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Effect of rapid thermal processing on high temperature oxygen precipitation behaviour in Czochralski silicon wafer

Xiangyang Ma, Lei Lin, Daxi Tian, Liming Fu and Deren Yang[1]

State Key Laboratory of Silicon Materials, Zhejiang University, Hangzhou 310027, People's Republic of China

E-mail: mseyang@dial.zju.edu.cn

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Abstract

The effect of rapid thermal processing (RTP) on the oxygen precipitation occurring at 1050 °C in a Czochralski (CZ) silicon wafer has been investigated. It has been proved that the RTP-induced vacancies only enhance the early stage oxygen precipitation at 1050 °C in terms of the precipitation rate. Furthermore, it is somewhat unexpected that after a lengthy 1050 °C anneal the oxygen precipitates generated in the CZ silicon wafer with prior RTP treatment had considerably lower density and larger sizes in comparison with those generated in the CZ silicon wafer without prior RTP treatment. The reason for this is that the prior RTP treatment will dissolve some of the grown-in oxygen precipitates, thus making the RTP-treated wafer possess fewer nuclei contributing to oxygen precipitation in the subsequent 1050 °C anneal. Moreover, the numbers of resulting precipitated oxygen atoms due to a lengthy 1050 °C anneal were nearly the same in the CZ silicon wafers with and without prior RTP treatment. Additionally, it has been illustrated that the high temperature RTP has superior capability to dissolve the existing oxygen precipitates. It is worthwhile to point out that, when addressing the effect of RTP on the oxygen precipitation behaviour during the subsequent anneal, two functions arising from the RTP treatment, that is, the injection of vacancies into the silicon wafer and the dissolution of grown-in oxygen precipitates existing in the silicon wafer, should be taken into account.

[1] Author to whom any correspondence should be addressed.

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1. Introduction

Rapid thermal processing (RTP) has been intensively employed in modern microelectronic device fabrication such as post-implant anneals [1, 2], thermal oxidation [3, 4], and shallow junction formation [5]. Over the years, the presentation of the magic denuded zone (MDZ) concept that is believed to be a milestone in the defect engineering of silicon wafers has exploited the application of RTP in the manufacture of silicon wafers [6, 7]. The underlying idea of the MDZ concept is to apply RTP for the introduction of a vacancy concentration profile increasing from the surface to the bulk of the wafer crossing the critical concentration at some desired depth, and the installed vacancies have full control over the oxygen precipitate profile of the wafer to form an ideal intrinsic gettering structure consisting of a bulk micro-defect (BMD) region and a precipitate-free zone (PFZ) [8]. From the basic science point of view, the effect of RTP on the oxygen precipitation behaviour in the CZ wafer in the subsequent anneal is an issue worthy of investigation, because the prior RTP treatment, on one hand, will install vacancies of a certain concentration to change the thermodynamics and kinetics of oxygen precipitation. On the other hand, it will eliminate the thermal history of the wafer to some extent, which certainly affects the oxygen precipitation behaviour in the subsequent heat treatment. Generally, for the creation of MDZ, a two-step (low–high) anneal is employed following the high temperature RTP. So far, the effect of RTP at different high temperatures for different times on oxygen precipitation during the subsequent two-step anneal has been investigated in detail, and it has been proved that the RTP-induced vacancies enhance oxygen precipitation [9–11]. The enhancement of oxygen clustering, i.e. nucleation of oxygen precipitation by RTP-induced vacancies at temperatures lower than 900 °C, has been well documented [8]. It is commonsense that at elevated temperatures such as 1050 °C the growth of oxygen precipitates prevails over the nucleation of oxygen precipitation. How the RTP-induced vacancies affect oxygen precipitation occurring at elevated temperatures is still a matter worthy of note. Herein, we have investigated the oxygen precipitation behaviour of a CZ wafer subjected to an extended anneal at 1050 °C following the high temperature RTP, the concerns of which include how the prior RTP treatment affects the oxygen precipitation kinetics, the amount of resultant precipitated oxygen, and the density of oxygen precipitates.

