



EE 143

Microfabrication Technology

Fall 2014

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Lecture Module 3: Film Deposition

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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
 - ↗ Electroplating
 - ↗ Atomic layer deposition (ALD)

Evaporation:

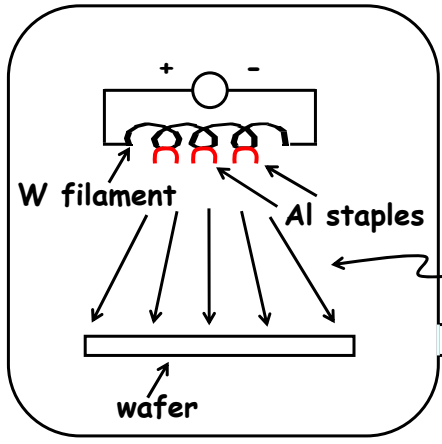
- Heat a metal (Al, Au) to the point of vaporization
- Evaporate to form a thin film covering the surface of the Si wafer
- Done under vacuum for better control of film composition

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Evaporation

Filament Evaporation System:



1. Pump down to vacuum
→ reduces film contamination and allows better thickness control
2. Heat W filament → melt Al, wet filament
3. Raise temperature → evaporate Al

mean free path = $\lambda = \frac{kT}{\sqrt{2\pi} Pd^2}$

Vacuum Pump

wafer

W filament

Al staples

k = Boltzmann Constant
 T = temperature
 P = pressure
 d = diameter of gas molecule

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Evaporation (cont.)

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- λ can be $\sim 60\text{m}$ for a 4\AA particle at 10^{-4} Pa ($-0.75\text{ }\mu\text{Torr}$)
 ↳ thus, at $0.75\text{ }\mu\text{Torr}$, get straight line path from Al staple filament to wafer

Problem: Shadowing & Step Coverage

Get an open

Source

Problem: line of sight deposition

Solns:

- Rotate wafer during evaporation
- Etch more gradual sidewalls

Better Solution: forget evaporation → sputter deposit the film!

Source

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Sputter Deposition

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- Use an energetic plasma to dislodge atoms from a material target, allowing the atoms to settle on the wafer surface

Not as low a vacuum as evaporation ($\sim 100\text{ Pa}$) (750 mTorr)

Vacuum Pump


Target (Al, SiO_2 , Si_3N_4 , ZnO, Ti, ...)

Ar⁺

plasma

wafer

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Sputter Deposition Process

- Step-by-step procedure:
 1. Pump down to vacuum
 $(\sim 100 \text{ Pa}) \rightarrow 1 \text{ Pa} = 9.8 \times 10^{-6} \text{ atm} \left(\frac{760 \text{ Torr}}{\text{atm}} \right) = 0.0075012 \text{ Torr}$
 \swarrow
750 mTorr
 2. Flow gas (e.g., Ar)
 3. Fire up plasma (create Ar⁺ ions) → 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, $\lambda \sim 60 \mu\text{m}$ for a 4 Å particle; plus, the target is much bigger
- Result: better step coverage!


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Problems With Sputtering

1. Get some Ar in the film
2. Substrate can heat up
 - ↳ up to $\sim 350^\circ\text{C}$, 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)**

- 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
 - ↳ Can be performed at pressures for which λ (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_2 , silicon nitride, SiGe, Tungsten (W), Molybdenum (M), Tantalum (Ta), Titanium (Ti), ...

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 **The CVD Process**

Reactant gas (+ inert diluting gases) are introduced into the reaction chamber

(a) Gas species move to the substrate **Gas Flow - Gas Stream**

(b) Reactants adsorb onto the substrate

(c) (d) **(e)** **Reaction by-products desorbed from surface**

Atoms migrate and react chemically to form films

Wafer

Energy required to drive reactions supplied by several methods: Thermal (i.e., heat), photons, electrons (i.e., plasma)

This determines the ultimate conformity of the film (i.e., determines step coverage)

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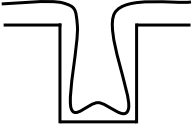
The CVD Process (cont.)

Step-by-Step CVD Sequence:

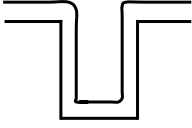
Gas phase processes

Surface processes

- a) Reactant gases (+ inert diluting gases) are introduced into reaction chamber
- b) Gas species move to the substrate
- c) Reactants adsorbed onto the substrate
- d) Atoms migrate and react chemically to form films
This determines to a large extent whether or not a film is conformal (i.e. better step coverage)



Not Conformal
low T
not enough adatom migration



Conformal
High T
Plenty of adatom migration

- e) Reaction by-products desorbed and removed from reaction chamber

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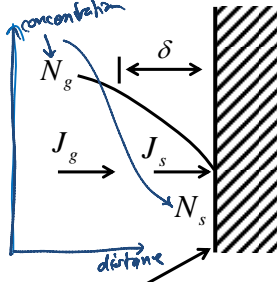
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CVD Modeling

