GENERAL PROCESS AND OPERATION SPECIFICATION

Mini Brute Oxidation and Annealing Furnaces

I. SCOPE

a. The purpose of this document is to describe requirements and basic operating instructions for the Mini Brute Oxidation and Anneal Furnaces. This tool is intended for oxidation and annealing of metallic films on samples. The equipment is supplied with ultra-high purity N2 and O2 gases and water vapor. This furnace is capable of continuous annealing at temperatures up to 1200°C.

II. SAFETY

a. Be sure that you are trained and signed off to use this equipment.
b. Be sure to keep all doors and protective shields in place before operating this equipment.
c. Use care when operating around high voltage or hot temperatures.
d. This equipment can reach 1200°C in the quartz tube heating chamber. Allow adequate time for samples to cool down before handling them. Allow system temperature to drop below an idle temperature (typically 600°C) and use respective quartz rod when moving sample boats.
e. Maximum operating temperature: DO NOT operate the furnace above 1200°C. DO NOT change the excess temperature setting to above 1240°C
f. Maximum time at peak temperature: DO NOT operate the furnace unattended after hours (5PM – 8 AM).
g. Contamination: Avoid touching any of the quartz surfaces with dirty gloves to prevent sodium and ionic contamination in the system. At high temperatures these contaminates can diffuse very quickly throughout the tube and process chamber, and in turn, contaminate samples of other users.
h. Contamination: Top chamber (tube 1) is intended for metal annealing. Middle and bottom chambers (tube 2 & 3) are intended for Si, SiN and SiO samples only. No other materials are allowed. No exceptions.
i. When not using the quartz push rod, place it in the rod holder.
j. When holding a rod (whether it's cold or hot), hold it vertically with the hot end pointing down.
k. Check the pressure level of the gas cylinders before starting.
l. Make sure to refill the bubbler with DI water before running the furnaces. DO NOT overfill it (3/4 full max) and DO NOT run empty.
m. Position the data cable away from the furnace tube opening.
n. If you are unsure about any procedure or indication while operating this equipment, contact a staff member or trainer for assistance.

III. APPLICABLE DOCUMENTS, MATERIALS AND REQUIREMENTS

a. For more information about the detailed operation of this tool refer to the electronic manual (Provided upon request).
b. Appendix A: Automatic Operation
c. Appendix B: Procedure Outlines
d. Appendix C: Gas Supply System

IV. OPERATION

a. Important Notes:
   i. Everything from Tube 1 should be quarantined from Tubes 2 & 3. This includes glass push rods, tube lids, elephants, boats, uncleaned samples, and more.
   ii. Be mindful of the temperature of every surface that interacts with the tube. It is easy to forget that surfaces can be hot. Use the heat gloves provided whenever possible.
   iii. Everything that interacts with the chamber must be kept clean to avoid contaminating it. This includes boats, samples, and your hands. Please clean your samples beforehand and wear double gloves when loading/unloading samples.
   iv. Remember the purpose of each tube:
      1. Tube 1: Annealing
      2. Tube 2: Dry nitride/oxide growth or wet oxide growth
      3. Tube 3: Dry nitride/oxide growth or wet oxide growth

b. Bubbler Setup (Wet Oxide Growth Only):
   i. Make sure the bubbler output valves are both closed (Figure 1).
   ii. Fill the bubbler ¼ of the way full for every 2 hours of growth needed (Figure 2).
      1. Minimum fill height: ½ full.
      2. Maximum fill height: ¾ full.
      3. Each bottle fills the bubbler ¼ of the way and last for 2 hours of growth.
   iii. Turn on the bubbler heater and set the power to 40% (Figure 2).
      1. It takes about 90 minutes for the water to boil.
      2. Water must be boiling prior to starting wet oxidation.
      3. Be careful when refilling the bubbler since filling it too quickly can cause the boiling to stop.

