Micro/Nanofabrication Facility

Cleanroom Training

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CLEAN ROOM LAYOUT
The clean rooms are divided into two spaces:

- The MicroFab (uFab) Clean Room divided into the optical lithography area and the wet etch area (acid hood)
- The NanoFab Clean Room (special permission is necessary to use this room)

For safety, and to keep particle counts low, the maximum number of occupants on the nanoFab side is 4 people.

GENERAL CLEANROOM INFORMATION, EQUIPMENT, AND PROCEDURES

- A keycard reader is at the door (381 or 381C MRL). Hold your keycard near the reader, wait a second, then enter.
- No short pants or open-toed footwear is allowed inside the gown-up area or inside the clean rooms.
- No food or drinks are allowed inside.
- Each user MUST wear head cover, safety eyewear (your prescription glasses DO NOT count), a gown, and a pair of clean room boots. The preferred garb is a one-piece garment with integrated boots and head cover. These are available in the MRL store room. This garb will require periodic replacement with normal use. If your garb is damaged or worn, you must replace it.
- You are required to write your user number and your name on the back of your gown with the large marker in VERY LARGE PRINT. When you replace your garb, you must repeat this.
- You may only wear YOUR garb. Do not disturb other users’ garments.
- You must also wear your USER BADGE on the outside front of your clean room garb at all times when in the clean room.
- Safety glasses are required at all times while in the cleanroom, even if you wear corrective eyewear.
- Ultraviolet-blocking safety glasses: Users should buy their own UV safety glasses, if desired. Confirm that they are the yellow-tinted, UV-blocking glasses (UV wavelength < 400nm).
- Store your clean room garb on a hangar located in the gown-up room.  You must reserve the hangar on a monthly basis.
- Users must log in using the computer in the gown-up area before entering, and must log out when leaving the clean room. This is an important safety measure.
- The room lights are filtered to block wavelengths shorter than 550nm (yellow). This prevents unwanted exposure of photoresist.
- Nitrile gloves (L&S) are available in the gown-up room. Users may bring their own powder-free gloves into the clean room.
- Do not bring Kleenex, card board, or any particle-producing products into the clean room. Do not rip paper.
- Several areas are available for open chemical work in the facility clean rooms (note that photoresist before bake out is considered an open chemical due to strong, unpleasant fume emission):
  A) One vented flow bench and two vented spin coaters on the uFab side in the optical lithography area.
  B) One chemical fume hood (the acid hood) in the wet etch area of the uFab side for working with HF, BOE, and Piranha.
  C) One small, vented flow bench on the nanoFab side.

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D) One chemical fume hood for acid work on the nanoFab side.
E) In addition several vented trash cans are provided for PR-contaminated waste (foil, pipettes, wipes).

- Acids may only be used in a chemical fume hood. Do not use acids in any of the flow benches.

- Benches/work areas that are not vented and are not for open chemical work include:
  A) Mask Aligner isolation tables (uFab lithography side and nanoFab side)
  B) Optical Microscope table (ufab lithography side and nanoFab side)
  C) Lab work benches throughout the facility

- If a user is signed-in on a piece of equipment or set up in a fume hood or flow bench, you should obtain permission from that user before working in that area. This equipment operates on a first-come, first-serve basis. Do not monopolize all the hot plates for yourself.

- Deionized (DI) Water is available from flow bench faucets. Let the water run a little to increase resistivity. Do not waste DI water. Turn off DI water once you are done.

- Cole Parmer hot plate temperature adjustment: Press the “SET” button (it lights up), then press the “TEMP” button (it lights up), then enter the temperature. To check the temperature, the “SET” light should be off. Push the “TEMP” button. It will light up and the display shows the current temperature. Other options work the same way.

- Cimarec hot plates: “TEMP” stops flashing when up to the set temperature.

- A refrigerator is available for chemical storage. Do not leave unused items in the refrigerator. Your items MUST BE LABELED, and must be stored in vapor-tight, sealed containers.

- Storage cabinets are provided to store small numbers of items such as glassware, solutions tools, and samples that you use often. You must store your items in a tub like the other tubs in these cabinets. These tubs are available in the MRL store room. Do not overfill tubs, or store unused items. Tubs must be marked with your user number and name on the end facing out so it is visible. Storage cabinets are subject to inspection. Users who do not satisfactorily maintain these spaces will be cited and risk losing clean room access.

