

*Principles of Micro- and Nanofabrication  
for Electronic and Photonic Devices*

# Cleanroom, Wafer Clean and Gettering

**Xing Sheng 盛兴**

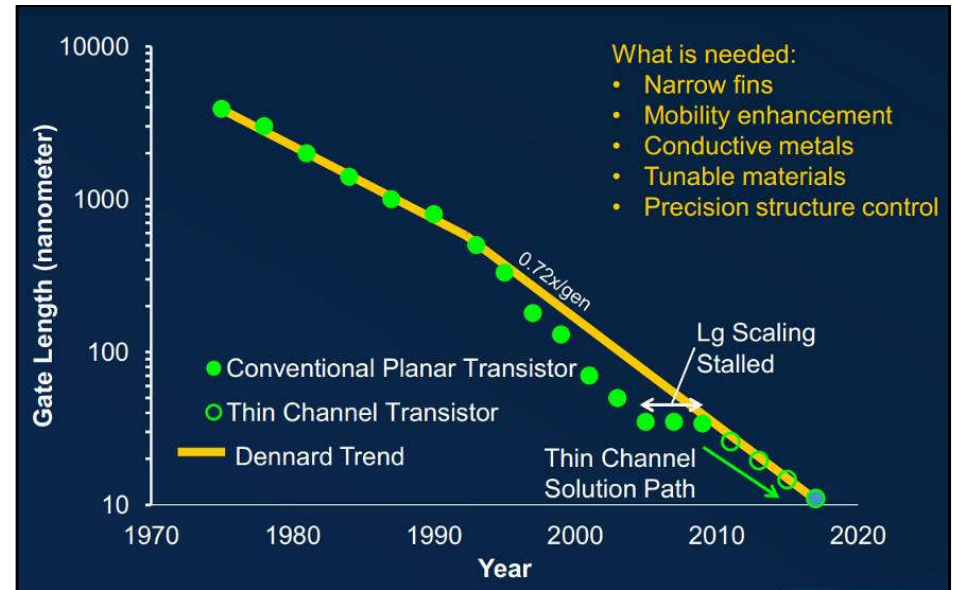
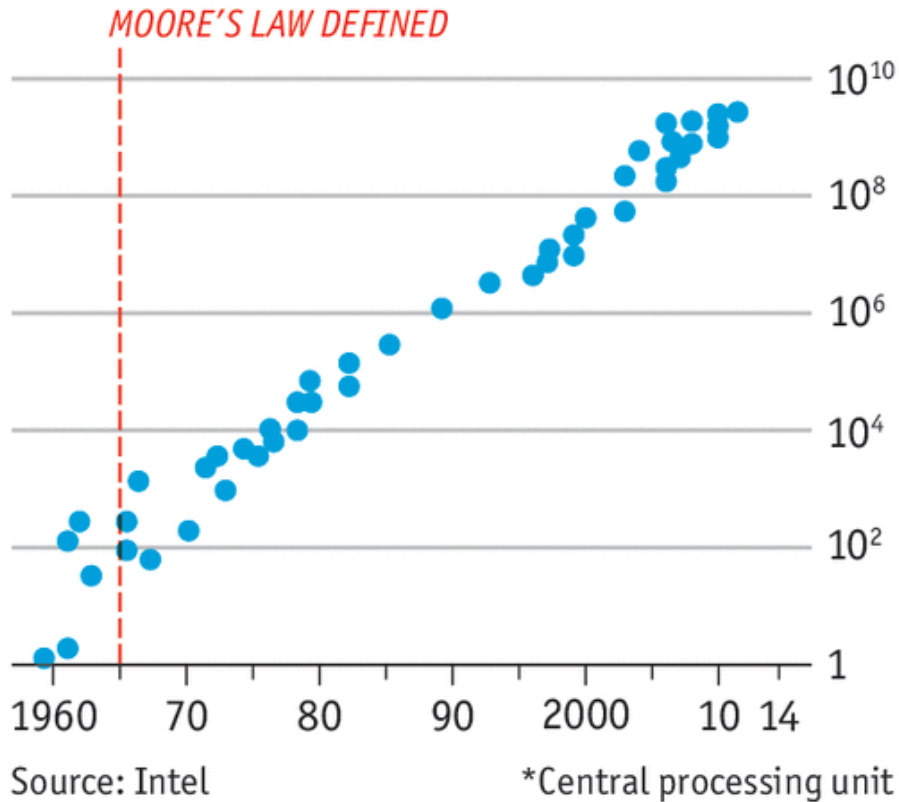


