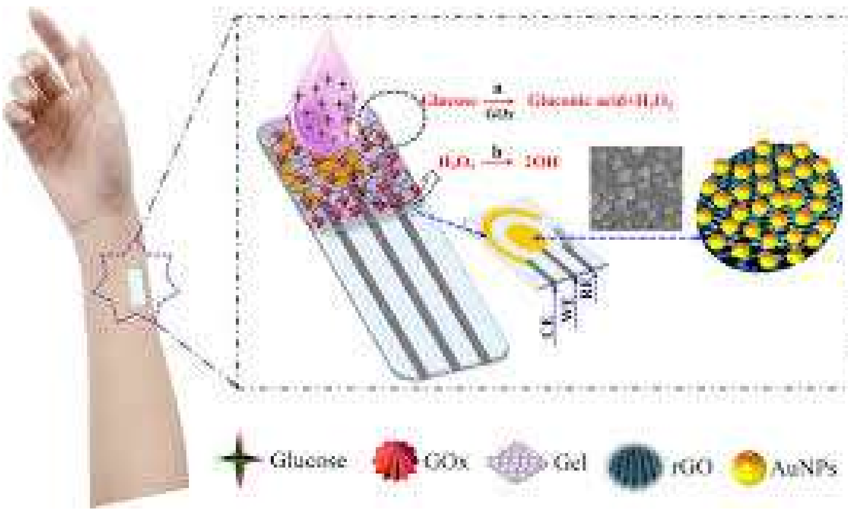
Biosensors

B. Tech.
Course No.: EEL 3050
L-T-P [C]: 3–0–2 [4]

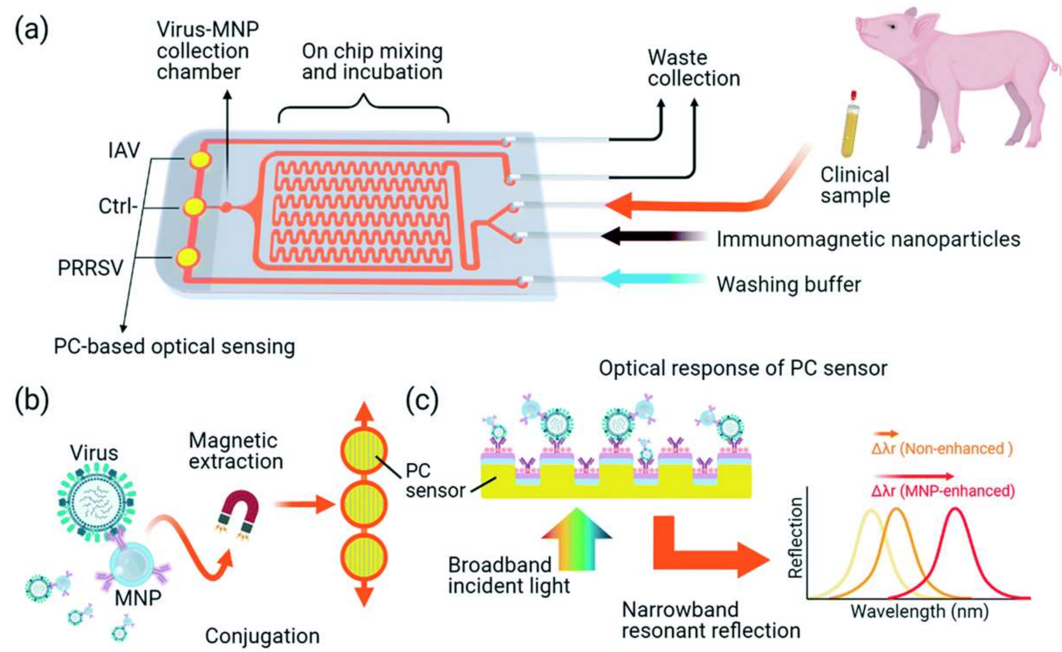
Prof. AJAY AGARWAL
ELECTRICAL ENGINEERING
IIT JODHPUR

Lecture 18 dated 23rd Sep. 2024

Biosensors



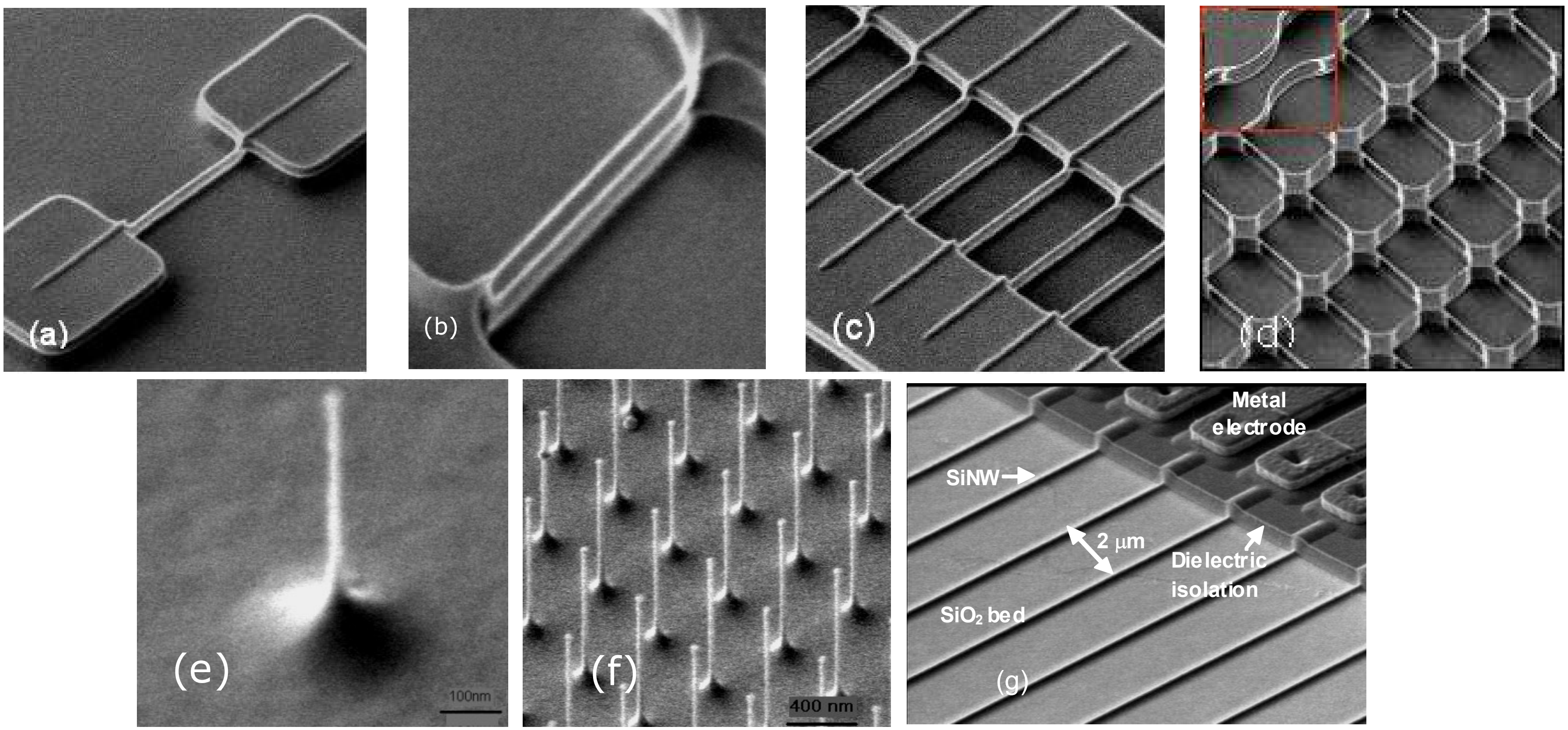
Wearable Glucose Biosensor



Microfluidic Biosensor for virus detection

Nano-structures/ Nano-materials

1- D nanostructures



Silicon nanowire: (a) Single SiNW, (b) vertically stacked twin SiNWs (c) An array of SiNW (d) large area regular mesh of nanowires. The inset: curved SiNW (e) & (f) 1.0 μm tall isolated & dense-array of vertical SiNW of dia. ~ 20 nm (g) **SiNW array for bio-chemical sensors**; length-to-cross section ratio up to 40,000:1.

Nano-structure array for *SERS*

ELSEVIER

Sensors and Actuators A: Physical

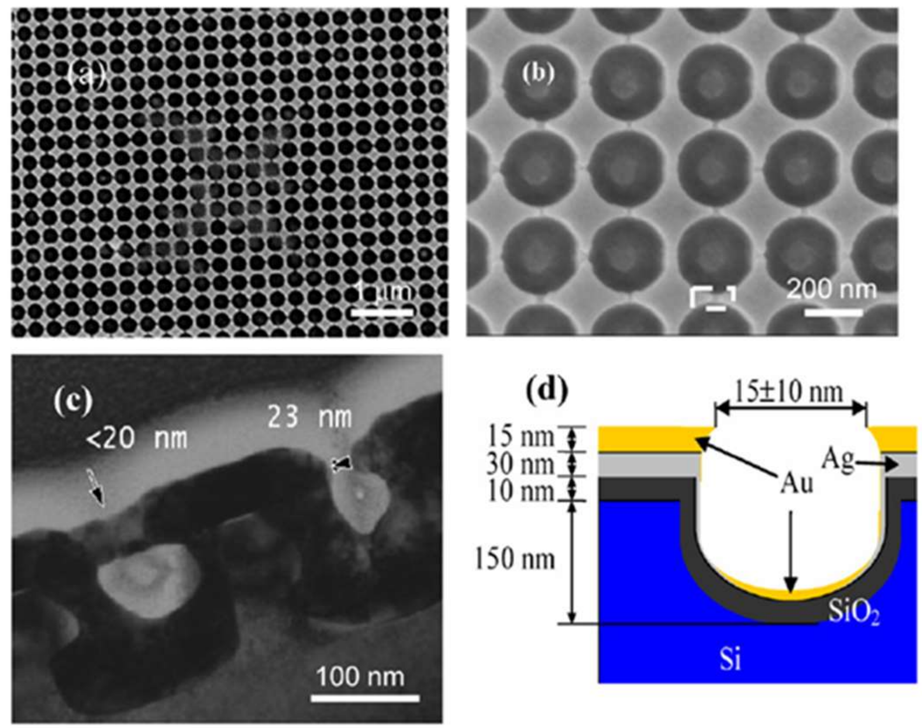
Volume 139, Issues 1–2, 12 September 2007, Pages 36-41

3D arrays of SERS substrate for ultrasensitive molecular detection

R.Z. Tan [a], A. Agarwal [a], N. Balasubramanian [a], D.L. Kwong [a], Y. Jiang [b], E. Widjaja [b], M. Garland [b]

Suitable for *trace level detection* of:

- **Biological warfare,**
- **Bio-markers,**
- **Explosives, etc.**



SERS substrates

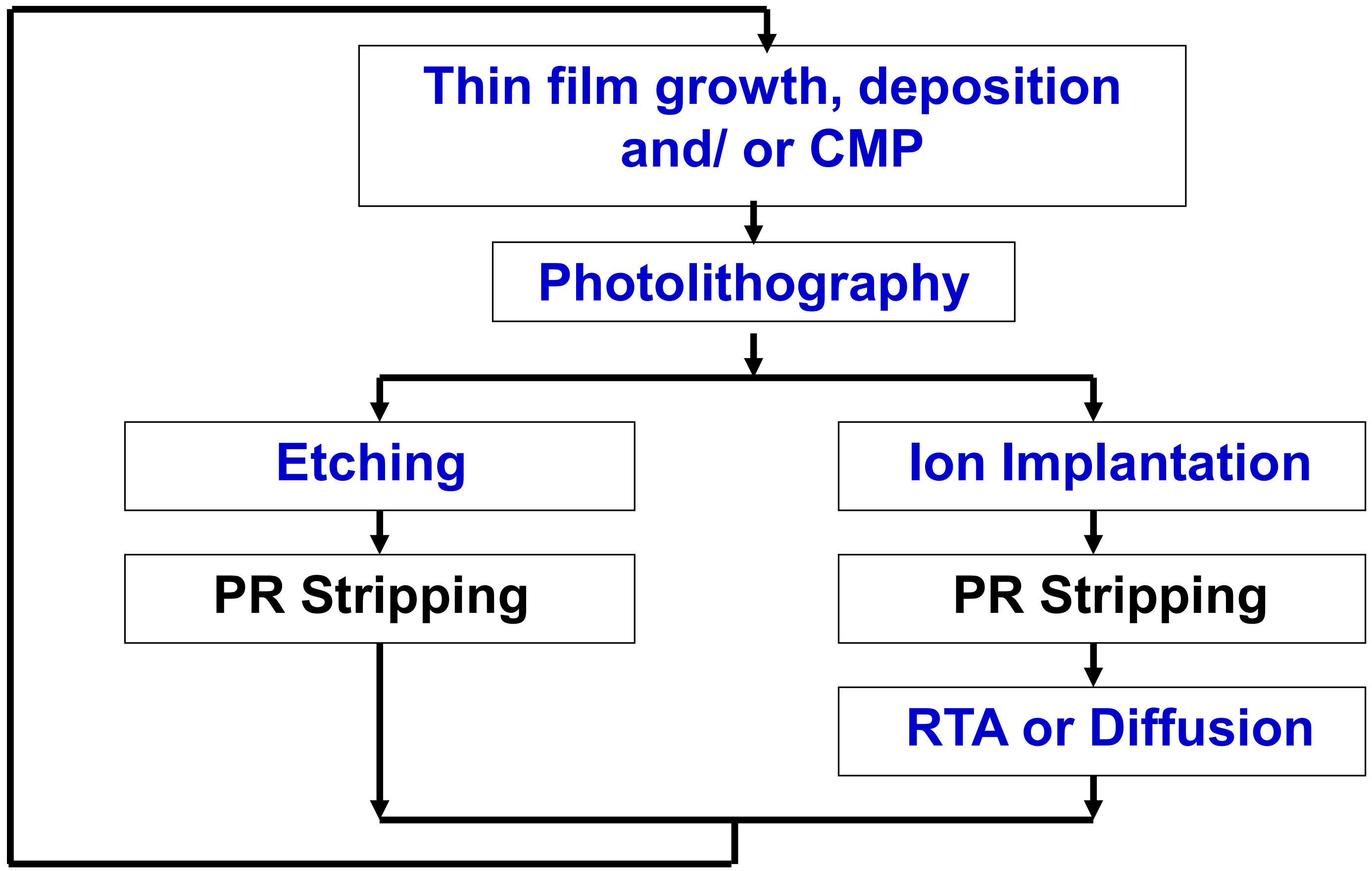
How are Biosensors made?

- Using micro fabrication techniques …

What is the substrate material for biosensors?

- **Silicon/ Silicon oxide**
- **Glass**
- **Polymers**
- **Paper, etc.**

Sensor Fabrication Processes



Photolithography

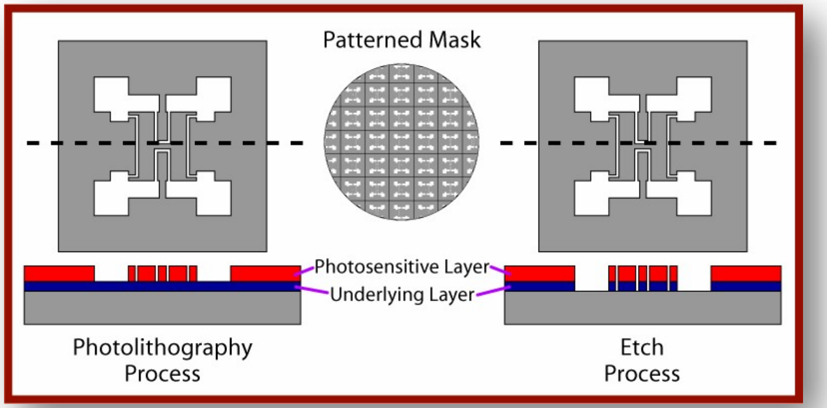
Lithography (from Greek - lithos: "stone" + grapho: "to write")

Lithography is a process used to selectively remove parts of a thin film.

It is an important process for industrial mass production of computer chips

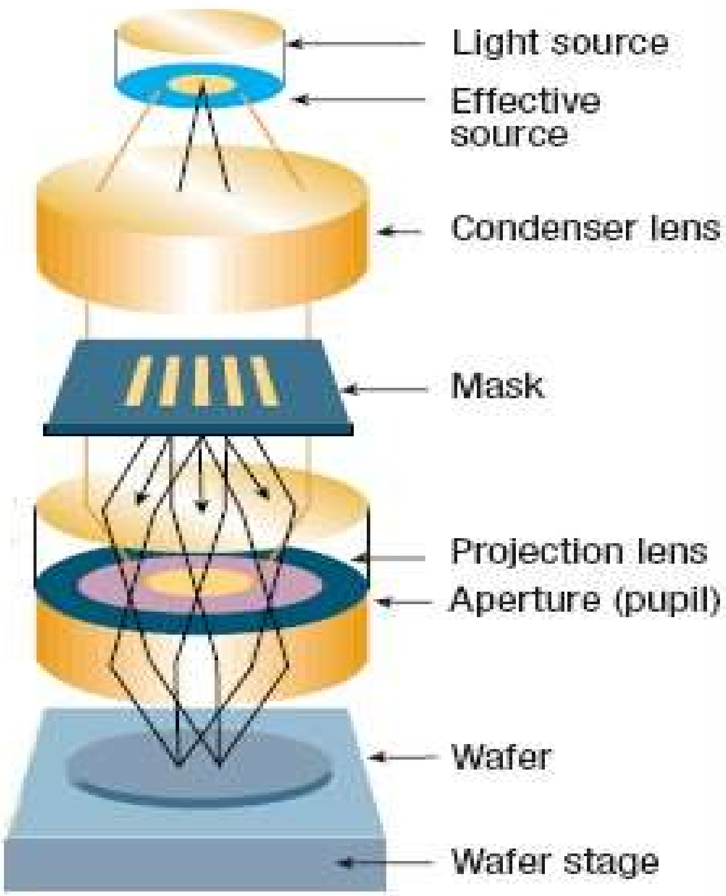
Different layers of a microsystem are realized as follows:

1. Photolithography transfers the pattern from a mask to a photosensitive layer
2. Then the pattern from the photosensitive layer is transferred into an underlying layer.
3. After the pattern transfer, the resist is stripped (removed).



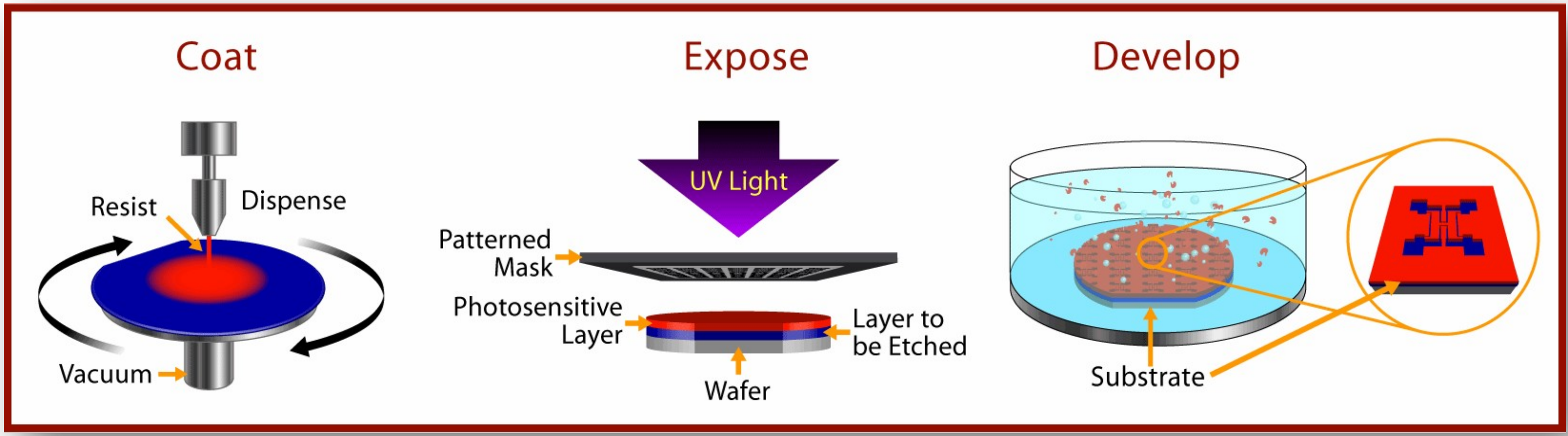
Pattern Transfer to Underlying Layer

The Optical System



Three Steps of Photolithography (*broadly*)

1. PR Coating
2. UV Exposure
3. Develop

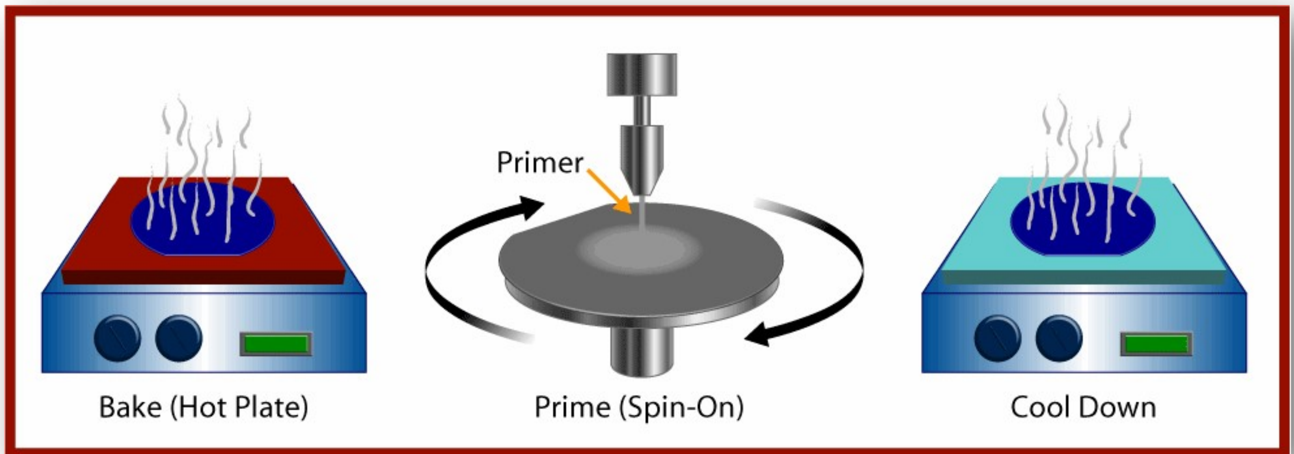


1. Surface Conditioning

In most applications, surface conditioning is required preceding to the photoresist coating

