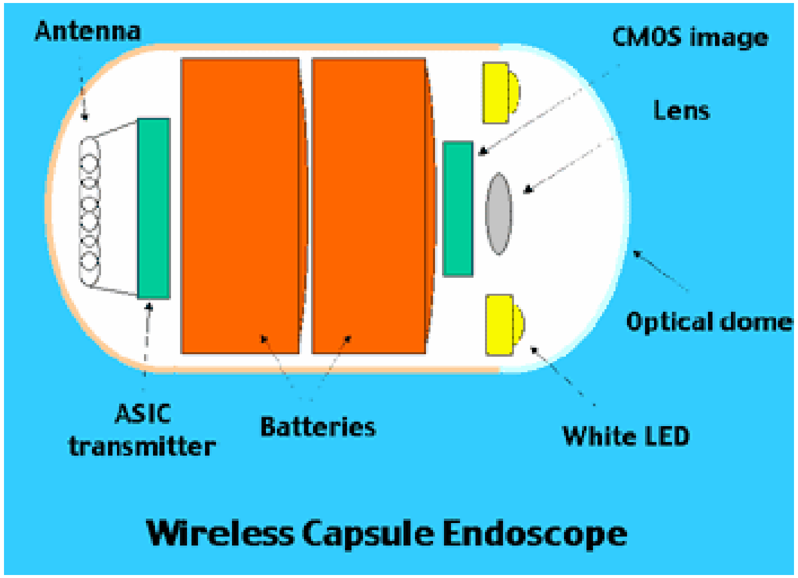
# Bio-MEMS Sensors technology and Fabrication

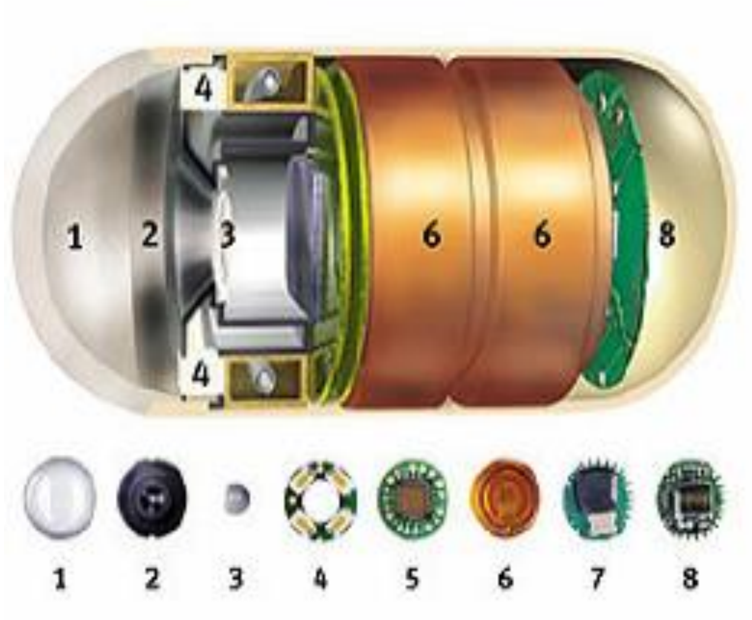
ENGG-6150 Bio-Instrumentation

# Applications

- Biomedicine
  - Drug delivery, DNA sequencing, chemical analysis
- Pressure sensors
  - Automotive, Medical, Industrial, …
- Accelerometers
  - Automotive, Medical, Industrial
- Gyros
  - Automotive
- Displays
  - TI DMD, SLM GLV
- Fiber optics
  - Switches, attenuators, alignment
- RF components
  - Relays, filters, tunable passive elements
- https://www.youtube.com/watch?v=iPGpoUN29zk
- https://www.youtube.com/watch?v=Cncmej_3ijg

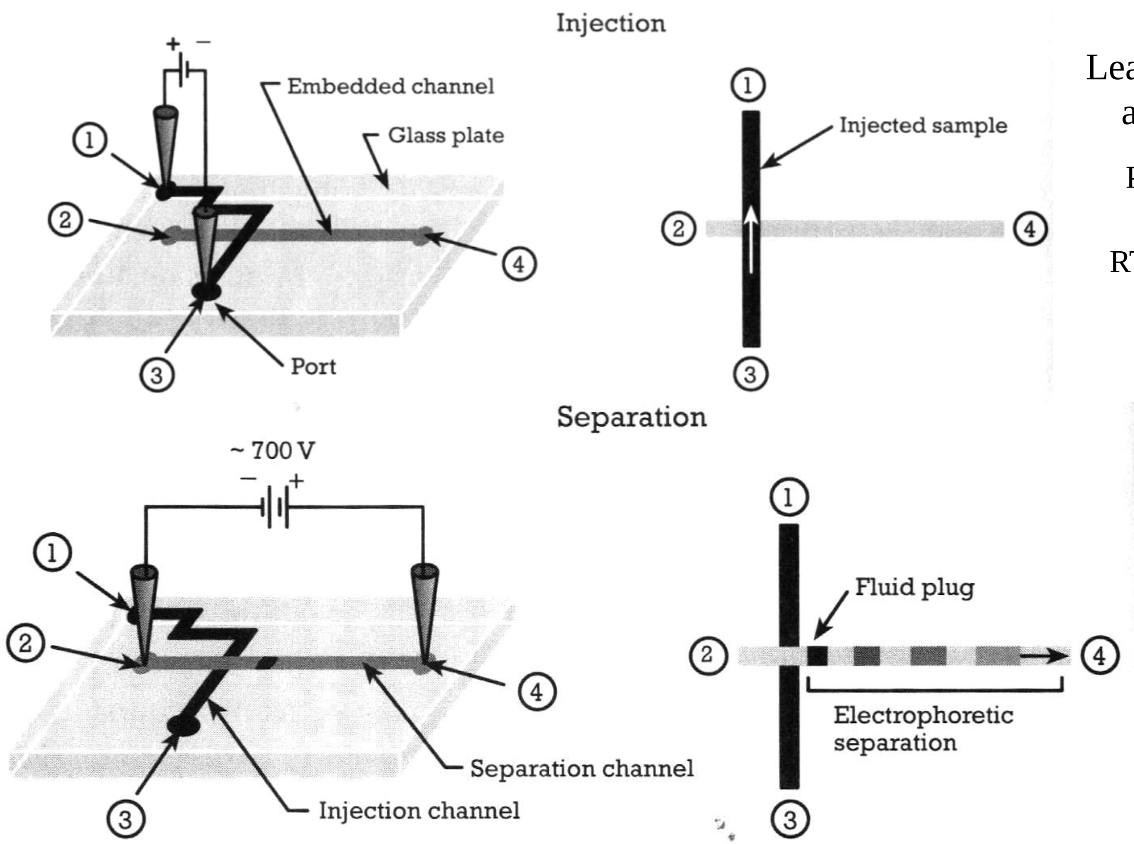
# Bio-medical Applications: Pill Camera

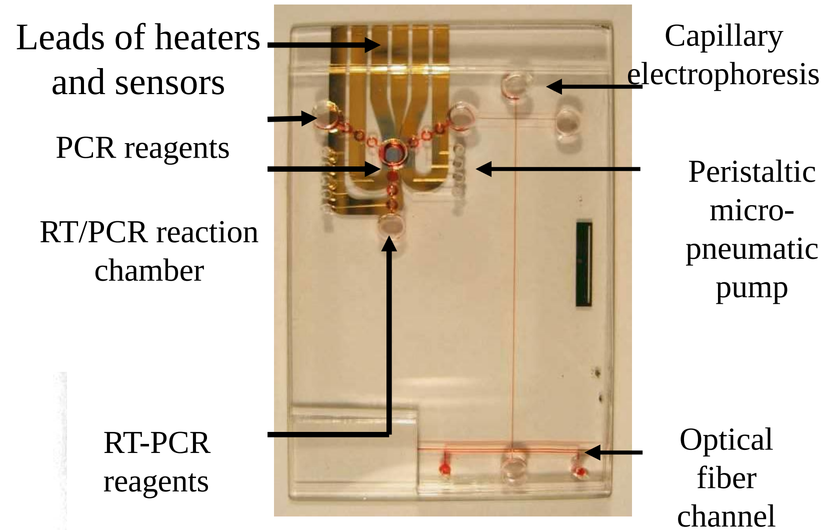



Pill camera are complete system with wireless communication channel and installed batteries inside

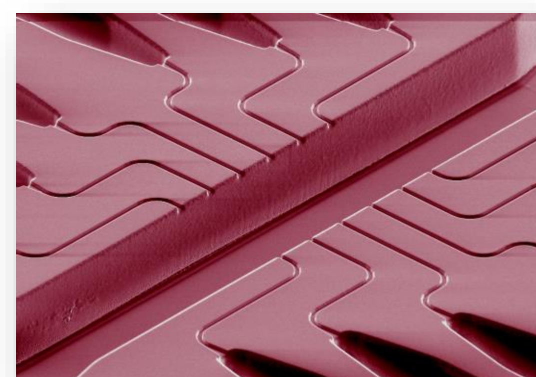
# Microfluidics / DNA Analysis




In the future, a complete DNA sequencing systems should include:

- Amplification
- Detection
- Fluid preparation and handling (pumps, valves, filters, mixing and rinsing)

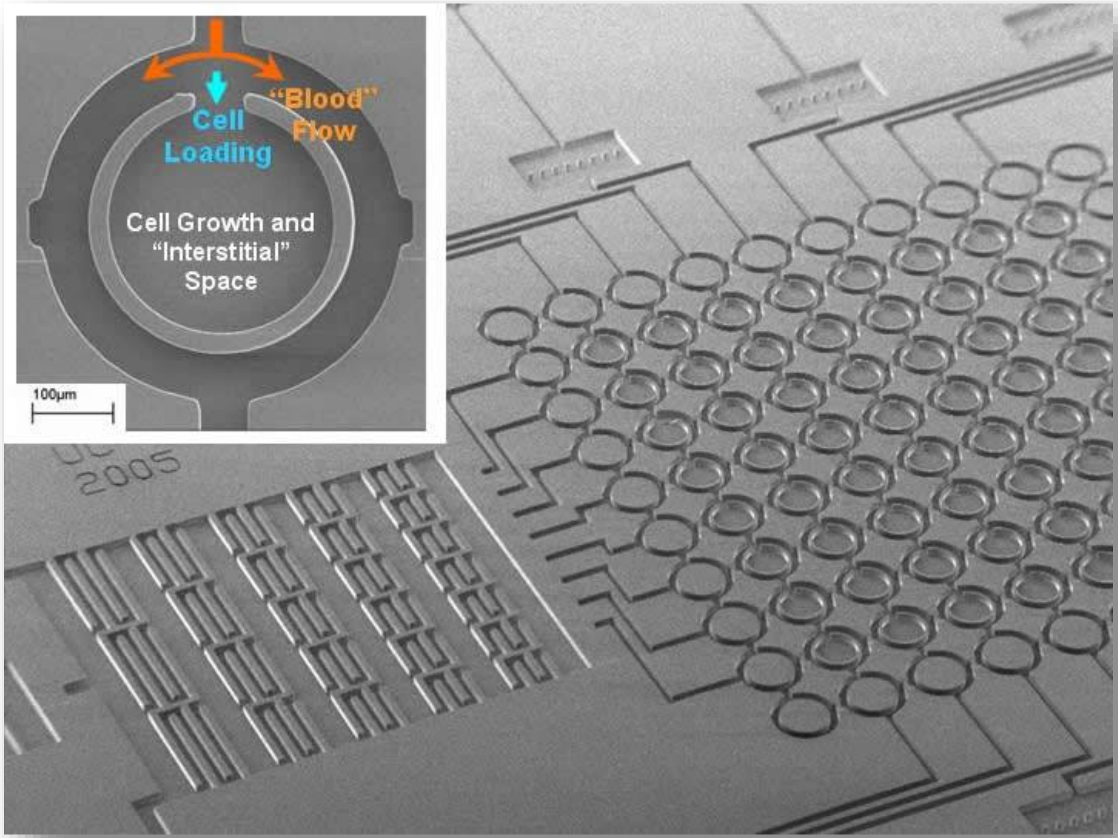


Microfluidics

*Channels and chambers*

*[Courtesy of Luke Lee @ UC-Berkeley]*

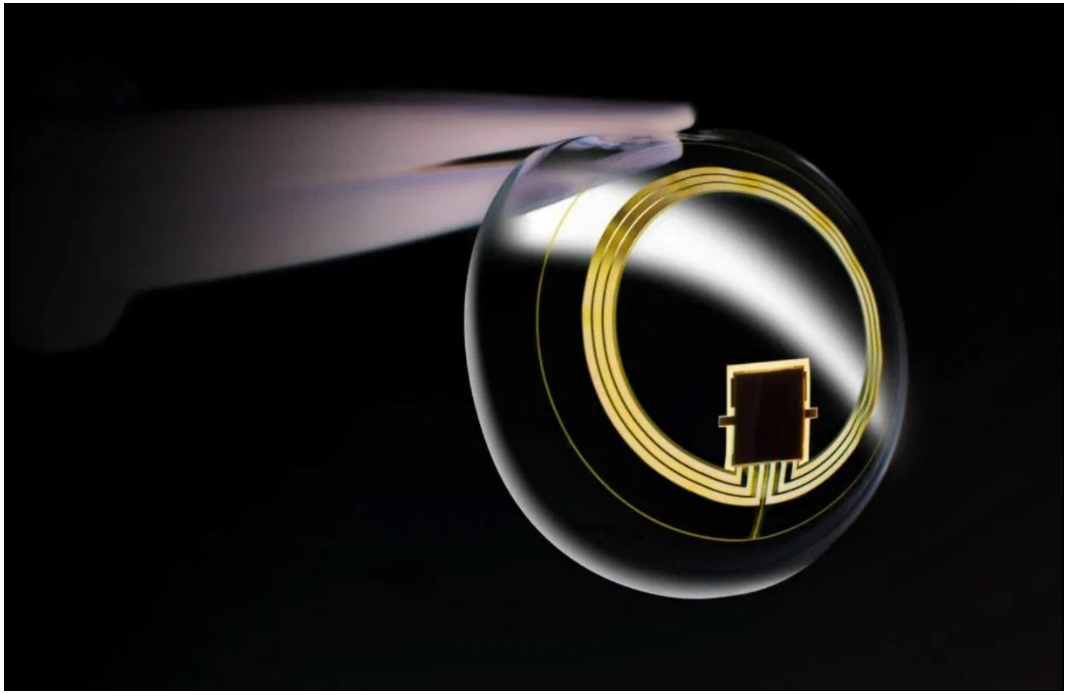
# Microfluidics / DNA Analysis




*A microenvironment for growing cells in vitro and in parallel, allowing for the analysis of multiple cell growth conditions.*
*[Developed by and courtesy of BioPOETS Lab, UC-Berkeley]*

