

# Characterization of energy levels related to impurities in epitaxial 4H-SiC ion implanted p<sup>+</sup>n junctions

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## Abstract

The distribution of energy levels within the bandgap of epitaxial 4H-SiC p<sup>+</sup>/n junctions was studied. The junction was obtained by Al ion implantation on a nitrogen doped n-type epitaxial substrate. Thermally Stimulated Currents/Capacitance (TSC/TSCAP) as well as Current/Capacitance Deep Level Transient Spectroscopy (I- and C-DLTS) were carried out over a wide temperature range (20-400 K). The two TSC/DLTS peaks associated with N-doping were detected for the first time and their trap signatures determined. Two hole traps relating to deep and shallow boron confirm that a boron contamination occurred during crystal growth. A negligible concentration of the  $Z_{1/2}$  level, which is usually the dominant level produced by irradiation of ion implant, was measured. The concentrations of all observed traps were significantly lower than nitrogen one, which determines the doping. This evidence supports the high quality of the processed junctions, making these devices particularly attractive for future use in particle detection as well as in optoelectronic applications.

Keywords: silicon carbide (SiC), implantation, defect characterization, detectors.

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71.55.Ht. Other nonmetals (impurity and defect levels)

81.05.Hd. Material science: other semiconductors

61.72.Ji. Point defects: vacancies, interstitials, color centers, etc. and defect clusters

## 1. Introduction

Single crystal Silicon Carbide (SiC) recently generated great interest because of its optical and electrical transport properties, in particular for high power [1], high temperature [2] high frequency applications [3] and radiation detection [4]. Schottky diodes fabricated on high quality epitaxial SiC substrates show promising performance [2,5], however, a technological process based on Schottky barriers is unsuitable for the production of complex radiation detectors featuring an on-board integrated electronics circuit. In the latter case, p/n junctions are needed together with MOSFET devices. Many research groups have analyzed ion implanted p<sup>+</sup>/n junctions [6-9] with low reverse leakage current and high breakdown voltages. To assess the production of junction-based SiC detectors, a detailed study on the crystalline quality of the epilayer and of the defect distribution in the device bulk is nevertheless mandatory. Traps and generation-recombination centers dominate the leakage current, effective doping concentration and charge collection properties of SiC diodes that are not yet unequivocally identified in spite of many studies dedicated to this investigation. Such defects may be native or induced by processes used in making the device. This paper aims to give a contribution to this field.

The characterization of ion implanted p<sup>+</sup>/n junctions has been carried out by Capacitance [10] and Current [11,12] Deep Level Transient Spectroscopy (C-DLTS and I-DLTS), Thermally Stimulated Current (TSC) and Thermally Stimulated Capacitance (TSCAP) [13,14]. The comparison to data in literature, when available, allowed us to hypothesize which defects were due to the technological steps applied for the bipolar diode construction.

## 2. Experimental Procedures

### 2.1. Samples

The samples studied in this work were ion implanted  $p^+/n$  4H-SiC diodes, produced in the frame of the collaboration between INSA-Cegely (Lyon, France) and CNR-IMM (Bologna, Italy). A  $\langle 0001 \rangle$   $8^\circ$  miss-cut oriented 4H-SiC  $n$ -type epitaxial wafer was used for this study. The  $p^+$  anodes were obtained by aluminum ion ( $Al^+$ ) implantation in the epilayer 40  $\mu m$  thick,  $n^-$  (nitrogen N) doped with density  $N_D = 1.1 \times 10^{15} cm^{-3}$ , as measured by C-V profiles.  $Al^+$  multiple energy implantation process with energies in the range 25–300 keV and fluence of  $0.9 - 8 \times 10^{14} cm^{-2}$  was used to produce an almost box shaped  $Al$   $4 \times 10^{19} cm^{-3}$  doping profile 0.45  $\mu m$  thick. The implantation temperature was 300  $^\circ C$ . The implanted wafer was annealed in an inductively heated J.I.P.ELEC<sup>TM</sup> furnace at 1650  $^\circ C$  for 30 min in high purity Ar gas at a partial pressure just above the atmospheric pressure. Al/Ti and Ni alloyed ohmic contacts were made on the  $p^+$  anode and the  $n^+$  base ( $\sim 10^{18} cm^{-3}$ ) respectively. The contact alloying process was done at 1000  $^\circ C$  for 2 min in vacuum [15]. Diodes of different areas were made. Circular diodes with 1mm diameter were used for the spectroscopy measurements presented in this paper. Two diodes with identical I-V curves were connected in parallel to increase the active area and, in turn, improve the signal to noise ratio during measurements. The reverse characteristics of each diode features a leakage current equal to  $\sim 10^{-12}$  A at 100 V and a breakdown voltage equal to 1.3 kV in air and 4 - 4.8 kV in Galden or SF6 [16].

