Ch. 10 Thin Film Process

陳國聲

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Outline

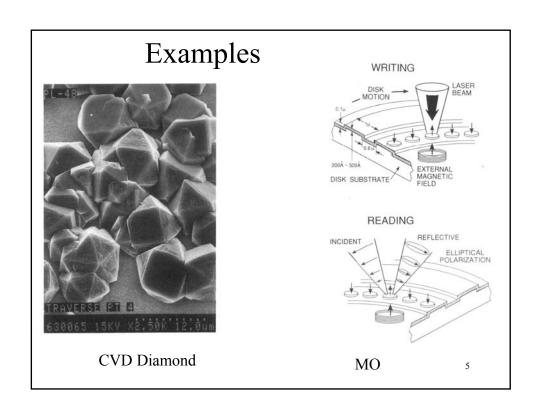
- Thin film applications
- Overview of deposition
- Physical vapor deposition
- Chemical vapor deposition
- Epitaxial growth
- Thin film materials sciences

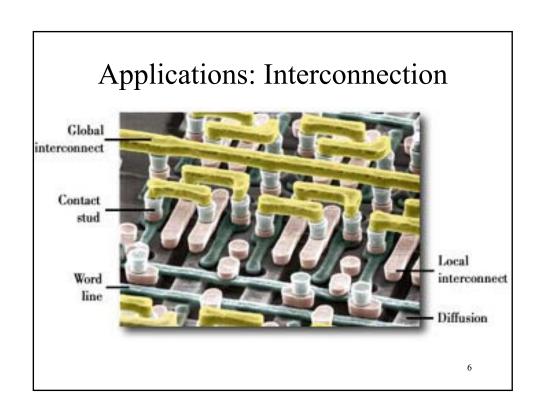
Part I:Thin film applications

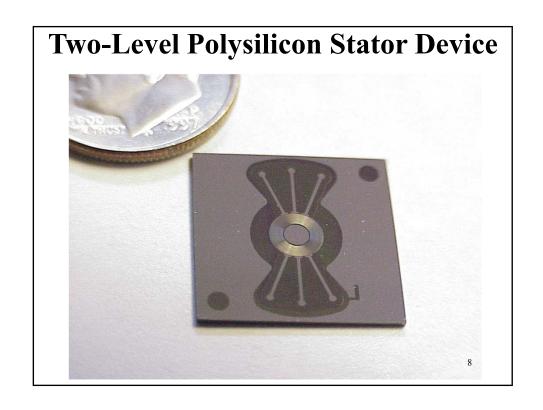
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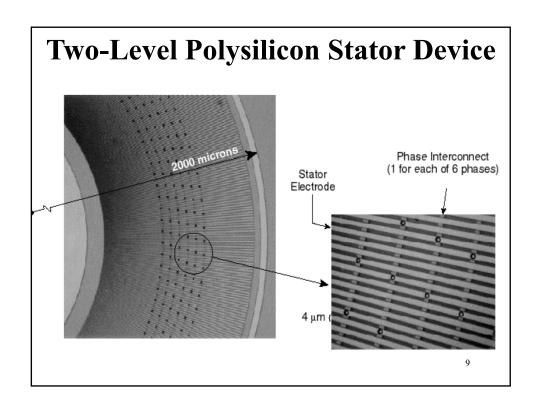
Why Thin Films?

- Thin films are required for many steps in semiconductor fabrication
 - Masks
 - Isolated elements
 - Metal interconnections
 - MOS gate
 - Barriers

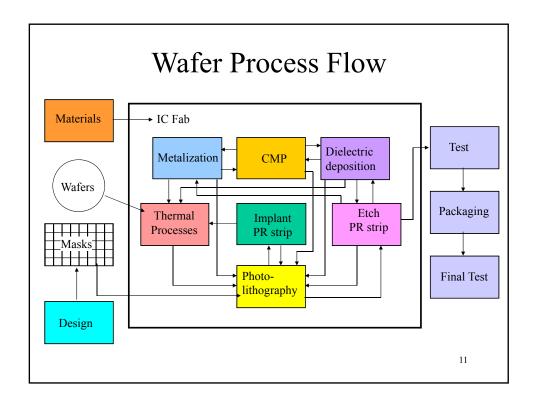






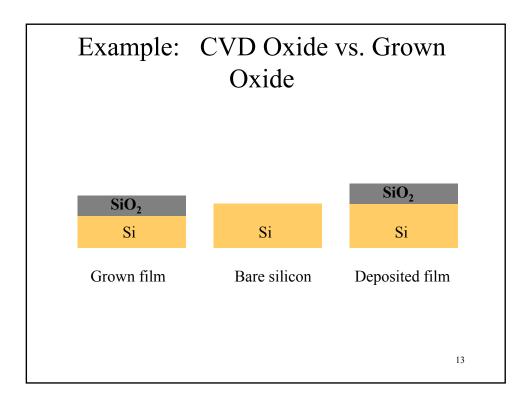


Part II: Overview of Deposition



Applications of Deposited Films

- Gate materials
 - polysilicon
- Conductive layers
 - Metal lines
- Dielectric layers
 - Inter-metallic dielectric (IMD)



Example: CVD Oxide vs. Grown Oxide

Grow CVD

- Oxygen is from gas phase
- Silicon from substrate
- Oxide grow into silicon
- Higher quality
- Both oxygen and silicon are from gas phase
- Deposit on substrate surface
- Lower temperature
- Higher growth rate

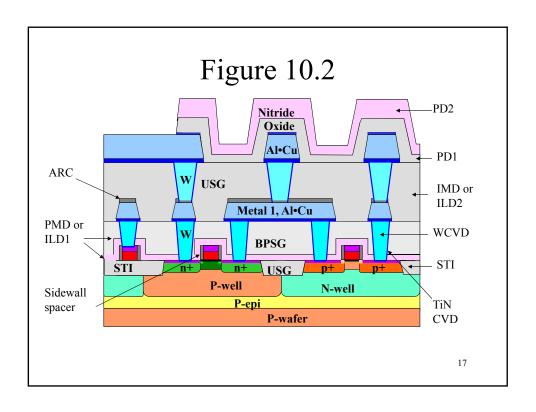
Dielectric Thin Film Applications

- Multi-level metal interconnection
- CVD and SOG plus CVD dielectrics
- Shallow trench isolation (STI)
- Sidewall spacer for salicide, LDD, and the source/drain diffusion buffer
- The passivation dielectric (PD)
- Dielectric ARC for feature size $< 0.25 \mu m$

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Dielectric Thin Film Applications

- Inter layer dielectric, or ILD, include PMD and IMD
- Pre-metal dielectric: PMD
 - normally PSG or BPSG
 - Temperature limited by thermal budget
- Inter-metal dielectric: IMD
 - USG or FSG
 - Normally deposited around 400 °C

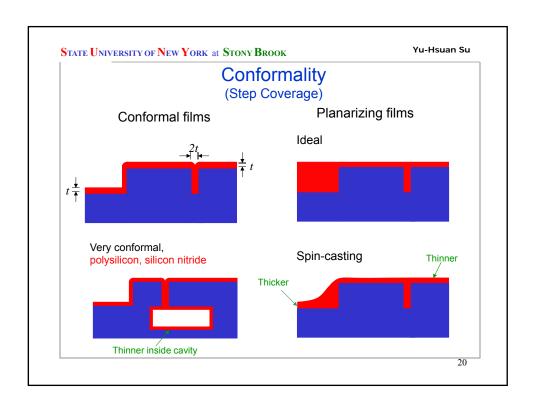


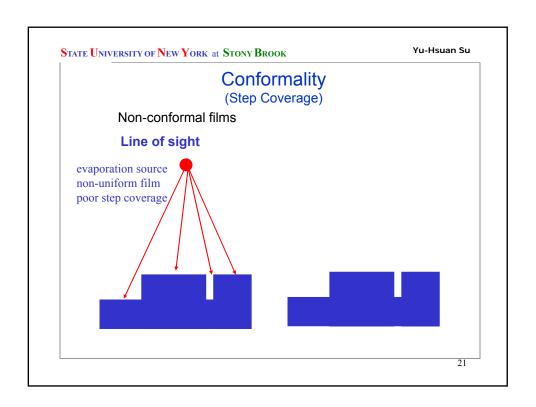
Key Performance Index of Thin Film Processes

- Deposition rate
- Dimension control
- Residual stress
- Comformality
 - Step coverage
 - Flatness
 - Surface roughness
- Adhesion of film to substrate