Simplified Schematic:



N_g = 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_g = flux of molecules diffusing in from the gas stream

Governing Equations:

Effective diffusion const. for the gas molecule

$$J_s = k_s N_s \quad [k_s = \text{surface reaction rate const.}]$$

Vapor phase mass-transfer coefficient

$$J_g = \left(\frac{\bar{D}_g}{\delta} \right) (N_g - N_s) = h_g (N_g - N_s)$$


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 **CVD Modeling (cont.)**


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

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

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

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

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 **CVD Modeling (cont.)**

- Case: $k_s \gg h_g$
 \hookrightarrow surface reaction rate \gg mass transfer rate

$\text{growth rate} = h_g \frac{N_g}{N}$

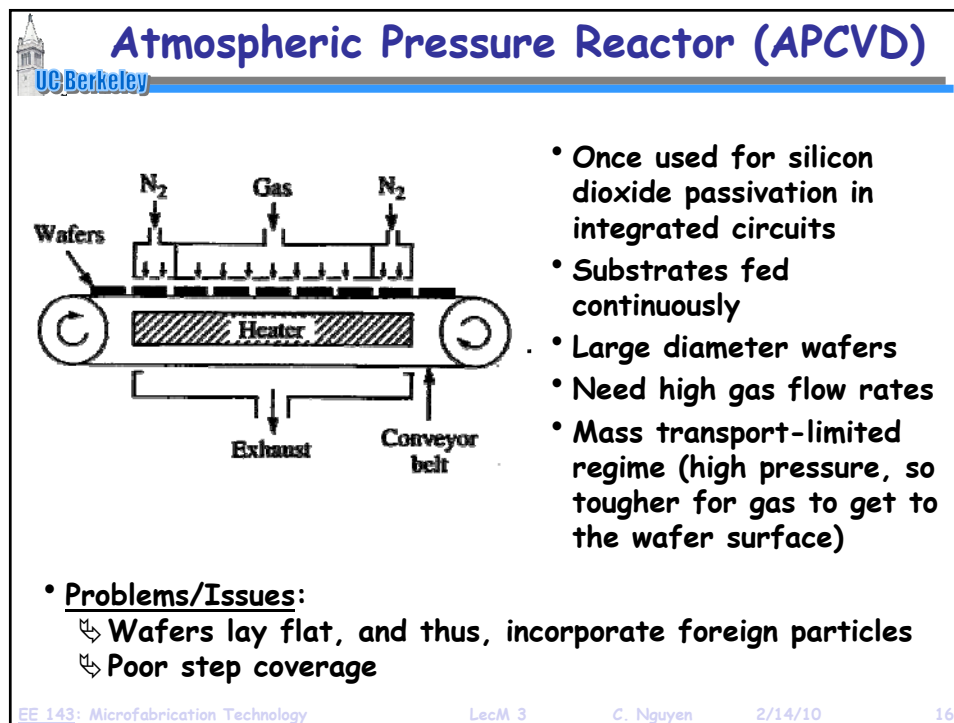
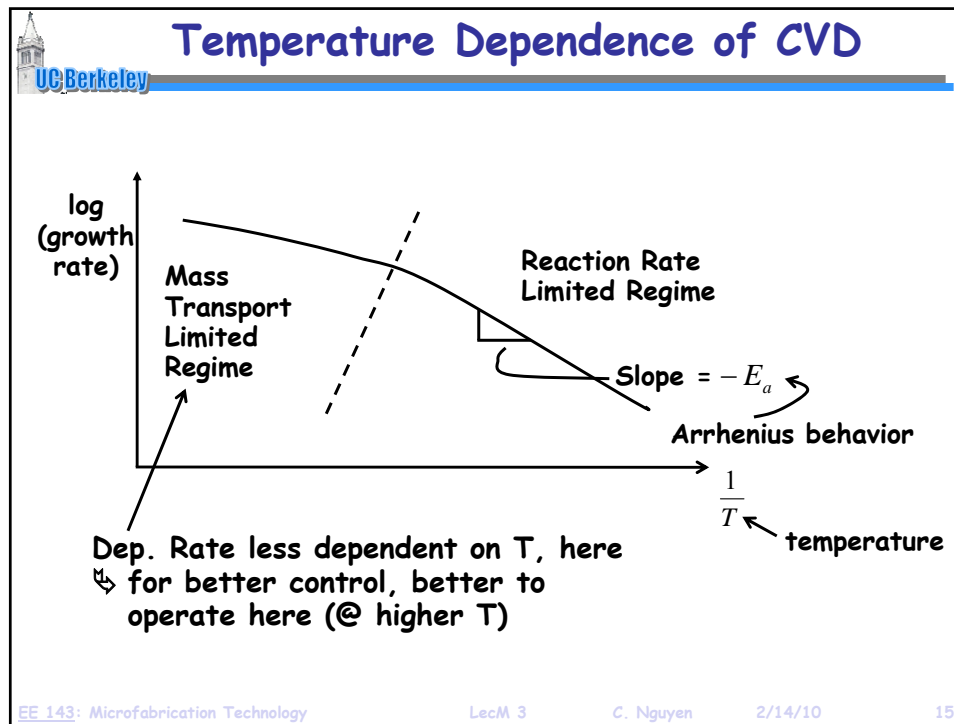
 (mass-transfer-limited)
- Case: $h_g \gg k_s$
 \hookrightarrow mass transfer rate \gg surface reaction rate


