ROCHESTER INSTITUTE OF TECHNOLOGY
MICROELECTRONIC ENGINEERING

Wet Etch for Microelectronics

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4-10-2008 wetetch.ppt

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OUTLINE

Wet Etch Basics
Etching of Oxide
Etching of Nitride
Etching of Silicon
Etching of Metals
RCA Clean
Wet Removal of Photoresist
Package Decapping
Defect Delineation
Cleaning
References

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TERMINOLOGY

Etching - the process by which material is removed from a surface
Mask Layer - Used to protect regions of the wafer surface. Examples are photoresist or an oxide layer
Wet Etching - substrates are immersed in a reactive solution (etchant). The layer to be etched is removed by chemical reaction or by dissolution. The reaction products must be soluble and are carried away by the etchant solution.
Dry Etching - Substrates are immersed in a reactive gas (plasma). The layer to be etched is removed by chemical reactions and/or physical means (ion bombardment). The reaction products must be volatile and are carried away in the gas stream.
Anisotropic - etch rate is not equal in all directions.
Isotropic - etch rate is equal in all directions.
Selectivity - the ability to etch desired film but not etch underlying substrate or mask layer.
Aspect Ratio - ratio of an etch geometries depth to width.

WET ETCH BASICS

Concentration: Often expressed as a weight percentage. That is the ratio of the weight of solute in a given weight solution. For example a solution containing 5 gms of solute in 95 grams of solvent is a 5 % solution.

Molarity: concentration expressed as moles of solute in 1 liter of solution. A solution containing one mole of solute in 1 liter of solution is termed a molar (1M) solution. A mole is the molecular weight in grams. Example: 10 gms of sulfuric acid in 500 ml of solution. H2SO4 has molecular weight of 1x2+32+16x4=98 so 10gms/98gm/M = 0.102M and 500ml is 1/2 liter, so this solution is 0.204 Molar

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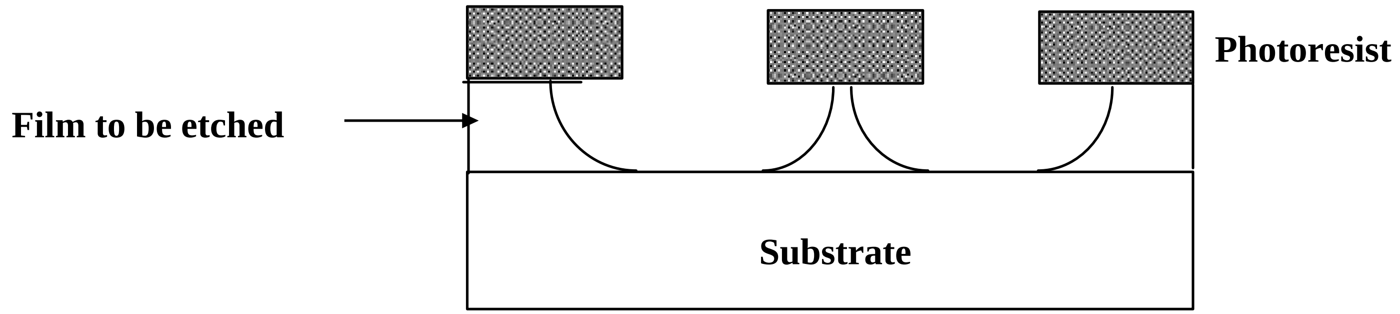
WET ETCH BASICS

Normality: concentration expressed as equivalents of solute in 1 liter of solution. One equivalent of a substance is the weight (1) which (as an acid) contains 1 gram atom of replaceable hydrogen; or (2) which (as a base) reacts with a gram atom of hydrogen; or (3) which (as a salt) is produced in a reaction involving 1 gram atom of hydrogen. Example 36.5 g of HCl contains 1 g atom of replaceable hydrogen and is an equivalent. 40 g of NaOH will react with 36.5 g of HCL which contains 1 g atom of hydrogen thus 40 g of NaOH is an equivalent. 98 grams of H2SO4 contains two gram atoms of hydrogen so 98/2 = 49 is one equivalent.

How many gams of sulfuric acid are contained in 3 liters of 0.5N solution?
(answer: 74.5g)

ISOTROPIC AND ANISOTROPIC ETCHING

Isotropic Etching - etches at equal rate in all directions

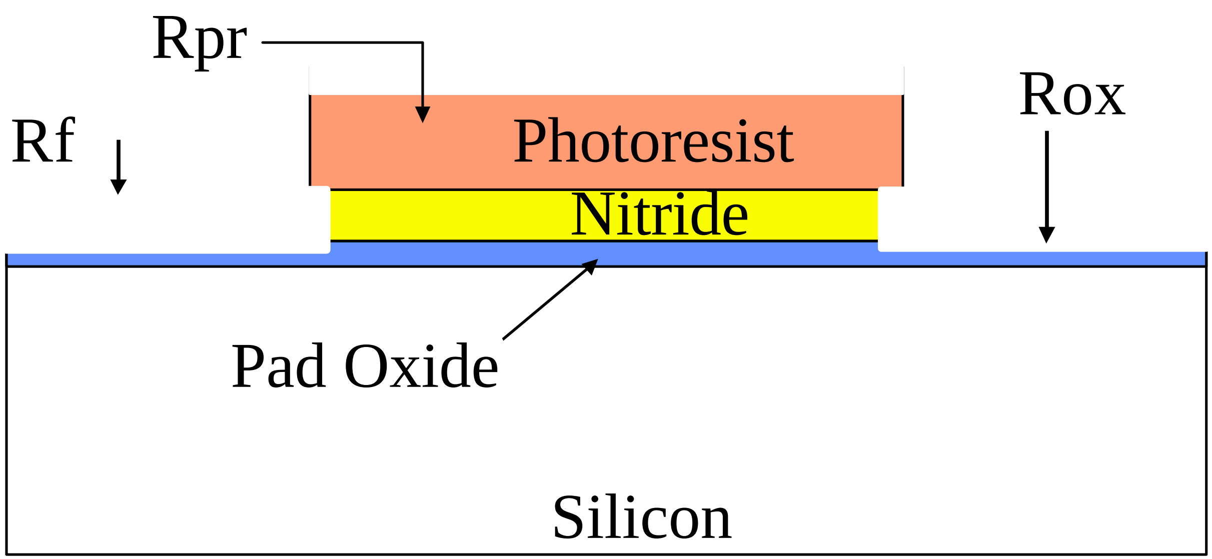


Anisotropic Etching - etches faster vertically than horizontally

Wet Chemical Etching - is isotropic (except in crystalline materials)

Plasma Etching (Dry Etch or Reactive Ion Etching, RIE) - is either isotropic or anisotropic depending on ion energy and chemistry of etch.

SELECTIVITY



Rf = etch rate for nitride film
Rpr = etch rate for photoresist
Rox = etch rate for pad oxide

We want Rf high and Rpr, Rox low

Selectivity of film to Photoresist = Rf/Rph

Selectivity of film to pad oxide = Rf/Rox

UNIFORMITY

$$\text{Etch rate uniformity (\%)} = \frac{\text{(Maximum etch rate-Minimum etch rate)}}{\text{(Maximum etch rate+Minimum etch rate)}} \times 100\%$$

Example: Calculate the average etch rate, etch rate uniformity given the etch rates at center, top left, top right, bottom right, bottom left are 750, 812, 765, 743, 798 nm

Answer: 773.6 nm
and 4.4%

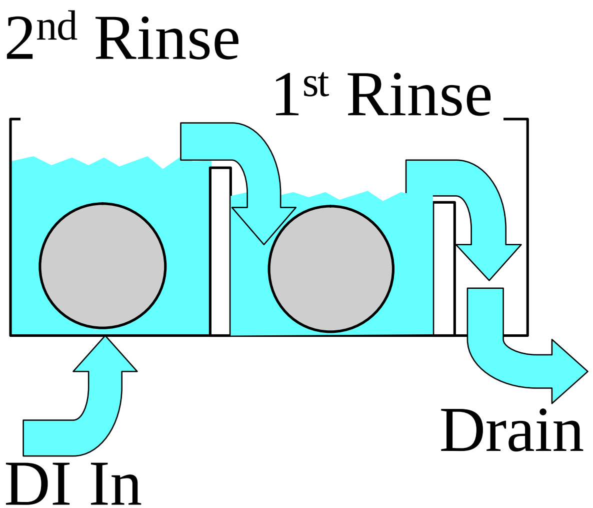
DI WATER

City Water In
Mixed Bed Filter
Water Softener
Charcoal Filter
Heat Exchanger
Reverse Osmosis Filters (6 Mohm)
Storage Tank
Recirculation Pumps
Resin Bed Filters (Rho = 18 Mohm)
Ultraviolet Light Anti Bacteria System
Final 0.2 um Particulate Filters
Special Piping