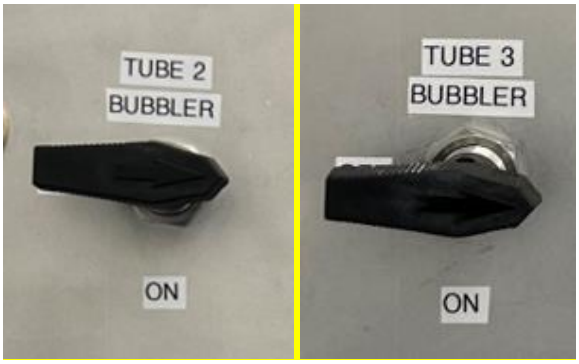

Figure 1: Bubbler Output Valves to Tube 2 and Tube 3

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Figure 2: Filling the Bubbler (Left) & Bubbler Power Control (Right)

c. N2 Gas Purge:

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i. Open the N2 gas cylinder (Figure 3).
   1. Use the metal valve on the top of the bottle.
   2. Make sure the valve on the regulator is also open and the output pressure is set to around 20 psi.

ii. Open the N2 supply valve for the desired tube (Figure 3).

iii. Using the turn knob on the flow controller, adjust the flow rate to 1 SLM of N_2.

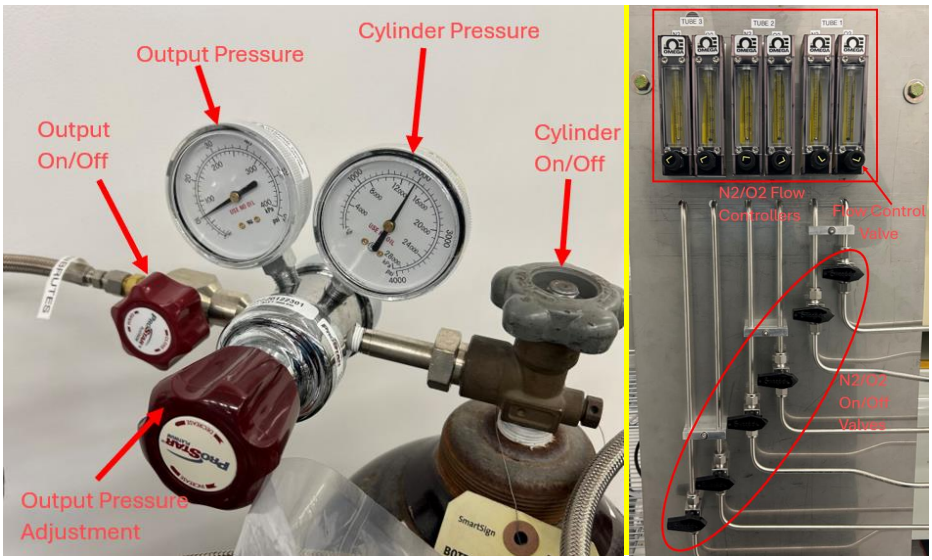

Figure 3: Gas Cylinder and Regulator Control (left) & N2/O2 Gas Supply Control (right)

d. Furnace Setup:
    i. Turn on the switch on the back of the furnace.
        1. This turns on the cooling fan inside. DO NOT operate the instrument without turning on this switch (Figure 4).
    ii. Turn on the "CONTROL" and "ELEMENT" switches.
    iii. Using the arrow keys and the "SET" button, set all three SOLO controllers to the idle temperature.
    iv. The idle temperature is dependent on the process you are running:
        1. **Wet Oxide Growth:** The recommended idle temperature for oxidation is 600°C. This will take ~25 minutes to reach.
        2. **Annealing:** Idle temperature is usually room temperature (20° C)
        3. **Dry Oxide/Nitride Growth:** Varies based on desired growth.

Figure 4: Fan switch (left), Power Switches & SOLO Controllers (right)

e. Load Samples onto Wafer Boat:
    i. Put on another layer of nitrile gloves.
        1. More gloves can be found in Bay 4, on the table next to the Minibrute, or in the gowning room.
        2. This is a precaution to keep the chamber clean and free of ionic contaminants.
        3. Keep in mind the more surfaces you touch, the more you risk contaminating the chamber.
    ii. Grab a sample boat from the glass cabinet
        1. **Tube 1:** Only use boats from the top shelf labeled 'Tube 1 only' or bring your own.
        2. **Tubes 2 & 3:** Only use boats from the middle and bottom shelves labeled 'Tubes 2 & 3'.
    iii. Load cleaned samples onto the sample boat.
        1. Do not put Kapton tape or sharpie marked samples in this tool.
f. Load Boat into Furnace
    i. Annealing, Tube 1:
        1. Wait for the chamber to reach idle temperature.

