R. F. Lever
H. M. Demsky

Water Vapor as an Oxidant in BBr_3 Open-tube Silicon Diffusion Systems

Abstract: The use of water vapor as an oxidant in place of oxygen enables a wide range of surface concentrations to be obtained in a single-step process. The concentration of boron at the silicon surface is found not to be constant throughout the diffusion process because, at the temperature used, the oxide growth is not parabolic.

Introduction

Boron tribromide is frequently used to introduce boron into silicon open-tube diffusion systems [1]. The BBr_3 vapor is mixed with oxygen, which causes it to oxidize in the high-temperature (950 – 1100 °C) deposition furnace to form B_2O_3 and bromine:

$$2BBr_3(g) + \tfrac{3}{2}O_2(g) = B_2O_3(g) + 3Br_2(g). \quad (1)$$

Since the vapor pressure of B_2O_3 is rather low (Fig. 1), being only about 10^{-7} atm at 1000 °C, liquid B_2O_3 is deposited on the furnace walls and on the silicon wafers. If care is taken to keep the initial partial pressure of BBr_3 low enough [1], the formation of "boron skin" (probably silicon boride) on the substrate wafers is avoided, and a boron-rich borosilicate glass (BSG) forms as a result of slowly depositing B_2O_3 onto silicon wafers in an oxidizing atmosphere. This glassy layer may be removed at this stage if desired, and the diffusion process then completed by heating in oxygen at temperatures that are usually somewhat higher (1050 – 1150 °C) than in the preceding diffusion step. This high-temperature step performs the dual function of drive-in, in which the boron that has entered the silicon in the first step diffuses into the desired depth, and re-oxidation, in which several thousand ångströms of oxide are grown on the wafer for use in subsequent processing steps.

This paper describes open-tube BBr_3 experiments in which water vapor, not oxygen, is used as the oxidant. In this case, we have [2]

$$BBr_3(g) + 2H_2O(g) = HBO_2(g) + 3HBr(g). \quad (2)$$

The HBO_2 may be regarded as a buffering agent for B_2O_3 vapor. If one considers the reaction

$$2HBO_2(g) = H_2O(g) + B_2O_3(g), \quad (3)$$

it is seen (Fig. 1, curve A) that

$$P_{B_2O_3}P_{H_2O}/P_{HBO_2}{}^2,$$

which is the equilibrium constant [2], is approximately 0.01. If the H_2O partial pressure is maintained at 10^{-2} atm, then a partial pressure of B_2O_3 of 10^{-8} atm results in a HBO_2 partial pressure of 10^{-4} atm. This is illustrated in Fig. 1 by curve B, a plot of the maximum HBO_2 pressure in the presence of 0.01 atm of water vapor, assuming that liquid B_2O_3 is present. While direct control of B_2O_3 partial pressure would be virtually impossible in a conventional open-tube $BBr_3 - O_2$ system because of the very low partial pressures involved, the use of HBO_2 as a buffering agent enables B_2O_3 pressures in the range 10^{-10} to 10^{-8} atm to be maintained by controlling the input BBr_3 levels in the 10 – 100 ppm range. When this is done, one expects to be able to vary the surface concentration of boron in the silicon at levels well below the solid solubility limit. This contrasts with the $BBr_3 - O_2$ system where the surface concentration before the drive-in step is invariably at the solid solubility limit of approximately 5×10^{20} atoms-cm^{-3}.

Experiments

Diffusions were made into the phosphorus-doped test wafers of 1–2 ohm-cm resistivity. The wafers were 1.25 in. (31.75 mm) in diameter, 0.008 in. (0.2 mm) thick, and oriented 3° off the (100) plane. They had been subjected to a chemical-mechanical polish and just before use were subjected to a ten-second immersion in hydrofluoric acid buffered by ammonium fluoride. The diffusions were all carried out at 1050 °C. The wafers were held vertically, parallel to the flow stream in a slotted silica oxidation boat in a rectangular silica furnace tube 5 cm wide by 8 cm high (Fig. 2).

