Accepted for publication in NIMB

# RELATION BETWEEN MICROSCOPIC DEFECTS AND MACROSCOPIC CHANGES IN SILICON DETECTOR PROPERTIES AFTER HADRON IRRADIATION

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#### Abstract

Silicon detectors produced from materials with different resistivity and oxygen concentration have been irradiated with energetic neutrons, protons and pions. Isothermal annealing studies have shown correlation between microscopic defect evolution and the macroscopic detector performance. It was found that the annealing behavior of the electron traps attributed to the single and double charged divacancy is strongly related to the current related damage parameter  $\alpha$ . In both cases the isothermal evolution is independent of the oxygen and doping concentration in the material under investigation  $(2 \times 10^{14} < [O] < 10^{18} \text{ cm}^{-3}$  and  $10^{12} < [P] < 4 \times 10^{13} \text{ cm}^{-3}$ ) and the absolute values do not depend on the particles used for the irradiation provided the fluence is properly normalized by the non ionizing energy loss (NIEL). In contrast to this result the introduction rates of the observed point defects VO<sub>i</sub> and C<sub>i</sub>C<sub>s</sub> were however found to depend on the particle type. Thus clear indication is given that the generation of point defects does not scale with NIEL. Compared to neutron irradiated samples the introduction rate after irradiation with charged hadrons was found to be higher by a factor around 2.

**Keywords:** silicon detector, radiation damage, neutron, proton, pion, defects, DLTS, generation current, annealing

#### **1. Introduction**

The long term operation of silicon detectors in the extremely intense hadronic radiation fields of high energy physics experiments leads to a degradation of the detector properties due to radiation damage in the silicon bulk. An increase of leakage current, a change of effective doping concentration and a decrease in charge collection efficiency are well known effects of hadron irradiation [1, 2]. However, only little is known about the nature of the radiation induced defects in the silicon bulk that lead to the different changes in the macroscopic detector properties [3-5]. Without such a knowledge, attempts to produce radiation harder silicon by defect engineering are based on a try and

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error strategy only. Thus, a more complete understanding of the relation between the microscopic defects and the macroscopic detector properties is essential.

It has been shown that the increase in leakage current as well as its annealing after irradiation with neutrons is independent of the used silicon material [6] and that an electron trap at about  $E_c$ -0.42eV is correlated with the leakage current [4]. Furthermore it was shown that in oxygen rich silicon the radiation induced changes of the effective doping concentration are suppressed in charged hadron damage while after irradiation with neutrons no or only little suppression is found [1, 2, 7, 8]. This important result was the motivation for the related microscopic investigations reported here for the first time.

## 2. Experimental procedure

The device parameters of the  $5x5mm^2$  diodes used in this experiment are listed in Tab. 1. The given oxygen and carbon concentrations have been measured by IR absorption and/or SIMS prior to the processing of diodes. As indicated in some of the cases these values are below the detection limits of the used methods. The broad range of [O] and [C] in the epitaxial p-type material produced by ITME reflects the inhomogeneous depth profile derived from SIMS-measurements.

The samples were exposed to different particle fields. Irradiation experiments have been performed with fast neutrons of the d+Be source (mean energy 5.3 MeV) at the PTB Braunschweig/Germany [9], reactor neutrons at the Ljubljana/Slovenia reactor facility [10], 192 MeV pions at the PSI/Villigen [11], 27 MeV protons at the Legnaro/Italy accelerator [12] and 23GeV protons at the PS/CERN [13]. All fluences given in this paper are normalized to a 1 MeV equivalent neutron beam using the appropriate hardness factors [14] if not explicitly mentioned otherwise.

For the investigation of the annealing process with the DLTS (Deep Level Transient Spectroscopy) technique an isothermal heat treatment at 60°C was chosen to accelerate the process with respect to room temperature. A commercially available DLTFS-apparatus [15] was used for the defect characterization which is described in more detail in [16]. The displayed spectra correspond to the sinus correlator function (b1) and were obtained with a time window of 200ms. The quiescent reverse bias was 10V and during the 100 ms lasting filling pulse 0V was applied to the sample. During the filling with holes a forward bias of 3V was applied.

Additionally, isothermal leakage current annealing studies were performed in parallel to the DLTS measurements at 60°C and at further temperatures in the range from 21°C to 106°C. Between the isothermal heating steps the I/V characteristics were measured at room temperature with the guard ring of the diodes properly connected to ground. All presented leakage current data are normalized to 20°C. Further details about the determination of the bulk generation current can be found in [6, 16].

