Computer Controlled Carbon Nanotube Growth Furnace

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ABSTRACT

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Carbon nanotubes (CNT) are used for various applications ranging from micro filters to low-reflection materials. However, reproducibility and predictability in CNT growth have been poor. One approach to improving reproducibility is to minimize process variations. Thus, we have built a new CNT growth system with computer control of the process. This includes computer control and monitoring of the heater temperature and gas flows, allowing more precise timing and monitoring of the process. We also keep specific logs of the process for further replication. We will discuss the design and build process and control benchmarks of the new system.

Keywords: Automation, Computer, Control, CNT, Carbon, Nanotube, Growth, Process, Furnace

ACKNOWLEDGMENTS

To Dr.Vanfleet, who stuck with me through all my ups and downs as a researcher. To my Professors Richard Sandberg and Chanel Earl, who gave me the motivation and instruction needed to write this report. To my God, who has strengthened me and made all of this possible.

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Chapter 1

Introduction

Carbon nanotubes (CNT) are carbon tube structures that are on the nanometer scale. Due to their size, CNT are used for a broad range of applications, including some here at BYU. Some applications here at BYU are MEMS (microelectromechanical systems), non-reflective material, and neural interfacing, as discussed in cited articles. [1] [2] [3] [4] However, CNT growth (production) can be difficult. This process requires careful control of gas flow and temperature, as mentioned in these articles. [5] [6]

1.1 Motivation

At BYU the CNT growth process used is inconsistent, giving different growth lengths for each student and even for different samples for the same student. Individual students will attempt to run the same gas flows and times, but still have inconsistencies due to errors in timing and other steps. Students will also tweak this process over time to achieve improved growth quality. This works for them; however, replication of their process is difficult. Additionally, even if two students attempt the same process, differing results will be achieved. To combat these differences, we are building a process that will automate the temperature, gas flow, and time at each step. This will produce consistency for the growth process. Additionally, this automated process will create an accurate log of the temperature and gas flows used for each growth, allowing for effective repeatability. We did this by wiring and building a system of mass flow controllers (MFC), a furnace, and valves. We also used LabVIEW to automate and log the process. In this report, we will discuss the development of this computer-controlled furnace, show effectiveness with a process run and logged by the computer-controlled furnace, and discuss the impact and future work associated with this project.

Chapter 2

Development

The development of this automated furnace has four main steps. First, we constructed the gas flow tubing that connects the Mass Flow Controller(MFC) to the furnace. Second, we constructed wiring to connect the devices to the computer. Third, we communicated with each device using RS-485 communication. Fourth, we created LabVIEW code to run a user-inputted furnace process.

2.1 Gas Flow Tubing

Our first problem was finding a way to connect all of the MFC cleanly and effectively, which avoided any gas leakage. We came up with the idea of having a metal plate, which we attached all of our parts with steel tubing and Swagelok valves, see Figure 2.1.

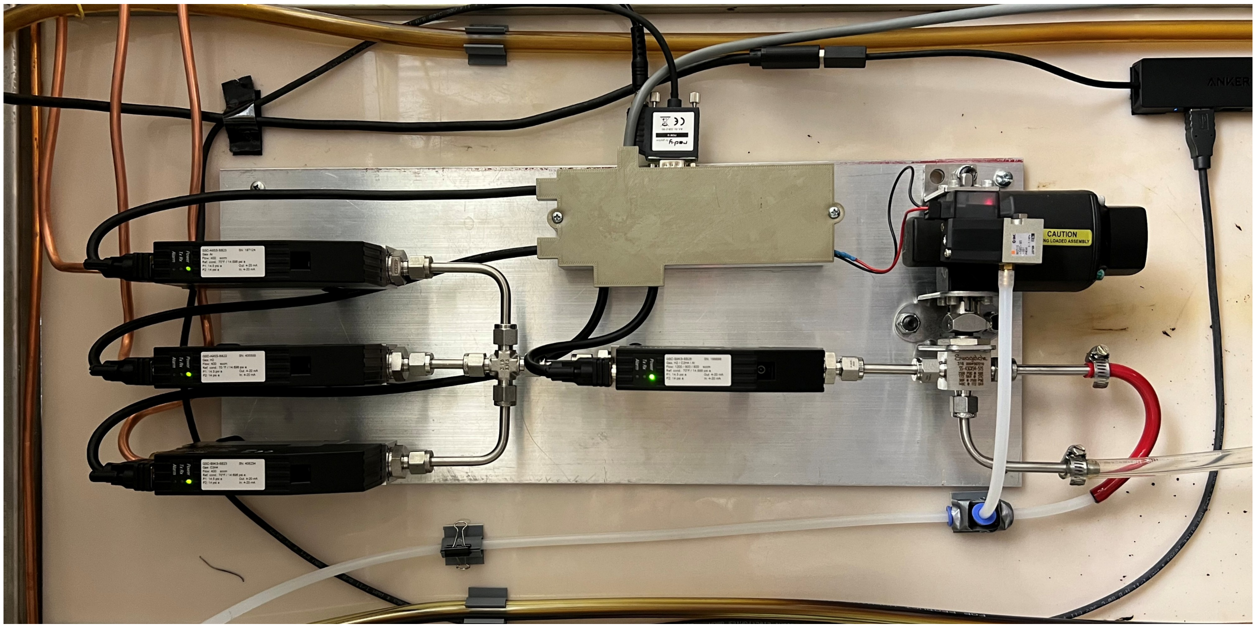

Figure 2.1 Picture of gas flow system plate. This is a machined plate with four MFC, a 3-D printed box with wiring, two valves, one solenoid, and one pneumatic. This is used for the control of gas and air flow.

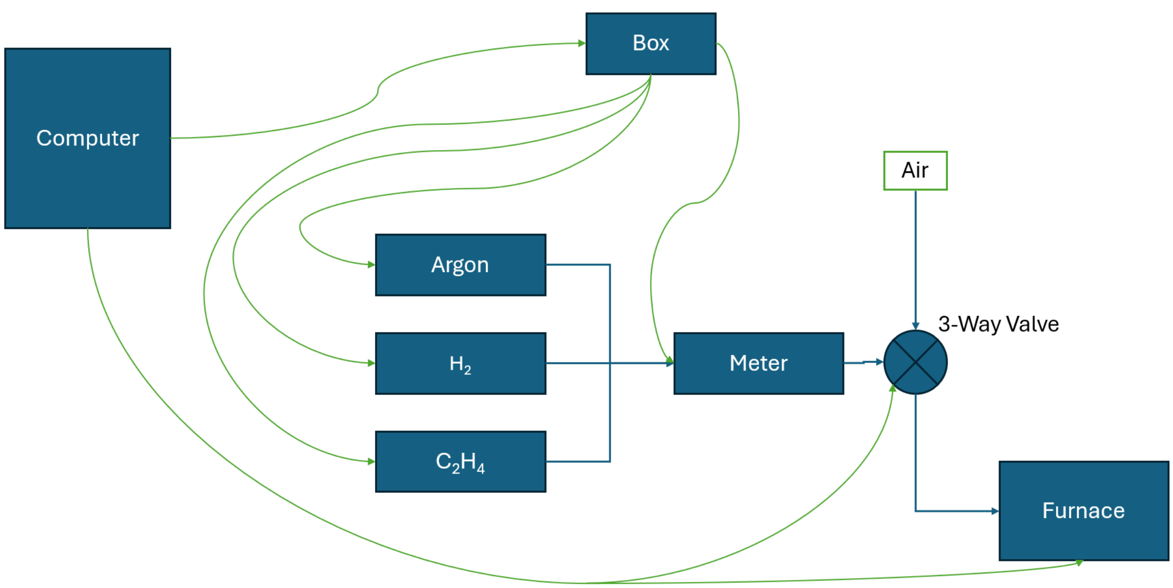

Figure 2.2 Schematic showing wiring for Furnace and MFCs. This is the same plate as figure 2.1 showing wiring to the furnace and computer. Green lines are wires, blue are gas lines.

