

# **NANOLAB ORIENTATION MEETING**

**August 29, 2008**

# NANOLAB OPERATIONS

- **STAFFING**

- \* Prof. Jack Judy—Lab Director
- \* Steve Franz—Lab Manager.....x68923...14–131A, EIV.
- \* Tom Lee- Etch Engineer.... x64641
- \* Hoc Ngo— Metals Engineer—X65528
- \* Huynh Do- PECVD Engineer...x64641
- \* Yuwei Fan Nanolithography Engineer-Leica,..X65528
- \* Wilson Lin- Furnaces and Thermal Processing Engineer-x68923
- \* Joe Zendejas-2nd shift Engineer-X65528
- \* Ghassan Malek-Lithography-X49329
- \* Brian Matthews-Training and management support
- \* Noah Bodzin-FIB Engineer- *nbodzin@physics.ucla.edu*
- \* Web Master, Mohammad Shahangian **X64641**
- \* Jamie Jung-Training Coordinator, x69908
- \* Lab Assistants
- \* Superusers for selected equipment

- **LAB HOURS (PHONE X69597)**

- \* 7 AM to 2 AM weekdays and weekends  
(requires building permit after 11PM)
- \* 1st floor EIV (18–132)
- \* Buddy system in force

- **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 Orientation (this meeting)**
- 2 Study Lab Usage Guide (Your Prof. has one and is online)**
- 3 Bring signed lab use permission slip with recharge ID and signed toxic gas acknowledgement to the test.**
- 4 Bring refundable deposit for badge ( \$25 check payable to UCLA EE Dept) to the test**
- 5 Pass safety test based on Lab Usage Guide and Handouts Safety (Section 1 only).**
- 6 Attend Lab Walkthrough (2.5 Hrs).**

***\* Industry users do not need to take test or pay badge deposit but DO need to sign indemnity form and gas acknowledgement, attend Lab Walkthru and present a valid PO # for not less than \$700***

# NANOLAB DESCRIPTION

- ~8000 sq ft class 1000 area
- Temperature and humidity controlled environment
- Yellow room lithography area
- Separate class 100 room for Leica lithography and SEM
- Badge-reader entry to dedicated gowning room
- 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 $\mu\text{m}$	0.2 $\mu\text{m}$	0.3 $\mu\text{m}$	0.5 $\mu\text{m}$	5.0 $\mu\text{m}$
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***

- \* Leica EBL 100, 100 Kev e-beam writer with laser stage and resolution to 30 nm
- \* 3 Karl Suss MA6 contact aligners up to 6 inch wafer with top and bottom side alignment
- \* 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
- \* HBr acid etching (special training required)
- \* HF/BOE acid etching (special training required)
- \* Heidelberg DWL66 LaserWriter for photomask making and direct-write
- \* Nova 600FIB for submicron cutting, etching 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
- \* 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)
- \* Gaertner automatic multiple-angle ellipsometer (fixed wavelength)
- \* Flexus film stress measuring system
- \* Omnimap 4 point probe with mapping capability
- \* Micromanipulator probe station with HP4145 tester

## ***DRY ETCHING***

- \* STS AOE ICP Thick Dielectric Etcher
- \* 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 (1300 °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