#### **3.** Experimental results

#### 3.1 Isothermal annealing studies

#### **3.1.1 Reverse current**

The radiation induced increase of the reverse current  $\Delta I$  is given by the difference between the currents measured at total depletion of the device before and after irradiation. It has been shown for numerous samples and different radiation fields that the increase of the current per unit of depleted volume  $\Delta I/V$  for a well defined annealing state after irradiation is proportional to the fluence  $F_{eq}$  and can thus be described by (see e.g.[6, 17, 18])

$$\Delta I/V = \mathbf{a} \, \mathbf{F}_{eq} \tag{1}$$

where the proportionality factor  $\alpha$  is called *current related damage rate*. This relation was found to be valid for a wide fluence range from  $10^{10}$  cm<sup>-2</sup> to  $10^{15}$  cm<sup>-2</sup> and therefore independent on the fact that some samples are inverted in conduction type after exposure to high fluences [6].

The annealing behavior of the current related damage rate a after irradiation is displayed in Fig. 1 for various temperatures in the range from 21°C to 106°C. The a value, respectively the leakage current, is continuously decreasing with increasing annealing time and only for the highest temperature, after a 2 months lasting annealing at 106°C, a saturation is indicated at a value of about  $6 \times 10^{-18}$  A/cm.

For room temperature the annealing curves were usually fitted to a sum of exponentials and a saturation value  $a_{\mathbf{x}}$  as indicated in Fig. 1 by the dashed-dotted line [17,18,19]. However such parameterization does not represent sufficiently the long term annealing data at room temperature for cumulated annealing times t>1 year. This is most obviously seen in the figure from the annealing at higher temperatures where no saturation value  $a_{\mathbf{x}}$  can be seen. Instead, in the long term annealing the  $\mathbf{a}$  value seems to follow a logarithmic function in time. Thus the long term annealing at room temperature and the annealing at elevated temperatures was chosen to be described by one exponential and a logarithmic term:

$$\mathbf{a}(t) = \mathbf{a}_0 \times exp(-t/\mathbf{t}_I) + \mathbf{a}_I - \mathbf{a}_2 \times \ln(t/t_0).$$
<sup>(2)</sup>

For each temperature the data were fitted according to Eq.(2) with  $t_0$  set to 1 min. The corresponding parameters are displayed in Tab. 2. For the exponential term the weighted average value for the amplitude  $\mathbf{a}_0$  was found to be  $\mathbf{a}_0 = (1.23\pm0.06)\times10^{-17} \text{ A/cm}$  and an Arrhenius plot of the time constant  $\mathbf{t}_I$  revealed [16]:

$$1/\mathbf{t}_I = k_{0I} \times exp(-E_I/k_B T_a) \tag{3}$$

with  $k_{0I} = 1.2^{+5.3}_{-1.0} \times 10^{13} s^{-1}$  and  $E_I = (1.11\pm0.05) eV$ . Thus, with a first order kinetics and a frequency factor  $k_{0I}$  close to the most abundant phonon frequency, it is very likely that a dissociation of a defect is responsible for the exponential part of the leakage current annealing.

The interpretation of the logarithmic part of Eq.(2) is far more complicated and it is noted here that the presented parameterization does not claim to be based on a physical model. The average value of the parameter  $\mathbf{a}_2$  is given by  $\mathbf{a}_2 = (3.07 \pm 0.18) \times 10^{-18} \text{ A/cm}$ , whereas the parameter  $\mathbf{a}_1$  clearly displays a temperature dependence which can be described by [16]:

$$\boldsymbol{a}_{l}(T_{a}) = \boldsymbol{a}_{10} + \boldsymbol{a}_{11} \times 1/T_{a} \tag{4}$$

with  $\mathbf{a}_{10} = -(8.9 \pm 1.3) \times 10^{17} \text{ A/cm}$  and  $\mathbf{a}_{11} = (4.6 \pm 0.4) \times 10^{-14} \text{ AK/cm}$ . It has to be noted that the given parameterization only holds for the time and temperature range indicated in Fig.1.

