EE-527: MicroFabrication

De-Ionized Water
Clean Water as a Processing Chemical

• Water (H₂O) is the most prevalently used material in microfabrication processes, and is used mainly for rinsing and cleaning of wafers.
• Approximately 6000 gallons of de-ionized (DI) water are required for each 6” CMOS wafer.
• DI water must be manufactured on site to achieve the quality and purity levels required by modern microfabrication.
• Each gallon of DI water may require as much as 4-6 gallons of raw city grade water to manufacture.
• DI water must be continuously recirculated in order to achieve the quality and purity levels.
Size Ranges of Suspended Particles

- Macro Particle Range
  - > 25 µm, visible to the naked eye

- Micro Particle Range
  - 1 - 15 µm, visible with an optical microscope

- Macro Molecular Range
  - 0.1 - 1 µm, visible with a high power optical microscope

- Molecular Range
  - 1 - 100 nm, visible with a scanning electron microscope

- Ionic Range
  - 0.1 - 1 nm, not visible with current technology
Size Range of Common Particles

- Atomic Radii: 1-6 Angstroms
- Metal Ions: 2-7 Angstroms
- Aqueous Salts: 2-20 Angstroms
- Sugars: 7-25 Angstroms
- Pyrogens: 20-250 Angstroms
- Collodial Silica: 6-250 nm
- Albumin Protein: 8-100 nm
- Viruses: 9-100 nm
- Carbon Black: 12-100 nm
- Tobacco Smoke: 20-1000 nm
- Paint Pigment: 0.1-5 μm
- Bacteria: 0.25-30 μm
- Lung Damaging Dust: 0.5-35 μm
- Coal Dust: 1.0-100 μm
- Milled Flour: 1.0-100 μm
- Yeast Cells: 2.0-50 μm
- Red Blood Cells: 5.0-9.0 μm
- Pollens: 10-100 μm
- Human Hair (Diameter): 25-200 μm
- Mist: 70-200 μm
- Beach Sand: 100-10,000 μm
Types of Water Filtration

– Particle Filtration
  • 1-75 \(\mu\text{m}\) cartridge filters: cellulose, fiberglass, or polypropylene fibers

– Microfiltration
  • 0.1-1.0 \(\mu\text{m}\) cartridge filters: ceramic or polymer membranes

– Ultrafiltration
  • 20-2000 Angstroms: chemically based

– Reverse Osmosis (Hyperfiltration)
  • 1-200 Angstroms: uses special membrane and high pressure to overcome osmotic pressure
Osmotic Pressure

Semipermeable membrane allows water to pass, but not dissolved ions.

Water flows in a direction so as to aid diffusion and dilute itself as much as possible. This produces the osmotic pressure.
Reverse Osmosis (RO)

- By applying a pressure greater than the osmotic pressure, water can be driven backwards through the membrane for purification (reverse osmosis process).

- Terminology:
  - TDS = total dissolved solids
  - “product” = pure water produced by RO
  - “concentrate” = rejected water which contains concentrated dissolved material
  - “recovery” = ratio of product to input water, typically about 75 %
  - “rejection” = fraction of TDS that is retained by the RO filter membrane, typically about 90-95 %

- Rejection and recovery are optimized by RO operating pressure and the type of RO membrane.
  - Higher pressures increase recovery, but decrease rejection.
Reverse Osmosis (RO) System

- Pure \( \text{H}_2\text{O} \)
- \( \text{H}_2\text{O} + \) dissolved ions

- Input supply water
- High pressure pump (125-175 psi, typ.)
- Adjustable pressure building valve
- Concentrate
- Product
Ultraviolet Sterilizers

- Standard is 25 mW-sec @ 253.7 nm; strongest Hg arc line.
- This bursts the cell membranes of bacteria and renders their DNA incapable of reproduction.
- Exploded remains of bacteria are called “pyrogens” and must be collected by a 0.2 µm filter downstream from the sterilizer.
Ion Exchange Columns

Based upon ion exchange resins:

- Cation exchangers: $\text{H}^+ \leftrightarrow \text{Na}^+, \text{K}^+, \text{Ca}^{++}$, etc.
- Anion exchangers: $\text{OH}^- \leftrightarrow \text{Cl}^-, \text{Br}^-, \text{F}^-$, etc.
- Both cation and anion exchange resins are built into a coaxial cartridge assembly. Exchanged $\text{H}^+$ and $\text{OH}^-$ ions combine to form $\text{H}_2\text{O}$. 

