Oxygen Precipitation in Highly Doped Silicon Substrates

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This paper reviews oxygen precipitation in heavily arsenic-doped silicon. A wide arsenic concentration range is explored, from $3 \times 10^{18}$ to $4 \times 10^{19}$ cm$^{-3}$. A precipitation retardation effect is observed in the arsenic doped samples when the dopant concentration is higher than $1.7 \times 10^{19}$ cm$^{-3}$ compared with lightly doped samples having the same initial oxygen content and grown under similar conditions. The oxygen precipitate density in the heavily arsenic-doped samples is uniform along the wafer radius, with no rings or cores, contrary to what is commonly observed in lightly doped samples grown with similar V/G values. This finding is discussed by considering the role played by vacancies in the formation of oxygen precipitates and the impact of the arsenic concentration on the equilibrium concentration of point defects in silicon, deduced from the experimentally observed voids revealed as light-scattering surface defects in polished wafers.

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Heavily doped silicon substrates are widely used for the fabrication of discrete devices for power applications. The basic wafer template is generally composed of an epitaxial layer over a heavily doped silicon substrate whose dopant type and resistivity vary depending on the final device characteristics. In either case of p- and n-type substrates, the demand is more and more oriented toward very low resistivity ranges, such as 0.5–5 m$\Omega \cdot$cm, in order to minimize the substrate contribution to the transistor drain-source resistance. Furthermore, the continuous effort to reduce manufacturing costs is moving discrete device fabrication from 100–150 mm in wafer diameter to 200 mm and even to 300 mm. Often a vertical device structure is chosen, where the electric current flows through the entire wafer thickness, therefore the properties of the wafer bulk affect the device performance and the bulk defectivity level has to be understood and controlled carefully. In this respect, oxygen precipitation is of utmost importance. The aim of this paper is to present a study of the behavior of oxygen precipitation in heavily doped silicon substrates, with particular focus on heavy arsenic doping.

Oxygen Concentration and Incorporation

Oxygen incorporation is of course the first factor to be considered when dealing with oxygen precipitation in silicon. Crystal growers have a number of process parameters that they can tune in order to achieve the desired oxygen concentration, such as crucible rotation rate, magnetic field intensity and so on. However, when the melt is heavily doped with volatile elements, such as arsenic, antimony or phosphorus, the oxygen concentration shows a strong dependence on the dopant concentration itself and it becomes very difficult to decouple these two parameters and achieve any desired combination of oxygen and dopant concentration. Specifically, a decrease in oxygen concentration with increasing dopant concentration is typically observed. This phenomenon was first reported in the literature in the case of antimony doping, and the responsible mechanism was concluded to be an enhanced oxygen evaporation from the melt. The crucible dissolution rate was found to be enhanced, too. The reasons of the increased oxygen evaporation are still under debate. A direct evaporation of antimony oxide species has been proposed by some authors but excluded by others on the basis of thermodynamic calculations and of the analysis of evaporated deposits. Some authors have proposed an accelerated SiO$_2$ evaporation caused by the simultaneous evaporation of elemental antimony, similar to the steam distillation of a substance of low volatility and to diffusion pumping. More recently, the enhanced SiO$_2$ evaporation was explained by the reaction of Sb with SiO$_2$ in the gas phase above the melt: the Sb$_2$O$_3$ formation lowers the SiO partial pressure in the gas boundary layer, favoring its evaporation from the melt. A deeper discussion is outside the scope of this paper. The same phenomenon is observed also in the case of arsenic and phosphorus doping: the oxygen concentration decreases with increasing dopant concentration, if all other process conditions are kept the same. An example relative to arsenic doping is shown in Figure 1. The effect of the arsenic concentration on the oxygen content becomes appreciable below 7 m$\Omega \cdot$cm (corresponding to a concentration of As of $8 \times 10^{18}$ at/cm$^3$).

