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# PROPOSAL FOR FURTHER WORK ON RADIATION HARDENING OF SILICON DETECTORS

The ROSE Collaboration
(R & d On Silicon for future Experiments)

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# 1. Introduction

Silicon detectors are key components of many future experiments. In recent years there has been a large amount of work aimed at understanding radiation damage effects in high resistivity silicon detectors. CERN's RD2 and RD20 collaborations have devoted much effort to this generic R&D work. Both collaborations have submitted final reports and most of the groups have joined LHC experiments. However, there has been a substantial amount of progress in the last year, and groups working on radiation effects in RD2 and RD20 are optimistic that more radiation tolerant detectors are possible by the use of "defect engineering". There is a clear desire and need for all the groups working in this area to collaborate on a common programme. This will prevent duplication of effort, focus the activities of the groups, and lead to efficient exchange of ideas, irradiation facilities, test facilities, and samples.

Safe operation at the LHC for 10 years is the main goal of current development work. This canot be guaranteed at present for the fluence levels expected over the whole radial coverage of the experiments. Moreover, there are uncertainties which need further study which affect our ability to project our present knowledge of radiation hardness data to the LHC operational scenario. Success in these areas will also be of great benefit to experiments at HERA-B and in space projects.

A Workshop on "Radiation Hardening of Silicon Detectors" was held at CERN in October 1995. Representatives from about 30 institutions attended. In addition, solid-state physicists are now showing a keen interest in this work, and several attended the workshop. Key staff from the silicon wafer and detector industry in Europe also attended and contributed some vital background information. A summary of the Workshop is appended to this proposal (see Appendix I). It contains a good summary of work performed to the end of 1995. The most recent results were presented at the Florence Conference in March 1996. Substantial progress has been made by all groups in many areas. Transparencies from this conference are available (CERN Building 13 3-005).

Two important issues highlighted by the October 1995 Workshop were addressed at the Florence conference. These were:

- a) Most results to date have used similar silicon which in most cases came from the same source Wacker. It is vital to obtain data using material that is significantly different. First results using epitaxial material are extremely promising. Diodes made on 900 ohm.cm, 100 micron thick n-type epitaxial silicon have been irradiated with 24 GeV/c protons. As usual, the effective doping concentration falls with irradiation, but by  $10^{14}$  p cm<sup>-2</sup> the diodes have still not inverted and the depletion voltage appears to have plateaued. Further data at higher fluences will be obtained in the near future. In addition, the leakage current damage parameter is about a factor two less than in high resistivity float-zone material and no reverse annealing is observed. Epitaxial material is thought to have substantially increased oxygen and carbon levels compared to float-zone silicon due to auto-doping from the CZ substrate. Measurements are in progress to determine the impurity levels in the epitaxial silicon used to date. More diodes are to be manufactured using this material and we are collaborating with RD19 because such silicon is suitable for pixel detectors.
- b) The measured leakage current in neutron irradiated detectors is larger than expected on the basis of a Shockley-Read-Hall (SRH) generation-recombination calculation using deep level defect concentrations measured by DLTS and other techniques. New calculations indicate that inter-centre charge transfer results in enhanced leakage current compared to estimates based on standard SRH processes. Such charge transfer is possible between divacancies in terminal clusters. One consequence of this idea is that the electron occupancy of the divacancies is enhanced and they can contribute significantly to the radiation induced build-up of negative space charge in the bulk silicon. Work is in progress to correlate defect levels in irradiated diodes with leakage current data. This together with ESR techniques and further theoretical calculations will allow us to validate these ideas.

The Florence Conference demonstrated yet again that our understanding of radiation effects in silicon detectors is improving rapidly and that real progress is being made because of regular contact between the groups.

The rest of this proposal describes the scientific objectives, plan of action, schedule, milestones, and request for resources.

#### 2. Objectives

- a) To develop radiation hard silicon detectors that can operate beyond the limits of present devices and that ensure guaranteed operation for the whole lifetime of the LHC experimental programme.
- b) To make recommendations to experiments on the optimum silicon for detectors and quality control procedures required to ensure optimal radiation tolerance.

#### 3. Plan of Action

The plan of action is given in the Workshop Summary. The schedule is also given in the summary but is repeated here for clarity. We have found a company in the Czech Republic (Polovodice) which can manufacture 3 inch silicon ingots with varying oxygen and /or carbon concentrations. In addition, ingots containing other impurities (Sn, Ge, N) which may act as gettering sites for radiation induced defects will also be grown. The ingots can then be cut and polished into wafers. Such wafers are vital to the project and enable us to check and improve device models, defect kinetics simulations, material characterisation techniques, and processing methods. The work at this company will be supervised locally by Prof. B.Sopko at the Czech Technical University in Prague. The work has been organised into two phases. In the first phase, the company will produce standard and oxygenated material. If this is successful, then more exotic variants will be tried.

To prevent the project from being dependent on this one company, material from various sources will be obtained and evaluated. This includes old stock, epitaxial, and silicon from Russia. Silicon containing various impurities is also being developed through Brookhaven National Laboratory. Wacker have produced oxygenated float zone silicon. Although this is not a commercial product, efforts will be made to evaluate this material. Contacts also exist with Topsil who have provided Si(Ge) wafers in recent months.

