



Characteristic photoluminescence properties of Si nanocrystals in SiO₂ fabricated by ion implantation and annealing

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Abstract

We have measured the implantation dose dependence as well as the oxidation effect of the photoluminescence behaviour of Si nanocrystals in SiO₂ layers fabricated by ion implantation and a subsequent annealing step. After annealing at high temperature, a characteristic photoluminescence band, peaking just below the 1.7 eV was observed. The peak energy and the intensity of the photoluminescence were found to be affected by the dose of implanted Si ions, but to be independent of annealing time and excitation photon energy. We also present experimental results of an oxidation induced continuous peak energy shift of the photoluminescence peak up to around 1.8 eV. This peak energy, however, was found to return to its previous position with re-annealing. These results indicate that whilst the excitation photons are absorbed by Si nanocrystals, the emission is not simply due to electron–hole recombination inside the Si nanocrystals, but is related to the presence of defects, most likely located at the interface between the Si nanocrystals and the SiO₂, for which the characteristic energy levels are affected by cluster–cluster interactions or the roughness of the interface. © 2001 Published by Elsevier Science Ltd.

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1. Introduction

In the past decade, there has been considerable interest in semiconductor nanostructures, especially porous Si [1–3] and Si nanocrystals [4–6] because of their potential applications toward Si-based optoelectronic devices. Si nanocrystals have been fabricated by a variety of methods and include such techniques as co-sputtering, chemical vapour deposition, molecular beam epitaxy, gas evaporation, laser ablation and so on. Nanometer-sized crystallites exhibit unique electrical, op-

tical, magnetic and thermal properties which are not observed in bulk materials. Although a considerable amount of research has been performed by many researchers world wide, the mechanism responsible for photoluminescence from these Si nanostructures is still unclear.

Utilization of porous Si in optoelectronic devices is doubtful because of its method of fabrication and structural fragility. However, one of the most promising approaches to producing Si nanocrystals, compatible with conventional microelectronic processing, may be by ion implantation. This technique has the advantage that a given number of ions can be placed at a controlled depth and distribution by changing the ion doses and acceleration energies [7,8]. Ion beam synthesis of Si nanocrystals is a potential candidate for manufacturing chemically stable and pure Si nanocrystals, not only for fundamental research, but also for applications in

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monolithically integrated Si-based optoelectronic devices.

The present authors have carried out a series of studies on the structural and the optical properties of high energy (1 MeV) Si-implanted silica glasses [9–11] and thermally grown oxide films on Si wafers [12–14]. We have shown that these specimens exhibit two luminescence bands in the visible range. One band is peaked around 2.0 eV, is observed in as-implanted specimens and those annealed around 600°C, and can be attributed to excess Si defects. The other peaks around 1.7 eV and is observed only after annealing at higher temperatures, and can be attributed to Si nanocrystals.

After our first reports [9], many reports [15–24] have appeared concerning the 1.7 eV luminescence in samples prepared employing the same technique of Si ion implantation into SiO₂ and subsequent high temperature annealing. More recently, a dose (implanted excess Si concentration) dependent photoluminescence peak energy shift has also been reported by present authors [25]. Although the 1.7 eV luminescence is evidently related to implanted Si nanocrystals formed by decomposition of the SiO_x phase with high temperature annealing, the detailed mechanism of the luminescence is not yet clear. The properties of the 1.7 eV luminescence band are similar in many respects to those of other Si nanostructures, and it is likely that the same mechanisms are responsible for the photoluminescence from these materials.

The photoluminescence arising from implanted Si nanocrystals in SiO₂ has been attributed by some investigations to quantum confinement [15–17], while others have concluded that surface states present in the interfacial layer between the Si nanocrystals and the surrounding oxide matrix play an important role in the emission processes [10,18,19]. This article extends our findings by reporting a wider range of dose dependence and oxidation effects on the photoluminescence and discussing a detailed mechanism for the photo-emission and tuning of the photoluminescence of the Si nanocrystals formed in SiO₂ by ion implantation, annealing and oxidation steps.