The mechanism responsible for the reduced oxygen concentration in the case of phosphorus and arsenic doping is likely the same as that proposed for antimony-doping: an enhancement in oxygen evaporation. The evaporating species could be either SiO$_2$ only, as suggested for the case of Sb, or a mixture of SiO$_2$ and volatile P and As oxides, respectively. Evidence in support of this mechanism is the formation of heavier oxide deposits on cold puller parts during the crystal growth process, as well as a faster crucible erosion, compared to undoped melts. In particular, in the case of arsenic doping, the crucible erosion rate at the melt line was measured and found to be twice as high compared to undoped melts: 22.4 $\mu$m/hour in the case of arsenic-doped melts vs 10.5 $\mu$m/hour in the case of undoped melts under the same process conditions. It is interesting to observe that in

Figure 1. Oxygen concentration in arsenic-doped silicon samples measured by Gas Fusion Analysis as a function of the sample resistivity. All data points refer to crystals grown under the same conditions, in particular the melt quantity in the crucible is the same (same melt aspect ratio). For reference, the average oxygen concentration of lightly doped silicon grown in the same conditions is 17 ppm. The GFA was calibrated against FTIR using lightly doped samples. The FTIR calibration coefficient used is $2.45 \times 10^{-17}$ atoms/cm$^3$ (ASTM F 121-83).
the case of heavy boron doping (boron is non-volatile), no appreciable change in oxygen concentration is generally observed.6,7

The strong coupling between dopant concentration and oxygen concentration in the case of volatile dopants poses a challenge when trying to assess the impact of the dopant concentration on the oxygen precipitation, because it is very difficult to obtain a good set of samples where only the dopant concentration varies. In this case, the use of undoped or lightly doped samples with the same oxygen content and grown under the same conditions is necessary.

Oxygen and Oxygen Precipitation Measurement in Heavily Doped Silicon

Oxygen measurement in silicon wafers down to $1 \times 10^{16}$ at/cm$^3$ is usually performed by Fourier Transform Infrared Spectroscopy (FTIR). However, the method cannot be applied when the dopant concentration exceeds $1 \times 10^{15} - 1 \times 10^{16}$ at/cm$^3$ due to excessive free carrier absorption. For heavily doped silicon, the most used methods are Gas Fusion Analysis (GFA) and Secondary Ion Mass Spectroscopy (SIMS). GFA is based on the reaction of oxygen with the carbon of the graphite crucible after having melted the silicon sample under test. GFA needs a significant amount of silicon for each analysis, a few mm$^3$. The detection limit is approximately $1 \times 10^{16}$ at/cm$^3$ for a standard GFA. For SIMS, the sample is bombarded by a primary ion beam under UHV conditions which are necessary to improve the detection limit; for oxygen this is around $1 \times 10^{17}$ at/cm$^3$. SIMS can best measure the oxygen concentration on polished wafers and can also perform a depth profiling of the oxygen concentration. Contrary to the FTIR technique which is fast and not destructive, GFA and SIMS are both destructive methods and require much longer measurement time, especially SIMS. GFA is often used in the industry for in-line product certification: the tool has affordable costs and reasonable measurement cycle time, of the order of one day including the sample preparation. SIMS is typically used when GFA is not possible, i.e. for the measurement of thin wafers, or for depth profiling. Both GFA and SIMS must be calibrated, this is usually accomplished using lightly doped silicon samples certified by FTIR. Finally, it must be reminded that both GFA and SIMS measure the total oxygen concentration of the sample, without the possibility to distinguish between the interstitial form and other oxygen species.

The most common method to determine the volume density of oxygen precipitates in heavily doped silicon is preferential etching and defect counting under a microscope. Usually, the sample is cleaved to expose a $<111>$ plane and it is etched with a preferential etch mixture that creates a pit on the surface of the sample in correspondence to each precipitate. The etching depth is measured and the defect volume density is calculated by dividing the total number of defects by the total volume sampled. Another method used to characterize oxygen precipitates in heavily doped silicon is transmission electron microscopy (TEM), but this method is mainly useful to characterize the defect morphology rather than the density, due to the small volume measured. It is also difficult to find defects with TEM when their density is less than $10^{10}$ cm$^{-3}$. Light scattering tomography (LST) can be also used when the investigated material is transparent to the wavelength of the laser. The scattered light is analyzed and the size, number and distribution of the light scattering defects can be calculated. Modern LST setups can detect defects larger than about 20 nm.

