Phosphorus and Arsenic Diffusions from Doped Polycrystalline Silicon

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Diffusions from phosphorus and arsenic doped polycrystalline silicon into crystalline silicon substrate have been investigated. Phosphorus profiles reveal two regions with different diffusion coefficients, *i.e.* 1.2×10^{-14} and 6.0×10^{-14} cm²/sec, that are almost similar to the values of doped oxide-diffusion obtained by Yoshida and conventional gas phase-diffusion by Tsai. The total arsenic concentration profile determined from electrically measured profile by means of Fair's method shows a good agreement with that obtained from doped oxide by Fair. No remarkable difference was recognized between profiles of diffusions in dry oxygen and nitrogen.

1 Introduction

In fabricating micro-wave transistors and integrated circuits using conventional planar techniques, it is required that the junction depth should be shallow such as $\sim 0.2 \mu$ depth of an emitter junction in order to improve the frequency characteristics. After the metallization of an aluminum electrode, however, an emitter-base short occurs very often because of such short distance between the junction and the aluminum electrode. Recently, Kamioka *et al.*¹⁾ have shown that shallow pn junction can be formed by diffusion from impurity-doped polycrystalline silicon deposited on single crystalline silicon substrate. Such Doped Poly Silicon technique (DOPOS) is very useful to prevent the above mentioned failure due to the existence of deposited poly-silicon between the pn junction and the aluminum electrode. Although the diffusion from a doped oxide has been studied by several workers^{2)~6)}, few studies⁷⁾ have been reported on diffusion from a doped poly-silicon.

This paper reports the study of diffusion characteristics from doped poly-silicon, such as concentration profile, junction depth and sheet resistivity. Phosphorus and arsenic diffusion coefficients are determined by Boltzmann-Matano

analysis⁸⁾ and the concentration dependence of the diffusion coefficient is compared with that obtained by other investigators^{9)~11)}.

2 Experimental Procedures

As the starting material, boron doped $1 \Omega\text{-cm}$ *p*-Si (111) wafers were mechanically and chemically polished and cleaned. The cleaning procedure involved rinsing in trichloroethylene and acetone and boiling in nitric acid and sulfuric acid. Immediately prior to the deposition of poly-silicon, the silicon wafers were dipped into an HF solution to remove a very thin silicon oxide layer.

The poly-silicon films were deposited in an rf induction-heated reactor. Phosphorus and arsenic doped poly-silicon was deposited on the silicon wafers from $\text{SiH}_4\text{-PH}_3\text{-N}_2$ and $\text{SiH}_4\text{-AsCl}_3\text{-N}_2$ mixtures at 690°C and 750°C, respectively. The temperature of the silicon wafers was measured with an infrared scope. In this experiment, phosphorus-doped poly-silicon films ranging from 500 to 4000 Å thick and arsenic doped poly-silicon films of 2500 Å thick were deposited. Other experimental deposition conditions are listed on Table 1. Before drive-in diffusion, phosphorus-doped poly-silicon films were coated with 5000 Å thick CVD SiO_2 films to prevent out-diffusion. Phosphorus and

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Table 1 Typical doped poly-silicon growth conditions

	P dope	As dope
SiH ₄ (cc/min.)	15	3
PH ₃ (cc/min.)	0.4	—
N ₂ (AsCl ₃ bubble) (cc/min.)	—	20(10°C)
N ₂ (carrier) (l/min.)	30	3
Growth Temperature (°C)	690	750
Growth Rate (Å/min.)	160	400
Reactor	horizontal	vertical

arsenic drive-in diffusions from doped poly-silicon films were carried out at 900°C and at 1000~1100°C, respectively, in dry nitrogen or oxygen atmosphere.