Since H_2O vapor reacts immediately with BBr_3 at room temperature, the gas streams containing these gases may not be mixed until they come together in the diffusion furnace. Even here they must be mixed at a temperature not significantly less than the diffusion temperature. If this is not done, B_2O_3 can precipitate out by reaction (3), so that the HBO_2 content of the gas stream could be limited by this effect rather than by the inlet concentration of BBr_3, as is desired. The gas flow velocity was typically 60 cm/s, and argon was used as the dilution gas. The BBr_3 was introduced into one gas stream by bubbling argon through the liquid held at 1 °C in an oil-jacketed bubbler and then further diluted with argon before mixing with the water-bearing gas stream in the furnace. The water was introduced into the other gas stream by bubbling argon through distilled water maintained at room temperature. After subjecting the wafers to the desired gas composition for the desired time, they were withdrawn and subjected to sheet resistance and junction depth measurements. In each experimental series, all the wafers were loaded at the start and were removed two at a time as desired. To determine the effect of the removal process, individual runs were made to isolate the same data points. No significant variation was found. It should be noted that no additional drive-in diffusion was performed in these experiments, as is always done after deposition of B_2O_3 in the $BBr_3 - O_2$ system.

Experimental results

A data acquisition matrix was developed that would produce a detailed cross-sectional characterization of a $BBr_3 + H_2O$ system. The matrix consisted of four series of runs. For all four series, the deposition temperature was fixed at 1050 °C. Good glass quality and diffusion uniformity were used as the criteria for selecting 1050 °C as the working temperature. The first series of runs was designed to characterize the reaction of the system to various partial pressures of BBr_3. Throughout this series of runs, and in the next series, deposition time was fixed at 120 minutes. Argon dilution gas was maintained at a flow velocity of 60 cm/s across the substrate for all runs.

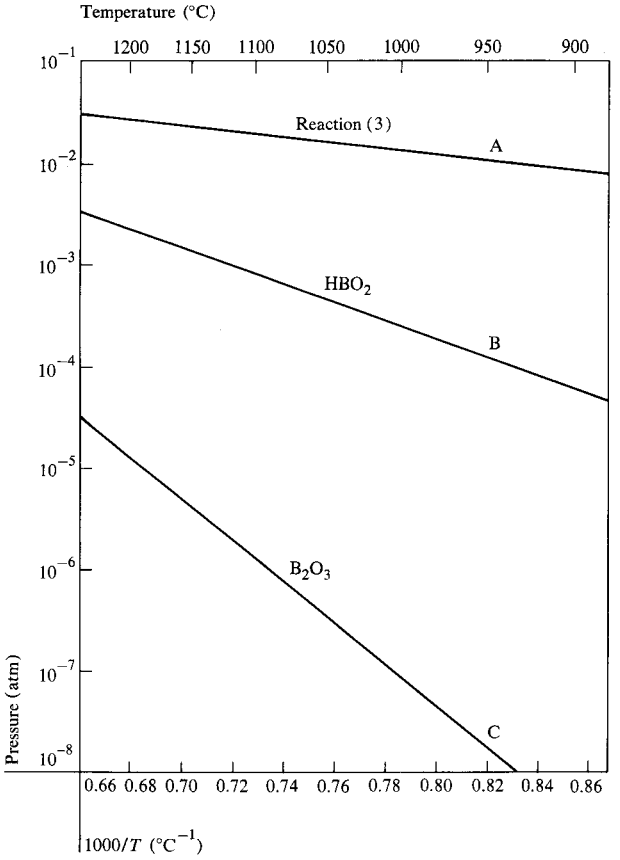


Figure 1 Partial pressures and equilibrium constants for the $B_2O_3 - H_2O$ system (see [2]). Curve A, the equilibrium constant for Eq. (3), shows that product of partial pressures of B_2O_3 and H_2O is considerably smaller than the square of the partial pressure of HBO_2. Curve B shows maximum partial pressure of HBO_2 when 0.01 atmosphere of water vapor is present. Any attempt to raise HBO_2 pressure above this level results in precipitation of liquid B_2O_3. Curve C shows partial pressure of B_2O_3 liquid.

