NAMANGAN DAVLAT UNIVERSITETI
ILMIY AXBOROTNOMASI, [2026-9]
ISSN:2181-1458
ISSN:2181-0427

01.00.00-FIZIKA-MATEMATIKA
journal.namdu.uz
PHYSICS AND MATHEMATICS

UDC 621.315.592

EFFECT OF TRADITIONAL AND RAPID THERMAL (TWO-STAGE) ANNEALING ON OXYGEN PRECIPITATES IN SILICON

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Abstract: *This study investigates the effects of conventional and rapid thermal annealing on the formation and dissociation of oxygen precipitates in monocrystalline and polycrystalline silicon samples. The concentrations of interstitial oxygen and substitutional carbon were determined using Fourier Transform Infrared Spectroscopy (FTIR). The results demonstrate that different heat treatment regimes significantly influence oxygen precipitation, precipitate dissolution, and the overall properties of silicon materials. Slow cooling conditions were found to promote oxygen precipitate formation, whereas rapid thermal annealing induced the dissociation of existing precipitates. The obtained findings contribute to a better understanding of oxygen diffusion processes in silicon, the mechanisms of thermal donor formation, and the evolution of oxygen-related clusters. These results are important for optimizing thermal processing conditions and improving the performance of silicon-based semiconductor devices.*

Keywords: *monocrystalline silicon, polycrystalline silicon, interstitial oxygen, substitutional carbon, rapid thermal annealing, conventional annealing, oxygen precipitation, FTIR spectroscopy, oxygen diffusion, thermal donors, oxygen clusters, precipitate dissociation.*

AN'ANAVIY VA TEZKOR TERMIK (IKKI BOSQICHLI) TAVLANISHNING KREMNIYDAGI KISLOROD CHO'KMALARIGA TA'SIRI

Annotatsiya: *Ushbu tadqiqotda monokristall va polikristall kremniy namunalarida an'anaviy hamda tezkor termik tavlashning kislorod cho'kmalari hosil bo'lishi va dissotsiatsiyasiga ta'siri o'rganildi. Namunalardagi interstitsial kislorod va o'rinbosar uglerod konsentratsiyalari Fourier transform infraqizil spektroskopiyasi (FTIR) yordamida aniqlandi. Issiqlik bilan ishlov berishning turli rejimlari kislorod cho'kmalarining shakllanishi, erishi va materialning xossalariga sezilarli ta'sir ko'rsatishi aniqlandi. Sekin sovitish kislorod cho'kmalari hosil bo'lishini rag'batlantirishi, tezkor termik tavlash esa ayrim cho'kmalarning dissotsiatsiyasiga olib kelishi kuzatildi. Tadqiqot natijalari kislorodning kremniydagi diffuziya jarayonlari, termik donorlarning hosil bo'lish mexanizmlari hamda kislorod klasterlarining shakllanish xususiyatlarini chuqurroq tushunishga xizmat qiladi.*

Kalit so'zlar: *monokristall kremniy, polikristall kremniy, interstitsial kislorod, o'rinbosar uglerod, tezkor termik tavlash, an'anaviy tavlash, kislorod cho'kmalari, FTIR spektroskopiyasi, kislorod diffuziyasi, termik donorlar, kislorod klasterlari, dissotsiatsiya.*

ВЛИЯНИЕ ТРАДИЦИОННОГО И БЫСТРОГО ТЕРМИЧЕСКОГО (ДВУХЭТАПНОГО) ОТЖИГА НА КИСЛОРОДНЫЕ ПРЕЦИПИТАТЫ В КРЕМНИИ

Аннотация: *В данной работе исследовано влияние традиционного и быстрого термического отжига на процессы образования и диссоциации кислородных преципитатов в монокристаллическом и поликристаллическом кремнии. Концентрации межузельного кислорода и замещающего углерода определялись методом Фурье-инфракрасной спектроскопии (Фурье-ИК спектроскопии). Установлено, что различные режимы термической обработки существенно влияют на формирование и растворение кислородных преципитатов, а также на свойства кремниевого материала. Медленное охлаждение способствует образованию кислородных преципитатов, тогда как быстрый термический отжиг приводит к диссоциации части существующих преципитатов. Полученные результаты расширяют представления о процессах диффузии кислорода в кремнии, механизмах формирования термических доноров и образовании кислородных кластеров. Результаты исследования могут быть использованы для оптимизации технологических процессов изготовления полупроводниковых приборов.*

