

# Notes for MICRO & NANO FABRICATION

《半导体工艺与检测（电镜）》笔记

**Jinwei (David) Li**

E-mail: [daviduoft.li@mail.utoronto.ca](mailto:daviduoft.li@mail.utoronto.ca)

University of Toronto 2025.12

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## Citations through out the book

- [1] S. Franssila, Introduction to microfabrication. Chichester, West Sussex, England: John Wiley & Sons, Inc., 2010, ch.x, pp.xx-xx.
- [2] Y. Lian, Semiconductor microchips and fabrication: a practical guide to theory and manufacturing. Hoboken, New Jersey: John Wiley & Sons, Inc., 2023, ch.x, pp.xx-xx.
- [3] A. Sarangan, Nanofabrication: Principles to Laboratory Practice. Boca Raton, Florida: CRC Press, 2019, ch.x, pp.xx-xx.

# 1. Introduction to Semiconductor Fabrication

TNFC – Toronto Nanofabrication Centre (ECE)

OCCAM - Open Centre for the Characterization of Advanced Materials (MSE)

1. Bardeen, Brittain, Shockley- Bell Lab

-1947 December, ACHIEVE TRANSISTOR ACTION IN A GERMANIUM POINT-CONTACT DEVICE, Later called the bipolar junction transistor (BJT).

-The base provides proper interaction between charge carriers of emitter and collector. The flow of majority carriers from emitter to collector controlled by the base of Transistor.

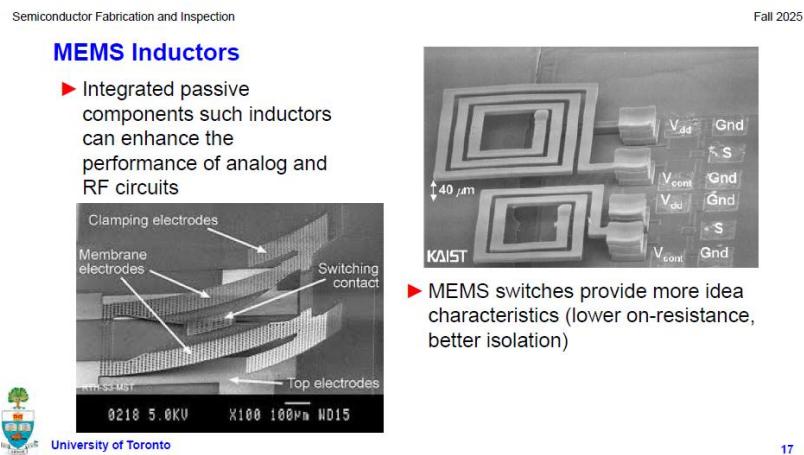
2. Jack Kilby and Robert Noyce

-1958 Jack Kilby (Texas Instruments) and Robert Noyce (Fairchild Semiconductor, co-founder of Intel) separately invented the integrated circuit.

-In 1958 (milestone), Jack Kilby of Texas Instruments built a circuit using germanium p-n-p transistor slices he had etched to form transistor, capacitor, and resistor elements.

- Noyce, first working monolithic ICs in 1960 (milestone).

Micro-electromechanical systems (MEMS) RELEASE STEP MAKES Cantilevers



Digital Micromirror Devices (DMD) (light steering device) is an optical micro-electrical-mechanical system (MEMS) that contains an array of highly reflective aluminum micromirrors. When coupled to an appropriate optical system, this DMD is capable of displaying images, video, and patterns.

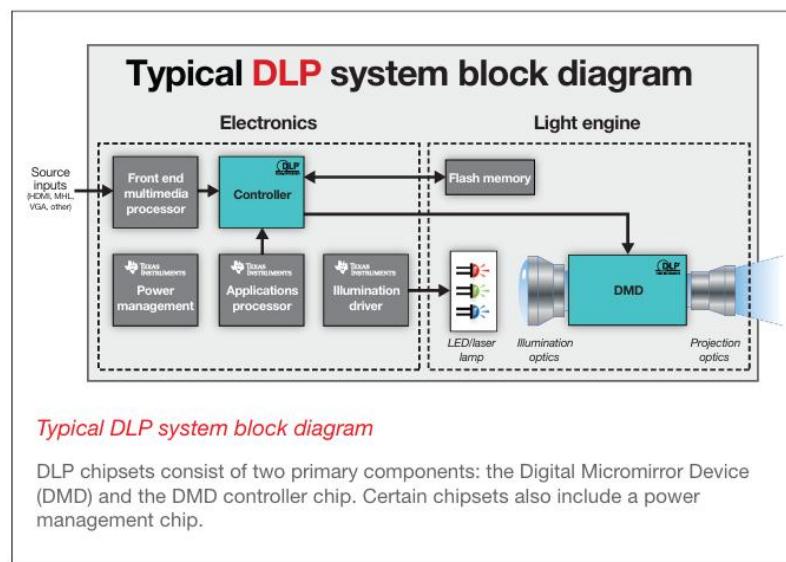
Silicon can be machined to make tilting mirrors, adjustable gratings. Due to silicon's smoothness and flatness for optics and its mechanical strength for tilting.

## DMD

### How DLP technology works

During operation, the DMD controller loads each underlying memory cell with a “1” or a “0”. Next, a micromirror clocking pulse is applied, causing each micromirror to switch to a plus or minus 12° landed state. In a projection system, the +12° landed state corresponds to an “on” pixel, and the -12° landed state corresponds to an “off” pixel. Grayscale patterns are created by programming the on/off-duty cycle of each mirror. And simultaneously, multiple light sources are multiplexed to create full RGB color images. In advanced light control applications, the ±12° states offer two general purpose output ports with a pattern and its inverse.

The DMD works in concert with an optical module containing optics and illumination to create the heart of the projection engine. The controller is



- **Digital exposure** – Systems based on DLP technology project digital patterns from the DMD that selectively cure and harden a layer of photopolymer or resin in one shot. These systems have higher throughputs than point-by-point technologies while achieving micron-scale patterns. PCB Lithography and 3D Printers use this capability today.



PCB lithography

### 3. Moore's Law

-Moore's law is the observation that the number of transistors in an integrated circuit (IC) doubles about every two years. Moore's law is an observation and projection of a historical trend.

-Named after Gordon Moore, co-founder of Fairchild semiconductor and was the CEO of Intel, whose 1965 paper described a X2/year in the number of components (C,R,Tran) per integrated circuit.

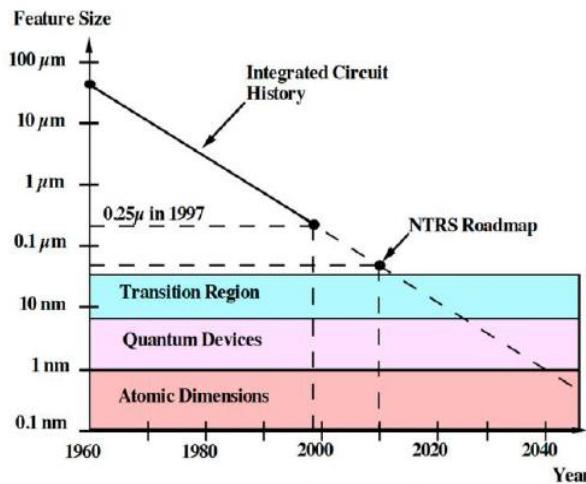


Fig. 1.2: Historical trends and future projections for the minimum feature size used in integrated circuits in manufacturing.

1 nm in future years, angstrom level, solution is to build in 3d,

Microfabrication is the collection of techniques used to fabricate devices in the micrometer range.

Bulk silicon wafers are single crystal pieces cut from larger single crystal ingots and polished.[1]

The most common dielectric thin films are silicon dioxide ( $\text{SiO}_2$ ) and silicon nitride ( $\text{Si}_3\text{N}_4$ ).

A special case of thin-film deposition is epitaxy: the deposited film registers the crystalline structure of the underlying substrate, and, for example, more single crystal silicon can be deposited on a silicon wafer but with different dopant atoms and different dopant concentration.

Microfabrication processes consist of four basic operations:

1. High-temperature processes to modify the substrate.
2. Thin-film deposition on the substrate.
3. Patterning of thin films and the substrate.
4. Bonding and layer transfer.

- **Ellipsometry and reflectometry** are distinct techniques primarily used for characterizing thin films, measuring film thickness and optical properties (refractive index and extinction coefficient).

Here is a breakdown of the differences:

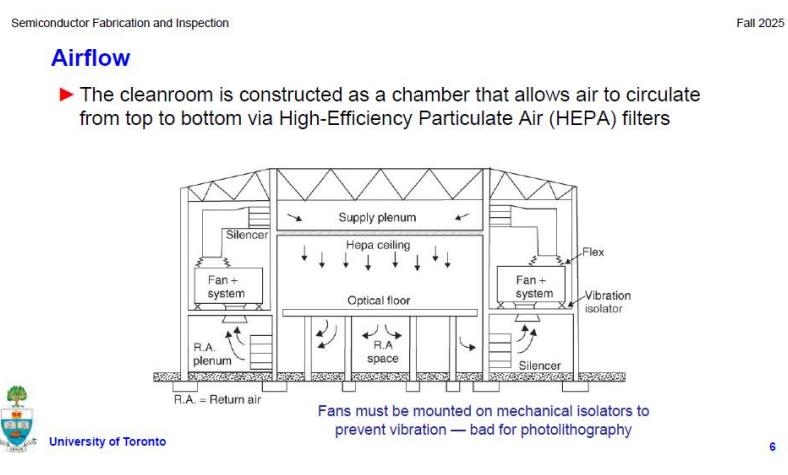
Feature	Optical Profilometry	Reflectometry	Ellipsometry
<b>Primary Goal</b>	3D surface topography (height variation)	Film thickness and optical properties	Film thickness and optical properties
<b>Measurement Principle</b>	Interferometry, confocal methods, structured light	Measures intensity of reflected light	Measures change in polarization state of light upon reflection
<b>Angle of Incidence</b>	Typically normal or near-normal	Typically normal	Requires an oblique angle
<b>Material Sensitivity</b>	Sensitive to material variations, which can cause measurement errors if not compensated for	Less sensitive, requires models and prior knowledge to separate thickness/index	Highly sensitive, can decouple thickness and refractive index without prior knowledge in many cases

## 2. Cleanroom Etiquette

Primary Interest is to reduce airborne particles. Vibration isolation of air fans.