The inlet partial pressure was maintained at 7.3×10^{-3} atm, while the BBr_3 partial pressure was varied between 8.6×10^{-6} and 9.5×10^{-5} atm. The results of this series are presented in Table 1. It can be concluded from this series that the partial pressure of B_2O_3, and therefore the doping level of boron in the silicon substrate, can be controlled over a wide range of values.

In all four series of runs, the results displayed are the averages obtained by measuring the sheet resistance R_S of the sample at five points on two wafers with a four-point probe and determining the junction depth x_j of the diffusion by staining the junction with copper after the sample had been beveled by conventional angle-lapping techniques. A virtual surface concentration C_S was computed, which, if maintained constant throughout a diffusion process in which the diffusion coefficient is independent of concentration, would yield the sheet resistance

Table 1 Effect of BBr_3 partial pressure on sheet resistance and erfc surface concentration for water vapor partial pressure of 7.3×10^{-3} atm, operating temperature of 1050 °C, and 120-min duration. Resulting borosilicate oxide thickness is also shown for these runs.

BBr (10^{-6} atm)	R_S (ohms)	G_S (mho)	x_j (10^{-6} m)	G_S/x_j (mho-cm^{-1})	$C_S(erfc)$ (10^{18} atoms-cm^{-3})	*BSG thickness* (Å)
8.6	1800	0.00056	0.7366	7.6	1.5	–
17.2	760	0.0013	0.6858	19	6.0	–
25.8	380	0.0026	0.9398	27	11	300
51.5	90	0.011	1.2192	90	42	700
94.5	8.5	0.118	1.4224	830	550	2500

and junction depth actually observed. Irvin [3] has computed this quantity in terms of average conductance and background doping. As indicated in the following section, we recognize that, in these experiments, the surface concentration is not constant throughout the experiment. However, C_S represents a good indication of the maximum dopant concentration in the sample at the completion of the experiment. Although the impurity distribution in our experiment is probably closer to the Gaussian form, we have used the complementary error function (erfc) concentration merely as a general guide to facilitate comparison with other diffusion techniques that are usually reported as having "error function" surface concentrations. In the experimental range under investigation, the "Gaussian" surface concentration is almost exactly two-thirds of the C_S given here.

After the first series of runs confirmed our expectations that the formation of HBO_2 could readily reduce the doping level below the solid solubility limit of boron in silicon, we proceeded to investigate the sensitivity of the system to changes in the partial pressure of water vapor. The second series of runs generated the data presented in Table 2. The partial pressure of BBr_3 was held constant at 5.15×10^{-5} atm and, as in the first series, $T =$ 1050 °C and diffusion time was 120 min. The partial pressure of water vapor was varied between 1.21×10^{-3} and 1.2×10^{-2} atm. Over this range C_S varied nearly two orders of magnitude.

The first and second series indicate the range of flexibility of the $BBr_3 - H_2O$ system. At appropriate concentrations of BBr_3 and H_2O, the solid solubility limit of boron in silicon (5.5×10^{20} atoms-cm^{-3}) at 1050 °C can be reached as in a conventional $BBr_3 - O_2$ system, except that the insoluble "boron skin" does not occur. With the same system, by decreasing the proportion of BBr_3 to H_2O, the diffused C_S can be moved significantly below the controllable limits of a conventional one-step $BBr_3 - O_2$ diffusion process.

The third part of the characterization matrix was designed to allow some conclusions to be drawn about the kinetics of the simultaneous diffusion-oxidation process. In this series of runs, the partial pressures of BBr_3 and H_2O were fixed at the intersection point of the first two series. Then, with BBr_3 pressure held constant at 5.15×10^{-5} atm and H_2O pressure maintained at 7.3×10^{-3} atm, the deposition time was varied from one minute to 840 minutes. Junction depth data could not be ascertained on runs with times of less than five minutes, since the diffusions were too shallow to see with the bevel and stain technique. However, the sheet conductance G_S as a function of time has been plotted in Fig. 3 for all runs.

