論文 88-25-7-6

A Study on the Nucleation, Growth and Shrinkage of Oxidation Induced Stacking Faults (OSF) – Part 2 : Role of SiO_2 Layer on the Shrinkage of Oxidation Induced Stacking Faults (OSF) in P-type CZ Silicon

(산화 적층 결함의 생성, 성장 및 소멸에 관한 연구 – 제 2 부 : P형 CZ 실리콘에서 산화 적층 결함의 소멸에 미치는 SiO_2 층의 역할)

金 龍 泰*, 閔 碩 基*

(Yong Tae Kim and Suk Ki Min)

要 約

본 연구에서는 산화 적층 결함의 성장과 소멸을 관찰하는데 필요한 간단하고 쉬운 새로운 방법을 고안하였다. 이 방법은 압력을 제어 할수 있는 바늘로 실리콘 웨이퍼 표면에 손상을 가한 후 이 손상된 영역에서 산화 및 열처리 공정에 따른 적층 결함의 거동을 관찰하는 것이다. 고안한 방법을 사용하여 산화 적층 결함을 성장시킨 후 산화막을 제거한 경우와 제거하지 않은 경우에서 산화막이 결함의 소멸에 어떤 작용을 하는지 조사하였다. 이 실험적 결과로부터 실리콘 interstitial 과 vacancy 의 재결합 기구에 의한 적층 결함 소멸 모형을 제시 하였다.

Abstract

We have proposed a new simple and easy method for the observation of OSF growth and shrinkage. This method is to observe the behavior of OSF in the damaged region during oxidation as well as annealing process after introducing mechanical damage on the silicon surface by pressure-controllable indentor. The effect of SiO_2 layer on the shrinkage of pregrown OSF generated by the proposed method has been investigated using the samples with or without SiO_2 layer. From the experimental data, we suggest a model for the shrinkage of OSF, which is based on the recombination mechanism between silicon interstitial and vacancy at the Si-SiO_2 interface.

*正會員, 韓國科學技術院 半導體 材料硏究室

(Semicond. Materials Lab., KAIST)

接受日字 : 1988年 2月 10日

I. Introduction

In recent years, many authors have reported about the growth and shrinkage (or annihilation) of OSF. However, several experiments concerning OSF growth and shrinkage are conflicting each other because both the growth and the shrinkage of OSF depend on such numerous parameters as oxygen concentration [1-4], dose and type of impurity [5-8], heat treatment cycle [9,10], surface conditions [11,12], stress evolution [13,14]. Furthermore, no report is still presented that the behavior of OSF is continously traced from the nucleation to the annihilation stage, because it is very hard to generate OSF intentionally on a certain region of silicon wafer. Consequently, it is also impossible to eliminate the entangled effects of the above mentioned parameters. In the view of these difficulties, we will suggest a new method, in which OSF is intentionally introduced on the silicon surface by the simple scratching without Si^+ implantation, and report the surface covering effect on the shrinkage of the pregrown OSF, which has occurred during annealing process.

Considering that it is difficult to find where OSF has occurred in a 4 or 5 inch-whole silicon wafer, it points out that the study of OSF is more easily carried out by the observation of pregrown OSF, which has grown on the predetermined region. Also, using the proposed method, it is possible to represent how the growth and shrinkage of OSF are influenced by the nature of Si-SiO_2 interface without any other mixed effects. In other words, we try to explain the OSF shrinkage in the way of that the unoxidized silicon interstitials generated in the Si-SiO_2 interface are absorbed into or emitted from the stacking fault depending on the surface conditions.