Ключевые слова: *монокристаллический кремний, поликристаллический кремний, межузельный кислород, замещающий углерод, быстрый термический отжиг, традиционный отжиг, кислородная преципитация, Фурье-ИК спектроскопия, диффузия кислорода, термические доноры, кислородные кластеры, диссоциация преципитатов.*

Introduction

The study of oxygen precipitates in silicon continues to be a subject of considerable interest today. It has been extensively investigated due to its role in strengthening the silicon lattice and in the gettering of impurities during device processing [1]. In general, the behavior of interstitial oxygen precipitates in silicon is determined by the interstitial oxygen concentration and the processing temperatures, especially the supersaturated oxygen content at these temperatures. Secondary factors are related to the impurity content, defects, and thermal history of the crystal [2]. Many fundamental aspects of the nucleation and growth of oxide precipitates have been studied, but the amount and morphology of oxygen precipitates represent a very complex issue. To date, the interactions between oxygen, carbon, and metallic impurities, point defects, and aggregates responsible for changes in the electrical properties of silicon during heat treatment have not been fully elucidated [3].

In this study, we compared the effects of traditional and rapid thermal annealing on interstitial oxygen precipitates in various silicon materials. The goal of this experiment is to understand the evolution of oxygen concentration and the mechanism of its precipitation in bulk silicon.

Literature Review

Oxygen precipitation in silicon has been extensively investigated because of its influence on the structural and electrical properties of semiconductor materials and its importance in the internal gettering of metallic impurities. Previous studies have demonstrated that the formation and evolution of oxygen precipitates depend on the interstitial oxygen concentration, thermal processing conditions, impurity content, and the thermal history of the crystal [1, 2]. Oxygen-related defects are particularly important in silicon produced by the Czochralski method, in which relatively high concentrations of interstitial oxygen can be incorporated into the crystal during growth.

Previous investigations have addressed the mechanisms of oxygen precipitation, the interaction of oxygen with other impurities, and the influence of thermal treatment on the properties of silicon. Studies by Hu and Patrick [4], as well as Newman and Willis [5], provide a basis for examining oxygen-related processes and impurity behavior in silicon. In addition, research on oxygen precipitation and defect interactions has highlighted the complexity of the relationships between oxygen, carbon, metallic impurities, and lattice defects [3]. These interactions may influence the distribution of oxygen-related defects and the electrical characteristics of the material during subsequent thermal processing.

The thermal history of silicon plays a significant role in determining the formation, growth, and dissolution of oxygen precipitates. Conventional thermal annealing with controlled heating and slow cooling can promote oxygen precipitation under appropriate conditions, whereas rapid thermal processing can modify the existing precipitate population through dissolution or dissociation processes [6]. The resulting changes in interstitial oxygen concentration provide useful information for evaluating the response of silicon to different thermal treatment regimes. However, the interpretation of these changes requires consideration of the initial oxygen concentration, treatment temperature, duration, and cooling rate.

Despite considerable progress in understanding oxygen-related defects in silicon, the mechanisms governing oxygen aggregation, thermal donor formation, and oxygen diffusion remain complex. In particular, the interactions between oxygen-containing complexes and other impurities require further investigation [7, 8]. Although carbon is an important parameter in characterizing silicon materials, its independent contribution to oxygen precipitation cannot be established solely from the initial impurity concentrations.