- Cell Culture Environments

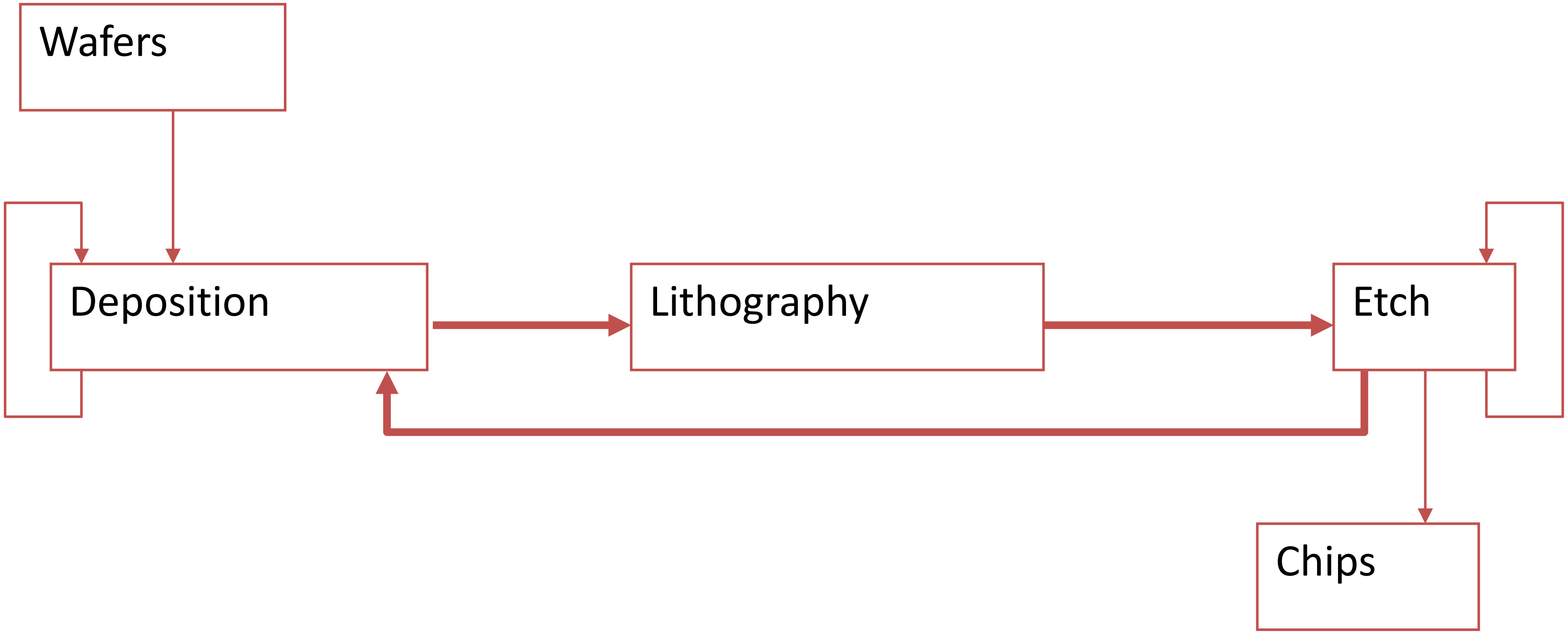
# MEMS Contact Lens



- A soft contact lens with a MEMS strain gage embedded for intraocular pressure monitoring.

# Process Flow

- Integrated Circuits and MEMS identical
- Process complexity/yield related to # trips through central loop



# Materials

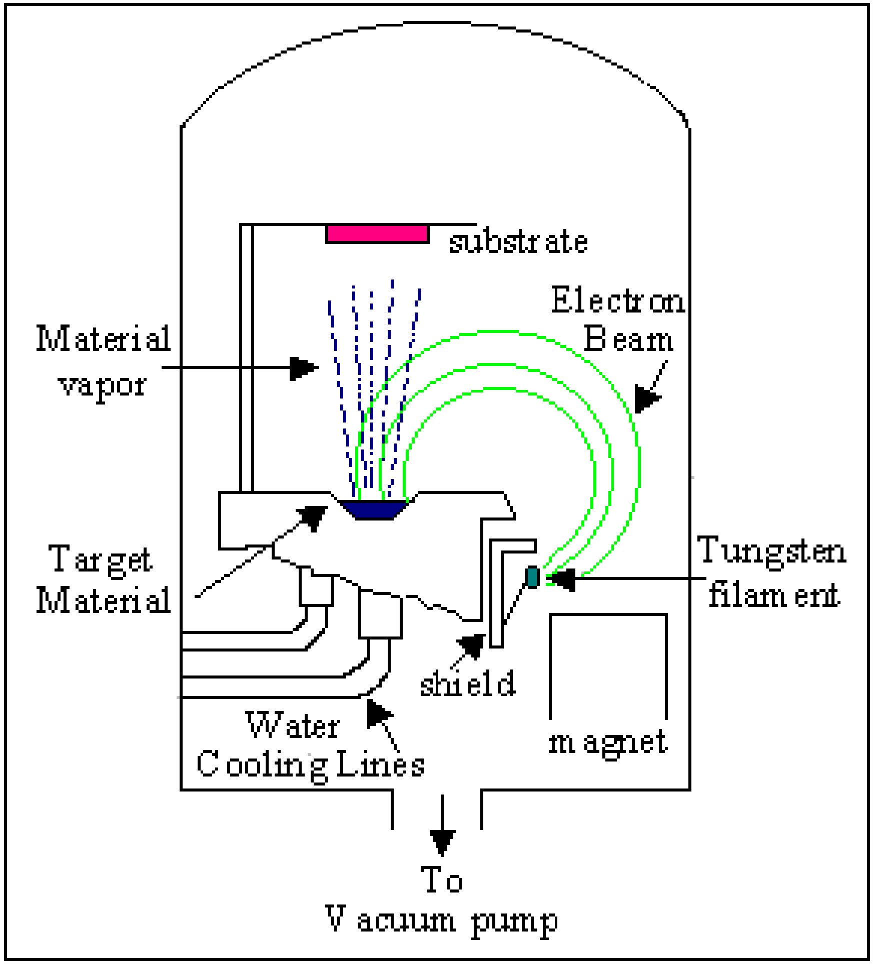
- Metals
  - Al, Au, Cu, W, Ni, TiNi, NiFe,
- Insulators
  - SiO2 - thermally grown or vapor deposited (CVD)
  - Si3N4 - CVD
- Polymers
- The King of Semiconductors: Silicon
  - stronger than steel, lighter than aluminum
  - single crystal or polycrystalline
  - 10nm to 10mm

# Process Flow

- Thin film deposition
  - Electron beam evaporation
  - Sputtering
  - Epitaxial grown
- Photolithography
  - Positive photoresist
  - Negative photoresist
- Pattern transfer through
  - Wet etching processes
    - Isotropic
    - Anisotropic
  - Dry etching processes
    - Isotropic
    - Anisotropic
  - Shadow masking
  - Lift Off

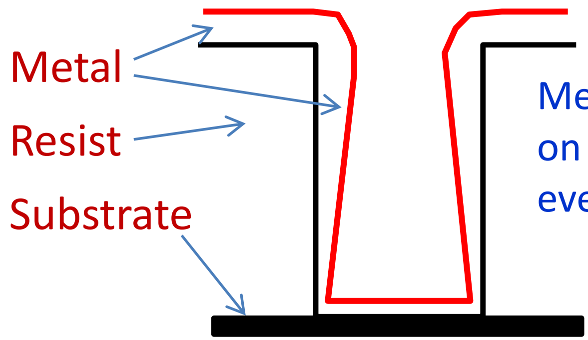
# Electron beam evaporation

- Using a focused electron beam to heat and evaporate metals,  electron temperature can be as high as 10,000 K.
- Suitable for high $T_{melt}$ metals like W, Ta, …
- Evaporation occurs at a highly localized point on the source surface, so little contamination from the crucible
- Composite films can be deposited using dual e-beams with dual targets
- Thickness uniformity can be improved by substrate rotation.
- Issues: radiation damage; not suitable for organic materials.



# Sputtering (overview)

- Sputter is carried out in a self-sustained glow discharge (plasma).
- Advantages:
  - Able to deposit a wide variety of metals, insulators and composites.
  - Replication of target composition in the deposited films.
  - Capable of in-situ cleaning prior to film deposition by reversing the electrode potential.
  - Better film quality and step coverage than evaporation, preferred deposition technique for *micro*-fabrication, where liftoff is not the easiest way for pattern transfer (can do wet etch using resist as mask, which is hard to control for nanofabrication).
- Disadvantages:
  - Substrate damage due to bombardment (usually not a serious problem)
  - More conformal coating than evaporation, not very suitable for liftoff.




Metal on top is connected to metal on bottom, so it is difficult to get off even though after resist is dissolved.

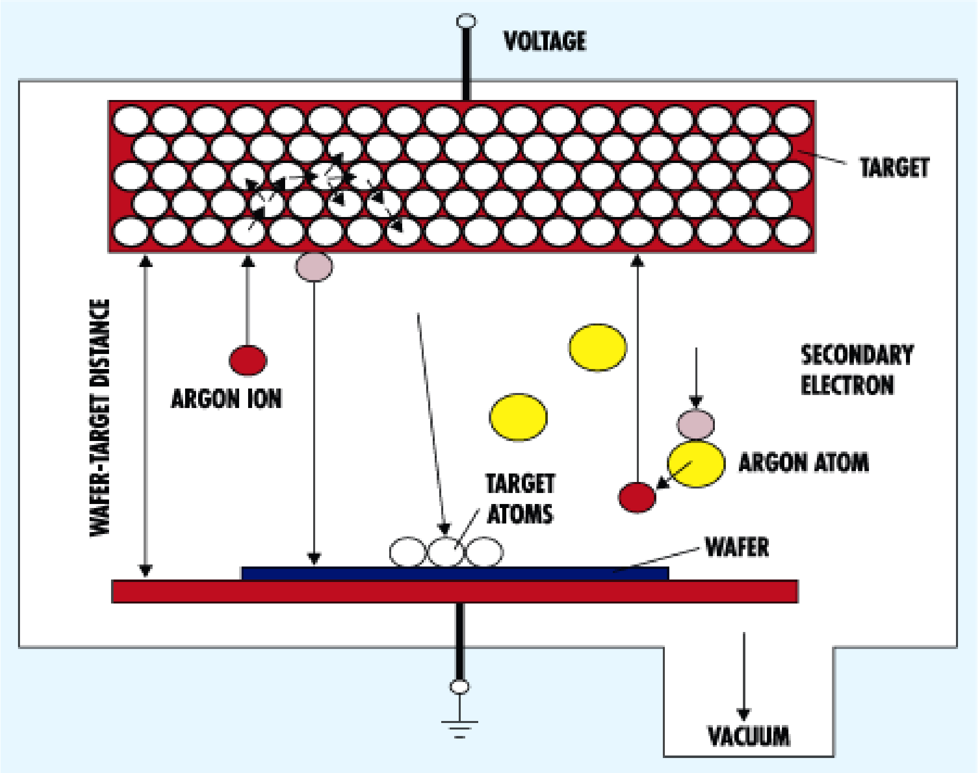
$$\boxed{P(\text{Torr})\times\lambda(\text{m}) = 7.8\text{x}10^{-5}}$$

Sputtering is typically done at order 10mTorr, so from above equation mean free path has order of 1cm, which means most metal atoms will experience some collisions with gas molecules before arriving at the wafer substrate. Collisions alter the directions, so the atoms hit the substrate from all directions. That is, the sidewall of resist structure will be covered with metals as well, which is usually connected to the metal on the trench/hole bottom (see figure above). This connection makes liftoff extremely difficult (even though resist is dissolved, the metals on top of resist is hard to get off since it is connected to the metal on the bottom through the metals on the sidewall).