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# Integrate Circuits

## Moore's law



Economist.com

transistor number

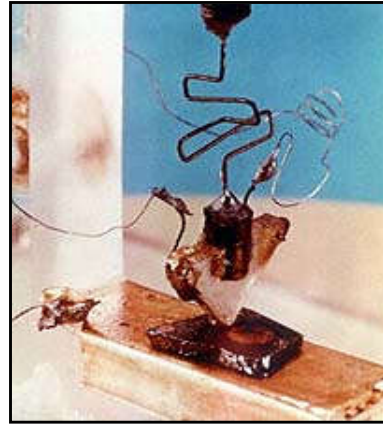


transistor size



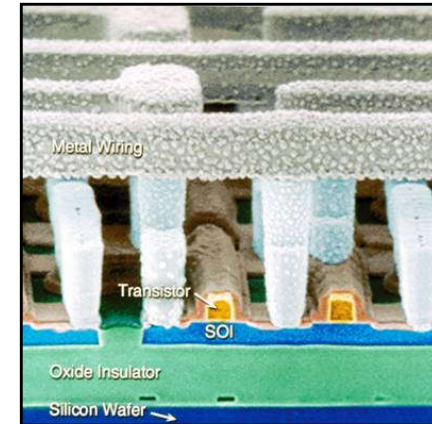
# Technology Evolution

1947



1 cm

today



< 100 nm



# Factory Evolution

## ■ cost of new fab

- 1967      2 million \$
- 2010      10 billion \$

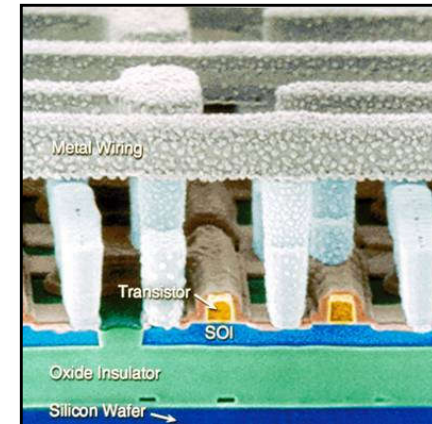
Video TSMC



武汉新芯集成电路制造有限公司 厂区鸟瞰图

**2016 长江存储, 武汉  
24 billion \$\$**

today

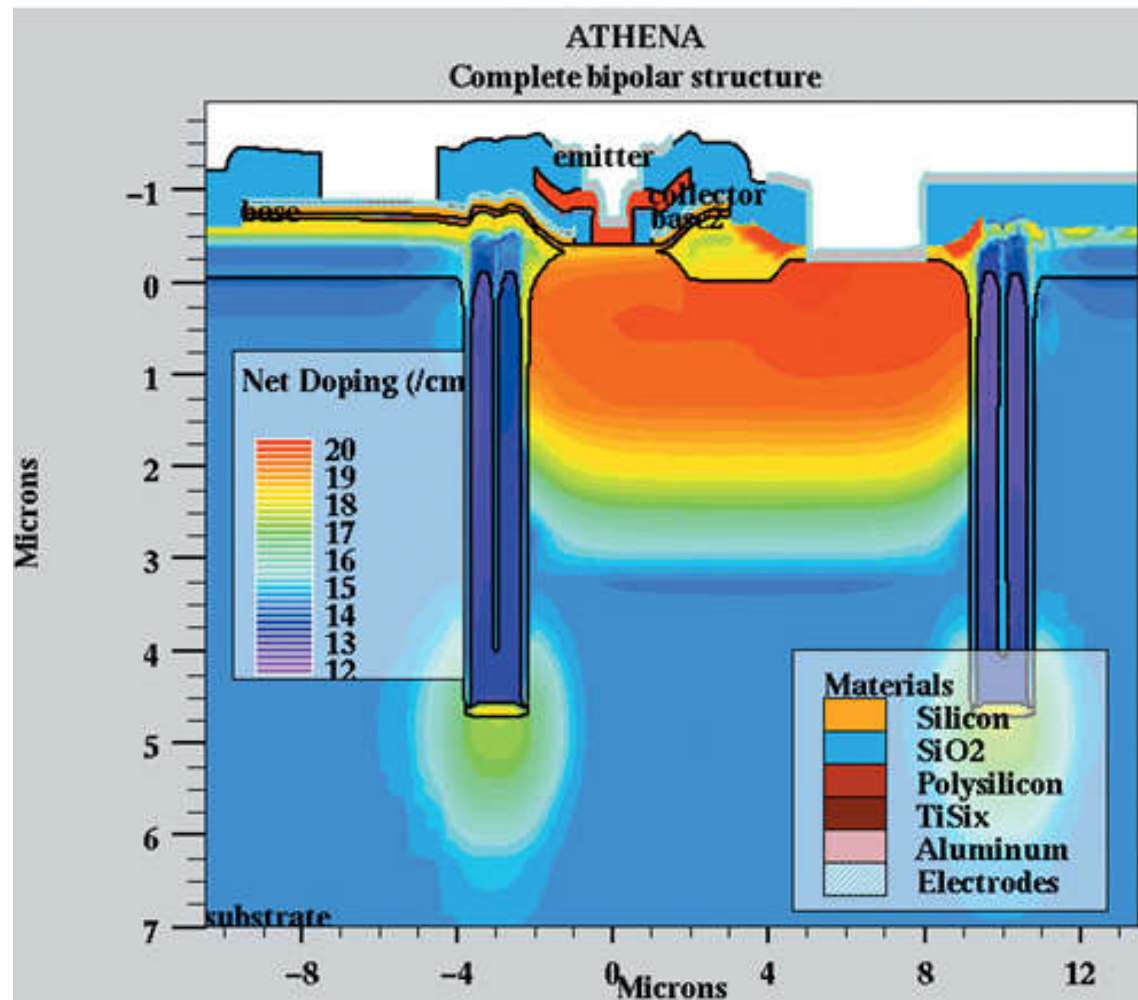


< 100 nm



# Process Simulations

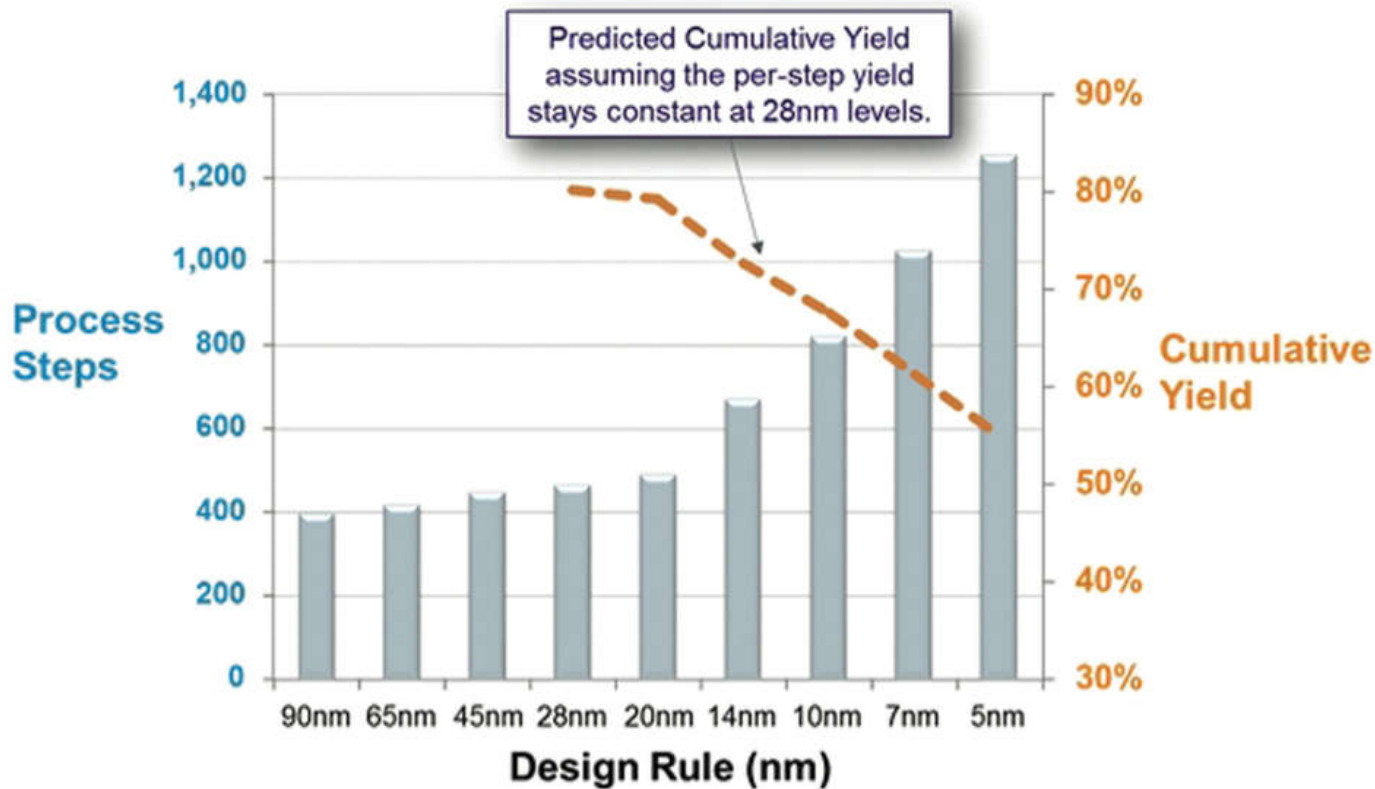
- Silvaco



# Manufacturing Yield

- Yield: rate of success

*assume yield = 99% per step:*

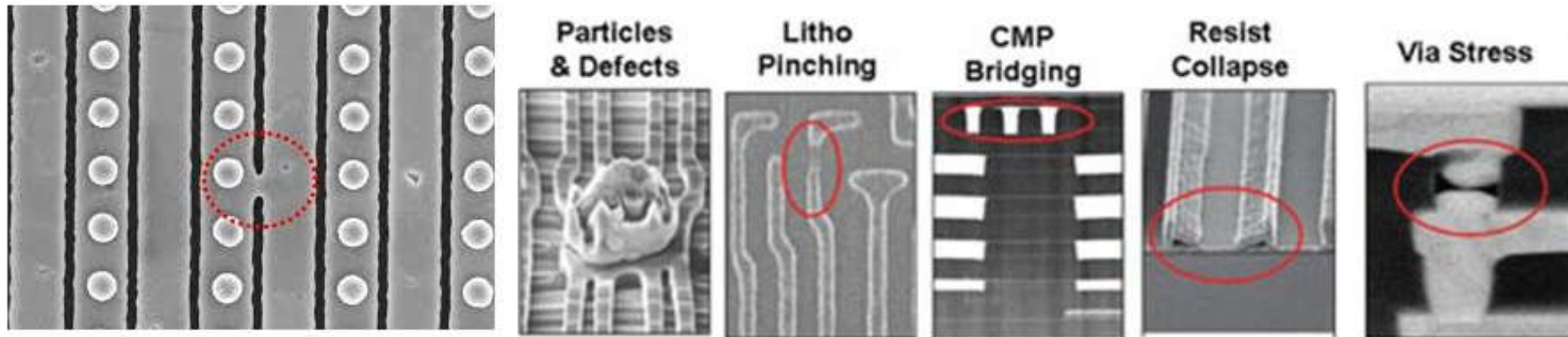
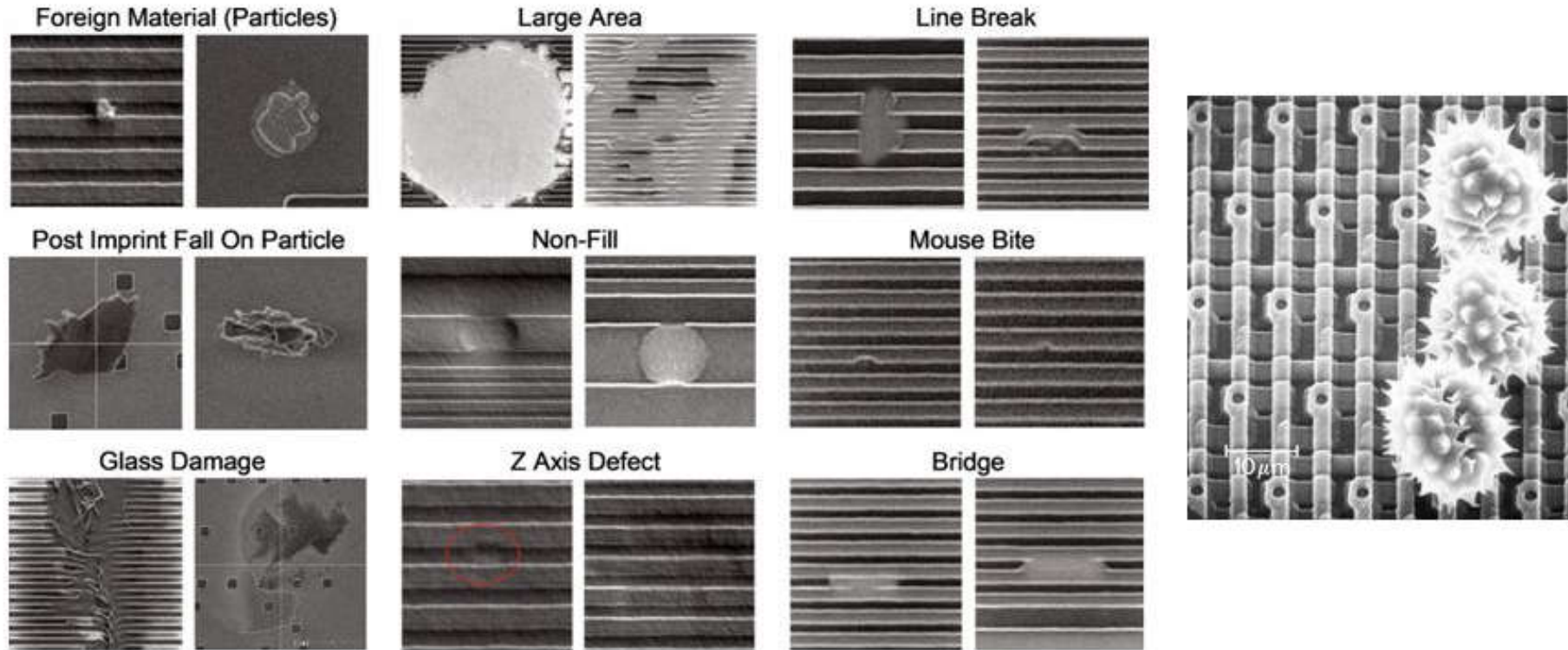


$$0.99^4 = 0.96$$

$$0.99^{400} = 0.02$$

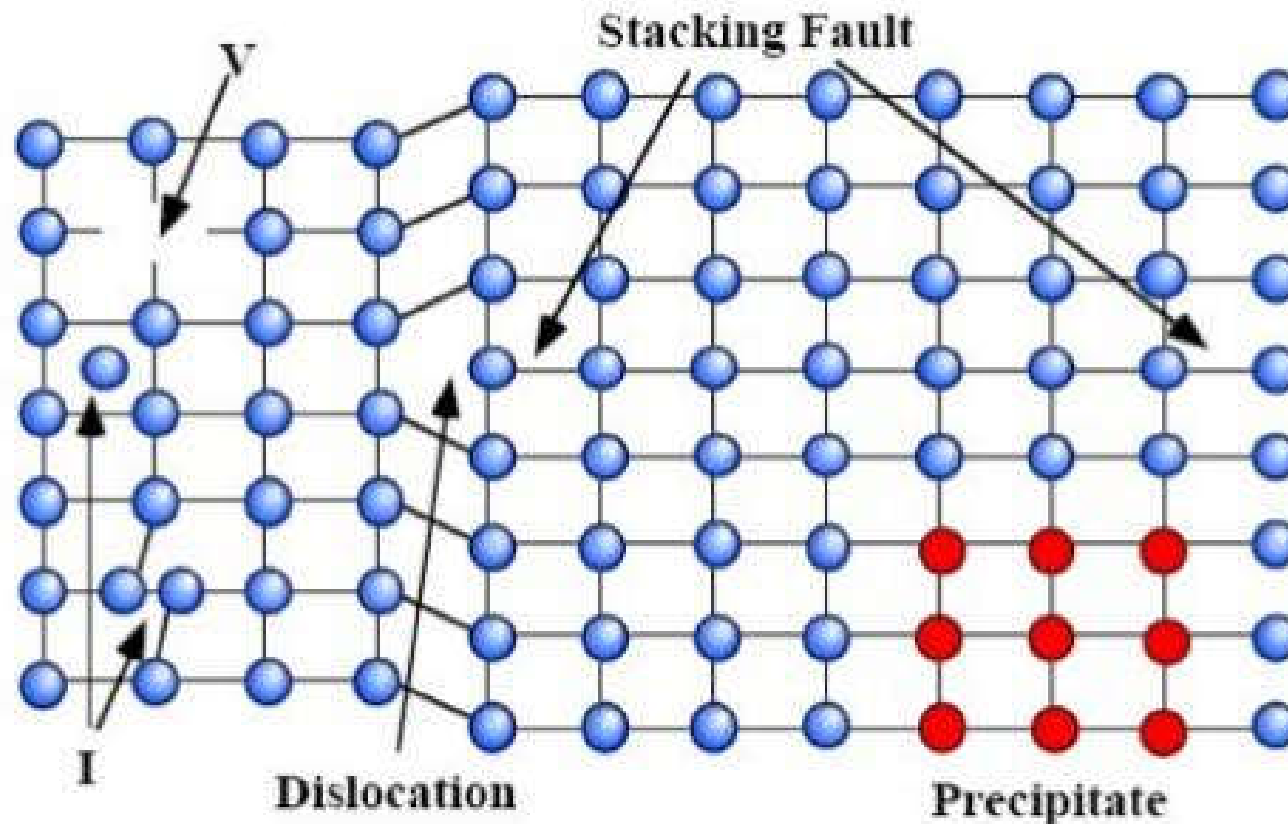
*every 1% yield means \$\$\$*

# Manufacturing Defects



# Defects in Silicon

- Point Defects e.g. Vacancies (V), Interstitials (I)
- Line Defects e.g. Dislocations
- Area Defects e.g. Stacking Faults (“extrinsic” or “intrinsic” form along {111} planes)
- Volume Defects e.g. Precipitates, Collection of Vacancies



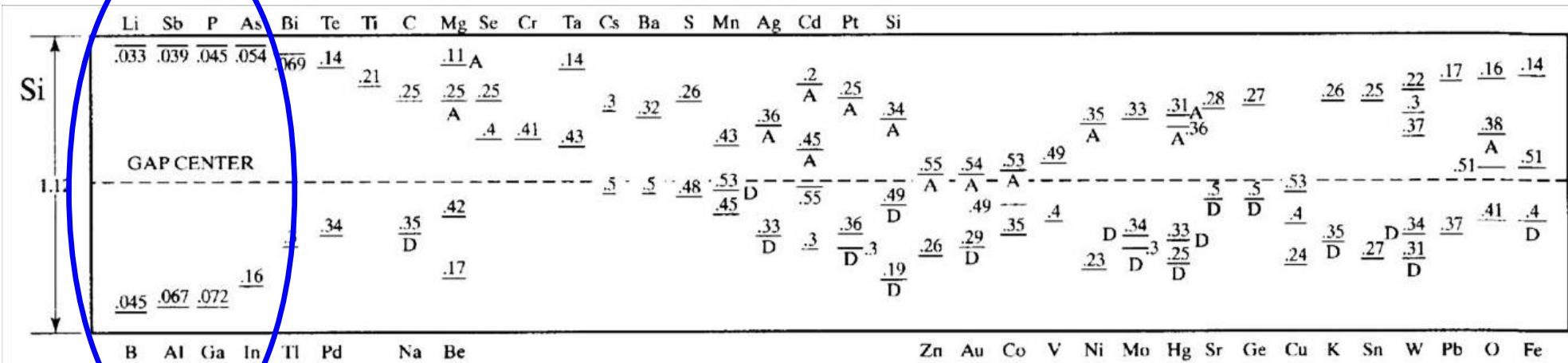


# Defects in Silicon

**Q: why?**

					2
5	6	7	8	9	10
B	C	N	O	F	Ne
13	14	15	16	17	18
Al	Si	P	S	Cl	Ar
31	32	33	34	35	36
Ga	Ge	As	Se	Br	Kr
49	50	51	52	53	54
In	Sn	Sb	Te	I	Xe
81	82	83	84	85	86
Tl	Pb	Bi	Po	At	Rn