Cleanroom facility consists of the cleanroom plus all supporting facilities for water, air-conditioning, chemical delivery.

High Efficiency Particle Air filter on ceiling, also make the airflow laminar.



## Classes of Cleanroom

### ► Classes and their typical uses

- Class 1 & 10      Semiconductor processing
- Class 100      Photo labs, production for medical supplies
- Class 10,000      Factory space for electronic products, hospital operating rooms

Maximum Number of Particles in Air  
(particles per cubic foot air)

Class	Particle Size (ISO 14644 Standard)				
	0.1 μm	0.2 μm	0.3 μm	0.5 μm	5.0 μm
1	35	7.5	3	1	
10	350	75	30	10	
100		750	300	100	
1,000				1,000	7
10,000				10,000	70
100,000				100,000	700



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International Organization for Standardization (ISO)

Class 100 refers to max 100 particles, 0.5um, per cubic foot.

Cleanroom personnel is an important source of contamination.

Only special lint-free paper should be used.

Never touch substrates or wafers with your hands – always use clean tweezers to manipulate them.

Hazardous properties of materials can be

## Hazardous Properties

- Flammable means “will burn or support combustion”. Flammable materials are those which have a flash point below 100°F (one of the standards), whereas combustible materials have a flash point in excess of 100°F
  - Flashpoint is the temperature at which the material can vaporize and become a combustible mixture
- Reactivity describes how a material will react in the presence of other materials. A good example of the importance of this knowledge are for acids and solvents
- Corrosivity refers to how quickly the material will attack another substance under a particular conditions (temperature, concentration, pressure)

## Hazardous Properties (cont'd)

- ▶ Corrosivity data will enable chemical users to design appropriate storage and use of appropriate vessels
    - For example, nitric acid ( $\text{HNO}_3$ ) can be stored in glass bottle whereas hydrofluoric acid (HF) has to be stored in plastic container, it is extremely corrosive to glass
  - ▶ Toxicity is the ability of a substance to do harm to the body. The severity depends on concentration, nature of the contact, etc.
- 

- Solvent: a material capable of dissolving another substance to form a solution
- A good solvent dissolves a broad range of other substances
- Water: an excellent solvent of many substances, especially ionic materials ( $\text{NaCl}$ ). Water molecules dissolve ionic-compounds present in the water by separating the ions, then surrounding and dispersing them into the liquid.

Water does this by overcoming the electrostatic force of attraction between the ions

- Common solvents used in micro-fabrication:
- DI water, Isopropyl-alcohol (IPA), Acetone, Xylene (PR edge-bead removal)

### De-ionized Water (Ultra-pure Water)

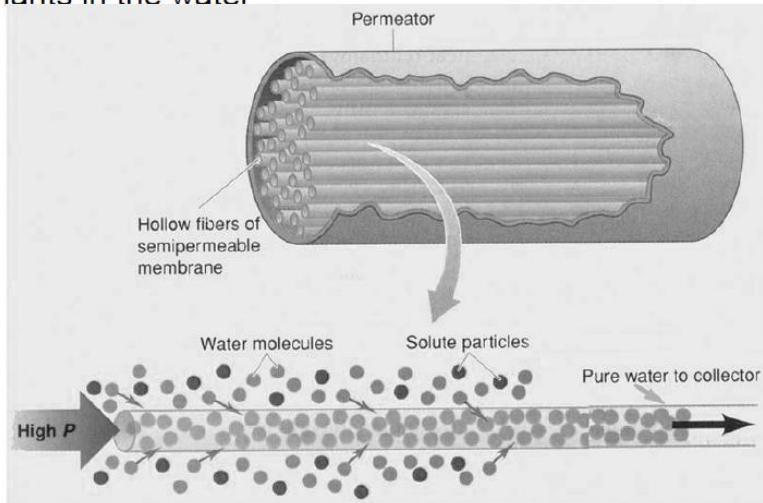
**DI water is water generated through filtering process. The de-ionizing process *does not remove non-ionic organic contaminants***

Large quantities of water are needed during microchip-fabrication – mostly for rinsing wafers after wet cleaning and etching

- Pure water is a neutral substance with a **pH of 7** — this is an important property for water to be used in wafer processing
- City water goes through a series of coarse sand and carbon filtering to remove large particles
- Reverse osmosis and ion exchangers are used to remove salts (ions)
- UV treatment is used to kill bacteria
- DI water quality is monitored by resistivity measurement (**>18 MΩ.cm**)

## Reverse Osmosis Technique

- Reverse Osmosis (RO) units can typically remove more than 90 % of the contaminants in the water



## Isopropyl alcohol

- Isopropyl alcohol dissolves a wide range of non-polar compounds. It evaporates quickly and is relatively non-toxic. It is used widely as a solvent and as a cleaning fluid, especially for dissolving oils
- Often used in cleaning electronic contact pins, magnetic tape and disk heads, optical lenses, etc.
- Also sold commercially as a whiteboard cleaner, and is a strong but safer alternative to common household cleaning products

## Acetone

- Acetone is a good solvent for most plastics and synthetic fibers including polystyrene, polycarbonate and some types of polypropylene
- It is also used as a volatile component of some paints and varnishes. As a heavy-duty degreaser, it is useful in the preparation of metal prior to painting
- It also thins polyester resins, vinyl and adhesives. It is also useful for high reliability soldering applications to remove solder resin after soldering is complete.

## Methanol

- Methanol is colorless, hygroscopic and completely miscible (mixable) with water, but much lighter (specific gravity 0.8)
- It is a good but volatile solvent. It is very toxic and extremely flammable.
- It can be used as a light fuel
- It is also used as an antifreeze in pipelines and windshield washer fluid

## Liquid Chemicals used in Microfabrication

### Common Liquid Chemicals

► Acids used in micro-fabrication:

- HF hydrofluoric acid
- HNO<sub>3</sub> nitric acid
- H<sub>2</sub>SO<sub>4</sub> sulfuric acid
- HCl hydrochloric acid
- H<sub>3</sub>PO<sub>4</sub> Phosphoric acid
- CH<sub>3</sub>COOH acetic acid (commonly found in vinegar)

► Bases used in micro-fabrication:

- KOH potassium hydroxide
- NH<sub>4</sub>OH ammonia

## CHEMICALS USED IN IC FABRICATION: ACIDS, BASES, & SOLVENTS

**TABLE 5.1 - Acids Used in Chip-Making**

Hydrofluoric acid	HF	Etching SiO <sub>2</sub> films and cleaning furnace tubes
Buffered Oxide Etch	BOE	Etching SiO <sub>2</sub> films
Nitric acid	HNO <sub>3</sub>	Used in mixtures of HF and HNO <sub>3</sub> to etch silicon
Sulfuric acid	H <sub>2</sub> SO <sub>4</sub>	Used in a mixture of H <sub>2</sub> SO <sub>4</sub> and H <sub>2</sub> O <sub>2</sub> to clean wafers
Hydrochloric acid	HCl	Used to clean wafers
Phosphoric acid	H <sub>3</sub> PO <sub>4</sub>	Used to etch Si <sub>3</sub> N <sub>4</sub> and Al
Acetic Acid	CH <sub>3</sub> COOH	Used in Al etch baths

**TABLE 5.3 - Solvents Used in Chip-Making**

Ultrapure Water (H <sub>2</sub> O)	UPW	Widely used to rinse wafers and dilute aqueous chemical solutions
Isopropyl Alcohol (C <sub>3</sub> H <sub>8</sub> O)	IPA	General-purpose cleaning solvent
Trichloroethylene	TCE	Cleaning solvent
Acetone		General-purpose cleaner
Xylene		Photoresist edge-bead removal

**TABLE 5.2 - Bases Used in Chip-Making**

Ammonium hydroxide	NH <sub>4</sub> OH	Used in wafer cleaning processes
Potassium hydroxide	KOH	Silicon etch
Tetramethyl-ammonium hydroxide	TMAH	An alkaline developer in photolithography



## **Gases used for Microfabrication**

Gases are classified into two groups:

- a) Bulk gases – used in large quantities
- b) Specialty gases – used as process gases, used in smaller quantities
  - Bulk gas purity is controlled to seven-nines purity (99.99999 %) – grade 5
  - Specialty gases must be controlled to better than four-nines (99.99 %)
  - Contaminants must be excluded from the process gases to extremely low levels of oxygen, water moisture, metals, etc.

