The Novel Biomaterial of Visible Photoluminescence by Saving Energy Technology of Rapid Thermal Annealing

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Abstract

In this article, the novel biomaterial of visible photoluminescence by saving energy technology of rapid thermal annealing is presented. A shift in the photoluminescence (PL) peak from blue to near-infrared region was observed in the Si^+-implanted 400-nm-thick SiO_2 film with the rapid thermal annealing (RTA) method only. As the Si^+-fluence was $1x10^{16}$ ions/cm^2, a blue band was observed in the films after RTA at 1050°C for 5 seconds in dry-N_2 atmosphere; then, the band shifted from blue to orange upon increasing the holding temperature of RTA to 1250 ^{o}C in the films after the isochronal RTA in dry N_2. Furthermore, while the fluence was increased to $3x10^{16}$ ions/cm^2 and the holding temperature was at the same range between 1050°C and 1250 ^{o}C, the PL peak occurred between red and near-infrared regions. Although the RTA and conventional thermal annealing (CTA) methods produce the similar mechanism, the CTA method needs a much longer annealing-time and a higher Si^+-implanted dose than the RTA method to observe the same range and intensity of PL peak from the as-implanted sample. Therefore, the RTA method can produce the mechanism in the Si^+-implanted sample with the energy of PL peak between blue and near-infrared band in palce of the CTA method. [Life Science Journal. 2009; 6(3):61–67] (ISSN: 1097 – 8135)

Keywords: rapid thermal annealing (RTA), Si^+ implantation, photoluminescence, SiO_2 films, FTIR spectra

1 Introduction

With the advancement of Si technology for electronic integrated circuits and in view of the potential applications in optoelectronics and photonics, there has been a significant interest in the structures consisting of silicon nanocrystals embedded in SiO_2[1]. Room temperature visible luminescence had been firstly observed from porous silicon (P-Si), however, several problems have been encountered in practical application [2, 3]. The plasma deposition technologies by using silane, gas evaporation, and termination of glass-melt reaction was used to produce visible light emission and silicon nanocrystals, but still none of them were appropriate for the manufacturing process of integrated device products. In addition to above methods, silicon-implantation into SiO_2 film on crystal Si with subsequent conventional thermal annealing (CTA) was another alternative method to fabricate Si-based luminescence structures. Broad emission bands between ~1.5 and ~2.5 eV had been observed by several research groups by implanting Si^+ into the SiO_2 films and following CTA at a temperature higher than 1000°C in vacuum, in Ar, or in N_2[4-9]. The $1x\ 10^{17}$ cm^{-2} Si^+-implanted films after the CTA in dry-N_2 at about 1000°C emits visible blue-light and the PL peaks were shifted from the blue ~2.5 eV to yellow ~2.2 eV upon increasing the annealing temperature from 1000°C to 1250°C[5]. The other light emission between red and near-infrared band was presented at the flux between $1x\ 10^{17}$ cm^{-2} and $3x\ 10^{17}$ cm^{-2} and were attributed to the quantum confinement effect in silicon nanocrystals that was confirmed by transmission electron microscopy [5-6,8]. In those reports[4-9], the flux of implanted silicon was relatively high and the whole annealing process required several hours. Although ion implantation of III-V compounds was routinely used in the production of optoelectronic integrated circuits, the methods using silicon implantation seem not suitable for applications in optoelectronics.

Recently, we successfully used the rapid thermal annealing (RTA) method only to observe the photoluminescence (PL) peak between blue and near-infrared regions in the Si^+-implanted 400-nm-thick SiO_2 films. At the Si^+-fluence of $1x10^{16}$ ions/cm^2, the light shifted from blue to orange band when the holding temperature of RTA was increased from 1050°C to 1250 ^{o}C in the films after RTA for 5 seconds in dry N_2. Furthermore, while the fluence was increasing to $3x10^{16}$ ions/cm^2 and the holding temperature was at the same range between 1050°C and 1250 ^{o}C, the ranges of PL peak were observed between red and near-infrared regions in the films after the RTA for 20 seconds. On the other hand, by means of RTA and CTA method, the PL

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peaks from the Si^+-implant SiO_2 films were both shifted with the increase of the Si^+ concentration in the oxide matrices and of the thermal annealing duration. However, the shifting affect using RTA method was much better than that using CTA method. In additional, the mechanisms achieved by RTA and CTA are both not dependent on hydrogen-related bonds and have similar existent ratio after isothermal and isochronal RTA test. The results show that the mechanisms in the Si^+-implanted SiO_2 films after the RTA and CTA methods are similar. However, for the same range and intensity of PL peak from the as-implanted sample, the CTA method needs a much longer annealing-time and a higher dose of Si^+-implantation than the RTA method. Therefore, using RTA method to produce the mechanisms in the Si^+-implanted samples with the energy of PL peak between blue to near-infrared band is better than using CTA method.

