The Properties of LPCVD SiO_2 Film Deposited by SiH_2Cl_2 and N_2O Mixtures

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ABSTRACT

The distribution of Cl in LPCVD SiO_2 film deposited by SiH_2Cl_2 and N_2O was measured by IMMA. Cl was distributed broadly near the SiO_2-Si interface even in unannealed film. In thick LPCVD SiO_2 films on Si, surface roughness was observed after thermal oxidation. Cl was markedly piled up near the interface between the thermally grown oxide and the Si substrate. In thin films, surface roughness induced by oxidation was not observed. Cl distribution after oxidation was almost the same as that of unannealed film. The Cl pile-up is not caused by oxidation, but by the existence of the Si-SiO_2 interface. The cause of surface roughness is attributed to a smaller concentration of oxygen than Cl at the interface.

CVD (chemical vapor deposited) SiO_2 film, which is indispensable to Si planar technology, has been formed conventionally by the reaction of SiH_4 and O_2 at about 400°C at atmospheric pressure. LP (low pressure)-CVD SiO_2 film formed by the reaction of SiH_2Cl_2 and N_2O at about 900°C has been examined, because it is denser, its thickness is more uniform, and because samples are easier to produce compared with conventional CVD SiO_2 film (1). Oxidation treatment after deposition of the LPCVD SiO_2 film on Si, however, induces surface roughness. Many bumps on the SiO_2 surface and many etch pits on Si are observed.

Surface roughness may be caused by HCl or Cl_2 produced by the decomposition of SiH_2Cl_2, since similar phenomena have been observed in HCl oxidation (2-4). There may be a high concentration of Cl ions in the LPCVD SiO_2 film. In this paper the Cl distribution in the LPCVD SiO_2 film was investigated by ion microprobe mass analyzer (IMMA). Cl distribution, the relation between surface roughness and Cl distribution, and the mechanism of surface roughness were studied before and after oxidation and before and after N_2 anneal. To investigate the Cl distribution in the LPCVD SiO_2, whose growth mechanism is different from that of thermal SiO_2, we studied the effect of Cl on the SiO_2-Si interface. We are thus able to determine under which conditions useful LPCVD SiO_2 film could be produced.

Experimental

LPCVD SiO_2 films were formed on silicon wafers by the reaction of SiH_2Cl_2 and N_2O at 0.6 Torr at about 900°C. The two reactant gases, controlled by mass flowmeters, were mixed in the reactor. The evacuation system consisted of a mechanical booster pump and rotary pump. The flow rate of SiH_2Cl_2 was varied from 15 to 50 cm^3/min. The N_2O flow rate was fixed at 100 cm^3/min. The growth rate was from 55 to 70 Å/min. The films were deposited on 2 in. Si wafers set vertically on a quartz boat, perpendicular to the gas flow. Thickness of the film was measured by ellipsometry. The index of refraction of the film, n, was from 1.44 to 1.46. Infrared spectrum of the film showed the absorption at 9.4 μm caused by the Si—O bond. The absorption of Si—N bond was not observed at 12 μm.

To investigate the Cl redistribution of the film, the samples were oxidized in dry O_2 ambient or annealed in N_2 ambient. Since the samples were oxidized or annealed in the furnace different from that used in deposition, the possibilities of Cl in these ambients were eliminated.

The different flow rates of SiH_2Cl_2 in our growth condition did not influence the chlorine concentration and distribution.

The depth profile was analyzed as follows. The surface of the sample was coated by Au evaporation to reduce charge accumulation and sputter-etched with a focused primary ion beam (~18.5 keV, O_2^+, 20 μm^{ϕ}). In order to get a uniform sputtering yield the beam was raster-scanned over a small area on the sample surface (400 × 320 μm). The electronic aperture system was used to eliminate the crater-wall effect. Integrated $^{35}Cl^+$ secondary ion intensity was measured during a 40 sec period. At the same time, $^{28}Si^+$ signals in the matrix were counted to normalize the Cl ion intensity. The depth of the crater was measured by Talystep with a mechanical stylus. The sputtering rates were approximately constant *i.e.*, R_s = 4 Å/sec. Although previous researchers have experienced insulator sample charging problems which have caused the redistribution of mobile ions, we did not experience this phenomenon, and we were able to confirm that the Cl ions had not been redistributed by the as-implanted profile of Cl in SiO_2 film about 1000Å thick. The instrument used in this study was an Applied Research Laboratories ion microprobe mass analyzer.

Results

Characteristics of Cl distribution.—Figure 1a shows Cl distribution in an LPCVD SiO_2 film of 2400Å. Figure 1b shows the Cl distribution after the N_2 anneal at 1100°C for 1 hr. The two films were chemically pre-etched to prevent accumulation of an electrical charge on the film. We found that Cl ions are not uniformly distributed at all depths and the peak concentration is about 10^{19} ions/cm^3.

