

CMMED Wet Cleaning Bench Operating Procedure

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Failure to follow procedures can and will result in contamination that will ruin your experiment and ruin the experiments of everybody after you

Privileges to use the facility will be revoked if procedures are not followed precisely

Failure to follow correct safety procedures will result in loss of use privileges

All equipment use, including the cleaning bench, must be logged

The bench and glassware must be cleaned to original state after use

No Glassware nor chemicals shall be left on the bench after use, period

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General Overview of Cleaning Processes

RCA clean: This is the standard cleaning procedure used in Si MOSFET processing developed by RCA corp. RCA1 is a $\text{H}_2\text{O}:\text{NH}_4\text{OH}:\text{H}_2\text{O}_2$ solution that chemically attacks organic impurities. The basic conditions also slowly etch Si, which removes small particles strongly attached to surface. RCA2 is a $\text{H}_2\text{O}:\text{HCl}:\text{H}_2\text{O}_2$ solution that readily dissolves metal impurities, in particular Fe and Na. Both steps are critical to make high quality gate oxides and insure that the oxidation furnace does not become contaminated. Once a furnace is contaminated, it may never form high quality oxides again as extremely small amounts of impurities have high mobility at 1000C.

Piranha clean: This is a mixture of $\text{H}_2\text{O}:\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ is highly oxidative and removes metals and organic contamination. This clean is suitable for putting samples in a furnace without contamination, however a few guidelines need to be followed. If a sample has a large amount of organic impurities (i.e. photoresist) the piranha etch will form an insoluble organic layer that can't be removed. It is recommended to follow photoresist cleaning procedure below 1st if photoresist was used on the sample. Another important factor is that the RCA1 step removes most particles strongly attached to Si wafers by etching underlying Si/SiO₂. Thus the RCA clean is preferred for MOS fabrication. However the piranha clean can be applied to a large number of materials

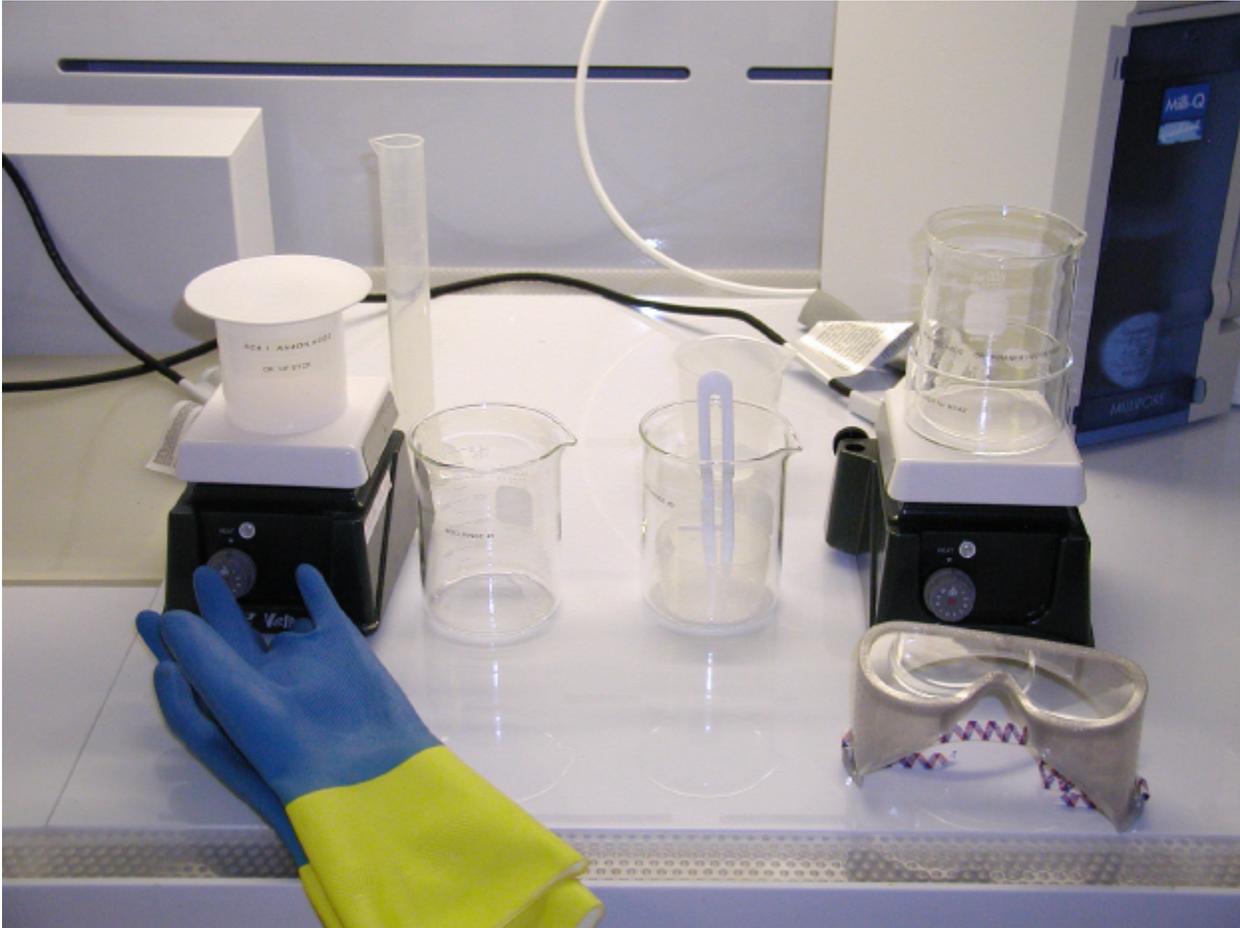
Photoresist/organic or liftoffstrip by boiling acetone: Shipley provides resist strip solution, however acetone works just as well. The reason the Shipley solution is used is because of low solvent vapor pressure that could interfere with the photo-resist. Acetone fumes will ruin a prepared sample so care must be taken to keep acetone in the fume hood while manipulating photoresist covered sample outside of the fume hood. Most organic contamination will be removed by boiling acetone for 5 minutes followed by DI water rinse. If photoresist is heated, it may polymerize and become insoluble, thus plasma etching is required. Excess photoresist can be observed in an optical microscope. Boiling acetone can remove photoresist below metal layer to 'lift-off' metal outside of the pattern of interest.