To determine the poly-silicon thickness and the interface between doped poly-silicon and the substrate¹²⁾, the doped poly-silicon was also deposited onto silicon wafers partially covered with thermally grown silicon oxide of 2000 Å thick. When the structure was immersed into an etch mixture containing 1 part HF (49%), 20 parts HNO₃ (70%) and 20 parts CH₃COOH (99.9%) at room temperature, the doped poly-silicon etch rate was 80~120 Å/sec while the silicon oxide etch rate was 3.5 Å/sec. Even after the drive-in diffusion above 900°C, the etch rate of the phosphorus doped and arsenic doped poly-silicon films was 60~80 Å/sec, while that of the single crystal silicon wafer with 1 Ω-cm resistivity was 30~40 Å/sec. The etching end-point of poly-silicon on silicon dioxide could indicate the substrate-poly-silicon interface within the errors of ±150 Å. The poly-silicon thickness was also confirmed with a Taylor Hobson Talystep.

To obtain the impurity profile, a layer removal technique was carried out in a mixture containing 1 part HF (49%) and 30 parts CuSO₄ solution (120 g CuSO₄ in 1000 cc water). Since the resistivity of each layer was given by

$$\frac{d(\rho_s^{-1})}{dx} = -\frac{1}{\rho} \quad (1)$$

where ρ_s is the measured sheet resistivity, x is the layer thickness.

Phosphorus and arsenic concentration profiles were obtained from Irvin's curves concerning impurity concentration *vs.* resistivity.¹³⁾ In the doped poly-silicon, the concentrations of the

phosphorus and arsenic were $4\sim6\times10^{19}$ atoms/cm³ without a correction which took into account the smaller electron mobility in the doped poly-silicon than that in the single crystal silicon¹⁴⁾. At the surface of the single silicon wafer, the concentration increased rapidly more than 1×10^{20} atoms/cm³. Therefore, the interface between the doped poly-silicon and the single crystal silicon wafer could be determined electrically. The junction depths obtained with the angle lapping and staining agreed with those determined from the concentration profiles within the error of 10%.

3 Results and Discussion

3.1 Phosphorus diffusion

3.1.1 Phosphorus concentration profiles

Phosphorus concentration profiles, obtained with diffusions at 900°C for 29, 100 and 225 minutes, are shown in Fig. 1A, B and C, respectively. There are two regions in each profile. One is near the surface (region I), where the concentration decreases rapidly, *i.e.* the diffusion coefficient D_I is small. The other is far from the surface (region II), where the concentration decreases slowly, *i.e.* diffusion coefficient D_{II} is larger than D_I . These two regions exist in cases of both 500 and 2000 Å thick films. The existence of two regions in the phosphorus profile has also been reported by Yoshida *et al.*¹⁰⁾, who carried out phosphorus diffusions from the phosphorus-doped-oxide. In the case of conventional diffusion from P₂O₅ or POCl₃, Tannenbaum¹⁵⁾, Duffy *et al.*¹⁶⁾ and Tsai⁹⁾ have also shown that a very high constant concentration region exists on the surface of the phosphorus-diffused layer. Such a flat region could not be observed in this doped poly-silicon case. Tsai⁹⁾ indicated that diffusion coefficient difference was attributed to the diffusions of the silicon phosphide and of phosphorus. Yoshida *et al.*¹⁰⁾ concluded that, in the doped oxide case, the phosphorus diffusion coefficient was dependent on both concentration and surface condition.

It has been reported¹⁷⁾ that the high concentration phosphorus diffusion into silicon induces the dislocations. The dislocations are not desirable for the semiconductor device fabrications

Fig. 1 Phosphorus concentration profiles obtained with diffusion from doped poly-silicon of 500 Å thick (●) and 2000 Å thick (Δ)

(A) 900°C, 29 minutes, (B) 900°C, 100 minutes and (C) 900°C, 225 minutes in N₂

¹⁶⁾. In the present diffusion process, the phosphorus diffused layers did not cause dislocation arrays, which is confirmed by Sirtl etch technique.

3.1.2 Film thickness effects As shown in Fig. 1, the junction depth does not depend on film thickness within this experimental range. Figure 2 shows the relation between junction depth and diffusion time for various film thicknesses, indicating that the junction depth is approximately proportional to the square root of diffusion time.

