

### EE C245 - ME C218 Introduction to MEMS Design Fall 2009

Prof. Clark T.-C. Nguyen

Dept. of Electrical Engineering & Computer Sciences
University of California at Berkeley
Berkeley, CA 94720

Lecture Module 3: Oxidation & Film Deposition

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#### Lecture Outline

- Reading: Senturia, Chpt. 3; Jaeger, Chpt. 2, 3, 6
  - SExample MEMS fabrication processes
  - **♥** Oxidation
  - **♥ Film Deposition** 
    - **◆** Evaporation
    - ◆ Sputter deposition
    - ◆ Chemical vapor deposition (CVD)
    - ◆ Plasma enhanced chemical vapor deposition (PECVD)
    - **←** Epitaxy
    - ◆ Atomic layer deposition (ALD)
    - Electroplating

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#### **MEMS** Fabrication

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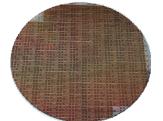
#### Making Mechanical Devices

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- How best does one make a mechanical product?
- Assembly line production?
  - \$ Pick and place parts
  - Used for many macroscopic mechanical products
  - ♥ Robotic automation greatly reduces cost
- <u>Problem</u>: difficult to do this with MEMS-scale parts (but not impossible, as we'll soon see ...)
- <u>Solution</u>: borrow from integrated circuit (IC) transistor technology
  - Use monolithic wafer-level fabrication methods
  - Harness IC's batch methods, where multiple devices are achieved all at once



**Automobile Assembly Line** 



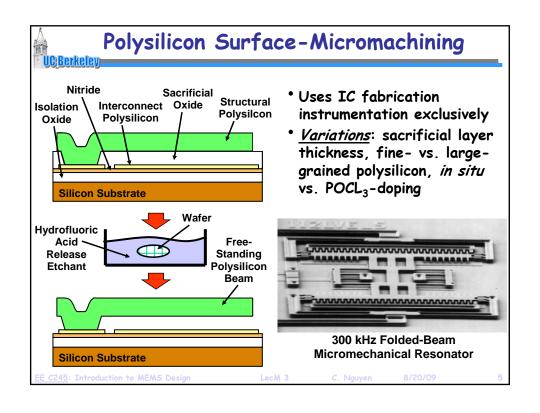
**CMOS Integrated Circuit Wafer** 

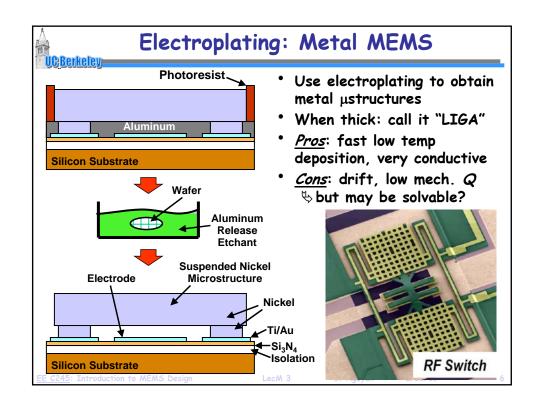
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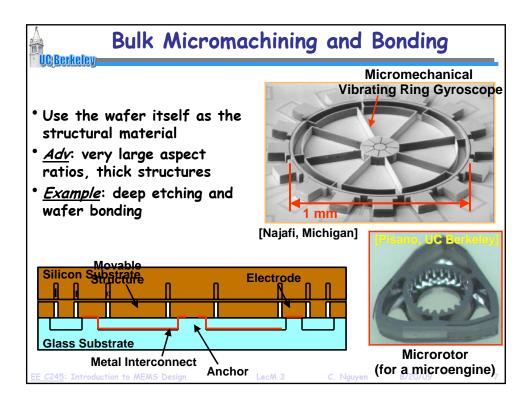
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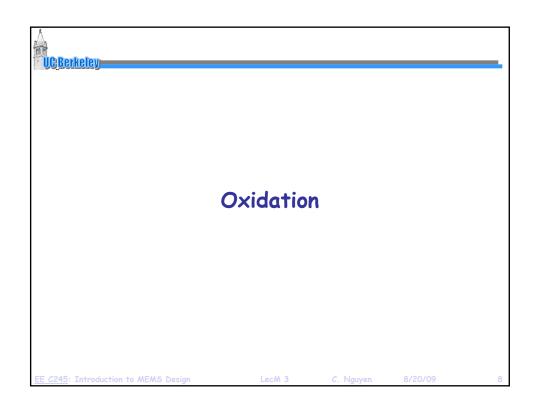
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#### Thermal Oxidation of Silicon

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- \* Achieved by heating the silicon wafer to a high temperature (~900°C to 1200°C) in an atmosphere containing pure oxygen or water vapor
- Enabling reactions:

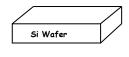
For dry oxygen:

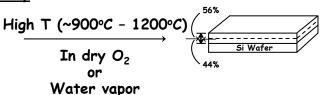
For water vapor:

Si + 
$$O_2 \rightarrow SiO_2$$

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

#### Schematically:





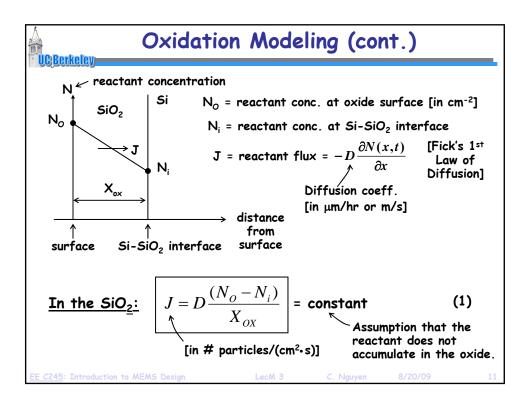
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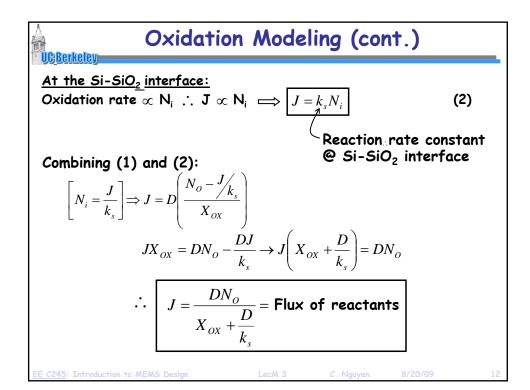
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silicon-oxide interface

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Oxidation Modeling **UC**Berkeley (1) <u>Initially</u>: (no oxide @ surface) gas stream ⋄ Growth rate determined by Si reaction rate @ the surface (2) As oxide builds up: gas stream Reactant must diffuse to Si oxide surface where the oxidation Si reaction takes place ⋄ Growth rate governed more by rate of diffusion to the





#### Oxidation Modeling (cont.)

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Find an expression for  $X_{OX}(t)$ : oxidizing flux

Rate of change of oxide layer thickness w/time 
$$= \frac{dX_{OX}}{dt} = \frac{J}{M} = \frac{DN_O/M}{X_{OX} + D/k_s}$$
 (3)

# of molecules of oxidizing species incorporated into a unit volume of oxide  $= 2.2 \times 10^{22} cm^{-3} \text{ for } O_2$   $= 4.4 \times 10^{22} cm^{-3} \text{ for } H_2O$ 

Solve (3) for  $X_{OX}(t)$ : [Initial condition  $X_{OX}(t=0)=X_i$ ]

$$\frac{dX_{OX}}{dt} = \frac{DN_O/M}{X_{OX} + D/k_s} \quad \text{Terms } \int_{X_s}^{X_{OX}} \left(X_{OX} + \frac{D}{k_s}\right) dX_{OX} = \int_0^t \frac{DN_O}{M} dt$$

#### Oxide Thickness Versus Time

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Result:

additional time required to grow 
$$X_i$$
 time required to grow  $X_i$  to go from  $X_i \to X_{OX}$  [ $X_i = \text{initial oxide thickness}$ ]
$$X_{OX}(t) = \frac{A}{2} \left\{ \left[ 1 + \frac{4B}{A^2}(t+\tau) \right]^{\frac{1}{2}} - 1 \right\}$$

$$X_{OX}(t) = \frac{A}{2} \left[ \left[ 1 + \frac{4B}{A^2} (t + \tau) \right]^{\frac{1}{2}} - 1 \right]$$

where  $A = \frac{2D}{k_s}$   $\tau = \frac{X_i^2}{B} + \frac{X_i}{(B/A)}$ 

$$B = \frac{2DN_O}{M} \qquad D = D_O \exp\left(-\frac{E_A}{kT}\right)$$

i.e., D governed by an Arrhenius relationship → temperature dependent

#### Oxidation Modeling (cont.)

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For shorter times:

$$\left[ (t+\tau) << \frac{A^2}{4B} \right] \Rightarrow X_{OX}(t) = \left( \frac{B}{A} \right) (t+\tau) \Rightarrow \text{oxide growth limited by reaction at the Si-SiO}_2 \text{ interface}$$

Taylor expansion (first term after 1's cancel)

 $^{
u}$  linear growth rate constant

For long oxidation times: oxide growth diffusion-limited

$$\left[ \left( t + \tau \right) >> \frac{A^2}{4B} \right] \Rightarrow X_{OX} \left( t \right) = \sqrt{B(t + \tau)} \approx \sqrt{Bt}$$

$$t >> \tau$$
Parabolic rate constant

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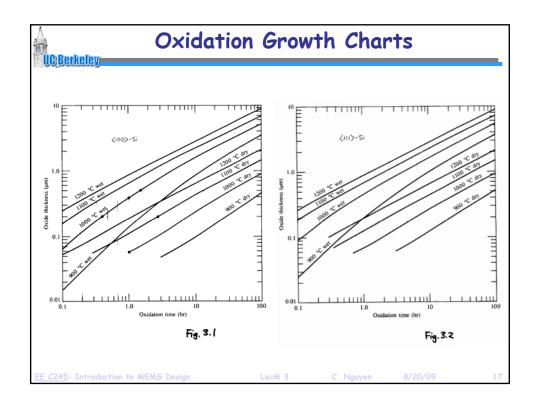
#### Oxidation Rate Constants

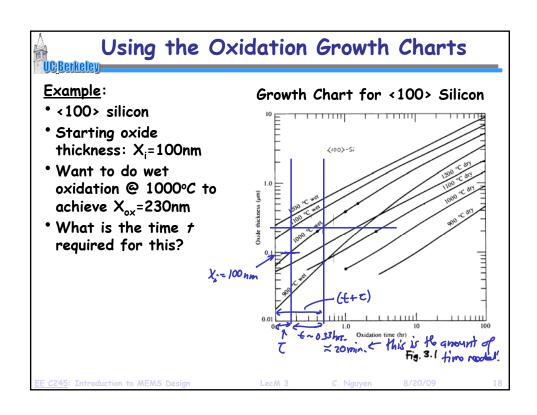
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**Table 6–2** Rate constants describing (111) silicon oxidation kinetics at 1 Atm total pressure. For the corresponding values for (100) silicon, all  $C_2$  values should be divided by 1.68.

