



### ME 141B: The MEMS Class Introduction to MEMS and MEMS Design

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### **Outline today**



- Introduction to thin films
- Oxidation
  - Deal-grove model
- CVD
- Epitaxy
- Electrodeposition



- Many techniques to choose from
- Differences:
  - Front or back end processes
  - Quality of resulting films (electrical properties, etch selectivity, defects, residual stress)
  - Conformality
  - Deposition rate, cost
- Physical techniques
  - Material is removed from a source, carried to the substrate, and dropped there
- Chemical Techniques
  - Reactants are transported to the substrate, a chemical reaction occurs, and the product deposit on the substrate to form the desired film



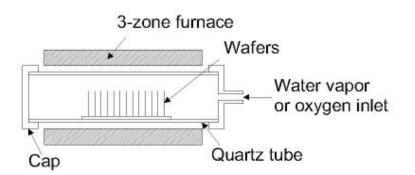


- Chemical
  - Thermal Oxidation
  - Chemical Vapor Deposition (CVD)
    - Low Pressure (LPCVD), Atomspheric pressure (APCVD), Plasma Enhanced (PECVD), Ultra High Vaccum CVD (UHCVD)
  - Epitaxy
  - Electrodeposition (Electroplating)
- Physical
  - Physical Vapor Deposition (PVD)
    - Evaporation
    - Sputtering
  - Spin-casting





- Most basic deposition technologies
- Oxidation of a substrate surface in an O<sub>2</sub> rich atmosphere
- Temperature is raised (800-1100C) to speed up process
- Only deposition technology which CONSUMES substrate
- Parabolic relationship between film thickness and oxidation time for films thicker than ~100 nm





# why is oxidation so important?



- Oxides are vital in device structures:
  - gate oxide in MOS transistors
  - ➢ field oxide for device isolation
- SiO<sub>2</sub>/Si has excellent electrical characteristics
- Other desirable characteristics: excellent adhesion, nonconductive, planar processing

\*the ability to easily and reproducibly grow highquality oxides on silicon is one of the principal reasons for the importance of silicon to semiconductor devices!



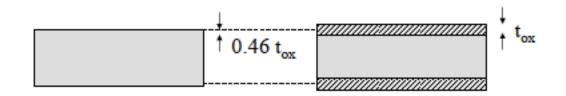


- Whenever you can!
- Best, highest quality oxide
- Unfortunately, high temperature makes it limited use
- Typically used to form films that are used for electrical insulation
- Also used as etch masks or etch stops for future processing steps





- Silicon forms a high quality, stable oxide
  - ➤ How it works:
    - Oxygen diffuses through oxide to SI/oxide interface
    - SI + O<sub>2</sub> + high temperature (~1100) furnace  $\rightarrow$  SiO<sub>2</sub>
    - Some Si is consumed



- Rate determined by diffusion of oxygen through oxide
- > Diffusion limits practical oxide thickness to < 2 um
- A key front end process



## **Oxidation II**



- Dry Oxidation (O<sub>2</sub>)
  - High quality, slow oxidation rate, smaller maximum thickness (i.e. gate oxide)
- Wet Oxidation (steam)
  - > H<sub>2</sub> to speed the diffusion
  - Lower quality, faster oxidation rate
- The Deal-Grove model describe the kinetics of oxidation quite well for oxides greater in thickness that about 30nm



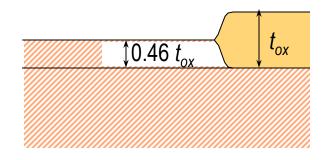
## oxidation reactions and oxide properties



- Two common oxidation reactions:
  - > Dry oxidation: Si (solid) +  $O_2$  (gas)  $\longrightarrow$  Si $O_2$  (solid)
  - > Wet oxidation: Si (solid) +  $2H_2O$  (vapor)  $\longrightarrow$  SiO<sub>2</sub> (solid) +  $2H_2$
- Crystal structure:
  - > crystalline SiO<sub>2</sub> exists in >11 distinct forms
  - > SiO<sub>2</sub> grown by thermal oxidation are amorphous
  - density of thermal SiO<sub>2</sub> (2.15 to 2.27 g cm<sup>-3</sup>) is less than crystalline SiO<sub>2</sub> (2.65 g cm<sup>-3</sup>) – the more "open" structure permits impurities (water, oxygen, sodium) to easily diffuse interstitially



A film of growing SiO<sub>2</sub> of thickness
 *t<sub>ox</sub>* will consume a layer of
 crystalline Si of about 0.46 *t<sub>ox</sub>*



- The oxide-Si interface is displaced from the original Si surface and is located deeper into the Si wafer
- With both O<sub>2</sub> and H<sub>2</sub>O the oxidizing species moves through the oxide layer to react
- The displaced and deeper interface + inward migration of oxidizing species provides a clean, contaminant-free oxide-Si interface





