Analysis of TSC spectra measured on silicon pad detectors after exposure to fast neutrons♣

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Abstract

We present thermally stimulated current (TSC) spectra measured on asymmetric p-n-junctions fabricated from detector grade silicon. A multitude of characteristic deep levels generated by fast neutron induced damage with fluences ranging from 10^{12} cm^{-2} to 10^{14} cm^{-2} were observed. The TSC spectra were found to depend strongly on both the filling conditions and the electric field strength in the device. The filling of the deep levels has been investigated in detail by varying the current, temperature, and duration of the free carrier injection pulse. The corresponding observations in conjunction with a delayed heating analysis allow a tentative identification of the complex defects VO_i, C_iO_i, C_iC_s, and the divacancy VV.

1 Introduction

It is now largely accepted that the radiation induced degradation, i.e. the doping changes, the leakage current increase, and the deterioration of the charge collection, decides on a successful operation of silicon detectors in future HEP experiments like LHC [1]. The groups engaged in the R&D for silicon particle detectors with improved radiation hardness have now started to systematically investigate the microscopic features of the radiation damage, relying on the yet unproved possibility of defect engineering. In this field the TSC technique has attracted much interest since it is easy to set up and remains applicable even for highest fluences [2-4]. In the low fluence range, where the deep level concentrations stay well below the concentration of shallow dopants, a good agreement between TSC data and the well established C-DLTS method was found [5]. For higher fluences, however, ambiguous results are obtained depending on the filling temperature and the length of the free carrier filling pulse [4] or the ratio between deep center and shallow dopant concentrations [6], both complicating the derivation of deep level parameters from TSC spectra, especially the concentrations. Therefore, the aim of this paper is on the one hand to systematically investigate the dependence of the TSC spectra on the filling conditions and the applied bias voltages and on the other hand to evaluate the deep level parameters and tentatively assign the chemical nature by inspecting the appropriate literature.

2 Experimental procedure

The samples used for this study are p^+-n-n^+ silicon pad detectors fabricated by the Max-Planck Institut (MPI) München from initial 3 kΩcm phosphorus doped silicon corresponding to a doping concentration of 1.4×10^{12} cm^{-3}. The square shaped active area has a size of 0.5×0.5 cm^2 and the device thickness is 280 µm resulting in a voltage for full depletion, V_{dep}, of around 80 V before irradiation. The samples were exposed to fast neutrons of an average energy around 5.3 MeV according to the ^{9}Be(d,n)^{10}B reaction (E_d = 13.26 MeV) at the Physikalisch-Technische Bundesanstalt (PTB) Braunschweig, Germany. All fluences Φ given in this paper are normalized to equivalent 1-MeV-neutron values using a hardness factor of 1.45, for details see [7]. For the TSC measurements the devices are mounted on a copper block (hot stage) that is thermally connected to the second stage cold station of a closed cycle helium cryostat via sapphire rods. A lowest temperature of around 15 K and a constant heating rate of up to 1 K/s is available. The current signals are measured with a

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Keithley 487 picoammeter and the total system noise is of the order of 20 fA. The voltage source is included in the same instrument and allows for bias voltages up to 500 V. Typically the devices are cooled down under a reverse bias not higher than 100 V. At a low temperature we then apply a sufficiently large forward bias (-100 V) for 30 s and limit the current by a series resistor (57 KΩhm). After an initial decaying period of 60 s the temperature is raised at a constant rate of 0.183 K/s and the TSC data is recorded.

Fig. 1 shows a typical TSC spectrum as measured for the device irradiated by the largest fluence of 8.96×10^{13} cm^{-2}. Note, that the steady state bulk generation current dominates the signal above 180 K. The very high resolution of this specific measurement allows an unambiguous assignment of peak labels which are introduced here in order to facilitate the discussion in the following paragraphs.