- NO open containers of liquids are permitted in the storage cabinets. Storage containers must be vapor-tight. Containers covered with foil are unacceptable. Solutions that cannot be stored in sealed containers MAY NOT be stored in the clean room cabinets. Clean up any spills in your tubs.

- Dropper bottles are NOT allowed anywhere in this facility.

- All unused, expired, and forgotten items (personal and group items) must be removed and properly disposed.

- Do not leave items lying around in the clean room without a good purpose that is obvious to others.

CLEANROOM SAFETY EQUIPMENT
- Fire Extinguishers are located inside the uFab door, inside the nanoFab door, and outside, at the east door to the gown up area.
- To operate a fire extinguisher, break the pull tab by pulling out the ring. Point the nozzle and squeeze the handle.

- Emergency pull shower is located in the hallway outside the clean room. An emergency exit door is near the sink in the wet etching area (uFab side, near the acid hood).

- The sink near the wet etching area has a hand-operated shower nozzle for emergency rinse-off or eyewash. Foot pedals allow for general use such as cleaning glassware. Do not touch these items with contaminated gloves.

- First Aid Kits are located in several areas, including near doorways.

- Spill kits: Acid and Base neutralizing solutions are located on the refrigerator (uFab side). Spill kit materials are located in the first aid cabinet outside the clean room (outside the east side door to the gown-up area)

GENERAL RULES FOR USE OF CHEMICALS
- Any “new” chemicals brought into lab, must first be approved by Staff. A Material Safety Data Sheet (MSDS) must be supplied by the user. The MSDS binder is located on the wall in the gown up area, near the uFab clean room door.

- The chemical log book must be filled in when bringing any chemical(s) in to the clean rooms. You must confirm that there is a MSDS for the chemical(s) you wish to bring in.

- CHEMICALS MAY NOT BE LEFT UNATTENDED IN WORK AREAS FOR MORE THAN 15 MINUTES.
  - If your process requires chemicals to be left unattended for longer than 15 minutes:
    - Notify Staff to get permission
    - You must add expected date and time of disposal on the label

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Labels must be legible
- The maximum time for unattended chemicals is 48 hours, subject to approval
- Dispose “Sharps” (glass slides, razor blades, etc.) in proper Sharps containers. **NEVER** dispose chemicals, PR-contaminated items, tissues, trash, plastic containers, pipettes, etc. in Sharps containers.
- **Empty Bottles** must be **triple-rinsed** and placed by the trash can.

**LABELING REQUIREMENTS**
All chemicals, glassware, etc., must always be labeled.
- Include **User Name**
- Include **User Number**
- Include **Group (PI) Name**
- Include **Contact Information: email or phone number**
- Include **Chemical name (use the chemical formula, not a common name).** For example, don’t label using the term “Aqua Regia.” Label as: “25% HNO3 + 75% HCl.” (That’s 25% Nitric Acid + 75% Hydrochloric Acid.)
- **Label ALL chemicals, even water (a clear liquid is often NOT water).**
- A **Label-printer and markers** are provided in the clean room to ensure that your containers can be labeled - and relabeled when the markings wear off. You are responsible for determining when relabeling is necessary.
- Very small containers that cannot be labeled must be kept in a larger, labeled container.
- **Containers that are not properly labeled will be disposed.** Any users caught violating this rule may be suspended from the clean rooms.
- Any person found working with an unlabeled substance in the facility will be cited.

**GENERAL CHEMICAL WASTE DISPOSAL GUIDELINES**
Dispose chemicals in appropriate waste bottles shown below. Most of these are stored in the chemical cabinets.
- **Halogenated Solvent waste** (F, Cl, Br), including Trichloroethane, Trichloroethylene
- **Non-halogenated Solvent waste** such as Acetone, Isopropyl Alcohol, 2-Propanol, Methanol, Ethanol, Propylene Glycol Methyl Ether Acetate (PGMEA), Methyl IsoButyl Ketone (MIBK).
- Non-halogenated solvents that have small amounts of photoresist in them go in the non-halogenated solvent waste container.
- **Photoresist** is a non-halogenated waste but goes in its own waste container or in the container for PR-contaminated Sharps. Do not put any PR-contaminated items in the regular waste containers; PR fumes will fill the room.
- **Developer waste** (generally Alkaline)
- **RCA 1** waste is defined as: H2O2 – NH4OH – H2O (an Alkaline solution)
- **RCA 2** waste is defined as: H2O2 – HCl – H2O (an Acidic solution)
- If your definition of RCA 1 or RCA 2 is different from the above, **DO NOT** use these waste. You must contact Staff to get an appropriate waste container.
  * RCA1 is also known as “Standard Clean 1” or “SC1.” RCA2 is also known as “Standard Clean 2” or “SC2.”

