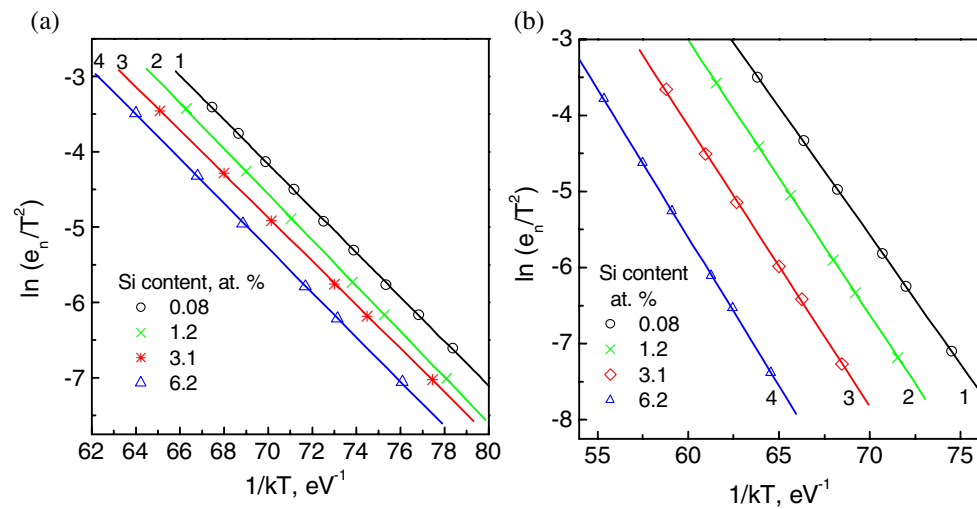
The samples have been subjected to 30 min isochronal annealing with no bias applied across the layer. The consequence of this is that the E centre is in the negative charge state during annealing. Table 3 presents the results of this study in terms of the temperature needed to reduce the concentration of the E centre to half its original value, in a 30 min anneal. For both the germanium and silicon results it is evident that there is a general trend to greater stability as the size of the dopant atom increases. This observation of annealing behaviour ranking with the covalent radius was first made by Brown *et al* (1959) in germanium and studied in the case of silicon by Hirata *et al* (1967).

## 5. The E centre in $\text{Si}_{1-x}\text{Ge}_x\text{:P}$ ( $0.938 \leq x \leq 1$ )

Figure 4(a) shows the conventional DLTS spectra from germanium rich silicon–germanium for three different silicon concentrations. Not surprisingly, there is great similarity to the spectrum of the E centre in germanium. Figure 4(b) shows the ‘injection’ DLTS spectra relating to hole emission from the same three samples. As in the case of germanium annealing and carrier capture, experiments confirm that these dominant peaks are the double- and single-acceptor states of the phosphorus E centre in silicon germanium. Figure 5 details the carrier emission data in the form of Arrhenius plots with the derived data for electron emission shown in table 4 and for hole emission in table 5. A representation of the activation energy as a function of composition is shown in figure 6. As mentioned for the case of germanium, a



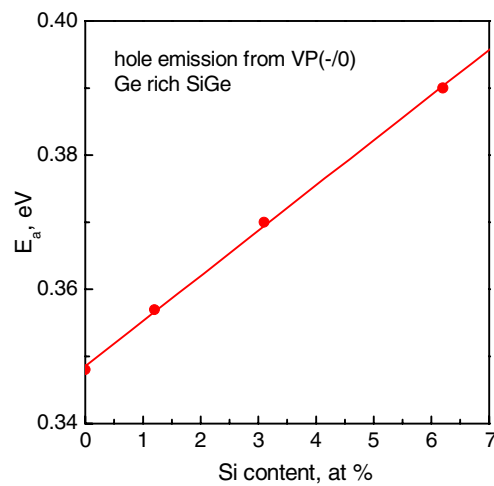
**Figure 4.** (a) DLTS spectra and (b) 'injection' DLTS spectra for gamma-irradiated P-doped  $\text{Ge}_{1-x}\text{Si}_x$  crystals with different Si content,  $x$  (at.%): (1)—0.08; (2)—3.1; (3)—6.2. The irradiation dose was  $2 \times 10^{17} \text{ cm}^{-2}$ .