Ambient	<b>B</b>	B/A
Dry O <sub>2</sub>	$C_1 = 7.72 \times 10^2 \mu\text{m}^2 \text{hr}^{-1}$	$C_2 = 6.23 \times 10^6 \mu \text{m hr}^{-1}$
	$E_{\rm I}=1.23~{\rm eV}$	$E_2 = 2.0 \mathrm{eV}$
Wet O <sub>2</sub>	$C_1 = 2.14 \times 10^2 \mu\text{m}^2 \text{hr}^{-1}$	$C_2 = 8.95 \times 10^7 \mathrm{\mu m}\mathrm{hr}^{-1}$
	$E_1 = 0.71 \text{ eV}$	$E_2 = 2.05 \text{ eV}$
H <sub>2</sub> O	$C_1 = 3.86 \times 10^2 \mu\text{m}^2 \text{hr}^{-1}$	$C_2 = 1.63 \times 10^8 \mathrm{\mu m}\mathrm{hr}^{-1}$
	$E_1 = 0.78 \mathrm{eV}$	$E_2 = 2.05 \mathrm{eV}$

 Above theory is great ... but usually, the equations are not used in practice, since measured data is available
 Rather, oxidation growth charts are used





#### Factors Affecting Oxidation

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- In summary, oxide thickness is dependent upon:
  - 1. Time of oxidation
  - 2. Temperature of oxidation
  - 3. Partial pressure of oxidizing species ( $\propto N_a$ )
- Also dependent on:
  - 4. Reactant type:

Dry O2

Water vapor ⇒ faster oxidation, since water has a higher solubility (i.e., D) in SiO<sub>2</sub> than O<sub>2</sub>

- 5. Crystal orientation:
  - <111> 

    faster, because there are more bonds available at the Si-surface
  - <100> ← fewer interface traps; smaller # of unsatisfied Si-bonds at the Si-SiO₂ interface

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#### Factors Affecting Oxidation

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- 6. Impurity doping:
  - P: increases linear rate const.

    no affect on parabolic rate constant
    faster initial growth 

    surface reaction rate limited
  - B: no effect on linear rate const.
    increases parabolic rate const.
    faster growth over an initial oxide → diffusion faster



#### Thin Film Deposition

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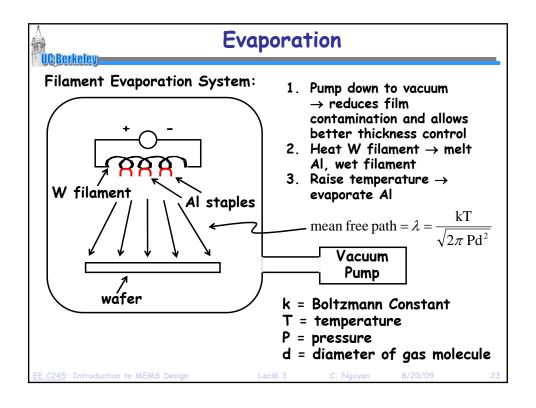
#### Thin Film Deposition

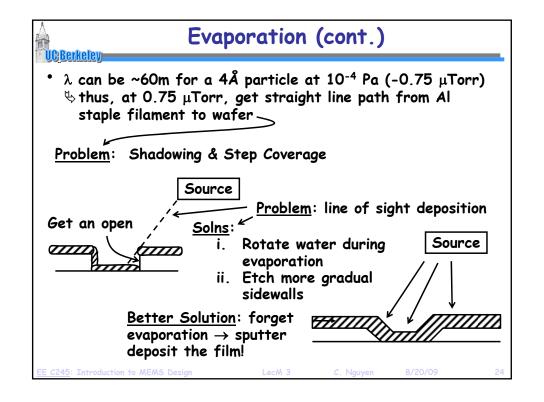
- Methods for film deposition:
  - **♥** Evaporation
  - ♦ Sputter deposition
  - \$ Chemical vapor deposition (CVD)
  - ♦ Plasma enhanced chemical vapor deposition (PECVD)
  - **⇔** Epitaxy

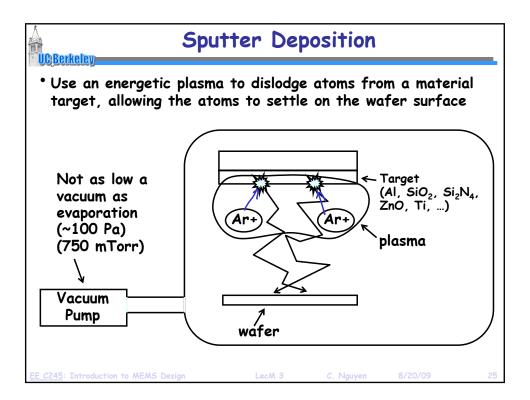
  - ♦ Atomic layer deposition (ALD)

#### **Evaporation:**

- Heat a metal (Al, Au) to the point of vaporization
- Evaporate to form a thin film covering the surface of the Si wafer
- Done under vacuum for better control of film composition







#### Sputter Deposition Process

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- <u>Step-by-step procedure</u>:
  - 1. Pump down to vacuum

(~100 Pa) 
$$\rightarrow$$
 1 Pa = 9.8×10<sup>-6</sup> atm  $\left(\frac{760 \text{ Torr}}{\text{atm}}\right)$  = 0.0075012 Torr

- 2. Flow gas (e.g., Ar)
- 3. Fire up plasma (create Ar+ ions)  $\rightarrow$  apply dc-bias (or RF for non-conductive targets)
- 4. Ar+ ions bombard target (dislodge atoms)
- 5. Atoms make their way to the wafer in a more random fashion, since at this higher pressure,  $\lambda$  ~60 $\mu$ m for a 4Å particle; plus, the target is much bigger
- Result: better step coverage!