The data presented in Table 3 and Figs. 3 and 4 clearly indicate that the C_S obtained, for one particular ratio of BBr_3 to H_2O, monotonically decreases with an increase in diffusion time. During the first five minutes of diffusion, the sheet conductance is rising rapidly. Between five and ten minutes into the diffusion process a leveling off occurs, and after ten minutes the sheet conductance essentially remains constant while the diffusion front continues to move deeper into the substrate. These data suggest that all of the boron that is diffused into the silicon is diffused into the surface of the wafer or is deposited near the $Si-SiO_2$ interface in the form of BSG during the first ten minutes of the process. After ten minutes, a barrier SiO_2 layer that is not rich in boron is sufficiently formed to block continued diffusion from the ambience. The remaining diffusion process is a redistribution of the boron diffused into the surface during the first ten minutes.

The fourth and final part of the experimental matrix was designed to shed more light on the nature of the barrier layer and the boron-doped glass and/or their reac-

Figure 2 Apparatus used for wafer preparation.

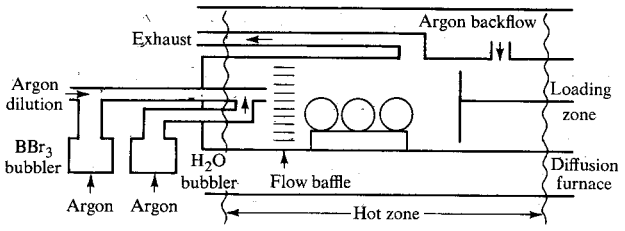


Table 2 Effect of varying partial pressure of water vapor on sheet resistance and erfc surface concentration, where BBr_3 partial pressure is held at 5.15×10^{-5} atm. Run duration was 120 minutes at a temperature of 1050 °C.

H_2O (10^{-3} atm)	R_S (ohms)	G_S (mho)	x_j (10^{-6} m)	G_S/x_j (mho-cm^{-1})	$C_S(erfc)$ (10^{18} atoms-cm^{-3})
1.21	8.9	0.112	1.2192	920	610
4.76	26.6	0.038	1.5748	242	140
7.3	90.0	0.011	1.2192	90	46
12.7	650	0.0015	1.0160	14.8	4.2

tion to changes in the make-up of the ambient gases. In this series, diffusion was carried on under the same conditions that were used to generate the data presented in Table 3. The mole ratio of BBr_3 to H_2O was held constant at 0.007 for a period of ten minutes. At this point, both reactant flow streams were terminated, and the wafers were allowed to remain in an argon ambience at 1050 °C for times up to two and one-half hours. As expected, the argon heat treatment allowed boron to diffuse through the thin barrier layer and into the silicon. Diffusion in this process is no longer a simple redistribution but is a continuous diffusion from a finite source. Table 4 indicates that C_S did not decrease as before but did in fact increase slightly to a value of 2×10^{20} atoms/cm^3 before declining again due to boron depletion from the thin BSG.

To find the maximum doping level possible from the BSG formed by the $BBr_3 - H_2O$ reaction, this experiment was performed again with the initial diffusion time (H_2O, BBr_3 flowing) increased from ten to 60 minutes to form a glass thickness sufficient to act as an infinite source. After 60 minutes, the reactants were again shut off and the wafers subjected to an argon drive-in of up to 60 minutes more. This experiment indicated that BSG soluble in HF, capable of doping silicon near the solid solubility limit, is formed from the reaction of BBr_3 and H_2O with a ratio BBr_3/H_2O of 0.007. Also, a period of ten minutes was found to be necessary before any new boron could diffuse into the substrate.

Table 3 Effects of varying time. Gas stream contained 7.3×10^{-3} atm of BBr_3 and 5.15×10^{-5} atm of water vapor; temperature was 1050 °C.

