#### Introduction to BioMEMS & Medical Microdevices

#### Silicon Microfabrication Part 2 – Deposition & Wet Etching

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#### Thin Films

#### Thermal Silicon Oxide

- Amorphous material.
- Insulating layer.
- Mask
- Sacrificial layer.
- Impurities such as Na<sup>+</sup> and K<sup>+</sup> diffuse through it.
- Most dopants except Ga diffuse poorly through it.
- Thicknesses usually less than 1 µm are produced.
- Silicon Dioxide (SiO<sub>2</sub>) by CVD
  - Insulator between conducting layers.
  - Diffusion and ion implantation masks.
  - Sacrificial material.
  - Chemical Vapor Deposition (deposition of a solid on a heated surface from a chemical reaction in the vapor phase.)





- Polysilicon Films (high-purity form of silicon with many silicon atoms)
  - Initially amorphous but may crystallize during deposition.
  - Transition from small grains at the film/substrate interface to columnar crystallites on top.
  - Dopants, impurities and temperature influence crystal orientation.
  - Dopants decrease resistivity to produce conductors and control stress.
  - Can be doped by diffusion, implantation, or by the addition of dopant gases during deposition.
  - Piezoresistive sensor elements may be fabricated from polysilicon.
  - Low Pressure Chemical Vapor Deposition (LPCVD).

### Thin Film Stress Gage...





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#### Silicon Nitride (Si<sub>3</sub>N<sub>4</sub>)

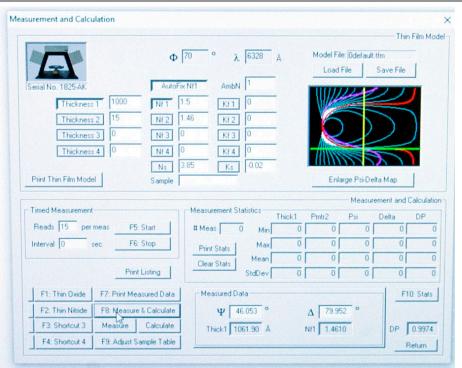
- Amorphous
- Insulator between conducting layers.
- Excellent water and ionic barrier.
- Can not be directly put on silicon.
- May be put on a silicon dioxide layer.
- Highly selective etch rates.
- Hard material that may be used for structural purposes.
- Useful in high temperature applications.
- An important mechanical membrane and isolation/buffer material.
- CVD Techniques

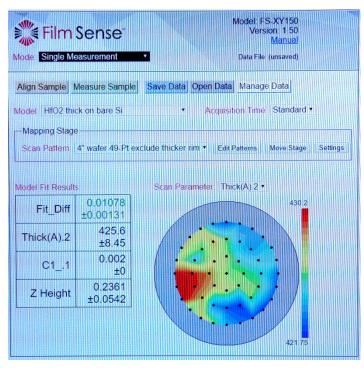
#### Phosphosilicate Glass (PSG)

- PSG and borophosphosilicate glasses (BPSG) soften and flow at lower temperatures, enabling smoothing of topography.
- Etch faster than silicon dioxide, hence more suitable sacrificial layer.
- Dielectric between conducting metal layers.
- Gettering (absorbing) of alkali ions and flow capabilities.
- Passivation coat to provide mechanical protection
- Creating by addition of phosphine to the gas steam.

#### Ellipsometer...







Measurement & Calculation Setup

Thickness in angstroms

Ellipsometers give *non-contact thickness* and *refractive index* measurements of thin transparent and semi-transparent films to *sub-angstrom precision*. It is based on the change of polarization upon reflection or transmission.

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#### Metal Films

- Aluminum and tungsten are commonly used.
- Metals with high reflectivity include Al (aluminum) and Au (gold).
- Metals with high mass density include W (tungsten), Au (gold) and Pt (platinum).
- Metals with specific adsorption and adhesion characteristics include Pd (palladium), Ir (iridium), Au (gold) and Pt (platinum).
- Tungsten will nucleate on silicon or metal surfaces, but not on dielectrics such as oxides and nitrides.
- CVD and PVD (Physical Vapor Deposition) techniques.

# Thin-Film Deposition Processes

#### 1. Chemical Vapor Deposition

 Chemical vapor deposition is the deposition of a solid on a heated surface from a chemical reaction in the vapor phase.

