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Effect of pressure and temperature on bulk micro defect and denuded zone in nitrogen ambient furnace

Young-Kyu Choi[*,**], Se-Young Jeong[*] and Bok-Cheol Sim[***,†]

*Department of Nano Fusion Technology, Pusan National University, Miryang 50463, Korea
**Crystal Growth Technology Team, LG Siltron, Simidong, Gumi 39400, Korea
***School of Mechanical and Automotive Engineering, Hanyang Cyber University, Seoul 04763 Korea

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Abstract The effect of temperature and pressure in the nitrogen ambient furnace on bulk micro defect (BMD) and denuded zone (Dz) is experimentally investigated. It is found that as pressure increases, Dz depth increases with a small decrease of BMD density in the range of temperature, 100~300°C. BMD density with hot isostatic pressure treatment (HIP) at temperature of 850°C is higher than that without HIP while Dz depth is lower due to much higher BMD density. As the pressure increases, BMD density is increased and saturated to a critical value, and Dz depth increases even if BMD density is saturated. The concentration of nitrogen increases near the surface with increasing pressure, and the peak of the concentration moves closer to the surface. The nitrogen is gathered near the surface, and does not become in-diffusion to the bulk of the wafer. The silicon nitride layer near the surface prevents to inject the additional nitrogen into the bulk of the wafer across the layer. The nitrogen does not affect the formation of BMD. On the other hand, the oxygen is moved into the bulk of the wafer by increasing pressure. Dz depth from the surface is extended into the bulk because the nuclei of BMD move into the bulk of the wafer.

Key words Bulk micro defect, Denuded zone, Nitrogen ambient furnace, Silicon wafer

1. Introduction

Recently, semiconductors are becoming more integrated and smaller for modern electronic application. Metal impurities within the silicon wafers are becoming increasingly critical to device yield and reliability. In general, metal impurities in silicon single wafer used for ultra large scaled integrated (ULSI) circuits have been believed to deteriorate the performance of device [1-3]. So, it is important to protect the active area of the device from metal contamination such as Cu, Fe, and Ni [2, 4, 5]. For the metal-free within the active area of the device, gettering techniques are used widely in semiconductor industries [6]. The denser BMD(Bulk Micro Defect) within the bulk of silicon single wafer can increase the gettering capability of the metal and improve the quality of ULSI devices [6, 7].

Intrinsic gettering using oxygen-induced BMD has been investigated since the realization of the potential to manage metal contamination [8, 9]. Because oxygen-induced BMD is the oxygen precipitate, it is influenced by oxygen concentration and heat treatment condition like temperature [10-15]. As the concentration of oxygen is higher, the density of BMD is increased. However, if BMD density is too much, BMD is generated within the active area of device. It causes the leakage current in the device. It is known that large oxide precipitates degrade the gate oxide integrity [16-18]. Micro-defects generally decrease the minority carrier lifetime and can cause leakage currents in devices [19, 20]. Defects in the active area of the device are harmful to the deep trenches of memory cells. However, if properly placed to getter metallic impurities, oxide precipitates can result in improved refresh time of dynamic memory devices [21]. Such micro-defect-free zones can be formed by oxygen out-diffusion from wafer surface at high temperature, which prevents oxygen precipitation in the region near the surface and the formation of secondary defects in the active device area [22]. The zone without BMD in the depth direction from the surface of wafer is required for the active area of the device. Such a zone is called Dz (Denuded zone). Thus, the wafer with denser BMD and sufficient Dz depth is required [7].

In the present study, we report experimental results on the formation of bulk micro defect (BMD) for the gettering effect. Samples are treated in the nitrogen ambi-

[†]Corresponding author
E-mail: sbcsim@naver.com, sbcsim@hycu.ac.kr

ent furnace with the various temperature and pressure conditions for 10 hours. BMD density and Dz depth are measured after 2-step heat treatment, which is to evaluate the oxygen precipitation performance of the silicon wafers. The effect of temperature and pressure on BMD and Dz depth in the nitrogen ambient furnace is experimentally investigated. Also, the effect of pressure on nitrogen behavior near the wafer surface is discussed.

