Chapter 1.10

**Miscellaneous Etchants**

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Aluminum Etchant Type A (Transene Co., Inc.)
  ► For VLSI aluminum etching, there is available a pre-mixed phosphoric/acetic acid mixture.
    Etch rate: ~ 100 Å/sec at 50°C.
  ► Corrosive. Avoid contact with eyes, skin and clothing. Avoid inhalation.

Aluminum Etchant for VLSI
  ► Etch rate ~ 2000 Å/min.
    16 parts phosphoric acid
    2 parts DI water
    1 part acetic acid
    1 part nitric acid

Aluminum Etchants - Others
  ► These will not etch gold, etc.
  ► Phosphoric acid at 60°C
  ► Sodium hydroxide (10% solution)
Miscellaneous Etchants

► Trisodium phosphate at 190°C
► These will not etch ZnO. Etch rate ~ 100 Å/sec.
► 10 g K₃Fe(CN)₆
► 1 g Potassium hydroxide (KOH) in 100 ml water at room temperature.

Antimony Etchant
► Etch (off of silicon): HNO₃
  H₂O : HCl : HNO₃ (1:1:1)
  H₂O : HF : HNO₃ (90:1:10)

Aqua Regia
► HCl : HNO₃ (3:1)
► Evaporation - removal:
  50% DI water
  45% HCl
  5% CuSO₄
► Dissolves gold.
► Never store in a tightly sealed container!

Bismuth Etchant
► 5 ml Sulfuric acid
  5 ml Hydrogen peroxide
  90 ml DI water
  No heat necessary. Etches quickly.
► H₂O : HCl (10:1)

Brass
► Use brass dip (Turco) for etching and cleaning.
► Ferric chloride (etch)
► Ammonium persulfate: 20 g to 100 ml H₂O

Cadmium Sulfide Etchant (CdS)
► Dislocation pits on the (0001). Distinguishes between A and B faces.
  HNO₃ : CH₃COOH : H₂O (6:6:1)

Cadmium Telluride (CdTe)
► Polishes
  10 ml HNO₃
  20 ml H₂O
  4 g K₂Cr₂O₇
► Pits
  5% Br₂ in methanol
  5 mg AgNO₃

Chromium Etchant
► HCl : H₂O₂ (3:1) - This will also etch gold film.
► HCl : H₂O (1:1) - Heat to 50°C, immerse substrate and touch with aluminum wire.

Chromium/Nichrome Etchant
► HCl : H₂O₂ (3:1) - This will also etch gold film.
► HCl and touch with aluminum wire.

Cobalt
► H₂O : HNO₃ (1:1)
► HCl : H₂O₂ (3:1)
Columbium

- HF : HNO₃ (1:1)

Copper

- Brass Dip, RT-2 Resist Stripper, FeCl solutions
- H₂O : HNO₃ (1:5)
- Oxide removal - cold solution of ammonium carbonate (slight etch)

Dislocation Etchants

- **Sirtl Etchant**
  
  1 part conc. HF or 50 g CrO₃ in 100 ml H₂O  
  1 part CrO₃ (5 M)  
  1:1 = HF : CrO₃ solution  
  500 g/L of solution

  Etch rate ~ 3.5 µm/min. Good on {111}, poor on {100}, faceted pits.

- **Secco Etchant**
  
  2 parts conc. HF  
  1 part K₂Cr₂O₇ (0.15 M)  
  44 g/L of solution

  Etch rate ~ 1.5 µm/min. Best with ultrasonic agitation. Good on all orientations. Non-crystallographic pits.

- **Wright-Jenkins Etchant**
  
  2 parts conc. HF  
  2 parts conc. acetic acid  
  1 part conc. nitric acid  
  1 part CrO₃ (4M)  
  400 g/L of solution  
  2 part Cu(NO₃)₂ + 3 H₂O (0.14 M)  
  33 g/L of solution

  Etch rate ~ 1.7 µm/min. Ultrasonic agitation not required. Good on all orientations. Faceted pits, good shelf life.

