

## **RULES FOR USING THE INESC MN CLEANROOM**

(Updated: 25 March 2022)

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# General information

## Contacts:

### INESC MN Supervisors:

Susana Cardoso de Freitas:	Ext. 2348 (213 100 348)
Paulo Freitas:	Ext. 2348 (213 100 348)
João Pedro Conde:	Ext. 2231 (213 100 231)
Virgínia Chu:	Ext. 2231 (213 100 231)

*The Ext (Extension) is only available on INESC MN telephones. Otherwise use the 9-digit number in parentheses.*

### Process Engineers:

Virgínia Soares, José Bernardo,	
Fernando Silva, Rita Macedo:	Ext. 2504

### Administrative:

Natércia Correia	Ext. 2237 (213 100 237)
Sandra Baptista	Ext. 2220 (213 100 220)

*The Ext (Extension) is only available on INESC MN telephones. Otherwise use the 9-digit number in parentheses.*

### Security Office:

Ext. 2222 (213 100 300)

*The Ext (Extension) is only available on INESC MN telephones. Otherwise use the 9-digit number in parentheses.*

### Emergency Medical Services (INEM):

112

## General Rules:

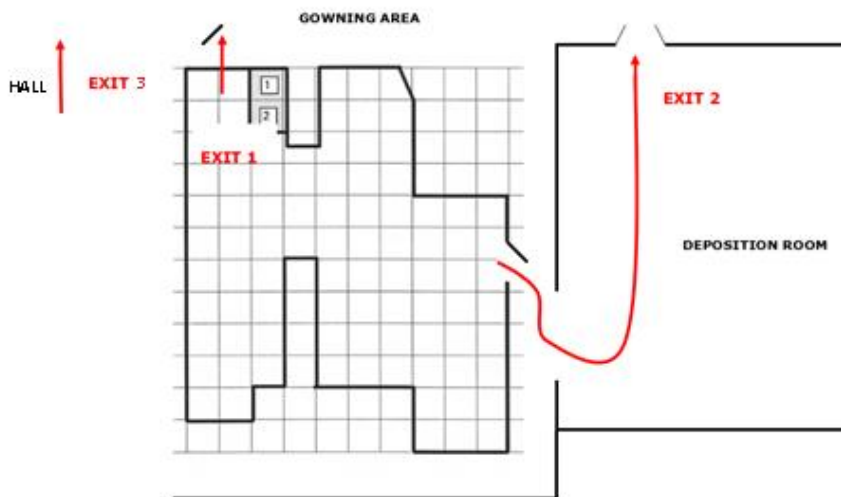
1. All personnel who will need to work in the cleanroom must undertake the training course and read all the rules in this document (\\microsrv02.INESC MN.pt\Transfer\INESC MN SAFETY). Only authorized personnel will be allowed inside the cleanroom
2. Locate a First Aid Kit in every room. If you injure yourself, be sure to seek assistance immediately
3. **Do not eat or drink** in the working area (chewing gum is not permitted as this can cause you an intoxication)
4. It is strongly recommended not to **use contact lenses** in the lab
5. **Open-toed shoes, sandals** and **shorts** are not allowed
6. Do not wear dangling jewellery or items that may get tangled with a tool
7. **Bare legs** and **arms** must be covered by wearing proper protective pants when working with chemicals
8. Keep your **hair tied** when working in the wet bench
9. Avoid entering the cleanroom if you are sick
10. The use of mobile phones inside the cleanroom is not permitted when working close to wet benches or chemicals. Phones should not be kept inside your cleanroom gowning if you plan to use it inside the cleanroom, as opening your suit will contaminate the clean environment
11. Only dedicated tools are allowed in the cleanroom
12. Take into the cleanroom only what is necessary to minimize contamination of the cleanroom. Any material or activity that makes dust or particles are prohibited in the cleanroom (such as: cleaving wafers, tearing paper/wipes, blocking airflows, exposing hair, impeding laminar flow, rapid movements). Materials that are not permitted inside the cleanroom include: Regular Paper, Cardboard, Wood, Make-up, Pencils, green paper towels, and packaging materials. Items must be removed from its cardboard packaging before going in. Paper must be either cleanroom paper or if using regular paper, it must be inside a plastic sleeve
13. Do not handle wafers/samples or bottles of chemicals without proper equipment (gloves; apron; etc)
14. Maintain good airflow management – do not block the airflow from the HEPA (High Efficiency Particulate Air) filters in the wet benches nor the exhaust at the back of the wet benches
15. **Work responsibly and clean after.** This is critical to avoid damaging or negatively impacting your work and those of other people working around you. If you finish any consumable (eg. resist,

acetone, IPA, clean room wipes, glass substrates, multimeter batteries, aluminum foil, parafilm, etc.), **you are responsible to refill it or immediately ask the person responsible to refill it.**