Key Considerations of Evaporation

- Deposition rate
- Temperature
 - some metals requires excessive high temperature
- Contamination
- Step Coverage
 - an important issue in evaluating performance of deposition, usually poor in evaporation
- Residual stresses
 - usually due to mismatch of CTE and micro structures transformation





Part III: Physical Vapor Deposition

PVD

- Deposit film by physical methods
 - Thermal evaporation
 - Material source is heated to sublimation temperature in a vacuum
 - Material is vapor transported to target in vacuum
 - Deposition is by "line-of-sight" (mean free path = 50m)
 - Sputtering
 - Material is removed from target by momentum transfer
 - Gas molecules are ionized in a glow discharge, ions strike target and remove mainly neutral atoms
 - · Atoms condense on the substrate
 - Vacuum level ~ 10 mtorr --> mean free path ~ 5 mm
 - Easy to deposit alloys

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蒸鍍原理

- 1. 控制蒸氣壓以調節原子到達的數量 $\log P \text{ (torr)} = A (\Delta H_v/2.3RT)$
- 2. 原子到達的能量
 Thermal Energy = 3kT/2
 e.g. 0.1 eV at 500°C
 0.2 eV at 1265°C

真空的要求

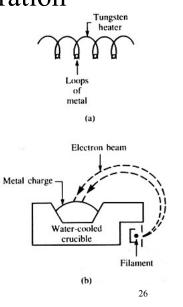
- □ 真空中殘留氣體分子有機會在薄膜成長過程中摻入,基板溫度愈高則機會愈小。
- 国 真空中原子碰撞的平均自由路徑(Mean free path) ,e.g. L=5 cm when $P=10^{-3}$ torr; L=5000 cm when $P=10^{-6}$ torr

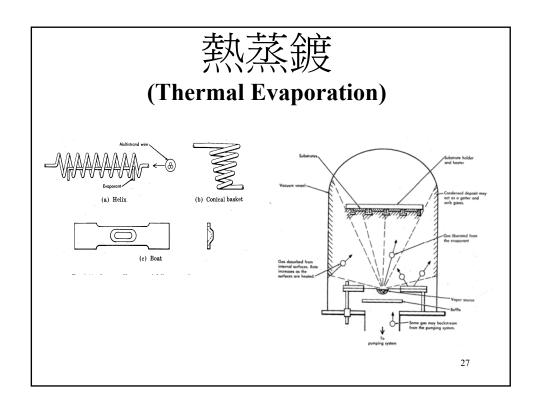
$$L(cm) = \frac{5 \times 10^{-3}}{P(torr)}$$

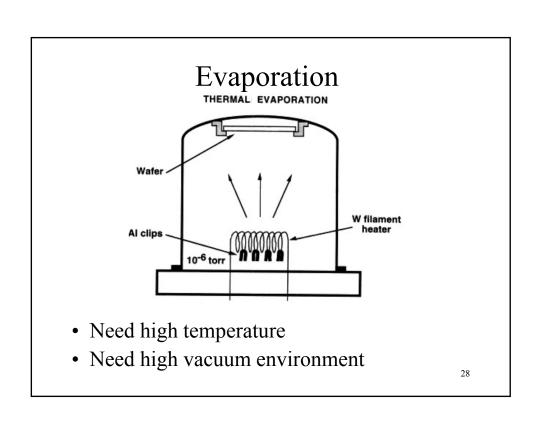
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Type of Evaporation

- Filament evaporation
 - major problems
 - high contamination level
 - hard to form composite films
- Electron-beam evaporation
 - using high density electron beam to evaporate metals
 - dual E-beams with dual target can be used to coevaporate composite materials
 - major problem: radiation damage

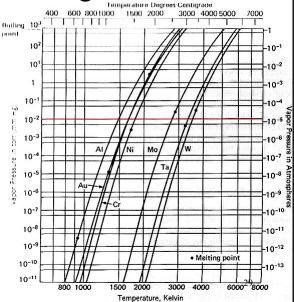








- Usually at least 10 mTorr is required to obtain reasonable deposition rate
- Refractory metals required very high temperature
 - W, Ta, Mo,



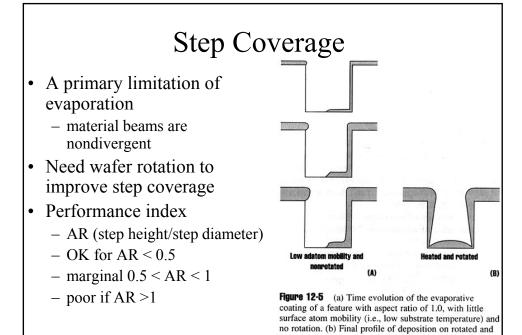
Applications

- Usually widely used in GaAs technology
 - in silicon, it was replaced by sputtering in most fields
- A necessary step for many other applications
 - SEM
 - to form a thin conduction layer on specimen surface
 - LIGA or LIGA-like MEMS processes
 - need to form a conduction layer on PMMA for the subsequent electroplating process
- Since high temperature is required, usually this technology is restricted on metal only

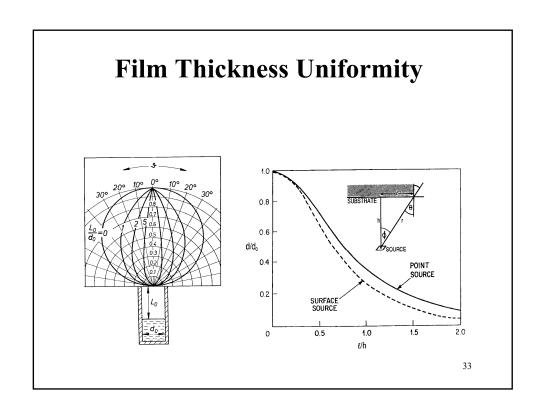
Key Considerations of Evaporation

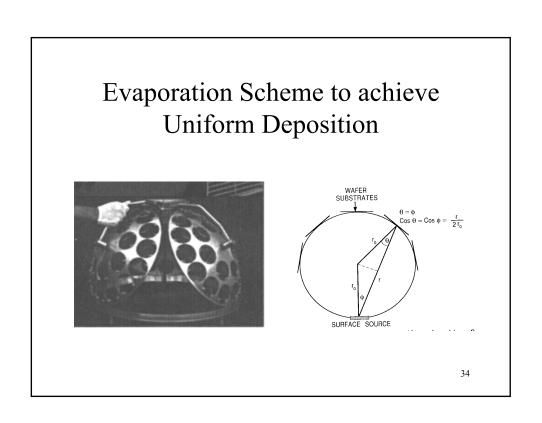
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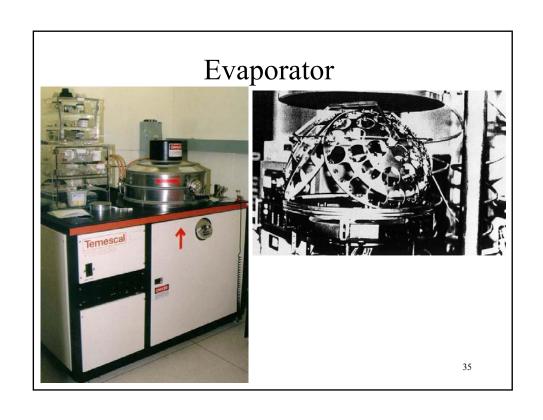
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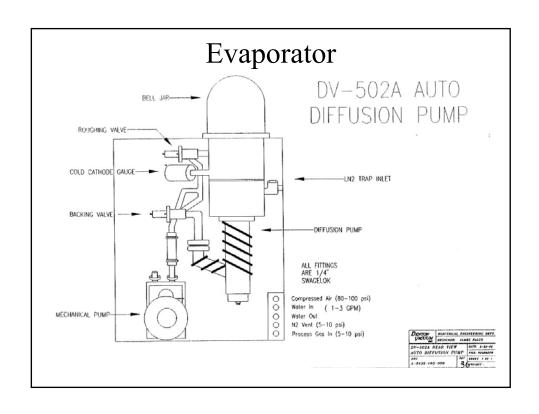


heated substrates.







