Cleanliness, Contamination, and Chemical Handling in the Stanford Nanofabrication Facility

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Outline

- Cleanliness
  - Why?
  - How?
- Cleaning
  - Cleans
  - General cleanliness
- Lab Policy
  - Personal
  - Equipment and Wafers
Why should you care?

Chemical contamination can ruin:
- Device and circuit performance
- Device reliability
- Process & equipment controllability/reliability

Particulate contamination can ruin:
- Device and circuit yield
- Process repeatability

Contamination affects not only YOUR research --
But EVERYONE ELSE’S too!

Device Issues

- Performance Degradation
  - gate oxide quality
  - carrier lifetime
  - threshold shifts
  - Low $I_{ON}$, high $I_{OFF}$

![Diagram showing the effects of contamination on device performance](image)
Device Issues

• Reliability Degradation
  – Oxide breakdown

- Contaminants act as "weak spots" in the oxide during stressing

Circuit Issues

• Yield Degradation
  – single defect can ruin entire circuit

• Performance Degradation
  – must design for worst case in performance space
MEMS Issues

• Release problems

Contaminant prevents proper release of overhang

Prevention: Personal Cleanliness

• Pre-suiting procedures
  – Wash your face and hands before you enter the lab. Use lots of water.
  – If you eat anything prior to entering the lab, drink water and rinse your mouth out.
  – Do not smoke just before entering the lab. Drink water if you have smoked recently.
  – Avoid muddy shoes
  – Wear gloves before suiting up
Prevention: Personal Habits & Hygiene

Gowning
• Follow gowning procedures
• Change your bunnysuit routinely

Bringing in materials
• Check new materials w/SpecMat
• Make sure things you use are cleanroom-compatible

Lab behavior
• Use appropriate handling tools (not fingers)
• Keep movements deliberate

Hygiene
• Cough and sneeze away from processing areas
• Avoid touching face and unprotected skin

Prevention: Wafer Handling

Poor handling practices are the primary cause of wafer defects!

Tweezers
• Use appropriate tweezers

Transferring wafers
• Practice roll transfers (cassette to cassette)
• Practice good wafer transfer methods (back to front unload, front to back load)

Transporting wafers
• Use appropriate cassettes for the process/station you plan to use
• Use wafer boxes when transporting from station to station
Cleaning - Surface Issues

**Contaminant**
- Organics
  - Skin oils
  - Resist
  - Polymers
- Metals
  - Alkali ions
  - Heavy and transition metals
- Native oxide
- Micro-roughness

**Sources**
- Room air, storage boxes residue from resist
- New wafers, chemicals, plasma etching, other equipment
- Room air, DI dryer, chemicals
- Chemicals, etching

**Effects**
- Oxidation rates, Interface properties
- Breakdown field, leakage, oxidation rates, interface properties, carrier lifetime
- Interface oxide under high-K
- Breakdown field, leakage, mobility

Origins of Metal Contamination

- Sources: equipment, processes, materials, and human
- Transition metals precipitation on Si surface is critical
Needs from Wet Clean

• Remove
  – Organics
  – Particles
  – Metals/ Ions
  – Native Oxide
• Passivate surface
• Dry surface
• Without roughening or contaminating surface

Si Process Cleans

Post-lithography rinse
Resist strip
• “Clean” substrates
  - Following simple etch
  - For hardened resist
  - For sidewall removal
• Metal-bearing substrates
  - Following simple etch
  - For hardened resist

Standard Pre-diffusion furnace clean
• Clean substrates

Standard Pre-deposition clean
• Clean substrates
• Metal-bearing substrates

Decontamination cleans
• Clean substrates
Post-lithography Rinse

Wafers undergoing dry etch following photolithography, may need to be rinsed prior to processing. The rinse will remove residual chemicals which may remain after develop. Residual developer can corrode substrate films and surfaces coming into contact with wafers.

Sulfuric/Peroxdie Clean: “Piranha”

• “Piranha” is a heated, boiling mixture of concentrated sulfuric acid ($\text{H}_2\text{SO}_4$) and ~30% hydrogen peroxide ($\text{H}_2\text{O}_2$).