## **Gases for Micro-fabrication**

- Common bulk gases used in wafer fab: N<sub>2</sub>, O<sub>2</sub>, H<sub>2</sub>, Ar, He

N <sub>2</sub>	Purging, blow dry wafers
Argon	Glow discharge gas used in PVD
He	Dry-etching gas, vacuum-leak checking
O <sub>2</sub>	Process chamber gas
H <sub>2</sub>	Carrier gas in epi-Si process, wet oxidation

## **Gases for Micro-fabrication**

- Common specialty gases:

Silane (SiH <sub>4</sub> )	Source of Si in deposition processes
Phosphine (PH <sub>3</sub> )	Dopant-gas for ion implanting phosphorus
Silicon tetrachloride (SiCl <sub>4</sub> )	Source gas used in Si-deposition processes
Silicon tetrafluoride (SiF <sub>4</sub> )	Source gas for Si & F-ions used in etch and implant steps
Carbon tetrafluoride (CF <sub>4</sub> )	Etch gas that produces fluorine ions

## Hazardous Process Gases

- ▶ The process gases are grouped into 4 hazardous categories:
  - ▶ **Toxic**: dangerous to human life,  
e.g. Arsine ( $\text{AsH}_3$ ), phosphine ( $\text{PH}_3$ ), and diborane ( $\text{B}_2\text{H}_6$ )
  - ▶ **Corrosive**: capability to destroy living tissues and equipment that comes into contact, e.g. HCl, HF
  - ▶ **Flammable**: substances that give off vapors that can be readily ignited if exposed to sparks and flames,  
e.g.  $\text{H}_2$ ,  $\text{PH}_3$ ,  $\text{SiHCl}_3$
  - ▶ **Pyrophoric**: ability to ignite spontaneously in air at 54 °C (or below), e.g. silane ( $\text{SiH}_4$  a gas widely used to deposit polysilicon,  $\text{SiO}_2$  or  $\text{Si}_3\text{N}_4$  by Chemical Vapour Deposition)
- 

## Gas Sources and Delivery Systems (cont'd)

- ▶ Gases are transported and stored in 100-lb metal containers called gas cylinders, usually in gas cabinets
- ▶ The cabinets are connected to an exhaust duct to minimize personnel exposure in the event of a leak
- ▶ Gases must also be properly purged from a gas system or delivery lines after every use



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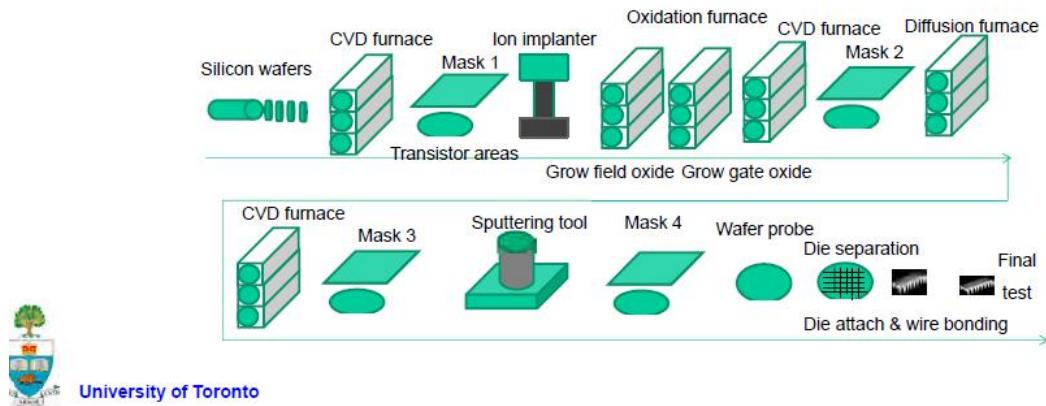
Gas Cabinet

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### 3. Making of Silicon Wafers

#### IC Manufacturing

- The manufacturing flow is basically a sequence of processing step to build up the IC layer by layer. Starting with the silicon wafer, layers of silicon dioxide, polysilicon, metal layers and dopants are added in strategic locations to form the final IC chip



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#### CZ and FZ techniques

##### Starting Material

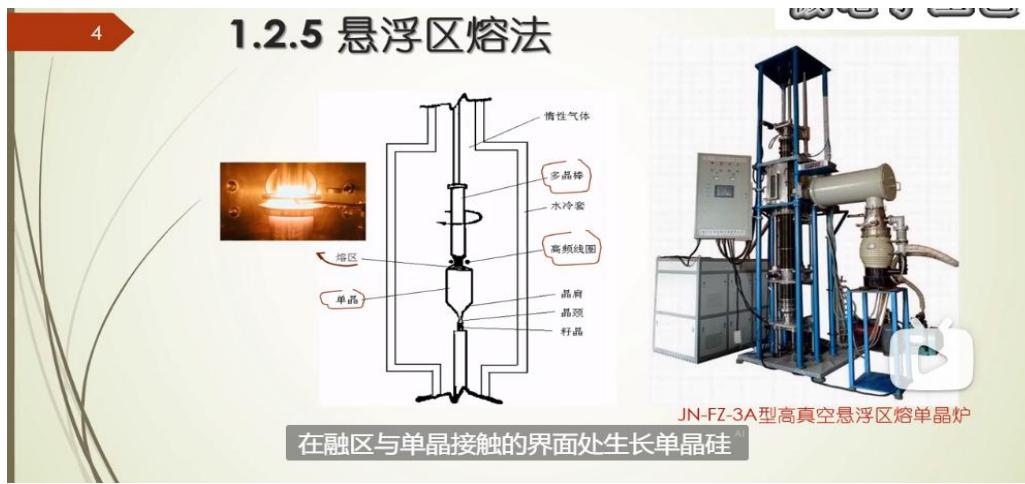
Electronic-grade polysilicon nuggets are melted in a quartz crucible at a  $T > 1400^{\circ}\text{C}$  in an inert gas atmosphere (e. g. argon).

**Czochralski-technique (CZ 直拉法)** is a method to pull a monocrystal ingot with the same crystallographic orientation of a small monocrystalline seed crystal out of molten silicon. Since the ingot is pulled out from the silica crucible (refer Figure 10.7a), some oxygen contamination is always present in the silicon. The seed crystal's rod is slowly pulled upwards and rotated simultaneously. Width is controlled by precise control of temperature, speeds of rotation etc.

##### After Ingot is made

**Float-Zone technique** (悬浮区技术) is used to re-fine/purify the silicon ingot.

An RF coil melts a small region of the polysilicon which, after cooling down, forms monocrystalline silicon with the crystallographic orientation of the seed crystal (e. g.  $<100>$ ,  $<110>$  or  $<111>$ )



Chemical-Mechanical Polishing (mech force and chemical etching)

CMP is used to remove micrometers only. Atomic bonds are weakened or broken, and removal is based on chemical reactions between the slurry and the surface and the mechanical effect of the abrasive particles (direct contact with wafer, high friction and no lubrication).

- mirror-like surface (surface irregularities are smoothed out, for wire bonding)
- planarization of topography (step height reduction)
- removal of hard-to-etch materials

Abrasive particles (silica SiO<sub>2</sub>, alumina Al<sub>2</sub>O<sub>3</sub>) are dispersed in a suitable liquid to create a slurry, which is fed in between a polishing pad and the wafer.

## 4. Wafer Cleaning and Surface Preparation

### [1] Chapter 12

Wafer cleaning is about removing particles and unwanted atoms and films, and it is also about leaving the surface in a known and controlled condition.

Acid, base, and solvent wet cleaning are the main methods of cleaning.

**Particles:** Physical wet cleaning: brush scrubbing. Jet scrubbing, rotating brush and spray of de-ionized water. (after CMP)

SPM: H<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>O<sub>2</sub> (Piranha) remove organic residues (such as photoresist films). Leaves difficult-to-remove sulfur residues. Followed by-SC1.

**Organics (contain carbon):** RCA-SC1, Ammonia ( $\text{NH}_4\text{OH}$ )-peroxide. Can be removed using solvent baths such as alcohol or acetone.

**InOrganic (metal Removal):** RCA-SC2, acidic solutions HCL-H<sub>2</sub>O<sub>2</sub>.

**Native oxide:** HF dip: Hydrofluoric (HF) acid is often used to remove the oxide. HF does not etch silicon LAB 1 done.

Rinsing in DI water and drying must be considered in wet cleaning process.

Bulk silicon wafers are single crystal pieces cut from larger single crystal ingots and polished. Polysilicon consists of grains or many orientations which have different oxidation rates.

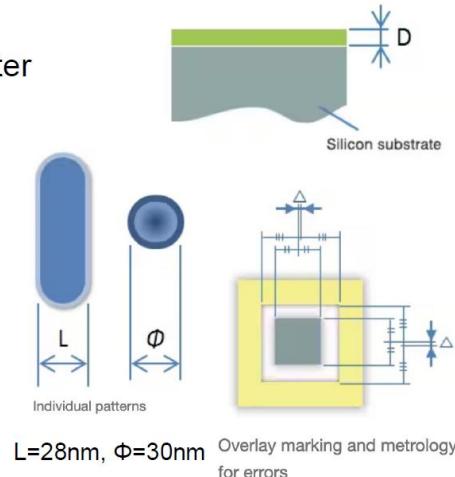
## 5. Introduction to crystallography (lattices, planes, etc)

Critical Dimensions: CD is the smallest feature size that a manufacturer can reliably produce and measure on a semiconductor wafer.

A Critical Dimension SEM (CD-SEM: Critical Dimension Scanning Electron Microscope) is a dedicated system for measuring the dimensions of the fine patterns formed on a semiconductor wafer. primary electron beam irradiating to the sample has low energy of 1keV or below to reduce damage to sample.