#### 3.1.2 DLTS studies

In parallel to the measurements of the current annealing described above the isothermal annealing at 60°C of neutron induced defects has been studied by the DLTS method. In this work the annealing of a detector fabricated from oxygen rich ntype Cz material (Cz-140, see Tab. 1) will be discussed in detail while similar results for the other materials are published elsewhere [16, 20]. Fig. 2 demonstrates the evolution of the DLTS spectrum for a Cz sample within a period of 85 hours. As typical for all investigated materials four peaks are detected in the majority carrier spectrum in the temperature range between 50K and 250K. They are labeled by the letter E and a number. However, in this special device the peak E1 can be attributed to a superposition of a thermal donor  $TD^{(0/+)}$  and the carbon interstitial  $C_i^{(-/0)}$  whose concentration decreases very quickly at 60°C while the concentration of the TD defect does not change with proceeding annealing time. The DLTS signal E2 is assigned to be the well known A-center  $VO_i^{(-/0)}$  and an increasing part which is associated with the  $C_iC_s^{(-/0)}(A)$  trap induced by the reaction  $C_i + C_s \rightarrow C_iC_s$ . The dynamic of such processes in high resistivity silicon has been described elsewhere [16, 21, 22].

In the following we will concentrate on the evolution of the peaks E3 and E4. Both peaks cannot be attributed to isolated point defects. E4 is usually assigned to be the singly charged divacancy  $VV^{(-/0)}$  and E3 to the doubly charged divacancy  $VV^{(-/-)}$  inside or nearby disordered regions or clusters [4, 23, 24]. Although both levels are due to the same defect their observed peak heights are different. According to a model described in [23] this is due to lattice strain in the vicinity of clusters which reduces the signal of the doubly charged divacancy (E3).

It is conspicuous that the peak height of the trap E4 is not only decreasing with cumulated annealing time but the peak maximum shifts also in its position to lower temperatures. This indicates that a trap level hidden in the right hand side shoulder of the peak anneals out. More information on this hidden trap level can be gained if one analyses the "difference spectra" which have been extracted by subtracting each spectrum measured after a certain annealing time from the spectrum taken after the first heat treatment of 5 min. Fig. 3 shows the corresponding evolution of the "difference spectra". Obviously, there is a trap level visible with a peak maximum at T = 212 K (E4b). The displayed increase with cumulated annealing time corresponds to a decrease in defect concentration. Thus the defect concentration extracted from the peak heights of peak E4a in Fig.3 of e.g. the difference spectrum  $\Delta(5\min-82000\min)$  corresponds to the decrease in concentration of defect E4a between the isothermal annealing steps of 5min and 82000 min at 60°C. The extracted trap parameters are included in Tab. 3. A similar annealing behavior is observed for the broad peak E4a. In contrast to E4b the peak shape of E4a cannot be described by a single defect level. The difference signal of trap E3 has an opposite sign which means that the defect concentration is increasing with progressing heat treatments. The "difference spectra" were used for determination of the defect parameters and trap concentrations of E4a and E4b (see Tab. 3) since they allow a more accurate analysis than the spectra shown in Fig. 2 in which E4a and E4b are superimposed with further levels. In Fig. 4 the trap concentrations normalized to the 1MeV neutron equivalent fluence  $\Phi_{eq}$  as function of cumulated annealing time are shown for all three electron traps. For comparison the annealing of the current related damage rate  $\alpha$ is included. The observed time dependence for the concentration of the E4b and E4a is quite similar to that found for the leakage current particularly for the short term component. A more direct correlation of the leakage current with the trapping center E4b can be seen if one plots the differences  $\Delta \alpha = \alpha(5 \text{ min}) - \alpha(t_a)$  versus the differences in the trap concentration  $\Delta N_T / \Phi_{eq} = N_T / \Phi_{eq}(5 \text{ min}) - N_T / \Phi_{eq}(t_a)$  taken at the same annealing time  $t_a$  (see Fig. 5). The proportionality between both quantities hold for the short term annealing period up to 640 min and the resulting slope is found to be  $(4.18\pm0.08)\times10^{-17}$ A. Such a correlation between the electron trap E4b and the leakage current has also been found in isochronal annealing experiments [4]. Since the hole capture cross section of the level E4b is unknown it is not possible to calculate the generation current produced by the defect from the level parameters measured by DLTS. However, using the standard Shockley-Read-Hall model and the relation between the defect concentration and the generation current given above the hole capture cross section can be estimated to be about  $1\times10^{-13}$ cm<sup>2</sup>. It is noted that this value would actually be too large if other mechanisms like the inter-center charge transfer suggested in [25] enhance the current generation.