- Surface cleaning to prepares the wafer to accept the photoresist
- Coating the wafer with a chemical that boosts adhesion of the photoresist to the wafer's surface (commonly used is Hexa-methyl-di-salizane or HMDS)

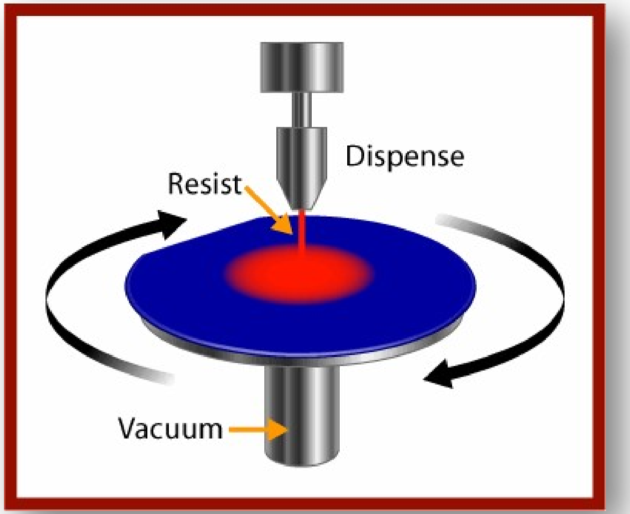
Surface Conditioning Steps



1. Wafer is baked to remove the water molecules on the wafer surface
2. HMDS is applied (prime) to create a hydrophobic surface
3. Wafer is cooled to room temperature

2. PR Spin Coating

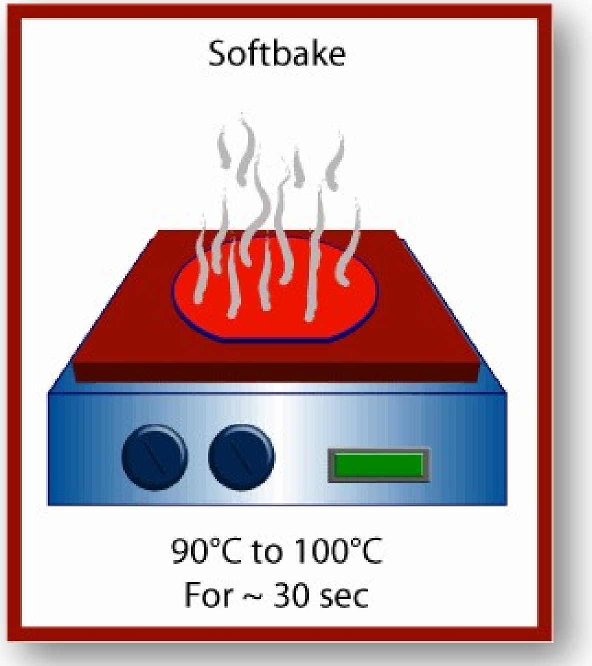
- Wafer is placed on a vacuum chuck
- Vacuum holds the wafer on the chuck
- Resist is applied
- Chuck accelerates for desired resist thickness
- Chuck continues to spin to dry film



PR Spin Coating

3. Soft bake

- After the photoresist is coated in the desired thickness, a softbake is used to remove the residual solvents of the photoresist
- After the softbake, the wafer is cooled to room temperature



Softbake after applying Resist

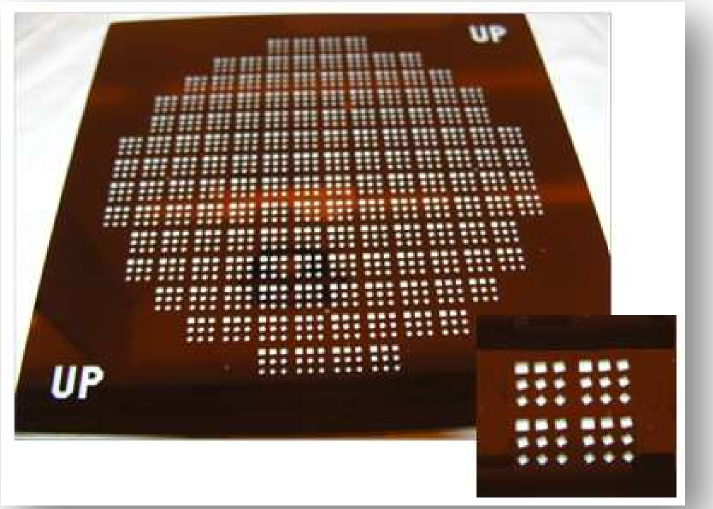
4. Alignment

- Alignment is one of the most critical steps in the entire micro-systems fabrication process
- A misalignment of one micron or smaller can destroy the device and all the devices on the wafer
- Each layer must be aligned properly and within specifications to the previous layers & subsequent layers

Mask *Aligner*

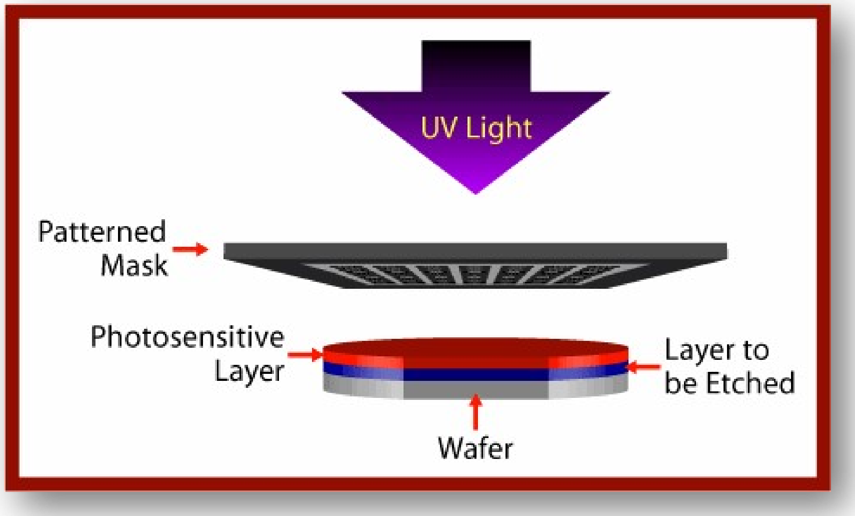
Mask vs. Reticle

- The patterned mask (or reticle) is a quartz or glass plate with the desired pattern (usually in chrome).
- Some equipment do not use a whole mask. Instead a smaller quartz plate is used with just a few die (inset). This plate is called a reticle.



Mask and Reticle (inset)

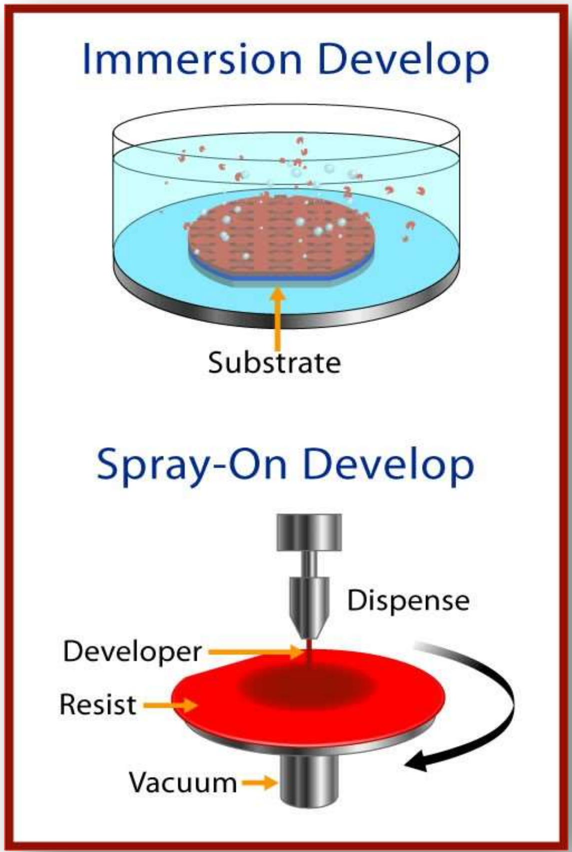
5. Expose



- The wafer is exposed by UV (ultraviolet) from a light source traveling through the mask to the resist
- A chemical reaction occurs in the resist due light exposure
- Only those areas not protected by the mask undergo a chemical reaction

6. Development

- Portions of the photoresist are dissolves in the developer
- With positive resist, the **exposed** resist is **dissolves** while the unexposed resist remains on the wafer
- With negative resist, the unexposed resist is dissolves while the exposed resist remains.

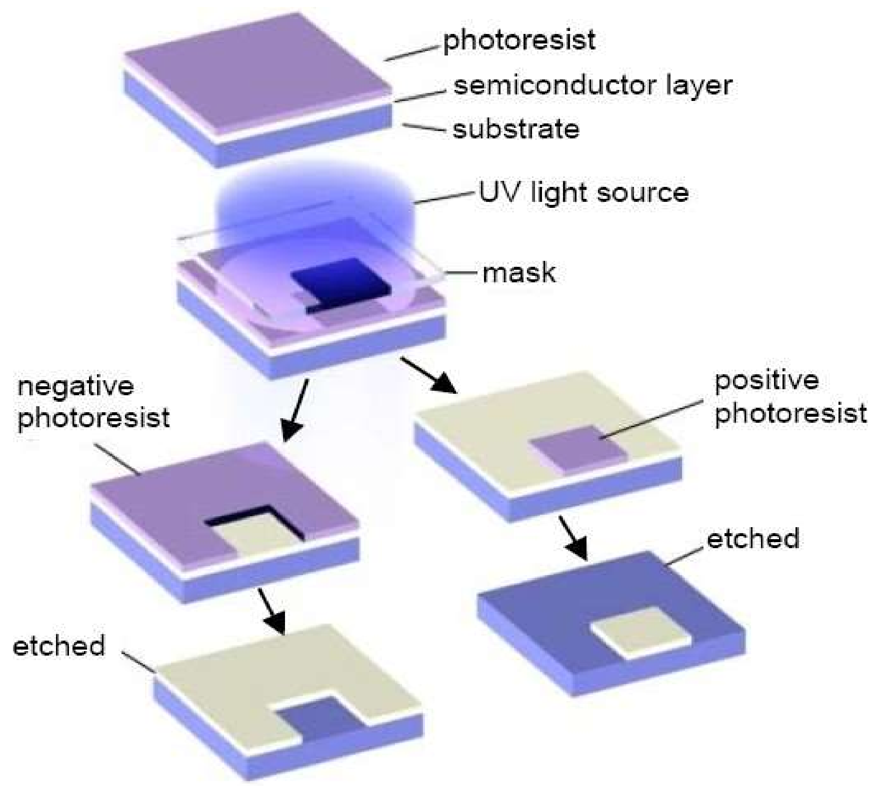


Photoresist (Resist)

Photoresist is a mixture of organic compounds in a solvent solution.

