NANOLAB ORIENTATION

October 6, 2014

NANOLAB OPERATIONS

- **STAFFING**

* Prof. Rob Candler	-Lab Director		
* Tom Lee	-Interim Manager	x64641	14-131A, EIV
* Wilson Lin	-Interim Asst. Manager	x68923	14-131A, EIV
* Noah Bodzin	-FIB/SEM	x33312	1129, EV
* Huynh Do	-PECVD, CMP	x64641	14-131A, EIV
* Hoc Ngo	-Metals Dep, Dry Strip	x65528	14-131A, EIV
* Joe Zendejas	-Mask Shop/2nd shift	x65528	14-131A, EIV
* Max Ho	-Safety, AFM	x49329	18-132, EIV
* Jamie Kahng	-Lab Administrator	x69908	56-125, EIV
* Brian Matthews	-IT, LabRunr	x65528	14-131A, EIV
* Lab Assistants			
	Nanolab	x69597	

- **LAB HOURS (PHONE X69597)**

 * 7 AM to 8PM weekdays and weekends (requires building permit after 8PM)
 * 1st floor EIV (18-132)
 * Buddy system enforced

- **LAB ACCESS QUALIFICATION**

 * Graduate UCLA students with support from Professor
 * Undergraduate students with Prof. support and Lab manager permission
 * Outside industry or other university researchers with proper paperwork

STEPS TO USE NANOLAB

1. **Attend The Fundamental Lab Safety course (offered by EH&S)**
2. **Watch the Orientation Video on-line**
3. **Study Lab Usage Guide (Online:www.nanolab.ucla.edu)**
4. **Bring signed New User Application with recharge ID and Chemical SOP Sign-off forms to the test.**
5. **If necessary, bring $25 check (nonrefundable) payable to UCLA Nanoelectronics Research Facility to the test. Professors can opt to charge the fee to their account.**
6. **Pass safety test based on Lab Usage Guide and Orientation Presentation Notes.**
7. **Attend Lab Walkthrough (2.0Hrs).**
8. **Badge allows lab access, but you still need training on specific machines**

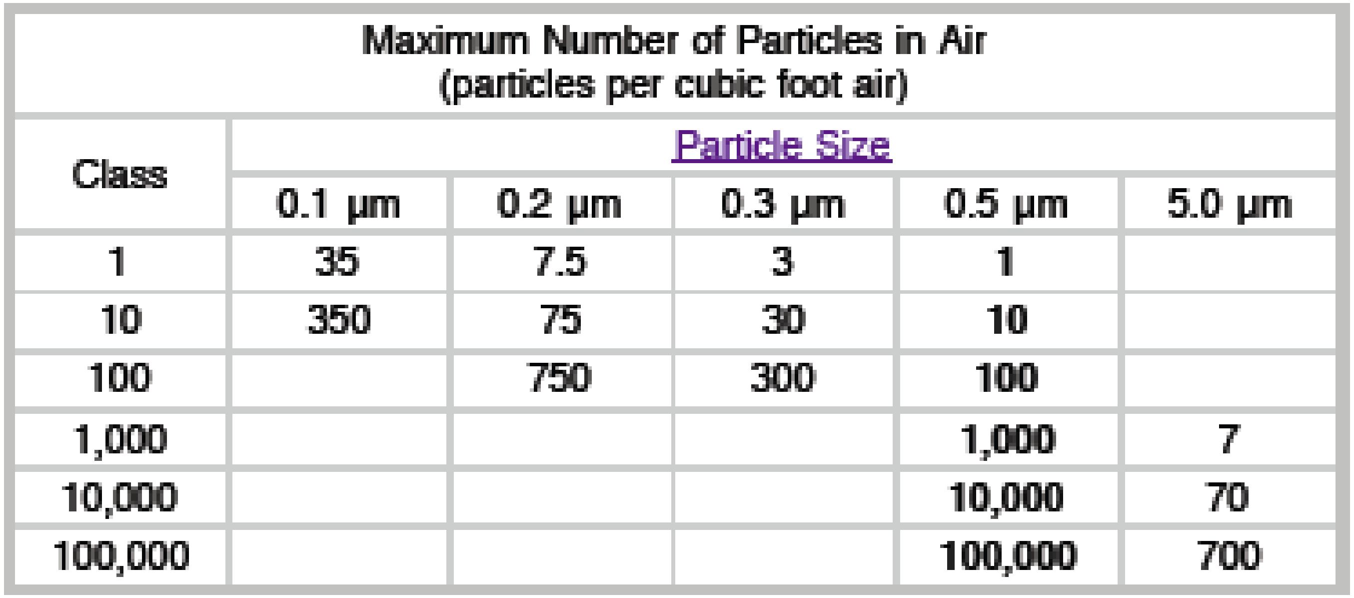
**** Industry users do not need to take test or bring check but DO need to sign new user form, attend Lab Walkthru and present a valid PO # for not less than $900***

NANOLAB DESCRIPTION

- ~8000 sq ft class 1000 area
- Temperature and humidity controlled environment
- Yellow room lithography area
- Separate class 100 room for AFM and SEM
- Badge-reader entry with real time user status
- Major equipment accessed thru touch screen interlock system
- Internet connections thruout lab for email and research
- Wafer pieces, 3 inch and 4 inch substrate processing (6 inch for a few processes eg furnaces)
- Lab accommodates III-V, Si and other substrates eg ferroelectrics
- Some equipment is dedicated for certain processes or substrates; Observe all signs
- Segregated hazardous chemical storage cabinets
- Specialized utilities throughout lab including: high purity deionized water, high purity gaseous and nitrogen drops, equipment cooling water, exhaust ducting etc.
- Remote toxic gas bunker with 24 hour-hazardous gas detection
- 7 fume hoods for working safely with chemicals