HF etch: **It is important to note that HF is extremely dangerous. If a spill occurs you may lose the exposed limb.** This is because HF destroys capillaries that supply blood to cells and gangrene can result. For the first few hours you will not feel anything unusual since the acid does not burn like HCl or H₂SO₄, it seems like water. If a spill occurs, treat the area with CaCl₂ solution (precipitates inert CaF₂) and get medical aid immediately. **It is extremely important to wear all safety wear!**

Since impurities may be in an oxide layer on top of Si wafer, a convenient way to further clean Si is to remove the relatively impure SiO₂ layer. HF etches are also commonly used as the last step to etch through SiO₂ to contact the Si wafer with your metal leads. Buffered HF (BHF) is NH₃/HF solution that has the highest SiO₂ etch rate, but can actually etch thin layers of Si. Another problem is that the high pH can precipitate metal contamination. Low pH unbuffered HF is much slower but the etch rate is highly variable. Importantly this does not etch Si, so can be used on very thin layers of Si.

Furnace care: The furnace is very easily poisoned by impurities. Impurities dissolve into the glass and release to ruin every sample after initial contamination (students thesis' become ruined). Your peers and friends demand that all rules are followed.

RCA Clean



Safety considerations: Proper safety attire when handling acid is thick chemical gloves, plastic apron with sleeves, and face shield or goggles. Boiling acid could ‘bump’ and splash, on to face and eyes. While acid is heating/boiling, the front of the fume hood sash must be down, preventing such accidents. Face shield must be in the down position when window sash is up or when pouring chemicals. Care must be taken not to mix concentrated acid and bases together.

General: This is the standard cleaning procedure used in Si MOSFET processing developed by RCA corp. RCA1 is a $\text{H}_2\text{O}:\text{NH}_4\text{OH}:\text{H}_2\text{O}_2$ (5:1:1) solution that chemically attacks organic impurities. The basic conditions also slowly etch Si, which removes small particles strongly attached to surface. RCA2 is a $\text{H}_2\text{O}:\text{HCl}:\text{H}_2\text{O}_2$ (5:1:1) solution that readily dissolves metal impurities, in particular Fe and Na. Both steps are critical to make high quality gate oxides and insure that the oxidation furnace does not become contaminated.

