UCIRVINE

Cleaning Procedures for Silicon Wafers

INRF application note
Process name: SOLVENTCLEAN + RCA01 + HFDIP

Overview
Silicon wafer are cleaned by a solvent clean, Followed by a dionized water (DI) rinse, followed by an RCA clean and DI rinse, followed by an HF dip and DI rinse and blow dry. This is a level-1 process and requires basic INRF safety certification. The use of dangerous chemicals requires that the user may not perform the process alone.

Time needed
This process takes one hour to complete in total.

Materials needed
- Acetone
- Methanol
- Ammonium hydroxide
- Hydrogen peroxide
- Dilute (2%) hydrofluoric acid
- Pyrex bath containers
- Hot plate
- Safety equipment
  Neoprene gloves
  Safety glasses
  Acid apron

Solvent clean [SOLVENTCLEAN]
Setup time for this process is about 5 minutes. This process takes about 15 minutes to complete.

Solvents can clean oils and organic residues that appear on glass surfaces. Unfortunately, solvents themselves (especially acetone) leave their own residues. This is why a two-solvent method is used.

Pour the acetone into a glass container. Pour the methanol in a separate container. Place the acetone container on to a hot plate to warm up (do not exceed 55 deg C). Place the silicon wafer in the warm acetone bath for 10 minutes. Remove and place in methanol for 2-5 minutes. Remove and rinse in DI water (DI water rinse is optional). Blow nitrogen. If the solvents are clean and you intend to use them again, store in appropriately labeled containers with name date and contents. Do not pour any solvents down the drain.

RCA-1 clean [RCA01]
Setup time for this process is about 5 minutes. This process takes about 20 minutes to complete.

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RCA clean is used to remove organic residues from silicon wafers. In the process, it oxidizes the silicon and leaves a thin oxide on the surface of the wafer. The general recipe is for RCA-1 cleanser is: 5 parts water (H2O), 1 part 27% ammonium hydroxide (NH4oH), 1 part 30%hydrogen peroxide (H2O2). Here is how to prepare it.

325 ml DI water
65 ml NH4OH (27%)
65ml H2O3 (30%)

Put 325 ml DI water in a Pyrex beaker, add 65 ml NH4OH (27%) and then heat to 70 + /- 5 deg C on hot plate. Remove from hot plate and add 65 ml H2O2 (30%). Solution will bubble vigorously after 1-2 minutes, indicating that it is ready for use. Soak the silicon wafer in the solution for 15 minutes. When finished, transfer the wafer to a container with overflowing DI water from a tap to rinse the solution. After several water changes, remove the wafer under flowing water. (Still water surface can contain organic residue that will redeposit on the water surface when removing wafer.) To dispose of the RCA-1 solution, dilute with cold water then pour down the drain with plenty of cold water to flush. Old RCA-1 cleaning solution cannot be used since it loses its effectiveness in 24 hours at room temperature (30 minutes at 70 deg. C).

HF dip [HFDIP]
Setup time for this process is about 5 minutes. This process takes about 5 minutes to complete.

Hydrofluoric acid (HF) is used to remove native silicon dioxide from wafers. Since it acts quickly, one needs to only expose the wafer fro a short time ("dip"). HF is a dangerous chemical and protective gear must be worn when using it, in particular, *neoprene or thick nitrile gloves and eye protection must be worn.*
A prepared solution should already be available to you. If not, here is how to prepare a 2% solution.

480 ml water
20 ml HF (49%)

Wear heavy protective gloves and protective eye gear. Add 480 ml water to polyproplene beaker then, add 20 ml HF. Never use a glass beaker with HF since HF attacks glass. Label the bottle "2% HF solution-Dangerous acid!" then, add your name and the date.

Soak the wafer for 2 minutes in this solution. Remove the wafer and rinse in running DI water. Check for hydrophobicity by performing a wetting test. Pour a little DI water on the surface. If the water beads up and rolls off, the surface is hydrophobic and water will

not wet it. Since oxide is hydrophilic and pure silicon is hydrophobic, a non-wetting surface is clean of oxides. Blow dry with nitrogen and store in a clean, dry environment.

The 2% solution may be saved for other cleaning. (It does not lose its effectiveness). To dispose the 2% HF solution, pour into an INRF labeled waste container for 2% HF. Very small amounts of 2% HF may be disposed by diluting with cold water, then flushing down the drain with plenty of cold water.

Be sure to wash your neoprene gloves and return to their proper location when finished.

Safety and Emergency

All INRF safety and procedural regulations must be followed. Review the INRF standard operating procedures for fire, chemical spill, acid exposure and HF exposure.

Acetone and methanol are flammable liquids. Handle with care. Do not let the solvent exceed 55 deg. C. Do not store the solvents near the hotplate or any other source of heat. Hydrogen peroxide is an explosive chemical. Keep it away from all solvents. Never leave the RCA process unattended.

Hydrofluorifc acid (HF) is an extremely toxic and dangerous acid. Use of HF required at least one other person in the clean room. HF should be handled in a laminar flow bench, using two pairs of nitrile gloves (or neoprene) and eye protection. Any small spills should be wiped up immediately with wipes and rinsed. Dispose of the wipe in the corrosive waste container. DO NOT LEAVE the etchant unattended.

A special INRF standard operating procedure for HF has been prepared. Follow the INRF SOP for HF exposure (summarized below).

In case of exposure **Seek medical attention immediately!** For skin exposure, flush immediately with water for 5 minutes, followed by liberal application of calcium gluconate gel to the skin. Remove all clothing that are exposed before and while flushing with water. For eye exposure, flush the eye with three times, 5 minutes each. Irrigate the eye repeatedly with 500-1000 ml of a 1% calcium gluconate solution applied through a syringe. Call for prompt emergency room transport. Apply ice-water compresses during transport.

References

Kevin M. Walsh, *University of Louisville Standard Operating Procedures,* http://mitghmr.spd.louisville.edu/lutz/resources/sops/
Prudent Practices in the Laboratory, National Research Council, 1995.

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W. Kern and J. Vossen, *Thin Film Processes,* Academic Press: New York, 1978, Ch V-1
W. Kern and Ed., *Handbook of Semiconductor Cleaning Technology,* Noyes Publishing: Park Ridge, NJ, 1993 Ch 1.

Checklist
The following checklist is designed to aid the researcher when performing this process.

Solvent clean

Prepare solvent baths: acetone and methanol

Prepare RCA bath: 5 parts water (H2O), 1 part 30% hydrogen peroxide (H2O2), 1 part 27% ammonium hydroxide (10 minutes).

Soak wafer in warm acetone (10 minutes)

Soak wafer in methanol (5 minutes)

DI rinse (optional) and blow dry

Clean up, dispose wastes

RCA #1 clean

Soak wafer in RCA bath at 70 deg C for (15 minutes)

DI rinse and blow dry

Clean up, dispose wastes

HF dip

Neoprene gloves required for HF dip

Dip wafer in 2% solution, (2 minutes)

Wettability test

DI rinse / blow dry

Clean up, dispose wastes