**Figure 5.** Arrhenius plots of the  $T^2$ -corrected carrier emission rates for (a) the dominant electron trap and (b) the dominant hole trap, in gamma-irradiated P-doped  $\text{Ge}_{1-x}\text{Si}_x$  crystals with different Si content,  $x$  (at.%): (1)—0.08; (2)—1.2; (3)—3.1; (4)—6.2.

temperature dependence of the electron capture cross-section of the double-acceptor state has been observed, together with a rather high value of entropy for this state, making the Gibbs free energy (the true energy from the conduction band) considerably smaller than the apparent activation energy. This issue is under investigation at the present time.

The annealing behaviour is very similar to that of pure germanium. Figure 7 shows the development of (a) the electron emission and (b) the hole emission spectra for a  $\text{Si}_{0.03}\text{Ge}_{0.97}$  sample, after 30 min of isochronal anneals at 20° intervals from 60 to 140 °C. Laplace DLTS measurements have been conducted at all stages of this annealing study and no structure has



**Figure 6.** Changes in the activation energy of hole emission from the dominant hole trap in gamma-irradiated P-doped  $\text{Ge}_{1-x}\text{Si}_x$  crystals versus changes in Si content.

**Table 4.** Activation energies and pre-exponential factors of electron emission for the double-acceptor state of the E centre in gamma-irradiated P-doped  $\text{Ge}_{1-x}\text{Si}_x$  crystals. The parameters were derived from least-squares fits of experimentally determined temperature dependences of electron emission rates ( $e_n$ ) to those described by the equation  $e_n/T^2 = A_n \exp(-\Delta E_n/kT)$ . Calculated values of apparent capture cross-section are also given.

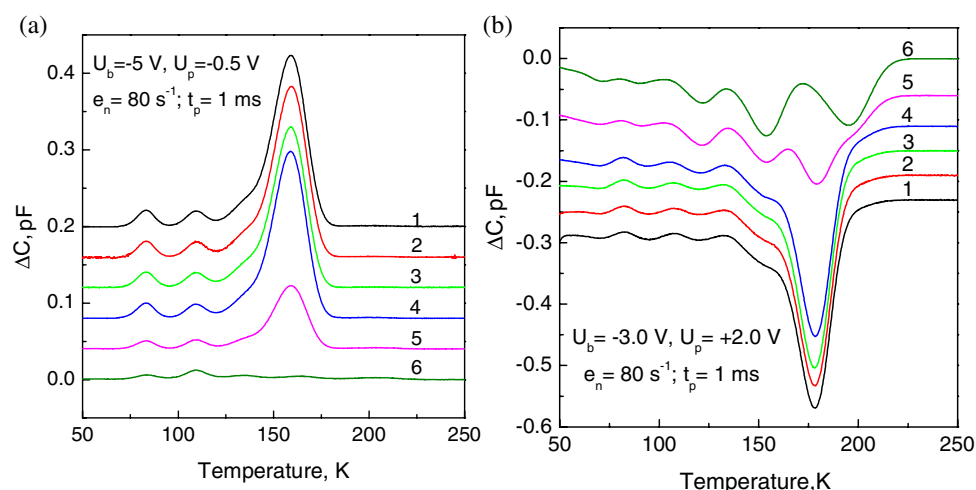
Ge content (at.%)	$\Delta E_n$ (eV)	$A_n$ ( $\text{s}^{-1} \text{K}^{-2}$ )	$\sigma_{na}$ ( $\text{cm}^2$ )
0.08	0.294	$1.35 \times 10^7$	$3.75 \times 10^{-15}$
1.2	0.303	$1.74 \times 10^7$	$4.8 \times 10^{-15}$
3.1	0.290	$4.82 \times 10^6$	$1.35 \times 10^{-15}$
6.2	0.296	$5.03 \times 10^6$	$1.4 \times 10^{-15}$

**Table 5.** Activation energies and pre-exponential factors of hole emission for the single-acceptor state of the E centre in gamma-irradiated P-doped  $\text{Ge}_{1-x}\text{Si}_x$  crystals. The parameters were derived from least-squares fits of experimentally determined temperature dependences of hole emission rates ( $e_p$ ) to those described by the equation  $e_p/T^2 = A_p \exp(-\Delta E_p/kT)$ . Calculated values of apparent capture cross-section are also given.