$\text{growth rate} = k_s \frac{N_g}{N}$

 (surface-reaction-limited)

$\sim R_0^{-E_a/kT}$ (Arrhenius character)

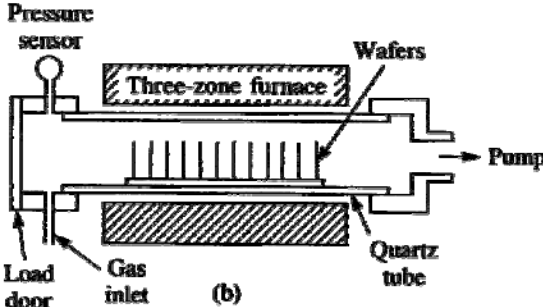
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Low Pressure Reactor (LPCVD)

- Many films available: polysilicon, SiGe, Si₃N₄, SiO₂, phosphosilicate glass (PSG), BPSG, W
- Temp.: 300 → 1150°C
- Press.: 30 → 250 Pa (200mTorr → 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




(b)

Problems:

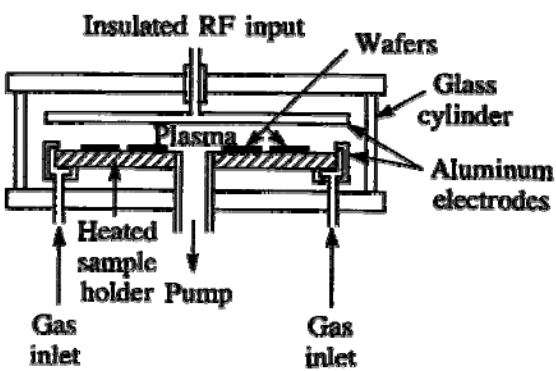
- ↪ Low dep. rate (compared to atm.)
- ↪ Higher T (than atmospheric)
- ↪ In hot wall reactors, get deposition on tube walls (must clean)

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Plasma-Enhanced CVD Reactor (PECVD)

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




(b)

Problems:

- ↪ Pin-holes
- ↪ Non-stoichiometric films
- ↪ Incorporation of H₂, N₂, O₂ contaminants in film; can lead to outgassing or bubbling in later steps

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Polysilicon CVD


Polysilicon Deposition:

$$\text{SiH}_4 \xrightarrow{600^\circ\text{C}} \text{Si} + 2\text{H}_2 \quad (\text{thermal decomposition of silane})$$

Fairly high temperature → conformal
 (conformal → high T)
 LPCVD (25 to 150 Pa) → 100-200 Å/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)

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Silicon Oxide CVD

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

{

$$\begin{aligned} \text{SiH}_4 + \text{O}_2 &\xrightarrow{300-500^\circ\text{C}} \text{SiO}_2 + 2\text{H}_2 \\ \text{(silane)} & \\ \\ 4\text{PH}_3 + 5\text{O}_2 &\xrightarrow{300-500^\circ\text{C}} 2\text{P}_2\text{O}_5 + 6\text{H}_2 \\ \text{(phosphine)} & \end{aligned}$$

Phosphosilicate glass (PSG)
- Above reactions: not very conformal step coverage → need higher T for this

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Silicon Oxide CVD (cont.)

- Phosphosilicate glass can be reflow
 - ↪ 6-8 wt. % allows reflow @ 1000-1100°C
 - ↪ Very useful to achieve smoother topography
 - ↪ Lower concentration → won't reflow
 - ↪ Higher concentration → corrodes Al 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

HTO { $\text{SiCl}_2\text{H}_2 + 2\text{N}_2\text{O} \xrightarrow{\sim 900^\circ\text{C}} \text{SiO}_2 + 2\text{N}_2 + 2\text{HCl}$


(dichlorosilane)

(Nitrous oxide)

(nice conformal step coverage)

or ...

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Silicon Oxide CVD (cont.)