## ***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
- \* Buelher MC 2000 cross-sectioning tool

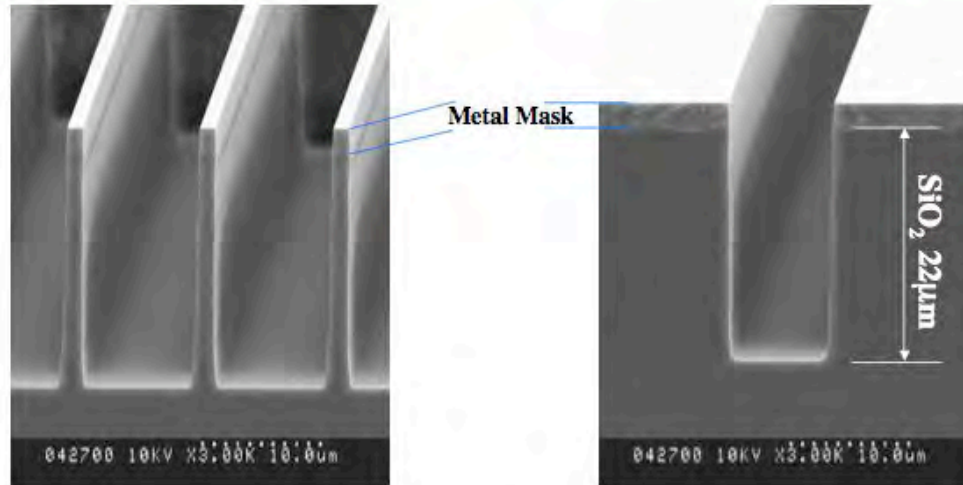
# NANOLAB CAPABILITIES

*Recently Added:*

- \* Ulvac NLD 570 Oxide Etcher  
Deep ( $>10\mu\text{m}$ ) Glass Etching

## Deep Trench Etching for Optical Waveguide

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# NANOLAB CAPABILITIES

## *Recently Added:*

- \* **FEI Nova 600 Dual Beam FIB**
  - Sub-micron etching
  - Deposition of C, Pt, W
  - Located in Knudsen
  - 2 Engineers:
    - Dr Sergey Prikhodko**
    - Noah Bodzin**
  - Open to all UCLA researchers and outside companies

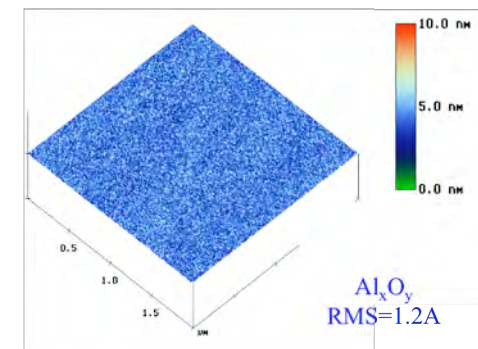
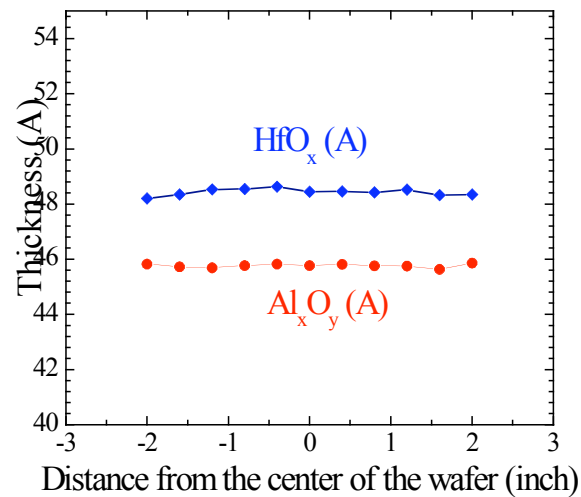
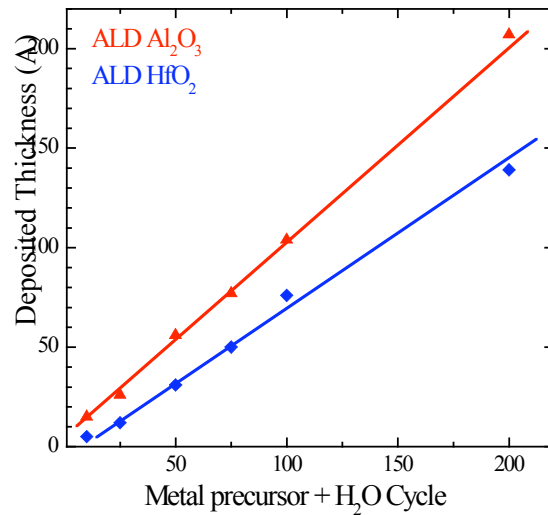


