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A contribution to the identification of the E5 defect level as tri-vacancy (V_3)

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ABSTRACT

Silicon particle detectors in tracking devices for the high luminosity Large Hadron Collider will suffer from an extremely intense radiation field of mainly hadronic particles. The main radiation induced deep defect centres in silicon, responsible for the increase of the dark current and corresponding noise, are the cluster related defect levels E5 and E205a. This work confirms the identification of the E5 level as tri-vacancy (V_3). This defect transforms into the tri-vacancy-oxygen complex (V_3O) at temperatures above 200 °C. The defect concentrations were obtained by means of Deep Level Transient Spectroscopy (DLTS) and Thermally Stimulated Current technique (TSC) performed on float zone (FZ), epitaxially grown (Epi) and Magnetic Czochralski (MCz) silicon diodes, irradiated with 1 MeV neutrons and 23 GeV protons.

1. Introduction

The Large Hadron Collider (LHC) is a proton accelerator at the European Organization for Nuclear Research (CERN). It addresses some of today's most fundamental questions of particle physics, like the existence of the Higgs boson and supersymmetry. Two large general-purpose experiments (ATLAS, CMS) are installed to detect the products of high energy proton–proton collisions with a centre of mass energy of 14 TeV and a luminosity of $L=10^{34}\,cm^{-2}\,s^{-1}$.

Silicon pixel and strip sensors are largely employed in the innermost region, the tracking area of colliding beam experiments. The proven technology and large scale availability make them the favourite choice.

Within the frame of the LHC upgrade to the high-luminosity LHC, the Luminosity will be increased by a factor of 10. In particular the pixel sensors in the innermost layers of the CMS and ATLAS silicon trackers will be exposed to an extremely intense radiation field of mainly hadronic particles with fluences of up to $\Phi=10^{16}\,cm^{-2}$. This will pose an unprecedented challenge to their radiation tolerance in order to guarantee the required lifetime of the experiments.

The radiation induced bulk damage in silicon sensors will lead to a severe degradation of the functionality during their operational time. In this work we focus on two acceptor levels: E4 at $E_C-0.39$ eV and E5 at $E_C-0.45$ eV, which are two different charge states of the same cluster related defect [1,2]. We reported earlier [3,4] that the E5 defect level is responsible for the bulk generation current, leading to an increase of the noise and a reduction of the signal to noise ratio in the particle detectors.

The E4 and E5 level concentrations were believed to decrease due to a dissociative process via first order kinetics even at room temperature [5,6]. However, Fleming et al. [2,7] reported that the defect concentrations in fact do not decrease. Instead the defect transforms into another atomic configuration, giving no rise to E4 or E5. Furthermore, they observed a recovery of the bistable E4/E5 levels after injecting a forward current density of $J=12.5\,A\,cm^{-2}$. The successive current injection after annealing would result in the initial concentration of the E4 and E5 defect levels. This approach allows to track the annealing kinetics at high temperatures, revealing information about the nature of the defect.

The vacancy-like character of the two defect levels has already been presented before [4] and an identification of the E5 defect as tri-vacancy (V_3) was already proposed by Markevich et al. [8]. In this work, we confirm the assumption and follow the annealing of V_3 and its transformation to the V_3O defect.

The main difficulty investigating cluster related defects is the general uncertainty concerning the structure of largely disordered regions. It is still an unsolved issue whether such defects are located inside or at the border region of a cluster. Furthermore, it is possible that multiple vacancies or interstitials are the origin of cluster related defects, but the available measurement methods only resolve certain defect components.

2. Materials and experimental techniques

Measurements presented here were performed on planar pad diodes. The pad with an area of $5\times5\,mm^2$ is surrounded by a guard ring structure, offering a well defined active sensor volume. The sensor thicknesses vary for the different n-type silicon materials used in this study.

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Epitaxial layers (EPI) with a resistivity of 150 Ω cm and a thickness of 74 μm were grown on 300 μm thick and highly Sb doped Czochralski (Cz) substrates. Magnetic Cz (MCz) devices of 300 μm thickness and a resistivity of 870 Ω cm have a similar oxygen concentration like the oxygen enriched EPI material (EPI-DO) of $[O_i] \approx 5 \times 10^{17}$ cm^{-3}, determined by *Secondary Ion Mass Spectroscopy*. In contrast to that, the float zone (FZ) material used in this study has a resistivity of 300 Ω cm and an oxygen content of $[O_i] \approx 9 \times 10^{15}$ cm^{-3} that is low compared to the other materials.

