# EE 527 MICROFABRICATION

Lecture 19 Tai-Chang Chen University of Washington



# NOVOLAC DISSOLUTION - 1

• A minimum concentration of [OH<sup>-</sup>] is required to produce a net forward rate:





© UWEE TC Chen

Winter 2014

 $1_{1}$ 

### NOVOLAC DISSOLUTION - 2

# Solution Dissolution Rate, Angstroms/second

Typical data for different developer solutions:

Joiution	Dissolution Nate, Angstroms/second	
	Unexposed	Exposed
0.15 M NaOH	20	1400
0.15 M KOH	10	860
0.15 M NaOH +	270	3400
$0.1 \text{ M Na}_2 \text{SiO}_3$		
0.15 M NaOH +	350	2800
0.1 M Na <sub>3</sub> PO <sub>4</sub>		
0.15 M NaOH +	270	2400
0.1 M Na <sub>2</sub> CO <sub>3</sub>		

WASHINGTON

© UWEE TC Chen

Winter 2014

# PHOTORESIST TUNING - 1

- While standard recipes exist and are a good starting point, optimal image formation and development requires small adjustments to prebake time, exposure time, and development time.
- The optimal photoresist recipe is pattern dependent!
- 50:50 line and space patterns are often used to tune resist since they provide a quick visual indication of the process.
- Usually the process conditions are kept constant (prebake temperature, exposure intensity, developer concentration), and the three key times are adjusted:
  - 1. Development time, t<sub>D</sub>
  - 2. Exposure time, t<sub>E</sub>
  - 3. Prebake time, t<sub>P</sub>



© UWEE TC Chen

Winter 2014

# PHOTORESIST TUNING - 2

A typical line and space pattern set for tuning the photoresist:



# PHOTORESIST TUNING - 3

- If the line/space pattern comes out over or under, which knob do you turn?
- Essential considerations:
  - The prebake needs to be long enough to drive off all of the coating solvent but not too long where it starts to thermally crosslink.
  - The exposure needs to bleach the resist all the way to the bottom, but not further.
  - The development needs time to clear the bottom of the features, and the edges and corners, but not so long as to etch laterally.
- While detailed theory can provide some guidance, in the lab the empirical approach often wins.
- Design of Experiments: (DOE)
  - Run a permutation of trials from a nominal operating point to explore which direction produces the best results.



© UWEE TC Chen

Winter 2014

#### STRIPPING POSITIVE PHOTORESIST

- High processing temperatures and irradiation can induce excessive thermal cross-linking of a photoresist, making it very difficult to remove.
- Subsequent processing relies heavily upon the complete removal of all prior photoresist and other organic residues.
  - Organic residues must never enter furnace tubes!
- Chemically-based stripping is used to achieve most of this.
- Oxygen plasma ashing is used to achieve final cleaning.



© UWEE TC Chen

Winter 2014

#### POSITIVE PHOTORESIST STRIPPERS

- Acetone, CH<sub>3</sub>COCH<sub>3</sub>
  - Generally used only in research labs, but very safe and effective when the photoresist is still fairly soft. Requires liberal flushing!
- Piranha Etch, 4:1 H<sub>2</sub>SO<sub>4</sub> : H<sub>2</sub>O<sub>2</sub> @ 90°C
  - Extremely aggressive etch that will remove all organics, but it has a rather short pot life and must be handled very carefully.
- Cyantek Nanostrip or Nanostrip 2X
  - Usually a better alternative to piranha etch: safer, long storage life, premixed, can be used either at room temperature or heated up to ~90°C, if needed.
    - Sulfuric acid H<sub>2</sub>SO<sub>4</sub> 90%
    - Peroxymonosulfuric acid H<sub>2</sub>SO<sub>5</sub> 5%
    - Hydrogen peroxide  $H_2O_2 < 1\%$

WASHING TWAter H2O 5%

© UWEE TC Chen

Winter 2014

44

# ACETONE (CH<sub>3</sub>COCH<sub>3</sub>)

- NFPA704M code = 1-3-0; CAS # [67-64-1]
- clear, colorless liquid
- It is a much stronger solvent than most alcohols, and it is capable of removing difficult oil, wax, and resin stains.
- It is used in nail polish removers and oil-based paint thinners.
- It has a slightly sweet odor.
- the vapor is heavier than air.
- It is highly volatile and evaporates very quickly: BP = 56°C.
- It has low toxicity: TWA-TLV = 1000 ppm for worker exposure. Primary hazards:
- It is extremely flammable and a serious fire and explosion risk!
- FP = -17°C; LEL = 2.6%; UEL = 12.8%.