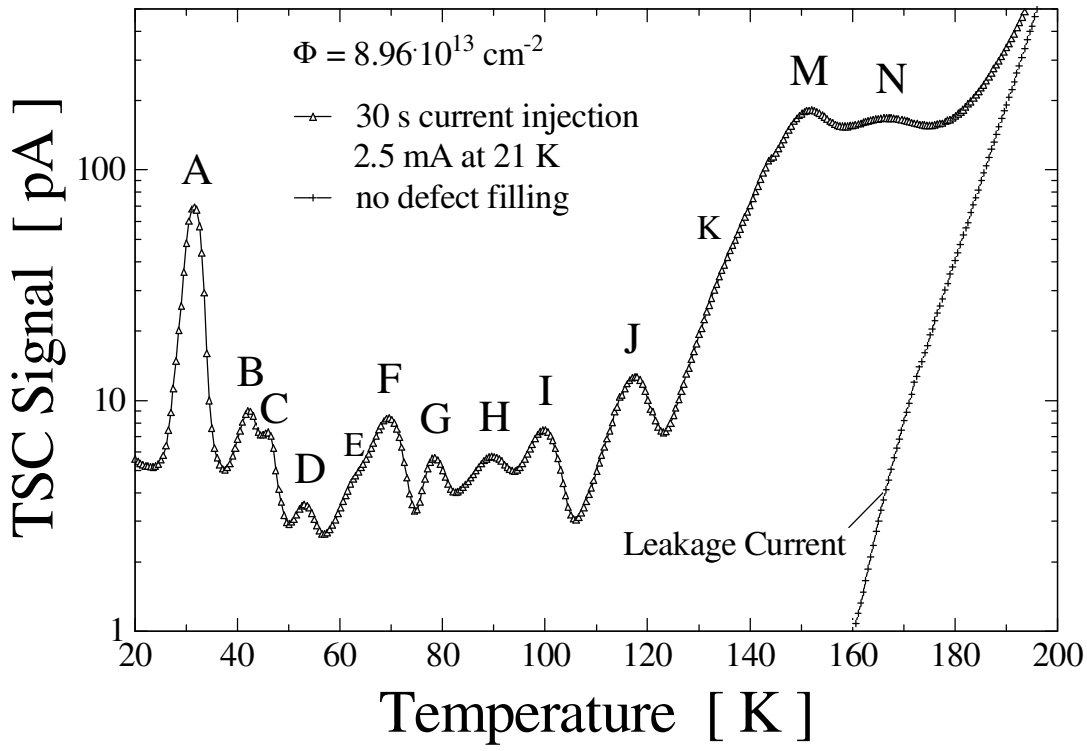

Fig. 1. Typical TSC spectrum for a detector irradiated by a fluence of 8.96×10^{13} cm^{-2} (device M20710). The filling of the deep levels was achieved by injecting a forward current of I_{fill} = 2.5 mA at T_{fill} = 21 K. A heating rate of β = 0.183 K/s and a reverse bias of 80 V was used. The depletion voltage at 294 K was $V_{dep} \approx$ 250 V after 8 months of room temperature annealing. The various emission peaks have been labeled with a capital letter.

3 Observed defect levels

Radiation induced defect levels have often been characterized with high accuracy in the literature, however the measurements were mainly performed on low resistivity silicon after electron irradiation. A particularly well suited method is the measurement of capacitance transients (C-DLTS) which has been used in [8] to verify that the same level parameters also apply to the defects in high resistivity silicon after neutron irradiation. Generally the thermal emission rate is determined which can be written

$$e_{n,p}(T) = B\left(\frac{m^*_{n,p}}{m_0}\right)\sigma_{n,p}T^2\exp\left(-\frac{\Delta E_{n,p}}{k_B T}\right), \quad (1)$$

where $m^*_{n,p}/m_0$, $\sigma_{n,p}$, and $\Delta E_{n,p}$ mean the effective masses (n : 1.08, p : 0.549), the capture cross section, and the ionization energy measured with respect to the conduction and valence band for electrons (n) and holes (p), respectively. T is the absolute temperature, k_B is the Boltzmann constant

and B is a numerical constant equal to 3.256×10^{21} $K^{-2}cm^2s^{-1}$ that absorbs parts of the effective density of states $N_{C,V}$ and of the thermal velocities $v_{th,n,p}$. Since most levels that are anticipated in the TSC spectra are well known, it is convenient to calculate the maximum temperature T_m of the corresponding peaks in order to assign the chemical nature:

$$T_m = \frac{\Delta E_{n,p}}{k_B \ln\left(\frac{B\sigma_{n,p}k_B T_m^4 m^*_{n,p}/m_0}{\beta(\Delta E_{n,p} + 2k_B T_m)}\right)}. \quad (2)$$

Given a heating rate β, eq. (2), which has been derived by Buehler [9], can be solved iteratively. The result of this procedure is presented in Table 1 for β = 0.183 K/s where most level parameters were taken from reference [8].