#### 2. Physical Vapor Deposition

- Material is transported in vapor form from a source to a substrate through a vacuum or low-pressure gaseous environment:
  - Evaporation,
  - Sputtering,
  - Arc vapor deposition
  - Laser ablation,
  - Ion plating.

#### 3. Epitaxial Deposition

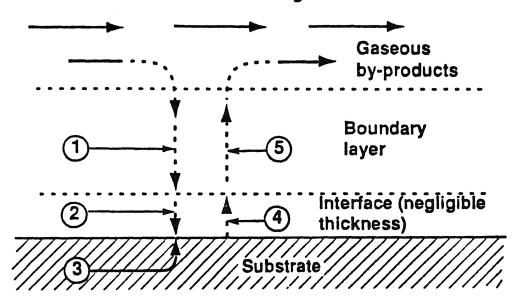
 A single crystal layer can be deposited onto the surface of a substrate wafer.

### Chemical Vapor Deposition (CVD)

- Chemical vapor deposition is the deposition of a solid on a heated surface from a chemical reaction in the vapor phase.
- Like PVD, the deposition species are atoms or molecules or a combination of these.
- Advantages:
  - High throwing power for ease of filling deep recesses, holes and other threedimensional shapes;
  - Deposition is not limited to line-of-sight;
  - Coatings up to several centimeters can be realized;
  - Ultrahigh vacuums are not necessary; and
  - Co-deposition of elements or compounds is achievable
- Disadvantages:
  - Use of temperatures above 600°C,
  - Requirement for chemical precursors with high vapor pressure and toxicity,
  - Toxic by-products.

#### CVD Process...

#### Main flow of reactant gases



- 1. Diffusion in of reactants through boundary layer
- 2. Adsorption of reactants on substrate
- 3. Chemical reaction takes place
- 4. Desorption of adsorbed species
- 5. Diffusion out of by-products

- 1. Reactant gases enter the reactor by forced flow.
- 2. Gases diffuse through the boundary layer.
- 3. Gases come in contact with the surface of the substrate
- 4. Deposition takes place on the surface of the substrate.
- 5. Gaseous by-products are diffused away from the surface

#### Methods...

- CVD Deposition Methods.
  - Atmospheric Pressure Chemical Vapor Deposition (APCVD).
  - Low-Pressure Chemical Vapor Deposition (LPCVD).
  - Plasma-enhanced Chemical Vapor Deposition (PECVD).

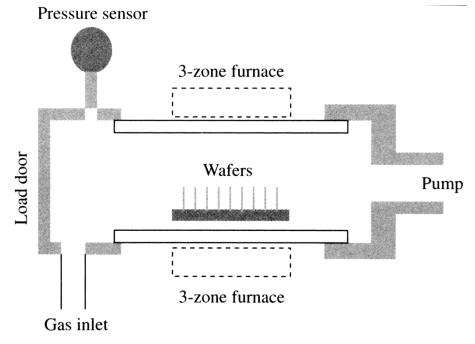
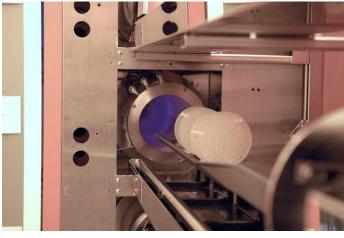


Image courtesy of Gardner JW.

# LPCVD...



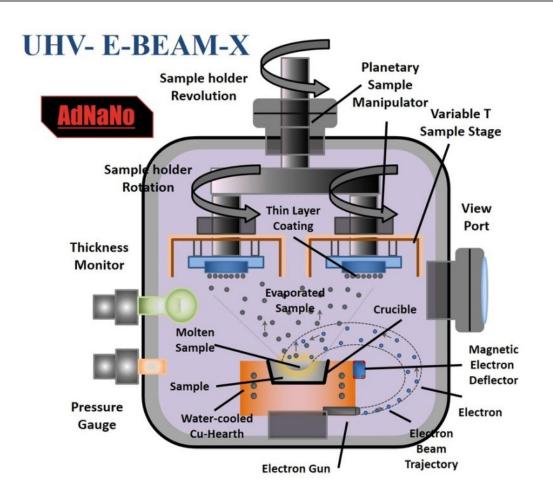




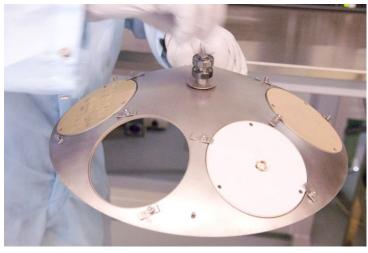
# Loading the LPCVD Machine...