Based on these considerations, the present study compares conventional annealing with slow cooling and rapid thermal annealing to investigate changes in interstitial oxygen concentration in monocrystalline and polycrystalline silicon samples. Fourier-transform infrared spectroscopy (FTIR) is used to evaluate interstitial oxygen and substitutional carbon concentrations. The study focuses on the relationship between thermal treatment conditions and the observed changes in oxygen concentration, providing experimental information relevant to understanding oxygen precipitation and possible precipitate dissolution in silicon.

Experimental procedure

In our experiments, we used monocrystalline and polycrystalline silicon samples. The polycrystalline material «P» was obtained from various ingots, «P,» designated by the letters 1, 2, 3, and 4 in the table and figures. The sample thicknesses ranged from 400 μm to 1 mm, and both surfaces were polished (double-sided polishing, DSP). Infrared absorption measurements at 1106 and 605 cm^{-1}, corresponding to the interstitial oxygen and substitutional carbon bands, respectively, were performed using a Perkin-Elmer 983G dual-beam infrared spectrophotometer. All spectra were recorded at room temperature using air as the reference beam, and calculations were performed using a computer station and PE-3600 software. The calibration coefficients were (3.03±0.19) $^{-}$ $10^{17}cm^{-2}$ for interstitial oxygen and (1.00±0.03) $10^{17}cm^{-2}$ for substitution carbon [4]. Particular care was taken to perform the infrared absorption measurements at exactly the same location within the samples to avoid the influence of possible non-uniform distribution of impurities [5]. Resistance was measured using a four-probe method. It should be noted that before each treatment, the samples were thoroughly rinsed in a 25% HF solution for 2 minutes. The first group of samples was subjected to successive isochronous 25-second heating steps from 450 to 1050°C, while the second group of samples was used to study oxygen dissociation and precipitates.

Research Methodology

The concentrations of interstitial oxygen and substitutional carbon found in the various materials used are summarized in Table 1, where the resistivity values are also given.

Table 1. Concentrations of interstitial oxygen and substitutional carbon			
Sample	Oi (ppma)	Ci (ppma)	ρ (Ω· cm)
1-CZ	12.0	<0.6	10
2	21.0	11.0	3.0
3	12.0	10.0	2.3
4	16.0	10.0	1.9
5	6.4	19.0	1.3

For the second point of our study, we attempted to confirm the process of precipitate formation and dissociation using two types of annealing. First, a traditional annealing with a slow cooling rate (2°C/min), which promotes precipitation formation, and second, a rapid annealing, characterized by a very high cooling rate (>300°C/s), which inherently causes a quenching effect. At this stage, dissolution of the precipitates and their dissociation can occur [6]. Figures 1 and 2 show the results of two-stage annealing. It is evident that the amount of oxygen precipitates after a slow cooling rate (2°C/min) varied significantly depending on the material used. For sample «1,» a significant decrease in oxygen concentration is observed after conventional annealing, confirming the presence of oxygen precipitates in this oxygen-rich sample. After subsequent annealing, some dissolution of these precipitates apparently occurs, which may explain the increase in intergranular oxygen concentration (Fig. 1).