### Examples of Metrology

- ▶ Measurement of the line width and hole diameter of a circuit pattern at a specified location of a semiconductor wafer
- ▶ Film thickness
- ▶ Overlay marking and metrology errors
- ▶  $1\text{nm} = 10^{-9}\text{ m}$
- ▶ In comparison:
  - Human hair:  $60 - 100\mu\text{m}$  ( $60,000 - 100,000\text{ nm}$ )
  - Bacteria:  $1\mu\text{m}$  ( $1,000\text{nm}$ )
  - Cigarette smoke (carbon black)  $100\text{ nm}$
  - Virus:  $50\text{ nm}$



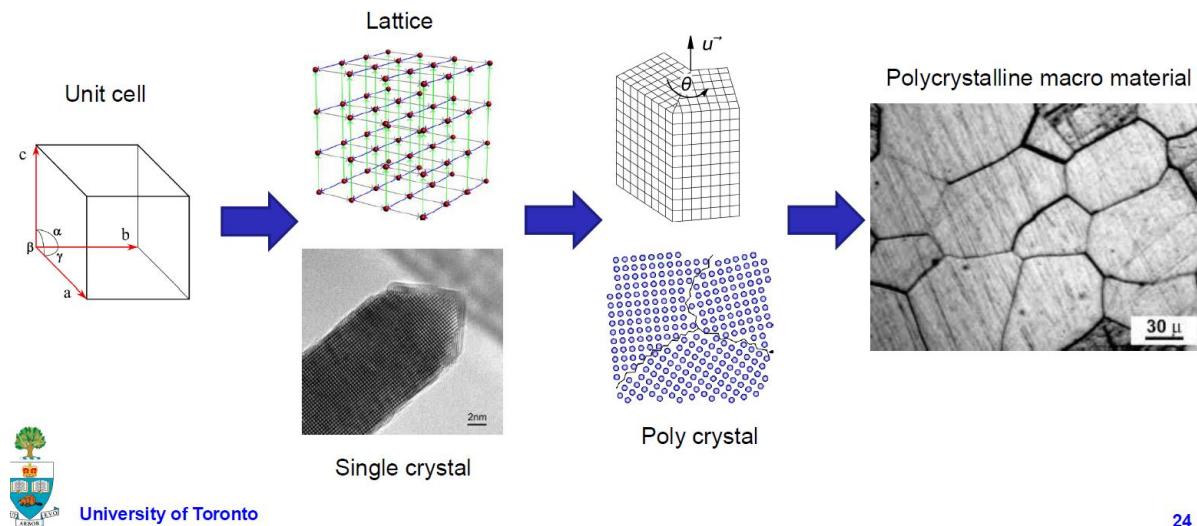
Materials properties and utilization may vary depending on the *crystallographic orientation, morphology and structure*.

### Crystal Lattice (晶格)

-Unit Cell(晶胞): the fundamental building block from which a crystal is constructed.  
Defined by 6 Lattice Parameters. (单晶体)

### Polycrystalline (多晶体)

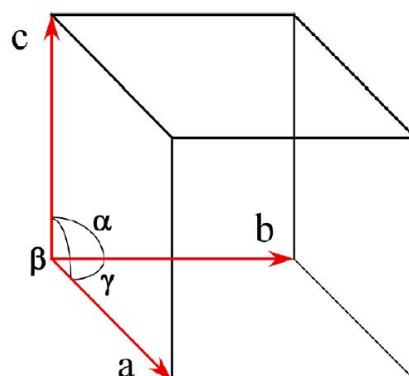
## The Crystal Lattice – Single- and Poly-Crystals



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- ▶ Seven crystal **types** are used to define the **subtypes** of Bravais lattices
- ▶ The unit cell possesses the same symmetry as the overall crystal structure

Cubic	$a=b=c$	$\alpha=\beta=\gamma=90^\circ$
Tetragonal	$a=b \neq c$	$\alpha=\beta=\gamma=90^\circ$
Orthorhombic	$a \neq b \neq c$	$\alpha=\beta=\gamma=90^\circ$
Hexagonal	$a=b \neq c$	$\alpha=\beta=90^\circ$ $\gamma=120^\circ$
Monoclinic	$a \neq b \neq c$	$\alpha=\beta=90^\circ \neq \gamma$
Triclinic	$a \neq b \neq c$	$\alpha \neq \beta \neq \gamma$
Rhombohedral	$a=b=c$	$\alpha=\beta=\gamma \neq 90^\circ$

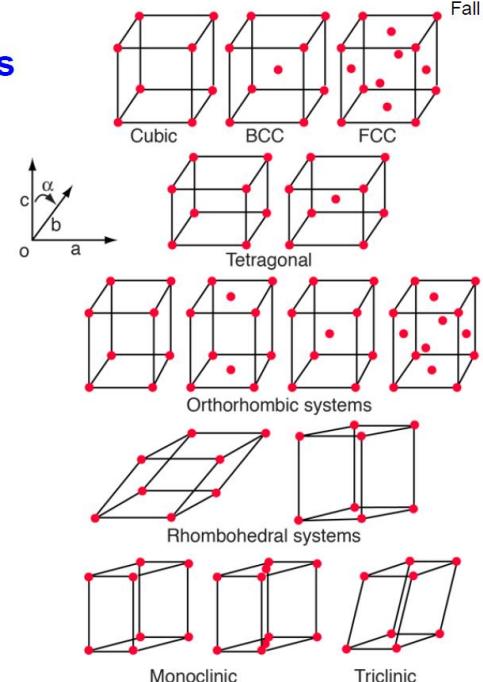


## The Crystal Lattice - Bravais Lattices

- The Bravais lattices provide the fundamental framework for describing the crystalline structure of solids
- A crystal is formed by placing a **basis** (an atom, ion, or molecule) at each point of a Bravais lattice.
- The **properties** of the crystal, such as its mechanical and optical characteristics, are directly related to the symmetry of its Bravais lattice



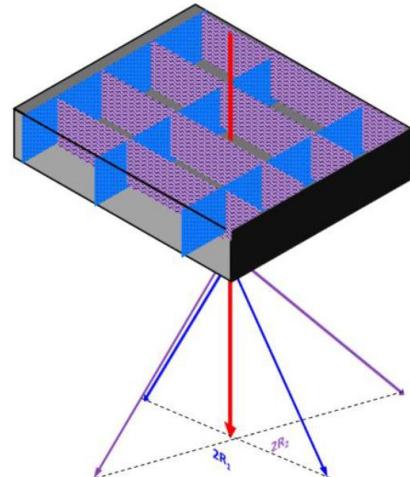
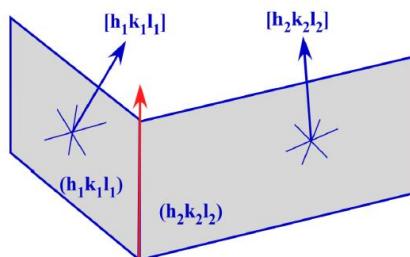
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## Zone Axis

- A lattice row parallel to the intersection of two (or more) families of lattices planes
- In electron microscopy (SEM/TEM), the zone axis tells you which direction the electron beam is traveling through the crystal
- Different zone axes give different diffraction patterns, helping identify **crystal structure and orientation**



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Suggested for you Add comments Highlight Add Text ...

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## 6. Electron-material interactions (Interaction volume)

Wavelength of electrons typically used in electron microscopy <10pm

Substituting:

$$\lambda = \frac{h}{(2m_0eV)^{1/2}}$$

**Key point: the higher the voltage, the shorter the wavelength**

Green is 3.7 pm and red is 1.97pm

### Basic properties of electrons

Accelerating Voltage (kV)	Non-relativistic wavelength (Å)	Relativistic wavelength (Å)	Mass (x m <sub>e</sub> )	Velocity (x 10 <sup>8</sup> m/s)	Velocity (x c)
100	0.0386	0.0370	1.196	1.644	0.54
200	0.0273	0.0251	1.391	2.086	0.69
300	0.0223	0.0197	1.587	2.330	0.78
400	0.0193	0.0164	1.783	2.484	0.83
1000	0.0122	0.0087	2.957	2.823	0.94

Magnification is a function of scan size: reduce scan size => increase magnification.

### Angles & Distances

Some things worth remembering ...

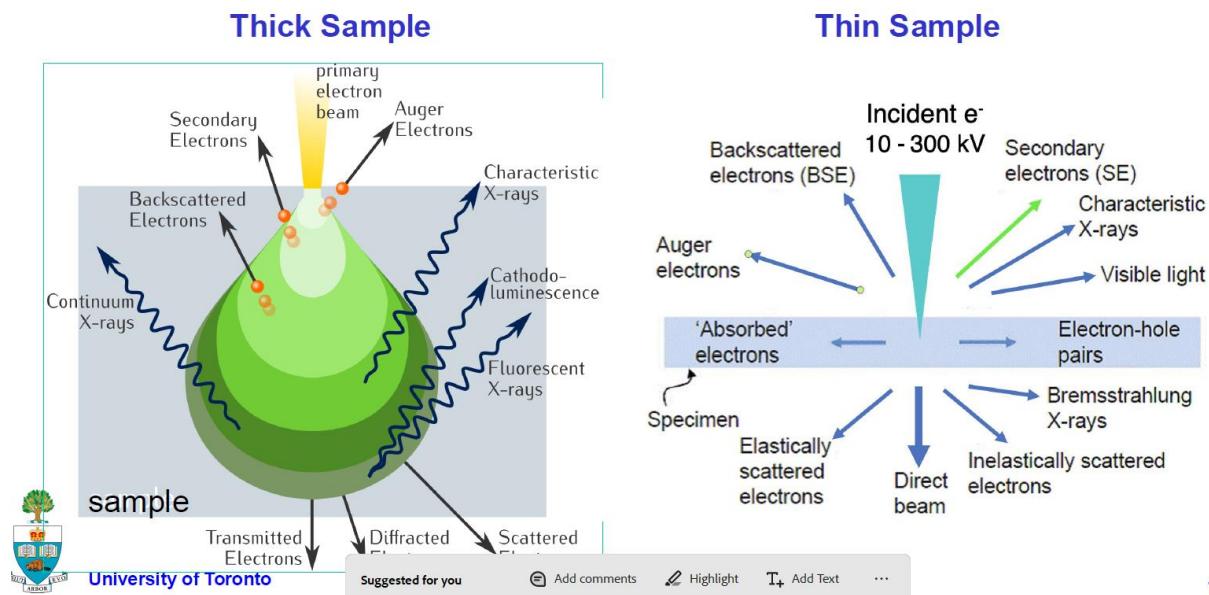
We deal often with very small angles:

Angle of 1° = 17.5 milliradians (mrad) ≈ 15 mrad

We use very high magnifications:

- At 1000 X, 1 cm = 10 μm
  - At 10,000X, 1 cm = 1 μm
  - At 50,000X, 1 cm = 200 nm
  - At 100,000X, 1 cm = 100 nm
  - At 500,000X, 1 cm = 20 nm = 200 Å
- Try to memorize these two

## Electron Beam ↔ Material Interaction

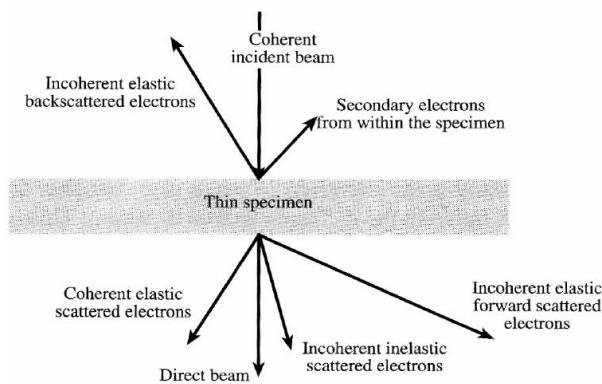


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## Scatter : 散射

## Electrons wave/particle duality

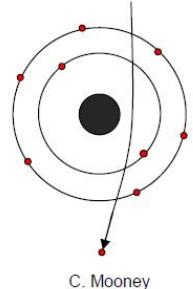
- ▶ Particle perspective:
  - Collision between electron and atom
  - No energy loss: elastic
  - Energy loss: inelastic
- ▶ Wave perspective:
  - Coherent - maintains phase
  - Incoherent - does not maintain phase
- ▶ Electron scattering is the underlying physics of EM
  - Diffraction: elastic scattering
  - Imaging: elastic & inelastic scattering
  - Spectroscopy: inelastic scattering



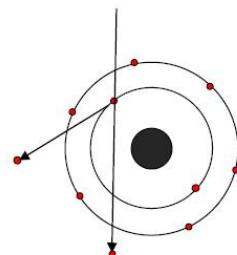
## Electron Beam-Sample Interactions

Interactions between primary electrons and the sample can be separated into two broad categories:

- Elastic scattering
  - Trajectory of the electron is affected
  - Energy of the electron is not (significantly) affected
  - Example: Backscattered Electrons
  
- Inelastic scattering
  - Trajectory of the electron is not (significantly) affected
  - Energy of the electron is transferred to an atom in the sample
  - Examples: Secondary electrons and X-rays



C. Mooney



### Elastic Scattering (弹性散射)

### Elastic scattering – Interaction Cross-section ( $\sigma$ )

- Interaction cross-section expresses the probability of a given scattering event.  
Generally:

$$\sigma = \pi r^2$$

- Elastic scattering radius has the form:

$$r_{electron} = \frac{e}{V\theta} \quad r_{nucleus} = \frac{Z e}{V\theta}$$

$\sigma$  cross-section  
Z atomic number

$V \uparrow$	$\sigma \downarrow$	e charge of the electron
$Z \uparrow$	$\sigma \uparrow$	V potential of the electron
$\theta \uparrow$	$\sigma \downarrow$	$\theta$ angle

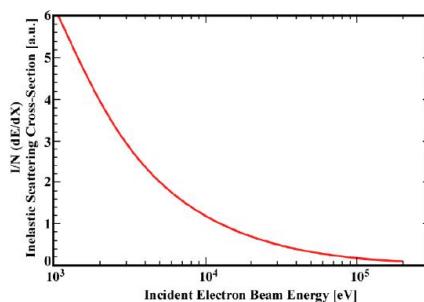


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If I throw a ball (a football) at a glass window one square meter in area, and there is one chance in ten that the window will break, and nine chances in ten that the window will not break, then there is an elastic cross-section of  $0.9m^2$  and an inelastic cross-section of  $0.1m^2$ .



Inelastic scattering cross-section as a function of incident electron beam energy



## Elastic scattering – Mean Free Path ( $\lambda$ )

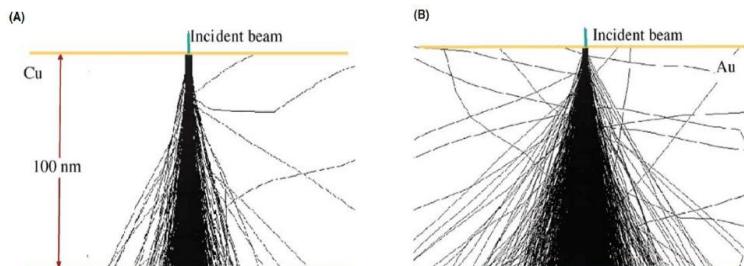
- The average distance an electron travels between successive interactions (collisions or scatterings)

$$\lambda = \frac{1}{n \cdot \sigma}$$

$\lambda$  = mean free path

n = number density of target particles

$\sigma$  = interaction cross section



**Monte Carlo simulation** of the paths followed by 103 100-keV electrons as they pass through thin foils of (A) Cu and (B) Au. Notice the increase in scattering angle with atomic number and the small number of electrons that are scattered through  $>90^\circ$ .

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## Elastic scattering – Bragg's Law – Electron Diffraction

- Describes the condition for **constructive interference** of waves scattered by a crystal lattice.

$$\lambda = \frac{2d \sin(\theta)}{n}$$

n = order of reflection (usually 1)

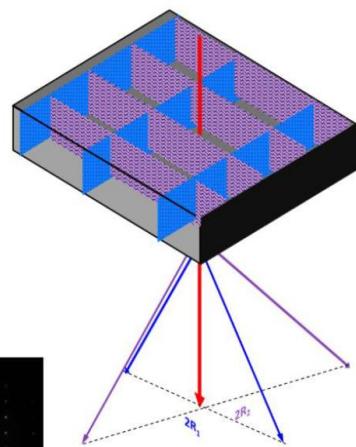
$\lambda$  = wavelength

d = spacing between crystal planes

$\theta$  = angle of incidence/reflection

$$\text{For } +100\text{keV:} \\ \lambda_e = \frac{h}{p} \approx 10^{-3} [\text{nm}]$$

$$\begin{array}{c} \downarrow \\ d\text{-space } [\text{\AA}] \\ \downarrow \\ \lambda \approx 2d\theta \end{array}$$



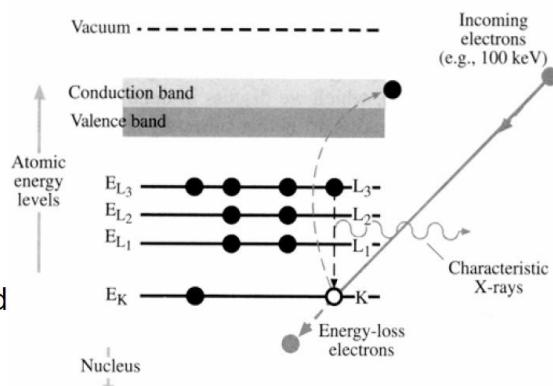
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## Inelastic Scattering (非弹性散射) and X-Rays

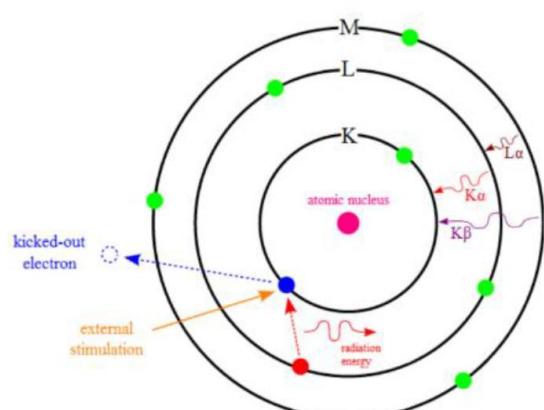
### Inelastic Scattering – Characteristic X-ray

- ▶ Interaction w/ inner shell electrons
- ▶ If energy sufficient, inner shell electron ejected
  - Atom is ‘ionized’
- ▶ Atom can return to its lowest energy state (ground)
- ▶ Electron from outer shell to fill the hole in the inner shell
- ▶ A characteristic X-ray photon is emitted
- ▶ Each ionization event sets off a cascade of transitions

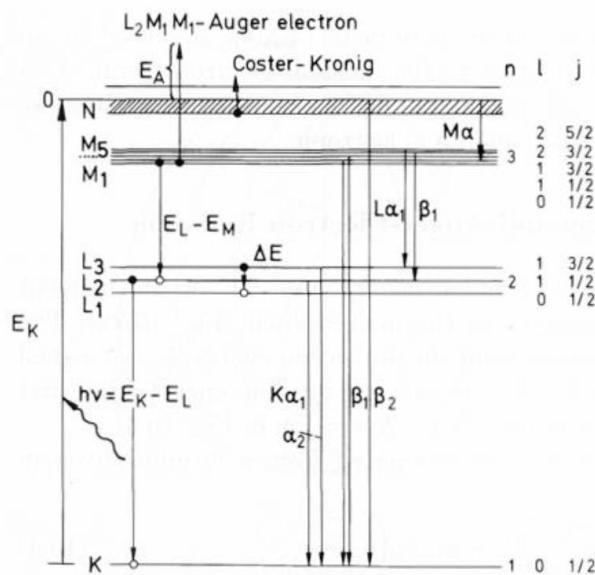


X-rays are generated in the sample by high energy electrons interacting with the sample