**DO NOT** overfill trash containers

*If you will generate any waste not identified above, you must first contact Staff for a new waste bottle. It will be labeled with the chemical composition and the starting date of waste generation.*

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CHEMICALS AND CONSUMABLES SUPPLIED

ACIDS:
- CE-5 M Chromium Mask Etchant with Surfactant (Perchloric Acid [HClO4] & Ammonium Nitrate)
- Iron Oxide Mask Etchant ME-10 (Hydrochloric Acid [HCl] & Ferrous Chloride)
- Hydrofluoric Acid (HF)
- Nitric Acid (HNO3) in the storage cabinet under the HF & BOE fume hood
- Hydrochloric Acid (HCl)
- Sulfuric Acid (H2SO4)
- Phosphoric Acid (H3PO4)

BASIC/ALKALINE chemicals:
- Potassium Hydroxide (KOH)
- Preferential Silicon Etchant (a Potassium Hydroxide solution)

Chemicals (SOLVENTS):
- Acetone
- Isopropyl Alcohol or 2-Propanol (IPA)
- Methyl Alcohol (Methanol)
- Methyl IsoButyl Ketone (MIBK)
- AZ 351 Developer (Sodium Borates & water)
- AZ 917 MIF Developer (Tetramethylammonium Hydroxide)

Refrigerator:
- AZ 5214-E IR photoresist bottles stored in the refrigerator or on the table are available for purchase. A bottle may be purchased by choosing an unclaimed bottle from the new bottle shelf and writing your name on the 5214 PR purchase sheet. ($30 / 8oz - 125ml bottle) Be sure to label your bottle as required!

You must see Microfab Staff if you want to use Chlorobenzene, PMMA-c,* or any other resists or chemicals that contain benzene (examples include Toluene, Xylene) in the cleanroom. * PMMA-c is Polymethylmethacrylate with chlorobenzene. Consider using PMMA-a. It contains anisole instead of chlorobenzene and is a safer substitute for PMMA-c.

ACID HOOD GUIDELINES

- All users must be trained by Staff before using the Acid Hood
- A buddy system is required at all times (especially during 5PM–9AM).

The Acid Cabinet for bulk storage and for waste storage is located the Wet Etch area. NO Nitric acid may be stored in this cabinet.
- Use the Top part for storage of bulk (large) acid bottles.
- Use the Lower part for storage of chemical waste and more bulk acid storage.

- The HF and BOE waste container is located in the fume hood (the acid hood) - not in this cabinet.
- Nitric acid must be stored separately in its own tub and container under the acid hood – not in this cabinet.

HF AND BOE USE

- HF may only be used in the HF (& BOE) designated use area. This is on the uFab side, in the Wet Etch fume hood (the acid hood). HF and BOE may NOT be used anywhere else in the facility.
- Before you begin using HF or BOE, make sure that the waste bottle has enough space to accept the waste that you will create. You may not overfill the waste bottle. If the bottle is (or will be) too full, You may NOT begin until this is corrected.

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HF BACKGROUND INFORMATION

- Concentrated HF is considered "extremely" toxic (4 out of 4 on the health hazard scale). A plain, concentrated ammonium fluoride solution (NH4F) is considered "very" toxic (3 on the health hazard scale), yet becomes "extremely" toxic when made more acidic, such as in BOE mixtures. For example 20:1 BOE has much less HF (about 7% of volume) than 49% HF. Because it also contains about 38% NH4F and it is acidic, it presents the toxic hazards of 49% HF.

- On contact, HF easily passes through skin and tissue. Because its action can be delayed for many hours, it can distribute throughout the body.

- Negatively charged fluorine ions bind very easily to positively charged calcium and magnesium ions to form insoluble salts. In the body, Ca and Mg ions are used to mediate a variety of physiological processes such as muscle movement. Calcium is also a chief component in bone. The capture of these ions by fluorine ions interferes with these important processes.