- Mathematically describes the growth of an oxide layer on the surface of a material
- Used to analyze thermal oxidation of silicon
- Physical assumptions
  - Oxygen diffuses from the bulk to the ambient gas to the surface
  - It diffuses through the existing oxide layer to the oxide-substrate interface
  - It then reacts with the substrate
- Model:
  - ➤ Henry's law
  - Fick's law of diffusion
  - First-order reaction
- Also assume steady state conditions





#### For oxides greater than about 30 nm thick:

$$\chi_{final} = 0.5 \left[ A_{DG} \left[ \sqrt{1 + \frac{4 B_{DG}}{A_{DG}^2} \left( t + \tau_{DG} \right) - 1} \right] \right]$$
  
where  $\tau_{DG} = \frac{\chi_i^2}{B_{DG}} + \frac{\chi_i}{B_{DG}^2} A_{DG}$ 

(Constants are given in the text; beware units of  $B_{DG}$ ,  $\mu$ m<sup>2</sup>/hour)

Growth goes approximately as *t* for short times, and approximately as  $\sqrt{t}$  for long times.



Temperature	A <sub>DG</sub>	B <sub>DG</sub>
920	0.05	0.203
1000	0.226	0.287
1100	0.11	0.510

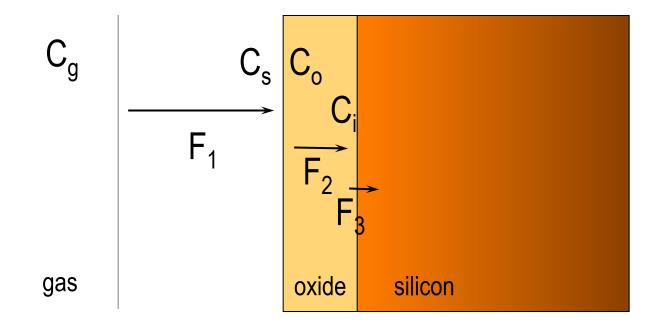
Determine how long it takes to grow 1um of oxide on a bare silicon wafer at 1000C under both wet and dry conditions

 $t = x_f^2 / B_{DG}$ 

47 minutes under wet conditions and 14.1 hours under dry conditions



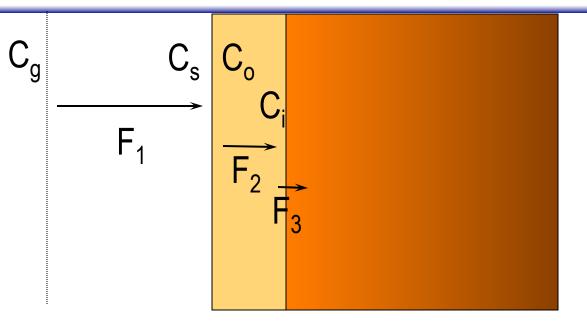




assumption: oxide growth occurs by diffusion of oxygen to the silicon interface, where the reaction Si +  $O_2 \rightarrow SiO_2$  occurs



## The Deal-Grove model inanolab



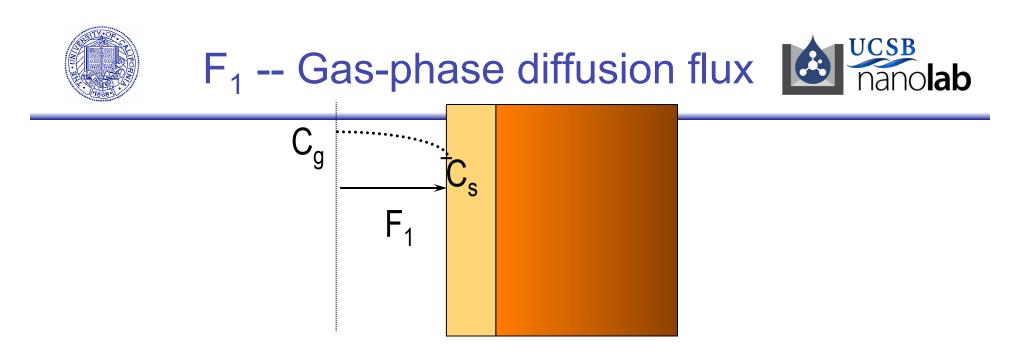
#### Fluxes:

 $F_1$  -- oxygen flux to the surface  $F_2$  -- oxygen flux through the oxide  $F_3$  -- oxygen flux reacting with the

silicon substrate

#### **Concentrations**:

 $C_g$  -- gas-phase concentration  $C_s$  -- oxygen conc. at surface  $C_o$  -- conc. inside oxide surface  $C_i$  -- conc. at Si/SiO<sub>2</sub> interface