#### Problems With Sputtering

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- 1. Get some Ar in the film
- 2. Substrate can heat up
  - □ up to ~350°C, causing nonuniformity across the wafer
  - but it still is more uniform than evaporation!
- 3. Stress can be controlled by changing parameters (e.g., flow rate, plasma power) from pass to pass, but repeatability is an issue

Solution: use Chemical Vapor Deposition (CVD)

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#### Chemical Vapor Deposition (CVD)

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- Even better conformity than sputtering
- Form thin films on the surface of the substrate by thermal decomposition and/or reaction of gaseous compounds
  - Desired material is deposited directly from the gas phase onto the surface of the substrate
  - $\diamondsuit$  Can be performed at pressures for which  $\lambda$  (i.e., the mean free path) for gas molecules is small
  - \$ This, combined with relatively high temperature leads to

Excellent Conformal
Step Coverage!

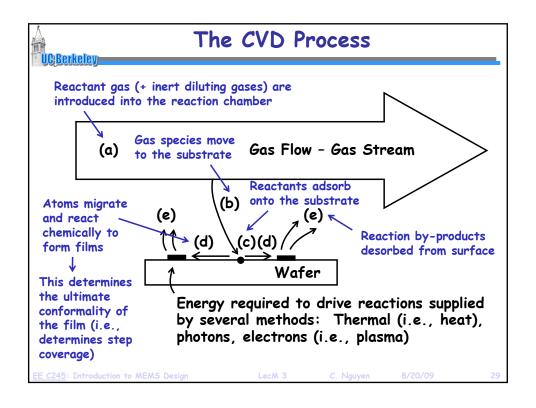
➡ Types of films: polysilicon, SiO<sub>2</sub>, silicon nitride, SiGe, Tungsten (W), Molybdenum (M), Tantalum (Ta), Titanium (Ti), ...

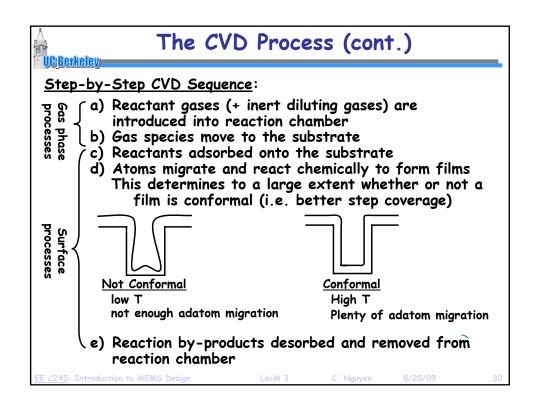
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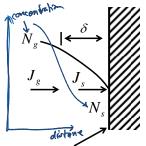






#### CVD Modeling

#### Simplified Schematic:



 $N_a$  = conc. of reactant molecules in the gas stream

 $N_s$  = conc. of reactant molecules at the surface

 $J_s$  = flux of gas molecules at the surface

 $J_a = flux$  of molecules diffusing in from the gas stream

surface

Effective diffusion const. for the gas molecule

$$J_s = k_s N_s$$
 [ $k_s$  = surface reaction rate const.]

$$J_{g} = \left( \frac{\overline{D}_{g}}{\delta} \right) \! \left( \! N_{g} - N_{s} \right) = h_{g} \! \left( \! N_{g} - N_{s} \right) \quad \text{Vapor phase mass-transfer coefficient}$$

Governing Equations:

CVD Modeling (cont.)

$$\underbrace{\left[J_s=J_g=J\right], \left[N_s=\frac{J_s}{k_s}\right]}_{ \text{Otherwise reactants will}} J=h_g \bigg(N_g-\frac{J}{k_s}\bigg)=h_g N_g-\frac{h_g J}{k_s}$$
 build up somewhere!

$$J\left(1+\frac{h_g}{k_s}\right) = h_g N_g \rightarrow \boxed{J = \frac{k_s h_g}{k_s + h_g} N_g = \left(\frac{k_s}{h_g}\right) N_g}$$

growth rate = 
$$\frac{\text{flux}}{\text{# molecules incorporated/unit volume}} = \frac{J}{N}$$

$$= \frac{J}{N} = \sqrt{\frac{k_s h_g}{k_s + h_g} \frac{N_g}{N}} = (k_s || h_g) \frac{N_g}{N} = \text{growth rate}$$

#### CVD Modeling (cont.)

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\* <u>Case</u>:  $k_s >> h_g$   $\stackrel{\mbox{$\mbox{$\mbox{$$$}}$}}{\mbox{$\mbox{$$$}$}}$  surface reaction rate >> mass transfer rate

growth rate = 
$$h_g \frac{N_g}{N}$$
 (mass-transfer-limited)