16. Read **MSDS** (Material Safety data sheet) before handling hazardous substances. You can find them in the folder \\microsrv02.INESC MN.pt\Transfer\INESC MN SAFETY\INESC MN MSDS. If you are using a new chemical, you will need to validate with the process engineers if you can take it to the clean room. Do not forget to add the MSDS on Transfer
17. Never block safety equipment such as eyewash, showers, or emergency exits. Keep aisles clear and free of tripping hazards
18. All equipments have log book. **Always review the log book before using the equipment, record your name, date, materials used, process run details, and any errors or maintenance done to the equipment.** Annotate when you are done using the equipment in order to let others know that the tool is available for the next user
19. Each equipment has specific sample holders, which are not inter-exchangeable (eg: sample holders from N7000 cannot be used in N3000). These should be kept cleaned and stored near the machine. Maintaining sample holders in order and cleaned will prevent cross-contamination and potential tool downtime
20. Small equipment/consumables at the cleanroom need to stay in the cleanroom. Do not remove pens, multimeter, diamond scribes, lamps, tools, aluminium foil, ... Keep everything from pens to tools (screwdriver, wrenches, etc.) in their respective places at each laboratory and toolboxes so they are easily accessible to everyone
21. Escorted Guests or Visitors: Guests should always be escorted in the cleanroom (they should never be left alone). Visitors that will work by themselves will need to go through the safety and cleanroom protocol training
22. **All processes should follow a detailed run sheet, with the step-by-step description of the microfabrication methods used.** The run sheet should be validated by the adviser/coordinator before starting and should be printed and carried with the sample throughout the process

### Emergency Procedures:

1. **Alarm:** if you hear an alarm and you are in the cleanroom:
  - a. Leave the cleanroom immediately. There are 2 exits of the cleanroom (see map below):
    - i. The normal door used to enter the cleanroom through the gowning area - use this door unless it is blocked (**Exit 1**).
    - ii. The door near the N3000 that leads to the grey area. If you use this door, you can exit through the door in the deposition room (**Exit 2**). This door is for emergency use only and should never otherwise be used except for moving large equipment.



b. Leave the basement by the stairs – if the stairs are blocked and you cannot exit that way, go up the ramp and remove the key that is located on the wall to the right of the garage door. An alarm will go off if this door is opened so only use it in case of an emergency.

c. Inform the security at the front door - report that there is an alarm to the security guard

d. Report the alarm to the Process Engineers, Supervisors, Administrative INESC MN:

- i. Virginia Chu, ext. 2231
- ii. Susana Freitas, ext. 2348
- iii. José Bernardo, Fernando Silva, Virginia Soares, Rita Macedo, ext 2504
- iv. Natércia Correia, ext 2237

Once the incident has been reported, the alarm will be checked for false or true. If it is true alarm, follow instructions and, if necessary, evacuate the building.

2. **Accident that requires medical attention:** if an accident occurs in the cleanroom or anywhere on the premises of INESC MN that requires medical attention, you need to:

- a. ask for help – never work alone in any of the labs/cleanroom. Always make sure you have a safety buddy (a person who may help you in case of need) in the same room as you. Tell him immediately what happened and ask to call for help. If the injury is serious and requires medical attention, immediately call for medical help.
- b. report the incident to INESC MN supervisors or Administrative: all incidents that involve the need to seek medical attention should be reported to INESC MN responsables.

3. **Off-hours Emergencies** - if an emergency occurs outside of normal working hours (after 7pm or on weekends),

- a. Contact the security guard immediately, tell him/her what happened and ask them to call for help.
  - INESC Security guard: ext. 2222
  - General emergency: 112
- b. Report the incident to INESC MN supervisors or Administrative: all incidents that involve the need to seek medical attention should be reported to INESC MN responsables. INESC

security guards have the home phone numbers of INESC MN responsables if they need to be contacted during off hours.

## Gowning

It is absolutely NOT PERMITTED to enter the cleanroom without first properly gowning up. Below are listed some rules regarding cleanroom gowning:

1. Follow this order when gowning:
  - a. Blue shoe covers
  - b. Hair net and Hood
  - c. Protective Coverall
  - d. Boots (at the bench)
  - e. Gloves
  - f. Safety Glasses (if a wet chemical process is to be done)



2. Always make sure that all head/facial hair is covered once inside the cleanroom – do not expose any facial/head hair. If you have beard or moustache, you are required to wear a face mask. For other people, a face mask is preferable, but not mandatory
3. The hood should be tucked inside the coveralls
4. Tuck the pant legs of your coverall inside your boots

5. Always wear gloves when entering the cleanroom. Gloves can be reused if they are not broken or covered with something (photoresist, tape, etc.)
6. Keep cleanroom garment closed at all times while inside in the cleanroom – do not expose the cleanroom environment to any street clothing
7. Always use a new face mask and put the old one to wash. Everything else (coverall, head cover, boots) can be reused, and send to wash only when dirty. A general rule is to exchange the garment for new ones after you have worn it for 40-50 hours. If you are in the cleanroom intensively, you may need to change the garments every week. If you only go in for a few hours per week, you can keep them longer
8. When leaving the cleanroom, hang your coverall using a designated hanger with your name. If you are a visitor, use the numbered hangers and write your name on the available user sheet taped near the gownroom door and hanging rack
9. Do not open the gown room and clean room doors simultaneously. Close one before opening the other to prevent air contamination
10. It is not permitted to wear your cleanroom garment in the grey area. If you need to leave the cleanroom, you must remove your cleanroom garment
11. If you need new garment elements, ask José Bernardo for them.