2. Remove the furnace lid and place it on the wire rack.
3. Place the boat into the tube.
    a. Do not use the elephants to load Tube 1.
4. Push the boat into the middle of the chamber using the glass push rod.
5. Replace the furnace lid.

ii. Oxide/Nitride Growth, Tube 2 & 3:

1. Wait for the chamber to reach idle temperature.
2. Use the furnace gloves to move the lid to an empty area of the wire rack.
    a. DO NOT PLACE THE LID OR OTHER HOT OBJECTS ON ALUMINUM FOIL. Aluminum melts at around 600 °C and can contaminate the chamber lid, boats, and samples. Only set cool objects on aluminum foil.
3. Place the sample boat in the elephant and align the elephant to the furnace opening.
4. Using the push rod, slowly push the sample boat until it is out of the elephant.
5. Move the elephant out of the way.
6. Slowly push the boat to the middle of the furnace (1 inch every 5 seconds)
7. Place the push rod back in its holder with the hook facing up.
8. Replace the furnace lid.

g. Ramp up to Process Temperature (if needed):
    i. **Manual operation:** Use each SOLO to slowly ramp up the temperature.
        1. Use the up and down buttons on all solos to change to the desired temperature and press “Set.”
            a. Make sure all SOLOs are set to the same value.
    ii. **Automatic Operation:** Refer to “Appendix A” to start a recipe.

h. Turn On Process Gas and Run Process:
    i. Once the process temperature is reached turn on process gas (refer to Appendic C for gas supply details):
        1. Nitride/Oxide Growth:
            a. Open the valve for the desired gas and tube (Figure 3).
            b. Adjust the flow rate using the knob at the bottom of the flow meter.
        2. Wet Oxide Growth:
            a. Open the bubbler output valve for the tube in use (Figure 1).
            b. Turn off N2 flow to the tube.
            c. Turn on the O2 gas cylinder that is next to the Minibrute table.
                i. Open the valve at the top of the cylinder.
                ii. Make sure the regulator is set to around 20 psi.
            d. Open the bubbler O2 supply valve.
            e. Set the O2 flow rate to 0.5 SLM using the needle valve under the flow meter.
                i. The bubbler must be boiling prior to starting the wet oxidation. (a few bubbles per second)
                ii. Be careful when refilling the bubbler since overfilling can cause the boiling to stop.

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ii. **Manual Operation:** Start a timer that is the amount of time your process needs to run.
iii. **Automatic Operation:** The recipe will automatically start.
1. Tips: “Hold” pauses the recipe, “Stop” ends the recipe.

i. Ramp Down to Idle Temperature: (if needed)
i. **Manual operation:** Use each SOLO to ramp down the temperature.
1. Use the up and down buttons on all solos to change to the desired temperature and press “Set”.
a. Make sure all SOLOs are set to the same value.
ii. **Automatic Operation:** Let the recipe continue running.
iii. Wet Oxide Growth:
1. Turn off the O2 supply to the bubbler.
2. Close the bubbler valve for the tube in use.
3. Turn on the N2 to purge the excess moisture out of the chamber.