$\text{Si}(\text{OC}_2\text{H}_5)_4 \xrightarrow{650-750^\circ\text{C}} \text{SiO}_2 + \text{by-products}$

(Tetraethylorthosilicate)
(TEOS)

(excellent uniformity & conformal step coverage)

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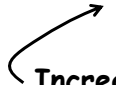
Silicon Nitride CVD

Silicon Nitride Deposition:

- First, note that thermal growth is possible:
 - Si in NH_3 @ 1000-1100°C
 - But very slow growth rate, thus, impractical
- LPCVD reactions:


$$\text{Silane reaction: } 3\text{SiH}_4 + 4\text{NH}_3 \xrightarrow[\text{(Atm. Press.)}]{700-900^\circ\text{C}} \text{Si}_3\text{N}_4 + 12\text{H}_2$$

$$\text{Dichlorosilane reaction: } 3\text{SiCl}_2\text{H}_2 + 4\text{NH}_3 \xrightarrow[\text{(LPCVD)}]{700-800^\circ\text{C}} \text{Si}_3\text{N}_4 + 6\text{HCl} + 6\text{H}_2$$


 Increase and T = 835°C → Si rich nitride → low stress

Problem: Clobbers your pumps! Expensive to maintain!

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Silicon Nitride CVD (cont.)

- Comments on LPCVD nitride films:
 - Hydrogen rich: ~8% H_2
 - 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 ($10^{16} \Omega\text{-cm}$) and dielectric strength (10 MV/cm)

PECVD Nitride:

$$\text{SiH}_4 + \text{N}_2 \xrightarrow{\text{Nitrogen discharge}} 2\text{SiNH} + 3\text{H}_2$$

or

$$\text{SiH}_4 + \text{NH}_3 \xrightarrow{\text{Ar plasma}} \text{SiNH} + 3\text{H}_3$$

PECVD films:

- Non-stoichiometric nitride
- 20-25% H_2 content
- Can control stress
- ($10^6 \Omega\text{-cm}$) resistivity

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Metal CVD

CVD Metal Deposition:

Tungsten (W) - deposited by thermal, plasma or optically-assisted decomposition

$$\text{WF}_6 \longrightarrow \text{W} + 3\text{F}_2$$

or via reaction with H_2 :

$$\text{WF}_6 + 3\text{H}_2 \longrightarrow \text{W} + 6\text{HF}$$

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

$$2\text{MCl}_5 + 5\text{H}_2 \longrightarrow 2\text{M} + 10\text{HCl},$$

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)

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Epitaxy

Epitaxy:

- Use CVD to deposit Si on the surface of a Si wafer
 - ↳ Si wafer acts as a seed crystal
 - ↳ Can grow a single-crystal Si film (as opposed to poly-Si)

Modeling -similar to CVD → in fact, the model discussed so far for CVD is more relevant to epitaxy than CVD!

↖ get similar curve:

Reactions - can use SiCl_4 , SiH_4 , SiH_2Cl_2 for vapor phase epitaxy.

SiCl_4 : Silicon tetrachloride

SiH_4 : silane

SiH_2Cl_2 : dichlorosilane

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Epitaxy (cont.)

Most popular: $\text{SiCl}_4 (\text{gas}) + 2\text{H}_2 (\text{gas}) \xrightarrow{1200^\circ\text{C}} \text{Si} (\text{solid}) + 4\text{HCl} (\text{gas})$

(Note that this is reversible!) \Rightarrow Reverse reaction (i.e., etching) if have excessive HCl \rightarrow sometimes used before deposition to clean the Si wafer surface.

Also get a competing reaction.

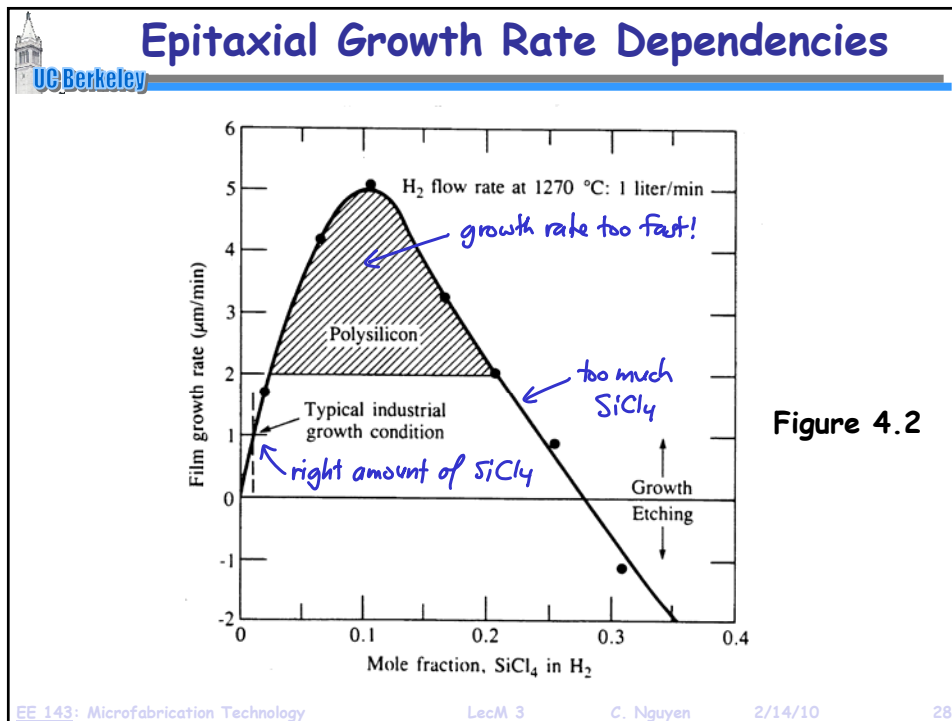
$\text{SiCl}_4 (\text{gas}) + \text{Si} (\text{solid}) \longleftrightarrow 2\text{SiCl}_2 (\text{gas})$


