

See discussions, stats, and author profiles for this publication at:
<https://www.researchgate.net/publication/232386162>

A comparative study of vacancies produced by proton implantation of silicon using positron annihilation and deep level transient spectroscopy

ARTICLE *in* NUCLEAR INSTRUMENTS AND METHODS IN PHYSICS RESEARCH SECTION B BEAM INTERACTIONS WITH MATERIALS AND ATOMS · APRIL 2001

Impact Factor: 1.12 · DOI: 10.1016/S0168-583X(00)00640-6

CITATIONS

4

READS

6

6 AUTHORS, INCLUDING:



Manon Lourenco

University of Surrey

80 PUBLICATIONS 1,076 CITATIONS

SEE PROFILE



Russell Gwilliam

University of Surrey

381 PUBLICATIONS 2,799 CITATIONS

SEE PROFILE



Peter Mascher

McMaster University

237 PUBLICATIONS 2,283 CITATIONS

SEE PROFILE



ELSEVIER

Nuclear Instruments and Methods in Physics Research B 175–177 (2001) 300–304

NIM B
Beam Interactions
with Materials & Atoms

www.elsevier.nl/locate/nimb

A comparative study of vacancies produced by proton implantation of silicon using positron annihilation and deep level transient spectroscopy

M.A. Lourenço ^{a,*}, A.P. Knights ^a, K.P. Homewood ^a, R.M. Gwilliam ^a,
P.J. Simpson ^b, P. Mascher ^c

^a School of Electronics, Computing and Mathematics, University of Surrey, Guildford, GU2 7XH, UK

^b Department of Physics and Astronomy, University of Western Ontario, London, Ont., Canada N6A 3K7

^c Department of Engineering Physics, McMaster University, 1280 Main Street West, Hamilton, Ont., Canada L8S 4L7

Abstract

Proton irradiation at an energy of 1 MeV has been used to create an approximately even distribution of simple point defects in both Cz and FZ, n-type silicon to a depth of greater than 10 μm . The implanted dose ranged from 1×10^{11} to $1 \times 10^{16} \text{ cm}^{-2}$. The vacancy component of the defect concentration has been quantitatively measured using positron annihilation and deep level transient spectroscopy. Through careful experimental design it has been possible to meaningfully compare the concentrations independently determined using the two approaches. Good agreement is found between the two techniques for the FZ material adding credence to the methods through which defect concentrations are obtained from the primary data. Somewhat less reasonable agreement is found for the Cz material for which one possible explanation is offered. © 2001 Elsevier Science B.V. All rights reserved.

1. Introduction

Understanding the structure, formation and evolution of implantation induced defects is important at both a fundamental and a technological level. For example, with the ever-decreasing size of devices the interaction of defects with intrinsic and extrinsic impurities (important in the gettering

process) and the effect of defects on the activation and redistribution of implanted dopants warrant a large effort by the research community (for example, see [1–3]).

The implantation of protons at energies in excess of a few hundred keV allows the controlled introduction of simple point defects to a depth of several microns and at an approximately uniform concentration (i.e. the large amount of damage produced by nuclear stopping at the ion end of range is several microns below the surface). This scenario represents one of the most straightforward implanted systems from which measurements of point defect concentrations can be

* Corresponding author. Tel.: +44-1483-879-860; fax: +44-1483-879-860.

E-mail address: m.lourenco@eim.surrey.ac.uk (M.A. Lourenço).

obtained. For example, deep-level transient spectroscopy (DLTS) has been applied recently to MeV He^+ [4] implanted Si, allowing the discrimination of the singly charged divacancy at $E_c - 0.41$ eV.

Although DLTS is a truly spectroscopic technique, it has some undesirable properties; namely the large effort required in sample preparation and the limitation to samples of certain doping concentrations. A far more versatile technique for the measurement of vacancy-type defects is positron annihilation spectroscopy (PAS) [5]. Measurement is not preceded by any significant sample preparation and defects may be detected in samples containing both doping types and across a wide range of doping concentrations. The application of PAS to proton irradiated silicon has been described recently by Kauppinen et al. [6]. They used not only beam based PAS but also positron lifetime spectroscopy and spreading resistance measurements to determine the profile of implanted defects, the species of the dominating defect (in this case the divacancy) and the effect of the implant on the generation of shallow donors at the end of implant range. These shallow donors appeared to be related to hydrogen-vacancy complexes. The high degree of confidence in their results was built on the use of both beam-based Doppler-broadening and positron lifetime measurements. Whereas the former provides useful information on the distribution of the defect profile (i.e. it has defect profiling capabilities), the latter has the ability to accurately determine the dominating defect type.