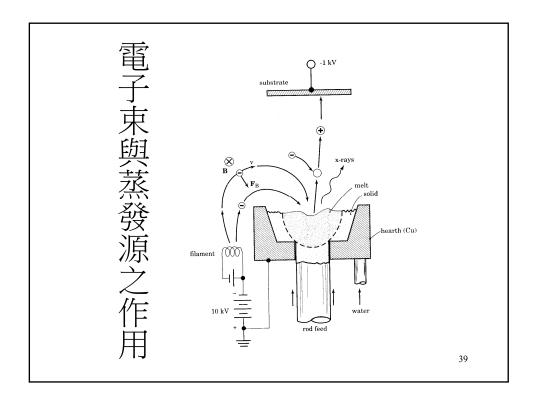


電子束蒸鍍 (E-Beam Evaporation)

- □可聚焦的電子束,能局部加溫元素源, 因不加熱其他部分而避免污染。
- □ 高能量電子束能使高熔點元素達到足夠 高溫以產生適量的蒸氣壓。

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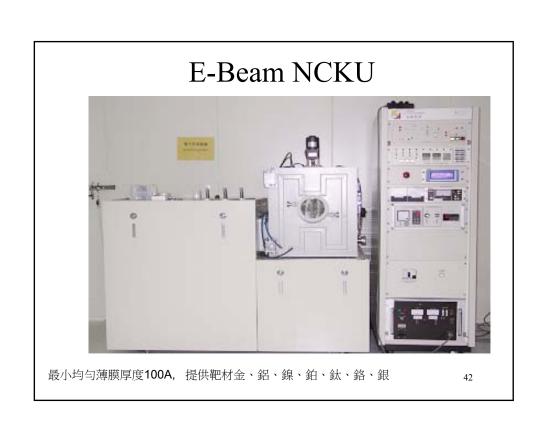
E-Gun Source



Beam-Material Interactions and Operations

- A raster scan of e-beam improves the film uniformity.
- For an accurate deposition-rate control, vapor flux monitoring with feedback control to the filament power should be used.
- The macroparticle-spitting problem may be minimized by using a vacuum-melted charge.
- The energies of vapor atoms are about 0.2 eV.
- X-rays are emitted and may cause charge-trapping defects in the dielectric materials.
- The fraction of vapor ionized varies with beam density and vapor composition and is of the order of 0.01 to 0.1. A negative bias may be applied to the substrate to affect the film structures so-called Ion Plating.



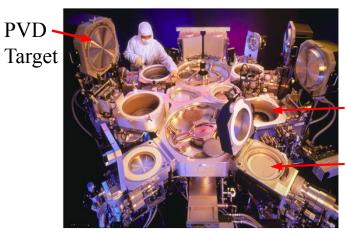


E-Beam NCKU



最小均勻薄膜厚度100A, 提供靶材金、鋁、鎳、鉑、鈦、鉻、銀

Endura® PVD System



PVD Chamber CVD Chamber

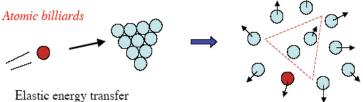
Sputtering

- The major PVD method in silicon technology
- Using ion bombardment to introduce mass transfer
- Basically, it is a low temperature process
 - can deposit virtually any materials, including metals, ceramics, and organic materials
 - can deposit composite film with controllable composition
- Substrate damage is the major disadvantage

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Sputtering process

Ar⁺ impact, momentum transfer at cathode $\Rightarrow e$ avalanche and released target atoms, ions.

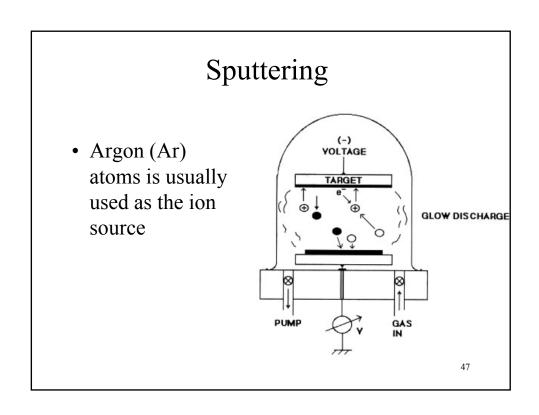


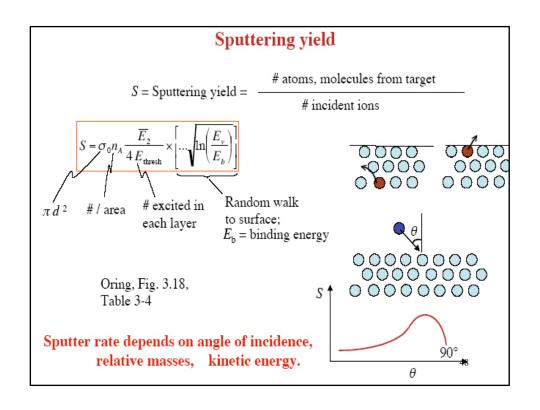
$$\frac{E_2}{E_1} \propto \frac{\frac{4M_1M_2}{(M_1 + M_2)^2} \cos^2 \theta}{E_2 \text{ greatest for } M_1 \cong M_2}$$

For e^- hitting anode, substrate, $M_1 << M_2$

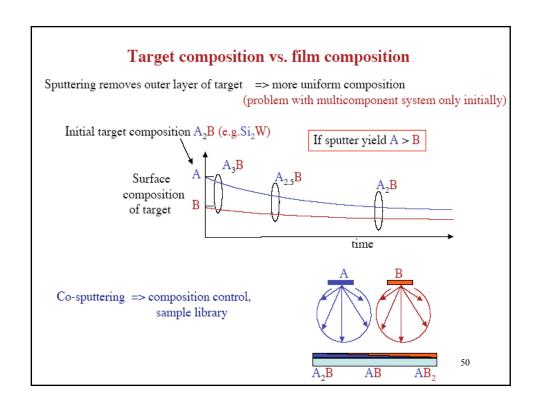
But e^- can give up all its E_K in **in**elastic collision:

$$\frac{1}{2}m_e v_e^2 \Rightarrow \Delta U$$
 Excitation of atom or ion





Sputtering Yield • See Campbell 12.8 for detail 10 Sputtering yield (atoms/Ar ion) • Each material has its sputtering threshold Sputtering threshold $-\sim 10$ - 30 eV Ion energy ↑, sputtering yield \(\) • However, if energy is too high, it becomes 10 103 104 10-1 102 "ion implantation" Normalized ion energy 49



Sputtering vs. Evaporation

- Temperature
 - evaporation requires high temperature
- Materials
 - evaporation: metal only
 - sputtering: virtually any materials
- Sputtering has better step coverage
- contamination problem
- Sputtering results more severe damage in substrate than evaporation

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Sputter



Magnetron Sputter NCKU



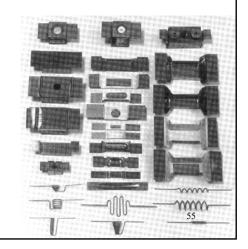
利用磁力控制增加電漿密度,以離子轟擊靶材濺鍍銅、鋅、鉻、鋁、鈦金屬薄膜(DC gun) 與金屬氧化物氧化鋅、二氧化矽薄膜(RF gun)於基板上,最小均勻厚度200A。 53

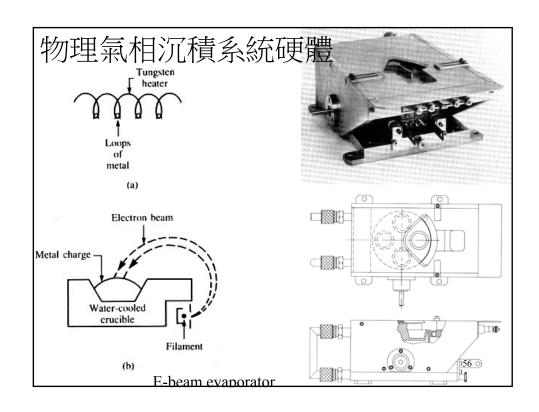
濺鍍(Sputtering) vs. 蒸鍍 (Evaporation)

- Temperature
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物理氣相沉積系統硬體

- Thermal Evaporator
 - Vacuum pump
 - Resistance heated evaporation sources
 - Sublimation furnaces, crucible sources
- E-beam evaporator
 - Vacuum pumps
 - Electron generators
 - Electrical bias system





物理氣相沉積系統硬體

- Sputter
 - Vacuum pump
 - Power supply for DC or RF glow discharge
 - Sputtering gas source