## Chemical Wet benches

- Clean up your glassware after each use
  - If weak acids are used, clean with: 1. DI water and 2. hang to dry
  - If organic solvents are used (e.g. microstrip) then clean with: 1. acetone 2. IPA 3. water, and 4. hang to dry
  - Handling strong acids requires special training. These include Nitric acid ( $\text{HNO}_3$ ), Hydrofluoric acid (HF), Sulfuric acid ( $\text{H}_2\text{SO}_4$ ), Hydrochloridric acid (HCL), etc. Piranha solution:  $\text{H}_2\text{O}_2$  is added to  $\text{H}_2\text{SO}_4$  and not the opposite. HF: glassware cannot be used. Instead use plasticware
- Special procedures which require long periods of wet bench usage, need to be scheduled in advance and well indicated
- Never place organic solvents (acetone, IPA, etc) close to or directly on hot plates because of fire-risk. Organic solvents may only be heated in a beaker inside the water bath
- Keep organic liquids strictly separated from acids, peroxides, alkalines, etc, because of explosion risk
- After resist strip, evaluate if the microstrip can be reused. If so, ask Virginia Soares for a container to keep your own microstrip for reuse. These containers must be kept under the wet bench with the following information written on the bottle: your name, date, and chemical identification (eg. Microstrip)
- The aluminium etchant can be reused for 2 or 3 days. Ask Virginia Soares for a bottle and funnel to transfer unwanted etchant. Label it with your name, date, and chemical name

- Do not obstruct the holes at the back of the wet bench, as this is the exhaust of the bench. Avoid Inhalation and exposure to vapour when working in the laminar flow of the clean benches
- When working with acids, never work alone. Wear goggles, apron and proper rubber gloves. Identify the chemicals you are using if you have to be away from the wet bench even for a small amount of time. Do not touch anything else with the gloves, you could be transferring acid to doorknobs, etc... If the gloves have been contaminated with acid, remove them, wash them with plenty of water and discard them
- Chemical Spill: If you have a major chemical spill be sure to call for assistance immediately.
  - If you spill chemicals on yourself:
    - Acids and alkalines: rinse directly with diphotérine (in First Aid Kit) and look for assistance. If you come in contact with HF, rinse 1 to 5 minutes with water and then apply the calcium gluconate gel located in the First Aid Kits. It is advisable to seek medical attention if you suspect a direct contact with HF or other strong acid.
    - Organic liquids: absorb the spill with a tissue and wash the spot well with soap and water. Let residues from the tissue evaporate on wet bench near ventilation. There is an eye wash and shower inside the cleanroom and near the outside wet bench.
- Waste disposal:
  - Do not throw anything in the drain (except for Alconox which is a detergent). There are dedicated labelled waste containers on the floor next to the wet bench. If the container is reaching 2/3 of its capacity inform Virginia Soares
  - The capacity of waste containers should never surpass 2/3.
  - Disposal of strong acids/bases has specific procedure and requires special training
- Wet bench located outside (in the Hall, near the entrance to the cleanroom):
  - All the rules above also apply here
  - If using the heated bath, be sure to check regularly that the system has sufficient water to prevent overheating which can lead to fire hazard. This also applies to the ultrasonic bath
  - The temperature of the heated bath should never surpass 65 °C to prevent overheating which can lead to fire hazard
  - Keep this area clean to prevent contamination in cleaner areas: Clean room Class 100 and Grey area Class 10000
  - The microstrip waste container should always be changed before the liquid reaches 2/3 of container capacity. If the container is reaching 2/3 of its capacity inform Virginia Soares. If reusing the microstrip, remember to ask for your own personal bottle to store it. Label it appropriately: your name, date, and chemical identification
  - If you drop samples or chemicals (e.g. Alconox or Microstrip) in the heated bath, you need to:
    - samples remove the samples to avoid clogging the drain;
    - chemicals drain the water, clean the tank and refill with clean water.



- Make sure to be careful not to clog the water drain with samples, sample pieces, etc. If you find it clogged, unclog it immediately for safety reasons and inform Virginia Soares.

## Disciplinary Action

The rules outlined in this document are to help safeguard your safety, the safety of your colleagues and to avoid damage to equipment or infrastructure. Some rules are simply guidelines to respectful behavior when working in close proximity to other people. All the rules should be followed.

Make sure you know all the rules before entering the cleanroom. If you have questions or doubts, ask before proceeding. Accidents happen, but accidents happening because you did not follow rules will not be tolerated. Depending on the gravity of your fault, you will be:

- warned and will be instructed to the correct behavior;
- suspended from using the cleanroom and will have additional training;
- expelled from INESC MN

## Micro and Nanofabrication Equipment and Processes

Equipment	Responsible
Alcatel magnetron sputtering system (targets: SiO <sub>2</sub> , Cr, Al, ITO, IZO, AZO, GeSbTe, Si, Ti, Ni, Au)	Fernando Silva
Autoprober setup for 6" wafer electrical characterization	Sofia Abrunhosa
Centrifuges: Thermo Fisher Scientific	Vania Silverio
CMP: Bruehler EcoMet 250 grinder/polisher	Susana Cardoso
Corona BD-20 discharge system, Electro-Technic	Virginia Soares
Device noise measurement setup (wire-bonded encapsulated samples)	Susana Freitas
Dicing saw: Disco DAD 321 automatic dicing saw, up to 6" wafer	Virginia Soares / Susana Cardoso
Diffractionmeter: Siemens D5000 X-Ray diffractometer	Fernando Silva /Susana Cardoso
Direct write laser lithography system (DWL ii) Heidelberg Instruments	José Bernardo
Electrical MEMS resonance setup	Virginia Chu
Ellipsometer Rudolf Auto EL IV-NIR-3	José Bernardo
Extrusion based 3D printing – Fused Deposition Modeling system	Vania Silverio
Incubator: Scientific Heratherm	Vania Silverio
LAM Research Rainbow plasma etcher system	Virginia Soares
Magnetic annealing setups	Susana Cardoso
Magnetoresistance measurement setup (140 Oe; 400 Oe; 2kOe)	Sofia Abrunhosa/ Pedro Araújo
Magnetoresistive scanner	Susana Cardoso/ Sofia Abrunhosa
Microscope: Leica DMLM fluorescence microscope	Virginia Chu
Microscope: Olympus CKX41 fluorescence microscope	Virginia Chu