• <u>Case</u>:  $h_g >> k_s$   $\$  mass transfer rate >> surface reaction rate

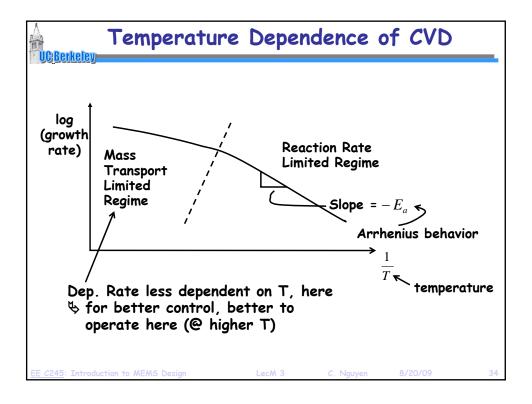
growth rate = 
$$k_s \frac{N_g}{N}$$
 (surface-reaction-limited)  $\sim R_o^{-E_a/kT}$  (Arrhenius character)

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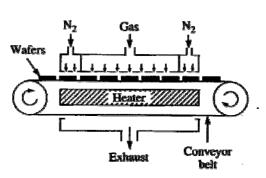
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#### Atmospheric Pressure Reactor (APCVD)

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- Once used for silicon dioxide passivation in integrated circuits
- Substrates fed continuously
- Large diameter wafers
- Need high gas flow rates
- Mass transport-limited regime (high pressure, so tougher for gas to get to the wafer surface)

- Problems/Issues:
  - ♥ Wafers lay flat, and thus, incorporate foreign particles
  - ♦ Poor step coverage

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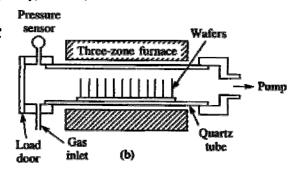
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#### Low Pressure Reactor (LPCVD)

 Many films available: polysilicon, SiGe, Si<sub>3</sub>N<sub>4</sub>, SiO<sub>2</sub>, phosphosilicate glass (PSG), BPSG, W

- Temp.:  $300 \rightarrow 1150$ °C
- Press.:  $30 \rightarrow 250 \text{ Pa}$ (200mTorr  $\rightarrow$  2Torr)
- Reaction rate limited; reduced pressure gives gas molecular high diffusivity; can supply reactants very fast!
- Can handle several hundred wafers at a time
- Excellent uniformity



- <u>Problems</u>:
  - \$Low dep. rate (compared to atm.)
  - ♦ Higher T (than atmospheric)
  - ♦ In hot wall reactors, get deposition on tube walls (must clean)

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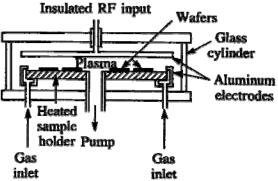
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#### Plasma-Enhanced CVD Reactor (PECVD)

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- RF-induced glow discharge + thermal energy to drive reactions → allows lower temperature deposition with decent conformability
- Still low pressure



- Problems:
  - ♥ Pin-holes
  - ♦ Non-stoichiometric films
  - $\$  Incorporation of  $H_2$ ,  $N_2$ ,  $O_2$  contaminants in film; can lead to outgassing or bubbling in later steps

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#### Polysilicon CVD

#### **Polysilicon Deposition:**

Fairly high temperature 
$$\rightarrow$$
 conformal 600°C SiH<sub>4</sub> $\longrightarrow$  Si + 2H<sub>2</sub> (thermal decomposition of silane) (conformal  $\longrightarrow$  high T) LPCVD (25 to 150 Pa)  $\rightarrow$  100-200Å/min

- In situ doping of polysilicon:
  - ⋄n-type: add PH₃ (phosphine) or Arsine gases (but greatly reduces dep. rate)
  - ⇔p-type: add diborane gas (greatly increases dep. Rate)

#### Silicon Oxide CVD

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#### Silicon Dioxide Deposition:

- After metallization (e.g., over aluminum)
  - ♦ Temperature cannot exceed the Si-Al eutectic pt.: 577°C
  - ♦ Actually, need lower than this (<500°C) to prevent hillocks from growing on Al surfaces
  - ♦ Similar issues for copper (Cu) metallization
- Low temperature reactions:

LPCVD  
LTO  
Reactions
$$\begin{cases}
SiH_4 + O_2 \longrightarrow SiO_2 + 2H_2 \\
(silane) & 300-500°C
\end{cases}$$

$$4PH_3 + 5O_2 \longrightarrow 2P_2O_5 + 6H_2 \\
(phosphine) & 300-500°C
\end{cases}$$
Phosphosilicate glass (PSG)

 $^{ullet}$  Above reactions: not very conformal step coverage ightarrow need higher T for this

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#### Silicon Oxide CVD (cont.)

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- Phosphosilicate glass can be reflown
  - \$6-8 wt. % allows reflow @ 1000-1100°C
  - ♦ Very useful to achieve smoother topography
  - Lower concentration → won't reflow
  - $\forall$  Higher concentration  $\rightarrow$  corrodes AI if moisture is present
  - ♦ 5-15% P can be used as a diffusion source to dope Si
- Before metallization:
  - ♥ Can use higher temperature → better uniformity and step coverage

or ...

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#### Silicon Oxide CVD (cont.)