#### 4. Timescale and Milestones

Oct. '95	Decide on material options to cover a broad range of oxygen, carbon, boron and phosphorus concentrations.
Jan. '96 onwards	Analysis of starting material.  Manufacture of test diodes on various material.
Apr. '96	Assessment of devices before and after irradiation. Microscopic and macroscopic evaluation.
Aug/Oct. '96	First results on macroscopic parameters of the test devices.  MILESTONE 1: Workshop on Defect Engineering and Radiation Hardening. Comparison of data with models.
Dec. '96	Decision on "best choice" of material. Obtain material.
Jan. '97 onwards	Analysis of starting material.  Manufacture of test diodes on "best choice" material.
Apr. '97	Assessment of devices before and after irradiation. Microscopic and macroscopic evaluation.
Aug/Oct. '97	Results on macroscopic parameters of the test devices.  MILESTONE 2: Workshop on Defect Engineering and Radiation Hardening.
Dec. '97	MILESTONE 3: Report providing recommendations on the silicon to be used for LHC detectors, including quality control procedures to be used during production. Work required for LHC experiments Technical Design Reports.

Although the project will not be considered by the LHCC until May 1996, work has started using "seedcorn" money in order to keep to the tight timescales required by the LHC experiments. Most material required for the first phase (see section 3) has been delivered to CERN. The project and phase 2 work at Polovodice cannot proceed beyond May without the resources detailed in Section 5.

#### 5. Request for resources

This is a two year project. The costs for each year are as follows:

	Cost (SFr)
"Pure" ingot with standard oxygen and carbon levels	1'000
7 ingots introducing various impurity atoms (O, C, Sn, N, Ge) (7 X 3000 SF)	21'000
Cut and polishing of wafers (800 wafers)	8'000
Processing	15'000
Impurity analysis of wafers	15'000
Travel and Meetings	30'000
Cost of irradiation facilities (see note)	10'000
Total for Year 1:	100'000 SFr

The second year of the project will have a similar breakdown.

Decisions on the material to order for the second year of the programme will be made at the end of 1996.

Total for Year 2: <u>100'000.-- Sfr</u>

Total cost of project: 200'000.-- SFr

Note: Most of the irradiations will use facilities that participating institutions can either resource from their own budgets or which are available at no cost. Facilities at CERN will also be used. However, it is clear that the project will have to pay for radiation facilities in order to keep to schedule. We estimate that access to suitable radiation facilities at short notice is about 1'200 SFr/day. As a result we have requested a contribution to these costs.

# 6. Industrial Collaboration and Observers

As mentioned above, silicon wafer and detector manufacturers from all over Europe attended the Workshop held at CERN in October 1995. Following this Workshop many manufacturers have indicated a willingness to contribute in some way to the project, and are keen to be kept informed of its progress. We consider it vital to maintain contacts with the semiconductor industry and will invite their representatives to all the Milestone Workshops and keep them up to date with progress.

Some institutions are keen to be kept informed of progress and have experience in radiation effects and material characterisation. However, they are not able to contribute directly to the project at present. These institutions will have "observer" status, which means that they will be invited to Milestone Workshops and will receive reports.

#### 7. Overview of the collaboration

The spreadsheet in the Table provides information about the collaborating groups. The key skills required are material characterisation, defect characterisation, detector characterisation, modelling, and access to radiation facilities. We believe that a key strength of the collaboration is that it contains physicists from the solid-state commmunity as well as detector experts.

# 8. Conclusion

In conclusion, although it may not be possible to prevent damage to silicon detectors by irradiation, recent work gives hope that process modifications might lead to harder detectors. Since oxygen and carbon are the dominant capture sites for vacancies and interstitials, these are the key ingredients to alter. Moreover, it will be essential for the LHC experiments to apply strict quality control to the detector starting material. Without such control, it would not be apparent until the detectors had operated for some time that due to variations in impurity levels in the silicon wafers, the radiation hardness had been compromised.

Table Summary of the skills of the collaborating groups

			1000	Pad Damage	Macroscopic	Detector	Special	Control
Intitute		Characterisation	Characterisation	Modeling	Parameters	Design	Skill(note 1)	Person
Univ. Bari	Italy	IR	ESR,DLTS,Cond(T),Hall		Yes			V. Augelli
Brookhaven Nat. Lab.	USA	PL	PL,I-DLTS,TSC,TCT,TchT	Yes	Yes	Yes	Processing	Z. Li
Brunel University	UK	SIMS	C-DLTS	Yes	Yes			S. Watts
Inst. N.P. Bucharest	Romania						Rad. Environment Calcs.	A. Vasilescu
Inst. Phys. Bucharest	Romania		TSC,OCS					T. Botila
Univ. of California	USA		ESR					E. Weber
Univ. of Catania	Italy		C-DLTS,Hall		Yes		30 MeV protons	R. Potenza
CERN-ECP	Switzerland	FTIR (Lausanne)			Yes			F. Lemeilleur
CERN-PPE	Switzerland				Yes	Yes		P. Weilhammer
Demokritos Inst. Nucl. Res.	Greece						Tandem accel., reactor n's	G. Fanourakis
Univ. Dortmund	Germany				Yes	Yes		R. Wunstorf
Univ of Florence	Italy		TSC,Hall,DLTS		Yes			M. Bruzzi
Hamburg Univ.	Germany		C-DLTS,I-DLTS,TSC,TCT,TchT	Yes	Yes	Yes	Processing	G. Lindstroem
Imperial College, London	UK			Yes	Yes	Yes		G. Hall
INFN PS	Italy				Yes	Yes		G. Tonelli
King's College, London	UK	PL, IR	PL.	Yes				G. Davies
Inst. Nucl. Research, Kiev	Ukraine				Yes			P. Litovchenko
Inst. App. Phys. Minsk	Belarus		DLTS	Yes	Yes			P. Kuchinski
Univ. Modena	Italy	Neutron Act. Analysis						G. Ottaviani
MPI. Semiconductor Lab.	Germany			Yes				G. Lutz
Univ. New Mexico	USA				Yes		Pion Irradiations	J. Matthews
Univ. Padova	Italy				Yes	Yes		D. Bisello
Czech Tech. Univ. Prague	Czech Rep.	IR						B. Sopko/S. Pospisil
PSI, Villigen	Switzerland					Yes		R. Horisberger
Ioffe PhysTech. Inst.	Russia		C-DLTS,TCT,TchT,EPR		Yes			V. Eremin
INFN, Perugia	Italy			Yes	Yes		Access to n-irradiation	G. Bilei
OBSERVERS:								
ESTEC	Holland						Rad. Effects/QCA	B. Johlander
IMEC	Belgium	Extensive Facilities	Extensive Facilities	Yes	Yes	Yes		C. Claeys
Univ. of Lancaster	UK	PL, IR?	PL,PICTS,I-alpha-DLTS	Yes	Yes			A. Chilingarov
LEPES, Grenoble	France	SIMS,IR,FTIR	ESR,PL,TSC					JC. Bruyere/B. Dezillie
MPI, Semiconductor Lab.	Germany			Yes	Yes	Yes	Processing	G. Lutz