Microscope: Nikon Eclipse LV-N metrology industrial microscope	Débora Albuquerque
Microscope: Olympus BH3-MJL industrial microscope	Virginia Soares
Milling: 3D CNC MiniTech micromilling system	Vania Silverio
Milling: 3D CNC Supertech micromilling system	Vania Silverio
Nanoplotter: GeSiM Nano-Plotter NP2.1	Débora Albuquerque
Nordiko 2000 magnetron and RF sputtering system (6 targets, e.g. Ta, Ru, MnIr, MnPt, NiFe, CoFe, CoFeB, Pt, MgO, AlOx, Ti)	Paulo Freitas / Fernando Silva
Nordiko 3000 Ion beam deposition and milling system (6 targets, e.g. Ta, Ru, MnIr, MnPt, NiFe, CoFe, CoFeB, Pt, MgO, AlOx, Al, Au, Ti, etc)	Susana Cardoso
Nordiko 3600 Ion beam deposition and milling system (6 targets, e.g. Ta, Ru, MnIr, MnPt, NiFe, CoFe, CoFeB, MgO, AlOx, Al, NiFeCr, etc.)	Susana Cardoso / Rita Macedo
Nordiko 7000 magnetron sputtering system (targets: Al <sub>98.5</sub> Si <sub>1.0</sub> Cu <sub>0.5</sub> , TiWN <sub>2</sub> )	Fernando Silva
Oxford PECVD System	Fernando Silva / Virginia Chu / Rita Macedo
PECVD: Aixtron NanoInstruments Black Magic PECVD system for graphene and carbon nanotubes	Fernando Silva
pH meter, Mettler Toledo	Débora Albuquerque
Plasma Cleaner: PDC-002CE, Harrick Plasma	Virginia Soares
Profilometer: DEKTAK 3030T	Virginia Soares
Profilometer: Tencor Alpha-Step 200	Virginia Soares
RAITH 150 e-beam lithography system	João Serra/ Pedro Araújo / José Bernardo
RAITH 150 SEM	José Bernardo
Scanning Electron Microscope: SEM Hitachi S-2500	Virginia Soares
Soft lithography Lab	Virginia Soares / Vania Silverio
SPTS Omega ICP etch system	Rita Macedo / Fernando Silva
Stratalinker UV Crosslinker	Vania Silverio
SVG resist coater and developer track	José Bernardo
Techne Prime Thermal Cyclers	Vania Silverio
UHV-I magnetron sputtering system (target: mosaic CoZrNb)	Fernando Silva / Susana Cardoso
UHV-II RF sputtering system (target: Al <sub>2</sub> O <sub>3</sub> )	Fernando Silva
UVO-cleaner: Model 144AX series, Jelight Company	Virginia Soares
Vapor prime oven: Yield Engineering YES 15 HMDS vapor prime oven	Virginia Soares
Vibrating Sample Magnetometer: DMS 880 VSM	Pedro Araújo
Wet benches	Virginia Soares
Wire bonding system, Kulicke & Soffa	José Bernardo

# Rules for Specific Equipment and Processes

## General

The following are rules and guidelines for using cleanroom equipment and processes. This is only for those people who have already been trained and authorized to use the equipment. No one should use equipment without having authorization from his/her supervisor and being trained and signed-off by the responsible for the equipment.

Anyone who is trained and authorized to operate an equipment alone but is not authorized to operate the machine in manual mode should not use the machine off hours (i.e., after 19h or on weekends). You should only operate equipment when the responsables are around to help if there should be any problems. If an equipment fails or does not operate properly, you need to inform the responsible of the equipment right away. Do not try to fix it yourself.

## Micro and Nanofabrication Equipment and Processes

### Photoresist Track [responsible: José Bernardo]

- (1) Use only clean wafers as support substrates. Clean the backside of the wafer with acetone if needed. Clean them at the wetbench so the acetone fumes are exhausted.
- (2) If the Silicon wafer is broken, it should be put in the trash can, never left on the table.
- (3) For developing, just open the nitrogen top line.
- (4) Turn ON the nitrogen black valve only if you need to coat.
- (5) To purge the photoresist line, just press the purge manual control 2 or 3 times. It is enough to fill the resist tube.
- (6) When the coat step is finished, you can see (on the heat station) if the resist is uniform. If this is the case, you can now turn OFF the black valve of nitrogen (bottom one).
- (7) Turn OFF nitrogen after you coat or develop.
- (8) If you need to strip the resist (prog# 8/6) you do not need to open the nitrogen black valve, use only the top nitrogen valve.
- (9) Remove tape from your sample and put it directly in the trash. Do not glue your tape in the SVG frame, in the support wafers or your gloves.
- (10) If you make a mess, clean it up. Return everything to its original condition, or leave conditions better than you found them.
- (11) Check the list before loading a wafer for coating/developing. This list is in the front part of the machine.
- (12) When using the track, please follow the following steps:
  - Open Compressed Air and Nitrogen (top line....if you want to **coat** please also open the Nitrogen **black line**
  - verify if vacuum and water are open. Usually they are ...
  - Power ON booth card cages ( if they are off).
  - Insert you **wafer(s)** on the **SEND cassette** and confirm that you are using the same **slot** number for each wafer – otherwise the 1 st arm will not pick up corretly.
  - Select the program to use in the corresponding card cage.
    - o Coater + oven
    - o Developer + oven

- Program Select is used to access one of the main process programs.
- Station Select toggles between the process stations controlled by the card cage.
- To coat you need to **purge the line 2 or 3 times** so press the pump red button and see if the resist tube is full with resist. You need to **WAIT 30 seconds** in between each purge.
  - o Select wafer processing mode **AUTO** and then press **START**.
  - o To process a wafer manually select first **MANUAL** and load your wafer in the corresponding station (coater/oven or oven/developer) – press **1/2** according to what you want to do and then press **START**
  - o Never leave the machine work alone. You should be always near the machine during a process
  - o At the end **turn OFF** Compressed Air and Nitrogen.
- Shut down sound alarm with **CLEAR** on Developer card cage keypad.

