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# ON DETERMINING THE CONCENTRATION OF OXYGEN PRECIPITATION IN SINGLE-CRYSTALLINE SILICON UNDER HIGH HEAT TREATMENTS

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## ABOUT ARTICLE

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**Abstract:** This paper investigates the formation of oxygen precipitates in Czochralski-grown (CZ) silicon wafers and their thermal stability during high-temperature manufacturing steps. The study focuses on the dissolution dynamics of oxygen nuclei formed at 700° and 800° C when subjected to subsequent heat treatments ranging from 950° and 1200° C. Experimental data demonstrate that the stability of oxygen clusters is fundamentally determined by their size relative to the critical radius at high temperatures. It was observed that high processing temperatures exert a limiting effect on the density of internal gettering sites, leading to an exponential decay of precipitate concentration as temperature increases. The research establishes an empirical relationship between the maximum precipitate density and the reciprocal of the process temperature. These findings are crucial for optimizing thermal cycles in semiconductor device processing to ensure effective metallic impurity gettering. The results highlight the

competition between precipitate growth and dissolution mechanisms in the silicon matrix.

**Introduction.** Sequences of low-temperature and high-temperature silicon manufacturing steps are widely used to create internal gettering sites during device processing. Low-temperature annealing, typically between 600° and 800° C, increases the oxygen nucleus density in the wafer bulk [1]. Several papers published in the literature describe the effects of low-temperature anneals and how they can be optimized for internal gettering purposes [2–4]. It has been found that the formation of oxygen nuclei, commonly referred to as nucleation, depends not only on the nucleation processing conditions but also mainly on the interstitial oxygen content [5] and the previous thermal history of the wafer [6]. Then, in subsequent high-temperature steps, the oxygen nuclei are converted into gettering sites for metallic impurities [7]. The growth of the deposit volume can be well described by diffusion-limited growth [8, 9]. Furthermore, the oxygen cluster must exceed the critical volume characteristic of the growth temperature to avoid dissolution during heat treatment. Only larger clusters grow by agglomeration of oxygen atoms to convert them into absorbents for metallic impurities. Very few studies have systematically examined the change in the concentration of oxygen precipitates in the wafer bulk upon heating to temperatures exceeding 1000°C. Of practical interest is the study of whether high process temperatures themselves have a limiting effect on the density of internal gettering centers. The aim of this work is to investigate the effect of high process temperatures on oxygen nuclei formed at 700 and 800° C, respectively.

**Materials and methods.** All experimental data were obtained on 100 mm phosphorus-doped Czochralski silicon (CZ) wafers with <100> orientation and resistivity greater than 2 Ω cm. All wafers were cut from the same location in the crystal to minimize the influence of thermal history and then processed using standard wafer fabrication technology. The initial oxygen content was measured using Fourier transform (FT-UT). The spectral density was found to be in a very narrow range from 7.75 x 10 17 to 8.00 x 10 17 atoms/cm3 using an IFS-113v infrared spectroscopy system from Bruker. The wafers were pre-annealed in a conventional oven at 700 or 800 ° C for periods ranging from 30 min to 6 h under nitrogen atmosphere. One set of wafers, the control group, was not pre-annealed. All samples were mounted and removed at the annealing temperature. The pre-annealed wafers and the control group were divided into several sets of samples, and each set was heat treated at one specific temperature from 950° to 50° C. The duration of this high-temperature cycle was typically 10 h. Annealing at 1150° C lasted only 8 h. Except for 950° C, the wafers were placed in an oven at 1000° C in a dry oxygen environment. After the oven was allowed to stabilize at 1000° C, the temperature was raised to

the final annealing temperature at a rate of 10° C/min. During the initial stage of the high-temperature annealing, a thin oxide layer was grown to protect the surface, while the remainder of the wafer heat treatment was carried out under a nitrogen atmosphere. At the end of the heat treatment, the temperature was raised to 1000° C, at which point the wafers were removed. Another batch of phosphorus-doped <100> wafers was pre- annealed at 700° C for 4 h and then processed using the simulated CMOS process, the conditions of which are given in Table I. After heat treatment, the entire oxide layer was removed from the surface using hydrofluoric acid boiling. The residual oxygen concentration was re-measured, and a reduction in interstitial oxygen concentration was determined.