The maximum Cl ion intensity in the LPCVD SiO_2 film appeared to be at about 500Å from the SiO_2-Si interface. This is subjected to an error of about 100Å, however, because the interface cannot be precisely monitored by Si ion intensity. Si ion intensity gradually changes near the SiO_2-Si interface because of the difference of the sputtering rate and the yield of SiO_2 and Si.

To verify the IMMA measurement, we measured the Cl ion intensity of Cl ion-implanted SiO_2 (implantation conditions: 60 keV, 5 × 10^{15} ions/cm^2) film deposited by SiH_4 and O_2 at 420°C. The measuring conditions were the same as Fig. 1. Figure 2 shows the resulting Cl distribution. This profile agrees with the Gaussian profile which is based on the LSS theory. Maximum ion intensity is near the theoretically predicted depth of 515Å. This result confirms that the ion intensity of

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Key words: CVD silicon dioxide low pressure, silicon dioxide chlorine distribution, Si-SiO_2 interface, thermal oxidation.

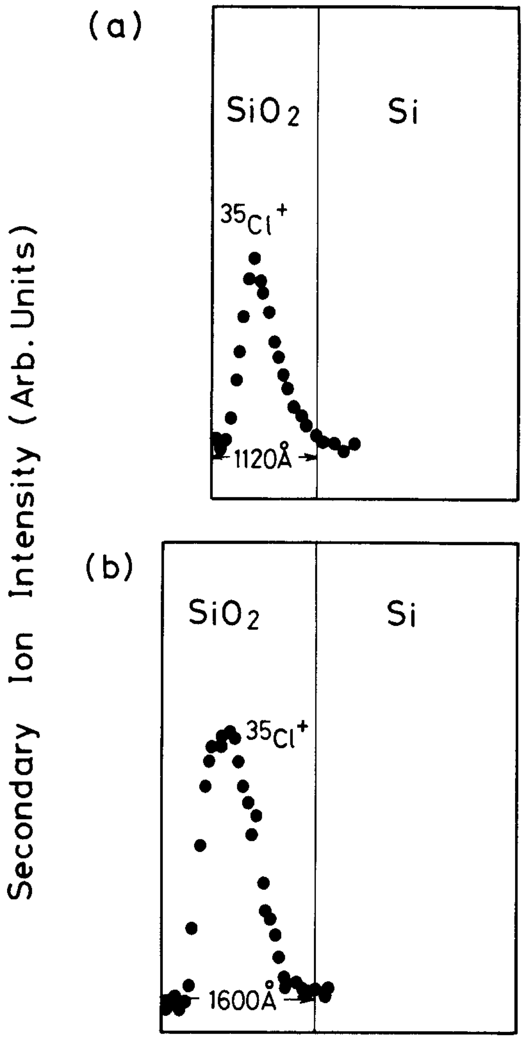

Fig. 1. Cl distribution of a chemically pre-etched LPCVD SiO_2 film (a) as deposited (b) after N_2 anneal at 1100°C for 1 hr.

secondary ion $^{35}Cl^+$ has a linear relation to the Cl concentration.

To verify the accuracy of the Cl^{35} in-depth profile in thick SiO_2 film, eight points (Q21 to Q28) on the sample surface were analyzed at different depths. The results are shown in Fig. 3a. In this figure, *N* represents the number of sampling times. Figure 3b shows the in-depth profile of the same sample, but the areas analyzed were ion etched prior to depth-profile analysis to determine the influence of ion etching on these points. The resultant profile of Cl distribution is reproducible. Distribution in nonetched sample is the same as that in etched samples. The number of Cl ions escaping from the side walls of craters is negligible and the knock-on Cl ions or electrical fields induced by sputtering do not affect Cl distribution. Figures 3a and b show that Cl has a flat distribution near the SiO_2 surface and piles up near the interface.

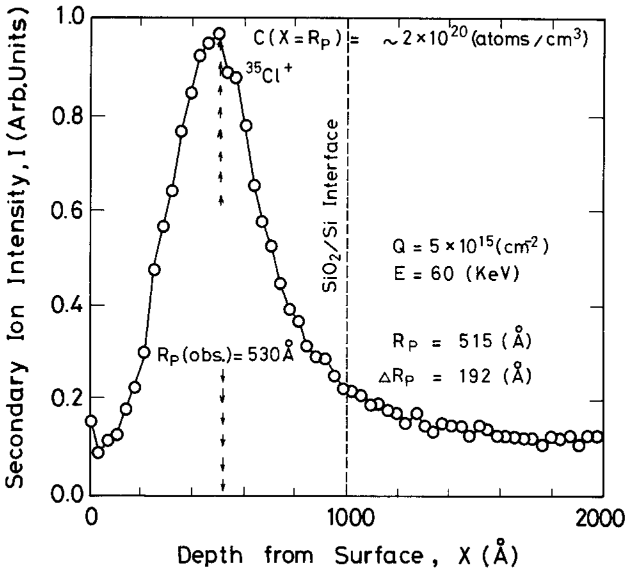

Fig. 2. Cl ion intensity of Cl ion-implanted CVD SiO_2 as deposited.

