NEW RESULTS FOR A NOVEL OXYGENATED SILICON MATERIAL

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Abstract

High resistivity FZ silicon diodes have been processed in order to increase their oxygen dimer (O_{2i}) concentration. DLTS measurements have been performed after proton irradiation showing that the formation of the VO centre is suppressed. The substrates had a starting resistivity of 2-4 k Ω cm, with an oxygen concentration of 10^{15} and 10^{17} cm⁻³. This new material has potential applications in very high radiation environments such as particle detectors in future particle physics experiments.

Keywords: Oxygen, Oxygen Dimer, Silicon Detector, DLTS

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1. Introduction

There has been much research in recent years to improve the radiation tolerance of silicon detectors for use in extreme radiation environments [1 - 4]. The "standard" silicon used for such detectors in particle physics experiments is typically 1-4 kocm FZ material with very low oxygen and carbon concentrations (~ 10^{15} cm⁻³). It has been found that increasing the oxygen concentration to $\sim 10^{17}$ cm⁻³ improves the radiation tolerance for detectors operating in environments dominated by charged particles. The simplest process to introduce oxygen into FZ silicon wafers, which are typically 300 µm thick, is high temperature diffusion, typically 24 hours at 1150 °C, after the growth of a thermal oxide [4]. This process does not affect key material parameters such as the resistivity. This material is referred to as "oxygenated silicon". Diodes are then processed normally on such wafers. Oxygenated silicon detectors have the following advantages; the build-up of defect related space charge due to irradiation in a charged hadron environment is a factor three less compared to standard (low oxygen) material. This is important in order to keep operating voltages reasonably low. A phenomenon known as reverse annealing, which is an increase of negative space charge in the device after irradiation, is reduced by a factor two in oxygenated silicon. Moreover, the time-constant of the annealing process is a factor two longer [4]. Reverse annealing causes the operating voltage to increase dramatically if detectors are left at room temperature over a period of months. The physical cause of this phenomenon is not fully understood, however, it has been linked to the photoluminescence W-line [5]. Recent theoretical calculations [6] indicate that the W-line may be the tri-interstitial, I₃.

An obvious reason for adding oxygen to silicon used by detectors in a radiation environment is that oxygen is a well-known vacancy getter. Defect modelling which explains all the beneficial effects described above is still incomplete. Moreover, neutron irradiation produces the same bulk effects in both standard and oxygenated devices [1]. However, a key result of modelling indicates that V_2O may play an important role in the irradiation induced space charge build-up, [1].

Recent work [7] has shown that it is possible to convert O_i to O_{2i} . There are two reasons why this is interesting for further developments of radiation tolerant detectors. Firstly, O_{2i} may be more effective than O_i at improving the radiation tolerance. For instance, V_2O_2 is electrically neutral unlike V_2O , which is thought to be an acceptor close to mid-gap [8]. Secondly, O_{2i} is thought to diffuse through silicon several orders of magnitude more rapidly than O_i . The migration energy of O_i is 2.53 eV whilst that for O_{2i} is estimated to be 0.8 eV lower - see ref. [9] and references therein. Thus a possible way to oxygenate silicon wafers in a short time is to introduce O_i into the surface of the wafer by a short high temperature diffusion, convert this O_i to O_{2i} , and then thermally diffuse the O_{2i} into the bulk of the wafer at a much reduced temperature. This would result in a shorter diffusion time and lower furnace temperature when preparing the oxygenated silicon material. A modification of this process would be to exploit the different diffusion rates of the O_i and O_{2i} to separate these species and then to remove the top part of the wafer where O_i and O_{2i} co-exist, leaving a wafer containing just O_{2i} .

2. Sample Preparation

The samples used for this experiment were all n-type, ion-implanted 0.25 cm², p⁺nn⁺ diodes processed on 300 μ m FZ material, with a bulk resistivity of 2-4 k Ω -cm. Some of the material had been oxygenated to a high level of ~10¹⁷ cm⁻³, diodes labelled 309, while some had standard low oxygen content (~10¹⁵ cm⁻³), diodes labelled 366. The starting wafers in all cases were high resistivity FZ material which is known to have a low carbon content, around 10¹⁵ cm⁻³. A set of samples of both types then underwent an additional *'timering''* process. This process involved the irradiation at elevated temperature, 350 °C, using a Cobalt-60 gamma source. The gamma's have an energy of ~ 1.2 MeV. Previously, a similar process has been tried using 2.5 MeV electrons [7]. The gamma source has the advantage of uniformly producing interstitial-vacancy pairs throughout the silicon. Moreover, divacancies (V₂) are produced a factor 50 times less than single vacancies by Colbalt-60 irradiation, [10]. The quasi-chemical reactions that are thought to lead to oxygen dimer formation are [7]:

$$V + O_i => VO_i, VO_i + O_i => VO_{2i}, I + VO_{2i} => O_{2I}$$

In addition, the main competing reactions for silicon and carbon interstitials are:

$$I + C_s => C_i, C_i + O_i => C_i O_i, C_i + C_s => C_i C_s, C_i + V O_{2i} => C_s O_{2i}$$

There are also many other reactions which may be found in ref. [7]. The amount of O_{2i} that forms is dependent on the concentrations of the various defects, the relative capture radii of the various defects, and the temperature. Results shown in Section 3 indicate that the reaction H + VO => VOH, is also seen to occur during the "dimering" process. The samples which have been "dimered" are labelled 309D and 366D for the high and low oxygen samples respectively.