2 Experimental Procedure

Samples were prepared by implantation of Si^+ into a 400-nm-thick wet SiO_2 film which was thermally grown on (100)-oriented p-doped Si substrates. The respective fluences of the Si^+-implantation are $1x10^{15}$, $4x10^{15}$, $1x10^{16}$, $2x10^{16}$, and $3x10^{16}$ ions/cm^2. The temperature of the samples during ion implantation was kept at liquid nitrogen temperature. The acceleration energy of ~ 160 keV was selected so that the maximum concentration was at a depth of ~250 nm below the surface and the standard deviation of the implanted region was ±60 nm. These samples were subjected to RTA treatments at substrate temperature of 1050°C, 1150°C, and 1250°C, respectively, under dry and wet N_2. The introduction of wet N_2 into the furnace was performed by heating liquid H_2O at 100 °C in a cylinder, which was connected to the furnace through a guiding tube, and by mixing up with dry N_2 in the guiding tube. Furthermore, the atmospheric pressure of 50 mbar was provided into the furnace and a heating rate of 100°C/s and the cooling rate of about 100°C /min was used. On the other hand, in order to display the advantage of RTA system, we also annealed the samples at temperatures between 1050°C and 1250°C by using a conventional electric oven. The atmospheric pressure of 50 mbar was provided but a maximum heating rate of 10°C /min and the cooling rate of 50°C /min were used in the CTA system. It shall be noted that the two thermal annealing treatments both include three procedures as heating, holding, and cooling. On the other hand, to detect the PL spectra, a He-Cd laser (3.8 eV) was used as the excitation and a lock-in amplifier was employed to improve the signal-to-noise ratio, in conjunction with a monochromator and cooled photomultiplier tube. The excitation was operated at a small and fixed power of 5 mW to not detect any PL sign in the $3x10^{16}$ cm^{-2} Si^+-implanted 400-nm-thick wet SiO_2 films without RTA. Finally, the FTIR measurements were performed to examine the presence or absence of hydrogen-related species in samples of oxide film. With the reference being the same silicon plate as the sample, the spectra were measured at room temperature in N_2 atmosphere at a 2 cm^{-1} resolution with 200 scan accumulations.

3 Results and Discussion

In order to demonstrate that the RTA method is better than the CTA method for producing the mechanism in Si^+-implanted SiO_2 films with the energy of PL peak between the blue and near-infrared band, the fluence of the silicon implantation in the films was kept at $1x10^{16}$ and $3x10^{16}$ ions/cm^2 and the experimental results are shown in Figs.1 and 2, respectively. From Fig. 1, when the holding temperature of RTA increases from 1050°C to 1250°C in the $1x10^{16}$ cm^{-2} Si^+-implanted 400-nm-thick films after the dry-N_2 RTA for 5 seconds, the PL peak shifted from the blue band of ~2.5 eV to the orange band around 2.1 eV. Furthermore, at the same range of holding temperature as in Fig.1, Fig. 2 shows that the PL peak shifted from the near-infrared band of ~1.85 eV to ~1.5 eV in the $3x10^{16}$ cm^{-2} Si^+-implanted 400-nm-thick films after the dry-N_2 RTA for 20 seconds. Keeping the holding temperature in the range between 1000 °C and 1250 °C, a similar shift of PL peak (~2.5 eV to ~2.3 eV) as shown in Fig. 1 was observed in the 160 Kev Si^+-implanted 430 nm-thick SiO_2 films after CTA by the group of Mutti. et al.[5]. However, the silicon fluence was 1 x 10^{17} ions/cm^2 and the duration at those high temperatures was 1 hr. On the other hand, a similar band of PL peak (~1.5 eV) as the result in Fig. 2 were observed in the 150 Kev Si^+-implanted 800 nm-thick SiO_2 films after CTA at 1100 °C by Garrido et al[8]; but, the silicon fluence must be increased to ~3 x 10^{17} ions/cm^2 and the duration at the high temperatures was 8 hr. Besides, At the silicon fluece of 3 x 10^{17} cm^{-2}, Mutti. et al. can only produce the PL peak of ~1.65 eV in the 160 Kev Si^+-implanted 430 nm-thick SiO_2 film after CTA at 1000 °C for 5 hrs[5].