On the contrary, evaporation is done at high vacuum, very directional, with very little metal on sidewall.

# Sputtering process

- Heavy inert gas is the major carrier, e.g. Ar.
- Reactive chemical species (e.g. $O_2$ to deposit oxide) may be introduced – reactive sputtering
- Sputtering process can be run in DC or RF mode (insulator must be run in RF mode)
- Major process parameters:
  - Operation pressure (~10-100mTorr)
  - Power (few 100W)
  - Substrate bias (in addition to self-bias)
  - Substrate temperature (20-500°C)




- Dielectric materials can also be deposited, but are usually formed by sputtering metals in $O_2$ , $N_2$, $CH_4$ gases, forming AlN, $Al_2O_3$, TiN, TiC…
- A wide range of industrial products use sputtering: computer disks, hard coatings for tools, metals on plastics.

# Material removal: etching processes

Etching is done either in “dry” or “wet” methods:

- Wet etch is cheap and simple, but hard to control (not reproducible), for pattern transfer purpose, not popular for *nano* (yet good for *micro*).
- Dry etching uses gas phase etchants in a plasma, both chemical and physical (sputtering process).
- Dry plasma etch can be used for many dielectric materials and some metals (Al, Ti, Cr, Ta, W…).
- For other metals, ion milling ($Ar^+$) can be used, but with low etching selectivity. (as a result, for metals that cannot be dry-etched, it is better to pattern them using liftoff)

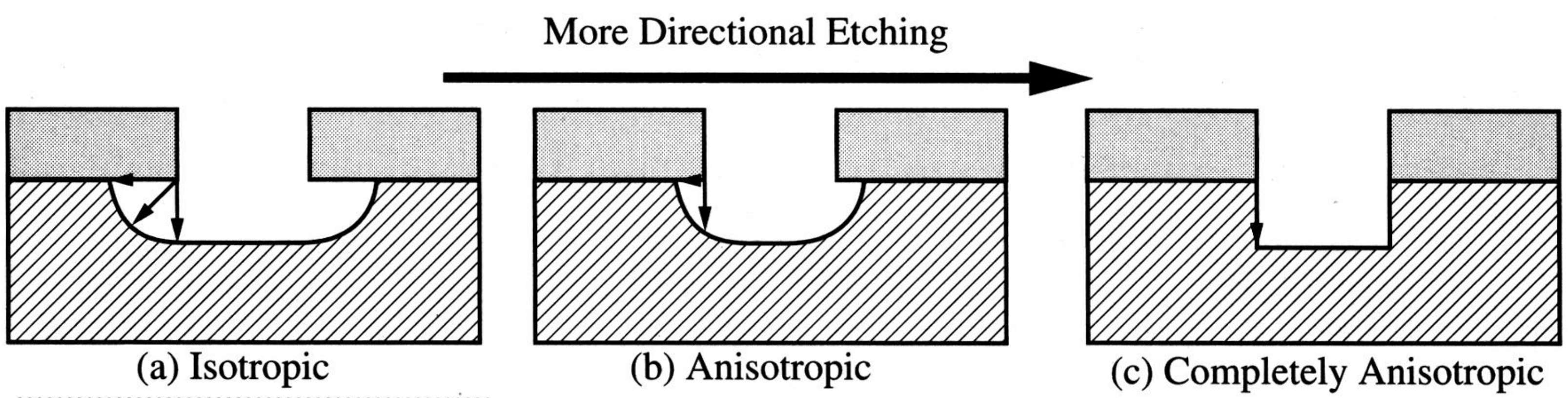



**Figure 10–3** Etch profiles for different degrees of anisotropic, or directional, etching: (a) purely isotropic etching; (b) anisotropic etching; (c) completely anisotropic etching.

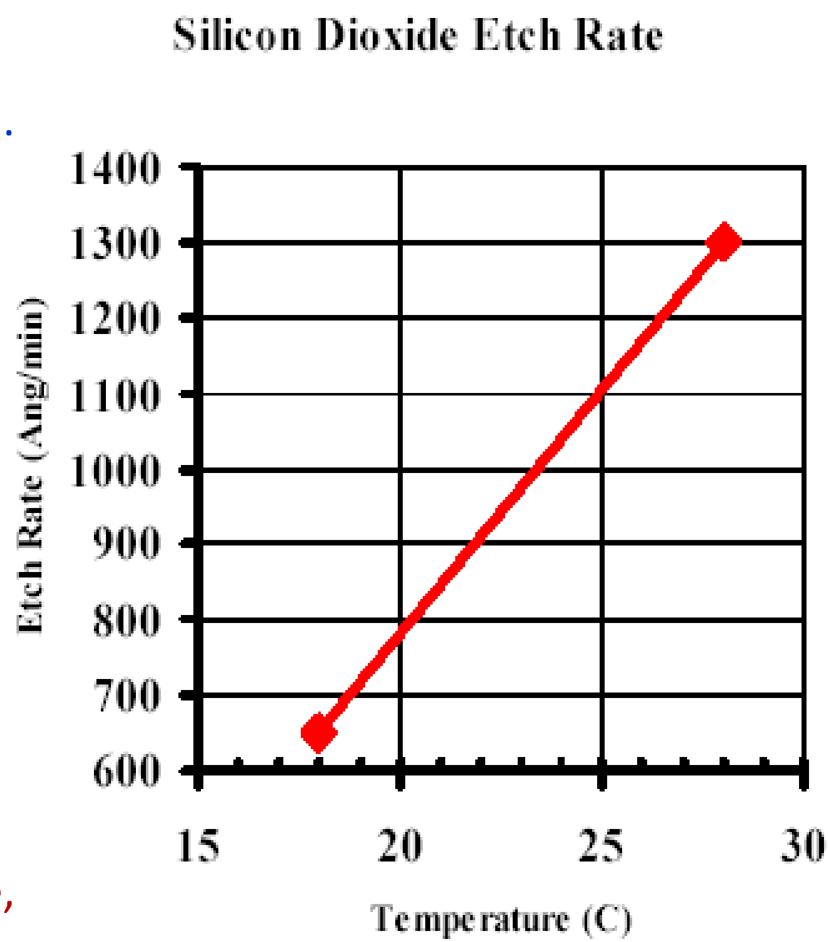
Generally speaking, chemical process (wet etch, plasma etch) leads to isotropic etch; whereas physical process (directional energetic bombardment) leads to anisotropic etch.

# Isotropic wet etching (silicon dioxide)

$$SiO_2 + 6HF \rightarrow H_2 \bullet SiF_6 + H_2O \rightarrow H_2 \uparrow + SiF_6 + 2H_2O$$

- Etch is isotropic and easily controlled by dilution of HF in $H_2O$.
- Thermally grown oxide etches at
  - 120nm/min in $6H_2O$:1HF
  - ~1 μm/min in 49 wt% HF (i.e. undiluted HF).
- Faster etch rate for doped or deposited oxide.
- High etch selectivity ($SiO_2$/Si) > 100
- Buffered HF (BHF) or buffered oxide etchant (BOE) provides consistent etch rates
  - In regular HF etches, HF is consumed and the etch rate drops.
  - HF buffered with $NH_4F$ to maintain HF concentration $6NH_4F$:1HF

$$NH_4F \rightarrow NH_3 \uparrow + HF$$



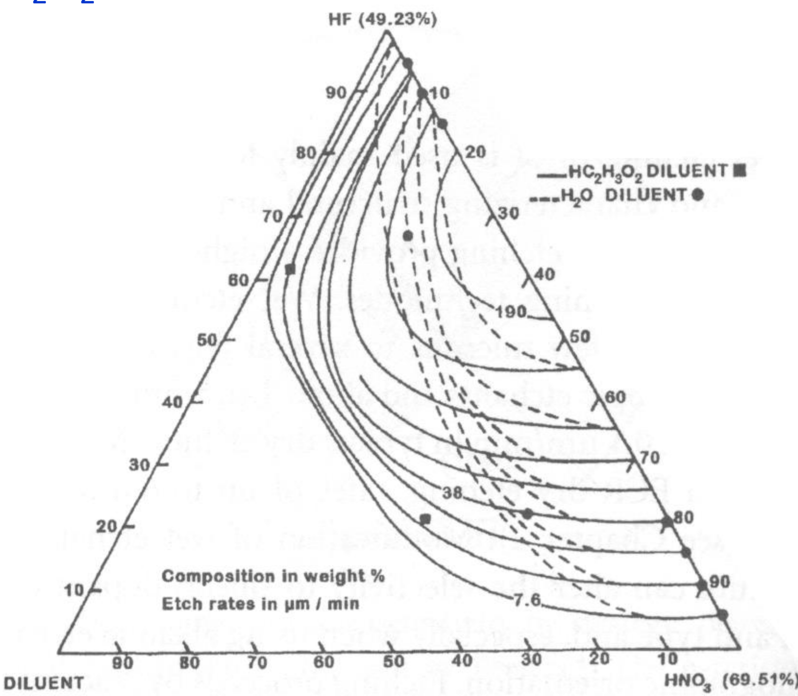

HF is very dangerous, it "etches" bones. One usually doesn't feel the pain before it is too late, which makes it even more dangerous.

# Isotropic etch (silicon)

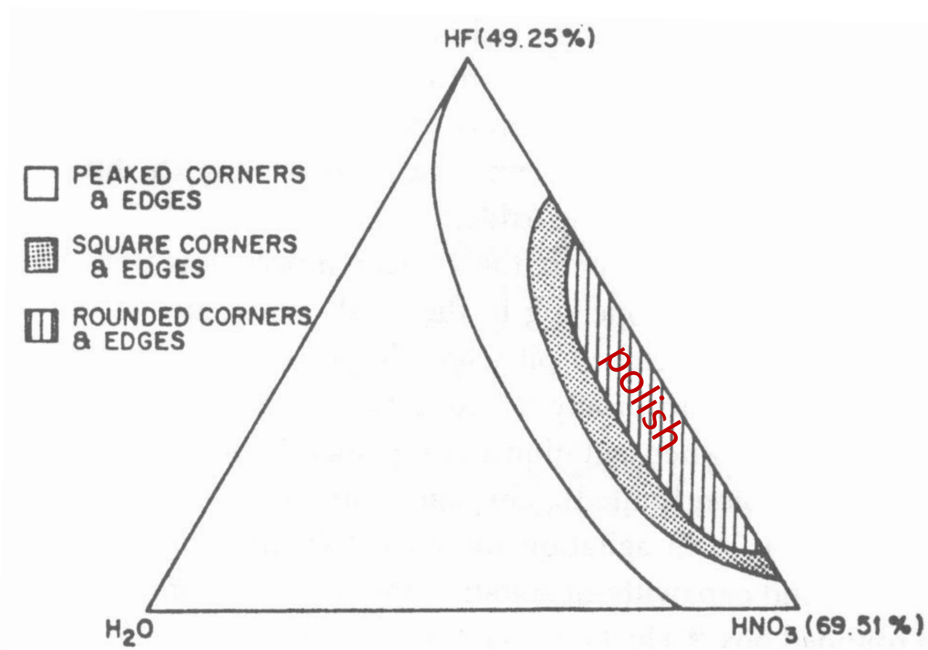
- Silicon is etched by nitric acid and hydrofluoric acid mixtures
- Use oxidant $HNO_3$ to oxidize silicon to form silicon dioxide, followed by HF etch of silicon dioxide.

$$Si + HNO_3 + 6HF \rightarrow H_2SiF_6 + HNO_2 + H_2O + H_2$$

- Excess nitric acid results in a lot of silicon dioxide formation and etch rate becomes limited by HF removal of oxide (polishing).
- $CH_3COOH$ or $H_2O$ can be added as diluents, but etch differently (acetic acid does not reduce oxidization power of the nitric acid, but water does).
- $H_2O_2$ (oxidant) and HF mixture can also etch Si.




