

5. Calculating the weight of solid necessary to achieve 99.8% conversion: Substituting into Equation (E11-3.4), we obtain

$$W = \frac{1 \times 10^{-6} \text{ m}^3/\text{s}}{(0.0627)(4.42 \times 10^{-10} \text{ m}^3/\text{m}^2 \cdot \text{s})(530 \text{ m}^2/\text{g})} \ln \frac{1}{1 - 0.998} = 423 \text{ g}$$

6. The reactor length is

$$L = \frac{W}{A_c \rho_b} = \frac{423 \text{ g}}{(2.03 \times 10^{-3} \text{ m}^2)(1.4 \times 10^6 \text{ g/cm}^3)} = 0.15 \text{ m}$$

11.7 The Determination of Limiting Situations from Reaction Data

For external mass transfer-limited reactions in packed beds, the rate of reaction at a point in the bed is

$$-r'_A = k_c a_c C_A \quad (11-72)$$

The correlation for the mass transfer coefficient, Equation (10-66), shows that k_c is directly proportional to the square root of the velocity and inversely proportional to the square root of the particle diameter:

$$k_c \propto \frac{U^{1/2}}{d_p^{1/2}} \quad (11-73)$$

We recall from Equation (11-55) that the variation of external surface area with catalyst particle size is

$$a_c \propto \frac{1}{d_p}$$

Consequently, for external mass transfer-limited reactions, the rate is inversely proportional to the particle diameter to the three-halves power:

$$-r'_A \propto \frac{1}{d_p^{3/2}} \quad (11-74)$$

From Equation (10-85) we see that for gas-phase external mass transfer-limited reactions, the rate increases approximately linearly with temperature.

When internal diffusion limits the rate of reaction, we observe from Equation (11-35) that the rate of reaction varies inversely with particle diameter, is independent of velocity, and exhibits an exponential temperature dependence which is not as strong as that for surface-reaction-controlling reactions. For surface-reaction-limited reactions the rate is independent of particle size and is a strong function of temperature (exponential). Table 11-1 summarizes the dependence of the rate of reaction on the velocity

If an industrial reaction is not mass transfer-limited, it is probably run incorrectly.

L. D. Schmidt, U. of Minn.

TABLE 11-1

Type of Limitation	Variation of Reaction Rate with		
	Velocity	Particle Size	Temp
External diffusion	$U^{1/2}$	$(d_p)^{-3/2}$	Exp
Internal diffusion	Independent	$(d_p)^{-1}$	Exp
Surface reaction	Independent	Independent	Exp

through the bed, particle diameter, and temperature for the limitations we have been discussing.

The exponential temperature dependence for internal reactions is usually not as strong a function of temperature as is for surface reaction limitations. If we would calculate an activation energy between 8 and 24 kJ/mol, chances are that the reaction is surface limited. An activation energy of 200 kJ/mol, however, indicates that the reaction is reaction rate-limited.

11.8 Chemical Vapor Deposition (CVD) Reactors

As discussed in Section 6.6, CVD is a very important process in the microelectronics industry. The fabrication of microelectronic devices include as few as 30 or as many as 200 individual steps to produce up to 10^6 transducers per chip. An abbreviated schematic of the process involved in producing a typical computer chip is shown in Figure 11-1.

Starting from the upper left we see that single crystal silicon is grown in a Czochralski crystalizer, then sliced into wafers, and physically polished. These polished wafers serve as a starting point for a variety of microelectronic devices. A typical fabrication process for processing the wafer beginning with the formation of a silicon layer on top of the silicon. The SiO_2 layer may be formed either by a silicon layer or by laying down a SiO_2 layer by Chemical Vapor Deposition (CVD). Next the wafer is masked with a polymer photoresist (PR) with the pattern to be etched onto the SiO_2 layer is placed over the wafer is exposed to ultraviolet irradiation. If the mask is a photoresist, the light will cause scission in the polymer so that the exposed areas will dissolve when the wafer is placed in the developer. On the other hand, if the mask is a negative PR mask is exposed to ultraviolet irradiation, crosslinking of the polymer chains occurs and the unexposed areas dissolve in the developer. The undeveloped portion of the PR (in either case) will protect the silicon from etching.