Two types of resist:

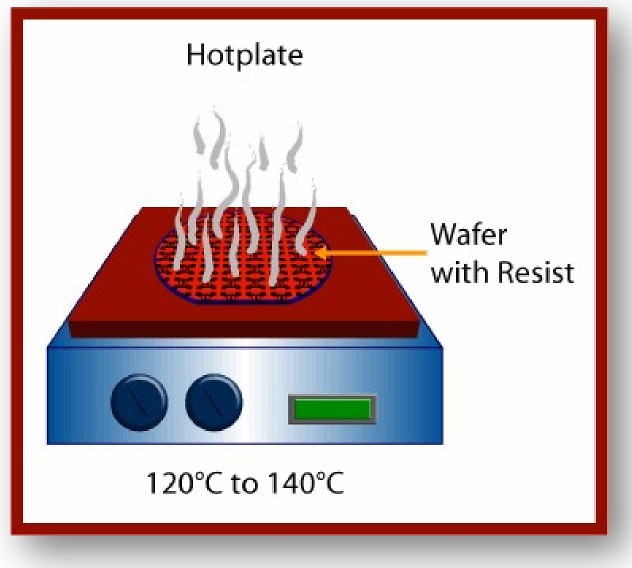
- **Positive resist** - **Exposed** regions become more **soluble**. A positive mask is left after development
- **Negative resist** - Exposed material harden. A negative mask is left after development



Photoresist- Positive vs. Negative

7. Hardbake

- Hardens the photoresist for the next process.
- The temperature of the hardbake is higher than that of the softbake after coat
- After the hardbake, the wafer is cooled to room temperature

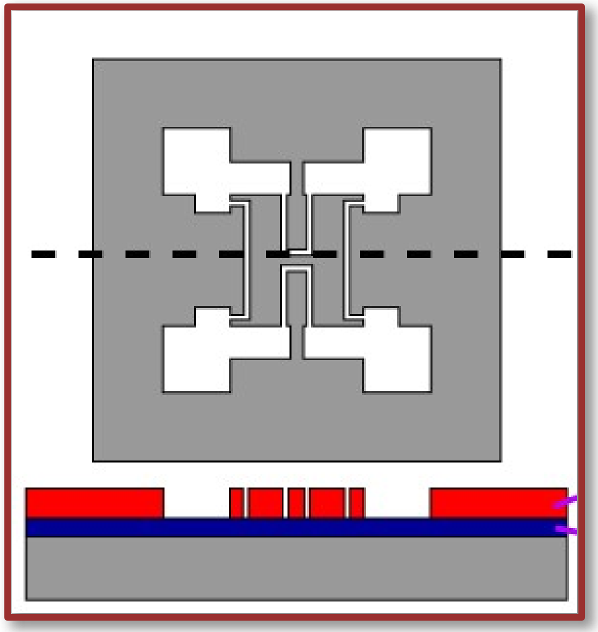


Hardbake

8. Inspect

Three critical parameters

- **Alignment:** the pattern must be positioned accurately to the previously layer
- **Line width or critical dimension (CD):** the pattern images are in focus and have the correct size
- **Defects:** things that could affect subsequent processes and eventually the operation of the devices



8. Inspect

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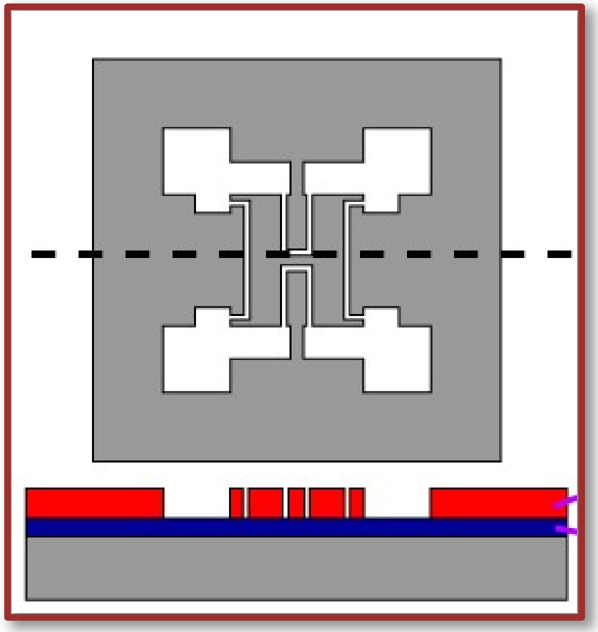
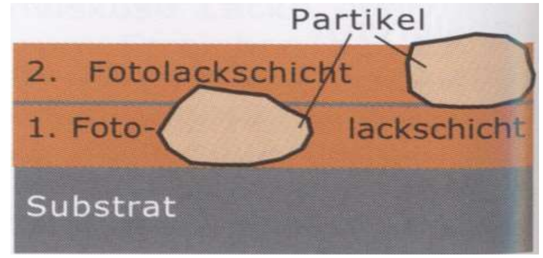
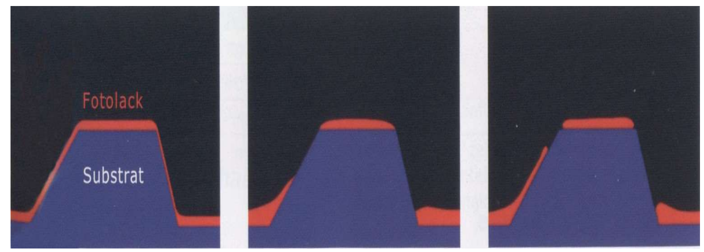


Defects

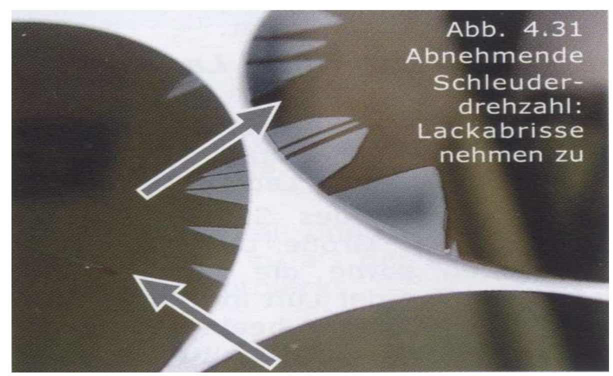
- Particles in the photoresist (dust, old photoresist)



- Bubbles



- Rough substrate
- Tear-off



Questions/ Discussion

Biosensors

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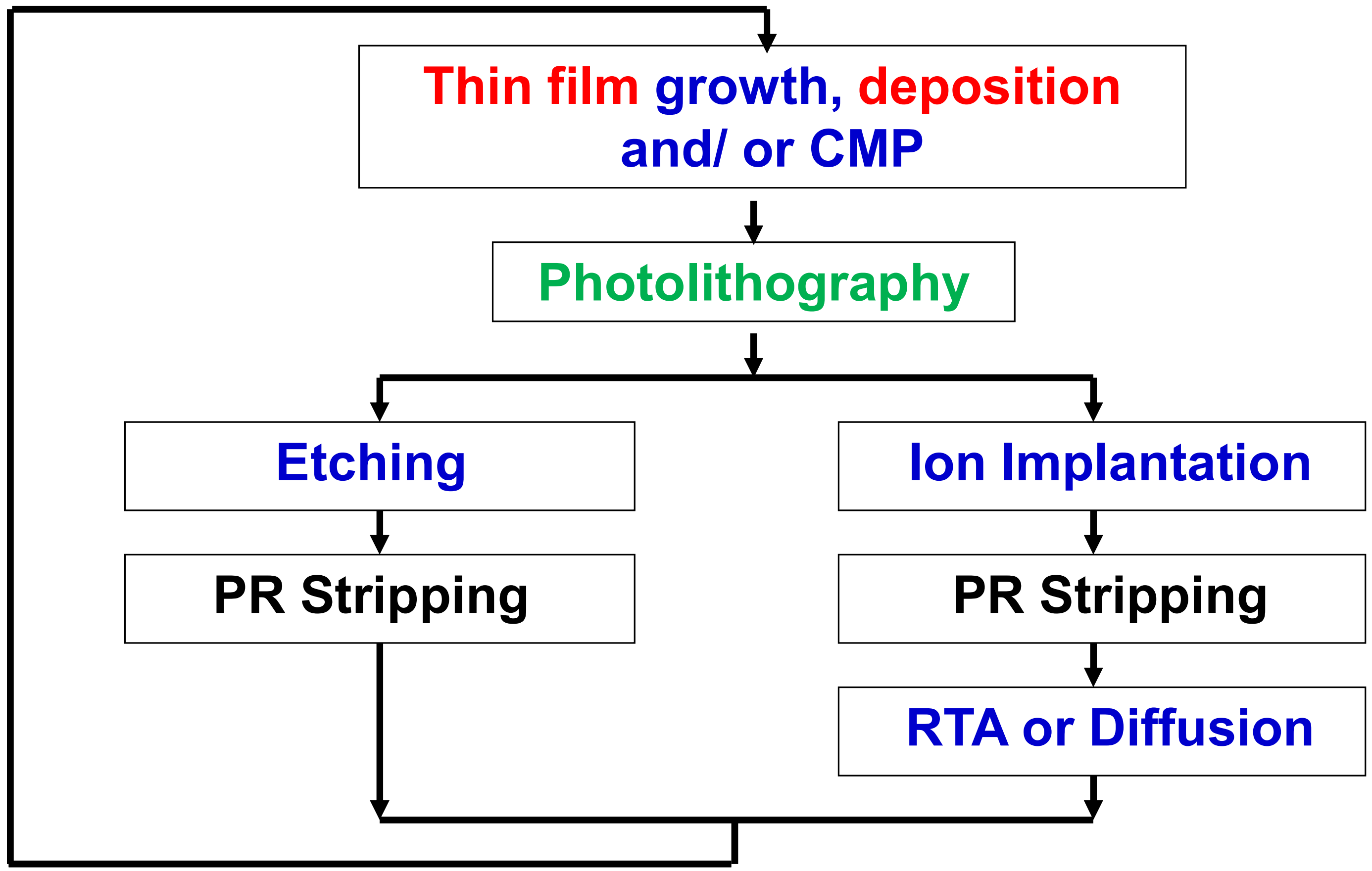
Lecture 19 dated 25th Sep. 2024

Sensor Fabrication Processes



Thin Film deposition
by CVD & PVD

Ref.: Marc J. Madou, (2011), Fundamentals of Microfabrication and Nanotechnology: The Science of Miniaturization, 3rd Edition, CRC Press

