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Experiment Safety Review Form
Review Number: NC-30-2011

PRINCIPAL INVESTIGATOR: Ming Lu
GROUP: NC
EXT: 4773 **E-MAIL: mlu@bnl.gov** **LIFE NUMBER: 24188**

Project Title: Oxford Reactive Ion Etcher-C
Location(s): 0735
Area(s): 0735-FIRST-1-1G05, 0735-FIRST-1-1L43
Proposed Start Date and Duration: 4/1/2011 - 1 years

SIGNATURES:

Principal Investigator: Ming Lu	**Date: 10/18/2011**
Experiment Review Coordinator: Robert Sabatini	**Date: 10/18/2011**
Reviewer: Frank Zafonte	**Date: 7/8/2011**
Reviewer: Aaron Stein	**Date: 8/2/2011**
Reviewer: Lorraine Davis	**Date: 9/21/2011**
Reviewer: Joy Haskins	**Date: 7/22/2011**
Reviewer: Nicole Chiu	**Date: 9/15/2011**
Approval: Emilio Mendez	**Date: 10/18/2011**

Review/Approval (ERC) Comments:
10/18/2011 3:19 PM
10/18/2011 3:19 PM

Final gas cylinder change out proceedure will be attached after first installation is complete.

Walkthrough Signature: Robert Sabatini	**Date: 11/1/2011**

Expiration Date (max 1 yr.): 10/18/2012
FUA Change Required? Yes
Fire Rescue Run Card Changes Required? Yes
Has a NEPA Review been Performed for this Project? Yes
Required Approvals (i.e., IACUC, IBC, etc.): NA

Project Termination Acceptance Signature: **Comments:**	**Date:**

I. Define the Scope of the Work

A. Description

This covers the work done with the second Oxford Plasmalab 100 etcher (internal name: Oxford-C). Films are etched via reactive ion etching. Since these tools reside in the clean room, full clean room garb as described in the attached SOP , including nitrile gloves, will be worn at all times. Entry into the clean room requires clean room orientation/training as per SOP# NC-2007-ELM-Ops-3 as well as COSA given by authorized CFN staff. Most chemical work in the clean room is covered under ESR NC-07.

All gases (incl. liquid nitrogen) are kept in the service galley and are piped through penetrations in the wall to the systems they supply. **Gas cylinders are to changed only by trained CFN Staff.**

In general, reactive ion etching (RIE) requires a plasma generated by applying an RF voltage to a gas in a vacuum chamber. The ions and other neutral species react with the substrate and selectively etch different materials based on the type of gas. The **Oxford Plasmalab System 100 (Oxford-C)** uses an inductively coupled plasma (ICP) to increase ionization without increasing the voltage across the plasma. The tool can operate in several modes: regular RIE, ICP RIE and cryo ICP process. When cryo ICP etching is used, the substrate is cooled by liquid nitrogen down to temperatures of -150C. Liquid nitrogen procedures are described below. The unit uses the following process gases: BCl_3, Cl_2, HBr, $SiCl_4$, CH_4, H_2, C_4F_8, CF_4, SF_6, Ar, O_2, and N_2. Gases are fed to the system by a gas pod which controls the flow of each gas. All functions of the DRIE machine are controlled by the system software. In addition, nitrogen and helium are used during operation of this tool for venting and cooling, respectively. The system operates under vacuum with a loadlock for exchanging wafers. Both loadlock and process chamber are pumped by their individual turbo molecular pump (TMP) and each TMP is backed with an individual dry scroll pump. Dry scroll pumps are located in the service galley. House chilled water is used to cool the system RF power generators. The liquid nitrogen from a liquid nitrogen tank in the service galley is used to cool the sample table. Alternatively a close-loop chiller in the service galley, backed by house chiller water, can be swapped to supply coolant to the sample table to stablize the sample temperature beyond 0 degree C.

Note: all plasma system have small windows to view the plasma process inside the vacuum chamber. These windows are all UV-shielded.

The highly toxic gases are stored and supplied from gas cabinets in the clean room service galley. The cabinets house the Cl2, HBr, BCl3, H2, CH4, and SiCl4. All cabinets (except the cabinet hosting SiCl4 vaporizer) have sprinklers and gas detectors. In addition to the detectors in the service galley, another set of detectors are installed in the cleanroom to monitor the cleanroom air. Each set of these gas detectors are connected to an individual Beacon Controller in the hallway outside the clean room. The Beacon controllers are wired to an interlock panel which controls the life-safety (emergecy shut-down) interlocks on all the gas cabinets. The interlock panel is also wired to the building fire alarm system and monitored by BNL fire department.