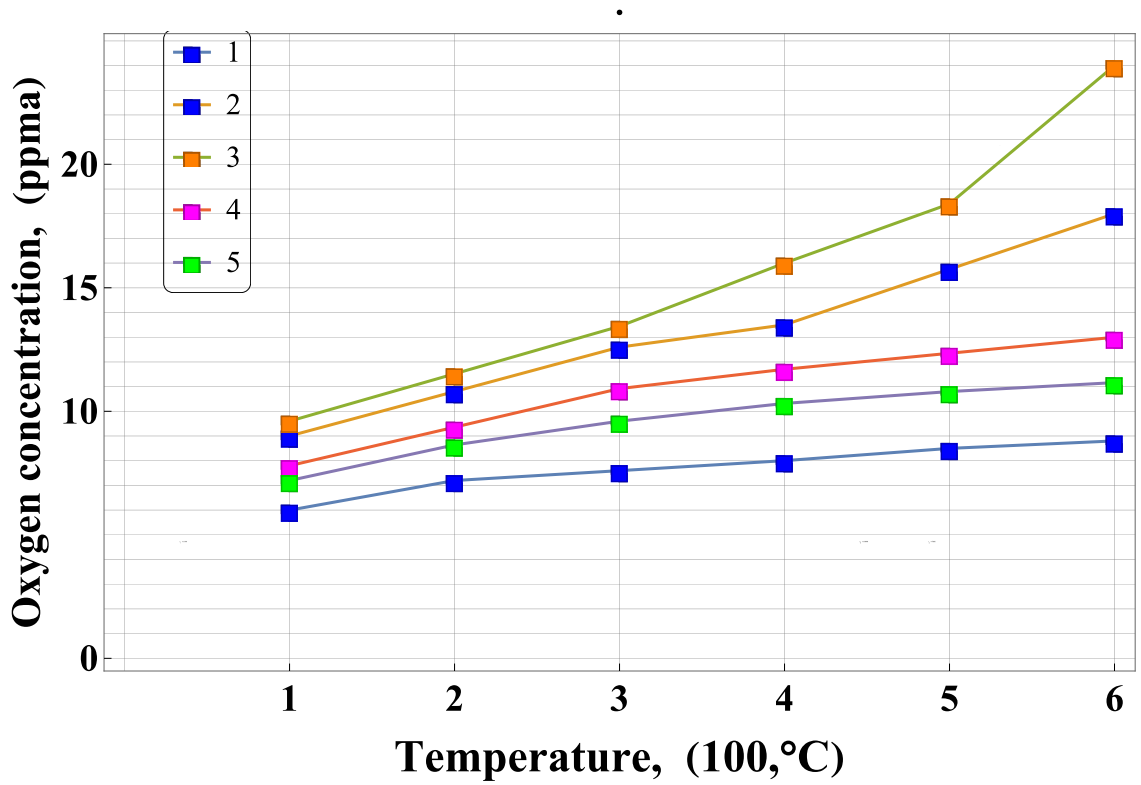

Fig. 1. Change in the concentration of interstitial oxygen depending on temperature during slow thermal annealing.

Thus, we observe a significant decrease in the [O_i] concentration after subsequent heat treatment, confirming the effectiveness of slow cooling in stimulating oxygen precipitates and likely other residual impurities. It should be noted that materials 1-4 have a large excess of oxygen compared to carbon; this type of material has already been shown to also exhibit significant recombination activity at grain boundaries [4]. Figure 2 shows the dependence of the interstitial oxygen content on temperature during rapid annealing, characterized by a very high cooling rate of 25 s. It can be seen that in sample 1, the oxygen concentration remains constant in the low-temperature range and increases at higher temperatures. When conducting a 25-second heat treatment at 1050 °C, we found an increase in the [O_i] content in materials 1-3, indicating that some precipitates may already exist at the initial stage and dissolve during heat treatment.

