

CORAL Name: OxidationTube

Type: Lindberg 6" Tube Furnace

Location: EML

July 2016

What it does: TRL CMOS oxidation if approved by PTC **OR** EML staff approved processing, as well as a low grade sintering tube, depending on which tube is installed.

Caution: This Technology is both Extremely Contamination Sensitive, due to the high temperatures and it has an awkward combination of Simple Concepts and a flexible, multipurpose frame, with cosmetically similar but dissimilar dedicated parts which are NOT interchangeable, which MUST be setup correctly. Plan on at least two training sessions!

(1) Introduction

The Lindberg 6" Tube Furnace is a 4" maximum substrate size, 1050 C maximum temperature furnace which is plumbed for N₂, O₂, and steam oxidation, located within the EML.

It has (3) selectable tubes, each with dedicated labware, allowing:

TRL "Green" processing, if a process flow is approved by ptc@mtl.mit.edu, with the TRL Oxidation Tube,

EML "Electronic Grade" processing, if "EML staff approved", with the EML Oxidation Tube, or

Organics, low melting point materials, or contaminated items if using the "Dirty" tube.

Each Tube is labeled. If items intended for the "Dirty" tube are used in one of the "Oxidation Tubes", YOU will be billed for new quartz tube and cassette, about \$1000. Please communicate with EML staff to ensure success, and schedule in advance to have the correct tube installed. Plan on having staff exchange tubes for you the first two times at least.

(2) Safety

(A) Emergency Shut-off Procedure

In case of an emergency, such as overheating or breaking, do the following:

Shut off the EMO circuit breaker on the temperature control unit.

Shut off Oxygen valve, open Nitrogen.

Find MTL Staff.

(B) Warnings about this system

This is a manually controlled machine, so you must be careful not to choose a destructive sequence and be aware of the machine status at each step. This is a high temperature research tool and YOU must check for fire or burn hazards.

- Everything which touches the quartz cassette or your wafers must be clean. Tweezers must be IPA cleaned on the tongs which touch the wafer. New vinyl gloves must be put over your existing vinyl gloves before touching the quartz.
- Clean the quartz tray and aluminum table with a fabwipe wetted with IPA prior to starting.
- Never use a temperature over 1050C, as the spike thermocouples tend to fail.
- Never open the chamber when the tube temperature is over 600C, as the quartz tube could be damaged.
- Do not run at high temperature, >600C overnight.
- Maximum combined gas flow is 4 SLM, which is 20% of the N₂ (MFC 2) “20 SLM” or 40% of the O₂ (MFC 1) “10 SLM”, so flows must be lowered if combining gases.
- Look for flammables and remove them from a 1 meter radius around the tool, especially resting on top of the furnace. This includes fabwipes or paper products, plastics, and especially solvent bottles.
- The end cap, cassettes, and wafers, will be hot after a run. Don’t ruin them by melting your gloves, plastic wafer boxes, or your hand on them, but instead use the thermal glove to remove a hot end cap, and put it on the aluminum tabletop, not on fabwipes, and let quartz cassette cool in the loading tray for 5 min before proceeding.

Following are instructions for correct operation of the Lindberg 6” Tube Furnace.

(3) Preparation

Make machine reservations in CORAL. Allow enough time to let your tube cool enough for unloading as part of your reservation time.

If requiring a tube change, communicate with lab staff in advance. We will require the tube to be cool before changing.

If doing a TRL process, please review PTC approved process with lab staff during training, including the details of where and when the Piranha/HF dip is scheduled.

If doing an EML process, make sure it is staff approved prior to training.

Identify the correct quartz cassette and confirm the correct quartz tube is installed, or allow ½ hour additional time to change tubes, in your scheduling.

If using an Oxidation Tube, pre-clean your substrate immediately before the furnace run, as contamination occurs during storage.