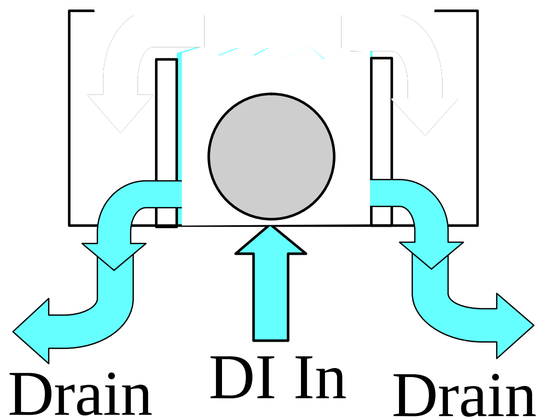
DI Water Plant at RIT

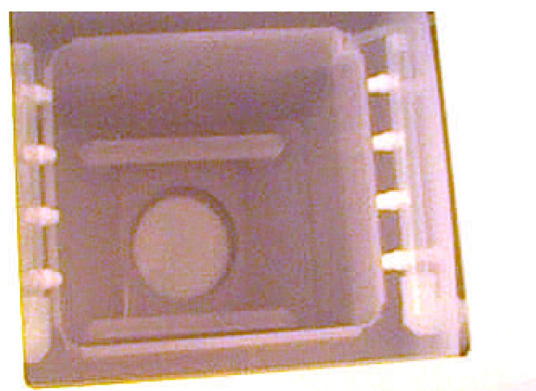
RINSE TANKS

Cascade Rinser



Dump Rinser





Spin Rinse Dry (SRD)

WET ETCHING OF SILICON DIOXIDE

HF with or without the addition of ammonium flouride (NH_4F). The addition of ammonium flouride creates a buffered HF solution (BHF) also called buffered oxide etch (BOE). The addition of NH_4F to HF controls the pH value and replenishes the depletion of the fluoride ions, thus maintaining stable etch rate.

$$SiO_2 + 6HF = H_2SiF_6 + 2H_2O$$

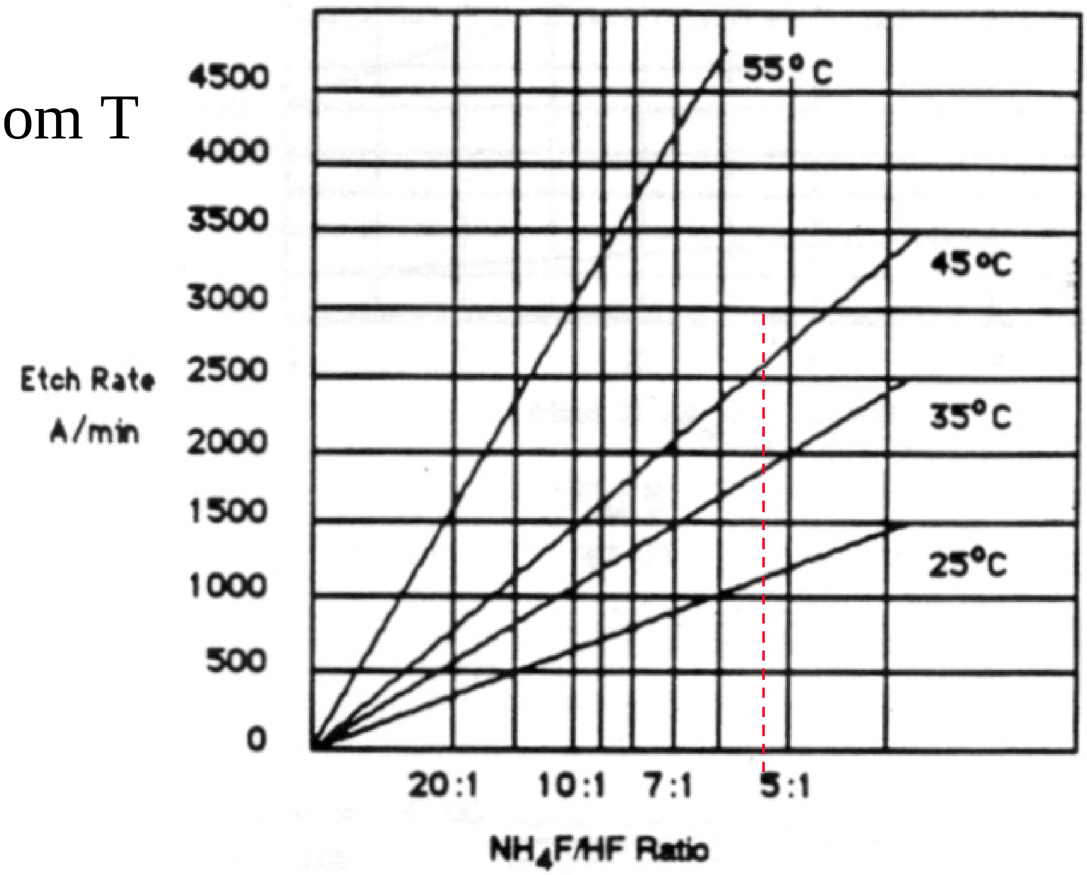
Types of silicon dioxide etchants:

- 49% HF - fast removal of oxide, poor photoresist adhesion
- BHF - medium removal of oxide, with photoresist mask
- Dilute HF - removal of native oxide, cleans, surface treatments
- HF/HCl or HF/Glycerin mixtures – special applications

TYPES OF BHF

7:1 NH_4F/HF gives about
1000 Å/min etch rate at room T

7 Parts 40% NH4F
and 1 part 48% HF



Oxide Etch Rates in Buffered Hydrfluoric Acid

ETCH RATES FOR VARIOUS TYPES OF SiO2

Thermal SiO2 * RIT data, Dr. Fuller, et.al. # from Madou Text ** from Journal of MEMs, Dec.'96, Muller, et.al.	BOE (7:1) 1:1 HF:HCl 49% HF KOH@ 72 °C KOH @ 90 °C	1,000 Å/min * 23,000 Å/min** 18,000 Å/min # 900 Å/min* 2500 Å/min*
CVD SiO2 (LTO)	BOE (7:1) 1:1 HF:HCl 49% HF	3,300 Å/min # 6,170 Å/min #
P doped SiO2 (spin-on dopant) (Photoresist adhesion problems)	BOE (7:1) 1:1 HF:HCl 49% HF	2000 Å/min 25,000 Å/min
Boron doped SiO2 (spin-on dopant)	BOE (7:1) 1:1 HF:HCl 49% HF	200 Å/min*
Phosphosilicate Glass (PSG) * RIT data, Dr. Fuller, et.al.	BOE (7:1) 1:1 HF:HCl 49% HF	10,000 Å/min # 11,330 Å/min # 28,000 Å/min

from Madou Text
** from Journal of MEMs, Dec.'96, Muller, et.al.

MEASURED ETCH RATES FOR OXIDE AT RIT

Rochester Institute of Technology	Dr. Lynn Fuller			
Microelectronic Engineering				
Summary of Etch Rates and Deposition Rates for RIT Processes				
Wet Etch Process Description	**Date**	**Rate**	**Units**	**Comment**
7:1 Buffered Oxide Etch of Thermal Oxide, 300°K	12/1/04	1122	Å/min	EMCR650
10:1 Buffered Oxide Etch of Thermal Oxide, 300°K	10/15/05	586	Å/min	Mike Aquilino
10:1 BOE Etch of PECVD TEOS Oxide, no anneal, 300°K	10/15/05	2062	Å/min	Mike Aquilino
10:1 BOE Etch of PECVD TEOS Oxide, anneal 1000C - 60 min, 300°K	10/15/05	814	Å/min	Mike Aquilino
10:1 BOE Etch of PECVD TEOS Oxide, anneal 1100C - 6 hr, 300°K	10/15/05	562	Å/min	Mike Aquilino
Pad Etch on Thermal Oxide, 300 °K	12/1/04	629	Å/min	EMCR650
Pad Etch of PECVD TEOS Oxide, 300°k	6/8/06	1290	Å/min	Dale Ewbank
Hot Phosphoric Acid Etch of Thermal Oxide at 175 °C	10/15/05	<1	Å/min	Mike Aquilino
Hot Phosphoric Acid Etch of TEOS Oxide, no anneal, at 175 °C	10/15/05	17	Å/min	Mike Aquilino
Hot Phosphoric Acid Etch of TEOS Oxide, 1000 C 60 min Anneal, at 175 °C	10/15/05	3.3	Å/min	Mike Aquilino
Hot Phosphoric Acid Etch of TEOS Oxide, 1100 C 6 Hr Anneal, at 175 °C	10/15/05	3.8	Å/min	Mike Aquilino
Hot Phosphoric Acid Etch of Si3N4 at 175 °C	11/15/04	82	Å/min	EMCR650
50:1 Water:HF(49%) on Thermal Oxide at room T	10/15/05	187	Å/min	Mike Aquilino
50:1 Water:HF(49%) on PECVD TEOS Oxide, no anneal, at room T	10/15/05	611	Å/min	Mike Aquilino
50:1 Water:HF(49%) on PECVD TEOS Oxide, anneal 1000 C -30 min, at room T	10/15/05	115	Å/min	Mike Aquilino
50:1 Water:HF(49%) of PECVD TEOS Oxide, anneal 1100C - 6 hr, 300°K	10/15/05	107	Å/min	Mike Aquilino
KOH 20 wt%, 85 °C, Etch of Si (crystaline)	2/4/05	30	µm/min	EMCR870
KOH etch rate of PECVD Nitride (Low σ)	2/4/05	10	Å/min	EMCR870