**dopants**



**deep level defects**  
**Na, K, Au, Cu, Fe, O, ...**

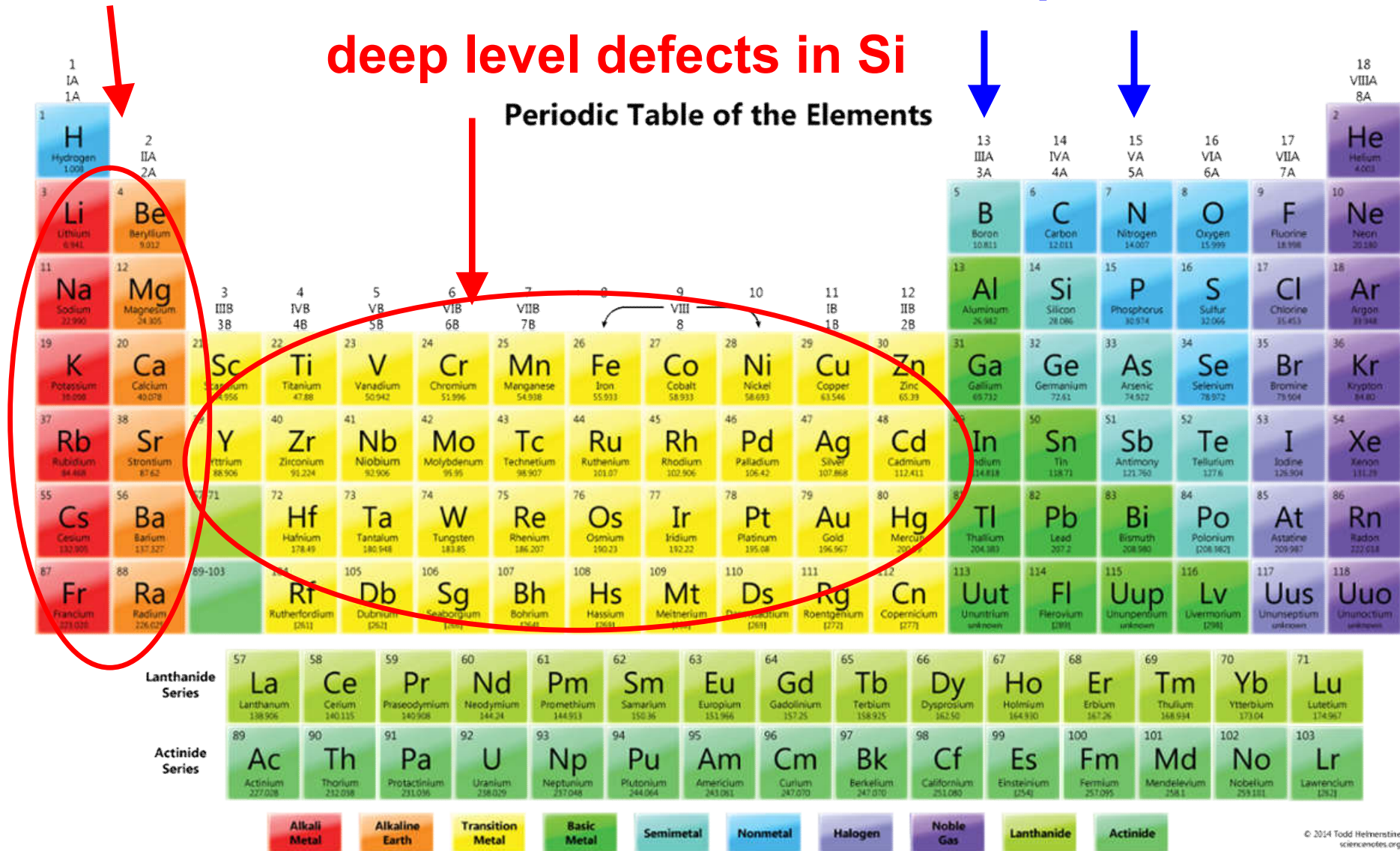
# Defects in Silicon

ions in gate oxide

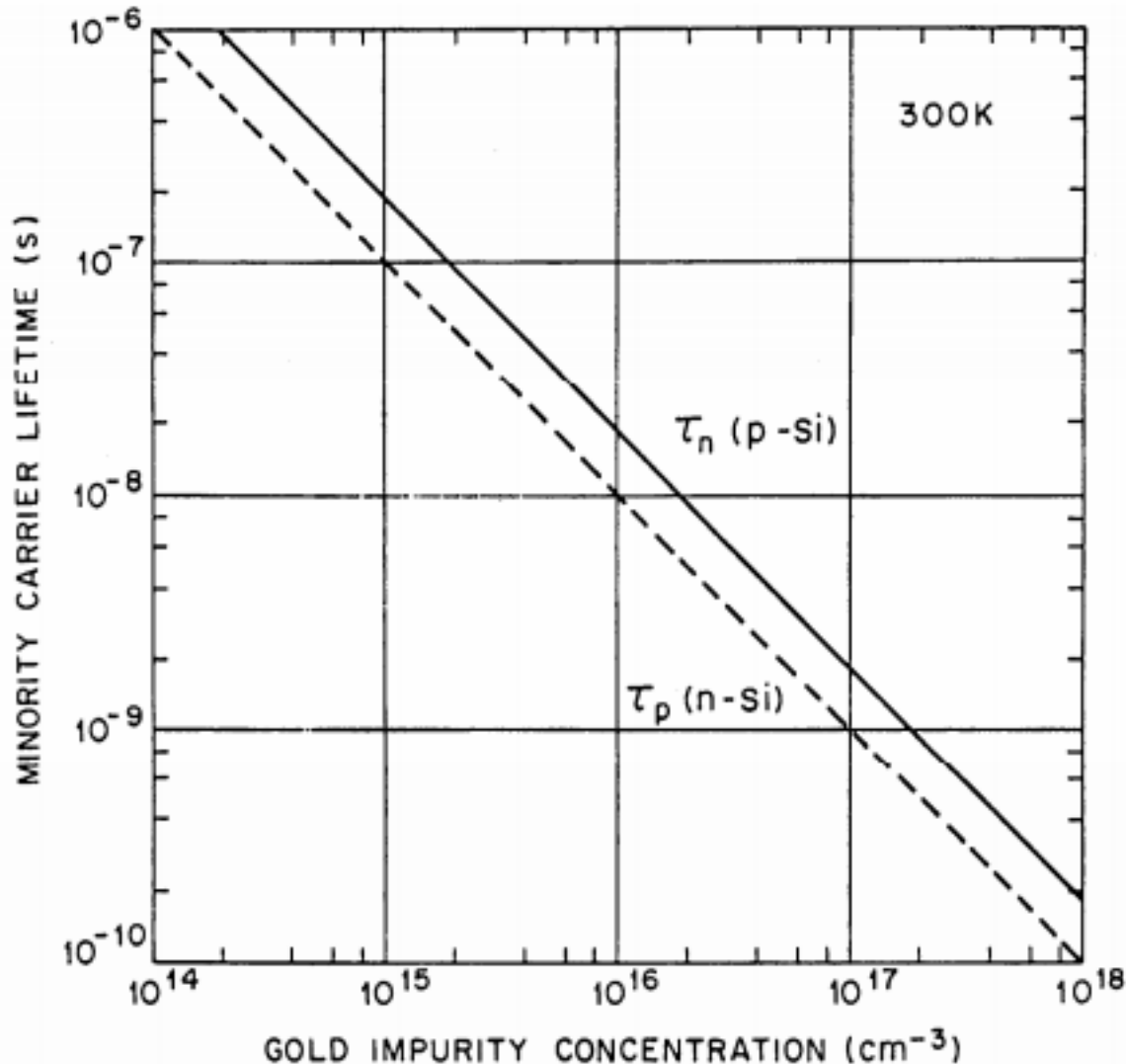
dopants

deep level defects in Si

Periodic Table of the Elements



# Defects in Silicon



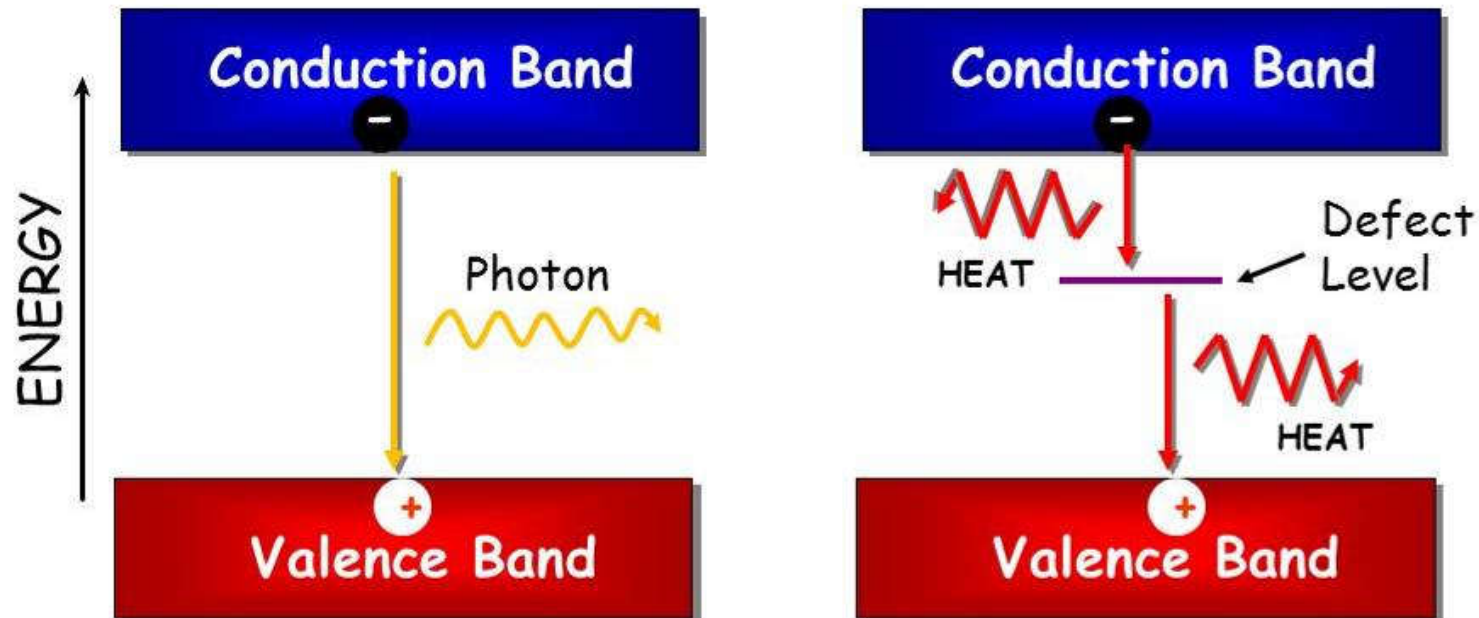
***Deep level defects  
(e.g. Au) reduce  
minority carrier  
lifetime in Si***

***bad for solar cells***

Fig. 16 Recombination lifetime versus gold impurity concentration in silicon.<sup>8</sup>