NANOLAB DESCRIPTION

Clean Room Classifications



Maximum Number of Particles in Air (particles per cubic foot air)					
Class	Particle Size				
	0.1 µm	0.2 µm	0.3 µm	0.5 µm	5.0 µm
1	35	7.5	3	1	
10	350	75	30	10	
100		750	300	100	
1,000				1,000	7
10,000				10,000	70
100,000				100,000	700

NANOLAB CAPABILITIES

LITHOGRAPHY

*** 2 Karl Suss MA6 contact aligners up to 6 inch wafer**
with top and bottom side alignment
*** Obducat NIL 6 imprinter**
*** 2 Thick PR Spinners with full programmability and ramp control**
*** Resist baking (Hot plate or oven)**
*** Microscope inspection, bright, dark field & Nomarski**
with video capture
*** Karl Suss SB6 Bonder (pieces up to 4 inch)**
*** Wet develop, etching, stripping & cleaning in custom wet benches**
*** HF/BOE acid etching (special training required)**
*** Heidelberg DWL66 LaserWriter for photomask making and direct-write**
*** Nova 600 FIB for submicron cutting, etching, TEM prep, and Pt, W, C deposition**

METALLIZATION

*** CVC 3 Target Sputtering with RF Etch capability (10 wafer load)**
*** 3 Electron beam evaporators ; Sloan and 2 CHAs, one with a**
thermal source. Both have 6 pockets for multilayer deposition
*** Denton Discovery 550 Research Sputterer with co-sputter and reactive**
sputter capability

NANOLAB CAPABILITIES

METROLOGY

* Hitachi S4700, Cold Field Emission SEM with X-RAY Attachment
* Veeco NanoMan Atomic Force Microscope
* Sopra GES 5 Spectroscopic Ellipsometer-multi wavelength/multi-angle
* Ulvac UNECS-2000 High-Speed Spectroscopic Ellipsometer
* Veeco NT3300 Optical Profiler for non-contact thickness measurement
* Filmtek 2000 for measuring thin layers and film stacks
* 2 Nanospecs to measure thin film thickness (transparent films)
* 2 Profilometers (Dektak 8 programmable & Dektak 6 manual)
* Flexus film stress measuring system
* CDE ResMap 178 4 point probe with mapping capability
* Micromanipulator probe station with HP4145 tester

DRY ETCHING

* STS AOE ICP Thick Dielectric Etcher
* Ulvac NLD 570 Dielectric Etcher for deep (>10µm) Glass Etching
* UNAXIS DSE II 3rd generation silicon etcher with SOI etch capability and smooth sidewall etch profile
* Unaxis SLR 770 for Deep Silicon RIE using ICP/Bosch process
* Unaxis SLR 770 Chlorine chemistry, ICP etcher of III-V & Al etching
* Oxford 80+ for RIE etching of oxides and nitrides using F chemistry
* XeF2 silicon etcher under computer control for multi-pulse Si etching
* Technics fluorine RIE etcher for removing oxides and nitrides
* Matrix 105 downstream, low-damage, high rate, plasma asher
* Oxygen Plasma Asher (Tegal 515 Barrel Etcher)

NANOLAB CAPABILITIES

THERMAL PROCESSING

- LPCVD furnaces with 6 inch capability for Phos doped LTO, doped polysilicon and silicon nitride (CMOS or low stress)
- Wet/Dry Thermal Oxidation for up to 6 inch substrates
- 2nd Oxidation/anneal tube for heavy doped substrates
- 2 Modular Process Rapid Thermal Annealers (Si and III-V)
- High Temperature (1200° C) Oxidation and Anneal-6 inch capable

LOW TEMPERATURE DEPOSITION

- STS Multiplex with stress control for silicon nitride. Recipes for oxides and amorphous silicon at Temp about 300C.
- PlasmaTherm 790 PECVD deposition system for oxides and nitrides
- Cambridge Savanah 100 ALD for aluminum oxide and hafnium oxide
- Ultratech Fiji Thermal and Plasma ALD for aluminum oxide, hafnium oxide, and zinc oxide
- BMR HiDep PECVD for silicon nitride and silicon oxide

MISCELLANEOUS

- Critical Point Dryer with 6 inch substrate capability
- Logitech CDP polishing and deplanarizing system
- Logitech PM 5 Lapper
- Sputter precoater for SEM imaging
- Porous Si etch setup with illumination and bias capability

CLEAN ROOM PROCEDURES

- **SUPPLIES**

  * Some supplies (eg substrates, glassware, safety gloves) supplied by users
  * Gowns, aprons, visors etc supplied by NanoLab
  * Nanolab supplies: acetone, alcohol, BOE, HF, HMDS, AZ5214E IR &AZ 4620 resists, AZ 400K developer, AZ Thinner, Aleg 380 Stripper, SU-8 resist and developer, PMMA, MIBK developer, Sulfuric Acid,Hydrogen Peroxide, AZ Developer, NH4OH, HCl
  * Au or Pt sources furnished for ebeam with extra charge. Al & Ti provided at no extra charge.

- **GOWNING/STORAGE**

  * Launderable jumpsuits and boots with hairnet and nitrile gloves; facemasks used at designated processes.
  * Hangers and assigned shelves inside cleanroom for storage.