See http://people.rit.edu/lffeee tools and processes

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BUFFERED OXIDE ETCH TANK

ETCHING GLASS OVER ALUMINUM WITH BOE AND GLYCERIN MIXTURE

In multilevel metal processes it is often necessary to etch vias through an insulating interlevel dielectric. Also if chips are given a protective overcoat it is necessary to etch vias through the insulating overcoat to the bonding pads. When the underlying layer is aluminum and the insulating layer is glass the etchant needs to etch glass but not etch aluminum. Straight Buffered HF acid will etch Aluminum.

A mixture of 5 parts BOE and 3 parts Glycerin works well. Etch rate is unaffected by the Glycerin. Original work was published by J.J. Gajda at IBM System Products Division, East Fishkill Facility, Hopewell Junction, NY 12533

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BOND PAD ETCHES FROM TRANSENE

Silox Vapox III – TRANSENE CO. (www.Transene.com)

This etchant is designed to etch deposited oxides on silicon surfaces. These oxides are commonly grown in vapox silox or other LPCVD devices and differ radically from their thermally grown cousins in many important ways. One way is their etch rate another is their process utility. The deposited oxide is many times used as a passivation layer over a metallized silicon substrate. Silox Vapox Etchant III has been designed to optimize etching of a deposited oxide used as a passivation layer over an aluminum metallized silicon substrate. This etchant has been saturated with aluminum to minimize its attack on the metallized substrate.

Deposited Oxide (Vapox/Silox) Etch Rate: 4000 Å / minute @ 22 °C

This product contains:

- Ammonium Fluoride
- Glacial Acetic Acid
- Aluminum corrosion inhibitor
- Surfactant
- DI Water

Pad Etch Measured etch rates at RIT:

Thermal Oxide 630 Å/min

TEOS PECVD TEOS Oxide 1290 Å/min

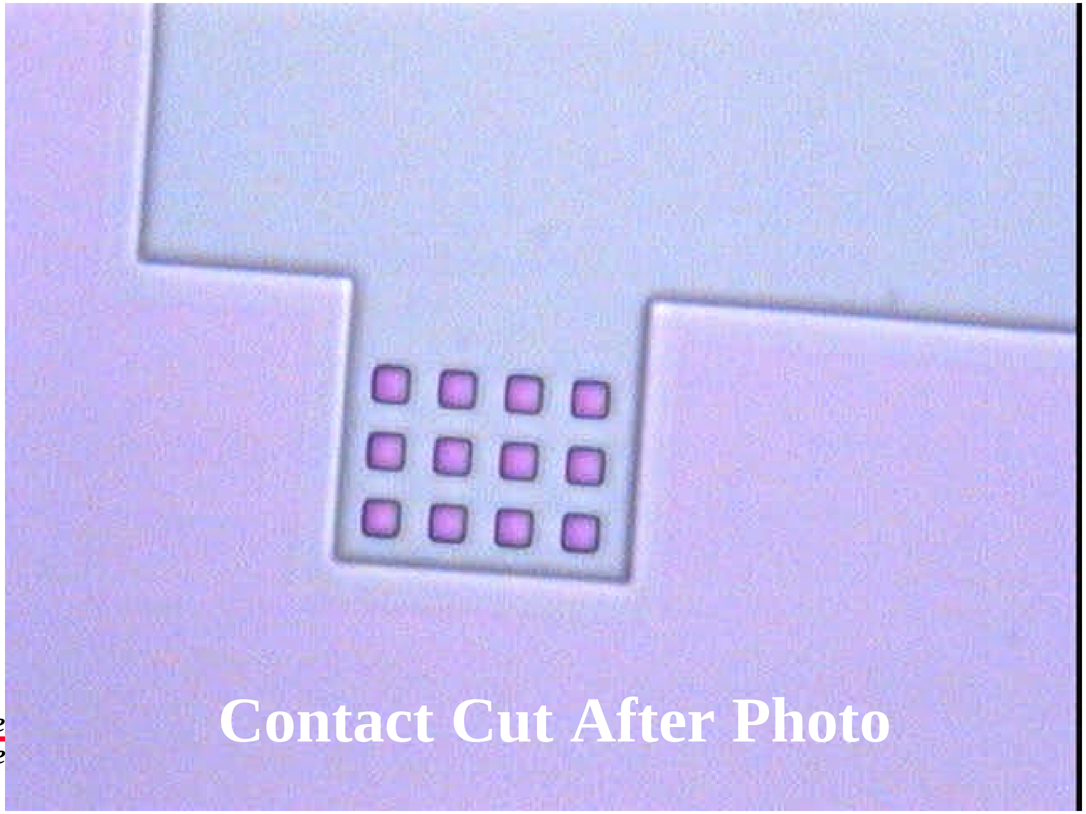
MAKING SMALL (2µm x 2µm) CONTACT CUT BY WET ETCH

Contact Cut Lithography is difficult because of the complicated film stack. The contacts are through 4000Å TEOS oxide on thermal oxide on poly on gate oxide. The poly has a silicide layer in the Advanced CMOS process. The poly thickness might be 4000Å or 6000Å. The TEOS may be annealed. Other contacts are to drain and source through 4000Å TEOS on thermally grown oxide of ~500Å (from poly reox step) plus gate oxide. The gate oxides are 330Å, 150Å, or 100Å depending on the exact process.

Contact cut etch is also difficult. Plasma etch is difficult because of the different oxide layers and thickness and the poor selectivity between etching oxide and the underlying poly or drain/source silicon. Wet etch has problems with blocking. That is where the BOE can not get into the small contact cut openings. Blocking depends on surface tension as measured by the wetting angle which depends on the type of photoresist used.

MAKING SMALL (2µm x 2µm) CONTACT CUT BY WET ETCH

To ensure that the photoresist is cleared in the bottom of all the contact cuts the exposure dose is increased to 285 mj/cm2 and track develop time is increased to 3 min. This makes the 2µm x 2µm contacts a little larger ~2.2µm by ~2.2µm but they are clear regardless of the underlying film stack.