Introduction

- Chemical Vapor Deposition is the formation of a non-volatile solid film on a substrate by the reaction of vapor phase chemicals (reactants) that contain the required constituents

- The reactant gases are introduced into a reaction chamber; they are decomposed and reacted at the heated surface, to form the thin film

Introduction

Examples of CVD films

- **Dielectrics**
  - Silicon dioxide, silicon nitride, …
- **Metals**
  - Tungsten, aluminum, copper, titanium, …
- **Semiconductors**
  - Epitaxial (a single crystal layer is grown on a single crystal substrate) silicon, gallium arsenide, poly-silicon, doped poly-silicon

Introduction

CVD Systems

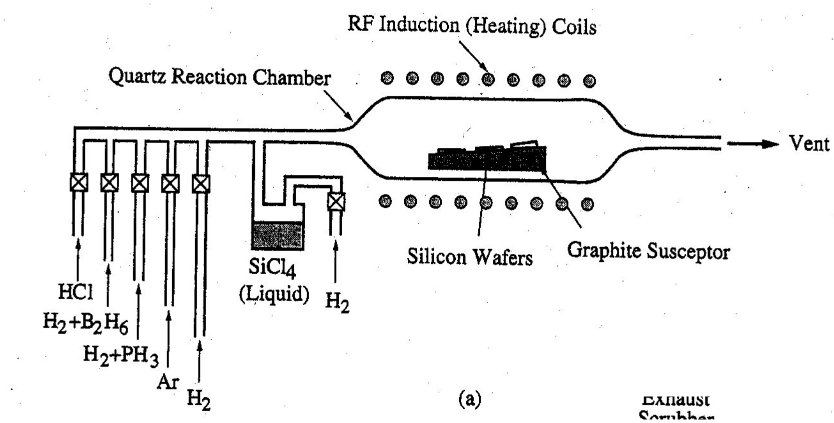
- **AP-CVD-** Chemical Vapor Deposition Process Running Under Atmospheric Pressures
- **SA-CVD-** Chemical Vapor Deposition Process Running Under Sub-atmospheric Pressures
- **LP-CVD-** Chemical Vapor Deposition Process Running Under Low Pressures
- **PE-CVD-** Chemical Vapor Deposition Process where reactions are Enhanced through the Development of a Plasma Energy Source

Introduction

CVD Systems

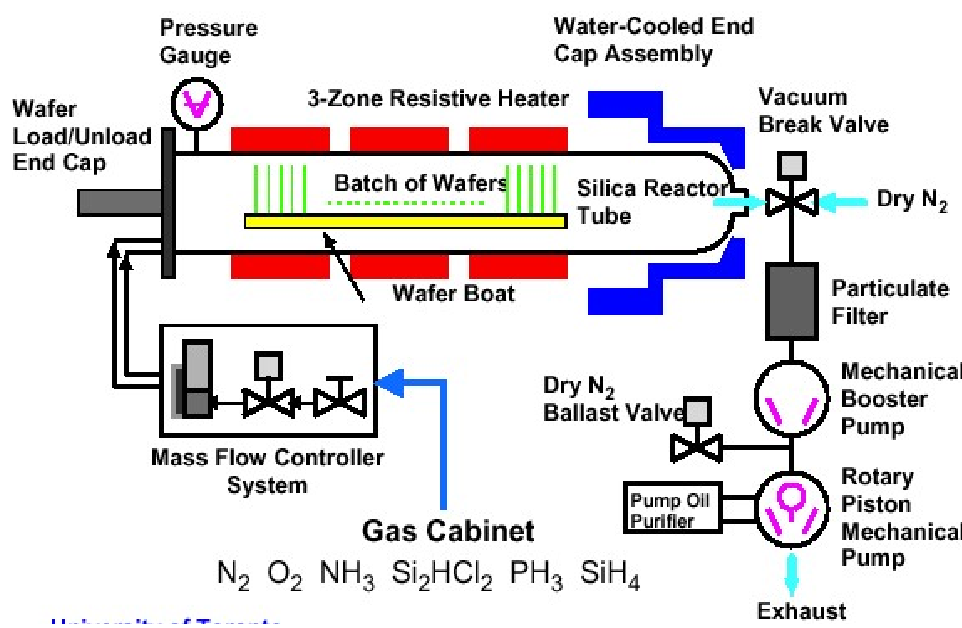
- **HDP-CVD- high density plasma chemical vapor deposition process are used for filling narrow gaps and greater depth/width aspect ratios of the underlying topography**
- **MOCVD- metal organic chemical vapor deposition process has the capability to deposit epitaxial films**
- **PH-CVD (CVD writing)- chemical vapor deposition process where *reactions are enhanced through* the photon or laser energy source**