Figure 3 shows that the PL peak is shifted from ~2.1 eV to ~1.7 eV when the 3 x 10^{16} cm^{-2} as-implanted films are treated by the dry-N_2 RTA at 1150°C and the RTA duration is increased from 5 to 20 seconds,. At the same time, Fig. 4 shows that the PL peak is shifted from ~1.75 eV to ~1.7 eV in the 3 x 10^{16} cm^{-2} as-implanted films after CTA at 1150°C for the duration from 4 to 12 hrs. Referring to Fig.3, the results indicate that the band

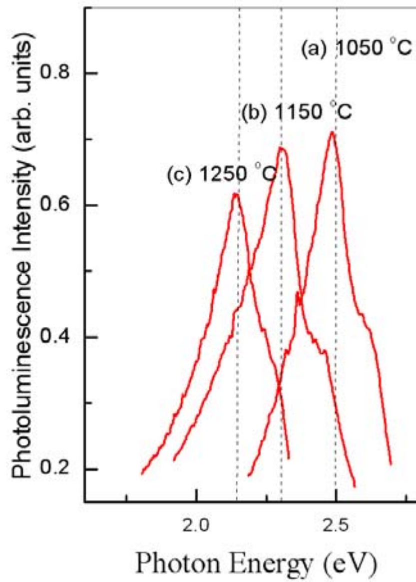

Figure 1. PL spectra of the Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si at a fluence of $1x10^{16}$ ions/cm^2 after dry-N_2 RTA for 5 seconds at (a) 1050°C, (b) 1150°C, and (c) 1250°C.

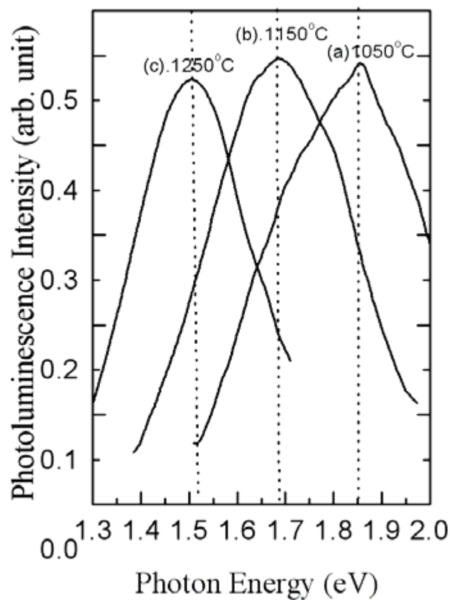

Figure 2. PL spectra of the Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si at a fluence of $3x10^{16}$ ions/cm^2 after dry-N_2 RTA for 20 seconds at (a) 1050°C, (b) 1150°C, and (c) 1250°C.

of PL peak can be decreased by increasing the RTA duration. However, the results in Fig. 4 indicate that the PL peak is weakly affected by the CTA duration although the intensity of PL spectra become large due to the increase in CTA duration,. The result agrees with that of the study by Shimizu-Iwayama et al.[6], in which the peak energy of the PL is found to be weakly dependent on annealing time, while the intensity of the luminescence increase as the annealing time increases. In addition, comparing Figs. 3 with 4, under the same Si^+-flux of $3x10^{16}$ ions/cm^{-2} and holding temperature of 1150 °C, the intensity of PL-peak in the as-implanted film after the RTA method for the holding duration between 5 and 20 seconds as shown in Fig. 3 is always bigger than that after the CTA method for the duration between 4 and 12 hrs as shown in Fig.4.