Contact Cut After Photo

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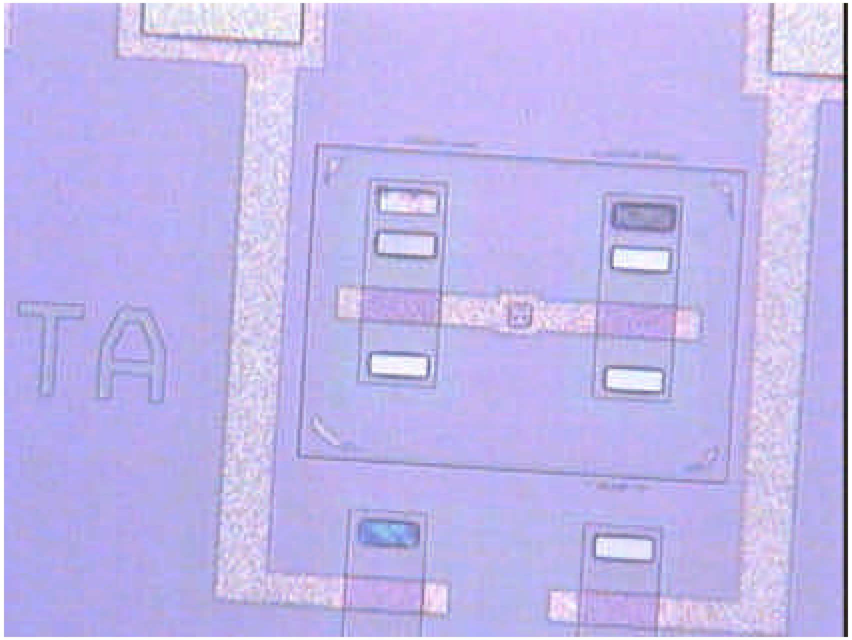
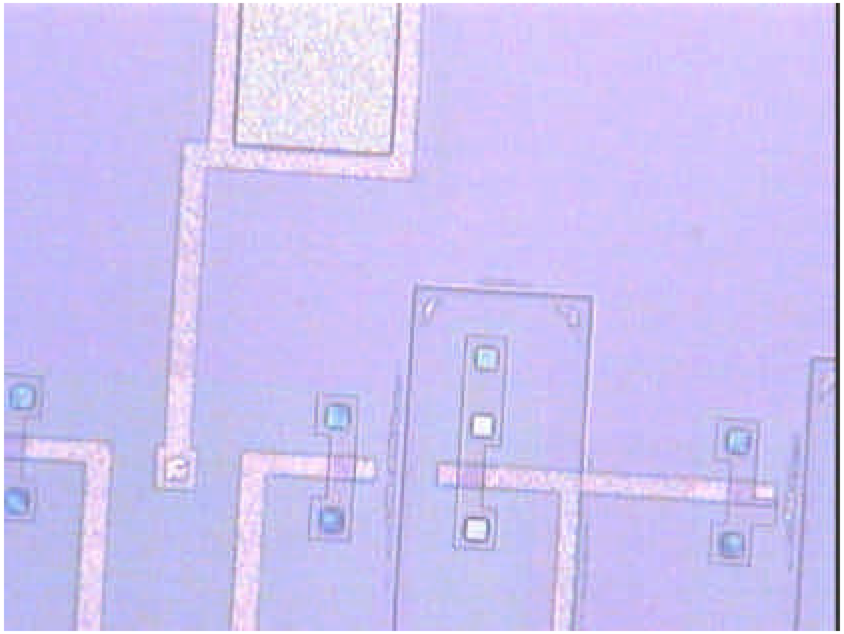
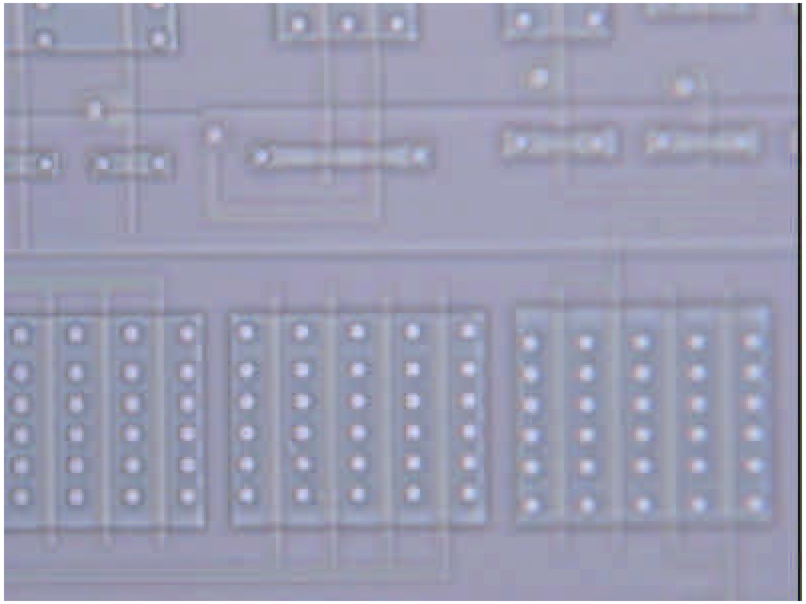
MAKING SMALL (2µm x 2µm) CONTACT CUT BY WET ETCH

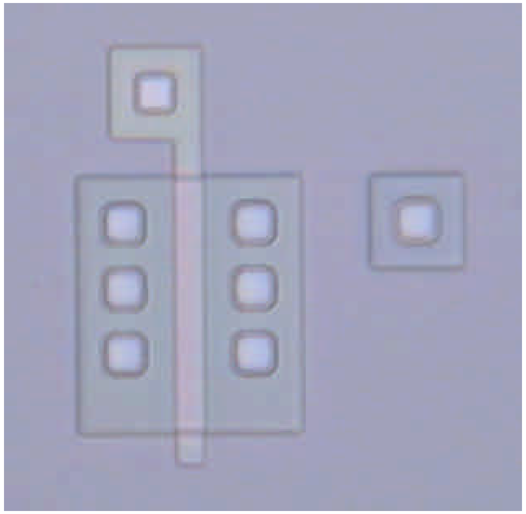




Wet etch has problems with blocking. That is where the BOE can not get into the small contact cut openings. Blocking depends on surface wetting angle. If blocking occurs some contact cuts will etch and clear while others will not etch as illustrated in the pictures above.

MAKING SMALL (2µm x 2µm) CONTACT CUT BY WET ETCH





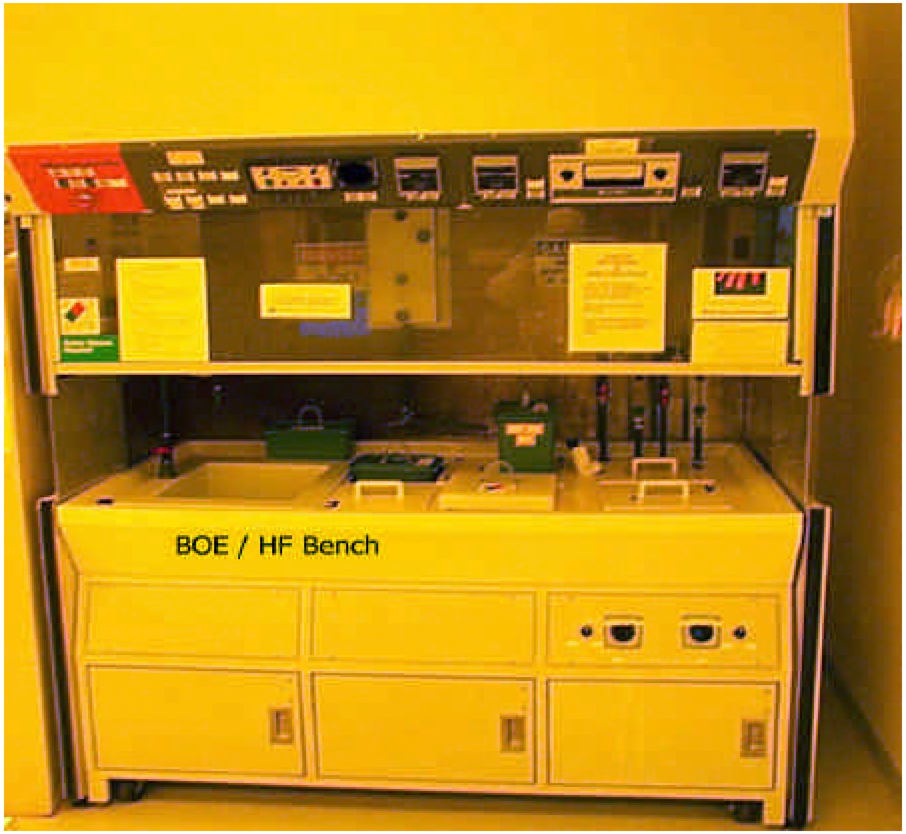
To overcome the blocking problem we raised the boat completely out of the BOE every 15 seconds throughout the entire etch. To be sure to clear all the contacts the etch time was extended to 5 minutes (approximately twice the expected etch time based on etch rates and approximate film thicknesses) This approach gave excellent results for all the various film stacks as shown in the pictures above.

SILICIDE ETCHES

Etching of Ti Metal:

Heat the Sulfuric Acid:Hydrogen Peroxide (1:2) mixture on a hotplate to 100°C (set plate temperature to 150°C)

Etch for 1 min 30 sec. This should remove the Ti that is on top of the silicon dioxide but not remove TiSi that was formed on the polysilicon and D/S regions. It also removes unreacted Ti metal over the TiSi on the poly and D/S regions.