2. Experimental details

One piece of 200 mm, ⟨100⟩ oriented, boron-doped, 670 μm thick silicon wafer with a resistivity of about 10 Ω cm was used for the experiment. One quarter of the wafer was subjected to a standard wet oxidation process and then preferentially etched by Schemmel etchant; as a result, no oxidation-induced-stacking fault ring (OSF ring) was found, indicating that the selected silicon wafer was of vacancy type. For the subsequent thermal treatments, four neighbouring slices were taken from the above-mentioned silicon wafer as the experimental samples, designated as samples A, B, C, and D respectively. The initial oxygen concentrations ($[O_i]$s) for these four samples were nearly the same, 1.03×10^{18} cm^{-3}, measured by Fourier transformation infrared (FTIR) spectroscopy using a conversion factor of 3.14×10^{17} cm^{-2}. It can be believed that these samples have the same thermal history. Samples A and B were first subjected to the RTP treatments at 1250 and 1200 °C for 50 s respectively, in which, on average, the temperature ramping rate was 50 °C s^{-1} and the cooling rate to 600 °C was 30 °C s^{-1}. Subsequently, samples A, B, and C were subjected to an extended anneal at the elevated temperature of 1050 °C. It should be mentioned that the samples were directly loaded into the furnace at 1050 °C. During the anneal, at different times the samples were withdrawn from the furnace to measure the $[O_i]$ by FTIR. Moreover, after the 1050 °C/128 h anneal,

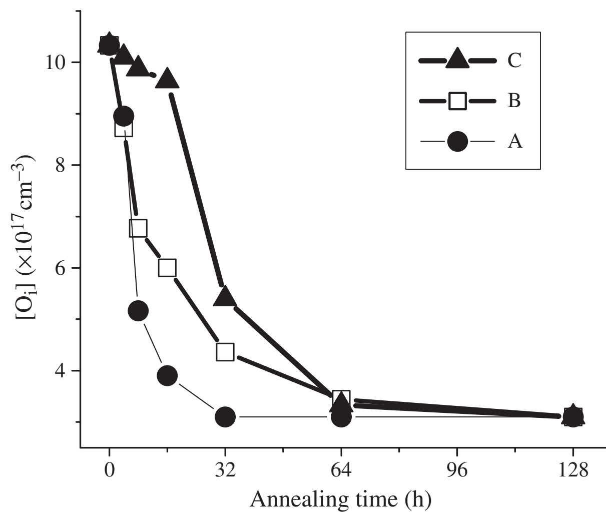

Figure 1. Interstitial oxygen concentration ($[O_i]$) in samples A, B, and C as a function of annealing time at 1050 °C. Samples A and B were respectively subjected to prior RTP treatments at 1250 °C and 1200 °C for 50 s.

samples A, B, and C were covered with wax on both sides and then cleaved, followed by a preferential etching for 3 min in Schemmel etchant. Subsequently, an Olympus MX-50 optical microscope (OM) was employed to take representative pictures of the bulk micro-defect (BMD) profiles within the samples.

In order to investigate the effect of RTP on the dissolution of grown-in oxygen precipitates existing in samples A and B, due to the difficulty in the measurement of the variation of $[O_i]$ in samples A and B before and after RTP by FTIR, we performed a 'magnified' experiment as follows: sample D as mentioned above was first subjected to a two-step anneal (650 °C/16 h plus 1000 °C/16 h) to generate oxygen precipitates, then was split into two pieces labelled D1 and D2. Sample D2 was further subjected to 1250 °C/50 s RTP, while sample D1 was not. Likewise, the $[O_i]$ and BMDs in sample D (D1 and D2) sequentially subjected to the two-step anneal and RTP as described above were respectively characterized by FTIR and OM. It should be mentioned that all the heat-treatments performed in the experiment were carried out in an argon ambient.