(13) If you need to dilute the e-beam resist, do it at the wet bench, with safety glasses and gloves, and label the beaker with your name, name of chemical, concentration, and date. Leave the beaker inside the wetbench until you are ready to use it. The fumes are extremely irritating.

## Direct Write Lithography System – DWL [responsible: José Bernardo]

- (1) Please schedule your runs in advance (<http://www.supersaas.com/schedule/INESC MN/DWL>). Do not sign-up for more time than you think you may need it.
- (2) Only people that are trained to use DWL are allow to access Schedule.
- (3) Use only clean wafers as support substrates. Clean the backside of the wafer with acetone if needed. The silicon wafers are expensive (even if dummy) so care should be taken to avoid breaking the wafers.
- (4) For the lithography, remove tape from the sample.
- (5) Control Panel:
  - a. Do InitStage before you load your sample.
  - b. Before focusing your sample, be pretty sure that it is below the nozzle.
  - c. The nozzle can be permanently damaged if
    - i. the stage moves while performing the **Focus** function, or
    - ii. focusing is done on the edge of a substrate, or
    - iii. focusing was done beside the substrate and the stage is then moved so that the substrate crashes against the nozzle.

With a damaged nozzle, exposure results will deteriorate dramatically, or exposures might even not be possible any more.
- (6) If the wafer from the previous exposure is still on the chuck when you arrive to do your exposure, please remove the wafer and place it inside the cassette next to the DWL computer, labelled EXPOSED. If you place it somewhere random and it has not been developed, it can be mistaken for a plain silicon wafer and be ruined.

## 8" Wafer Coater and Developer System [responsible: José Bernardo]

### COATER – 8"

1. turn ON vacuum pump (grey area)
2. turn ON nitrogen (yellow room)
3. turn ON coater
4. load recipe " 8resist "
5. load wafer and confirm that is well center apply vacuum and close the cover START dispense resist, manually, from the Top window
6. in the end you MUST turn OFF nitrogen
7. in the grey area turn OFF vacuum pump

### DEVELOPER – 8"

- |    |  |
|----|--|
| 1. | turn ON vacuum pump (grey area)  |
| 2. | turn ON DI water (grey area)   |
| 3. | turn ON nitrogen (near Nano-Plotter)   |
| 4. | turn ON nitrogen (yellow room)   |
| 5. | turn ON developer  |
| 6. | load recipe "Center"   |
| 7. | load wafer and confirm that is center - use the special tool to apply vacuum and close the cover |
| 8. | START  |

*\*\* if needed, repeat this process until the wafer is well center load recipe " 8develop ", then START*

In the end you MUST turn OFF nitrogen (both lines) in the grey area turn OFF vacuum pump and close DI water valve

## Vapour Prime Oven [responsible: Virginia Soares]

- (1) Do not put samples mounted on a 6 inch wafer inside. We do not know how the tape reacts to the HMDS.
- (2) Do not open the vapour prime until the display indicates it is in step 41 or 42. Otherwise the purging of the chamber might not be complete.
- (3) The vapor prime is not a storage facility. Samples left inside for several days will be removed..

## Reactive Ion Etching System – LAM [responsible: Virginia Soares]

- (1) Check the backside of the support wafers you use: it should be clean, check for tape or PDMS residues. If the backside is not clean, wipe it with acetone or IPA. Tape or PDM residues in the backside may lead to wafer breakage in the chamber as the arm will not pick them up correctly. They should not have metal deposited on the backside either. Do not handle them without gloves.
- (2) For etching, the support wafers should always be covered with photoresist (it would be perfect if you can use the wafer you used for lithography). Do not mark it with ink because this will contaminate the machine.
- (3) For PDMS processing remember that the sample thickness should be 1mm at the most, otherwise it will be, in the best of cases, stuck when it goes inside the Entrance Load Lock. It can also contaminate the chamber.
- (4) If you need to change the etch time in the recipe, save it either as a temp file or change it back to the original time when you are done.

## Metallization System – Nordiko 7000 [responsible: Fernando Silva]

- (1) Write down in the log book the base pressure of the modules before starting a process and the machine status in a daily basis (include the date).
- (2) Write down the read parameters during the processes (not only the set point values).
- (3) Write down your name, batch number, sample reference and project related.
- (4) Do not change pre-defined functions. If power or other deposition conditions are needed, a new function needs to be set.
- (5) Check the backside of the support wafers: tape residues in the backside may lead to wafer breakage in the chamber as the arm will not pick them up correctly. Greasy residues (PDMS or resist) are contaminants for the high vacuum systems.
- (6) Always keep the metallic holders clean and stored near the machine.
- (7) Always pump the loadlock IMMEDIATELY after placing/removing a wafer (this saves Nitrogen). Loadlock door must be closed carefully to avoid damaging sealing O-Ring.
- (8) After lithography, samples cannot be mounted directly onto the Nordiko holders for metallization: sample back side MUST be cleaned with acetone to avoid contamination of the metallic holders.