The neutron irradiation was carried out at the TRIGA reactor of the Jožef Stefans Institute Ljubljana in Slovenia [9], while 24 GeV/c proton irradiations were provided by the Proton Synchrotron (PS) at CERN [10]. In order to compare the damage caused by the different particles it is common to use the Non-Ionising Energy Loss (NIEL) scaling approach, normalising the damage inflicted by a particle to that of a 1 MeV neutron, resulting in an equivalent fluence Φ_{eq} [11].

In this work the detection and analysis of electrically active defects were performed by either DLTS [12] or TSC [13]. DLTS can be applied on sensors with defect concentrations of $N_t \ll N_D$, where N_t is the defect concentration and N_D the doping concentration. TSC on the other hand can be applied on sensors irradiated to high fluences up to $\Phi_{eq} = 10^{15}$ cm^{-2} [14].

Fig. 1 presents a DLTS spectrum recorded directly after the annealing at 240 °C (black line). The prominent defects are the vacancy–oxygen complex (VO) and an overlap of the two charge states of the di-vacancy (V_2) with the di-vacancy–oxygen complex (V_2O). As mentioned earlier, the defect levels E4 and E5 exhibit a bistability. Directly after the annealing process the other configuration, the shallow E75 level at $E_C - 0.075$ eV can be observed [8]. It is possible to recover the E4/E5 configuration in our samples by injecting a forward current density of $J = 4$ A cm^{-2} ($I = 1$ A) (compare [2,7]) for 20 min. The recovered defect concentrations due to this treatment are illustrated by red squares in Fig. 1. The E4/E5 defect levels are unfortunately overlapping with the di-vacancy ($V_2^{-/0}$), hence the evaluation of the defect concentrations for single defects is not trivial. At annealing temperatures below 200 °C even a further deep acceptor level (E205a, acting as generation/recombination centre, not presented here) [3] overlaps with both defects, as well.

As a result of the overlap of E4/E5 with $V_2^{-/0}$, the defect concentrations of the E4 and E5 levels had to be extracted from the difference of both DLTS spectra, before and after the injection of 1 A forward current. The resulting difference spectrum is presented in Fig. 1 (blue dashed line). Based on this procedure a special treatment was adopted for all diodes. This was performed in three steps: First an isochronal annealing step at a high temperature, followed by the injection of 1 A ($J = 4$ A cm^{-2}) forward current and subsequent annealing at 80 °C for 60 min.

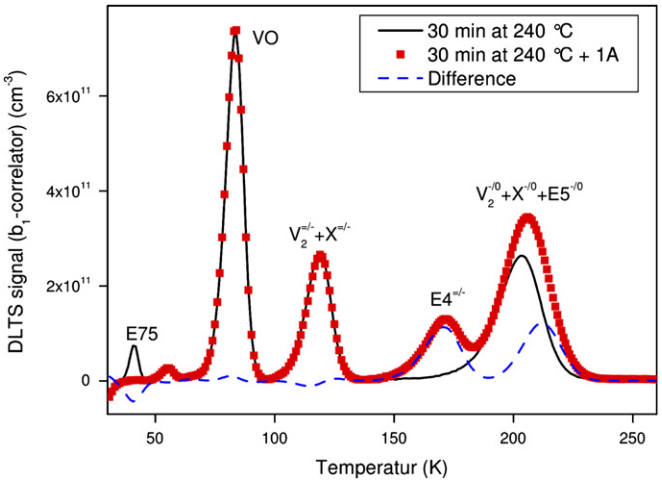

Fig. 1. Evaluation of the concentration of the E4 and E5 defects as result of the difference between the DLTS spectra at an annealing step of 240 °C and the successive injection of 20 min with 1 A. Material: FZ, $\Phi_{eq} = 6 \times 10^{11}$ p cm^{-2}. Measurement: Reverse bias voltage $V_R = -10$ V, pulse voltage $V_P = -0.1$ V, time window $t_w = 200$ ms. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Isochronal annealing studies were performed at various temperatures to gather information about the reaction kinetics of the E4 and E5 levels. After each annealing step capacitance–voltage (C–V) and current–voltage (I–V) characteristics as well as DLTS or TSC measurements were performed.