This machined plate we built, containing four MFC, a solenoid valve, a 3D-printed box holding wiring, and a 3-way pneumatic valve, is shown in Figure 2.1. Three MFC flow gases, Argon, hydrogen gas, and ethylene, respectively, and the fourth is used as a meter. The three gas flow MFC control and read the gas flow for each of the gases. The meter is there to verify gas flow and potentially check calibration. As shown in Figure 2.2, the three MFC connect with tubing continuing to a 4-way junction. They then flow from that to the meter. From the meter, they go to the 3-way valve where it switches between airflow and gas flow. The pneumatic valve is activated with pressurized air. The pneumatic valve is actuated with a solenoid valve, which turns on and off digitally. When the solenoid is on, it presurizes the 3-way valve, which flows air to the furnace. When the solenoid valve is off, the 3-way valve flows gas to the furnace.

We needed a way to both run pressurized air to the pneumatic valve for actuation and unpressurized air to the furnace. To do this, we needed pressurized air at about 70 psi, and we needed to significantly decrease this to a small amount of air going into the furnace, similar to the controlled 20 psi gases from the MFC. The air going into the furnace just needed to be low enough flow and pressure that it wouldn't cause problems in the furnace. We tried to have a flow comparable to the other gases when looking through a bubbler(a beaker of liquid used to show gas flow from the furnace visually). To do this, we used a 100-micron orifice. We used pressurized house air and split it into two lines, one going to the valve to activate it, and the other going to the furnace. For the air entering the furnace, we added a 100 micron orifice between the tube and the 3-way valve junction. The orifice was connected to the end of the tube with epoxy. This orifice reduces the pressure from 70 psi to a safe flow usable and seen through the bubbler.

2.2 Wiring and RS-485 Communication

After finishing the gas plate, we needed to communicate with and automate everything. First we start by communicating with the furnace. To do this, we wired 2-wire communication according to the instructions given in the furnace manual [7]. Once we could communicate with the furnace using LabVIEW code given by Eurotherm [8], we needed to communicate with the MFC. We were able to do this using a 4-wire RS-485 communication protocol. We also used a 24-Volt power source to bring power into the MFC as described, along with other communication protocols by MFC communication manuals [9] [10]. First, we used LabVIEW RS-485 protocol to communicate with one MFC, then connected multiple MFC together with one line as done with RS-485 communication. We tried many ways to connect the 2-wire furnace communication and the 4-wire MFC communication, but came to know that it is not possible to have both 2-wire and 4-wire on the same line. In order to deal with that we used a 2-wire RS-485 line to communicate with the furnace and a separate 4-wire line to communicate with the MFC. Then we got the valve, which requires the power from the MFC power supply, but is a 2-wire communication which we took off of the furnace line. This required two separate RS-485 communication lines. One with the furnace and valve, and the other for the MFC. This is shown in Figure 2.2, the box is where we connected all of the MFC wiring and the power to the 3-way valve.

2.3 Labview

Now that we have the hardware, we need to communicate with the furnace. We did this with LabVIEW. We needed code to communicate with the furnace and MFC to run a CNT growth as needed. We used LabVIEW first for convenience, it is an effective method for communication with lab equipment, and Eurotherm already had LabVIEW code for communicating with the furnace, which we used. We will discuss the use of this code and computer-controlled furnace system in the next chapter.

Chapter 3

Use of Computer Controlled Furnace

In this chapter, we will describe the use of the Computer Controlled Furnace. Previously, users had to go through a long process, carefully following steps. Now we have developed a user interface that takes them through the process. This provides a much simpler process for the users.

3.1 Recipe and Setup

The process we have developed goes as follows. First, the user will insert a Carbon Nanotube(CNT) sample into the furnace and turn on the gases used for CNT growth, hydrogen, ethylene, and argon. Once that is done, they will run the LabVIEW automated process file we created, see A. Once they run the file, they will see a recipe tab shown in Figure 3.1. They can edit the recipe by inserting lines, removing lines, adding an infiltration, and editing the flow time, temperature, and gases flown. Once they have done this, they can run the process.

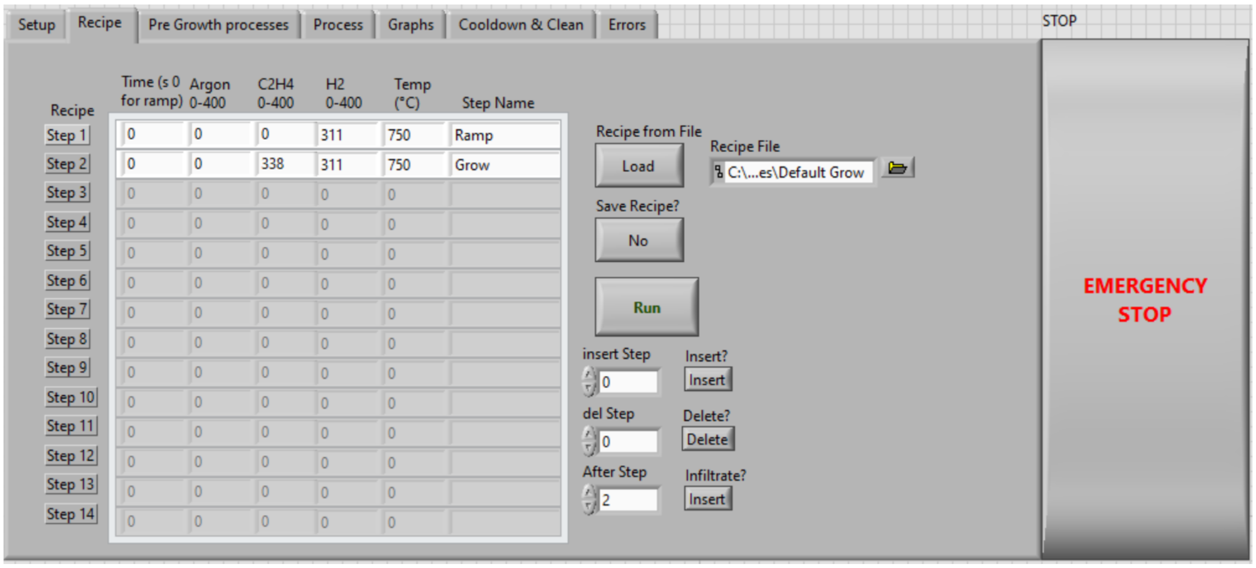

Figure 3.1 Recipe input user interface. Shown is the recipe array with steps, flow time, Gases, temperature, and step names along with buttons to load, save, or run the recipe. At the bottom, we have options to insert and delete steps and add an introduction with a stop button on the right.

The program then runs the user through a setup process. First, it runs all the gases, telling the user to check the gas cylinders' pressure, which should be at 20 psi. Then it has them check the bubblers as it runs each gas in order: argon, then hydrogen gas, then ethylene. Then it requires the user to leak check while it runs hydrogen gas and ethylene using a flammable gas checker on all the joints. Once they have done that, it starts going through the step-by-step growth process that the user previously input into the recipe.

