

Magnetic Czochralski silicon as detector material

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The Czochralski silicon (Cz-Si) has intrinsically high oxygen concentration. Therefore Cz-Si is considered as a promising material for the tracking systems in future very high luminosity colliders. In this contribution a brief overview of the Czochralski crystal growth is given. The fabrication process issues of Cz-Si are discussed and the formation of thermal donors is especially emphasized. $N^+/p^-/p^+$ and $p^+/n^-/n^+$ detectors have been processed on magnetic Czochralski (MCz-Si) wafers. We show measurement data of AC-coupled strip detectors and single pad detectors as well as experimental results of intentional TD doping. Data of spatial homogeneity of electrical properties, full depletion voltage and leakage current, is shown and n and p-type devices are compared. Our results show that it is possible to manufacture high quality $n^+/p^-/p^+$ and $p^+/n^-/n^+$ particle detectors from high resistivity Czochralski silicon.

1. Introduction

Particle detectors made of high-resistivity silicon wafers are widely used in high-energy physics experiments. Segmented silicon detectors provide excellent spatial resolution while being cost-effective due to well-established manufacturing technology. Nevertheless, particle radiation causes irreversible crystallographic defects in silicon material deteriorating the detector performance. First, the donor doping concentration of silicon is compensated by dominantly acceptor type defects. This may cause space charge sign inversion (SCSI) in n-type silicon material, which is followed by the increase of the detector full depletion voltage (V_{fd}). Second, the leakage current of the detector (I_{leak}) increases linearly as the function of accumulated radiation fluence. Third, due to the trapping of charge carriers, overall charge collection efficiency (CCE) degrades [1]. The deterioration of the CCE due to the trapping of charge carriers will be the most severe obstacle for the use of silicon sensors in future very high luminosity colliders with extremely harsh radiation environment. Possible upgrade scenario of the LHC to a luminosity of $10^{35} \text{ cm}^{-2} \text{s}^{-1}$, corresponding to expected total fluences of fast hadrons above 10^{16} cm^{-2} and reduced bunch-crossing interval of about 10 ns [2].

The detectors used in particle tracking systems must be fully depleted at reasonably low operating voltages. A practical limit of the operating voltage is 500 V. Detectors have traditionally been fabricated on n-type high resistivity wafers made by Float Zone (Fz-Si) crystal growth technique. The low oxygen concentration in Fz-Si is a drawback since oxygen has experimentally been found to improve the radiation hardness of silicon detectors, as demonstrated by the R&D work performed in the framework of CERN RD48 collaboration [3,4].

Developments in the crystal growth technology of Czochralski silicon (Cz-Si) have enabled the production of Cz-Si wafers with sufficiently high resistivity and with well-controlled, high concentration of oxygen. In addition to the high oxygen concentration, the Cz-Si is available commercially in large quantities and many commercial detector foundries are familiar with processing of it, since the Cz-Si is basic raw material of the microelectronics industry. These aspects are important when designing a future large scale tracker system.

2. Crystal growth and material properties

In the Czochralski method, polycrystalline silicon fragments are melted inside a silica crucible. During the process, argon gas is continuously flushing the interior of the crucible and the surface of the silicon melt. Silicon single crystals are grown by slowly pulling a crystal seed up from the molten silicon, thus developing an ingot. Later, wafers are cut from the ingot.

Oxygen concentration is one of the most important parameters of silicon wafers. For example, oxygen precipitates bind unwanted metallic impurities present during the processing of silicon devices [5, 6]. Furthermore, stress induced during high temperature processing can lead to the formation of slip defects in the wafer. The presence of oxygen stabilizes the wafer and thus Cz-Si wafers are less prone to slip defect than Fz-Si wafers [7].

During the crystal growth the silica crucible gradually dissolves releasing oxygen, carbon and other impurities, namely aluminum and boron, into the silicon melt. Most of the oxygen vaporizes from the melt as silicon monoxide (SiO) that is transported away by the protection gas, but the rest stays in the melt and can dissolve into the single-crystal. The concentrations of interstitial oxygen and carbon impurities in the single crystal are typically $1\text{-}10 \times 10^{17} \text{ cm}^{-3}$ and $1\text{-}5 \times 10^{16} \text{ cm}^{-3}$, respectively. If the oxygen concentration in silicon exceeds $1 \times 10^{18} \text{ cm}^{-3}$, the oxygen starts to precipitate, i.e. form clusters.

