Introduction of high oxygen concentration in silicon wafers by high temperature diffusion

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Abstract

The tolerance to the hadron irradiation of silicon detectors can be improved by the introduction of high oxygen concentration into the starting material. High resistivity FZ silicon is required for the detectors used in high energy physics applications. Significantly high oxygen concentration (> 10^{17} atoms cm^{-3}) can hardly be achieved during the FZ silicon refinement.
The diffusion of oxygen at high temperature from a SiO_2 layer grown on both sides of a silicon wafer is a simple and effective technique to achieve high oxygen concentrations throughout the bulk of a 300 µm thick silicon wafer.

1. Introduction

The damage inflicted to silicon detectors by the intense hadron fluxes predicted in the future high energy physics experiments [1,2] can lead to the failure of these devices before the lifetime of the experimental programme. The radiation tolerance of silicon can be improved by the deliberate introduction of impurities into the silicon crystal [3]. Impurities can react with the primary induced defects (vacancies and interstitials) and effect the formation of electrically active defect centres. Suitable impurities can thus control the degradation of the electrical parameters of devices, as the increase of the full depletion voltage due to the changes in the effective spatial charge in irradiated devices. In particular, oxygen acts as a sink for vacancies. The rate of formation of di-vacancy (or multi-vacancy) complexes, responsible for the degradation of the electrical properties of detectors, can therefore be reduced in presence of high oxygen concentration in silicon [4].

Oxygen is present in low resistivity Czochralski silicon up to ~ 10^{18} atoms cm^{-3}, but concentration of only ~ 10^{15} cm^{-3} are found in the purer and high resistivity Floating-Zone (FZ) silicon used for the fabrication of particle detectors.

The high temperature diffusion technique can be used to enhance the oxygen concentration in the 250-300 μm thick silicon wafers produced from FZ grown ingot.

2. Impurity diffusion in silicon

Impurities can diffuse in the silicon crystal through vacancy or interstitial sites. The isotropic diffusion process is described in term of the diffusion coefficient D, as defined by the first Fick's law [5]:

$$\bar{j} = -D\nabla N \qquad (1)$$

where N is the impurity concentration. The silicon devices are plane and parallel structures, therefore the analysis can be limited to the simple one dimensional case. By applying the continuity equation to eq. 1 we obtain, for the one-dimensional case:

$$\frac{\partial N}{\partial T} = D\frac{\partial^2 N}{\partial x^2}\,. \qquad (2)$$

Whether the diffusing impurities enter the body through the surface (plane $x = 0$) so as to maintain a constant concentration N_0 at the surface, the boundary conditions are:

$$N(x > 0, t = 0) = 0\,, \qquad N(x = 0, t \geq 0) = N_0 \qquad (3)$$

and the solution of eq. 2 is:

$$N(x,t) = N_0 erfc\frac{x}{2\sqrt{Dt}}\,. \qquad (4)$$

This solution describes reasonably well an impurity diffusing in silicon from a gas or a vapour phase.

The solution of eq. 1 when the impurity is introduced in the body before the diffusion, forming a "rectangular" box in a semi-infinite body, is:

$$N(x,t) = \frac{N_0}{2}\left[erfc\left(\frac{x-h}{2\sqrt{Dt}}\right) - erfc\left(\frac{x+h}{2\sqrt{Dt}}\right)\right]. \qquad (5)$$

The Taylor's series expansion of 5 about $\frac{x}{2\sqrt{Dt}}$ is:

$$N(x,t)=\frac{Q}{\sqrt{\pi Dt}}\exp\left[-\left(\frac{x}{2\sqrt{Dt}}\right)^2\right]\cdot\left[1+\frac{1}{3!}\cdot\left(\frac{h}{2\sqrt{Dt}}\right)^2 H_2\left(\frac{x}{2\sqrt{Dt}}\right)+\frac{1}{5!}\cdot\left(\frac{h}{2\sqrt{Dt}}\right)^4 H_4\left(\frac{x}{2\sqrt{Dt}}\right)+\ldots\ldots\right]. \qquad (6)$$

The oxygen diffusion experiment

The velocity of the diffusion depends on the coefficient D and the achievable concentration depends on the solubility of the impurity in silicon. The expression of D as a function of the temperature is:

$$D(T)=D_0\exp\left(-\frac{E_d}{k_B T}\right). \qquad (7)$$

Table 1 lists the values of the diffusion parameters of oxygen in silicon as reported by many workers.