Despite the successful application of PAS to ion implanted systems on numerous occasions [7], the extraction of quantitative information from PAS measurements is still viewed with a degree of scepticism. By using MeV proton implanted Si we have fabricated a sample set which allows a unique (to our knowledge) comparison of PAS and DLTS. The aim of the paper is then twofold. Firstly, it is our intention to instil a greater degree of confidence in the ion beam community in the defect concentrations determined in PAS measurements, and secondly to determine the useful limits of the two techniques to implanted systems similar to those used here.

2. Experimental

2.1. Sample preparation

The starting material consisted of either FZ or Cz, n-type (phosphorous doped) silicon quoted as having resistivity of between 20 and 40 $\Omega \text{ cm}$ (1×10^{14} – $2 \times 10^{14} \text{ P cm}^{-3}$) and 1–3 $\Omega \text{ cm}$ (1.5×10^{15} – $5 \times 10^{15} \text{ P cm}^{-3}$), respectively. The 1 inch square samples were cleaved from a 4 inch diameter wafer.

Protons of energy 1 MeV were implanted at room temperature at doses ranging from 1×10^{11} to $1 \times 10^{16} \text{ cm}^{-2}$. The degree of variation in dose prevented the use of a constant dose-rate for the preparation of all of the samples. The dose-rate varied from 0.01 $\mu\text{A cm}^{-2}$ for a dose of $1 \times 10^{11} \text{ cm}^{-2}$ to 0.1 $\mu\text{A cm}^{-2}$ for a dose of $1 \times 10^{16} \text{ cm}^{-2}$. This variation most seriously effects the samples with the higher doses which require a dose-rate great enough to induce heating of the sample during implantation. Even so, with adequate heat sinking the temperature of the samples implanted to a dose of $1 \times 10^{16} \text{ cm}^{-2}$ was not greater than 70°C.

Range calculations for 1 MeV protons in Si predict an R_p of approximately 16 μm . The peak in the implanted ion profile is preceded by a level concentration of point defects extending from the surface to at least 10 μm .

2.2. Deep-level transient spectroscopy

DLTS experiments were performed in the dark using a Bio-Rad DL4600 system. Samples were mounted on a stage in a liquid nitrogen cryostat. The temperature was monitored by a platinum resistance thermometer attached directly to the stage, giving an uncertainty of $\pm 0.5 \text{ K}$ on the measured value. All DLTS spectra were taken twice (ramping the temperature up and then down) to account for any temperature lag which might have occurred. Arrhenius plots were obtained from the average values of the up and down peak positions. The DLTS biasing conditions were chosen such that under reverse bias the depletion layer extended up to 1.2 and 8 μm , for the CZ and FZ structures, respectively (the fill pulse was kept

constant, i.e. $V_F = 1$ V and $t = 30$ μ s). Prior to the DLTS measurements capacitance–voltage (CV) and current–voltage (IV) measurements were performed, and standard diode characteristics were obtained.

2.3. Positron lifetime spectroscopy

Positron lifetime spectroscopy has been described many times before [5]. It is based on the fact that a thermalized positron in a solid can be trapped at an open volume defect. This occurs due to the lack of the positive core of the atom at the vacancy, which creates an attractive potential. Once the positron is trapped at the defect its annihilation rate decreases due to the lower electron density. The resulting lifetimes and trapping rates are thus related to the size of the defects and their concentrations, respectively.

In the present study positron lifetime spectroscopy was performed using a spectrometer with a resolution of 235 ps (FWHM) and channel widths of approximately 19 ps. An encapsulated $^{22}\text{NaCl}$ source was used to generate the positrons. To account for annihilations in the Al foil and the source material itself, lifetimes of 155 and 256 ps with absolute intensities of 1.7% and 4.4% were subtracted from the spectra. The positron lifetime spectra were analyzed using PATFIT88 [8]. Several spectra were measured for each sample and $5\text{--}7 \times 10^6$ counts were taken in each spectrum to be able to resolve defect related components.