After the exposed areas of SiO_2 are etched to form trench structures, wet etching (see P5-11) or plasma etching, the remaining PR is removed. Next the wafer is placed in a furnace containing gas molecules

5. Calculating the weight of solid necessary to achieve 99.8% conversion: Substituting into Equation (E11-3.4), we obtain

$$W = \frac{1 \times 10^{-6} \text{ m}^3/\text{s}}{(0.0627)(4.42 \times 10^{-10} \text{ m}^3/\text{m}^2\cdot\text{s})(530 \text{ m}^2/\text{g})} \ln \frac{1}{1 - 0.998} = 423 \text{ g}$$

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If an industrial reaction is not mass transfer-limited, it is probably run incorrectly.

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TABLE 11-1

Type of Limitation	Variation of Reaction	
	Velocity	Particle Size
External diffusion	$U^{1/2}$	$(d_p)^{-3/2}$
Internal diffusion	Independent	$(d_p)^{-1}$
Surface reaction	Independent	Independent

through the bed, particle diameter, and temperature limitations we have been discussing.

The exponential temperature dependence for reactions is usually not as strong a function of temperature for surface reaction limitations. If we would calculate between 8 and 24 kJ/mol, chances are that the reaction is limited. An activation energy of 200 kJ/mol, however, reaction is reaction rate-limited.

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A negative photoresist is exposed to ultraviolet irradiation and the unexposed portion of the photoresist is dissolved. The undeveloped portion of the photoresist (in either case) is used as a mask for etching. After the exposed areas of SiO_2 are etched to form the desired pattern, the photoresist is removed by etching (see P5 11) or plasma etching) the exposed areas of the wafer are etched to form the desired pattern.