### 2.2. Experimental Setup

In C-DLTS measurements, capacitance transients were converted into voltage by a SULA Tech. Deep Level Spectrometer, with 1MHz test signal frequency and 50mV amplitude. Then a digital oscilloscope (Tektronix TDS520D) performed the acquisition of the transients at selected sampling

times. In I-DLTS experiments the spectrometer is replaced by a pulse generator (Systron Donner 110D) providing reverse bias and pulsed injection, and by a custom readout circuit [17]. In TSCAP measurements, capacitance is measured directly by the SULA spectrometer. An HP4284A LCR meter measures the  $C(V)$  characteristics at room temperature. Thermally Stimulated Currents (TSC) experiments were carried out using an apparatus similar to the C-DLTS one, where the spectrometer is replaced by the Keithley 6517A electrometer, providing sample bias, low temperature forward injection and current reading.

Two possible schemes for sample cooling were used in our measurements. In the first, the sample sits inside a vacuum chamber where a pressure below  $10^{-3}$  mbar is maintained. The cryogenerator (Officine Galileo K1) cools the sample-holder, while a resistor wound around it heats the sample. This cooling system allows one to reach the minimum temperature of 20 K. A constant heating rate  $\beta$  - slow enough not to affect the shape of the spectrum ( $\beta \leq 0.07$  K/s) - was always adopted in current and capacitance DLTS experiments. A second cooling was used to achieve lower temperatures. It also ensure a better thermal coupling between sample and temperature sensor. The sample holder, mounted on a long steel hollow tube placed inside a liquid He dewar at different heights above the liquid surface achieves temperatures between 4.2K and room temperature.

All the measurements were carried out at 10V reverse bias and priming was performed using a forward bias limited to a 3mA forward current. Taking into account of the depletion layer thickness in the  $p^+n$  junction ( the built-in potential is 2.7V), the doping density of the epilayer, the 4H-SiC relative dielectric constant ( $\epsilon_r = 9.7$ ) and the specimen polarization during the measurements, the traps responsible of the spectra presented in this paper stayed in a 4H-SiC 4 $\mu$ m-thick layer.

### **3. Experimental Results and Discussion**

The capacitance DLTS spectrum measured in the range 30-220 K is shown in Fig. 1. Sampling times and duration of injection pulse were  $t_1 = 5$  ms,  $t_2 = 45$  ms and  $t_p = 10$ ms respectively. Several peaks were observed in the experimental C-DLTS spectrum: two negative peaks caused by electron traps labeled  $E_2$  and  $E_3$  and two positive peaks corresponding to hole traps labeled  $H_1$  and  $H_2$ . The bottom of Fig.1 shows the junction capacitance versus temperature  $C(T)$  measured without carrier injection. The corresponding vertical scale is plotted on the right axis. When decreasing  $T$  below 60K the junction capacitance sharply decreases and then reaches a plateau value corresponding to the geometric capacitance  $C_g \cong 8$  pF. From  $C(T)$  measurements we infer that the ionization of nitrogen occurs in the range 30-60 K, where the junction is formed. The  $E_2$  peak falls in this range too, which suggests a relationship of the  $E_2$  peak with nitrogen. It is known that nitrogen produces two energy levels close to 50 meV and 100 meV. These levels were inferred in past by different experimental evidences, as capacitance- and current-voltage measurements as a function of the temperature in the range 100-200K [18], photoluminescence [19] and Hall effect measurements [20-22]. The two nitrogen levels correspond to the substitutional hexagonal and cubic sites in the lattice respectively.