# Defects

recombination at defect sites  
reduce efficiencies of LEDs / solar cells

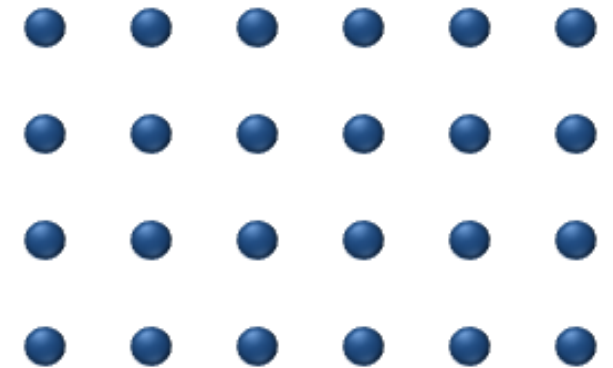


# Diffusion of Defects

Liquid



Solid



**$C$**  concentration ( $\text{mol}/\text{m}^3$ )

**$J$**  diffusion flux ( $\text{mol}/\text{m}^2/\text{s}$ )

**$D$**  diffusivity ( $\text{m}^2/\text{s}$ )

# Diffusion of Defects

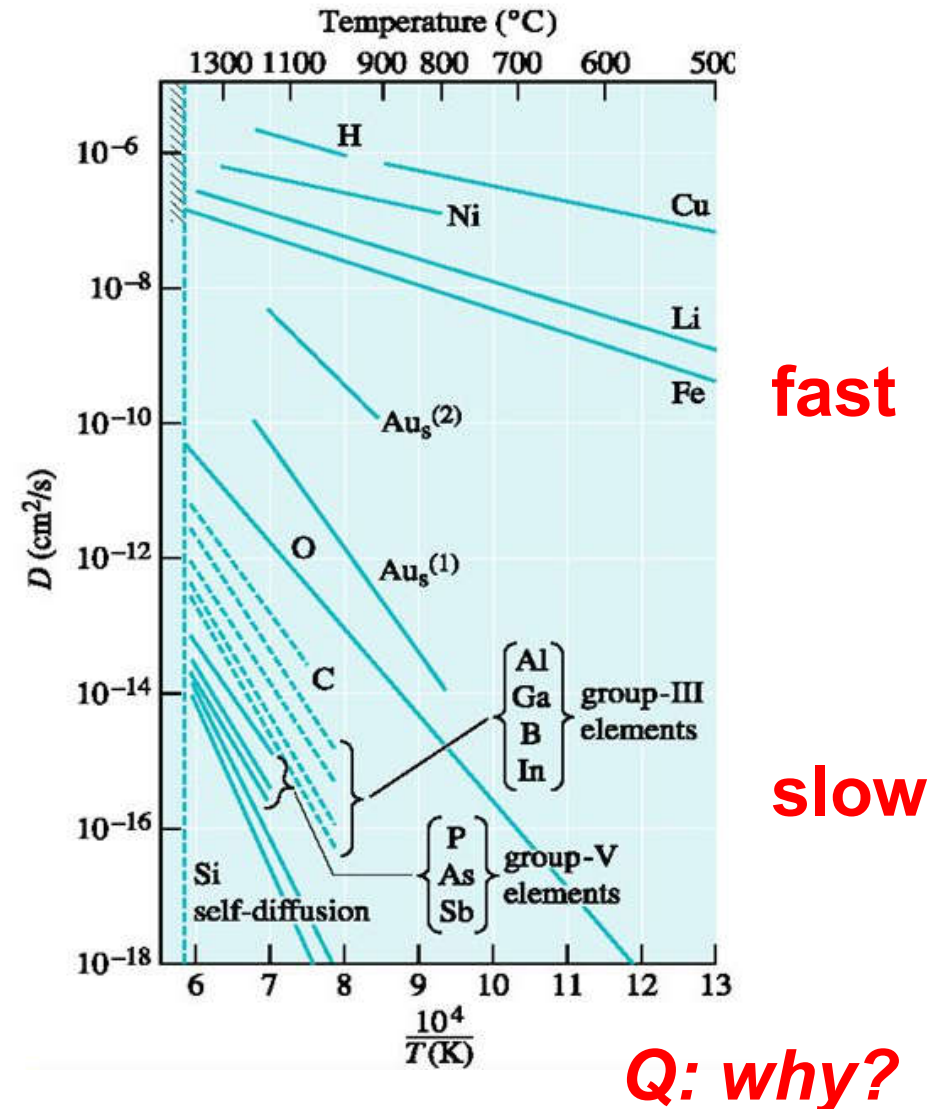
## ■ Diffusivity (扩散系数) $D$

- rate of spread
- unit:  $\text{cm}^2/\text{s}$

$$D = D_0 \exp\left(-\frac{E_A}{kT}\right)$$

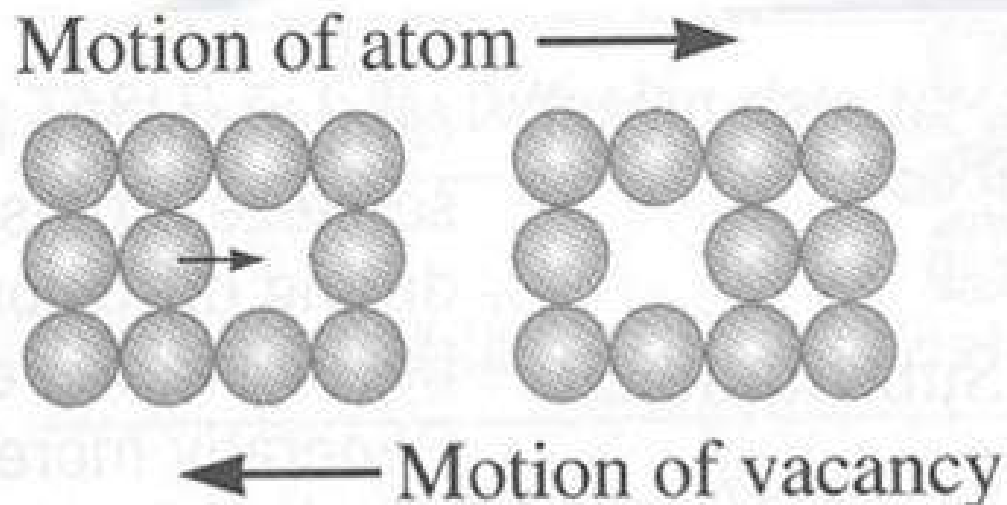
## ■ Diffusion length $L$

$$L = \sqrt{Dt}$$



diffusivity of defects in Si

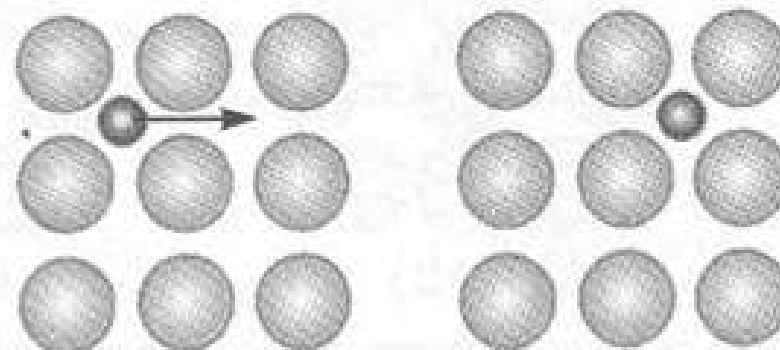
# Defect Diffusivity in Silicon



(a) Vacancy mechanism

Si, B, P, As, Sb, ...

**Slow**



(b) Interstitial mechanism

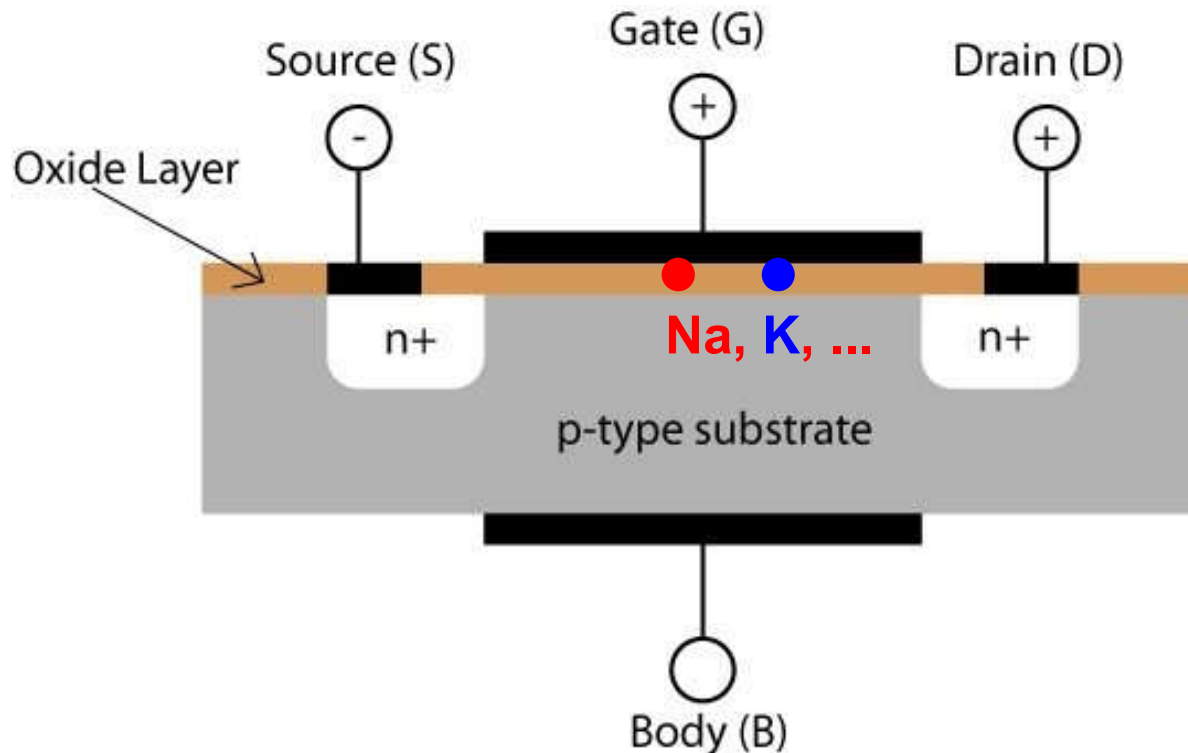
Cu, Fe, Li, H, Au, ...

**Fast**

# Defects in Oxide

$$V_{th} = V_{FB} + 2\Phi_f + \frac{\sqrt{2\varepsilon_s q N_A (2\Phi_f)}}{C_{ox}} - \frac{qQ_M}{C_{ox}}$$

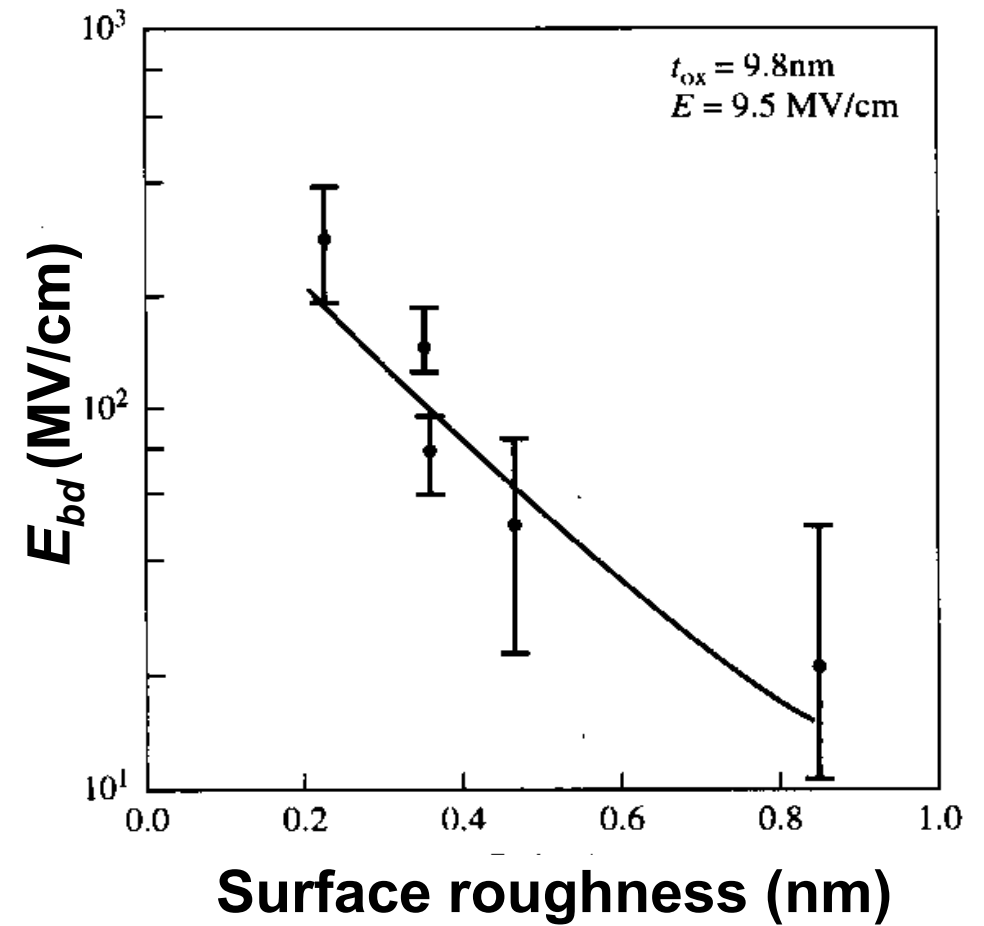
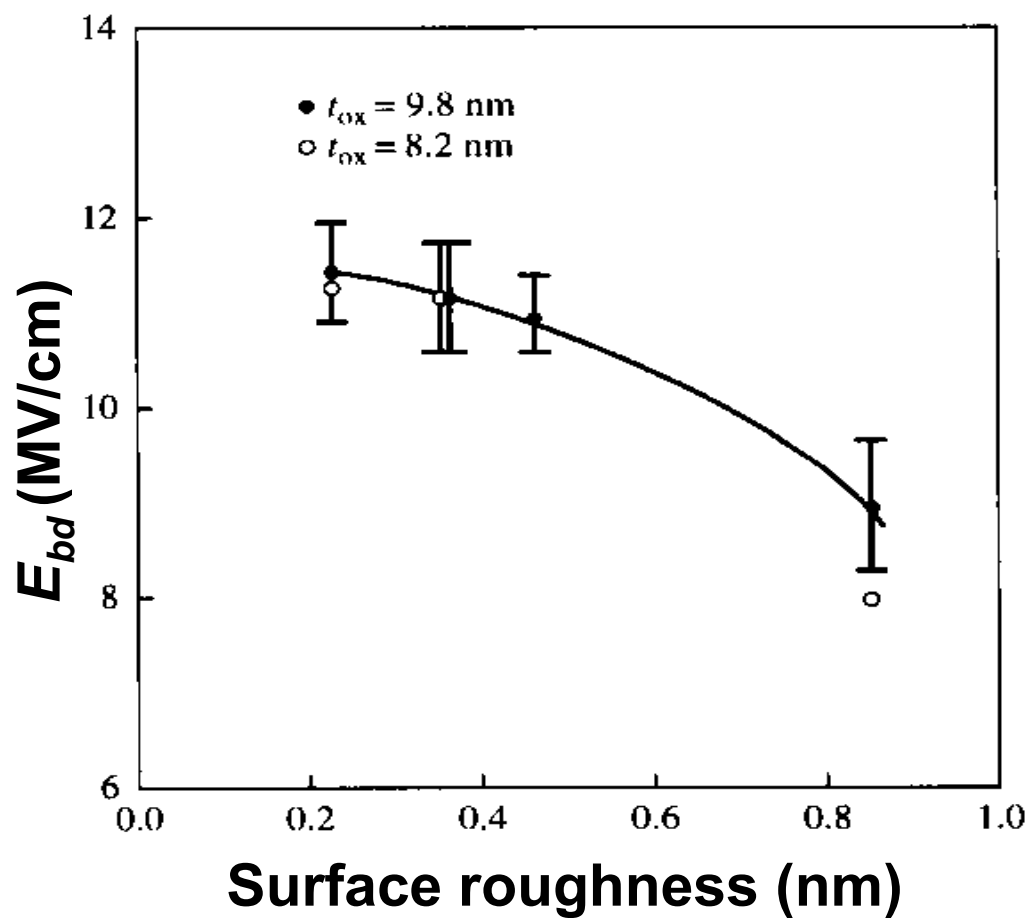
defect density



alkali ions (Na, K, ...) in gate oxide



# Defects on Surface



Surface roughness reduces breakdown voltages ( $E_{bd}$ )