\hookrightarrow Too much $\text{SiCl}_4 \rightarrow$ etching rather than growth takes place!
 \hookrightarrow Growth rate too fast \rightarrow get polysilicon instead of Si. ($> 2\mu\text{m}/\text{min.}$)

See Figure 4.2

Important that the right conc. of SiCl_4 is used!

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Epitaxy (cont.)

Alternative reaction: pyrolytic decomposition of silane:

$$\text{SiH}_4 \xrightarrow{650^\circ\text{C}} \text{Si} + 2\text{H}_2$$


↙ 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
 - ↘ causes silica formation

Doping of Epitaxial Layers:

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

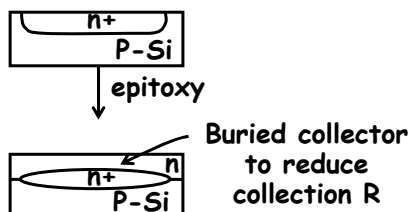
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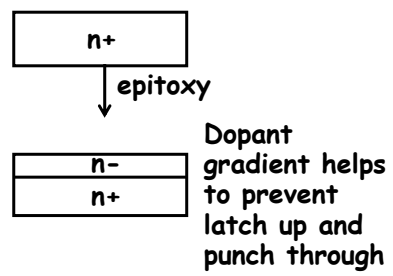
Doping of Epitaxial Layers

2. Use "autodoping" → when growing own heavily-doped substrates
 - ↙ Impurity evaporates from wafer (or liberated by Cl etching of surface during dep.)
 - ↙ Incorporates into gas stream
 - ↙ Impurities dope new layer
 - ↙ Examples of autodoping:

Bipolar Processing:



MOS:



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Atomic Layer Deposition (ALD)

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Atomic Layer Deposition (ALD)

• Fundamental Components:

- Self-limiting surface reactions of suitable precursor compounds A & B
- A & B then form the desired product S in a binary reaction cycle consisting of two sequential half-reactions


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Atomic Layer Deposition (ALD)

- **Remarks:**
 - ↪ Both half-reactions must be *complete* and *self-limiting* at the monolayer level
 - ↪ The total film thickness $d(\text{tot})$ can be “digitally” controlled by the number of applied deposition cycles $N(A/B)$:
$$d(\text{tot}) = d(\text{mono}) \cdot N(A/B)$$
 - ↪ 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

