

GENERAL PROCESS AND OPERATION SPECIFICATION

Minibrute Oxidation and Annealing Furnaces

I. SCOPE

- a. The purpose of this document is to describe requirements and basic operating instructions for the Minibrute 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 a temperature 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 high temperature.
- d. This equipment can reach 1200°C on the quartz tube heating chamber. Be sure to allow adequate time for cool down of samples 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 contaminants 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 gas cylinders to prevent running out of gas during the process run.
- 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 be sure to 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. Gas setup: The furnace tube should be filled with the needed gas at the beginning of the process. Refer to Figure 1.
 - i. Air: No additional step required
 - ii. N₂: turn the three-way valve towards the nitrogen side prior to opening the nitrogen inlet valve. Adjust the needle valve to obtain the required flow. Typically, 1 SLM of N₂ is used.
 - iii. O₂: turn the three-way valve towards the oxygen side prior to opening the oxygen inlet valve. Adjust the needle valve to obtain the required flow. Typically, 0.5 SLM O₂ is used.
 - iv. Water vapor: disconnect the bubbler from the furnace tube so that vapor doesn't leak into the anneal chamber. Fill the bubbler with DI water up to $\frac{3}{4}$ full using the provided squirt bottle. (~3 bottles = $\frac{3}{4}$ full, each bottle lasts approximately 2 hours). Turn on the heater and set the power to 40%. It takes about 90 minutes until water boils. Water must boil prior to starting the wet oxidation.

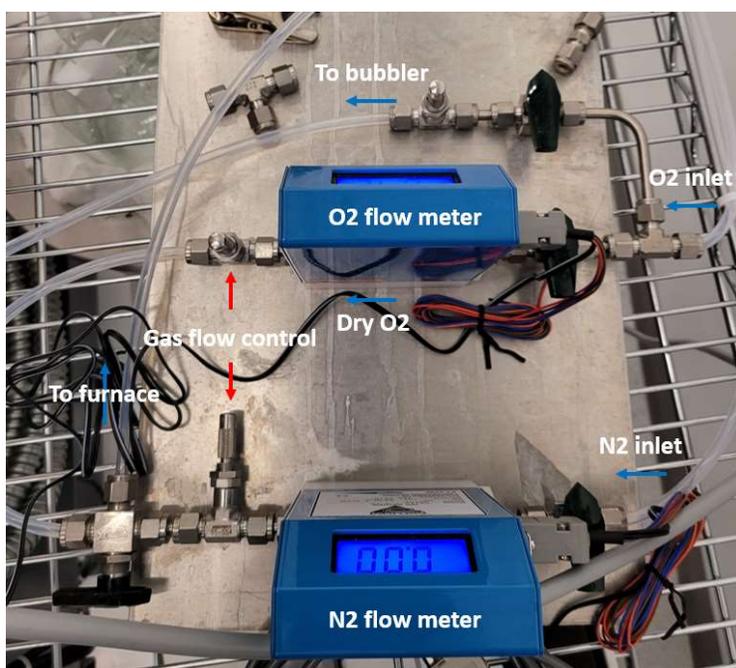


Figure 1. Gas lines

- b. Furnace setup:
 - i. Turn on the main 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 furnace by switching on the controller (left) and heating element (right) switches. (Figure 3)
 - iii. Change the temperature to the idle temperature on all three controllers using the arrow keys and the set button.
 - iv. Wait until the idle temperature is reached (~25 minutes for 600°C).
 1. Typical idle temperature for oxidation is 600°C.
 2. Higher idle temperature may lead to undesirable outcomes.

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- Other low temperature processes may have room temperature as the idle temperature.



Figure 2. Fan switch on the back of furnace.



Figure 3. Controller & heating element switches.

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c. Load Samples:

- i. Grab a sample boat from the glass cabinet (or bring your own for tube 1). The boat from the top shelf labeled 'Tube 1 only' can only be used with the top furnace (tube 1). Other boats on the middle and bottom shelves can be used with tube 2 or 3 but not with tube 1. No exceptions applicable.
- ii. Load cleaned samples onto the sample boat.
- iii. Once the idle temperature is achieved, use the furnace gloves to open the lid and place it on the wire rack. Do not place it on aluminum foil as this can melt aluminum foil and contaminate the lid.
- iv. Place the sample boat in the elephant and align the elephant to the furnace opening.
- v. Using the designated push rod for the furnace tube, slowly push the sample boat into the furnace until the entire sample boat is securely inside the furnace opening.
- vi. Remove the rod and the elephant.
- vii. Use the push rod to slowly push (1 inch per 5 seconds) the boat to the middle of the furnace until the line indicator on the push rod is aligned to the edge of the furnace opening.
- viii. Place the rod back in its holder.