• This is a strongly oxidizing acid mixture that is used for removal of organics. It is also used for removing gross particle contaminants (e.g., scribe dust) and is used in as part of the pre-furnace/pre-deposition clean.
  – $\text{H}_2\text{SO}_4$ - reduces organics to carbon
  – $\text{H}_2\text{O}_2$ - oxidizes carbon to form $\text{CO}_2$

• This clean will consume some metals to form sulfates, and is thus not metal-compatible

• Peroxide evaporates rapidly and is consumed in the reaction, so needs to be replenished frequently.
**Metallic / Alkali clean**

\[ \text{H}_2\text{O}:\text{H}_2\text{O}_2:\text{HCl} \quad 5:1:1 \quad \text{Typically at 70}^\circ\text{C} \]

- This clean removes metallic contaminants and alkali ions
  - \( \text{H}_2\text{O}_2 \) - Oxidizes surface (metal and hydrocarbons)
  - \( \text{HCl} \) - Reacts with most metals to form soluble chlorides
  - Removes metals and ionics from surface
  - Leaves chemical oxide on Si surface

**Oxide Clean/Etch**

HF (Hydrofluoric Acid)

- This clean removes oxides from the wafer surface. It may sometimes be buffered in \( \text{NH}_4\text{F} \), and is then called BOE.
- Note that this clean attacks several metals and is metal-incompatible
Passivation:
HF Terminated Surface

- HF, NH₄F or HI will passivate Si surface with a H termination
- Passivation reduces organic pickup from air
- Passivation can last for hours
- Degraded by UV or ozone in air

Resist Strip: Nonmetal-bearing Wafers

Resist hardening: Resist can get heavily cross-linked, e.g., due to heavy ion implantation and in some plasma etchers

For non-hardened photoresist, 20 minutes of immersion in 10:1 ratio of conc. sulfuric:30% hydrogen peroxide is used.

For most hardened photoresists, plasma O₂ clean may be required prior to standard or abbreviated (10 min) 10:1 piranha resist strip.

Following certain plasma etch processes in which sidewalls may form as a side reaction, additional sidewall stripping may be required, in addition to plasma O₂ and standard 10:1 piranha resist strip.

Always inspect your wafers after a resist strip to ensure cleanliness
Resist Strip for Metal-bearing Wafers

Piranha cannot be used because it corrodes most metals. PRX-127 is a commercial chemical product used for stripping resist. It is partly a solvent and partly a base; introduction of water will cause metal corrosion.

For non-hardened photoresist (i.e., wafers undergoing standard wet etch), 20 minutes of immersion in heated PRX-127.

For most hardened photoresists, plasma O₂ clean may be required prior to resist strip in PRX-127.

Always inspect your wafers after a resist strip to ensure cleanliness.

Post Plasma Etch Side Wall Cleaning

- Poly-Si/Si etch (HBr/Cl₂)
  - 50:1 HF 10 sec before resist strip
- Oxide etch
  - Polymer cleanup process
- STS Si etch
  - Oxygen plasma
- Al etch
  - Passivation/strip in AMT P5000/chamber D or PRX127 clean
Standard Pre-diffusion Furnace Clean
For Clean Wafers only

A. 4:1 Conc. H2SO4: 30% H2O2
B. 50:1 HF dip
C. 5:1:1 Water: 30% H2O2: conc. HCl
D. 50:1 HF dip (before gate oxide growth)

Mixture A: Removes residual organics.
Mixture B: Removes oxide formed by exposure to Mixture A, which is chemically oxidizing
Mixture C: Removes trace metals

• Rinses are done between each step.
• This is comparable in function to the industry-standard RCA clean and is performed for clean wafers prior to diffusion furnace processing (anneals and oxidations, not deposition.)
• Mixtures A and C need to be frequently replenished or changed.

Standard Pre-deposition Clean
For Clean wafers only

A. 4:1 Conc. H2SO4: 30% H2O2
C. 5:1:1 Water: 30% H2O2: conc. HCl
B. 50:1 HF dip

• Rinses are done between each step.
• Same function as the pre-Diffusion furnace clean, but with the HF dip done last to minimize native oxide formation between substrate and deposited film. (Some lab members prefer to do the standard pre-diffusion furnace clean process followed by an additional HF dip at the end.)
• This process is performed for clean substrates prior to LPCVD or metal film deposition.
Standard Pre-deposition Clean
For wafers with Standard Metals

Wafers are dipped in heated PRS-1000 for 20 minutes, followed by double-rinse cycle, then spin-rinse-dried.

PRS-1000 is a largely solvent-based, commercial stripper, similar to PRX-127.

Decontamination

Decontamination can be done for some substrates which have been exposed to alkali or other metal contaminants. Wafers must not contain any contaminating films. This is most commonly done following KOH etching of silicon. Following decontamination, wafers are considered to be “clean”.