Oxygen Precipitation Behavior in Heavily Doped Silicon

Heavily doped N+ silicon wafers generally exhibit very low oxygen precipitation when analyzed after a typical nuclei stabilization followed by growth thermal cycle, such as the standard precipitation treatment consisting of 4 hours at 800°C followed by 16 hour at 1000°C.8 An example of this behavior in the case of heavily arsenic-doped silicon is shown in Figure 2, where the oxygen precipitate density is plotted as a function of the sample resistivity. An abrupt drop by 4 orders of magnitude is observed below 4 mΩ·cm (corresponding to a dopant concentration of $1.6 \times 10^{15}$ cm$^{-3}$).

From the plot in Figure 2 it cannot be immediately concluded that the drop in oxygen precipitation is due to the increased dopant concentration, since also the oxygen content decreases with increasing dopant concentration, as shown previously in Figure 1, although not so sharply. To some extent, the decrease in oxygen content can be corrected by tuning the crystal pulling process, but not completely.

In order to fully decouple the two factors, a comparison with lightly doped samples was done. For this purpose, a group of lightly boron doped samples grown under similar conditions was chosen. Attention was put to compare samples not only with the same oxygen content, but also with similar growth thermal histories, to reduce as much as possible the influence of other factors. More details on this study can be found in Ref. 11. The results are shown in Figure 3, where the oxygen precipitate density is plotted as a function of the oxygen content, comparing lightly boron doped and heavily arsenic doped samples. The reduced oxygen precipitation in the case of arsenic doped samples is confirmed. For completeness, it should be mentioned that the possibility of an incomplete detection of precipitates does exist, in principle, and one could also explain the reduced oxygen precipitation observed in the heavily arsenic-doped samples as a result of their size becoming smaller than the detection limit of the technique, rather than their volume density becoming lower. Although this possibility
cannot be ruled out completely, to the authors’ knowledge no evidence is available that oxygen precipitates become smaller with increasing arsenic concentration, therefore this work will assume that the detected defects are well representative of the total number of defects for both sample groups.

Regarding the radial distribution of oxygen precipitates, the samples did not show any presence of rings or cores: defects were distributed quite uniformly on the whole sample radius, with the exclusion of few millimeters near the edge. A quantitative measurement is reported only for the center, where also the oxygen concentration was measured.

**Impact of Heavy Donor Doping On Point Defects**

The precipitation of oxygen is strongly affected by the concentration of vacancies. For this reason, before moving to the discussion of the oxygen precipitation in heavily doped silicon, we will review the impact of donors on point defects in silicon.\(^{12-15}\)

Experimentally it was found that increasing the concentration of donors at first shifts the silicon growth regime toward the vacancy-rich regime, until a critical dopant concentration is reached, and then toward the interstitial-rich regime for even higher concentrations.\(^{16,17}\) In the case of antimony, the impact on point defect concentration was observed already at a concentration of the order of \(10^{18} \text{ cm}^{-3}\).\(^{15}\) At these concentrations, a downward shift in the critical V/G (ratio between pull speed and axial thermal gradient) was measured. The maximum of the vacancy concentration (considering the number of voids counted by wafer surface laser inspection as indicator) is achieved at a critical concentration of \(1.7 \times 10^{19} \text{ cm}^{-3}\) (3.9 m\(\Omega\)·cm) in the case of arsenic, and at approximately \(2.9 \times 10^{19} \text{ cm}^{-3}\) (2.4 m\(\Omega\)·cm) for phosphorus.\(^{15}\)