- **BRINGING MATERIALS INTO LAB**

  * NO pencils, wood, cardboard or dirty/dusty material should be brought into Lab; ballpoint pens, writing paper OK. See lab management for specific cases
  * Cleaning station to blow off and wipe down material brought into Lab is located near gowning room inside of lab

CLEAN ROOM PROCEDURES

WASTE DISPOSAL

* Dispose ALL chemicals including solvents & HF waste into prelabeled carboys and store in appropriate waste cabinet
* 2 disposal locations (1 in yellow room and 1 near PFC sink)
* Acid and base waste stored in polypro (plastic) cabinets; flammables eg acetone, organic solvents etc stored in metal cabinets
* Vaporous waste eg H_2O_2 solutions, Cl and NH_3 solutions must go in vented (polypro) cabinet and use vented caps

EQUIPMENT TRAINING

* Approval/training required for all equipment
* Training and supervision usually done by superuser or staff
* Training sign up done on line by adding name to training wait list on LabRunr
* Scheduling done thru nanotraining@seas.ucla.edu
* More complicated equipment requires 2 stage training
* 2nd qualifying session must be done one-on-one with superuser or staff. The same superuser who trained cannot do the qualification. Staff may hold both trainings.
* All log books must be used and filled out legibly and completely
* Read and obey ALL signs
* Report all broken or "sick" equipment to lab management
* Volunteer to help in the lab and be a superuser
* WHEN IN DOUBT, ASK

CLEAN ROOM PROCEDURES

- **SAFETY**

* Use of Yellow Room fume hoods, HF/BOE require special training
* Know where fire extinguishers, first aid, spill kits and emergency exits are
* Read MSDS sheet on any chemical you are unfamiliar with.
* Verify fume hood exhaust before using hoods
* Wear proper safety equipment: Visor, safety gloves and aprons at wet stations; wear safety glasses when in lab; Wear goggles or visor when filtering resists
* Be especially wary of Hydrofluoric Acid (HF) and HF mixtures (eg BOE) These are weak acids which may not be felt immediately upon contact but the fluorine can work its way to your bone and do serious damage. Wash exposed area with large amounts of water for at least 15 min and notify lab management. Use special calcium gluconate cream.
* Store all chemicals in their proper cabinets and label all chemicals
* Label any beakers left out with their contents, your name and date and your phone number, start time and finish time
* There must be 2 people in Nanolab at all times (Buddy System)
* Read and understand Lab Usage Guide
* Do not touch equipment you are not trained on- WHEN IN DOUBT, ASK

NOTE: USE OF HF, BOE REQUIRES SPECIAL TRAINING

CLEAN ROOM PROCEDURES

OUTSIDE CUSTOMERS & GUESTS

* Guests such as sponsors and collaborators are allowed in the lab with the following restrictions:

- Advanced notification to lab manager
- One time visit (ie not several consecutive days)
- Three or less visitors at a time
- Visitor to be properly gowned, obey all rules and be with host at all times
- Visitor must only observe and not participate or touch equipment or chemicals

* Consulting with or working with outside companies:

- You must notify and get concurrence from advisor
- You must also notify lab manager
- You may need to get a separate badge with company ID

TRAINING PROCEDURES

- **Log on to the Equipment Reservation and Training System. (User name: badge #, Password: nanolab).**
- **Must fill out the Personal Information Form completely.**
- **Add your name to the waitlist (please do not add your name to every equipment, only equipment that you NEED training on).**
- **Upon receiving training announcement, must reply to the announcement to be considered for the training list. (Priority will be given to those on the waiting list.)**
- **Training list will be sent out consisting of individuals who will be trained.**
- **Prior notice of absence to a training session must be made to avoid being blacklisted. (Blacklisted user will have the lowest priority when considered for a training session.)**
- **For equipment with a 2nd qualifying training session, user needs to set up an one-on-one training session with a superuser or staff after completing the first session.**
- **Upon completion of training, you may log on to the Equipment Reservation and Training Session and reserve the equipment.**
- **Please be patient with the training process as there are many students to be trained and limited number of trainers.**
- **Read training process on Nanolab website for details and direct all training matters to Jamie (jamie@seas.ucla.edu).**
- ***It is mandatory to fill out all log books completely and legibly."***

Lab Etiquette

* Clean up after yourself
* Leave room for others at fume hoods
* Do NOT borrow other users equipment or material without their permission
* Label unattended setup with: your name, chemicals in beaker, phone #, time and date started, time and date you will finish
Remember: Special permission required for more than 24 hours
* Read and obey ALL signs
* Get to know your fellow researchers outside your group
* Volunteer to help in the lab and be a superuser
* Put things back when you are done
* Use phones and paging system in lab
* Report broken equipment, safety issues etc.
* Turn off DI water when finished otherwise we run out.

NANOLAB COMMUNICATIONS

* Extensive use made of E-Mail
- Request Webmaster to add your name to “nanoall” list

* Web site: http://nanolab.ucla.edu for:
- Superuser info,
- Training documents and general information,
- Training waiting list-self sign up
- Web Calendar
- On-line Equipment Reservation

* Semi-annual user meetings held among group leaders

NANOLAB COMMUNICATIONS

* Online Reservation System

- Your name is submitted after training by Nanolab staff
- You will be given user name (badge #) and password (first-time password: nanolab)
- You then sign in from http://nanolab.ucla.edu and can reserve time for ONLY the equipment you are authorized on
- NOT all equipment is on reservation list

* Equipment Training Wait List

- Read all information on web on our procedures 1st
- Fill out personal information on wait list second
- Use LabRunr to sign up for training sessions