Si iso-etch curves using $CH_3COOH$ or $H_2O$ diluent



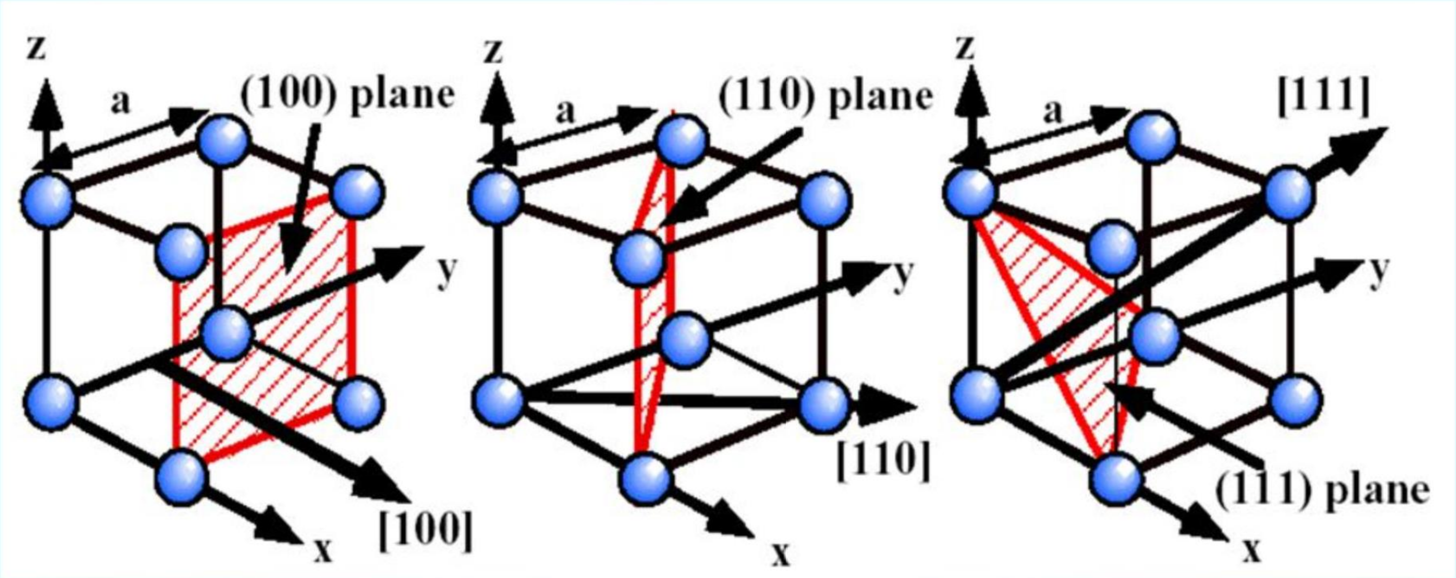

Topology of etched Si surfaces



Marc Madou, “Fundamentals of microfabrication”, 1997 page 164-165

# Silicon Lattice Planes

## Silicon Crystal Structure

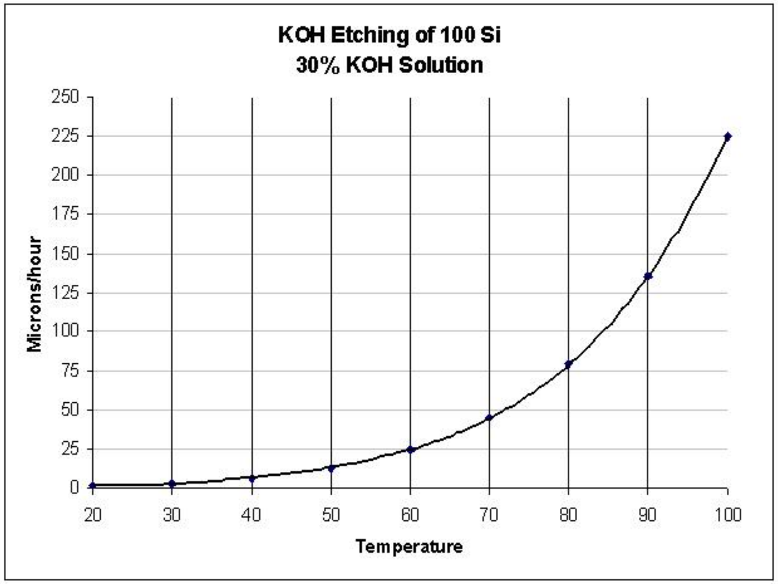



Crystals are characterized by a unit cell which repeats in the x, y, z directions.

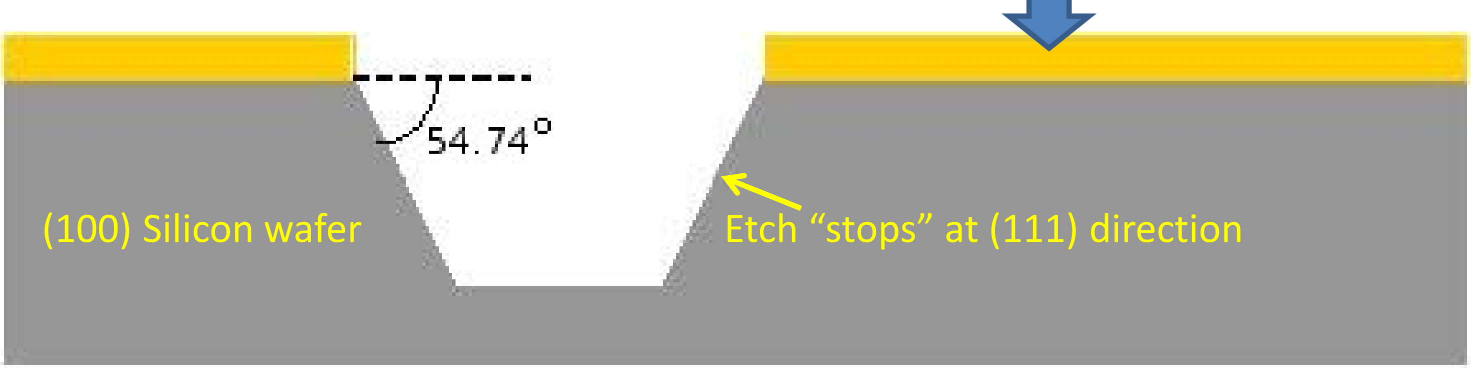
- Planes and directions are defined using x, y, z coordinates.
- [111] direction is defined by a vector of 1 unit in x, y and z.
- Planes defined by "Miller indices" – Their normal direction (reciprocals of intercepts of plane with the x, y and z axes).

# An-isotropic wet etching (silicon)

- Orientation selective etch of silicon occur in hydroxide solutions because of the close packing of some orientations relative to other orientations
  - Etch rate: R(111) << R(100) < R(110)
- <100> direction etches faster than <111> direction, with etch rate
  - R(100) > 100 × R(111)
  - It is reaction rate limited
- Most useful for $Si_3N_4$ membrane fabrication (etch through Si wafer thickness, stop at nitride).




Etch mask: $SiO_2$ or $Si_3N_4$ or Cr/Au




Popular etchant: KOH, TMAH (tetra methyl ammonium hydroxide)

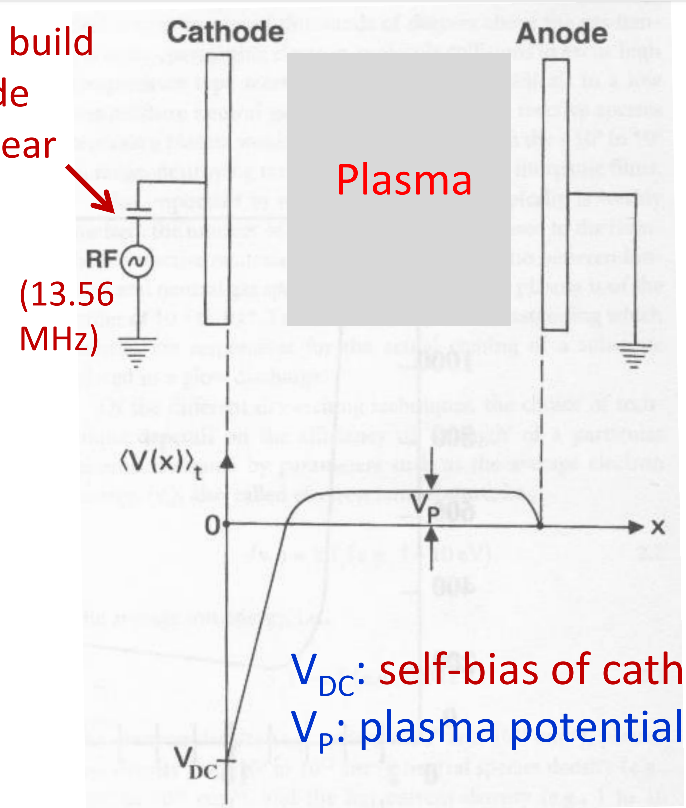
# Wet “etch” for wafer cleaning

1. Solvent cleaning: acetone, then methanol, then iso-propanol (2-propanol).
2. Pirahna: $H_2SO_4 : H_2O_2 = 3 : 1$, remove organic and metals, can heat to above 60$^o$C.
3. RCA clean: (all three steps are needed for thermal oxidation or LPCVD nitride)
   - $H_2O_2$:$NH_4OH$:$H_2O$=1:1:5, 80$^o$C for 10min. (remove organics and particles)
   - $H_2O_2$:HCl:$H_2O$=1:1:5, 80$^o$C for 10min. (remove metals)
   - HF:$H_2O$=1:50, 30sec (for Si, remove native oxide and contaminants on top).

Usually, only the first step is enough for such as nanoimprint mold fabrication.

# RF plasma (RF: radio frequency)

Due to blocking capacitor, electrons build up on the cathode, but not on anode (grounded). So large *self*-bias only near cathode.




$V_{DC}$: self-bias of cathode

$V_P$: plasma potential

Time-averaged potential distribution

If BOTH electrodes are blocked by a capacitor:

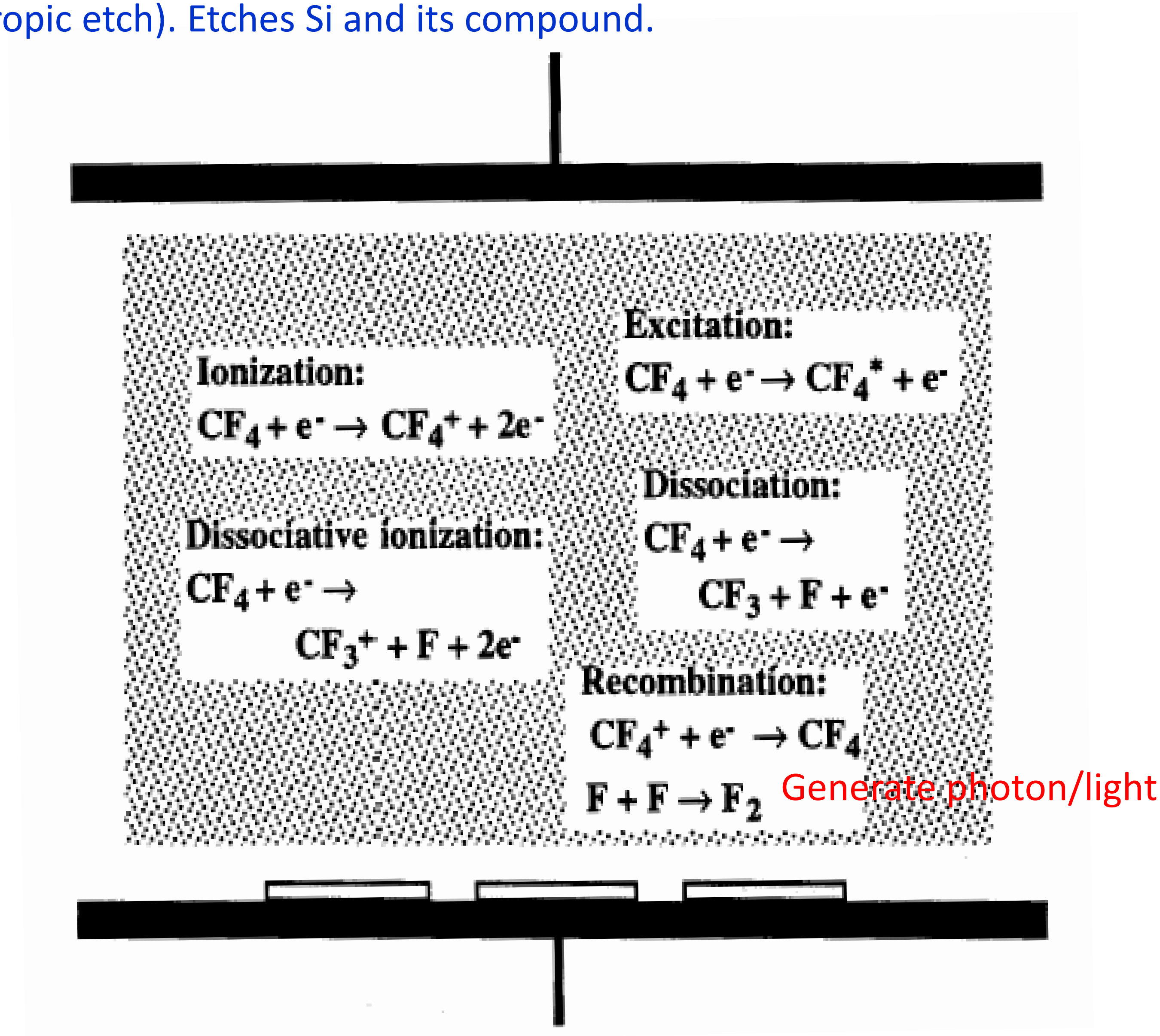
- Relative voltage drop near each electrode depends on its area A. In theory, $V \propto (A)^{-4}$; but experimentally $V \propto (A)^{-2}$.
- Thus if connect anode to chamber wall (very large A), DC voltage drop near cathode will be very high.

In a plasma, electrons and ions are NOT in thermal equilibrium.