Table 1
Tentative assignment of the chemical nature by calculating the peak maximum temperature according to eq. (2) for a heating rate of 0.183 K/s. If no extra reference is given, the level parameters are taken from [8].

peak label	B [10]	C	F	H [11]	I	J	M	N
T_m	<47 K	49 K	70 K	92 K	97 K	115 K	147 K	159 K, 170 K
assignment	$C_iC_s(A)^{(+/0)}$	$C_iC_s(B)^{(-/0)}$	$VO_i^{(-/0)}$+$C_iC_s(A)^{(-/0)}$	$VV^{(+/0)}$	$VV^{(--/-)}$	$C_i^{(+/0)}$?	$C_iO_i^{(+/0)}$	?+$VV^{(-/0)}$

The assignment of peak J to the C_i donor level is however speculative since the interstitial carbon anneals out rapidly at 300 K and should not be present in the measurements shown here which were always performed after a considerable period of room temperature storage.

It is noted here that the delayed-heating method introduced by Muller et al. [12] can be used to obtain the ionization energy and capture cross section from TSC measurements. Results were found in [13] for the peaks F (ΔE_n = 0.172 eV, σ_n = 1.35×10^{-14} cm^2) and M (ΔE_p = 0.363 eV, σ_p = 2.45×10^{-15} cm^2) which are in good agreement with the C-DLTS data.

The origin of all other peaks in Fig. 1 is not known in the literature. On the one hand, this may be due to the different filling process compared to DLTS which may bring to daylight yet unknown metastable defect levels. On the other hand, the absolute sensitivity of current based spectroscopy methods is very high [14]. Thus these levels may have been overlooked in the C-DLTS measurements up to now.

4 Results from different modes of deep level filling

In order to understand the effect of defect filling, it is necessary to write down the fraction of a trap occupied with electrons in the space charge region in the steady state condition which is established in the TSC measurement at sufficiently high temperatures

$$n_t/N_t = \frac{1}{1+e_n/e_p}. \quad (3)$$

Here e_n and e_p are the rates for the emission of electrons and holes from the trap level under consideration, given by eq. (1), and N_t is the trap concentration. We have assumed that the concentration of free carriers is negligible. Since the emission rates exhibit an exponential dependence on the position of the energy level within the forbidden gap, it is clear from eq. (3), that levels in the upper half of the gap (electron traps) are empty and the ones in the lower half (hole traps) are occupied by electrons, i.e. empty of holes. It is only when the occupancy of a defect can be altered from the value given by eq. (3) by a suitable mode of filling that a corresponding peak in the TSC spectrum will arise. In the following sections we will discuss experimental results obtained with different modes of defect filling, always bearing in mind that we are dealing with high resistivity

silicon where the concentration of deep levels after irradiation can largely exceed the shallow doping level.

4.1 Cooling down with zero bias

Filling the defects by cooling the sample down in thermal equilibrium (0 V bias, sufficiently slow cool down rate), where the occupation of a trap is controlled by the Fermi level, is widely used in junction space charge spectroscopy in order to completely fill majority carrier traps. However, due to the high resistivity of our samples the Fermi level is very close to the mid of the gap at room temperature. Consequently, if small concentrations of defect levels are present after irradiation, the Fermi level will move towards the conduction band in n-type material upon cooling and finally pins at an electron trap [15]. For fluences $\Phi > 1\times10^{12}$ cm^{-2} this will be the level N, in our case, whose concentration then exceeds the phosphorus doping level. Thus, only peak N is observed in the corresponding TSC measurements. For very heavily irradiated devices ($\Phi > 1\times10^{13}$ cm^{-2}), virtually no levels are filled, which substantiates that the Fermi level stays in the middle of the band gap. This means that there are deep levels very close to the middle of the band gap with large concentrations.

4.2 Forward current injection

Consider one single trap whose emission rate at the filling temperature is small, say, $e_{n,p} << 1$/s. Then the steady state electron occupation ratio is given by

$$n_t / N_t = \frac{1}{1 + \frac{\sigma_p v_{th,p}}{\sigma_n v_{th,n}} \frac{p}{n}}, \tag{4}$$

when free electrons and holes with concentrations n and p are injected. Thus, according to the aforementioned, an electron/hole trap is filled only in the case that $\sigma_p v_{th,p} p$ is smaller/larger than $\sigma_n v_{th,n} n$ at the filling temperature. It is clear that this requirement may not be fulfilled in all cases, i.e. the trap is not detected.