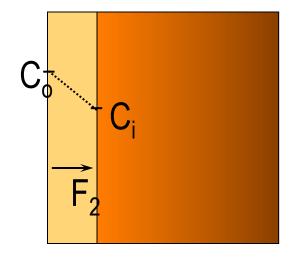


Oxygen must diffuse across a stagnant boundary layer to the surface:

$$F_1 = D_{O_2} \frac{C_g - C_s}{t_{sl}}$$
$$= h_g \left( C_g - C_s \right)$$

where  $h_g$  is the mass transport coefficient



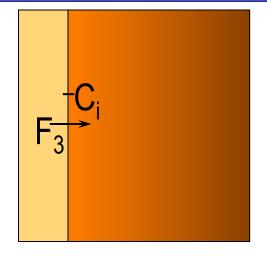


• Oxygen must diffuse through the oxide to the silicon interface:

$$F_2 = D_{O_2} \frac{C_o - C_i}{t_{ox}}$$

(note: the diffusivity is for oxygen in the oxide)





 The oxygen at the Si/SiO<sub>2</sub> interface reacts to form the oxide:

$$F_3 = k_s C_i$$

where  $k_s$  is the chemical rate constant for the oxidation reaction



## Solution to the Deal-Grove model (1)



 at steady state (*not equilibrium!*) the fluxes will be equal:

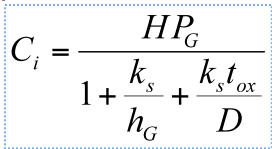
$$F_1 = F_2 = F_3 \equiv F$$

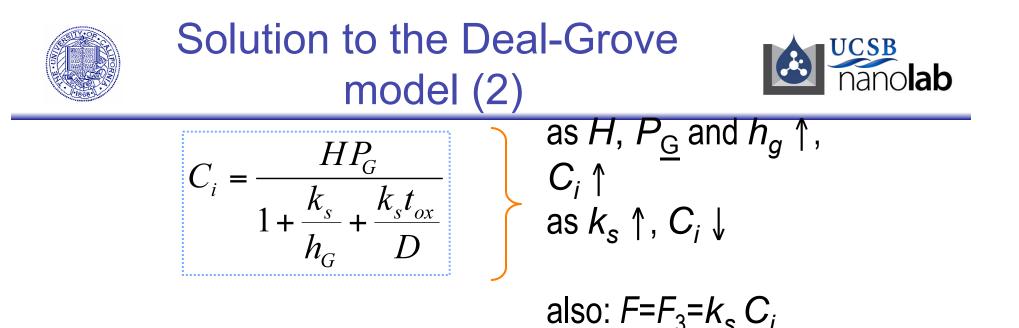
 note that the oxygen concentration at the oxide surface (C<sub>o</sub>) is proportional to the gas pressure (Henry's Law)

$$C_o = HP_G = HkT C_G$$

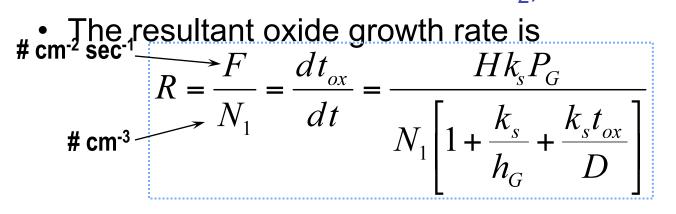
since  $C_G = n/V = P_G/kT$  (ideal gas law)

• "After some algebra..." one obtains:





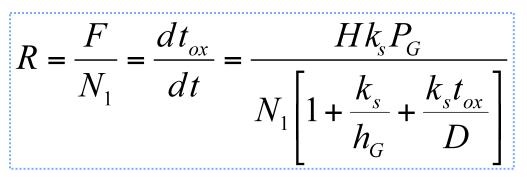
 The oxide growth rate will be given by: (flux at interface)÷(number of O<sub>2</sub> molecules per unit vol. of SiO<sub>2</sub>)