- **ASTM Dislocation Etchant**
  
  600 ml HF  
  30 ml HNO₃  
  0.2 ml Br₂  
  28 g Cu(NO₃)₂ + 3 H₂O  
  Dilute 1:10 with H₂O

Gallium Arsenide

- 1-2% Br₂ in ethanol  
  H₂SO₄ : H₂O₂ : H₂O (5:1:1)

  Good polishing etches

- Fused KOH at 300°C

  Good crystallographic dislocation pits on the (100) surfaces

- 1 ml HF  
  2 ml H₂O  
  8 mg AgNO₃  
  1 g CrO₃

  Dislocation lines and striations
Gallium Phosphide

- Behaves similarly to GaAs and the above etches may be used.
- HF : Acetic Acid : Saturated KMnO₄ sol’n (1:1:1)
  Good striations, free from pits on (110) surfaces

Germanium Etchant (and Germanium-Silicon)

- H₂O₂ (30%) at 90°C
  Etch rates:
  100% Ge 4000 Å/min
  80% Ge 1000 Å/min
  60% Ge and less do not etch

- H₂O at 90°C
  Etch rates:
  100% Ge ~ 200 Å/min
  < 60% Ge does not etch

- RCA SC-1 (NH₄OH : H₂O₂ : H₂O) at 75°C
  Etch rates:
  100% Ge ~ 4 um/min
  80% Ge ~ 9000 Å/min
  60% Ge ~ 500 Å/min
  40% Ge ~ 30 Å/min
  20% Ge ~ 10 Å/min
  0% Ge ~ 5 Å/min

Gold

- Aqua Regia: HCl : HNO₃ (3:1)
- Saturated solution of KI in H₂O, 1 iodine crystal

Indium

- Reacts with acids (HCl)
- Slow etch (1000 Å/min.)
  HNO₃ : H₂O (1:1)
  Hot HCl : HNO₃ (3:1)

Indium Antimonide

- HNO₃ : HF : Acetic Acid (5:3:3)
  Polishes rapidly as it does most semiconductors, but bubble formation can ruin the polish.

- 0.2N solution of FeCl₃ in HCl
  Develops pits.

- HF : Acetic Acid : 2N HMnO₄ (1:1:1)
  Good pit-free striations of (211) surfaces

Indium Phosphide

- Cut on diamond saw using slow feed. Lap using 5u powder. Degrease in acetone, then methanol. Chemical etch using 5% bromine by weight for about 2 minutes using a swirling motion. Rinse in methanol, DI water, N₂ dry.

Indium Tin Oxide (ITO)

In order to etch ITO it is needed to reduce it to a metallic state. The reactions are:

Zn + HCl = H₂ + ZnCl₂
H₂ reduces ITO
SnO₂ + H₂ = Sn or SnOₓ with x smaller than 1
Sn + HCl = H₂ + SnCl₄ which is soluble

► The procedure:
conc. HCl: H₂O=1:1 at 50ºC.

Add a small amount of Zn powder (on edge of a spatula). Put the wafer in the solution for about 1 min. Watch for turbidity of the ITO. Transfer the wafer to another beaker containing conc. HCl (no dilution), for about 1 min.

Take the wafer out and check if all the film was etched. Return to first solution if needed, at 50 degrees.

► HCl:HNO₃ (3:1)

Iron Etchant

► H₂SO₄ : H₂O (1:1)
HCl : H₂O (1:1)
HNO₃ : H₂O (1:1)

► To remove rust: saturated oxalic acid solution.

Kovar

► Cleaner:
Ferric ammonium sulfate 50 g
H₂SO₄ 125 ml
HCl 150 ml

Heat to 60-80ºC

► Electrolysis:
HCl and salt, alternating voltage. Kovar or carbon electrode
10% solution of HCl and a handful of salt

Lead

► Acetic : H₂O (1:1)
► Lead deposited on glass can be removed with dilute HNO₃.