3. Results and discussion

Figure 1 illustrates the variation of $[O_i]$ in samples A, B, and C subjected to 1050 °C anneal for different times. As can be seen, during the first 32 h anneal, the reduction of $[O_i]$ in samples A and B was much more significant than that in sample C, and furthermore the decrease of $[O_i]$ in sample A was the most pronounced. It should be noted that prior to the 1050 °C anneal the vacancy concentration was the highest in sample A among the three samples. According to the fact mentioned above, it is believed that the vacancies introduced by RTP remarkably accelerate oxygen precipitation at elevated temperature in CZ silicon, which is positively dependent on the vacancy concentration. Of note is that the $[O_i]$s in samples A, B, and C became nearly the same after 1050 °C/64 h anneal, and afterwards they did not change any more, indicating that oxygen precipitation at 1050 °C in the three samples reached equilibrium after 64 h anneal. Moreover, figure 1 indicates that in sample C the $[O_i]$ decreased

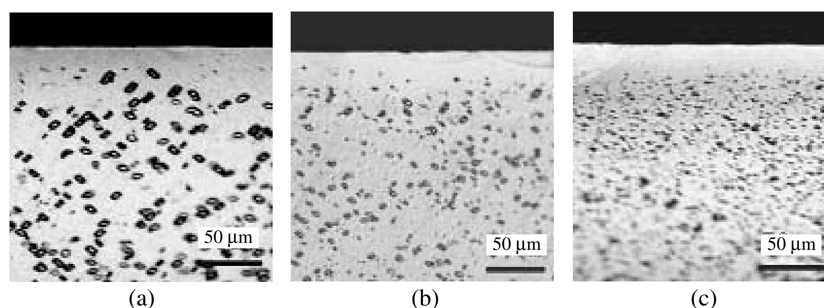

Figure 2. Representative cross-sectional pictures of bulk micro-defects (BMDs) arising from the oxygen precipitates generated in the samples subjected to 1050 °C/128 h. (a) Sample A with a prior 1250 °C/50 s RTP treatment, (b) sample B with a prior 1200 °C/50 s RTP treatment, and (c) sample C without a prior RTP treatment.

slightly during the early 16 h anneal but decreased dramatically afterwards, suggesting that the incubation of oxygen precipitation occurred in the early 16 h anneal. From the oxygen precipitation behaviour of samples A and B, it is explicit that the introduction of vacancies eliminated the incubation stage of oxygen precipitation. Therefore, strictly speaking, it was the early stage of oxygen precipitation at elevated temperature that was enhanced by vacancies. It has been documented that the vacancies and oxygen could combine into O_2V species at the temperatures 1020–1070 °C [12, 13]. Consequently, for samples A and B, during the cooling down after RTP, oxygen and vacancies combined into O_2V species. Moreover, even if the combination of oxygen and vacancies could not be finished during the cooling down after RTP, it would continue at the very early stage of the subsequent 1050 °C anneal. Because the O_2V species could provide space to relieve the strain arising from the growth of oxygen precipitates, the early stage oxygen precipitation was significantly enhanced at a price of depletion of O_2V species. Therefore, during the subsequent anneal, there were hardly any differences among samples A, B, and C. Once the 1050 °C anneal was long enough, the three samples had nearly the same numbers of resultant precipitated oxygen atoms.

Figure 2 shows the representative cross-sectional pictures of BMDs generated in samples A, B, and C subjected to 1050 °C/128 h anneal. As revealed in figure 1, the three samples possessed nearly the same numbers of precipitated oxygen atoms; nevertheless, as illustrated in figure 2, the densities of BMDs pertaining to the oxygen precipitates and their extended defects formed in samples A, B, and C in turn increased from about 1.8×10^9, 3.2×10^9, to 8.0×10^9 cm^{-3}. It is obvious that the super-saturation of O_i at the elevated temperature of 1050 °C is small so that the fresh oxygen precipitate nuclei are difficult to generate. Therefore, the oxygen precipitation in sample C was primarily based on the grown-in oxygen precipitates. As for samples A and B, it is evident that the concentration of RTP-induced vacancies was higher in sample A than in sample B. Therefore, prior to the prolonged 1050 °C anneal, the concentration of O_2V species was higher in sample A than in sample B. However, as revealed in figure 2, sample A had lower BMD density than sample B. Accordingly, it is reasonably believed that the O_2V species did not aggregate further to create fresh oxygen precipitate nuclei but contribute to the growth of existing oxygen precipitates during the early stage 1050 °C anneal. Moreover, of interest is that the widths of PFZ in samples A, B, and C were nearly the same. It thus seems that the prior RTP has no effect on the formation of PFZ in samples A and B. However, if samples A and B were subjected to a two-step anneal such as 800 °C/4 h plus 1050 °C/16 h, a PFZ as wide as the vacancy depletion region generated during