Clean the tweezers you will use, the quartz loading tray and the table with alcohol and a fabwipe, prior to starting



Oxidation Tube and “Dirty” Tube

*** If doing wet oxidation, see appendix for set up procedure. You must return to normal dry oxidation when done***

(4) Verify the Proper Starting and Ending Parameters

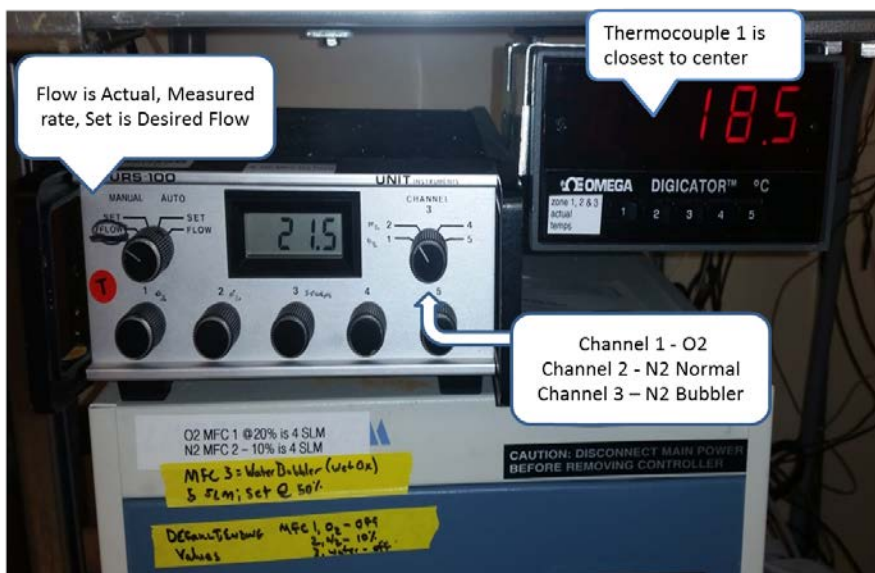
Idle temperature is either Off or up to 300C.

End cap is on, but Not Tightly, or it will fuse to the tube.

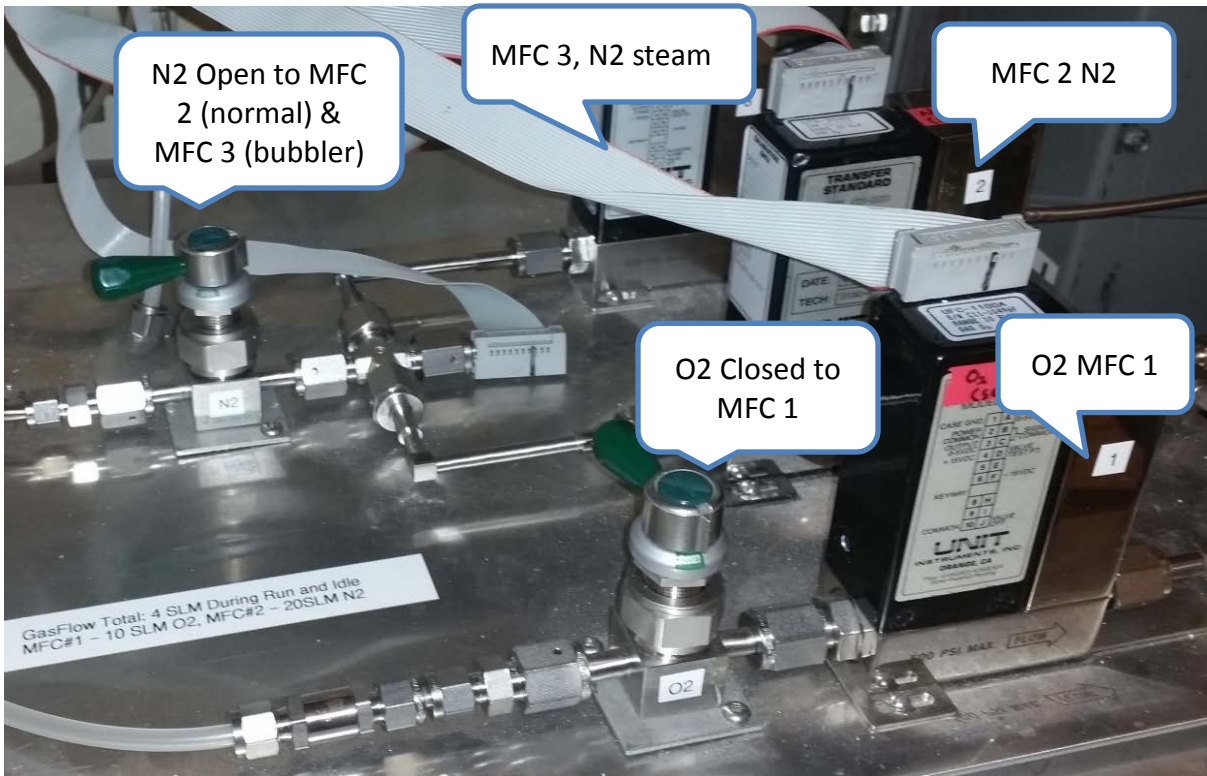
Gas flow is N2 at 20%, of 20 SLM, or 4 SLM, with N2 valve Open