Due to the high resistivity of the samples used for this study, the concentration of the free carriers can easily reach the doping level, which is known as the high-level injection case [16]. Furthermore, injection of holes from the p$^+$ contact as well as injection of electrons from the n$^+$ contact may occur, which has been described in terms of a double-injection p-i-n diode in the literature [16,17]. Both mechanisms imply that $p/n = 1$, a condition being clearly desirable for the experimenter since eq. (4) is greatly simplified. If however it is not possible to inject a sufficient amount of free carriers, it is necessary to take into consideration the charges trapped in the defects which then have to be absorbed in the difference of the concentrations of free electrons and holes in order to maintain the approximate space charge neutrality [18]. This may easily happen unless the filling current is very large. Therefore, one major shortcoming of this filling mode is that the value of p/n is not easily controlled. In addition, this ratio may vary with distance and consequently the filling of defects becomes inhomogeneous. It is noted here that this problem may best be overcome by generating the free carriers with a laser [4].

4.2.1 Filling temperature

We have observed a very strong influence of the filling temperature on the TSC spectra. Unfortunately the behavior is not unique for different fluences and annealing times. Three possible sources have to be discussed here: i) the initial filling of defects, ii) metastable defects, and iii) variation of the active volume as function of the temperature during the measurement. In order to exclude the last point, which will be discussed in Section 5, we shall focus on a weakly irradiated device, where the applied bias always suffices to activate the whole detector volume. Fig. 2 shows the corresponding result for filling temperatures, T_{fill}, ranging from 35 K to 85 K. It should be noted here that the sample is warmed up to 290 K after each measurement in order to guaranty the same initial conditions.

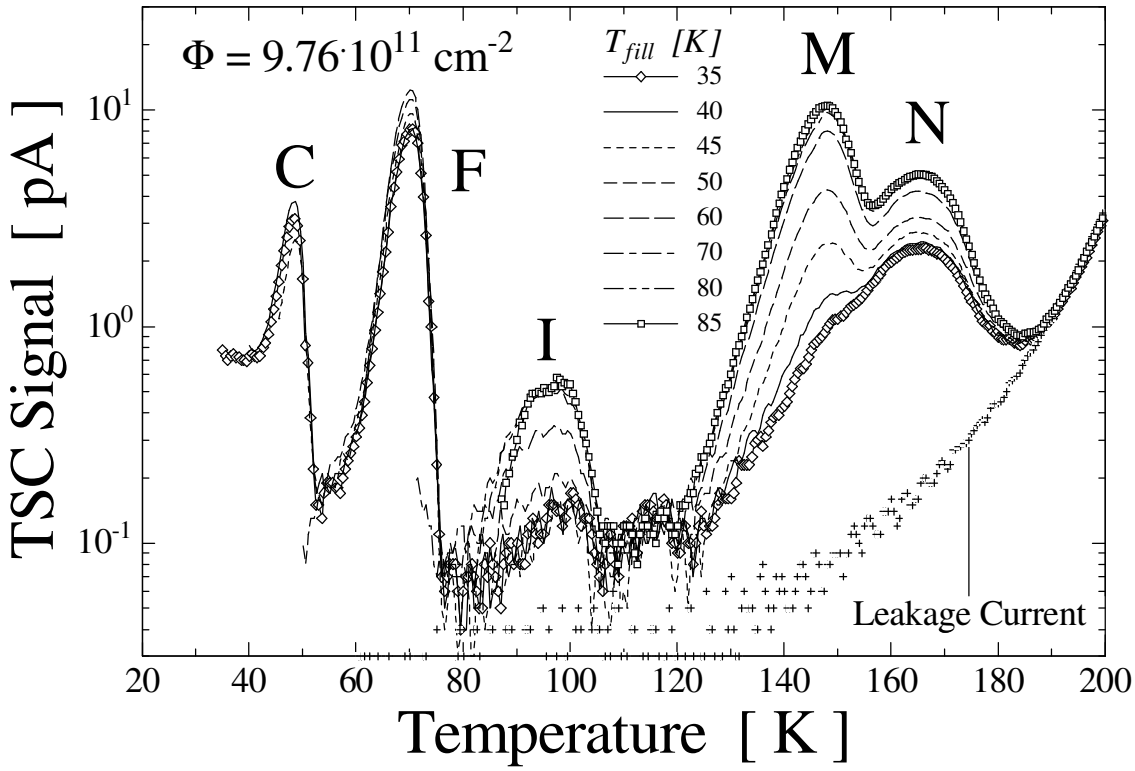

Fig. 2 Variation of the TSC spectrum as function of the filling temperature, T_{fill}. I_{fill} = 1.8 mA, t_{fill} = 30 s, β = 0.183 K/s, bias = 75 V. V_{dep} = 72 V at 294 K after 6.5 months of room temperature annealing (device M20609).