2. Experiment

The samples were prepared by implanting Si ions into oxidized Sb doped Si epitaxial layer (10 Ω cm, 10 μm) grown on p⁺-type Si wafers with a resistance of around 0.01 Ω cm (oxide thickness of 300 and 600 nm). Ion implants were made with a Whickham ion implanter over the energy range from 25 to 200 keV with a beam current of 570 μA (current density of about 28.5 μA/cm²). The results for two different types of samples are presented here. One type of sample (type A) was pre-

pared by the implantation of Si ions into an oxide film of 600 nm thickness at an energy of 180 keV with doses from 1.0×10^{16} to 2.5×10^{17} Si ions/cm². The other type of sample (type B) was prepared by implanting Si ions at six energies between 25 and 200 keV with varying doses to produce uniform excess Si concentrations of 5%, 10%, 15% and 20% in an oxide film of 300 nm thickness thermally grown on Si wafers. The expected depth profiles of implanted Si atoms in thermal oxide films on Si wafers of both types were estimated using the stopping and range of ions in matter (SRIM) [26], as shown in Fig. 1(a) and (b). All implementations were performed at room temperature.

The implanted samples were subsequently annealed at 1050°C in a flowing N₂ atmosphere for several hours to induce precipitation and the formation of Si nanocrystals. Some of oxide films containing Si nanocrystals were then etched off in a series of subsequent etch steps of several minutes, using serous HF solution at room temperature. Other samples were then oxidized at 1000°C in a flowing O₂ atmosphere for up to 90 min,

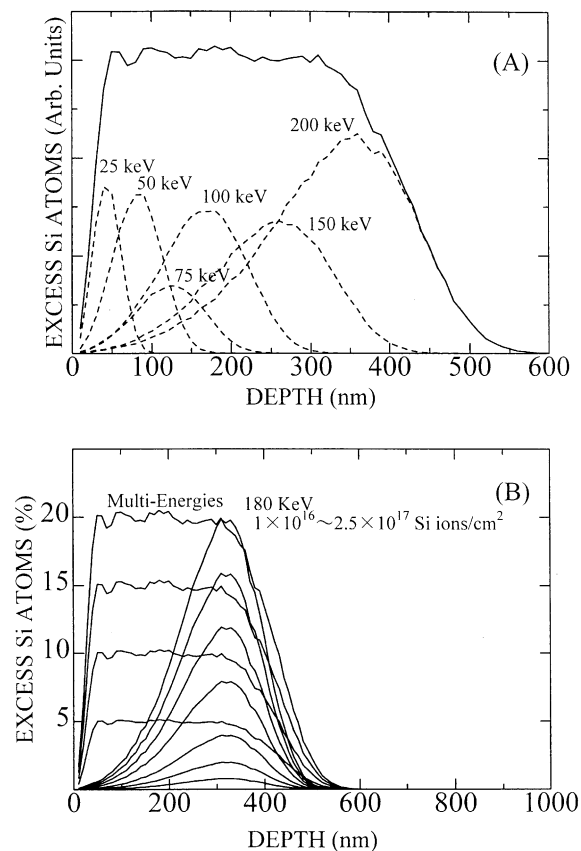


Fig. 1. Estimated depth profiles obtained by using SRIM program: (A) uniform profiles (sample B) and (B) all of samples used in the experiments.

and then re-annealed at 1050°C in a flowing N₂ atmosphere for 1 h. Other oxide films containing Si nanocrystals were etched. Conventional room temperature photoluminescence spectra were measured at various stages of the processing. An Ar-ion laser (2.41 or 2.54 eV) was used as the excitation source and the luminescence was detected by a cooled photomultiplier tube, employing the photon counting technique. Time-resolved photoluminescence measurements were performed using a YAG laser (2.33 eV) of 10 ns pulse width. Photoluminescence was detected by a cooled photomultiplier tube and stored by a digital oscilloscope.