- Local tissue damage (at the point of contact) results from free hydrogen ions which cause corrosive chemical burns and from free fluorine ions which cause deep tissue damage, including erosion of bone.

- Systemic damage can occur when fluorine becomes distributed throughout the body. These conditions include hypocalcaemia (loss of calcium) and hyperkalemia (too much potassium). Since calcium and potassium regulate heartbeat, irregular heartbeat and cardiac arrest are possible! Deaths have been reported from concentrated acid burns to as little as 2.5% body surface area.

- Pure hydrogen fluoride is an extremely toxic gas which very easily dissolves in water. This solution is hydrofluoric acid. HF easily passes between the gas and liquid phases, so HF- and NH4F-containing solutions will emit toxic fumes. Avoid inhalation of the fumes by always working inside the acid fume hood (never in the flow benches).

- Exposure to HF is treated by applying Calcium Gluconate gel. This gel provides calcium ions which can scavenge free fluorine ions before they penetrate and damage tissue. In cases where systemic damage is a risk, calcium gluconate is administered intravenously by a healthcare professional.

HF AND BOE HANDLING

- HF & BOE should only be kept in Nalgene containers because they will etch silicon-based containers. (Glass & Pyrex are SiO2-based).

HF AND BOE PPE GUIDELINES

- DO NOT enter the hood area without wearing additional PPE as described below.

- You MUST wear a Full Face Shield when working with HF and BOE.

- You MUST wear an Acid Apron when working with HF and BOE. Aprons are located near the acid hood.

- You MUST double-glove when handling HF and BOE. This means that you MUST wear acid-resistant neoprene gloves over your nitrile gloves. These Neoprene gloves are located near the acid hood on the wire rack and next to the sink in a wire basket. They are also available in the MRL store room. Do not put your bare hands into the neoprene gloves; always double-glove.

HF & BOE STORAGE

- Always store HF in closed Nalgene containers in the acid storage cabinet located next to the acid fume hood.

- Store BOE in the supplied (Nalgene) containers in the acid cabinet

HF EMERGENCY PROCEDURES

- If you are exposed to HF, IMMEDIATELY apply Calcium Gluconate Gel to the area of contact. Calcium Gluconate Gel is located on TOP of the base storage cabinet - just to the right of the acid fume hood.

- Seek medical attention immediately (call 9-911 and report that you have been exposed to hydrofluoric acid) and notify Microfab Staff.

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HF WASTE DISPOSAL

- There is a separate container in the acid work area for HF and BOE\(^*\) waste. It is marked for this purpose.
  \(^*(6\text{ to }1\text{ NH}_4\text{F to HF})\)
- Do not dispose any BOE in the HF waste container that is a different chemical formula than NH4F + HF (ammonium fluoride + hydrofluoric acid). Other BOE etch solutions may be different and may create a safety hazard when combined.

PIRANHA OPERATIONAL AND SAFETY INFORMATION

Piranha use is being discontinued. Only current, APPROVED users may use piranha. No new users are allowed to use piranha.

Piranha may only be used Monday through Friday between 8am and 6pm.

Piranha is commonly a 3:1 or 5:1 mixture of concentrated sulfuric acid and 30% hydrogen peroxide. NEVER USE HYDROGEN PEROXIDE IN CONCENTRATIONS OVER 30%. THIS MAY CAUSE AN EXPLOSION!

PIRANHA SHOULD BE HANDLED WITH EXTREME CARE. ITS COMPONENTS ARE ACIDIC AND HIGHLY CORROSIVE. THEY REACT VIOLENTLY WITH MOST ORGANIC MATERIALS.

PIRANHA CONTAINER SEALS MUST BE VENTED TO PREVENT BUILD UP OF PRESSURE. DO NOT STORE PIRANHA IN SEALED CONTAINERS.

Mixing Sulfuric Acid and Hydrogen Peroxide H2O2 causes two different chemical reactions which can accelerate out of control. The solution may foam out of the beaker or explode with a huge shock wave. Glassware and other objects can become sharp flying objects that can inflict serious injury - even though you’re wearing protective clothing.

PIRANHA HANDLING

THE MIXING AND USE OF ANY HAZARDOUS OR CORROSIVE SUBSTANCE (eg. PIRANHA) REQUIRES THE PRESENCE OF A SECOND MICROFAB TRAINED USER AT ALL TIMES (BUDDY SYSTEM).