The Cl distribution in samples of three different thickness, 1500, 2600, and 6000Å, before and after thermal oxidation is shown in Fig. 4. The characteristics of the Cl distribution are as follows: (*i*) Cl ions are distributed broadly near the interface as the film is deposited. Cl distribution is flat near the surface. (*ii*) Before annealing the magnitude and profile of Cl distribution near the interface are independent of the film thickness. (*iii*) Cl ions move toward the newly formed interface as the thermally oxidized layer grows. (*iv*) In the 6000Å film, Cl concentration near the interface increases and its profile becomes sharp after oxidation. The additional Cl ions come from outside the pile-up region. In the 1500 and 2600Å films, the Cl profile remains unchanged after oxidation, although, of course, distribution shifts to the new interface. (*v*) Cl distribution remains unchanged after the N_2 anneal at 1100°C for 1 hr.

Surface roughness is observed only in the 6000Å film. This can be attributed to the remarkable piling-up of Cl at the interface. The surface roughness induced by oxidation is accompanied by the redistribution of Cl. (In thin films surface roughness is not observed after a 1 hr oxidation.) Pancake shapes in thick LPCVD SiO_2 films, as shown in Fig. 5a, are observed after oxidation. Their diameter is from 5 to 10 μm, measured by Talystep. The center of the risen area is about 3000Å high. Many etch pits are observed on the silicon surface after removal of SiO_2 by diluted HF, as shown in Fig. 5b. The approximately 300Å deep pits are square or rectangular because of the anisotropic etching to the Si(100) wafer. A schematic drawing of a section of the SiO_2-Si structure is shown in Fig. 6. We can assume that SiO_2 thickness is constant over the convex and concave regions because the oxygen intensity in these regions, measured by EPMA, is constant.

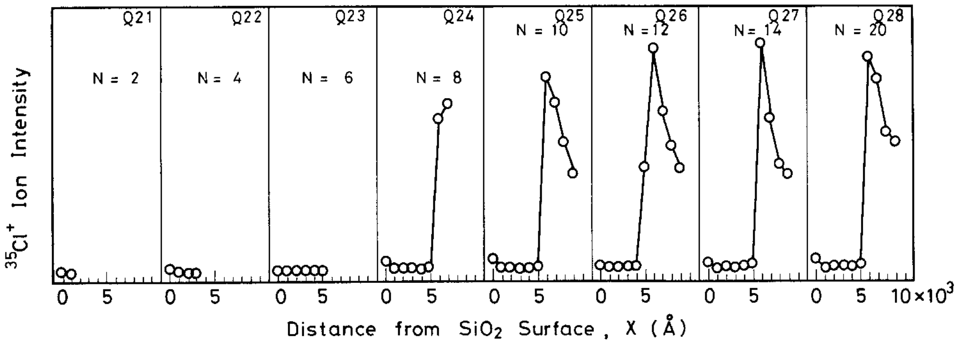

Fig. 3. (a) Cl ion distribution at eight different places (Q21-Q28) on LPCVD SiO_2 surface.

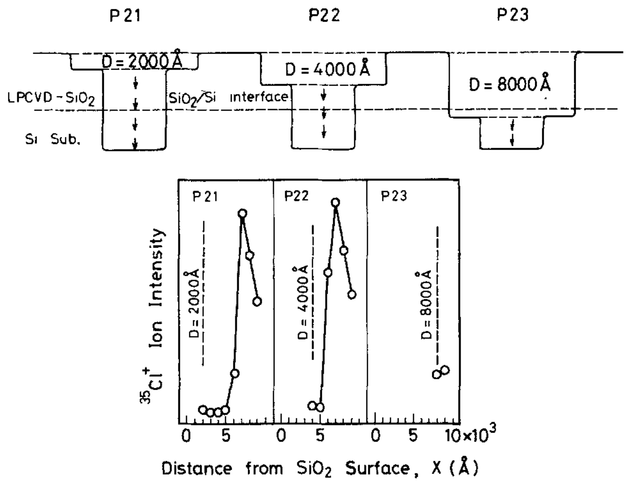

Fig. 3 (b) Cl distribution after ion etching of LPCVD SiO_2 to three different depths, DÅ.

After N_2 annealing of the film surface roughness is not observed. Oxidation following N_2 annealing, however, induces surface roughness. The fact that Cl distribution remains unchanged after N_2 annealing suggests that no surface roughness would be produced after the annealing, but that surface roughness would be induced after oxidation.