### Inelastic Scattering – Characteristic X-ray – Nomenclature



**Cu-K $\alpha$**



## Secondary Electrons (SE)

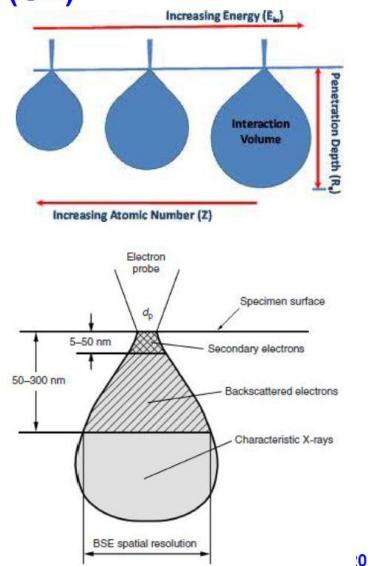
### Inelastic Scattering – Secondary Electrons (SE)

► Incident electrons:

- Impart energy to electrons in the crystal
- Slow secondary electrons
  - Electrons from the conduction or valence band
  - Require little energy: yields slow, unenergetic electrons (<50 eV)
  - These are the electrons used in the SEM for morphology detection
- Fast secondary electrons
  - Inner shell electrons are ejected
  - Requires much energy, yields fast, energetic (~50-200 keV) electrons
  - Result in additional scattering processes, degrade microanalysis resolution



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## Interaction Volume

Electrons do not stop at the surface – they can penetrate microns into a sample and generate signals throughout the interaction volume

-generally determined via Monte Carlo simulations

The dimensions of the interaction volume depend on:

- Primary electron energy
- Atomic weight
- Density

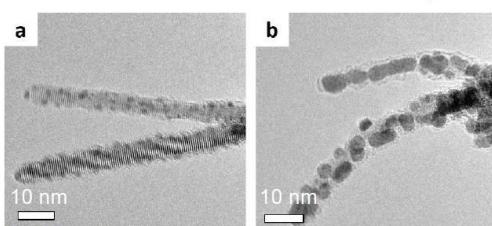
## Beam Damage

## Beam Damage

- ▶ High-voltage, high-current-density electron beam can cause considerable damage to the sample
- ▶ Two types:
  - Radiolysis: Inelastic scattering  $\Rightarrow$  ionization which breaks bonds
  - “Knock-on” damage: direct displacement of atoms
- ▶ Increasing voltage: less radiolysis, more ‘knock-on’
- ▶ High energy may propagate decomposition and unwanted reaction while inspecting



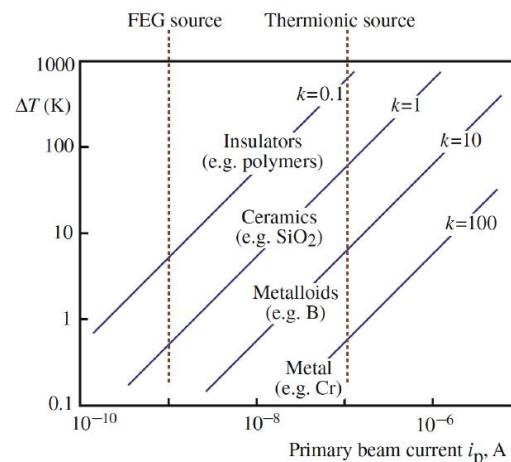
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[youtube.com/watch?v=oMTMtmQaw\\_k](https://youtube.com/watch?v=oMTMtmQaw_k)

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## Beam Damage – Specimen Heating

- ▶ Generally, not a large issue for metals and semiconductors
- ▶ Is a problem for ceramics, polymers, and biological samples
- ▶ Possible solutions:
  - Reduce the cross section
  - Thinner samples
  - Higher voltage



**FIGURE 4.11.** The increase in specimen temperature as a function of the beam current and the thermal conductivity of the specimen ( $k$ , in  $\text{W}/\text{m K}$ ). Typical materials are noted, but should not be considered representative, since  $k$  varies substantially in any class of materials.



## 7. Electron Source

**Beam Energy:** up to 30keV. The parameter represents the initial energy of the electrons as they enter the SEM chamber or sample.

-1 eV is the energy acquired by an electron when it is accelerated through a potential difference of 1 v.

Beam Diameter: range of 1nm to 1um, symbol of  $d_p$ ,  $d_o$ .

-small diameter, higher spatial resolution imaging. However, beam current is reduced as the inverse square of the beam diameter. Visibility is compromised.

### Source characteristics

#### Brightness

$$\text{Brightness: } \beta = \frac{4i_e}{(\pi d_0 a_0)^2}$$

- ☞ Beam diameter:  $d_o$
- ☞ Cathode emission current:  $i_e$
- ☞ Semi-angle of divergence from source:  $a_0$

#### Brightness ( $\text{A/m}^2\text{s}\cdot\text{sr}$ )

Thermionic	$10^9$
Schottky	$5 \cdot 10^{10}$
Cold Field Emission	$10^{13}$

#### This is a key parameter:

- ☞ impacts exposure times
- ☞ analytical work



### Source characteristics

#### Temporal coherency & energy spread

Temporal coherency refers to the energy spread of the source

☞ Analogue to light optics is “color”

☞ Coherence length:  $\lambda_c = \frac{vh}{\Delta E}$  (5.4)

$v$ : electron velocity,

$\Delta E$ : energy spread of the beam

$h$  is Planck's constant

#### Typical $\Delta E$

☞ Tungsten thermionic: 3 eV

☞ LaB<sub>6</sub> thermionic: 1 eV

☞ Schottky field emission:  $\approx 0.8$  eV

☞ Cold field emission: 0.3 eV

☞ Note this is on top of 200 to 300 keV

Important with respect to EEL spectroscopy



## Source characteristics

### Spatial coherency & source size

Spatial coherency is associated with the physical 'point of origin' of the electrons

Related to the "effective source size" ( $d_c$ ):  $d_c \ll \frac{\lambda}{2\alpha}$

To improve spatial coherency:

$d_c \downarrow$ (FEG);  $\lambda \downarrow$ ( $\uparrow kV$ );  $\alpha \downarrow$ ( $\downarrow$ aperture)

Improved spatial coherency:

↳ Helps with high resolution imaging

↳ Gives sharper diffraction patterns

↳ Gives better diffraction contrast images

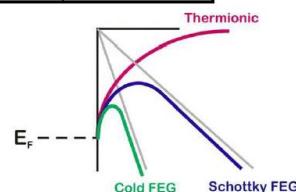


## Emission physics

### Summary

	$\beta$ (A/m <sup>2</sup> sr)	$\Delta E$ (eV)	d	Vacuum (Pa)
W	$10^9$	1.5 - 3	20 - 50 $\mu m$	$10^{-3}$
LaB <sub>6</sub>	$5 \cdot 10^9$	1 - 2	10 - 20 $\mu m$	$10^{-4}$
Schottky FEG	$5 \cdot 10^{10}$	0.7	15 nm	$10^{-6}$
Cold FEG	$10^{13}$	0.3	2.5 nm	$10^{-8}$

- Conventional TEM - LaB<sub>6</sub> / CeB<sub>6</sub>
- Schottky FEG - Conventional analytical
- Cold FEG - Very high end analytical



2 Types of Electron gun: -Thermionic (SU3500). -Schottky Field Emission (SU7000). -Cold Field Emission

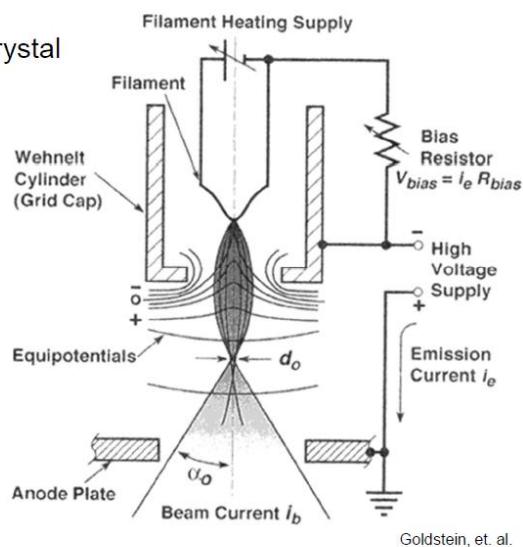
Thermionic electron guns

- Heat a cathode filament (~2700K) (Tungsten) until electrons boil off the surface, then accelerate them down the electron optical column
- Use thermal energy to overcome a cathode's work function (EW) and release electrons.

## Thermionic Electron Gun

Components of a Thermionic Electron Gun:

- **Filament** (plus heating supply)
  - Typically W wire or a LaB<sub>6</sub>/CeB<sub>6</sub> crystal
  - Source of electrons
  - Electrons escape surface via thermal energy
- **Wehnelt** (plus bias resistor)
  - Simple electrostatic lens
  - Forms first crossover
  - Also inhibits emission from filament not at apex
- **Anode** (plus HV supply)
  - Accelerates electrons



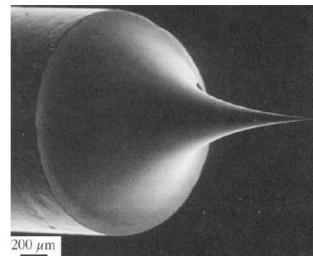
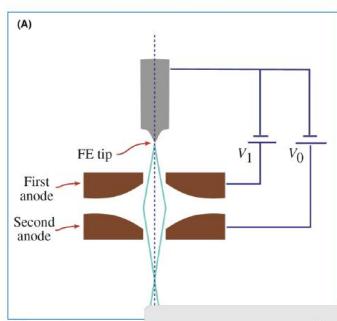
Goldstein, et. al.