Decontamination consists of

A. Immersion in 5:1:1 H2O:H2O2:HCl for 20 minutes (done at wbsilicide – sink must be decontaminated afterwards.)
B. Process through standard piranha resist strip at wbnonmetal
C. Process through standard pre-diffusion or pre-dep clean at wbdiff
Cleaning Vessels and Carriers

- Cleaning Vessel for SC1/SPM/SC2
  - Always use Quartz or Teflon containers
  - Never use Pyrex – 10% Na and large % B
- HF Vessel
  - Teflon preferred. Natural plastics without coloring and HF compatible are OK. HF can not go down drain
- Wafer/sample carriers
  - Teflon or quartz holders only
  - Wafers much easier to clean than pieces
  - Cassettes – more repeatable results
    - Compatible with spin rinser dryers
    - Require large vessels
  - Non-cassettes – Cheaper and small bath sizes
    - Usually requires blow of each wafer/piece

Rinsing

- Rinsing -- Always done in quartz or “clean” plastic
  - Must be long enough to remove all trace of previous chemical
    - Use pH meter or resistivity meter to test rinse
  - Beakers
    - Need to manual dump several times for each rinse step
    - Results vary a lot with user
    - Uses simple sink – Lowest cost
  - Up-flow and Cascade Rinse Tanks
    - Big improvement over beakers
  - Dump Rinser
    - Faster and more effective
    - Much higher cost
Drying

• Blow drying
  • Simplest setup and lowest cost
  • Needs point of use filter on nitrogen gun
  • Need clean surface under sample
    • Clean wipes or filter paper
    • Particles and water marks common problem

• Isopropyl drying
  • Beaker on hot plate with isopropyl at bottom
  • Wafers dry as slowly pulled out of breaker

• Spin Rinser Dryers
  • Requires full wafers and cassettes
  • Simplest to use but most expense

Storage:
Cassettes, Storage Boxes, Ozone

• Be careful about storing cleaned wafers
• Cassettes adsorb chemicals and release them later
• Plastic boxes can outgas -> organic deposition
• Lab air is filled with organics -> organic deposition
• Labs have lots of ozone
  ➢ Ozone removes H passivation
The three classes of equipment at SNF are:

- **Clean**: Most stringent level of chemical cleanliness, compatible with front-end CMOS processing.

- **Semi-clean**: Next level of chemical cleanliness, compatible with backend electronics process. Substrates with standard metals (Al, W, Ti) and their silicides are acceptable.

- **Gold-Contaminated**: Non-standard materials may be processed (however, any new material must receive prior approval before processing.) Substrates may be processed in sequence through equipment belonging to the groups "Clean" -> "Semi-Clean" -> "Gold-Contaminated", but generally not the reverse (exceptions listed on the next slide.)

- Wafers run in semiclean equipment are thereafter considered semiclean, even if there are no metal films present on the wafer. Likewise, wafers run in gold-contaminated equipment are then considered gold-contaminated.

- Some equipment (generally, some analytical and most litho tools) belong to all three categories. This is because there are built-in safeguard cleans in place to prevent cross-contamination at critical steps.

- Substrates run through some contaminating processes can be decontaminated (for ex., KOH etching.)

- SpecMat approves new materials and processes.
Lab policy - Cleanliness Levels

- Non-standard materials / gold?
  - No
  - Yes: No mainline equipment (except some photo, etc)
- Resist, other organics?
  - No
  - Yes: Dirty equipment only
- Metal status is accumulated - once contaminated, always contaminated
- Standard Metals?
  - No
  - Yes: Metal equipment only

Must be exposed <1 hour

Diffusion Clean?
- Yes: Any mainline equipment
- No: Any mainline equipment except post-diff

To remove organic contamination, use the appropriate clean

Lab Policy - Wet Cleans

- Cleans must be done less that 1 hour before equipment loading and wafers must be stored in clean Teflon cassettes
- Wafers can move between equipment of equal cleanliness provided
  - interval is less than 1 hour
  - wafers are in a clean Teflon cassette (not plastic)
Lab policy - equipment and wafers

• No wafers of unknown or inappropriate history

• All new materials and chemicals must undergo review by the SpecMat committee prior to use in the lab. SpecMat attempts to ensure that potential issues of cross-contamination and safety are carefully considered before new processes are approved. SpecMat can be contacted at specmat@snf.stanford.edu

• “New” includes not only materials that have not been used in the lab before, but also:
  ▪ Standard materials or chemicals from an unproven source
  ▪ Standard materials or chemicals used in a novel way

Don’t shortcut policy to get your own work done. If people break the rules, everyone suffers.

Take home message

Don’t allow bad habits to propagate. Be conscientious about your own habits and don’t be shy about correcting other people’s.

We ALL make mistakes. Be professional – take responsibility. Help by informing staff so we can work to minimize the effect on other people’s work.

Ask questions if you have any doubts. Everyone wants you to be successful.

Remember: This is a shared facility. Everything you do affects not only your work, but that of your fellow labmembers.