3.2 Growth Process

The program then runs the user's growth process recipe, to do this, it goes through each step of the recipe. Setting the temperature and gas flows, then dwells for the given time or ramps to the given temperature if no dwell time is given, and repeats this process for each step. The user can see the

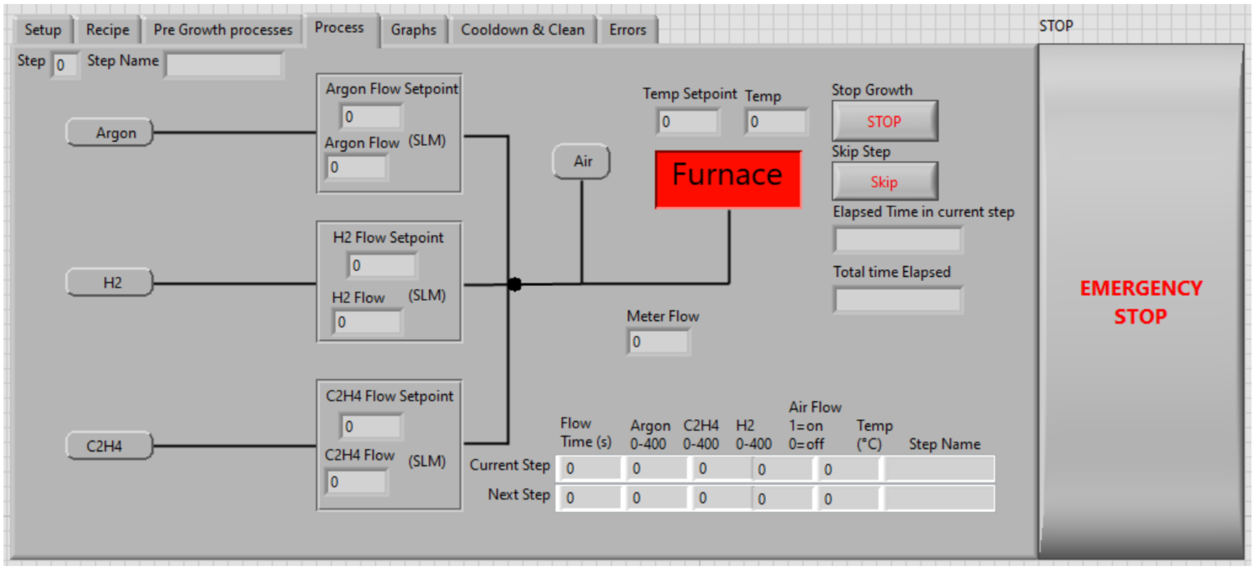

Figure 3.2 Here we have the Growth Process User Interface. This shows what gases are flowing to the furnace with the same format as figures 2.1 and 2.2, along with the setpoint and actual temperature of the furnace. At the bottom, it shows the current and next steps in the recipe. There are Skip, Stop, and emergency stop buttons shown on the right, along with the elapsed time in the current step and total time.

current step and next step along with all of the MFC and furnace setpoints and values as shown in Figure 3.2. In addition to that it shows a graph of the process up to that point as seen in Figure 4.1.

Once the growth has finished, the furnace is cooled to 200°C, allowing the user to take out the sample. Once the sample is removed, the furnace heats back up to temperature, running air to clean the furnace, and has the user move the tube to help clean. After that is finished, it cools down, and once the furnace reaches 200°C, the program stops. This leaves the furnace set to 0°C with all gases turned off and the 3-way valve switched to air, which is the ideal state of the furnace when it is not being used.

Chapter 4

Application

To test this automated process we developed, we ran this process on three samples. A graph of the process is shown in Figure 4.1. This shows all the flow rates and temperature over time, along with the reading of the meter. The process that we ran was a growth of 5 minutes. The program ran hydrogen gas (the green line) until the furnace reached the temperature of 750 °C (the yellow line). Then, in addition to hydrogen, it flowed ethylene (the red line) for 5 minutes. The blue line is the reading of the meter. We did this process three times, and the resulting parts are shown in Figure 4.2. This is proof that the automated furnace process succeeded in growing CNT.

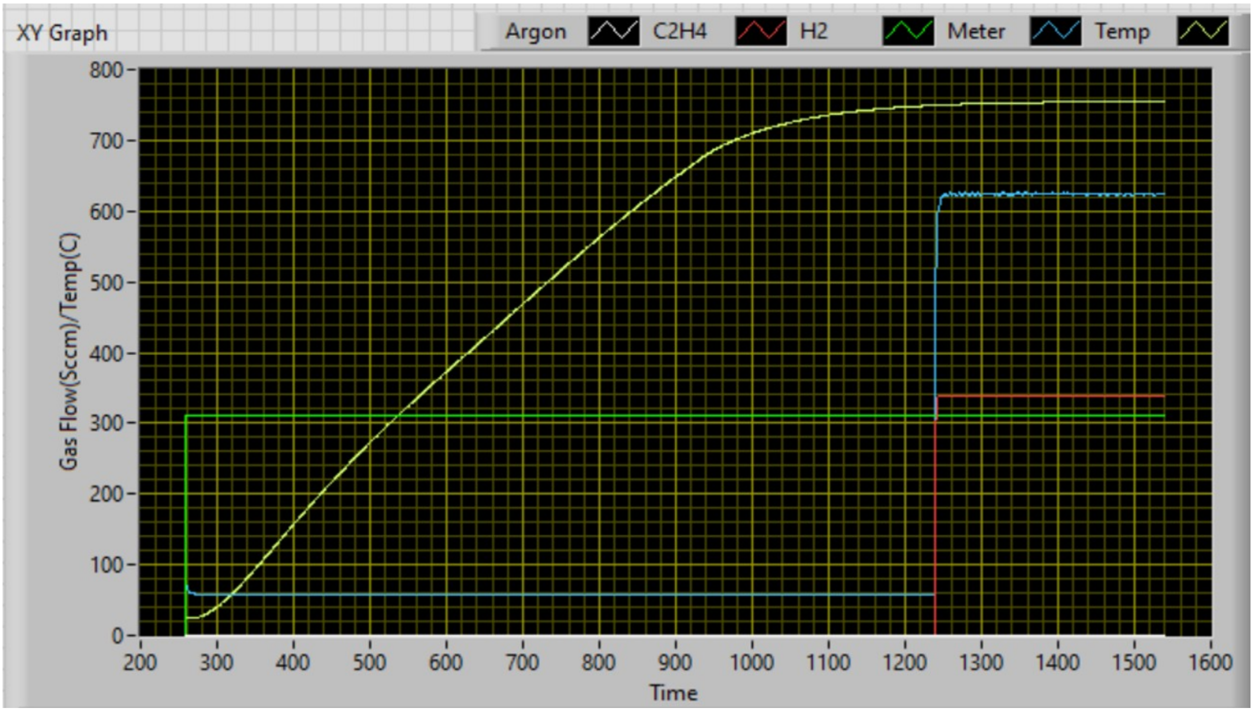

Figure 4.1 Graph of Growth Process we ran on the Automated Furnace. In this graph, we see the flow of hydrogen and ethylene (red and green lines), the meter reading (blue line), and the measured Temperature (yellow line).

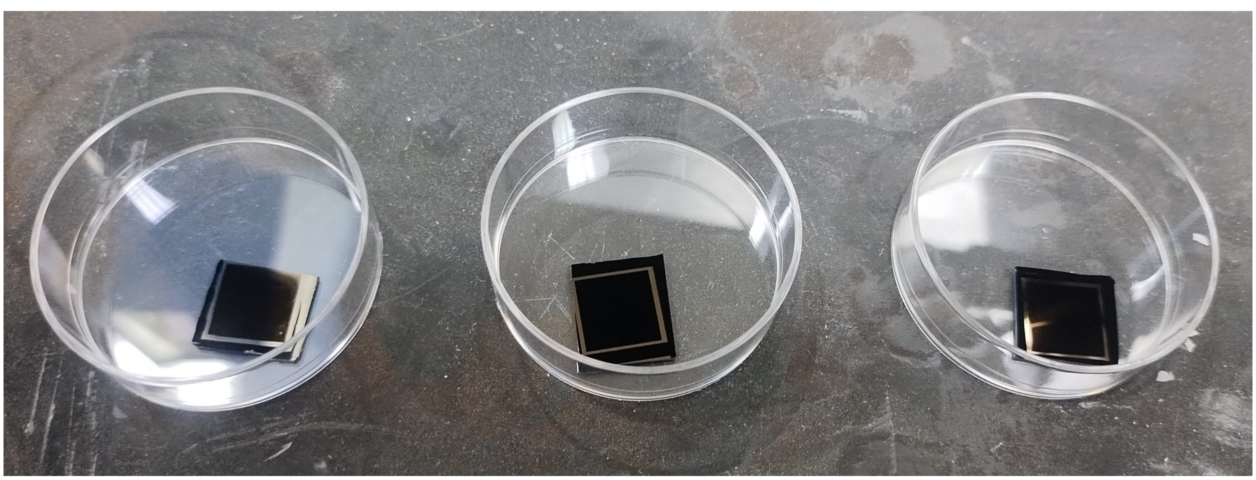

Figure 4.2 Three CNT Samples Grown in Computer Controlled Furnace. The black substance on each of the samples is the CNT.