Figure 1 also indicates that, at region I, the phosphorus concentration diffused from a 500 Å poly-silicon film is lower than that from a 2000

Fig. 2 Junction depth obtained with diffusion from phosphorus doped poly-silicon at 900°C vs. square root of diffusion time

Fig. 3 Sheet resistivity obtained with diffusion from phosphorus doped poly-silicon at 900°C vs. square root of diffusion time. Poly-silicon thicknesses are given as parameters

Å film. It suggests that sheet resistivity of diffused silicon surface depends on the source film thickness. Figure 3 shows the relations between sheet resistivity and diffusion time. Sheet resistivity decreases with film thickness increasing, corresponding to Fig. 1. Yasuda *et al.*¹⁸⁾ have also confirmed that the boron concentration in the doped poly-silicon with the film thickness below 1000 Å was lower than that of a film thickness above 1000 Å. The concentration difference at region I also corresponds to this phenomenon.

As shown in Fig. 3, the sheet resistivities of diffused layers from 500 Å and 1000 Å films begin to saturate as the diffusion time becomes longer than 100 minutes. However, sheet resistivities obtained with 2000 Å and 4000 Å films do not saturate even for 169 minutes diffusion. This means that, in the case of film thickness greater than 2000 Å, the diffusion source has sufficient amounts of phosphorus for a long diffusion time of at least up to 169 minutes.

3.1.3 Phosphorus diffusion coefficient

As described in Sec. 3.1.1, the phosphorus diffusion coefficient depends on the concentration. Thus, Boltzmann-Matano analysis⁸⁾ was used to determine the diffusion coefficient, that is, the diffusion coefficient is represented by

$$D(n) = -\frac{1}{2t} \frac{dx}{dn} \int_{n(x=\infty)}^{n(x)} x dn \quad (2)$$

where D is the diffusion coefficient and t is the diffusion time. Here, as explained by Yoshida *et al.*¹⁰⁾, it is also confirmed that the concentration is a function of $x/\sqrt{t}$.

Figure 4 shows the phosphorus diffusion coefficients determined from Eq. (2) using the profiles of 2000 Å poly-silicon film indicated in Fig. 1. In the region of a concentration lower than 10^{19} cm⁻³, the diffusion coefficient was given as a large value ($D_{II} \cong 6 \times 10^{-14}$ cm²/sec), while in higher concentration of $\sim 10^{20}$ cm⁻³, it decreased and reached a small value ($D_I \cong 1.2 \times 10^{-14}$ cm²/sec). This tendency coincides qualitatively with Tsai's⁹⁾ and Yoshida *et al.*'s¹⁰⁾ results.

Tsai has suggested quantitatively that the phosphorus diffusion profiles were derived from

Fig. 4 Dependence of phosphorus diffusion coefficient on phosphorus concentration in 2000 Å poly-silicon film

Fick's diffusion equations using a model where a moving boundary separates two distinct phases, and has determined the small and large coefficients, as 2.7×10^{-15} cm²/sec and 5.7×10^{-14} cm²/sec. Although a good agreement was obtained between this doped poly-silicon case and conventional gas phase diffusion case in a large coefficient region, a slight difference was observed in a small coefficient region.

3.2 Arsenic diffusion

3.2.1 Arsenic concentration profiles

Figure 5 A, B, C and D shows arsenic concentration profiles obtained with the diffusion from the 2500 Å arsenic-doped poly-silicon films with various gas phase-molar ratios of AsCl₃/SiH₄. Concentration gradients are small in the high concentration region ($\geq 1 \times 10^{20}$ cm⁻³) and increase rapidly near the junction. These phenomena are similar to those found in the studies reported by many workers.^{1), 4), 7), 11), 19), 20)} In addition, the concentration slightly decreases near the surface at AsCl₃/SiH₄ of 7×10^{-2} , as reported by Fair²⁰⁾. In the case of 0.7×10^{-2} AsCl₃/SiH₄ ratio, the arsenic concentration is lower than that