II. Experiment

We grew a boron doped, (100) oriented silicon ingot by CZ method. After fine polishing, resistivity, interstitial oxygen and carbon concentrations of wafers are measured by spreading resistance probe and FTIR spectroscopy, respectively. Resistivity is 5-7 ohm-cm, oxygen concentration is 14-18 ppm and carbon is 0.3-0.5 ppm. In order to introduce mechanical damage on the silicon surface, a pressure controlled Os-W alloy tipped indentor is used.[15] We have run this indentor across a polished silicon surface in both directions close to [100] and [001]. After scratching, samples are cleaned and oxidized at 1100°C in dry oxygen ambient for 3 hrs.. Observation of OSF on the damaged region is carried out by the Wright etching method, which is introduced in part 1. The SiO_2 covering effects on the shrinkage of OSF are investigated as follows: In the first set of samples, we have removed the SiO_2 layer grown by first oxidation and then annealed these samples in N_2 ambient for different annealing times, ranging from 1 to 10 hrs., and annealing temperatures are 850, 950, 1050, 1100 and 1150°C. In the second set of samples, the SiO_2 layer is kept on the silicon surface and the SiO_2 covering effect on the shrinkage of pregrown OSF is investigated through the same annealing conditions of the first case. The third group is selected from the previous two sets and reoxidized in dry oxygen ambient for 2 hrs. to study whether or not the shrunken OSF becomes to regrow during the re-oxidation process. After annealing and re-oxdation, we have also measured the length of OSF with SEM and Nomarski interference microscope. The oxygen and carbon concentrations are checked to study oxygen and carbon out diffusion effects.

III. Results and discussion

Fig. 1 shows the temperature dependence of pregrown OSF on the samples with or without

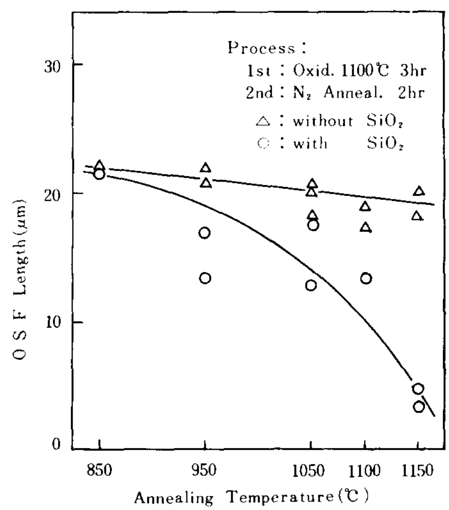
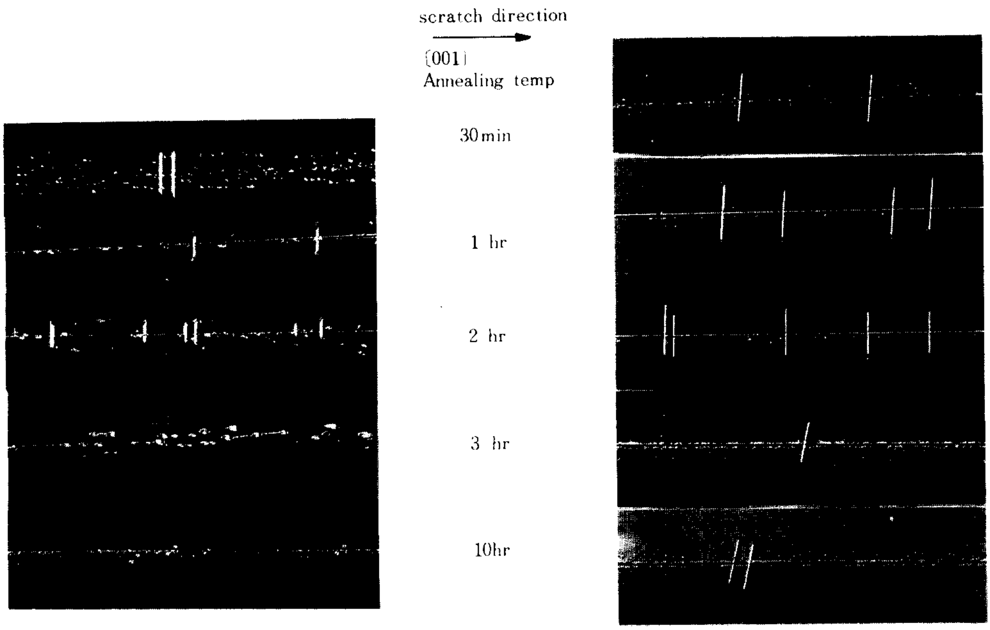
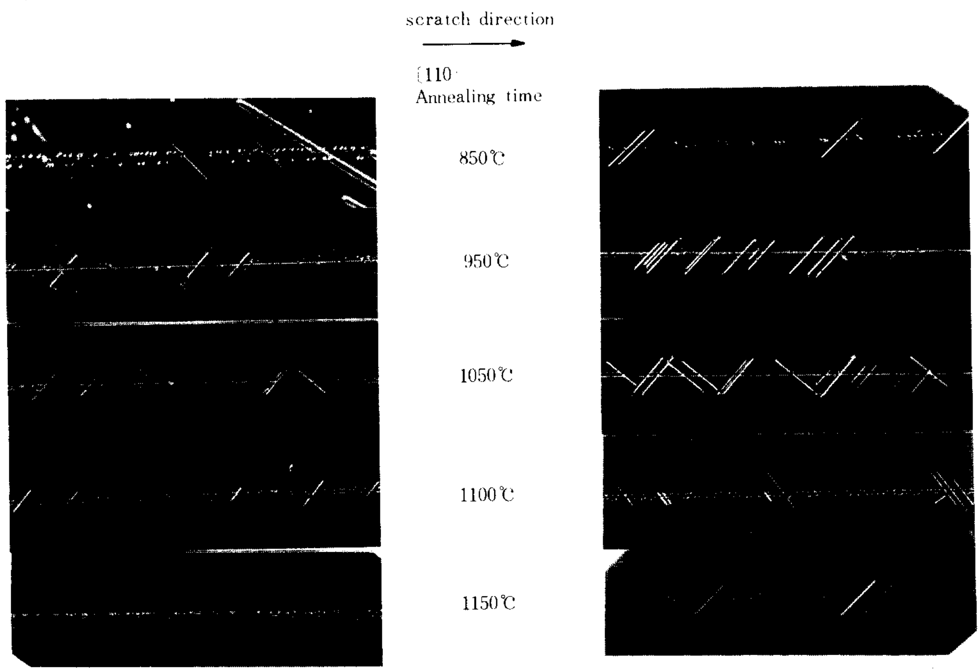