Figure5 shows the effect of Si^+ fluence on the luminescence in the RTA-treated film, where the PL spectrum of the Si^+-implanted 400-nm-thick SiO_2 films grown on silicon crystal at three different fluences after RTA in dry N_2 at 1150°C for 20 seconds. As the Si^+ fluence into the as-implanted film was $1x10^{16}$ ions/cm^2, the PL spectrum has a peak at ~1.95 eV shown in Fig.5(a); as those films have the fluence of $2x10^{16}$ and $3x10^{16}$ ions/cm^2, the PL spectra have the peaks at ~1.8 eV and ~1.7 eV, shown in Figs. 5(b) and 5(c), respectively. The results indicate that the PL peak can be red-shifted with the increase of fluences. The peak energy of the PL spectra is obviously decided by the fluence of the Si^+-implantation. Thus, the Si^+ concentration plays the important role during the production of the respective mechanisms in the RTA-treated films. On the other hand, Fig.6 shows the effect of Si^+ fluence on the luminescence

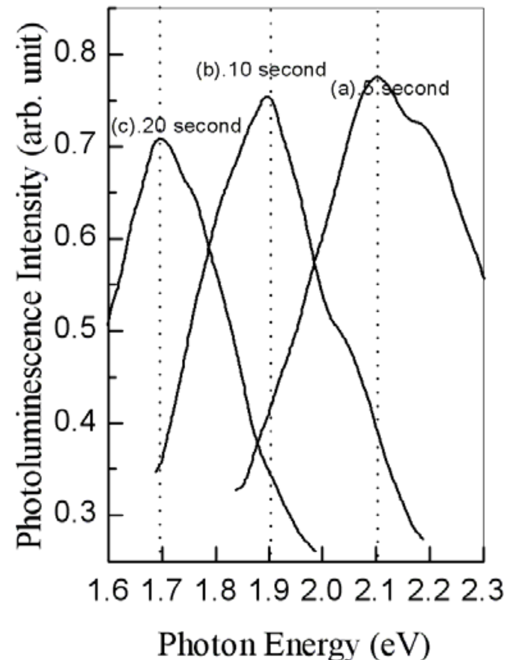

Figure 3. The PL spectra of the Si^+-implanted 400-nm -thick SiO_2 film grown on crystal Si at a fluence of $3x10^{16}$ ions/cm^2 after dry-N_2 RTA at 1150 °C for (a) 5 seconds, (b) 10 seconds, and (c) 20 seconds.

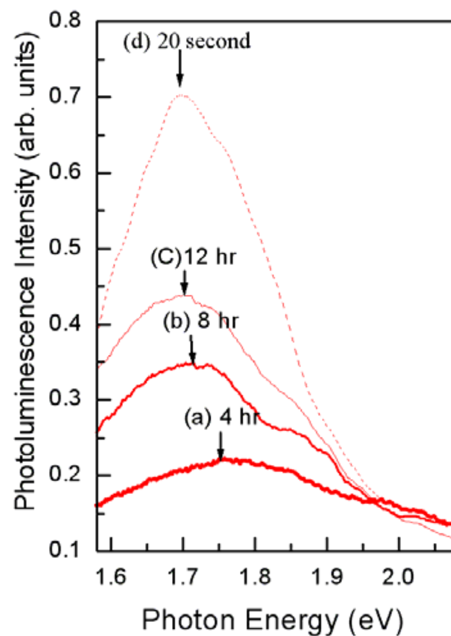

Figure 4. The PL spectra of the Si^+-implanted 400-nm -thick SiO_2 film grown on crystal Si at a fluence of $3x10^{16}$ ions/cm^2 after dry-N_2 CTA at 1150 °C for (a) 4 hours, (b) 8 hours, and (c) 12 hours and (d) after isothermal dry-N_2 RTA for 20 seconds.

in the CTA-treated film, where is also the PL spectrum of the Si^+-implanted 400-nm-thick SiO_2 films grown on silicon crystal at three fluences as above but after CTA in dry N_2 at 1150°C for 12 hrs. Referring to Figs.5 and 6, under the same holding temperature of 1150°C and the Si^+ -fluence between $1x10^{16}$ and $3x10^{16}$ ions/cm^2, the PL intensity of the as-implanted film by CTA has obvious increase upon increasing with the dose of implanted Si^+ but the respective PL Peak shifts only from ~1.75 eV to~1.70 eV as shown in Fig.6, which are always weaker than that by RTA as shown in Fig.5. The results show that the intensity of the PL spectrum is obviously affected

