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Radiation damage studies on MCz and standard and oxygen enriched epitaxial silicon devices ☆

E. Fretwurst[a,*], F. Hönniger[a], G. Kramberger[b], G. Lindström[a], I. Pintilie[a,c], R. Röder[d]

[a]Institute for Experimental Physics, University of Hamburg, Hamburg 22761, Germany
[b]Jozef Stefan Institute, University of Ljubljana, Ljubljana 100, Slovenia
[c]National Institute for Materials Physics, Bucharest 077125, Romania
[d]CiS Institut für Mikrosensorik gGmbH, Erfurt 99099, Germany

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Abstract

N-type epitaxial layers of 72 μm thickness and a resistivity of 150 Ω cm have been grown on highly Sb-doped Cz-substrates at ITME (Warsaw). The diode processing was performed at CiS-Erfurt. For comparison a batch of 280 μm thick n-type MCz wafers with a resistivity of >600 Ω cm from Okmetic (Finland) was added to the process run. Depth profiles of the oxygen and carbon concentration were measured via the SIMS-method on as grown epi-layers and after different device-process steps at CiS including an oxygen enrichment at 1100 °C for 24 h. For the MCz material the profiles were measured on untreated samples and after the full device process. Irradiation runs were performed with neutrons at the TRIGA reactor of Ljubljana up to a fluence value of 10^{16} cm^{-2}. The development of the macroscopic device parameters (effective doping concentration and charge collection for α-particles) as function of fluence is presented for the standard and oxygenated epi-devices and compared with the MCz-diodes. The results are discussed in the frame of defect studies resulting from TSC-measurements.

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1. Introduction

Silicon particle detectors are mandatory for vertex detectors in high energy physics experiments at future colliders, in particular, for the proposed luminosity upgrade of the Large Hadron Collider at CERN (S-LHC) or the International Linear Collider (ILC) [1–3]. Especially the upgrade of the LHC will demand the inner most layers of the vertex detector to sustain fluences of about 10^{16} charged hadrons cm^{-2} [4]. Due to the required very high rate capability and spatial resolution small area pixel detectors are the logical choice.

A considerable reduction of the pixel size, essential for the inner layers of the vertex detector, allows correspondingly a smaller detector thickness while maintaining the same input capacitance and hence its influence to the electronic noise. Also the shot noise due to the leakage current will be reduced and thin active layers will certainly reduce the voltage for total depletion. This allows to establish a high electric field strength throughout the sensitive sensor volume and the degradation of the charge collection efficiency due to trapping can be partly compensated.

For the development of thin detectors different technological approaches are possible and under investigation in the frame of the RD50 collaboration [5]. These are thin epitaxial layers grown on highly doped Czochralski substrates [6–8], thinning of high purity magnetic Czochralski (MCz) or FZ devices by chemical etching [9] and processing of thin FZ devices using the so-called wafer bonding technology [10]. In this work we present results on the radiation hardness of 72 μm thick epitaxial devices

☆Work performed in the frame of the CERN-RD50 collaboration.
*Corresponding author. Tel.: +49 40 8998 2956; fax: +49 40 8998 2959.
E-mail address: eckhart.fretwurst@desy.de (E. Fretwurst).

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manufactured on as grown wafers and on oxygen-enriched layers. For comparison MCz detectors had been included in this study since the oxygen concentration was measured to be in the same order as found in the oxygen-enriched epitaxial layers.

2. Devices and experimental procedures

N-type epitaxial layers of 72 μm thickness with a nominal resistivity of 150 Ω cm were grown on highly Sb-doped Cz-substrates (0.01 Ω cm) by ITME. Pad diodes ($5\times 5\,\mathrm{mm}^2$ or $2.5\times 2.5\,\mathrm{mm}^2$ active area) were processed on these epitaxial layers by CiS using either the standard process technology (denoted as EPI-ST) or a preceding oxygen enrichment of the epi-layer by a heat treatment for 24 h at 1100 °C (denoted as EPI-DO). Depth profiles of the oxygen concentration have been measured by the SIMS-method and are displayed in Fig. 1 for both epi-materials and for a 300 μm thick sample of a fully processed MCz wafer. The depth profiles of the EPI-DO and the MCz sample are quite similar with respect to the absolute values of the oxygen concentration as well as their shape, showing in both cases a homogenous distribution throughout the bulk material and a decrease of the oxygen concentration near to the surface, caused most likely by an out-diffusion of oxygen. On the other hand the oxygen concentration of the EPI-ST sample exhibits a strong non-homogeneous profile typically observed for samples of fully processed epi-layers [8].

