# Lecture 1 Clean room, wafer cleaning, and Thermal oxidation

#### • Clean room environment

- 1. Contaminates effect on micro fabrication:
  - a. Chemical sensitivity: Resistivity of silicon to dopants
  - b. Physical sensitivity: defect from otpical image process, and short circuit from particular contaminates
  - c. Yield Loss
    - ⇒ need special cares on process environment, water, and chemicals used.

#### 2. Particles control:

- a. Ordinary room contains around 10<sup>7</sup> particles/ft<sup>3</sup>
- b. The gravitational settling velocity of particles in air:

Settling velocity u<sub>s</sub> can be expressed by Stokes' law:

$$u_s = \frac{\rho_p g d_p^2}{18\mu}$$

 $\rho_p$ =particle density,  $\mu$ =viscosity of surrounding fluid,  $d_p$ =particle diameter.

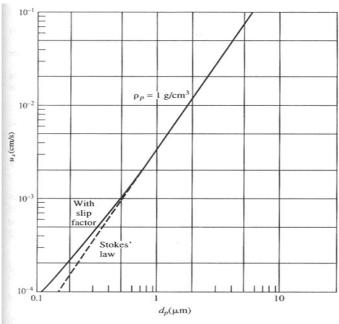


FIGURE 4-1
Gravitational settling velocity of particles in air at 1 atm.

c. Particles smaller than 0.05 µm is not significant in fabrication process. Because of the low settling speed in minuscule, they form airbone flying in the air and not easily to be deposited on wafers. Particle size above 0.5 µm is very important.

#### 3. Clean room

- a. Class x clean room has fewer than X particles/ft³ of size above 0.5 μm.
- b. Schematic of a clean room: (using <u>filtration methods</u> and <u>laminar flow</u> to filter out particles)

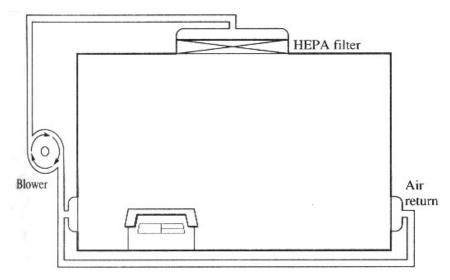
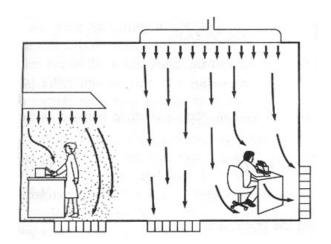
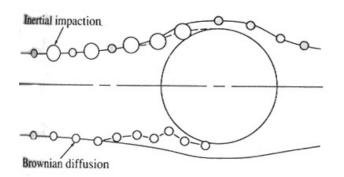


FIGURE 4-3 Schematic of a clean room.

c. The goal of laminar flow: prevent turbulence mixing, particles cross the boundary through <u>convective</u> and <u>diffusive fluxes</u>.



d. Tow mechanisms for filtration: <u>block</u> particle larger than the pore of filter, <u>stick</u> particles for smaller sizes (for HEPA (Hight efficiency Particular air) filter pore size of 5 µm fiber radius, some circumstance can have most popular particle size in 0.13 µm)



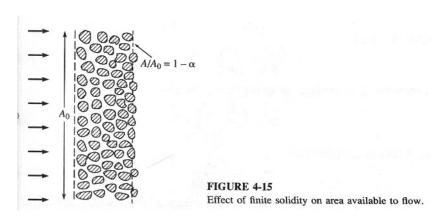
#### FIGURE 4-9

Filtration mechanisms of particles.
Inertialess particles follow a streamline.
Heavy particles can deviate from
streamlines and impact on the filter. A
particle may also have a sufficiently large
Brownian diffusion coefficient that it will
wander across streamlines and strike the
filter.

e. Pressure drop, Darcy's law:

$$\Delta P = k\mu U_0 Z$$

 $\mu\text{=air}$  viscosity, Z=thickness of the filter medium,  $U_0\text{=superficial}$  velocity (approach velocity to the filter.)



# Wafer cleaning process

The goal of wafer cleaning: to remove contamination from wafer before next process.