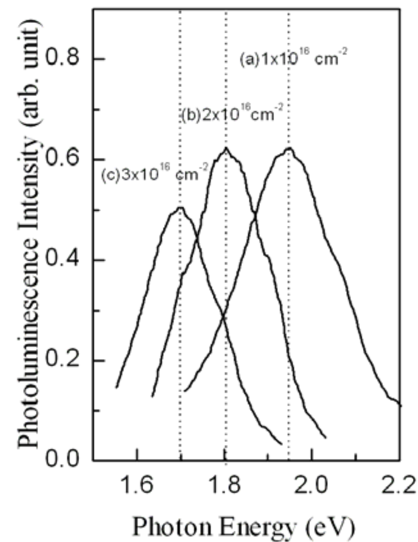

Figure 5. PL spectra of the Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si at a fluence of (a) $1x10^{16}$ ions/cm^2, (b) $2x10^{16}$ ions/cm^2, and (c) $3x10^{16}$ ions/cm^2 after dry-N_2 RTA treatment for 20 seconds at 1150°C.

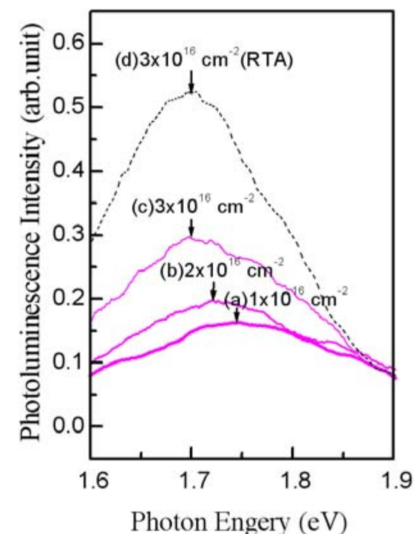

Figure 6. PL spectra of the Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si at a fluence of (a) $1x10^{16}$ ions/cm^2, (b) $2x10^{16}$ ions/cm^2, and (c) $3x10^{16}$ ions/cm^2 after dry-N_2 CTA for 12 hours at 1150°C and at a fluence of (d) $3x10^{16}$ ions/cm^2 after dry-N_2 isothermal RTA for 20 seconds.

by the concentration of implanted Si^+, but the peak energy is weakly affected by the concentration of implanted Si^+ in the CTA-treated film. Otherwise, the result shown in Fig.6 is consistent with the observation of several other research groups[6, 8, 14-15]. According to the observation of Shimizu-Iwayama et al.[6], although the intensity of PL is strongly affected by the dose of implanted Si^+, the peak energy of the PL that is weakly

affected by the Si^+-dose shifted from 1.75 eV to 1.7 eV as the Si^+-fluence implanted into the 600 nm-thick SiO_2 film with the energy of 180 KeV increases from $1x10^{16}$ to $2x10^{17}$ ions/cm^{-2} and the thermal annealing was carried out by the CTA at 1050 ^oC for 4 hrs. Furthermore, the shift of PL peak from 1.7 eV to 1.5 eV was obsedved by Fernandez et al.while the Si^+-fluence implanted into the 800 nm-thick SiO_2 film with the energy of 150 KeV increases from $1x10^{17}$ to $3x10^{17}$ ions/cm^2 and the thermal annealing was treated by the CTA at 1100 ^oC for 8 hrs [8]. Anyway, the red-shifting phenomena of PL can be observed by RTA, which can decrease the fluence of the Si^+- implantation and shorten the thermal annealing time as compared with CTA. Moreover, although the growth of PL mechanism in the as-implanted SiO_2 film treated by CTA is much slower than that treated by isothermal RTA, the mechanism in the film treated by the RTA or CTA method is dependent of Si^+ concentration in the oxide matrices and of the thermal annealing temperature and duration.

Remarkably, besides of the enhanced diffusion and aggregation of Si^+ implanted in oxide matrix during RTA [21], we believe that the procedure of Si^+-nanocrystals formation all be enhanced by using the RTA method but the effect is very sensitive to the heating rate of RTA. According to our initial observation, the PL intensity from the as-implanted film by RTA with the heating rate of 100 ^oC/s was much stronger than that by RTA with the heating rate of 50 ^oC/s and no significant intensity could be found after the heating rate of 25 ^oC/s. At the same time, the shift of PL peak from the as-implanted film by RTA with the heating rate of 100 ^oC/s could occur at the holding duration less than 20 seconds. However, after the heating rate decreasing to 50 ^oC/s, the shifting phenomena could occur only at the holding duration less than 5 seconds. The results conform to the observation by Shimizu-Iwayama et al.. They found that, under the Si^+ fluence of $\geq 5x10^{16}$ ions/cm^{-2}, the PL intensity of sample only after RTA at $\geq$ 1050 ^oC for 5 min with the heating rate of 30 ^oC/s was much weaker than that treated by the same RTA at first and achieved finally by CTA at $\geq$ 1050 ^oC for $\geq$ 1 hr [21].

