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Nuclear Instruments and Methods in Physics Research A 568 (2006) 66–71
NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH Section A
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Epitaxial silicon detectors for particle tracking—Radiation tolerance at extreme hadron fluences ☆

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Available online 27 June 2006

Abstract

Diodes processed on n-type epitaxial silicon with a thickness of 25, 50 and 75 μm had been irradiated with reactor neutrons and high-energy protons (24 GeV/c) up to integrated fluences of $\Phi_{eq} = 10^{16}\,cm^{-2}$. Systematic experiments on radiation-induced damage effects revealed the following results: in contrast to standard and oxygen-enriched float zone (FZ) silicon devices no space charge sign inversion was observed after irradiation. It is shown that the radiation-generated concentration of deep acceptors, dominating the behavior of n-type FZ diodes, is compensated by creation of shallow donors. Thus a positive space charge is maintained throughout the irradiation up to the highest fluence and even during prolonged elevated-temperature annealing cycles. Defect analysis studies using thermally stimulated current measurements attribute the effect to a damage-induced shallow donor at $E_C - 0.23$ eV. It is argued that, as in the case of thermal donors, oxygen dimers, out diffusing from the Cz substrate during the diode processing, are responsible precursers. Results from extensive annealing experiments at elevated temperatures are verified by comparison with prolonged room-temperature annealing. These results showed that in contrast to FZ detectors, which always have to be cooled, room-temperature storage during beam off periods of future elementary particle physics experiments would even be beneficial for n-type epi-silicon detectors. A dedicated experiment at CERN-PS had successfully proven this expectation. It was verified, that in such a scenario the depletion voltage for the epi-detector could always be kept at a moderate level throughout the full S-LHC operation (foreseen upgrade of the large hadron collider). Practically no difference with respect to FZ-silicon devices was found in the damage-induced bulk generation current. The charge trapping measured with ^{90}Sr electrons (mip's) is also almost identical to what was expected. A charge collection efficiency of 60% (2500 e) in 50 μm n-type epi-diodes after 24 GeV/c proton irradiation with $\Phi_{eq} = 6.2 \times 10^{15}\,cm^{-2}$ was reported recently, independent of the operating temperature down to $-20\,°C$.

PACS: 85.30.De; 29.40.Wk; 29.40.Gx; 61.80.Fe; 61.82.Fk; 71.55.Cn; 29.40.G

Keywords: Tracking detectors; Epitaxial silicon; Neutron and proton damage; Radiation tolerance for S-LHC; Defect spectroscopy; Shallow-donor creation; Oxygen dimers; Depletion voltage; Annealing; Charge collection efficiency; Current-related damage rate

1. Introduction

Silicon detectors will be largely employed in the tracking area of future colliding beam experiments. The proven technology, low cost and large-scale availability make them the favorite choice. However the very large hadron fluence of up to 10^{16} hadrons cm^2 expected for the upgrade of the

☆work performed in the frame of the CERN RD50 collaboration

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doi:10.1016/j.nima.2006.05.203

Large Hadron Collider (S-LHC) will pose an unprecedented challenge as to their radiation tolerance not yet met by present devices [1]. Small-sized pixel detectors will be used to cope with the much larger multiplicity of events. Thus a reduced thickness could be tolerated for keeping the capacitance at a low level. Thin detectors would then allow a much higher doping concentration at moderate depletion voltages. A large donor reservoir in n-type silicon would in turn delay the type inversion effect by radiation-generated acceptors. These considerations had been the main motivation to start a project with diodes processed on thin epitaxial silicon layers [2–5]. On top of the expected benefits it was found that damage-generated shallow donors are created in epi-diodes, compensating the build up of negative space charge by deep acceptors [5,6]. In the following, data are presented extending recent results obtained for 24 GeV/c proton irradiation [4] to investigations with reactor neutrons. The applicability of the proposed thin n-type epi-detectors for the foreseen S-LHC operational scenario was then tested by a dedicated experiment at CERN. It was aimed to verify that the depletion voltage of epi-detectors could be kept at moderate levels throughout the foreseen 5 yr S-LHC operation

2. Samples and experimental procedure

25, 50 and 75 μm thick epitaxial n-type layers with a nominal resistivity of 50 Ω cm were produced by ITME, using highly doped Cz-silicon substrates (0.01 Ω cm) and pad diodes ($5 \times 5\,\mathrm{cm}^2$ active area) were processed by CiS. Depth profiles of resistivity and oxygen concentration are displayed in Figs. 1 and 2, respectively. Oxygen is outdiffusing from the Cz substrate mainly during the high-temperature diode process steps as verified by comparison of SIMS results with process simulations [7,8].