Fig.1. The dependence of OSF length on annealing temperatures in the samples with and without SiO_2 layer.



(a) OSF shrinkage in the samples covered with SiO_2 layer

(b) OSF non-shrinkage in the SiO_2 removed samples



(c) Annealing temperature dependence of OSF shrinkage in the samples covered with SiO_2 layer

(d) Annealing temperature dependence of OSF non-shrinkage in the SiO_2 removed samples

Fig.2. Sequential micrographs representing OSF shrinkage and non-shrinkage.

SiO_2 layer. For the samples with SiO_2 layer, it is observed that OSF becomes to shrink with increasing annealing temperature, where as the shrinkage of OSF did not apparently occurred in the samples without SiO_2.

As shown in Fig.2 (a), 2(b), 2(c) and 2(d) OSF is extended across the scratch line and gradually, OSF disappears in the samples with SiO_2 layer. It is apparent that OSF becomes annhilated under the SiO_2 layer when annealing is done at 1150°C. From these results, we can conclude that good evidence for the shrinkage of pregrown OSF can be derived when the both samples are annealed at 1150°C for long time.

Fig. 3 shows both the variation of OSF length with annealing times at 1150°C and the reoxidation results for the samples with SiO_2 layer. In this figure, it is found that the pregrown OSF under the SiO_2 layer is directly shrinking with increasing annealing times, 1 to 10 hrs. . However, for the samples without SiO_2 layer, as shown in Fig.4, OSF does not shrink but rather slowly expand. On the other hand, when oxidation is carried out again after annealing at 1150°C for each annealing time, OSF grows again for the both samples. This phenomenon indicates that a certain kind of interaction exists between the Si-SiO_2 interface and the OSF shrinkage.