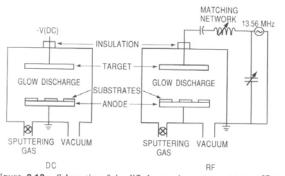
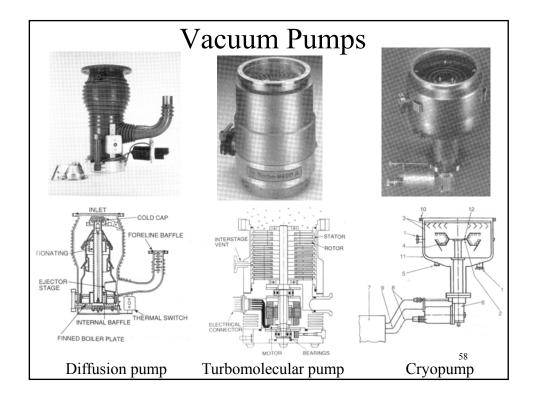
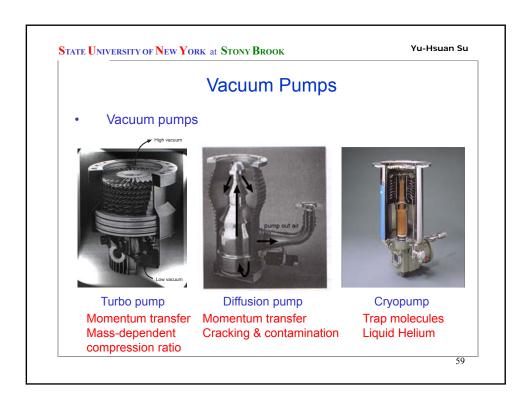
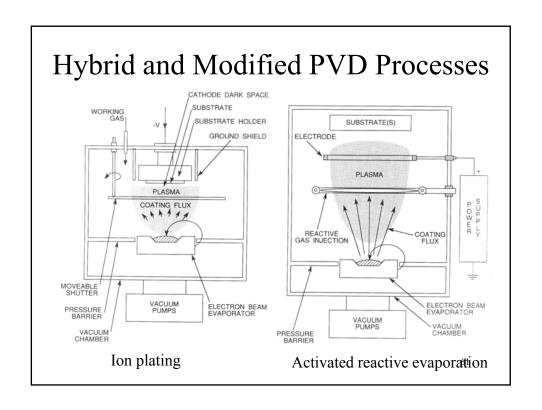


Figure 3-13. Schematics of simplified sputtering systems: (a) dc, (b)/RF.

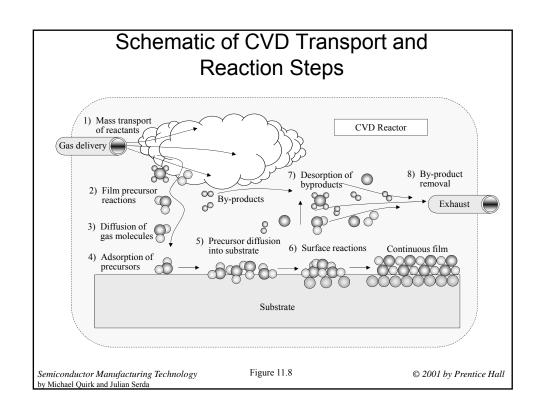


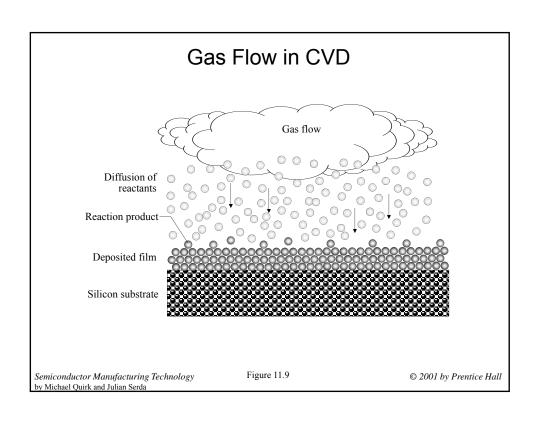


Hybrid and Modified PVD **Processes** • Ion beam assisted deposition SUBSTRATE HOLDER • Use ion bombardment to SHUTTER adjust PVD films • Ion plating - Thermal evaporation + ion supttering PUMP - Provide excellent adhesion Reactive evaporation – Metal vapor reacts with a gas GAS NLET to form compound deposits Ion-beam-assisted deposition



Part IV: Chemical Vapor Deposition



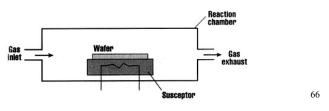


CVD Applications

	FILMS	PRECURSORS
	Si (poly)	SiH ₄ (silane)
Semiconductor		SiCl ₂ H ₂ (DCS)
	Si (epi)	SiCl ₃ H (TCS)
		SiCl ₄ (Siltet)
		LPCVD SiH ₄ , O ₂
	SiO ₂ (glass)	PECVD SiH ₄ , N ₂ O
Dielectrics		PECVD Si(OC ₂ H ₅) ₄ (TEOS), O ₂
		LPCVD TEOS
		APCVD&SACVD TM TEOS, O ₃ (ozone)
	Oxynitride	SiH ₄ , N ₂ O, N ₂ , NH ₃
	a:	PECVD SiH ₄ , N ₂ , NH ₃
	Si ₃ N ₄	$\begin{array}{ccc} \text{LPCVD} & \text{SiH}_4, \text{N}_2, \text{NH}_3 \\ \text{LPCVD} & \text{C}_8\text{H}_{22}\text{N}_2\text{Si} \left(\text{BTBAS} \right) \end{array}$
		10 22 21 (11)
	W (Tungsten)	WF ₆ (Tungsten hexafluoride), SiH ₄ , H ₂
	WSi_2	WF ₆ (Tungsten hexafluoride), SiH ₄ , H ₂
Conductors	TiN	$Ti[N (CH_3)_2]_4 (TDMAT)$
	Ti	TiCl ₄
	Cu	64

Introduction to CVD

- Form thin films on the surface of a substrate by thermal decomposition and/or reaction of gaseous compounds
- Usually performed at high temperature
 - Can be performed at various pressure and with assistance of plasma
 - Usually at viscous flow regime



Type of CVD

- APCVD
 - high deposition rate, poor uniformity, high contamination level, 250-450 °C
 - for dielectrics
- LPCVD
 - low deposition rate, high uniformity, 575-650 °C
 - for polysilicon
- PECVD
 - for extremely low deposition temperature
 - e.g, oxide and nitride
 - quality is poor

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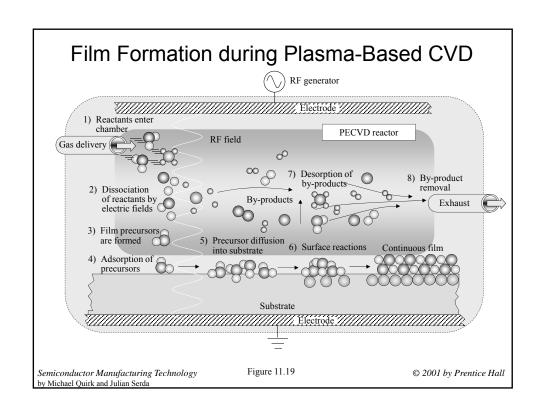
Plasma Enhanced CVD

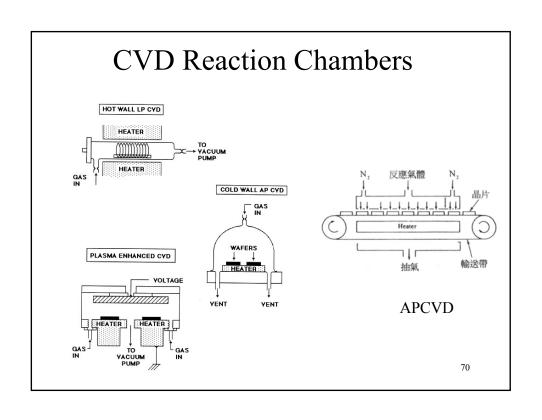
• PECVD with SiH₄ and NO₂ (laughing gas)

$$e^- + SiH_4 \rightarrow SiH_2 + 2H + e^-$$

 $e^- + N_2O \rightarrow N_2 + O + e^-$
 $SiH_2 + 3O \rightarrow SiO_2 + H_2O$

- Plasma enhanced chemical reaction
- PECVD can achieve high deposition rate at relatively lower temperature