According to eq. (4) the initial filling depends on the ratio *p*/*n* which in principle can be derived from the forward current voltage characteristics obtained at low temperatures (Fig. 3). Only at the highest temperature is the IV curve in agreement with the familiar diffusion theory [16]. The negative differential resistance measured at 173 K is probably due to the dependence of the recombination lifetime on the injection level as outlined in [18]. For temperatures T < 25 K there is a very large voltage drop across the diode, which however also was observed in unirradiated devices. Also we found that the filling via forward biasing is very bad for almost all defects in this temperature range. The reason could be that both contacts become blocking and therefore a space charge region is established, which makes the density of free carriers very small. In addition it may happen that the current does not flow through the bulk of the detector.

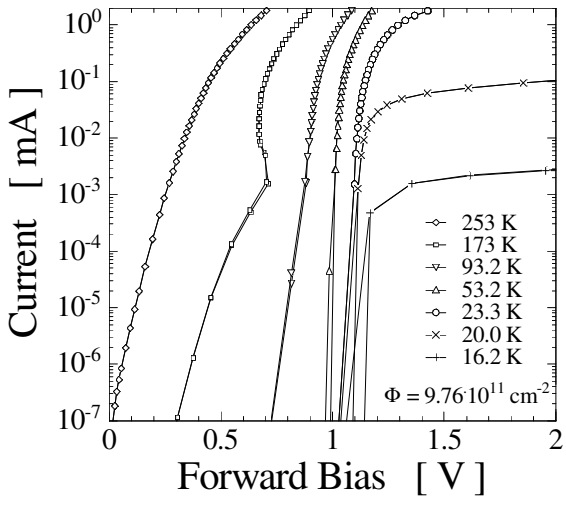

Fig. 3 Forward I-V characteristics at low temperatures (device M20609).

For temperatures higher than 25 K and currents larger than 10^{-2} mA there seems to be a more or less general behavior where the effects of the series resistance and high-level injection dominate. According to Section 4.2 we therefore assume that *p*/*n* has a value close to unity for all TSC measurements presented in Fig. 2.

Thus, by inspection of eq. (4), it is evident that the marked variation of peak M in Fig. 2 reflects a strong temperature dependence of the ratio between the cross sections for hole and electron capture, σ_p/σ_n. Such thermally activated carrier capture processes have been described in detail by Pässler [19] and probably apply to the C_iO_i donor level. The same arguments may be employed for the peak N.

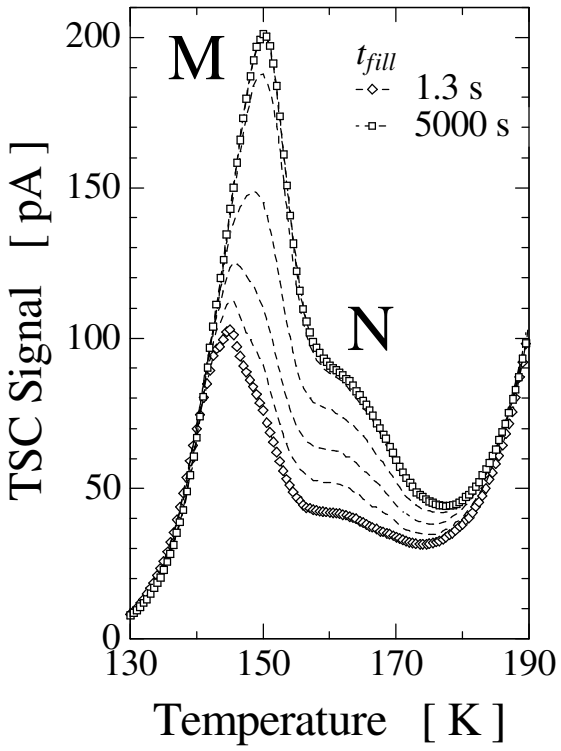

Fig. 4 Influence of the duration of the injection pulse, t_{fill}, on the TSC spectrum. I_{fill} = 1.7 mA, T_{fill} = 50 K, β = 0.183 K/s, bias = 50 V, V_{dep} = 65 V at 294 K after 7.5 months of room temperature annealing (device M20708, $\Phi = 3.82\times10^{13}$ cm^{-2}).