Note 1: Special skill of group, if not mentioned already under other categories

#### Appendix I

#### **WORKSHOP ON RADIATION HARDENING OF SILICON DETECTORS CERN 3-4 OCTOBER 1995**

#### **SUMMARY**

#### 1. Introduction

The aim of the meeting was to:

- a) inform silicon wafer and silicon detector manufacturers about requirements for future experiments at CERN.
- b) provide an overview of recent radiation damage work on silicon detectors,
- c) describe R&D plans for defect engineering more radiation tolerant silicon.

Manufacturers and solid state physicists were invited to the meeting (Appendix A). because their expertise was needed to draw up a sensible plan of action.

Recent work has pointed to the importance of carbon and oxygen levels on the radiation tolerance of silicon detectors. In addition, the use of Si-Ge and high-resistivity epitaxial material has also been added to the growing list of material options. There is much optimism that better material for detectors is possible. It is clear that quality assurance of the starting material will be necessary for the detectors manufactured for LHC experiments if we are to ensure that we get the required radiation tolerance.

The output of this meeting was an agreed plan of action for the next two years. Surface and interface effects due to ionising radiation were briefly discussed. See Section 6 for more details.

This summary brings together important information and conclusions from the various talks (Appendix B). For full details one should refer to the transparencies of the meeting [1].

#### 2. Background

Silicon detectors will be widely used in experiments at the CERN Large Hadron Collider where high radiation levels will cause significant bulk damage. In addition to increased leakage current and charge collection losses which worsen the signal to noise, changes in the effective doping concentration have been observed which are not fully understood and which represent the limiting factor to long term operation. Radiation levels are expected to be around 1 Mrad/year plus a hadron dose equivalent to 10<sup>13</sup> one MeV neutrons cm<sup>-2</sup> per year. Radiation levels for vertex detector systems are at least a factor three worse. Silicon detectors at HERA-B also face such problems. About 150 m<sup>2</sup> of silicon detectors will be used in the LHC experiments - or about 100,000 four inch wafers. This is a large amount of silicon for the HEP community, but is only a small fraction of the silicon produced by wafer manufacturers for the semiconductor industry worldwide.

#### 3. Overview of radiation damage - macroscopic behaviour

# 3.1 Experimental Observations

The principal observations are:

a) high resistivity n-type detectors apparently invert to p-type after a fluence of around  $10^{13}$  cm<sup>-2</sup> fast neutrons, high energy protons or pions. The effective doping concentration,  $N_{eff}$ , is inferred from the voltage required to obtain full depletion,  $V_{FD}$ . Thus,

 $V_{FD} = (Detector Thickness)^2 e N_{eff} / (2\varepsilon).$ 

In an n-type diode, the depletion region grows from the  $n^+$  rather than  $p^+$  contact after type inversion. However, in equilibrium, both n-type and p-type wafers become almost intrinsic after around  $10^{13}$  cm<sup>-2</sup> fast

neutrons. Moreover, p<sup>+</sup> strips on an n-type substrate do not short out after type-inversion. This apparently conflicting information, can be explained by the "deep acceptor" model described in the Section 3.3.2.

For diodes which have been rapidly irradiated (1 day) by 24 GeV/c protons, so that long term annealing is avoided, the inversion fluence ,  $\phi$ INV is  $18*N_{eff,0}$ . This is for diodes with starting effective doping densities,  $N_{eff,0}$ , of up to 3  $10^{12}$  cm<sup>-3</sup>. There is evidence that diodes with a high level of boron compensation, but the same initial resistivity, have a lower inversion fluence. The constant term has been found to be different by other groups using various radiation sources and material. It is also sensitive to annealing effects. As a rule of thumb, one can use  $10*N_{eff,0}$  for the inversion fluence.

- b) after irradiation at room temperature, the effective doping density, N<sub>eff</sub>, initially decreases for some days, then gradually increases over the next few months. This "reverse annealing" can be slowed down considerably if the detector is cooled below about 5°C.
- c) Leakage current for detectors is defined in terms of the parameter  $\alpha$ . This is the leakage current increase per unit volume per incident particle fluence:  $\Delta I/V$  olume =  $\alpha \varphi$ .  $\alpha$  is around (4 to 9)  $10^{-17}$  A cm<sup>-1</sup> immediately after irradiation. Leakage currents anneal over several weeks. At room temperature, most of the annealing occurs over about 10 days and results in approximately a 50% reduction of the leakage current.
- d) Loss of signal induced by a m.i.p. due to charge trapping depends on the applied voltage and shaping time constant. However, surprisingly enough, all measurements are consistent with a degradation of around 10% at room temperature after 1 MeV neutron equivalent fluence of 10<sup>14</sup> cm<sup>-2</sup>. Moreover, this value is independent of whether this equivalent fluence is delivered by neutrons, protons or pions. The data indicates a linear dependence with fluence. However, recent data from PSI shows that detectors are still producing signals even up to 1 MeV neutron equivalent fluence of 10<sup>15</sup> cm<sup>-2</sup>.