CVD systems



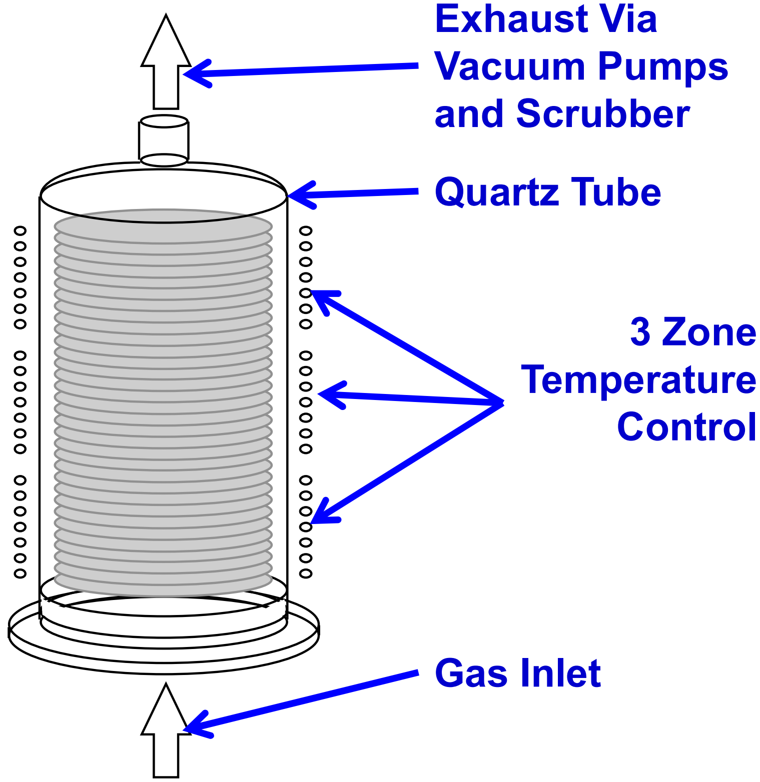
Horizontal APCVD Reactor

Horizontal LPCVD Reactor

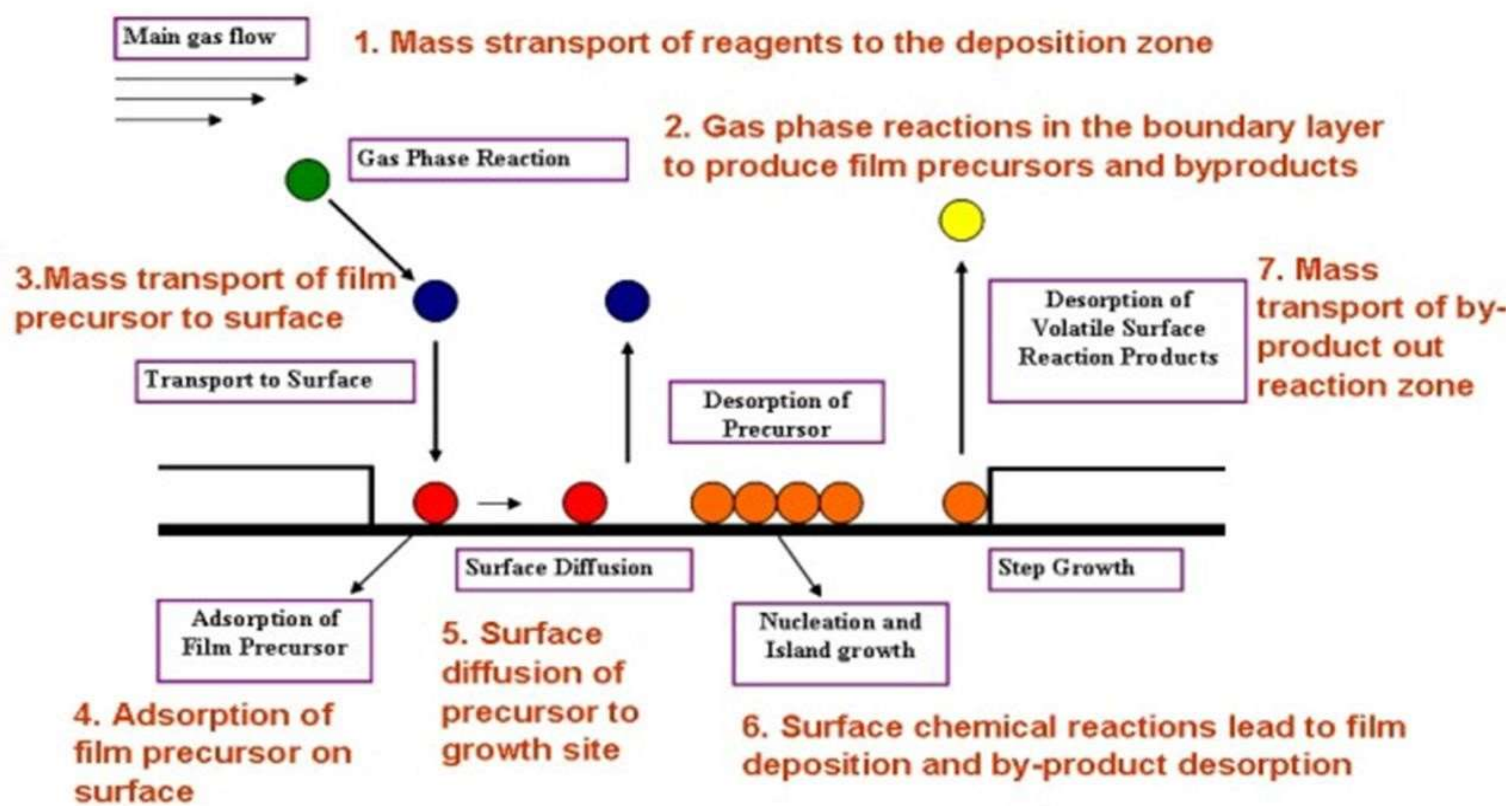


CVD systems

Vertical LPCVD Furnace

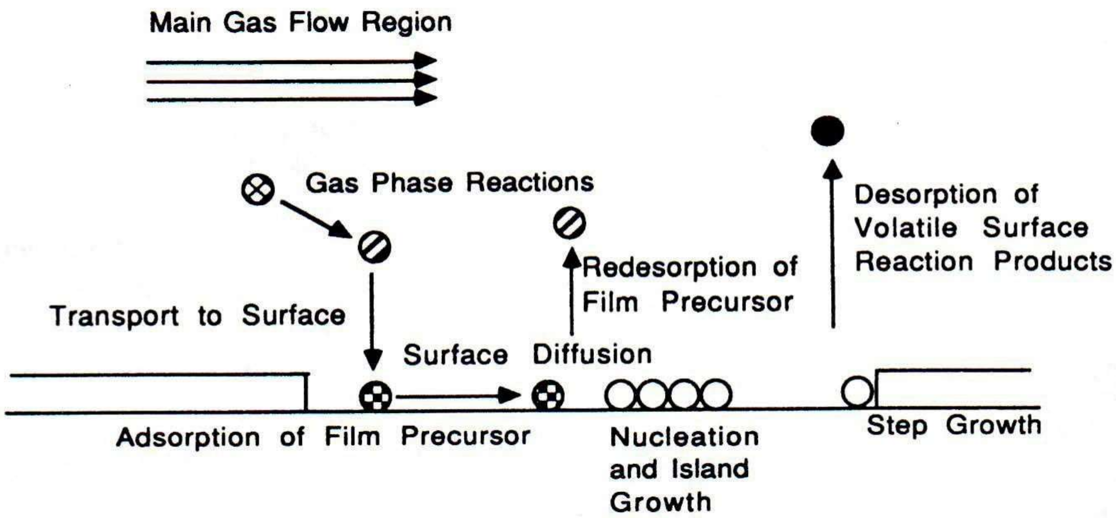


Chemical Vapour Deposition Mechanism



Transport and Reaction Processes of CVD

CVD of Si in a Si-Cl-H system



1. $SiCl_{4(g)} + 2H_{2(g)} \leftrightarrow Si_{(s)} + 4HCl_{(g)};$

2. $SiCl_3H_{(g)} + H_{2(g)} \leftrightarrow Si_{(s)} + 3HCl_{(g)};$

3. $SiCl_2H_{2(g)} \leftrightarrow Si_{(s)} + 2HCl_{(g)};$

4. $SiClH_{3(g)} \leftrightarrow Si_{(s)} + HCl_{(g)} + H_{2(g)};$

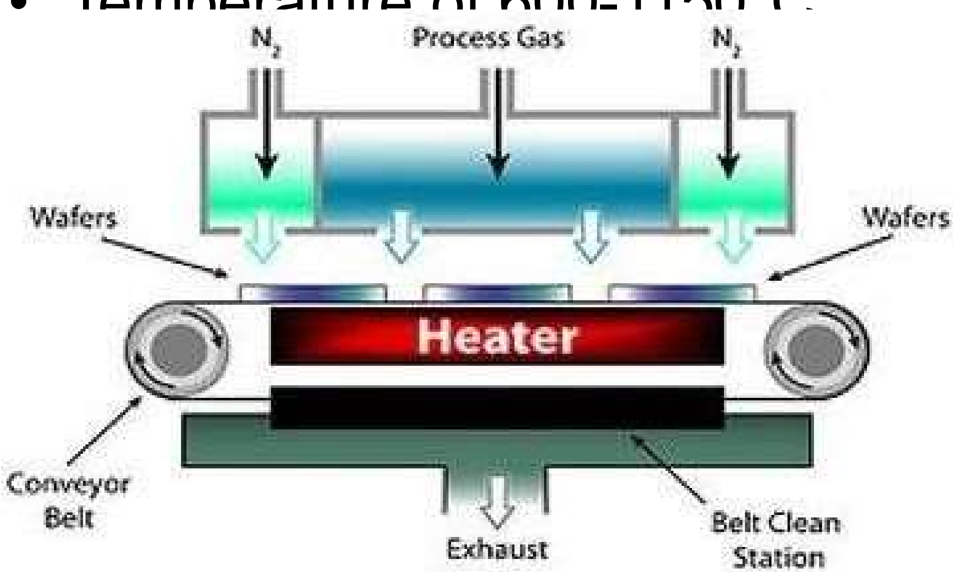
5. $SiCl_{2(g)} + H_{2(g)} \leftrightarrow Si_{(s)} + 2HCl_{(g)};$

6. $SiH_{4(g)} \leftrightarrow Si_{(s)} + 2H_{2(g)};$

Chemical Vapour Deposition Techniques - 1

Atmospheric Pressure Chemical Vapor Deposition (APCVD)

- Dielectrics and metals
- Atmospheric pressure or partial pressure in N_2
- Low film purity
- Temperature of 600-1150°C

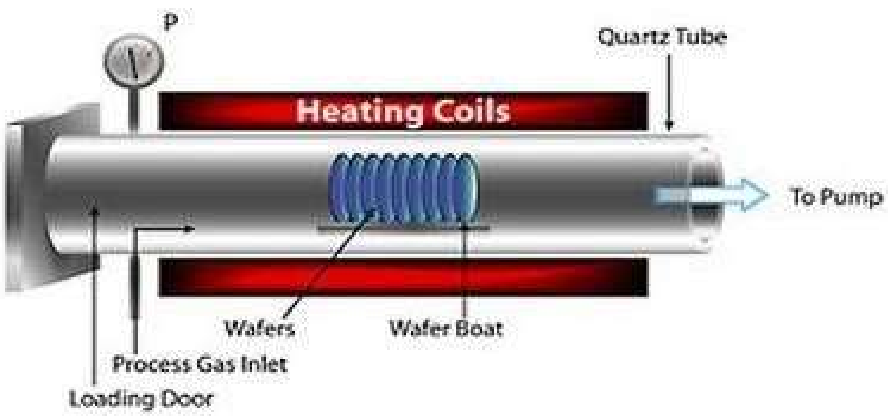


SierraTherm APCVD Furnaces

Chemical Vapour Deposition Techniques - 2

Low-Pressure Chemical Vapor Deposition (LPCVD)

- Dielectrics and metals
- Performed at reduced pressure or "rough vacuum"
- 10^{-3} to 10^{-5} Torr (1 atm = 760 Torr)
- High purity
- High temperature



Tystar LPCVD Tube Furnaces

LPCVD Processes

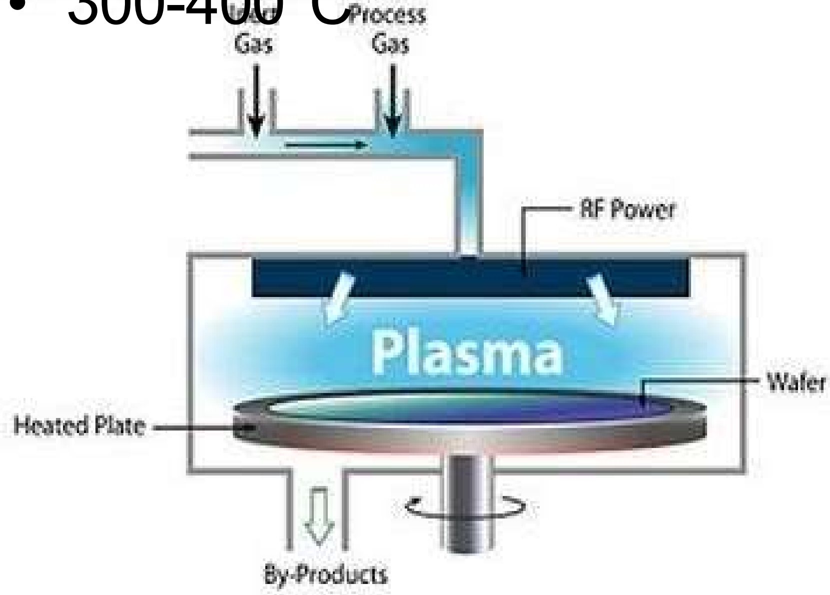
Layer		Reaction equations	Temperature (ºC)
SiO_2	LTO TEOS HTO	$SiH_4 + O_2 \rightarrow SiO_2 + 2H_2$ $Si(OC_2H_5)_4 \rightarrow SiO_2 + gas$ $SiCl_2H_2 + N_2O \rightarrow SiO_2 + 2N_2 + 2HCl$ $SiH_4 + CO_2H_2 \rightarrow SiO_2 + gas$	400-450 650-700 850-900 850-950
Si_3N_4		$3SiH_2Cl_2 + 4NH_3 \rightarrow Si_3N_4 + 6HCl + 6H_2$	700-900
Polysilicon & a-Si		$SiH_4 \rightarrow Si + 2H_2$	550-650

•LPCVD pressures are around 300mT (0.05% atmosphere)

Chemical Vapour Deposition Techniques - 3

Plasma Enhanced Chemical Vapor Deposition (PECVD)

- Dielectrics only
- ‘High’ vacuum (10^{-6} Torr)
- 300-400°C



Plasma-Therm 790 PECVD

Chemical Vapour Deposition Techniques

APCVD	LPCVD	PECVD
Advantages	**Advantages**	**Advantages**
• Relatively low operating cost since no vacuum needed	• Lower reaction temperatures than APCVD reactors • Good step coverage and uniformity • Less dependence on gas flow dynamics	• Combination of vacuum pressures and lower temperature produces better uniformity in the deposited layer • Reactor can be used in other microelectronic production process steps
Disadvantages	**Disadvantages**	**Disadvantages**
• Uniformity of deposited layer compromised at higher temperatures and pressures • Gas flow dynamics hard to control at high pressures	• More expensive than APCVD reactors • Downstream depletion can occur in horizontal designs	• More process variables to be controlled compared to other CVD reactors • Cost of operation is increased with increased number of components

Questions and Discussion?

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Lecture 20 dated 26th Sep. 2024

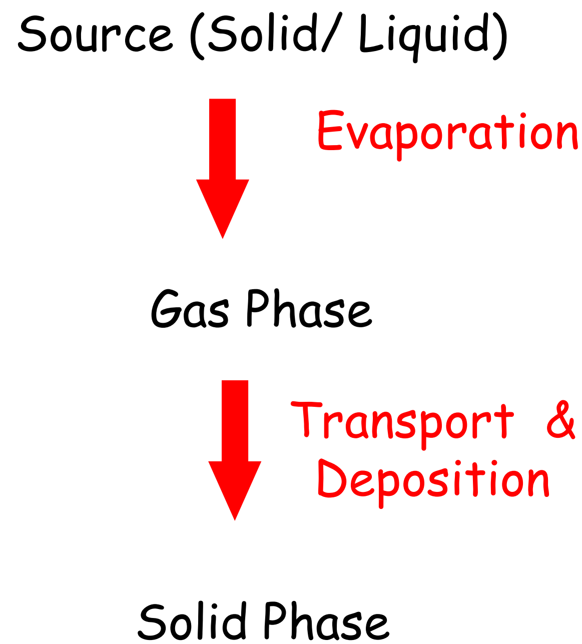
Thin Film deposition by CVD & PVD

Marc J. Madou, (2011), Fundamentals of Microfabrication and Nanotechnology: The Science of Miniaturization, 3rd Edition, CRC Press

Introduction

- Physical Vapor Deposition (PVD) is also called physical vapor transport; accomplished by a variety of vacuum deposition methods, to produce thin films and coatings

- In PVD, the material is transported from a condensed phase to a vapor phase & then back to a thin film condensed phase.