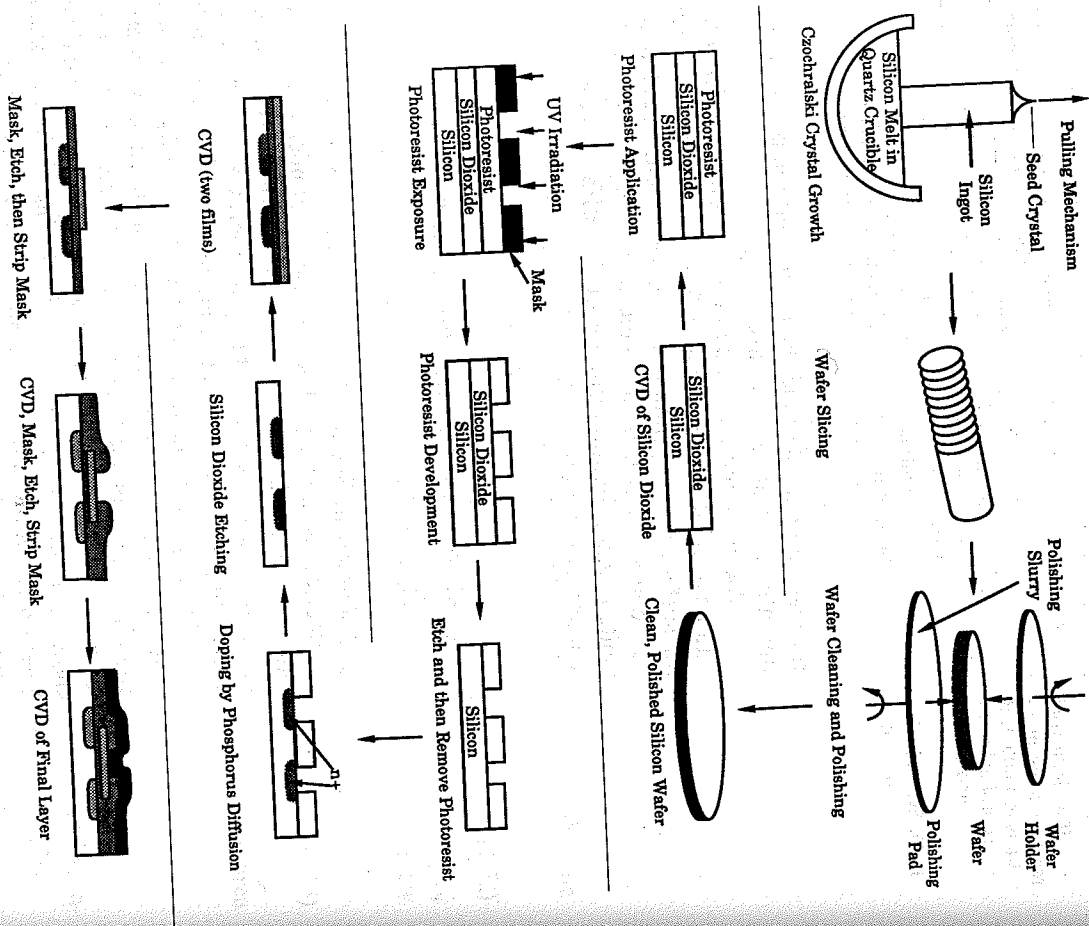


Figure 11-11 Microelectronic fabrication steps.

dopant, which then diffuse into the exposed silicon. After diffusion of dopant to the desired depth in the wafer it is removed and then covered with SiO₂ by CVD. The sequence of masking, etching, CVD, and metallization continues until the desired device is formed. A schematic of a final chip is shown in the lower right-hand corner of Figure 11-11.

One of the key steps in the chip making process is the deposition of different semiconductors and metals on the surface of the chip. This step can be achieved by CVD. CVD mechanisms were discussed in Chapter 6, consequently this section will focus on CVD reactors. A number of CVD

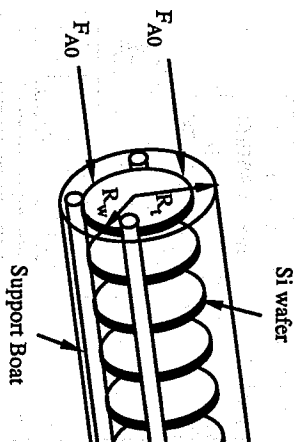
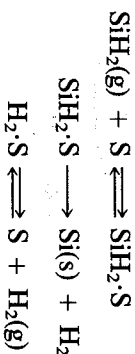


Figure 11-12 LPCVD boat reactor

horizontal and vertical reactors. A description of the equations are given by Jensen.⁶

One of the more common CVD reactors is the LPCVD (LPCVD) reactor. This reactor operates at pressures of 100 Pa. The main advantage of the LPCVD is its capability to deposit a large number of wafers without detrimental effects to the large increases in the diffusion coefficient to the large number of wafers without detrimental effects to transfer. A schematic of a LPCVD reactor is shown in Figure 11-12. To illustrate LPCVD modeling we shall use as an example, the deposition of silicon from a gas stream

CVD Reaction Sequence in Silicon Deposition



Here we have assumed that the equilibrium for the first reaction lies far to the right. The corresponding rate law is

$$r_{\text{Si}}^{\text{CVD}} = \frac{k_1 P_{\text{SiH}_2}}{1 + K_1 P_{\text{H}_2} + K_2 P_{\text{SiH}_2}}$$

Recalling that the adsorption constants K_1 and K_2 are functions of temperature, an excellent approximation at high temperatures is

$$1 \gg (K_1 P_{\text{H}_2} + K_2 P_{\text{SiH}_2})$$

consequently, the deposition rate can be modeled as

$$r_{\text{Si}}^{\text{CVD}} \approx k_1 P_{\text{SiH}_2} \approx k_C P_{\text{SiH}_2} \approx k_C A$$