j. Unload Samples:
i. **Annealing, Tube 1:**
1. Wait until the boat and tube have fully cooled to room temperature.
2. Put on a new pair of clean nitrile gloves to prevent contamination.
3. Remove the furnace lid and place it out of the way on the wire rack.
4. Using the push rod, slowly pull the sample boat (1 inch per 5 seconds) until the boat is sitting securely in the furnace opening.
5. Carefully remove the boat from the tube.
6. Remove samples from the boat and place it back in the glass cabinet.
ii. **Oxide/Nitride Growth, Tube 2 & 3:**
1. Wait until the idle temperature is reached.
2. Put on a new pair of clean nitrile gloves to prevent contamination.
3. Put on the furnace gloves.
4. Remove the furnace lid and place it on the wire rack.
a. Remember to avoid placing hot objects on aluminum foil.
5. Using the push rod, slowly pull the sample boat (1 inch per 5 seconds) until the boat is sitting securely in the furnace opening.
6. Place the push rod back in its holder. (With the hook facing up)
7. Align the elephant to the furnace opening.
8. Use the push rod to pull the sample boat into the elephant.
9. Place the elephant away from the furnace and let cool for 10-15 minutes.
10. When the boat is cool, remove it from the elephant.
11. Remove the samples from the boat
12. Put the boat back into its respective cabinet.

k. Close Gases and Shutdown the Tool:
i. Set each solo to 20 °C.
ii. Flip the “Control” and “Element” switches to the off position
iii. Close all supply valves.
iv. Turn all gas cylinders off (knob on top of cylinder)

V. SIGNATURES AND REVISION HISTORY

a. Author of this document: Elijah Colter
b. Author Title or Role: Student Technician
c. Date: January 10, 2022
d. Revision A: Updated automatic recipe instructions. General clarity revisions.
e. Revision B: Updated procedure to reflect new gas supply system. Format revisions. Added Appendix C.

Approvals:

Technical Manager Signature: Sandra G Malhotra _____

Date: __8/6/2024_____

Revision History:

Revision	**Author**	**Date**
Original Issue	J. Woo	February 12, 2021
Rev A	Elijah Colter	January 10, 2022
Rev B	John Slentz	August 2, 2024
Rev C		
Rev D		
Rev E		

Appendix A: Automatic Operation

1. Set up SOLOS:
    a. Connect the cable's serial connection to the furnace being used.
    b. Connect the USB cable to the computer.

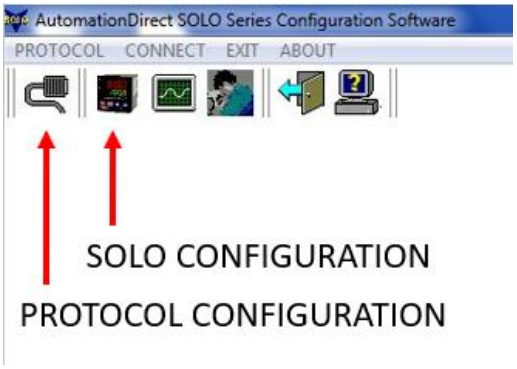

Figure 5: SOLO main screen.

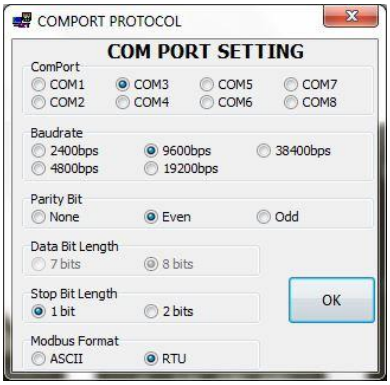

Figure 6: COM port configuration

    c. Optional: Click on "PROTOCOL CONFIGURATION" (Figure 5), double-check the values match Figure 6, and click "OK".
    d. Click on 'SOLO CONFIGURATION' (Figure 5) to go to the main working page.
2. Change each SOLO to automatic operation settings:
    a. Starting from left to right, set the "Address" to 1, 2 and 3.
    b. Click "Connect" on the first SOLO.
        i. Only connect to one SOLO at a time. Connecting to multiple SOLOs at once will slow down the interface considerably.
    c. Set "Control Mode" to "Ramp / Soak"
    d. Set "RUN/STOP" to "Stop Program"
    e. Click "Disconnect" and repeat for the other SOLOs.
3. Edit and save the recipe:
    a. Connect to a SOLO.
    b. Click on "Edit ramp/soak pattern" to edit the recipe.
        i. For "Step 1 SV" and "Step 1 Time" enter the idle temperature and 1 minute.
        ii. For "Step 2 SV" and "Step 2 Time" enter the process temperature and the ramp up time.
            1. The rest of the temperature/time steps should be straightforward.
        iii. Set "Last step number" to the number of the last step.
        iv. Make sure "Next pattern number" is set to "Prog End" unless the process needs more than 7 temperature/time points.
    c. Click "Write to File" to save the recipe.
        i. Note: The software may save the data on the desktop instead of the folder chosen.
    d. Disconnect from the SOLO.