## Solution to the Deal-Grove model (3)

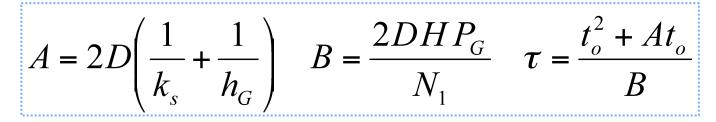


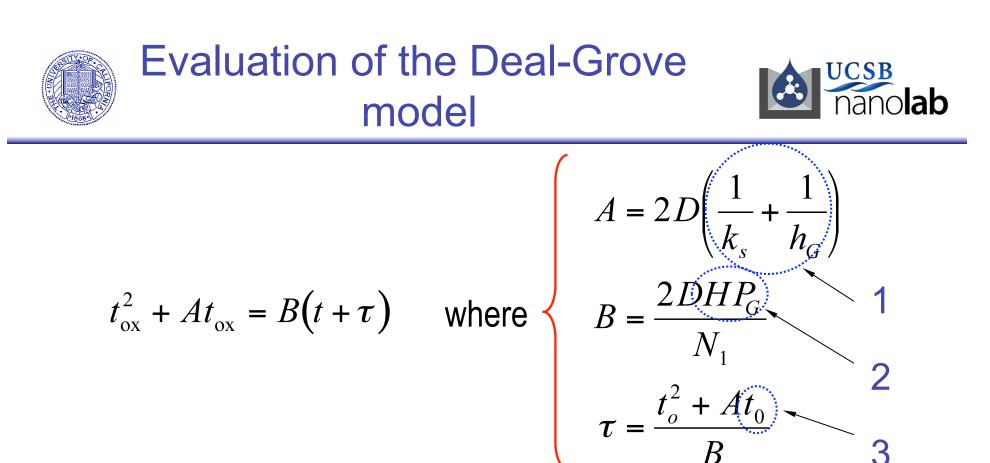


The solution to this differential equation is:

$$t_{ox}^2 + At_{ox} = B(t + \tau)$$

where





- 1. In most cases, oxidation is performed at atmospheric pressure where  $h_G \rangle k_s$  and  $A \approx 2D/k_s$  (units: cm)
- 2.  $C_o = HP_G$  so  $B = 2DC_o/N_1$  (units: cm<sup>2</sup> sec<sup>-1</sup>)

**3**.  $t_o$  is the oxide thickness at t = 0





$$t_{\text{ox}}^{2} + At_{\text{ox}} = B(t + \tau) \implies t \approx 0 \text{ and } t_{\text{ox}}^{2} \Rightarrow 0$$
  
or: 
$$t_{\text{ox}} = \frac{B}{A}(t + \tau)$$

- Early in the oxidation process the oxide thickness is *linearly proportional* to the oxidation time
- The ratio *B*/*A* is often called the "linear rate constant"
- B/A is equal to  $C_o k_s / N_1$  and has units of cm sec<sup>-1</sup>  $\rightarrow$ *independent* of D
- $\tau$  arises from the boundary condition at t = 0 -- either the oxide thickness at t = 0 is  $t_o$ , or the thickness is zero at time  $-\tau$





$$t_{ox}^{2} + At_{ox} = B(t + \tau) \implies t_{ox}^{2} >> t_{ox} \text{ and } t >> \tau$$
  
or:  $t_{ox}^{2} = Bt$ 

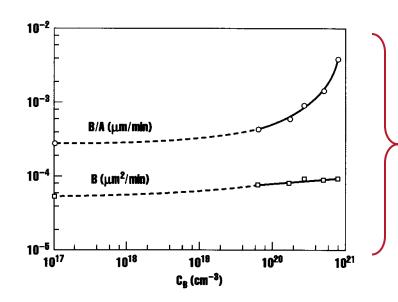
- *B* depends on the product of *D* and  $C_o \rightarrow$  a function of reactant concentration within the oxide and its ability to diffuse through the oxide
- *B* is often called the "parabolic rate constant" since a plot of the square of the thickness versus time would look like a parabola
- Systems that exhibit parabolic growth kinetics typically are *diffusion controlled*



- Linear regime between the growth rate is constant in time and is independent of the amount of gas and substrate that are consumed
- Parabolic regime a thermally-activated diffusion process is controlling the growth rate
- In general, the Deal-Grove model does an excellent job of describing the oxidation process under a wide variety of conditions



- Since the linear rate depends on the surface reaction rate, it can be sensitive to the properties of the substrate
  - the linear rate constant for growth on the (111) Si surface is 1.7× greater than for growth on the (100) surface (reason: larger number of available bonds on the (111) surface)
  - heavy doping of the silicon substrate can increase the linear rate constant by a factor of 10 -- doping enhanced oxidation, or DEO (reason: doping alters the interstitial and vacancy concentrations)



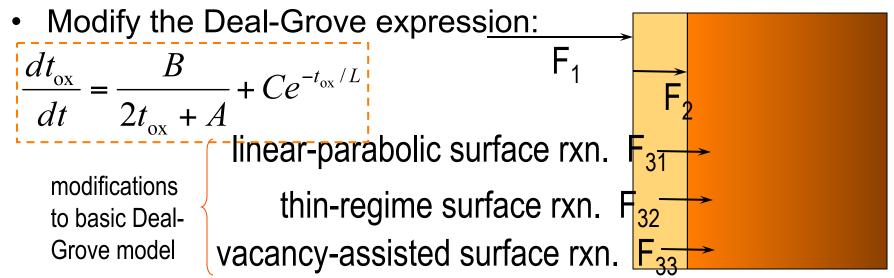
note: no effect on the parabolic rate constant (more to come on doping effects in oxidation...)