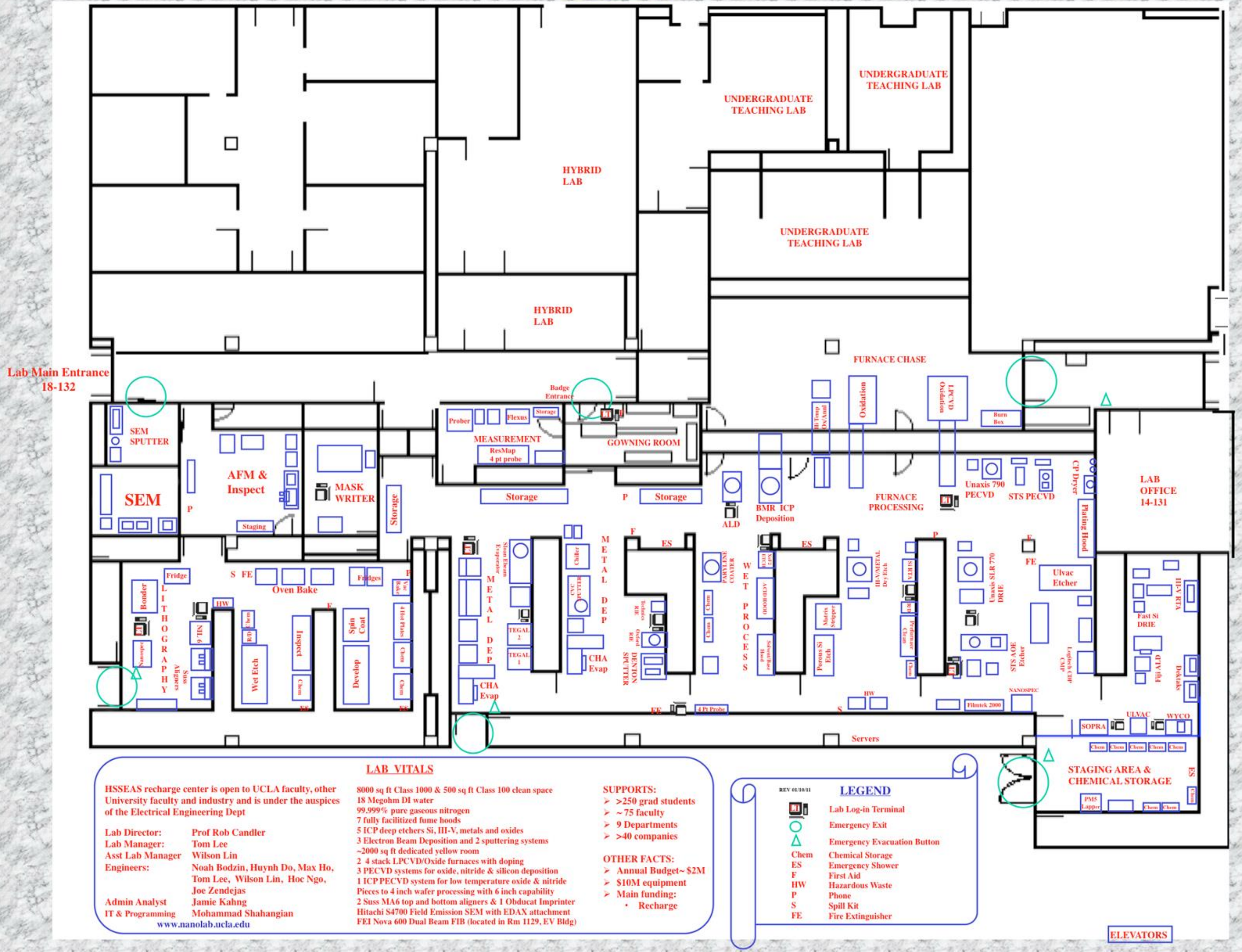
* Nanoall Email: Lab status sent out as news develops

- Equipment status
- Problems and fixes
- Special lab events, training etc

* Web calendar: training, orientations etc

UCLA Nanoelectronics Research Facility

EST 1992



LAB VITALS

HSSEAS recharge center is open to UCLA faculty, other University faculty and industry and is under the auspices of the Electrical Engineering Dept

Lab Director: Prof Rob Candler
Lab Manager: Tom Lee
Asst Lab Manager Wilson Lin
Engineers: Noah Bodzin, Huynh Do, Max Ho, Tom Lee, Wilson Lin, Hoc Ngo, Joe Zendejas
Admin Analyst Jamie Kahng
IT & Programming Mohammad Shahangian

www.nanolab.ucla.edu

8000 sq ft Class 1000 & 500 sq ft Class 100 clean space
18 Megohm DI water
99.999% pure gaseous nitrogen
7 fully facilitized fume hoods
5 ICP deep etchers Si, III-V, metals and oxides
3 Electron Beam Deposition and 2 sputtering systems
~2000 sq ft dedicated yellow room
2 4 stack LPCVD/Oxide furnaces with doping
3 PECVD systems for oxide, nitride & silicon deposition
1 ICP PECVD system for low temperature oxide & nitride
Pieces to 4 inch wafer processing with 6 inch capability
2 Suss MA6 top and bottom aligners & 1 Obducat Imprinter
Hitachi S4700 Field Emission SEM with EDAX attachment
FEI Nova 600 Dual Beam FIB (located in Rm 1129, EV Bldg)

SUPPORTS:
- >250 grad students
- ~ 75 faculty
- 9 Departments
- >40 companies

OTHER FACTS:
- Annual Budget~ $2M
- $10M equipment
- Main funding:
  - Recharge

REV 01/10/11

LEGEND

LT	Lab Log-in Terminal
○	Emergency Exit
△	Emergency Evacuation Button
Chem	Chemical Storage
ES	Emergency Shower
F	First Aid
HW	Hazardous Waste
P	Phone
S	Spill Kit
FE	Fire Extinguisher

General Chemical Handling

General Overview of Cleaning Process

Chemical Properties and Exposure Effects

- **Definitions**
  - **Chemical Properties Terms**
  - **Types of Exposure**
  - **Types of Effects**
  - **Exposure Levels**
  - **Toxic Effects**
- **Chemicals Used**
  - **Acetone and Flammable Solvents**
  - **Hydrofluoric Acid**
  - **Piranha and Nanostrip**
  - **Chlorinated Solvents**
  - **Photoresists---Glycol Ethers**
  - **Peroxides**
  - **Silane**
  - **Chlorine**
  - **Liquid Nitrogen**
- **Highly Toxic Gases**
  - **Phosphine**
  - **Diborane**

All material was taken from Cornell and Stanford web site with slight modification.