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Features of DI water systems - 1

- Water must flow continuously through recirculation loops at approximately 10 linear feet per minute to keep bacteria from forming on walls.
- Dead-legs must be avoided in plumbing-- stagnant regions will provide culture zone for bacterial growth.
- Distance from loop to taps must be minimized.
- All piping must be PVDF (teflon) with fusion welded joints.
- No metal parts must be in contact with loop, except for a few stainless steel parts around the UV sterilization units which are unavoidable.
- Let DI faucets run until resistivity monitor reads > 18 MΩ-cm.
- High usage during day may require make-up system to run overnight to replenish supply in recirculation tank.
- If system “goes green” it must be sterilized with H₂O₂ and then restarted. H₂O₂ is reduced to H₂O and O₂ upon passing through UV sterilizer. (Turn the UV unit off for system sterilization.)
Features of DI water systems - 2

• Because of its zero ion and mineral content, DI water is a powerful leaching agent for metals. Metals in contact with DI water are often eroded in very short periods of time. Metal pump impellers last only a few months if they are not teflon coated.

• Molecular chlorine (Cl₂) and chloride ion (Cl⁻) are removed differently from incoming water.
  – Cl⁻ is removed via the ion exchange columns
  – Cl₂ is removed via an activated carbon filter
  – Cl₂ must be removed prior to the RO, or it will destroy the membrane

• Final polishing is performed with 0.2 µm filters.

• Breach of an upstream filter often sends particles and debris down stream in sufficient quantity to damage subsequent filters.

• Change cartridge filters when the pressure difference across them exceeds more than about 10 psi.
## Integrated Circuit Processing Requirements

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<tbody>
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<td>125</td>
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<td>10</td>
<td>1</td>
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<td>environment</td>
<td>0.1 mini environment</td>
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<td>Utility Purity (ppb)</td>
<td>1,000</td>
<td>500</td>
<td>100</td>
<td>50</td>
<td>5</td>
<td>1</td>
<td>0.1</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Data from Chang and Sze, *ULSI Technology*. 

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Water Purity Measures

- Resistivity ($\rho$), measured in M$\Omega$-cm
  - Perfectly pure water at 20°C has $\rho = 18.2$ M$\Omega$-cm
  - Perfectly pure water at 20°C has pH = 7.00
- Total Organic Carbon (TOC), measured in ppm
- Total Dissolved Oxygen (TDO), measured in ppm
  - Pure water in equilibrium with air has TDO of around 20 ppm, depending upon agitation conditions
- Total Heavy Metals (THM), measured in ppm
- Bacteria, measured in colony forming units (CFU) per liter per day
ASTM D-1193-91 Type 1 Reagent Water

- Specifications:
  - $\rho > 18$ M$\Omega$-cm
  - TOC $< 100$ ppb
  - $Na^+ < 1$ ppb
  - $Cl^- < 1$ ppb
  - Silica $< 3$ ppb

- Basic de-ionized water available at taps in EE MicroFab Lab.

- De-ionization is different from distillation!!
  - De-ionization removes charged ionic species, not neutral molecules.
  - Distillation removes molecules of differing vapor pressure, such as alcohols and other solvents, but does not de-ionize.
  - Distillation is more commonly used in biological applications.
SEMI recommended DRAM1 pure water guidelines

- $\rho > 18.2 \text{ M\(\Omega\)-cm}
- TOC < 5 ppb
- THM < 1 ppb
- TDO < 20 ppm
- < 200 particles 0.3-0.5 \(\mu\)m/L
- < 1 particle > 0.5 \(\mu\)m/L

<table>
<thead>
<tr>
<th>Max ions (ppb)</th>
<th>Max metals (ppb)</th>
</tr>
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<tbody>
<tr>
<td>Na(^+)</td>
<td>0.05 Li</td>
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<tr>
<td>K(^+)</td>
<td>0.1 Na</td>
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<tr>
<td>Cl(^-)</td>
<td>0.05 K</td>
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<tr>
<td>Br(^-)</td>
<td>0.1 Mg</td>
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<tr>
<td>NO(_3)(^-)</td>
<td>0.1 Ca</td>
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<tr>
<td>SO(_4)(^-)</td>
<td>0.1 Sr</td>
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<tr>
<td>total</td>
<td>0.5 Ba</td>
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Semiconductor Equipment and Materials International (SEMI) is a standards organization for the microelectronics industry