Silicon wafer oxygenation from SiO₂ layers for radiation hard detectors


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Abstract

Radiation produces lattice damage in silicon by displacing the atoms from their original positions and thereby generating the corresponding defects. As a result, new states are created in the semiconductor forbidden band gap, affecting negatively the electrical performance of devices. The endurance to radiation can be improved by having a high oxygen concentration in the silicon. For detector fabrication high resistivity silicon is also needed, thus Float Zone wafers are preferable, however, this kind of material exhibits low oxygen concentration. Although different ways to incorporate oxygen in Float Zone silicon have been proposed, all of them imply modifications during the ingot growth. Thermal diffusion from SiO₂ layers on polished wafers is an interesting alternative solution to improve their oxygen content. Different thermal processes aimed at obtaining oxygen enriched silicon for the fabrication of radiation hard detectors have been tested. Attention has also been paid to carbon introduction during processing since high concentrations of this element has been proved deleterious.

Keywords: Silicon oxygenation, radiation hardening, radiation damage, FZ silicon

1. Introduction

Radiation produces lattice damage in silicon by displacing the atoms from their original positions and thereby generating silicon interstitials (I) and the corresponding vacancies (V). As a result, new states are created in the semiconductor forbidden band gap. Some of these defects have a negative impact on the electrical performance of the silicon: shorter minority carrier lifetime, increased leakage currents and, a feature specially important for silicon radiation detectors, the full depletion voltage of the silicon substrate changes due to acceptor-like radiation induced defects.

After a high energy particle impact, a cascade of interactions occur, generating several thousands of I-V pairs. Most of them recombine in a very short time and some form stable complexes, which are immobile, forming clusters of localised defects. The pairs surviving this initial recombination diffuse through the crystal reacting with other defects and impurity atoms, particularly oxygen and carbon. Due to this fact, it was hypothesised that carbon and oxygen impurities would improve the radiation tolerance of the silicon as it was thought that the mentioned combination with interstitials and vacancies would generate less detrimental defects.

Experience showed that while oxygen indeed improved the silicon strength against radiation, the presence of carbon in the silicon lattice diminished
its performance [1,2,3]. The minimum recommended oxygen concentration for improving radiation hardness has been empirically seen to be around $10^{17} \text{cm}^{-3}$ [2].

For radiation hard silicon detectors the starting material should combine high oxygen concentrations and high resistivity. This last feature is necessary to obtain wide depletion zones, thus increasing the detection efficiency. Unfortunately, high resistivity requires Float Zone silicon which has low oxygen concentrations. Czochralski wafers have higher oxygen concentration but cannot be made with a resistivity as high as needed for this application (above 1000 $\Omega \cdot \text{cm}$). There have been proposed several methods to increase the silicon oxygen concentration during the ingot growth such as gas jet doping and quartz ring on the Float Zone set up. Unfortunately all of them involve complicated procedures for the starting silicon at the wafer factory level.

There is another option, easier and cheaper, consisting of enriching the oxygen content of standard silicon by diffusion of oxygen from a thick oxide layer [2,4]. In this work we have focused on this last option analyzing different processing conditions, crystal orientations and carbon contamination.

2. Experiments

Experiments were made at two different laboratories, IMB-CNM and SINTEF, on both $<100>$ and $<111>$ orientations. The wafer processing is detailed in table 1. All the wafers were Float Zone from Wacker and from Topsil, with an 1-6 k$\Omega \cdot \text{cm}$ resistivity range.

It is well known that adding small amounts of chlorine to the oxidation ambient improves the oxide quality and usually fastens its growth. The original source of chlorine, HCl, has been nowadays replaced by carbon-chlorine compounds (e.g. $\text{C}_2\text{H}_3\text{Cl}_3$ or $\text{C}_2\text{H}_2\text{Cl}_2$). There is the possibility of carbon diffusion from the SiO$_2$ layer using these kinds of compounds. The same hazard could exist if the diffusion tube material is SiC. To examine these hazards, oxygen and carbon concentrations were measured by SIMS.

The processing consisted of an initial oxidation, followed by an optional diffusion step in $\text{N}_2$ ambient. All the oxidations were made in quartz tubes, dry ($\text{O}_2$) or wet ($\text{H}_2 + \text{O}_2$) ambient were used. In some cases trichloroetane (TCA) was added. The diffusion step was made in a quartz or in a SiC tube. The diffusion temperature was always 1150$^\circ\text{C}$, and the diffusion times ranged from 12 to 72 hours.

3. Results

Oxygen and carbon concentrations measured by SIMS are presented for the different processes described in the section above. All the wafers were 300µm thick, and oxidations and diffusion took place from both sides simultaneously, so measurements were made up to 150 µm, which is the middle of the wafer. The measurement background was $1.6 \times 10^{16}$ cm$^{-3}$ for oxygen and $6.3 \times 10^{15}$ cm$^{-3}$ for carbon, and is indicated with a dashed line in all the graphs.

Figure 1 shows that it is not necessary to diffuse in $\text{O}_2$ atmosphere during the whole process to achieve a high oxygen concentration. The same result can be achieved with just an initial thick oxide and a subsequent long nitrogen annealing, which is a much cheaper process.