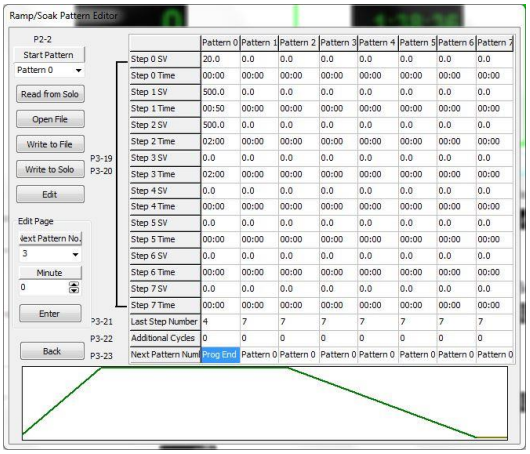

Figure 7: Recipe editor

4. Another way to edit recipes (if the above way doesn't work):
    a. Open a previously save recipe via Notepad.
    b. The recipe file is double space ('__') delimited number arrays.
        i. Start Pattern: User may choose the pattern to begin the ramping. Choose from 0-7 (Figure 8, A)
        ii. Last Step Number: ignores any input after this step. (Figure 8, B)
        iii. Additional Cycles: repeats this pattern by the input number. (Figure 8, C)
        iv. Next Pattern: If you run out of space in Pattern 0, you may continue in Pattern 1 and indicate 1 here. 8 represents the end of recipe, "Prog End." (Figure 8, D)
        v. Temperatures: The columns in the editor correspond to rows in the notepad. (Figure 8, E)
        vi. Times: The columns in the editor correspond to rows in the notepad. (Figure 8, F)

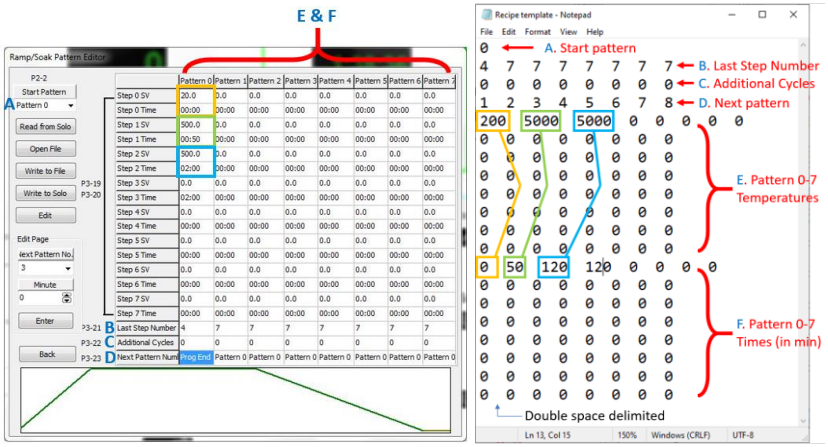

Figure 8: Ramp/Soak Pattern Editor

5. Load the recipe:
    a. Connect to a SOLO.
    b. Click “Edit ramp/soak pattern”.
    c. Click “Open file”.
    d. Click “Send to SOLO” to write the recipe to it.
    e. Make sure the recipe isn’t running.
        i. RUN/STOP under Operation mode should be set to “Stop Program”.
    f. Disconnect from the SOLO.
    g. Repeat for the other two SOLOs.
    h. Set SOLOs 1, 2, and 3 to display Pattern-Step, set temperature, and time respectively.
        i. This will act as a “heads-up display” while the recipe runs. (Figure 9)

Figure 9: Control panel showing step number (left), set temperature (middle), and time remaining (right).