Lucite

► Softens with acetone
► Acetone : formaldehyde

Magnesium

► Hot H₂O : NaOH (10:1 by weight)
Follow with H₂O : CrO₃ (5:1 by weight)

Magnesium Fluoride

► Dissolves (sometimes) in hot commercial ferric chloride.

Mercury

► Dissolves and reacts in HNO₃.
► To clean (purify), bubble air through mercury, filter and vacuum distill.

Molybdenum (Moly)

► Hot concentrated H₂SO₄
► Aqua Regia
► HCl : H₂O₂ (1:1) (etches stainless steel)
Electrolysis 15 V ac moly or carbon electrode in pure H₂SO₄
- Dissolves in H₂O : HNO₃ : H₂SO₄ (1:1:1) cold
- 45% formic acid : 45% H₂O₂ : 10% H₂
  Heat 2 min at 80°C.

**Monel**
- Clean with 50% HNO₃ : salt. Wash with water, then dip in 50% solution HNO₃, then rinse in water, then dip in ammonium hydroxide and dry.

**Nichrome**
- HCl : copper chloride (1:1)
- Ce(SO₄)₂ 7.9 g
  Water 130 ml
  Add:
  35 ml HNO₃

**Nichrome Etchant** (Transene Co., Inc.)
- Contains nitric acid. Slightly irritating to skin. Wash area thoroughly if contacted.

**Nickel**
- HF : HNO₃ (1:1)
- Electrolysis:
  dc nickel electrode. H₂SO₄ or H₃PO₄. Reverse polarity several times, finish with nickel part as electrode.

**Nickel Etchant** (Transene Co., Inc.)
- Contains nitric acid. Highly irritating to eyes, skin and mucous membranes, avoid inhalation of vapors.
- Avoid contact with reducing agents.

**Nickel Oxides**
- HCl

**Niobium**
- HF : HNO₃ (1:1)

**P-Etchant** (**Phospho-Silicate Glass [PSG] Etchant**)
- 3 parts HF
  2 parts HNO₃
  60 parts DI water

**Palladium**
- HCl : HNO₃ (3:1) Hot

**Picein Wax**
- Withstands all acids (including HF)
- Thin/dissolve in trichloroethylene (TCE)

**Piranha**
- Excellent oxidant; removes most organic residues.
  5 parts H₂SO₄
  1 part H₂O₂
- **Note:** Always add peroxide to sulfuric acid, never vice versa! This is a self-heating solution.
**Platinum**

- Dissolves in Aqua Regia
  
  \[ \text{HCl : HNO}_3 \ (3:1) \ 85^\circ\text{C} \]

**Polish - Fairchild's "Magic Polish"**

- A - 2.5 g I\(_2\) in 1100 ml acetic acid
- B - HNO\(_3\) : HF (3:1)
  
  Add A to B (1:1) just prior to use.

**Polysilicon Etchant** (See also Silicon Etchant)

- 64% HNO\(_3\) / 33% H\(_2\)O / 3% NH\(_4\)F
- 189 ml HNO\(_3\) / 96 ml H\(_2\)O / 7.5 ml NH\(_4\)F

**Preferential Etch** (See Dislocation Etchant Wright-Jenkins Etchant)

**Rhodium**

- HCl : HNO\(_3\) (3:1) Hot

**Ruthenium**

- HCl : HNO\(_3\) (3:1) Hot

**Silicon Etchant - Polycrystalline Silicon (Bell Labs)**

- This solution is mixed and bottled by Microlab staff. Bottles are stored in the tall white acid cabinet next to sink 432C (old lab).
  