## Ion Beam Systems – Nordiko 3000 and N3600 [responsible: Susana Freitas]

- (1) Always use the dedicated metallic holders, and respect the sample dimensions allowed for each machine. Check the backside of the support wafers: tape residues in the backside may lead to wafer breakage in the chamber as the arm will not pick them up correctly. Greasy residues (PDMS or resist) are contaminants for the high vacuum systems.
- (2) The Nordiko 3000 and 3600 machines are not general purpose tools, and are dedicated to thin film deposition.
- (3) Number your samples sequentially, using the machine name convention (eg: 36svxxxx or TJxxxx). Never use your own number as this helps maintain a control of process.
- (4) After venting loadlock always close the vent valve manually. Loadlock doors must be closed carefully, to avoid damaging the sealing O-ring.
- (5) In the N3000 system, the cassette must be placed correctly (= horizontally) to avoid loading problems.
- (6) Write down in the log book the base pressure of the module before starting a process, your name, sample description, and project related. Write down the read parameters during the processes (not only the set point values).
- (7) Do not change pre-defined functions. If different process conditions are needed (beam current, power, gas flow, angle), a new function needs to be set by the responsible.
- (8) These systems are priority for film deposition. Milling has to be done after the deposition processes finish, to avoid unnecessary cleaning of the targets (targets are very expensive). Several users should combine their milling processes (= several samples together) to minimize the machine usage time and gas waste.
- (9) After lithography, samples cannot be mounted directly onto the Nordiko holders for etching: sample back side MUST be cleaned with acetone to avoid contamination of the metallic holders.

## Oxford Plasma Pro PECVD [responsible: Fernando Silva / V. Chu]

- (1) Only authorized persons can operate this system. If you want to deposit films or be trained to be an authorized operator contact Fernando Silva or Virginia Chu.
- (2) All depositions must be reserved online  
([https://www.supersaas.pt/schedule/Oxford\\_Plasma\\_Pro\\_PECVD/Oxford\\_Plasma\\_Pro\\_PECVD](https://www.supersaas.pt/schedule/Oxford_Plasma_Pro_PECVD/Oxford_Plasma_Pro_PECVD)).

- (3) Plasma cleaning must be done between silicon dioxide / silicon nitride and amorphous silicon depositions so you must check what was previously deposited and if a plasma clean is needed, you must contact Fernando Silva or Virginia Chu.
- (4) Plasma cleaning must be done every
  - i. 8-10 microns of deposition of SiO<sub>2</sub> and/or SiN or;
  - ii. 5-6 microns of deposition of a-Si:H
- (5) All depositions and plasma cleans need to be noted in the notebook. The total amount of deposited film must be noted in the notebook in order to know the accumulated amount of deposited film for the purpose of scheduling the plasma clean.
- (6) If anything out of the ordinary occurs while you are using the machine, note this in the notebook and inform Fernando and Virginia Chu

## Chemical Mechanical Polishing System [responsible: Susana Freitas]

- (1) Before starting the CMP please always check the following:
  - the container in the back of the CMP: if it's almost full you should throw away its contents or it will spill onto the table without your noticing (that's why the table is full of dirt).
  - the collection system that delivers the liquid excess to the container: if it is too full of solid residues it will end up being obstructed. In that case you should try to remove them as best as you can.
  - the table contour: check that there is no accumulation of solid waste.
- (2) After using the CMP, you should remember to do/check the following:
  - clean the pad carefully: the slurry that is being used is difficult to clean so we have to be extra careful.
  - clean the cup used to spill the slurry.
  - clean the sample holder and the holder responsible to keep the rotation.
  - remove the excess of water from the pad border with a piece of paper to prevent accumulation.

## ***Biological Processes and BioLab***

### **General**

- a) Cleanliness is a must for using biolab equipment and materials. Please remember that even a minute contamination (including not only bacterial contamination but also reagents from previous experiments) might have drastic effects reflected in biological experiments.
- b) Clean the area with 70% ethanol (EtOH) or Isopropanol (IPA) before use and after the experiments. Always leave the experimental area clean.
- c) Use of a lab-coat is strongly recommended. Do not handle materials, such as syringe tips, pipette tips and eppendorfs without gloves.
- d) The use of corrosive chemicals and toxic volatiles needs to be brought to the attention of the biolab users, for safety and health reasons.
- e) Ask for using a chemical in case you do not know about it, as there might also be potential carcinogens and other hazardous chemicals present in the list.
- f) Ask for instructions before using any of the pieces of equipment present in the biolab if it is the first time you intend to use them.



- g) Be cautious not to interfere with on-going experiments (for example, switching the lights on before checking with the colleagues, as there might be some experiments involving fluorescent molecules in progress).
- h) In case you take something out of the fridge or freezer, please make it a point to ensure that the doors are closed properly. Lethargy or indifference in this might lead to the loss of very expensive materials stored inside from all the users.
- i) Do inform and alert the colleagues in case of any accidental abnormality (for example, the door of the freezer found open, some -20°C chemicals/boxes found outside, etc.) so that appropriate measures can be taken to rectify the damage caused.
- j) Always use the centrifuge with a balancing tube on the opposite side.
- k) The same applies to multi-channel syringe pumps: the syringes must be placed from the outer to the inner positions and always equilibrated on both sides.
- l) If some material is the last piece or vial or packet you use, make it a point to inform the person responsible for purchase, so that nobody runs out of materials or time due to delayed ordering or delivery.
- m) Do not leave vials or tubes or syringes with cells or microbes just like that in the fridge – it is forbidden and might be a bio-hazard! Discard as per bio-disposal norms.