There is a status light tree, green, yellow, and red in the clean room and galley.

1. The green light indicates all systems are normal.
2. Yellow indicates a fault which needs attention by the system owner.
3. Red indicates a Level 1 Alarm, which indicates either a gas detection or problem with exhaust system. This will stop the process until the problem is resolved.

If **Level 2 Alarm** is tripped, there will be a **special temporal 3 alarm** and a message **to evacuate the building due to a Toxic Gas release**. The message will tell everyone to exit out the north doors (front) only.

Must follow the SOP for cyclinder changeout. 2-man rule at all times during the changeout. During the changeout of the highly toxic gases workers will don SCBA.

No modifications to this system or componets can be made without permission from the ES&H Coordinator.

This RIE-C can only be used during normal working hours at the CFN, no after hours work is to be done unless authorized by the CFN ES&H Coordinator.

Equipment manuals or procedures that are controlled documents:

- The paper copy of the user manuals for the Oxford ICP metal etcher are stored in a cabinet in front of the Lesker sputtering tool in 1L43
- The electronic copy of the complete user manual is in the Oxford etcher computer
- At the present time, SOP's have been written for:
  - Liquid nitrogen filling
  - gas cylinder changing

SOP-NC-2007-EM-OPS-1 (Clean Room Gowning Procedure); SOP-NC-2007-ELM-PECVD-1 (Clean room silane and ammonia gas handling procedure).; Ref ODH Analysis Case # 3a,5c and 7.

B. Human Performance Factors

The Oxford Plasmalab 100 system has a loadlock and is presently configured for use with 3" or 4" wafers only. Because many users are using chip-sized pieces, they are required to attach them to a wafer for etching. This is often down with a little bit of Fomblin oil or indium (to ensure thermal contact) and a waste "carrier wafer." After several uses, the carrier wafer is unusable and must be discarded. If this practice is not followed, it is possible for the wafer to break inside the chamber or to slip. In both instances, the chamber must be vented, causing interruptions in service which can be detrimental to user projects. We try to avoid this situation by training all users in the importance of using "fresh" carrier wafers.

The process chamber directly contacts the hazardous gases. The tool is password-protected to only allow tool manager to vent the process chamber. To avoid any possible hazardous gas release, the gas pods are interlocked to avoid feeding gas into the chamber if it is not under vacuum or the chamber lid is open.

C. Waste Minimization/Pollution Prevention

The corrosive gases not completely reacted in the process chamber is pumped to a waste gas abatement unit (PureAir) and is chemically turned into neutral solid waste left in the unit.

The chemical trap canister in the scrubber is sent back to the manufacturer to be reprocessed.

D. Materials Used /Waste Generated

Materials Used	Disposal Method	Amount per Use	Amount per Year	Comments
Chlorine Gas	Other	3.10 g	35.00 lb	this is a new process. The annual quantity is yet to be determined. "Per use" quantity is based on 10 min/use at maximal flow rate.
Hydrogen Bromide	Other	5.20 g	30.00 lb	this is a new process. The annual quantity is yet to be determined. "Per use" quantity is based on 10 min/use at maximal flow rate.

Boron Trichloride	Other	3.60 g	35.00 lb	this is a new process. The annual quantity is yet to be determined. "Per use" quantity is based on 10 min/use at maximal flow rate.
Silicon Tetrachloride	Other	3.80 g	25.00 lb	this is a new process. The annual quantity is yet to be determined. "Per use" quantity is based on 10 min/use at maximal flow rate.
nitrogen gas		0.00	0.00	
		0.00	0.00	

II. Identify and Analyze Hazards Associated with the Work

The following hazards were identified:

Physical Hazards:

- Oxygen deficient atmosphere
  (Area: 0735-FIRST-1-1G05)
- Cryogens (any substance or device capable of producing temperatures <= 170K)
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)
- Compressed gases (lecture bottles, cylinders, gas lines)
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)
- Compressed gas-Flammable
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)
- Compressed gas-toxic
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)
- Compressed gas-highly toxic
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)