Ge content (at.%)	$\Delta E_p$ (eV)	$A_p$ ( $\text{s}^{-1} \text{K}^{-2}$ )	$\sigma_{pa}$ ( $\text{cm}^2$ )
0.08	0.338	$6.85 \times 10^7$	$5.7 \times 10^{-14}$
1.2	0.361	$1.255 \times 10^8$	$1.05 \times 10^{-13}$
3.1	0.370	$7.0 \times 10^7$	$5.8 \times 10^{-14}$
6.2	0.390	$5.47 \times 10^7$	$4.6 \times 10^{-14}$

been detected which can be attributed to a difference in the nearest neighbour sites as has been found previously for the case of Au and Pt in SiGe (Gościński *et al* 2001). However, the maximum Si content in the samples measured was 6% and so some degree of preferential siting of the E centre near silicon would be necessary for the structure to be evident in the Laplace measurement. It seems that this is not the case.





**Figure 7.** Development of (a) electron emission DLTS spectra and (b) hole emission 'injection' DLTS spectra for a gamma-irradiated P-doped  $\text{Ge}_{0.969}\text{Si}_{0.031}$  sample upon 30 min isochronal annealing with temperature increments of 20 °C. The spectra were measured after (1) irradiation and anneals at (2) 60 °C, (3) 80 °C, (4) 100 °C, (5) 120 °C and (6) 140 °C.

## 6. Discussion and conclusions

In the case of the silicon E centre we have used high resolution Laplace DLTS to obtain a clear separation of the electron emission from the E centre and the di-vacancy. In consequence, these measurements should provide very reliable data in comparison to the wide spread of values in the literature. For the pure germanium case, our results give strong support to the assignments of the single- and double-acceptor states of the E centre associated with phosphorus and antimony proposed previously (Fage-Pedersen *et al* 2000, Markevich *et al* 2004b). Key issues in this assignment are that it is the dominant centre in terms of concentration in the DLTS measurement and that the concentrations of the electron and hole traps have been found to be nearly identical after irradiation and at different stages of isochronal annealing. This strongly supports the view that the assignment of the traps to two different energy levels of the same defect must be the case.

Direct measurements of the capture cross-section of the electron trap are indicative of electron capture into a negatively charged state, and hence the assignment of this level as the double acceptor seems sound and the other charged state of the defect lower in the gap must then be the single acceptor. This is of course the correct order for a positive- $U$  centre. In consequence, it seems that in germanium the single acceptor observed in silicon is pushed towards the valance band and becomes a hole trap, while the double acceptor emerges from the conduction band as an electron trap.

In silicon–germanium only germanium rich alloys have been examined in this study with a maximum silicon content of 6%. There is a systematic change in the activation energy of the hole emission from the single acceptor, which without correction for the as yet unknown temperature dependence of the cross-section and for the entropy suggests that the level follows the conduction band edge.

The activation energy for electron emission of the double acceptor changes only slightly with composition, but a preliminary study not reported in detail here suggests that when the capture cross-section dependence and the entropy are taken into account the Gibbs free energy

is constant, indicating that this level is also pinned to the conduction band. Previous work on silicon rich silicon–germanium over the range 0–25% Si by Kringhøj and Nylandsted Larsen (1995), suggests that this pinning also occurs for the single-acceptor state in the silicon rich material.

Annealing studies confirm that the centre is more stable the larger the donor atom associated with the E-centre is, but that the E-centre in germanium anneals out at lower temperatures than in silicon. All anneals in Ge have been done so that the E centres have been in the singly negatively charged state and the results may well be quite different for the neutral and doubly negatively charged states as indicated in previous studies by Fage-Pedersen *et al* (2000) and by Kimerling *et al* (1975).

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