## Microscopes

- a) As there are two microscopes (one inverted and the other non-inverted) available for fluorescence modes, check for the purpose and availability.
- b) Do not use the microscope without any orientation or user-guidelines from the colleagues, in case you are new to the equipment and/or software.
- c) If you change any settings in the software (image field view, binning, image type, etc.) or any settings in the microscope (use of DLF, mirrors, etc.), please make it a point to put them back to the same parameters as how you found them before use.
- d) The power supplies beside each of the Olympus and Leica microscopes are connected to short-arc mercury lamps, contained inside appropriate protective housings, which are to be used for fluorescence measurements ONLY.
- e) Mercury short-arc lamps irradiate at a high intensity in the near UV range, which is very harmful for your eyes. Thus, if you have no fluorescence filter (position 1 on the Leica) or using a UV fluorescence filter (position 2 on the Leica or UV labelled cube in the Olympus), DO NOT look directly at the light without some protection (filter glasses or dark plastic board in the Olympus). This is even more relevant if you are imaging highly reflective surfaces in the Leica microscope, such as a silicon piece.
- f) Do not move the microscopes and the fluorescence power supply for any reason. In the case of the Leica microscope, this can lead to lamp misalignment.
- g) Clean the microscopes, objectives and other accessories only with appropriate wipes and cleaning solutions. Do not leave the objectives or the microscopic stage dirty, in case there was a spill from some solution used.
- h) After use, carefully move the stage far away from the objective lenses to avoid accidents breaking the objective nose-piece.
- i) Make entries in the log-book regularly without fail, for every use of the mercury lamp power supplies.
- j) Before switching on the mercury lamps, always check the registration of the previous user in the log-book. The lamp must have been switched off **at least before the past 20 minutes**. Otherwise, one needs to wait for a **minimum of 20 minutes** (from the previous use) before switching it on again. In the same way, when the lamp is switched on, it must not be switched off immediately. One needs to **wait at least until 20 minutes** for this. Failure to comply with the above may compromise the work of everyone due to lamp intensity degradation or even risk of explosion (and release of harmful mercury vapors) if the lamp is not allowed to cool down before turning it on again.



## Micropipettes

- a) Get to know about the micropipette you use, its measuring capacity, value set-up/regulation and how it works.
- b) Clean the pipettes before and after use, and check them for any obvious damage (nose of the barrel – where the tip is fitted).
- c) Never put the pipette on its side with liquid in the tip.
- d) Use only well-fitting tips. Poorly fitting tips allow air to escape when drawing up and dispensing, leading to inaccurate results.
- e) Use a sensible pipette for the volume you need to dispense, and be gentle while pressing and releasing the push button to avoid the fluid entering the filter.
- f) Beware of the two stop positions in the micropipette, and use them accordingly for wetting and reverse-pipetting.
- g) Never set volumes lower or higher than the recommended pipette range and leave the pipettes in their maximum position only after use.
- h) Do not pipette corrosive chemicals (concentrated acids or bases) with micropipettes.

## Cell Handling

People working with microbial cells must follow strict security rules to minimize contamination risks.

- (1) Consumable waste (eppendorfs, pipette tips, gloves, paper wipes...) and glassware must be discharged in proper containers for sterilization purposes;
- (2) Liquid waste must be poured in a proper container with bleach;
- (3) Whole working area must be carefully cleaned up with ethanol at 70 % before and after using microbial cells;
- (4) Avoid spreading contamination outside the strict working area (e.g. answering the phone with gloves on.)
- (5) People working with microbial cells must restrict their use of equipments (eg. pipettes) and glassware to a set of designated material for this purpose.

## Check-List / To-Do List before leaving the Biolab

- ☐ The fridge and the freezer doors are properly closed.
- ☐ All temperature-sensitive reagents are back in the fridge/freezer and not left outside.
- ☐ The fluorescence and bright field lamp is switched off.
- ☐ The micropipettes are in maximum position.
- ☐ The used area is left clean.

## Microfluidics Processes (PDMS Room)

### General rules

- a) Most processes performed in this room require precise handling of materials and relative cleanliness of the surroundings, thus, keep all tables clean and clear of all types of materials such as gloves, petri plates and microfluidic structures.
- b) Everything must be properly identified if left unattended for more than a couple of days, otherwise it will be disposed of.

- c) Clean your table before and after you use it, using a normal paper wipe embedded with IPA. This will protect everybody's work and avoid spreading PDMS that may stick to the bottom of your materials.
- d) Since volatile chemicals are used inside this room on a regular basis, the door should be left open, if possible, at all times. In addition, the air conditioner should be turned ON to ventilate the room.
- e) All use of volatile chemicals, such as PGMEA, is restricted to the inside of the laminar flow hood.
- f) Gloves dirty with PDMS base (siloxane oligomers) or SU-8, which are highly viscous and greasy, should not contact other materials in the room without being cleansed with isopropanol or acetone, respectively.
- g) Do not take any of the materials (such as scissors, cutting blade, tape...) from the room without leaving a clear note. Also, if there is only one unit of these items available, do not take it at all since they are essential for most of the processes.
- h) Keep track of the solvents in the general storage inside the PDMS room and notify the responsible when something is about to run out (i.e. acetone, isopropanol, PGMEA...)