Courtesy of SMFL

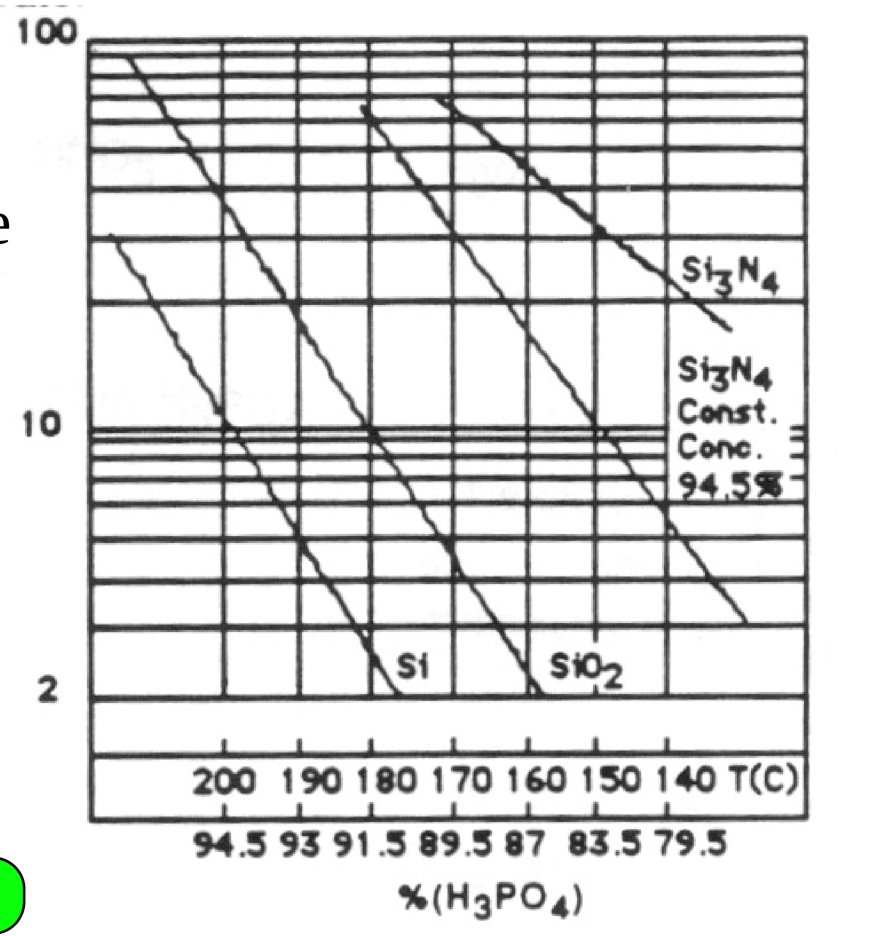
WET ETCHING OF SILICON NITRIDE

Silicon Nitride - BOE (7:1) 20A/min,
1:1 HF:HCL 120A/min,
49% HF 140 A/min
165°C Phosphoric Acid 55A/min (BOE dip first to remove oxynitride layer), etches silicon dioxide at 10 Å/min and silicon at 1Å/min

Hot phosphoric acid etch of nitride can not use photoresist as an etch mask. One can use a thin patterned oxide (or oxynitride) to act as the etch mask. Etch rate for silicon is even lower than the etch rate of oxide.

HOT PHOSPHORIC ACID ETCH OF SI3N4

The boiling point of phosphoric acid depends on the concentration of H_3PO_4 in water. So if you heat the solution until it boils you can find the corresponding concentration. If you operate at the boiling temperature (temperature is controlled without a closed loop control system) and the water boils off, the concentration increases making the boiling point hotter. Thus reflux condensers and drip systems replace the water to control the concentration and boiling temperature.



Silicon Nitride Etch Rate in Boiling Phosphoric Ac

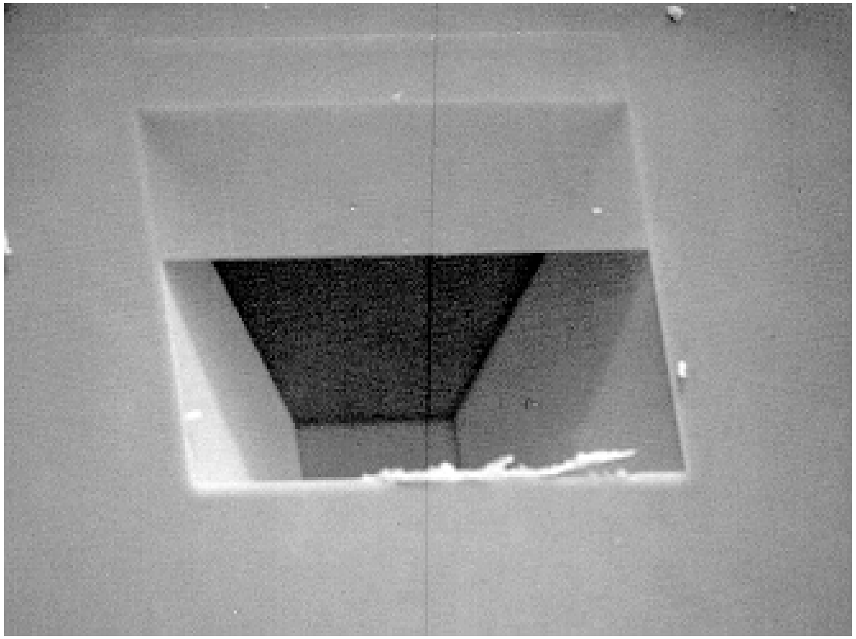
MORE

HOT PHOSPHORIC ACID NITRIDE ETCH BENCH

- Warm up Hot Phos pot to 175º
- Use Teflon boat to place wafers in acid bath
  - 3500Å +/-500 → 50 minutes
  - 1500Å +/- 500 → 25minutes
  - Etch rate of ~80 Å/min
- Rinse for 5 min. in Cascade Rinse
- SRD wafers

KOH ETCHING OF SINGLE CRYSTAL SILICON

KOH etches silicon along the (111) crystal plane giving a 53° angle.



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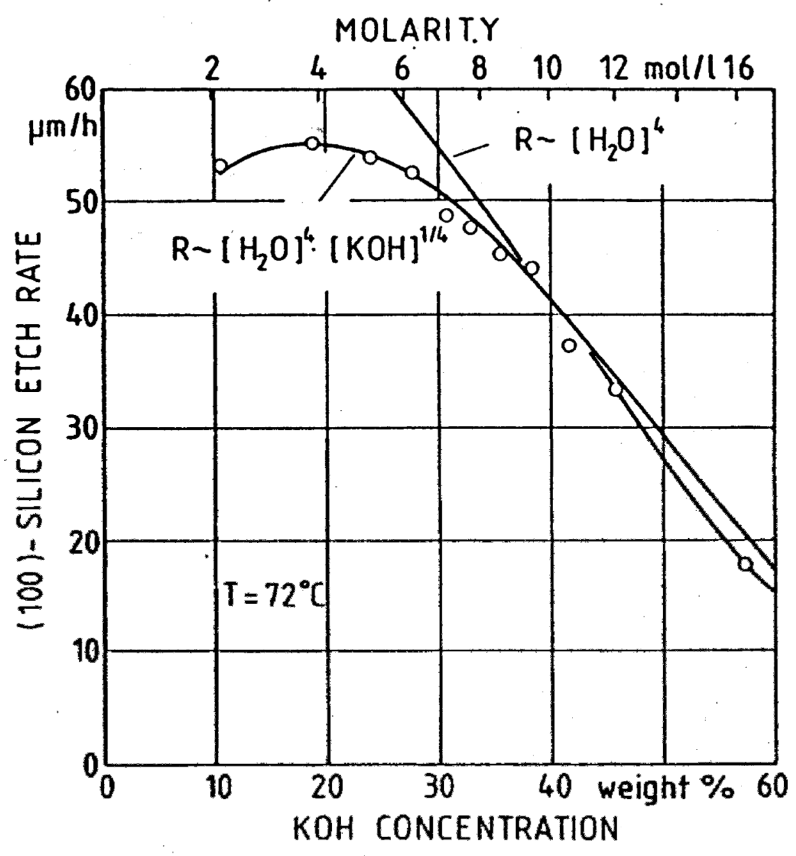
KOH ETCHING OF SINGLE CRYSTAL SILICON