  - Etch rate ~ 100 Å/sec
  
  - 33% DI water / 3% NH\(_4\)F / 64% HNO\(_3\)
  
  - Bottle content:
    
    - 960 ml DI water
    - 75 ml NH\(_4\)F (ammonium fluoride)
    - 1890 ml HNO\(_3\) (nitric acid)

**Big Batch Silicon Etch** (staff only)

- Big batch silicon etch is used by staff to rework Tylan dummies in the heated bath, left side of sink 7.
- 48% DI water / 48% HNO\(_3\) / 2% HF at 50ºC

**Silicon Etchants - Single-Crystal (Sensors)**

- **EDP Etchant for Single Crystal Silicon**
  
  EDP etchant can be used on p-type wafers with <100> orientation, masked with either silicon dioxide or silicon nitride. It leaves a cleaner, smoother silicon surface with partial etch than KOH (see below). Heavy boron doping acts as an etch stop for EDP. Since EDP does not etch oxide, it is important to remember to dip off any native oxide from the silicon surfaces to be etched in HF solution. Etch rates and temperatures are given below. Complete instructions on the use of EDP are given in Chapter 1.3 of the lab manual.
  
  - Ethylenediamine \( \text{N H}_2\text{O(C H}_2\text{)}_2\text{N H}_2 \) 1 mole = 50.10 g
  - Pyrocatechol \( \text{C}_9\text{H}_4\text{(O H}_2\text{)} \) 1 mole = 109.1 g
  - Water \( \text{H}_2\text{O} \) 1 mole = 18.02 g

  
  - Ethylenediamine 500 ml 35.1 mole%
  - Pyrocatechol 88 g 3.7 mole%
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Water 234 ml 61.2 mole%
Etch temp: 110°C
Etch Rate ratio: <100>:<110>:<111> 50:30:3
Initial Etch Rate: 28 µm/hr
Oxygen Exposure: up to > 50 µm/hr
Mask Resistance: SiO₂ 200 Å/hr

This is the earliest reported EDP (or EPW) composition. It is generally used in the temperature range 100-118°C. At lower temperatures it develops insoluble residues. This composition, as well as other uncatalyzed EDP compositions, tends to 'age' rapidly with exposure to oxygen. The etch rate increases with time to 50 µm/hr and higher.

The addition of pyrazine increases the <100> etch rate while making it less sensitive to oxygen exposure. Pyrazine has a very small effect on the <111> etch rate so the <100>/<111> ratio increases with pyrazine content. The selectivity to boron content is reported by Reisman et al. to be similar for the F & K, B, and S etches. Also, the smoothness of the etching surface is improved by the addition of 0-6 g/L pyrazine. 8 g/ has shown some unevenness, <111> pyramids form, possibly due to the very high <100>/<111> etching ratio. IBM recommends 4 g of pyrazine to every liter of ethylene diamine for a smooth surface.


<table>
<thead>
<tr>
<th>Ethylenediamine</th>
<th>500 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrocatechol</td>
<td>80 g</td>
</tr>
<tr>
<td>Water</td>
<td>160 ml</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>100-118°C</td>
</tr>
<tr>
<td>Boiling Point:</td>
<td>18°C</td>
</tr>
</tbody>
</table>

<100> Etch Rate (with pyrazine added):

<table>
<thead>
<tr>
<th>Pyrazine per 500 ml Ethylenediamine</th>
<th>0 g</th>
<th>1.0 g</th>
<th>3.6 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>at 100°C</td>
<td>14 µm/hr</td>
<td>42 µm/hr</td>
<td>50 µm/hr</td>
</tr>
<tr>
<td>at 115°C</td>
<td>26 µm/hr</td>
<td>65 µm/hr</td>
<td>75 µm/hr</td>
</tr>
</tbody>
</table>

► Mask Resistance:
  
  SiO₂ 150 Å/hr
  Si₃N₄ 80 Å/hr

This composition with or without pyrazine provides residue-free etching above 100°C.