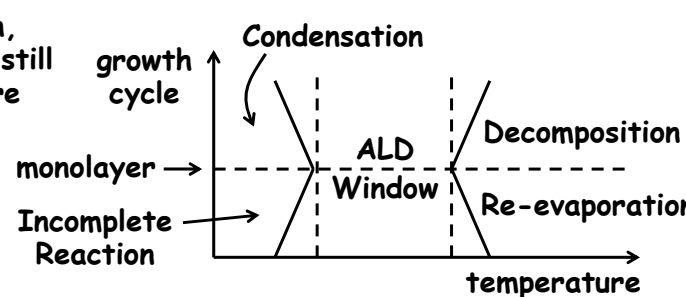
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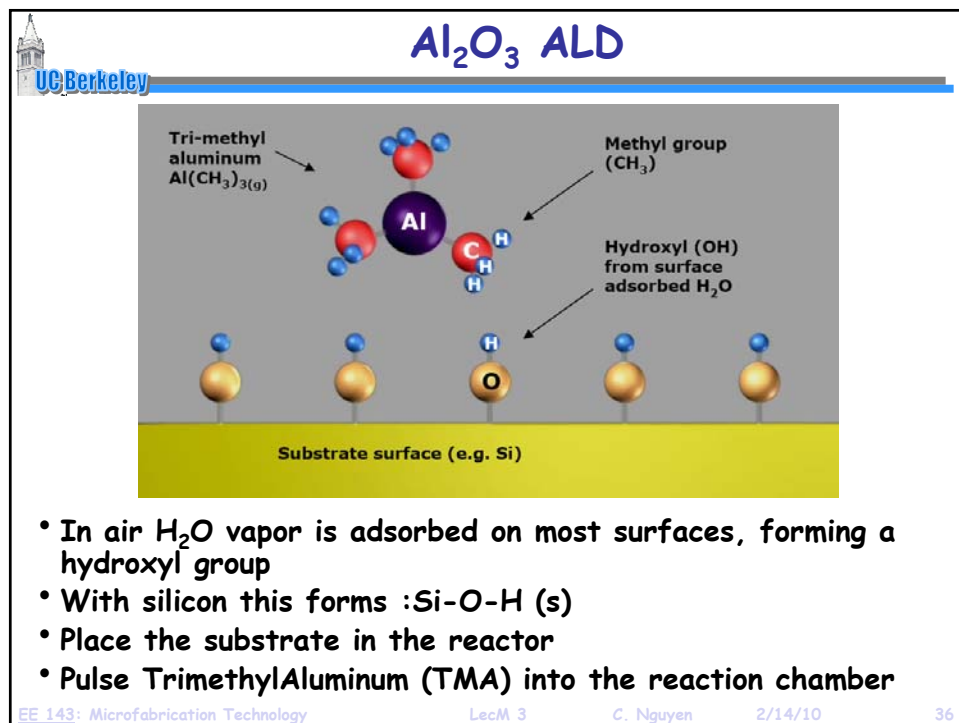
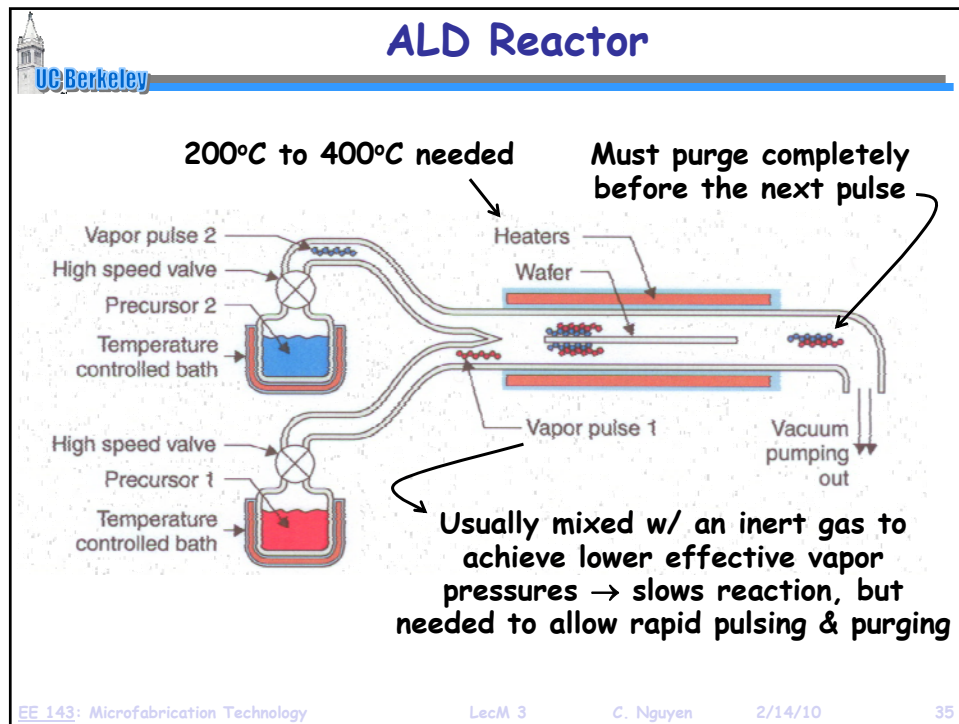
Advantages of ALD

- Surface limited reaction
 - ↪ excellent step coverage and refilling
- Self-limiting mechanism
 - ↪ Monolayer deposition
 - ↪ Composition control
 - ↪ Thickness control (\propto # of cycles)
 - ↪ Less sensitive to flow rate & temperature

Note, though, that there's still a temperature window:



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Al₂O₃ ALD

The diagram shows a substrate surface (e.g., Si) with several hydroxyl groups (Si-OH). A Trimethylaluminum (TMA) molecule, represented as a central purple sphere (Al) bonded to three red spheres (C), each of which is bonded to three white spheres (H), is shown reacting with one of the hydroxyl groups. An arrow labeled "Reaction of TMA with OH" points to the reaction site. Another arrow labeled "Methane reaction product CH₄" points to a methane molecule (one red sphere bonded to four white spheres) being released. The resulting surface has a monolayer of Al(CH₃)₂ groups.