Irradiations had been performed at CERN-PS with 24 GeV/c protons as well with MeV neutrons from the

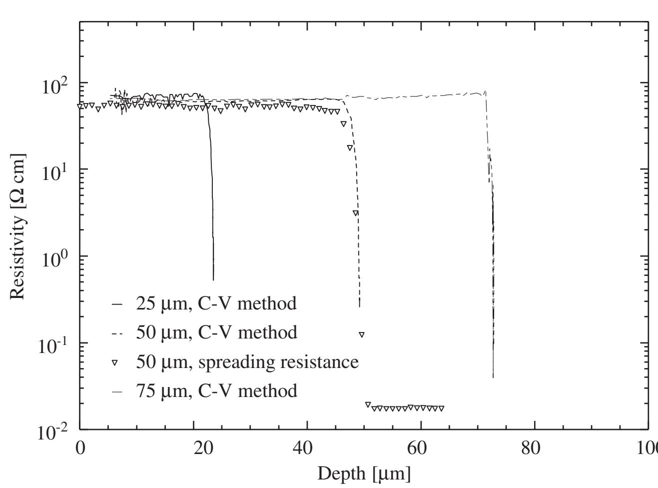

Fig. 1. Resistivity depth profiles as measured by spreading resistance (symbols) and C/V methods (lines) for 25, 50 and 75 μm n-type epi-diodes.

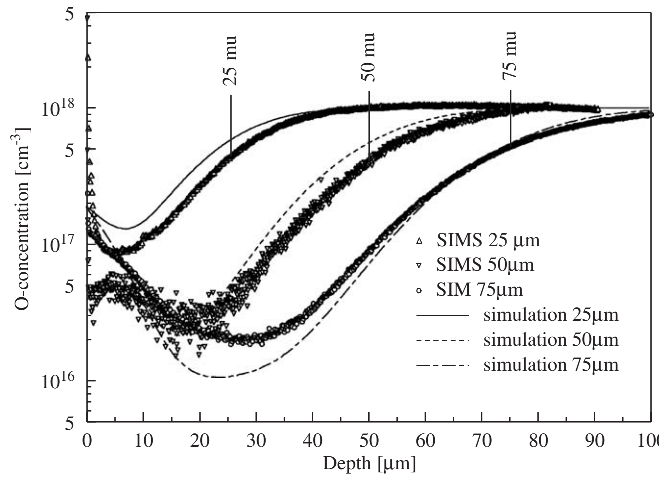

Fig. 2. Oxygen concentation profiles measured with SIMS (symbols) in comparison with process simulation results (lines) in the n-type epi-diodes under test.

research reactor of the Jozef Stefan Institute in Ljubljana. In both cases 1 MeV neutron equivalent fluences of up to $10^{16}\,\mathrm{cm}^{-2}$ were reached. The annealing experiments at elevated temperatures and the details of the analysis had been described in a recent paper [3]. In addition, room-temperature (RT) annealing had been performed for a total period of 250 d. Finally a dedicated CERN scenario experiment, simulating the actual S-LHC conditions, with high-fluence irradiation steps and intermittent annealing was carried out. For all three n-type epi-samples (25, 50 and 75 μm) thermally stimulated current (TSC) measurements were performed with special emphasis on the defect concentration of the shallow donor, identified recently both in Cz and epi-diodes [6]. Results of the charge collection efficiency were already reported in a previous paper showing that a most probable mip signal of 2500 electrons could be reached after proton irradiation with $\Phi_{\mathrm{eq}} = 6.2 \times 10^{15}\,\mathrm{cm}^{-2}$ with adequately short pulse shaping (25 ns) [4]. Also the measurements on the bulk generation current will not be reported here. The obtained data show no difference to the well-known current-related damage rate of $\alpha = 4 \times 10^{-17}\,\mathrm{A\,cm}^{-1}$, measured after an initial annealing for 8 min at 80 °C [9].