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$$Si(OC_2H_5)_4 \longrightarrow SiO_2 + by-products$$
 $650-750^{\circ}C$ 
(Tetraethylorthosilicate) (excellent uniformity & conformal step coverage)

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#### Silicon Nitride CVD

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#### Silicon Nitride Deposition:

- \* First, note that thermal growth is possible:
  - § Si in NH<sub>3</sub> @ 1000-1100℃
  - ♦ But very slow growth rate, thus, impractical
- LPCVD reactions:

Silane reaction: 
$$3SiH_4 + 4NH_3 \longrightarrow Si_3N_4 + 12H_2$$
 (Atm. Press.)

Dichlorosilane reaction:

$$700-800^{\circ}C$$

$$3SiCl_2H_2 + 4NH_3 \xrightarrow{(LPCVD)} Si_3N_4 + 6HCl + 6H_2$$

Increase and  $T = 835^{\circ}C \longrightarrow Si \text{ rich nitride} \longrightarrow low stress}$ 

<u>Problem:</u> Clobbers your pumps! Expensive to maintain!

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#### Silicon Nitride CVD (cont.)

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- Comments on LPCVD nitride films:
  - ⇔ Hydrogen rich: ~8% H₂
  - High internal tensile stresses: films >1000Å crack and peel due to excessive stress
  - & Can get 2μm films with Si-rich nitride
  - $\$  LPCVD gives high resistivity (10<sup>16</sup>  $\Omega$ -cm) and dielectric strength (10 MV/cm)

#### PECVD Nitride:

$$\begin{array}{c} \text{Nitrogen discharge} \\ \text{SiH}_4 + \text{N}_2 & \longrightarrow 2 \text{SiNH} + 3 \text{H}_2 \\ \text{or} \\ \text{SiH}_4 + \text{NH}_3 & \longrightarrow 3 \text{SiNH} + 3 \text{H}_3 \end{array} \end{array} \\ \begin{array}{c} \text{PECVD films:} \\ \text{$\forall$ Non-stoichiometric nitride} \\ \text{$\forall$ 20-25\% H}_2 \text{ content} \\ \text{$\forall$ Can control stress} \\ \text{$\forall$ (10^6 \Omega-cm) resistivity} \end{array}$$

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#### Metal CVD

#### **CVD Metal Deposition**:

<u>Tungston (W)</u> - deposited by thermal, plasma or optically-assisted decomposition

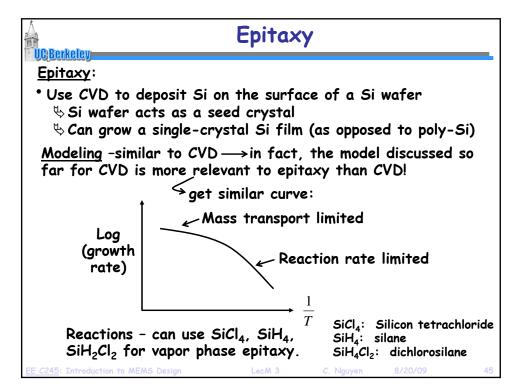
$$WF_6 \longrightarrow W + 3F_2$$
  
or via reaction with  $H_2$ :  
 $WF_6 + 3H_2 \longrightarrow W + 6HF$ 

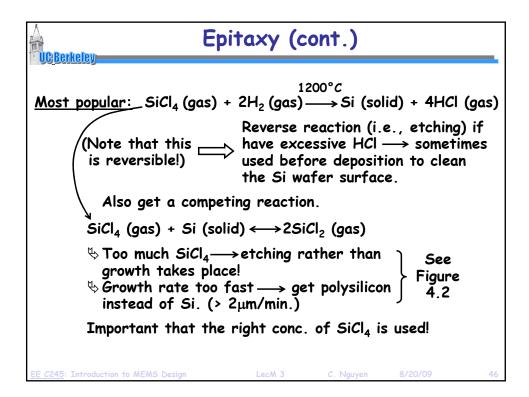
Other Metals - Molybdenum (Mo), Tantalum (Ta), and Titanium (Ti)

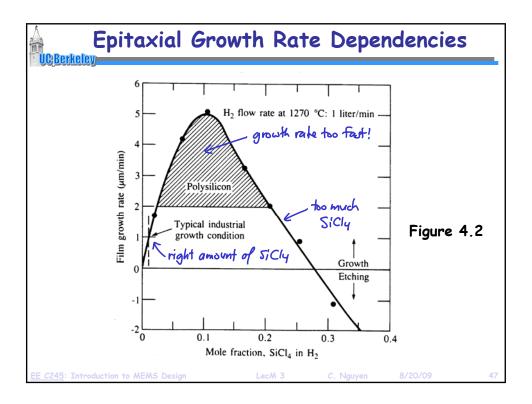
$$2MCl_5 + 5H_2 \longrightarrow 2M + 10HCl$$
, where M = Mo, Ta, or Ti

(Even Al can be CVD'ed with tri-isobutyl Al ... but other methods are better.)