Equipment: Pictured below. Glassware in oxidation furnace kit are to be used only for new or previously cleaned wafers only! This is the cleanest set of glassware.

Procedure:

Turn on DI water purifier. Clean all glassware. Fill all beakers 1/3 full and swirl rinse 5 times. Insure wafer basket is in one of the beakers during rinse. Rinse graduated cylinder 5 times.

Never set Teflon tweezers on counter. Always place tweezers in one of the clean beakers. This easy to make mistake will contaminate everything! If tweezers are contaminated you must use this procedure to clean them, then make new solutions to clean the sample.

An optional Acetone clean of sample can be performed at this time if the wafer had previously been coated with photoresist or known to be in an environment with many organic chemicals.

An optional HF etch can be performed to remove the thin layer of oxide from Si wafer (and

Into 500ml PFT beaker add 20ml of electronics grade NH_4OH . (*Do Not use glass beaker as high pH will leach Na^+ from glassware and contaminate your sample/furnace*) Add 20 ml electronics grade H_2O_2 . Add 100 ml DI H_2O . Rinse graduated cylinder 3 times (for the first rinse of graduated cyl. pour waste into acid waste tub, other rinses can go into sink).

Into glass 500ml beaker add 250 ml DI H_2O . Add 50 ml electronics grade H_2O_2 . Add 50ml of electronics grade HCl . Rinse graduated cylinder 5 times with 20-30 ml DI H_2O . Mix chemicals in this order to avoid accidentally mixing acid and bases.

Set each heat plate to setting **7** and place beakers onto them. Ensure that both beakers are in a crystallization glass dish since solutions can boil over easily. Solutions should be visibly boiling. The hotplate is much hotter than the solution ($\sim 180\text{C}$, *so be careful not to put hot glassware on plastic counter (melting) or touch with hands/gloves*)

Place wafer into basket and place into $\text{NH}_4\text{OH}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ solution (RCA1) for five minutes. Transfer to one of the H_2O baths. Remove and place into other beaker for water. Turn off hotplate. Rinse previous beaker of water 5 times and half fill with DI H_2O . Transfer basket to freshly rinsed H_2O beaker and rinse 'dirty' beaker 5 times. Repeat transfer process 5 times.

Place wafer into basket and place into $\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ solution (RCA2) for five minutes. Turn off hotplate. Transfer to one of the H_2O baths. Remove and place into other beaker for water. Rinse previous beaker of water 5 times and half fill with DI H_2O . Transfer basket to freshly rinsed H_2O beaker and rinse 'dirty' beaker 5 times. Repeat transfer/rinse process 5 times.

At this time an optional HF etch may be performed to remove thin chemical oxide on Si wafer.

Dry wafer with N_2 gun. The wafer is now very clean

Let solutions cool 5 minutes. Pour waste RCA1 and RCA2 solution into the same container to neutralize acid/base. Make sure that waste container has pressure relief, as H_2O_2 can build up hazardous pressure.

Piranha Clean

Safety considerations: Proper safety attire when handling acid is thick chemical gloves, plastic apron with sleeves, and face shield or goggles. Boiling acid could 'bump' hitting face and eyes. While acid is heating/boiling front of fume hood sash must be down, preventing such accidents. Face shield must be in the down position when window sash is up or when pouring chemicals. Make sure that no organics (especially acetone) are in the fume hood with boiling H_2SO_4 .

Piranha clean: This is a mixture of $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ (10:1) is highly oxidative and removes metals and organic contamination. This clean is suitable for putting samples in a furnace without contamination, however a few guidelines need to be followed. If a sample has a large amount of organic impurities (i.e. photoresist) the piranha etch will form an insoluble organic layer that can't be removed. It is recommended to follow photoresist cleaning procedure below 1st if photoresist was used on the sample. Another important factor is that the RCA1 step removes most particles strongly attached to Si wafers by etching underlying Si/SiO₂. Thus the RCA clean is preferred for MOS fabrication. However the piranha clean can be applied to a large number of materials.

Procedure:

Unless this clean is for oxidation furnace, use the other set of glassware. This will keep the oxidation cleaning kit as clean as possible

Turn on DI water purifier. Clean all glass ware. Fill all beakers 1/3 full and swirl rinse 5 times. Insure wafer basket is in one of the beakers during rinse. Rinse graduated cylinder 5 times.