Chapter 5

Conclusion

Carbon Nanotubes (CNT) are used in a variety of applications, including medical and non-reflective materials. However, growing CNT requires consistency across samples. To achieve this, it is essential to control user-induced inconsistencies. Additionally, precise monitoring of gas flow, temperature, and timing at each step is necessary. So we developed a computer controlled CNT growth furnace that controls and monitors gas flow, temperature, and timing of each step.

5.1 The Computer Controlled CNT Growth Furnace

This computer-controlled furnace provides consistent growth conditions and logs temperature and gas flow data for each run. It also enhances safety and ease of use. Since the program guides users through the process, minimal training is required before they can successfully grow CNT. Furthermore, the program enforces safety protocols by preventing the simultaneous flow of air and hydrogen gas, which could otherwise lead to combustion.

5.2 Future Work

Several improvements are needed to enhance the program. First, an error-handling and alarm system should be implemented. Second, the user interface requires further refinement through tweaking and beta testing to improve flexibility. Additionally, the PID controller differs from that of the previous furnace and will require tuning to achieve comparable temperature control. Another important step is to evaluate the furnace's consistency in CNT growth. A comparative study between the computer-controlled furnace and user-operated systems would provide valuable insights into their reliability.

Appendix A

Automated Process LabVIEW Code

This appendix gives screenshots of the LabVIEW code we created to run the automated furnace process. For each screenshot, there is an explanation of the use and or function of the code.

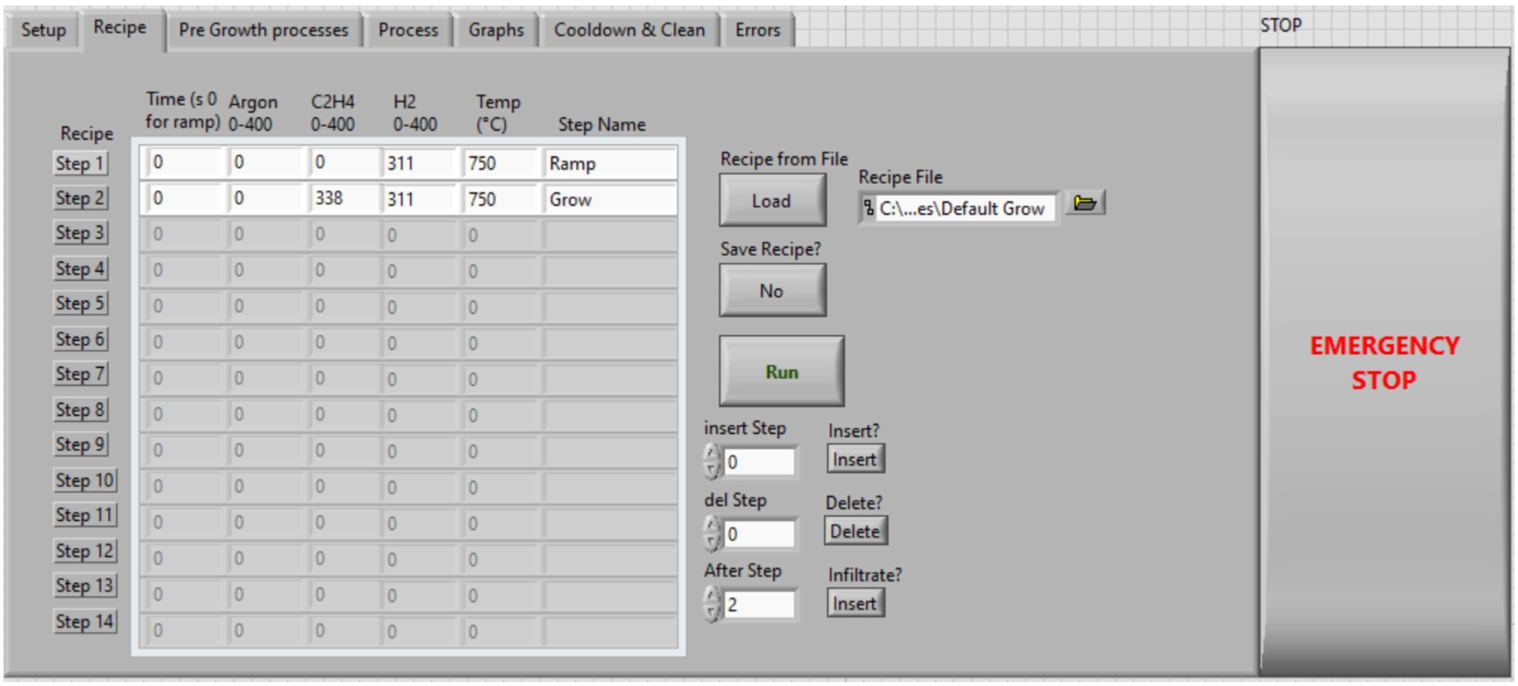

Figure A.1 User interface panel used for recipe editing. Steps and flows are shown in the array on the left. To load a recipe from a file click the load button which will load the recipe from the Recipe File given. To save the Recipe to the given Recipe File click Save Recipe this will override the file that was loaded unless you choose a new file. To insert a step into the recipe choose which step you would like to insert and then click insert. Similar to the insert before option in a spreadsheet. To delete a step select the step to delete then click delete. If you want to add an infiltration select the step you want to add it after then click Insert. If you need to stop the program at any time click Emergency Stop.

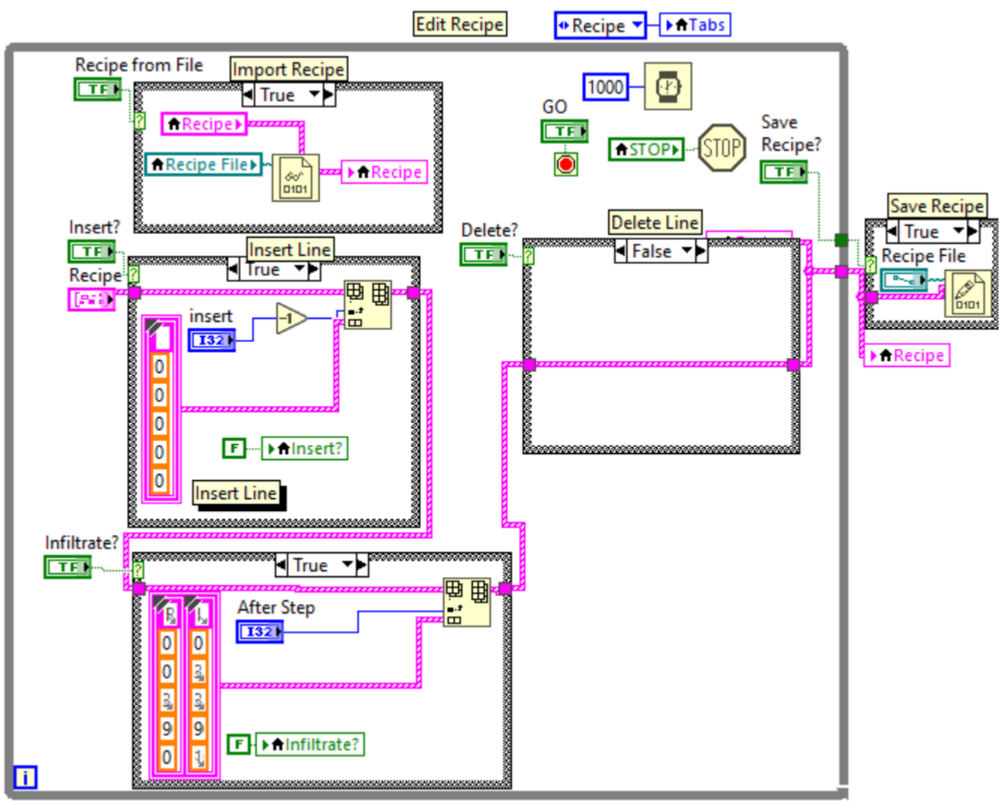

Figure A.2 Code Used to edit the recipe. Import recipe imports a bit file based on the file received from the user when they indicate. Insert line inserts a line of zeros at the spot indicated by the user. Infiltrate adds the lines to the recipe required to infiltrate the sample. Delete line deletes the line imputed by the user. When run is pressed it leaves the while loop and saves the recipe if the user specifies.