# Defects Control

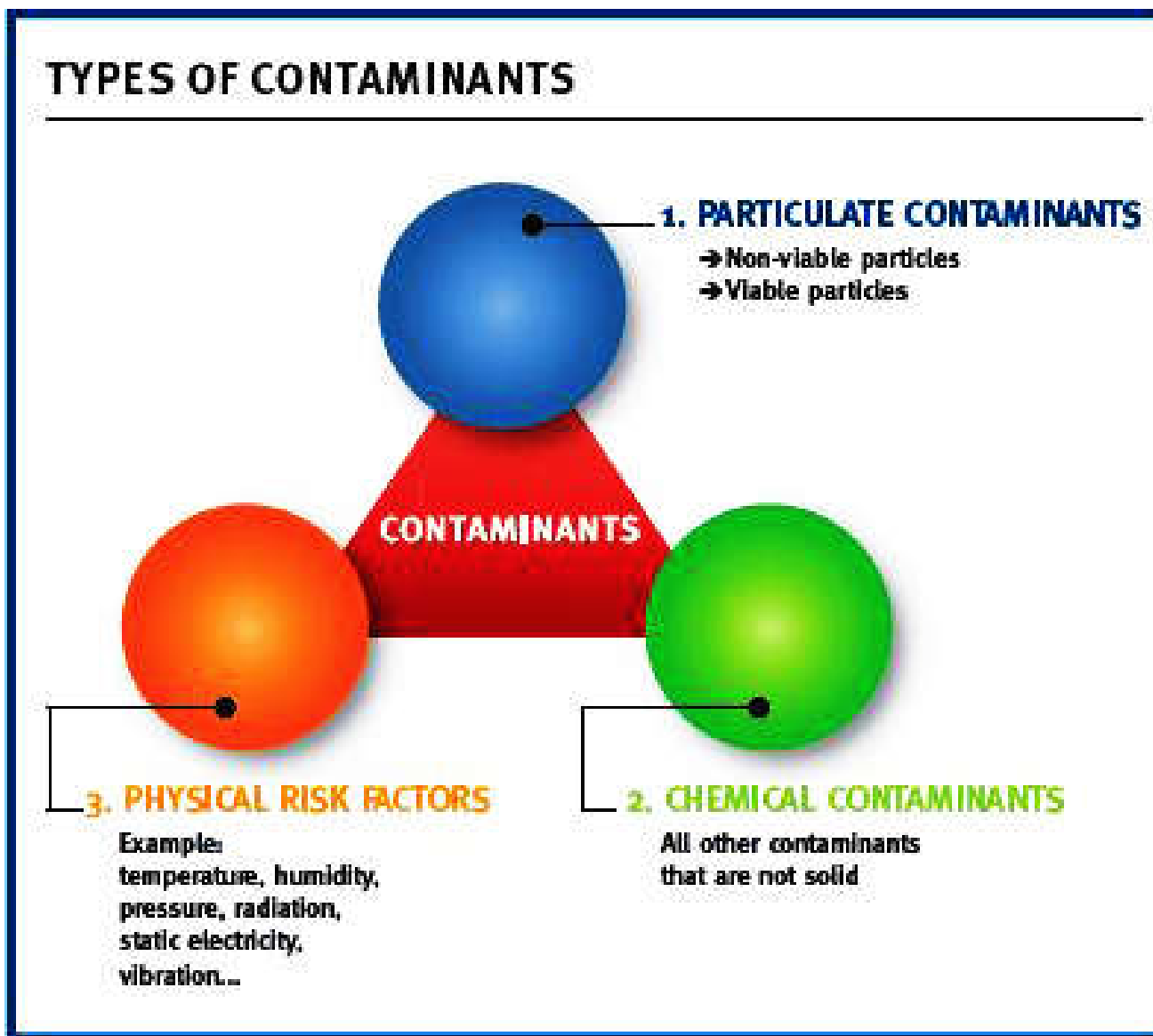
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**environment** ← **cleanroom control**

← **surface clean**

**Si wafer** ← **gettering**

# Contaminations



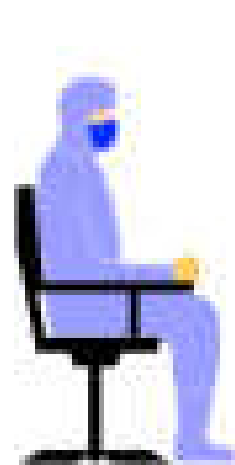
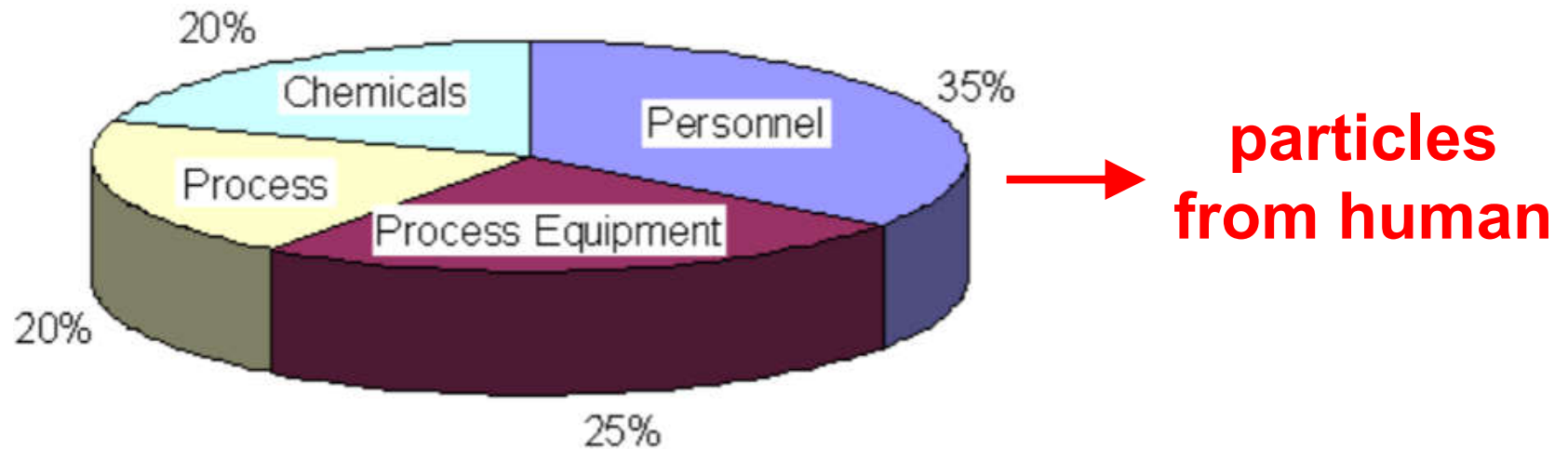
## particles:

- hair
- pollen
- bacteria
- PM2.5
- ...

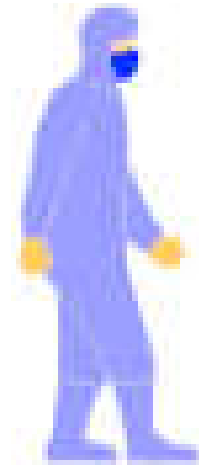
## chemicals:

- organics
- Cu, Au
- Na, K
- ...

# Sources of Contaminations



**sitting**  
100,000 /min



**walking**  
1,000,000 /min



**running**  
10,000,000 /min

# Particles



Open circuit

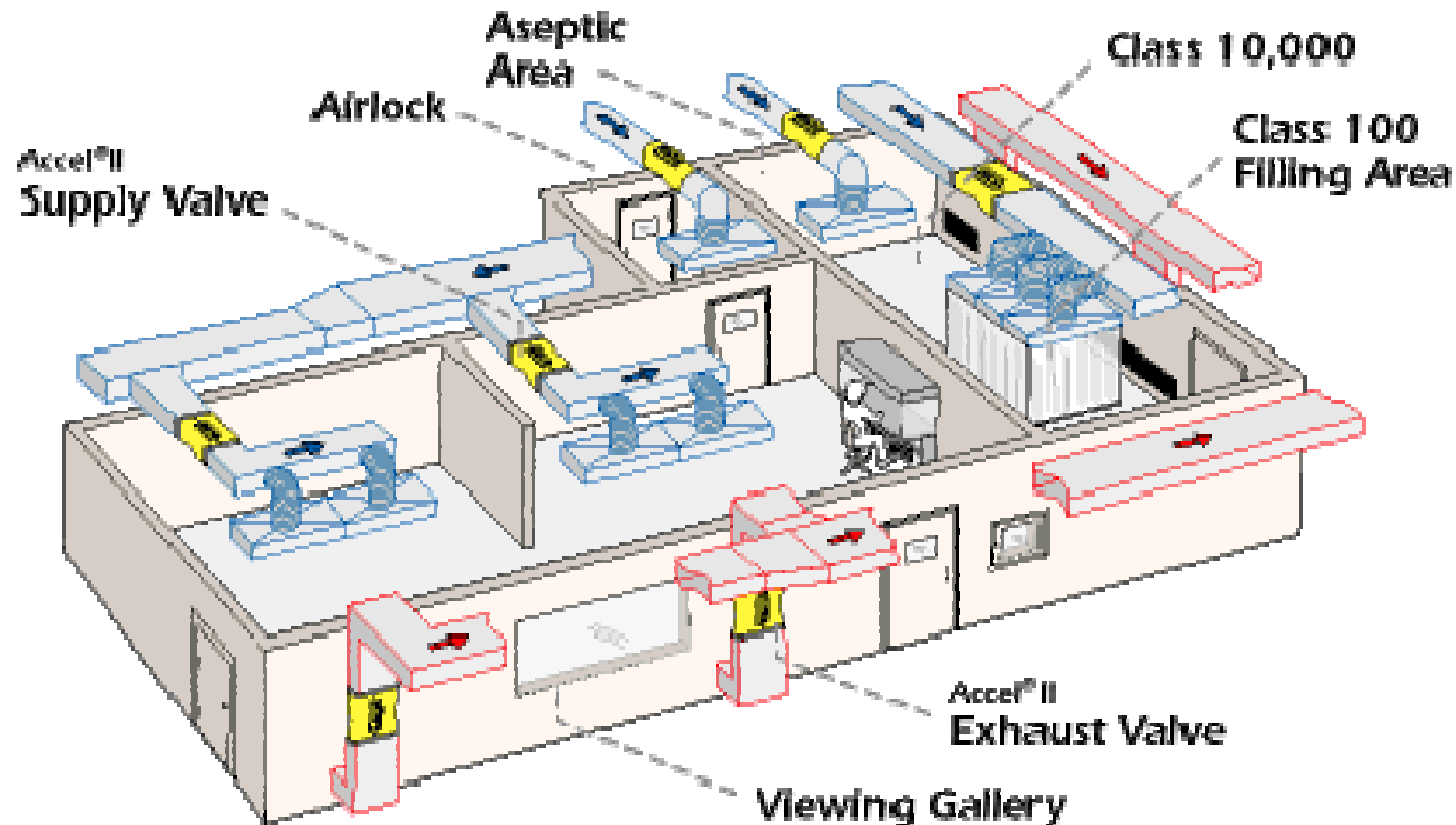


Short circuit

# IC Roadmap

Year of 1st DRAM Shipment	1997	1999	2003	2006	2009	2012
Minimum Feature Size	250nm	180nm	130nm	100nm	70nm	50nm
Wafer Diameter (mm)	200	300	300	300	450	450
DRAM Bits/Chip	256M	1G	4G	16G	64G	256G
DRAM Chip Size (mm <sup>2</sup> )	280	400	560	790	1120	1580
Microprocessor Transistors/chip	11M	21M	76M	200M	520M	1.40B
<b>Critical Defect Size</b>	<b>125nm</b>	<b>90nm</b>	<b>65nm</b>	<b>50nm</b>	<b>35nm</b>	<b>25nm</b>
<b>Starting Wafer Total LLS (cm<sup>-2</sup>)</b>	<b>0.60</b>	<b>0.29</b>	<b>0.14</b>	<b>0.06</b>	<b>0.03</b>	<b>0.015</b>
<b>DRAM GOI Defect Density (cm<sup>-2</sup>)</b>	<b>0.06</b>	<b>0.03</b>	<b>0.014</b>	<b>0.006</b>	<b>0.003</b>	<b>0.001</b>
<b>Logic GOI Defect Density (cm<sup>-2</sup>)</b>	<b>0.15</b>	<b>0.15</b>	<b>0.08</b>	<b>0.05</b>	<b>0.04</b>	<b>0.03</b>
<b>Starting Wafer Total Bulk Fe (cm<sup>-3</sup>)</b>	<b>3x10<sup>10</sup></b>	<b>1x10<sup>10</sup></b>	<b>Under 1x10<sup>10</sup></b>	<b>Under 1x10<sup>10</sup></b>	<b>Under 1x10<sup>10</sup></b>	<b>Under 1x10<sup>10</sup></b>
<b>Metals on Wafer Surface After Cleaning (cm<sup>-2</sup>)</b>	<b>5x10<sup>9</sup></b>	<b>4x10<sup>9</sup></b>	<b>2x10<sup>9</sup></b>	<b>1x10<sup>9</sup></b>	<b>&lt; 10<sup>9</sup></b>	<b>&lt; 10<sup>9</sup></b>
<b>Starting Material Recombination Lifetime (μsec)</b>	<b>≥ 300</b>	<b>≥ 325</b>	<b>≥ 325</b>	<b>≥ 325</b>	<b>≥ 450</b>	<b>≥ 450</b>