### Table 1
Wafer processing. Process identification refers to the wafer supplier (W=Wacker, T=Topsil), crystal orientation, and thermal processes.

<table>
<thead>
<tr>
<th>Process</th>
<th>Oxidation</th>
<th>Oxide thickness</th>
<th>Diffusion</th>
<th>Tube material</th>
</tr>
</thead>
<tbody>
<tr>
<td>W111-A; T111-A</td>
<td>$\text{H}_2 + \text{O}_2$, 1100$^\circ\text{C}$</td>
<td>700nm</td>
<td>72 hours, $\text{N}_2$</td>
<td>quartz</td>
</tr>
<tr>
<td>W111-B; T111-B</td>
<td>$\text{O}_2 + \text{TCA}$, 72 hours, 1150$^\circ\text{C}$</td>
<td>1.5µm</td>
<td>No additional diffusion</td>
<td>quartz</td>
</tr>
<tr>
<td>W111-C; T100-C</td>
<td>$\text{O}_2$, 6 hours, 1100$^\circ\text{C}$</td>
<td>330nm</td>
<td>No additional diffusion</td>
<td>quartz</td>
</tr>
<tr>
<td>W111-D; T100-D</td>
<td>$\text{O}_2$, 12 hours, 1150$^\circ\text{C}$</td>
<td>600nm</td>
<td>No additional diffusion</td>
<td>quartz</td>
</tr>
<tr>
<td>W111-E; T100-E</td>
<td>$\text{O}_2$, 12 hours, 1150$^\circ\text{C}$</td>
<td>600nm</td>
<td>48 hours, $\text{N}_2$</td>
<td>quartz</td>
</tr>
<tr>
<td>W111-F; T100-F</td>
<td>$\text{O}_2$, 12 hours, 1150$^\circ\text{C}$</td>
<td>600nm</td>
<td>48 hours, $\text{N}_2$</td>
<td>SiC</td>
</tr>
</tbody>
</table>
Fig. 1: SIMS Oxygen content on some <111> samples.

In figure 2, the oxygen profiles of <111> wafers after different processing times are shown. As can be observed, concentrations higher than $1 \times 10^{17}$ cm$^{-3}$ can be got throughout the wafers with a 48h extra diffusion step after a long enough oxidation (12h).

Fig. 2: Oxygen concentrations obtained with different processing times.

As shown in figure 3, qualitative similar results were achieved with <100> samples, although the <111> ones consistently yielded a higher oxygen content. A last remark regarding these figures is that higher oxygen contents were also obtained in the quartz tube in comparison to the SiC tube, although a temperature mismatching between them explaining this fact can not be ruled out at this point.

Fig. 3: Differences in oxygen content between <100> and <111> samples and between SiC and quartz tubes.

Finally, with regard to the carbon content, it is evident from figure 4 that the use of TCA during the oxidation promoted its incorporation to the silicon. It is also clear from figure 5 that processing in a SiC tube did not.

Fig. 4: Carbon concentration on <111> samples with and without TCA.

Fig. 5: Carbon concentration on <111> samples processed in quartz and SiC tubes.
4. Further measurements

Float Zone wafers exhibit a radial resistivity variation higher than Czochralski ones \[5,6\], reaching up to a 20% non-uniformity. The oxygenation process has the additional advantage of homogenizing impurity wafer concentration, feature of maximum importance in the fabrication of large area detectors.

A potential problem is mechanical deformation due to so high thermal budgets. Wafer curvature was used as an indication of deformation.

Wafer resistivity and curvature was measured before and after thermal processing, and results are shown in table 2. Resistivity was measured using a four point equipment, and curvature with and optical system.

Oxygen diffusion improves significantly the resistivity homogeneity, as shown on wafermaps on figures 6 and 7, without any important deformation. It is important to point out that some thermal donor activation occurs, decreasing silicon resistivity, but the final value is high enough to fabricate high quality detectors.

5. Conclusions

Diffusion from a thick SiO2 layer is a suitable method for silicon wafer oxygenation. The presence of carbon-chlorine compounds in the oxidation ambient is harmful since it promotes carbon incorporation into the silicon.

Concerning to the time, at least 48 hours diffusion at 1150°C is needed to achieve an oxygen concentration above \(10^{17} \text{ cm}^{-3}\) in the middle of 300 µm wafers, which is high enough to achieve radiation hardening. SiC tubes behave in a similar way to quartz tubes and do not produce carbon incorporation. This is a technological important point since in the long term SiC tubes withstand better the extreme thermal conditions required by the oxygenation.

Acknowledgements

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References


Table 2
Four point resistivity and curvature measurements

<table>
<thead>
<tr>
<th>Measure</th>
<th>Resistivity (Ω·cm)</th>
<th>Carrier concentration ( (\text{cm}^{-3}) )</th>
<th>Homogeneity</th>
<th>Mean radius of curvature (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td>4470</td>
<td>(1.1 \times 10^{12})</td>
<td>12.9%</td>
<td>234</td>
</tr>
<tr>
<td>After thermal process</td>
<td>2519</td>
<td>(1.9 \times 10^{12})</td>
<td>3.3%</td>
<td>164</td>
</tr>
</tbody>
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