Electrons don't lose energy efficiently by collision due to the huge mass difference, so electrons are very "hot" with energy 1-10eV (equivalent to $10^3$-$10^4$K); to a certain degree, plasma is very reactive because the electrons are "hot", as if the reactants were heated. (ions lose energy by collision, so they are "cold" with energy ~0.04eV)

# RF plasma chemistry

$CF_4$ plasma, most popular gas. Other popular F-gases are $CHF_3$ and SF6 (isotropic etch). Etches Si and its compound.



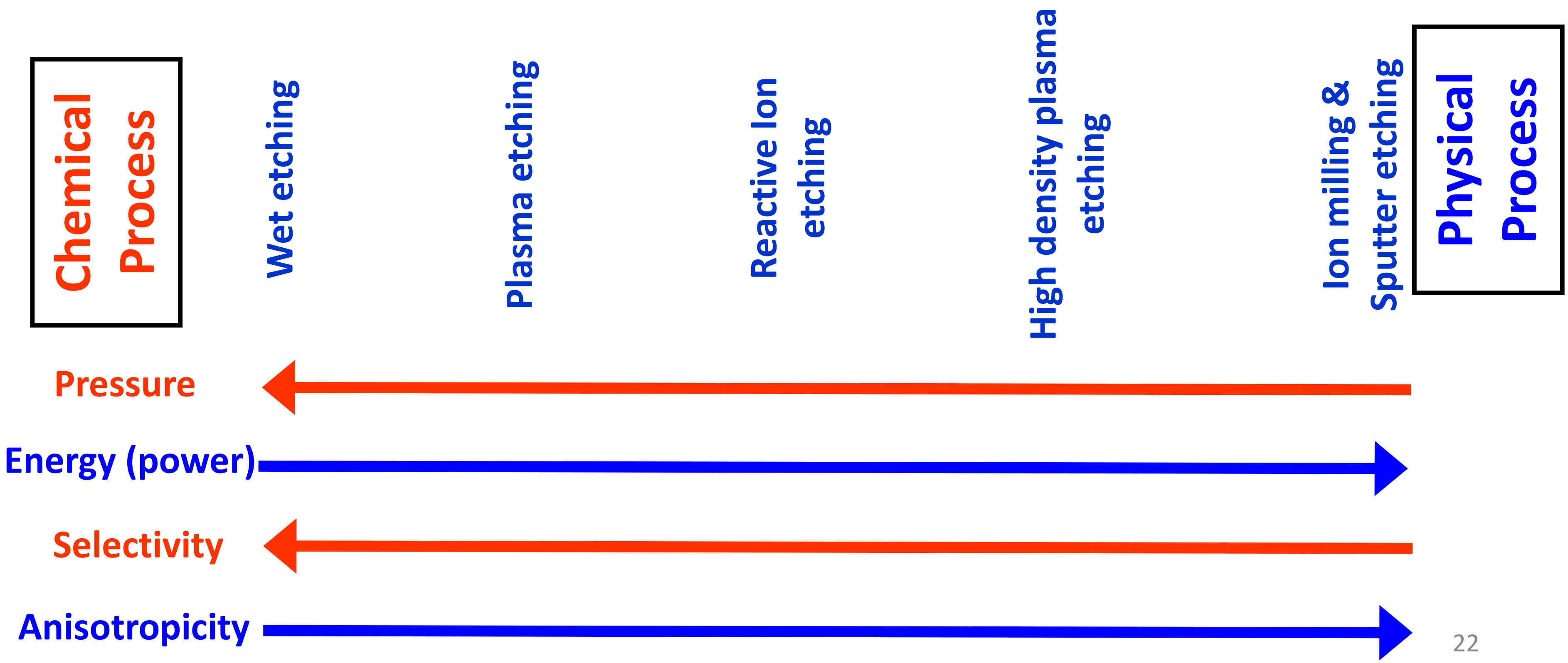
# Types of dry etching processes

| Type of Etching | Excitation Energy | Pressure |
| --- | --- | --- |
| **Gas/Vapor Etching** | none | high (760-1 torr) |
| - *isotropic, chemical, very selective* (e.g. $XeF_2$ gas etch Si even without plasma) | | |
| **Plasma Etching** | 10's to 100's of Watts | Medium (>100 torr) |
| - *isotropic, chemical, selective* | | |
| **Reactive Ion Etching** | 100's of Watts | Low (10-100 mtorr) |
| - *directional, physical & chemical, fairly selective* | | |
| **Sputter Etching** | 100's to 1000's of Watts | Low (~10 mtorr) |
| - *directional, physical, low selectivity* (e.g. ion beam etching/milling using $Ar^+$) | | |

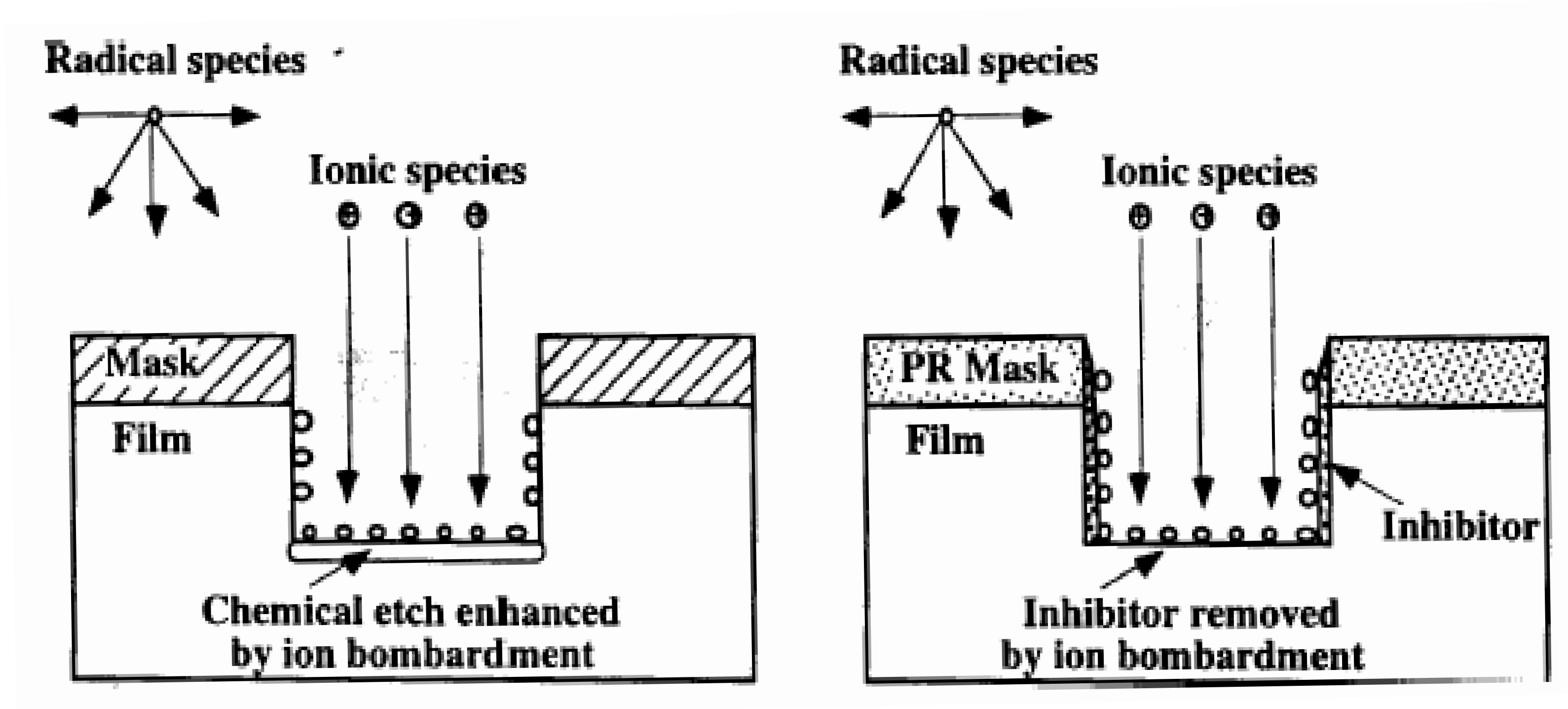
RIE is most popular for nanofabrication.
Plasma etching can be used to remove a layer such as photoresist residue.

# Plasma etching mechanism

- Chemical etching: free radicals react with material to be removed.
- Physical etching or sputtering: ionic species, accelerated by the built-in electric field (self-bias), bombard the materials to be removed. E.g. sputter cleaning using Ar gas in sputter deposition system, extremely slow unless very high bias voltage.
- Ion enhanced etching: combined chemical and physical process, higher material removal rate than each process alone. E.g. reactive ion etching (RIE), which is the most widely used dry etching technique.



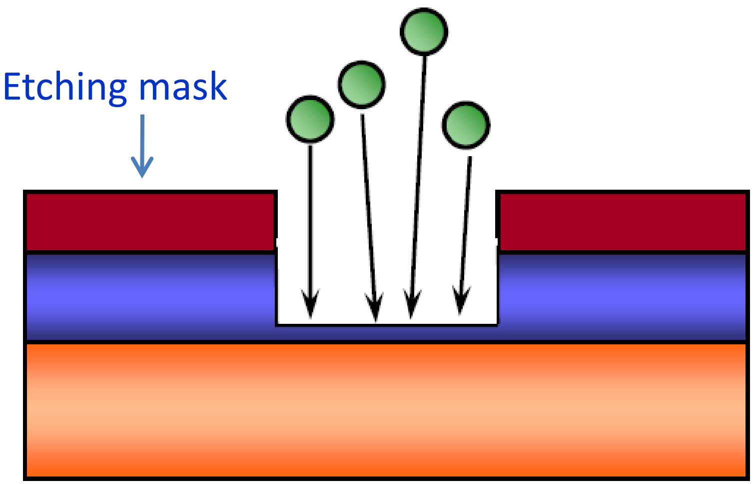
# Ion enhanced plasma etch (IEE): chemical etch assisted by physical bombardment



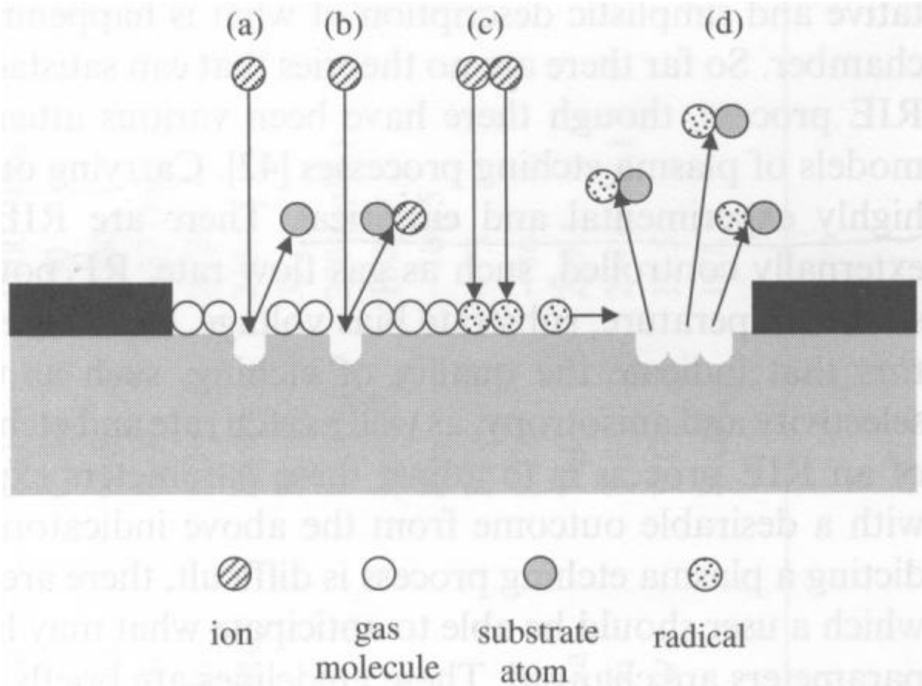

- IEE is an anisotropic and highly selective etching process.
- Reactive ion etch (RIE) is the most popular IEE.
- Ion bombardment can enhance one of the following steps during chemical etch: surface adsorption, etching reaction, by-product formation, by-product removal (inhibitor layer) and removal of un-reacted etchants.

# Reactive ion etch (RIE)

Anisotropic etching with vertical sidewall




Schematic RIE process




a) Ion sputtering, b) reactive etching, c) radical formation, d) radical etching (most important)

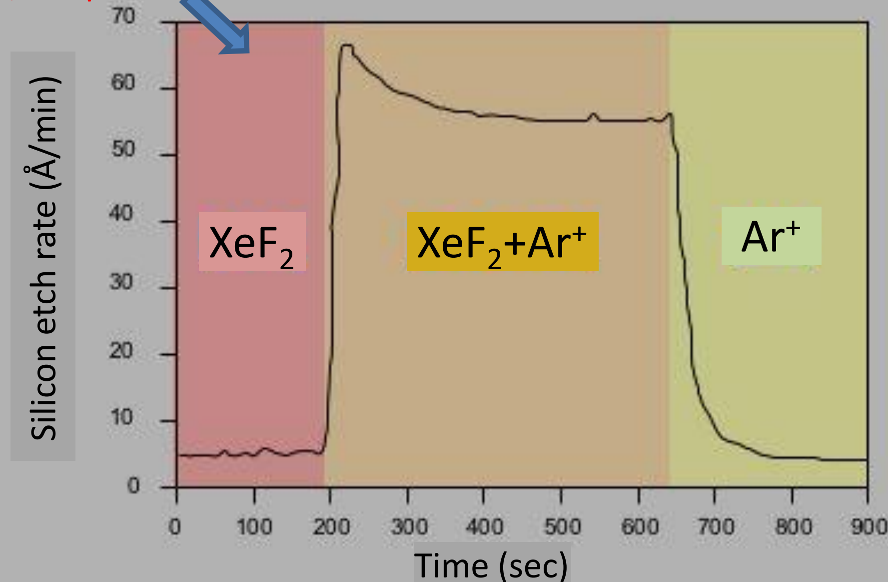
- The high anisotropy in RIE is due to the directional nature of the ion bombardment.
- High selectivity arises from the chemical interactions.
- Volatile product of etching prevent redeposition.
- Well known for semiconductors and dielectrics.