#### 3.2. Parametrisation of radiation effects

A huge amount of data has been parametrised and fitted by the RD2 collaboration, ref. [2]. These fits not only allow predictions to be made for detector systems but also provide important clues to the underlying physical causes of the radiation effects. From these fits one can make the following conclusions:

- a) Bulk damage constants once normalised to a 1 MeV neutron equivalent fluence using Non Ionising Energy Loss (NIEL), are similar for neutrons, protons and pions. A small discrepancy 25% may exist for protons.
- b) The temperature dependence of  $\alpha$  is given by an  $E_g$  =1.24  $\pm$  0.06 eV. This is consistent with an  $n_i$  dependence see later.
- c) The activation energy for annealing of the leakage current is  $1.09 \pm 0.14$  eV.
- d) The activation energy for reverse annealing is  $1.31 \pm 0.04$  eV. Reverse annealing can accelerated by heating samples up to 100 °C.
- e) The amount of reverse annealing is proportional to irradiation fluence. It saturates after greater than 10 years. With the exception of two low resistivity Micron Semiconductor diodes (see below), reverse annealing is similar for all diodes tested to date.
- f) Introduction rates for stable defects,  $g_c$  (slope of  $N_{eff}$  versus fluence after inversion), and reverse annealing,  $g_y$ , are about 0.017 and 0.05 cm<sup>-1</sup> respectively.
- g) The leakage current damage constant after annealing has occured,  $\alpha$  (infinity), is about 2.5  $10^{-17}$  A cm<sup>-1</sup>.

#### 3.3. Models of bulk damage

#### 3.3.1 Leakage current

The leakage damage constant  $\alpha$ , can be written in terms of the damage constant,  $K_{\tau}$ , for the effective lifetime,  $\tau$ , in the depletion region, ref. [3]:

$$\alpha = e n_1 / 2K_T$$

where  $1/\tau = \phi/K_\tau$  at high fluences. This shows that the temperature dependence of  $\alpha$  goes as  $n_t$  (i.e.  $exp(-E_\gamma/2kT)$ ) and predicts that it should be approximately the same in any depletion region - i.e. material independent. The data confirm this model.

If one knows the density of the electrically active defects in the silicon then it is possible to calculate  $K_{\tau}$  and thus  $\alpha$ . Using a defect kinetics model to estimate defect concentrations after 1 MeV gamma irradiation, one arrives at a reasonable estimate of  $\alpha$ , [4]. In this calculation, the main contribution to the leakage current comes from the divacancy-oxygen centre,  $V_2O$ . The energy level of this defect is based upon a single experiment, ref. [5], performed many years ago. The same calculation for 1 MeV neutrons is a factor 100 too low; this is an important problem which needs to be understood.

#### 3.3.2 Type inversion

Type inversion has been parameterised by hypothesising donor removal, due to the creation of vacancy-phosphorus complexes, and the generation of shallow acceptors. However, there is no direct evidence of significant phosphorus removal, and defect kinetics considerations indicate that donor removal should be suppressed because the ratio of oxygen to phosphorus is large. Deep Level Transient Spectroscopy (DLTS) measurements confirm these conclusions. Moreover, a shallow acceptor would cause the material to become p-type in equilibrium. An alternative hypothesis, [3], is that the introduction of deep acceptor levels, close to the centre of the band gap, causes n-type silicon to become effectively p-type *under bias*. A device model which includes a single acceptor in the Poisson equation has been successfully used to describe the observed evolution of N<sub>eff</sub> with neutron fluence for both p- and n-type detectors in terms of one parameter, the introduction rate of the acceptors. The charge state of a deep acceptor depends on its energy level, and also on the density of free carriers. Thus the material will behave differently in equilibrium and under bias. The model can accomodate phosphorus removal and additional acceptor states.

This "deep acceptor" model has been confirmed in many ways. Firstly, by observing the depletion behaviour of diodes under illumination; irradiated detectors behave differently to non-irradiated ones in a manner consistent with deep traps being filled by photo-generated carriers, [6]. Secondly, one expects a correlation between N<sub>eff</sub> and the leakage current, [7]; this is clearly seen (see section on annealing).

#### 4. Overview of radiation damage - microscopic behaviour

# 4.1 Defect kinetics modelling

Further understanding can be achieved by numerical calculations of the evolution of complex defects formed during irradiation. The elementary defects produced are vacancies (V), interstitials (I) and divacancies ( $V_2$ ). Divacancies are static until about 600 K whereas vacancies and interstitials are mobile except at very low temperatures. Those escaping initial recombination diffuse through the crystal reacting with other defects and impurity atoms, particularly oxygen and carbon. Reaction rates are controlled by the concentration of impurities and defects and their relative capture radii. Davies et al. [8] have explained infra-red absorption spectra of electron irradiated silicon by means of a small number of reactions. A kinetics model, based on this work and suitably extended, has been used to predict the evolution of defects during neutron irradiation [3,9]. The main reactions, which include those of interstitial and substitutional carbon ( $C_i$  and  $C_s$ ) are listed below:

I Reactions	V reactions	C <sub>i</sub> Reactions
		·
Group A		
$I + C_s \rightarrow C_i$	$V + O \rightarrow VO$	$C_i + C_s \rightarrow CC$
$I + V_2 \rightarrow V$	$V + P \rightarrow VP$	$C_i + O \rightarrow CO$
$I + VP \rightarrow P$	$V + VO \rightarrow V_2O$	$CO + I \rightarrow COI *$
$I + V_3O \rightarrow V_2O$	$V + V_2O \rightarrow V_3O$	$CC + I \rightarrow CCI *$
		*Not thought to be electrically active
Group B		
$I + V \rightarrow Si$ (annihilation)	$V + V \rightarrow V_2$	

Group A reactions are for vacancies and interstitials diffusing throughout the crystal. The Group B reactions only have a significant chance of occurring during a Primary Knock-on Atom (PKA) cascade. The densities of primary defects in the small volume of the displacement damage region are large compared to impurity atom concentrations in the high resistivity silicon. For this reason the relative introduction rate of  $V_2$  in neutron irradiation is greatly in excess of that in electron irradiation.