2. Experiments

Firstly, the vacancy-rich 300 mm (100) silicon single crystal (called silicon ingot) is manufactured in commercial crystal growth furnace. At the same position of the ingot, some wafers are selected. The resistivity is 22.0 Ω cm and the oxygen concentration is 13.5 ppma at the center of these wafers. A piece of sample of 2 inch by 2 inch size is abstracted at the center of the wafer. Then the sample is treated in the nitrogen ambient furnace that can control temperature and isostatic pressing. This treatment is called HIP (Hot Isostatic Pressure) treatment. In these experiments, the temperature conditions are 100°C, 300°C and 850°C, and the pressure conditions are 10 bar, 50 bar, 100 bar and 200 bar. The samples are treated for 10 hours in the furnace.

Usually, oxygen precipitate in silicon wafer are produced using two-step annealing. First step is to nucleate new precipitates or to enlarge already existing ones, and second step is to make the precipitates large enough to be detected. This annealing procedures are used to evaluate the oxygen precipitation performance of wafers [15]. In order to estimate BMD in each sample, the sample is done 2-step heat treatment (800°C for 4 hours and 1000°C for 16 hours). The temperature of 800°C is to make the nucleus of the oxygen precipitate, and the temperature of 1000°C is to grow the oxygen precipitate. The density of BMD and Dz depth are measured by SIRM (Scanning Infrared Microscopy) and microscope after etching. Also, the nitrogen concentration toward depth from the surface of the sample is measured by SIMS (Secondary Ion Mass Spectroscopy).

3. Results and Discussion

Fig. 1 shows the density of BMD and Dz depth with and without HIP treatment, where ref1 means without HIP. The conditions of the temperature, T, are 100°C, 300°C and 850°C, and the conditions of the pressure, P, are 10 bar and 50 bar. The temperature of 850°C is treated in the pressure of 50 bar only. In Fig. 1, it is shown that Dz depth and BMD density are in inverse proportion, that is, Dz depth increases when BMD density decreases. Compared to BMD density and Dz depth without HIP treatment, HIP treatment makes BMD density lower and Dz depth higher except the results at the highest temperature, T = 850°C. BMD density with HIP is lower than that without HIP because the nuclei of oxygen precipitates is dissolved in T = 100°C and 300°C. In T = 850°C, which is near T = 800°C to make

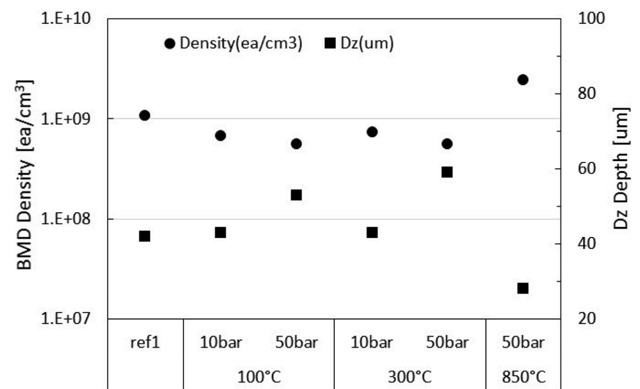

Fig. 1. BMD density and Dz depth after HIP with various temperatures and pressures, where ref1 means without HIP.