## Initial rapid growth rate



- Deviations from the Deal -Grove linear-parabolic oxidation theory for very thin oxide layers (short-time growth) -growth *faster* than expected
- Possible causes included: space-charge effects (fieldassisted diffusion); structural defects in the oxide; stress effects leading to increased diffusivity; increased solubility of the oxidant in the thin oxide; and diffusion of silicon to the oxide surface





# Observations on the parabolic regime



- The parabolic rate constant is sensitive to factors that alter the ability of the oxidizing species to diffuse through the oxide
  - H<sub>2</sub>O is a smaller molecule than O<sub>2</sub> and thus will have a larger diffusion coefficient
  - The oxide formed by wet oxidation is more porous and less dense than that formed without water (dry oxidation) and permits higher diffusivities at lower temperatures

ppm H <sub>2</sub> O	B/A (Å min <sup>-1</sup> )	<i>B</i> (Ų min⁻¹)	
<1	2.2	4350	
1000	2.9	8400	
assing the o	vugan nrassur	a will also increas	ΔR

 Increasing the oxygen pressure will also increase B and decrease the time needed to grow a thick oxide



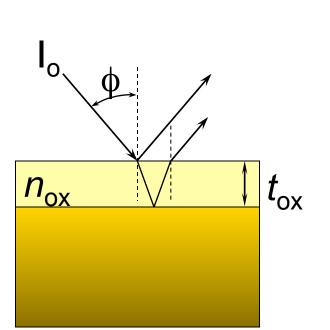


A silicon wafer is initially covered by an SiO<sub>2</sub> film 0.3 µm thick. What is the time required to increase the thickness to 0.5 µm?  $t_{ox}^2 + At_{ox} = B(t + \tau) \approx Bt$  (ignore  $\tau$  for thick oxide) For a wet oxidation at 1200°C,  $A = 0.05 \mu m$  and  $B = 0.720 \ \mu m^2/hr$ , so:  $(0.2)^2 + (0.05)(.2) = (0.720)t$  or t = 0.069 hours or ~ 4.2 minutes If a dry oxidation at is performed at 1200°C,  $A = 0.04 \mu m$  and  $B = 0.045 \,\mu m^2/hr$ , so:

(0.2)2 + (0.04)(.2) = (0.045)t or t = 1.067 hours or ~ 64 minutes



By measuring the intensity of reflected light as a function of wavelength, the oxide thickness can be determined from:



$$t_{\rm ox} = \frac{\Delta \lambda}{2n_{\rm ox}}$$

strictly true only at normal incidence

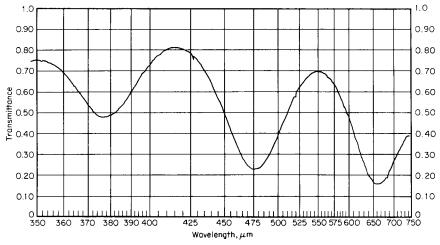


Fig. 6.12. Reflected intensity of an  $SiO_2$  layer on a Si substrate.





- Interference effects can give a thin transparent layer a characteristic color
- Color depends on:

   film thickness
   index of refraction
   spectral distribution of the viewing light
- Experienced viewers can estimate the thickness of a film to about 100Å

Film		Film	
thickness, μm	Color and comments	thickness, μm	Color and comments
0.05	Tan	0.63	Violet-red
0.07	Brown	0.68	"Bluish" (Not blue but borderline
0.10	Dark violet to red-violet		between violet and blue-green.
0.12	Royal blue		It appears more like a mixture
0.15	Light blue to metallic blue		between violet-red and blue-greer
0.17	Metallic to very light yellow-		and looks gravish)
	green	0.72	Blue-green to green (quite broad)
0.20	Light gold or yellow-slightly	0.77	"Yellowish"
	metallic	0.80	Orange (rather broad for orange)
0.22	Gold with slight yellow-orange	0.82	Salmon
0.25	Orange to melon	0.85	Dull, light red-violet
0.27	Red-violet	0.86	Violet
0.30	Blue to violet-blue	0.87	Blue-violet
0.31	Blue	0.89	Blue
0.32	Blue to blue-green	0.92	Blue-green
0.34	Light green	0.95	Dull yellow-green
0.35	Green to yellow-green	0.97	Yellow to "yellowish"
0.36	Yellow-green	0.99	Orange
0.37	Green-yellow	1.00	Carnation pink
0.39	Yellow	1.02	Violet-red
0.41	Light orange	1.05	Red-violet
0.42	Carnation pink	1.06	Violet
0.44	Violet-red	1.07	Blue-violet
0.46	Red-violet	1.10	Green
0.47	Violet	1.11	Yellow-green
0.48	Blue-violet	1.12	Green
0.49	Blue	1.18	Violet
0.50	Blue-green	1.19	Red-violet
0.52	Green (broad)	1.21	Violet-red
0.54	Yellow-green	1.24	Carnation pink to salmon
0.56	Green-yellow	1.25	Orange
0.57	Yellow to "yellowish" (not yellow	1.28	"Yellowish"
	but is in the position where	1.32	Sky blue to green-blue
	yellow is to be expected. At	1.40	Orange
	times it appears to be light	1.45	Violet
	creamy gray or metallic)	1.46	Blue-violet
0.58	Light orange or yellow to pink	1.50	Blue
	borderline	1.54	Dull yellow-green
0.60	Carnation pink		-