Time (min)	R_S (ohms)	G_S (mho)	x_j (10^{-6} m)	G_S/x_j (mho-cm^{-1})	C_S (*erfc*) (10^{19} atoms-cm^{-3})
1	480	0.00208	–	–	–
2	350	0.00286	–	–	–
3	280	0.00358	–	–	–
4	230	0.00435	–	–	–
5	176	0.00565	0.1143	495	31
10	115	0.00869	0.3048	286	17
20	115	0.00869	0.4318	202	11
60	100	0.0100	0.8382	119	6.3
120	95	0.0105	1.270	83	4.2
150	110	0.0090	1.3208	69	3.3
300	97	0.0103	1.8034	57	2.7
840	117	0.0855	3.0734	27.8	1.0

Table 4 Effect of terminating flow of BBr_3 and H_2O after 10 minutes at levels maintained in Table 3. Note appreciable rise in sheet conductance and depletion of boron source as indicated by decreasing C_S.

Argon drive-in time (min)	R_S (ohms)	G_S (mho)	x_j (10^{-6} m)	C_S (*erfc*) (10^{19} atoms-cm^{-3})
0	118	0.00850	0.3048	17
10	49	0.0204	0.6096	21
60	45	0.0222	0.8890	14
140	43	0.0232	1.3208	9.4

Figure 3 Sheet conductance as a function of time from Table 3. Note that after ten minutes conductance levels off, suggesting that after this time no more boron enters the silicon from the glassy layer.

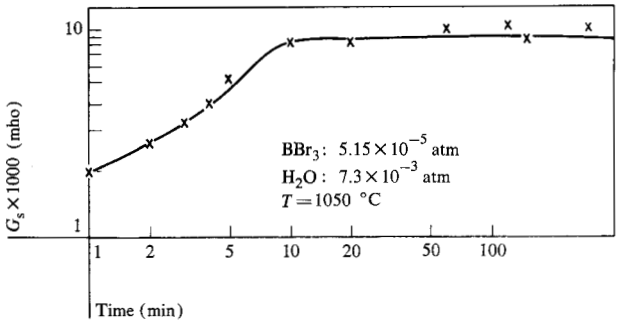
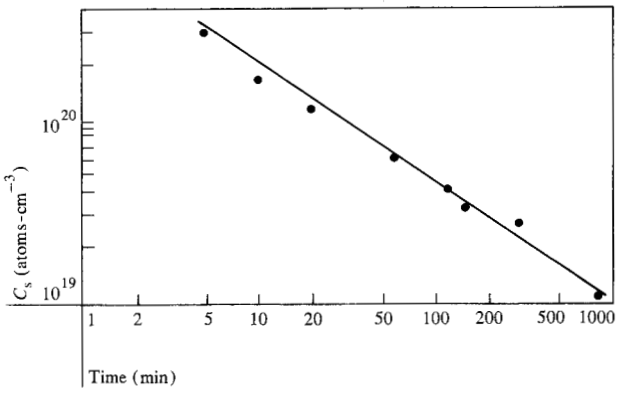


Figure 4 Effective surface concentration as a function of time from Table 3.



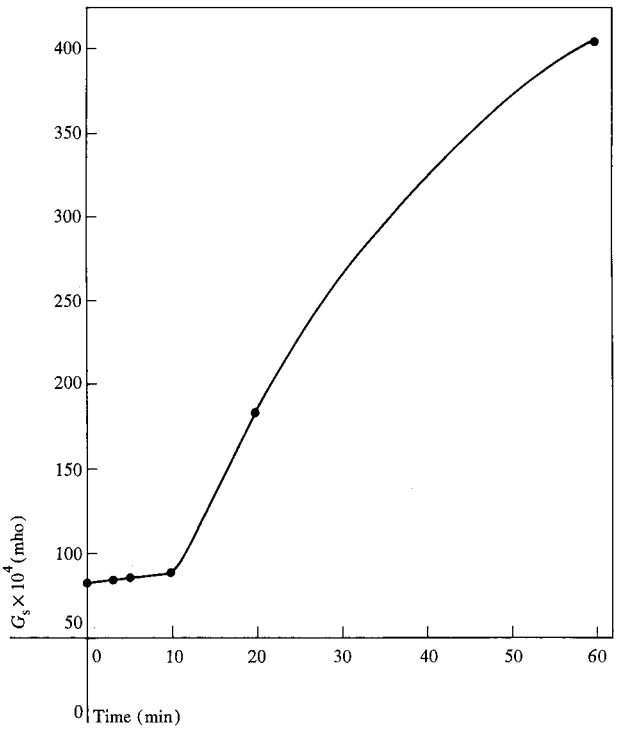


Figure 5 Rise in sheet conductance after heating in pure argon following 60 minutes of the treatment shown in Fig. 2.