3. Results and discussion

3.1. Annealing function at 80 °C

Typical annealing curves for the full depletion voltage V_{FD} respectively, the effective doping concentration N_{eff} for an annealing temperature of 80 °C are shown in Fig. 3. In contrast to float zone (FZ)-silicon diodes [10], the doping concentration stays always positive immediately after irradiation [3]. As known from studies with FZ-silicon diodes part of this initial change, attributed to acceptor generation, is annealing out within a short time after irradiation (“beneficial” annealing) such that a relative

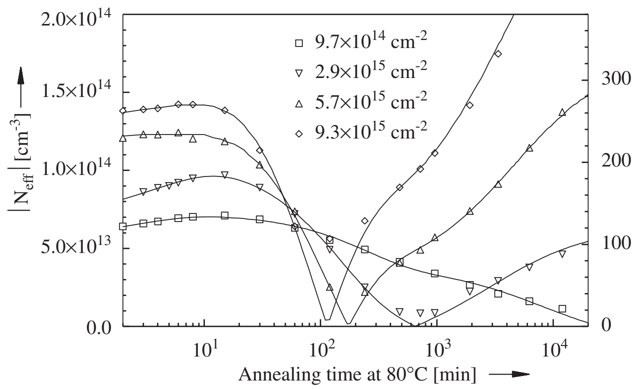

Fig. 3. Examples of annealing function for 50 μm thick n-type epi-diodes at 80 °C after irradiation with different 1 MeV neutron equivalent fluences of 24 GeV/c protons. Lines represent model fits (see text).

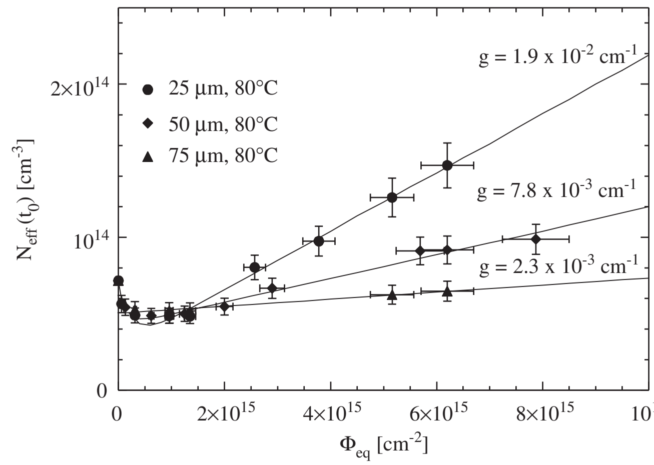

Fig. 4. Effective doping concentration, as measured after the end of beneficial annealing as function of fluence after 24 GeV/c proton irradiation. Solid lines represent fits according to a model description [3], slope values assigned to the curves represent the donor generation rates.

maximum of $N_{\rm eff}$ is observed at about 10 min. With increasing annealing time negatively charged acceptors are prevailing (similar to effects in FZ-silicon) compensating the initial positive space charge and finally leading to type inversion. For an irradiation with $\Phi_{\rm eq} = 9\times10^{15}\,{\rm cm}^{-2}$, type inversion occurs however only at about 100 min, which would correspond to a storage time of 500 d at RT [11]. In contrast, the usually used FZ detectors presently installed in the CMS and ATLAS experiments undergo type inversion immediately after irradiation already for an equivalent fluence below $2\times10^{13}\,{\rm cm}^{-2}$ and subsequently undergo severe reverse annealing effects, thus increasing the necessary full depletion voltage dramatically. A drastic example for the opposite annealing behavior of FZ-diodes in comparison to epi-silicon devices was shown in a recent publication (Fig. 3 in [3]).

3.2. Shallow-donor generation

The build up of positive space charge in epi-diodes with increasing fluence is displayed in Fig. 4. Here the effective doping concentration after 10 min at 80 °C, corresponding roughly to the end of the short-term annealing (see Fig. 3), is plotted as function of the equivalent fluence for all three n-type epi-devices. It should be noted that the results are not parameters extracted from fits, with unevitable additional error margins, but directly taken from the measured depletion voltage. Due to experimental difficulties with C/V measurements at 10 kHz for the 75 μm diodes, all data in Fig. 4 are obtained with 100 kHz. Contrary to the damage effects for FZ silicon the effective doping concentration stays always positive and after crossing a slight minimum even increases almost linearely with fluence. For the chosen annealing stage both the beneficial annealing and also reverse annealing effects do not play a significant role. The values plotted in Fig. 4 are therefore only approximately caused by the change from the initial effective doping concentration due to the stable damage component.