## Schottky Field Emission

### Electron guns

#### Field Emission Gun (Schottky)

- First anode ( $V_1$ ) is extraction voltage  
Reduces the work function of the tip (increases the electron emission from the tip)
- Second anode ( $V_2$ ) acts as an electrostatic lens
- Different extraction voltages for different operation modes



FE electron guns are capable of very high resolution – high end

-FESEM resolution is on the order of 0.5nm! (very high good)

-Requires ultra-high vacuum ( $10^{-6}$  Pa)

– Suspect 0.5 nm is a fundamental resolution limit

-Also, Schottkey guns have very high “**Brightness**.” This means that more electrons per unit area are emitted. As the beam in a thermal emitter is made smaller and smaller, the number of electrons in the beam becomes so small that no usable signal can be obtained from the sample. **Also means current density can be high with a FE-SEM.**

A Schottky source can produce more actual current than a cold or thermal FE source

## Electron Gun - Comparison

Thermionic	Thermal-Field (Schottky) Emission	Cold Field Emitter
Major Advantages:	Major Advantages:	Major Advantages:
<ul style="list-style-type: none"> <li>Very high probe currents obtainable           <ul style="list-style-type: none"> <li>Stable probe, especially W</li> <li>Less complex vacuum system</li> <li>Lowest overall cost / easy to maintain</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>Very high brightness source</li> <li>High probe currents obtainable (few hundred nA)</li> <li>Long potential source lifetime (few years)</li> <li>Excellent short and long term stability</li> </ul>	<ul style="list-style-type: none"> <li>Highest brightness SEM source available</li> <li>Very long potential source lifetime – many years</li> </ul>
Disadvantages:	Disadvantages:	Disadvantages:
<ul style="list-style-type: none"> <li>Lower brightness</li> <li>Relatively short lifetimes</li> </ul>	<ul style="list-style-type: none"> <li>Requires ultra-high vacuum in gun area</li> <li>Source heating is continuous, 24/7 (finite life)</li> <li>Cost (initial and maintenance)</li> </ul>	<ul style="list-style-type: none"> <li>Lowest maximum probe current</li> <li>Requires ultra-high vacuum in gun area</li> <li>Cost (initial)</li> </ul>



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Suggested for you

Add comments

Highlight

Add Text

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Aberrations – Lens Defect 2025.12 caused by magnetic field (2 types)

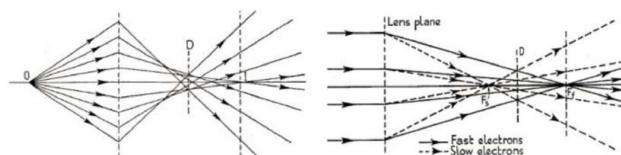
-Can be corrected using stigmators or mini lenses!

## Aberrations

Lens defect caused by magnetic field asymmetry

- Spherical

- Chromatic



$$r_{sph} = C_s \beta^3$$

$$r_{chr} = C_c \frac{\Delta E}{E} \beta$$

## **Electron Lenses – Chromatic Aberrations**

- Lorentz Force Law

$$\vec{F} = -e(\vec{v} \times \vec{B})$$

- We want all the electrons to have the same energy, which is expressed as velocity, however, there are often small differences in energy between emitted electrons
- A delta in the velocity will result in:

$$\Delta\vec{F} = -e(\Delta\vec{v} \times \vec{B})$$

- Electrons with different velocities get focused to different places, which is like not being in focus!
- This is called a **chromatic aberration**, or Ca

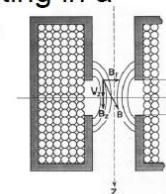
## **Electron Lenses – Spherical Aberrations**

- Lorentz Force Law

$$\vec{F} = -e(\vec{v} \times \vec{B})$$

- The B-field varies radially across the bore of the lens, resulting in a delta in the B-field (in addition to any potential errors in the windings)
- A delta in the B-field will result in:

$$\Delta\vec{F} = -e(\vec{v} \times \Delta\vec{B})$$



- Electrons going through different parts of the lens will get focused to different places, which is like not being in focus!
- This is called a **spherical aberration** or Cs

## 8. Scanning Electron Microscopy (SEM)

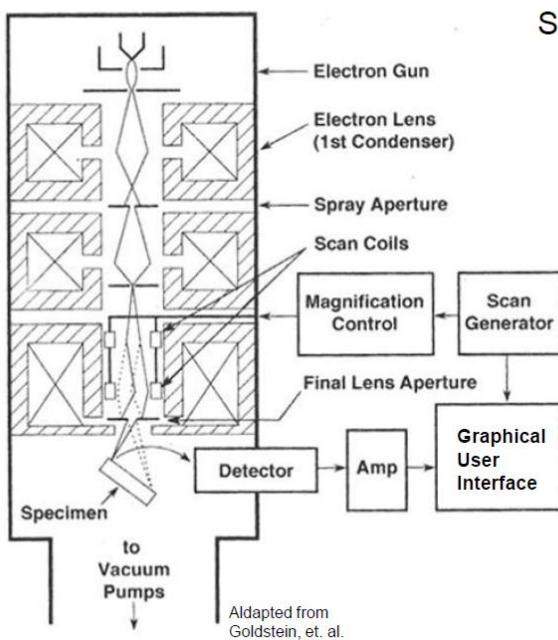
Scanning an *electron probe* (*the electron beam*) over a sample and mapping probe position vs. sample-probe interaction where the displayed image is larger than the scanned region.

Optics form the probe! (SEM probe = electron beam)

- Optics = source, lenses, apertures, and correctors
- Probe characteristics are determined by the optics parameters
- Optics parameters include beam energy, current, spot size, and convergence angle
- The probe is aka the beam or the spot (terms used interchangably)

**Resolution** is a function of optics parameters: Smaller probe (spot) on the sample = higher (potential) resolution

### SEM Schematic



#### SEM Parts:

- **Electron gun**
  - Source of electrons
- **Condenser lens(es)**
  - Spot size/current control
- **Objective lens**
  - Focus control
- **Objective aperture**
  - Helps control depth of field
- **Scanning coils**
  - Magnification control
- **Stigmator coils**
- **Scan generator**
- **Detector(s)**
- **Graphical User Interface (GUI)**
  - Typically a computer control interface
  - Displays images and other data

Scanning Electron Microscope (SU7000) SEM

Back-Scattered Electron Detector (BSED)

[https://serc.carleton.edu/research\\_education/geochemsheets/bse.html](https://serc.carleton.edu/research_education/geochemsheets/bse.html)

Why do we need vacuum

-Electron Mean free path. The mean free path of a particle is the distance that the particle can travel in a medium without scattering event.

Mean free path of an electron in vacuum is 1m!

-In order for field emission to happen. Prevent gas particles from absorbing onto the sharp tip (Schottky Emitter) , which can cause instability, contamination.

## 9. Thermal Oxidation

[1] Chapter 13 (950 degrees – 1100 degrees)

Dry oxidation: thin oxide (1-20nm): dielectric layer. Better thickness control, have lower interface charges and trap states.

Wet Oxidation: thick oxide (100-1000nm): diffusion/etch masks.

This leads to a significant number of broken bonds (dangling bonds)

Hydrogenation is a common method to fulfill these dangling bonds, but the interface charge cannot be eliminated completely. (hydrogen anneal – hydrogen attaches to the free valence of the silicon and eliminates further charge trapping).

High pressure oxidation can reduce oxidation time significantly.

Thermal oxidation produce uniformity, density, Ebreak good oxide. BUT CVD oxide allow lower process temperatures, and thick oxide only take few mins. However, CVD oxide lower breakthrough voltage, higher wet etch rate in HF, and rougher surfaces.

Plasma is used to grown thin oxides, AlO etc.

LOCOS: Si<sub>3</sub>N<sub>4</sub> (silicon nitride) is an effective mask against oxygen during thermal oxidation.

Process flow: thermal oxidation (pad oxide), LPCVD nitride deposition, lithography, nitride etching, photoresist strip, cleaning, wet oxidation.

Pad oxide serves as a stress-relief layer, and it diminishes the stress-induced dislocation that thick nitride exerts in silicon.

Nitride acts as diffusion barrier for oxygen diffusion.

## 10. Diffusion and Ion Implantation

[1] Chapter 14, 15

**Diffusion:** locally vary the dopant concentration. Thermal diffusion 900-1200 degrees, batch process. Done fromg as phase, and junction depth is the depth where diffused dopant concentration equals substrate dopant concentration.

CMOS well diffusion could be 16 hours, 1150 degrees, 5um deep.

Diffusion is usually carried out in two steps: pre-deposition and drive-in.

Pre-deposition is a constant source diffusion where a continuous flow of impurity is dissolved into the wafer surface.

During drive-in, the source of impurity is removed. O<sub>2</sub> & N<sub>2</sub> introduced.

**Ion Implantation** (mostly used ROOM TEMPERATURE): where ionized dopants are accelerated into silicon, can be considered as a pre-deposition step for diffusion. An ion implanter produces ions of the desired impurity, accelerates them by an electric field, and allows them to strike the silicon surface.

**The dose of ions** implanted (specified in cm<sup>-2</sup>) can be controlled by varying the beam current.

Ion implantation energy 10-200kev. The higher, the deeper the ions will penetrate.

The accurate dose control of implantation is used to perform pre-deposition, and thermal drive-in also required to reduce crystal lattice damage, which determines final dopant profile.