#### 1. Contamination classification

#### **Particulates:**

### (1) Particulate matter

(airborne bacteria, dust, abrasive particles: SiC, Al<sub>2</sub>O<sub>3</sub>, diamond power, lint from filter, human skin or clothing),

## (2) Traces of organic

(photoresist, Grease, Wax from cutting oil or physical handling, finger print, plasticizers from containers and wrapping materials)

## (3) Light metal ion

(Na, K... from etchant impurities)

## (4) Heavy metal impurities

(Ca, Co, Hg, Cu, Au, Fe, Ag, Ni...electrodeposition from etchant)

#### Films:

## (2) Solvent residues

(leave stain, causing poor resist adhesion and increasing undercutting during etching), fully rinsing and apply next rinsing step before drying out of the previous rinsing solvent is necessary

# (3) <u>Photoresist developer residues</u>

(overconcentration of dissolved photoresist, positive PR: react with carbon dioxide in air, negative PR: solvent evaporated), fully rinse and 2 step developing can help.

# (4) oil films

(from not filtered nitrogen, air or other gas lines)

# (5) silicone films

(causing resist wetting and adhesion problems, coming from hand cream, lubricants, cutting oils, human skin oils, detergents, usually give the wafer a hydrophobic surface resisting aqueous cleaning), using <u>atomizing</u> a layer of water on the surface to check the uniformity.

# (6) Metallic films

( from the ion in etchants and resist stripper, those ions may replace silicon or entrapped on wafers): good three steps rinse and a spry rinse can reduce the later, but not the former.

# (7) Water stains

(caused by the impurity of water, posing resist adhesion problem)

- (8) Native oxide (~ 50 Å) Using dilute HF to remove.
- 2. DI Water (deionized water, 18 Mohm-cm at 25° C, <0.25  $\mu$  m particles, <1.2 colonies of bacteria/mL) is desired throughout the fabrication process to reduce ion contimation.
- 3. Silicon wafer cleaning methods:

## Silicon wafer Standard cleaning:

For organic removal by Solvent, necessary before any process steps:

- 1. Immerse in acetone for 3 min
- **2.** Immerse in methyl alcohol (or IPA, iso-proponal alcohol) for 3 min
- 3. Wash in DI water for 3 min
- **4.** Nitrogen blow dry

# Silicon wafer **Piranha cleaning**:

For remove light-metal-ion contamination and Residual Organic( not good if the wafer already has metal structures):

- 1. Immerse in a (6:1:1) solution of  $H_2O$ -HCl- $H_2O_2$  (can be replaced by (Piranha bath:  $H_2O_2$ :  $H_2SO_4$  =1:10) for 10 min at temp of 75-80° C
- 2. Quench the solution under running DI water for 1 min.
- 3. Wash in DI water for 20 min

Note: to Remove hard baked PR without damage the metal structures already on the wafer, using organic stripper (for example, AZ 300T at 80°C for AZ resist removal)

# Silicon wafer **Pre-furnace cleaning** (RCA cleaning):

A. Organic removal by Solvent:

- 1. Immerse in boiling trichloroethylene (TCE) for 3 min (not use in school)
- 2. Immerse in boiling acetone for 3 min (room temp in the lab)
- 3. Immerse in boiling methyl alcohol for 3 min (room temp in the lab)
- 4. Wash in DI water for 3 min

## B. Removal of Residual Organic/Heavy metal impurities

- 1. Immerse in a (5:1:1) solution of  $H_2O-HH_4OH-H_2O_2$  at 75-80° C for 10 min. (RCA I)
- 2. Quench the solution under running DI water for 1 min.
- 3. Wash in DI water for 5 min

#### C. Hydrous Oxide removal:

- 1. Immerse in a (1:50) solution of HF-H<sub>2</sub>O for 40-60 sec
- 2. Wash in running DI water with agitation for 30 sec.

## D. Light-metal-ion contamination removal:

- 1. Immerse in a (6:1:1) solution of  $H_2O$ -HCl- $H_2O_2$  (can be replaced by (Piranha bath:  $H_2O_2$ :  $H_2SO_4$  =1:10) for 10 min at temp of 75-80° C
- 2. Quench the solution under running DI water for 1 min.
- 3. Wash in DI water for 20 min

Note: chloride ions is easier to be removed for weak van der waals forces than fluoride ions.