Irradiations had been performed with neutrons from the research reactor of the Jozef Stefan Institute in Ljubljana. A 1 MeV neutron equivalent fluence value up to $10^{16}\,\mathrm{cm}^{-2}$ was achieved. For both epi-materials as well as the MCz-silicon thermally stimulated current (TSC) measurements were performed with special emphasis on the radiation induced generation of shallow donors, found in former studies on low resistivity (50 Ω cm) epi-material after irradiation with 24 GeV/c protons [8,11]. Charge collection efficiency measurements with α-particles of a ^{244}Cm source were performed for both types of epi-devices. Special charge collection studies with 670 nm laser light pulses were added in order to prove whether the neutron irradiated devices had undergone space charge sign inversion after a certain fluence value or not.

3. Experimental results and discussion

3.1. Effective doping concentration

The development of the effective doping concentration as function of the equivalent fluence for both epi-materials (EPI-ST, EPI-DO) and MCz devices is displayed in Fig. 2. Here the values are taken after annealing for 8 min at 80 °C, corresponding roughly to the stable damage component [7,8]. Since the thickness of the MCz devices is 300 μm the largest fluence was limited to $8\times 10^{14}\,\mathrm{cm}^{-2}$ due to the resulting increase of the full depletion voltage, which is limited to 1000 V by the used voltage source. For the 72 μm thick epi-samples a maximal fluence of $10^{16}\,\mathrm{cm}^{-2}$ could be achieved resulting in full depletion voltages of about 300 V. The solid and the dashed-dotted lines in Fig. 2 represent fits to the epi- and MCz-data according to:

$$N_{\mathrm{eff}}(\Phi_{\mathrm{eq}}) = |N_{\mathrm{eff},0}\times \exp(-c\cdot\Phi_{\mathrm{eq}}) - g_{\mathrm{eff}}\times\Phi_{\mathrm{eq}}| \tag{1}$$

where $N_{\mathrm{eff},0}$ is the initial doping concentration, c is the donor removal coefficient and g_{eff} is the effective introduction rate of defects which are charged in the space charge region. In Eq. (1) it is assumed that g_{eff} represents an effective introduction of negative space charge leading to type inversion above a certain fluence value. As can be seen from the fit lines in Fig. 2 type inversion occurs at about $8\times 10^{13}\,\mathrm{cm}^{-2}$ for the MCz-material and $8\times 10^{14}\,\mathrm{cm}^{-2}$ for both epi-materials. Two main reasons are responsible for this difference. At first, the lower initial doping concentration of the MCz-samples ($4.9\times 10^{12}\,\mathrm{cm}^{-3}$) compared to the epi-samples ($2.5\times 10^{13}\,\mathrm{cm}^{-3}$) results in an earlier

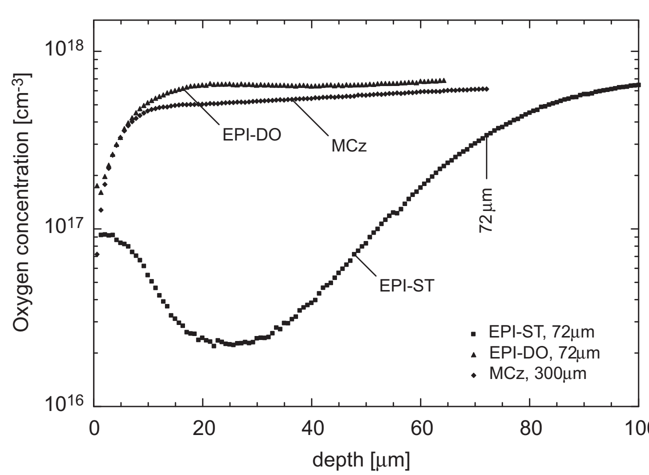

Fig. 1. Depth profiles of the oxygen concentration measured after full detector process with SIMS for standard and oxygen-enriched epi-material (see text) as well as MCz-material.