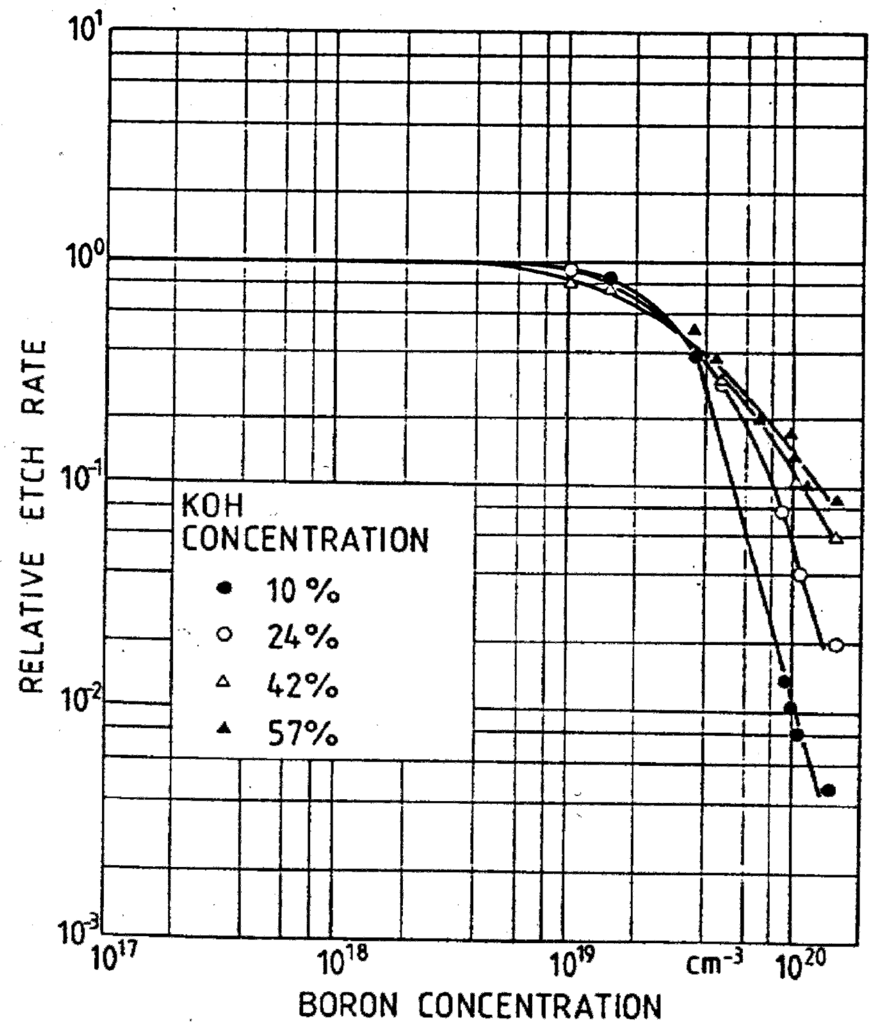
Si_3N_4 is the perfect masking material for KOH etch solution. The etch rate for Silicon Nitride appears to be zero.

When SiO_2 is used as a masking layer with a KOH solution both temperature and concentration should be chosen as low as possible. LTO is not the same as thermal oxide and can be attacked by KOH at a much higher rate. KOH etch rate is about 50 to 55 µm/min at 72 °C and KOH concentrations between 10 and 30 weight %. The Si/SiO etch ratio is 1000:1 for 10% KOH at 60 °C, at 30% it drops to 200:1. The relative etch rate of doped silicon to lightly doped silicon decreases for doping concentrations above 1E19 and at 1E20 the relative etch rate is 1/100 for 10% concentration. (on (100) wafer the angle is 50.6°)

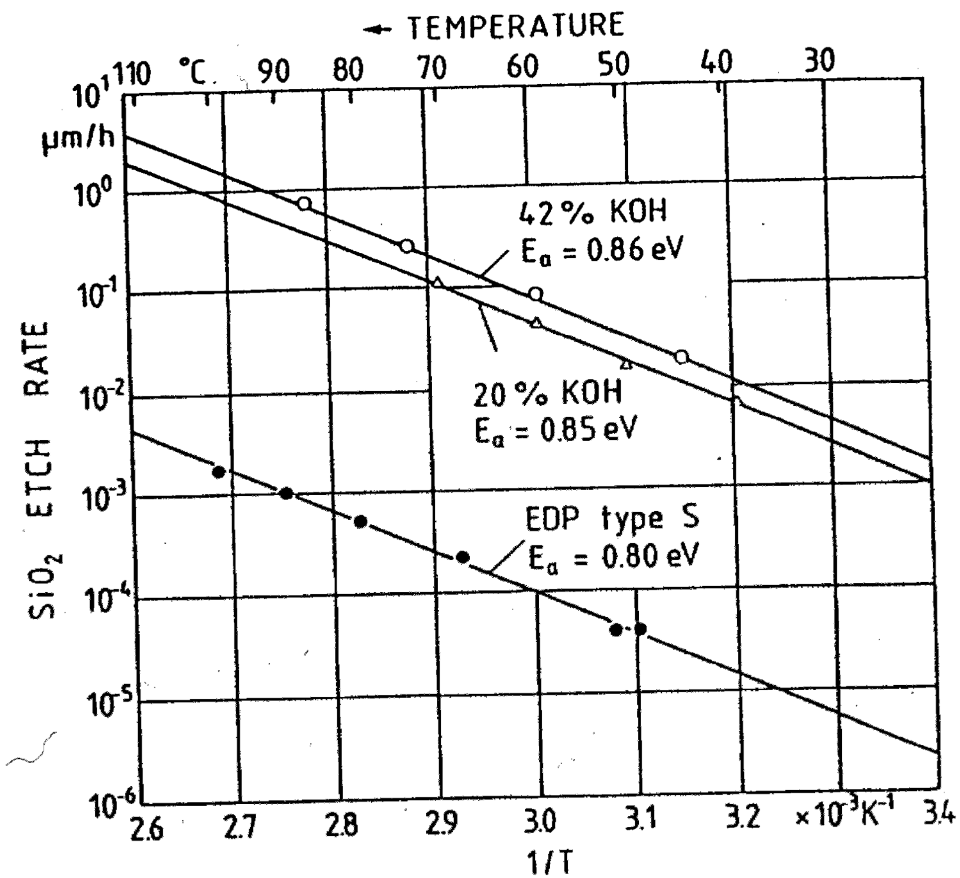
C. Strandman, L. Rosengren, H. Elderstig, and Y Backlun uses Isopropyl Alcohol (IPA) added to the KOH mixture at 30 wt% before IPA was added. 250 ml of IPA per liter of KOH was added giving an excess of IPA on the surface of etchant during etching.