For quantitative calculations the oxygen, carbon and phosphorus concentrations ([O], [C], and [P]) and introduction rates of I, V and  $V_2$  are required. Introduction rates of I, V and  $V_2$  due to neutron irradiation calculated from DLTS measurements are 11.5, 2.1 and 4.7 cm<sup>-1</sup> respectively. These compare with values of 0.167, 0.133 and 0.017 cm<sup>-1</sup> observed in 2 MeV electron irradiation [10]. Typical uncertainties are  $\pm 10\%$ . Ratios of capture radii are reasonably well known, e.g. Davies et al. [8]. Neutron irradiation introduction rates vary by factors of 5 between various groups (see Section 6.0).

Because of its concentration and capture radius, oxygen is the dominant capture site for vacancies; carbon behaves similarly as a sink for interstitials. The effectiveness of vacancy capture at oxygen and related defects prevents significant VP production, despite the relatively large capture radius of phosphorus.

This modelling indicates possible candidates for the deep level acceptor(s) responsible for type inversion. The strongest candidate is the  $V_2O$  centre.

Apart from the photo-EPR experiment, ref. [5] mentioned earlier, there is no direct evidence for this centre.

If the defect responsible for type inversion is impurity related, then a necessary condition would be the observation of such behaviour in a diode subjected to gamma irradiation. The deep acceptor model shows that  $N_{eff}$  is proportional to  $(N_{deep}/\tau)^{0.5}$ . Introduction rates are a factor 1000 times lower than for neutrons. Moreover, the effective lifetime degrades a factor  $10^6$  less at the same incident fluence. Consequently there should be a factor 3  $10^4$  difference between the gamma and neutron fluence necessary for type inversion; a dose of 150 Mrad would be required. It has been confirmed experimentally that type inversion does occur in gamma irradiated detectors, ref. [6]. No reverse annealing is observed.

#### 4.2 Impurities in float zone silicon

Phosphorus concentrations are usually close to  $N_{\rm eff}$ , typically  $10^{12}~{\rm cm}^{-3}$  for standard n-type detectors. This is because Wacker material is usually only lightly compensated. The best method to determine the level of compensation is photoluminescence. Oxygen and carbon levels can be inferred from DLTS measurements after gamma irradiation. This method can be used on processed diodes and is non-destructive. Typical oxygen and carbon concentrations are  $\sim 5 \times 10^{15}~{\rm cm}^{-3}$ . Infra-red absorption can also be used to obtain these concentrations. Boron concentrations in n-type material are never more than a few  $10^{11}~{\rm cm}^{-3}$ ; too low to explain reverse annealing effects in heavily irradiated detectors.

#### 4.3 Defect characterisation of irradiated detectors

A large amount of data has been accumulated over the last two years. Defect characterisation is time consuming and fraught with technical difficulties. Nevertheless, considerable progress has been made. The groups working in this area (including non-HEP groups), and the technique that they use is given below:

Group	Methods
Hamburg	C-DLTS, I-DLTS, TSC, TCT
BNL	I-DLTS, TSC, TCT, TchT
Florence	TSC
Brunel	C-DLTS
Kings College London	PL, IR
IMEC	Many, including DLTS, PL, and noise characterisation.

#### Key:

C-DLTS: Capacitance Deep Level Transient Spectroscopy I-DLTS: Current Deep Level Transient Spectroscopy

TSC: Thermally Stimulated Current TCT: Transient Current Technique TchT: Transient Charge Technique

PL: Photoluminescence IR: Infrared absorption

EPR: Electron Paramagnetic Resonance

C-DLTS is probably the most precise technique; it gives the energy level and cross-section for both electron and hole traps. One needs this information to model the electrical behaviour of the device. However, it is only useable for neutron fluences up to few 10<sup>10</sup> neutrons cm<sup>-2</sup>. TSC can only probe to about 0.4 eV below (above) the conduction (valence) band. Moreover, TSC cannot tell whether the defect is a hole or electron trap. Only PL, IR and EPR can directly determine the chemical nature of a defect. DLTS and TSC identify the energy level of the defect - this is not always a unique characteristic. A strategy of using C-DLTS at low fluence and other techniques at high fluence is proving very fruitful. TCT and TchT are techniques that study time resolved current/charge transients induced by laser pulses. These techniques are very useful for determining the electric field throughout the material, estimating N<sub>eff</sub> and the Fermi level, and for studying the carrier mobility. The following conclusions can be made from the measurements made by these groups:

- a) It is vital to have a defect kinetics model to provide a framework in which the measurements can be interpreted.
- b) There is good agreement between the various groups concerning the defects produced by radiation. Electron traps seen include VO, CC, and  $V_2$ . Hole traps seen include  $C_i$  and CO. Some unknown defect is found at  $E_c$ -0.4 eV, which is only caused by neutron irradiation. This defect slowly anneals at room temperature.
- c) TSC data from Florence shows shallow acceptor and donor levels in the material before irradiation. Similar concentrations of these levels are seen even after high neutron fluences.
- d) Carbon reactions occur with a timescale of about 14 days at room temperature. The activation energy for the carbon interstitial reaction is  $1.0 \pm 0.1$  eV. This behaviour is remarkably similar to the annealing time and activation energy for the leakage current. It is highly probable that the carbon interstitial is responsible for the short term annealing of the leakage current. Additional evidence for this hypothesis has been gathered by investigating the annealing behaviour of gamma irradiated diodes. After gamma irradiation, the only defect that is known to be mobile is the carbon interstitial. Annealing of leakage current after gamma irradiation is also consistent with movement of the carbon interstitial. However, the carbon interstitial has an energy level which is too shallow in the bandgap to cause significant leakage current.