Chemical Vapor Deposition Tool

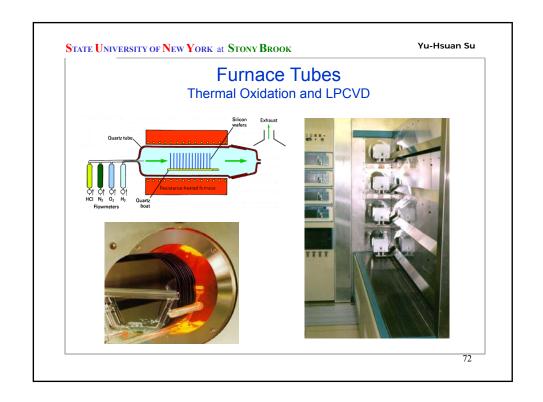


Photograph courtesy of Novellus, Sequel CVD

Semiconductor Manufacturing Technology by Michael Quirk and Julian Serda

Photo 11.3

© 2001 by Prentice Hall



LPCVD NCKU

(1)多晶矽,主要氣體 SiH4、N2;溫度約 620℃、壓力約300 mtorr,

(2)低應力氮化矽,主要氣體 NH3、DSC、N2;溫度約810℃、 壓力約200 mtorr

(3)低溫氧化矽,主要 氣體 SiH4、O2、N2; 溫度約450°C、壓力約 200 mtorr



STATE UNIVERSITY OF NEW YORK at STONY BROOK

Poly-Silicon LPCVD

Process:

 $\mathrm{SiH_4,\,H_2\colon\ SiH_{4\,(g)}} \rightarrow \mathrm{SiH_{2\,(g)}} + \mathrm{H_{2\,(g)}}\,,\ \frac{\mathrm{SiH_{2\,(a)}}}{\mathrm{SiH_{2\,(a)}}} \rightarrow \mathrm{Si\,_{(s)}} + \mathrm{H_{2\,(g)}}$

pressure :~500 mtorr.

temp.: 540 °C(amorphous) ~ 650 °C (polysilicon, 0.03~0.3 μ m)

dep. rate: 100~200 Å/min.

Conducting poly-Si: PH_3 , AsH_3 reduce deposition rate, while B_2H_6 increases deposition rate.

Conformality: spectacular, excellent uniformity

Compatibility: thermal issue (no Al)

SiH₄ is extremely toxic gas and extremely *flammable* at atmospheric

pressure! (Osaka University)

amorphous Si (solar cells & TFT), poly-Si (LCD projector)

STATE UNIVERSITY OF NEW YORK at STONY BROOK

LTO LPCVD

Process:

 $SiH_4,\ O_2\ \ (PH_3,AsH_3,\ B_2H_6)$ Note SiH_4 and $\ O_2$ at 350 mtorr. will not explode!

pressure :~350 mtorr. temp.: 300 $^{\circ}\text{C}$ ~ 400 $^{\circ}$ C

dep. rate: 175 Å/min. for 4" wafer

Planarization \rightarrow flow \rightarrow add dopants to reduce melting point

PSG (Phosphosilicate): phosphine doped LTO

 $(6\sim8\% \text{ wt., } 1000 \,^{\circ} \text{ C})$, "gettering" of small positive ions BPSG (Borophosphosilicate):

(4~5% wt. each, 900°C), contact dielectrics

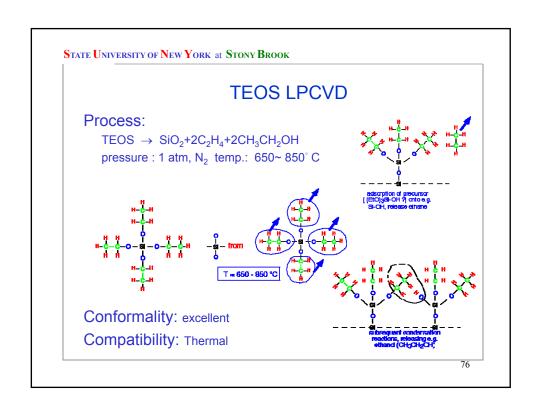
flow process may be replaced by CMP planarization

Conformality: not so good (P glass reflow)

Compatibility: good for Aluminum

 PH_3 , AsH_3 , B_2H_6 are all extreme toxic gases, usually shipped with

SiH₄ for safety (explosive alert !).



STATE UNIVERSITY OF NEW YORK at STONY BROOK

Nitride LPCVD

Process:

 SiH_4 , $SiCl_2H_2$, NH_3 pressure :~250 mtorr. temp.: ~ 800 $^{\circ}$ C dep. rate: 35 Å/min. for 4" wafer 1100MPa tensile stress

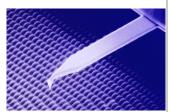
 $3SiH_4 + 4NH_3 \rightarrow Si_3N_4 + 12H_2 \ (700 \sim 900\ ^{\circ}\ C\)$ $3SiCl_2H_2 + 4NH_3 \rightarrow Si_3N_4 + 6HCl + 6H_2$

(700~800 °C)

Conformality: good to excellent Compatibility: Thermal

Olympus, Tetra Tip





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Oxide

· Silane based

$$SiH_4 + O_2 \rightarrow (300 - 500^{\circ}C) SiO_2 + 2H_2$$

• PSG

$$4PH_3 + 5O_2 \rightarrow 2P_2O_5 + 6H_2$$

• TEOS (LPCVD 650-700 °C) (PECVD 350

 $Si(OC_2H_5)_4 \rightarrow SiO_2 + by \ product$

Silane based PECVD

$$SiH_{4(g)} + 2N_2O_{(g)} \xrightarrow{RF \atop \Delta} SiO_{2(s)} + 2N_{2(g)} + 2H_{2(g)}$$

Oxide

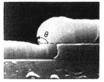
Table 6.1 Properties of Various Deposited Oxides. (After ref. [2].)

Source	Deposition Temperature (°C)	Composition	Conformal Step Coverage	Dielectric Strength (MV/cm)	Etch Rate (Å/min) [100:1 H ₂ O:HF]
Silane	450	SiO ₂ (H)	No	8	60
Dichlorosilane	900	SiO ₂ (Cl)	Yes	10	30
TEOS	700	SiO ₂	Yes	10	30
Plasma	200	SiO, 9(H)	No	5	400

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PSG and BPSG

- Phosphosilicate glass (PSG)
 - reducing stress
 - improve step coverage
 - flow at high temperature (1000-1100 °C) to create smooth surface
- Borophosphosilicate glass (BPSG)
 - flow temperature is reduced to 700 °C
 - for isolation and surface planarization





(a) 0% P

(b) 2.2%

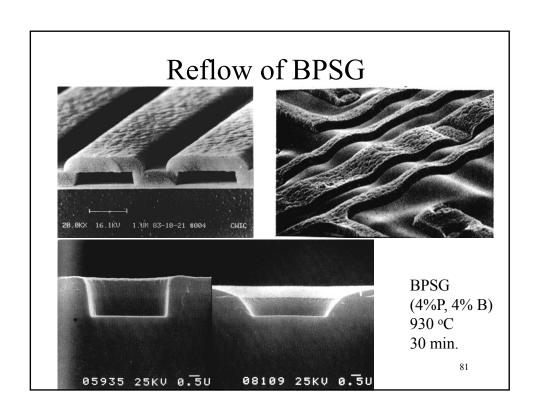


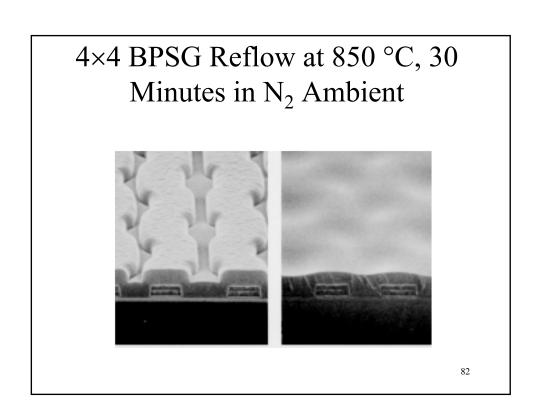
Λθ.

(c) 4.6%

7.2%

1100 °C for 20 min.