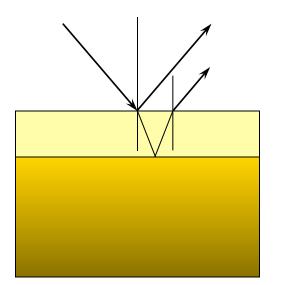
Table 6.6. Color Chart for Thermally Grown  ${\rm SiO}_2$  Films Observed Perpendicularly under Daylight Fluorescent Lighting

After Pliskin and Conrad.<sup>57</sup>

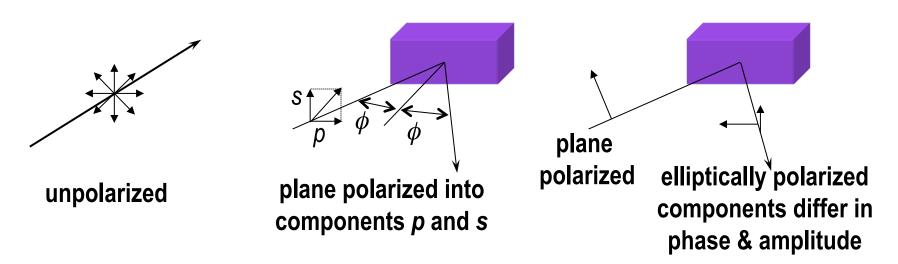


#### Physical characterization of oxides -- ellipsometry





- As light is reflected from a surface, it is normally reduced in amplitude and shifted in phase
- If there are multiple reflecting layers, the various beams will interact further with each other





## Ellipsometry for oxide thickness measurement



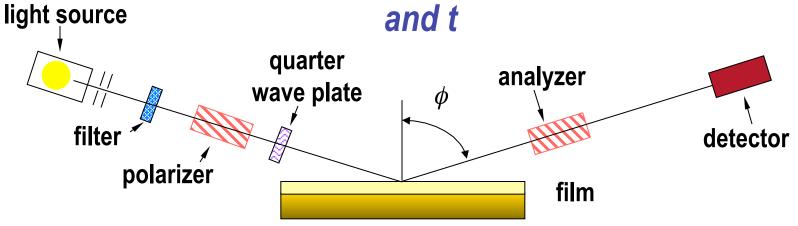
Most useful for studying thin (<  $1\lambda$ ) layers on thick substrates

 $\Delta$  = differential phase change =  $\Delta_p$  -  $\Delta_s$ 

$\Psi = \tan^{-1}($	$\left(\frac{R_p}{R_s}\right)$
---------------------	--------------------------------

 $R_p$  and  $R_s$  are the reflection coefficients for the *p* and *s* components

### $\Delta$ and $\Psi$ can be directly related to n







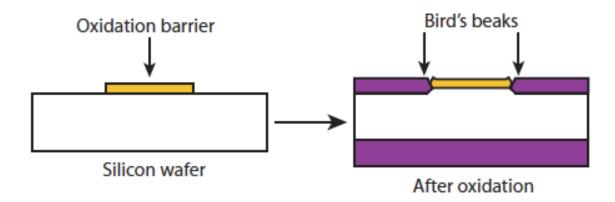
- Works well for single=crystal silicon
- Thin oxides grow more quickly in O<sub>2</sub>
- Fails for polycrystalline silicon
  - Random orientation of the crystal grains makes it difficult to choose value for linear rate constant
  - Oxidant molecules diffuse rapidly along grain boundaries oxidizes more rapidly
- Dopants strain silicon lattice making it easier for oxygen to enter – oxidizes faster



Local Oxidation



- Oxidation can be masked locally by an oxidation barrier, such as silicon nitride
- Oxide undercuts edge of mask layer to form a "bird's beak"
- Oxidation followed by an oxide etch can also be used to sharpen silicon features



Senturia, Microsystem Design.