6. Run the recipe:
    a. On each SOLO set “RUN/STOP” to “Run”.
        i. (Oxide/Nitride Growth) Remember to open the process gas and set the flow once the process temperature is reached and turn it off when the process is done.
    b. When the recipe is completed set “Run/Stop” to “Stop Program”.
7. Return all SOLOs to default settings:
    a. Set “Control Mode” to “PID”, “SV” to “20” °C, and “RUN/STOP” to “Run” for all controllers.
        i. This is so the idle temperature can be easily set and for users who don’t know how to operate the SOLOs manually.

Appendix B: Procedure Outlines

AFNF Standard Wet Thermal Oxide Growth Recipe

1. Turn on bubbler (Do this ~1.5 hours before loading).
2. Clean substrate beforehand using piranha for 15 min or acetone/IPA/DI water.
3. Turn on furnace (600 °C) & N2 (1 SLM) (Do this ~0.5 hours before loading).
4. Load boat into furnace.
5. Ramp up @10 °C/min to 1100 °C (50 min).
6. Open bubbler valve to the furnace in use & the O2 supply to the bubbler. Adjust flow to 0.50 SLM (a few bubbles per second).
7. Soak @1100C (50 min = 580 nm or 60 min = 640 nm).
8. Ramp down @10 °C/min to 600 °C (50 min).
9. Close bubbler and close O2 valve. Open N2 to furnace (1 SLM).
10. Unload & keep boat in elephant for 10-15 min. Close N2.
11. Transfer boat to foil to further cool down.
12. Store wafers and clean up the workspace.

400° C Annealing Sample Recipe

1. Clean substrate beforehand.
2. Turn on furnace (20 °C) & N2 (1 SLM).
3. Load boat into furnace.
4. Ramp up @10 °C/min to 400 °C.
5. Ramp down @10 °C/min to 20 °C. Open N2 to furnace (1 SLM).
6. Unload & keep sample in the boat for 10-15 min or until cool. Turn off N2.
    a. Transfer boat to foil to further cool down if needed
7. Store wafers and clean up the workspace.

Appendix C: Gas Supply System

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1. Gas Cylinder Controls:
    a. All gases are supplied by gas cylinders located near the Minibrute in Bay 2.
    b. The available gases are N2, O2, and a 5% H2/95% Ar mix.
    c. These gas cylinders are connected to the gas control panel behind the Minibrute.
    d. The locations of the cylinders and the panel are shown below (Figure 10).

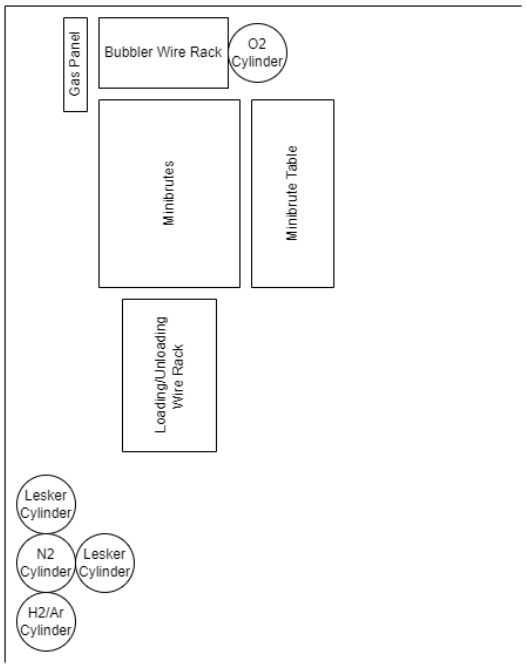

Figure 10: Overhead Layout of the Minibrute System

    e. Each of these cylinders has:
        i. An on/off valve located at the top of the cylinder.
        ii. A regulator that can be used to control the output pressure.
        iii. Gauges that show the output pressure and supply pressure.
    f. The N2 and H2/Ar cylinders also have an output on/off valve.