The influence of Cl distribution.—The influence of Cl ions in SiO_2 on the etching rate and on flatband voltage V_{FB} was investigated. The rate of etching to LPCVD SiO_2 and to conventional CVD SiO_2 is shown in Fig. 7. The rate of etching to CVD SiO_2 (deposited by SiH_4 and O_2 at 420°C at atmospheric pressure) is constant, since this film does not include Cl ions. The rate of etching to LPCVD SiO_2 film decreases when the film becomes less than 1500Å thick. This tendency is also observed in another etchant, NH_4F:HF = 60:1.

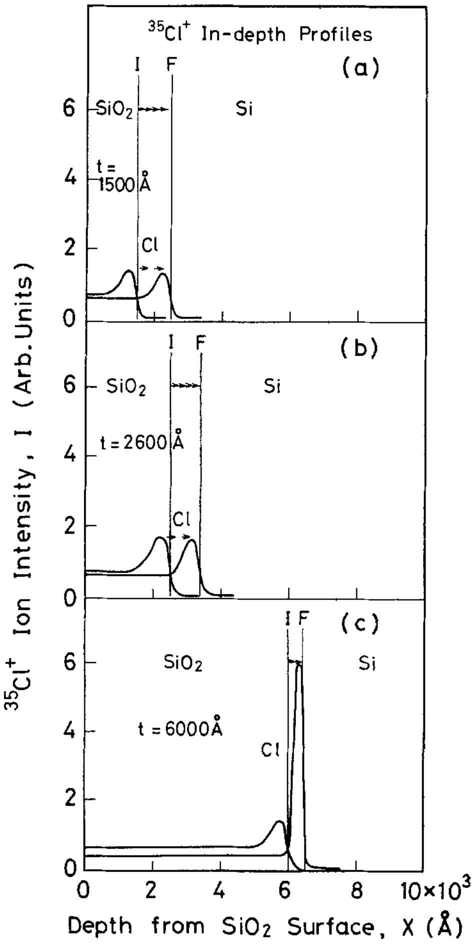

Fig. 4. Cl in-depth profile for three different thickness as deposited. I indicates the interface as deposited. F indicates the interface after oxidation at 1000°C for 1 hr.

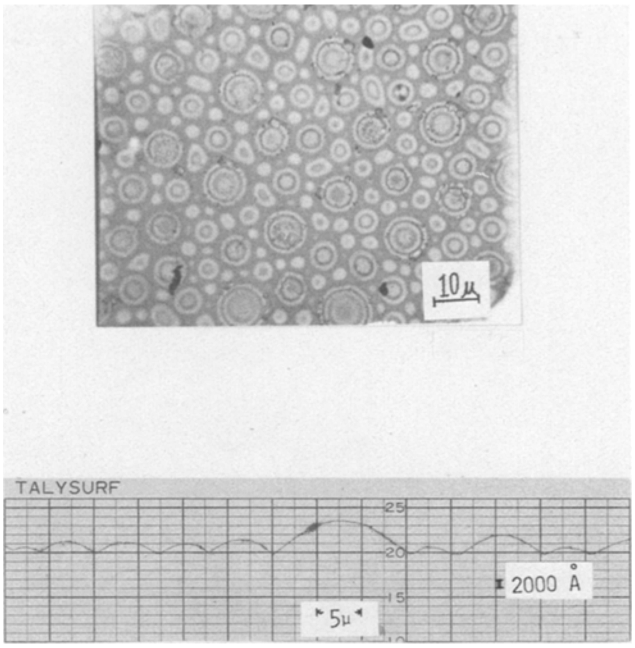

Fig. 5. (a) Surface of a thick LPCVD SiO_2 film after 1000°C anneal.

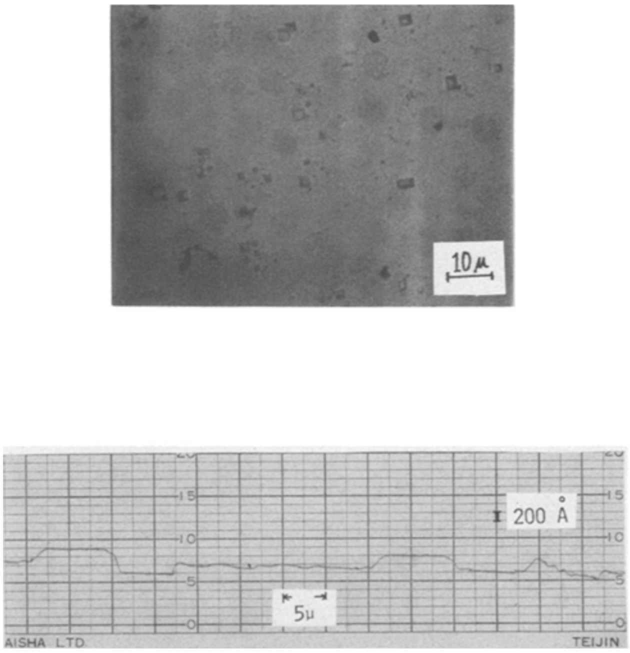

Fig. 5. (b) Surface of Si after removal of SiO_2 layer of the same sample as (a).