### Physical Vapor Deposition - Thermal Evaporation







CHA E-Beam evaporator.

Planetary wafer holder.

Image courtesy of Adnanotek

### Metals for Evaporation in Graphite Crucibles...







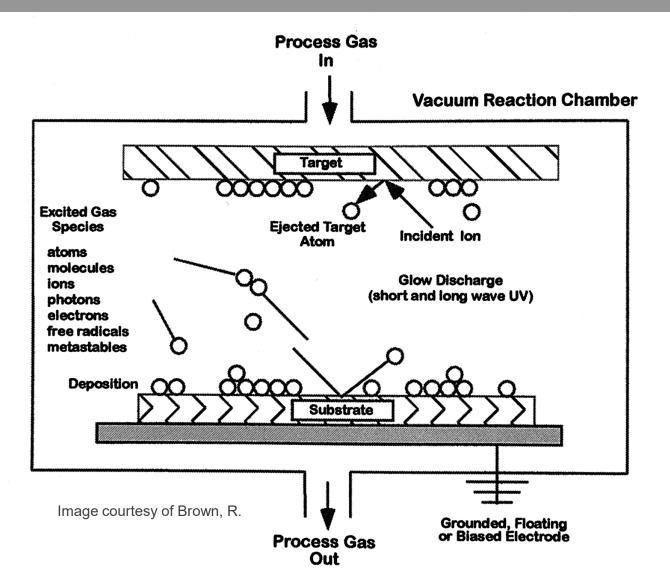






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### Alternatively, Sputter Deposition...



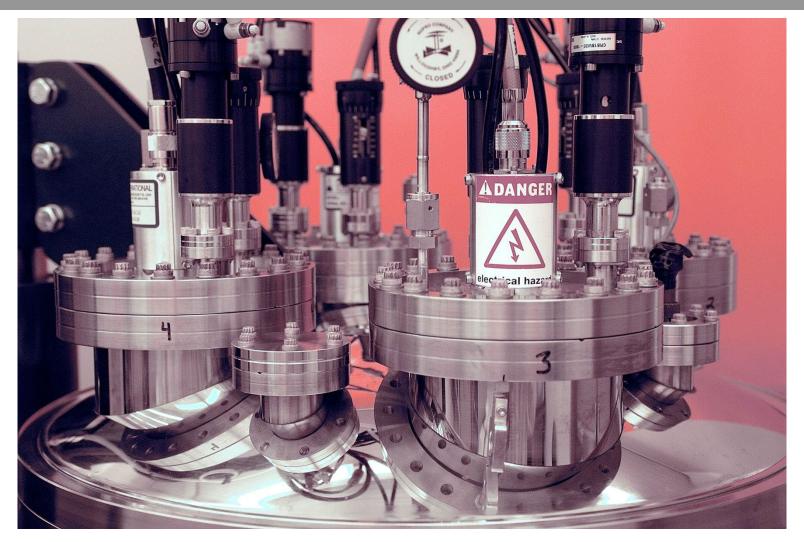


# Sputtering Equipment...











DC metal Sputter System – Aluminum, Titanium and Niobium.



Cluster Sputter System.

# Some Example Select Thin-Films

SiO<sub>2</sub> can be deposited with CVD by reacting silane and oxygen in a LPCVD reactor as shown:

$$SiH_4 + O_2 \rightarrow SiO_2 + 2H_2$$

 SiO<sub>2</sub> can be deposited with LPCVD by decomposing tetra-ethyl-ortho-silicate, Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>, also known as TEOS. This is vaporized from a liquid source.  SiO<sub>2</sub> can also be deposited with LPCVD using dichlorosilane:

$$SiCl_2H_2 + 2H_2O \xrightarrow{900^{\circ}C} SiO_2 + 2H_2 + 2HC1$$

 Si<sub>3</sub>N<sub>4</sub> can be deposited with LPCVD using dichlorosilane and ammonia:

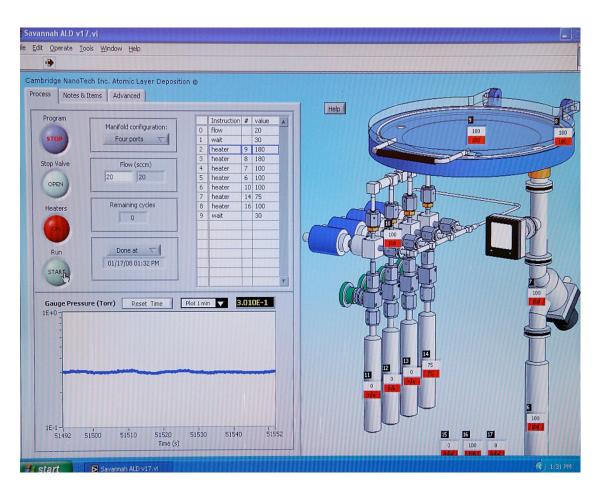
$$3SiCl_2H_2 + 4NH_3 \xrightarrow{800^{\circ}C} Si_3N_4 + 6HCL + 6H_2$$

### CVD Thin-Film Properties...

- There are three factors that control the nature and properties of the deposit:
  - Epitaxy
  - Gas-phase precipitation
  - Thermal expansion
- CVD structure can be controlled by manipulation of temperature, pressure, supersaturation and the CVD reaction.
- Ceramics, including SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Si<sub>3</sub>N<sub>4</sub> and most dielectrics are amorphous.
- Metal deposits tend to be crystalline.

# Atomic Layer Deposition (ALD)





### Materials That Can Be Deposited...

- Oxides: Al<sub>2</sub>O<sub>3</sub>, HfO<sub>2</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, SrTiO<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub>, Gd<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, Ga<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, Co<sub>3</sub>O<sub>4</sub>, ZnO, ZnO:Al, ZnO:B, In<sub>2</sub>O<sub>3</sub>:H, WO<sub>3</sub>, MoO<sub>3</sub>, Nb<sub>2</sub>O<sub>5</sub>, NiO, MgO, RuO<sub>2</sub>
- Fluorides: MgF<sub>2</sub>, AlF<sub>3</sub>
- Organic-hybrid materials: Alucone
- Nitrides: TiN, TaN, Si<sub>3</sub>N<sub>4</sub>, AlN, GaN, WN,
   HfN, NbN, GdN, VN, ZrN
- Metals: Pt, Ru, Pd, Ni, W
- Sulfides: ZnS

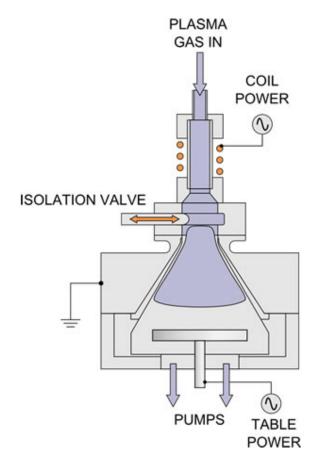


Image courtesy of Oxford industries

#### Select ALD Features...

- 100% film density guarantees ideal material properties.
- Insensitive to dust (grows underneath dust!).
- Oxides, nitrides, metals, semiconductors possible (Cambridge Nanotech provides standard recipes).
- Amorphous or crystalline depending on substrate and temperature.



Steven S. Saliterman Cambridge NanoTech Inc.

#### ALD Features...

- Digital thickness control to atomic level (no rate monitor needed, just set the number of atomic layers).
- Perfect 3D conformality, 100% step coverage: uniform coatings on flat, inside porous and around particle samples.
- Low defect density.
- Gentle deposition process for sensitive substrates, no plasma.
- Low temperature deposition possible (RT-400C).
- Low stress because of molecular self assembly.

Cambridge NanoTech Inc.

# Ion Implantation

- In ion implantation, the dopant element is ionized, accelerated to a kinetic energy of several hundred keV, and then driven into the substrate.
- The electrical conductivity of an intrinsic semiconductor can be increased through doping. The charge carrier density can be increased through impurities of either higher or lower valence.
- Doping can be used to control etching by reducing etch rates.
- Sources for *n*-type doping include antimony, arsenic and phosphorous; and for *p*-type doping, boron.

# Controlled Etching with Chemicals

- Anisotropic etchants (potassium hydroxide, ethylene diamine pyrochatechol, and tetramethyl ammonium hydroxide):
  - Etch silicon preferentially along preferred crystallographic directions.
  - Show a reduction of etch rate in heavily doped p-type regions.
- Boron typically is incorporated using ion implantation for this purpose.