The homogeneity of the material is strongly affected by the thermal gradients present in the melt crucible. The thermal gradients cause strong buoyancy-driven convection and surface-tension-driven flows at the free surfaces of the melt. These melt-convective flows are at least partially turbulent and they are largely responsible for the crystal imperfections and radially and microscopically non-uniform distributions of dopant and impurity atoms in the crystals [8]. In addition, the silicon melt is enriched by dopants and therefore the resistivity decreases as a function of the length of the ingot. For these reasons, the maximum resistivity of the standard Czochralski wafers is rather limited.

The magnetic Czochralski (MCZ) method is in principle the same as the standard Czochralski crystal growth method, but the ingot is grown in a strong magnetic field, which is used to dampen the oscillations in the silicon melt. That is, the Lorentz force resulting from the applied magnetic field influences the flow and reduces the amplitude of the melt fluctuations [9,10]. In this method the concentration and the distribution of the oxygen can be controlled better than in the standard CZ method, which is important for the large-area detector applications. The method is today widely used in the semiconductor industry and therefore wafers grown with magnetic Czochralski method have become available also for the detector applications.

3. Processing of MCz-Si detectors

Pad detectors and segmented AC-coupled microstrip detectors have been processed on n and p-type MCz-Si substrates at the Microelectronics Center of Helsinki University of Technology [11,12]. The wafers are 100mm diameter double-side-polished $300 \pm 2 \mu\text{m}$ -thick having $<100>$ crystal orientation. The nominal resistivity, measured by the four point probe method, of the boron-doped wafers is $1800 \Omega\text{cm}$ and $900 \Omega\text{cm}$ in phosphorous doped wafers. The oxygen concentration of these wafers was measured by the Fourier Transformation Infrared (FTIR) spectroscopy. The measurements were done on a thick reference wafer at the Institute of Electronic Materials Technology (ITME), Warszawa, Poland. The following oxygen concentrations were measured: $4.95 \times 10^{17} \text{ cm}^{-3}$ (center), $4.89 \times 10^{17} \text{ cm}^{-3}$ (right), $4.93 \times 10^{17} \text{ cm}^{-3}$ (left) and $4.93 \times 10^{17} \text{ cm}^{-3}$ (bottom).

The process sequence is basically similar when processing MCz-Si and standard Fz-Si devices: ion implantation of the segmented side and the back plane of the wafers, implantation activation thermal treatment, processing of bias resistors and strip dielectric, contact metallization, aluminum sintering and passivation. The essential difference between MCz-Si and Fz-Si is, however, the oxygen concentration. The oxygen can form at the temperatures between 400° C – 600° C complexes consisting of four or more oxygen atoms. These complexes are called thermal donors (TD) and they influence on electrical properties of detectors. When processing p⁺/n/n⁺ pin-diode detectors on n-type phosphorous-doped Cz-Si substrates, the TD formation decreases the effective bulk resistivity and consequently increases the full depletion voltage (V_{fd}). On the other hand, it is possible to tailor the V_{fd} of the n⁺/p⁻/p⁺ device made of p-type MCz-Si wafer by deliberate introduction of thermal donors TDs. The ion implantations doses in our process are all $1 \times 10^{15} \text{ cm}^{-2}$ while the implantation energies were 70 keV and 30 keV for phosphorous and boron, respectively. The thermal dry oxidations in our process were done at 1100° C. After the oxidations, the temperature was ramped down at the rate 4° C/min. The pull-out of the wafers took place at the 700° C. Thus the wafers were cooled as fast as possible in order to avoid temperature range 400-600 °C. All detectors were passivated by approximately 60 nm thick silicon nitride (Si₃N₄) film grown by Plasma Enhanced Chemical Vapor Deposition (PECVD) method. The deposition temperature of PECVD Si₃N₄ is 300° C.

Figure 1 a) and b) show the results of the capacitance-voltage (CV) and leakage current measurement of two n-type MCz-Si strip detectors.

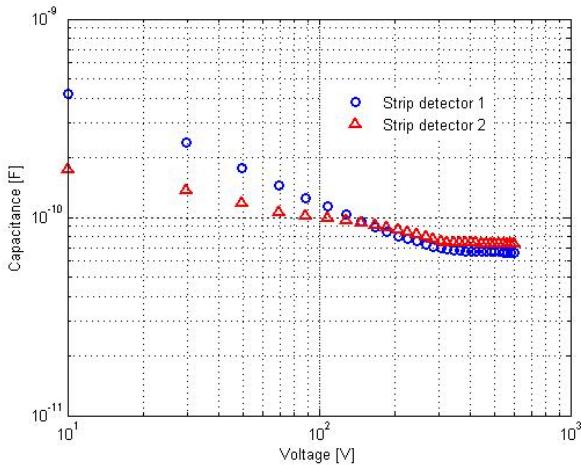


Figure 1a) CV measurement of two mini strip detectors. The area of detectors is 2.5 cm² and there are 128 1.5 cm long AC-coupled strips. The V_{fd} of detectors are 307 V (1) and 304 V (2).