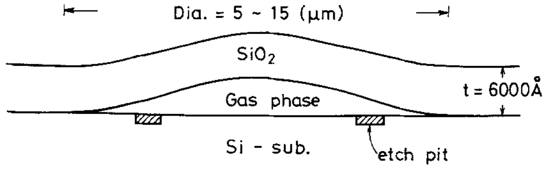

Fig. 6. Schematic illustration of the section of SiO_2-Si when surface roughness is observed.

Figure 8 shows the thickness dependence of V_{FB} on MOS structures [n-type (100) 2-3 Ω-cm Si] obtained

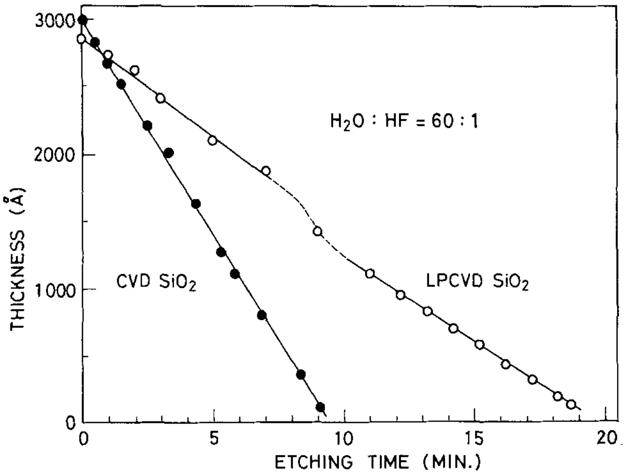

Fig. 7. Etching rates of LPCVD SiO_2 and atmospheric pressure CVD SiO_2.

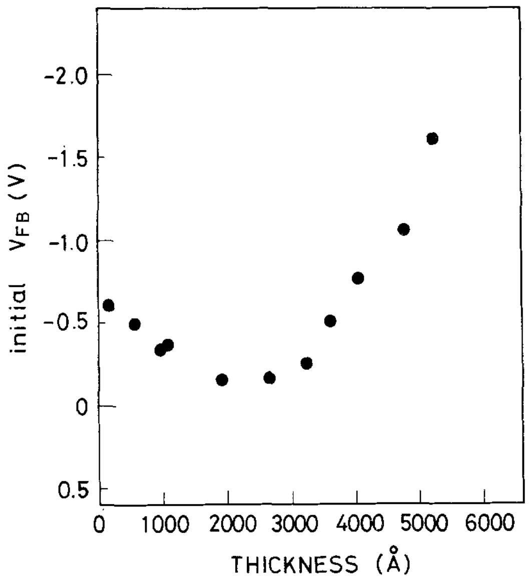

Fig. 8. Flatband voltage of Al-LPCVD SiO_2-Si structures obtained from step-etching of thick film about 6000Å thick.

from the step etching film about 6000Å thick. These data show that negative charges accumulate near the interface. To estimate the magnitude of these charges, we assumed that the charges were distributed as in Fig. 9. The negative charge density ρ is defined as

$$\rho = -b\rho_0 \quad (b > 0) \qquad [1]$$

where ρ_0 is the density of uniformly distributed positive charge and b is a constant. The flatband voltage shift, ΔV_{FB}, caused by fixed charges in SiO_2 is given

$$\Delta V_{FB} = -\frac{1}{C_0}\int_0^d \frac{x\rho(x)}{d}\,dx \qquad [2]$$

where C_0 is the oxide capacitance and d is the oxide thickness. ΔV_{FB} has a minimum value at $d = ab$. The difference of ΔV_{FB} between $d = 0$ and $d = ab$ is

$$\delta V_{FB} = \frac{\rho_0}{2\epsilon_0}\,b\,(1-b)\,a^2 \qquad [3]$$

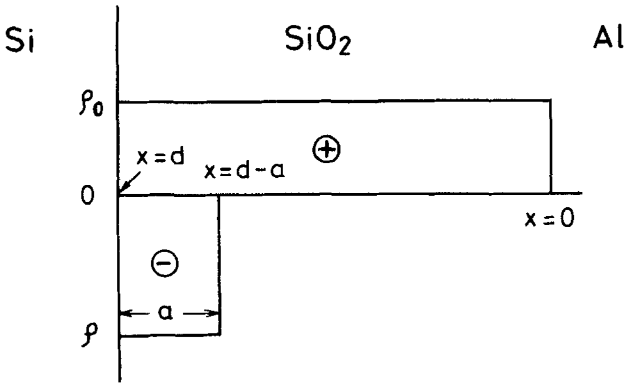

Fig. 9. Assumed charge distribution in LPCVD SiO_2 film

The results show that $\delta V_{FB} = -0.5V$ and $ab = 2500Å$. If $a = 1000Å$, then $b = 2.5$, $\rho = 1.4 \times 10^{16}$ qcm^{-3} and $\rho_0 = 0.56 \times 10^{16}$ qcm^{-3}. Since the order of magnitude of Cl concentration is 10^{19} cm^{-3}, the small portion of Cl has a negative charge.