Two main effects attribute to the stable damage (see Ref. [3] for details). The donor removal reduces the positive space charge by formation of E centers (vacancy–phosphorus complex). This component, responsible for the first part of the fluence dependence, is exponentially saturating due to the exhaustion of available P donors. Donor removal is present in both n-type FZ and epi-silicon diodes. A large value of the doping concentration, as possible in thin diodes, will however delay this exhaustion. For larger fluences in epi-diodes the possible creation of acceptors is obviously always overcompensated by donors causing the almost linear increase of $N_{\rm eff}$ with fluence.

It is however striking that the effect of stable donor generation is largely depending on the thickness of the device. Indeed the differences seen in Fig. 4 between 25 μm (large increase) and 75 μm diodes (only small increase) are obviously correlated with the oxygen concentration profiles displayed in Fig. 2. A direct correlation has to be excluded, because a similar effect was not observed in oxygen-enriched silicon (DOFZ) diodes with the same average O concentration. A first understanding was provided by defect spectroscopy investigations, revealing a shallow donor at $E_{\rm C}-0.23$ eV, which is not detectable in FZ diodes [5,6,12]. Fig. 5 shows results of TSC spectra for all three n-type epi-diodes. While the well-known point defects like e.g., C_iO_i, the double vacancy as well as the peak at 115 K, known from previous work in FZ diodes [11] are measured with concentrations independent of the diode type and hence independent of the O concentration, the TSC signal due to the shallow donor (denoted BD) has a similar dependence on the material as the average O concentration (Fig. 2) and the stable damage generation (Fig. 4).

In Fig. 6 a comparison of the effect after proton and neutron irradiation is displayed for the example of the 50 μm diodes. It is well known that, due to Coulomb interaction, the generation of point defects after proton

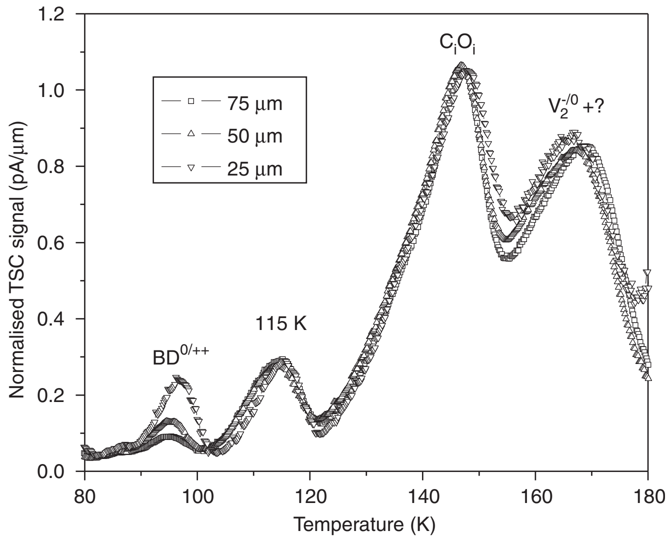

Fig. 5. TSC spectra for 25, 50 and 75 μm n-type epi-diodes after 24 GeV/c proton irradiation with $\Phi_{eq} = 1.2 \times 10^{14}\,cm^{-2}$ and 120 min annealing at 60 °C (top curve: 25 μm, bottom one: 75 μm).

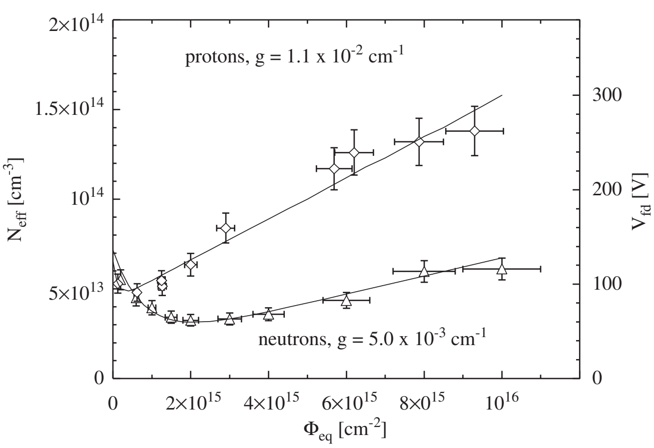

Fig. 6. Comparison between donor generation in 50 μm epi-diodes after 24 GeV/c proton and reactor neutron irradiation, measured at the end of the beneficial annealing. Solid lines are fits using a model description [3]. Given slope values represent the donor generation rates.