3. Results

The photoluminescence spectra of type A samples after annealing at 1050°C for 8 h are shown in Fig. 2. The 2.54 eV laser line was used as the excitation source to obtain these spectra unless indicated otherwise. To compare the peak energies of the luminescence, the spectra are normalized at the peak height and the zero lines of some curves are shifted vertically, as shown in Fig. 3. Here we summarized the dose dependence of the photoluminescence peak energy and intensity (Fig. 4). It is clear from the figures that the peak energies of the luminescence spectra are strongly affected by the doses of implanted Si ions in the high dose range. The peak energies are close to 1.7 eV in samples with lower doses (below a dose of 5×10^{16} Si ions/cm²), but are shifted to lower energies with increasing dose of implanted Si ions. Moreover, the intensity of the luminescence is also

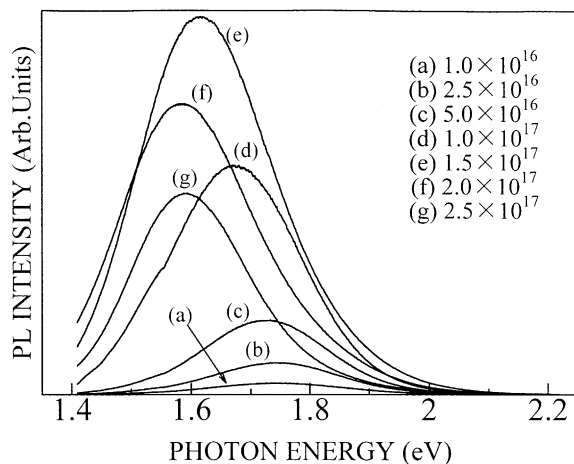


Fig. 2. Photoluminescence spectra of 180 keV Si implanted 600 nm thermal oxide films to doses of (a) 1.0×10^{16} , (b) 2.5×10^{16} , (c) 5.0×10^{16} , (d) 1.0×10^{17} , (e) 1.5×10^{17} , (f) 2.0×10^{17} and (g) 2.5×10^{17} Si ions/cm² and excited with a 2.54 eV laser, after annealing at 1050°C for 8 h in a N₂ atmosphere.

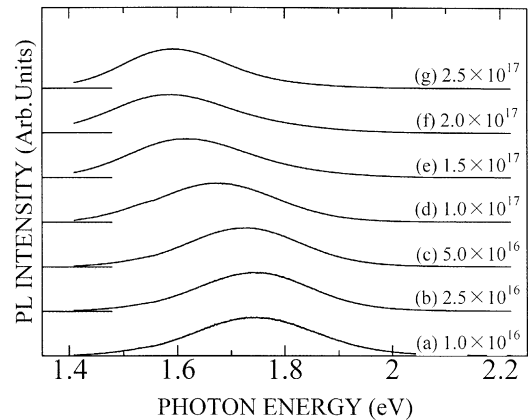


Fig. 3. Normalized photoluminescence spectra of 180 keV Si⁺-implanted 600 nm thermal oxide films at doses of 1.0×10^{16} , 2.5×10^{16} , 5.0×10^{16} , 1.0×10^{17} , 1.5×10^{17} , 2.0×10^{17} and 2.5×10^{17} Si ions/cm² (from bottom to top) and excited with a 2.54 eV laser, after annealing at 1050°C for 4 h. The zero lines of some curves are shifted vertically.

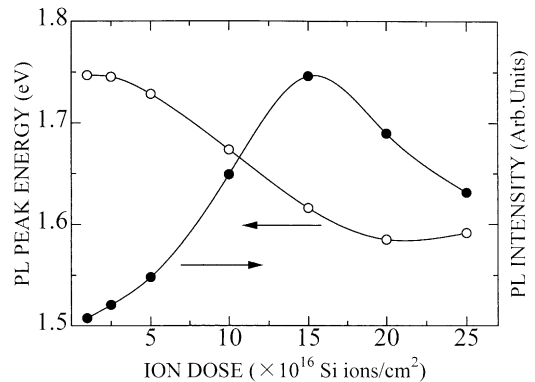


Fig. 4. Comparison of the photoluminescence peak energy and the photoluminescence intensity for Si nanocrystals formed by implanting 180 keV Si to different doses, after annealing at 1050°C for 8 h in a N₂ atmosphere.

affected by the dose of implanted Si ions. Maximum luminescence intensity can be obtained at a level around 1.5×10^{17} Si ions/cm².