### 3.2 Material dependence

Similar studies have been performed on devices fabricated from different materials (see Tab. 1). As an example in Fig. 6 DLTS spectra for such different samples after neutron irradiation and annealing for 80 minutes at 60°C are shown. Here the peak amplitudes of the individual E4 signals are normalized to corresponding introduction rates  $N_T(E4))/\Phi_{eq}$ . A comparison of the introduction rates for all three trapping centers E2, E3 and E4 is demonstrated in Fig. 7 and shows that the rates for the traps E3 and E4 are independent on the impurity or doping concentration of the material under investigation. On the other hand the introduction rate of the point defects  $VO_i$  and  $C_iC_s$  seen in peak E2 show a distinct dependence on the impurity concentration of the material. For devices with high oxygen concentration the introduction rate is about a factor two smaller compared to material with a standard oxygen content. This effect is related to the dependence of the defect kinetics on the impurity concentration [16, 26]. A high oxygen concentration leads to a shift of the C<sub>i</sub> sharing between the two reactions  $C_i + C_s \rightarrow C_i C_s$  and  $C_i + O_i \rightarrow C_i O_i$  in favor of the second one, i.e. the  $C_i C_s$  signal is reduced in oxygen rich material compared to standard material.

For numerous detectors fabricated from the materials mentioned above and additional p-type material (see Tab. 1) the isothermal annealing of the leakage current at  $60^{\circ}$ C has been measured.

The extracted  $\alpha$  values after annealing for 80 minutes are plotted in Fig. 8 as function of the effective doping concentration  $N_{eff}$ . In the legend of Fig. 8 the oxygen concentrations of the different materials are given. It is very striking that the current related damage rate  $\alpha$  does not depend on the material properties of the silicon, although the oxygen concentration is varying by about 4 orders of magnitude ( $10^{14}$  cm<sup>-3</sup> < [O<sub>i</sub>] <  $10^{18}$  cm<sup>-3</sup>) and the resistivity is ranging from about 400  $\Omega$ cm to 4 k $\Omega$ cm in p-type and from about 100  $\Omega$ cm to 25 k $\Omega$ cm in n-type materials. Since such strong variation in impurity content has an influence on the defect kinetics of migrating vacancies and interstitials (see above) it is concluded here, that after irradiation with neutrons mainly intrinsic defects composed of vacancies and interstitials inside or close to the clusters (DLTS peak E4) are responsible for the increase of the generation current but not impurity related defects.

#### 3.3 Dependence on particle type

In this section we present DLTS measurements performed on oxygen enriched FZ material which has been irradiated with 23 GeV protons, 27 MeV protons, 192 MeV pions and 5.3 MeV neutrons. The results will be compared with observed changes in the leakage current induced by the same particles. The aim of these studies was twofold. First, it should be clarified whether the correlation between the current damage rate  $\alpha$  and the DLTS signal E4 could also be established for charged hadrons and whether the NIELhypothesis (non ionizing energy loss) for the scaling of the damage parameter  $\alpha$  and the introduction rate of the trap E4 is valid for all hadrons under study. Secondly, it was recently found by the ROSE collaboration [1, 7] that the radiation induced change of the effective doping concentration  $N_{eff}$  after exposure to charged hadrons is strongly reduced for oxygen rich material compared to material with standard oxygen concentration. However, after neutron irradiation no difference was observed between oxygen rich and standard material.

The extracted current damage rates  $\alpha$  as a function of fluence for different particles are summarized in Fig. 9. The  $\alpha$  values correspond to the annealing state of 80 min at 60°C. On the right hand side ordinate of Fig. 9 the so called hardness factors  $\mathbf{k}$  of the different particle fields are given which coincide with values calculated from published NIEL-functions [14]. All plotted values are derived from individual devices irradiated to the given fluences. These results demonstrate that the radiation induced increase of the bulk generation current scales with the displacement damage cross section (NIEL) for charged and neutral hadrons.

DLTS measurements have been performed on detectors fabricated from oxygen enriched silicon (II-800, see Tab. 1) after irradiation with different particles as mentioned above. Also in this study all samples have been annealed to 80 min at 60°C. The spectra shown in Fig. 10 are scaled in such a way that the E4 peak gives identical peak maximum values. The 1 MeV neutron equivalent introduction rates of the defects are given in Tab. 4. As can be seen the introduction rate of the E4 center is nearly independent of the damaging particle type. In Fig. 11 the introduction rates of E4 are compared with their corresponding  $\alpha$  values. A nice correlation between both quantities can be stated which substantiates the assumption that the E4 center can be attributed to intrinsic defects in clusters and that the introduction rate scales with the non ionizing energy loss. The significantly larger E3 signal (see Fig.10) for charged hadrons indicates that the lattice strain is less pronounced in charged particle damage.