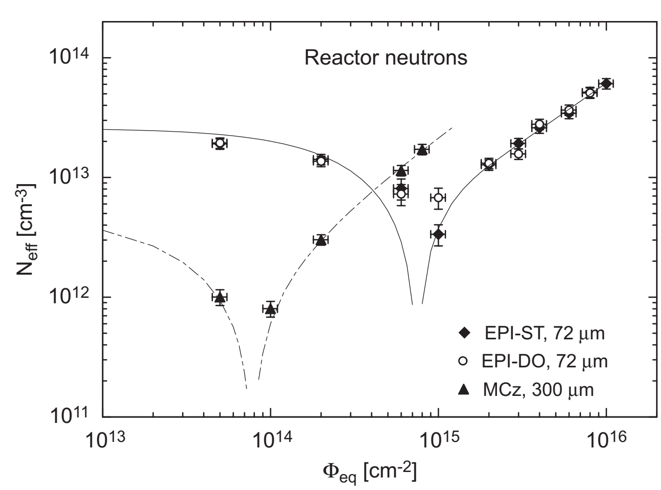

Fig. 2. Effective doping concentration as function of fluence, as measured after annealing for 8 min at 80 °C. Lines represent fits according to Eq. (1).

exhaustion of donors in MCz-material and secondly the introduction rate for MCz ($g_{\mathrm{eff}} = 2.3 \times 10^{-2}\,\mathrm{cm}^{-1}$) is much lager in comparison to both epi-materials ($g_{\mathrm{eff}} = 6.0 \times 10^{-3}\,\mathrm{cm}^{-1}$), to be discussed below.

In order to prove the occurrence of type inversion TCT (transient current technique) measurements were performed by illuminating the p^+ contact with 670 nm laser light pulses. Since the absorption length of 670 nm light in silicon is about 3 μm electron hole pairs are created near the p^+ surface and the charge collection for an inverted device should only become efficient when the applied bias voltage approaches the value for total depletion. In Fig. 3 the collected charge as function of bias voltage normalized to the individual full depletion voltages ($V_{\mathrm{bias}}/V_{\mathrm{fd}}$) is shown for two EPI-ST devices irradiated with $2 \times 10^{14}\,\mathrm{cm}^{-2}$ (non-inverted) and $2 \times 10^{15}\,\mathrm{cm}^{-2}$ (inverted) as well as for one MCz-diode irradiated up to $2 \times 10^{14}\,\mathrm{cm}^{-2}$ (inverted). The voltage dependence of the non-inverted diode follows approximately a square root behavior, as expected for highly irradiated but not inverted diodes, and if the free charge carriers are created near to the p^+-electrode. In contrast to that, for both inverted samples the voltage dependence of the collected charge is governed by three regions. In the first region the increase of the collected charge can be described by a saturating development followed in region 2 by a nearly linear increase up to $V_{\mathrm{bias}}/V_{\mathrm{fd}} = 1$, and finally a further rise is observed in region 3 where the diodes are fully depleted. The voltage dependence in the first two regions is typically observed for inverted diodes and can be explained by the so-called double junction effect in combination with charge carrier trapping as described in different papers [12–14]. On the other hand the behavior in region 3 ($V_{\mathrm{bias}}/V_{\mathrm{fd}} > 1$) can be attributed to charge carrier trapping in the fully depleted diode which is more pronounced in the MCz-device due to the larger thickness and thus longer collection time compared to the inverted epi-device.