Now, the question is what the interaction between the Si-SiO_2 interface and the OSF is. From the growth kinetics, which is depicted in

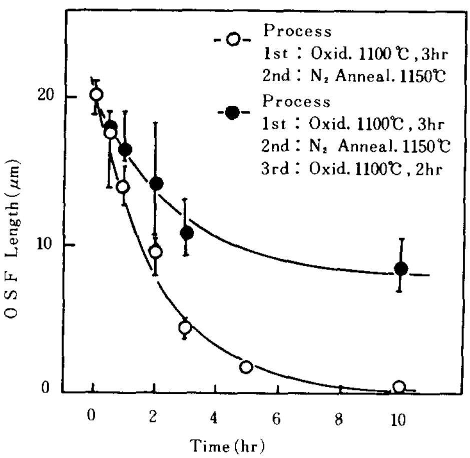


Fig.3. The time dependence of OSF length on the annealing and reoxidation processes for the samples with SiO_2 layer.

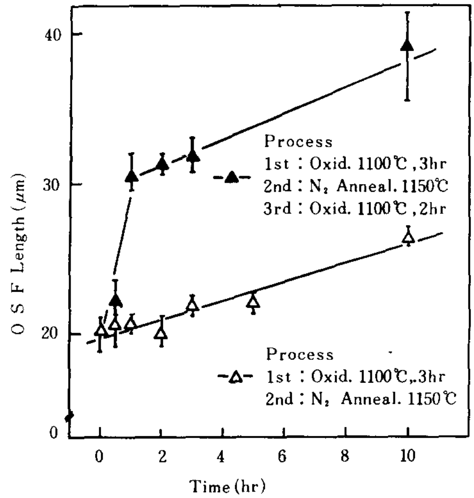


Fig.4. The time dependence of OSF length of the annealing and reoxidation processes for the samples without SiO_2 layer.

equation (1) of part 1, it is known that length of OSF depends on the relative interstitial concentration Ci/Ci^{O} Antoniadis[12] also showed that the growth rate of OSF is related to Ci/Ci^{O} and in the same analogy, the shrinkage rate of OSF can be calculated from the relative difference in the interstitial concentration, providing the related constant values obtained from the OSF shrinkage rate under the oxide mask surrounded by a phosphorus-doped layer. Therefore, we can suggest that the shrinkage rate of OSF is also correspondent upon Ci/Ci^*, where Ci^* is the initial interstitial concentration of pregrown OSF and Ci is the reduced interstitial concentration of shrinking OSF. However, in this paper, we can not derive the exact equation for the shrinkage kinetics because the activation energy for interstitial to leave the fault is unknown and we do not reciprocally apply the equation for the growth of OSF, Eq.(1) of part 1, to the OSF shrinkage kinetics.

Consequently, it is possible to describe the shrinkage rate of OSF as follows:

$$L/L^* \propto Ci/Ci^* \tag{1}$$

where, L is the length of OSF shrinkage and L^* is the initial length of OSF. As shown in Fig. 5,

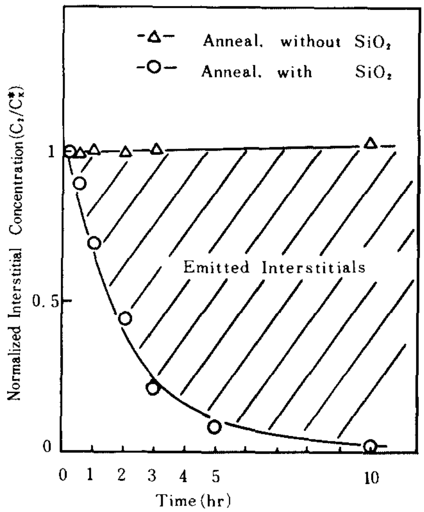


Fig.5. Relation of normalized interstitial concentration vs annealing time.

empirically, Ci/Ci^* can be expessed as follows:

$$Ci/Ci^* \propto \exp(-t/\tau) \qquad (2)$$

where, t is the annealing time and τ is the relaxation time for the shrinkage. Antoniadis reported that Ci/Ci^* decreased linearly with the annealing time. But, we point out that in his report the effect of phosphorus diffusion is involved in the shrinkage of OSF and as a result, there should be a compressive stress around the heavily phosphorus doped layer. In our result, it can be concluded that under the SiO_2 layer, Ci decreases exponentially with increasing annealing time, which indicates that only Si-SiO_2 interface play some role to leave silicon interstitial from the stacking fault.