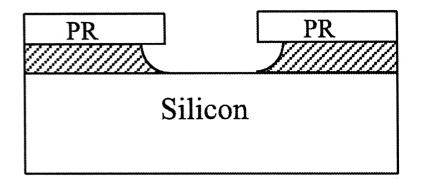
# Wet Bulk Surface Micromachining

- In wet bulk micromachining features are sculptured in bulk materials like silicon, quartz, sapphire, ceramics, SiC, GaAs, InP and Ge by orientation independent (isotropic) or orientation-dependent (anisotropic) wet etchants.
- Integrated circuits typically have aspect ratios of 1-2, while in BioMEMS the ratio may be up to 400.

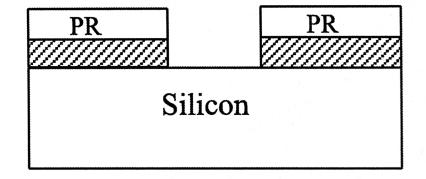
### Recall Silicon Crystal Orientation...

- Orientation is important because of different characteristics and etching speeds in different planes and resulting etching angles.
  - The (111) plane has the highest atom packing density and is relatively non-etching compared to the others.
  - The angles between planes, {100} and {110} are 45 or 90 degrees, between {100} and {111} are 54.74 degrees, and between {111} and {110} planes 35.26, 90 or 144.74 degrees.

### Isotropic vs. Anisotropic Etching...

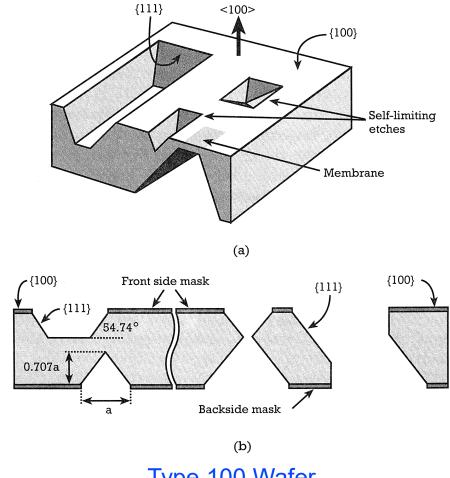


**Isotropic Etching** 

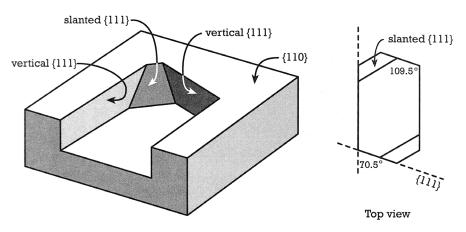


**Anisotropic Etching** 

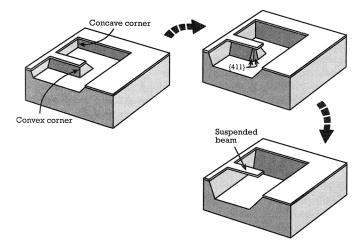
### Geometric Shapes...



Type 100 Wafer



Type 110 Wafer



Images courtesy of Madou M,

### Isotropic Etching Agents...

 Isotropic etchants are usually acidic, and lead to rounded features (HNA = HF (hydrofluoric acid) + HNO<sub>3</sub> (nitric acid) + CH<sub>3</sub>COOH (acetic acid)):

$$Si + HNO_3 + 6HF \rightarrow H_2SiF_6 + HNO_2 + H_2O + H_2$$
  
(H<sub>2</sub>SiF<sub>6</sub> is water soluble)

### Anisotropic Etching Agents...

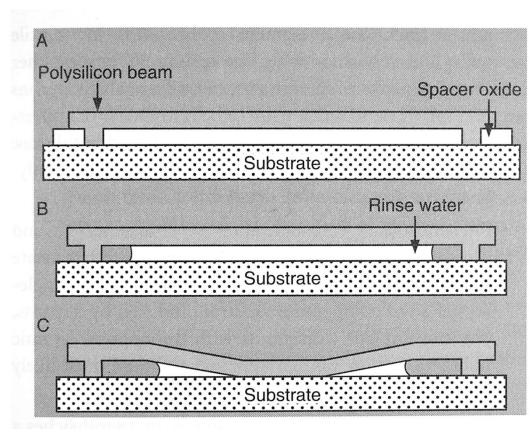
Anisotropically etchants are alkaline:

$$Si + 2OH^{-} \rightarrow Si(OH)_{2}^{2+} + 4e^{-}$$
 $4H_{2}O + 4e^{-} \rightarrow 4OH^{-} + 2H_{2}$ 
 $Si(OH)_{2}^{2+} + 4OH^{-} \rightarrow SiO_{2}(OH)_{2}^{2-} + 2H_{2}O$ 
 $\therefore Si + 2OH^{-} + 2H_{2}O \rightarrow Si(OH)_{2}^{2+} + 2H_{2}$ 