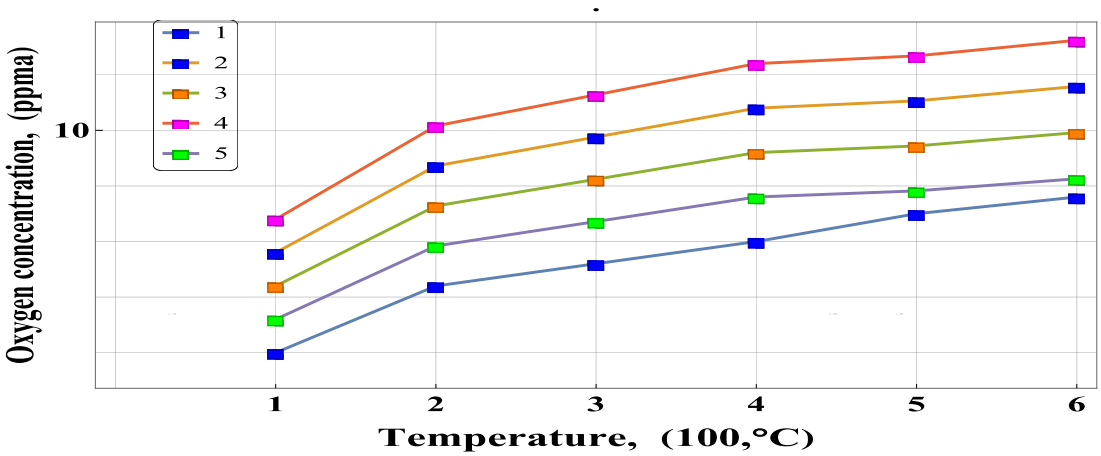

Fig. 1. Change in the concentration of interstitial oxygen depending on temperature during fast thermal annealing.

Results and Discussion

The results obtained to date can be summarized as follows: first, the effect of heat treatment on oxygen precipitation depends significantly on the thermal history of the material, the initial oxygen content, and the oxygen species present in the material. Second, rapid thermal annealing can lead to the dissociation of some oxygen precipitates present in the silicon material. This dissociation is accompanied by a deterioration in the bulk diffusion length of the material. It is well known that oxygen is present in high concentrations (~10 18 cm -3) both in the original crystalline silicon obtained by the Czochralski method (CzSi) and is effectively introduced into Si crystals during various high-temperature manufacturing operations. Although an electrically inactive impurity, oxygen can nevertheless significantly affect the material's characteristics. Oxygen precipitation has long been used for the internal gettering of rapidly diffusing metallic impurities. On the other hand, even short-term heat treatments of oxygen-containing Si crystals in the temperature range of 300-700°C lead to the formation of various types of electrically active centers (thermal donors, etc.). Such heat treatments, typically used in the final stages of the manufacturing process, can significantly alter the electrical properties of the material and, consequently, the performance characteristics of the devices produced. For this reason, the behavior of oxygen impurity atoms in silicon has been intensively studied for several years.

However, despite the large number of studies in this area (see, for example, [7, 8] and references therein) and the use of a wide variety of research methods, a number of key points still remain unclear. This applies primarily to the mechanism of thermal donor formation, as well as to the processes of direct and reverse oxygen diffusion at T < 700 °C. While in the temperature range T = 700-1300 °C the processes of oxygen mass transfer and precipitation are limited by the usual (normal) diffusion of single Oi atoms, the situation is more complex at T = 300-700 °C. In this temperature range, both the formation of oxygen agglomerates and the development of diffusion profiles occur at an accelerated rate and can only be described using an effective oxygen diffusion coefficient that exceeds the normal value by several orders of magnitude. It is assumed that this is due to the existence of rapidly diffusing oxygen-containing complexes. However, the nature of such complexes and the mechanism of accelerated oxygen diffusion have not yet been fully elucidated. Recently, two important results have been obtained that give hope that a solution to this problem can be found. First, local vibrational modes of a complex containing two oxygen atoms (oxygen dimer, O_{2i}) [7] and local vibrational modes of the first three centers from the family of doubly charged oxygen-containing thermal donors [8] were identified. In this work, the formation of small oxygen clusters in hydrogenated silicon is investigated using local vibrational mode spectroscopy with the aim of elucidating the mechanism of hydrogen-accelerated oxygen diffusion and clustering.

CONCLUSION

In conclusion, heat treatment and reverse annealing cycles proved to be very useful for identifying precipitation and dissolution effects. Possible causes for the variability in the response of these samples to heat treatment remain to be elucidated. The problem is very complex due to the presence of various defect reactions and the large number of parameters involved. Further, more detailed research is needed to address this issue.

ACKNOWLEDGMENT

We thank researchers of the laboratory Kyungwon university (within the framework foreign internship program) (Korea Republic) for assistance in obtaining some experimental results.

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