irradiation is much larger than for neutron damage, even after normalizing both to NIEL (non ionizing energy loss) equivalent fluence values [13]. It is therefore no surprise and supports our arguments, relating the effect to a point defect, that the positive space charge build up after proton irradiation is much larger than for neutron damage. It is also evident from Fig. 6 that in the case of proton damage the initial donor removal is much faster than after neutron irradiation, a fact which is of course also the result of the relatively much larger point defect generation.

Further evidence for the arguments given above is provided by results comparing the effect after irradiation with different particle types, for which even larger point defect contributions could be expected as e.g., after low-energy proton or Li-ion damage [14,15]. In all these cases it was found that the positive space charge build up is strongly related to the Coulomb interaction part of the damage function.

The following tentative explanation for the generation of the BD donor and hence the superior radiation tolerance of n-type epi-diodes had been proposed in Refs. [5,6]. A strong similarity of the BD complex to thermal double donors and the well-known fact that oxygen dimers O_{2i} are precursors for the formation of thermal donors (see Ref. [16]) leads to the assumption that dimers are involved in the damage-produced BD defects. It is also known that oxygen dimers have a much larger diffusion constant than oxygen interstitials. Thus it can be expected that they outdiffuse from the Cz substrate predominantly after the last high-temperature process step, thus leading to a relatively larger dimer concentration in the epi-diodes than could be expected from the oxygen concentration alone. In fact the ratio between the O_i concentrations in 25 and 50 μm diodes was measured to be considerably lower (1.8) than that for the BDs (2.2), giving further evidence to this assunption [5]. In addition, by monitoring the oxygen dimers with measurements of the IO_{2i} concentration an appreciable enhancement of the relative dimer ratio in n-type epi-diodes with respect to that found in n-type Cz silicon by at least a factor of 2 was shown. In standard and oxygen-enriched n-type FZ diodes no IO_{2i} defect was found supporting the general picture [17].

In conclusion it is therefore very likely that the shallow donor (the BD complex), found in epi-silicon after irradiation, is indeed responsible for the dominating positive space charge build up in these devices and that this donor is generated via the enhanced concentration of oxygen dimers outdiffusing from the Cz substrate.

4. Application of epi-detectors for S-LHC

In a a first paper [2] on systematic investigations of damage effects in epi-diodes it was argued that the effect of a fluence-depending increased positive space charge creation could be partly compensated by the generation of acceptors during annealing, as can also be seen in Fig. 3. It was suggested that in contrast to FZ-silicon detectors which would always have to be stored at low temperatures in order to avoid an intolerable increase of the depletion voltage, in the case of epi-detectors such low-temperature storage is not necessary. In addition, keeping the detectors at RT during beam-off periods could be very profitable because the increase of the depletion voltage as resulting from donor generation during the beam periods would be partly compensated by annealing-induced acceptor generation. In order to prove this suggestion the following dedicated experiment had been performed. Both 50 and 25 μm epi-diodes had been irradiated with 24 GeV/c proton fluence steps of $\Phi_{eq} = 2.2 \times 10^{15}\,cm^{-2}$. After each damage step the diodes were annealed for 50 min at 80 °C corresponding approximately to the 265 d beam-off period at RT. The compression factor between 20 and 80 °C was taken to be 7500 for this evaluation, according to an activation energy for the involved kinetics of 1.31 eV [11]. On the other hand a simulation was used calculating the depletion voltage along the full irradiation and annealing cycles. Values extracted from a detailed parameterization fit of extensive annealing experiments at 60 and 80 °C had been employed [3]. After each annealing step the depletion voltage had been measured followed by the next irradiation step. The meaured data of this experiment together with the simulations are given in Fig. 7. An excellent agreement was obtained both for the 25 μm as well as the 50 μm diodes. It is clearly seen that the depletion voltage of the 25 μm diode is increasing with fluence (by roughly a factor of 2) while that of the 50 μm diode is even slightly decreasing. This is of course due to the fact that in the case of the thin 25 μm diode the positive space charge is much more increasing with fluence and hence less compensated by the annealing-induced acceptors than in the case of the thicker (50 μm) one.