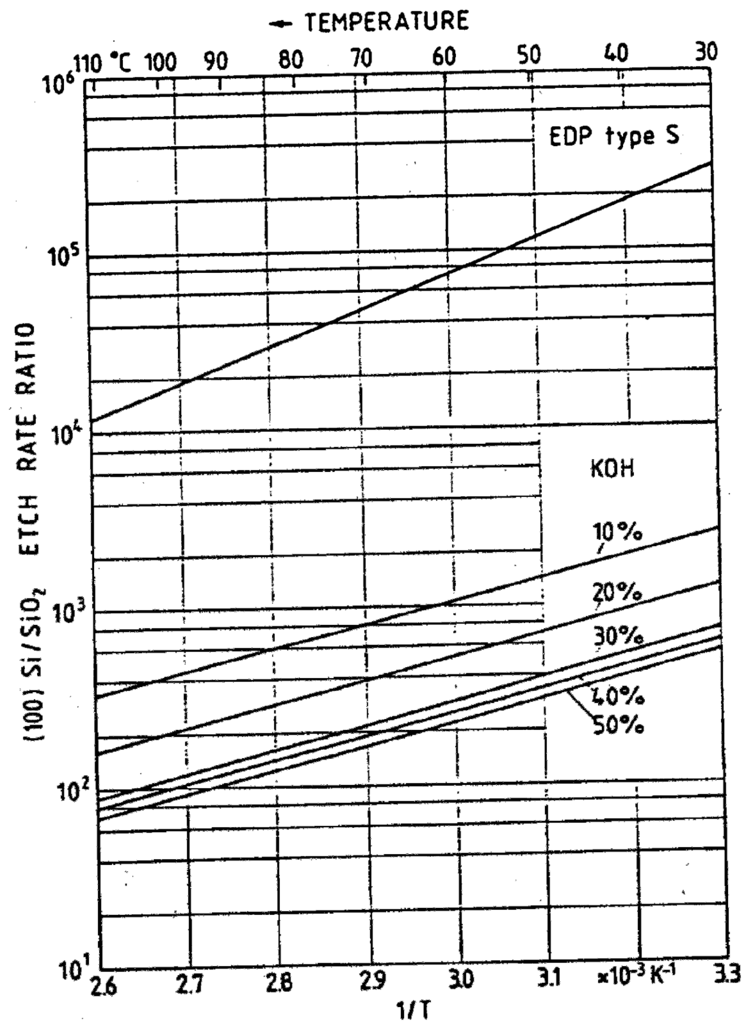
KOH ETCHING OF SILICON



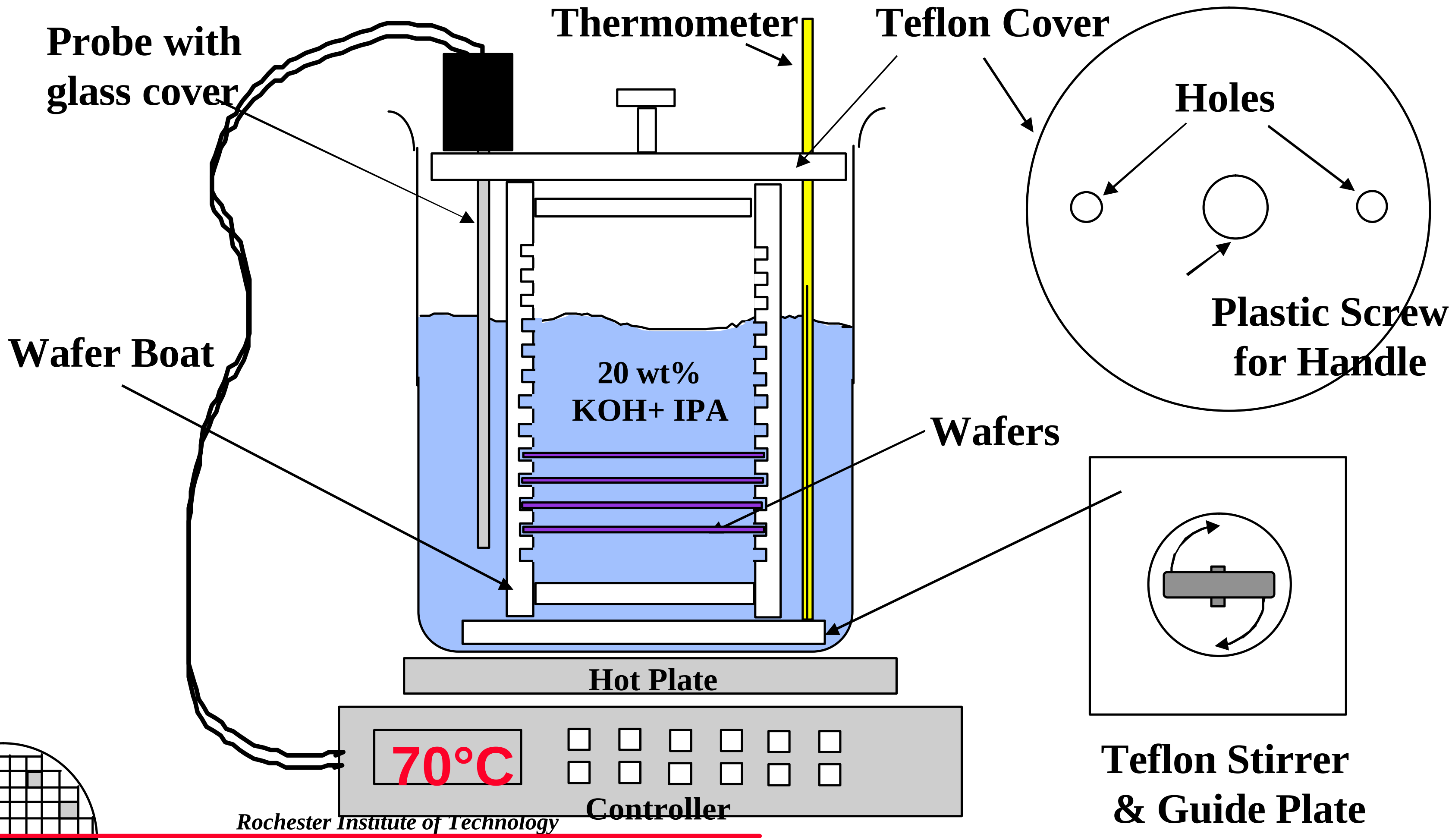


KOH ETCHING OF SILICON OXIDE





KOH ETCH APPARATUS



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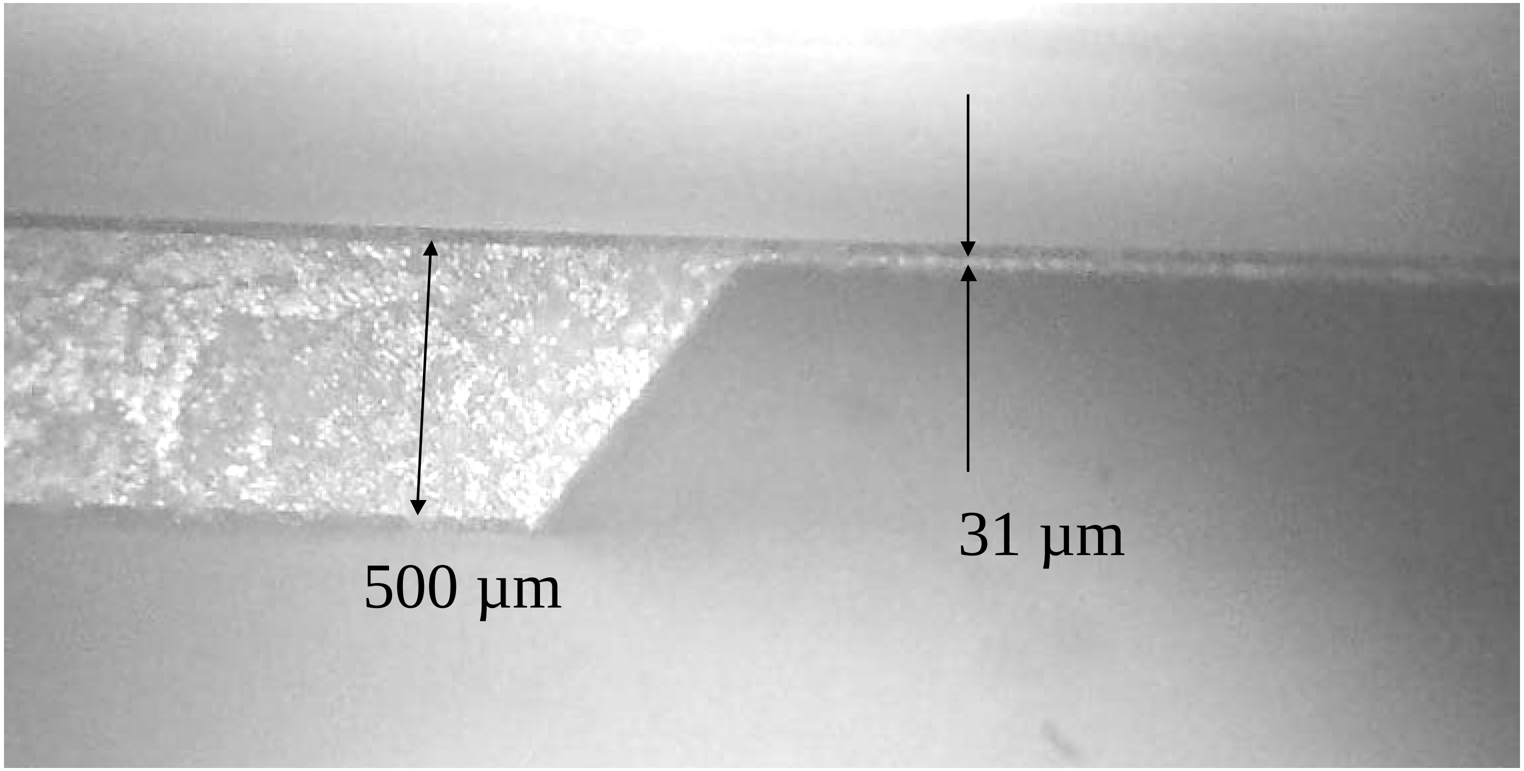
SINGLE SIDED KOH ETCH APPARATUS

Dual 4 inch wafer holder with "O" ring seal to protect outer ½ " edge of the wafer. Integral heater and temperature probe for feedback control system. Stainless steel metal parts do not etch in KOH.

3-28-02 Fuller et.el.

50 um in 57 min at 72 C

PICTURES OF DIAPHRAGM ETCH



20% KOH Etch, @ 72 C, 10 Hrs.

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WET ETCHING OF POLYSILICON

Poly – KOH etches poly at the same rate it etches single crystal silicon. Poly does not etch with 53° angle because the crystal orientation is random.

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WET ETCHING OF ALUMINUM

Aluminum - "Aluminum Etchant Type A" from Transene Co., Inc. Route 1, Rowley MA, Tel (617)948-2501 and is a mixture of phosphoric acid, acetic acid and nitric acid. Al/1%Si leaves behind a silicon residue unless the aluminum etch is heated to 50C. For multilayer metal processes it is often necessary to etch vias through an insulating interlevel dielectric. When the underlying layer is aluminum and the insulating layer is glass the preferred etchant is 5 parts BOE and 3 parts Glycerin. (straight BOE etches aluminum) See J.J.Gaida, IBM System Products Division, East Fishkill Facility, Hopewell Junction, NY 12533.