The lifetime system requires the use of unmoderated positrons with a continuous energy spectrum up to 540 keV. This results in an implantation profile extending from the surface of the sample to a depth of approximately 250 μ m and hence only 10% of the positrons interact with the defective layer created during the proton irradiation. Although this precludes the extraction of accurate defect concentrations, enough positrons are trapped in implantation induced defects to enable a determination of the positron lifetime specific to those defects (and hence the structure of the positron trapping defects can be confidently determined).

2.4. Positron annihilation spectroscopy (beam-based Doppler-broadening mode)

In contrast to the positron lifetime measurements described above, beam-based, slow positron spectroscopy utilises a monoenergetic beam of positrons which can be accelerated to energies of a few tens of keV. This in turn permits the sample under study to be profiled from the surface to a depth of a few microns resulting in depth-resolved information on the defect concentration. The application of slow positrons to the study of ion implantation defects has been comprehensively described elsewhere [5,7]. In the current study the Doppler-broadening S parameter was extracted as a function of positron energy (and therefore depth), to a maximum energy of 60 keV (approximately 12 μ m).

3. Results and discussion

The DLTS measurements indicated an electron trap, with an activation energy (ΔE) of 246 ± 10 meV, for the Cz implanted samples (the activation energy obtained for the sample implanted at $10^{11} \text{ H}^+ \text{ cm}^{-2}$ was not taken into account when calculating this average, due to the low DLTS signal/noise ratio and large errors). This level has been previously observed in n-type Cz structures and is attributed to a double charge divacancy [4]. The DLTS spectra of the FZ structures were also dominated by a majority (electron) trap, with an activation energy in the range of 300–350 meV. We believe that this trap is likely representative of the singly charged divacancy, although the trap level is less than that of 0.41 eV given in [4]. Reasons for this discrepancy are unclear and beyond the scope of this study. Trap concentrations were calculated from the peak of the DLTS spectra [9].

Positron lifetime measurements found a dominant positron trap (in those samples with defects concentrations greater than the sensitivity limit) with a specific positron lifetime of approximately 312 ± 12 ps. This value is known to be that associated with the Si divacancy [5].

Defect concentrations were calculated from the beam-based Doppler-broadening measurements

assuming a two-state trapping model, i.e. positron trapping was dominated by a single defect type for all of the implanted doses (as indicated by the positron lifetime measurements). In this case, the fraction of positrons trapped at defects (F) is given by

$$F = \nu C / (\lambda + \nu C), \quad (1)$$

where ν is the defect specific trapping rate (given as $1 \times 10^{15} \text{ s}^{-1}$ for divacancies [10]), C is the fractional defect concentration and λ is the annihilation rate of positrons in defect free silicon (well known to be $4.55 \times 10^9 \text{ s}^{-1}$ [5]). The value of F can be extracted from the primary positron data using

$$S = F(S_d) + (F - 1), \quad (2)$$

where S is the measured S parameter, and S_d is the S parameter specific to the positron trapping defect. For the divacancy this has recently been determined to be 1.052 for the beam apparatus at the University of Western Ontario.

Fig. 1 shows the defect concentrations obtained from the two techniques for the Cz and FZ material, respectively. The data overlap only for the samples implanted with $1 \times 10^{13} \text{ cm}^{-2}$. For DLTS these samples maintain the requirement that the number of free carriers are greater than the num-

ber of defects. For PAS, these samples are at the lower sensitivity limit for defect detection. In general, reasonable agreement is observed between the two techniques for the FZ material. The agreement is less satisfactory for the Cz material for which PAS appears to yield defect concentrations at a dose of 10^{13} cm^{-2} more than an order of magnitude greater than the DLTS measurements. It is possible that the defect specific positron trapping rate is under estimated by an amount great enough to account for the seemingly larger defect concentration measured by PAS. However this is only a postulate and it is unclear at the moment which technique is yielding the greatest deviation from the actual defect concentration and future measurements are planned.