Fig. 5 Arsenic concentration profiles obtained with diffusion at 1000°C for 60 minutes in O₂ on molar ratios of AsCl₃/SiH₄. Dotted lines indicate total arsenic concentration profiles calculated by Fair's method

(A) AsCl₃/SiH₄ = 0.7×10^{-2} , (B) 2.1×10^{-2} , (C) 3.8×10^{-2} and (D) 7.0×10^{-2}

from the others and the junction depth is also shallower than that from the others. On the other hand, arsenic concentration profiles obtained with diffusion at 1000°C for 60 minutes from the arsenic-doped poly-silicon films with 1000, 2000, and 4000 Å thicknesses agree with one another.

3.2.2 Molar ratio ($\text{AsCl}_3/\text{SiH}_4$) dependence

Figure 6 shows the sheet resistivity and the junction depth obtained with the diffusion from the 2500 Å doped poly-silicon for various molar ratios of $\text{AsCl}_3/\text{SiH}_4$. In the case of $\text{AsCl}_3/\text{SiH}_4$ of 0.7×10^{-2} , the sheet resistivity is higher, which corresponds to the shallower junction depth. The sheet resistivity decreases as the ratio of $\text{AsCl}_3/\text{SiH}_4$ increases and it begins to saturate above 3.8×10^{-2} . On the contrary the junction depth increases and saturates as the ratio of $\text{AsCl}_3/\text{SiH}_4$ increases. These facts are consistent with the profiles in Fig. 5 A, B, C and D.

Fig. 6 Molar ratio ($\text{AsCl}_3/\text{SiH}_4$) dependence of sheet resistivity and junction depth obtained with diffusion at 1000°C for 60 minutes

Fair²¹⁾ showed that, for a constant C_s diffusion,

$$C_s = \frac{1.56 \times 10^{17}}{\rho_s x_j} \quad (\text{atoms/cm}^3) \quad (3)$$

where C_s is the electrically active surface concentration. Equation (3) is always valid in dependent of the type of arsenic diffusion²¹⁾. Then the surface concentrations of electrically active arsenic, C_{AO} , are calculated for the various molar ratios of $\text{AsCl}_3/\text{SiH}_4$ by using ρ_s and x_j shown in Fig. 6. Calculated C_{AO} values are 8.3×10^{19} atoms/cm³ for $\text{AsCl}_3/\text{SiH}_4 = 0.7 \times 10^{-2}$, 1.9×10^{20} atoms/cm³ for $\text{AsCl}_3/\text{SiH}_4 = 2.1 \times 10^{-2}$, 2.2×10^{20} atoms/cm³ for $\text{AsCl}_3/\text{SiH}_4 = 3.8 \times 10^{-2}$ and $2.2 \times$

10^{20} atoms/cm³ for $\text{AsCl}_3/\text{SiH}_4 = 7.0 \times 10^{-2}$, respectively. These calculated C_{AO} values are coincident with those shown in Fig. 5 within the limit of errors of 10~15%, except for the case of $\text{AsCl}_3/\text{SiH}_4 = 7.0 \times 10^{-2}$. Therefore, it is ascertained that Eq. (3) does not lose the validity for the diffusion from the arsenic doped poly-silicon. When the molar ratio of $\text{AsCl}_3/\text{SiH}_4$ is larger than 3.8×10^{-2} ,^{*1} the total arsenic concentration might exceed a value above the solubility limit, corresponding to the saturations of the sheet resistivity and junction depth.

3.2.3 Arsenic diffusion coefficient

It has been shown that, in the high-concentration arsenic-diffused-layer obtained by another diffusion technique, the total arsenic concentration C_T is higher than the electrically-active arsenic-concentration C_A ^{11), 12), 23)}. Figure 7 shows profiles of C_A diffused from the doped poly-silicon with

Fig. 7 Diffusion temperature dependence of arsenic concentration profiles obtained with diffusion from arsenic doped poly-silicon with $\text{AsCl}_3/\text{SiH}_4$ of 7.6×10^{-2}

Diffusion conditions are 1000°C, 49 minutes (○), 1050°C, 20 minutes (△) and 1100°C, 20 minutes (□)

*1 By using atomic absorption spectrophotometry, it becomes clear that the arsenic concentration in the doped poly-silicon (deposited with a molar ratio of 3.8×10^{-2}) is 7.1 wt%.