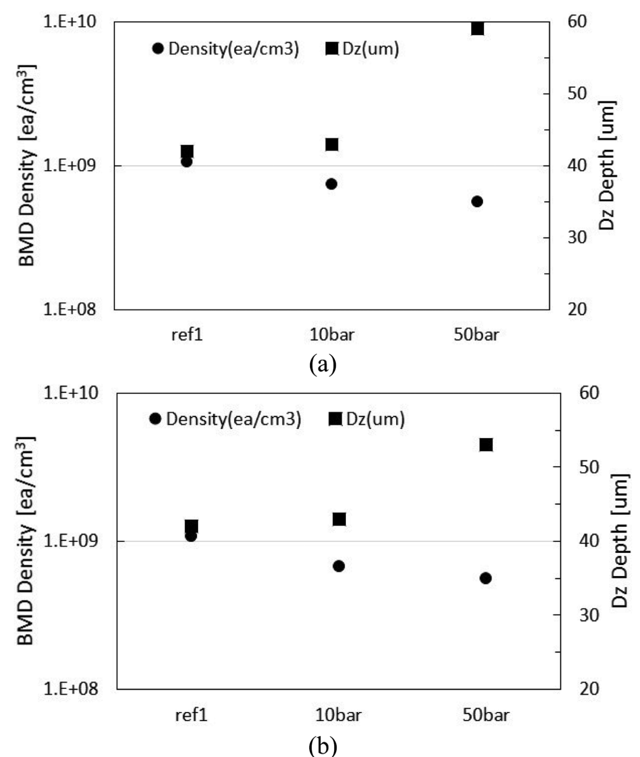

Fig. 2. BMD density and Dz depth after HIP with various pressures at the fixed temperature of (a) 100°C and (b) 300°C, where ref1 means without HIP.

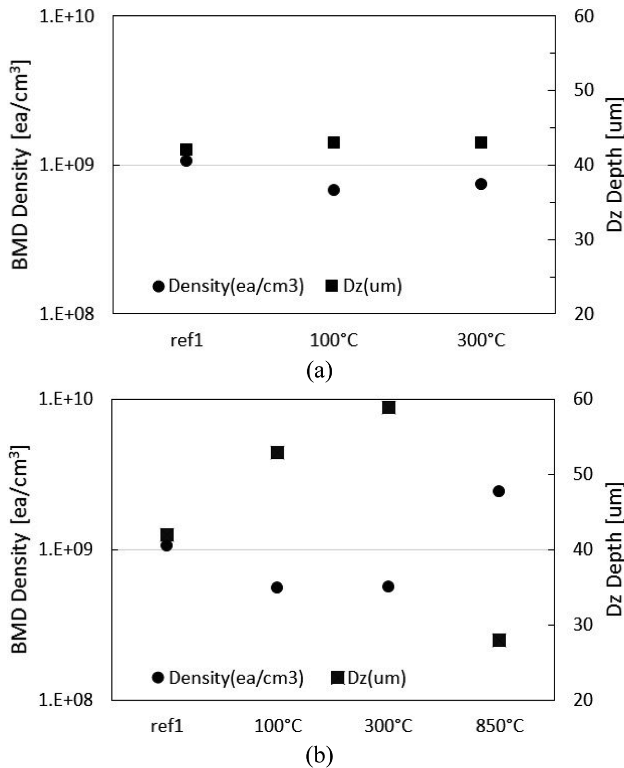

Fig. 3. BMD density and Dz depth after HIP with various temperatures at the fixed pressure of (a) 10 bar and (b) 50 bar, where ref1 means without HIP.

the nuclei of oxygen precipitates, BMD density is highest due to generation of nuclei of oxygen precipitates. In Fig. 1, it is difficult to show the effect of temperature and pressure on BMD density and Dz depth. Fig. 1 is separated into Figs. 2 and 3 to investigate the influences of temperature and pressure.

Fig. 2 shows the effect of pressure on BMD density and Dz depth at the fixed temperatures, (a) T = 100°C and (b) T = 300°C. At the fixed T = 100°C or T = 300°C, BMD density with P = 50 bar is a little lower than that with P = 10 bar, and Dz depth with P = 50 bar is much higher than that with P = 10 bar as shown in Figs. 2(a) and 2(b). It is found that Dz depth increases and BMD density decreases a little as pressure increases.

The effect of temperature on BMD density and Dz depth is shown in Fig. 3. As shown in Fig. 3(a), BMD density and Dz depth do not change with increasing temperature at P = 10 bar. Except results at highest temperature, T = 850°C, BMD density does not change with increasing temperature at P = 50 bar, while Dz depth increases with increasing temperature in Fig. 3(b). It is found that Dz depth is dependent on temperature at P = 50 bar and is independent of temperature at less than P = 10 bar in our experimental range of temperature,

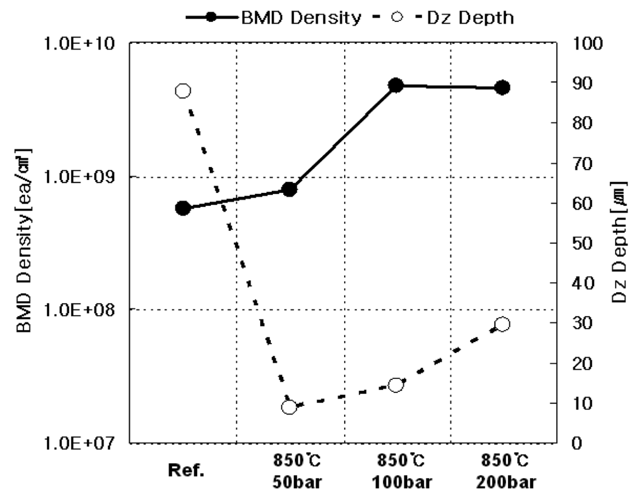

Fig. 4. BMD density and Dz depth after HIP with various pressures at fixed temperature of 850°C, where Ref means without HIP.