As described in [20], the increase of the height of peak F towards higher filling temperatures can be attributed to the metastability of the C_iC_s pair. This peak is composed of the well known VO_i complex and the above mentioned carbon pair in its configurational state (A). It has been shown in [10] that cooling of the sample with a reverse bias applied will generally freeze the C_iC_s in its state (B). Consequently, a signal according to an electron trap around 0.11 eV below the conduction band, which is our peak C, but no contribution of the C_iC_s defect to peak F is awaited. If, however, at a sufficiently high temperature electrons are injected into the diode, the carbon pair is transformed from its state (B) to the state (A) and an electron trap at 0.17 eV occurs, thus giving rise to a larger TSC signal around 70 K (peak F, compare Table 1).

4.2.2 Filling time

During injection of free carriers the occupation of a trap changes according to the relation $1-\exp(-t/\tau_{fill})$ until the steady state value given by eq. (3) is reached. Thus, if the duration of the injection pulse, t_{fill}, is of the order of the trap filling time constant, τ_{fill}, which is given by

$$\tau_{fill}^{-1} = nv_{th,n}\sigma_n + pv_{th,p}\sigma_p \qquad (5)$$

a dependence of the corresponding peak height on t_{fill} is expected. This behavior was observed only for the peaks M and N and only in heavily damaged detectors. Fig. 4 gives a magnified view of the according temperature range. The TSC spectra presented were measured after injecting a current of 1.7 mA at 50 K. The maximum of peak M shifts, which is probably not an indication of a yet unknown peak, but rather due to the improved filling of the C_iO_i defect, since, according to Section 5, the TSC peak shape is not well defined in heavily damaged detectors. Obviously it takes times of up to several 1000 s in order to completely fill these peaks. On the other hand, peak F in the same spectra does not depend on t_{fill}, which means that the corresponding filling time constant is smaller than 1 s. Therefore the large filling time constants of the peaks M und N, given by eq. (5), can only be explained by very small capture cross sections of the corresponding defects for both types of free carriers, electrons and holes. Moreover we found that the spectrum does not change when the product of filling time and filling current, $t_{fill}\times I_{fill}$, is kept constant [13]. Because the filling of a defect depends only on the ratio t_{fill}/τ_{fill} we conclude from this that the concentration of free carriers in eq. (5) is proportional to the injected current, as would be awaited for a p-i-n diode [16].

We noticed that the voltage which drops across the detectors when a large forward current is injected is increasing monotonically with the irradiation fluence. Consequently the free carrier densities decrease because the current flow is due to a drift process in the corresponding electric field. Thus, the filling time constant given by eq. (5) is an increasing function of the irradiation fluence, which explains why the dependence of the TSC spectrum on the filling time was not observed for weakly damaged detectors.

4.3 Switching to zero bias

Due to the fact that in high resistivity n-type silicon detectors radiation damage can change the sign of the space charge from positive to negative, one is able to distinguish between electron and hole traps,

as will be shown in the following. For all spectra plotted in Fig. 5 the corresponding sample has been cooled down to the filling temperature under reverse bias. The solid lines refer to TSC signals after injection of electrons and holes at the lowest temperature (forward bias), whereas for the other curves a bias of 0 V has been applied for 30 s prior to the measurement. In the latter case the space charge is frozen during the cooling down period and consequently free carriers of the opposite sign will flow into the device when the bias is removed resulting in the predominant filling of either electron or hole traps. For the low fluence device, the space charge is positive due to the ionized phosphorus atoms. Therefore the peaks A, C, F, and N are considered to be electron traps. For the inverted detector (high fluence), on the other hand, we find the peaks G+H, J, K, and M filled. Thus, the corresponding deep levels should be hole traps. From the comparison of all spectra it can furthermore be concluded that peak I is best assigned to an electron trap. These findings are also in agreement with the assignments previously presented in Table 1.

