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: Recipe Editing

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. Try using the heat gloves provided whenever possible.
   iii. Everything that interacts with the chamber must be kept clean to avoid contaminating it. This includes boats, user samples, and your hands. Please clean your samples beforehand and wear double gloves when loading samples.
   iv. Remember the purpose of each tube:
      1. Tube 1: Annealing
      2. Tube 2: Dry nitride/oxide growth
      3. Tube 3: Dry nitride/oxide growth or wet oxide growth

b. Bubbler setup: (Wet Oxide Growth)
   i. Disconnect the top of the bubbler from the furnace tube. (Figure 3)
   ii. Fill the bubbler ¼ of the way full for every 2 hours of growth needed.
      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%.
      1. It takes about 90 minutes (about 1 and a half hours) 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.

c. N2 Gas Purge: (if a process gas is desired)
   i. Connect the gas line to the chamber you are using.
      1. Wet Oxide Growth: Connect the gas to the bottom port. The top port is reserved for the bubbler.
   ii. Open the N2 gas cylinder.
      1. Use the metal valve on the top of the cylinder.
   iii. Turn the black three-way valve towards the desired gas.
      1. An arrow on the top of the valve shows which way it is flowing.
   iv. Open the green inlet valve for the N2 gas.
   v. Using the respective needle valve, adjust the flow rate to “1.00” SLM of N_2.

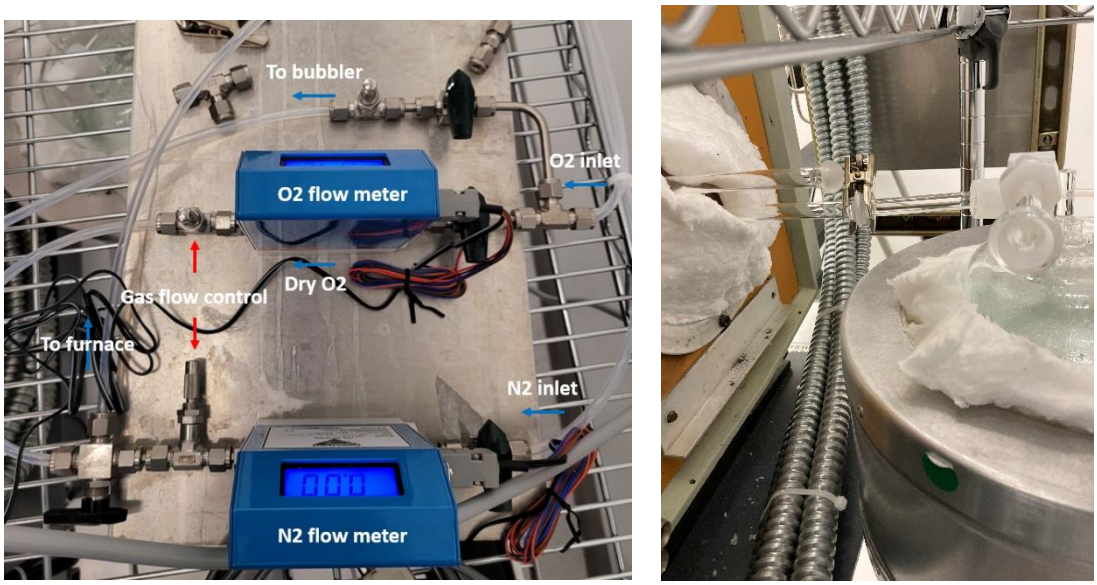

Figure 1: *O2 and N2 gas flow control (left) Disconnected bubbler (right)*

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 2)
    ii. Turn on the "CONTROL" (left) and "ELEMENT" (right) switches. (Figure 3)
    iii. Using the arrow keys and the "SET" button, set all three SOLO controllers to the idle temperature.
        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)