$\text{AsCl}_3/\text{SiH}_4$ of 7.6×10^{-2} at 1000, 1050 and 1100°C, respectively. C_A at 1100°C is higher than C_A at 1000°C, *i.e.* in the doped poly-silicon case, again, C_T is higher than C_A at 1000°C. C_A slightly decreases near the surface for the diffusion at 1000°C and 1050°C.

In this paper, C_T was calculated by applying Fair's empirical equations^{2,4)} as

$$C_T(x) = C_A(x) [1 + \beta(T)] \quad \text{for } 0 \leq x \leq \frac{1}{2}x_j \quad (4)$$

$$C_T(x) = C_A(x) [1 + \beta(T) (C_A(x)/C_{AO})^3] \quad \text{for } \frac{1}{2}x_j < x \leq x_j \quad (5)$$

where β is a fitting parameter and C_{AO} is the surface concentration of electrically active arsenic. Dotted lines of Fig. 5 A, B, C and D shows the total arsenic profiles corresponding to the solid line profiles shown in Fig. 5 A, B, C and D respectively, using $\beta(1000) = 2^{2,4)}$.

Figure 8 shows arsenic diffusion coefficients D_{As} determined by the Boltzmann-Matano analysis as described in Sec. 3.1.3. It should be noted that D_{As} depends strongly on the total arsenic concentration C_T and is also sensitive to the molar ratio of $\text{AsCl}_3/\text{SiH}_4$. In addition, D_{As} has a maximum value at C_T of $2 \sim 4 \times 10^{20} \text{ cm}^{-3}$. These values agree with the study reported by Fair¹¹⁾ using a doped oxide method.

Table 2 represents C_T/n_i values and $D_{\max}/D_i$ values at $D_{As} = D_{\max}$ for various ratios of $\text{AsCl}_3/$

Fig. 8 Relation between arsenic diffusion coefficients and total arsenic concentration

$\text{AsCl}_3/\text{SiH}_4$ are 0.7×10^{-2} (●), 2.1×10^{-2} (△), 3.8×10^{-2} (■) and 7.0×10^{-2} (○)

Table 2 Ratios of total to intrinsic arsenic concentrations and ratios of maximum to intrinsic arsenic diffusion coefficient. Diffusion temperature is 1000°C

$\frac{\text{AsCl}_3}{\text{SiH}_4}$	$\frac{C_T}{n_i}$ (at $D = D_{\max}$)	$\frac{D_{\max}}{D_i}$
0.7×10^{-2}	21	12
2.0×10^{-2}	37	27
3.8×10^{-2}	50	52
7.0×10^{-2}	37	50
doped oxide*	34	43

* : After Fair¹¹⁾ $C_{TO} = 8.5 \times 10^{20} \text{ (atoms/cm}^3\text{)}$

SiH_4 , where n_i is the intrinsic electron concentration at a given diffusion temperature, D_i is the intrinsic arsenic-diffusion-coefficient at the diffusion temperature and $D_{\max}$ is the maximum value of D_{As} . It indicates that C_T/n_i and $D_{\max}/D_i$ values increase as the molar ratio of $\text{AsCl}_3/\text{SiH}_4$ increases. These values saturate at a molar ratio of larger than 3.8×10^{-2} .