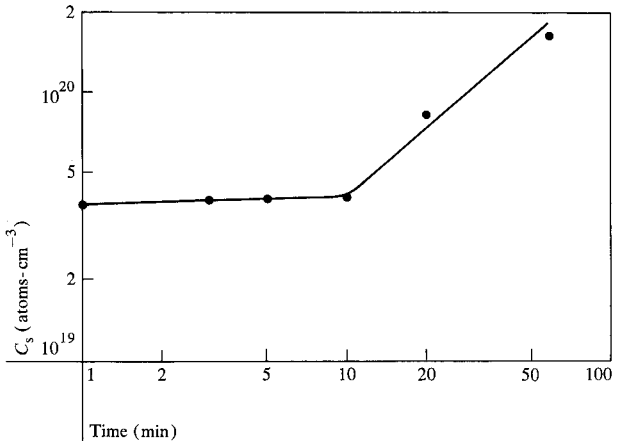


Figure 6 Rise in effective surface concentration corresponding to Fig. 4. It is clear that after a ten-minute blocking period, boron enters silicon from glassy layer.

This result is consistent with the assumption that a barrier layer, only lightly doped with boron, is constantly formed as long as the H_2O reactant stream remains on. As is discussed in the concluding section of this paper, an oxide film of approximately 80 Å thickness acting at the interface between the heavily doped BSG and the silicon substrate can cause the behavior exhibited by this fourth series of runs. The results of the final series of runs are presented in Table 5 and Figs. 5 and 6.

Table 5 Effect of terminating flow of BBr_3 and H_2O after 60 minutes of treatment described in Table 3. Note considerable rise in sheet conductance and "effective" surface-concentration after a 10-minute incubation period.

Argon drive-in time (min)	R_S (ohms)	G_S (mho)	x_j (10^{-6} m)	C_S (*erfc*) (10^{19} atoms-cm^{-3})
0	120	0.00835	1.0668	3.9
3	120	0.00835	1.0668	3.9
5	118	0.00850	1.0922	3.95
10	115	0.00870	1.1176	4.0
20	55	0.01820	1.2446	8.0
60	25	0.0400	1.4732	16.0

Discussion

As described in the introduction, the use of water vapor as the oxidant for BBr_3 enables effective pressures of B_2O_3 to be maintained in the system, which are well below the vapor pressure of liquid B_2O_3. We may assume, therefore, that as the partial pressure of BBr_3 in the gas stream is reduced, the concentration C_0 of boron at the surface of the growing glassy layer is also reduced. We assume that the surface of the layer is in equilibrium with the gas phase, so that C_0 stays constant during a diffusion run, at a value determined by the B_2O_3 level in the gas phase. Even with this simplification, the process by which the boron enters the silicon is rather complex. Water diffuses through the glassy layer and reacts with the silicon at the interface to cause oxide growth, while at the same time boron diffuses through the glass and into the silicon. The diffusion coefficients of both H_2O and boron in the glass are dependent on the local glass composition, being larger in highly doped borosilicate glass than in pure SiO_2. The boron enters the silicon by means of a surface reaction in which boron oxide in the glass is reduced by the silicon, while the kinetics of the oxide growth are influenced by the kinetics of the reaction between silicon and the water-derived oxidizing species diffusing through the glass. Finally, the diffusion constant of boron in the silicon is concentration-dependent. It is instructive, however, to consider a simplified model in which diffusion coefficients are not concentration-dependent and where equilibrium is assumed to exist at the Si-BSG interface. It is then possible to show in simple mathematical form how the concentration N_i of boron at the surface of the silicon is influenced by the relative rates at which water vapor and boron are able to diffuse through the growing BSG film.

