

# Tube Furnace Procedure

## 1. Tube Selection

### 1.1. Anneal Tube (Max 4" wafer)

1.1.1. Used for sample annealing

1.1.2. **DO NOT USE** Organic materials

1.1.3. **DO NOT USE** any materials that will out-gas

1.1.4. **DO NOT USE** low melting point metals

### 1.2. Oxidation Tube (Max 4" wafer)

1.2.1. Used EXCLUSIVELY for growing oxide layers on samples

### 1.3. Phosphorous Tube (Max 4" wafer)

1.3.1. Used for N-type doping

### 1.4. Boron Tube (Max 4" wafer)

1.4.1. Used for P-type doping

### 1.5. Dirty Tube (Max 3" wafer)

1.5.1. Used when sample would contaminate other tubes

1.5.1.1. Organic materials

1.5.1.2. Any other materials that will out-gas

1.5.1.3. Low melting point metals

## 2. General information

2.1. Standby temperature is 400°C

2.2. Idle Nitrogen flow is 37 (6L/min) read by the black ball.

2.3. **DO NOT EVER USE PLASTIC TWEEZERS!**

2.4. **Do Not shut off the tube heaters.**

2.4.1. Cycling temperatures from below 275°C to above 1100°C changes the quartz crystalline structure, making it vulnerable to break.

2.5. Max operating temperature of the tubes is 1150°C.

2.6. **ALWAYS USE FURNACE GLOVES.** Quartz can be very hot without looking hot.

## 3. Operation

3.1. Verify the specific tube being used is at the proper temperature.

3.1.1. See step 3.9. Temperature Control

3.2. Verify the nitrogen is flowing into the tube.

3.2.1. See Step 3.10. Gas Control

3.3. Verify the flow for the other desired gases.

3.3.1. See step 3.10. Gas Control

3.4. Put on Furnace Gloves.

3.5. Open the door of the tube using the furnace gloves.

3.6. Take the cap off the end of the tube.

3.6.1. The cap sometimes sticks to the tube.

3.6.1.1. A gentle jiggle of the cap is enough to loosen its grip.

3.7. Insert the quartz rod into the tube.

3.7.1. Each tube has its own rod.

3.7.1.1. Do not switch these rods.

3.7.2. Hook onto the boat.

3.7.3. Slowly and gently retract the boat to the opening of the tube.

# Tube Furnace Procedure

- 3.7.4. Allow the rod to cool for a few seconds.
- 3.7.5. Place the rod back into its holder, being careful not to jam the rod.

**3.8. Dirty Tube**

- 3.8.1. Install the elephant (loading shelf) onto the front of the dirty tube.
- 3.8.2. Use the dirty rod to pull the boat into the elephant.
- 3.8.3. Replace the rod into its storage cylinder.
- 3.8.4. Move the elephant so it is lengthwise on the furnace table.
- 3.8.5. Use the tongs to pull the boat out of the elephant.
  - 3.8.5.1. Do not pull the boat completely out of the elephant.
  - 3.8.5.2. Pull out only enough to allow loading and unloading of the samples.
  - 3.8.5.3. NEVER USE THE PLASTIC TWEEZERS.**
- 3.8.6. Proceed with sample loading/unloading.
- 3.8.7. Follow steps 3.8.1 through 3.8.5 in reverse order to place boat back into the tube.
  - 3.8.7.1. If loading samples, use the rod to push the boat to the middle of the tube.

**3.9. Specific Tubes**

- 3.9.1. Insert the fork into the holes on the boat.
- 3.9.2. Remove the boat from the tube.
- 3.9.3. Place the boat on the table.
- 3.9.4. Remove the fork from the boat.
- 3.9.5. Load/Unload samples.
  - 3.9.5.1. NEVER USE THE PLASTIC TWEEZERS.**
  - 3.9.5.2. In the Oxidation and Anneal tube use the quartz plates for small samples.
- 3.9.6. Use the fork to reload the boat into the tube.
- 3.9.7. Use the rod to push the boat to the middle of the tube.
- 3.9.8. Return the rod to its holder.
- 3.9.9. Place the cap back on the end of the tube.

**3.10. Temperature Control**

- 3.10.1. Adjust the appropriate middle controller.
  - 3.10.1.1. Use the up and down arrows to set the desired temperature.
  - 3.10.1.2. The left and right controllers on each tube are incorporated into the middle controller and therefore do not need to be set.
- 3.10.2. A thermocouple is located on the front of the tube furnace.
  - 3.10.2.1. This may be inserted into the tubes to achieve a more accurate temperature reading.
  - 3.10.2.2. Use the TC meter in the tool box with the thermocouple.

**3.11. Gas Control**

- 3.11.1. Use the rotameters on the back side of the tube furnace to adjust the desired gas flows.
  - 3.11.1.1. Gas needs to be flowing through the tubes at all times.
  - 3.11.1.2. The quarter-turn valves are used for shutting off gases.
    - 3.11.1.2.1. If a gas is wanted other than Nitrogen, turn on that gas first before turning off the Nitrogen and visa versa.
- 3.11.1.3. Do not use the rotameter flow adjustment to turn off any gases.

# Tube Furnace Procedure

## 3.12. Completion

- 3.12.1. When process is completed, using the same procedure as before, remove the samples.
- 3.12.2. If Boron or Phosphorous tube was used, push the boat to the center of the tube after the samples are removed
- 3.12.3. Change gases back to initial state
- 3.12.4. Set temperature to 400°C
- 3.12.5. Complete logbook entry.

## Flowmeter Calibration Tables

Argon and oxygen supplied by manufacturer specs. Nitrogen flows calculated based on air/nitrogen ratios and air data supplied by manufacturer.

Scale Readings	Nitrogen Flow [mL/min] Glass / Stainless	Argon Flow [mL/min]	Oxygen Flow [mL/min]
150	24279 / 46250	14131	6992
140	22627 / 42917	13307	6561
130	20846 / 39182	12375	6131
120	19073 / 35968	11480	5624
110	17369 / 32371	10581	5168
100	15417 / 28884	9634	4712
90	13848 / 25730	8643	4231
80	12232 / 22701	7764	3724
70	10484 / 19488	6805	3268
60	8832 / 16369	5896	2787
50	6938 / 13282	4886	2280
40	5217 / 10267	3855	1773
30	3785 / 7420	2940	1267
20	2299 / 4774	1926	735
10	809 / 2231	826	228

## Useful website

Dry oxidation calculator:

<http://www.lelandstanfordjunior.com/dealgrove.html>

Doping wafer

<http://www.bn.saint-gobain.com>