Both the etching result and V_{FB} result do not bear directly on the profile of Cl distribution but do indicate that the film is not uniform.

Discussion

Cl ions are distributed broadly near the SiO_2-Si interface as the LPCVD SiO_2 film is deposited. It is well known that Cl ions distribute at the interface in HCl/O_2 oxidation. In thermal oxidation, SiO_2 films grow by the reaction of Si and O_2 and the growth rates depend on the film thickness. However, since the growth of SiO_2 at the interface by HCl/O_2 oxidation is simultaneous with the piling up of Cl, it is not clear in the case of thermally grown film whether the cause of the Cl pile-up is oxidation or the existence of the interface. On the contrary, CVD films grown by the chemical reaction of gases without consuming Si substrate and the growth rates are constant. The difference between the growth mechanism of thermally grown film and CVD film leads us to conclude that, in the case of CVD film, Cl distribution is determined not by the mechanism of Si oxidation but by the existence of the SiO_2-Si interface. If the cause of the Cl pile-up is existence of the interface, then it is clear that the profile of Cl concentration is independent of the thickness of unannealed LPCVD film. If the cause of Cl pile-up were the oxidation, Cl in LPCVD film would pile up at the SiO_2 surface or distribute uniformly because the oxidation of SiH_2Cl_2 in gas phase occurs at the surface.

The effect of the interface on Cl distribution can also be seen in the redistribution of Cl after the N_2 annealing of Cl-implanted SiO_2 film as is shown in Fig. 10. The distribution of Cl in conventional CVD films before annealing is shown in Fig. 2. Cl redistributes

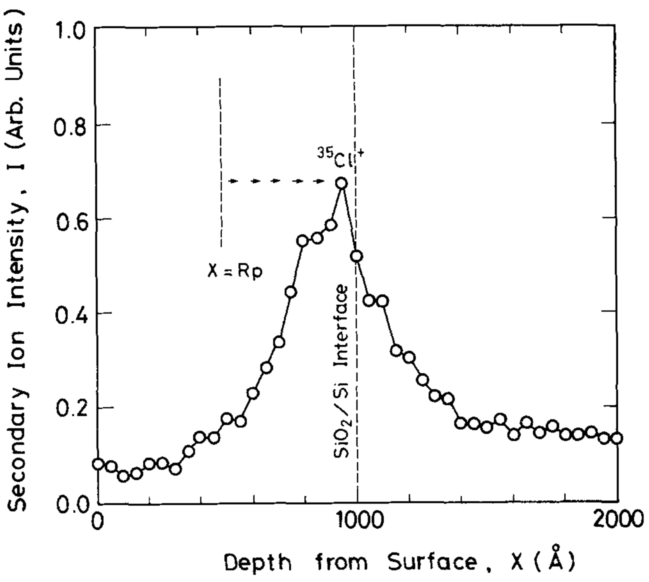

Fig. 10. Cl ion intensity of Cl ion implanted CVD SiO_2 after oxidation at 1000°C N_2 for 1 hr.

at the interface after N_2 annealing, although the extra implanted Cl ions are diffused out. The peak concentration of Cl after N_2 annealing of the Cl-implanted SiO_2 film is 10^{19} ions cm^{-3}. This value is equal to that of Cl in unannealed LPCVD SiO_2 film. Although we cannot explain the mechanism for Cl peak formation near the SiO_2-Si interface, we believe that Cl ions in SiO_2 are attracted to the interface by the existence of the interface.

Cl profile after heat-treatment.—Because thermal oxidation in some way causes the movement of the SiO_2-Si interface, Cl ions move toward the interface as the thermally oxidized layer grows. The velocity of Cl ions moving toward the interface is higher than the velocity of the interface movement caused by oxidation, as shown in Fig. 4. In this sense Cl ions are mobile in SiO_2 film.

Because the Cl profile is unchanged in thin films and the total number of Cl ions is preserved after oxidation and N_2 annealing, Cl ions do not diffuse out by thermal treatment. Cl ions are stably incorporated in SiO_2 film.

Cause of surface roughness.—After the oxidation of thick LPCVD SiO_2 film, the Cl profile near the interface changes remarkably. This behavior is explained as follows: In thermal oxidation oxygen diffuses through oxide and reacts on silicon. The concentration of diffused oxygen ions at the interface depends on the thickness of the film, but the concentration of Cl ions near the interface is independent of the thickness of unannealed film. Oxygen competes with Cl in reaction on Si. Since the oxygen concentration at the interface is sufficiently large in thin films, the reaction of Si-O is dominant and SiO_2 is formed even though Cl reacts on Si. In a thick film, because of the insufficient supply of oxygen to the interface, the Si-Cl formed by the reaction of Si and Cl cannot be completely transformed into SiO_2 and remains as a gas phase of Si-O-Cl. Pancake shapes are then formed. The gas of Si-O-Cl lifts the SiO_2 layer. Cl induces etch pits on the Si wafer. It is for this reason that surface roughness is observed only in thick films.