where $A \equiv \text{SiH}_2$

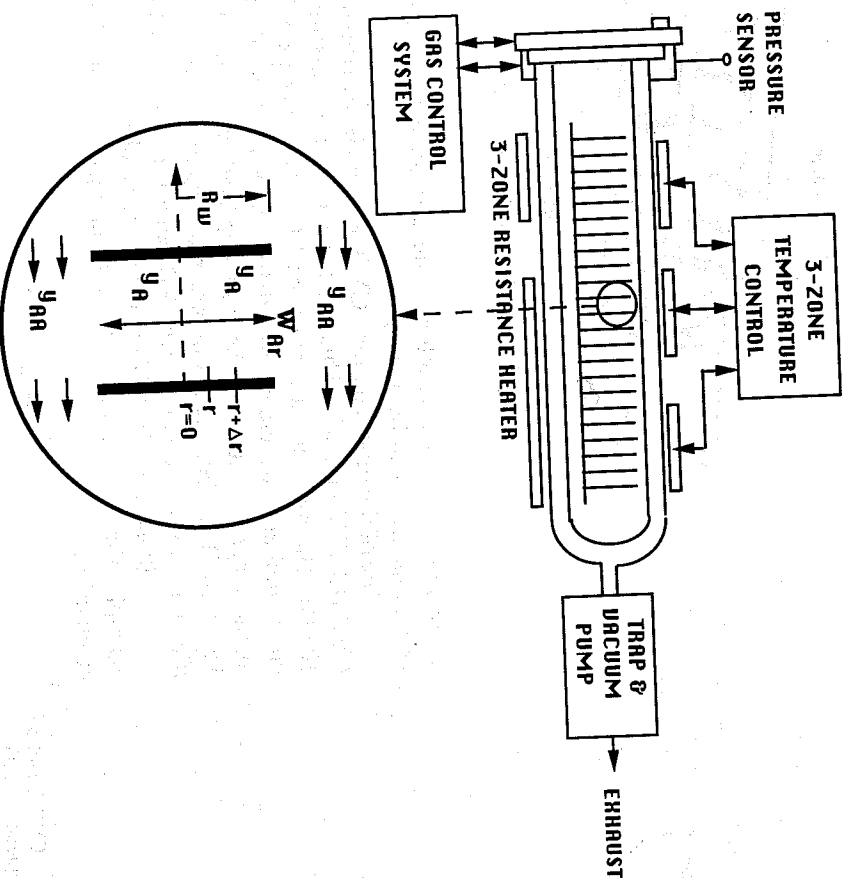


Figure 11-13 LPCVD boat reactor with peripherals.

Modeling Concepts We shall model the axial flow in the annular region as being laminar. This assumption is reasonable because a typical Reynolds number for flow in a LPCVD reactor is less than 1. As the reactant gases flow through the annulus, the reactants diffuse from the annulus radially inward between the wafers to coat them.⁷

The reacting gas flows through the annulus between the outer edges of the cylindrical wafers and the tube wall (see Figure 11-13). The corresponding cross-sectional area of the annulus is

$$A_c = \pi(R_t^2 - R_w^2)$$

Flow in the Annulus

where R_t and R_w are the radii of the tube and wafer, respectively. Because SiH_2 is being consumed by CVD, the mole fraction of SiH_2 (i.e., the reactant) in the annulus, y_{AA} , decreases as the reactant flows down the length of the annulus.

The reacting gases diffuse out of the annular region between the wafers where the mole fraction is represented by y_{AA} . The reactants diffuse radially inward from the wafers to the wafer surface. The reaction products then diffuse back into the gas stream axially flowing in the annulus. This is a manner analogous to flow through a packed bed reactor where reaction gases diffuse into the catalyst pellets. In this case, the effectiveness factor to determine the overall rate of reaction (i.e., mass) of reactor bed. We can extend this idea to LPCVD where the reactants diffuse from the annular flow channel to the wafers.

11.8.1 Effectiveness Factor for a LPCVD Reactor

Silicon will deposit on the wafers, the reactor support. Deposition on the walls and support will take place. Concentrations in the annulus. However, the concentration of the wafers is less than the concentration in the annulus. Rate of deposition on the wafer will be less than the rate of deposition in the annulus. Fortunately these two concentrations can be related by an effectiveness factor. We can determine the effectiveness factor by determining the concentration profile in the region between the wafers i

Actual rate of reaction

$\eta = \frac{\text{Rate of reaction when entire wafer surface is exposed to } C_{AA}}{\text{the concentration in the annulus } C_{AA} \text{ (i.e., } C_{AA} \text{)}} = \frac{2\pi R_w \int_0^{R_w} (-r'_{AA}(r)) dr}{2\pi R_w^2 (-r'_{AA}(R_w))}$

$$\eta = \frac{2 \int_0^{R_w} 2\pi r (-r'_{AA}(r)) dr}{2\pi R_w^2 (-r'_{AA}(R_w))} = \frac{2\pi R_w \int_0^{R_w} (-r'_{AA}(r)) dr}{2\pi R_w^2 (-r'_{AA}(R_w))}$$

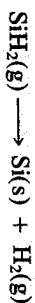
where ℓ is the distance between wafers, and $-r'_{AA}$ is the rate of consumption of A in the annular region. We use η to express the actual rate of reaction per unit area of wafer in terms of the rate of reaction at conditions in the annulus.