<table>
<thead>
<tr>
<th>Ethylenediamine</th>
<th>500 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pyrocatechol</td>
<td>160 g</td>
</tr>
<tr>
<td>Water</td>
<td>160 ml</td>
</tr>
<tr>
<td>Temperature Range:</td>
<td>100-118°C</td>
</tr>
</tbody>
</table>

<100> Etch Rate (with pyrazine added):

<table>
<thead>
<tr>
<th>Pyrazine per 500 ml Ethylenediamine</th>
<th>0 g</th>
<th>1.0 g</th>
<th>3.0 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>at 115°C</td>
<td>27 µm/hr</td>
<td>68 µm/hr</td>
<td>81 µm/hr</td>
</tr>
</tbody>
</table>

**Miscellaneous Etchants**

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Ethylene diamine 500 ml  
Pyrocatechol 80 g  
Water 66 ml  
Temperature Range: 50-115ºC

<100> Etch Rate (with pyrazine added):

<table>
<thead>
<tr>
<th>Pyrazine per 500 ml Ethylene diamine</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>at 50ºC</td>
<td>4.5 µm/hr</td>
</tr>
<tr>
<td>at 75ºC</td>
<td>13 µm/hr</td>
</tr>
<tr>
<td>at 95ºC</td>
<td>26 µm/hr</td>
</tr>
<tr>
<td>at 105ºC</td>
<td>34 µm/hr</td>
</tr>
<tr>
<td>at 115ºC</td>
<td>45 µm/hr</td>
</tr>
</tbody>
</table>

► **"M" (Medium) Etchant** - Based on A. Reisman et al., as above.

This etch is useful for etching below the boiling point in order to minimize agitation of the wafer. It etches at a rate between the "F" and "S" etches (hence "M" for medium). This etch prevents the formation of residues by the "F" etch by slowing oxidation of the surface through the reduction of the water content.

<table>
<thead>
<tr>
<th>Ethylene diamine 500 ml</th>
<th>Pyrocatechol 160 g</th>
<th>Water 125 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature Range:</td>
<td>105ºC</td>
<td></td>
</tr>
</tbody>
</table>

<100> Etch Rate (with pyrazine added):

<table>
<thead>
<tr>
<th>Pyrazine per 500 ml Ethylene diamine</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>at 115ºC</td>
<td>63 µm/hr</td>
</tr>
</tbody>
</table>

Other references:

► **KOH Etchant for Single Crystal Silicon**

KOH is a strongly anisotropic etch, preferring the <100> crystal plane. (The differential etch rate at 80ºC is on the order of 400:1.) Lines of rectangular areas to be etched must be parallel or perpendicular to the wafer flat. It is possible to etch around rectangular geometries, i.e., leave islands of silicon, if the proper convex corner compensation is used to prevent rounding off of the corner due to undercutting along the <411> plane.

<table>
<thead>
<tr>
<th>KOH: 750 g</th>
<th>Water: 1500 ml H₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature:</td>
<td>80ºC</td>
</tr>
<tr>
<td>Etch Rate:</td>
<td>1 µm/minute</td>
</tr>
</tbody>
</table>

► **TMAH Etchant for Single Crystal Silicon**

TMAH solution is commonly stocked in a 25% concentration. Calculate the amount of 25% solution and DI water to make the desired etchant concentration (often 3%-15%). Under the hood, open TMAH container, add the desired amounts of solution to a DI water to bath, and heat to 80ºC. Silicon nitride and silicon dioxide are both good masks for TMAH.
Silicon Dioxide Etchant (Buffered HF)
► NH : HF₄ (6:1)
   Etch rate: ~1000 Å/min.

Silicon and Germanium Etchant
► CP-8 (fast) HNO₃ : HF (5:3)
  CP-6 HNO₃ : HF (5:1)
  CP-4 HNO₃:HF : acetic acid (540 ml : 200 ml : 200 ml)

Silicon-Germanium (polycrystalline)
► H₂O₂ (30%) at 90°C
  Etch rates:
  - 100% Ge 4000 Å/min
  - 80% Ge 1000 Å/min
  - 60% Ge and less do not etch

► H₂O at 90°C
  Etch rates:
  - 100% Ge ~200 Å/min
  < 60% Ge doesn't etch

► RCA SC-1 (NH₄OH: H₂O₂: H₂O) at 75°C
  Etch rates:
  - 100% Ge ~ 4 um/min
  - 80% Ge ~ 9000 Å/min
  - 60% Ge ~ 500 Å/min
  - 40% Ge ~ 30 Å/min
  - 20% Ge ~ 10 Å/min
  - 0% Ge ~ 5 Å/min

Silicon Monoxide Etchant
► Saturated solution of NaOH

THIN FILMS OF SiO REACT EXPLOSIVELY WITH HF!