Never set Teflon tweezers on counter. Always place tweezers in one of the clean beakers. This easy to make mistake will contaminate everything! If tweezers are contaminated you must use this procedure to clean them, then make new solutions to clean the sample.

Set heat plate to setting 7 and place beakers onto them. Ensure that $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$ is in a crystallization glass dish since it boils over easily. Solution should be visibly boiling.

Place wafer into basket and place into $\text{HCl}:\text{H}_2\text{O}_2:\text{H}_2\text{O}$ solution for five minutes. Transfer to one of the H_2O baths. Remove and place into other beaker for water. Rinse previous beaker of water 5 times and half fill with DI H_2O . Transfer basket to freshly rinsed H_2O beaker and rinse 'dirty' beaker 5 times. Repeat transfer/rinse process 5 times.

Dry wafer with N_2 gun. The wafer is now very clean

Let solution cool for 15 minutes. Slowly pour into acid/base waste bucket.

Photoresist/organic or liftoff strip by boiling acetone

Safety considerations: Proper safety is cleanroom suit and protective eyewear. Boiling acetone could 'bump' hitting face and eyes. While acetone is heating/boiling front of fume hood sash must be down, preventing such accidents. Make sure that no strong oxidizers (ie. Parhana clean, concentrate H₂O₂) are in the fume hood at the same time.

General: Shipley provides resist strip solution, however acetone works just as well. The reason the Shipley solution is used is because of low solvent vapor pressure that could interfere with the photo-resist. Acetone fumes will ruin a prepared sample so care must be taken to keep acetone in the fume hood while manipulating photoresist covered sample outside of the fume hood. Most organic contamination will be removed by boiling acetone for 5 minutes followed by DI water rinse. If photoresist is heated, it may polymerize and become insoluble, thus plasma etching is required. Excess photoresist can be observed in an optical microscope. Boiling acetone can remove photoresist below metal layer to 'lift-off' metal outside of the pattern of interest.

Procedure: Fill beaker with 100-300ml of acetone. If doing metal liftoff procedure be sure to use only the labeled 'liftoff' beaker. Set on hotplate at "4" setting and wait to boil. Add basket as 'boiling chip. Rinse DI H₂O beaker 5 times. Add sample into basket and boil for 2-3 minutes. Remove acetone beaker from hotplate and place basket/sample in DI water. Alternately transfer/wash/rinse beakers 3 times. Pour waste into acetone waste bottle. (Do not mistakenly pour into acid waste as this is potentially flammable, if this occurs make sure that the small cap is unfastened to relieve pressure.)

HF etch

Safety precautions: It is important to note that HF is extremely dangerous, if a spill occurs you may lose the exposed limb. This is because HF destroys capillaries that supply blood to cells and gangrene can result. For the first few hours you will not feel anything unusual since the acid does not burn like HCl or H₂SO₄, it seems like water. If an exposure to skin occurs, treat the area with CaCO₃ solution (precipitates inert CaF₂) and get medical aid immediately. **It is extremely important to wear all safety wear! You must wear acid gloves, plastic apron over the cleanroom suit, eyewear, and a faceshield.** HF must be disposed of in proper container.

For large spills of HF acid, immediately evacuate the room and keep others from entering. Call hazardous waste. For smaller spills use paper towels and seal in plastic bag. Double seal bag inside another plastic bag and label. Fill out waste disposal ticket and inform CMMED staff.

General information: Since impurities may be in an oxide layer on top of Si wafer, a convenient way to further clean Si is to remove the relatively impure SiO₂ layer. Buffered HF (BHF) is NH₃/HF solution that has the highest SiO₂ etch rate, but can actually etch thin layers of Si. Another problem is that the high pH can precipitate metal contamination. Low pH unbuffered HF is much slower but the etch rate is highly variable. Importantly this does not etch Si, so can be used on very thin layers of Si. HF etches are also commonly used as the last step to etch through SiO₂ to contact the Si wafer.

Procedure: Pour required HF directly into the Teflon PFT beaker and add sample for the desired time. Rinse sample in 1/4 full (200-300ml) glass DI H₂O beaker and transfer to second glass DI H₂O beaker. Pour the HF acid and the first DI rinse into the HF etch. Rinse 1st DI H₂O beaker 3 times and fill with 200-300ml DI H₂O. Alternate DI H₂O rinses 5 times.