One further note of interest is the increased number of vacancy-type defects observed by both PAS and DLTS in the Cz material relative to the FZ material. This would suggest that the higher levels of impurities in the Cz material help to stabilise the defects. The positron lifetime for defects in both FZ and Cz was measured at 312 ps indicating that significant positron trapping was only observed at defects with a structure close to that of the divacancy allowing us to dismiss the presence of oxygen–monovacancy complexes as the reason for the increased defect levels. We further note that no significant oxygen related signal was observed in the DLTS spectra for the Cz material (maybe somewhat surprisingly).

4. Summary and conclusions

A unique comparison of DLTS and PAS has been performed. The vacancy component of the defect concentration following MeV proton irradiation of both Cz and FZ Si has been quantitatively measured using the two techniques. Through careful experimental design it has been possible to meaningfully compare the independently determined concentrations. Good agreement is found between the two techniques for the FZ material adding credence to the methods through which defect concentrations are obtained from the primary data. Somewhat less reasonable agreement is found for the Cz material possibly due to the use

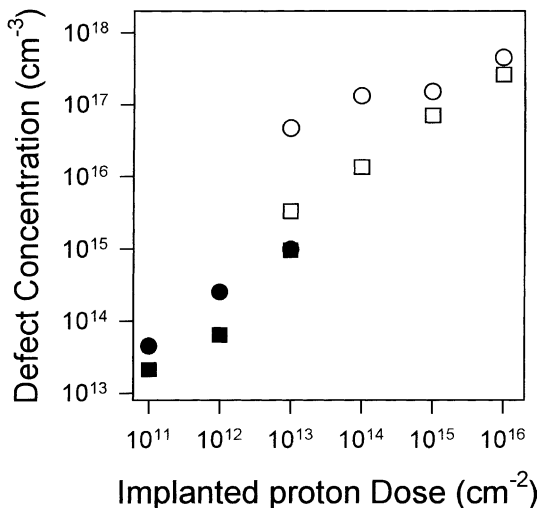


Fig. 1. Vacancy-type defect concentrations measured using DLTS (filled symbols) and PAS (open symbols) following 1 MeV proton irradiation of Cz (circles) and FZ (squares) silicon.

of an incorrect value for the defect specific positron trapping rate used in the calculation of defect concentration using the primary PAS data. Both techniques indicate a relatively larger concentration of defects in Cz compared to FZ material.

References

- [1] E. Chason, S.T. Picraux, J.M. Poate, J.O. Borland, M.I. Current, T. Diaz de la Rubia, D.J. Eaglesham, O.W. Holland, M.E. Law, C.W. Magee, J.W. Mayer, J. Melngallis, A.F. Tasch, J. Appl. Phys. 81 (1997) 6513.
- [2] P.A. Stolk, H.J. Gossmann, D.J. Eaglesham, J.M. Poate, Nucl. Instr. and Meth. B 96 (1995) 187.
- [3] V.C. Venezia, D.J. Eaglesham, T.E. Haynes, A. Argawal, D.C. Jacobson, H.-J. Gossmann, F. Baumann, Appl. Phys. Lett. 73 (1998) 2960.
- [4] S. Coffa, V. Privitera, F. Priolo, S. Libertino, G. Mannino, J. Appl. Phys. 81 (1997) 1639.
- [5] P. Asoka-Kumar, K.G. Lynn, D.O. Welch, J. Appl. Phys. 76 (1994) 4935.
- [6] H. Kauppinen, C. Corbel, K. Skog, K. Saarinen, T. Laine, P. Hautajarvi, P. Desgardin, E. Ntsoenzok, Phys. Rev. B 55 (1997) 9598.
- [7] A.P. Knights, P.G. Coleman, Defect and Diffusion Forum 183–185 (2000) 41.
- [8] P. Kirkegaard, N.J. Pedersen, M. Eldrup, PATFIT-88: A Data-Processing System for Positron Annihilation Spectra on Mainframe and Personal Computers, Risø National Laboratory, 1989.
- [9] P. Blood, J. Orton, in: The Electrical Characterisation of Semiconductors Majority Carriers and Electron States, Academic Press, London, 1992, Chapter 7.
- [10] R. Krause-Rehberg, H.S. Leipner, Appl. Phys. A. 64 (1997) 462.