The oxygen precipitation density in the wafer bulk was estimated by decorative etching of the cleaved cross-section with Secco solution for 2.5 minutes. The defect density was measured at the same location where the FTIR measurements were performed by counting defects under an optical microscope at 900x magnification. The number of defects was then converted to bulk density by dividing the surface density by the amount of material removed during decorative etching.

**Results and discussion.** Figure 1 shows a graph of the change in the interstitial oxygen content as a function of high-temperature annealing. The lower curve of the figure shows the amount of released oxygen in samples pre-annealed at 700 ° C for 1 h, and the two upper curves represent data for long-term pre-annealing at 700 ° C and 1 h at 800 ° C. The presented values clearly show that the amount of released oxygen decreases with an increase in the annealing temperature from 950 ° C to 1100 ° C. Although the increase in release should occur faster at higher temperatures [9], the decrease in oxygen release with temperature indicates significant dissolution of oxygen clusters at higher temperatures.

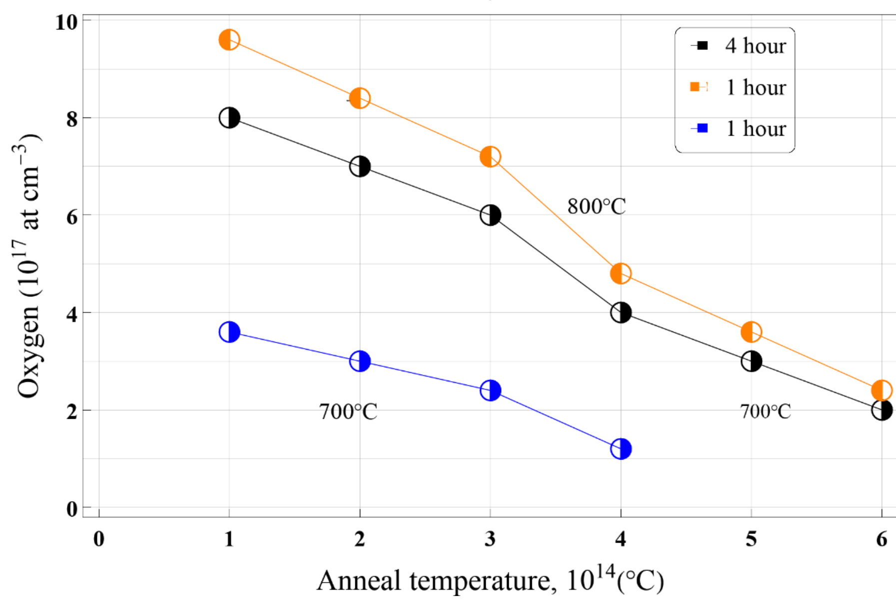

**Fig. 1. Interstitial oxygen concentration as a function of pre-annealing time at 650-1200° C, measured after a subsequent high-temperature cycle.**

Analysis of the bulk defect density shows that oxygen clusters formed at 700°C or 800° C do indeed dissolve at higher temperatures, as shown in Fig. 2. The density of precipitates decreases by orders of magnitude with increasing temperature. In fact, all nuclei formed within 1 h at 700° C are completely dissolved after a 10-h cycle at 1050° C, and their density is equal to the number of precipitates observed in the control wafers that were not subjected to nucleation.