General Chemical Handling:

Wear the appropriate safety apparel; apron, chemical goggles or face shield and neoprene gloves with a flexiglove over them (for particulate control). Change gloves often when working in a critical area.

Always **A**dd **A**cid to water (AAA) when pouring chemicals.
Acids commonly used in the Cleanroom are hydrochloric, nitric and sulphuric acids.

Sulphuric acid represents the highest degree of danger as the exothermic reaction with water constitutes an extreme hazard; the mixture can explode. **NEVER ADD WATER TO ACID, ALWAYS ADD ACID TO WATER SLOWLY** so that the heat of dilution is released in a controlled manner. This warning especially applies to making up Hydrogen Peroxide-sulphuric acid mixture.

Do not mix solvents and acids. This may cause a violent reaction. You may use solvents only at a solvent bench. Do not dispose of acid or alkali mixtures into the solvent waste bottles/bins. Large quantities of noxious fumes can result as well as possible explosive reactions. Always work with acids or solvents in an exhausted hood. Be sure you open the bottle up far enough back in the wetbench or workstation so that the exhaust draws off the fumes.

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 H2O:NH4OH:H2O2 solution that chemically attacks organic impurities. The basic conditions also slowly etch Si, which removes small particles strongly attached to surface. This step is one of the critical steps necessary 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 H2SO4:H2O2 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 pirana 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.

Chemical Properties and Exposure Effects

Definitions: Chemical Properties Terms

Pyrophoric chemicals spontaneously ignite in air. No source of ignition (spark) is needed. They react spontaneously when exposed to oxygen. Silane is an example of a pyrophoric gas used in the facility.

Flash point is the minimum temperature of a liquid at which it gives off sufficient vapor to form an ignitable mixture with air. Liquids with a flash point near room temperature can be ignited very easily during use.

Exothermic Reaction- A reaction which produces heat (releases energy).

Definitions: Types of Exposure

Acute Exposure as used in toxicology refers to a short term exposure. It has nothing to do with either the severity of the exposure or the severity of the effect. The type of exposure occurring during an accidental chemical spill is properly described as an acute exposure.

Chronic Exposure as used in toxicology refers to a long term exposure. Again, it has nothing to do with the severity of the exposure, the severity of the consequences, or the duration of the consequences. Chronic exposures can be the result of chemicals in the workplace, the home, or the environment. Chronic exposures are usually the result of carelessness, ignorance, or neglect, and not the result of an accident.

Local Exposure refers to exposure limited to a small area of skin or mucous membrane.

Systemic Exposure means exposure of the whole body or system, through adsorption, ingestion, or inhalation.

Definitions: Types of Effects

Acute Effects refers to the duration of the symptoms. Acute means symptoms lasting a few hours or days. Again, it has nothing to do with the severity of the effects.

Chronic Effects are long term effects, manifested by prolonged duration and continuing injury.

Local Effects occur in a small area, at the place of contact.

Systemic Effects occur throughout the body, or at least away from the point of contact.

Allergies and Hypersensitivity are reactions by particular individuals to particular chemicals, caused by heredity or prior overexposure. Hypersensitive individuals should avoid exposure to the offending agents.

Definitions: Exposure Levels

TLV - Threshold limit value. This is actually TLV-TWA (time weighted average) but is commonly called just TLV. It is the (averaged) level to which you can be exposed 8 hours a day, 5 days a week forever, without adverse health effects. These levels are set by ACGIH (governmental and industrial hygienists), and adopted into law by OSHA (Occupational Safety and Health Administration). This level is most relevant to chronic (long term) exposure to chemicals in the work place. Short term exposures in excess of TLV are thus not necessarily hazardous. This value is not particularly relevant to our laboratory situation. It is sometimes used as a guideline, however, since short term exposure to < TLV should be very safe.

IDLH - Immediately Dangerous to Life and Health. This level represents the maximum value for which a 30 minute exposure will result in no irreversible or escape impairing effects, i.e. the maximum level which will not cause you to pass out or sustain irreversible organ damage. It is the value most appropriate to sudden, one time accidental exposures. For your information, a short table of values for relevant chemicals is listed below.

IDLH

Ammonia 500 ppm
Carbon Monoxide 1500 ppm
Chlorine 25 ppm
Hydrogen Fluoride 20 ppm
Diborane 40 ppm
Phosphine 200 ppm

STEL- Short Term Exposure Limit- Actually TLV-STEL. Maximum concentration to which you can be exposed for 15 minutes, up to 4 times a day without adverse effects.

PEL- Permissible Exposure Limit- The statutory equivalent of TLV.
LD_{50}- The dose at which 50 % of those exposed will die. Separate levels apply to various modes of exposure (inhalation, dermal, etc.). Usually expressed in terms of mg per kg of body weight; often measured for mice and rats, for obvious reasons.
All these levels are approximate, with considerable inconsistency between various sources. It is obvious that one cannot do well controlled experiments on human subjects. It is thus wise to be conservative in estimates using these numbers.

Definitions: Toxic Effects

Carcinogen- A substance producing or inciting cancerous growth.
Mutagen- Capable of inducing mutations .
Teratogen- A substance causing damage or death to a fetus.