Either it has another energy state, or due to some unknown mechanism, it acts as an efficient recombination centre.

- e) The interstitial introduction rate should equal the vacancy introduction rate plus twice the divacancy rate. Using the VO and CC/CO defects to measure the vacancy and interstitial introduction rates respectively, this consistency check has been found to be good to 10% for neutron and gamma irradiated samples.
- f) In neutron irradiated diodes, there is evidence of vacancy release, presumably from defect clusters, at a temperature of 360 K. Neff measurements on irradiated diodes show accelerated reverse annealing at this temperature. There is no evidence of interstitial release, although further measurements are required.
- g) Gamma irradiation is useful in two ways. Firstly, the defect kinetics is simpler than for neutron irradiation. As a result, it is easier to check both defect kinetics simulations, and device models against measurements. Secondly, such irradiation together with defect characteristion, can be used to extract information about impurity levels in processed diodes.
- h) Both the BNL and Florence groups see a defect level growing at about 0.4 eV with a similar time constant to reverse annealing. Because TSC cannot identify in which half of the bandgap the defect resides, its identification is not clear. Moreover, unless it is an acceptor in the bottom half of the bandgap (unlikely), then it cannot on its own explain the doping changes. However, it is an important clue to the nature of reverse annealing and more work is required. It is very unlikely to be related to the carbon interstitial because these react with carbon and oxygen in the first two weeks after irradiation. There are several vacancy-oxygen related defects with such an energy level. These have only been studied by just one group using EPR and photo-EPR [11, 5]. This is an experiment that should be performed on detector silicon.

#### 4.4 Reverse annealing behaviour

To complete the picture any model should also explain the annealing behaviour of the detectors. The deep acceptor model provides the basis for such an understanding. For detectors irradiated past type inversion, a simple expression between the deep acceptor concentration and effective carrier lifetime in the depletion region has been analytically derived. The same lifetime controls the detector leakage current. As a result one can write that,

$$N_{eff} / \phi = constant x ((N_{deep} / \phi) x (\alpha / n_i))^{1/2}$$

where  $N_{\text{Deep}}$  is the concentration of deep acceptors,  $\phi$  is the fluence,  $\alpha$  is the leakage current damage constant and  $n_i$  is the intrinsic carrier concentration. Only a small fraction of the deep acceptors are filled. Higher leakage currents result in more acceptors being filled which increases  $N_{\text{eff}}$ . The RD2 collaboration have carefully measured both  $N_{\text{eff}}$  and  $\alpha$  as a function of time from 1 hour to 200 days for detectors at temperatures of 20°, 10°, 0° and -20 °C. The initial annealing of  $N_{\text{eff}}$  is proportional to  $\alpha^{0.5}$ . In other words, in the early phase after irradiation, the density of deep acceptors is approximately constant but fewer remain filled as the leakage current reduces during annealing.

 $N_{eff}$  starts to exhibit reverse annealing (become more p-type) at room temperature once the leakage current reduction has ceased. This implies that  $N_{Deep}$  is increasing. What is the microscopic cause of this behaviour? Obviously something has to "move" in the silicon. Carbon and boron interstitials are ruled out - section 4.3 and 4.2 respectively. The most likely reason, which is supported by measurements, ref. [12], is release of vacancies from defect clusters. This would imply that gamma irradiated detectors would not show reverse annealing behaviour as has been confirmed by experiment. This new source of vacancies then generates more deep acceptors which causes  $N_{eff}$  to increase. This successfully explains the observed features of reverse annealing, namely the strong temperature dependence and eventual saturation of the effective doping concentration.

One can speculate that the identification of the  $V_2O$  centre as a possible candidate for the deep acceptor is also consistent with this picture; the "delayed" vacancies react with VO centres to form more  $V_2O$  defects.

TSC data, Section 4.3, does show a defect concentration increasing with a time constant similar to that for reverse annealing. However, the identity of the centre has still to be determined. A considerable amount of

further work is needed to confirm the role of vacancy-oxygen complexes and to clearly identify the defect(s) causing reverse annealing.

# 4.5 Radiation hardening - predictions from defect kinetics considerations.

If the current models are correct, then the key ingredients to change are oxygen and carbon. If the carbon concentration was lowered, then there would be more interstitials available to suppress multi-vacancy complexes. More detailed modelling also suggests that higher carbon concentrations would be beneficial.

A higher oxygen concentration would suppress phosphorus removal even further, and encourage VO production at the expense of  $V_2O$ . Great care should be taken: the authors of ref. [11] suggest that their data may be evidence for the direct production of  $V_2O$  in heavily oxygenated CZ material.

There is an urgent need to obtain material with differing oxygen and carbon levels in order to verify and adjust our current defect kinetics model.