Sample of Si implanted to a dose of 1.5×10^{17} ions/cm² and annealed at 1050°C in a flowing N₂ atmosphere for 8 h was then etched off in HF solution, and photoluminescence spectra were measured at various stages of the etching processing. Fig. 5 shows normalized difference photoluminescence spectra obtained by subtracting the photoluminescence spectra for subsequent etch steps. As the etching ratio depends on the local excess Si concentrations, here, we only indicate the etching time. As you can see in the figure, the peak energies of the photoluminescence once shift to lower energy side and

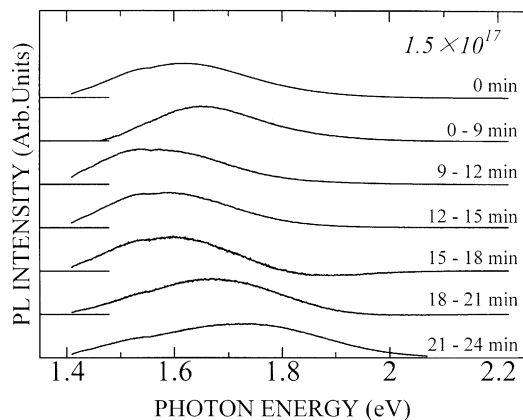


Fig. 5. Normalized difference spectra of 180 keV Si implanted 600 nm thermal oxide films to a dose of 1.5×10^{17} Si ions/cm² after annealing at 1050°C for 8 h, obtained by subtracting the photoluminescence spectra for subsequent etch time indicated in the figure.

back to higher energies. This results are consistent with those shown in Fig. 3.

For the purpose of discussing the detail correlations between the concentrations of implanted excess Si atoms and the luminescence, we also obtained the photoluminescence spectra of Si⁺-implanted layer with uniform excess Si concentrations. The photoluminescence spectra of type B samples after annealing at 1050°C for 8 h are shown in Fig. 6. It is also clear from the figure that the peak energies and the intensities of the luminescence are strongly affected by the concentrations of implanted excess Si atoms. The peak is shifted to lower energies with increasing concentration of excess Si atoms, similar to that observed in type A samples.

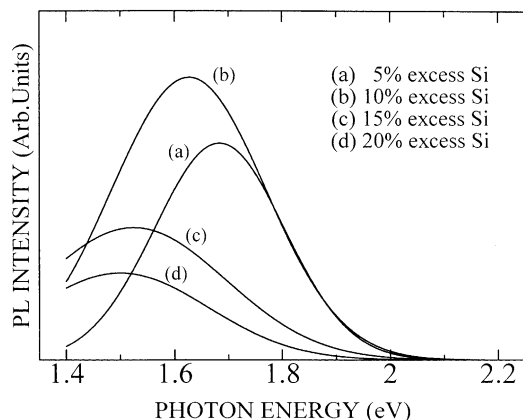


Fig. 6. Photoluminescence spectra of type B samples with the concentrations of 5%, 10%, 15% and 20% implanted excess Si atoms and excited with a 2.54 eV laser, after annealing at 1050°C for 8 h.

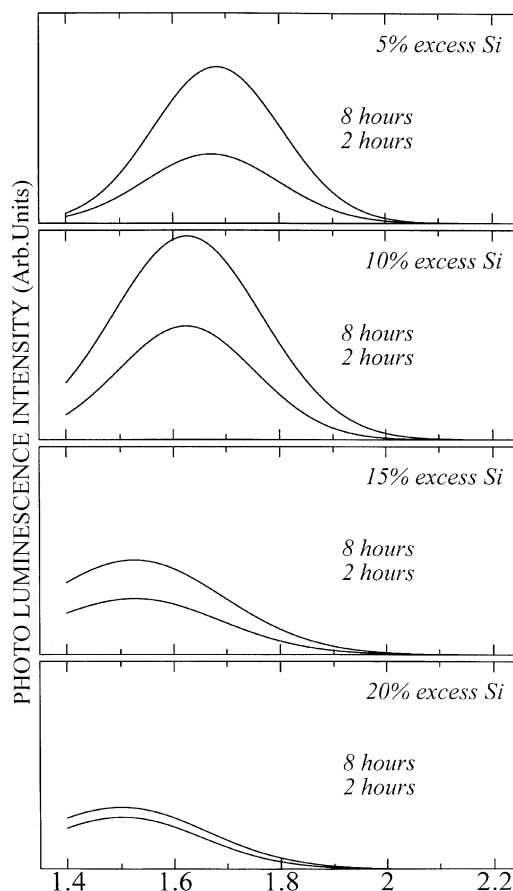


Fig. 7. Photoluminescence spectra of type B samples. 5%, 10%, 15% and 20% excess implanted Si atoms and excited with 2.54 eV laser, after annealing at 1050°C for 2 (down) and 8 h (up).