On the other hand, Fig. 7 shows the FTIR spectra of the films with the different treated process. Figure 7(a) shows the FTIR spectrum in the 3750 cm^{-1} range of original 400-nm-thick SiO_2 film grown on the Si crystal without RTA. Besides, for the Si^+-implanted 400-nm-thick SiO_2 films with the same fluence of $3x10^{16}$ ions/cm^2, but proceeding the RTA in dry N_2 at 1150°C for 20 seconds and the CTA at 1150°C for 12 hours, the FTIR spectra in the 3750 cm^{-1} range are show in Figs. 7(b) and 7(c), respectively**.** Moreover, in order to confirm the absence of Si-O-H structures in the as-implanted samples by the RTA and CTA**,** the spectra in the 3750 cm^{-1} range of the Si^+-implanted 400-nm-thick SiO_2 films with the respective fluences of $1x10^{15}$ and $4x10^{15}$ ions/cm^2 after the RTA but in wet N_2 are displayed in Figs. 7(d) and 7(e). According to these FTIR spectra in Figs.7(b) and 7(c), the absorption valleys of Si-O-H structures in the dry-N_2-annealed films after above RTA and CTA, which are indicated by the signature at ~ 3750 cm^{-1} in the FTIR spectra[16-20], are both shallow as the sample of original wet SiO_2 films without RTA treatment shown as Fig. 7(a) and seem un-change with the increase of silicon implantation fluence. However, from Figs. 7(d) and 7(e), we observed that the absorption valleys of Si-O-H structures in the respective $1x10^{15}$ cm^{-2} and $4x10^{15}$ cm^{-2} Si^+-implanted SiO_2 films after RTA in a wet N_2 are deepened with the increase of Si^+-implantation fluence and so their respective mechanism in the films, that lead to the luminescence, are related closely to the Si-O-H structures[11, 12]. In addition, no samples with the above mechanisms in this study display an FTIR signature at ~2100 cm^{-1} and~1450 cm^{-1} (or ~2300 cm^{-1}), related to the absorption of SiH_x (x=1,3) and CH_x structures, respectively[13, 16]. Hence, the above mechanisms in the $3x10^{16}cm^{-2}$ Si^+-implanted SiO_2 films after the above dry-N_2 RTA and CTA are all independent of the Si-O-H related bonds and the SiH_x or CH_x structures according to the FTIR spectra.

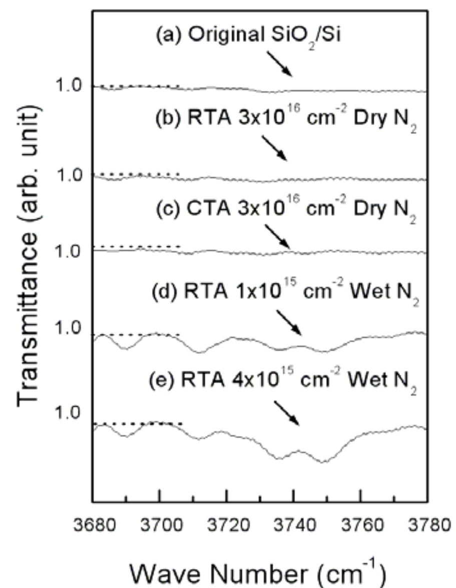

Figure 7. FTIR spectra in the 3750 cm^{-1} range of (a) original 400-nm-thick SiO_2 film grown on Si crystal without RTA, the Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si at a fluence of $3x10^{16}$ ions/cm^2 (b) after Dry-N_2 RTA at 1150°C for 20 seconds and (c) after Dry-N_2 CTA at 1150°C for 12 hours, and that at a fluence of (d) $1x10^{15}$ ions/cm^2 and (e) $4x10^{15}$ ions/cm^2 with RTA as in (b) but in wet N_2.