3. Experimental Results

The samples prepared as described in Section 2 were studied after the dimering process and after irradiation at room temperature by 24 GeV/c protons at the CERN Proton Synchrotron. Deep level transient spectroscopy (DLTS) and bulk measurements were performed.

3.1. DLTS of samples irradiated by gammas at elevated temperature – "dimering process".

The DLTS spectra of the 309 and the 366 samples after the dimering process are shown in Figs. 1 and 2. The majority carrier (electron) trap spectrum is identical for both samples – Fig. 1. The E(170) peak in the spectrum corresponds to an energy level of E_c –0.32 eV and is most likely the VOH complex [11]. The concentration is 5.0 x 10¹¹ cm⁻³ and this is the first evidence of the presence of hydrogen in this type of material. The spectrum recorded for the minority carrier (hole) traps is shown for the 309D sample in Fig. 2. The measured peaks correspond to $C_iO_i - H(200)$ - and an unknown defect with an energy of E_r +0.20 eV – H(144). The quantitative evaluation of the peaks is difficult in the case of the minority carrier traps since the defect concentration is large relative to the doping concentration. A further peak, H(80?), is apparent in the data. It is not fully resolved due to the temperature limit of the cryostat. The energy level of this defect is very approximately estimated to be around E_V +0.1 eV.



Fig. 1. DLTS majority carrier (electron) trap spectra of the 309D and 366D samples after the *dimering* process. The E(170) peak seen in both samples is most probably the VOH complex.



Fig.2 DLTS minority carrier (hole) spectra of the 309D and 366D samples after the dimering process. The H(200) peak is most likely the C_iO_i defect.

3.2 DLTS of processed samples after 24 GeV/c proton irradiation at room temperature

Proton irradiation was performed on a 24 GeV/c beamline at the CERN Proton Synchrotron¹. The samples were exposed together with identical non-processed samples, which were used for comparison. The total dose received by the samples was 1.1×10^{11} p/cm². The samples were kept refrigerated after exposure, except for a brief room temperature period of about 12 hours when they were transported from Geneva to the UK. The DLTS spectra of the dimered samples, 309 D, 366 D and the non-processed diodes 309 and 366 are shown in Fig. 3 for the majority (electron) traps. It is interesting to note that the E(90) peak which comprises the VO_i and C_iC_s defects is almost completely suppressed in the case of the processed samples 309D and 366D whilst it is present for the non-processed 309 and 366 diodes. The E(225) peak which is associated with cluster damage and contains the divacancy in the single negative charge state, $V_2^{(-0)}$, plus other defects, has different magnitudes for the various diodes, giving an indication, for the first time, that a material process may have altered the cluster formation. Note that the magnitude of the E(225) peak is the same for the high oxygen, 309 and 309D samples, and is smallest for the low oxygen 366D sample. Table 1 summarises the various defects found by DLTS. The E(170) peak existed in the 366D and 309D samples prior to proton irradiation. The E(170) peak is truncated in Fig. 3 in order to show the E(90) and E(225) data more clearly.



Fig 3. DLTS majority carrier (electron) spectra for the 309D and 366D processed samples, after proton irradiation. The spectra are compared with identical non-processed diodes (309 and 366). The 309D and 366D samples show no significant E(90) peak. This implies that VO_i is not formed in these samples. The E(225) peak is associated with cluster damage and is different for the various samples.

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3.3 Bulk properties of samples heavily irradiated by 24 GeV/c protons at room temperature.

The space charge, N_{eff} and the leakage current have been measured up to a fluence of 4 x 10¹⁴ p/cm². Reverse annealing and charge collection efficiency data are still being analysed. The results will be published elsewhere [12].

5. Conclusion

Oxygenated and standard FZ, high resistivity, n-type diodes have been processed with ⁶⁰Co gammas at elevated temperature in order to increase the concentration of O_{2i} clusters in the bulk. The properties of these diodes were not affected by this process. After a subsequent 24 GeV/c proton irradiation at room temperature, the DLTS spectra for the processed diodes show a suppression of VO_i production. This indirectly implies that the O_i concentration in the diodes has been significantly reduced by the high temperature gamma irradiation. Defect production associated with cluster damage (divacancy and other defects) is also different in the various samples. The low oxygen FZ material which has undergone the "dimering" process shows the least cluster related defect damage. This process opens new possibilities for radiation hardening of silicon detectors by defect engineering. It offers the possibility of suppressing radiation-induced defects like V₂O and VO, which may result in an increase of the radiation hardness of silicon detectors required by future high energy physics experiments.

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Table 1 DLTS peaks seen in the various samples

H(80?)

Electron (E) or Hole (H) trap	Energy Level (eV)	Identification
E(170)	E _c - 0.32	Probably VOH [11]
H(200)	$E_{v} + 0.36$	C_iO_i (0/+) Charge state [1]
H(144)	$E_{v} + 0.2$	Unknown

Not fully resolved. Unknown.

As Processed (309D and 366D) - High temperature irradiation with 1.2 MeV gamma source

All samples after room temperature high energy proton irradiation

Approx. $E_v + 0.1$

Electron (E) or Hole (H) trap	Energy Level (eV)	Identification
E (90)	$E_{c} - 0.17$	$VO_i + C_iC_s$ [1] Note 2.
E(225)	~ E _c - 0.4	Cluster related. $V_2^{(-/0)}$ + Others.
		[1].

Note1: Hole traps could not be studied as the DLTS technique could not be used. Defect concentrations were greater than the doping density.

Note 2: E(90) peak was suppressed in the 366D and 309D samples.