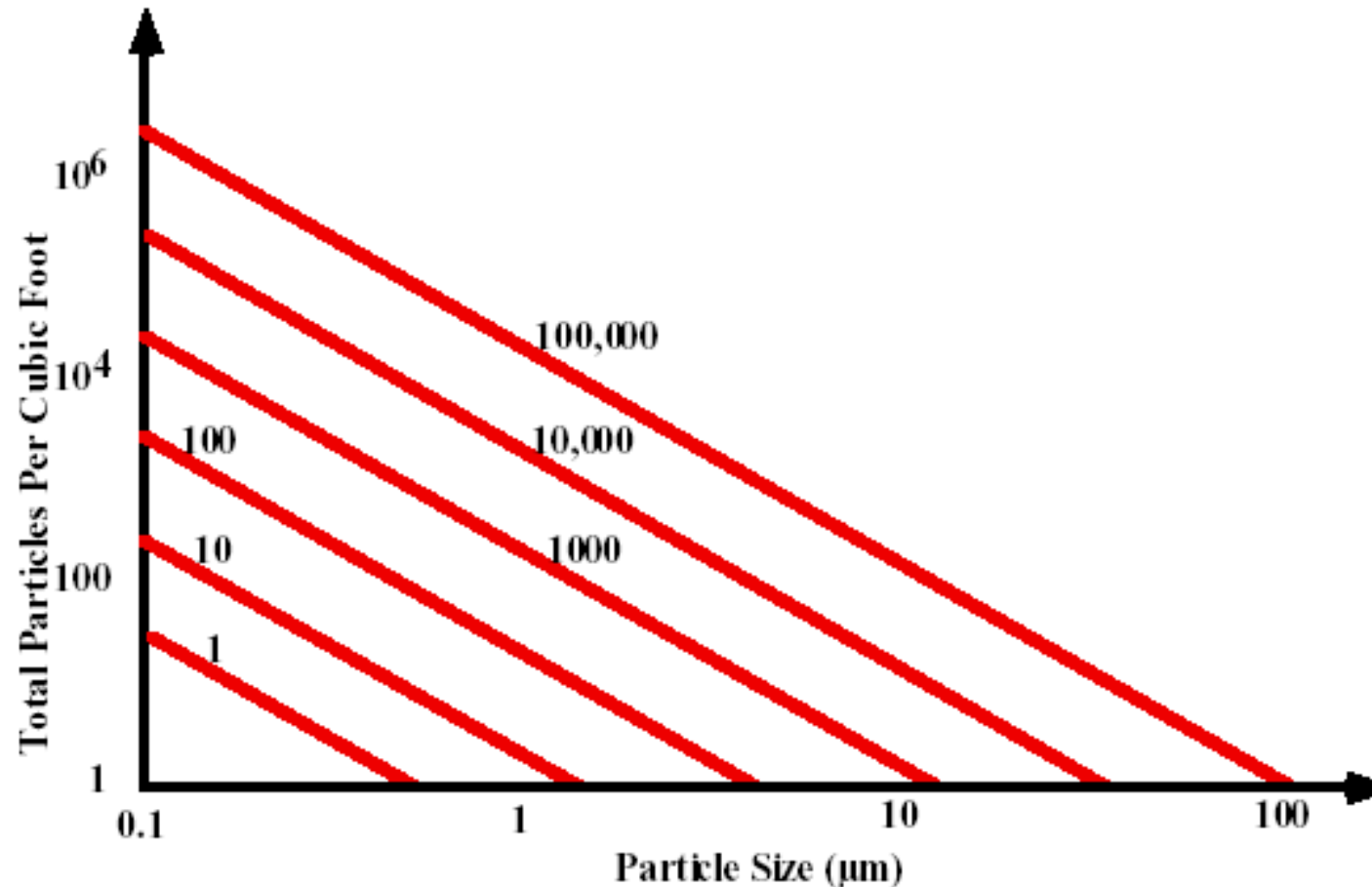
# Cleanroom



**class X:**

less than X particles larger than  $0.5 \mu\text{m}$   
per cubic feet

# Cleanroom



**class X:**

less than **X** particles larger than 0.5 µm  
per cubic feet



# Cleanroom

Class	Particle Diameter ( $\mu\text{m}$ )			
	0.1	0.3	0.5	5.0
1	35	3	1	
10	350	30	10	
100		300	100	
1,000			1,000	7
10,000			10,000	70
100,000			100,000	700

'PM2.5 index'  
 $\ll 1 \mu\text{g}/\text{m}^3$

**class X:**

less than **X** particles larger than  $0.5 \mu\text{m}$   
 per cubic feet

# Defects Control

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**environment** ← **cleanroom control**

← **surface clean**

**Si wafer** ← **gettering**

# Defects in Water

defects in water



*effects of water cleaning on transistor performance*

water resistivity ( $M\Omega \cdot \text{cm}$ , at 25 °C)	leakage current ( $\text{A}/\mu\text{m}^2$ )
5	$12 \cdot 10^{-9}$
10	$10 \cdot 10^{-9}$
13	$5 \cdot 10^{-9}$
15	$1 \cdot 10^{-9}$

# Water

## ■ Types

- purified water, distilled water, tapping water, ...
- 自来水, 矿泉水, 纯净水, 超纯水, 蒸馏水, ...

## ■ In cleanroom, **deionized (DI) water (去离子水)** is used

- free of any mineral ions
- only  $H^+$ ,  $OH^-$

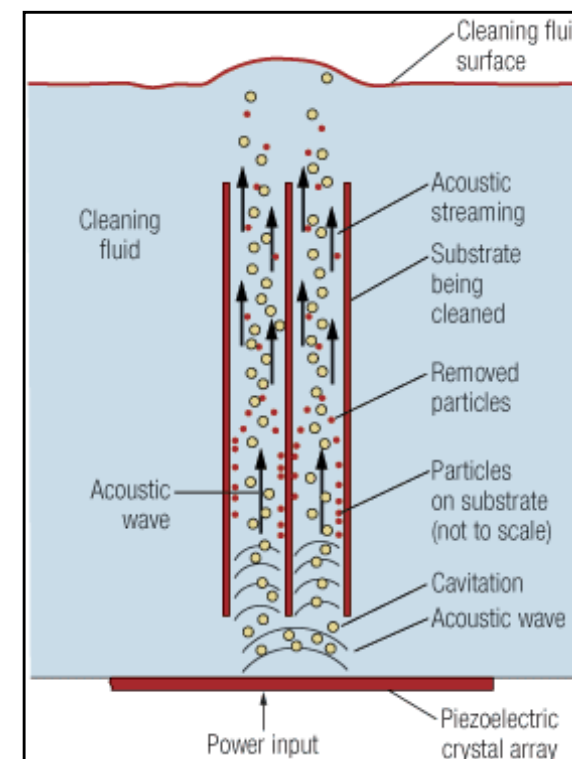


## ■ In water, at 25 °C

- $[H^+][OH^-] = K_w = 10^{-14} \text{ (mol/L)}^2$
- in DI water,  $[H^+] = [OH^-] = 10^{-7} \text{ mol/L}$ , **pH = 7.0**
- **resistivity = 18.5 MΩ\*cm**

# Si Wafer Clean

- Ultrasonic / megasonic clean in DI water



**remove: large particles,  
water soluble ions (Na, K, Cl, ...)**

# Standard Si Wafer Clean (RCA)

---

- **Step 1 (SC-1)**
  - $\text{NH}_4\text{OH} : \text{H}_2\text{O}_2 : \text{H}_2\text{O} = 1:1:5$ , at 80 °C, 10 mins
  - remove organic residues
  
- **Step 2**
  - $\text{HF} : \text{H}_2\text{O} = 1:50$ , at 25 °C, 20 secs
  - remove native  $\text{SiO}_2$
  
- **Step 3 (SC-2)**
  - $\text{HCl} : \text{H}_2\text{O}_2 : \text{H}_2\text{O} = 1:1:6$ , at 80 °C, 10 mins
  - remove metals
  
- **Step 4**
  - clean in DI water

# Metal Removal

**Table 4-3** Oxidation-reduction reactions for a number of species of interest in silicon wafer cleaning

Oxidant/ Reductant	Standard Oxidation Potential (volts)	Oxidation-Reduction Reaction
Mn <sup>2+</sup> /Mn	1.05	Mn ↔ Mn <sup>2+</sup> + 2e <sup>-</sup>
SiO <sub>2</sub> /Si	0.84	Si + 2H <sub>2</sub> O ↔ SiO <sub>2</sub> + 4H <sup>+</sup> + 4e <sup>-</sup>
Cr <sup>3+</sup> /Cr	0.71	Cr ↔ Cr <sup>3+</sup> + 3e <sup>-</sup>
Ni <sup>2+</sup> /Ni	0.25	Ni ↔ Ni <sup>2+</sup> + 2e <sup>-</sup>
Fe <sup>3+</sup> /Fe	0.17	Fe ↔ Fe <sup>3+</sup> + 3e <sup>-</sup>
H <sub>2</sub> SO <sub>4</sub> /H <sub>2</sub> SO <sub>3</sub>	-0.20	H <sub>2</sub> O + H <sub>2</sub> SO <sub>3</sub> ↔ H <sub>2</sub> SO <sub>4</sub> + 2H <sup>+</sup> + 4e <sup>-</sup>
Cu <sup>2+</sup> /Cu	-0.34	Cu ↔ Cu <sup>2+</sup> + 2e <sup>-</sup>
O <sub>2</sub> /H <sub>2</sub> O	-1.23	2H <sub>2</sub> O ↔ O <sub>2</sub> + 4H <sup>+</sup> + 4e <sup>-</sup>
Au <sup>3+</sup> /Au	-1.42	Au ↔ Au <sup>3+</sup> + 3e <sup>-</sup>
H <sub>2</sub> O <sub>2</sub> /H <sub>2</sub> O	-1.77	2H <sub>2</sub> O ↔ H <sub>2</sub> O <sub>2</sub> + 2H <sup>+</sup> + 2e <sup>-</sup>
O <sub>3</sub> /O <sub>2</sub>	-2.07	O <sub>2</sub> + H <sub>2</sub> O ↔ O <sub>3</sub> + 2H <sup>+</sup> + 2e <sup>-</sup>



# Other Si Clean Recipes

## ■ Piranha clean

- SPM: Sulfuric-Peroxide Mixture
- $\text{H}_2\text{SO}_4 : \text{H}_2\text{O}_2 = 3:1$ , 10–30 mins
- extremely exothermic, self heating up to 80 °C
- remove organic residues and some metals



**DANGER**

## ■ Ozone ( $\text{O}_3$ ) clean

- $\text{H}_2\text{O} + \text{O}_3$
- remove organic residues

## ■ Organic solvent

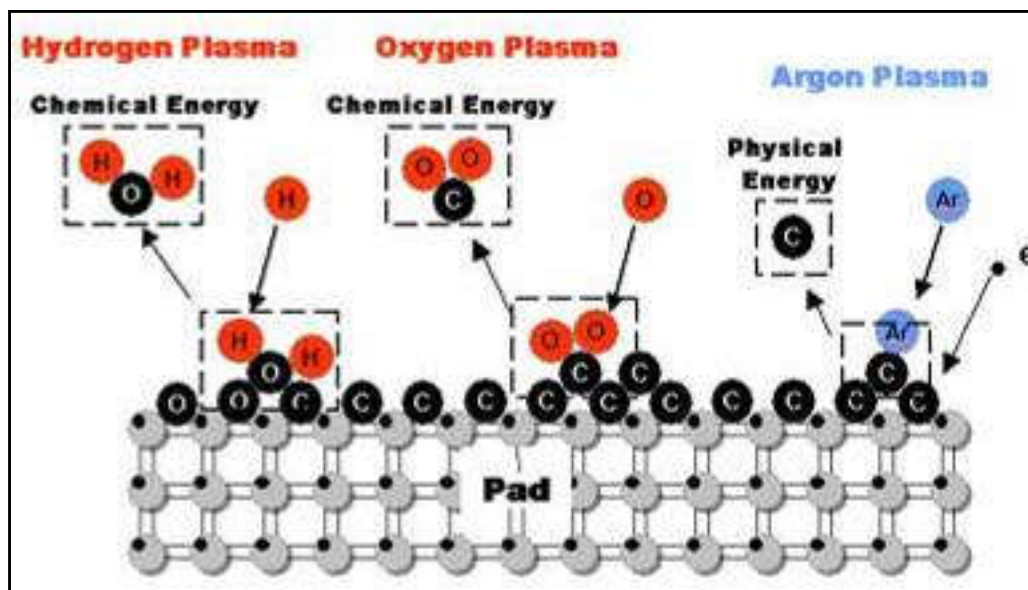
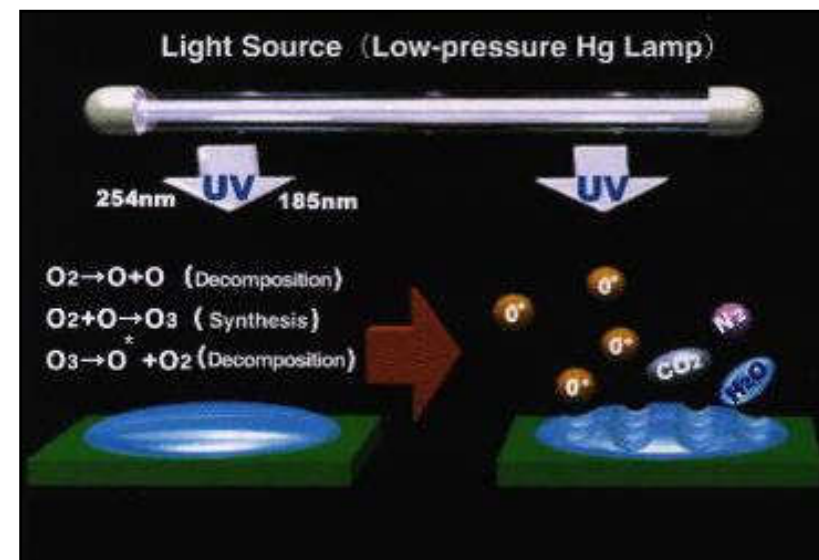
- Acetone / Isopropanol / DI water
- remove organic residues
- not used for standard CMOS process!