A more pronounced difference is observed for point defects as demonstrated by the different peak heights of the E2 signal (associated with the superposition of VO<sub>i</sub> and C<sub>i</sub>C<sub>s</sub> signals) and the H1 signal (associated with the C<sub>i</sub>O<sub>i</sub> signal). The introduction rates of all 4 trapping centers are summarized in Fig. 12 and Tab.4. The defects E2 and H1 are well known point defects while the signal E4 is best related to defects located in clusters. Therefore, the ratio (E2+H1)/E4 given in Tab.4 can be regarded as a measure of the ratio of point defects to clusters. As can be seen the value for 23 GeV protons and 192 MeV pions is 2.4 and for 27 MeV protons 3.3. However, neutrons have only a ratio of 1.4. Such an enhanced introduction of point defects by charged particles compared to neutrons lead to the suggestion that this effect is caused by the coulomb interaction of charged particles with silicon atoms in the lattice. Coulomb interaction will create primary knock-on atoms (PKAs) with much smaller recoil energies leading finally to an enhanced generation of isolated point defects. Since it is still unknown which defects are responsible for the change of  $N_{eff}$  it is difficult to understand why the change in  $N_{eff}$  is suppressed in oxygen rich material after irradiation with charged hadrons. So far we can only speculate. Since the change in  $N_{eff}$ is mainly due to the generation of negatively charged defects in the space charge region one may think e.g. of an enhanced generation of oxygen related positively charged donors after charged hadron irradiation partly compensating the negative space charge. However, another possibility would be a suppressed generation of an oxygen related negatively charged acceptor. This possibility was recently discussed and modeled under the assumption that the V<sub>2</sub>O defect is an acceptor responsible for a part of the radiation induced negative space charge [27, 28].

### 4. Conclusion

Systematic isothermal annealing studies on the radiation induced increase of the leakage current and defect formation measured by the DLTS technique have been presented for detectors fabricated from silicon materials with different impurity and doping concentrations after exposures to neutral and charged hadrons. It has been proven that the current related damage rate  $\alpha$  and the introduction rate of the electron trap E4 which is usually attributed to the VV<sup>(-/0)</sup> defect does not depend on the material properties. We have also demonstrated in an experiment with a CZ silicon sample that both quantities scale properly with the non ionizing energy loss and that a nearly perfect correlation between the short term annealing component of  $\alpha$  and the annealing of an electron trap (E4b) at  $E_C - E_t = 0.46$  eV exist. From all these results we conclude that the observed defects E4, E4a and E4b induced by neutral or charged hadrons are intrinsic defects composed of vacancies and possibly interstitials inside or close to clusters and that they are essentially responsible for the increase of the generation current.

The recently observed effect that in charged hadron fields oxygen rich silicon is more radiation hard with respect to the change of the effective doping concentration than standard material and the fact that this effect is not present in neutron damage initiated first DLTS studies on this subject. There is clear evidence that the introduction of point defects by charged particles is much larger compared to the introduction by neutrons. However, there is so far no clear understanding of the underlying defect processes which can explain the observed reduction of the change of  $N_{eff}$  in oxygen rich silicon induced by charged hadrons. In order to clarify this problem further studies on the formation and kinetics of defects are needed.

#### Acknowledgements

We gratefully appreciate the manifold assistance of R.Böttger, H.J.Brede and H.Klein in using the irradiation facility at the Physikalisch-Technische Bundesanstalt Braunschweig, V.Cindro and M.Mikuz for the exposures at the Ljubljana TRIGA reactor, G.Casse, B.Dezillie, M.Glaser, F.Lemeilleur and A.Ruzin for the great support atthe irradiation facility at CERN, K.Gabathuler and R.Horisberger for providing the pion irradiation facility at the Paul Scherrer Institut Villigen and D.Bisello and J.Wyss for the exposures at the Legnaro proton irradiation facility. Financial support is acknowledged from the CERN LHCC to the RD48 collaboration and from the BMBF to our group under contract 05 7HH17I.

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