Actual Rate = $-r'_{AA} \eta$

Letting 'a' be the wafer surface area per unit volume of reactor, the rate of consumption of species A by the wafer per unit volume of reactor is

Example 11-4 Diffusion Between Wafers

Derive an equation for the reactant gas concentration as a function of wafer radius and then determine the effectiveness factor.



In terms of the diffusing gas phase components, we can write this reaction as:



Solution

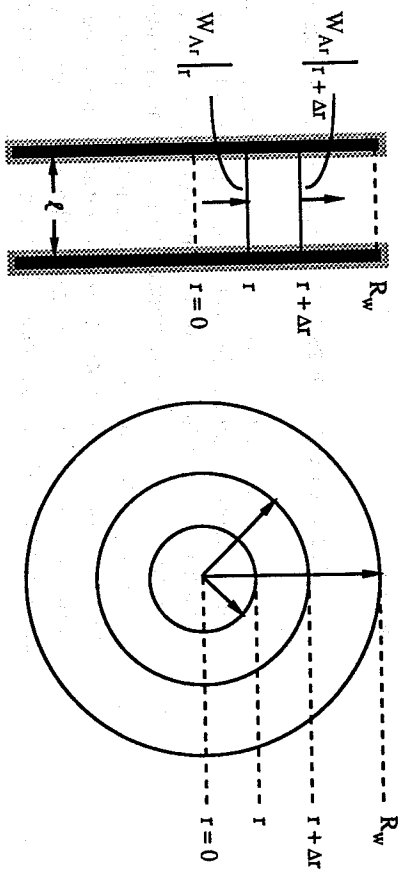


Figure E11-4.1

The shell balance on the reactant diffusing between two wafers separated by a distance ℓ shown in Figure E11-4.1 gives

$$\text{In} - \text{Out} + \text{Generation} = 0$$

$$W_{Ar} 2\pi r \ell - W_{Ar} 2\pi (r + \Delta r) \ell + 2(2\pi r \Delta r r_{Aw}') = 0$$

where r_{Aw}' = rate of generation of species A per unit wafer surface area. The factor of 2 appears in the generation term because there are two wafer surfaces exposed in each differential volume element. Dividing by $2\pi r \Delta r \ell$, taking the limit as Δr approaches zero, and then rearranging gives

$$\frac{1}{r} \frac{d(W_{Ar} r)}{dr} = \frac{2r_{Aw}'}{\ell} \quad (\text{E11-4.1})$$

Recalling the constitutive equation for the molar flux W_{Ar} in radial coordinates

$$W_{Ar} = -c D_{AB} \frac{dy_A}{dr} + y_A (W_{Ar} + W_{Br}) \quad (\text{E11-4.2})$$

For every one molecule of SiH_2 (i.e., species A) that diffuses *IN* one molecule of H_2 (i.e., species B) diffuses *OUT*.

$$W_{Br} = -W_{Ar}$$

Then

Diffusion with
reaction between
wafers

For a first-order reaction

$$-r_{Aw}' = kC_A$$

Substituting equations (11-76) and (E11-4.3) into Equation

$$\frac{1}{r} \frac{d}{dr} \left(r \frac{dC_A}{dr} \right) - \frac{2k}{D_{AB} \ell} C_A = 0$$

The corresponding boundary conditions are

$$\text{At } r = R_w \quad C_A = C_{AA}$$

$$\text{At } r = 0 \quad \frac{dC_A}{dr} = 0 \text{ and } C_A \text{ is finite}$$

Let $\lambda = r/R_w$ and $\Psi = C_A/C_{AA}$

then

$$\frac{1}{\lambda} \frac{d}{d\lambda} \left(\lambda \frac{d\Psi}{d\lambda} \right) - \phi_1^2 \Psi = 0$$

where $\phi_1^2 = \frac{2kR_w^2}{D_{AB} \ell}$

The boundary conditions are

$$\text{at } \lambda = 0: \quad \frac{d\Psi}{d\lambda} = 0 \text{ and } \Psi \text{ is finite}$$

$$\text{at } \lambda = 1 \quad \Psi = 1.0$$

Equation (E11-4.7) is a form of Bessel's Equation.