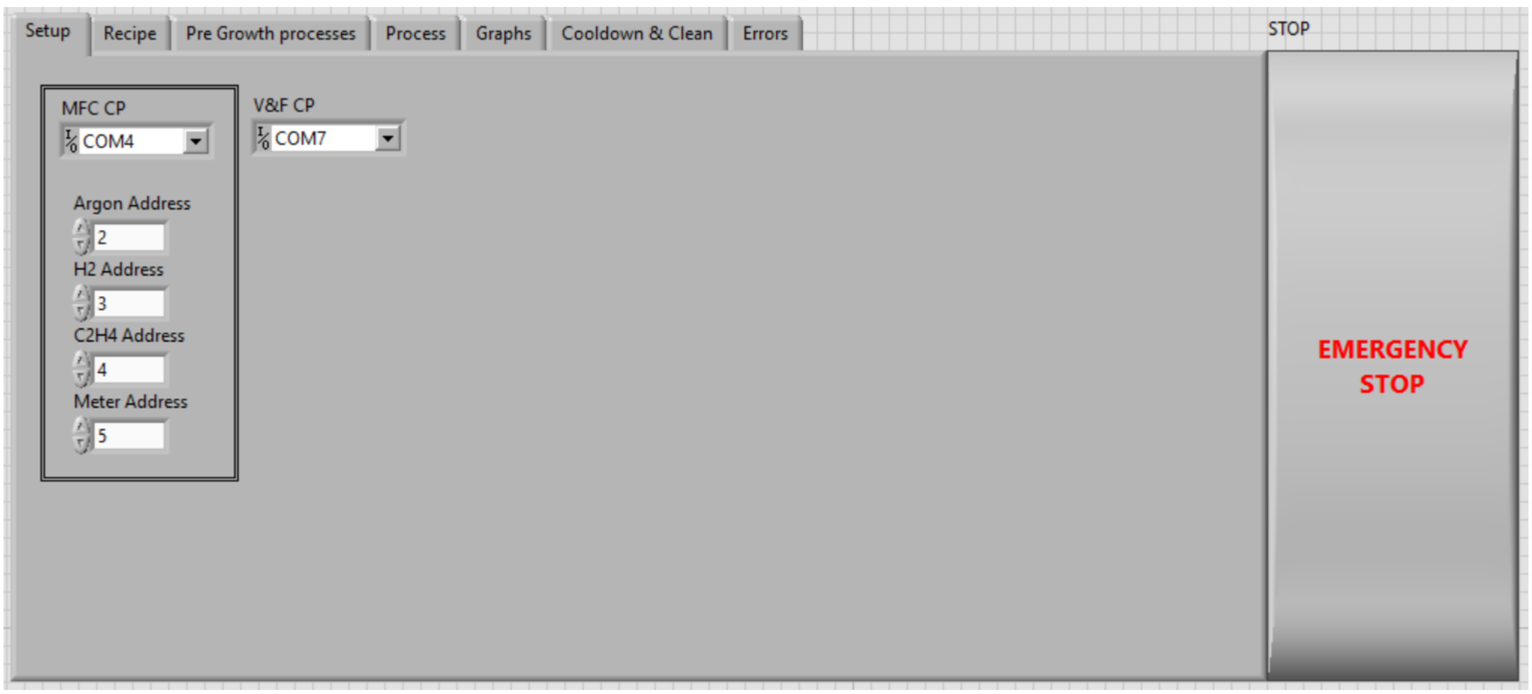

Figure A.3 panel indicates communication ports and slave addresses for Furnace, Valve, and MFC.

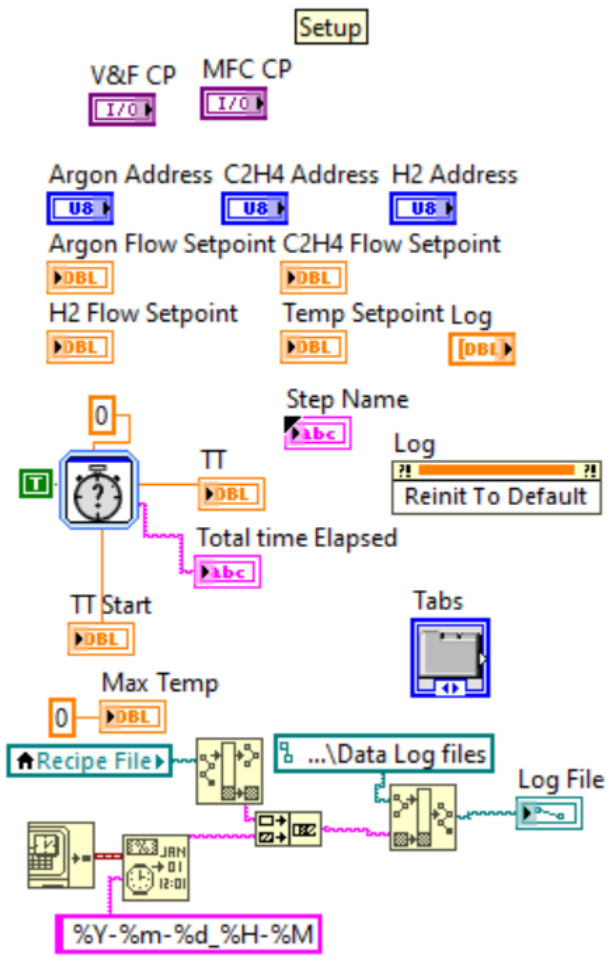

Figure A.4 Code initializes variables and starts the time for the process to be run. The bottom is the code to create a log file based on the location of the Recipe file. If there is an error make sure Data Log files are in the same folder as the recipes folder.

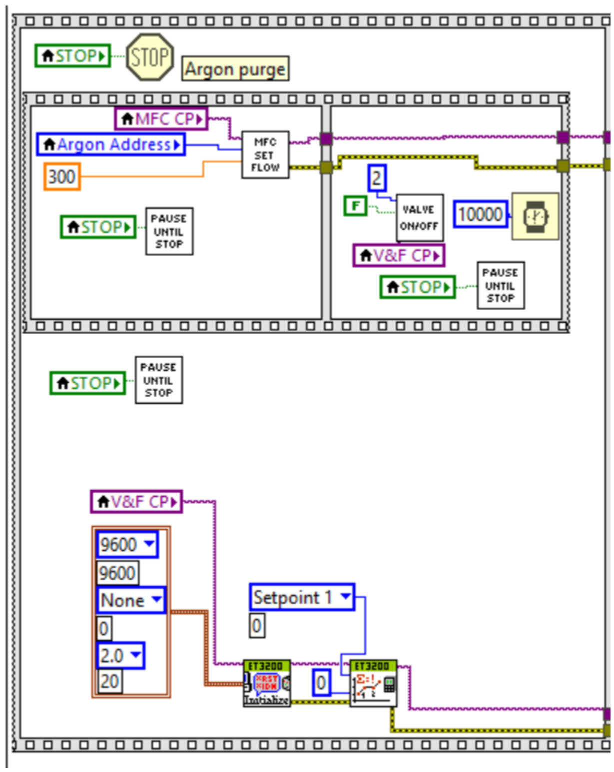

Figure A.5 Code to run Argon purge. Sets argon MFC to run at 300 sccm then turns the 3-way valve to run gasses and waits 10 seconds. Initializes furnace communication and sets the setpoint to 0.