Oxygen diffusion parameters			
E_d [eV]	**D_0 [cm^{2}s^{-1}]**	**D(1150 °C) [cm^{2}s^{-1}]**	**D(1200 °C) [cm^{2}s^{-1}]**
3.5	135	$5.44 \cdot 10^{-11}$	$1.43 \cdot 10^{-10}$
2.55	0.21	$1.96 \cdot 10^{-10}$	$3.97 \cdot 10^{-10}$
3.5	83	$3.35 \cdot 10^{-11}$	$8.82 \cdot 10^{-10}$
2.4	0.091	$2.88 \cdot 10^{-10}$	$5.60 \cdot 10^{-10}$
3.15	22.6	$1.58 \cdot 10^{-10}$	$3.78 \cdot 10^{-10}$
2.44	0.07	$1.60 \cdot 10^{-10}$	$3.15 \cdot 10^{-10}$
2.77	1.5	$2.33 \cdot 10^{-10}$	$5.01 \cdot 10^{-10}$
2.54	0.17	$1.72 \cdot 10^{-10}$	$3.47 \cdot 10^{-10}$
2.51	0.11	$1.42 \cdot 10^{-10}$	$2.85 \cdot 10^{-10}$
2.43	0.033	$8.19 \cdot 10^{-11}$	$1.60 \cdot 10^{-10}$
2.53	0.14	$1.54 \cdot 10^{-10}$	$3.10 \cdot 10^{-10}$

Table 1 *Diffusion parameters and solubility for O, C and Sn in silicon* [6].

The value of oxygen solubility in silicon at 1250 °C is ≈ $8.4{\cdot}10^{17}$ [6].

The oxygen diffusion tests have been performed at the Technion University in Haifa (Israel) using 1x1 cm^2 and 300 μm thick samples of standard silicon produced by POLOVODICE [7]. Layers of ≈ 3000 Å of silicon oxide were grown on both sides by the dry oxidation technique. The samples were then heated to 1150 °C in an inert (N_2) atmosphere, to allow the dissociation of oxygen from the SiO_2 at the interface, and its diffusion into the silicon bulk. The samples were maintained at 1150 °C during 20 hours.

Figure 1 shows the obtained oxygen profile as a function of the depth in silicon, as measured by the Secondary Ion Mass Spectroscopy (SIMS) technique, by the Evans Europa Company. The value of D and N_0 (maximum concentration at the surface) obtained by fitting the data are $2.25 \cdot 10^{-10}$ cm^2 s^{-1} and $5.6 \cdot 10^{17}$ cm^{-3}, respectively. The SiO_2 layer disturbs the SIMS signal for the shallower points and those points have been excluded from the fit.

Figure 2 shows the oxygen concentration as a function of the depth into the silicon wafer as calculated using the fitted parameters for tree different diffusion times at 1150 °C.

The high temperature diffusion technique was used to introduce oxygen into 3” silicon n-type wafers, 300 μm thick, with initial resistivity of ~ 2.5 kΩ cm, suitable for the detector processing. The single crystal wafers were provided by POLOVODICE and were diffused by ITE [8] during 24, 48 and 72 hours at 1150 °C. The profile of the oxygen concentration as a function of the depth in silicon is shown in Fig. 3. It can be noticed the large enhancement of the oxygen concentration compared to the prediction, particularly evident for the 24 hours diffusion.

ITE processed the diffused wafer to produce 5x5 mm^2 diodes. The resistivity of the diffused material, as measured using the capacitance-voltage technique on the diodes, was between 2.2 to 2.7 kΩ cm, in the range of the fluctuation normally found for FZ wafers and then comparable to the initial resistivity. This indicates that the formation of small clusters of oxygen atoms that arise from the diffusion at 450 °C and act as thermal donors [9] is relatively negligible.

3. Conclusion

The high temperature diffusion is an effective technique to introduce fast diffusing impurities in silicon. High oxygen concentration in 300 μm thick silicon wafers can be reached with relatively short diffusion time at 1150 °C from SiO_2 layers grown on both surfaces of the wafers. The radial symmetry of this technique guarantees an optimal radial homogeneity of the oxygen concentration. The experimental results also exhibit a good homogeneity of the concentration as a function of the depth, better than the prediction. Actually, already with 24 h of diffusion time, the obtained profile is rather smooth, and the O concentration in the middle of the silicon wafer (150 µm) is $1.7 \cdot 10^{17}$ cm^{-3}.

A negligible reduction of the initial resistivity due to oxygen complex acting as thermal donor has been found in n-type silicon wafers after the high temperature diffusion process. This technique is therefore suitable for the introduction of oxygen in the high resistivity silicon required for the fabrication of detector for high energy physics.

4. Additional remarks

The negligible changes of the resistivity measured on the O-diffused diodes imply low formation of O-related thermal donor. This result is achieved avoiding long time annealing around 450 °C during the decreasing temperature cycle after the diffusion. The diffusing wafers should be removed from the furnace when the temperature reaches 600-700 °C after the decreasing cycle.

After the diffusion, the deposited silicon oxide is removed. This can be obtained by chemical etching or by polishing. Polishing the detector could probably recover a better quality of the surface of the wafer, because it remove the Si-SiO_2 interface layer that could present high density of defects after the high temperature treatment.