T = 100~300°C. BMD density at T = 850°C and P = 50 bar is highest because T = 850°C is near T = 800°C, which generates the nuclei of oxygen precipitations. Many nuclei generated at T = 850°C grow to BMD during 2-step heat treatment. The very high BMD density results from very low Dz depth at T = 850°C.

In order to verify the effect of pressure on BMD density and Dz depth, another set of samples is made from wafers with the resistivity of 16.0 Ω cm and the oxygen concentration of 13.3 ppma. This set of samples is different from the previous one (with the resistivity of 22.0 Ω cm and the oxygen concentration of 13.5 ppma). The samples are treated for 10 hours in the nitrogen ambient furnace with P = 50 bar, 100 bar and 200 bar at fixed T = 850°C. The temperature is fixed at T = 850°C for the sufficient density of BMD. After 2-step heat treatment, BMD density and Dz depth are shown in Fig. 4, where Ref means without HIP. BMD density with HIP at T = 850°C is higher than that without HIP while Dz depth is lower due to much higher BMD density as shown in Fig. 4. As the pressure increases, BMD density is increased and saturated to a critical value, and Dz depth increases. At more than P = 100 bar, BMD density is saturated while Dz depth still increases with increasing pressure. It is evident that Dz depth is controlled by the pressure in the furnace even if BMD density is saturated.

Fig. 5 shows the images of BMD and Dz about the samples of Fig. 4. As the pressure increases, the density of BMD increases and Dz depth is extended toward the bulk of wafer. The result shows that the area of forming BMD can be adjusted by controlling the pres-

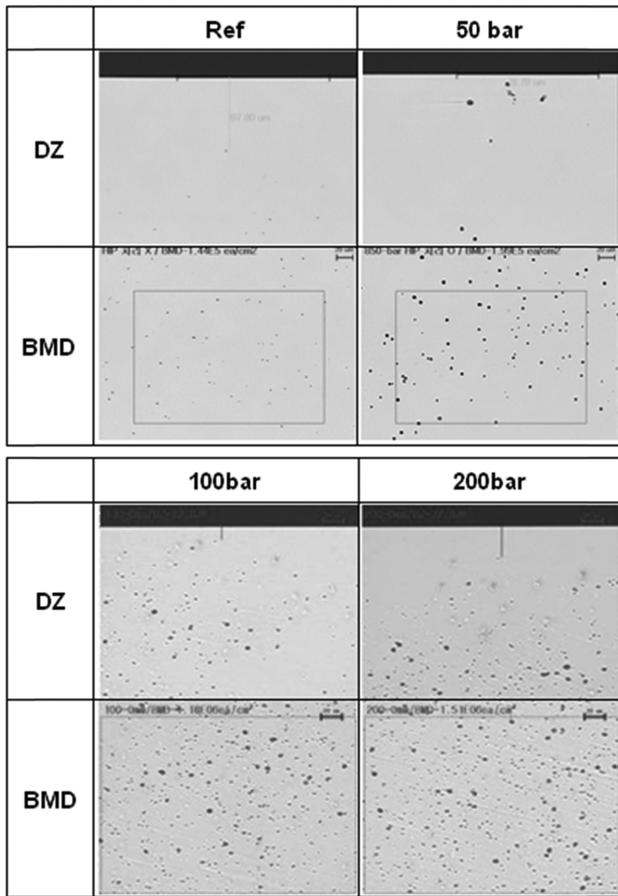

Fig. 5. Image of Dz depth and BMD corresponding to Fig. 4.

sure. The nitrogen concentration near the wafer surface is measured by SIMS in Fig. 6. As the pressure increases, the concentration of nitrogen increases, and the peak of the concentration moves closer to the surface. The nitrogen is gathered near the surface, and does not become in-diffusion to the bulk of the wafer as shown in Fig. 6. Thus, BMD density in the bulk of the wafer is not related to in-diffusion of nitrogen from the near surface. We conclude that BMD density in the bulk of the wafer is only due to oxygen precipitation. Voronkov et al. [15] reported that in rapid thermal annealing (RTA) of a nitride-coated wafer with high temperature T = 1220°C, the nitrogen did not become in-diffusion to the bulk from the wafer surface.