3.2. TSC studies

In earlier studies on 50 μm n-type epi-devices with an initial doping of about $7 \times 10^{13}\,\mathrm{cm}^{-3}$ it was demonstrated that this material does not undergo type inversion during exposures to 24 GeV/c protons or reactor neutrons up to equivalent fluences of $10^{16}\,\mathrm{cm}^{-2}$ [6–8,15,16]. Detailed TSC studies had shown that this effect could be correlated with an introduction of shallow donors which always overcompensate the creation of deep acceptors being negatively charged in the space charge region [8]. Thus, the question is why the 72 μm epi-material, under investigation in this work, undergoes type inversion, no matter of the type EPI-ST or EPI-DO. A natural explanation would be that in this case the introduction rate of shallow donors is suppressed or strongly reduced to a level sufficiently below that of acceptors. TSC measurements on all materials demonstrate that shallow donors (denoted as bistable donor $\mathrm{BD}^{0/++}$ in Fig. 4) are generated in both epi-materials but in a much lower concentration in MCz material. All spectra shown in Fig. 4 are taken for a reverse bias voltage which guaranties that the diodes stay fully depleted in the overall TSC temperature range. While the well-known point defects like C_iO_i, the divacancy V_2 and the hole trap H (116 K) are measured with concentrations nearly independent of the diode type, the signal attributed to the VO defect has a

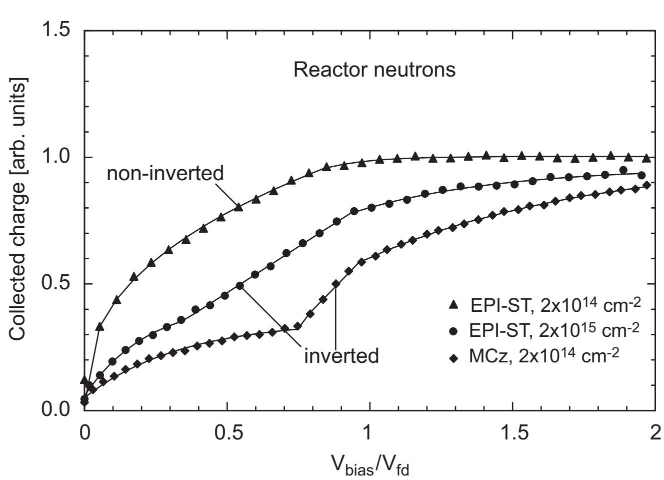

Fig. 3. Collected charge as function of normalized bias voltage $V_{\mathrm{bias}}/V_{\mathrm{fd}}$ (V_{fd} = full depletion voltage) for an inverted MCz, a non-inverted and an inverted EPI-ST diode irradiated with different fluences (see legend). The charge carriers were created by illuminating the p^+-electrode with 670 nm laser light pulses.

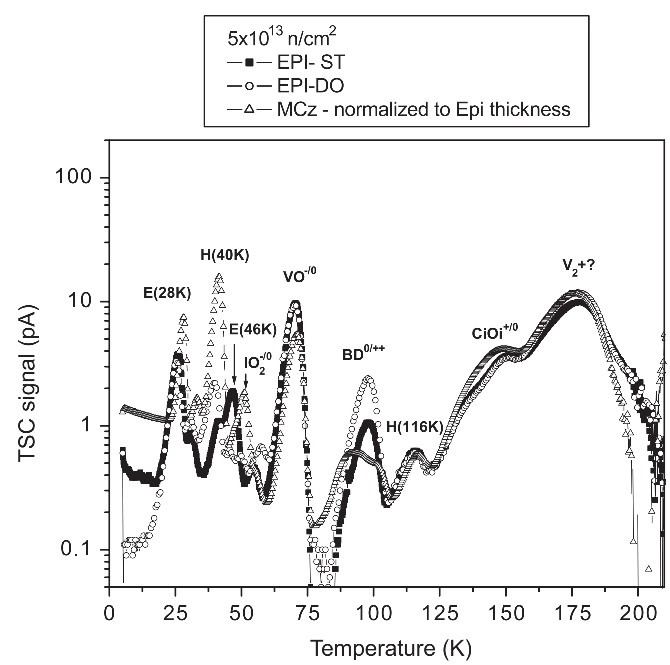

Fig. 4. TSC spectra after irradiation with $5 \times 10^{13}\,\mathrm{n\,cm}^{-2}$ recorded on an EPI-ST, EPI-DO and MCz diode with a reverse bias of 150 V for the EPI diodes and 300 V for the MCz diode. The TSC current of the 300 μm thick MCz diode is normalized to the thickness of the EPI diodes (72 μm). Injection of charge carriers for filling the traps was performed by forward biasing at 5 K.