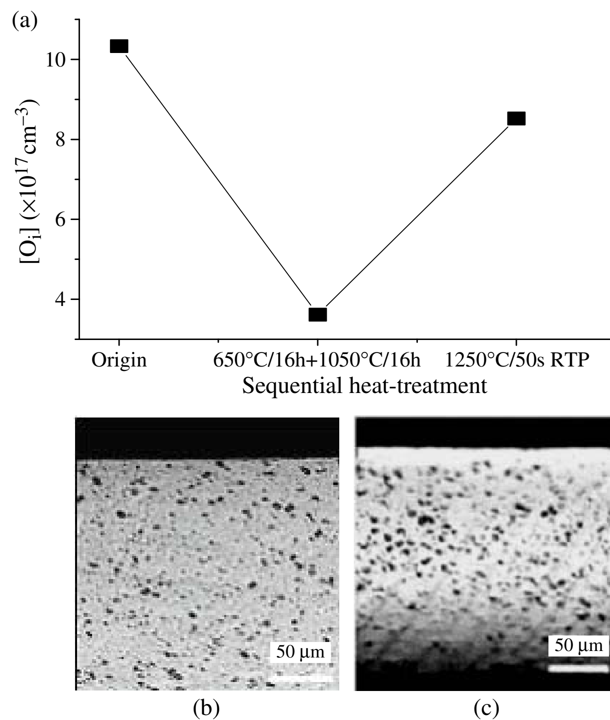

Figure 3. (a) Variation of interstitial oxygen concentration ($[O_i]$) in sample D (D1 and D2) subjected to the sequential anneal of 650 °C/16 h + 1000 °C/16 h and 1250 °C/50 s RTP, (b) representative cross-sectional picture of bulk micro-defects (BMDs) in sample D1 subjected to two-step anneal of 650 °C/16 h + 1000 °C/16 h, and (c) representative cross-sectional picture of bulk micro-defects (BMDs) in sample D2 subjected to 1250 °C/50 s RTP following two-step anneal of 650 °C/16 h + 1000 °C/16 h.

RTP would be formed in these samples. Therefore, the anneal scheme subsequent to RTP is essential to the width of PFZ. If the two-step anneal mentioned above is employed, after the 800 °C/4 h anneal, an oxygen precipitate nuclei profile that is almost geometrically the same as the vacancy profile created by RTP is formed [8]. Consequently, the width of PFZ formed by the two-step anneal is nearly the same as that of the vacancy depletion region, while in the present work no low step anneal of 800 °C/4 h anneal other than the high temperature anneal was employed for samples A and B. Thus, the oxygen precipitate nuclei profile determined by the RTP-created vacancy profile was not formed in samples A and B. Furthermore, during the 1050 °C anneal, the O_2V species could also move outward. As a result, the width of PFZ in samples A and B only subjected to 1050 °C anneal was not determined by the initial vacancy profile but most likely determined by the out-diffusion of oxygen.