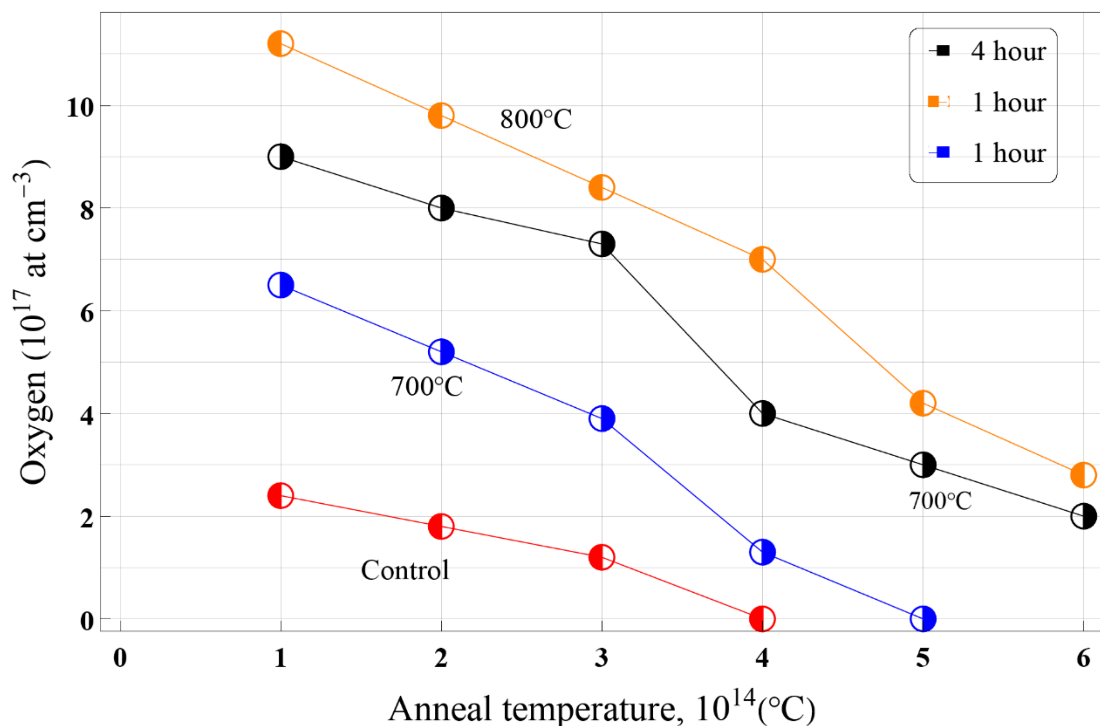


**Fig. 2. Interstitial oxygen concentration as a function of pre-annealing time at 560-1200° C, measured after a subsequent high-temperature cycle.**

The graph in Figure 2 also shows that both increasing the time and temperature of the nucleation cycle increases the precipitate density manifold, as expected from wafer processing in this temperature range. However, the concentration quickly drops again to a fraction of the number observed at lower processing temperatures.

The combined results of Figs. 1 and 2 indicate that the stability of oxygen clusters depends on the actual cluster size at the end of the nucleation treatment. It is evident that nuclei created at 700° C dissolve faster, while the higher oxygen diffusion coefficient at 800° C effectively enhances survival at temperatures above 1000° C.

The effect of cluster size on stability at high processing temperatures was further studied by increasing the annealing time at 800 ° C. It can be expected that the cluster size will increase significantly during annealing at 800 ° C and, consequently, a higher amount of oxygen evolution should be observed after the high-temperature cycle. Figure 3 shows the decrease in the amount of interstitial oxygen as a function of annealing time at 800 ° C with high temperature as a parameter. Initially, the amount of evolved oxygen increases with time, but then it reaches a plateau characteristic of each high-temperature annealing. The degree of oxygen evolution decreases again with increasing heat treatment temperature. At 1050 ° C, the drop in the amount of interstitial oxygen even decreases with annealing time at 800 ° C, indicating a lag phenomenon. Reports in the literature attribute this to the bulk placement of the evolution in the silicon lattice [10].