d. Run a process:

- i. The below instructions are for manual operation, for automatic operation, refer to appendix A
- ii. Use each SOLO to ramp up the temperature
 1. Make sure all SOLOs are set to the same value
- iii. Once the process temperature is reached, start flowing the process gas and soak for a desired time.
- iv. At the end of the session, turn off the process gas and flow the ramping gas, if applicable.
- v. Ramp down the temperature to the idle temperature

e. 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.
- iv. Align the elephant to the furnace opening.
- v. Use the push rod to load the sample boat into the elephant.
- vi. Place the elephant away from the furnace and let cool for 10-15 minutes.
- vii. When the sample boat is cool, use the push rod to unload the sample boat.
- viii. Remove the samples from the boat and put the boat back in its cabinet.
- ix. Turn off the furnace, close all gas valves, and turn off the bubbler.

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V. SIGNATURES AND REVISION HISTORY

- a. Author of this document: Jung Hwan Woo
- b. Author Title or Role: Graduate Student Worker
- c. Date: February 12, 2021
- d. Revision: Original Issue

Approvals:

Technical Manager Signature: _____

Date: _____

Revision History:

Revision	Author	Date
Original Issue	J. Woo	February 12, 2021
Rev A		
Rev B		
Rev C		

Appendix A: Automatic Operation

- a. Connect the cable's serial connection to the furnace you wish to control.
- b. Connect the USB cable to the computer.
- c. (Optional) Click Windows Start button -> type 'Device Manager' then enter -> click on 'Ports (COM & LPT)' -> note the port number on USB Serial Port (COM X) -> Open 'SOLO Connect' -> click on 'PROTOCOL' (Figure 4) -> check to make sure the correct COM port is selected. -> check the rest of the settings: Baud rate – 9600, Parity Bit – Even, Data Bit Length – 8 bit, Stop Bit Length – 1 bit, Modbus Format – RTU. (Figure 5)

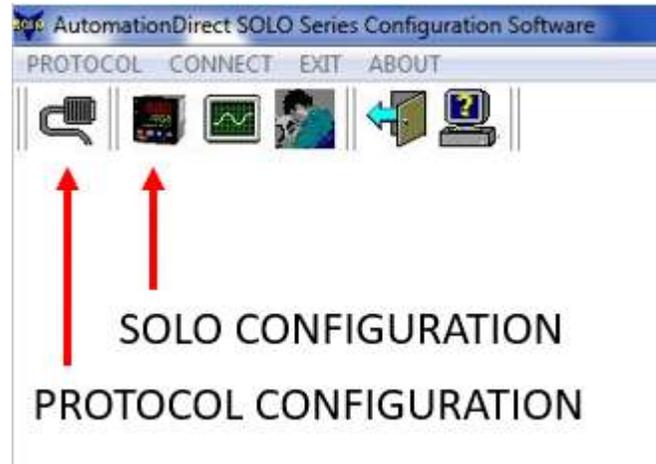


Figure 4. SOLO main screen.

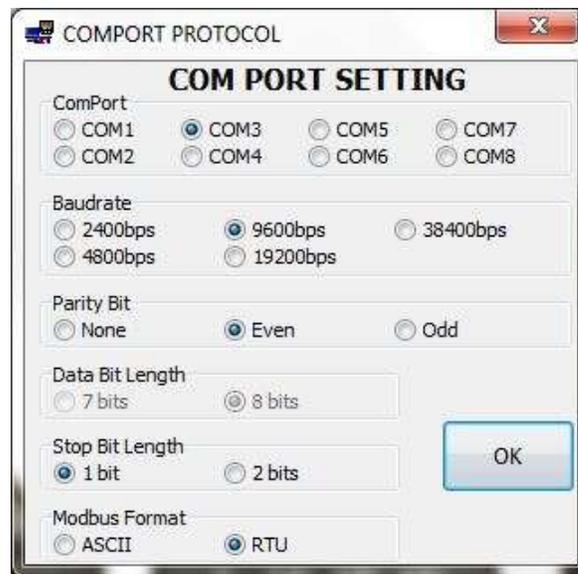


Figure 5. COM port configuration

- d. Click on 'SOLO CONFIGURATION' (Figure 4).
- e. Starting from left to right, set the address to 1, 2 and 3, and click "connect" on each one. (Figure 6).
 - a. Zone # (in Figure 3) = address # (in Figure 6)
- f. Click on a controller image to select it.