A variety of C-DLTS measurements were carried out using different sampling times: resulting Arrhenius plots of the peaks  $E_2$  and  $H_2$  are shown in Fig. 2. Here  $\ln(T_p^2/e)$  is plotted versus  $1/K_B T_p$ , where  $T_p$  is the peak temperature of the C-DLTS signal,  $K_B$  is the Boltzmann constant and  $e$  the emission coefficient of the trap at the temperature  $T_p$ . The resulting trap parameters of  $E_2$  are  $E \cong 0.10$  eV and  $\sigma \sim 10\text{-}200 \cdot 10^{-15}$  cm<sup>2</sup>. Even if these values are considered a first approximation (because the modification of junction capacitance during charge emission from traps is not negligible) they confirm that  $E_2$  corresponds to the nitrogen level close to 100 meV. This energy level can produce a significant C-DLTS signal because the junction is already formed during the ionization of the shallower nitrogen level, which occurs at lower temperatures. On the contrary, the

shallower nitrogen level cannot be observed because the junction forms after its ionization starts to occur.

From the Arrhenius plot, the signature of the H<sub>2</sub> peak was found to be :  $E = 0.28 \pm 0.01$  eV,  $\sigma \sim 0.2-1 \cdot 10^{-15}$  cm<sup>2</sup>. This emission is associated with shallow boron. Many studies targeted boron because this impurity may be a p-type dopant for SiC [23,25]. Boron occupies both the silicon and the carbon sites with a preference for the Si site for samples grown with a low Si/C ratio [25,26]. This produces a deep acceptor center at  $E_V+0.55$  eV and a shallow level at  $E_V+0.26-0.30$  eV. The deep boron peak usually appears at a temperature slightly lower than the Z<sub>1/2</sub> peak. Boron at a silicon site is usually associated with a shallow acceptor while boron at a carbon site is associated with a deep acceptor. Thus H<sub>2</sub> may be related to shallow boron, which could have contaminated the sample during growth and processing. This relationship is reinforced by the observation of the deep boron peak described below. Note, however, that the cross section value measured by us is smaller than the value reported in [23] as calculated from Minority Carriers Transient Spectroscopy (MCTS) technique ( $\sigma=2 \cdot 10^{-14}-3 \cdot 10^{-13}$  cm<sup>2</sup>). Such a large  $\sigma$  value is not consistent with our measurements. Fig. 1 (dashed lines) shows the fit corresponding to the E<sub>2</sub> and H<sub>2</sub> trap parameters superimposed on the measured spectrum.

Two minor components in the C-DLTS spectra are observed close to room temperature. The spectrum measured in the range 220-400 K is shown in Fig. 3. The spectral feature observed in this temperature range is composed of a hole emission, labeled H<sub>3</sub>, and an electron emission, E<sub>4</sub>. We did not attempt to draw the Arrhenius plots of levels E<sub>3</sub> and H<sub>1</sub>, or H<sub>3</sub> and E<sub>4</sub> because their signals are extremely weak and a reliable analysis based on Arrhenius plots cannot be carried out. We obtained a rough estimate of the E<sub>3</sub> signature by fitting the position and the linewidth of the C-DLTS peak. The result is:  $E=0.15 \pm 0.1$  eV,  $\sigma \sim 10^{-17}$  cm<sup>2</sup>. We will discuss H<sub>1</sub> later in conjunction with additional measurements. H<sub>3</sub> and E<sub>4</sub> were identified by comparing the fits produced by the signatures reported

in literature.  $E_4$  was fitted using the signature of the  $Z_{1/2}$  center. The  $Z_{1/2}$  DLTS peak near 300 K is often reported to be the dominant feature of SiC after ion implantation [27] or irradiation with different particles, such as electron and protons [28-30]. The corresponding deep level signature was  $E=0.63-0.67$  eV,  $\sigma=3-20 \cdot 10^{-15}$  cm<sup>2</sup>. The measured concentration of  $Z_{1/2}$  is usually in the range  $10^{14}-10^{15}$  cm<sup>-3</sup> depending on irradiation and annealing procedure. The  $Z_{1/2}$  center is sometimes observed in non-irradiated epilayers [23,31]. It was suggested to be the superposition of the two-electron emission processes ( $Z_1^{-/+}$  and  $Z_2^{-/+}$ ) from a negative-U system [32, 33]. Although the study of the  $Z_{1/2}$  center in 4H-SiC was reported quite extensively [34], the exact microscopic structure of this defect is still unknown. The current consensus is that the  $Z_{1/2}$  center is related to a vacancy-type defect.  $H_3$  relation to deep boron confirms that a boron contamination occurred during processing. In our samples the concentrations of  $H_3$  and  $E_3$  were of the order of  $10^{12}$  cm<sup>-3</sup>.