# Silicon wafer dry cleaning:

- a. To avoid residue left behind wet chemical etching, and chemical disposal problem
- b. High temp burn off: in oxidation tube at 900-1200°C
- c. Low temp ashing: Oxygen plasma—descum after development. (however, dry oxygen plasma may also deposit polymer type residue onto wafers surface, depending on the chamber cleanness.)

- d. Dry etching plasma: CF<sub>4</sub>:O<sub>2</sub>
- e. To cleanly remove negative resist: 650°C heat treatment (ash) in air.
- f. Resist film wet stripper: Piranha ( $H_2SO_4$ : $H_2O_2$ =10:1), or  $HNH_4$ : $H_2O_2$ : $H_2O$ =1:1:5.

## • Thermal oxidation

- 1. SiO<sub>2</sub> melting point: 1732 C, growth 1 μm SiO<sub>2</sub> Consume 0.44 μm Silicon. It is a high quality insulator and good barrier material during impurity diffusion.
- 2. Diffusivity:

$$D = D_0 e^{-E_A/kT}$$

 $D_0$ =Diffusion constant,  $E_A$ =activation energy of the diffusion species in eV/Molecule. K=Boltzmann's constant,  $8.62e^{-5}$  eV/K, T= temperature.

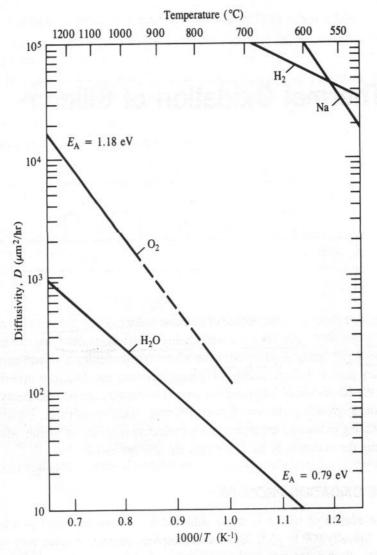


Fig. 3.1 Diffusivities of hydrogen, oxygen, sodium, and water vapor in silicon glass. Copyright John Wiley & Sons, Inc. Reprinted with permission from ref. [2].

#### 3. Oxide formation:

Dry Oxide:

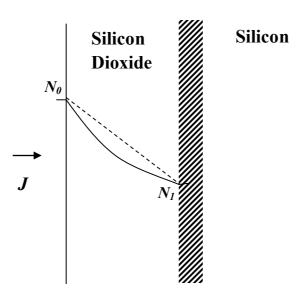
$$Si+O_2 \longrightarrow SiO_2$$

Wet Oxide:

$$Si+2H_2O \longrightarrow SiO_2+2H_2$$

- Note: 1. wet thermal oxide is faster in long time oxide growth, but slower in short times (<160 A), because H<sub>2</sub>O's diffusion rate is slower than O<sub>2</sub>. However, water can form hydroxyl groups within the Si-O-Si network, which opens the solid structure and permits much more rapid diffusion of the oxidants H<sub>2</sub>O and O<sub>2</sub> the reactive interface.
  - 2. Wet thermal oxide is more porous than dry oxide, so is not as good as dry oxide for gate oxide.

### 4. Kinetics of Oxide Growth:



# **Diffusion process:**

$$J = -D\frac{\partial N}{\partial x} \cong \frac{D(N_0 - N_1)}{x} \tag{2-1}$$

where x is thickness of the oxide at a given time. This is *Henry's law*.

## Reaction process:

$$J = kN_1 \tag{2-2}$$

Where k is the interfacial reaction rate constant. Combining Eqs. (2-1) and (2-2) gives:

$$J \cong \frac{DN_0}{x + D/k} \tag{2-3}$$

The rate of change of the oxide layer thickness is given by:

$$\frac{dx}{dt} = \frac{J}{n} = \frac{DN_0/n}{x + D/k} \tag{2-4}$$

Were n is the number of molecules of the oxidizing impurity that are incorporated into unit volume of the oxide. Solving this equation to the boundary condition that x=0 at t=0, gives

$$x^2 + \frac{2D}{k}x = \frac{2DN_0}{n}t\tag{2-5}$$

So that

$$x = \frac{D}{k} \left[ \left( 1 + \frac{2N_0 k^2 t}{Dn} \right)^{1/2} - 1 \right]$$
 (2-6)