**Nova 600 NanoLab**  
DualBeam™-SEM/FIB for  
Nanoscale Prototyping, Machining,  
Characterization, and Analysis  
of Structures below 100 nm

# NANOLAB CAPABILITIES

## Recently Added:

- \* Cambridge Savannah 100 ALD
  - Monolayer Tk control
  - Highly conformal, smooth films
  - Gate Dielectric Quality
  - Deposition @ ~250 °C



# **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 355 Stripper, Sulfuric Acid,Hydrogen Peroxide, HBr, 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 pre-labeled 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<sub>2</sub>O<sub>2</sub> solutions, Cl and NH<sub>3</sub> 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
- \* Training sign up done thru Jamie  
(name will be added to waiting list)
- \* Badge log-in/log-out required for single door entry
- \* Log books must be used for major pieces of equipment
- \* 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, HBr, 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
- \* 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
- \* 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 OR HBr 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 2<sup>nd</sup> 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@ee.ucla.edu](mailto:jamie@ee.ucla.edu)).**

# 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**

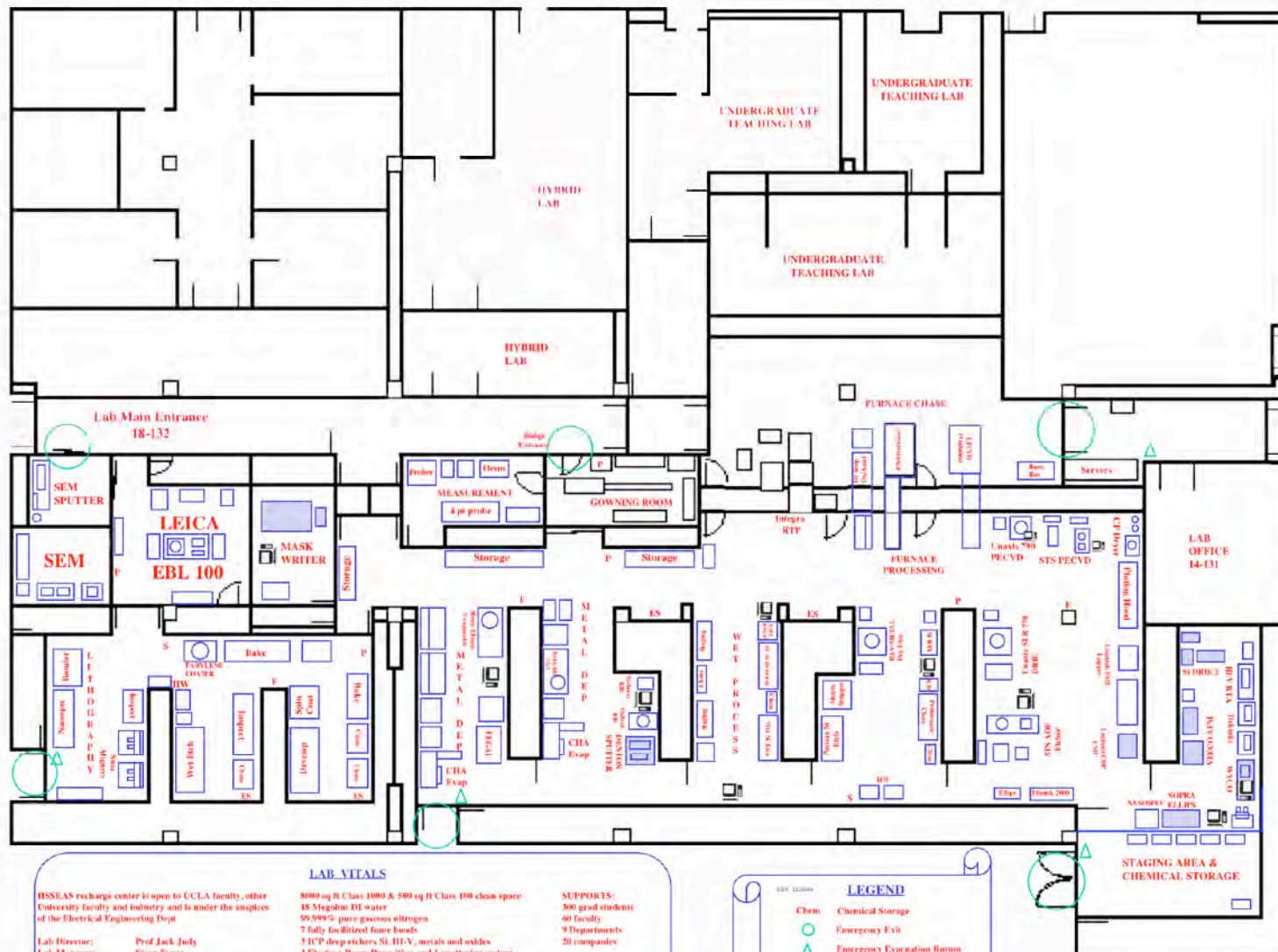
# **NANOLAB COMMUNICATIONS**

- \* **Online Reservation System**
  - Your name is submitted after training to Nanolab staff
  - You will be given user name (badge #) and password (last name in Caps)
  - 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 Wait List**
  - Read all information on web on our procedures 1st
  - Fill out personal information on wait list second
  - Email Jamie and put your name on training wait list
  
- \* **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**

BSSEAS 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. Jack Judy  
**Lab Manager:** Steve Franz  
**Engineers:** Dr. Ivan Alvarado, Huzah De Tam Lee, Dr. Wilson Lin, Hue Ngo, Joe Zendejas, Jamie Jung

**Admin:** Mohammad Shahoungian  
**Web:** Justin Thomas, Jeff Chen,  
**Lab Assist:** Harold Kim, Craig Chi

8000 sq ft Class 1000 & 500 sq ft Class 100 clean space  
 18 Megahm DI water  
 99.999% pure gaseous nitrgen  
 7 fully faciltized fume hoods  
 3 ICP deep richers, Si, III-V, metals and oxides  