3. Experimental results

Similarity of E5 and di-vacancy: An identification of the chemical structure of defects with the DLTS technique is not possible, but DLTS indirectly provides information about a possible defect assignment. For the E5 defect level the information is mainly derived from a comparison of its annealing behaviour with that of the di-vacancy. In oxygen rich material this defect transforms at high temperatures to the X defect, which is commonly assigned to V_2O although there is still no direct proof [15].

The E5 defect transforms in oxygen rich material into a defect, labelled L defect [16] for annealing temperatures above 200 °C. The defect concentration can be extracted from DLTS spectra taken directly after the annealing. In particular this dependance of the E5→L reaction on the oxygen concentration of the studied material is the same as observed for the transition $V_2 \rightarrow X$ (V_2O), as will be shown later.

There are further similarities between the electrical properties of E4/E5 and the di-vacancy. The E5 level was proven to be the single negatively charged sate of the defect (E5$^{-/0}$) and the E4 called defect is the double negatively charged state of the defect (E4$^{=/-}$) [1,2]. Two charge states were also observed for both acceptor levels of V_2. The DLTS peak suppression of E4 compared to E5 after irradiation with neutrons is similar to the observation of the two different charge states of the V_2 [4].

Annealing behaviour: The transformation of $V_2 \rightarrow V_2O$ and the increase of the L defect concentrations can be observed unaffectedly from the annealing behaviour of the E4/E5 defect levels in DLTS spectra taken directly after annealing. Defect properties of V_2 and V_2O are well known, therefore it is possible to extract their concentrations by a two levels fit to the overlapping peaks. It should be noted that in oxygen lean materials the annealing of the V_2 defect is delayed and the transition to V_2O suppressed. This is an expected result due to the lack of reaction partners.

If E5 is indeed the V_3 defect, a transformation $V_3 \rightarrow V_3O$ should be possible. The transformation of E5→L seems to be a good candidate for this reaction. Both reactions ($V_2 \rightarrow V_2O$ and $V_3 \rightarrow V_3O$) would strongly depend on the oxygen concentration.

The observation of both transformations during annealing in oxygen rich material is presented in Fig. 2 for MCz and Fig. 3 for EPI-DO material. Defect concentrations were determined as described earlier and are presented as function of the isochronal annealing temperatures for $V_2^{=/-}$, X$^{=/-}$ ($V_2O^{=/-}$), E5 and L. There is a striking similarity between the reactions $V_2 \rightarrow V_2O$ and E5→L.

Finally, Fig. 4 represents the annealing behaviour of $V_2^{=/-}$, X$^{=/-}$ ($V_2O^{=/-}$), E5 and L in oxygen lean FZ material. Hence, it is no surprise that both the transformation of $V_2 \rightarrow V_3O$ and E4/E5→L are suppressed in this material. It was reported by Mikelsen et al. [16] that the L centre was only found in oxygen rich material. This can be confirmed by the measurements presented here. Because of all similarities and the very likely reaction E4/E5→L = $V_3 \rightarrow V_3O$ we conclude that E5 is indeed the

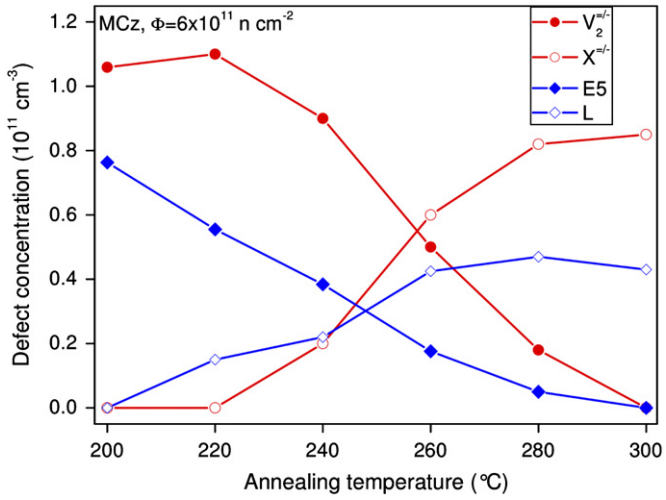

Fig. 2. Annealing behaviour of E5 compared to the V_2 defect for $\Phi_{eq} = 3 \times 10^{11}$ n cm^{-2} irradiated MCz material. Concentrations were obtained by means of DLTS.