The time evolution of the photoluminescence spectra of type B samples with annealing at 1050°C for several hours are shown in Fig. 7. It is clear from the figures that the luminescence intensity grows as the annealing time increases and the peak energies of the luminescence spectra are independent of the annealing time. Luminescence spectra were also generated using both the 2.41 and 2.54 eV excitation laser lines for these samples. One of the results is shown in Fig. 8. It is clear that the peak luminescence energies are independent of excitation energy. The only effect of the different excitation energies is a decrease in the luminescence intensity. Similar experimental results were obtained in all the samples investigated in the present experiments.

Fig. 9 shows an example of recorded photoluminescence decays at some selected photon energies for a type B sample with 10% excess Si after annealing at 1050°C in a flowing N₂ atmosphere for 8 h. We cannot fit them to simple exponential. The lifetime of the photoluminescence increase with decreasing emitted photon energy.

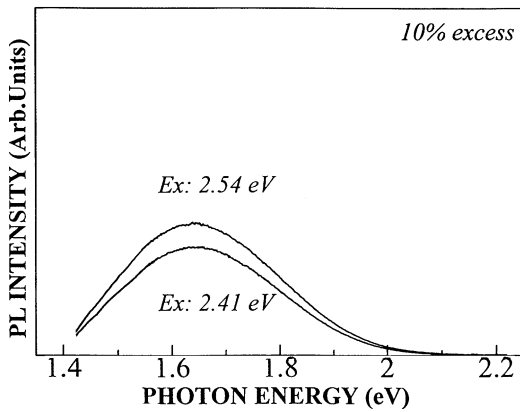


Fig. 8. Photoluminescence spectra of type B sample with 10% excess Si and excited with 2.41 and 2.54 eV laser, after annealing at 1050°C for 8 h.

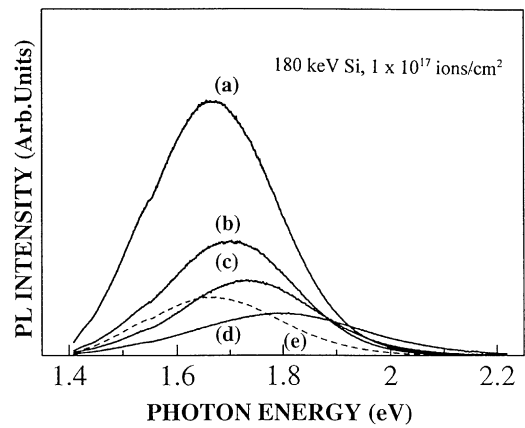


Fig. 10. Photoluminescence spectra of samples implanted with 180 keV Si to a dose of 1.0×10^{17} ions/cm² and excited with a 2.54 eV laser, (a) after annealing at 1050°C for 8 h in a N₂ atmosphere and subsequent annealing at 1000°C for (b) 30, (c) 60 and (d) 90 min in an O₂ atmosphere, and (e) re-annealing at 1050°C in a N₂ atmosphere for 1 h.