Chemicals Used: Acetone and Flammable Solvents

Acetone is widely used throughout the facility. It is a very flammable solvent with a low flash point, (i.e. it can be ignited at a low ambient temperature). Because of this it presents a significant fire hazard. A spill of a gallon bottle of acetone could cause a catastrophic fire or explosion. It should not be transported except in chemical buckets. Solvents should also be handled with care in the hoods and not used near hot plates. Spilled solvent can be ignited by the hot plates. The resulting fire could easily be drawn up into the exhaust ducts, again with catastrophic consequences. Spilled solvents can react explosively with chemical oxidizers present, e.g., peroxides, nitric acid. Spilled solvents should be contained immediately with spill control pillows. Environmental Health and Safety should be called for emergency response and to assist in clean up.

Chemicals Used: Hydrofluoric Acid

Hydrofluoric acid, HF, presents a significant hazard for personal injury. It is widely used in the lab in its pure form, diluted, and as the active component of BOE, Buffered Oxide Etch. It is used for etching silicon dioxide and particularly for stripping the native oxide prior to further processing. 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**.**

HF, however, is a very hazardous chemical, much more so than any of the other acids. Its danger comes from its effect on flesh.

At the concentrations used in the laboratory, an HF “burn” is initially painless. You may not even know that you have gotten a splatter on your hands, arms, face, or in your gloves. The acid however will silently eat away at your flesh. The fluoride ion is not consumed in this process and is soluble in tissue, so the damage penetrates deeper and deeper, until it comes to the bone. About this time the excruciating pain begins. It is too late , however, to reverse the considerable tissue damage. At some point, it enters your blood stream and goes everywhere scavanging Ca ions, totally messing up the ionic chemistry of your nervous system. **At some point, if left untreated, you die.**
Simple washing of an HF splash is not sufficient to prevent damage. It does not wash off; it is already dissolving you and will continue to do so until you receive **medical attention specific to HF burns** (including deep injections to neutralize the penetrated acid). **Be sure that medical personnel know that it is an HF burn and know that it requires specific treatment different from a common acid burn.**
You thus have only 2 choices; 1) be absolutely certain that you don’t get it on you by being very

careful and wearing Nitrile gloves, apron, mask, etc., or 2) visiting the emergency room after you use it. **We prefer #1.** . 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! HF etches silicon dioxide very well. Therefore, it also etches glass. It must not be kept in a glass bottle, used in a glass beaker or disposed in a glass waste bottle. Plastic labware is available for this purpose. HF, like all other chemicals, must only be used in the chemical hoods.

Chemicals Used: Piranha and Nanostrip

Liquid **piranha** is a common name applied to a mixture of Hydrogen Peroxide and Sulfuric Acid (typically 1:5). It is extremely aggressive toward carbonaceous materials (e.g. flesh and photoresist residue, equally). It also removes heavy metal contamination. It is used by some users for cleaning wafers.

We have difficulty disposing of this mixture, however, as the waste continues to react and decompose for a long period of time. This builds up pressure in the waste bottles causing them to burst. Also if the solution is mixed very peroxide rich, one can make unstable compounds.

Chemicals Used: Chlorinated Solvents

Chlorinated solvents **(chlorobenzene, trichloroethylene, and methylene chloride)** are used in various resist processes. They are particularly bad for you, causing cancer, organ damage, etc. They should not be mixed with normal solvents in waste bottles. There are separate waste bottles for chlorinated solvents. As with most solvents, they can be readily absorbed through the skin.

Chemicals Used: Photoresists---Glycol Ethers

Commercial photoresists and electron beam resists are dispersed in a variety of solvents. The composition of these mixtures is generally not disclosed on the bottle; you must look on the **MSDS** for it. One family of chemicals, the **glycol ethers**, commonly used in photoresists, masquerades under a variety of names. It is not often clear that many of these are the same chemical; oh, the wonders of organic chemistry nomenclature. In addition, the common trade name **"Cellosolve"** is often thrown in. Anyway,

Methyl Cellosolve
Ethylene glycol mono methyl ether
2-methoxyethanol

are all the same thing. Similarly,

Cellosolve
Ethyl Cellosolve
2-ethoxyethanol (2EE)
Ethylene glycol mono ethyl ether

are all the same solvent. To further complicate things, each solvent has an acetate relative, so we have

Cellosolve Acetate
Ethyl cellosolve acetate (ECA)
Ethylene glycol mono ethyl ether acetate
2-Ethyoxy ethyl acetate

which are again all identical. I won't even go into the methyl cellosolve acetates or any of the butyl cellosolves. I think you get the picture. Most photoresists contain one or more of these as solvents.

Members of this family of chemicals have been shown to be teratogens and have other effects on reproduction in laboratory animals. A number of recent studies funded by IBM and others have found evidence that these chemicals can lead to miscarriage and other reproductive effects. To quote from the MSDS for AZ 2131 Thinner (2 Ethoxyethyl Acetate and N-Butyl Acetate)

" In studies with laboratory animals, 2-ethoyxethyl acetate caused birth defects, increased fetal death, delayed fetal development, caused blood

effects, testicular damage and male infertility."
The liquid and vapor are eye and respiratory tract irritants and may cause kidney damage, narcosis, and paralysis (in simple terms, it damages your kidneys, eyes, lungs and brains). Primary routes of exposure are inhalation, skin absorption, and skin and eye contact with vapors. **N-butyl Acetate**, the other component of this thinner, has a similar list of possible systemic effects. As with all chemicals, these are only the effects we know about. These experimental laboratory exposures were large amounts but nonetheless it is prudent to be careful with these solvents.
If after reading this section, you still do not have sufficient respect for these chemicals, please go back and read it again.
Many users have become sloppy with resist. **Don't be!!!!! If you can smell resist in the resist room, somebody is doing something wrong!!** Find out what it is and stop it. The conclusion is that you should be careful with the use of even these seemingly innocent chemicals, wear the proper protective equipment, and work in a well ventilated area at all times.