References

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Figure caption

Fig. 1 Oxygen concentration profile, measured by SIMS, in a silicon sample diffused during 20h at 1150 °C in N_2 atmosphere from a 3000 Å thick layer of SiO_2.

Fig. 2 Oxygen concentration as a function of the depth into the silicon wafer calculated for tree different diffusion times at 1150 °C. The diffusion parameter used have been obtained from the fit shown in Fig.1.

Fig. 3 SIMS measurement of the oxygen concentration profile as a function of the depth in silicon after different diffusion times from SiO_2 surface layers grown on both sides of the silicon wafer. Diffusion times: 24h, 48h, 72h; diffusion temperature: 1150 °C.

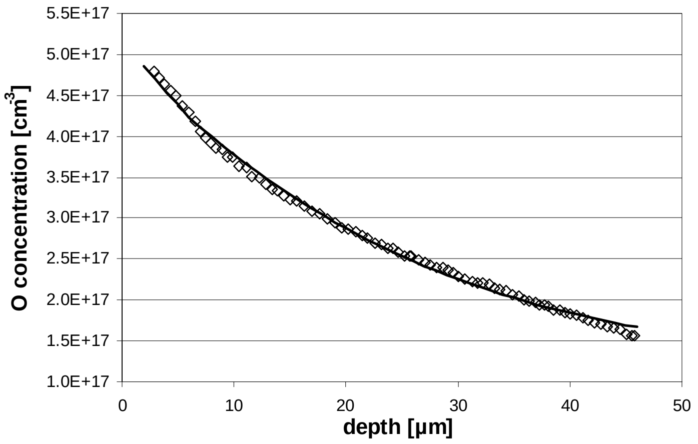

5.5E+17
5.0E+17
4.5E+17
4.0E+17
3.5E+17
3.0E+17
2.5E+17
2.0E+17
1.5E+17
1.0E+17
O concentration [cm-3]
0
10
20
30
40
50
depth [µm]

Fig. 1

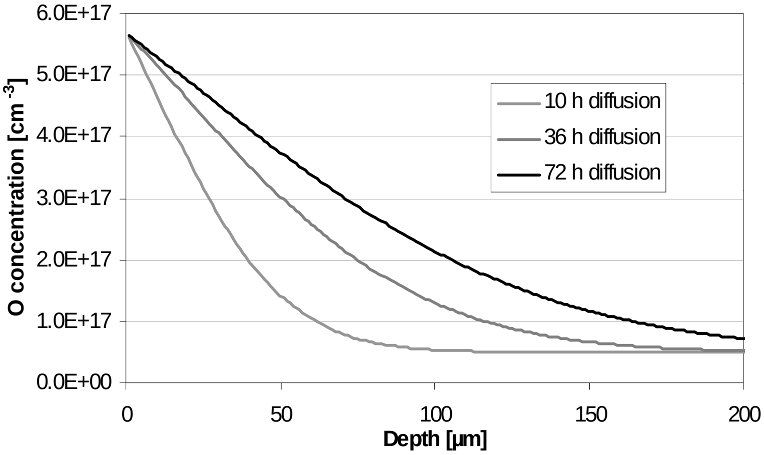
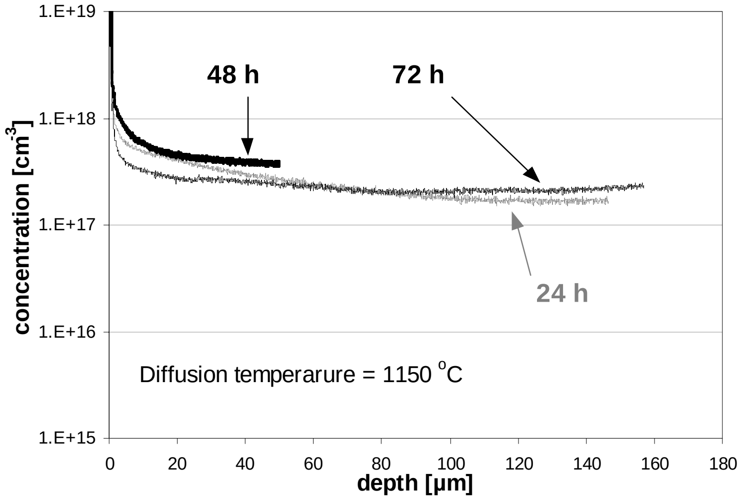

6.0E+17
5.0E+17
4.0E+17
3.0E+17
2.0E+17
1.0E+17
0.0E+00
O concentration [cm -3]
10 h diffusion
36 h diffusion
72 h diffusion
0
50
100
150
200
Depth [µm]

Fig. 2



1.E+19
1.E+18
1.E+17
1.E+16
1.E+15
concentration [cm-3]
48 h
72 h
24 h
Diffusion temperarure = 1150 °C
0
20
40
60
80
100
120
140
160
180
depth [µm]

Fig. 3