- CVD is a chemical process used to produce high-purity, high-performance solid materials
- Typical CVD process
  - > Wafer exposed to one or more volatile precursers
  - These react and/or decompose on surface
  - This produces desired deposit
- Can deposit in various forms
  - Monocrystaline
  - Polycrystalline
  - Amorphous
  - Epitaxial
- Materials include silicon, carbon fiber, carbon nanofibers, filaments, carbon nanotubes, SiO<sub>2</sub>, silicon-germanium, tungsten, silicon carbide, silicon nitride, etc...



- How CVD works:
  - Gaseous reactants, often at low pressure
  - Long mean free path; reactants reach substrate
  - Reactants react and deposit products on the substrate
  - Unlike Oxidation, does not consume substrate material
- Energy sources facilitate CVD reactions:
  - > High temperature, plasma, laser
- Processing temperatures vary widely
- Commonly deposited films: Oxide, silicon nitride, polysilicon
- CVD results depend on pressures, gas flows, temperature
  - Film composition, uniformity, deposition rate, and electrical and mechanical characteristics can vary



Types of CVD



- Atmospheric pressure CVD (SPCVD) CVD processes at atmospheric pressure
- Low Pressure CVD (LPCVD)- CVD processes at subatmospheric pressures.
  - Reduced pressures tend to reduce unwanted gas-phase reaction and improve uniformity across the wafer
  - Most modern CVD processes are LPCVD or UHCVD
- Ultrahigh vacuum CVD (UHCVD) CVD process at a very low pressure, ~10<sup>-8</sup> torr
- Plasma-enhanced CVD (PECVD) CVD process that utilize a plasma to enhance chemical reaction rates of the precursors
  - Allows low temperatures
- 10/6/10 Other types, MPCVD, ALCVD, MOCVD



- Oxide formation:
  - > To get a thicker layer than thermal oxidation can provide
  - To create oxide on a wafer that can't withstand high temperatures (for example because of metal features)
  - ➤ To create oxide on top of a material that is not silicon
- For film formation in general
  - To tailor the film properties (like form stress) by adjusting pressures, flow rates, external energy supply, ratios of different precursor gases (to adjust proportions of different materials in the final product)
  - Conformally : (more of less) even coating on all surfaces
- Drawbacks:
  - Films deposited at low temperature are often lower quality than high temp versions, and have less predictable properties
- $_{0}$  > Flammable, toxic or corrosive source gases





- CVD is a common MEMS tool for creating thick films on the wafer surface
  - In practice, film stress limits thickness (film delamination or cracking, or curvature of underlying structures)
  - Can deposit thick oxides; nitrides are still typically submicron
  - Must anneal deposited oxides for some applications lose low stress property on anneal



#### Commonly Deposited Substances



- Polysilicon
  - > Deposited from silane (SH<sub>4</sub>) (SiH<sub>4</sub> $\rightarrow$  Si + 2H<sub>2</sub>)
  - Usually preformed in LPCVD systems
  - ➢ Growth rate 10-20 nm per minute
- Silicon dioxide
  - Source gases include silane and oxygen, dichlorosilane, nitrous oxide, or TEOS (tetraethlyorthosilicate)
  - Choice of source depends on thermal stability of substrates
    - ie. aluminum is sensitive to high temperature
  - TEOS is the best, but needs 650-700C, silane is lower quality. Thermal oxidation is best
  - Ozone may deposit TEOS at lower temperatures being explored



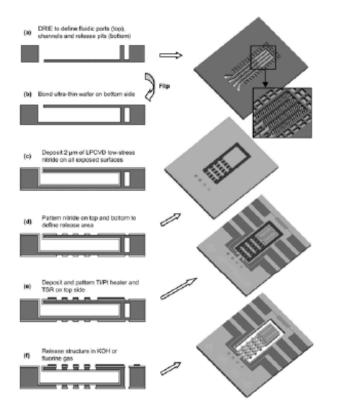
#### Commonly Deposited Substrates

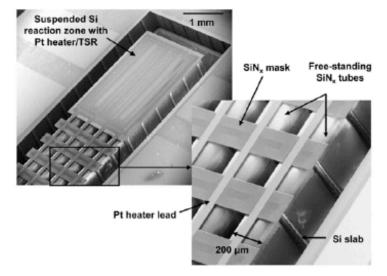


- Silicon Nitride
  - LPCVD generally used here
- Metals
  - > Molybdenum, tatalum, titatnium, nickel and tungsten
  - Deposited by LPCVD



# CVD enables conformal coating inanolab





MEMS-based fuel processor.