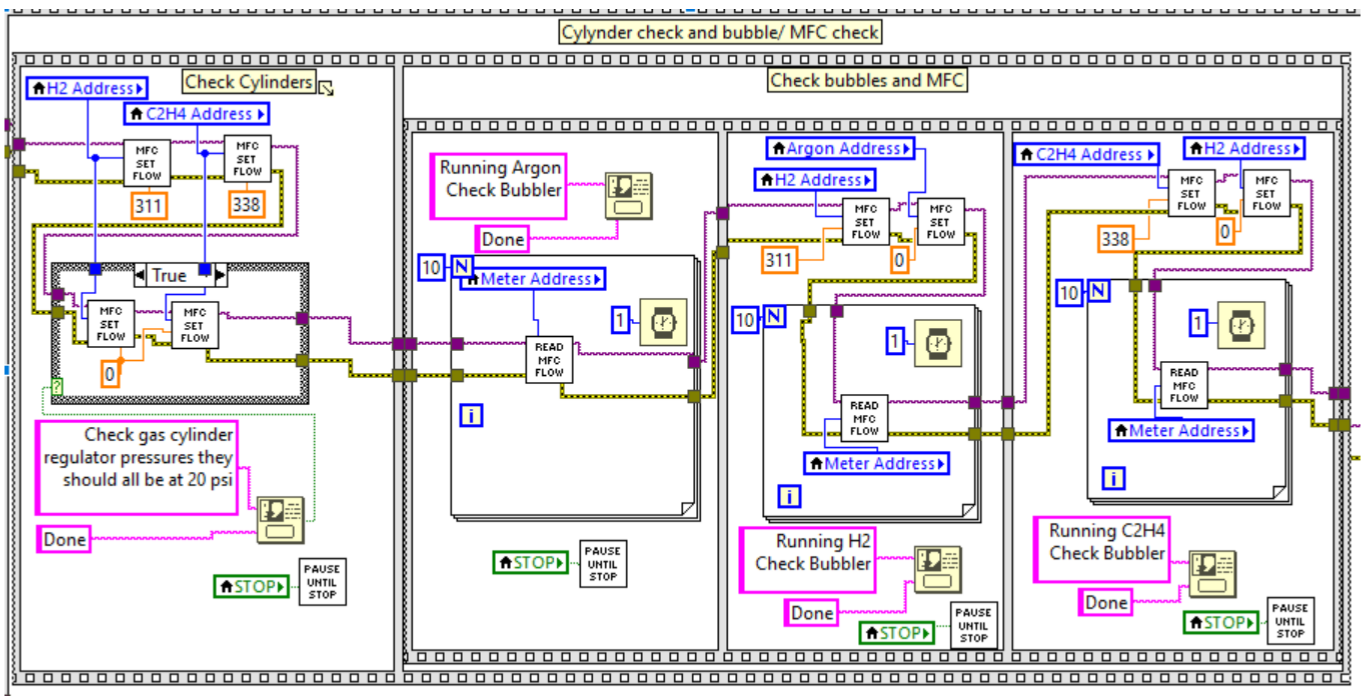

Figure A.6 Code to allow users to check gas cylinders and Gas flow. Sets both H2 and C2H4 to 311 and 338 sccm respectively remember Argon is still running. Sends popup message telling user to check cylinder pressure. When they select Done turns off H2 and C2H4 flow. Prompts the user to check bubbler to see if argon is flowing. Uses a for loop to check MFC flow with the meter. Then turns on the H2 flow and turns the argon flow off. Has the user checked the bubbler and checked the meter flow again. Turns on C2H4 flow and turns off H2. Has the user checked the bubbler and checked meter flow. Once all gasses are flowing move to the next step.

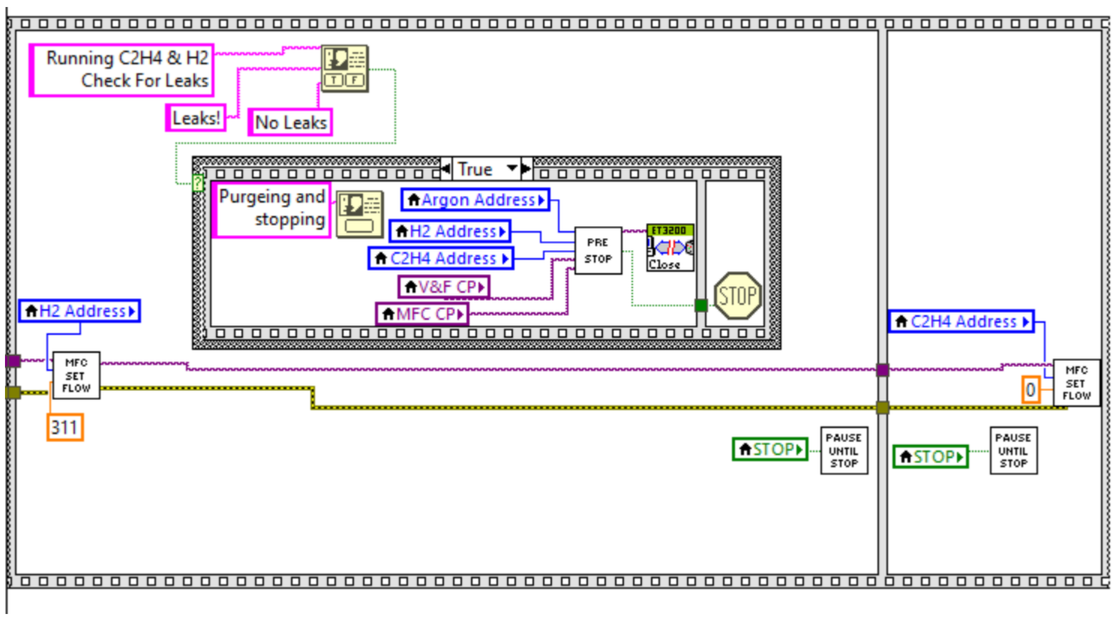

Figure A.7 Leak Check Code. Turns on H2 since C2H4 is already running and prompts the user to preform a leak check. If the user says there are leaks it goes through emergency stop process. If there are no leaks it continues and turn off C2H4 flow.