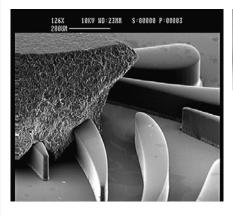
Silicon Nitride

- Application:
 - masks to prevent oxidation for LOCOS process
 - final passivation barrier for moisture and sodium contamination
- APCVD $3SiH_4 + 4NH_3 \rightarrow Si_3N_4 + 12H_2$
- LPCVD

$$3SiCl_2H_2 + 4NH_3 \rightarrow Si_3N_4 + 6HCl + 6H_2$$

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CVD SILICON CARBIDE PROCESSING

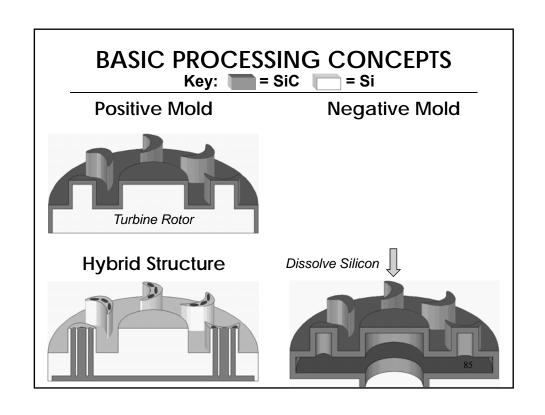


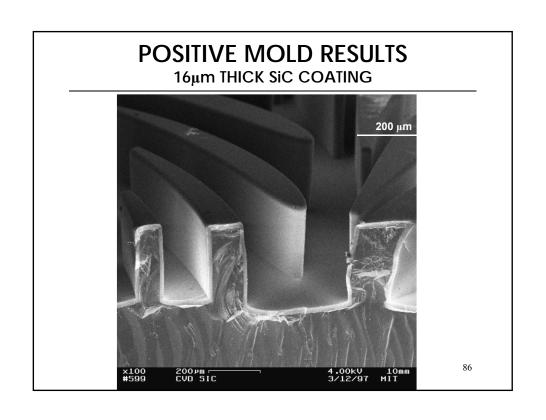


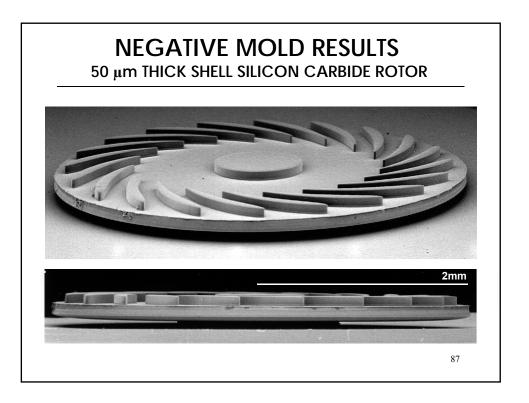
Procedure:

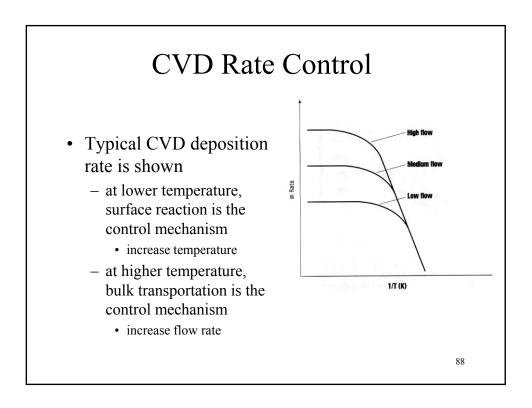
- 1. DRIE silicon substrate
- 2. Wafer bonding
- 3. CVD silicon carbide
- 4. Etch silicon

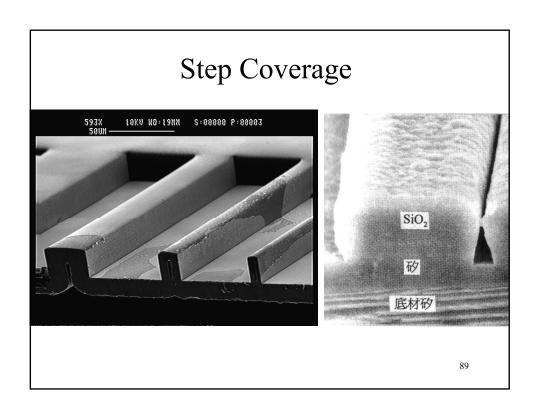
Lohner, MIT 1999

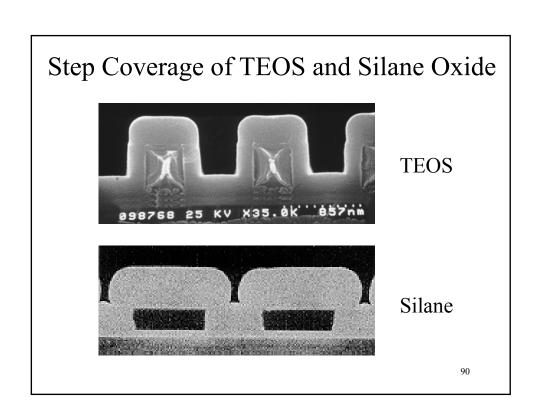


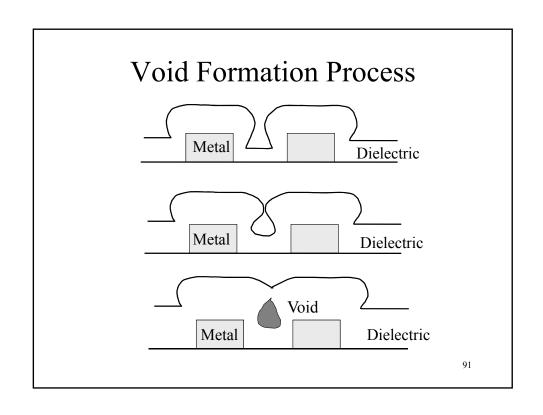


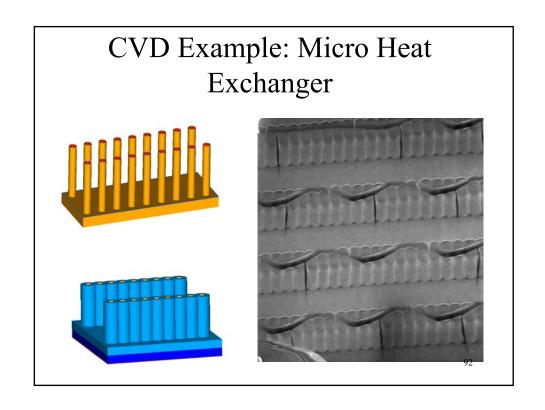


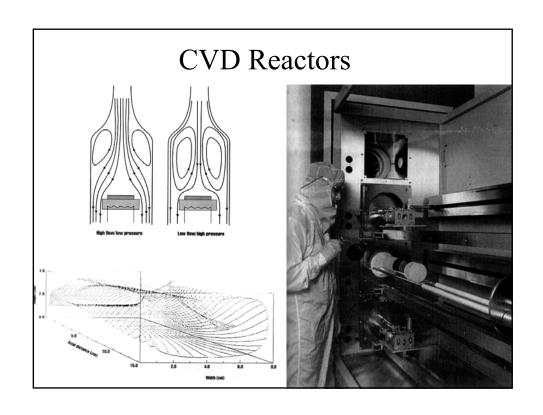






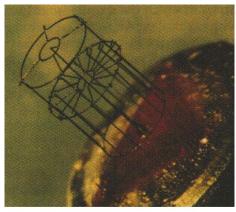






Laser Assisted Deposition Pyrolytic By heating substrate to enhance reactivity Photolytic Direct dissociation of molecules by energetic photons PHOTOLYTIC PHOTOLYTIC

LCVD



 $\label{eq:Figure 6.} \textbf{A 3-dimensional structure of alumina} grown with the LCVD process.$

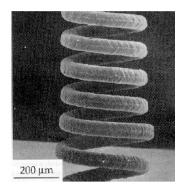


Figure 7. Tungsten coil made by LCVD.