Chemical Hazards:

- Chemicals, Hazardous (General)
- Corrosives
- Toxic metals (e.g., As, Ba, Be, Cd, Cr, Hg, Pb, Se, Ag)
  (Area: 0735-FIRST-1-1L43)

Ionizing and Non-ionizing Radiation Hazards:

- Radio frequency (RF) or Microwave sources exceeding 10 mW radiated output
  (Area: 0735-FIRST-1-1L43)

Biological Hazards:

- None

Offsite Work:

- None

Other Issues (Security, Notifications, Community, etc.):

- None

Significant Environmental Aspects

- Any amount of hazardous waste generation
  (Area: 0735-FIRST-1-1G05)
- Any atmospheric discharges that require engineering controls
  (Areas: 0735-FIRST-1-1G05, 0735-FIRST-1-1L43)
- Process Assessment Form required (determined by ECR or other qualified person)

III. Develop and Implement Hazard Controls and Assess Risk

A. Physical Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level

Hazard: **Oxygen deficient atmosphere** **Default Controls:** Contact ESH Coordinator for ODH determination and associated controls. Comply with Subject Area "Oxygen Deficiency Hazards (ODH), System Classification and Controls" **Task Specific Info:** CR & CR Galley Calculations show the level with ventilation is safe.	**Negligible (0-20)**
Hazard: **Cryogens (any substance or device capable of producing temperatures <= 170K)** **Default Controls:** General Requirements: • Evaluate location oxygen deficiency • Store/transport only in approved containers (i.e. DOT/ASME or BNL LESHC) • Never pour from above chest level • PPE: Long Sleeve Shirt (or Lab Coat), long pants (or skirt covering ankles) and closed shoes Pressurized transfer to open (vented) container; Or-Pouring > 5 liter volumes of LN2 between open containers: • Face shield along with either Safety Glasses (w/side shields) or Goggles • Gloves (Cryo or Heavy Leather) Pouring small (5 liters or less) volumes of LN2 between open containers: • Safety Goggles (face shield recommended if possible) • Gloves (Cryo or Heavy Leather) Work with samples immersed in LN2 in small (~1 liter) dewars: • Use Tongs (tools) to manipulate/handle cryogenic samples (do not touch with gloves)• Use insulated non-absorbent gloves with dexterity (cotton/nylon gloves under disposable nitrile gloves) • Safety Goggles **Task Specific Info:** State in training that there is no skin approach to the LN2 outlet when the LN2 is being used.	**Negligible (0-20)**
Hazard: **Compressed gases (lecture bottles, cylinders, gas lines)** **Default Controls:** • Any systems >15psi must be SME Approved • Transport cylinders using a cylinder cart • Secure cylinders to a fixed object/wall • Use regulator, hoses, and components compatible with gas • Use hoses and clamps rated for maximum regulator output or use pressure relief device • Wear safety glasses with side shields when installing/removing/or adjusting regulator • Label piping/tubing	**Negligible (0-20)**
Hazard: **Compressed gas-Flammable** **Default Controls:** In addition to compressed gas requirements: • Flash arrestor/backflow device • Separate 20 ft from oxidizers or barrier	**Acceptable (21-40)**

• Electrically ground lines/equipment • Non-Sparking tools **Task Specific Info:** State in training that user shall check the status of the tool to make sure gas pods are off before vent the machine. This is to let the user to be aware of any interlock fault.	
Hazard: **Compressed gas-toxic** **Default Controls:** Controls specific to the task are required. **Task Specific Info:** State in training that user shall check the status of the tool to make sure gas pods are off before vent the machine. This is to let the user to be aware of any interlock fault.	**Acceptable (21-40)**
Hazard: **Compressed gas-highly toxic** **Default Controls:** In addition to compressed and toxic gas controls: • Need to be used with a gas cabinet • Separate at least 20 ft. from flammable or pyrophoric gases or install a metal barrier Controls specific to the gas and task are required. **Task Specific Info:** gas cabinets are sprinklered gas detection monitors tied into the building fire alarms 2-man rule for chageout. SCBA to be used for connecting and opening cylinders. State in training that user shall check the status of the tool to make sure gas pods are off before vent the machine. This is to let the user to be aware of any interlock fault.	**Acceptable (21-40)**