# RIE: first proof of etching mechanism

## The Role of Ion Bombardment in Plasma Etching

- Ion / Neutral Synergy in Plasma Etching -

Gas phase etch, NO plasma




**From:** J.W. Coburn, H.F. Winters, J. Appl. Phys. 50, 3189, (1979)

Very slow etch when pure chemical or physical etch *alone*

In this classical experiment, the silicon etch rate increased by one order of magnitude when the ion beam is turned on. This shows the importance of the ion/neutral synergy in plasma etching.

The reaction of XeF2 with silicon is described by the following reaction scheme:

g: gas, p: ?, s: solid

$XeF_2(g) \quad XeF_2(p)$

$XeF_2(p) + Si(s) \quad Xe(g) + SiF_2(s)$

$XeF_2(p) + SiF_2(s) \quad Xe(g) + SiF_4(g)$ ⇨ Enhanced by ion bombardment

$SiF_2(s) \quad SiF_2(g)$ (over 600K)



Note: $XeF_2$ gas can etch Si even without plasma

For more information on the reaction of XeF2 with silicon see website of TU Eindhoven: http://www.phys.tue.nl/aow/Pages/Research/spontaneous_etching.htm

# Anisotropy due to ion bombardment

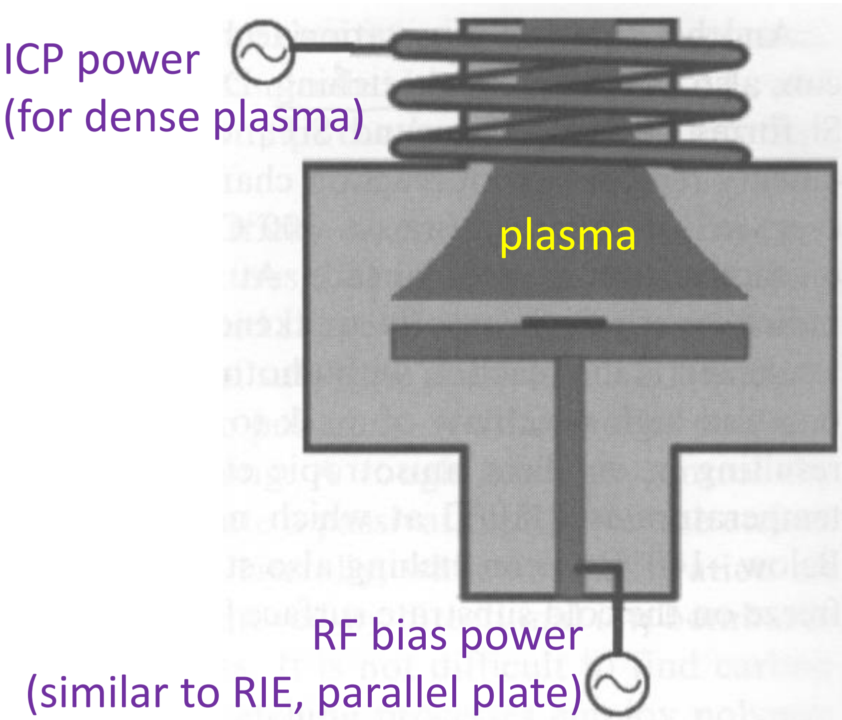
- Anisotropic etch with vertical sidewall is desired for micro- and nano-fabrication.
- But directional ion bombardment doesn't always lead to noticeable anisotropic etch.
- For instance, $SF_6$ etch of Si is pretty isotropic with large undercut like wet etch.
- To achieve anisotropy, there are two mechanisms:
    - Energy-driven anisotropy: bombardment by ions disrupts an un-reactive substrate and causes damages such as dangling bonds and dislocations, resulting in a substrate more reactive towards etchant species (electrons or photons can also induce surface activation).
    - Inhibitor-driven anisotropy: in this case etching leads to the production of a surface covering agent (a passivation/inhibition layer). Ion bombardment removes this layer from horizontal surface (vertical surface - sidewall remain passivated), and reaction with neutrals proceed on these un-passivated surfaces only. E.g. RIE of $SiO_2$ using $CHF_3$ gases to form fluorocarbon passivation layer.

# Review of thin film deposition and etching techniques important for nanofabrication

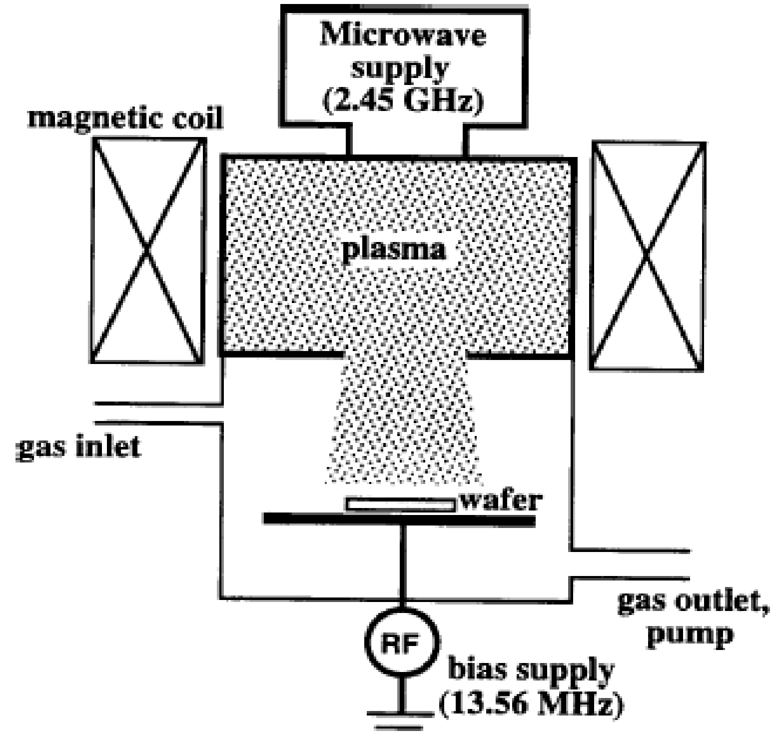
- Physical vapor deposition (PVD): evaporation and sputtering.
- Wet chemical etching.
- Plasma etching – reactive ion etching
- ICP and deep Si etching
- Stencil mask lithography

# High density plasma system

## Inductively coupled plasma (ICP)
(four systems at Waterloo)



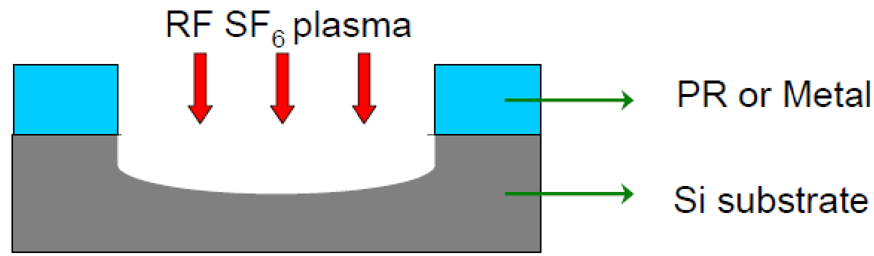

## Electron cyclotron resonance plasma
(less common)



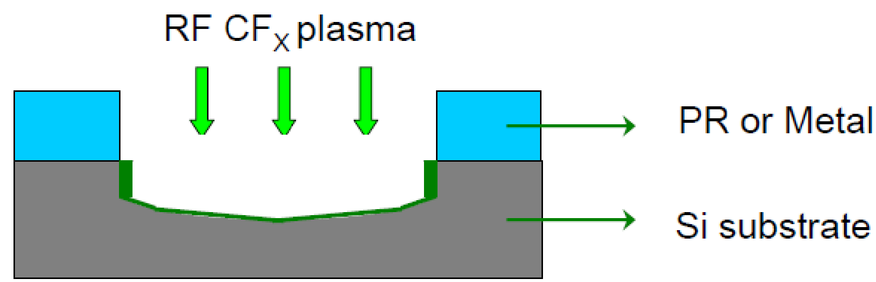

- High magnetic field in the coil, so electrons move in circles with long path, leading to higher collision and ionization probability.
- Plasma density $10^{11}$ - $10^{12}$ ions/cm$^3$; may have low operation pressure 1 - 10mTorr.
- Independent control of RF bias (ion energy, directionality) and ion density (plasma density, chemical etching rate).
- High etching rate than RIE, but may be less anisotropic due to increased chemical etching.
- ICP etcher can be used as a pure RIE etcher by turning off the ICP component.

# Deep Si etch: ICP - “BOSCH” process

**(a) Etch Step**

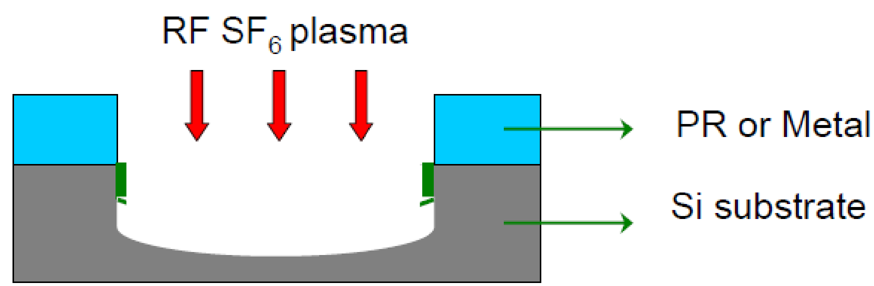



**(b) Passivate Step**




The passivation layer (dark green color) on the *bottom* (not that on the sidewall) is removed during the etch step due to ion bombardment (directional physical etching).

**(a’) Etch Step**




- Alternating etch and (inhibitor/passivation layer) deposition step.
- $SF_6$ etch 5-13 sec; followed by $C_4F_8$ fluorocarbon polymer deposition 5-10 sec.
- Etch rate several $\mu$m/min, capable of etching several hundred $\mu$m with vertical walls.
- Side wall is rough, depending on cycle times (longer cycle, rougher).
- More popular for MEMS, less common for nano-fabrication due to sidewall roughness.

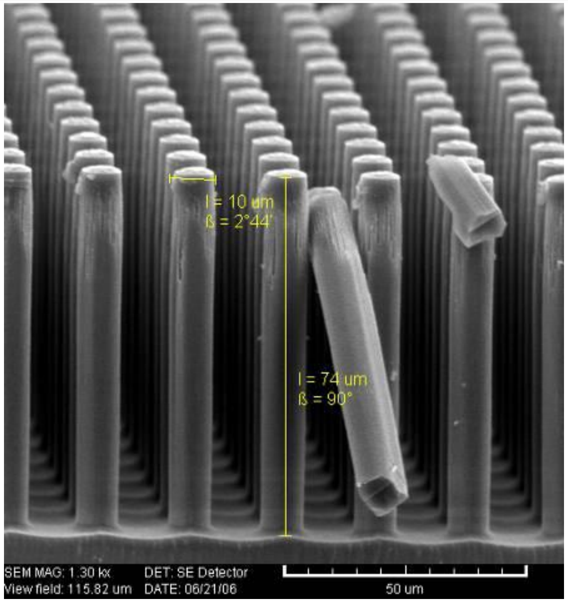
MEMS: micro electro mechanical system

# Deep Si etch - Bosch process

Table 6.4 Typical "Bosch" process conditions

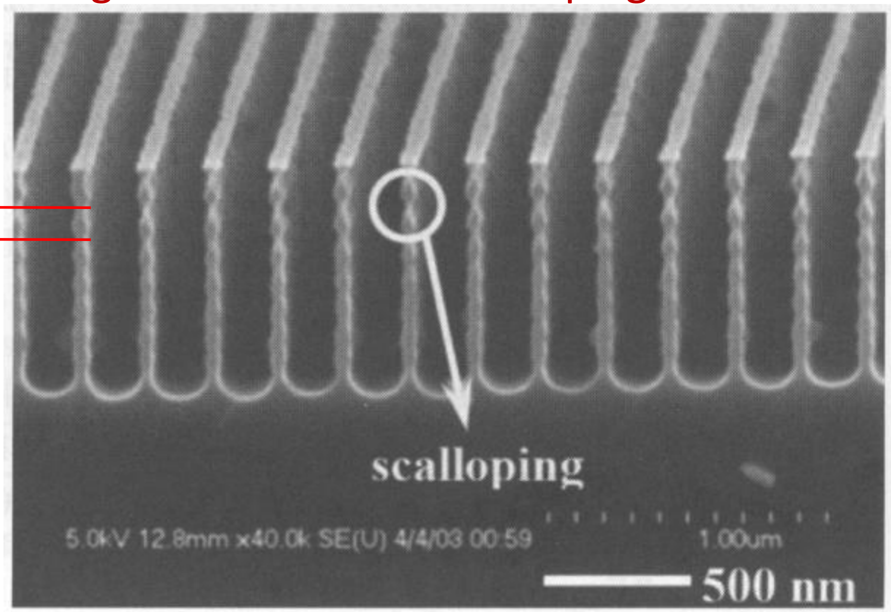
| Process parameters | Passivation | Etching |
|---|---|---|
| $C_4F_8$ | 85 sccm | 0 sccm |
| $SF_6$ | 0 sccm | 130 sccm |
| RF power at stage | 0 W | 12 W |
| RF power from coil | 600 W | 600 W |
| Cycle time | 7.0 s | 9.0 s |
| Delay time | 0.5 s | 0.5 s |
| Etch rate | | 1.5–3 $\mu$m min$^{-1}$ |