smaller peak height for the MCz diode than observed for both epi-types. This is due to an incomplete filling of the VO center due to the much lower doping concentration of the MCz material in comparison to the epi-diodes. Strong differences between the TSC spectra of the diodes are seen in the low temperature range (6–60 K): (a) the signal labeled IO_2 is only observed in the MCz diode. The O_2 is known to be a precursor for the formation of shallow donors like the early thermal double donors TDD1 and TDD2 [17,18]; (b) at very low temperatures the spectra recorded for the MCz and the EPI-ST diode show a "tail" in the TSC current which is much more pronounced in the MCz than in the EPI-ST diode. This tail might be attributed to the existence of the BD center in a different configuration [11]. For the first time the bistability of the BD center could be directly observed in the EPI-DO diode as shown in Fig. 5. In the TSC spectrum measured directly after irradiation a peak at 49 K (attributed to $BD^{+/++}$) is recorded which vanishes after a short storage time of 3 h at room temperature, but during a second measurement a strong increase of the $(0/++)$ transition of the BD center is observed. The donor activity of both BD transitions had been proven by the temperature shift of their peak position as function of the applied bias voltage (Poole–Frenkel effect) which is, for example, shown in Fig. 6 for the $BD^{+/++}$ transition. For this diode the BD introduction rate could be accurately evaluated resulting in a value of $g(BD^{0/++}) = 1.7 \times 10^{-2}\,cm^{-1}$. This value represents the rate of positive charge introduced by the BD in the space charge region. Since we assigned the BD as a double donor like TDD2 the introduction rate for the BD concentration would be half of the value given above.

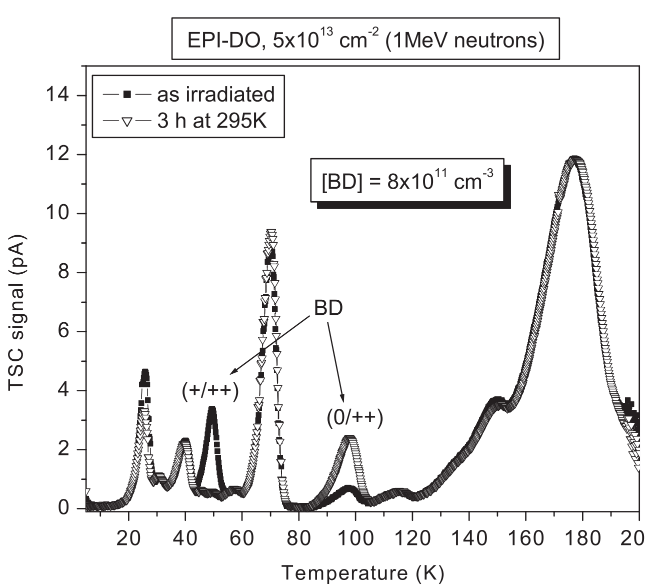

Fig. 5. TSC spectra recorded on an EPI-DO diode as irradiated to $5 \times 10^{13}\,n\,cm^{-2}$ and after storage for 3 h at 295 K. The applied reverse bias and the filling of traps are the same as given in the caption of Fig. 4. For details see the text.