The following experiment shows clearly the thickness dependence of surface roughness. After the deposition of LPCVD SiO_2 film 2600Å thick, conventional CVD SiO_2 8000Å thick is deposited by the reaction of SiH_4 and O_2 at 420°C. The Cl distribution of LPCVD SiO_2 is unaffected by the low temperature deposition of CVD SiO_2. Wafers without the CVD SiO_2 deposition and wafers with the deposition were oxidized in dry O_2 for 1 hr at 1000°C. Though the integrated Cl concentrations are the same in both samples, the surface roughness is observed only in the film with addition of CVD SiO_2. Thus we can conclude that the surface roughness does not depend on the total amount of Cl, but does depend on the oxygen concentration at the interface.

Though we do not identify the gas at the SiO_2-Si interface, it seems to be the same as that produced by HCl/O_2 oxidation. Monkowski *et al.* (2) have suggested that the gas formed by HCl/O_2 oxidation is oxygen-rich chlorosiloxane. Lin (5) has discussed this oxygen-rich chlorosiloxane, $Si_nO_{2n-3}Cl_6$ (n = 2-11), which is formed in reduced oxygen pressure and appropriate chlorine atmosphere.

Surface roughness is not observed after N_2 annealing in spite of there being no O_2 concentration at the interface. This can be explained in the following manner: Silicon is etched by HCl

$$\text{Si (solid)} + \text{HCl (gas)} \leftrightarrows \text{SiCl}_4\text{ (gas)},\ \text{SiHCl}_3\text{ (gas)}$$

This reaction is reversible. Etching by Cl will not cause surface roughness. When oxygen is present, however, the formation of Si-O-Cl continues irreversibly.

Comparison between oxidation rate and etching rate.—Let us now consider the oxidation rate of Si under LPCVD SiO_2 film xÅ thick. Oxygen flow F_1 in SiO_2 can be expressed as

$$F_1 = D\,\frac{C_o - C_i}{x} \qquad [4]$$

where D is the diffusion constant of oxygen, C_o and C_i are the oxygen concentrations at the oxide surface and at the SiO_2-Si interface, respectively. Oxygen flow at interface F_2 is

$$F_2 = K_sC_i \qquad [5]$$

where K_s is the chemical surface-reaction rate constant for oxidation. In a steady state, that is where $F_1 = F_2$

$$C_i = \frac{DC_o}{K_sx + D} \qquad [6]$$

The growth rate of SiO_2 is given by

$$\frac{dx}{dt} = \frac{B}{A + 2x} \qquad [7]$$

where $A \equiv 2D/K_s$, $B \equiv 2DC_o/N_1$, and N_1 is the O_2 concentration in SiO_2. We can determine the values of A and B from oxidation. After dry O_2 oxidation for 1 hr at 1000°C of 2630Å thick LPCVD film, oxide thickness increased to 3370Å. Average oxidation rate and average thickness are 12.3 Å/min and 3000Å, respectively. The oxidation rates of films with three different initial thickness are plotted in Fig. 11. We can obtain A = 3000Å and $B = 11.3 \times 10^4$ Å²/min. In HCl/O_2 oxidation of (100) Si, using the data of Deal *et al.* (6, 7) A and B are given as follows: A = 2530Å, $B = 2.6 \times 10^4$ Å²/min at 1% HCl and A = 4300Å, $B = 4.4 \times 10^4$ Å²/min at 4.5% HCl. Linear rate of oxidation $B/A = (C_0/N_1)k_s$ is 10 Å/min in HCl oxidation both at 1% and 4.5%, whereas it is 37 Å/min in LPCVD SiO_2. If C_0 is the same in both these films, the values of k_s and D of LPCVD SiO_2 are large compared with those of HCl/O_2 oxidized SiO_2.

The rate of Si consumption, R_o, is derived from the oxidation rate

$$R_o = \frac{N_1B}{N(A + 2x)} = \frac{B}{2(A + 2x)} \qquad [8]$$

where N is the density of Si, 5×10^{22} cm^{-3}.