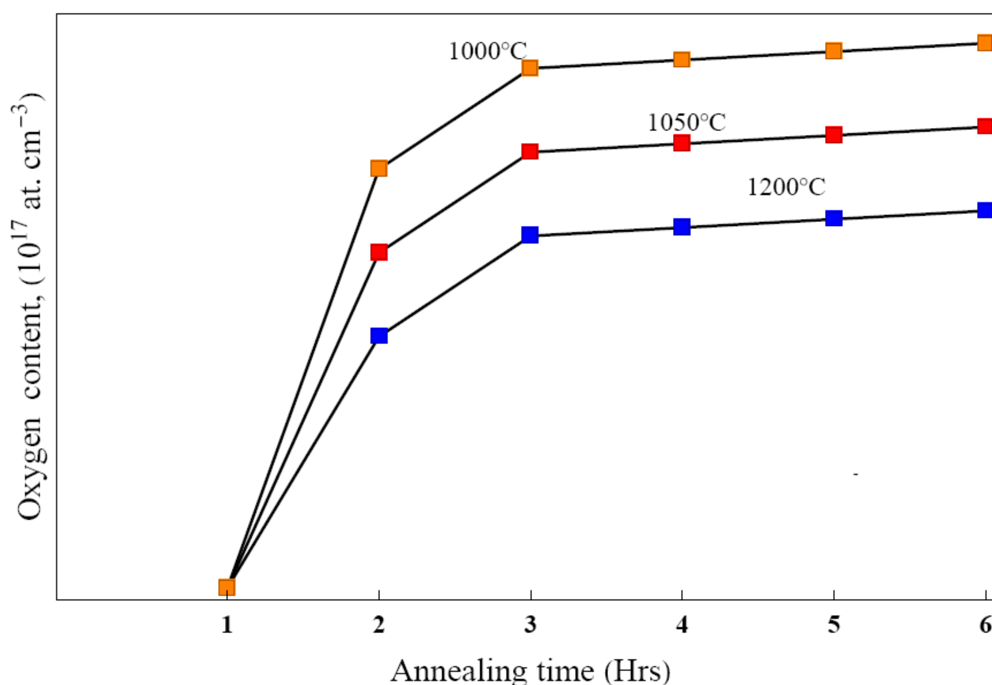


**Fig. 3. Reduction in interstitial oxygen content as a function of pre-annealing time at 800° C, measured after a subsequent high-temperature cycle.**

The plot of the bulk density of the precipitates versus the nucleation time at 800° C again shows a monotonic decrease in density with high-temperature treatment, as shown in Fig. 4. At 1000° C, a concentration of approximately 10 N $cm^{-3}$ is determined. At high temperatures, the density initially increases with nucleation time, as at 1200° C. However, at longer times, the volume defects reach a maximum [O_i]~3.8·10^10 cm^(-3)at 1050°C and then saturate after [O_i]~2.4·10^10 cm^(-3)4 h of nucleation, as shown in Fig. 4.

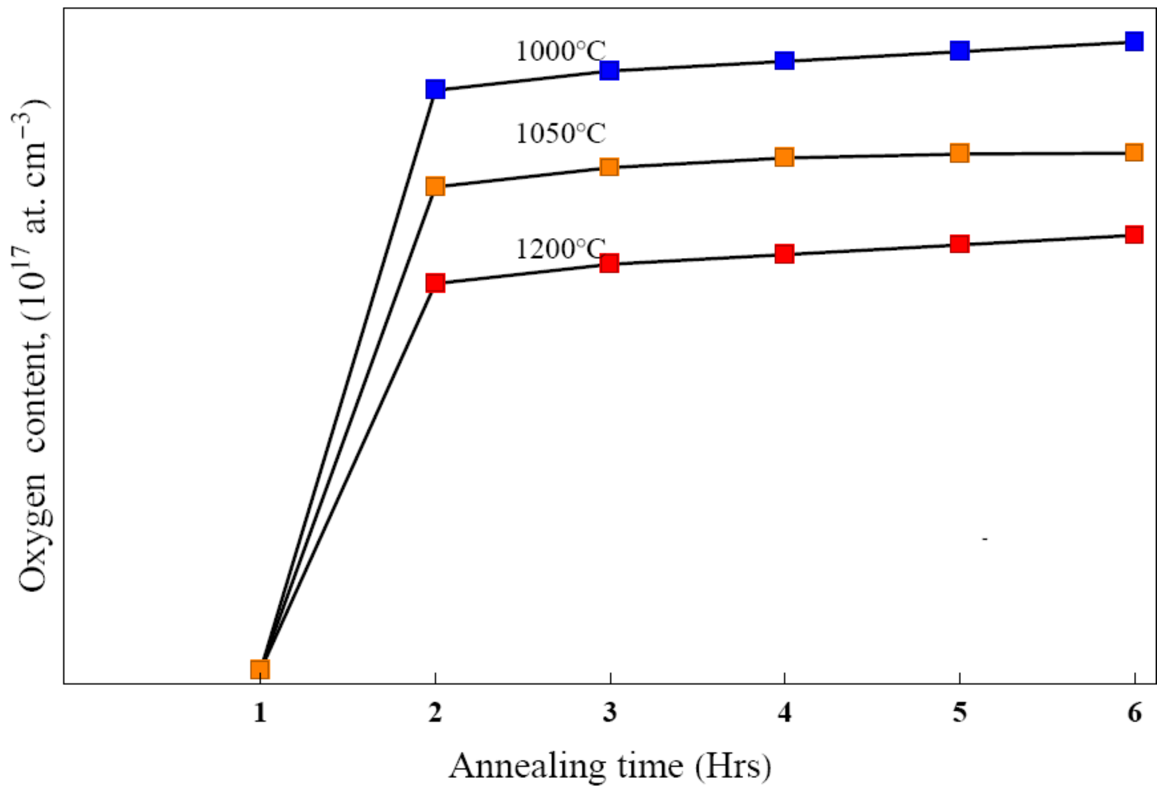


**Fig. 4. Density of interstitial oxygen percipitates as a function of pre-annealing time at 800° C, measured after a subsequent high-temperature cycle.**