Further information, complementary to those obtained from C-DLTS, was gained from TSCAP spectra shown in the main plot of Fig. 4 (solid line). The spectrum is compared to the capacitance profile  $C(T)$  measured without any low temperature carrier injection. Below 20 K,  $C(T)$  is fixed to the geometrical value. In the range 20-50 K the nitrogen freeze out occurs, the junction settles and the capacitance reaches a value close to 30 pF. Above 50K, the  $C(T)$  characteristic exhibits a slow monotonic increase not related to charge emission from defect levels. The TSCAP spectrum, measured with low temperature carriers injection, matches  $C(T)$  above 150 K, while at lower temperatures these two signals exhibit some differences, which are related to the discharge of traps  $H_1$ ,  $H_2$  and  $E_3$ . The monotonic growth of  $C(T)$  is superimposed on the discharge of  $E_3$  producing an increase in capacitance. For this reason the contribution of  $E_3$  to the TSCAP spectrum cannot be used as evidence. On the contrary, the hole emissions due to  $H_1$  and  $H_2$  produces a sharp decrease in capacitance that is easily recognized in Fig. 4. The transition due to  $H_1$  is visible at 65 K.  $H_2$  is related to the capacitance decrease above 100 K seen where the curve measured after carriers



injection matches the background capacitance, because its position agrees with the signature obtained from DLTS. In the inset, the temperature range 50-150 K is enlarged and the capacitance transitions due to H<sub>1</sub> and H<sub>2</sub> discharge are indicated. An additional curve (dotted line) corresponding to a fractional annealing stage is shown in this plot. The measurement was obtained by stopping the spectrum measurement at 70 K, cooling down again and repeating a new measurement without performing any further injection. Thus, this measurement is influenced by the charge trapped by H<sub>2</sub> alone, which starts as filled and emits at 120 K, and not by the charge trapped by H<sub>1</sub>, which discharged during the first heating. This measurement allows one to isolate the contribution of H<sub>2</sub>. The trap concentrations can be estimated from TSCAP spectra using the formula:

$$N_t = N_d \frac{(C_i^2 - C_f^2)}{C_i^2} , \quad (2)$$

where  $N_t$  is the trap concentration, and  $C_i$  and  $C_f$  are the capacitance values measured before and after trap discharge. If the background capacitance  $C(T)$  varies during deep level emission, this fact must be accounted for when evaluating  $C_i$  and  $C_f$ . In our situation,  $C_i$  is the maximum value of capacitance before hole emission while  $C_f$  is the capacitance measured at the same temperature when the trap is not filled. The difference,  $C_i - C_f$ , is indicated by vertical bars in the inset. We find  $N_t \approx 2 \cdot 10^{14} \text{ cm}^{-3}$  for H<sub>1</sub> and  $N_t \approx 5 \cdot 10^{13} \text{ cm}^{-3}$  for H<sub>2</sub>. The latter estimate agrees well with C-DLTS results.

The shallower nitrogen level cannot be observed by measuring the junction capacitance as pointed out earlier. To gain further information we carried out current measurements (i.e. TSC and I-DLTS). Fig. 5 shows the I-DLTS signal measured in the temperature range 20-300 K. We used

$t_1=1\text{ms}$  and  $t_2=9\text{ms}$  for the sampling times, and  $t_p=10\text{ ms}$  for the injection time. Note that the position of the peaks in I-DLTS and C-DLTS spectra do not match. They differ because the sampling times are different and the signals are described by slightly different equations. N ionization distorts the feature below 50 K. In this case, the N signal exhibits a double peak structure due to the emissions from shallow ( $E_1$ ) and deep ( $E_2$ ) nitrogen levels. The peak labeled as  $H_2$  at 180 K relates to shallow boron. Two other peaks are observed near 80 K and 160 K, which are assigned to levels  $H_1$  and  $E_3$  respectively. The position and the shape of the  $E_3$  peak are consistent with the signature previously estimated.