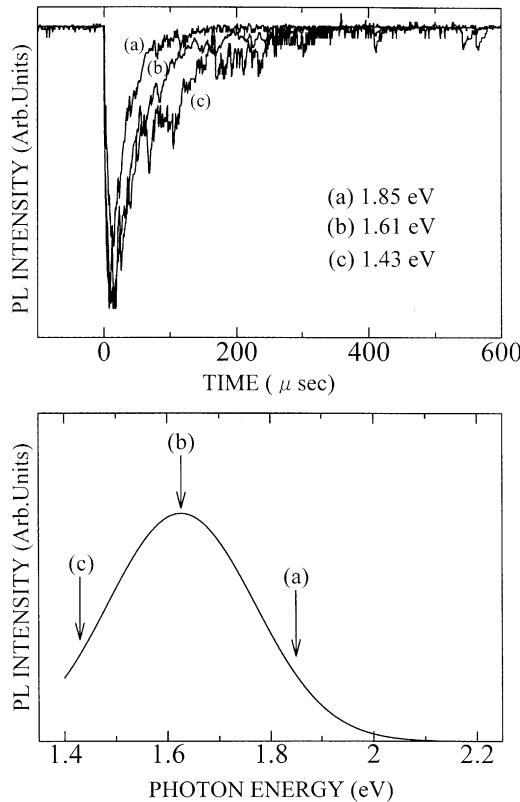


Fig. 9. Time-resolved photoluminescence decay from type B sample with 10% excess Si and excited with 2.33 YAG laser, after annealing at 1050°C for 8 h. The arrow indicate the position of the measurements.

These lifetime dependencies on the photon energy are also very similar to the ones observed for porous Si and reveal nonexponential shape. Detailed time-resolved

photoluminescence measurement results will be reported elsewhere [27].

The photoluminescence spectra of type A sample implanted to a dose of 1.0×10^{17} ions/cm² at room temperature after annealing at 1050°C in a N₂ atmosphere for 8 h, and subsequently annealing at 1000°C in an O₂ atmosphere for 30, 60 and 90 min are shown in Fig. 10. In the figure, the spectra of samples re-annealed at 1050°C in a N₂ atmosphere for 1 h are shown as dashed lines. It is clear from the figure that the peak energies of the photoluminescence spectra continuously shift to higher energies up to around 1.8 eV and the tails of the spectra extend to 2.2 eV. Moreover, the photoluminescence bandwidth becomes much broader and the peak intensity of the photoluminescence decreases with increasing oxidation time. It is also clear that the peak energy of the photoluminescence go back to almost the same energy observed before oxidation, while the intensity levels do not recover with re-annealing in a N₂ atmosphere. It is noted that similar peak shift with oxidation were observed in samples with other Si doses.

4. Discussion

In this section, the correlation between the microstructure of the Si-implanted layer and the luminescence band, and the detailed mechanism for the photoemission and the peak energy shift of the photoluminescence is discussed. Initially, it is useful to review our previous experimental results of cross-section high-resolution transmission electron microscopy [14], together with

those of others [28–30]. We have previously reported that no trace of the formation of crystalline Si is evident in the Si-implanted samples before high temperature heat treatment. After treatment at high temperatures, however, electron micrographs indicated the presence of Si nanocrystals in an amorphous SiO₂ matrix for samples implanted with 1 MeV Si ions to a dose of 2×10^{17} ions/cm² (peak excess Si concentration of about 8%) around the depth of the projected range of the implanted Si in SiO₂. Moreover, we also observed an increase in the size of Si crystallites after extending the annealing time.

The increase in size and also the number of Si nanocrystals when increasing the ion dose for samples implanted with a few hundreds keV energy Si ions was confirmed by White et al. [28,29]. More recently, we obtained cross-section high-resolution transmission electron micrograph of the present samples [30] and confirmed the increase of size with increasing ion concentration and annealing time. Brongersma et al. [31] have also reported that annealing of 35 keV Si-implanted samples in an O₂ atmosphere at 1000°C after high temperature annealing without O₂ results in oxidation and a reduction in the size of the Si nanocrystals in SiO₂. These results clearly indicate that the photoluminescence band observed around 1.7 eV is related to the formation and behaviour of Si nanocrystals created from the implanted species.