Chemicals Used: Peroxides

All peroxides are highly oxidizing materials. Considerable energy can be released in their reactions with common materials. Some peroxide compounds are unstable, and can explode. We have hydrogen peroxide in the facility. Extreme care should be used in mixing solutions containing peroxides. Peroxides are incompatible with all forms of organic solvents and flammable materials.

Chemicals Used: Silane

Silane (SiH_4) is used for the deposition of CVD silicon nitride and silicon dioxide in the MOS area and at the PECVD system in the thin film area. **Silane is pyrophoric.** It spontaneously ignites in air at concentrations between 4 % and approximately 90 %. The silane gas cylinder is located in a ventilated cabinet. The bottle is fitted with a flow restricting orifice and a flow limit valve. These limit the flow of gas from the bottle so that even under catastrophic system failure the concentration is kept below the lower explosive limit.

Chemicals Used: Chlorine

Chlorine gas is used in several of the etching systems, where it is confined in ventilated cabinets. Chlorine is severely corrosive and is choking to breathe. Chlorine forms HCl in the lungs, causing severe tissue damage which can be fatal. As with many other corrosive gases, the effects of exposure may not be noticed for a few days. In all cases, medical attention should be sought immediately following exposure, not at the onset of symptoms. For your reference the following values relate to chlorine exposure.
TLV 1 ppm
Odor Threshold 0.1 ppm
Coughing 30 ppm
Dangerous in 30 min. 40-60 ppm
Fatal with a few breaths 1000 ppm
Because of the small amounts used, the ventilation used, and the low odor threshold for chlorine exposure, accidental chlorine gas exposure is not considered a significant risk.

Chemicals Used: Liquid Nitrogen

Nitrogen, a hazardous gas ??? Yes, it is true. More people die of asphyxiation by good old nitrogen than by any of the "toxic" gases discussed here. We use liquid nitrogen for many things in the laboratory. It is transferred to 160 liter dewars for cold traps, and the boil off is used for purge gas for all vacuum pumps. A large liquid storage tank is located outside the loading dock. Smaller tanks are filled on the loading dock.

Highly Toxic Gases Used

Several highly toxic gases are used in the silicon processing area of the facility. These are limited to **dilute diborane** (0.2% B_2H_6 in nitrogen) and **dilute phosphine** (2 % PH_3 in nitrogen). Extreme care must be exercised with these gases, as exposure to small amounts can be fatal. Odor provides a poor warning for these gases as they are toxic at levels near or below the odor threshold. These gases are widely used in the semiconductor industry. Because of the extensive safety precautions normally taken, no accidental fatalities have been reported in the semiconductor industry using these gases.

Highly Toxic Gases: Phosphine

Phosphine gas is a severe pulmonary irritant and an acute systemic poison. Overexposure can cause either sudden or delayed death due to lung destruction. It is a colorless gas with a fishy odor. Olfactory (smell) warning properties are better, however, than for arsine or diborane. It is toxic at levels near the odor threshold so it must be treated with great care. The OSHA standard PEL (permissible exposure limit) is 0.3 ppm averaged over an 8 hour shift. It is slightly heavier than air. Phosphine, like silane, is pyrophoric; i.e., spontaneously flammable in air. We usually don’t dwell on this, however, as its acute toxicity is a concern at much lower levels. Exposures of 2000 ppm for a few minutes are lethal; exposures of 7 ppm for several hours can be tolerated. The odor threshold for phosphine is near 1 ppm. The phosphine used in the facility is diluted to 2% in nitrogen in the bottle. This adds an extra measure of safety to any accidental release of phosphine.

Sources quoted:

http://www-snf.stanford.edu/Process/WetProcessing/ChemPolicies.html
http://www.engr.uky.edu/~cmmed/CMMEDpdf/WetBench.pdf
http://www.ee.ucl.ac.uk/~cleanrm/CleanRmSafety.html
CNF Laboratory Usage and Safety Manual, 8th Edition
http://www.cnf.cornell.edu/cnf/CNFSafetyManual8.pdf

NANOLAB CHEMICAL HANDLING RULES

Note 1 Common sense and engineering knowledge is used as a guide. There may be situations not covered by the following rules. Consult with lab management for any situation that is unclear. Do NOT proceed if you have any doubt about the correct procedure.

*Note 2: Use of **HBr** and **HF** requires special training and only authorized users may work with HBr at the designated location.*

1 All chemicals must be poured at a working fume hood. Magnehelic gauges should be checked BEFORE pouring chemicals to insure adequate exhaust. Do NOT pour chemical if exhaust is inadequate.
2 Chemical-resistant gloves and face shields must be worn when pouring chemicals. Aprons should also be worn.
3 Only approved chemicals (those already listed in the MSDS book) may be used in the lab. New chemicals (those not already in use in lab) must be approved by lab management and MSDS sheets must be obtained before bringing new chemicals into the lab.
4 MSDS sheets must be read and understood before working with a chemical. All warnings and procedures regarding storage, disposal etc. must be followed.
5 Chemicals poured in open beakers must remain under a fume hood. Do NOT move them out of the fume hood. Beakers must be labeled as to chemical name, user name, date and time started, date and time expected to finish and phone number.
6 Do NOT heat highly flammable liquids such as acetone and alcohol. Turn off hotplates when finished.
7 Use rubber or nalgene carriers when transporting chemicals thru lab. Do NOT carry a bottle without a carrier unless it is directly across from the fume hood being used. Use a plastic cart with containment lip if you are bringing chemicals in from another lab.
8 All chemicals must be stored in clearly marked, sealed containers of a material compatible with the chemical (e.g. do NOT use glass with HF). Markings must include:
chemical name, professor and date chemical was brought into lab.
9 Do NOT move or dispose hot chemicals, let them cool first.
10 Chemicals must be stored in the appropriate cabinet except when in actual use. Acids must be stored in the acid cabinet, bases in the base cabinet and solvents in the solvent cabinet.
11 Waste chemicals must be stored in the chemical waste cabinet in clearly marked carboys; Do NOT overfill. Use funnels where necessary.
12 Chemicals must NOT be stored or left for appreciable amounts of time on the floor or tables and must be labeled as to chemical name and/or strength of mixture.
13 Chemicals must NOT be stored underneath fume hoods.
14 Strongly fuming chemicals such as HCL and Ammonium Hydroxide must be used only in the recessed hot pot areas of the fume hood so that lip exhaust helps contain odors.