Introduction

Examples of PVD films

- **Metals**
- **Alloys**
- **Dielectrics**
- **Ceramics**
- **Semiconductors**
- **other Inorganic compounds, &**
- **even certain polymers …**

Introduction

PVD techniques

Can be grouped into five principal types:

1. Thermal evaporation
2. Sputtering
3. Ion plating and cluster deposition
4. Laser sputter deposition or laser ablation deposition, &
5. Aerosol deposition

Introduction

PVD Process	Features	Coating Materials
Thermal evaporation	• Equipment is relatively low cost and simple • deposition of compounds is difficult • coating adhesion not as good as for other PVD processes	Ag, Al, Au, Cr, Cu, Mo, W
Sputtering	• Better coating adhesion than vacuum evaporation • can coat compounds • slower deposition rates & more difficult process control than vacuum evaporation	Al_2O_3, Au, Cr, Mo, SiO_2, Si_3N_4, TiC, TiN
Ion plating & cluster deposition	• Best coverage & coating adhesion of PVD processes • most complex process control • higher deposition rates than sputtering	Ag, Au, Cr, Mo, Si_3N_4, TiC, TiN

Introduction

PVD Process	Features	Coating Materials
Laser sputter deposition or laser ablation deposition	Best for complex compound deposition	high-temperature superconductor $YBa_2Cu_3O_{7-x}$ and biocompatible calcium hydroxylapatite, or $Ca_{10}(PO_4)_6(OH)_2$
Aerosol deposition	Lowest temperature deposition	ceramic coatings on all types of substrates

1. Evaporator Deposition:

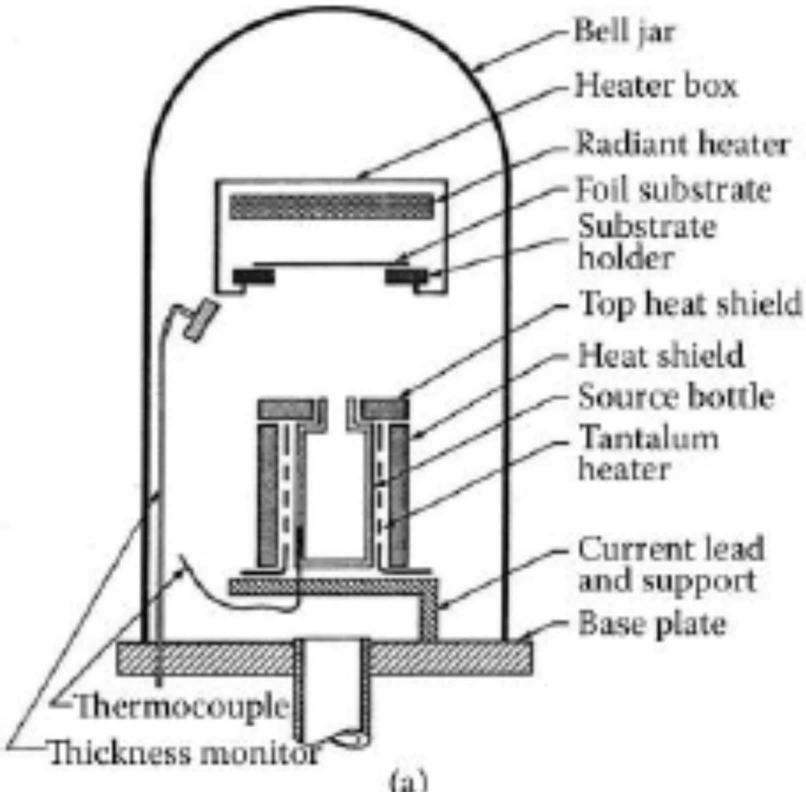
The deposited materials are evaporated or sublimated, either by an electron beam or a other heat source.

- Mainly metals
- High vacuum ($\geq 10^{-6}$ Torr)

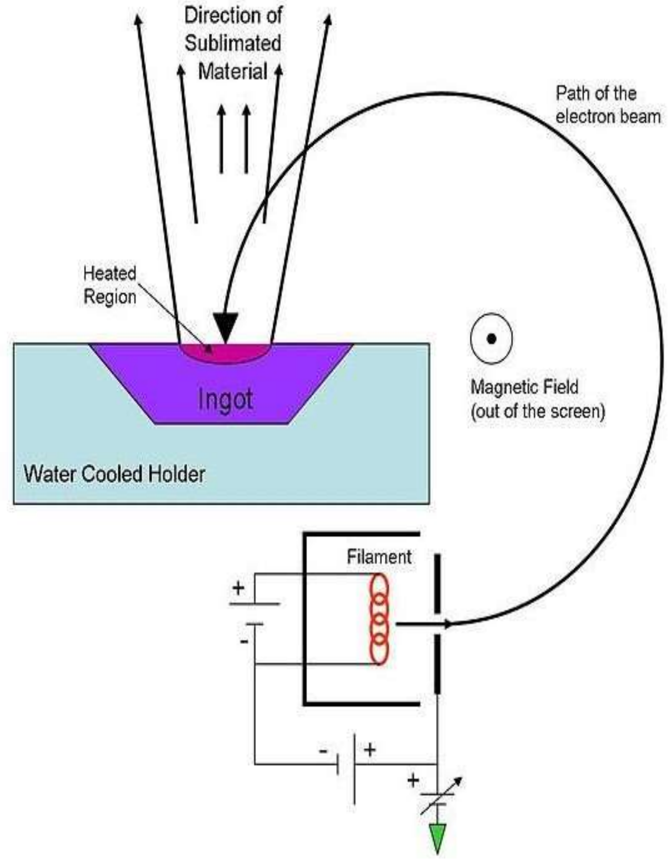
Edwards E306A thermal evaporator evaporator

Denton DV-502A e-beam

Evaporation



Typical evaporation setup



Magnetized deflection electron-beam evaporation system

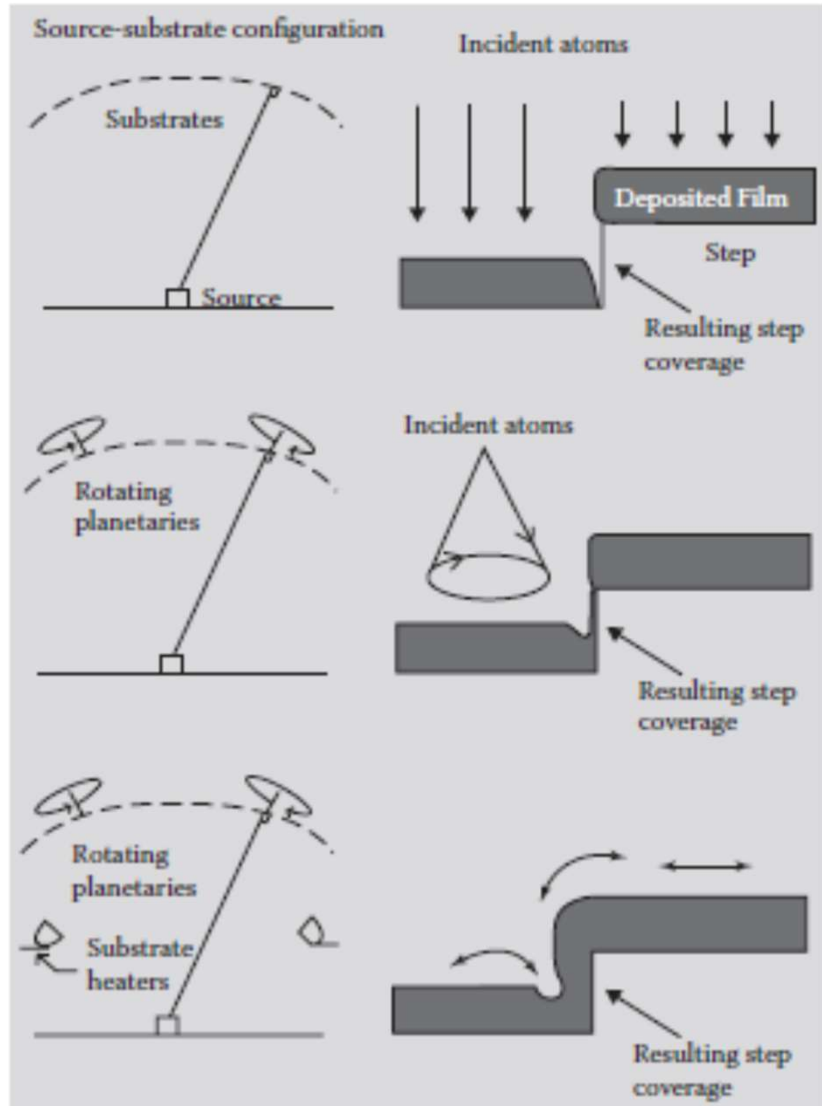
Comparison of Heat Sources for Evaporation

Heat Sources	Advantages	Disadvantages
Resistance	No radiation	Contamination
Electron beam	Low contamination	Radiation
RF	No radiation	Contamination
Laser	No radiation, low contamination	Expensive

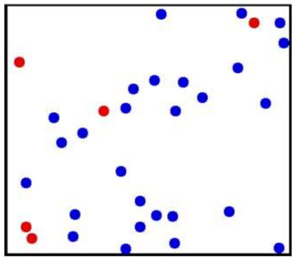
Shadowing Effect

Shadowing effect may be overcome by using:

- rotating planetaries &
- heating of the substrate



Mean Free Path



λ = k * T / (√2 * π * d² * p)

where,

- λ is the mean free path expressed in the length units
- T is the temperature of the gas
- p is the pressure of the gas
- d is the diameter of a particle
- k is the Boltzmann constant
  k = 1.380649 * 10^(−23) J / K.