The result of the TSC measurements in the range 20-80 K is shown in Fig. 6. At temperatures higher than 80 K the background current swamps the weak signal coming from deep levels. Since the heating rate used in these experiments is the same adopted in TSCAP, the peak of the TSC spectrum and the inflection point in the TSCAP spectrum occur at the same temperature. The feature around 30-40 K is due to nitrogen. In this case, the N signal also exhibits a double peak structure. The peak due to  $H_1$  seen near 60 K satisfies both TSC and I-DLTS measurements [35]:  $E=0.11\text{ eV}$ ,  $\sigma=0.1\cdot 10^{-16}\text{ cm}^2$ . Since the activation energy of aluminum in 4H SiC is 0.19 eV [20,21], neither  $H_1$  nor  $H_2$  relate to Al, which is the p-type doping impurity in our sample. All these results are summarized in Table 1.

#### 4. Conclusions

TSC, TSCAP DLTS current and capacitance measurements were carried out on 4H SiC  $p^+/n$  diodes obtained by Aluminum ion implantation on a nitrogen doped n-type epitaxial substrate. The measurements were done over a wide temperature range, 20-400 K, to investigate the material quality of these devices.

Several charge emissions below room temperature related to dopants and impurities were observed. The deeper nitrogen level was observed in all spectra near 0.1 eV. Moreover, the shape of the TSC and I-DLTS signals exhibits a double peak structure because the shallower nitrogen level close to 50 meV was superimposed on the deeper level in current spectra. To our knowledge, this is the first time the TSC/DLTS peaks related to these two energy levels were observed. We estimated the capture cross section of deep nitrogen to be  $(1-20) \cdot 10^{-14} \text{ cm}^2$ .

A clear signal was observed in all the spectra at higher temperatures corresponding to the level labeled here as H<sub>2</sub>. From C-DLTS and TSCAP we inferred this was a hole trap with a concentration  $N_t = 0.4-1 \cdot 10^{14} \text{ cm}^{-3}$ . The signature of this level is  $E=0.28 \text{ V}$ ,  $\sigma \sim 1 \cdot 10^{-15} \text{ cm}^2$ . This signal is related to the shallower level of boron. The hole trap H<sub>3</sub> detected by C-DLTS close to room temperature is related to deep boron, thus confirming a boron contamination occurred during crystal growth.

A hole trap (H<sub>1</sub>) with concentration  $N_t = 2 \cdot 10^{14} \text{ cm}^{-3}$  was also observed. The signature was found to be  $E=0.11 \pm 0.1 \text{ eV}$ ,  $\sigma = 0.1-1 \cdot 10^{-16} \text{ cm}^2$ . To our knowledge, there is no evidence of such a hole trap in literature.

The emission from the Z<sub>1/2</sub> level, the dominant level produced by irradiation of ion implant, is extremely weak denoting a concentration of  $\sim 10^{12} \text{ cm}^{-3}$ . This confirms the quality of the implant process and of the additional annealing procedures making these devices particularly attractive for future use in particle detection and optoelectronic applications.

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## Figures and Tables

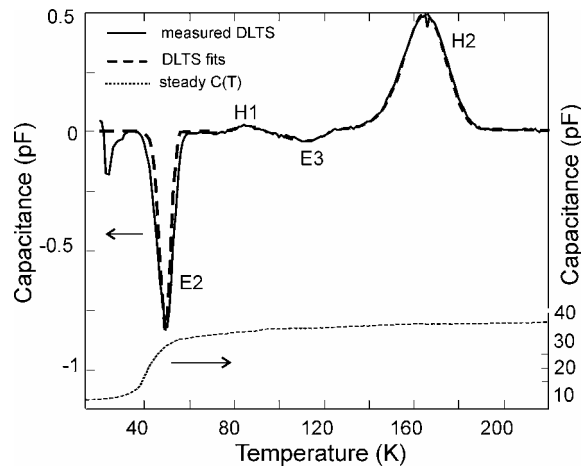


Fig. 1. DLTS Capacitance spectrum measured in the range 20-220 K (solid line). The trap signature fit (dashed lines) is superimposed to the measured values (solid line). Sampling times and duration of pulse injection are  $t_1=5$  ms,  $t_2=45$  ms and  $t_p=10$  ms respectively. The steady state capacitance  $C(T)$  is shown in the bottom of the figure (dotted line). The vertical scale corresponding to the latter curve is shown on the right axis. All the measurements were made with  $V_{rev}=10$  V.