If annealing at 800° C is followed by precipitate growth at 1100° C, the measured density is in the range of 10 -10 cm -3. A cycle at 1150° C further dissolves the oxygen clusters to a level of [O_i]~3.8·10^9 cm^(-3), regardless of the nucleation conditions. The data show that only a fraction of the oxygen nuclei formed at 800° C survive at the high process temperature, and the duration of the nucleation cycle does not have a significant effect. If the initial size of the oxygen clusters determines the number of stable precipitates, the precipitate density should increase with increasing nucleation time. Instead, saturation of the precipitate density is observed at longer nucleation times. Saturation rather indicates that the concentration of internal gettering sites is limited by the density characteristic of the higher annealing temperature. If the fraction of precipitated oxygen reaches a value of unity, then complete precipitation occurs. At 1050° C and below, oxygen precipitates near the solid solubility limit. Lifshitz et al. demonstrated that precipitate nuclei coalesce as the solid solubility limit is approached [11]. This phenomenon is easily understood if we assume that the critical radius increases sharply in solid solubility with increasing temperature, and only large precipitates with a radius exceeding the critical radius survive.

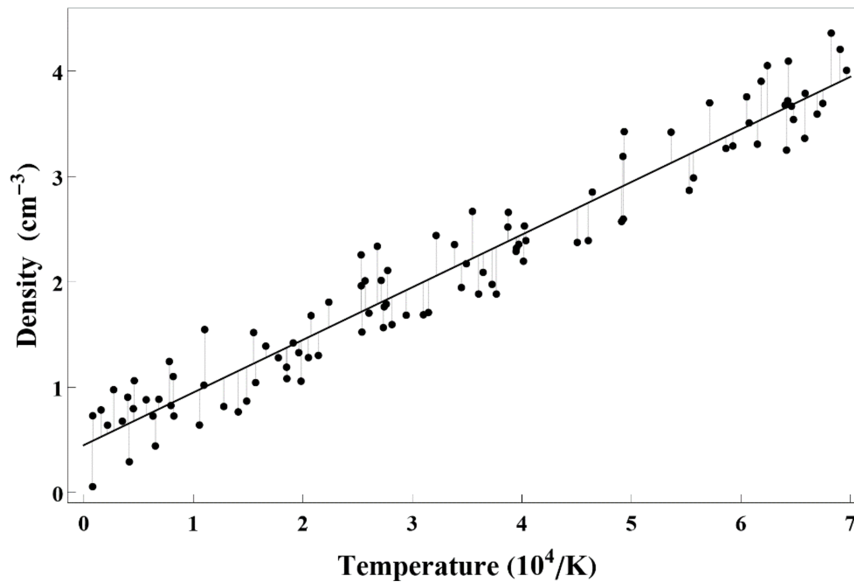


**Fig. 5. Density of peripatites as a function of nucleation time at 800°C, observed after high-temperature annealing.**

Although we observe an exponential decay of precipitate nuclei with increasing temperature, as shown in Fig. 5, the final oxygen content at higher temperatures is not close to the solid solubility reported in the literature [12]. Therefore, it is unclear what mechanism is driving the phenomenon observed here. Figure 5 shows a semilog plot of the precipitate density from Table II versus the reciprocal of the precipitation temperature. The data fit a straight line, and the error bars represent one standard deviation of the data points. Fitting the data leads to an empirical relationship of the form

$$[N_i]\approx 0.02\cdot \exp(3.14 eV/(k_B T))\, cm^{-3}.$$

where is $k_B$the Boltzmann constant, and T the temperature. This empirical relationship expresses the fact that the maximum density Nmax of precipitation nuclei or internal gettering sites is essentially determined by the highest temperature to which the wafer is exposed, especially over long cycle times.

**Conclusion.** The obtained results show that during long high-temperature cycles, temperature significantly influences the dynamics of oxygen precipitation. It was observed that during short anneals at 650-700° C, clusters quickly dissolve at higher temperatures due to insufficient size. Increasing the annealing time at 700° C and raising the temperature to 800-1200° C increases the number of stable oxygen precipitates. Analysis of the bulk density of precipitates after heat treatment confirms that the maximum measured density of internal gettering centers in CZ silicon is determined by the highest process temperature to which the wafer is subjected.

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