It is assumed that the nitrogen makes the compound like silicon nitride to react with silicon. Silicon nitride layer moves closer to the surface of wafer with increasing pressure. The silicon nitride layer near the surface prevents to inject the additional nitrogen into the bulk of the wafer across the layer. The nitrogen does not affect the formation of BMD. On the other hand, the oxygen is estimated to move into the bulk of the wafer with

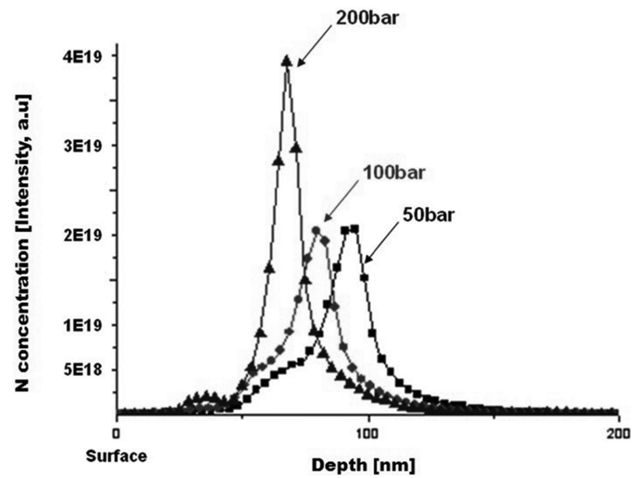

Fig. 6. Nitrogen concentration near surface by SIMS corresponding to Fig. 4.

increasing pressure. The oxygen moved by pressure causes the nucleus of BMD. Therefore, Dz depth from the surface is extended into the bulk because the nuclei of BMD move into the bulk of the wafer. It is found that by controlling the temperature and pressure, BMD with the sufficient density can be formed in the desired area within a wafer. This concept is different from the formation of Dz layer by out-diffusion in annealed wafer. The oxygen precipitates are not out-diffused near surface in HIP, and the oxygen as the nuclei of BMD is moved into the bulk of wafer at high pressure.

4. Conclusions

The effect of temperature and pressure on BMD and Dz is experimentally investigated in the nitrogen ambient furnace. Compared to BMD density and Dz depth without HIP treatment, HIP treatment makes BMD density lower and Dz depth higher in T = 100°C and 300°C. It is found that as pressure increases, Dz depth increases with a small decrease of BMD density. BMD density and Dz depth do not change with increasing temperature at P = 10 bar in the range of temperature, T = 100~300°C. Dz depth is dependent on temperature at P = 50 bar.

BMD density with HIP at T = 850°C is higher than that without HIP while Dz depth is lower due to much higher BMD density. As the pressure increases, the density of BMD is increased and saturated to a critical value, and Dz depth increases. It is found that Dz depth increases with increasing pressure in the furnace even if BMD density is saturated.

As the pressure increases, the concentration of nitrogen increases near surface, and the peak of the concentration moves closer to the surface. The nitrogen is gathered near the surface, and does not become in-diffusion to the bulk of the wafer. It is assumed that the nitrogen makes the compound like silicon nitride to react with silicon. Silicon nitride layer moves closer to the surface of wafer with increasing pressure. The silicon nitride layer near the surface prevents to inject the additional nitrogen into the bulk of the wafer across the layer. The nitrogen does not affect the formation of BMD. On the other hand, the oxygen is estimated to move into the bulk of the wafer with increasing pressure. The oxygen moved by pressure causes the nucleus of BMD. Therefore, Dz depth from the surface is extended into the bulk because the nuclei of BMD move into the bulk of the wafer. It is found that by controlling the temperature and pressure, BMD with the sufficient density can be formed in the desired area within a wafer.

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