In addition to the reverse annealing experiments at elevated tempertures reported in Ref. [3], for a few cases RT annealing had also been carried out both after high-fluence proton and neutron irradiation [18]. An example is shown in Fig. 8. In addition to the experimental data a fit had been performed, using the model description detailed in Ref. [3]. The relevant parameters, known from the 80 °C annealing experiment, are reproduced by the 20 °C fit within reasonable error margins, e.g., for $\Phi_{eq} = 6.3 \times 10^{15}\,cm^{-2}$ the fit in Fig. 8 gives a stable damage component of $8.9 \times 10^{13}\,cm^{-3}$, identical to what had been found from annealing at 80 °C. Also the first reverse annealing amplitude (the fit in Fig. 8 gives $2.2 \times 10^{14}\,cm^{-3}$) is in good agreement with the 80 °C value of $1.9 \times 10^{14}\,cm^{-3}$ as taken from Ref. [3]. The RT annealing appears however to be faster by a factor of approximately 3 than expected from 80 °C if an activation energy of 1.31 eV is assumed as given in Ref. [11]. Evidently more annealing studies also at lower temperatures and in the time domain

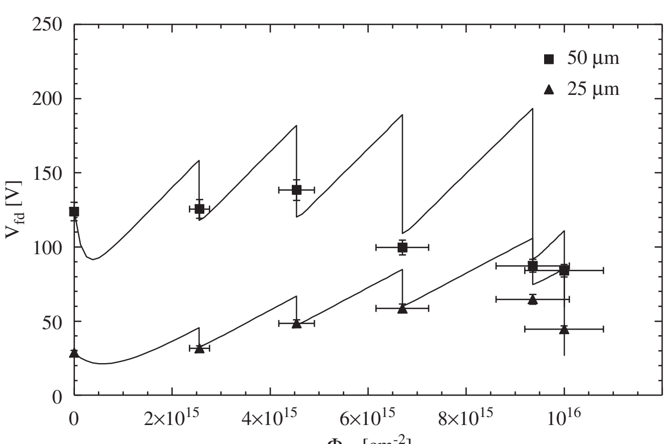

Fig. 7. Results of successive irradiation with 24 GeV/c protons with steps of $\Phi_{eq} = 2.2 \times 10^{15}\,cm^{-2}$ followed by annealing for 50 min at 80 °C. Symbols: experimental points, solid lines: simulations (see text).

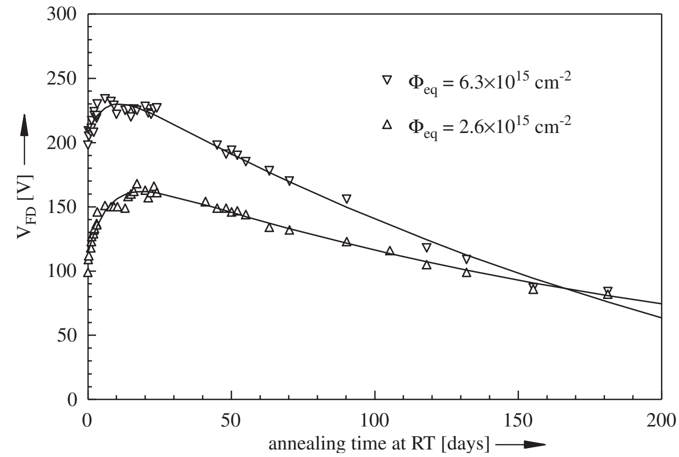

Fig. 8. RT annealing curves (20–23 °C) for the full depletion voltage of 50 μm n-type epi-diodes after 24 GeV/c proton irradiation with 2 different fluences. Symbols: experimental points, solid lines: fits according to a model description [3], see text.