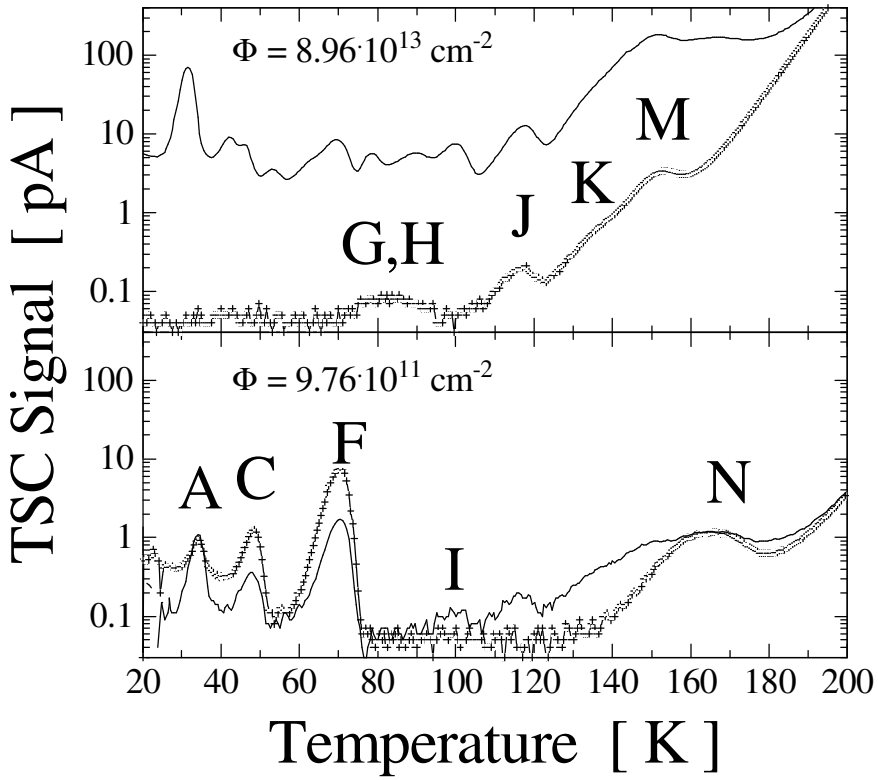

Fig. 5. The type of a defect (electron or hole trap) can be inferred from TSC spectra, provided special modes of defect filling are used. The injection of electrons and holes (—, forward bias) or the injection of only one type of charge carriers (+++, zero bias) is compared for detectors before (bottom, device M20609) and after (top, device M20710) radiation induced inversion of the space charge sign.

5 Reverse bias dependence

Fig. 6 shows the variation of the TSC spectrum as function of the reverse bias i.e. of the electric field in the device for a heavily damaged diode. First of all it is observed that the signal grows monotonically as function of the bias voltage which is easily explained by the corresponding increase of the depletion layer width. Also the shape of the peaks, especially M and N, changes but never looks like the corresponding measurement in Fig. 2 where the defect concentrations are lower by a factor of around 100. The deformed peaks can be explained by the alteration of the effective doping concentration in the space charge region, N_{eff}. At a certain time t during the heating cycle of the TSC measurement N_{eff} is given by

$$N_{eff}(t) = \sum_{donors} \left(N_t - n_t(t)\right) - \sum_{acceptors} n_t(t) . \qquad (6)$$

In this expression also shallow levels are included, e.g. the phosphorus donor which easily releases its electron at the temperatures discussed here and therefore $n_t = 0$ for all times in eq. (6). However all deep levels will enter into eq. (6) according to their initial state of filling and will modify the space charge at the corresponding emission temperature. If the external bias is not sufficient to totally deplete the detector at all times, which would require more than 5000 V for the device of Fig. 6, the depletion layer width will be modified accordingly. Hence the peaks are shifted and their shapes are distorted. In addition the junction may even switch from the p^+ side to the n^+ side and vice versa after emission of a dominant hole or electron trap, which we think is the origin of the apparent kink around 145 K in Fig. 1 and Fig. 6.