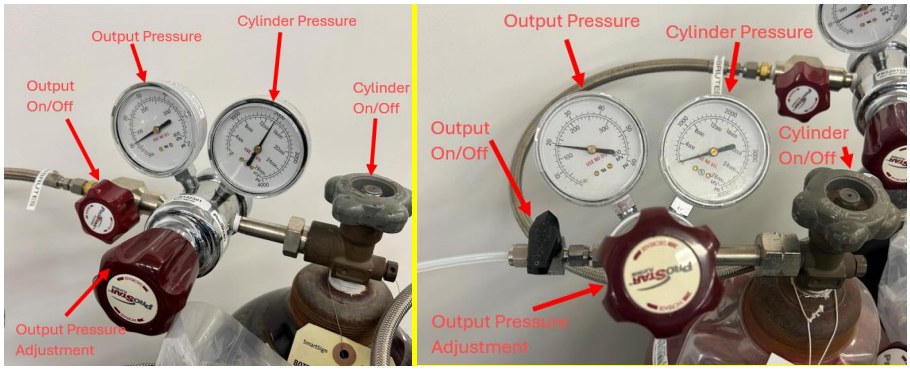

Figure 11: Gas Cylinder Controls for N2 (left) and H2/Ar (right)

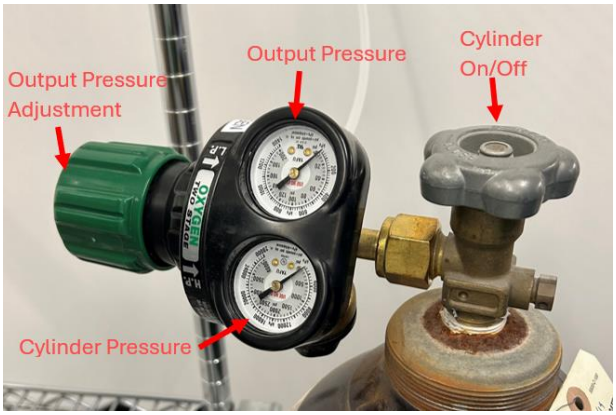

Figure 12: Gas Cylinder Controls for O2

g. To use one of these cylinders:

i. Open the on/off valve at the top of the cylinder.
ii. Open the output valve if the cylinder has one.
iii. Adjust the output pressure if needed.

2. N2/O2 Process Gas Controls:
   a. All furnaces are supplied N2 and O2.
   b. These gases can be turned on via the on/off valves on the gas control panel.
   c. The flow of N2/O2 can be controlled by the flow control valve at the bottom of the flow meters.
   d. There are valves and flow controllers for each gas and each tube (Figure 13).

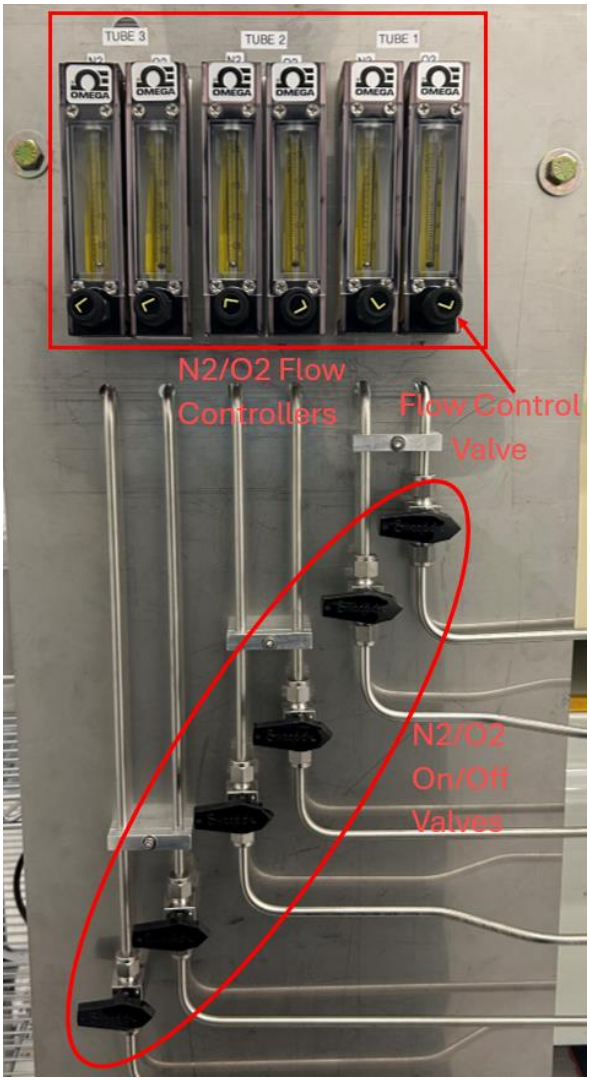

Figure 13: N2/O2 Supply On/Off Valves (bottom) and Flow Controllers (top)