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- g. Set the control mode under control setup to 'Ramp / Soak' and RUN/STOP under operation mode to "stop program."
- h. Click on "Edit ramp/soak pattern"
- i. Click on "Edit" to change the recipe. (Figure 7)
- j. You can also import/export ramp recipes from the computer if desired.
- k. Click on "send to SOLO" to write the recipe to the controller.
- l. Repeat e.ai - k for the other two controllers.

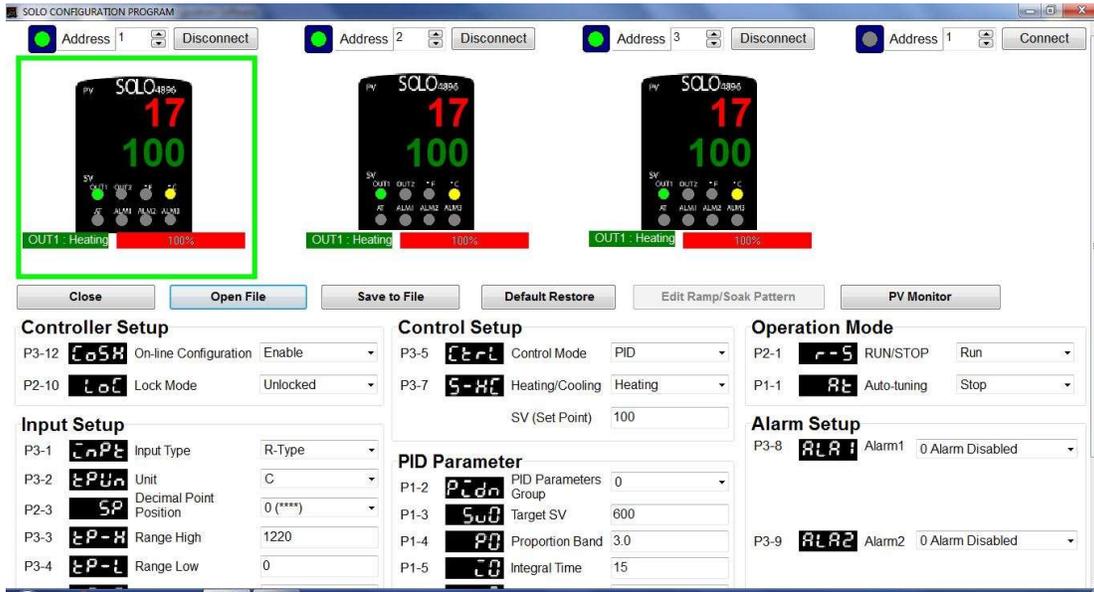


Figure 6. SOLO configuration screen

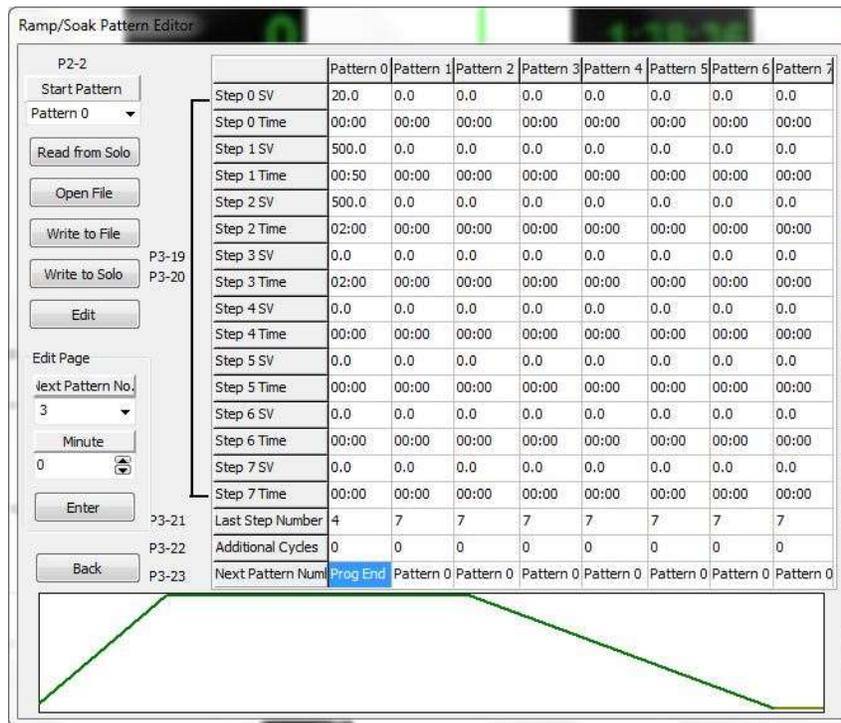


Figure 7. Ramp/Soak Pattern Editor

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- m. To start the run, change RUN/STOP under Operation mode to “Run” for all three controllers. Operate the gas flow as necessary for each period. At the time of writing, the controllers are configured to display the step number in Pattern-Step (X-XX), set temperature in °C, and time remaining in hour.minute (hh.mm) from left to right. (Figure 8)
- n. At the end of your session, set all controllers to the following configuration:
 - i. Control mode – PID
 - ii. SV – 20°C
 - iii. RUN/STOP – Run
 - iv. Failure to do so will cause a problem with the next user who potentially did not receive the supplementary controller training, as they do not know how to change back to the PID mode.



Figure 8. Control panel showing step number (left), set temperature (middle), and time remaining (right).

Appendix B: Recipe Editing

Recipe file:

- a. Recipe file can either be edited within the software or written using a text editor.
- b. The recipe file is double space ('__') delimited number arrays
- c. Component descriptions (Figure 9):
 - A. Start Pattern: User may choose the pattern to begin the ramping. Choose from 0-7
 - B. Last Step Number: ignores any input after this step.
 - C. Additional Cycles: repeats this pattern by the input number.
 - D. 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."
 - E. The rows in txt file represents the temperature values in the editor columns.
 - F. Same as E for time. Time in minutes.

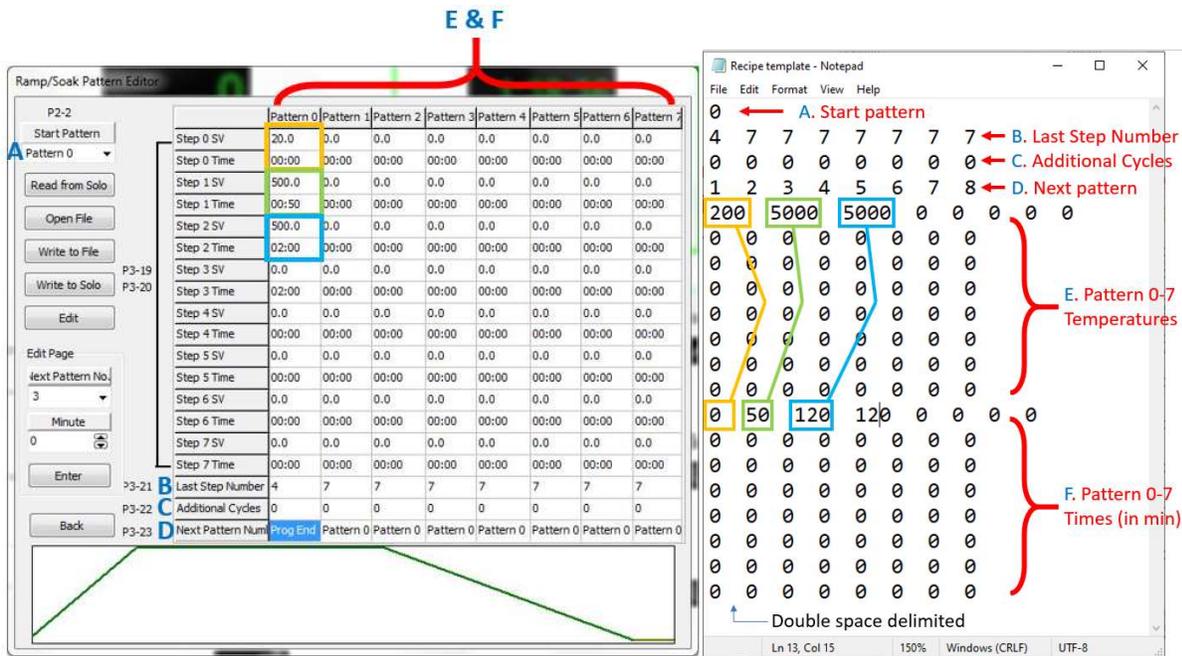


Figure 9. Recipe text file.