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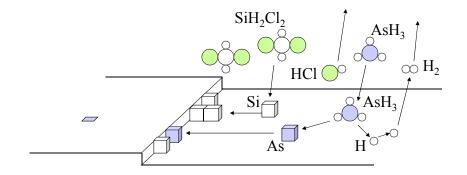
Part V: Epitaxial Growth

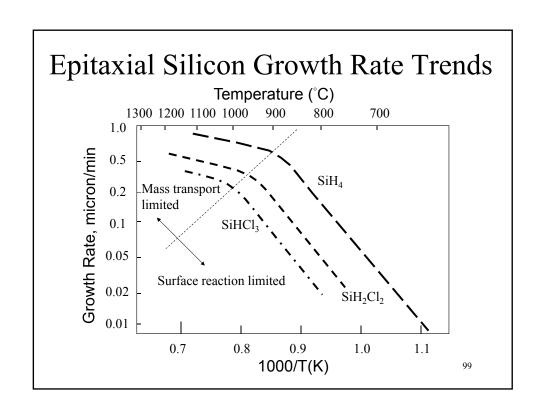
Epitaxy

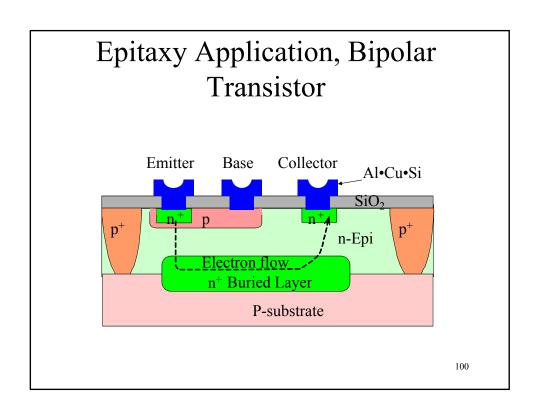
- Deposition of single crystal films on single crystal substrate
 - Single silicon acts as a seed
 - This is called vapor phase epitaxy (VPE)
 - Other technologies include liquid phase epitaxy (LPE) and molecular beam epitaxy (MBE), they are widely used in GaAs process
- To grow single crystal n-type layers on p-type substrates for bipolar processing
- Prevent CMOS latchup

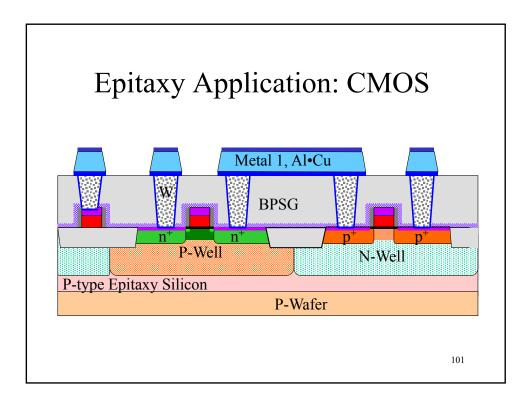
97

Schematic of DCS Epi Grow and Arsenic Doping Process



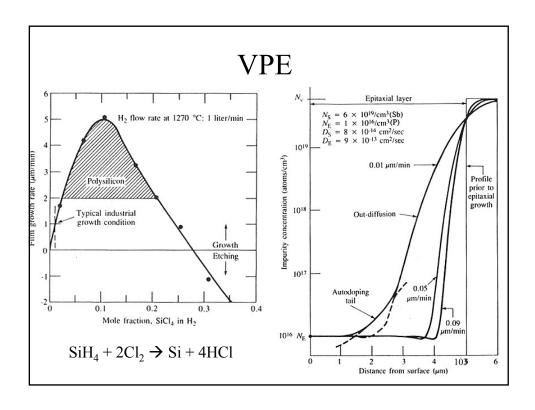






VPE (Vapor Phase Epitaxy)

- Single silicon from single silicon
- Use the same raw materials as polysilicon CVD
- Careful control is required to prevent polysilicon formation
- Resistivity can be adjusted by doping
- Buried layer
 - Heavily doped layer first, then epitaxy layer
 - Auto doping
 - Out-diffusion



LPE and MBE

• LPE:

- Substrate is brought into contact with a solution containing the material to be deposited in liquid form
- Material crystallizes directly from the solute
- $-0.1 1 \mu m/min.$

• MBE:

- Crystalline layer is formed by deposition from a thermal beam of atoms or molecules
- Deposition is performed in ultrahigh-vacuum condition (10-8 Pa)
- $-0.001-0.3 \mu m/min$

CVD Vs. Epitaxy

- Crystalline
 - CVD: Polysilicon or amorphous
 - Epitaxy: single crystal
- Fluid dynamics
 - CVD: viscous flow regime
 - Epitaxy: molecular flow regime
- Technology
 - CVD: quite popular in Si technology
 - Epitaxy: mostly used in GaAs and BJT technology

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Part VI: Thin Film Materials Sciences

Step Coverage

- A primary limitation of evaporation
 - material beams are nondivergent
- Need wafer rotation to improve step coverage
- Performance index
 - AR (step height/step diame)
 - OK for AR < 0.5
 - marginal 0.5 < AR < 1
 - poor if AR >1

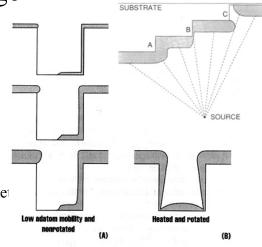
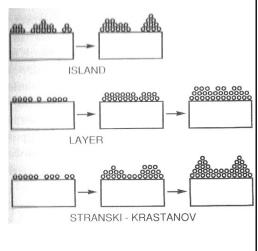
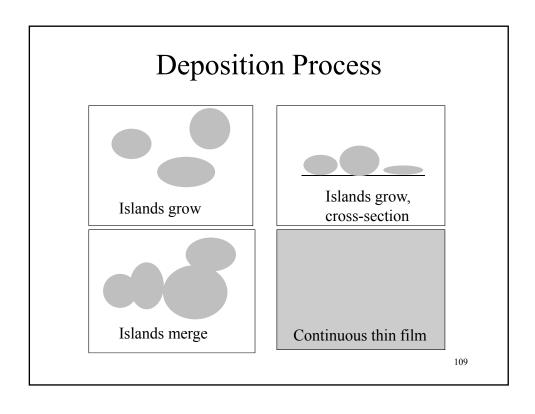


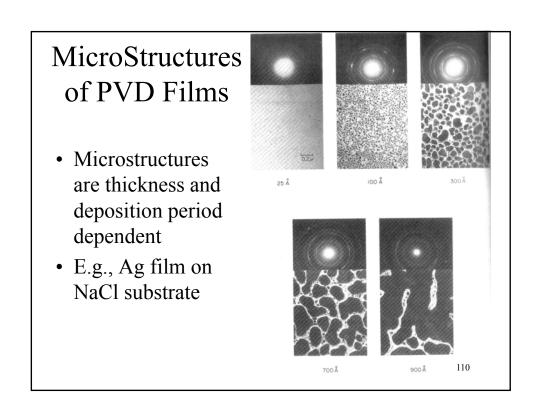
Figure 12-5 (a) Time evolution of the evaporative coating of a feature with aspect ratio of 1.0, with little surface atom mobility (i.e., low substrate temperature) and no rotation. (b) Final profile of deposition on rotated and heated substrates.

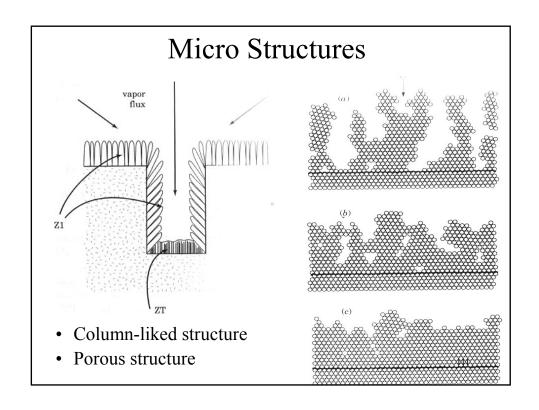
Thin Film Growth Modes

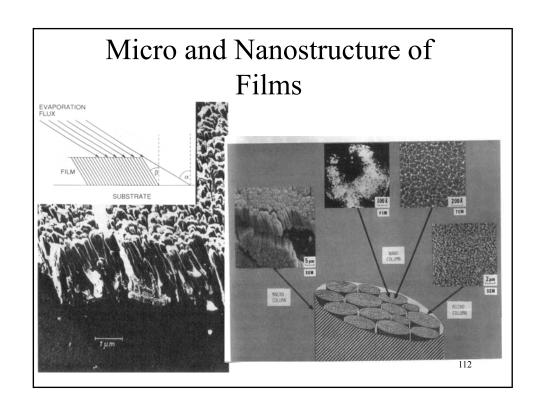
- Island (Volmer-Weber)
 - Film materials have stronger bonds
 - E.g., metals on insulators
- Layer (Frank-Van der Merwe)
 - Film / substrate has stronger bonds
 - E.g., epi of semiconductor
- Stranski-Krastanov