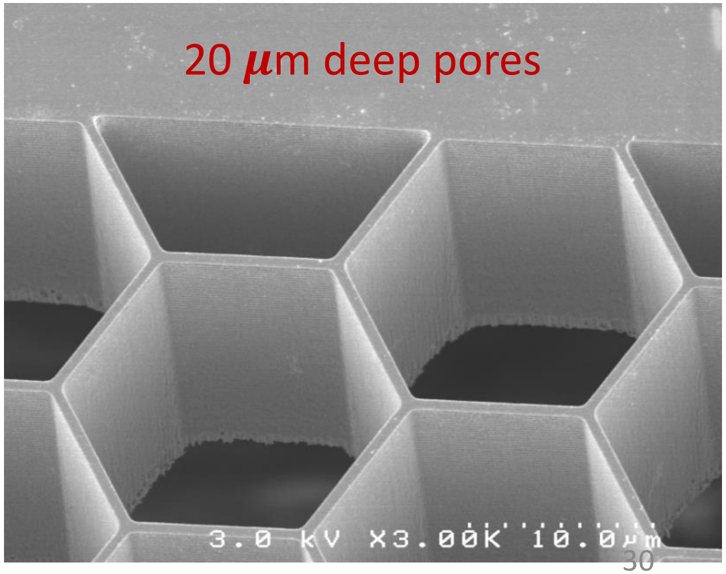
sccm: standard cubic centimeter per minute




Rough sidewall due to scalloping effect.

1 cycle

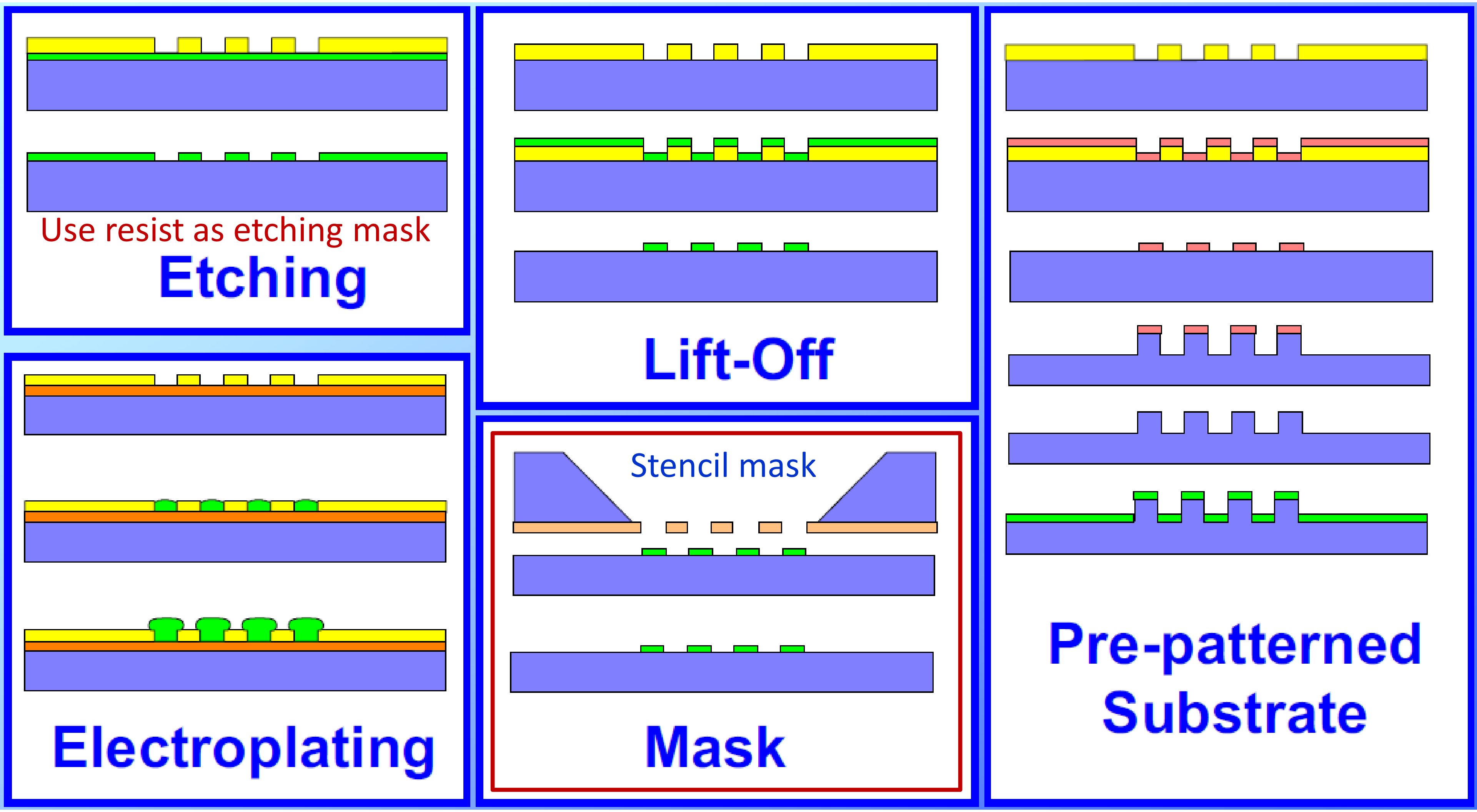


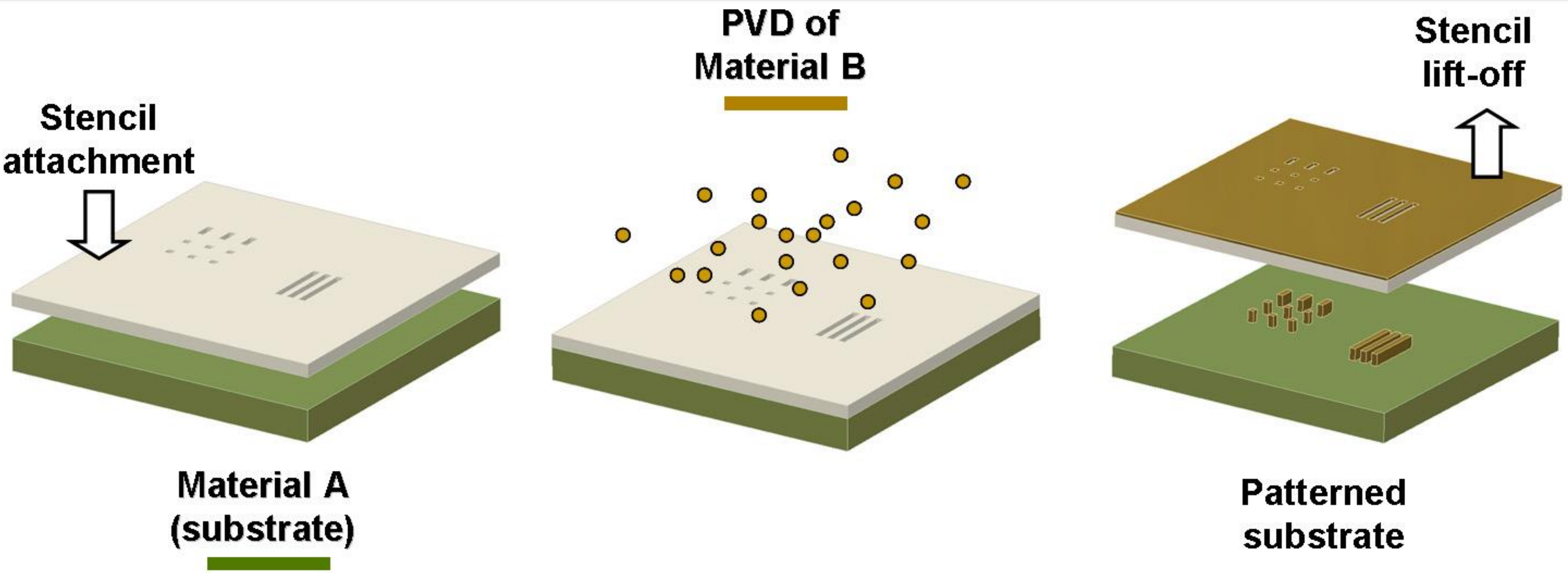
# Review of thin film deposition and etching techniques important for nanofabrication

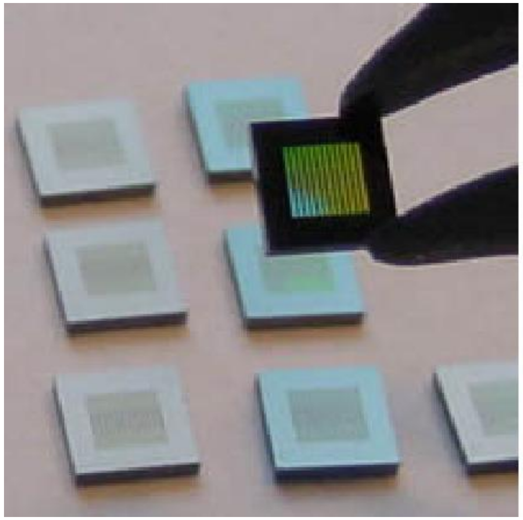
- Physical vapor deposition (PVD): evaporation and sputtering.
- Wet chemical etching.
- Plasma etching – reactive ion etching
- ICP and deep Si etching
- Stencil mask lithography