The Figure 4 illustrates the impact of arsenic on the formation of voids detected as Crystal Originated Particles (COP) on the surface of 200 nm diameter polished silicon wafers by an automatic laser inspection tool. A similar trend was found also for phosphorus.\(^{15}\) The sudden drop is explained by a quick increase in the concentration of interstitial arsenic \(\text{As}_i\) and a corresponding sharp transition from the vacancy growth mode (with the dominant \(\text{VAs}_i\) point defects) to the interstitial mode (with the dominant \(\text{As}_i\) point defects). According to Ref. 18, the concentration of \(\text{VAs}_i\) (at the interface) increases in proportion to the doping level \([\text{As}]\) while the concentration of \(\text{As}_i\) increases much faster, roughly as \([\text{As}]^{1.5}\) - due to the assumed charge difference between substitutional \(\text{As}^+\) (positive) and interstitial state \(\text{As}_i\) (negative). For this reason, \(\text{As}_i\) will dominate at sufficiently high doping level.

A complementary view is shown in Figure 5, where the diameter of the vacancy-rich region is plotted as a function of the resistivity for several arsenic-doped crystals. The diameter of the vacancy-rich region is determined by visual inspection under collimated light of samples subjected to defect decoration (by copper) and preferential etching. At the highest resistivity explored of 25 m\(\Omega\)·cm (corresponding to an arsenic concentration \([\text{As}] = 8.6 \times 10^{17} \text{ cm}^{-3}\)), the silicon crystal has a small vacancy core of approximately 50 mm diameter surrounded by an annulus of interstitial-type dislocation loops. This behavior is similar to that of undoped or lightly doped silicon grown under the same V/G conditions, confirming that at this low concentration arsenic does not have an important impact on point defects concentration. In the range between 4 and 12 m\(\Omega\)·cm ([\(\text{As}] = 1.6 \times 10^{18}\) to \(3.3 \times 10^{18} \text{ cm}^{-3}\)) the crystal is fully vacancy-rich. From 4 to 2.5 m\(\Omega\)·cm ([\(\text{As}] = 2.7 \times 10^{18} \text{ cm}^{-3}\)) the density of the defects drops quite abruptly to zero while remaining substantially radially uniform, apart a few cases where the density at the crystal edge is already zero while some rare defects can still be observed in the central area. No rings or cores are observed.

Moving further down in resistivity, another feature becomes visible by copper decoration in samples with resistivity <1.8 m\(\Omega\)·cm ([\(\text{As}] > 3.9 \times 10^{19} \text{ cm}^{-3}\)], i.e. the presence of a pattern that resembles closely the dopant striations as far as the macroscopic pattern is concerned, but that is formed by tiny individual copper decorated points instead of being continuous lines, as shown in Figure 6. A possibility is that it consists of tiny arsenic clusters or precipitates. In the literature, the formation of arsenic clusters/precipitates has been studied mainly in silicon wafers implanted with arsenic at levels higher than the solubility limit, up to \(4 \times 10^{21} \text{ cm}^{-3}\), where they are considered...
Et al. have studied one arsenic-doped sample with a resistivity of 5·10⁻¹⁸ cm. The gradual transition from a regime where vacancy-related defects can easily form (top part of the image, higher resistivity) to a nearly defect-free region (bottom part of the image, lower resistivity) can be observed in this image.

The presence of arsenic precipitates at very high arsenic concentrations would support the hypothesis of a switch from vacancy-rich supersaturated (while the substitutional component is undersaturated). Reduced oxygen precipitation in heavily donor doped silicon.— The impact of vacancies on oxygen precipitation in heavily doped silicon was studied recently by means of high temperature rapid thermal anneal (RTA)-induced vacancy injection. Arsenic doped samples with a resistivity of 3·10⁻¹⁸ cm are typically observed in lightly doped silicon. For the arsenic doped samples, the enhancement was found only if the subsequent precipitation cycle is at 800°C (32h) or at 1000°C (32h), while no precipitates are observed for a cycle at 900°C (32h) or at 1000°C (32h). In the case of antimony doped samples, all thermal cycles showed an RTA-induced precipitation enhancement. According to the authors, in the antimony doped samples, less RTA generated vacancies are trapped by the dopant atoms and more vacancies remain available to reduce the size of the oxide precipitates compared to the arsenic-doped samples.