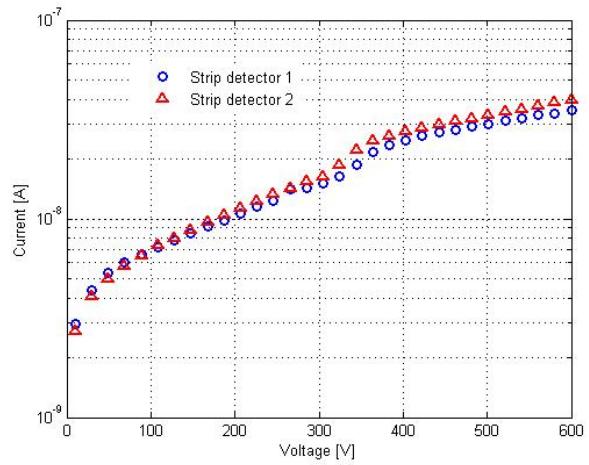


Figure 1b) IV measurements of detectors (1) and (2). The leakage current at the room temperature is about 16 nA corresponding about 6 nA/cm²

The bias resistors of detectors have been processed of sputtered tungsten nitride (WN_x). The reason to use WN_x instead of commonly adopted polysilicon resistor technology is twofold. First, WN_x process is very simple. The resistors don't need ion implantation and subsequent dopant activation in order to adjust resistance value. The resistance value is adjusted by controlling the film thickness, which easily can be done with nanometer scale accuracy. Additionally, WN_x can be wet etched at the room temperature completely selectively against underlying SiO₂ by hydrogen peroxide [11]. Second, WN_x resistor is a room temperature process containing no hydrogen ingredients like silane (Si₃H₄) used as a precursor in polysilicon process. This is important in scope of controlling the TD formation, which is known to be enhanced if hydrogen is present in the fabrication process at the

temperatures between 400° C – 600° C. For same reason, the strip insulators, 1 μm thick stack of SiO₂ and Si₃N₄, have been deposited by the PECVD at the 300° C.

The process cleanliness has been monitored on the regular basis by the minority carrier recombination lifetime measurements carried out by the microwave photoconductivity decay (μPCD) method [13,14]. The measurements show no lifetime degradation in MCz-Si with respect of standard Fz-Si experienced same process conditions and thermal history. Additionally, the results of recombination lifetime injection level dependence reported in Reference [15] give reason to assume that the lifetime in MCz-Si is not limited by the deep levels, introduced e.g. by copper or iron, but rather by relatively shallow levels.

4. P-type MCz-Si detectors

The radiation hardness of silicon particle detectors can also be significantly increased by using n⁺/p⁻/p⁺ structures instead of conventional p⁺/n⁻/n⁺ detectors [16]. The essential advantage is that no space charge sign inversion (SCSI) occurs in p-type bulk, resulting in the collective junction to remain on the segmented side of the detector. Thus, the signal comes dominantly from electrons having three times higher mobility than that of holes and, consequently, the trapping of charge carriers within their trapping lifetime (inversely proportional to the irradiation fluence) is reduced. This allows higher charge collection in n⁺/p⁻/p⁺ devices than in conventional detectors. If p-type Czochralski silicon is used as detector starting material, it is possible to engineer the full depletion voltage by compensating boron doping by thermal donors.

In order to study the thermal donor generation, five n⁺/p⁻/p⁺ pad detector wafers were sintered at 430 °C for different times, i.e. 35, 45, 60, 70 and 80 minutes, at the peak temperature [12]. The thermal profile of sintering is push-in 2 minutes followed by the 10 minutes stabilization while the furnace settles again at the peak temperature (the furnace door is open during the push-in). After heating at the peak temperature the wafers were pulled-out in 2 minutes. In order to study the spatial uniformity of V_{fd}, ten diodes along the each wafers diameter were picked for the CV measurements. The full depletion voltage of devices was extracted from the CV curve measured at the room temperature and at the 10 kHz frequency. The effect of thermal treatment on effective doping concentration N_{eff} is shown on Figure 2.