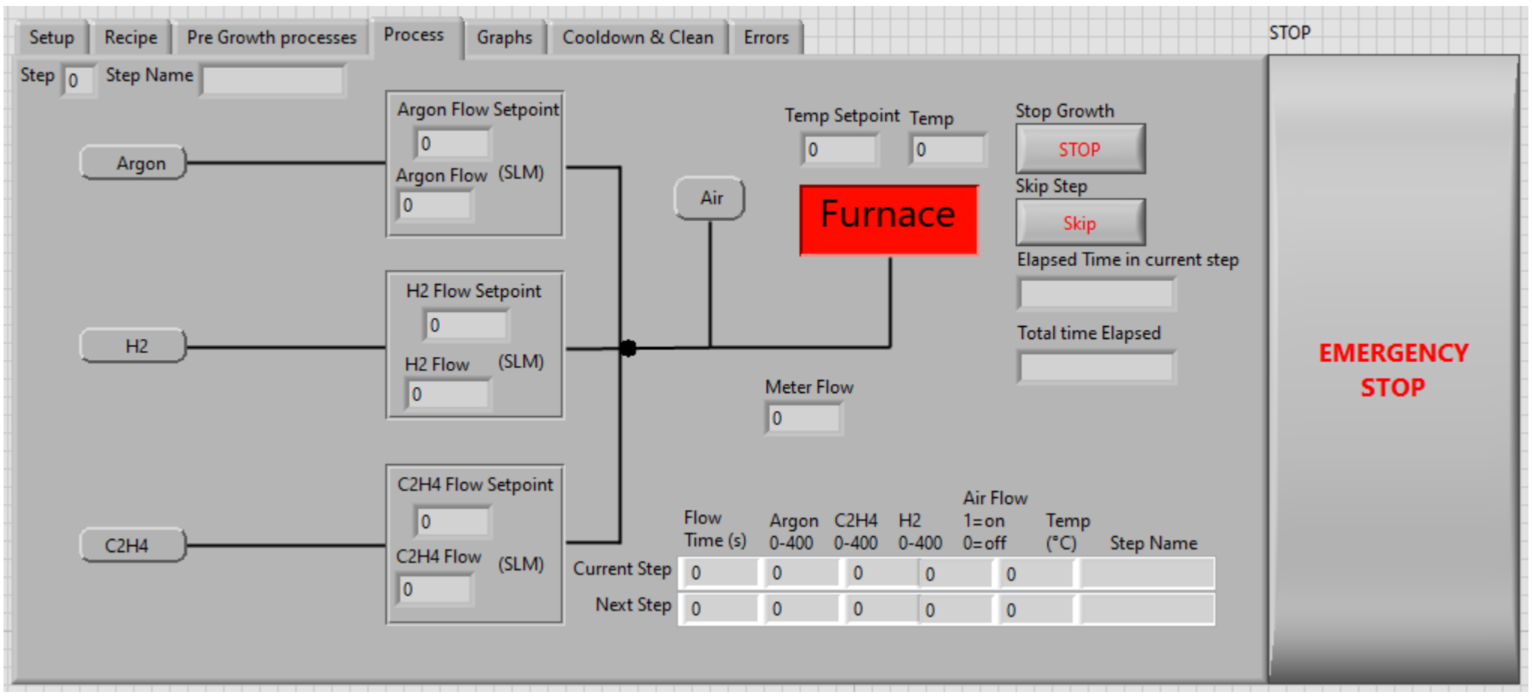

Figure A.8 User interface for growth process. Top left shows the step number and the name of the step. shows the setpoints and flows for all the gasses. Shows the flow reading for the meter. Shows the furnace setpoint and actual temperature. Shows the total elapsed time and the time elasped in the current step. Shows the current and next step parameters. Stop growth is used to stop the growth which just skips the growth this is to be used when you don't need to emergency stop so you can properly shut down the furnace and everything. Skip step will skip to the next step if needed. Emergency stop is used when you need the furnace to stop and gas to go to argon if used get a grad student help to shut everything else down.

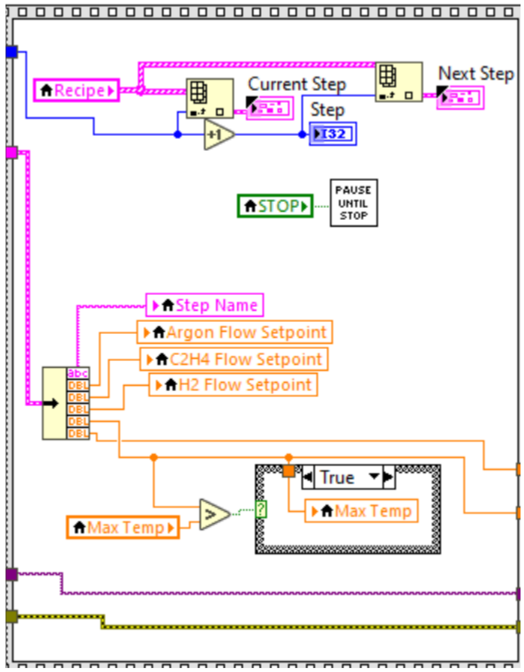

Figure A.9 Code for Reading the Recipe. It starts by reading the Recipe and displaying the current step and next step. Check the given temperature to confirm it is within the range of the furnace.

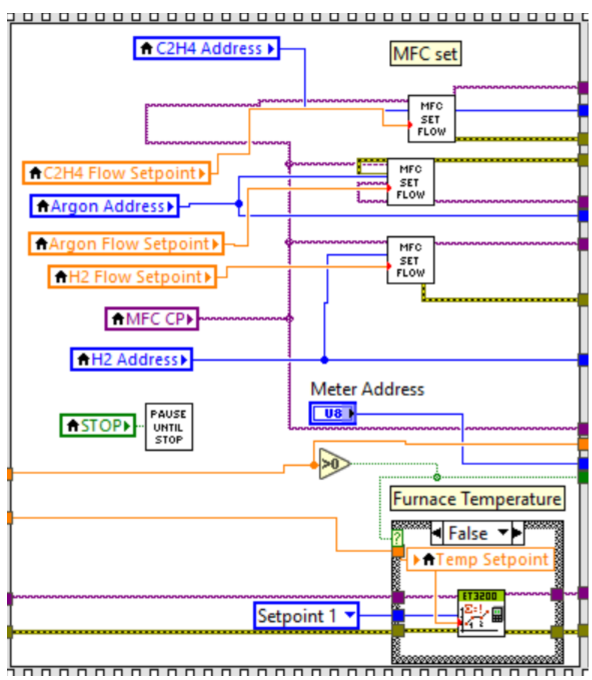

Figure A.10 Code to set the MFC flow rates and furnace temperature for the step.

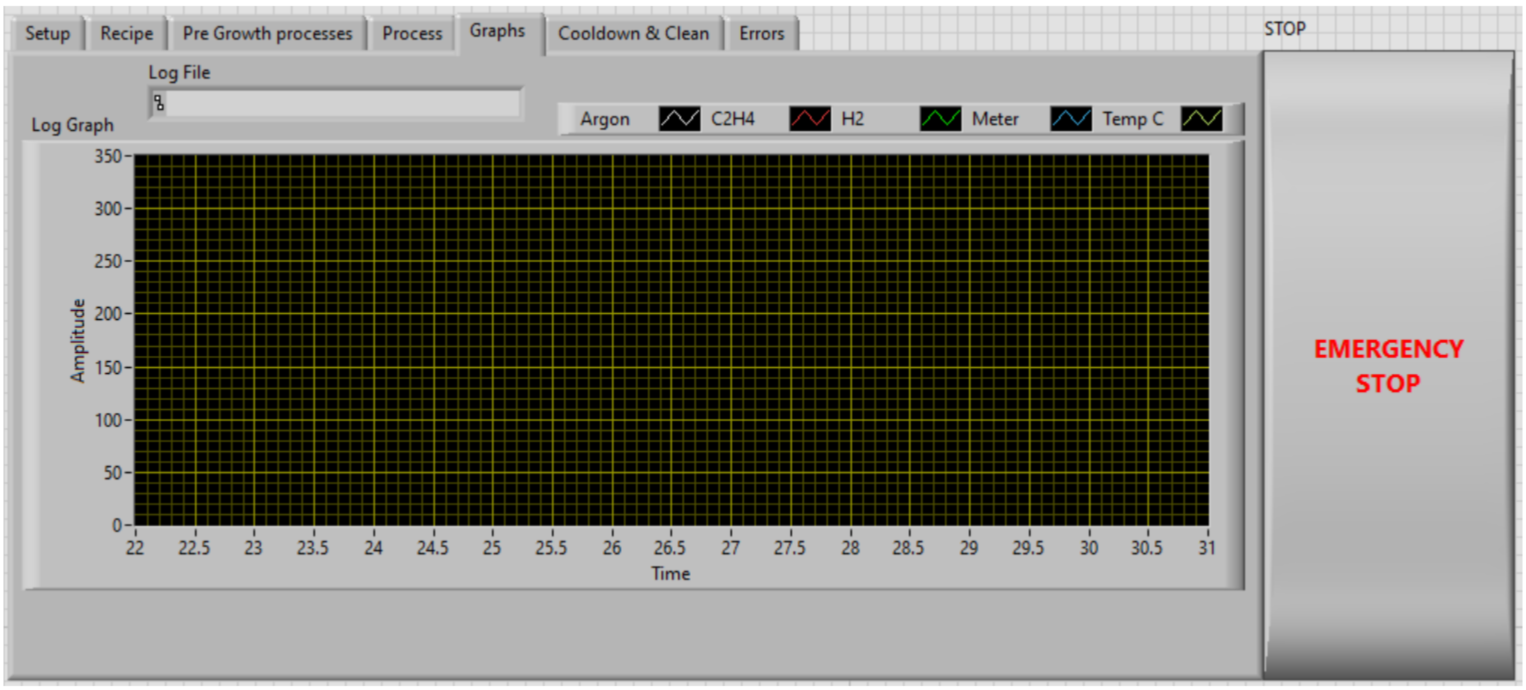

Figure A.11 User interface shows all the current values of gas flow and Temperature. Additionally shows the name of the log file.