e. To use the N2/O2 process gasses:

i. Open the cylinder. Ensure the cylinder output valve is also open for N2 (the O2 cylinder does not have an output valve).
ii. Turn on the supply valve for the desired tube and process gas.
iii. Adjust the accompanying flow controller for that tube/gas to the desired flow rate.

3. Bubbler Controls:
    a. The bubbler can be used for wet oxide growth in tube 2 and/or tube 3.
    b. The bubbler is supplied with O2 from the same cylinder as the O2 process gas.
    c. On the gas panel there is:
        i. An on/off valve for the bubbler O2 supply.
        ii. A needle valve to control the flow of O2.
        iii. A flow meter to show the flow of O2 to the bubbler.
        iv. Two on/off valves that control the output of the bubbler. One for tube 2 and one for tube 3.

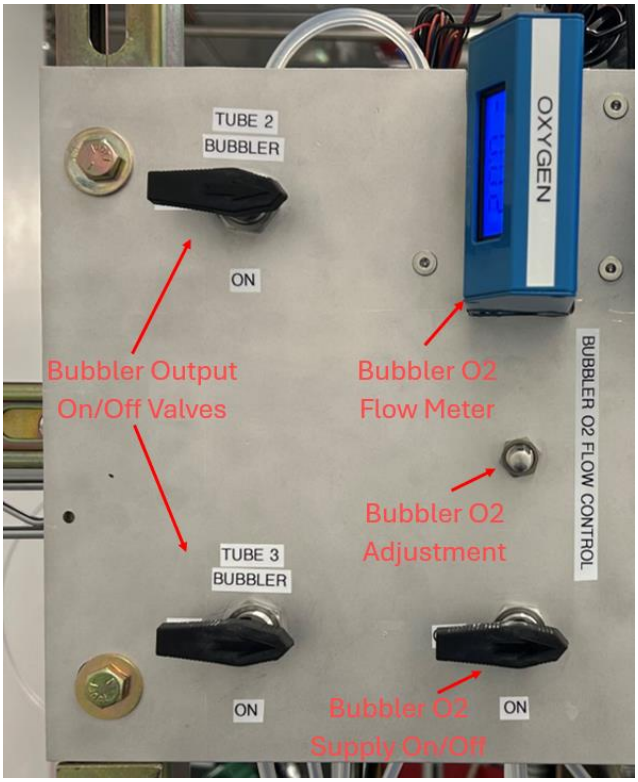

Figure 14: Bubbler Output Controls (Left) and Bubbler O2 Supply Controls (right)

d. To use the bubbler:
    i. Set up the bubbler and ensure the water is boiling with a few bubbles a second.
    ii. Turn on the O2 cylinder.
    iii. Open the output valve to the desired tube.
    iv. Make sure all other process gases are turned off for the desired tube.
    v. Turn on the O2 supply valve to the bubbler.
    vi. Adjust the needle valve to get the desired flow displayed on the flow meter.

4. H2/Ar Mix Process Gas Controls:
    a. Along with N2 and O2, Tube 1 can also be supplied with the 5% H2/95% Ar mix.
    b. The on/off valve for this process gas is located at the gas cylinder.
    c. On the gas panel, there is:
        i. A needle valve to control the flow of the gas.
        ii. A flow meter to measure the flow of gas.

Figure 15: H2/Ar Flow Controls

d. To use the H2/Ar process gas:
    i. Turn on the H2/Ar cylinder. Ensure the cylinder output valve is also open.
    ii. Adjust the needle valve until the desired flow rate is displayed on the meter.