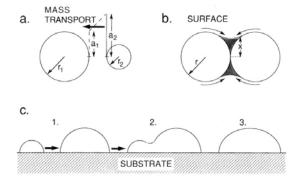








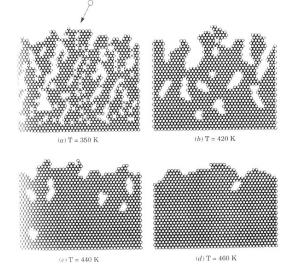
Coalescence of Clusters and Stress Generation



• Cluster migration causes the elimination of grain boundary and induces tensile strains

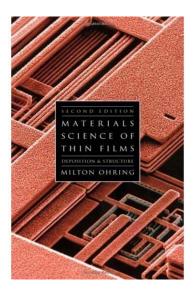
113

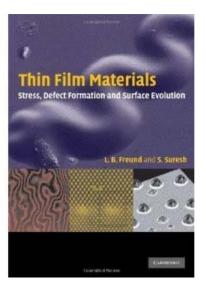
Molecular Dynamics Simulation



Low substrate temperature will result in porous structure

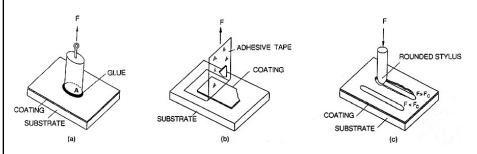
Two Good Textbooks





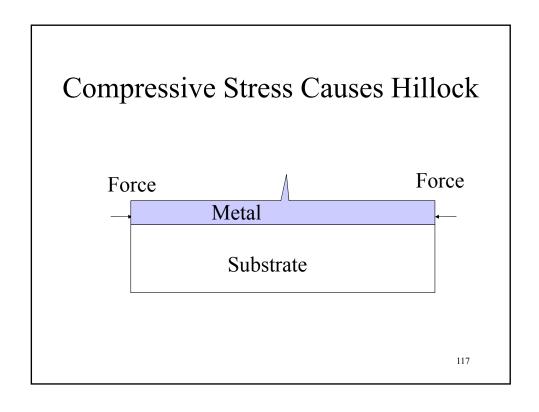
115

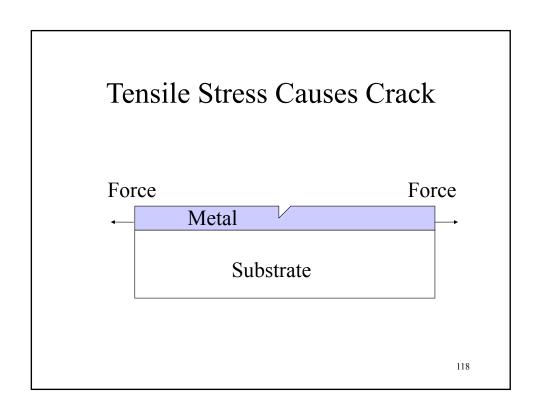
Adhesion



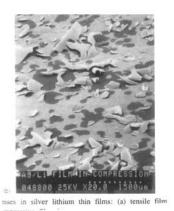
(a)pull test, (b)shear test, (c)scratch test

• Metal film must have enough adhesion force to substrate





Failure of Thin Films



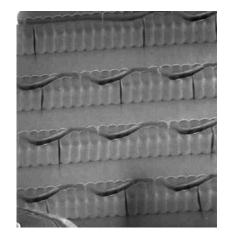
- Compressive failure
 - Bulking
 - Hillocks formation

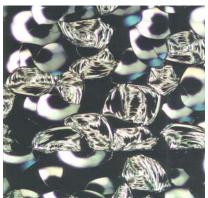


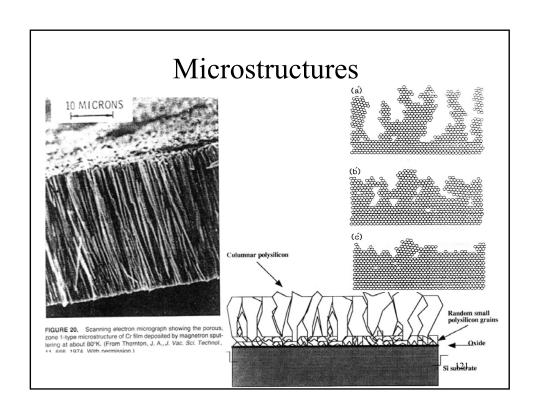
- tensile failure
 - Cracking

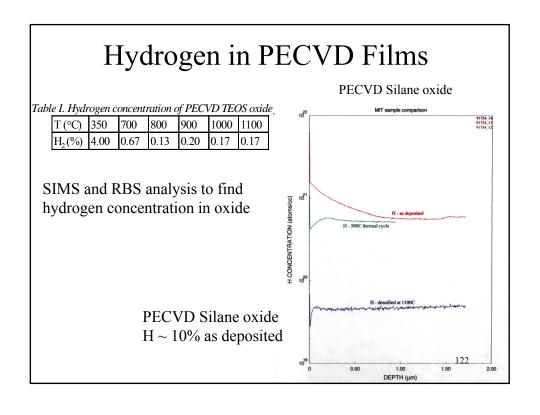
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Failure of Oxide Films









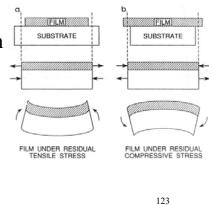
Stoney Formula

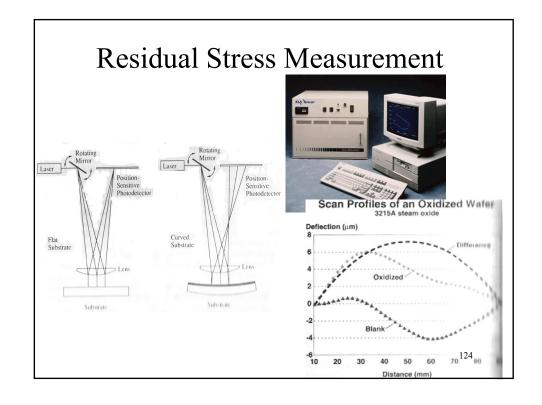
• Simple 1-D thermal stress

$$\sigma = (\alpha_f - \alpha_s)(T_1 - T_2)E_f/(1 - \nu_f)$$

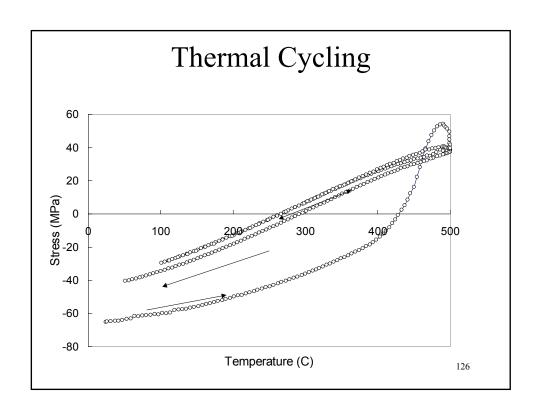
- Stoney formula for thin film material
 - by curvature measurement to find residual stress

$$\sigma_{\rm f} = \frac{E_{s}h_{s}^{3}}{6(1-\nu_{s})Rh_{\rm f}^{2}(1+h_{s}/h_{\rm f})}$$

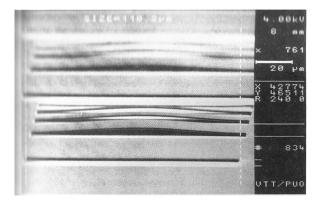






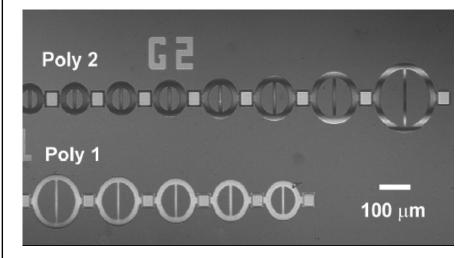


Buckling



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薄膜製程殘留應力量測



Guckel's Rings: for tensile residual stress evaluation