#### 5. Information from the manufacturers

Oxygen and carbon levels in FZ silicon are around 5  $10^{15}$  cm<sup>-3</sup>. The limit is set by the quality of the starting material (polysilicon), the process gas and the furnace. The industry trend is to lower levels. Wacker have produced oxygen doped FZ although this is not available as a commercial product. Topsil have produced Si-Ge (0.05% max.) FZ material. Detectors made on this material have similar bulk damage parameters to pure Si material. Work in Russia is ongoing with possibilities for higher oxygen and carbon levels: FZ ( $[O] \sim 10^{17}$  and  $[C] \sim 10^{16}$  cm<sup>-3</sup>) and photon transmutation doping of Magnetic CZ ( $[O] \sim 5 \cdot 10^{17}$  and  $[C] \sim 10^{17}$  cm<sup>-3</sup>). Charge coupled devices are made on Wacker p-type epitaxial material. Up to 100 micron thick several thousand ohm.cm material has been used. The CZ substrate is useful for gettering impurities during processing. SIMS measurements indicate that the epitaxial layer is autodoped by the CZ substrate. This leads to higher oxygen and carbon levels than in FZ material - around  $3 \cdot 10^{17}$  and  $5 \cdot 10^{16}$  cm<sup>-3</sup> respectively.

Transient enhanced diffusion (TED) is a problem that needs to be solved if device dimensions are to be made even smaller. High carbon levels are known to supress the diffusion of boron implants [13]. Thus defect physics and higher carbon levels are issues that the semiconductor industry cannot ignore.

Micron Semiconductor have supplied detectors made on old silicon stock - circa 1984. These show a factor 2 improvement in the reverse annealing behaviour. Because the time to obtain material can be around one year, it would be useful for detector manufacturers to look for old stock.

All the companies emphasied the commercial considerations which motivate their developments. High energy physics detectors are a very small part of the silicon market. This market is very buoyant and there is little hope of changing their manufacturing processes unless the changes coincide with their own requirements. However, at least one major company showed some willingness to provide samples of material with differing oxygen and carbon concentrations. In addition, they confirmed that the volumes of high resistivity material required for the LHC experiments should be available on the necessary timescale. Users should be warned that orders need to be placed at least one year in advance.

# 6. Other Issues

- a) Intercalibration of various irradiation sources is being studied in Bucharest. Hardness factors are sensitive to the low energy cut-off.
- b) There is disagreement between introduction rates estimated using different defect characterisation equipment. There is a clear need to find a defined "standard" and also to "cross-calibrate" by exchanging samples between the groups.

- c) Surface and interface effects have been studied by many groups and are reasonably well understood. They do not represent a serious threat to operation provided detectors are carefully designed. The design of detectors will be dependent on decisions specific to a given experiment. Moreover, surface and interface effects are closely related to the process technology. For these reasons, there was no enthusiasm for joint R&D.
- d) Reliability is an area which has yet to be fully investigated.

#### 7. Plan of action

The strategy for defect engineering work is shown in Fig.1. Material with differing carbon and oxygen levels is used to manufacture test diodes. Impurity concentrations for each material is checked before and after processing using photoluminescence, infra-red absorption and DLTS. It is important to determine the degree of compensation of the starting material. The diodes are then irradiated with gammas and neutrons. Both macroscopic and microscopic measurements on these irradiated diodes are then compared with defect kinetics simulations and device models.

Material with different impurity levels to the silicon used for current detectors is required in order to tune up our present kinetics model.

Readily available material which is likely to be different includes epitaxial, old stock, and silicon from Russia. Once a "best option" has been identified, approaches can be made to obtain such material.

#### 7.1 Timescales and Milestones

Oct. '95	Decide on material options to cover a broad range of oxygen, carbon, boron and phosphorus concentrations.
Jan. '96	Analysis of starting material.
onwards	Manufacture of test diodes on various material.
Apr. '96	Assessment of devices before and after irradiation.
	Microscopic and macroscopic evaluation.
Aug Oct. '96	First results on macroscopic parameters of the test devices.  MILESTONE 1: Workshop on Defect Engineering and Radiation Hardening.  Comparison of data with models.
Dec. '96	Decision on "best choice" of material. Obtain material.
Jan. '97	Analysis of starting material.
onwards	Manufacture of test diodes on "best choice" material.
Apr. '97	Assessment of devices before and after irradiation.
•	Microscopic and macroscopic evaluation.
Aug Oct. '97	Results on macroscopic parameters of the test devices.  MILESTONE 2: Workshop on Defect Engineering and Radiation Hardening.
Dec. '97	MILESTONE 3: Report providing recommendations on the silicon to be used for LHC detectors, including quality control procedures to be used during production. Work required or LHC experiments.  Technical Design Reports.

#### 7.2 Organisation

All the groups attending the workshop showed enthusiasm for joint work on defect engineering of silicon. It was agreed that the workshop organisers would summarise the meeting and investigate ways of obtaining different material. Bringing groups together from both ATLAS and CMS had clearly been of great benefit. Joint meetings with a 3 to 6 month interval would benefit everyone. The interest and collaboration with solid-state physicists is an important development. The project would benefit if it were supported by the LHCC. The organisers will approach the relevant CERN committee to discuss such support.

#### 8. Conclusion

In conclusion, although it may not be possible to prevent damage to silicon detectors by irradiation, recent work gives hope that process modifications might lead to harder detectors. Clearly it is desirable to prevent formation of the  $V_2O$  defect. Since oxygen and carbon are the dominant capture sites for vacancies and interstitials, these are the key ingredients to alter. Moreover, it will be essential for the LHC experiments to apply strict quality control to the detector starting material. Without such control, it would not be apparent until the detectors had operated for some time that due to variations in impurity levels in the silicon wafers, the radiation hardness had been compromised.

# Acknowledgements

The organisers are grateful to the representatives from industry for attending the Workshop and providing valuable background information. The contribution of physicists from outside high energy physics is also much appreciated. Financial help from ECP Division is acknowledged with thanks.