**'wet' method**



# Other Si Clean Recipes

- UV Ozone clean
  - clean organic residues
  
- Plasma clean
  - clean organic residues



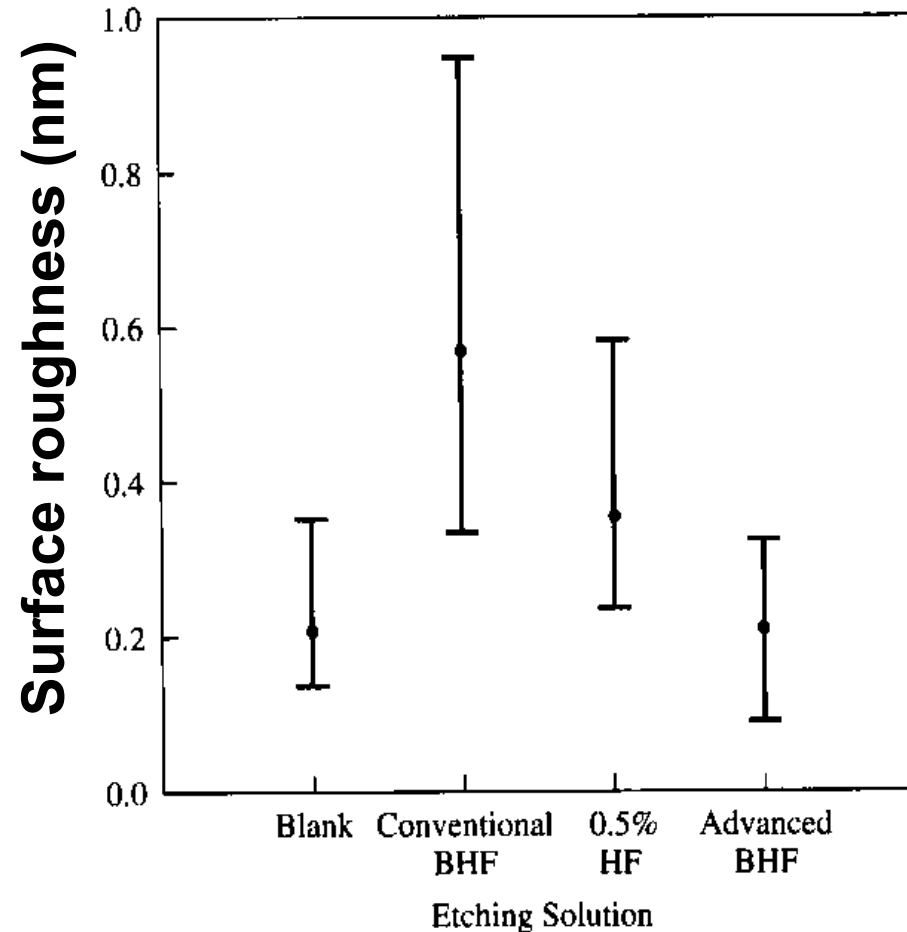
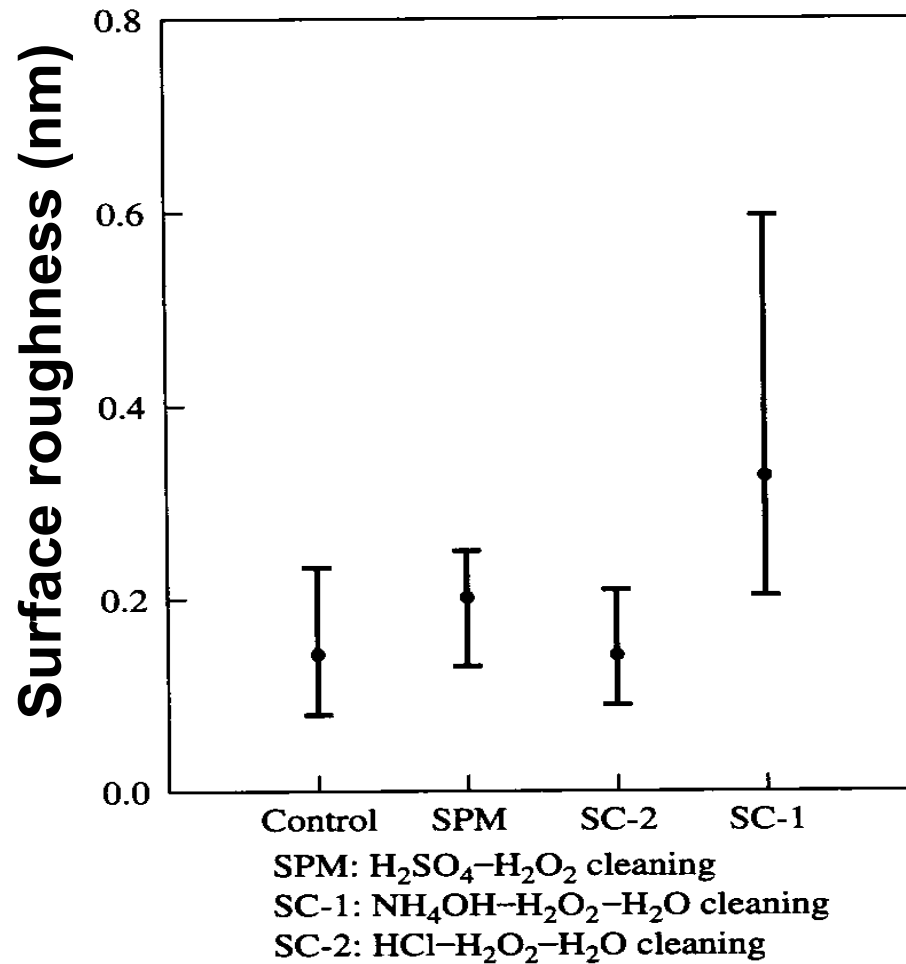
'dry' method

# Clean other Materials

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- **SiO<sub>2</sub> (glass, quartz, ...)**
  - piranha clean, H<sub>2</sub>SO<sub>4</sub> : H<sub>2</sub>O<sub>2</sub> = 3:1, 10–30 mins
  - SC-1, NH<sub>4</sub>OH : H<sub>2</sub>O<sub>2</sub> : H<sub>2</sub>O = 1:1:5, at 80 °C, 10 mins
- **GaAs**
  - NH<sub>4</sub>OH : H<sub>2</sub>O = 1:10, for stoichiometric surface (Ga/As 1:1)
  - H<sub>3</sub>PO<sub>4</sub> or HCl, for As rich surface
- **Acetone / Isopropanol / DI water**
  - generally works well for most non-CMOS process

# Surface Roughness



**Surface Roughness of Si after cleaning ammonia ( $\text{NH}_4\text{OH}$ ) and HF slightly etches Si**

# Defects Control

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**environment** ← **cleanroom control**

← **surface clean**

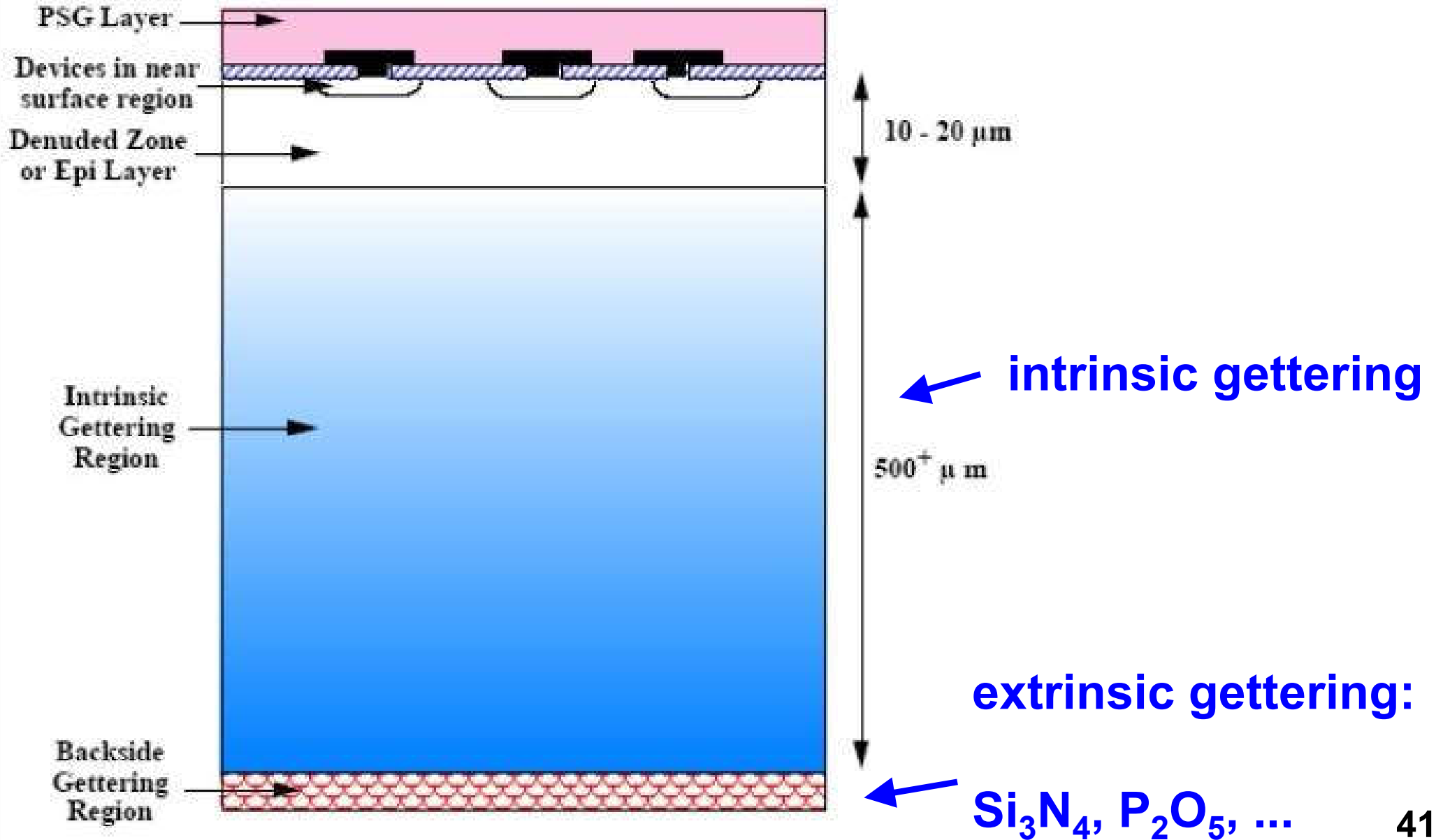
**Si wafer** ← **gettering**

# Gettering (吸杂)

- 'gettering' in the vacuum tube
  - use titanium to absorb gases in vacuum tubes