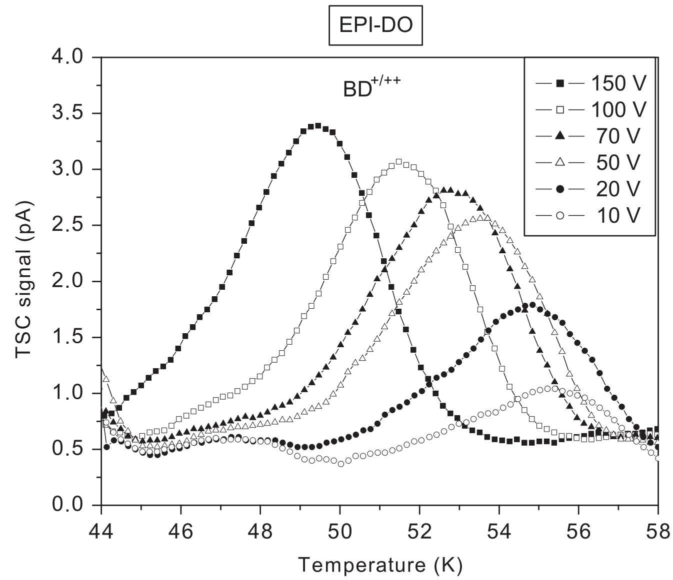

Fig. 6. Donor activity of the BD(+/++) transition. The TSC peak is shifting to lower temperatures when increasing the reverse bias applied during TSC measurement.

In order to get an insight into the influence of the BD on the development of the effective doping concentration as function of fluence for the EPI-DO material as presented in Fig. 2, Eq. (1) can be rewritten as [11]:

$$N_{eff} = N_{eff,0} + [BD] - [E] - [Z] \tag{2}$$

where the term [BD] represents the concentration of positive charge introduced by the ionized BD centers, [E] the concentration of the E center (V-P defect, removal of P donors) and [Z] the concentration of negative space charge at RT introduced, e.g. by the deep acceptor I_p [19], clusters or other unknown defects. A quantitative evaluation for [Z] from TSC- or DLTS-studies could not be given so far. Therefore, as a first guess the introduction rate of [Z] has been deduced from $N_{eff}(\Phi_{eq})$ measurements for neutron irradiated high purity FZ devices. In Ref. [13] a value of $g([Z]) = 2 \times 10^{-2}\,cm^{-1}$ is reported which holds for standard FZ as well as oxygen enriched FZ material. At high fluences, when all P donors are exhausted due to the formation of E centers, N_{eff} is determined by the difference [BD]–[Z] only. For the EPI-DO material with an effective introduction rate for negative space charge of $g_{eff} = 6 \times 10^{-3}\,cm^{-1}$ we would get an introduction rate for positive space charge which is given by the difference $g([Z]) - g_{eff} = 1.4 \times 10^{-2}\,cm^{-1}$ This value is in sufficiently good agreement with the measured introduction rate for the BD center presented above. For the EPI-ST material the BD concentration could not be evaluated with sufficient accuracy and, therefore, a quantitative comparison between the macroscopic parameter g_{eff} and the introduction rate of BD's could not be given. Further, the unexpected large value of g_{eff} for the MCz material, even being larger than that obtained for standard FZ material (see above) could so far not be explained, especially keeping in mind that the oxygen concentration is approximately equal to that measured in the EPI-DO material and assuming that the BD center is an oxygen or oxygen dimer related defect. From the TSC-spectra shown in Fig. 3, it can only be stated that in the temperature range where the $BD^{0/++}$ transition appears the signal measured for the MCz-diode is extremely small and seems to be composed by two defect levels. Further studies are needed for a clarification of the introduction of defects in MCz material.

3.3. Charge collection efficiency

One of the most important issues for the application of silicon sensors in the innermost part of the tracking area of the S-LHC experiments is the charge collection efficiency. As discussed in the preceding Section 2, the required electric field strength throughout the sensitive volume can be readily achieved for epi-devices within the S-LHC fluence range using moderate bias voltages. Degradation of the charge collection due to trapping of the drifting carriers is therefore a major problem. In this work, measurements with a collimated beam of 5.8 MeV α-particles had been performed (see Section 2). Their penetration depth in the devices is 24 μm taking an air gap of 3 mm between the source and the detector surface into account. Thus both electrons and holes contribute to the induced charge. The collected charge is extracted from voltage scans performed very similar to what is shown in Ref. [15]. In our case the maximal bias voltage was kept sufficiently above the value for full depletion such that the drift velocity of the charge carriers approaches nearly the saturation values. As a consequence, the collection time becomes independent of the bias voltage leading finally to a saturation of the charge loss by trapping. The charge collection efficiency CCE is plotted as function of fluence in Fig. 7. The solid line in Fig. 7 presents a fit to the experimental data according to: $CCE = 1-\beta_c \cdot \Phi_{eq}$. For the parameter β_c a value of