Why the oxygen precipitate density is often of the same “magic” value.— In lightly doped silicon, the oxygen precipitation induced by a standard annealing cycle 800 + 1000°C is controlled by the vacancies. The vacancies can be introduced into a wafer by Rapid Thermal Annealing (RTA) in a concentration C_v that is higher at a higher RTA temperature. The precipitate density N_p is an increasing function of C_v that seems to saturate at a level of about 10⁻¹⁹ cm⁻³. As-grown crystals are often composed of vacancy and self-interstitial regions. In the vacancy-dominated regions, the incorporated point defects are vacancies; they aggregate into voids that consume most of the space. In the interstitial regions, a crystal with a large oxygen content can form oxygen dimers. The precipitates at the vacancy-dominated regions are VO₂, which is generally higher at a higher RTA temperature.


10^{10} \text{ cm}^{-3} in the vacancy regions, due to the residual vacancies. In our As-doped crystals the precipitate density was found to be the same as in lightly doped vacancy-type crystals: around 10^{10} \text{ cm}^{-3} up to some high As concentration; above this threshold, the density is however strongly reduced.

The existence of a “magic” density value, \( \approx 10^{10} \text{ cm}^{-3} \), is further supported by the data on oxygen precipitation in boron doped silicon.\(^{12,13}\) The boron impurity is known\(^{14}\) to induce, at fixed growth conditions, a transition from vacancy to interstitial growth mode; this effect is manifested, in particular as a shrinkage of the OSF ring\(^{15}\) that is located within the vacancy region, close to the boundary between a vacancy and an interstitial region. In moderately doped crystals, when the OSF ring is still present (and thus the crystal still consists of an inner vacancy region and a peripheral interstitial region), the precipitate density was found to be \( 10^{10} \text{ cm}^{-3} \) uniformly through a wafer including the interstitial region (see Fig. 4b in Ref. 32). In the interstitial region, the self-interstitials (I) are replaced, upon lowering oxide precipitates. These sites, of a density always contain some heterogeneous sites for nucleation of crystals, seem to be enough for full activation of the sites: the observed density is quite rapid and can be explained by a strongly reduced vacancy availability in this resistivity range, as indicated also by the rapid disappearance of COPs. When the vacancy concentration is high enough, the density of oxygen precipitates is correspondingly high and tends to saturate at approximately \( 10^{10} \text{ cm}^{-3} \) also in the case of arsenic doped silicon. This can be explained by a heterogeneous precipitation mechanism controlled by the activation of specific sites with density of the order of \( 10^{10} \text{ cm}^{-3} \).

**Conclusions**

The phenomenon of the reduced oxygen precipitation in heavily donor doped silicon has been reviewed, with special emphasis on heavily arsenic-doped silicon. An impact of arsenic on oxygen precipitation is experimentally confirmed in heavily arsenic doped silicon below 4 m\(\Omega\) \cdot \text{cm} compared to lightly doped silicon with the same oxygen content and growth conditions. For resistivities higher than 4 m\(\Omega\) \cdot \text{cm}, no appreciable difference is observed between heavily arsenic doped and lightly doped silicon. The drop in oxygen precipitation below 4 m\(\Omega\) \cdot \text{cm} is quite rapid and can be explained by a strongly reduced vacancy availability in this resistivity range, as indicated also by the rapid disappearance of COPs. When the vacancy concentration is high enough, the density of oxygen precipitates is correspondingly high and tends to saturate at approximately \( 10^{10} \text{ cm}^{-3} \) also in the case of arsenic doped silicon. This can be explained by a heterogeneous precipitation mechanism controlled by the activation of specific sites with density of the order of \( 10^{10} \text{ cm}^{-3} \).

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