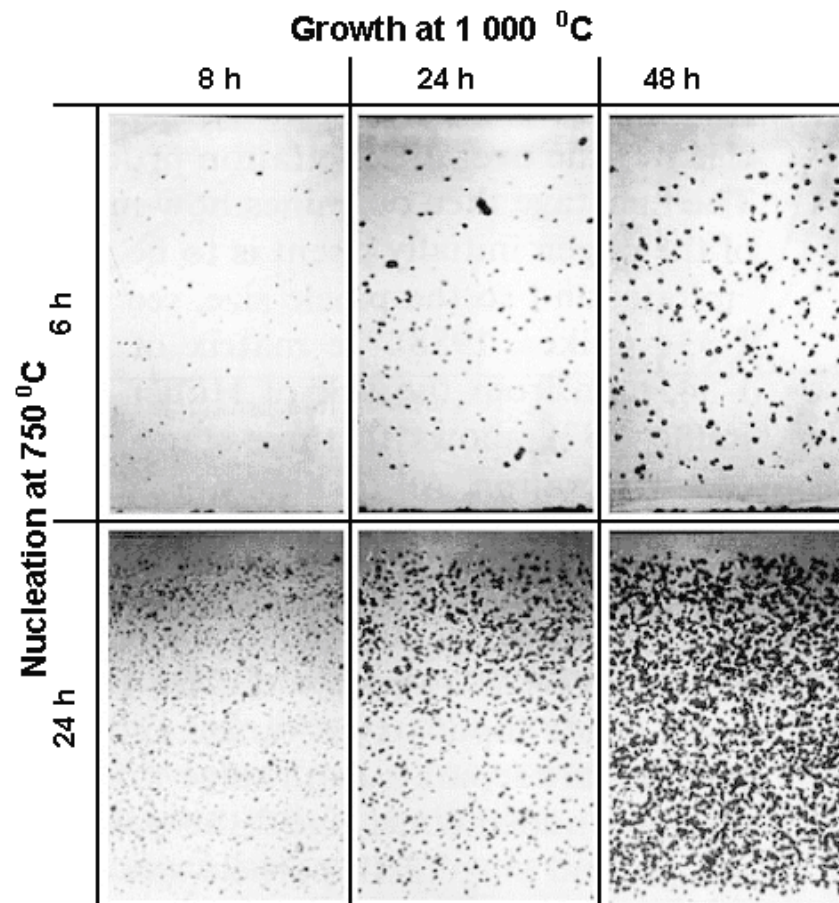


# Si Wafer Gettering (吸杂)

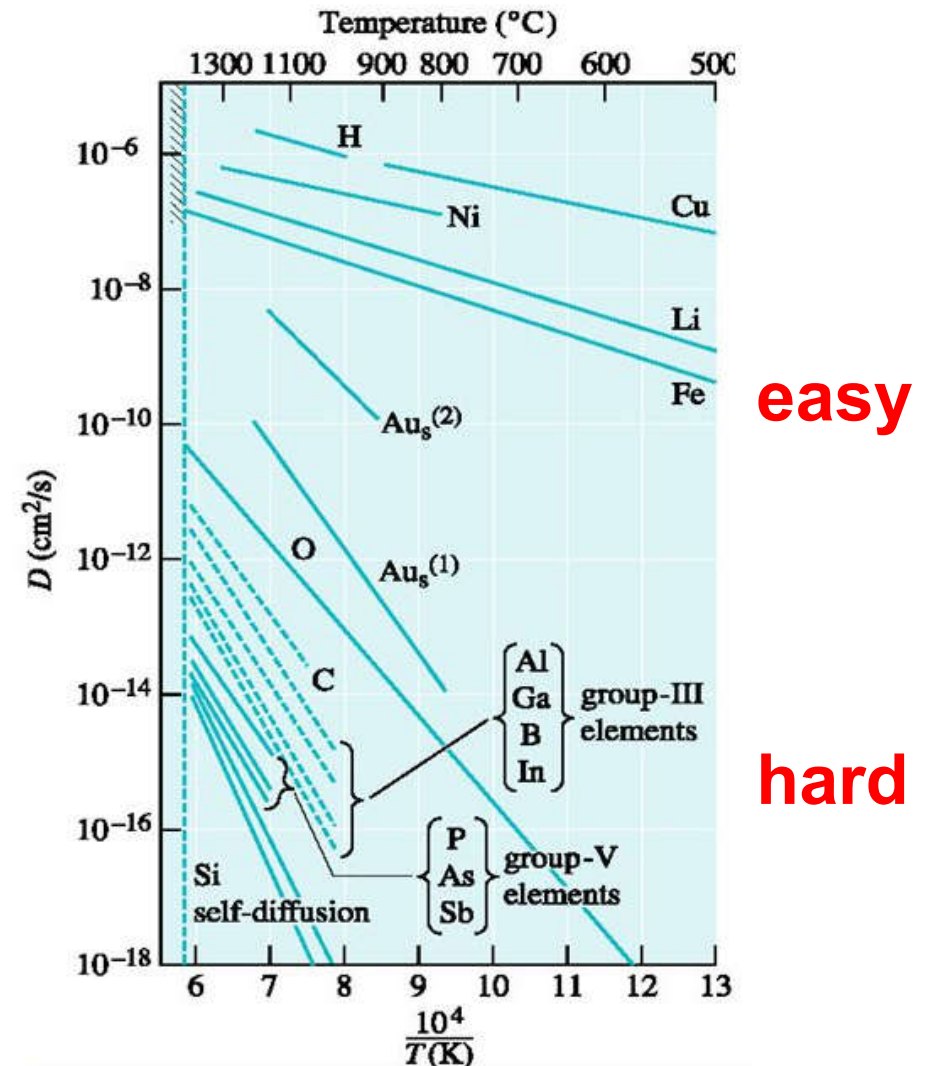


# Si Wafer Gettering (吸杂)

## ■ intrinsic gettering



O defects in Si



diffusivity of defects

# Si Wafer Gettering (吸杂)

---

- Minority carrier lifetime

**Au doped Si:**  $10^{-9}$  s

**Typical Si:**  $10^{-6}$  s

**Gettered Si:**  $10^{-3}$  s



# *Principles of Micro- and Nanofabrication for Electronic and Photonic Devices*

## Lab Safety

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# Lab Safety

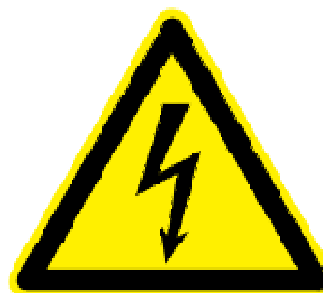
## ■ Chemicals

- HF, H<sub>2</sub>SO<sub>4</sub>, ...
- KOH, NH<sub>4</sub>OH, ...
- Acetone, ...



## ■ Electricity

- instruments, ...



## ■ Fires

- Acetone, Alcohol, ...

## ■ Sharps

- silicon, glass, ...

■ ...



# Lab Safety

- Lab orientation

- exits, showers, ...

- Proper protection

- gloves, goggles, aprons, ...



- Materials Data Safety Sheets (MSDS)

- ...



# Lab Safety

## ■ Materials Data Safety Sheets (MSDS)

### Material Safety Data Sheet Hydrofluoric Acid, 48% MSDS

#### Section 1: Chemical Product and Company Identification

**Product Name:** Hydrofluoric Acid, 48%

**Catalog Codes:** SLH2227

**CAS#:** 7664-39-3

**RTECS:** Not applicable.

**Contact Information:**

Sciencelab.com, Inc.

14025 Smith Rd.

Houston, Texas 77396

US Sales: 1-800-901-7247

International Sales: 1-281-444-4400

#### Section 3: Hazards Identification

##### Potential Acute Health Effects:

Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

# Chemical Safety

## ■ NFPA diamond

- 0: no hazard
- 4: highest risk



HF



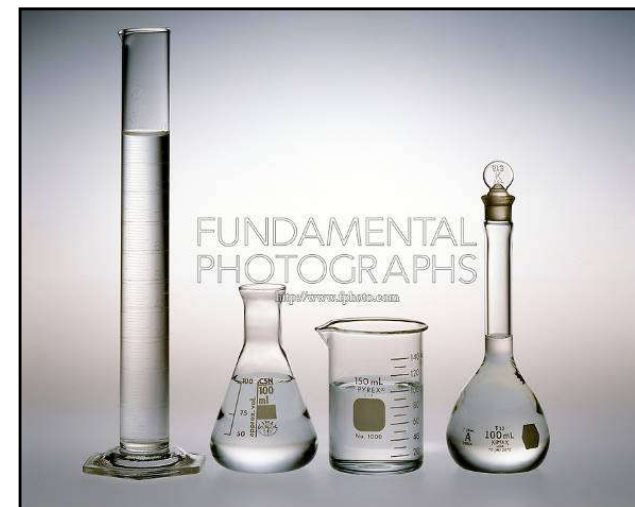
acetone



H<sub>2</sub>O<sub>2</sub>

# Chemical Safety

- Choose proper containers
- Most solvents
  - glass, Teflon, ...
- Be careful
  - alkali (NaOH, etc) slowly etches glass
  - HF strongly etches glass!



glass art by HF etch



# Chemical Safety

宣城郎溪中学化学备课组举办玻璃画制作比赛

2011年01月11日11时08分 来源：中安教育网 分享到

为提高学生学习化学兴趣，增强学生的动手能力，激发学生思维发散能力。日前，宣城郎溪中学高一化学备课组举办玻璃画制作比赛，百余人参加了此次活动。图为玻璃画制作现场。(宗京能)

编辑：李其平



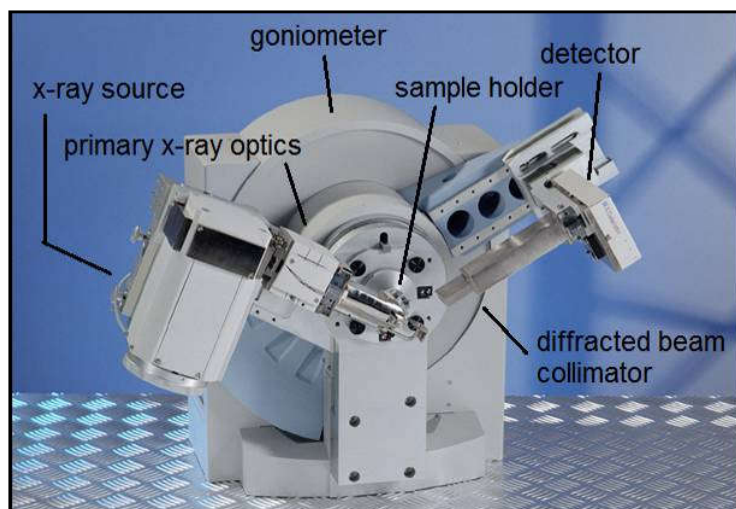
学生氢氟酸中毒事件：校方N宗罪

网易 - Jan 1, 2014

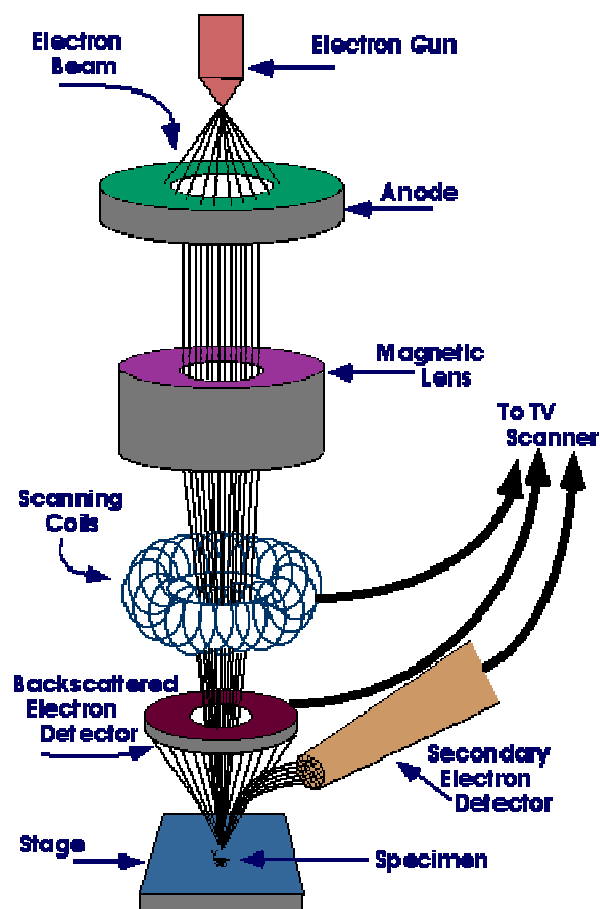
学校即使使用20%的低浓度氢氟酸，同样有致命风险。氢氟酸被列为危险化学品管制名单，这种高危剧毒物本来就不应该出现在高中实验室，将问题指向学生是推卸...

glass art by HF etch

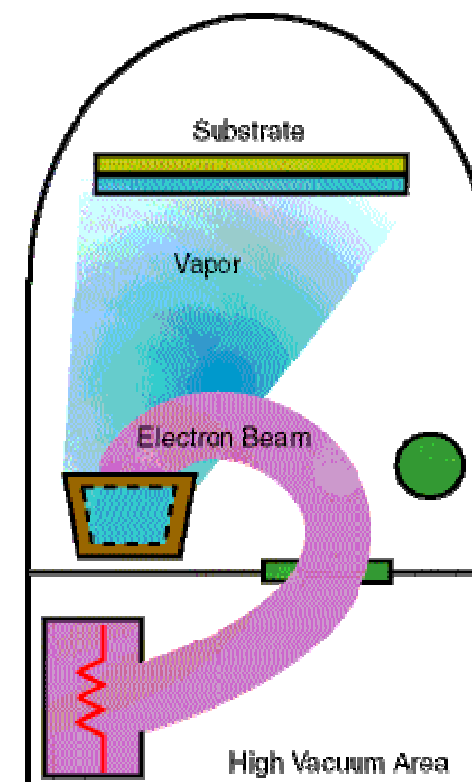
# Radiation Safety



## XRD



## SEM & TEM



## Ebeam Evaporator

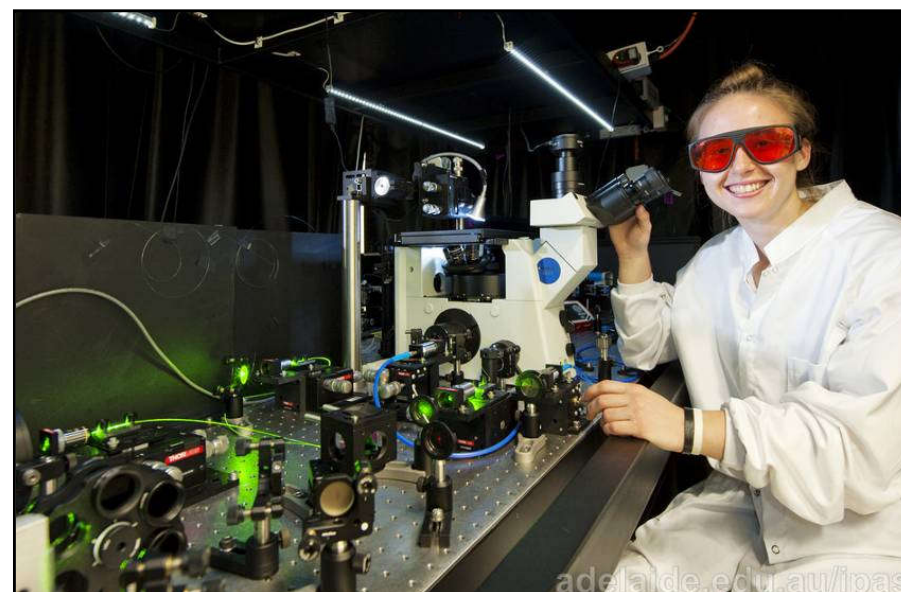


# Laser Safety

<b>Class 1</b>	CD/DVD Player/Recorder, Laptop or Personal Computer
<b>Class 2</b>	Presentation Laser Pointer, Barcode Reader
<b>Class 3R</b>	Some Measuring & Targeting Devices, Higher Power Pointers
<b>Class 3B</b>	Higher power laser products intended for professional applications
<b>Class 4</b>	Medical Lasers, Industrial Cutting/Welding, Scientific Applications and most <i>Laser Light Show</i> Equipment



**wear goggles**



# Biological Safety



↑  
**Basic  
Facility**

- **BSL-1**
  - Safe microorganisms
  - Not known to cause disease in healthy adult humans
- **BSL-2**
  - Moderate-risk microorganisms
  - Potentially hazard to humans and the environment
  - e.g. inactivated virus that Causes Foot and Mouth Disease

↑  
**Containment  
Facility**

- 
- **BSL-3, BSL-3 Enhanced & BSL-3 Ag**
    - High-risk microorganisms, foreign and emerging agents
    - Serious and potential lethal consequences for livestock
    - Not harmful to humans because of protective measures
    - e.g. live virus that Causes Foot and Mouth Disease
  - **BSL-4**
    - High-risk agents microorganisms
    - No known vaccine or therapy
    - e.g. Nipah and Hendra viruses

# Cleanroom Orientation

## Video



"YOU CAN'T COME INTO THE CLEAN ROOM LOOKING LIKE THAT!"



***always gown up!***

# Process References

## BYU Cleanroom

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<https://cleanroom.byu.edu>