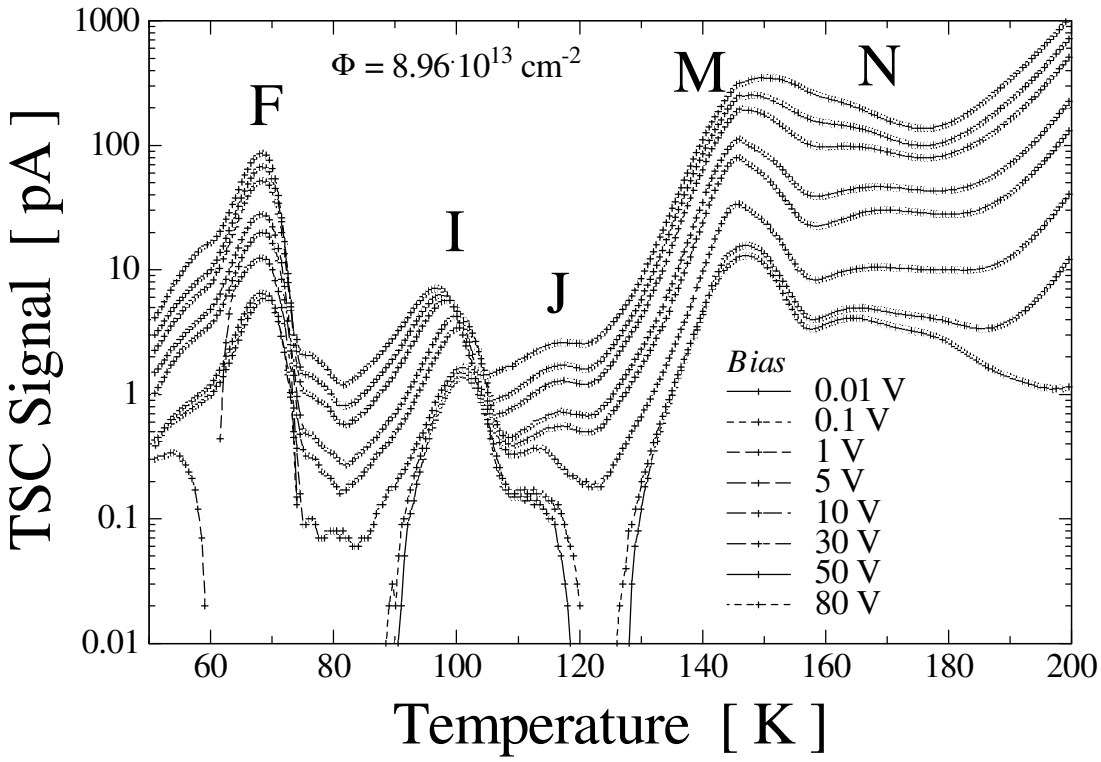

Fig. 6. The reverse bias dependence of the TSC spectrum reveals the field enhanced emission of peak I. β = 0.183 K/s, t_{fill} = 30 s, T_{fill} = 50 K, I_{fill} = 2.5 mA, cooled down under reverse bias 100 V for each measurement, after 7 months of room temperature annealing (device M20710).

The most striking feature of the set of measurements presented in Fig. 6 is the obvious temperature shift of the maximum of peak I. It is concluded that the emission rate is enhanced for a large electric field strength which is most likely explained by the Poole-Frenkel effect. In order to observe a significant influence on the emission rate, even for the low electric fields present here, there must be a long range Coulomb interaction between the ionized defect and the emitted carrier. This supports the previous assignment of peak I to the transition from the double negative to the single negative charge state of the divacancy VV.

6 Conclusions

It has been shown that the TSC method is a powerful technique for surveying deep levels produced in high resistivity silicon by radiation damage. However, it must be clearly stated that deep level parameters can only be derived unambiguously, for example using the delayed-heating method, if their concentration is small compared to that of the shallow dopants. In this case also the C-DLTS method is applicable which furnishes much more accurate data.

Still, TSC measurements are very sensitive and may discover deep levels that have been overlooked in the C-DLTS data. Moreover the TSC method uses a completely different way of defect filling and the measured spectra depend strongly on the corresponding filling parameters. Although this makes the determination of defect concentrations almost impossible the variation of peak heights

as function of the filling pulse duration t_{fill}, current I_{fill}, and temperature T_{fill} can give important additional information concerning the other deep level parameters.

The main benefit of the TSC method in the search for silicon with improved radiation hardness is that the measurements can be performed on standard silicon detectors without any preparation and in the fluence range that is of interest for future HEP applications. Therefore a direct correlation of the well known macroscopic changes with the TSC spectrum should be possible. The basic work in this paper has shown that all peculiarities of the spectra measured on high resistivity silicon detectors with large concentrations of radiation induced deep levels can be understood, provided the initial filling and the variation of the space charge width during the measurement is taken into account.

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