4.1. Implantation dose dependent photoluminescence peak energy shift

Firstly, we discuss the implantation dose dependence of the photoluminescence of Si nanocrystals. From the experimental results of the dose dependent peak energy shift of the photoluminescence alone, it seems reasonable to assume that the origin of the photoluminescence is due to quantum confinement effects and the peak shift could be explained by the change in size of the Si nanocrystals. However, as we mentioned above, the size of Si nanocrystals depends on both the implantation dose (concentration) and the annealing time. An important point to note is that the luminescence intensity grows during annealing, without changing the peak energy of the spectra, as shown in Fig. 7. Since the Si nanocrystals grow as the annealing time increases, the lack of dependence of the peak energy on the annealing time excludes the possibility that the luminescence is simply due to the direct recombination between electrons and holes confined inside the Si nanocrystals. Other groups have suggested that the absorption of photons leads to the generation of electron–hole pairs which are confined within the Si nanocrystals, whilst the emission of photons arises from surface states localized at the interface between the Si nanocrystals and the SiO₂ matrix [32,33]. However, with these models, we

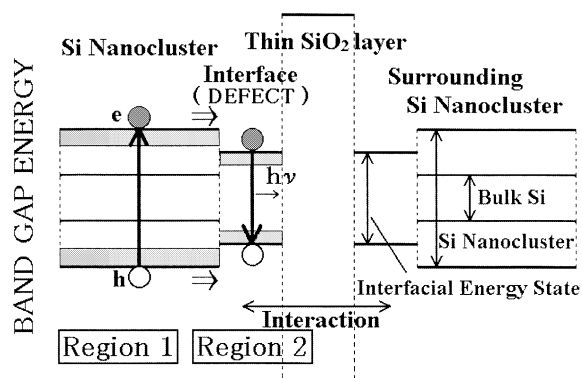


Fig. 11. Schematic illustration of a reactive nanocluster model of photoluminescence in which the nanoclusters react via a thin oxide interface.

cannot explain our observed dose dependent shift of the photoluminescence.

We have recently proposed an alternative model (reactive nanocluster model) [25] to explain the peak shift of the photoluminescence, as shown in Fig. 11. In this approach, we consider that the band gap widening due to the quantum confinement effect plays an essential role in the photoabsorption process and the interface energy state between Si nanocrystals and thin SiO₂ layer, for which the energy levels are affected by cluster–cluster interactions via a thin oxide layer, plays an essential role in the luminescence process. Here we would like to stress the importance of the local concentration of Si nanocrystals. If the Si nanocrystal population is very dense, the nanocrystals interact with each other via a thin oxide and a decrease in the interface energy level should be expected. With larger implanted Si doses, of course, the local concentration of Si atoms before annealing is much higher, and this contributes to an increase in both the size and the number of Si nanocrystals after annealing, as observed by others [28,29]. However, once the nucleation of Si aggregates occurs by decomposition of SiO_x, a point would be reached where the Si nanocrystals formed would not migrate within the SiO₂ matrix.

Based on these assumptions, we can explain the implantation dose dependent peak shift of the photoluminescence as follows: because the number of Si nanocrystals formed is almost fixed after prolonged annealing and only growth in the size of crystallites occurs, the change in the distance between adjacent nanocrystals is negligible, even if the size increases. In contrast to this, with an increase in the dose of implanted Si ions, number of Si crystallites increases. Therefore the distance between the nanocrystals changes, and cluster–cluster interactions via a thin oxide can be expected. Below a dose of 5×10^{16} ions/cm² at an energy of 180 keV, the peak energy is almost fixed at around 1.7 eV. This energy seems to be the interfacial

energy level between Si nanocrystals and the SiO₂ matrix without any cluster–cluster interactions.

Now, we discuss the dose dependent change in photoluminescence intensity. For the case Si nanocrystals, the luminescence intensity is determined by the number of (proper-sized) Si nanocrystals and/or their luminescence efficiency, if we assume that their absorption cross-section is the same at the excitation energy (2.54 eV). Up to a dose around 1.5×10^{17} ions/cm², the photoluminescence intensity increases as the dose increases, as shown in Fig. 4. The number and the growth of the Si nanocrystals are expected to increase as the dose increases. Thus the initial intensity increase with implanted Si ion dose, as shown in Fig. 4, is consistent with the presumption. Above a dose of 1.5×10^{17} ions/cm², the intensity of photoluminescence reduces as the dose increases. There are several possible reasons for this. Firstly the band-to-band transition energy of the confined Si nanocrystal system should be smaller than the emission energy with the growth of the Si nanocrystals. Secondly the interaction between the nanocrystals affects the photoluminescence efficiency. Thirdly as the size increases the interface-to-volume ratio decreases. Fourthly the probability of the energy transfer to the interface will decrease, particularly if the nanocrystals include imperfections. In any case we expect that the highest photoluminescence intensity is observed from the samples when the Si nanocrystals have been properly distributed in an SiO₂ matrix at an appropriate size.