Substrate surface (e.g. Si)

- TrimethylAluminum (TMA) reacts with the adsorbed hydroxyl groups, producing methane as the reaction product

$$\text{Al}(\text{CH}_3)_3 (g) + \text{:Si-O-H} (s) \rightarrow \text{:Si-O-Al}(\text{CH}_3)_2 (s) + \text{CH}_4$$

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Al₂O₃ ALD

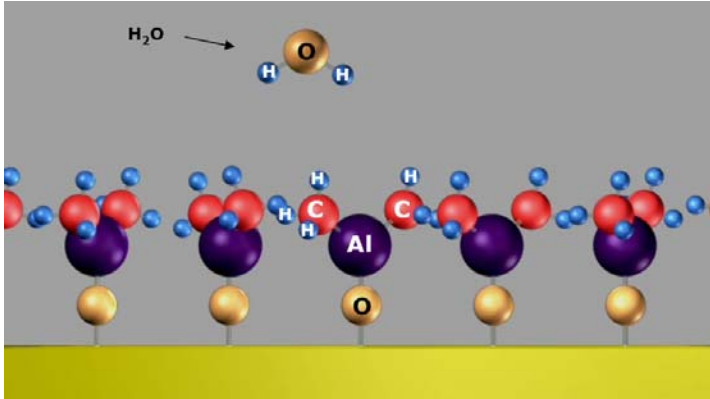
The diagram shows the substrate surface after the first step, with a monolayer of Al(CH₃)₂ groups. "Excess TMA" molecules are shown above the surface. An arrow labeled "Methane reaction product CH₄" points to a methane molecule being released. The diagram illustrates that the reaction is self-limiting, forming only one layer of Al(CH₃)₂ groups.

Substrate surface (e.g. Si)

- 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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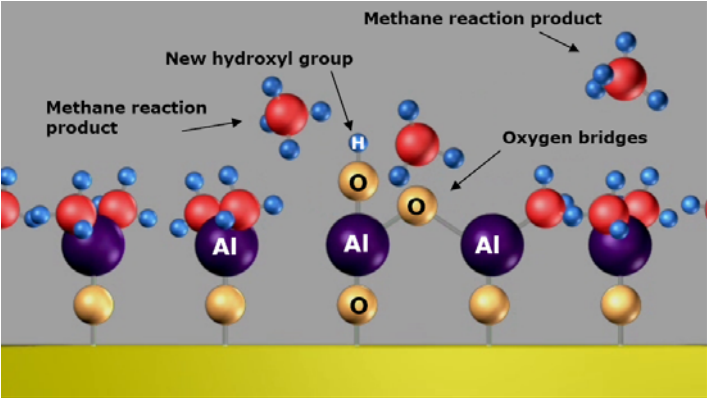
Al₂O₃ ALD



- After the TMA and methane reaction product is pumped away, water vapor (H₂O) is pulsed into the reaction chamber.

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Al₂O₃ ALD



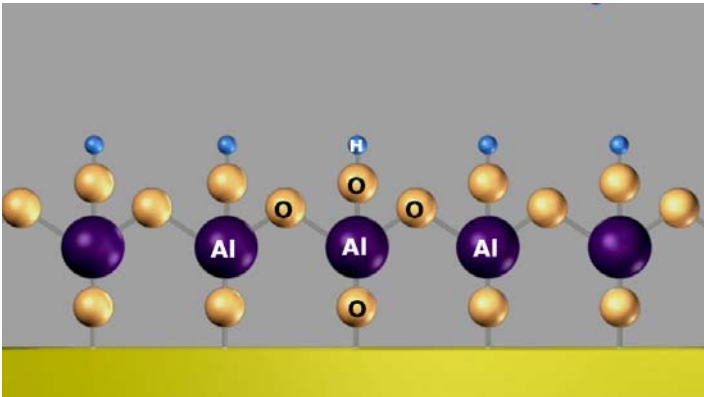
- H₂O reacts with the dangling methyl groups on the new surface forming aluminum-oxygen (Al-O) bridges and hydroxyl surface groups, waiting for a new TMA pulse
- Again methane is the reaction product

$$2 \text{H}_2\text{O (g)} + \text{:Si-O-Al(CH}_3)_2 \text{(s)} \rightarrow \text{:Si-O-Al(OH)}_2 \text{(s)} + 2 \text{CH}_4$$

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Al₂O₃ ALD

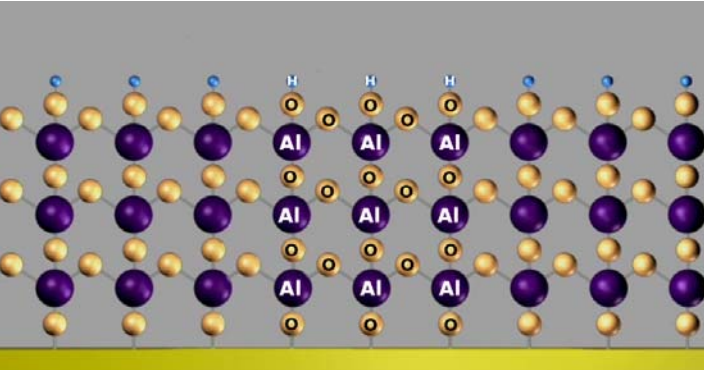


- The reaction product methane is pumped away
- Excess H₂O vapor does not react with the hydroxyl surface groups
- Again, get perfect passivation to one atomic layer