Pressure (Torr)	Mean Free Path (cm)	Number Impingement Rate ($s^{-1}. cm^{-2}$)	Monolayer Impingement Rate (s^{-1})
10^{-2}	0.5	3.8×10^{18}	4400
10^{-4}	51	3.8×10^{16}	44
10^{-5}	510	3.8×10^{15}	4.4
10^{-7}	5.1×10^{4}	3.8×10^{13}	4.4×10^{-2}
10^{-9}	5.1×10^{6}	3.8×10^{11}	4.4×10^{-4}

- The number of atoms per unit area corresponding to a monolayer for a metal is about 10^{15} atoms/cm^2

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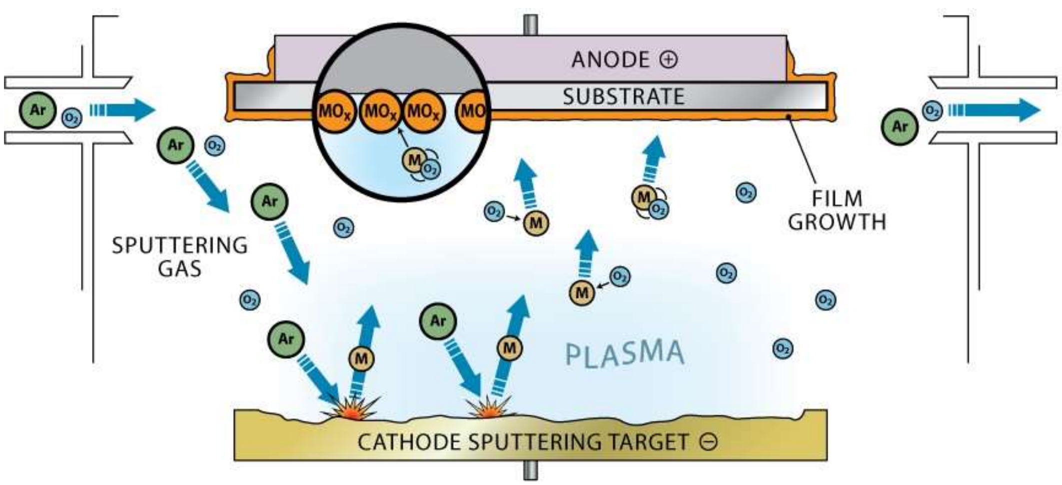
Lecture 21 dated 30th Sep. 2024

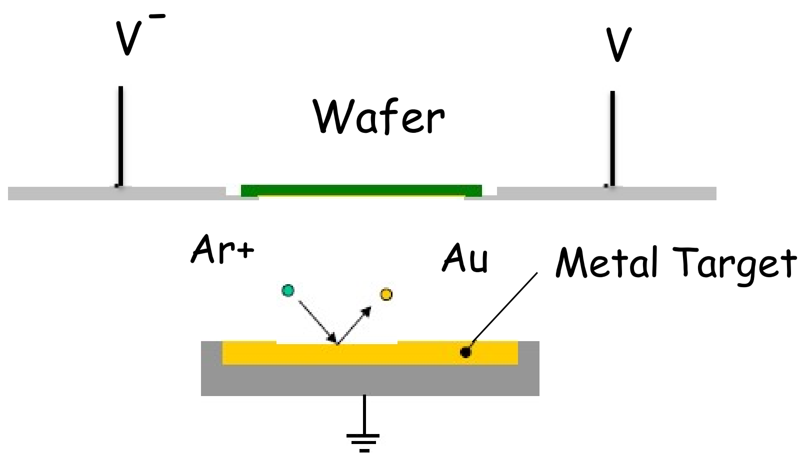
2. Sputter Deposition:

Sputtering involves the collisions of ions (Ar·) with target material, leading to the ejection of target atoms that are collected on a substrate.

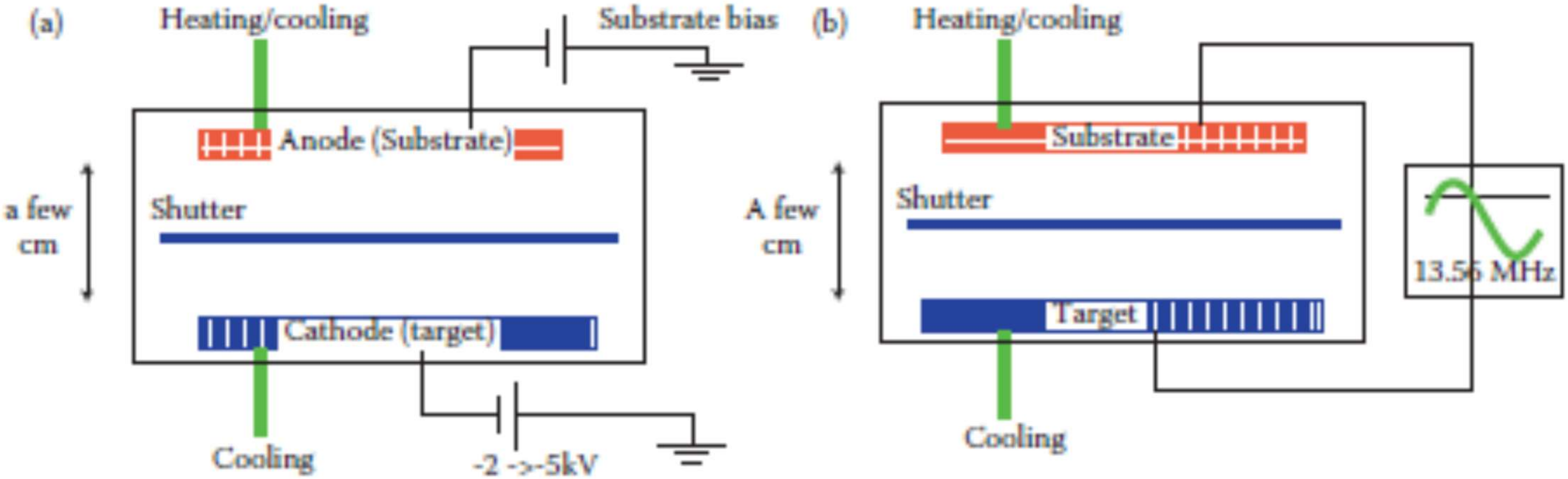
- Metals and dielectrics
- High vacuum (10^{-6} Torr)



A high electric field ionizes argon atoms and accelerates them into a metal target



- Forms very uniform films
- Excellent step coverage (distributed angles of impact)
- Sputtering allows easy deposition of alloys (Al-Cu-Si)
- Wafer heating less than 300°C
- Sputtering of dielectrics uses both DC and RF fields



The DC (a) and RF (b) sputter deposition setups

Example materials deposited by sputtering:

Type of Material: Examples
Metals: Al, Cu, Zn, Au, Ni, Cr, W, Mo, Ti
Alloys: Ag-Cu, Pb-Sn, Al-Zn, Ni-Cr
Nonmetals: graphite, MoS_2, WS_2, PTFE
Refractory oxides: Al_2O_3, Cr_2O_3, Al_2O_3-Cr_2O_3, SiO_2, ZrO_2-Y_2O_3
Refractory carbides: TiC, ZrC, HfC, NbC
Refractory nitrides: TiN, Ti_2N, ZrN, HfN, TiN-ZrN, TiN-AlN-ZrN
Refractory borides: TiB_2, ZrB_2, HfB_2, CrB_2, MoB_2
Refractory silicides: $MoSi_2$, WSi_2, Cr_3Si_2

Comparison of Evaporation & Sputtering Technology

	Evaporation	Sputtering
Rate	Thousand atomic layers per second (e.g., 0.5 μm/min for Al)	One atomic layer per second
Choice of materials	Limited	Almost unlimited
Purity	Better (no gas inclusions, very high vacuum)	Possibility of incorporating impurities (low-medium vacuum range)
Substrate heating	Very low	Unless magnetron is used substrate heating can be substantial
Surface damage	Very low, with e-beam x-ray damage is possible	Ionic bombardment damage
In situ cleaning	Not an option	Easily done with a sputter etch
Alloy compositions, stoichiometry	Little or no control	Alloy composition can be tightly controlled
X-ray damage	Only with e-beam evaporation	Radiation and particle damage is possible
Changes in source material	Easy	Expensive
Decomposition of material	High	Low
Scaling up	Difficult	Good
Uniformity	Difficult	Easy over large areas
Capital equipment	Low cost	More expensive
Vacuum path	High: few collisions, line-of-sight deposition, little gas in film	Low: many collisions, less line-of-sight, gas inclusions
Number of depositions	Only one deposition per charge	Many depositions can be carried out per target
Thickness control	Not easy to control	Several controls possible
Adhesion	Often poor	Excellent
Shadowing effect	Large	Small
Film properties (e.g., grain size and step coverage)	Difficult to control, larger grain size, fewer grain orientations	Control by bias, pressure, substrate heat, smaller grain size, many grain orientations

Questions and Discussion?

Etching

Film Etching

Etching is partial or complete removal of a film, mainly to realize desired pattern of the film.

A **masking layer** is used to protect the film surface from etching; the masking material has very low etch rate or is etch resistant in the etchant.

Materials of interest:

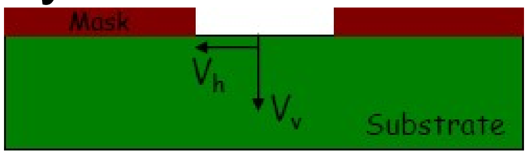
- Conductors: Al, Cu, Au, Ag, Cr, Ti, …
- Dielectrics: SiO_2, Si_3N_4, Polyimide, PR, …
- Semiconductors: Si, GaAs, Polysilicon, …

Film Etching

Etches are characterized by the verticality and anisotropy.

- If Vh is the horizontal etch rate & Vv is the vertical etch rate,
- The anisotropy can be given by:

$$A = 1 - \frac{V_h}{V_v}$$



- A=1 for fully anisotropic etches (Vh =0)
- A=0 for fully isotropic etches (Vv =0)

Etches can be performed using chemical solutions (wet) or plasmas (dry).

Etching Process Categories

1. Dry etching
2. Wet chemical etching & wet bulk Micromachining
3. Thermal energy-based removing
4. Mechanical energy-based removing

Isotropic Wet Etching

Wet etching of amorphous (polycrystalline) materials is usually isotropic, meaning no direction is favored.

- SiO_2: buffered hydrofluoric acid (HF)
- Si_3N_4: hot phosphoric acid (65-70°C)
- Al: nitric/ phosphoric/ acetic acid (can't use if on GaAs) or hydrochloric acid (HCl; OK on GaAs)
- PolySi: HF and nitric acid
- Cr: potassium permanganate
- Au: potassium iodide and iodine (KI/ I_2)

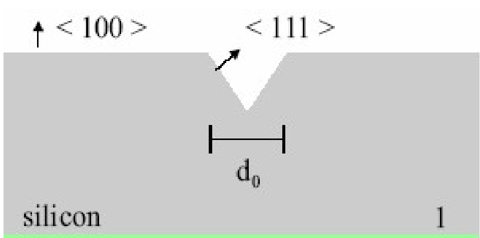
Anisotropic Wet Etching

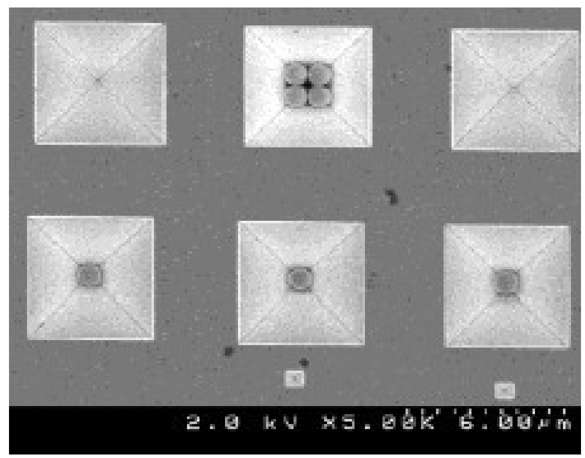
Wet etching of crystalline (single-crystal) materials is usually anisotropic, meaning some crystallographic directions are favored than others.

- Silicon – anisotropic etch: basis of silicon micromechanics.
- GaAs – anisotropic etch: bromine and methanol (highly exothermic!!) or hydrogen peroxide/sulfuric acid.

Single-crystal

Multi-crystal





Pyramid shaped pits in Si (100)

Questions and
Discussion?