4.2. Oxidation induced photoluminescence peak energy shift

Next, we discuss the oxidation induced peak energy shift of the photoluminescence spectra of the Si nanocrystals. Since the size of the Si nanocrystals evidently decreases as the oxidation time increases, it seems that the origin of the peak energy shift of the photoluminescence is due to quantum confinement effects, as others have already concluded [31]. An important point, however, to note is that the photoluminescence peak energy returns to its previous peak position after re-annealing in a N₂ atmosphere. With annealing in an O₂ atmosphere, of course, the size of the Si nanocrystals decreases and this also contributes to an oxidation of remaining excess Si atoms. If we assume that the origin of the peak energy shift of the photoluminescence is due to quantum confinement effects and the photoluminescence is simply due to the direct recombination between electrons and holes confined inside of Si nanocrystals, it means that the size of the Si nanocrystals returns to their previous size after re-annealing in a N₂ atmosphere. The recover of the size of the Si nanocrystals to their previous size seems not to be so easy by annealing samples with such a deficient excess Si concentration and it strongly suggests that other factors, but not the size of

the Si nanocrystals, affect the peak energy shift of the photoluminescence spectra with oxidation.

As we have already discussed, a photoluminescence peak energy above ~ 1.7 eV will not be expected with our model. It seems, therefore, that a change in the interfacial energy states with oxidation induces the observed change in the photoluminescence peak energy. It is noted that the excitation energy dependence of this photoluminescence band is different from that observed in the as-implanted samples and is not related to the Si nanocrystals perse but can be attributed to excess Si defects. The peak energy of the photoluminescence band observed before thermal annealing depends on excitation energy, but that observed after oxidation does not. This indicates that the origin of the photoluminescence is different from that observed before annealing and it seems that the incident photons are absorbed by Si nanocrystals.

It is known that the luminescence emitted by recombination of the self-trapped excitons in crystalline SiO₂ peaks at 2.8 eV [34–36]. This luminescence is thought to be due to the recombination of an electron on the Si dangling bond and a hole on the O dangling bond, which are generated by breaking of Si–O bond [37,38]. The similar luminescence band in amorphous SiO₂ has been shown to peak around 2.3 eV [39]. Moreover, the luminescence band observed around 2.0 eV in Si-implanted SiO₂ before high temperature annealing, in which the peak energy of the luminescence depends on both the implantation dose and the substrate temperature during implantation, can be attributed to the Si excess defects formed in SiO₂ [10–12]. It is suggested that the peak energy of the luminescence in all these Si–O systems is sensitive to a variety of local disorders which include both structural and bond disorder. The arguments above suggest that the photoluminescence observed above 1.7 eV after oxidation is due to the recombination of electrons and holes at the interface between Si nanocrystals and SiO₂, with different oxidation states to those before oxidation. These oxidation states seem to be metastable and easily transformed to more stable states by annealing in a non-oxygen ambient. These experimental results indicate that the peak energies of the photoluminescence above 1.7 eV are strongly affected by the roughness of the interface.

5. Conclusion

We have measured the implantation dose, implantation temperature dependence and oxidation effect of the photoluminescence of Si nanocrystals in SiO₂ layers, fabricated by ion implantation and subsequent annealing and oxidation steps. We found that the peak energy of the photoluminescence was almost independent of the size of the Si nanocrystals if they have an appropriate

size (i.e. the band-to-band transition energy of the confined Si system should be smaller than the incident photon energy and larger than the emission photon energy), but strongly affected by the distance between them (i.e. their concentration) for the photoemission below 1.7 eV, or the roughness (oxidation state) of the interface for the photoemission above 1.7 eV. This method of fabricating Si nanocrystals by ion implantation and subsequent high temperature annealing is found to be an important technique that enables the discrimination of some factors which affect the peak energy of the photoluminescence spectra. Further studies of the size and distribution of the Si nanocrystals in these Si implanted SiO₂ layers by cross-section high-resolution transmission electron microscopy are now in progress.

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