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Al₂O₃ ALD

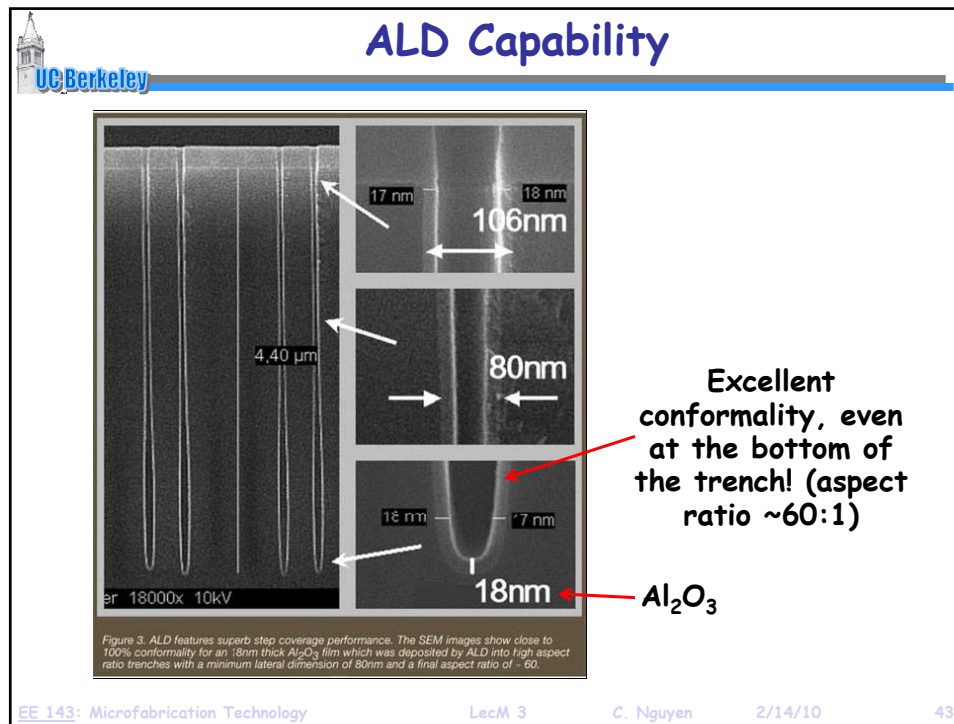


- One TMA and one H₂O vapor pulse form one cycle
- Here, three cycles are shown, with approximately 1 Å per cycle
- Each cycle including pulsing and pumping takes, e.g., 3 sec

$$\text{Al}(\text{CH}_3)_3 \text{ (g)} + \text{:Si-O-H (s)} \rightarrow \text{:Si-O-Al}(\text{CH}_3)_2 \text{ (s)} + \text{CH}_4$$

$$2 \text{ H}_2\text{O (g)} + \text{:Si-O-Al}(\text{CH}_3)_2 \text{ (s)} \rightarrow \text{:Si-O-Al}(\text{OH})_2 \text{ (s)} + 2 \text{ CH}_4$$


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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
Interface 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

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Electroplating

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Metal Electroplating

Electroplating: the process using electrical current to coat an electrically conductive object with a thin layer of metal

- ↳ Useful when very thick ($>1\mu\text{m}$) metal films are needed
- ↳ Evaporation and sputtering generally suffer from excessive stress when films get too thick → get peeling

1. Switch on external supply of direct current
2. Metal at anode is oxidized to form cations with a (+) charge
3. Cations are attracted to the (-) charge on the cathode
4. Cations get reduced by e^- 's at the cathode, depositing the metal (in this case, Cu)

Labels in diagram: Anode (often made of the metal to be plated), Cathode (part to be plated), Electrolyte, $\text{CuSO}_4(\text{aq})$, Cu^{2+} , SO_4^{2-} , Cation, Anion, Spoon, Battery, e^- .

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Wafer-Level Implementation

Wafer Preparation: areas where plating is to occur must have electrical access to the DC voltage source

- ↳ Often use a seed layer that accesses all plating locations

Labels in diagram: Ti/Au, Nickel, Photoresist, Aluminum, Silicon Substrate.

Al layer insures electrical contact to plating areas, despite patterned Ti/Au

Labels in diagram: DC Voltage Source, Electrical Connector, Container, Electrolyte Solution, Counter Electrode, Wafer, Wafer Holder.

- Need not be the metal to be electroplated
- ↳ Often just a platinum electrode
- ↳ In this case, must replenish electrolytic solution after time

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