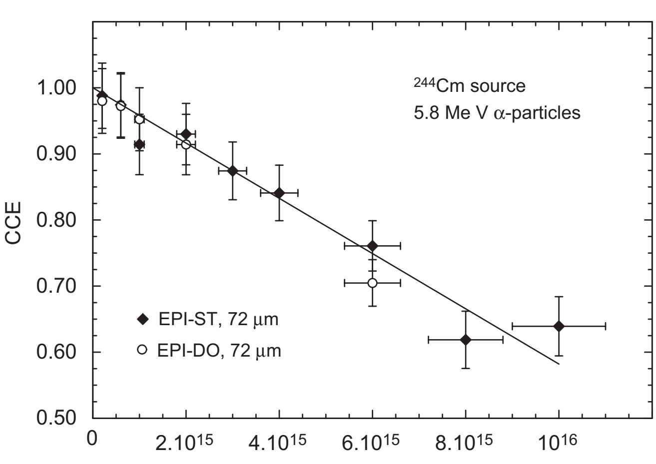

Fig. 7. Charge collection efficiency as function of fluence for EPI-ST and EPI-DO diodes as measured with 5.8 MeV α-particles.

$4.2 \times 10^{-17}\,cm^2$ is derived, resulting in a CCE value of 58% at $10^{16}\,cm^{-2}$. From charge collection measurements with β-particles of an ^{90}Sr source, representing minimum-ionizing particles (mip's), a charge collection of $3200\,e^-$ was measured at $8 \times 10^{15}\,cm^{-2}$ for epi-devices of the same production batch [20]. This corresponds to a CCE value of 56% which is quite similar to that observed for α-particles. These values are in quite good agreement with those reported for 50 μm epi-diodes irradiated with 24 GeV/c protons with the same equivalent fluence [7].

4. Conclusions

The changes of the effective doping concentration and the corresponding full depletion voltage in 72 μm thick 150 Ω cm standard and oxygen-enriched epi-devices as well as MCz-silicon detectors have been measured after irradiation with reactor neutrons in the fluence range up to 10^{16} and $8 \times 10^{14}\,cm^{-2}$, respectively. In contrast to former results on 50 μm epi-detectors with a resitivity of 50 Ω cm type inversion is observed for both 72 μm epi-materials. Also for the 800 Ω cm MCz material this effect is seen but at a 10 times lower fluence as measured for the epi-material. Type inversion had been proven by charge collection measurements with 670 nm laser light pulses. From the analysis of the doping concentration as function of fluence $N_{eff}(\Phi_{eq})$ generation rates for the introduction of negative space charge were evaluated resulting in the same value for both epi-types ($6 \times 10^{-3}\,cm^{-1}$), although the BD concentration derived from the $BD^{0/++}$ transition in the EPI-ST material is smaller than that observed in the EPI-DO material. This might be due to the non-homogeneous oxygen depth profile and related defect concentrations. The larger generation rate observed for the oxygen rich MCz–Si remains an open question which needs to be clarified by further investigations. TSC measurements have clearly shown that in epi-material the introduction of negative space charge is partly compensated by the generation of shallow donors (BD) exhibiting a bistability which could be detected for the first time in the oxygen-enriched epi-diode. Further, the donor activity of the BD's had been proven by the occurrence of the Pool–Frenkel effect.

The charge collection efficiency measured for both epi-types with α-particles is found to be in the same order as measured by β-particles and coincides well with values observed in 25 and 50 μm epi-devices or thin FZ-diodes after irradiation with 24 GeV/c protons.

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