Now, We try to explain the above results as follows: First, silicon interstitials, which are generated from Si-SiO_2 interface during oxidation process, establish the extra Si planes on the damaged region. Second, when oxidation process is finished and annealing process is continously carried out, interstitials are not supplied from the Si-SiO_2 interface but absorbed in to the interface. In this mechanism, we suppose that the potential motive to leave interstitial from the stacking fault is the recombination process between interstitials and vacancies at the interface. In other words, during annealing process interstitials are emitted from the stacking fault and migrated into the Si-SiO_2 interface, while vacancies move from the interface to the stacking fault. From the above discussion, it is found that the quantity of interstitials emitted with increment of the annealing time is to be the shadow region of Fig.5. Fig.3 shows that for the SiO_2-covered samples, the larger the shrinkage is, the higher the regrowth rate of OSF and for the SiO_2-removed samples, regrowth rate seems to be equal, although there is some deviation in the length of OSF. It implies that interchange process between interstitials and vacancies at the interface is not a perfect one-to-one corresponding interchange mechanism and interstitials, which is migrating to the interface and remaining after interchanging with vacancies, start to re-enter into the fault as oxidation process begins. As we metioned earlier, there have been several reports to support our result. Murarka [16], Hu [3], Tiller [17], Leroy [18] and Antoniadis[12] Jaccodine[19] found that OSF growth/shrinkage could be explained in terms of interstitial and vacancy. However, it is noteworthy here that the previous experiments was complicated and had uncertainties in determining the dominant reasons.

IV. Conclusion

We have studied the effect of Si-SiO_2 interface on OSF growth and shrinkage with or without SiO_2 layer. Comparing with the previous works, the proposed method, where OSF is intentionally introduced by the mechanical damage, is simple and easy for observation of OSF because it needs not the implantation of Si ions and TEM observation. From the results of our experiment, it is shown that the Si-SiO_2 interface play the sink-site for silicon interstitials to leave from the extra Si-planes, which is so called stacking fault, when annealing process is carried out for the samples covered with SiO_2 layer. However, oxidation process is done, the interface becomes as the generation site to give excess interstitials to the stacking fault.

We suggest the recombination mechanism between interstitials and vacancies to explain the effect of Si-SiO_2 interface. However, there are some remaining problems to find parameters, to establish the exact equation, which is necessary for understanding of OSF shrinkage.

References

[1] C.W. Pearce and G.A. Rozgonyi, "Source of oxidation induced stacking faults in CZ silicon wafers: II. the influence of oxygen content," Semicond. silicon 1977, p. 606, 1977.

[2] P.E. Freeland, et al, "Precipitation of oxygen in silicon," *Appl. Phys. Lett.* vol. 30, no. 1, p. 31, Jan. 1977.

[3] S.M. Hu, "The shrinkage and growth of oxidation stacking faults in silicon and the influence of bulk oxygen," *J. Appl. Phys.* vol. 51, no. 7, p. 3666, July 1980.

[4] S.M. Hu, "Precipitation of oxygen in silicon: some phenomena and a nucleation model," *J. Appl. Phys.*, vol. 52, no. 6, p. 3974, June 1981.

[5] S. Prussin, "Role of sequential annealing, oxidation, and diffusion upon defect generation in ion implanted silicon surfaces," *J. Appl. Phys.*, vol. 45 no. 4, p. 1635, Apr. 1974.

[6] G.A. Rozgonyi and T.E. Seidel, "Influence of wafer orientation and ion implantation on surface and bulk stacking faults," Semicond. silicon 1977, p. 616, 1977.