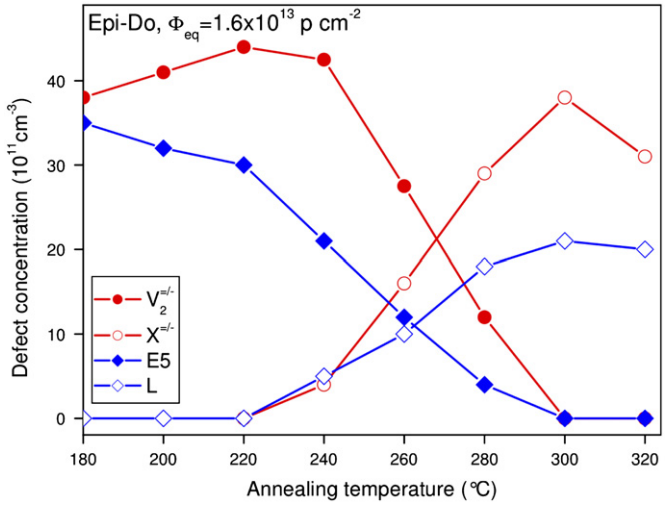

Fig. 3. Annealing behaviour of E5 compared to the V_2 defect for $\Phi_{eq} = 1.6 \times 10^{13}$ p cm^{-2} irradiated EPI-DO material. Concentrations were obtained by means of TSC.

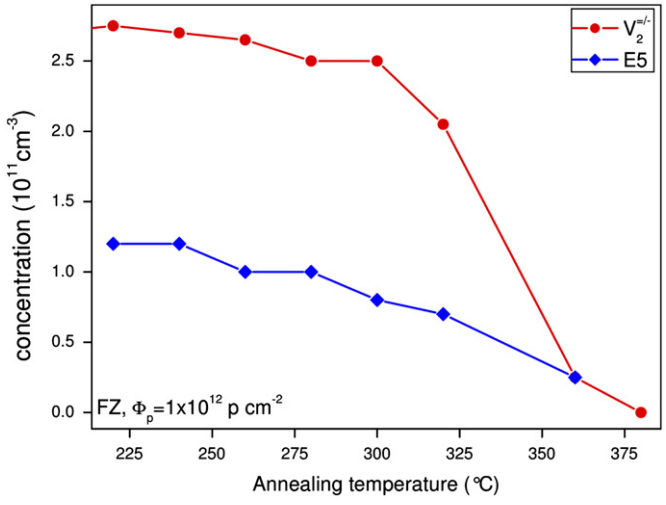

Fig. 4. Annealing behaviour of E5 compared to the V_2 defect for $\Phi = 1 \times 10^{12}$ p cm^{-2} irradiated FZ material. Concentrations were obtained by means of DLTS.

single negatively charged state of V_3 and the L centre can be attributed to the V_3O defect.

Very recently also Murin et al. correlated the increase of the V_3O concentration to the reaction $V_2 \rightarrow V_2O$ by means of IR-*spectroscopy* [17]. Their results match our findings concerning the V_3O. Unfortunately no defect concentrations for the decrease of V_3 were presented. However, as for IR-*spectroscopy* a direct correlation to the chemical structure of the defects is possible, the assignment of the L defect to V_3O is confirmed by this study.

Moreover, Markevich et al. [8] performed ab initio modelling of the atomic structure of the $V_3^{=/-}$, $V_3^{-/0}$ and V_3O defect levels. They observe a bistability for the V_3 defect, with the two orientations. The E4/E5 states can be attributed to the planar configuration, while the shallow state E75 [8], can be attributed to the four-fold configuration.

4. Summary

The two deep acceptor levels E4 at $E_C-0.39$ eV and E5 at $E_C-0.45$ eV have been proven to be the doubly and the singly charged state of the tri-vacancy (V_3). At temperatures above 200 °C and depending on the oxygen concentration of the material, the V_3 transforms into the tri-vacancy–oxygen complex (V_3O). This identification is based on the striking similarity of the reaction E5 → L to the known transformation $V_2 \rightarrow V_2O$ and the significant oxygen dependence of the generation of the L defect. As a result, the L defect can be assigned to the V_3O complex.

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