 3 Electron Beam Deposition and 1 sputtering system  
 ~2000 sq ft dedicated yellow room  
 2 4 stack LPCVD/Oxide furnaces with dopng  
 Pieces to 4 inch wafer processing with 6 inch capability  
 Leica EBL100 direct beam writer with 30nm resolution  
 2 Sus M46 top and bottom aligners  
 Hitachi S4700 Field Emission SEM with EDAX attachment

**SUPPORTS:**  
 300 grad student  
 60 faculty  
 9 Departments  
 20 companies

**STAFF:**  
 7 full time engineers  
 4 student assistants  
 1 part time web masters  
 1 administrative assistant

[www.nanolab.ucla.edu](http://www.nanolab.ucla.edu)



■ New Equipment '04-05

ELEVATORS

## **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 Add Acid 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 H<sub>2</sub>O:NH<sub>4</sub>OH:H<sub>2</sub>O<sub>2</sub> 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 H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O<sub>2</sub> 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/SiO<sub>2</sub>. 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<sub>50</sub>-** 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 SiO<sub>2</sub> layer. HF etches are also commonly used as the last step to etch through SiO<sub>2</sub> to contact the Si wafer with your metal leads. Buffered HF (BHF) is NH<sub>3</sub>/HF solution that has the highest SiO<sub>2</sub> 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 scavenging 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 CaCl<sub>2</sub> solution (precipitates inert CaF<sub>2</sub>) 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-Ethoxy 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-ethoxyethyl 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<sub>4</sub>)** 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<sub>2</sub>H<sub>6</sub> in nitrogen) and **dilute phosphine** (2 % PH<sub>3</sub> 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.

**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<sub>4</sub>):** 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<sub>3</sub>):** 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<sub>3</sub>, 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<sub>2</sub>H<sub>2</sub>):** 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<sub>3</sub>) :** 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 poison. It 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.**