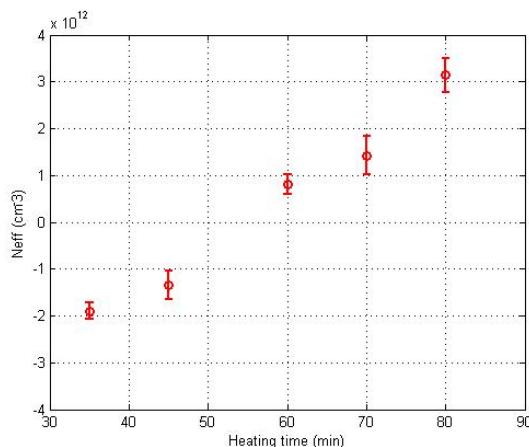


Figure 2. Effective doping concentration N_{eff} versus heating time at 430°C. The heating time data points correspond to 10 diodes picked along the wafer diameter, as shown in Fig 2. The standard deviation of N_{eff} is displayed with error bars.

Almost linear increase of N_{eff} with respect of heating time can be seen in Figure 2. The TD formation rate is discussed more in detail in References [12,17,18].

The drawback of $n^+/p^-/p^+$ devices is the more complex fabrication technology. Conventional detectors can be processed with 6-7 lithography mask levels while $n^+/p^-/p^+$ devices require two more levels and additional ion implantations. The complexity is due to the positive dielectric charge that accumulates electrons near the surface inducing and inversion layer that short connects the segmented n^+ areas and draws very high leakage current from sawing damaged edges. Two methods are commonly adopted in order to suppress. P-spray is a light field implantation that results in the vicinity of the surface negative space charge that repulses electron accumulation. So called p-stop is simply heavily doped pn-junction isolation between segmented n^+ areas. Five $n^+/p^-/p^+$ pad detector wafers have been processed with different surface terminations. The active pad implanted area of the diodes is $5 \times 5 \text{ mm}^2$. It is surrounded by two wide guard rings ($100 \mu\text{m}$) having opposite implantations (except wafer #5) and 16 small guard-rings (each $16 \mu\text{m}$ wide) where every second guard ring has opposite doping. The distance between the active area implant and the first guard ring is $10 \mu\text{m}$. The V_{fd} of as-processed diodes were measured by CV. The V_{fd} of diodes originating one p-type and one n-type wafer with respect of distance from wafer centre are shown if Figure 3.

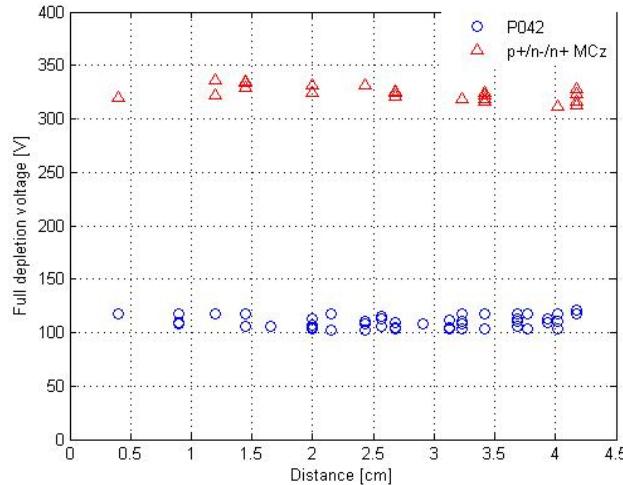


Figure 3. V_{fd} as function of distance of a p-type MCz-Si diodes (\circ) and n-type MCz-Si diodes (Δ).

The properties of wafers are listed in Table 1.

Table 1. P-spray and p-stop combinations of five pad detector wafer. Each wafer consists of 80 diodes from which 57 were measured. The values of V_{fd} and I_{leak} are expressed average \pm standard deviation. The yield percentage refers to the number of bad samples out of 57 excluded from data. The sample is considered to bad if breakdown occurs before the full depletion.

	P-stop $1 \times 10^{15} \text{ cm}^{-2}$	P-spray	V_{fd}[V]	I_{leak}[nA]	Yield [%]
P042	X	-	110 ± 5.5	3.2 ± 2.1	93
P068	X	$1 \times 10^{12} \text{ cm}^{-2}$	115 ± 5.7	2.6 ± 2.0	91
P069	X	$3 \times 10^{12} \text{ cm}^{-2}$	115 ± 4.4	2.2 ± 1.8	84
P082	X	$5 \times 10^{12} \text{ cm}^{-2}$	112 ± 4.6	3.3 ± 2.4	86
P083	-	$3 \times 10^{12} \text{ cm}^{-2}$	106 ± 4.4	2.6 ± 2.4	58

The leakage current with respect of position in the wafer in n and p-type diodes are shown in Figure 4.