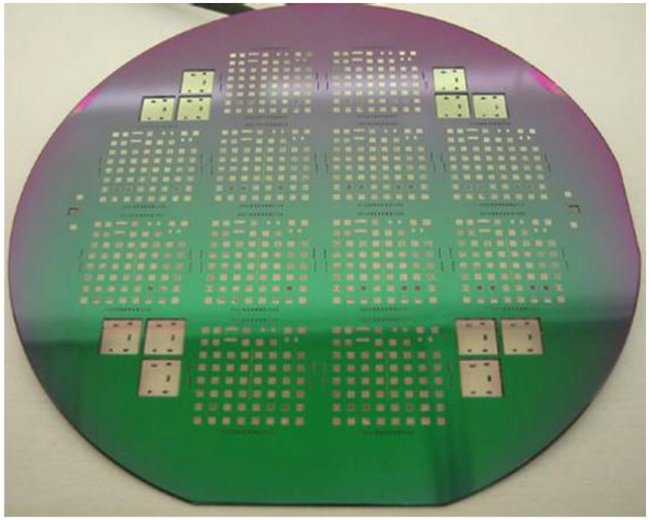
# Pattern transfer: summary



# Stencil “Shadow” mask lithography

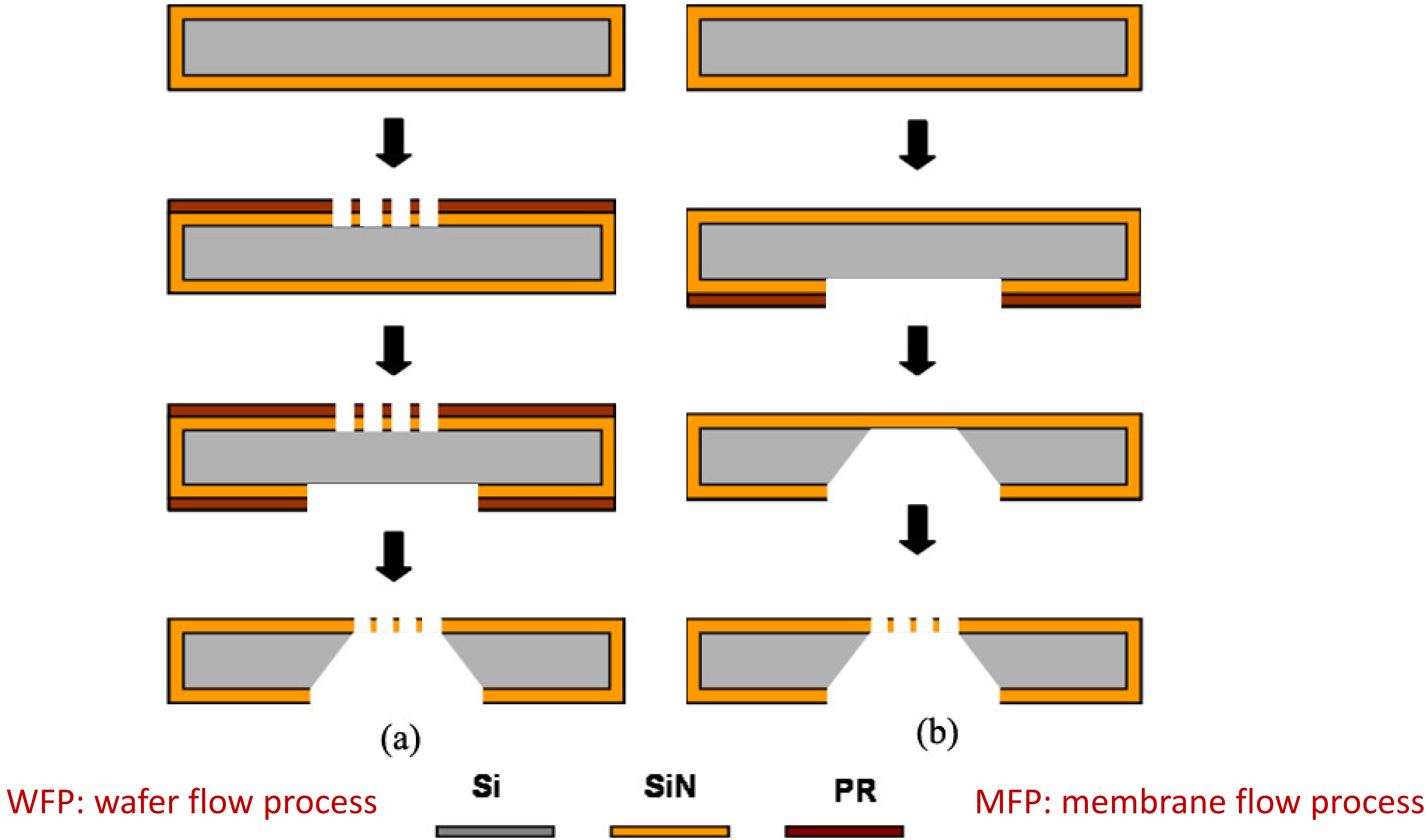




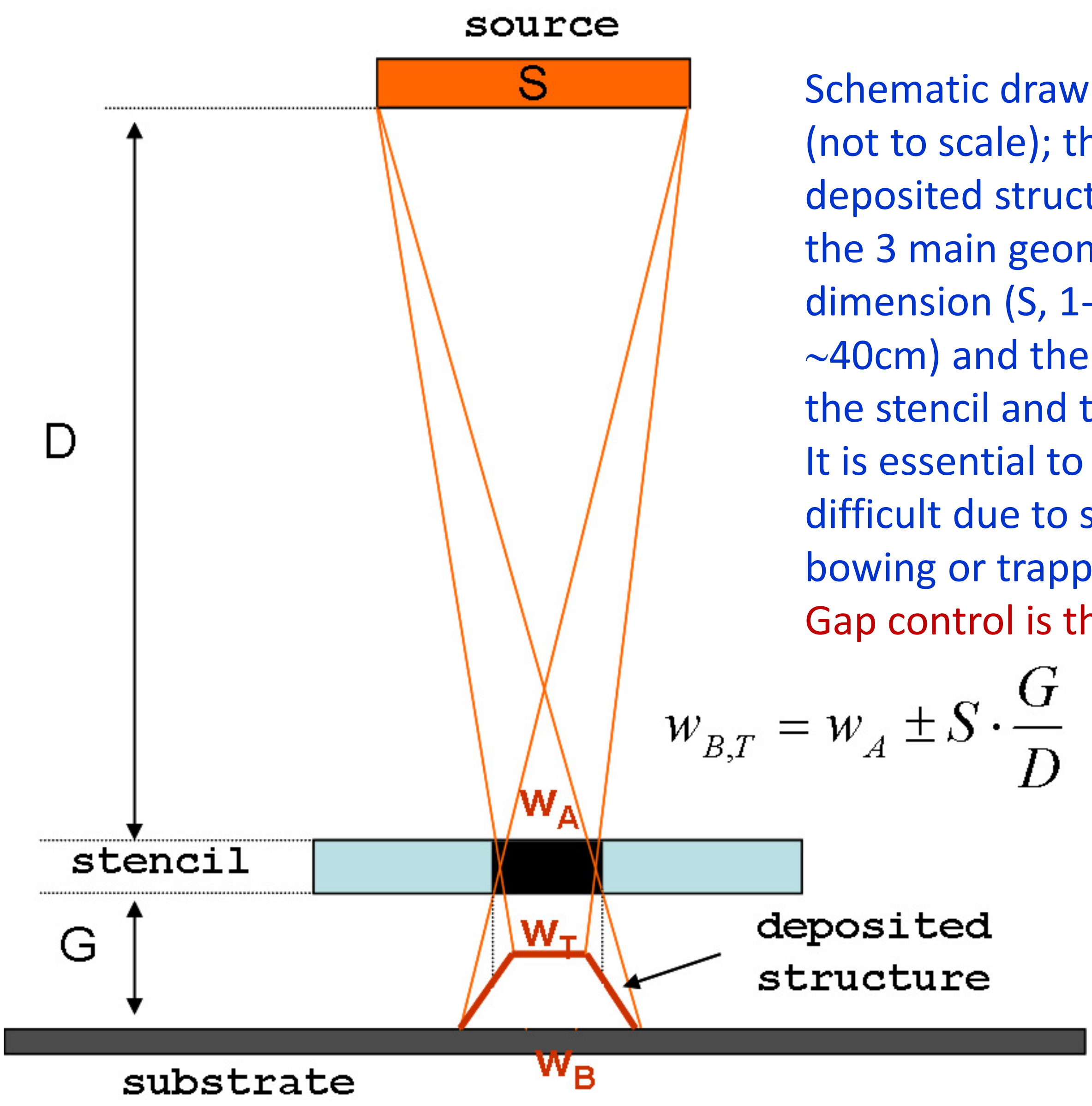
Stencil masks

# Stencil “Shadow” mask fabrication




Schematic drawings of WFP (a) and MFP (b) sequences used for nanostencil fabrication.
Colors chart: grey- silicon wafer; orange- low-stress LP-CVD SiN; brown- photoresist.

# Deposition through shadow mask




Schematic drawing of shadow-mask deposition (not to scale); the enlargement of the deposited structure $W_B$ is highly dependent on the 3 main geometric parameters: the source dimension (S, 1-2cm); distance source-stencil (D, ~40cm) and the gap (G, ideally G≈0) between the stencil and the substrate.

It is essential to control the gap, which is very difficult due to stress-induced membrane bowing or trapped particles.

Gap control is the main issue for this technique.

$$w_{B,T} = w_A \pm S \cdot \frac{G}{D}$$

# Photomask

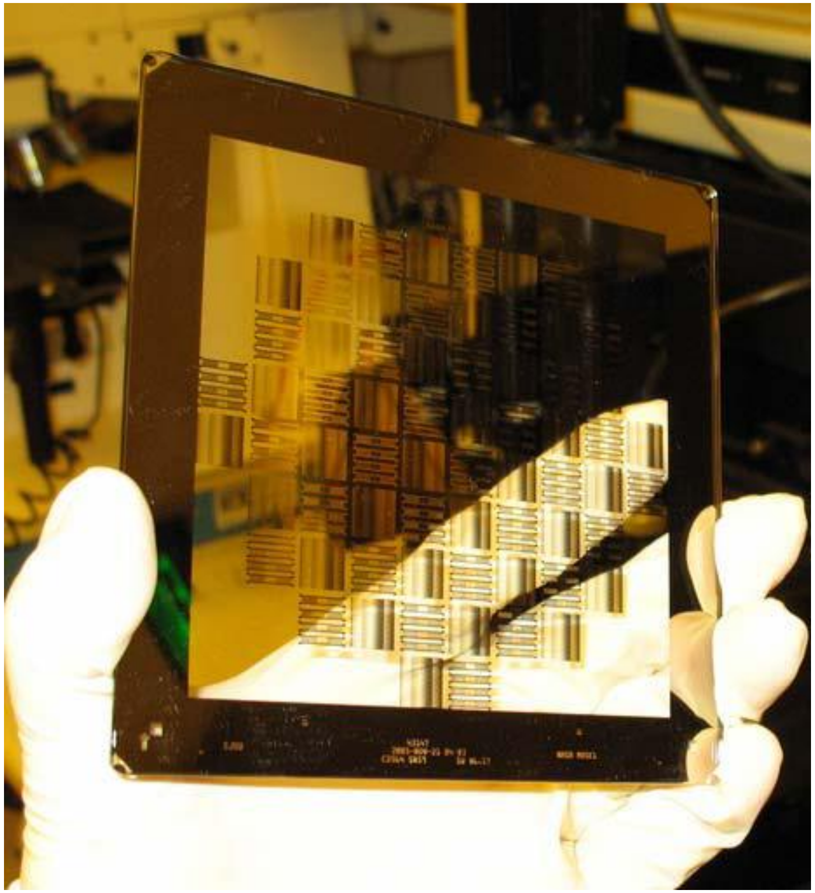
Types:

- Cr on soda lime glass and on quartz glass (most popular).

  (Quartz has low thermal expansion coefficient and low absorption of light, but more expensive; needed for deep UV lithography).

Polarity:

- Light-field, mostly clear, drawn feature is opaque.
- Dark-field, mostly opaque, drawn feature is clear.

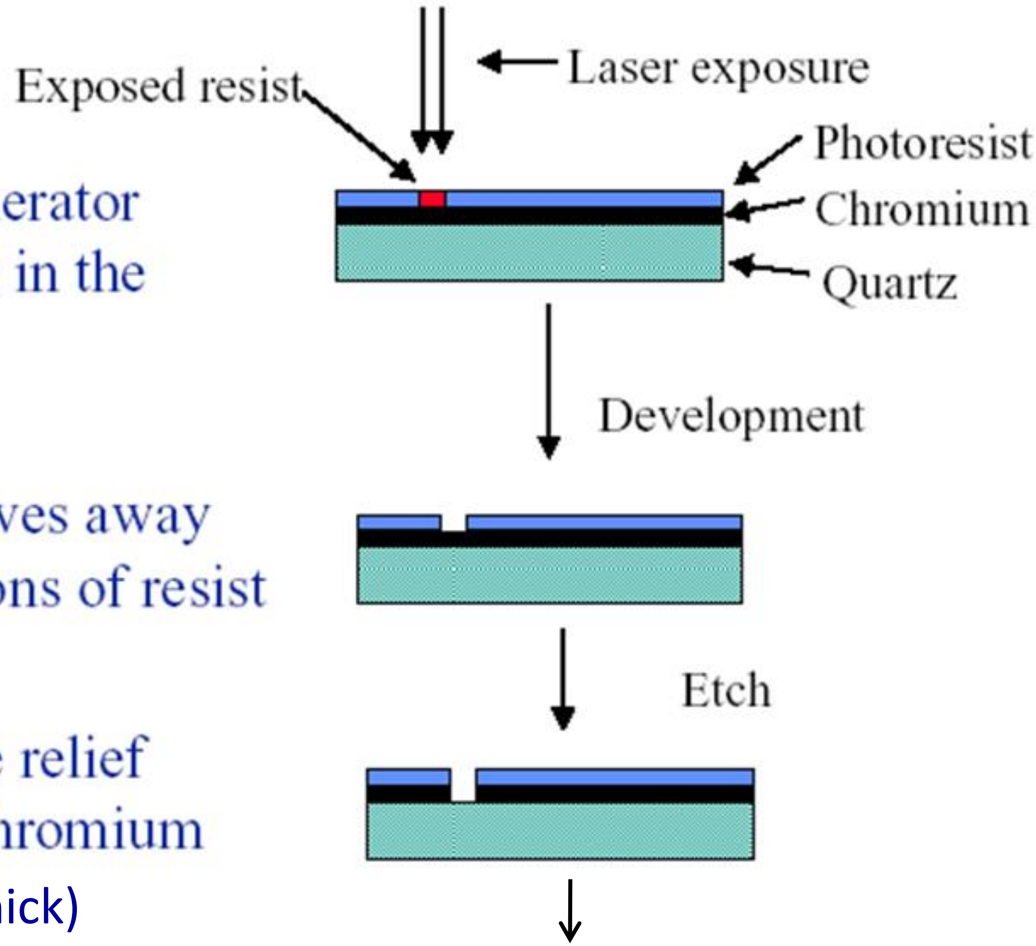


*Light-field* photomask

# Photomask (Cr pattern on quartz) fabrication

Laser beam writing:

- Similar to photolithography, but use a *focused* laser beam.
- It is a direct-write technique - no mask is needed.
- Resolution down to a few 100nm, cheaper than electron-beam writing.




- Laser pattern generator exposes a pattern in the resist
- Developer dissolves away the exposed regions of resist
- Etch transfers the relief image into the chromium

(Cr is ~100nm thick)

**Remove the resist.**

# Photomask fabrication by electron beam lithography