WET ETCHING OF METALS

Nickel - Use Aluminum Etch at 50 C Temperature, Etch Rate is about 1 min for 2000 Å, The aluminum etch solution we use is "Aluminum Etchant Type A" from Transene Co., Inc. Route 1, Rowley, Mass 0196, Tel (617)948-2501 and is a mixture of phosphoric acid, acetic acid, nitric acid.

WET ETCHING OF METALS

Chromium - CR-9 Etch, Cyantek Corp., 3055 Osgood Court, Fremont, CA 94539-5652, (510)651-3341

Gold - Gold Etch, J.E.Halma Co., 91 Dell Glen Ave, Lodi, NJ 07644

Copper - Ferric Chloride or mix Etchant from 533 ml water, add 80 ml $Na_2S_2O_3$, Sodium Persulfate, (white powder, Oxidizer), prepare in glass pan, place pan on hot plate and heat to 50 C (plate Temp set at 100 C) Etch Rate ~ 1.66 µm/min Copper Coated Board about 30 min.

Platinum – Try 7ml HCl, 1ml HNO_3, 8 ml H_2O at 85 C approximate etch rate 450 Å/min. (seems to work as reported by Tom Brown 10-7-94)

TYPES OF CASSETTS AND CARRIERS

Black, Blue, Red wafer cassetts:
- Fluoroware PA182-60MB, PA72-40MB
- STAT-PRO© 100 (polypropylene)
- 6" wafers (SSI track, Canon stepper)
- 4" wafers (SVG track, GCA stepper)
- DO NOT USE IN WET CHEMISTRY

medium heavy

Teflon Chemical Process Wafer Cassett:
- Fluoroware A182-60MB
- PerFluoroAlkoxy (Teflon®) – heavy, medium
- High resistance to chemicals and temperature
- Can be used in wet chemistry processes (RCA clean, BOE etch, wet nitride etch, wet aluminum etch)

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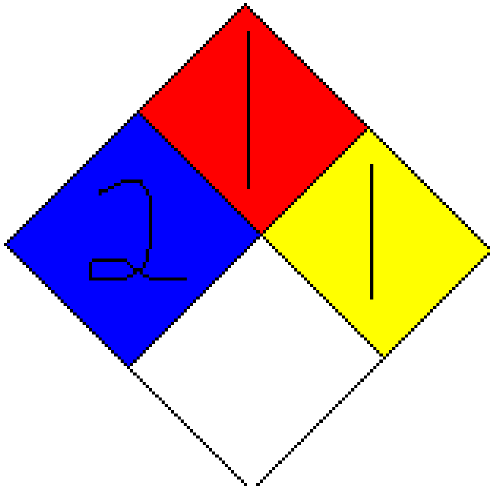
TYPES OF CASSETTS AND CARRIERS

Shipping Cassette:
- Empak PX9150-04
- Thin high purity polypropylene
- Available in 6" and 4" wafer sizes
- Not for use in processing
- DO NOT USE IN WET CHEMISTRY

Metal Cassetts:
- Stainless steel
- Available for 6" and 4" wafers
- Use on Branson Asher only
- DO NOT USE IN WET CHEMISTRY

WET RESIST STRIP WITH BAKER PRS-1000



What steps will use wet strip?

- **Eventually all steps after Gate Oxide Growth**

Why use wet strip?

- **Lower temperatures**
- **No electric field from plasma**
- **Gate oxide reliability increases**

Harmful if swallowed or inhaled. Causes irritation to skin, eyes and respiratory tract.

Flashpoint 96C

- **Explosive vapors can be formed above this temperature (sealed container)**

BAKER PRS-1000 PHOTORESIST STRIP

Processing temperature is 95C

Etch rate calculated to be 760A/min

- **Process designed for 1000A/min**

Inhibition layer could slow etch at start

10 minutes fully stripped most wafers

PHOTORESIST DEVELOPERS

CD-26 DI water

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RCA CLEAN WAFERS

APM

H_2O – 4500ml
NH_4OH–300ml
H_2O_2 – 900ml
75 °C, 10 min.

DI water rinse, 5 min.

H_20 - 50
HF - 1
60 sec.

DI water rinse, 5 min.

HPM

H_2O–4500ml
HCL-300ml
H_2O_2 – 900ml
75 °C, 10 min.

DI water rinse, 5 min.

SPIN/RINSE DRY

What does RCA stand for?

ANSWER

PLAY

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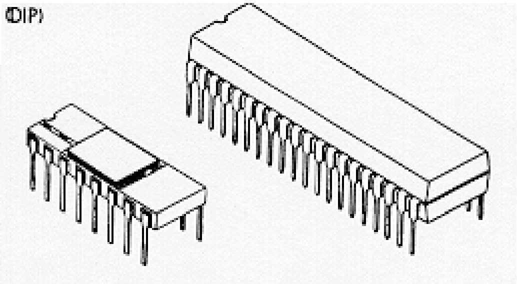
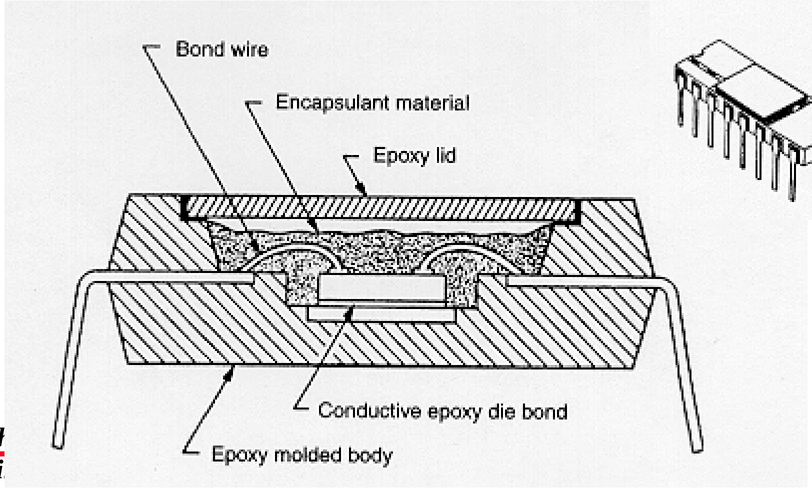
ETCHING THE DUAL-IN-LINE PLASTIC PACKAGE OFF OF PACKAGED CHIPS (DECAPSULATING)

Hot H_2SO_4 will etch the plastic package and not etch the metal wire bonds or other metal parts as long as no water is present. Straight H_2SO_4 heated to 100 C for 3 hours to remove all water. Allow to cool to 80C. This etch will remove a plastic package in 30 minutes. Immerse briefly in room temperature H_2SO_4 to cool the part, then rinse in DI water.





ETCH FOR SILICON DEFECT DELINEATION

Sirtl Etchant: 1 part 49%HF, 1 part CrO3 (5M) (ie 500 g/liter) Etch rate ~3.5um/min. good on (111), poor on (100) faceted pits.

Secco Etchant: 2 parts 49%HF, 1 part K2Cr2O7 (0.15M) (ie 44 g/liter). Etch rate ~1.5 um/mon. Best with ultrasonic agitation. Good on all orientations. Non-crystallographic pits.

Wright Jenkins Etchant: 2 parts 49%HF, 2 parts conc. Acetic acid, 1 part con. Nitric acid, 1 part CRO3 (4M) (ie 400 g/liter), 2 part Cu(NO3)2 +3H2O (0.14M) (ie 33 g/liter). Etch rate ~1.7 um/min. Faceted pits, good shelf life.

MASK CLEANING SOLUTION

CA-40 Photomask Cleaning Solution
Used as a soap with texwipe similar to cleaning dishes.

CLEANING SOLUTIONS

Used for CMP clean. Used as a soap with texwipe similar to cleaning dishes.

Used in dishwasher.

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4. Scotten W. Jones and Steven T. Walsh, Wet Etching for Semiconductor Fabrication, Private Publication. **MORE**
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HOMEWORK

1. Determine the etch rates for the following materials and etches:
Thermal Oxide at room T in Pad Etch
Thermal Oxide at room T in BOE 7:1
Thermal Oxide at room T in 50:1 DI:HF
PECVD TEOS Oxide at room T in Pad Etch
PECVD TEOS Oxide at room T in BOE 7:1
PECVD TEOS Oxide at room T in 50:1 DI:HF

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