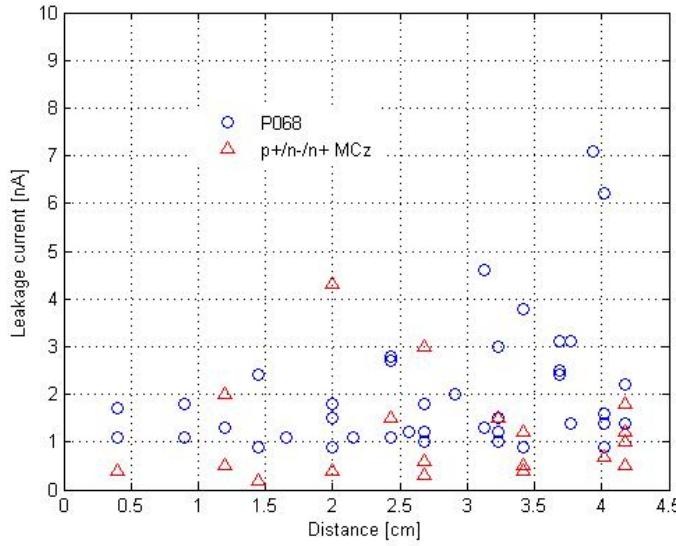


Figure 4. Leakage current of 0.25 cm^2 area diodes as function of distance from wafer center of a p-type MCz-Si diodes (\circ) and n-type MCz-Si diodes (Δ).

It can be seen in Figures 3-4 and Table 1. that the electrical properties of $n^+/p^-/p^+$ pad detectors are homogenous. The standard deviation of the V_{fd} along the wafer diameter is less than 5%. The average V_{fd} value of five wafers undergone same thermal history and processing condition is from 106V to 115V. For comparison, in n-type devices the average V_{fd} is 324V and the standard deviation along the wafer diameter is 7V. The leakage current is also satisfactory. The I_{leak} is slightly higher in p-type diodes than in n-type references. That is obviously caused by surface current. A small trend of increasing I_{leak} towards the wafer edges can be seen in Figure 4. That is possibly due to the lower carrier lifetime near the wafer edges due to contaminants diffusion from the wafer carriers during the high temperature processing. The surface current isolation technology does not seem to influence I_{leak} significantly. The breakdown properties are, however, worst in P083 wafer having no p-stop and best in P042 without any p-spray.

5. Summary

It is possible to manufacture high quality particle detectors from high resistivity Czochralski silicon. Electrical properties, i.e. full depletion voltage and leakage current, are comparable to those of traditional Float Zone silicon detectors. A characteristic feature of Cz-Si is the formation of thermal donors at certain temperatures. The kinetics of TD formation depends strongly on the oxygen concentration of Cz-Si material, on the presence of hydrogen in semiconductor fabrication process and on the temperature. Therefore it is essential to control the thermal history and temperature homogeneity of the detector process. The use of the magnetic field during the crystal growth might be necessary to adjust the oxygen concentration homogeneity to be sufficiently good for detector applications.

The $n^+/p^-/p^+$ and $p^+/n^-/n^+$ detectors show similar performance in terms of leakage current. However, if p-type MCz-Si wafers are used to produce $n^+/p^-/p^+$ devices, the TD formation can be utilized to tailor the V_{fd} . The compensation of the acceptor doping does not increase the leakage current of the detector, as showed in Figure 4. and Reference [12]. This is expected because the thermal donors form shallow levels in the band gap of silicon and thus

their contribution to the leakage current is much smaller than e.g. that of metallic impurities induced during the device processing. Therefore, detectors with high oxygen concentration and with low V_{fd} can be fabricated without additional process complexity or additional high temperature diffusion oxygenation.

Many investigations performed within the framework of CERN RD50 Collaboration have showed that MCz-Si is less sensitive to the changes in V_{fd} in proton radiation, but no or very little improvement in neutron radiation. Also, no significant improvement in trapping lifetime and leakage current has been observed [19]. The lower V_{fd} under harsh radiation conditions is, however, itself an important improvement, because in the future large tracking systems upgrades, the power dissipation will be one of the difficult engineering issues. Furthermore, MCz-Si is available in large quantities and if necessary, in large diameter substrates. Thus, MCz-Si can be considered as a serious candidate for the detector material in future very high luminosity colliders.

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