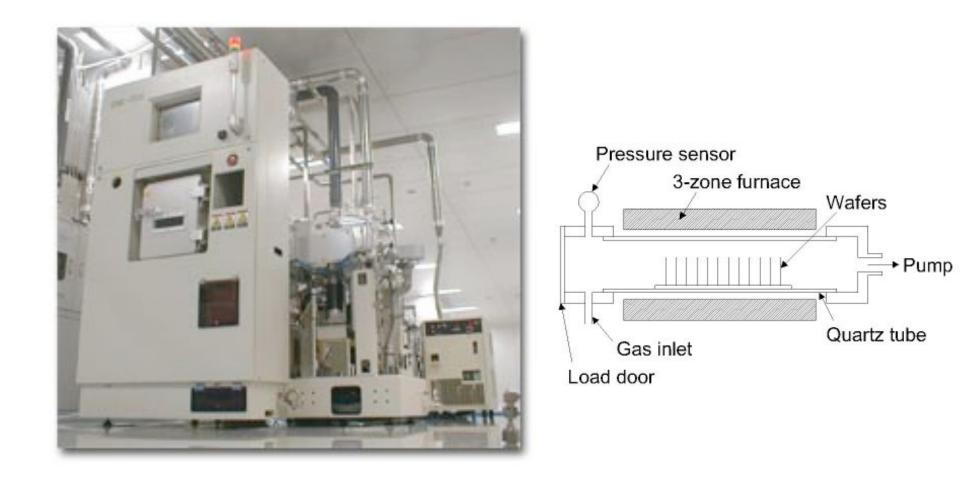


- Amorphous at lower deposition temperatures and high deposition rates
  - ➤ Typical temperature: ~590 C
- Polycrystalline at higher deposition temperatures and lower deposition rates
  - ➤ Typical temperature: ~625 C
- Grain size and structure depend on detailed deposition conditions
  - $\succ$  E.g. thicker films  $\rightarrow$  larger grains
- Structure, electrical properties, and mechanical properties also vary with post-deposition thermal processing
  - Grain growth
- 10/6/10 > Dpoant activation or diffusion



## **CVD** Machine











- CVD deposition process in which atoms move to lattice sites, continuing the substrate's crystal structure
  - ➤ Homoepitaxy: same material, i.e. Si on Si
  - Heteroepitaxy: different materials, i.e. AlGaAs, on GaAs
- How it happens
  - Slow deposition rate (enough time to find a lattice site)
  - High Temperature (enough energy to move a lattice site)
- Selective epitaxy is possible through masking
- Can grow a doped Si layer of known thickness

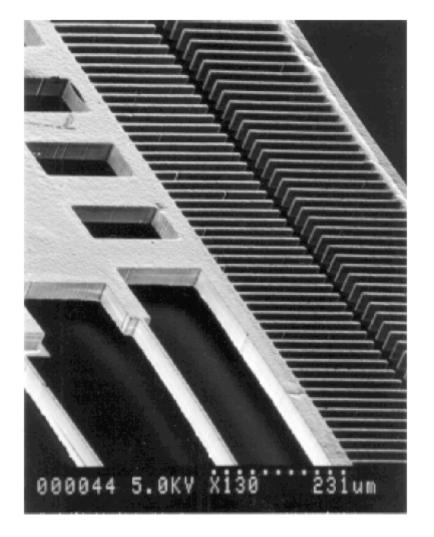


- Pass a current through an aqueous metal solution
  - Anode is made of the meta that you want to deposit
  - Cathode is the conductive seed material on your wafer
  - Positive metal ions travel to the negatively charged cathode on your wafer and deposit there
- Preparing your wafer
  - If you want to plate metal in some places and not in others, you will need a patterned metal seed layer (and typically a "sticky" metal adhesion layer under that)
  - For very short features, just plate onto the seed layer
  - ➢ For taller features, need to plate into a mold
  - Molds can be photoresist, silicon, SU-8, et.. Depending on the needs of your device



## Electroplating



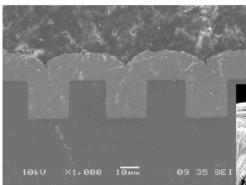


#### **Electroplating for LIGA**

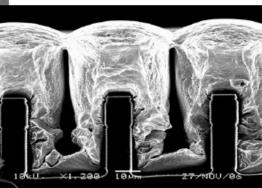
40 μm thick films of nickel fabricated by electroplating into a mold



# Electroplating realities

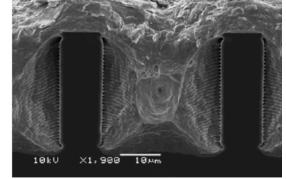


Test run w/ bump plating - perfect



#### Real device forms keyholes – different loading pattern

Courtesy of Dariusz Golda. Used with permission.



Solution: Cu damascene fill, with additives/agitation to promote fill at bottom

# Conformality and keyholes Manolab

- To lowest order, conformal films coat sidewalls and horizontal surfaces at the same rate
- But high aspect ratio trenches are prone to keyholes (CVD, electroplating, etc..)