In the case of doped oxide diffusion, C_T/n_i and $D_{\max}/D_i$ at $D_{As} = D_{\max}$ have been given by¹¹⁾

$$\frac{C_T}{n_i} = \frac{0.6 C_{TO}}{\sqrt{\beta(T)} [1 + \beta(T)] n_i} \quad (6)$$

$$\frac{D_{\max}}{D_i} = \frac{\sqrt[3]{2} C_{TO}}{3 \sqrt[3]{\beta(T)} [1 + \beta(T)] n_i} \quad (7)$$

where C_{TO} is the surface concentration of total arsenic. If these relations hold in the doped poly-silicon case, the molar ratio dependences of C_T/n_i and $D_{\max}/D_i$ correspond to those of C_{TO} . In the case of molar ratio above 3.8×10^{-2} , C_{TO} was approximately $7 \times 10^{20} \text{ cm}^{-3}$, which was nearly equal to that obtained with an arsenic-doped-oxide by Fair¹¹⁾.

3.3 Effect of atmosphere during diffusion

Figure 9 shows the arsenic diffusion profiles obtained with the diffusion in dry O_2 and in dry N_2 atmospheres. A profile obtained with the diffusion in a dry O_2 atmosphere agrees well with that obtained with the diffusion in a dry N_2 atmosphere.

As shown in Table 3, the sheet resistivity obtained with diffusion in dry O_2 is almost equal to that obtained in dry N_2 , and the junc-

Fig. 9 Arsenic concentration profiles obtained with diffusion in O_2 ($\circ$) and in N_2 ($\blacktriangle$) at 1000°C for 60 minutes

$\text{AsCl}_3/\text{SiH}_4$ is 3.8×10^{-2} and poly-silicon thickness is $2500 \text{ \AA}$

Table 3 Sheet resistivity and junction depth for 1000°C , 60 minutes diffusion. Molar ratios of $\text{AsCl}_3/\text{SiH}_4$ and PH_3/SiH_4 are 3.8×10^{-2} and 2.6×10^{-2} , respectively

Diffusion condition	Sheet resistivity ρ_s ($\Omega/\square$)	Junction depth X_j (μm)
As 1000°C 60 min	15 (O_2)	0.48 (O_2)
	14 (N_2)	0.45 (N_2)
P 1000°C 60 min	9.6 (O_2)	1.27 (O_2)
	9.7 (N_2)	1.16 (N_2)

tion depth obtained with diffusion in dry O_2 is almost equal to that obtained in dry N_2 , corresponding to diffusion profiles shown in Fig. 9. In the case of phosphorus doped poly-silicon, the sheet resistivity and the junction depth are almost equal to those obtained in both ambients.

4 Summary and Conclusion

Phosphorus and arsenic doped poly-silicon

films were prepared from $\text{SiH}_4\text{-PH}_3\text{-N}_2$ and $\text{SiH}_4\text{-AsCl}_3\text{-N}_2$ mixtures, respectively, and then, drive-in diffusions were carried out in dry nitrogen or dry oxygen ambient. Junction depths, sheet resistivities, concentration profiles and diffusion coefficients have been investigated.

Two regions have been observed in the phosphorus profiles, e.g. one region has a diffusion coefficient D_I of $1.2 \times 10^{-14} \text{ cm}^2/\text{sec}$ and the other has D_{II} of $6.0 \times 10^{-14} \text{ cm}^2/\text{sec}$ through the diffusion at 900°C from doped poly-silicon deposited with the molar ratio PH_3/SiH_4 of 2.6×10^{-2} . This deviation from a simple complementary error function is qualitatively similar to the cases of doped oxide and conventional gas-phase-diffusion technique.

The arsenic profile diffused from doped poly-silicon has also shown a good agreement with the profile from doped-oxide reported by Fair²⁰⁾. The total arsenic concentration profile determined from the electrically measured profile by means of Fair's method has provided the same diffusion coefficient at 1000°C as that of Fair's doped-oxide-diffusion.

Especially, since the phosphorus profile has such a kink point, in comparison with the arsenic profile, only the sheet resistivity measurement on the surface is not enough to control the junction depth. In the case of arsenic diffusion, for constant surface concentration, the junction depth could be controlled by measuring the sheet resistivity.

No remarkable difference has been found between the sheet resistivity and junction depth diffused in both nitrogen and oxygen ambients.

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