NANOLAB CHEMICAL HANDLING RULES

15 If strong odors are present during chemical processing, stop, turn off hot plate, if on, call lab management and evacuate area. Do NOT continue processing if you smell fumes.
16 It is recommended that you have your own personal chemical-resistant gloves. Wash gloves in DI water after each use.
17 Triple rinse empty chemical bottles including cap and affix rinse label. Date and initial it and place rinsed bottle back in appropriate cabinet if there is room or on the floor near the cabinet out of the way if there is not room.
18 Chemical waste must be poured AT THE FUME HOOD into the appropriately labeled waste bottle. Allow the chemical to cool first and wear safety visor and special gloves. Wipe down outside of waste bottle with DI water and clean wipe.
19 Use only vented caps (a small hole is visible in the top) or a waste bottle with a pop out plug in the spout for hydroxide and peroxide solutions.
20 If chemical processing at a fume hood needs to continue in your absence, you MUST place the setup towards the back of the hood out of the way and label it (use clean tape) with: your name, chemical(s) used, time and date poured, your phone # and when you will finish.
21 Report all spills or chemical exposure to lab management. For small spills, spill kits are located in yellow room above fire extinguisher and near LPCVD furnaces.
22 Use appropriate fume hood (e.g. solvent hood for solvents, acid hood for acids).
23 Report unsafe conditions or safety violations by others to lab management.

Rev 4/22/04 **Toxic Gas Usage In ENG IV Building**

Introduction:

This information is supplied to you as your "Right to Know" and is believed to be accurate. It was taken from the Matheson Gas Catalog and the Air Products Gas Reference. MSDS sheets on all gases are stored at the Nanolab entrance and in the Lab Manager Office. For more information call x68923.

The Nanoelectronics Research Facility (NRF) located on the 1st floor of the ENG IV building uses small quantities of pyrophoric and toxic gases. These gases are stored in small steel cylinders in the toxic gas bunker located between Boelter Hall and ENG IV (GS 130). This special bunker is built to handle small leaks and/or fires and was specially designed to accommodate the gases used. The bunker is kept locked at all times and may only be entered by trained personnel.

The gases are piped into the NRF in specially welded steel tubes which have been helium leak checked and are connected directly to certain equipment in the NRF. Gas sensors are strategically located to sense the smallest leak. Under a high alarm condition, the sensors shut off the gas, sound a building alarm and automatically call the fire department. This entire system has passed thorough testing.

Hazardous Gases:

The following hazardous gases are used in small amounts (typically 10 lbs or less) and are stored in the toxic gas room.:

Silane (SiH_4): Is a highly flammable (pyrophoric) gas and toxic which spontaneously combusts with air or water vapor. It can combust at concentrations as low as 2%. It produces a white powder (glass) as a byproduct of its burning which is non toxic.

Ammonia (NH_3): Is a corrosive, toxic gas with a pungent odor. Unlike phosphine, your nose is sensitive to ammonia before it becomes lethal. Ammonia is also flammable in the 15-28% concentration range.

BCl_3, Chlorine: are highly toxic, corrosive gases. They are greenish, yellow liquefied gas and are strong oxidizers with a choking odor. Chlorine is in a double contained tube (tube within a tube) because of its toxicity.

Dichlorosilane ($SiCl_2H_2$): Is a highly toxic, corrosive and flammable gas with a sharp, pungent odor. It hydrolyzes with water to form hydrochloric acid. This gas is also in a double contained tube because of its toxicity.

Hydrogen: Is a non toxic, odorless gas which ignites readily. It burns with an almost invisible flame and can be explosive.

Phosphine (PH_3) : Is a highly flammable and highly toxic gas with a decaying fish smell. Concentrations as small as a few hundred ppm can be dangerous to life and health. Because of its toxicity, phosphine is mixed with silane so that a leak will burn rather than posion. Is is also double-contained for its toxicity.

Safety Features and Alarms:

A number of safety precautions and special features have been implemented for our safety including:

- An exhausted, specially-designed bunker remote from the building where the gases are stored.
- Individual sprinklered, exhausted gas cabinets for each gas.
- Orbital-welded stainless steel tubing with double containment for the most toxic gases
- An integrated gas detection system capable of sensing extremely small amounts of each gas. If a gas leak is detected, an alarm is activated and a fire dispatch signal is sent automatically.
- Excess flow sensors shut the gas bottle down in the event of a leak.
- Restrictive flow orifices built in to each bottle limit the leak rate in the event of a problem.
- A seismic sensor shuts down all gas cabinets in the event of a major seismic event.
- Small quantities of each gas to limit the possibility of our exposure.

For Your Safety:

- **If the Toxic Gas Alarm (Buzzer) sounds, evacuate the building calmly and quickly via your nearest emergency exit.**

- **Do NOT loiter near the Toxic Gas Bunker even when there is no emergency.**

- **If you suspect something is wrong in the toxic gas room, (noises, smoke, funny smells etc.) contact Nanolab Manager @ x68923 or EH&S @ x55689.**

- **Do NOT attempt to enter the toxic gas room.**