O2 valve is off. MFC 3, N2 bubbler, is at 0%

Quartz cassettes are stored in their dedicated holders, not left in the tube or on the loading tray.



MFC Channel #2, N2, @21.5%. Top right is more accurate temperature display, at zone 1



O2 Valve to MFC 1 Closed and N2 Valve to MFC 2 or 3, Open

(5) Loading

Choose the quartz cassette which matches the process and tube you had approved or you will be buying new ones. If you don't know, ask staff!

IMMEDIATELY before removing the quartz boat from the protective storage, put on new gloves over the gloves you are wearing. Once these gloves touch your uniform, glasses, or the keyboard, they are instantly decertified for quartz ware handling.

The quartz boats are stored in their dedicated holders, and are removed only for loading into the tube, and are never left out, nor are they left in the tube when finished, but are put back when finished

Never touch the interior of the tube or end cap, they will be ruined. Put the end cap face down on the cleaned metal surface, never on the black counter or a fabwipe.

Hold the quartz loading tray snug to the tube end, and using the quartz rod on top of the furnace, push the quartz wafer cassette about halfway into the tube.

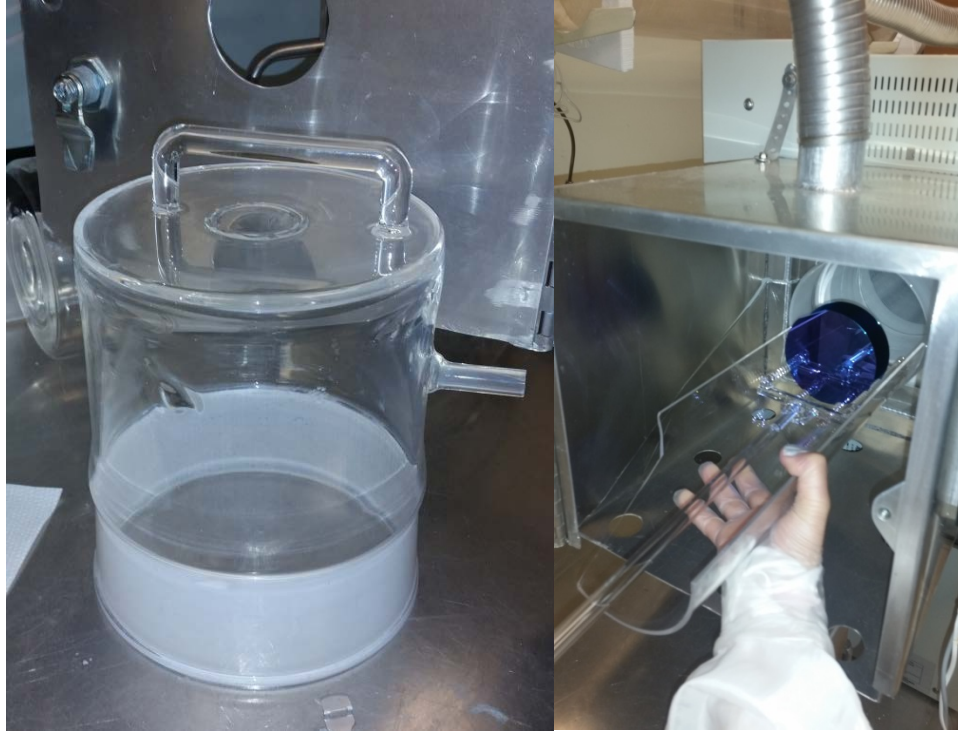
Put the quartz rod back into the quartz sheath, and put the endcap on, loosely, but not tightly, or it will fuse closed.