Otherwise, Fig. 8 shows the peak-intensity of the PL spectra as a function of the re-annealing temperature by RTA for 5 min in nitrogen. The PL peaks from the respective $1x10^{16}$ cm^{-2} as-implanted films achieved by the CTA (shown as Fig. 8(a)) and RTA (shown as Fig. 8(b)) both decay nearly one-half intensities after isochronal re-annealing at the temperature of 1000 ^oC

and both disappear since the temperature of 1100 °C. Furthermore, when the fluence is increased to $3x10^{16}$ ions/cm^2, the PL mechanism in the as-implant films achieved by the CTA (shown as Fig. 8(c)) and RTA (shown as Fig. 8(d)) can persist even after re-annealing at the temperature of >1100°C and both disappear at 1200 °C. The results of the above PL mechanism imply that the ratio of remainder number after the above re-annealing is ambiguously dependent of productive method (RTA or CTA) but depends on the Si^+ dose implanted into the SiO_2 film. Hence, under the same dose of implanted Si^+, the PL mechanism in the film achieved by RTA and CTA have similar existent ratio after isothermal and isochronal RTA test.

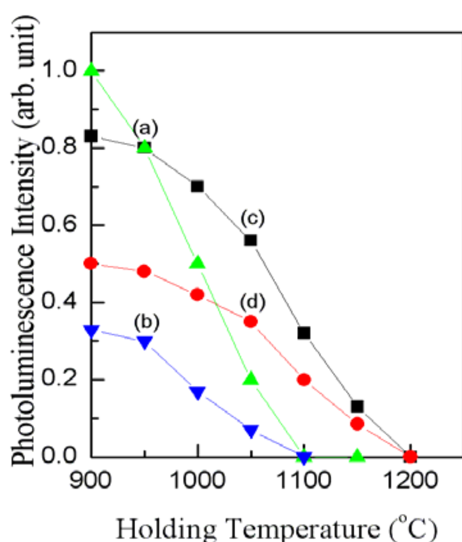

Figure 8. Changes in the PL intensity from the $1x10^{16}$ cm^{-2} Si^+-implanted 400-nm-thick SiO_2 film grown on crystal Si (a) after RTA at 1150°C for 20 seconds and (b) after CTA at 1150°C for 12 hrs and from the $3x10^{16}$ cm^{-2} as-implanted sample (c) after RTA at 1150°C for 20 seconds and (d) after CTA at 1150°C for 12 hrs as a function of the re-annealing temperature, respectively. Re-annealing of the these groups of specimens was achieved by RTA for 5 min with heating rate of 30 °C/s .

Finally, both the RTA and CTA methods can produce the mechanisms in the Si^+-implant SiO_2 film with the energy of PL peak between blue and near-infrared band. In addition, their respective PL peaks are both shifted with the increase of Si^+ concentration in the oxide matrices and of the thermal annealing temperature and duration. The two mechanisms are both independent of hydrogen-related bonds and have the similar existent ratio after the test of RTA at high temperature. Therefore, the respective mechanism in the Si^+-implanted SiO_2 films after RTA and CTA are similar. It is well known that Si ion implanted into SiO_2 and subsequent high-temperature CTA induce the formation of embedded luminescence Si nanocrystals[4-9, 21]. Hence, the mechanism in the as-implanted film,produced by RTA,was also attributed to silicon nanocrystals embedded in SiO_2.

4 Conclusion

The PL peak between ~2.5 eV and ~1.5 eV can be observed in the Si^+-implanted 400 nm-thick SiO_2 film after dry-N_2 RTA only. The studies indicate that light emission in the lower dose Si^+-implanted film with the ~30 minutes RTA has the similar shifting phenomena like that in the higher dose as-implanted film with the several-hours CTA method. Under the implanted Si^+ dose between $1x10^{16}$ cm^{-2} and $3x10^{16}$ cm^{-2} and the holding temperature of 1150 °C, the intensity of PL spectrum from the as-implanted film treated by the RTA method is always stronger than that treated by the CTA method. Furthermore, light emission from the as-implanted film by the two methods, in which the peak positions are both shifted with the variation of Si^+ concentration in oxide matrices and the thermal annealing temperature and duration. The PL mechanisms in the film achieved by RTA and CTA are both independent of hydrogen-related bonds and have similar existent ratio after isothermal and isochronal RTA test. The phenomena imply that the two methods produce the similar PL mechanism in the oxide matrix, that were both attributed to silicon nanocrystals embedded in SiO_2. Therefore, using RTA method to produce the mechanisms in the Si^+-implanted films with the energy of PL peak between blue and near-infrared band is better than using the CTA method.

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