Figures 2 & 3: *Fan switch (left) Controller & heating element switches (right)*

e. Load Samples:
    i. Go to the back of bay 4 and put on another layer of gloves.
        1. This is a precaution to keep the chamber clean and free of ionic contaminants.
        2. Keep in mind the more surfaces you touch, the more you risk contaminating the chamber.
    ii. Grab a sample boat from the glass cabinet (or bring your own for tube 1).
        1. Tube 1: Only use boats from the top shelf labeled 'Tube 1 only'.
        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.
    iv. Load boat into furnace (Annealing, Tube 1):
        1. Wait for the chamber to reach idle temperature.
        2. Slide the boat into the middle of the chamber using the glass push rod.
            a. Do not use the Tube 2 or Tube 3 elephant to load Tube 1.
    v. Load boat into furnace (Oxide/Nitride Growth):
        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. 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.
f. 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.
g. Run process:
    i. Manual Operation: Once process temperature is reached, start a timer.
    ii. Automatic Operation: The recipe will automatically start.
        1. Tips: "Hold" pauses the recipe, "Stop" ends the recipe.
    iii. Wet Oxide Growth:
        1. Connect the bubbler to the chamber.
        2. Close the green valve supplying N2.
        3. Turn the black three-way-valve to the O2 side.
        4. Open the green valve supplying O2.
        5. Set the O2 flow rate to 0.5 SLM.

a. The bubbler must be boiling prior to starting the wet oxidation. (a few bubbles per second)
b. Be careful when refilling the bubbler since overfilling can cause the boiling to stop.

h. Ramp down to idle 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.
            b. 10 °C/min is a starting ramp rate.
    ii. Automatic Operation: Let the recipe continue running.
    iii. Wet Oxide Growth:
        1. Turn off the O2 going to the bubbler.
        2. Turn on the N2 to purge the excess moisture out of the chamber.

i. Unload Samples:
    i. When the idle temperature is reached, open the furnace lid using the furnace gloves and place the lid on the wire rack.
    ii. Slowly unload the sample boat (1 inch per 5 seconds) until the boat is sitting securely in the furnace opening.
    iii. Place the push rod back in its holder. (With the hook facing up)
    iv. Align the elephant to the furnace opening.
    v. Use the push rod to load the sample boat into the elephant.

j. Cool off Samples:
    i. Place the elephant away from the furnace and let cool for 10-15 minutes.
    ii. When the boat is cool, remove it from the elephant.
    iii. Remove the samples from the boat
    iv. Put the boat back into its respective cabinet.

k. Close gases and shut off the tool:
    i. Set each solo to 20 °C.
    ii. Flip the "Control" and "Element" switches to the off position
    iii. Turn the green knob on the gas controls to off. (Figure 1)
    iv. Turn the gas cylinder 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: Updated automatic recipe instructions. General clarity revisions.

Approvals:

Technical Manager Signature: Sandra G Malhotra _____

Date: __1/10/2022_____

Revision History:

Revision	**Author**	**Date**
Original Issue	J. Woo	February 12, 2021
Rev A	Elijah Colter	January 10, 2022
Rev B		
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 1: SOLO main screen.

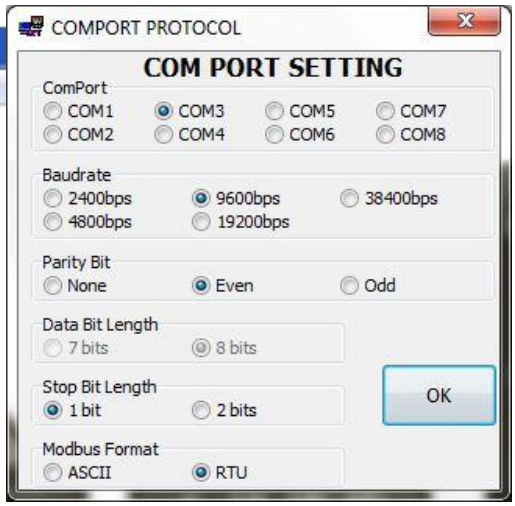

Figure 5: COM port configuration

    c. Optional: Click on "PROTOCOL CONFIGURATION" (Figure 4), double-check the values match Figure 5, and click "OK".
    d. Click on 'SOLO CONFIGURATION' (Figure 1) 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. (**Error! Reference source not found.**).
        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.