Assuming equilibrium to exist at the silicon-BSG interface and neglecting concentration effects, one may assume an oxide growth rate given by

$$z_i = 2.3y_i = kt^{\frac{1}{2}}, \tag{4}$$

where k is the usual parabolic growth constant, z_i is the

position of the interface relative to the oxide surface, y_i is the position relative to the original silicon surface, and 2.3 is the ratio of the density of pure silicon to that of Si in SiO_2. For such a moving boundary diffusion problem, one may adopt Neumann's solution [4] as used by Grove, Leistiko, and Sah [5] for the similar problem of impurity redistribution during thermal oxidation. The boron concentrations in both media have the form of an error function complement, and the concentration N_i of boron at the interface is constant. The concentration C_i of the glass side of the interface is a single-valued function of N_i, because equilibrium is assumed. This type of solution is shown in Fig. 7. The concentration C of boron in the oxide is referred to coordinates having origin at 0 on the oxide surface, and the depth below the surface is denoted by z. The concentration N of boron in the silicon is referred to coordinates at rest relative to the silicon, having origin at 0′, the initial position of the silicon surface. The depth in the silicon below this original surface is denoted by y. We then have

$$C = C_\infty + (C_0 - C_\infty) \text{ erfc } (z/z_0), \tag{5}$$

$$N = N_\infty + (N_0 - N_\infty) \text{ erfc } (y/y_0), \tag{6}$$

where $z_0 = 2(D_1 t)^{\frac{1}{2}}$, $y_0 = 2(D_2 t)^{\frac{1}{2}}$, and D_1 and D_2 are the diffusion coefficients of boron in BSG and silicon, respectively.

Equations (4), (5), and (6) yield constant values for C_i and N_i. If one adds the condition for conservation of boron at the boundary,

$$D_1 \frac{dC}{dz} - D_2 \frac{dN}{cy} = N \frac{dy_i}{dt} - C \frac{dz_i}{dt}, \tag{7}$$

one obtains the following expression:

$$\frac{2.3(C_0 - C_i)}{G(z_i/z_0)} - \frac{N_i - N_\infty}{F(y_i/y_0)} = 2.3\ C_i - N_i, \tag{8}$$

where

$$F(x) = \pi^{\frac{1}{2}} x \text{ (erfc } x) \exp (x^2), \tag{9}$$

$$G(x) = \pi^{\frac{1}{2}} x \text{ (erf } x) \exp (x^2). \tag{10}$$

The ratios y_i/y_0 and z_i/z_0 are simply given by $0.22kD_2^{-\frac{1}{2}}$ and $0.5kD_1^{-\frac{1}{2}}$, respectively, while, in our experiments, N_∞ may be taken as equal to zero. Provided $N_S = f(C_S)$ is known, N_S and C_S may be found from Eq. (8) and substituted into Eqs. (5) and (6) to give C_∞ and N_0. For boron in silicon, $k \ll D_2^{\frac{1}{2}}$ so that $N_0 \simeq N_i$. We expect N_i/C_i to be approximately 0.05 [6].

Graphs of F and G are given by Crank [4]. For $F(x)$ the effect of increasing x is to increase $x \exp (x^2)$ while decreasing erfc (x). Hence $F(x)$ tends asymptotically to unity as $x \to \infty$. The behavior of $G(x)$ is very different, however, being completely dominated by the term exp (x^2). For $x > 1$, G is liable to be large since $G(1) = 4.06$, $G(2) = 193$, $G(3) = 4308$, $G(4) = 6.3 \times 10^7$, etc. That is, if k is greater than $2\ D_1^{\frac{1}{2}}$, very little boron will reach the interface. This is also intuitively obvious from the very rapid fall off of erfc (x) for $x > 1$.