Silicon Nitride Etchant
► Hot phosphoric acid ~ 150°C

Silver
► NH₄OH: H₂O₂ (1:1)
► Remove with HNO₃
► Clean with dilute HNO₃ : NH₄ (1:1)

Stainless Steel
► HF: HNO₃
► Aqua Regia (depends upon grade of stainless steel)
► HCl
► Electrolytic in diluted HCl

Tantalum
► HF: HNO₃ : H₂O (1:1:1)
  10 parts 30% KOH solution at 90°C
  1 part 30% H₂O₂
This mixture etches Ta<sub>2</sub>O<sub>5</sub> and tantalum nitride at rates of 1000-2000 Å/min. Attacks photoresists and must therefore be used with a metal mask (e.g. gold).

**Tin**
- HF : HNO<sub>3</sub> (1:1)
- HF : HCl (1:1)
- Clean with ammonium chloride
- Remove with HCl

**Titanium**
- H<sub>2</sub>O : HF : HNO<sub>3</sub> (50:1:1)
- H<sub>2</sub>O : HF : H<sub>2</sub>O<sub>2</sub> (20:1:1)
- HF : H<sub>2</sub>O : ethylene glycol (20:10:220)

No heating necessary. Rate ~ 1600 Å/min.
- For evaporation, deposit Al before Ti to facilitate cleaning of glass cylinder.
- Titanium dioxide is soluble in hot H<sub>2</sub>SO<sub>4</sub>.

**Titanium/Tungsten**
- Hydrogen peroxide

**Tungsten**
- 45% formic acid : 45% H<sub>2</sub>O<sub>2</sub> : 10% H<sub>2</sub>O
  Heat 2 minutes at 80ºC.
  Bleach
  HNO<sub>3</sub> : HF (1:10-15). This will not etch gold.
  Boiling hydrogen peroxide
  Fused NaOH (pellets, melted, 318ºC)
- Electrolytic NaNO<sub>2</sub>
  HF : HNO<sub>3</sub> (1:1)
  15 V ac with iron electrode (NaNO<sub>2</sub> for polished finish)
- Cleaning
  Boil in 20% solution NaOH for 15 minutes of HNO<sub>3</sub> : HF (1:1) for a few seconds.
- Potassium ferricyanide-based etch
  K<sub>2</sub>HPO<sub>4</sub> 34.0 grams
  KOH 13.4 grams
  K<sub>3</sub>Fe(CN)<sub>6</sub> 33.0 grams
  H<sub>2</sub>O ~1.0 liter
  Etches tungsten without significantly attacking resist.

**Turpentine**
- Insoluble in water. Soluble in alcohol, chloroform, ether, acetic acid.

**Vanadium**
- H<sub>2</sub> : HNO<sub>3</sub> (1:1)
- HF : HNO<sub>3</sub> (1:1)

**Westinghouse Etchant (Si Polish Etch)**
- HF : acetic acid : HNO<sub>3</sub> (3:5:15)

**Zinc**
- Reacts with HCl.
ZnO

- acetic acid : phosphoric acid : H₂O (1:1:30)
  The etch rate is approximately 5000 Å/min.

Zirconium

- H₂O:HF : HNO₃ (50:1:1)
- H₂O:HF : H₂O₂ (20:1:1)

See also:
  Dislocation Etches, Secco Etch
  Silicon and Germanium Etchant, CP-4, 6 and 8