B. Chemical Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: **Chemicals, Hazardous (General)** **Default Controls:** • All operations with large (>250ml, health hazard 3) quantities of hazardous chemicals (pouring, mixing, evaporation, etc) in hood, or use snorkel when hood is impractical • Register Commercial Chemicals in CMS • Work alone after hours only if permitted by supervisor or ESR • Identify containers so contents are identifiable unless being actively used (ex. 1 shift) • Food, beverage, smoking, and cosmetics are prohibited • Handle glassware properly: no mouth suction, no drinking from labware. **Task Specific Info:** State in training that user shall check the status of the tool to make sure gas pods are off before vent the machine. This is to let the user to be aware of any interlock fault.	**Negligible (0-20)**

Hazard: **Corrosives** **Default Controls:** Use hazardous chemicals controls plus: - Unobstructed access to Emergency Eyewash and Shower **Task Specific Info:** State in training that user shall check the status of the tool to make sure gas pods are off before vent the machine. This is to let the user to be aware of any interlock fault.	**Negligible (0-20)**
Hazard: **Toxic metals (e.g., As, Ba, Be, Cd, Cr, Hg, Pb, Se, Ag)** **Default Controls:** As for chemicals, plus need for SHSD and OMC monitoring and surveillance must be evaluated BURF for Beryllium operation **Task Specific Info:** Cr used for thin films.	**Negligible (0-20)**

C. Environmental Hazards, Tasks and Controls (include on/off site transportation and products/services)

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: **Any amount of hazardous waste generation** **Default Controls:** Engineering Controls • Waste will be accumulated in chemically compatible containers that appropriately contain/protect the waste. • Waste containers will be closed in a tray (secondary containment) in the Satellite Accumulation Area (SAA). Administrative Controls • All hazardous waste containers will have a (red) “Hazardous Waste Label” that has the generator’s name and the chemical contents (trade name/formula not acceptable). • All waste will be accumulated in closed containers and kept in an established and posted SAA until ready for transfer to the 90-Day Haz Waste Area for pick-up by Waste Management. • For pick-up by Waste management, complete the Nonradioactive Haz Waste Control Form and consult the 90-Day Area Manager to gain access/transfer the waste to the 90Day Area. Training: Hazardous Waste Gen. (HP-RCRIGEN3). PPE: When handling waste materials follow PPE requirements specified for the specific materials. Comply with the SBMS Subject Area: “Hazardous Waste Management”. **Task Specific Info:** The waste is chemically turned into neutral solid waste which is hazardless by the PureAir abatement unit and stay there until replacement of the scrubber chemical.	**Negligible (0-20)**
Hazard: **Any atmospheric discharges that require engineering controls**	**Negligible (0-20)**

Default Controls: • Maintain controls and emissions according to permit requirements, if applicable. • Maintain control equipment (such as filters) as per manufacturer's recommendations if not a permitted emissions point. Comply with the SBMS Subject Area: "Nonradioactive air emissions". **Task Specific Info:** Scrubber has a chemical trap cartridge. It has an end of life indicator.	
Hazard: **Process Assessment Form required (determined by ECR or other qualified person)** **Default Controls:**	**Negligible (0-20)**

D. Radiation Hazards, Tasks and Controls

Hazard, Default Controls, Task Specific Info	Risk Level
Hazard: **Radio frequency (RF) or Microwave sources exceeding 10 mW radiated output** **Default Controls:** Administrative Controls • Request RF survey/evaluation through SHSD • Complete JAF or Non-Employee Static Magnetic Field Questionnaire for exposed employees/workers • Training: TQ-RF/MW-SAFE for people routinely working in area • Evaluate medical device wearers • Appropriate posting (see Subject Area) Comply with Subject Area "RF and Microwave Safety" **Task Specific Info:** The unit is shielded.	**Negligible (0-20)**

E. Biological Hazards, Tasks and Controls

None

F. Offsite Work Hazards, Tasks and Controls

None

G. Other Issues (Security, Notifications to Other Organizations, Community Involvement, etc.)

None

H. Recommended Exposure Monitoring

- RF/Microwave

Description or comments:

Need initial survey.