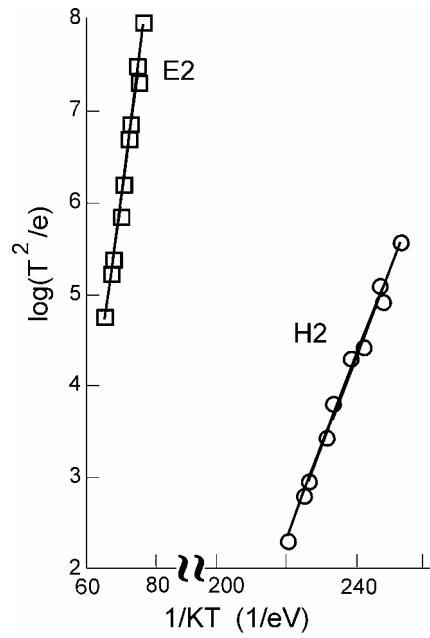


Fig. 2. Arrhenius plots obtained from C-DLTS spectra measured with different sampling times. The squared markers refer to the E<sub>2</sub> peak and the circles to the H<sub>2</sub> peak.

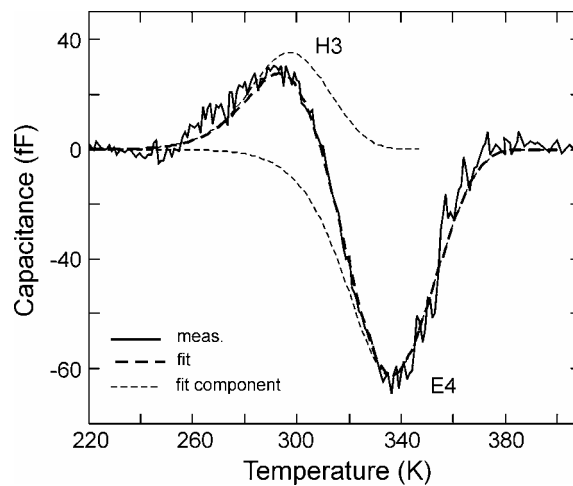


Fig. 3. DLTS Capacitance spectrum measured around room temperature (solid line) using  $t_1=1$  ms,  $t_2=9$  ms,  $V_{rev}=10$  V and  $t_p=10$  ms. of the two components H<sub>3</sub> and E<sub>4</sub> (thin dashed lines) accounts for the spectrum, The sum of the components is plotted with a smooth and thick dashed line.



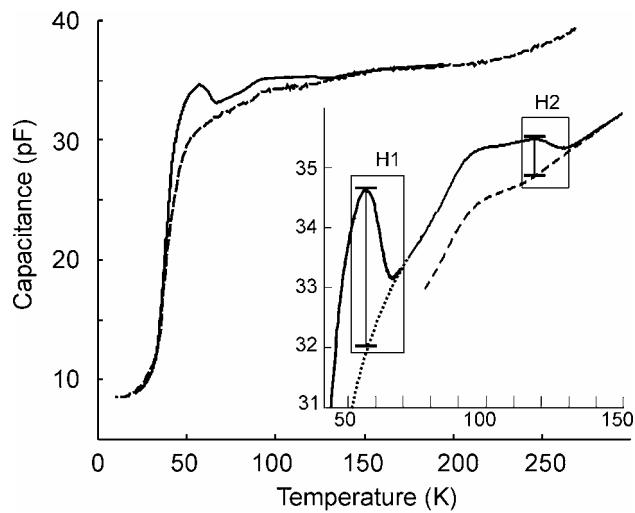


Fig. 4. A TSCAP spectrum (solid line) is compared to the  $C(T)$  profile measured without performing any injection (dashed line). An enlargement of the region between 50 K and 150 K is shown in the inset. The emissions from two hole traps  $H_1$  and  $H_2$  are highlighted. An additional trace (dotted line) shows the measurement obtained by stopping the spectrum measurement at 70 K, cooling again and performing a new measurement without injection ( $V_{\text{rev}}=10$  V,  $\beta=0.15$  K/s). Injection: 1 minute of forward bias ( $i=10$  mA). Vertical bars indicate the amplitude of capacitance transition ( $C_f - C_i$ ) due to each trap.