(Cu is normally electroplated)







#### Epitaxy (cont.)

Alternative reaction: pyrolytic decomposition of silane:

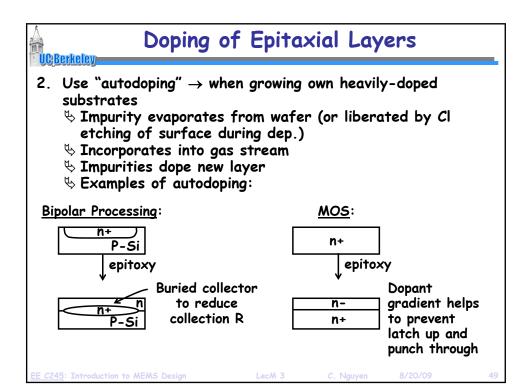
$$SiH_4 \xrightarrow{650^{\circ}C} Si + 2H_2$$
not reversible, low T, no HCl formation

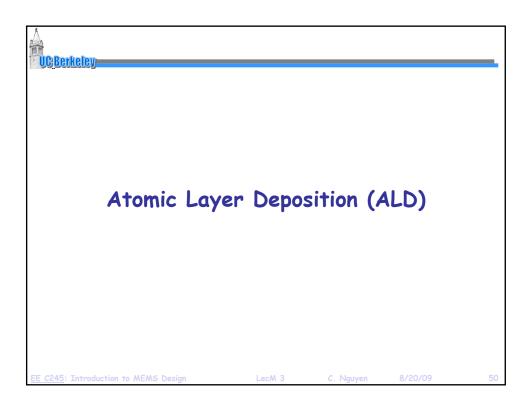
- however, requires careful control of the reaction to prevent formation of poly-Si
- 🔖 also, the presence of an oxidizing species

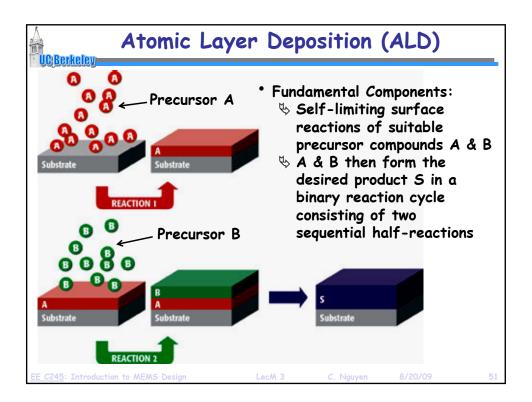
#### **Doping of Epitaxial Layers:**

causes silica formation

- 1. Just add impurities during growth: Arsine, diborane, Phosphine
  - Scientification Control resistivity by varying partial pressure of dopant species
    - i. Arsine, Phosphine  $\rightarrow$  slow down the growth rate
    - ii. Diborane → enhances growth rate







#### Atomic Layer Deposition (ALD)

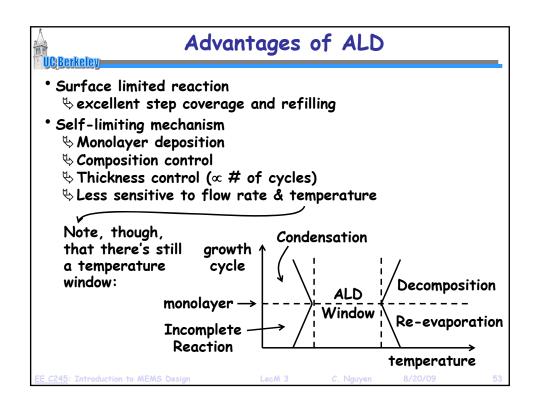
#### • Remarks:

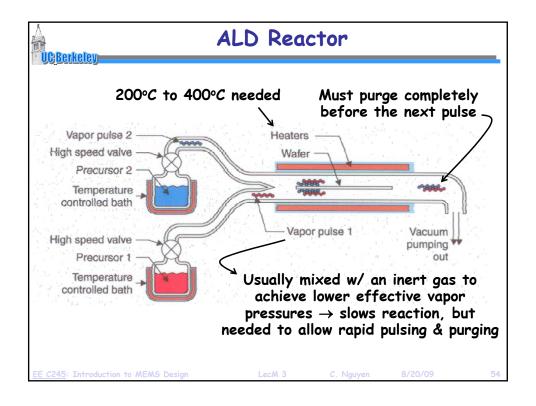
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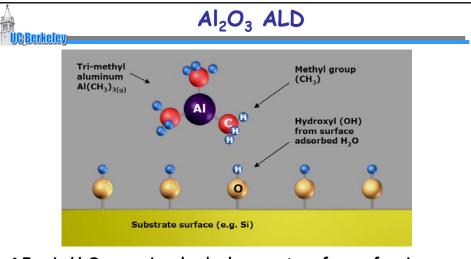
- ♥ Both half-reactions must be complete and self-limiting at the monolayer level
- ♦ The total film thickness d(tot) can be "digitally" controlled by the number of applied deposition cycles N(A/B):

$$d(tot) = d(mono) \cdot N(A/B)$$

- The reagents A & B in the half reactions are normally chemical reactions
  - → But they don't need to be
  - ◆ They can also represent a physical process, e.g., heating, irradiation, electrochemical conversion







- In air H<sub>2</sub>O vapor is adsorbed on most surfaces, forming a hydroxyl group
- With silicon this forms :Si-O-H (s)
- Place the substrate in the reactor
- Pulse Trimethyl Aluminum (TMA) into the reaction chamber

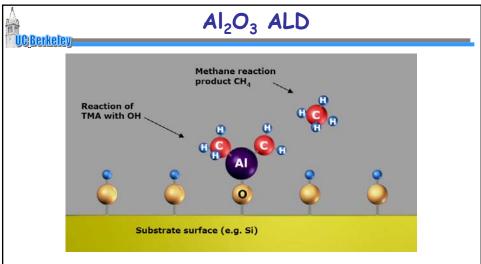
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\* Trimethyl Aluminum (TMA) reacts with the adsorbed hydroxyl groups, producing methane as the reaction product