WASHINGTON

# SULFURIC ACID (H<sub>2</sub>SO<sub>4</sub>) - 1

- most commonly used industrial chemical
- colorless, oily, syrupy liquid, specific gravity = 1.84
- standard reagent concentration is 98%, yellow bottle cap
- NFPA704M code = 3-0-2-\; CAS # [7664-93-9]

Primary hazards:

- when dissolved into water, H<sub>2</sub>SO<sub>4</sub> liberates considerable heat
- H<sub>2</sub>SO<sub>4</sub> is a strong acid
- always add acid to water to avoid spattering
- concentrated H<sub>2</sub>SO<sub>4</sub> has a great affinity for H<sub>2</sub>O: hygroscopic
- acid burns to skin dehydrate the tissue with localized evolution of heat. skin becomes charred, like burnt wood

WASHINGTON



Red Wash Bottle

#### HYDROGEN PEROXIDE (H<sub>2</sub>O<sub>2</sub>)

- a very strong oxidizer
- NFPA704M code = 3-0-2-OXY; CAS # [7722-84-1]
- $2H_2O_2 \rightarrow 2H_2O + O_2$
- typically lasts only a few months under refrigeration (~5°C)
- standard reagent concentration is 30%, white bottle cap

Primary hazards:

- decomposes violently at 144°C
- readily provides oxygen in redox reactions
- can cause spontaneous ignition of combustible materials when > 50 %
- greatly speeds up many reactions, turning some into explosions
- can cause severe skin burns or bleaching
- drug store hydrogen peroxide is only 1-3 %



# PLASMA ASHING

- When simple chemical treatments fail, plasma ashing is usually the technique used to strip stubborn photoresist and its residues.
  - It is frequently used as a final clean up step, regardless.
- Plasma ashing is (dry) plasma etching using oxygen.
  - Barrel (full cassette) or single wafer chambers are most common.
  - Operating pressures are typically 1-15 mTorr.
  - Relatively low RF power is required, ~50-200 Watts.
  - Cycle times are also fairly short, ~5-15 minutes.
- Ashing is a bit of a misnomer.
  - No "ash" is left behind.
  - The O<sup>=</sup> oxygen radicals are extremely reactive with organic hydrocarbons, usually producing volatile H<sub>2</sub>O and CO<sub>2</sub> which gets removed in the vapor phase by the vacuum pump.



© UWEE TC Chen

Winter 2014



#### SU-8 PHOTORESIST - 1

- SU-8 is a negative, epoxide, near-UV photoresist.
- Originally developed at the IBM T. J. Watson Research Center in NY in 1989.
- When exposed to near UV (~350-400 nm), SU-8's long molecular chains cross-link causing the solidification of the material.
- The inertness and mechanical stability of cross-linked resins allows SU-8 to be used to form permanent structures.
- It is now mainly used in the fabrication of microfluidics and MEMS systems parts. It is also one of the most biocompatible materials known and is often used in bio-MEMS.



© UWEE TC Chen

Winter 2014

#### SU-8 PHOTORESIST - 2

- SU-8 is currently marketed by Micro-Chem as their Nano SU-8 product line.
- Several viscosities for a wide range of coating thicknesses:
  - SU-8 2000.5 The last digits in the part number give the nominal
  - SU-8 2002 thickness of the resist for a spin speed of ~3000 rpm.
  - SU-8 2005
    SU-8 2007
    Thicker layers are possible on a single coating by using slower spin speeds, down to as low as ~1000 rpm.
  - SU-8 2010
    High aspect ratios are possible, up to ~100:1.
  - SU-8 2025
    SU-8 2035
    SU-8 2035
  - SU-8 2050 Practical considerations:
  - SU-8 2075 SU-8 is extremely goopy and difficult to clean up.
  - SU-8 2100 Once applied and cross-linked, SU-8 is extremely difficult to
    SU-8 2150 remove.



It is generally considered to be a "permanent" photoresist.