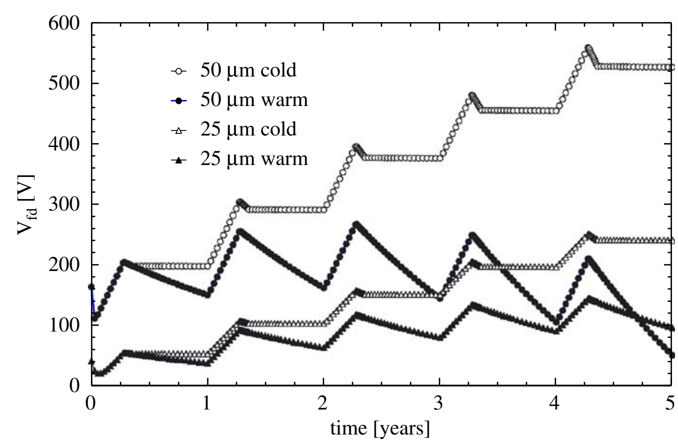

Fig. 9. Simulation results for the depletion voltage of 25 and 50 μm n-type epi-diodes kept at −7 °C during the operational periods of 100 d yr^{-1} and at RT (20 °C) during the beam-off periods of 265 d every year for the full S-LHC operation (full symbols). In comparison the behavior is shown if the devices are stored at −7 °C during the entire 5 yr (open symbols).

of interest here, i.e. equivalent to several 100 d at RT are necessary.

This experimental evidence for the validity of our parameter representation regarding the damage and annealing behavior gave confidence to a simulation describing the evolution of the depletion voltage for epi-detectors in the real S-LHC scenario for the innermost pixel layers at $R = 4$ cm. In this case the parameters extracted from the proton experiments were used for the pion damage while a separate investigation revealed the parameters needed for neutron damage [19]. The total fluence per year was assumed to be $\Phi_{eq} = 3.48 \times 10^{15}\,cm^{-2}$ containing a neutron contribution of 10%. Again RT storage was assumed during the beam-off periods every year. In comparison the calculations have also been done for cold storage throughout the entire 5 yr operation. The results of this simulation are shown in Fig. 9. As explained above (Fig. 7) the compensation effect of the increasing positive space charge build up by negatively charged acceptors created during annealing is more pronounced in the 50 μm diode than for the 25 μm one. As the thin n-type epi-diodes are rather unlikely to be used because of the high capacitance and small signal the behavior of the 50 μm epi-diode would give a better view of what could be expected via an optimization of thickness, technology (dimer concentration) and beam-off storage conditions. It seems to be quite plausible that a dedicated development along these lines will result in pixel devices for the innermost layers easily withstanding the extremely intense radiation at moderate operational voltages while maintaining an acceptable signal response for mip identification.

5. Conclusions

Thin epitaxial-grown low resistivity silicon diodes have been investigated after 24 GeV/c proton and reactor neutron damage up to equivalent fluences of $10^{16}\,\mathrm{cm}^{-2}$. It is shown that these devices exhibit an unexpected large radiation tolerance superior to any FZ silicon. The effect is attributed to the damage-induced generation of a shallow donor which is very likely created via the presence of oxygen dimers similar to the thermal donors for which the dimers are known to be precursors. It is argued that these dimers are outdiffusing from the Cz substrate during the diode processing and due to their larger diffusion constant lead to a comparatively large concentration not seen in any FZ material. While the positive space charge build up by these donors is unique for epi-silicon the acceptor creation during annealing is very similar to what had been observed in FZ diodes. The compensation of the positive space charge generation during the irradiation by negatively charged acceptors during annealing provides a distinct possibility for keeping the depletion voltage at moderate levels, if the low-temperature operation during the beam periods is accompanied with RT storage during beam-off times every year. A dedicated experiment with successive 24 GeV/c proton irradiation steps and intermittent room temperature equivalent annealing had validated this expectation. Simulations for a realistic S-LHC 5 y operational scenario have shown that detectors built on the basis of epi-silicon could withstand the extreme radiation environment while keeping the operational voltage at very moderate levels.

Further developments will be undertaken regarding the epi-layers (effective doping concentration, including p-type doping, thickness), diode processing (possible control of dimer concentration) and storage conditions during beam-off times (temperature regime).

Acknowledgments

Many thanks are due especially to M. Glaser at the CERN PS irradiation facility for his invaluable help. Likewise we like to thank the team at the research reactor of the Jozef Stefan Institute in Ljubljana. A. Barcz from the Physics Institute of the Polish Academy of Sciences in Warsaw had executed the very reliable SIMS measurements and Li Long from the Institute for Micro Sensors CiS at Erfurt provided the process simulations. Many thanks are expressed to both of them. This work was performed in the frame of the RD50 collaboration. It was partly funded by German Research Foundation DFG under Contract FR1547/1-2 which is gratefully acknowledged.

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