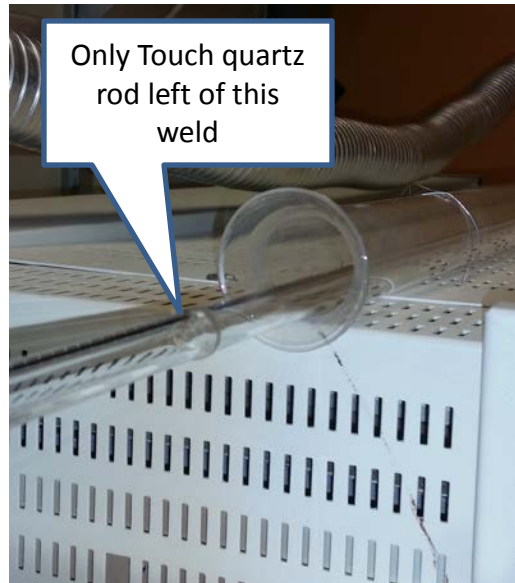
TRL dedicated quartz boat in storage, left, and Dummy wafers for holding pieces, baffling, right



Endcap removed with thermal glove



Endcap on Al countertop, L, and quartz Boat being loaded or unloaded, R



(6) Running

Start ramping up the temperature, in increments no more than 200C, allowing approximate temperature stabilization, or the gas source end of the furnace, Zone 3, will lag too far behind the center Zone 2, and an error under rate (UR) will occur. To cure the UR error, set the temperature down 200C and allow zone 3 to turn back on, when the differential is lower between zones 2 and 3. Often you will see a Low Alarm error, saying zone 3 is low, but it will still be working unless the UR error occurs

When at the correct run temperature, switch to the correct gas and flow rate for the calculated time. At completion, return to N2 at 4 SLM and return the temperature to 300C.

(7) Ending

After the temperature has dropped to the targeted temperature, always less than 600C, remove the end cap, holding only the handle, and place on the freshly re-cleaned metal surface.

Use quartz rod, never touching the rod in front of the weld, to remove the quartz cassette onto the quartz loading tray.

Let the wafers cool before touching them, even with metal tweezers, or you will melt them into your cassette or holder.

Do NOT touch quartz boats unless you have new gloves on top and let the boat cool.

Return furnace to the correct Starting / Ending Parameters, item #4 above

Appendix:

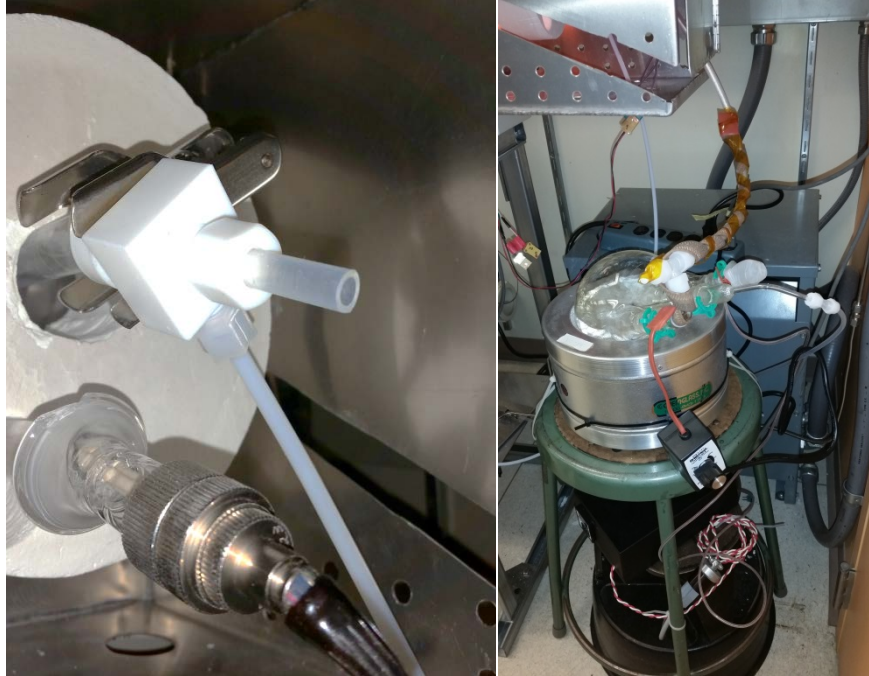
Wet Oxidation Instructions:

Setup

This is the **starting** and **ending** configuration for the gas input into the tube. Notice there is the combined N2 and O2 feed coming into the tube on a 1/4" line from the bottom, and there is a blocked 3/8" line from the end, as a plug.



Plug removed from Ball Valve prior to wet oxidation. Replace before disengaging!



Default, starting and ending 1/4" line feed, and plug, L, and installed Bubbler, R

Replacing Plug with Bubbler 3/8" feed

To allow Wet Oxidation, the 3/8" plug must be removed, and replaced with the Teflon line coming from the bubbler, underneath.

Do Not Lose the plug, as it must be returned before you finish!

Note the Bubbler, set to 85C, and heat tape, on lowest setting 5%, are turned on with the power strip on the gray platform behind it. Note, also, the slope going from the bubbler to the Oxidation Tube doesn't contain any traps which could cause water collection. These take 30 min to heat before starting.

Processing

1. Load your samples, typically at 600C with 4 SLM N2 (20% of 20 SLM) flowing, then ramp up to 950 to 1050C typically, depending on recipe
2. When target temperature reached, turn N2 flow to 0, and turn On O2, for 10 min, at 4 SLM, (40% of 10 SLM) to get a uniform thin initial growth
3. After 10 min, turn Down MFC#1 O2 to 2 SLM and turn On MFC#3, N2 to the Bubbler, which should be 85C, at 2 SLM (40% of 5 SLM). It should be vigorously bubbling but water shouldn't be going up the 3/8" tube. Adjust flow or angle of N2 inlet into bubbler or of 3/8" outlet if necessary

4. Grow for time calculated by oxidation calculator, for instance <http://www.cleanroom.byu.edu/OxideTimeCalc.phtml>



Bubbler with 2.5 SLM N2 Bubbling away, but Water not “climbing” tube, nor condensing

When growth finished:

Turn Off MFC #3 and O2 valve

Turn On N2, at 4 SLM

Set temperature to 0 (turn off Circuit Breaker to allow fast cooldown, otherwise takes forever)

Turn off the power strip, feeding the Bubbler and Heat Tape

When temperature goes below 600C, unload samples and measure thickness with Filmetrics F20 interference tool

When temperature goes below about 200C, put the plug back into the 3/8” feed Angle 3/8” tube, still attached to bubbler, downwards, to protect from debris, with protective cap reattached.



Kurt Broderick 7/2016