Define A=2D/k,  $B=2DN_0/n$ , when t is small (<<1, thickness <500), equation (2-6) can be reduced to:

$$x = \frac{B}{A}t$$
 (reaction limit) (2-7)

and for large t:

$$x = \sqrt{Bt}$$
 (diffusion limit) (2-8)

# 5. Thickness vs. Time and Temp:

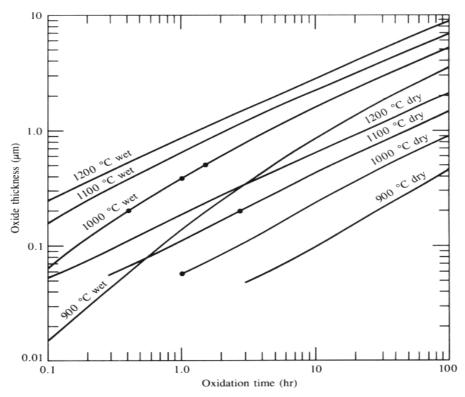


Fig. 3.6 Wet and dry silicon dioxide growth for  $\langle 100 \rangle$  silicon calculated using the data from Table 3.1.

# 7. Selective Oxidation:

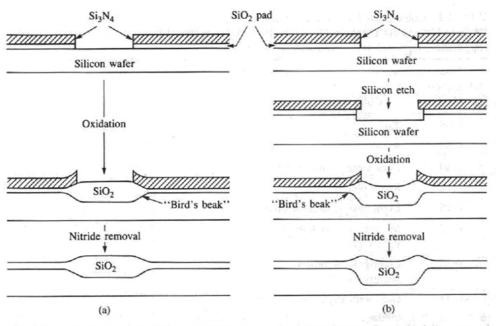


Fig. 3.12 Cross section depicting process sequence for (a) semirecessed and (b) fully recessed oxidations of silicon.

Table 3.2 Color Chart for Thermally Grown SiO<sub>2</sub> Films Observed Perpendicularly Under Daylight Fluorescent Lighting. Copyright 1964 by International Business Machines Corporation; reprinted with permission from ref. [9].

Film Thickness (µm)	Color and Comments	Film Thickness (µm)	Color and Comments
0.05	Tan	0.54	Yellow green
0.07	Brown	0.56	Green yellow
0.10	Dark violet to red violet	0.57	Yellow to "yellowish" (not yellow but is in the position where yellow is to be expected; at times appears to be light creamy gray or metallic)
0.12	Royal blue		
0.15	Light blue to metallic blue		
0.17	Metallic to very light yellow green		
0.20	Light gold or yellow; slightly metallic	0.58	Light orange or yellow to pink borderline
0.22	Gold with slight yellow orange	0.60	Carnation pink
		0.63	Violet red
0.25	Orange to melon	0.68	"Bluish" (not blue but borderline between violet and blue green; appears more like a mixture between violet red and blue
0.27	Red violet		
0.30	Blue to violet blue		
0.31	Blue		
0.32	Blue to blue green	0.72	green and looks grayish)  Blue green to green (quite broad)
0.34	Light green	0.72	
0.35	Green to yellow green	0.77	"Yellowish"
0.36	Yellow green	0.80	Orange (rather broad for
0.37	Green yellow		orange)
0.39	Yellow	0.82	Salmon
0.41	Light orange	0.85	Dull, light red violet
0.42	Carnation pink	0.86	Violet
0.44	Violet red	0.87	Blue violet
0.46	Red violet	0.89	Blue
0.47	Violet	0.92	Blue green
0.48	Blue violet	0.95	Dull yellow green
0.49	Blue	0.97	Yellow to "yellowish"
0.50	Blue green	0.99	Orange
0.52	Green (broad)	1.00	Carnation pink

#### **Reference:**

- 1. Introduction to Microelectronic Fabrication, Richard C. Jaeger, Addison-Wesley Publishing Company, Inc, 1998.
- 2. Integrated circuit fabrication technology, David J. Elliott, second edition, McGRAW-HILL, 1989.
- 3. Process engineering analysis in semiconductor device fabrication, Stanley Middleman, and Arthur K. Hochberg, McGRAW-HILL, international editions,

Advance Micro System Fabrication and Lab Lec1, Fall/2016,

NTHU ESS5810 F. G. Tseng p13 1993.