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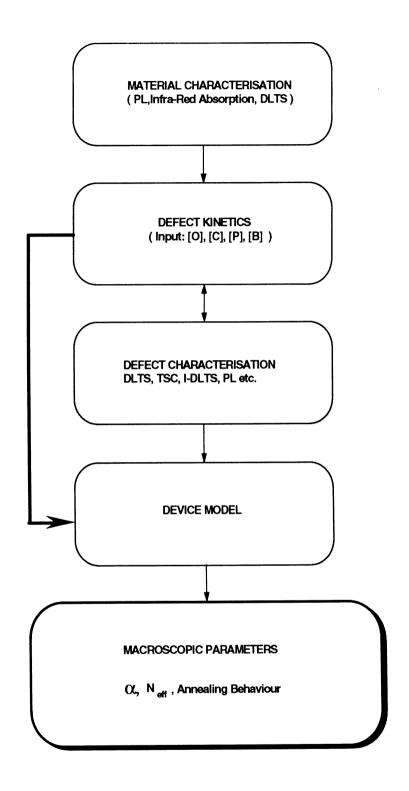


Figure 1: Defect Engineering Strategy

# APPENDIX A

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# APPENDIX B

# WORKSHOP ON RADIATION HARDENING OF SILICON DETECTORS CERN, 3-4 October 1995

# Tuesday morning 3 October 1995 Council Chamber Bat 503 (Main Building) 1st Floor

10.00	Welcome F. Lemeilleur/G. Lindstroem/S. Watts
Session 1	Chair: Gunnar Lindstroem Theme: Background information
10.05	Overview of requirements for silicon detectors at CERN Peter Weilhammer (CERN)
10.35	Overview of radiation damage - macroscopic behaviour 1 Francois Lemeilleur (CERN)
11.05	COFFEE BREAK
11.20	Overview of radiation damage - macroscopic behaviour 2  Eckhart Fretwurst (Hamburg Univ.)
11.50	Question Time
12.00	Overview of radiation damage - microscopic behaviour Steve Watts (Brunel Univ.)
12.45	Discussion
12.55	Organisational details
13.00	LUNCH
	Tuesday afternoon 3 October 1995 Council Chamber Bat 503 (Main Building) 1st Floor
Session 2	Chair: Geoff Hall Theme: Background from silicon wafer and detector manufacturers
Contributions fr	om silicon wafer manufacturers
14.00	Overview of silicon production at Siltronix B. Meier (Siltronix)
14.20	Overview of silicon production at Topsil P.E.Schmidt (Topsil)
14.40	Overview of silicon production at Wacker W. Hensel (Wacker Chemitronics Burghausen)
15.00	Overview of silicon production in Russia - 1 N. Zamiatin (JINR Dubna)
15.20	Overview of silicon production in Russia - 2 G. Bashindzaghian (Silab, MSU Moscow)

# 15.40 TEA BREAK

# Contributions from silicon detector manufacturers

16.00	P. Burger (Canberra)
16.15	A. Perret/R. Della Marina (CSEM)
16.30	P. Pool (EEV Ltd)
16.45	M. Lampert/P. Rohr (Eurisys Mesures)
17.00	C. Wilburn (Micron Semiconductor)
17.15	L. Evensen (Sintef)
17.30	R&D on process technology at MPI G. Lutz/J. Kemmer (Semicond. Lab. MPI Munich and KETEK)
17.50	Discussion/End of first day
	Wednesday morning 4 October 1995 <u>Building 13 2-005 ECP Amphitheatre</u>
Session 3	Chair: François Lemeilleur Theme: Defect characterisation and kinetics
9.00	Defect kinetics in electron irradiated silicon Gordon Davies (Kings College, London)
9.20	Defect characterisation work at IMEC Eddy Simoen (IMEC, Leuven)
9.50	Conclusions from defect kinetics modelling of irradiated detectors <i>Geoff Hall</i> (Imperial College, London)
10.10	Radiation Hardness of Raw Silicium material and Si detectors. P.Litovchenko (Kiev)
10.20	COFFEE BREAK
Session 4	Chair: Steve Watts Theme: Round Table Discussion
10.35	What material options are possible? How can we best characterise the starting material, the processed wafers, and irradiated detectors?
11.30	Plan of Action
12.30	Lunch

# Wednesday afternoon 4 October 1995 Building 304 1-001B

Session 5 Chair: Gunnar Lindstroem

Theme: Calibration and defect analysis

Most of the afternoon will be left free for discussions and private meetings, however, 2 topics need special attention:

- 1) intercomparison of results obtained with different neutron sources.
- 2) intercomparison of results from microscopic defect analysis.

# Intercomparison of different neutron sources

13:30 Comparison of various neutron sources used for irradiation studies and the NIEL problem. *A. Vasilescu* (INPE Bucharest)

# Intercomparison of microscopic defect analysis

<b>r</b>	
13:50	Survey of the Semiconductor Microscopic Defect Analysis System at BNL and Recent Results. <i>Zheng Li</i> (BNL)
14:10	Survey of the Semiconductor Microscopic Defect Analysis System at INFN Florence and Recent Results. <i>Mara Bruzzi</i> (INFN Florence)
14:30	Survey of the Semiconductor Microscopic Defect Analysis System at Hamburg University and Recent Results. <i>Eckhart Fretwurst</i> (Univ. of Hamburg)
14:50	Survey of the Semiconductor Microscopic Defect Analysis System at Brunel University and Recent Results. <i>Steve Watts</i> (Brunel University)
15:10	Discussion
15:40	TEA BREAK
16.00	Further discussions and private meetings.
18.00	END OF MEETING