- When your buddy is using Piranha, keep track of his/her movements and take immediate action in case of an emergency. Remember, you are there to help them react correctly when an accident occurs. **They will be counting on YOU!**
- Conversely, if you are using Piranha, make sure your buddy is still with you as you perform your work.
- **Always use a FULL FACE SHIELD, ACID APRON, and ACID RESISTANT GLOVES when handling piranha.** These guidelines are the same as the HF & BOE PPE guidelines above.
- Fill in the piranha logbook with time, date, name, user number, concentration of each piranha mixture made, quantity of each piranha mixture made, substrate, piranha disposal time, and waste bottle swirl.
- Use only Pyrex containers. MAKE SURE CONTAINERS ARE VERY CLEAN AND HAVE NO ORGANIC OR OTHER RESIDUES.
- All containers must be CLEARLY LABELED with required information (see labeling section) visible to any user working or entering the acid hood area.
- **DO NOT PLACE WASH BOTTLES OR OTHER GLASSWARE CONTAINING ORGANIC COMPOUNDS SUCH AS ACETONE, ALCOHOL, ETC. IN THE ACID HOOD WHEN USING PIRANHA.**
- The fume hood sash must be lowered to the working height shown AND the splash shield must be placed between you and the solution.
- **BEFORE you create a piranha solution, make sure that the piranha waste container has enough empty volume to accept your solution without passing the 3/4 full level. If it does not, you MUST contact Staff for a new waste container. NEVER proceed otherwise.**
- Mix only the minimum amount of solution necessary. **A MAXIMUM OF 250 mL is permitted.**
- To create a piranha mixture, first add sulfuric acid to the Pyrex container. Next, SLOWLY add peroxide to the acid. This will produce an exothermic reaction (the temperature may rise above 100°C) with gas release.
- Place your sample in the solution for the required time and then remove it.

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• After piranha etch is complete, the substrate should be thoroughly rinsed with DI water and dried. Do not splash the DI water into the container holding your piranha mixture!
• Leave the hot piranha solution to cool off.
• Adding any acids or bases (including photoresist) to piranha, or spraying it with water will accelerate the reaction.
• Mixing hot piranha with organic compounds such as acetone, photoresist, isopropyl alcohol, or nylon may cause an explosion!
• Any accident or spill of >50 ml involving the use of Piranha solutions must be reported without delay to MicroFab Staff.

PIRANHA STORAGE
• Do not store piranha. Mix fresh solution for each use.

PIRANHA WASTE DISPOSAL
• Leave the hot piranha solution until it cools to a temperature of 40°C. Do not remove your labels.
• NEVER ADD HOT PIRANHA SOLUTION TO THE PIRANHA WASTE CONTAINER. THIS MAY CAUSE AN EXPLOSION!
• Never close the piranha waste container. Piranha stored in a closed waste container can cause an explosion due to pressure buildup.
• Once a temperature of 40°C has been reached, the peroxide should be mostly depleted. The solution can then be transferred to the glass piranha container for waste storage. The container is clearly labeled with the solution name and composition.
• CHECK THE WASTE CONTAINER TO ENSURE THAT IT HAS SUFFICIENT EMPTY VOLUME FOR YOUR WASTE PIRANHA SOLUTION. IF THE WASTE CONTAINER APPEARS 3/4 FULL, DO NOT ADD SOLUTION TO THE CONTAINER! CONTACT MICROFAB STAFF FOR A NEW PIRANHA WASTE CONTAINER.
• Gently swirl the waste bottle to mix the piranha waste and then set it back in its place. DO NOT lift the waste bottle if it is too heavy. Ensure that no heat or gas is evolved from the waste bottle after swirling. If this happens, contact Staff immediately.

PIRANHA EMERGENCY PROCEDURES

Emergency showers are located at the SW corner of room 384 (on the sink), and in the hallway outside room 384.
• In case of skin contact, flush with copious amounts of water for at least 15 minutes. Seek medical attention to assess the extent of skin burns.
• In case of eye contact flush contaminated eye(s) immediately with copious quantities of water for at least 15 minutes. You must seek IMMEDIATE medical attention. Have your buddy CALL 911.
• In case of inhalation get to an area with fresh air – your buddy may have to help you. Seek medical attention in the event of respiratory irritation, cough, or tightness in the chest. These symptoms may be delayed.

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