The general form of the solution to Bessel's equation

$$\Psi = A I_0(\phi_1 \lambda) + B K_0(\phi_1 \lambda)$$

Where I_0 is a modified Bessel function of the first kind K_0 is a modified Bessel function of the second kind second boundary condition requires Ψ to be finite B must be zero because $K_0(0) = \infty$. Using the first we get $1 = A I_0(\phi_1)$, then $A = 1/I_0(\phi_1)$. The concentration between the wafers is

$$\Psi = \frac{C_A}{C_{AA}} = \frac{I_0(\phi_1 \lambda)}{I_0(\phi_1)}$$

$$\eta = \frac{-W_{Ar}|_{r=R_w} (2\pi R_w \ell)}{2\pi R_w^2 (-r_{Aw}')}$$

$$\eta = \frac{-\left(-D_{AB} \frac{dC_A}{dr} \Big|_{r=R_w}\right) (2\pi R_w \ell)}{2kC_{AA}\pi R_w^2} = 2 \frac{d\psi}{d\lambda} \Big|_{\lambda=1} \frac{d\psi}{d\lambda} \Big|_{\lambda=1} \frac{2\pi R_w^2 \ell}{D_{AB} \ell} \quad (\text{E11-4.11})$$

$$\eta = 2 \frac{d\psi}{d\lambda} \Big|_{\lambda=1}$$

$$\eta = \frac{2I_1(\phi_1)}{\phi_1 I_0(\phi_1)}$$

(E11-4.12)

The concentration profile along the radius of the wafer disk as well as the wafer shape is shown in Figure E11-4.2 for different values of the Thiele modulus.

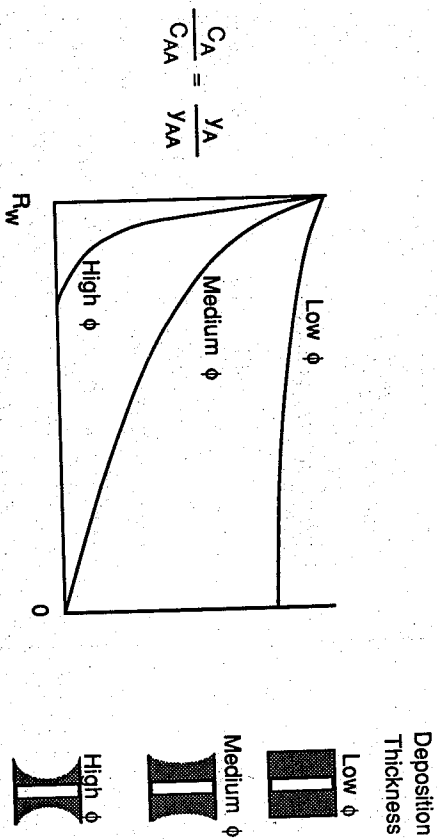


Figure E11-4.2 Radial concentration profile.

Deposition on the Peripherals Silicon will deposit on the walls of a reactor and on the boat support in addition to the wafers. This rate of deposition on the walls and support is

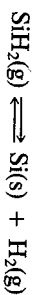
$$-r'_{Si,p} = -r'_{AA} = (1 + \alpha)kC_{AA}$$

$$\alpha = \frac{\text{surface area of boat support}}{\text{surface area of tube}}$$

Owing to high temperature and low pressure, radiation is the dominant heat transfer mechanism, therefore small temperature differences exist between the wafer and reactor wall. Consequently, there is no need to couple the

Example 11-5 CVD Boat Reactor

Silicon is to be deposited on wafers in a LPCVD reactor. an analytical solution for the silicon deposition rate and reactor profile for the simplified version of the LPCVD reactor. Analytical solutions of this type are important in that an engineer has an understanding of the important parameters and their sensitivity making a number of 'runs' on the computer. The reaction is