Regarding the reason why the BMD densities of samples A, B, and C in turn increased, it is worthwhile to evaluate the RTP effect on the dissolution of existing oxygen precipitates. The significant effect of RTP on the dissolution of oxygen precipitates is essentially reflected in figure 3(a), in which the formation of oxygen precipitates by a two-step anneal (650 °C/16 h + 1000 °C/16 h) and the partial dissolution of the above oxygen precipitates due to a subsequent 1250 °C/50 s RTP occurring in sample D are illustrated. Figures 3(b) and (c) are respectively the cross-sectional pictures of BMDs in sample D1 subjected to the two-step anneal (650 °C/16 h + 1000 °C/16 h) and of those in sample D2 subjected to RTP

(1250 °C/50 s) following the two-step anneal as mentioned above. It is evident that after the RTP the shallow pits of BMD disappeared to a considerable extent, indicating that the oxygen precipitates formed by the prior two-step anneal were dissolved to a considerable extent by the subsequent RTP. Furthermore, it seems that figure 3(c) reveals a PFZ of a certain width, because the oxygen precipitates in the vicinity of the wafer surface were dissolved. The plausible mechanism for the rapid dissolution of oxygen precipitates caused by RTP is described below. The dissolution of oxygen precipitates is quite different from the growth of oxygen precipitates; the latter is controlled by oxygen diffusion and therefore is strongly time dependent, while the former is determined by the breakage of Si–O bonds, which is most likely an instantaneous event. During the RTP, the rapid increase to high temperature enabled the oxygen precipitates to expand violently, thus exerting great strain that was beyond the strength of Si–O bonds. Ultimately, a considerable concentration of oxygen atoms were dissociated from the oxygen precipitates because of the breakage of Si–O bonds. Evidently, the smaller oxygen precipitates were readily dissolved by RTP, and moreover more oxygen precipitates would be dissolved by higher temperature RTP. In view of the above analysis, it is reasonable to believe that during the prior RTP treatments of samples A and B, the grown-in oxygen precipitates being smaller than the critical size for the oxygen precipitation occurring at the temperature of RTP were substantially dissolved. Obviously, more grown-in oxygen precipitates were dissolved in sample A which received higher temperature RTP.

So far, it is clear that from sample A to B to C the numbers of grown-in oxygen precipitates contributing to oxygen precipitation occurring at 1050 °C increased in turn. After a long 1050 °C anneal, from sample A to B to C, the densities of BMDs pertaining to the resultant oxygen precipitates accordingly increased in turn, as revealed in figure 2. On the other hand, as illustrated in figure 1, the amounts of precipitated oxygen in samples A, B, and C subjected to 1050 °C/128 h anneal were nearly the same; if it is assumed that the precipitated oxygen atoms were uniformly distributed into the growing oxygen precipitates, the sizes of resultant oxygen precipitates from sample A to B to C should decrease in turn, which is actually reflected in figure 2 where the sizes of etch pits for BMDs pertaining to the oxygen precipitates generated in samples A, B, and C decreased in turn.

4. Conclusion

In summary, through the investigation of the RTP effect on the oxygen precipitation in a CZ silicon wafer occurring at the elevated temperature of 1050 °C, it has been clarified that the early stage oxygen precipitation is significantly enhanced by the RTP-induced vacancies which combine with oxygen atoms into O_2V species at the beginning of the 1050 °C anneal. This substantial enhancement effect is due to the fact that the O_2V species provide the space to relieve the strain arising from the growth of oxygen precipitates. However, when the oxygen precipitation in the CZ silicon wafer reaches an equilibrium state by a long 1050 °C anneal, the RTP-induced vacancies do not lead to more precipitated oxygen atoms. Furthermore, it has been found that after a long 1050 °C anneal the oxygen precipitates in the CZ silicon wafer with prior RTP treatment have considerably lower density but are larger in size than those in the control CZ silicon wafer without prior RTP treatment. This is because the prior RTP treatment will dissolve some of the grown-in oxygen precipitates that will contribute to oxygen precipitation in a subsequent anneal. In a word, the enhancement effect of vacancies introduced by RTP on oxygen precipitation at elevated temperature is only manifested at the early stage in terms of precipitation rate. Incidentally, it has been proved that the high temperature RTP has superior capability to dissolve oxygen precipitates existing in the CZ silicon wafer. Furthermore, it is worthwhile to point out that, when addressing the effect

of RTP on the oxygen precipitation behaviour during the subsequent anneal, two functions arising from the RTP treatment, that is, the injection of vacancies into the silicon wafer and the dissolution of grown-in oxygen precipitates existing in the silicon wafer, should be taken into account.

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