We concluded that the Cl etching rate, R_e, was

$$R_e = 5\text{ Å/min}$$

because Si was etched to a 300Å depth by Cl at 1000°C for 1 hr. From the above estimation, we can approxi-

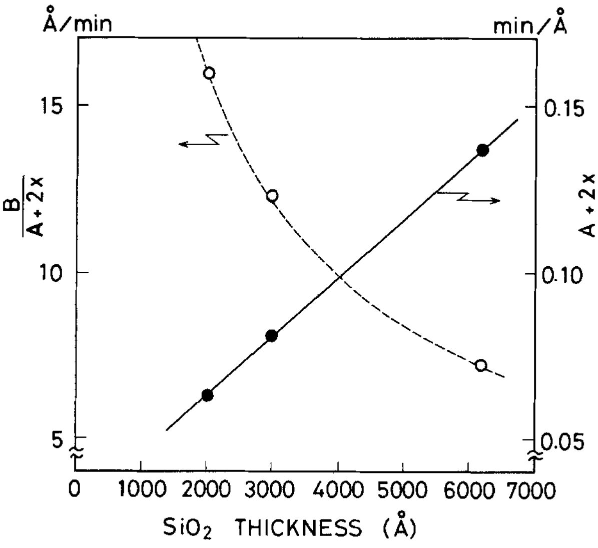

Fig. 11. Oxidation rate $B/A + 2x$ and its reciprocal rate of three different LPCVD SiO_2 thicknesses.

mate the point at which the film becomes too thick to be without surface roughness. The Cl etching rate is smaller than the oxidation rate

$$R_e < \frac{B}{2(A + 2x)} \qquad [9]$$

By substituting the experimental values for A, B, and R_e

$$x < 4100Å \qquad [10]$$

This value agrees with the experimental results. It is reasonable to assume that the existence of surface roughness depends on whether the Cl etching rate or the oxidation rate is higher.

By measuring surface state density, we confirmed that the microstructure of surface roughness does not appear after oxidation in thin LPCVD SiO_2 film on Si. Surface state density of the film as deposited and after N_2 annealing at 1100°C for 1 hr is comparable to that of thermally grown SiO_2.

Comparison of Cl profiles.—The Cl distribution in LPCVD SiO_2 film is broader than that in HCl/O_2 oxidized film. In HCl/O_2 oxidized film, Cl piles up within 200Å from the SiO_2-Si interface (6). In Cl_2/O_2 oxidized film, however, Cl ions distribute broadly within 1000Å (7). The difference between the Cl distribution in HCl/O_2 and Cl_2/O_2 oxidized films may be caused by H_2O produced by HCl/O_2 oxidation (8). In LPCVD SiO_2 films, judging from the reaction of SiH_2Cl_2 and N_2O and from the deposition pressure, H_2O is not produced. The Cl distribution in LPCVD SiO_2 is almost the same as that in Cl_2/O_2 oxidized film.

Summary

Cl distribution in LPCVD SiO_2 film deposited by SiH_2C_2 and N_2O was measured by IMMA. We found that Cl ions are distributed approximately 1000Å from the SiO_2-Si interface. The rate of etching to SiO_2 and the flatband voltage are affected by Cl distribution. The Cl distribution is independent of the thickness of unannealed film. After the oxidation Cl ions move toward the newly formed interface. In films thinner than 4000Å, the Cl profile remains unchanged after the oxidation, but in thick films Cl concentration near the interface is increased and surface roughness is observed. Cl distribution remains unchanged after N_2 annealing at 1100°C. From these results, it is apparent that the phenomenon of Cl pile-up is not caused by the mechanism of oxidation but by the existence of the interface itself. Surface roughness after oxidation depends on whether the Cl etching rate of the oxidation rate is higher. In a high oxygen concentration at the interface, the oxidation rate is larger than the Cl etching rate and surface roughness is not observed. In a low oxygen concentration, Si is etched by Cl and Si-O-Cl is formed. The surface of SiO_2 films is lifted by the gases and etching pits are observed. In this case Cl ions markedly pile up at the interface. When there is no O_2 (such as in N_2 annealing), even if Si is etched by Cl, Si-O-Cl is not formed and surface roughness is not observed.

Taking into account our estimation of the Cl etching rate and the oxidation rate, we can conclude that oxidation at 1000°C for 1 hr does not induce surface roughness when LPCVD film is less than 4000Å thick. We believe that LPCVD SiO_2 will be useful as the gate oxide of Si devices.

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Au/Ni/SnNi/n-GaAs Interface: Ohmic Contact Formation

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ABSTRACT

The Au/Ni/SnNi/GaAs interface has been studied in order to understand ohmic contact formation. Depth profiling in conjunction with Auger electron spectroscopy has been used to determine impurity distribution in as-deposited and alloyed films and were correlated to electrical parameters. We find that ohmicity can be achieved without Sn due to Au-induced Ga vacancies and Ni-induced breakup of background Si covalent bonds. However, addition of Sn to the system lowers the specific contact resistances greatly.

Various studies have been conducted on metal-GaAs ohmic contact systems. Most of these are concerned with electrical and metallurgical properties of the contacts as well as their long-term reliability. The majority of the systems studied include Au-based metal contacts. Among them Ni/Au-Ge/GaAs is favored in many applications and have been exten-

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Key words: interfaces, bonding, ohmicity.