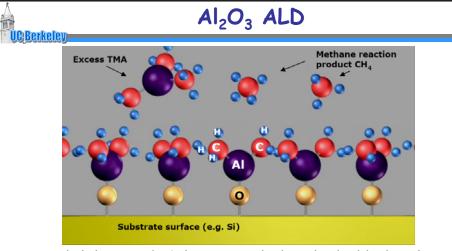
$$Al(CH_3)_3$$
 (g) + :Si-O-H (s)  $\rightarrow$  :Si-O-Al(CH<sub>3</sub>)<sub>2</sub> (s) + CH<sub>4</sub>

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- TrimethylAluminum (TMA) reacts with the adsorbed hydroxyl groups, until the surface is passivated
- TMA does not react with itself, so terminates the reaction to one layer
- This leads to the perfect uniformity of ALD.
- The excess TMA and methane reaction product is pumped away

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chamber.

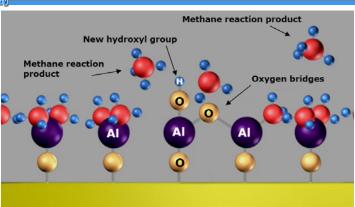
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# \* After the TMA and methane reaction product is pumped away, water vapor (H<sub>2</sub>O) is pulsed into the reaction

#### Al<sub>2</sub>O<sub>3</sub> ALD



- $^{\bullet}$   $H_2O$  reacts with the dangling methyl groups on the new surface forming aluminum-oxygen (Al-O) bridges and hydroxyl surface groups, waiting for a new TMA pulse
- \* Again methane is the reaction product

2 
$$H_2O_{(g)}$$
 + :Si-O-Al(CH<sub>3</sub>)<sub>2 (s)</sub>  $\longrightarrow$  :Si-O-Al(OH)<sub>2 (s)</sub> + 2  $CH_4$ 

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## Al<sub>2</sub>O<sub>3</sub> ALD

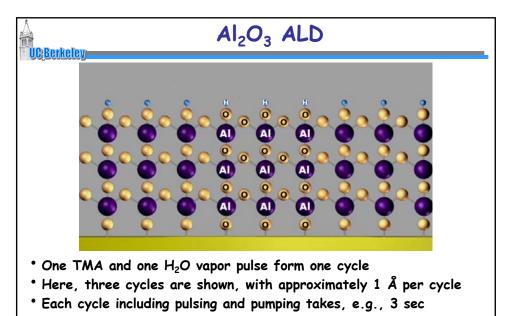
- The reaction product methane is pumped away
- Excess H<sub>2</sub>O vapor does not react with the hydroxyl surface groups
- \* Again, get perfect passivation to one atomic layer

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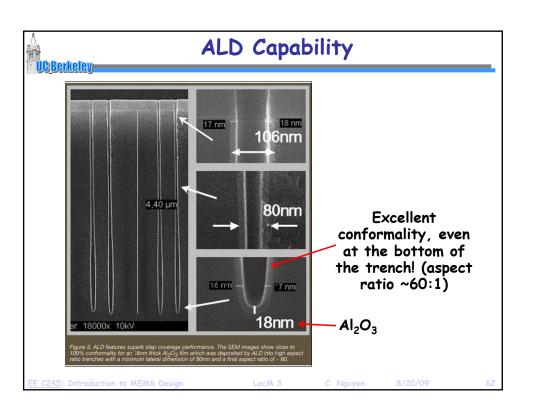
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 $\begin{array}{l} \text{Al(CH}_3)_3 \ _{(g)} \ + \ : \text{Si-O-H} \ _{(s)} \ \longrightarrow \ : \text{Si-O-Al(CH}_3)_2 \ _{(s)} \ + \ \text{CH}_4 \\ \text{2 H}_2O \ _{(g)} \ + \ : \text{Si-O-Al(CH}_3)_2 \ _{(s)} \ \longrightarrow \ : \text{Si-O-Al(OH)}_2 \ _{(s)} \ + \ \text{2 CH}_4 \end{array}$ 



ALD Versus CVD						
ALD	CVD					
Highly reactive precursors	Less reactive precursors					
Precursors react separately on the substrate	Precursors react at the same time on the substrate					
Precursors must not decompose at process temperature	Precursors can decompose at process temperature					
Uniformity ensured by the saturation mechanism	Uniformity requires uniform flux of reactant and temperature					
Thickness control by counting the number of reaction cycles	Thickness control by precise process control and monitoring					
Surplus precursor dosing acceptable	Precursor dosing important					
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ALD Versus Other Deposition Methods								
Method	ALD	MBE	CVD	Sputter	Evapor	PLD		
Thickness Uniformity	Good	Fair	Good	Good	Fair	Fair		
Film Density	Good	Good	Good	Good	Poor	Good		
Step Coverage	Good	Poor	Varies	Poor	Poor	Poor		
Inteface Quality	Good	Good	Varies	Poor	Good	Varies		
Number of Materials	Fair	Good	Poor	Good	Fair	Poor		
Low Temp. Deposition	Good	Good	Varies	Good	Good	Good		
Deposition Rate	Fair	Poor	Good	Good	Good	Good		
Industrial Apps.	Good	Fair	Good	Good	Good	Poor		