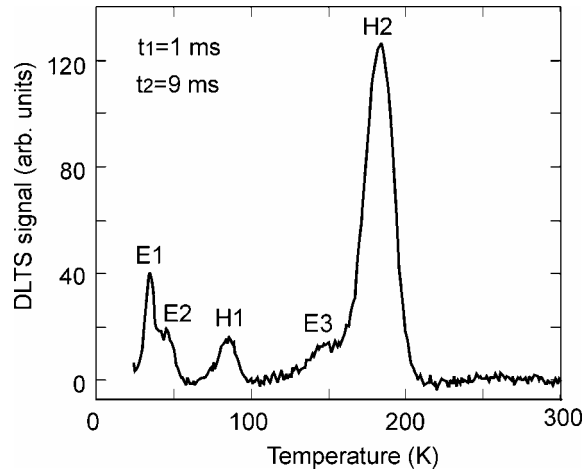


Fig. 5. I-DLTS spectrum measured in the range 20-300 K using the sampling times  $t_1=1$  ms,  $t_2=9$  ms ( $V_{\text{rev}}=10$  V,  $t_p=10$  ms).

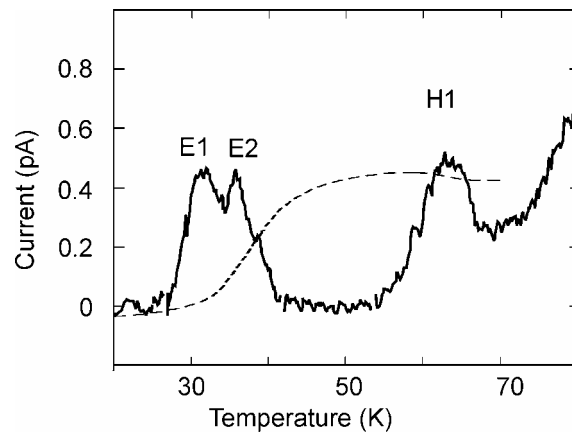


Fig. 6. TSC spectrum measured in the range 20-80 K with  $V_{\text{rev}}=10$  V and  $\beta=0.15$  K/s (solid line). The TSCAP spectrum (dashed line and arbitrary units) measured with the same heating rate is shown in the plot in order to compare the relative positions of spectral features.

Level	Defect	$E$ (eV)	$\sigma$ ( $\cdot 10^{-15}$ $\text{cm}^{-2}$ )	$N_t$ ( $\cdot 10^{15} \text{cm}^{-3}$ )	Notes
E <sub>1</sub>	Shallower N	$\sim 0.05^a$	-	1.1	Observed only by current techniques.
E <sub>2</sub>	Deeper N	$0.1 \pm 0.01$	10-200		( $E, \sigma$ ) deduced from Arrhenius plot. Overall $N_t$ from $C(V)$ at 300 K.
E <sub>3</sub>	-	$0.15 \pm 0.01$	$\sim 0.01$	$< 0.1$	
E <sub>4</sub>	Z <sub>1/2</sub>	$0.63 - 0.67^a$	$3 - 20^a$	$< 0.01$	
H <sub>1</sub>	-	$0.11 \pm 0.01$	0.01-0.1	0.2	Data from I-DLTS, TSC and TSCAP correlation. Not observed by C-DLTS.
H <sub>2</sub>	Shallower B	$0.28 \pm 0.01$	0.2-1	0.04-0.1	( $E, \sigma$ ) from Arrhenius plot.
H <sub>3</sub>	deeper B	$0.58 - 0.63^a$	$10 - 100^a$	$< 0.01$	

Table 1. Summary of the observed energy levels together with the results from data analysis. The superscript (a) indicates data taken from literature - these cases were checked for agreement with our measurements.