[7] D.A. Antoniadis, A.M. Lin, and R.W. Dutton, "Oxidation enhanced diffusion of arsenic and phosphorus in near-intrinsic (100) silicon," *Appl. Phys. Lett.*, vol. 33, no. 12, p. 1030, Dec. 1978.

[8] S. Mizuo and H. Higuchi, "Effect of back side oxidation on B and P diffusion in Si directly masked with Si_3N_4 films," *J. Electrochem. Soc.*, vol. 129, no. 10, p. 2292, 1982.

[9] F. Shimura and H. Tsuya, "Multistep repeated annealing for CZ-silicon wafers: oxygen and induced defect behavior," *J. Electrochem. Soc.*, vol. 129, no. 9, p. 2089, 1982.

[10] S. Isomae, S. Aoki, and K. Watanabe, "Depth profiles of interstitial oxygen concentrations in silicon subjected to three-step annealing," *J. Appl. Phys.*, vol. 55, no. 4, p. 817, Feb. 1984.

[11] C. Claeys, et al, "Impact of a chlorine oxidation on the properties of silicon bulk defects," Semicond. silicon 1981, p. 730, 1981.

[12] K. Nishi and D.A. Antoniadis, "Observation of silicon self interstitial super-saturation during phosphorus diffusion from growth and shrinkage of oxidation-induced stacking faults," *J. Appl. Phys.*, vol. 59, no. 4, p. 1117, Feb. 1986.

[13] J.M. Fairfield and G.H. Schwuttke, "Strain effects around planar diffused structures," *J. Electrochem.* Soc., vol. 115, no. 4, p. 415, Apr. 1968.

[14] G. Charitat and A. Martinez, "Stress evolution and point defect generation during oxidation of silicon," *J. Appl. Phys.*, vol. 55, no. 4, p. 909, Feb. 1984.

[15] Y.T. Kim, et al, "Effect of mechanical damage on the nucleation of oxidation induced stacking faults in n-type (100) Si," Bull. of Kor. Phys. Soc., vol. 2, no. 3, p. 204, 1984.

[16] S.P. Muraka, "Role of point defects in the growth of the oxidation-induced stacking faults in silicon," Phys. Rev. B, vol. 16, no. 6, p. 2849, Sep. 1977.

[17] W.A. Tiller, "On the kinetics of the thermal oxidation of silicon," *J. Electrochem. Soc.*, vol. 127, no. 3, p. 619, Mar. 1980.

[18] B. Leroy, "Kinetics of growth of the oxidation stacking faults," *J. Appl. Phys.*, vol. 50, no. 12, p. 7996, Dec. 1979.

[19] U.S. Kim and R.J. Jaccordine, "Fast shrinkage of oxidation stacking faults during O_2/NF_3 oxidation of silicon," *Appl. phys. Lett.*, vol. 49, p. 1201, 1986. *

著 者 紹 介

金 龍 泰(正會員)
1954年 3月 3日生. 1981年 2月 경북대학교 전자공학과 졸업(B. S) 1982年 2月 경북대학교 대학원 전자공학과 석사학위 취득. 1982年 3月~12月 경북대학교 전자공학과 강사. 1984年 3月~1987年 12月 한국과학기술원 전기 및 전자공학과 박사과정. 현재 한국과학 기술원 반도체 재료연구실 선임연구원 주관심분야는 PECVD, ECRCVD, RIE기술, 3차원 구조 VLSI기술 등임.

閔 碩 基(正會員)
1938年 12月 14日生. 1964年 2月 고려대학교 물리학과 졸업. 1966年 고려대학교 대학원 고체물리학 전공 이학석사 학위취득. 1968年 고려대학교 물리학과 조교. 1969年~1970年 고려대학교 물리학과 강사. 현재 한국과학기술원 반도체 재료 연구실 실장. 주관심분야는 Si, GaAs 단결정성장기술 MOCVD, HEMT 관련기술 개발분야 등임.