**Channeling:** charged particles penetrate deeper into a crystalline solid, like silicon (Si), by moving through the relatively empty spaces, or channels, channeled ions deep into the substrate. Channeling can be reduced by tilting the <100> silicon by approximately 7° relative to the ion beam, leading to SHADOWING EFFECT. Solve: rotating the wafer during the implant

Al cannot be used in self-aligned gate process since the anneal step after implantation requires 1000+ tem.

## 11. Wet and Dry Etching

[1] Chapter 11, 20, 21

Selectivity and degree of anisotropy.

**Wet Etch** uses liquid chemical (result in an isotropic profile):

Buffered Hydroflouric Acid (BHF) for SiO<sub>2</sub>

Potassium Hydroxide (KOH) for Silicon

Phosphoric Acid for Aluminum

Hot Phosphoric Acid (180°C) for Silicon Nitride64

Slope of side wall govern by etchant concentration and temperature.

Undercut can be compensated by making the initial mask feature larger than the desired width, for light-field structures, and smaller for dark-field structure.

Wet Etch has various limitation for small geometries: limited to pattern sizes of 3μm or larger, Requires rinse and dry steps, wet chemicals are hazardous and/or toxic.

**Dry Etch** is a generic term that refers to the etching techniques in which gases are the primary etch medium. The wafers are etched without wet chemicals or rinsing.

**Plasma etching (RIE)**: combination of plasma etching and ion-milling principles. Done in a vacuum chamber by reactive gases excited by RF fields.

**Ion-milling** uses ion-beam (inert gas, Ar) to physically bombard the wafer surface. Etch all material so poor selectivity. Can make inclined structure.

## 12. Thin-film Materials and Processes

[1] Chapter 7

Epitaxial films remain single crystalline during annealing.

Polycrystalline films experience grain growth and phase transitions into other crystal structures.

Amorphous films stay amorphous or crystalize.

### **THIN FILM**

Properties such as refractive index, resistivity, are thickness dependent!

Thin-Film Resistivity always higher than bulk.

## **PVD**

Material ejection from a solid target material, transported in a vacuum to the substrate surface where film deposition takes place. By means of resistive heating, electron beam heating, ion bombardment.

All Al films are deposited by PVD.

**Evaporation:** hot metals have high vapor pressure and In high vacuum the evaporated atoms will be transported to the substrate. Electron beam heating (localized heating) , but deposition rate is very low. 0.1-1nm/s. Evaporation is performed in a High vacuum (means low pressure).

< 10<sup>-5</sup> Torr, atoms do not experience collision, so line-of-sight route, therefore sidewalls not coated!

**Sputtering** is most important PVD: Argon ions hits the negatively biased target and eject one target atom. The atom will be transported to the substrate wafer in vacuum.

Rate 1-10nm/S, higher !

Sputtering pressure are quite high 1-10mTorr, sputtered atoms will experience many collisions before reaching the substrate. Called Thermalization.

Losely bound atoms will be knocked out, improve adhesion and making the film denser.

Reactive sputtering :forming oxide films. A critical challenge in reactive sputtering (using a metal target and introducing oxygen gas) is that the target surface itself becomes oxidized. This is called "target poisoning".

## **CVD**

The source materials are brought into the reactor in GAS PHASE, they are activated in the plasma, diffuse to wafer surface and react there to deposit film.

Silane and oxygen create CVD oxide. Reactor wall must be cleaned since Silane and oxygen can already react in the gas phase.

LTO, 425 degrees to form CVD oxide.

## **PECVD**

To enhance source gas decomposition and reactions by plasmas. At 300 degrees, same 0.1-10nm/s, enabling deposition on metals.

### **Molecular Beam Epitaxy (variant of evaporation)**

**Heated single-crystal sample (400-800 °C) is placed in an ultra-high vacuum (10-11 Torr) in the path of a stream of atoms from heated cells containing the material of interests. The atom beam coming out from the orifice is much more stable.**

**MBE is the most sophisticated form of PVD, deposition rate is very slow, 1µm/hr.**

Crystal lattice of the film and the substrate must be identical.

Heteroepitaxy: epitaxy on dissimilar materials.

## **13. Optical Lithography**

[1] Chapter 8, 9

### **Photolithography**

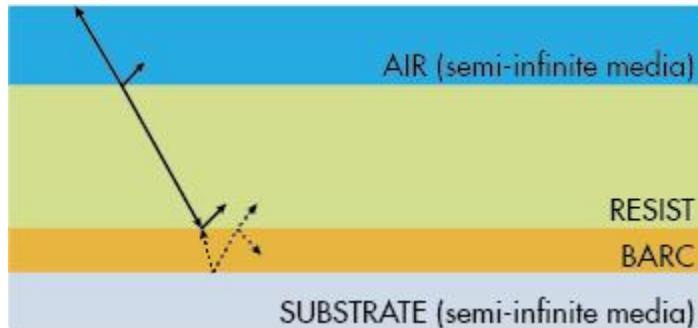
Hexamethyldisilazane (HMDS) – promotes adhesion between oxides (silicon dioxide) and the photoresist (光刻胶). Acting as a primer layer, it is a common primer that is applied before a BARC to improve adhesion and prevent defects, forming a more uniform BARC layer with better reflectivity and pattern resolution in semiconductor manufacturing.

-HMDS reacts readily with silicon oxide surfaces removing adsorbed water and reducing surface silanols, thus preventing future adsorption of water and other polar materials. During this process, small amounts of ammonia are liberated.

-Photoresists wet HMDS-treated surfaces uniformly. It forms a hydrophobic (non-wetting) layer that improves the adhesion of the anti-reflective coating to the substrate.

The *developers and etchants* used in subsequent steps are unable to penetrate the HMDS treated SiO<sub>2</sub>/resist interface. This prevents lifting and minimizes undercutting of the resist.

### **Bottom Antireflective Coatings**

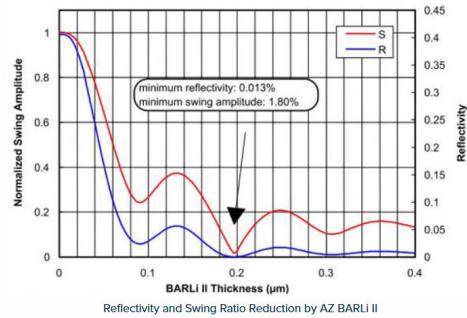


<https://www.brewerscience.com/products/tarc-vs-barc/>

The highly reflective substrate (typically a polished silicon wafer) would cause light from the exposure to bounce back into the photoresist, resulting in phenomena such as standing waves and reflective notching. These defects could ruin the fidelity of the pattern in the photoresist.

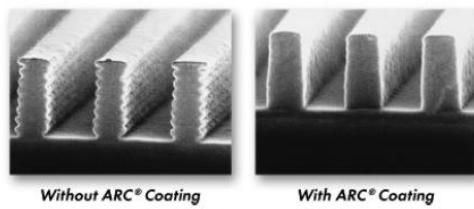
#### AZ® BARLi II™ Series Bottom Anti-Reflective Coatings for i-line

The AZ BARLi II Series products are bottom anti-reflective coating (BARC) materials optimized for i-line (365nm) exposures. At optimum film thickness, BARLi II layers can reduce substrate reflectivity by >98% at 365nm. BARLi II films also greatly improve substrate adhesion (no adhesion promoter necessary) and strip easily in most standard photoresist strippers. AZ EBR 70/30 is recommended for edge bead removal.



## Photoresist BARC

- Bottom anti-reflective coatings (BARC) are polymer based liquid chemistries that have three primary functions in the creation of a semiconductor
  1. The light absorbing chemistry controls light as it passes through the photoresist during the photolithography stage of chip manufacturing. The result is a dampening of reflected light on the surfaces and inside the material (destructive interference)
  2. Reduces or eliminates reflective notching and standing waves
  3. The liquid chemistry typically flattens surface topography



<http://www.brewerscience.com/products/arc>

## S1813 Photoresist (Positive)

-maximum coating uniformity is typically attained between spin speed of 3500-5000 rpm

A positive photoresist is a type of photoresist in which a portion is exposed to light and becomes soluble to the photoresist developer. The unexposed portion of the photoresist remains insoluble in the photoresist developer.

## **MF312 Developer (显影剂)**

-Contains C<sub>4</sub>H<sub>13</sub>NO TMAH, which acts as a strong **alkaline** base., used to etch silicon, but the HMDS layer prevents this developer to etch silicon substrate.

## **Wafer Exposure**

The photoresist is exposed to UV light through a mask, triggering a chemical reaction. For a common positive photoresist, the photo-active compound (PAC), a diazoquinone (DNQ) derivative, undergoes a conversion.

Photochemical reaction: Photo-triggered Wolff rearrangement: When a DNQ molecule is exposed to UV radiation, it absorbs the photons and becomes energized.

**Exposure reaction:** In the exposed regions, UV light converts the hydrophobic, alkali-insoluble DNQ into **hydrophilic, alkali-soluble indene carboxylic acid**.

**Hydrolysis reaction:** DNQ + H<sub>2</sub>O + UV energy → indene carboxylic acid + N<sub>2</sub>.

Water comes from film or air

## **Wet chemical stripping reaction**

-Once the indene carboxylic acid is formed in the exposed areas, the TMAH base in the MF312 developer initiates the dissolution process.

-Acid-base reaction: The TMAH ( $\text{[(CH}_3\text{)}_4\text{N]OH}$ ) reacts with the newly formed indene carboxylic acid. The TMAH's hydroxide ( $\text{OH}^-$ ) ion deprotonates the carboxylic acid.

**Dissolution rate:** The rate at which the exposed resist dissolves is determined by the concentration of TMAH, the degree of exposure, and the development temperature.

## 14. Advanced Lithography

[1] Chapter 10