## Laminar flow hood [responsible: Virginia Soares]

- (1) Always keep the hood clean: use IPA to clean PDMS and acetone to clean photoresist residues;
- (2) PDMS must be cleaned immediately, before it has time to cure;
- (3) If something drops in the holes on the grid, try to recover it immediately (including pieces of solid PDMS). You should lift the grids and clean underneath (using IPA or acetone) if you let any liquids drip down the grids;
- (4) Work only in the area with laminar flow – i.e. the area of the grid with the circular holes – and keep the air intake zone – i.e. the area with the slits – uncluttered;
- (5) The inside of the spinner must be coated with aluminum foil to prevent the accumulation of PDMS and photoresist;
- (6) Always clean the spinner after use. You can clean the inside of the spinner with IPA and acetone. Dried residues will require hours of hard work to remove if they are left to accumulate.
- (7) The vacuum port in the nozzle of the spinner should be protected from residues at all times, since dried residues can damage the vacuum generation permanently.
- (8) ALWAYS register the use of the laminar flow hood in the specified sheet.
- (9) The HEPA filters of the laminar flow hood have a limited lifetime. If nobody is going to use the laminar flow hood after you, turn it off and put on the front “door” to avoid contamination of the hood. **Don't let the laminar flow hood working overnight.**

## SU-8 Mold processing

- (1) For SU-8 mold fabrication, put all the materials you need inside the laminar flow hood before starting in order to minimize contaminations of the substrate during the initial steps of processing (pre-exposure).
- (2) Clean the substrate properly (generally acetone + IPA + alconox + rinse with water) for SU8 2015 and the same plus a UVO cleaning for 10 min for SU8 50 in order to increase the fabrication yield. **DO NOT USE MICROSTRIP** to clean the substrate, acetone works just fine to remove photoresist residues and is MUCH less expensive.
- (3) Plan ahead before starting a mold fabrication, make sure that everything is fine with the mask (SU-8 is a negative photoresist!) and make sure the spinning conditions are correct for the desired height. Most SU-8 heights have already been tested and optimized for spin velocity, bake and exposure times by someone at INESC MN, so ask before trying. SU-8 is VERY EXPENSIVE ( $\approx 2000\text{€}/\text{L}$ ) and should not be wasted.
- (4) SU-8 takes a very long time to arrive ( $\approx 2$  months), keep track of the general usage. It is essential to have an extra bottle after the one in use reaches about 1/2 of the total volume. **Tell Virginia Soares when the volume reaches the line written on the bottle.**

## PDMS processing:

PDMS is highly greasy and viscous, so you should take special precautions when handling it. Also, you should use disposable cups to prepare the elastomer base + curing agent mix and ONLY the materials present in the PDMS processing tables.

- (1) Press the tap of the PDMS container to release the oligomer solution (base) into a plastic cup and be patient to collect the majority of the leftover flow after releasing the tap. PDMS is expensive ( $\approx 80\text{€}/\text{L}$ ) and should not be wasted;
- (2) To prepare PDMS use only disposable or identified material for this purpose;
- (3) When weighing PDMS, be very careful not to smudge the balance, and place a piece of wipe to protect the dish of the balance;
- (4) When pipetting the curing agent always use the 3 mL disposable plastic Pasteur pipettes;
- (5) Cover the plastic cup with aluminum foil or parafilm after mixing to avoid getting impurities into the PDMS prior to degassing.
- (6) Always wipe your work area with IPA after preparing PDMS.
- (7) Discard your gloves after you finish working with PDMS since most likely they have residues of uncured PDMS that can easily spread to other materials on the labs and compromise someone else's work.

## Corona discharge generator

- (1) Due to ozone generation during discharge, the corona generator must only be used inside the laminar flow hood
- (2) Before switching on the corona, always check if all the neighboring electronic devices are turned off and the lights are turned off. The corona discharge can cause interference to other electronics and may damage its display;
- (3) Before using, remove any flammable compounds that may be in the surrounding area;
- (4) Do not use it for periods longer than 20 minutes;
- (5) After using it, remove and store the electrodes properly;
- (6) Report any problems with the discharge generation or broken electrodes.

## Plasma cleaner (located in the grey area)

- (1) The regulator nozzle (at the left of the oxygen bottle) **MUST NOT** be operated and is already at an optimum position (0,9 bar). **DO NOT TOUCH THE REGULATOR!** If the pressure at the exit of the regulator is not at 0.9 bar, please contact one of the persons responsible for the equipment.
- (2) Handle the glass tray inside the chamber with care to avoid damaging it and/or the glass seal around the entrance of the chamber. If the glass from the chamber is chipped at this position, there will be a leak due to an improper seal against the O-ring on the door and appropriate vacuum may not be achieved, requiring the replacement of the expensive glass chamber.
- (3) When turning the equipment ON in the main power switch, make sure the intensity regulator is in the OFF position and not in LOW, MED or HIGH.
- (4) When the intensity regulator is shifted from the OFF to the LOW position, look inside the chamber to make sure the plasma is being generated. The plasma appears visually as a very faint blueish glow and may not be formed instantaneously ( $\approx 5$  second delay).
- (5) The power switch in the extension cord should be switched OFF after using the equipment.
- (6) Always turn the main nozzle of the oxygen bottle OFF after using the equipment.

## **UVO cleaner (located in the grey area)**

- (1) Always set program 1 for minimum 5 minutes, because program 1 is for gas exhaust. If you do not do this, ozone will come to the grey area when you open the door!!!;
- (2) Ozone is VERY dangerous and may cause serious respiratory and eye irritations!!! NEVER open the drawer while the UV lamps are turned on or before the 5 min exhaustion time has ended.
- (3) After using the equipment always turn it off and fill the log book.