Sections of the reactor are shown in Figures E11-5.1 and E11-5.2

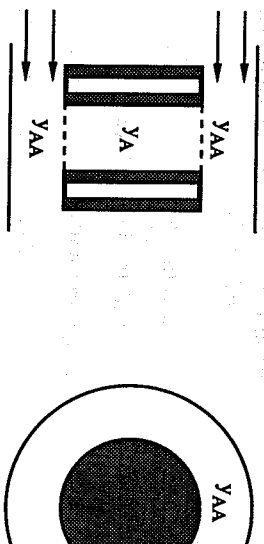


Figure E11-5.1

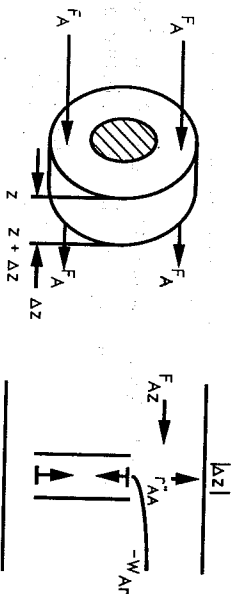


Figure E11-5.2

1. Balances: In forming our shell balance on the annulus assume there are no radial gradients in the annulus. The outer tube walls and the boat which consume some silicon by deposition on the walls in the balance. In addition, there may be any dispersion or diffusion in the axial direction.

Balance on reactant A

IN	OUT	GENERATION	GENERATION
By Convection	By Convection	By reaction on wafer surface	By reaction on tube walls
$F_{Az} _z$	$-F_{Az} _{z+\Delta z}$	$+r'_{AA}\eta\pi R_w^2\Delta z$	$+r'_{AA}2\pi R_w\Delta z$

Dividing through by Δz and taking the limit as $\Delta z \rightarrow 0$

$$- \frac{dF_{Az}}{dz} + \underbrace{r''_{AA} [(1 + \alpha)2\pi R_i + \eta a \pi R_i^2]}_{R''_{AA}} = 0 \quad (E11-5.2)$$

2. **Rate laws:** The rate of silicon deposition, r''_{Si} , (mol/dm²·s) is equal to the rate of depletion of SiH₂.

$$r''_{Si} = -r''_A = kC_A \quad (11-76)$$

where the units of C_A and k are (mol/dm³) and (dm/s), respectively. Deposition takes place on the reactor walls, the support, and on the wafer surfaces. The corresponding depletion of reactant gas on each of these surfaces is

Wafer (mol/s): $kC_{AA}\eta a \pi R_i^2 \Delta z$

Reactor wall (mol/s): $kC_{AA}2\pi R_i \Delta z$

Boat support (mol/s): $kC_{AA}\alpha 2\pi R_i \Delta z$

Total: $-R''_{AA}\Delta z = -r''_{AA}(2\pi R_i + \alpha 2\pi R_i + \eta a \pi R_i^2)\Delta z$

$$-R''_{AA} = kC_{AA}(2\pi R_i + \alpha 2\pi R_i + \eta a \pi R_i^2) \quad (E11-5.3)$$

Radial concentration profile

3. **Concentration profile and effectiveness factor:** From Example 11-4 we derived the radial concentration profile between the wafers as

$$\frac{C_A}{C_{AA}} = \frac{I_0(\phi_1 \lambda)}{I_0(\phi_1)} \quad (E11-4.9)$$

the corresponding effectiveness factor was

$$\eta = \frac{2I_1(\phi_1)}{\phi_1 I_0(\phi_1)} \quad (E11-4.12)$$

4. **Concentration profile in the annular region:** Combining equations (E11-5.2) and (E11-5.3) yields

$$- \frac{dF_{Az}}{dz} = kC_{AA}[(1 + \alpha)2\pi R_i + \eta a \pi R_i^2] \quad (E11-5.4)$$

Writing F_{Az} and C_{AA} in terms of conversion we have

$$F_{Az} = F_{A0}(1 - X)$$

$$C_{AA} = \frac{C_{A0}(1 - X)}{(1 + \epsilon X)} \quad (E11-5.5)$$

Mole balance on the reacting gas

Axial concentration profile

Combining equations (E11-5.4) and (E11-5.5)

$$F_{A0} \frac{dX}{dz} = kC_{A0}[(1 + \alpha)2\pi R_i + \eta a \pi R_i^2] (1 - X) \quad \epsilon = \gamma_{A0}\delta = 1 \cdot (1 - 1 - 0) = 0$$

Collecting terms we can obtain an expression involving number, Da.