I. EPHA Determination

Chemical Name	Quantity (lbs, gal)	Location (Bldg/Room#)
Chlorine Gas	40 lbs	735 Clean Room Galley

Hydrogen bromide	35 lbs	735 Clean Room Galley
Boron Trichloride	40 lbs	735 Clean Room Galley
Silicon Tetrachloride	17 kgs	735 Clean Room Galley

IV. Perform Work Within Controls

A. Recommended Training and Medical Surveillance Summary

- Laboratory Standard (HP-IND-220)
- Cryogen Safety (HP-OSH-025)
- Hazardous Waste Generator (HP-RCRIGEN3)
- Respirator Medical Surveillance (OM-MEDSURV-RESP)
- Compressed Gas Safety (TQ-COMPGAS1)
- Oxygen Deficiency Hazard (TQ-ODH)

B. Personnel Training, Qualification, and Authorization List

Employee/Guest Name	Life/Guest#	Dept	Required Training Course(s)	Signed
Ming Lu	24188	NC	Oxygen Deficiency Hazard (TQ-ODH) [EXPIRES: 4/6/2013] Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 4/8/2012] Laboratory Standard (HP-IND-220) [EXPIRES: 4/11/2013] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 4/7/2012] Respirator Medical Surveillance (OM-MEDSURV-RESP) [UNASSIGNED: EXPIRES: 2/22/2013]	9/23/2011 1:37:47 PM
Aaron Stein	22947	NC	Oxygen Deficiency Hazard (TQ-ODH) [EXPIRES: 1/5/2014] Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 3/2/2013] Laboratory Standard (HP-IND-220) [EXPIRES: 2/28/2013] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 4/11/2012]	
Fernando Camino	23799	NC	Oxygen Deficiency Hazard (TQ-ODH) [EXPIRES: 8/24/2013] Cryogen Safety (HP-OSH-025) [EXPIRES: NEVER] Compressed Gas Safety (TQ-COMPGAS1) [EXPIRES: 3/4/2013] Laboratory Standard (HP-IND-220) [EXPIRES: 8/24/2013] Hazardous Waste Generator (HP-RCRIGEN3) [EXPIRES: 8/17/2012] Respirator Medical Surveillance (OM-MEDSURV-RESP) [UNASSIGNED: INCOMPLETE]	1/4/2012 2:30:50 PM

C. Emergency Procedures

DO NOT DISROBE FIRST.

Following the Bldg. 735 Local Emergency Plan. Spill kits located in NE Service Galley and in 90-day area. Eyewash and shower in Lab and galley.

HF emergency kit located next to hood in 1L-43, refer to special provisions detailed in standard operating procedure for HF.

There is a status light tree, green, yellow, and red in the clean room and galley. The green light indicates all systems are normal. Yellow indicates a fault which needs attention by the system owner. Red indicates a Level 1 Alarm, which indicates either a gas detection or problem with exhaust system. This will stop the process until the problem is resolved.

If **Level 2 Alarm** is tripped, there will be a **special temporal 3 alarm** and a message **to evacuate the building due to a Toxic Gas release**. The message will tell everyone to exit out the north doors (front) only.

In the event of a TOXIC GAS release. There will be a temporal 3 alarm followed by a message to exit out the north(front) doors only. After evacuation go to the outdoor assembly area on the west side of the building.

D. Transportation

Laboratory users bringing samples into the laboratory for processing should be aware of the SBMS requirements for transporting hazardous materials and transporting nanomaterials. Sample preparation other than that described in this document must be done in an environment appropriate for chemical handling. Samples must be shipped through the BNL Shipping Office.

E. Logistical Interactions

In an emergency call x2222 or 911. From a cell phone call 631-344-2222. Notify the ES&H Coordinator x3509.

F. Termination/Decommissioning

This is an ongoing research facitlity. If any equipment is to be decommissioned it would go throgh an EMR. Waste will be handled and wasted through WMD.

V. Provide Feedback

This is a new experiment.

VI. Attachments

Attached Files:

SOP-NC-2007-ELM-Cryo5.doc

SOP CR orientation.pdf

ODH Calc 1G05.mht

ODH Clean Room 1L43.mht

Maintenance of Oxford RIE_Monthly Checks.doc

Maintenance of Oxford RIE_6_month_yearly.doc

Maintenance of Oxford RIE_Weekly Checks.doc

Install lecture bottles in RIE gas cabinets.doc

RIE-C_process_cylinder_changeout_20110621_RLS_mod_20110706 (2).docx