### Structural Element/Sacrificial Layer/Etchant

- Common structural elements include polysilicon, polyimide, silicon nitride and tungsten.
  - The polysilicon and its sacrificial layer, silicon dioxide can be applied by LPCVD. Silicon dioxide can be etched away with hydrofluoric acid (HF) solution without etching the polysilicon.
  - Polyimide can be used with aluminum as the sacrificial layer, the latter being dissolvable with acid-based etchants.
  - Silicon nitride is both a good structural material and electrical insulator. Polysilicon can be used as the sacrificial layer, in which case KOH and EDP can be used as the etchants.
  - Tungsten can be applied by CVD over silicon dioxide, and again HF is a suitable etchant to remove the silicon dioxide sacrificial layer.

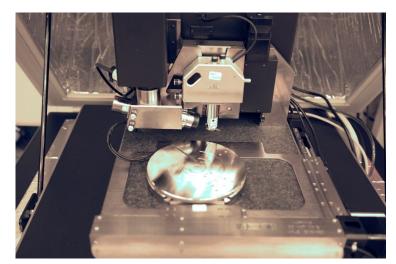
# Stiction and Critical Point Dryer

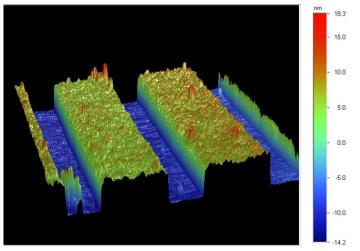


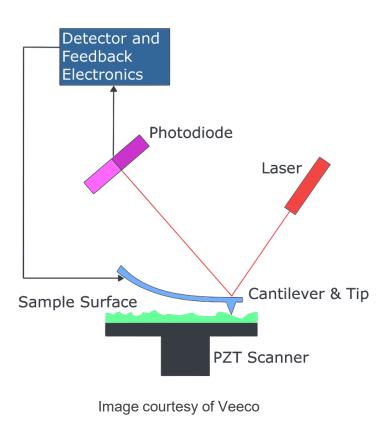




# **Atomic Force Microscopy**











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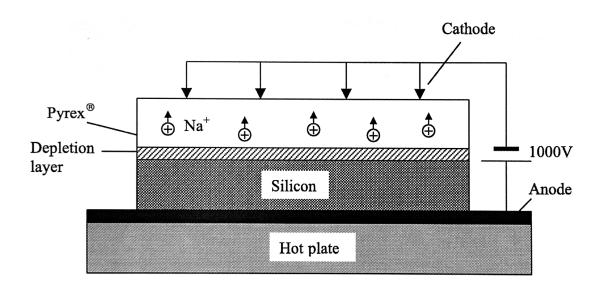
# Substrate Bonding

- 1. Silicon Direct Bonding (or Fusion Bonding)
  - Silicon to Silicon
  - Silicon on Insulator (SOI)
  - No intermediate layers
  - Requires heating (450°)
- 2. Anodic Bonding
  - Silicon to Glass (Pyrex 7740)
  - Intermediate Adhesive Layers
- 3. Lasers

# Wafer Bonding



Wafer bonder - anodic or direct (fusion).



# Summary

- Important thin-films include:
  - Thermal Silicon Oxide
  - Silicon Dioxide (SiO<sub>2</sub>)
  - Polysilicon
  - Silicon Nitride (Si<sub>3</sub>N<sub>4</sub>)
  - Phosphosilicate Glass (PSG)
  - Metal films (e.g., tungsten and aluminum)
- Thin films may be produced by:
  - Thermal oxidation
  - Physical Vapor Deposition
  - Chemical Vapor Deposition
  - Epitaxial Deposition
  - Atomic Layer Deposition (ADL)

- Ion implantation may be used to improve electrical conductivity or to control etching characteristics.
- Wet bulk micromachining
  - Isotropic and anisotropic etching
  - 3-D structure and sacrificial layers
- Characterization with Atomic Force Microscopy
- Wafer bonding
  - Silicon direct (or fusion) bonding
  - Anodic bonding