$$\frac{dX}{dz} = \frac{Da}{L} (1 - X)$$

where

$$Da = \frac{\text{Rate of consumption of A by reactor}}{\text{Rate of transport of A by convection}}$$

and $Da = \frac{kC_{A0}[(1 + \alpha)2\pi R_i + \eta a \pi R_i^2]L}{F_{A0}}$

Solving for conversion as a function of distance along reactor yields

$$X = 1 - \exp(-Da z/L)$$

or, in terms of concentration

$$C_{AA}/C_{A0} = \exp[-Da(z/L)]$$

The deposition rate as a function of r and z can follow. The deposition rate at a location r and z is

$$-r''_{Aw}(r, z) = kC_A(r, z)$$

First, using Equation (E11-4.9) to relate $C_A(r, z)$ and C_{AA}

$$-r''_{Aw}(r, z) = kC_A(r, z) = kC_{AA}(z) \frac{I_0(\phi_1 r/L)}{I_0(\phi_1)}$$

Next, we use Equation (E11-5.9) to determine the rate as a function of z down the reactor.

$$-r''_{Aw}(r, z) = kC_{A0} \frac{I_0(\phi_1 r/L)}{I_0(\phi_1)} \exp[-Da(z/L)]$$

The thickness, T , of the deposit is obtained by integrating with respect to time, i.e.,

$$dT = 2(-r''_{Aw}) dt$$

where ρ = molar density of the material deposited, gmole/cm³. The two accounts for deposition on both sides of the wafer. Integrating, we obtain

$$T(r, z) = \frac{-r_{Aw}^n(r, z)}{\rho} 2t \quad (\text{E11-5.12})$$

The reactant concentration profile and deposition thickness along the length of the reactor is shown schematically below in Figure E11-5.3 for the case of small values of the Thiele modulus ($\eta \approx 1$).



$$\frac{C_{AA}}{C_{A0}} = \frac{Y_{AA}}{Y_{A0}}$$

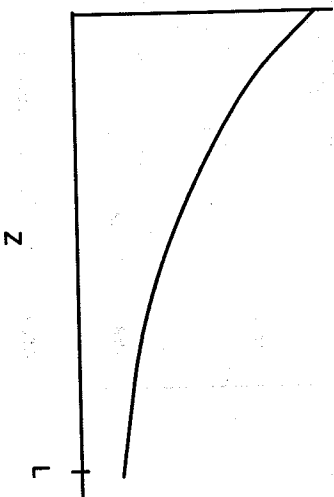


Figure E11-5.3 Axial concentration profile.

SUMMARY

1. The concentration profile for a first-order reaction occurring in a spherical catalyst pellet is

$$\frac{C_A}{C_{As}} = \frac{R}{r} \left[\frac{\sinh(\phi_1 r/R)}{\sinh \phi_1} \right] \quad (\text{S11-1})$$

where ϕ_1 is the Thiele modulus. For a first-order reaction

$$\phi_1^2 = \frac{k_p S_a R^2}{D_e} \quad (\text{S11-2})$$

2. The effectiveness factors are

$$\text{internal effectiveness} = \eta = \frac{\text{actual rate of reaction}}{\text{reaction rate if entire interior factor}}$$

overall effectiveness = $\Omega = \frac{\text{actual rate of reaction}}{\text{reaction rate if entire factor exposed to bulk concentration}}$

3. For large values of the Thiele modulus,

$$\eta = \left(\frac{2}{n+1} \right)^{1/2} \frac{3}{\phi_n}$$

4. For internal diffusion control, the true reaction order by

$$n_{\text{true}} = 2n_{\text{apparent}} - 1$$

The true and apparent activation energies

$$E_{\text{true}} = 2E_{\text{app}}$$

5. The Weisz-Prater parameter is

$$C_{WP} = \frac{-r_A^{\prime}(\text{observed})}{D_e C_{As}}$$

The Weisz-Prater criterion dictates that

If $C_{WP} \ll 1$ no internal diffusion limit

If $C_{WP} \gg 1$ internal diffusion limit

6. The deposition thickness on wafers in a boiler down the reactor and at a wafer radius r

$$T = \frac{k C_{A0} I_0(\phi_1 r/R_w)}{\rho I_0(\phi_1)} \left[\exp(-1) \right]$$

QUESTIONS AND PROBLEMS

P11-1c Make up an original problem using the concept of η (your instructor will specify the section).

P11-2c Review the example problems in this chapter. Use a software package such as POLYMATH for sensitivity analysis. Perhaps reconsider the LP Example 11-4. What are the effects of wafer space deposition support area, α , and gas velocity in shape of the wafer? If there were a significant length of the reactor, how would the wafer shape the effects of increasing or decreasing the temperature on the shape of the wafer?