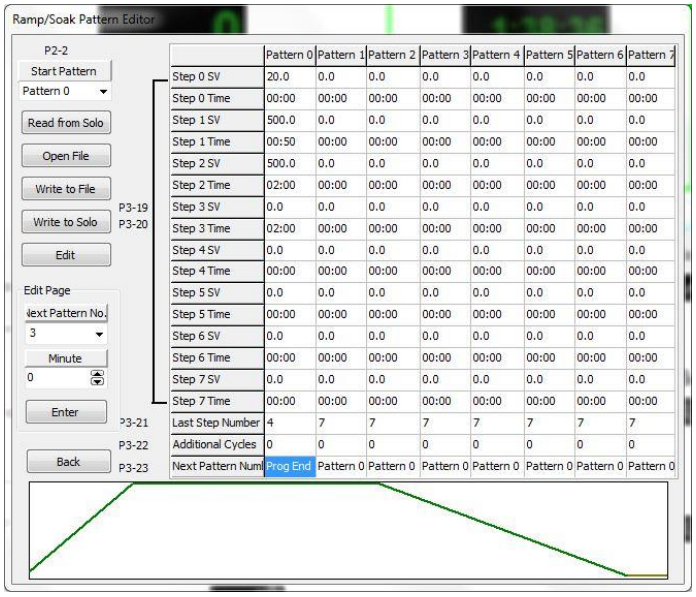

Figure 6: Recipe editor

4. <u>Another way to edit recipes</u> (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 (A, fig 7)
        ii. Last Step Number: ignores any input after this step. (B, fig 7)
        iii. Additional Cycles: repeats this pattern by the input number. (C, fig 7)
        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." (D, fig 7)
        v. Temperatures: The columns in the editor correspond to rows in the notepad. (E, fig 7)
        vi. Times: The columns in the editor correspond to rows in the notepad. (F, fig 7)

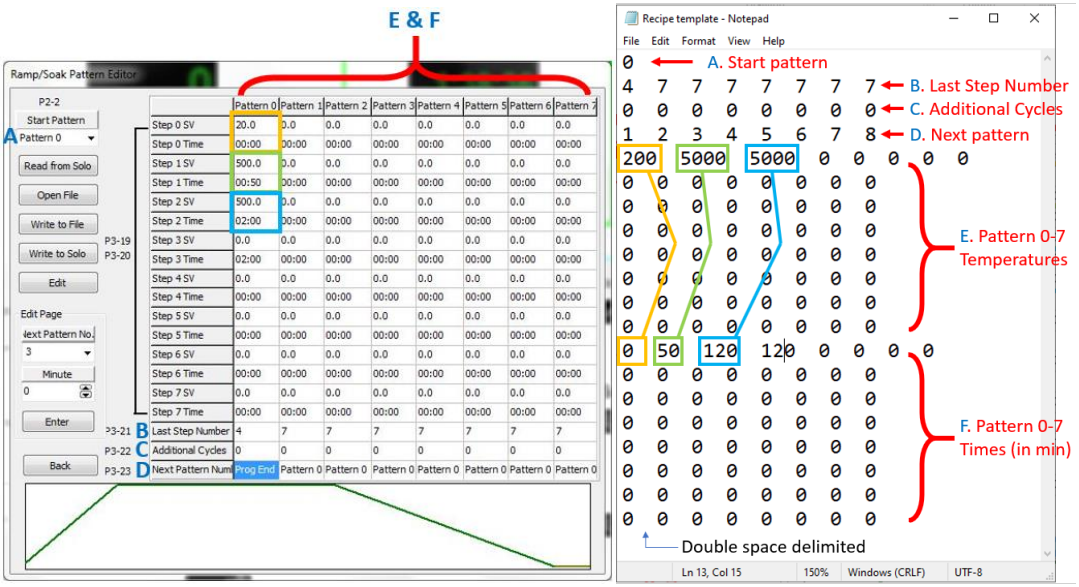

Figure 2. Ramp/Soak Pattern Editor

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 3)

Figure 3. 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: Recipes

AFNF Standard Wet Thermal Oxide Growth Recipe

1. Clean substrate beforehand using piranha for 15 min or acetone/IPA/DI water.
2. Turn on bubbler (Do this ~1.5 hours before loading).
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. Soak @1100C (50 min = 580 nm or 60 min = 640 nm). Connect bubbler to the furnace & open the O2 to bubbler (0.50 SLM) (a few bubbles per second).
7. Ramp down @10 °C/min to 600 °C (50 min). Disconnect bubbler and close O2. Open N2 to furnace (1 SLM).
8. Unload & keep boat in elephant for 10-15 min.
9. Transfer boat to foil to further cool down.
10. 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.
    a. Transfer boat to foil to further cool down if needed
7. Store wafers and clean up the workspace.