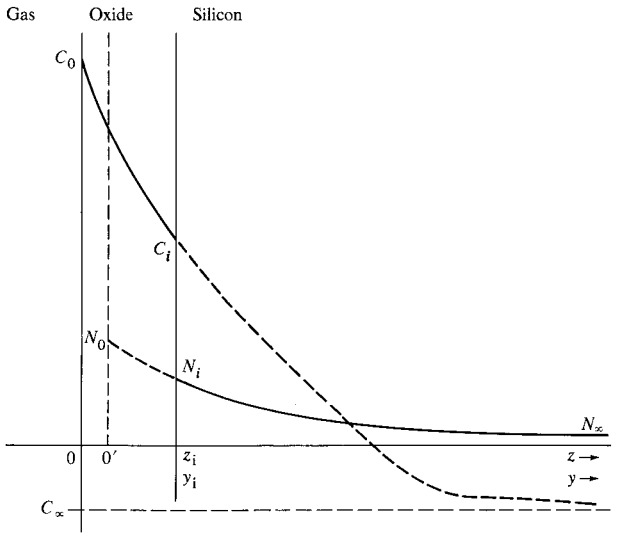


Figure 7 Diffusion profiles for diffusion through an oxide into silicon, where surface concentration of oxide is constant and growth rate is parabolic.

Horiuchi and Yamaguchi [7] give $D_1^{\frac{1}{2}}$ as about 1.7×10^{-3} μm-h$^{-\frac{1}{2}}$ for the penetration of boron through thermal SiO_2 at 1050 °C, while Schwenker [6] gets approximately 10^{-2} μm-h$^{-\frac{1}{2}}$ for diffusion in vapor-deposited BSG containing approximately 10% B_2O_3.

From the literature [8, 9], one obtains a value of k, the parabolic rate constant for oxide growth, of 0.65 μm-h$^{-\frac{1}{2}}$. Hence, for the conditions of our third run, with a partial pressure of H_2O equal to 7.3×10^{-3} atm, one may estimate $k = 0.06$ μm-h$^{-\frac{1}{2}}$. From Table 1, it is seen that for a BBr_3 partial pressure of 5.15×10^{-5}atm, 700 Å of BSG grows in 2 h. If we assume a simple parabolic law, this gives $k = 0.05$, in reasonable agreement with other derived values in the literature. Our estimated z_i/z_0 ranges from 2.5 to about 15 depending on our estimate of the value of D_1. Because this yields values of G in the range of 2,300 to 10^{100}, application of Eq. (8) would lead us to expect no detectable diffusion of boron into the silicon. However, it is known that the growth of SiO_2 is not strictly parabolic, particularly at oxide growth temperatures as low as 1050 °C [8] where equilibrium is *not* obtained at the Si-SiO_2 interface. In the earlier stages of oxide growth, the growth rate is considerably less than would be expected by a simple application of Eq. (4). Hence, during the first few minutes of growth, one expects boron to diffuse through the oxide layer and into the silicon. At the later stages, the fact that the oxidizing

species is able to diffuse through the growing oxide considerably more rapidly than is boron leads to the diffusion being cut off. In the experiment shown in Fig. 6, ten minutes were required for the boron to diffuse through the barrier layer after oxidation had ceased. The diffusion length $2(Dt)^{\frac{1}{2}}$ associated with the process is, using $t = 1/6$ h and $D_1^{\frac{1}{2}} = 0.01$ μm-h$^{-\frac{1}{2}}$ [6], simply 80 Å. Since this is somewhat smaller than the total oxide thickness observed, the formation of a barrier layer of that order of magnitude during oxidation is quite plausible. An exact model of the system would be extremely complex and would require knowledge of surface processes and concentration-dependent diffusion coefficients that are not available. The influence of surface limitations on the early stages of oxide growth has, however, been very well established [8, 9] and is bound to permit enhanced diffusion during the early stages of the $BBr_3 - H_2O$ process described in this paper.

Conclusions

1. Using the $BBr_3 - H_2O$ process, diffusions having a final C_S in the range 10^{18} to 5×10^{20} atoms-cm^{-3} are readily obtained.
2. The results are strongly influenced by the oxide growth kinetics in the early quasi-linear stages of growth.

Acknowledgments

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The authors are located at the IBM System Products Division East Fishkill Facility, Hopewell Junction, New York 12533.