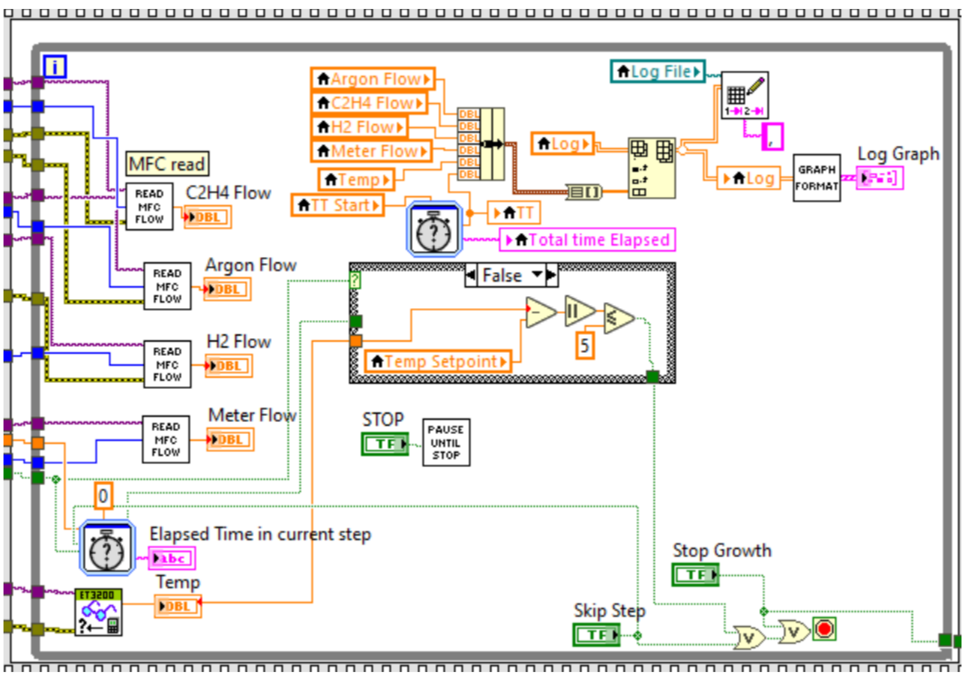

Figure A.12 Code Goes through a while loop running the step. Reads each MFC flow and outputs it to the process tab. Checks if the Temperature is reached or the step time has passed. This also logs the process and outputs a graph to the log tab. This goes to the next step when the current step is finished or the skip step button is pressed.

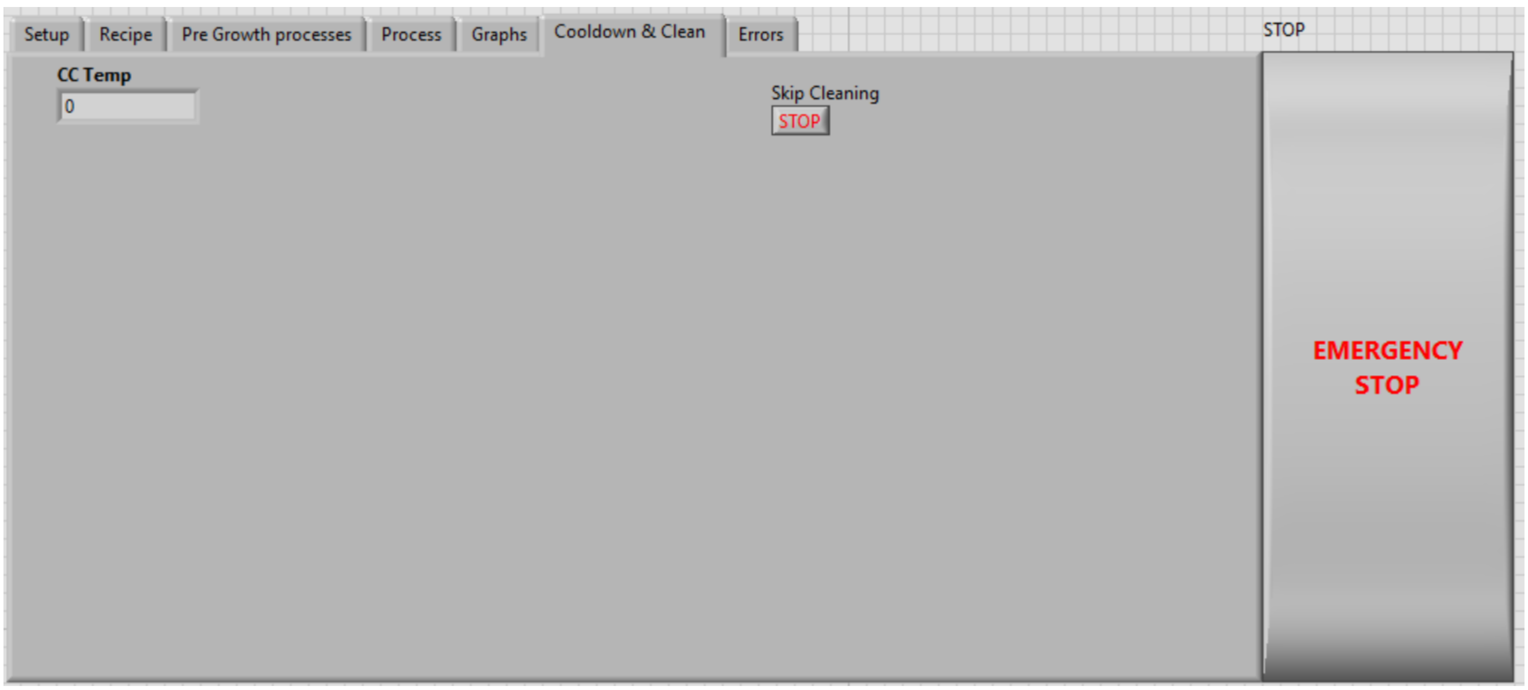

Figure A.13 Pannel for cleaning and cooldown. Shows the furnace temperature. Skip cleaning button will just send it to final step.

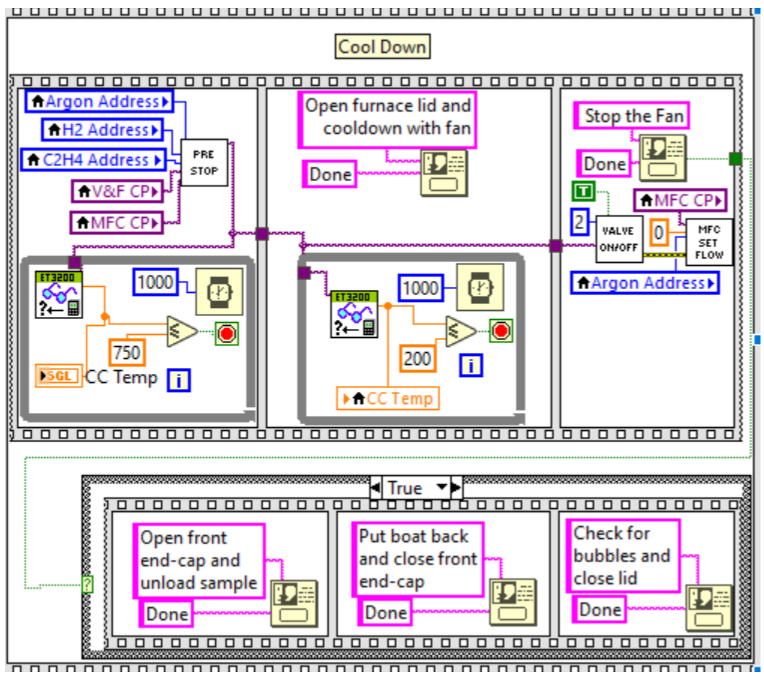

Figure A.14 Runs pre stop which sets the only flow to argon and the furnace temp to 0. reads furnace temp until it is below 750.Then prompts user to open lid and waits for furnace temp to get below 200. Then changes 3-way valve to air and sets argon flow to 0. Then guides the user through removing their sample.

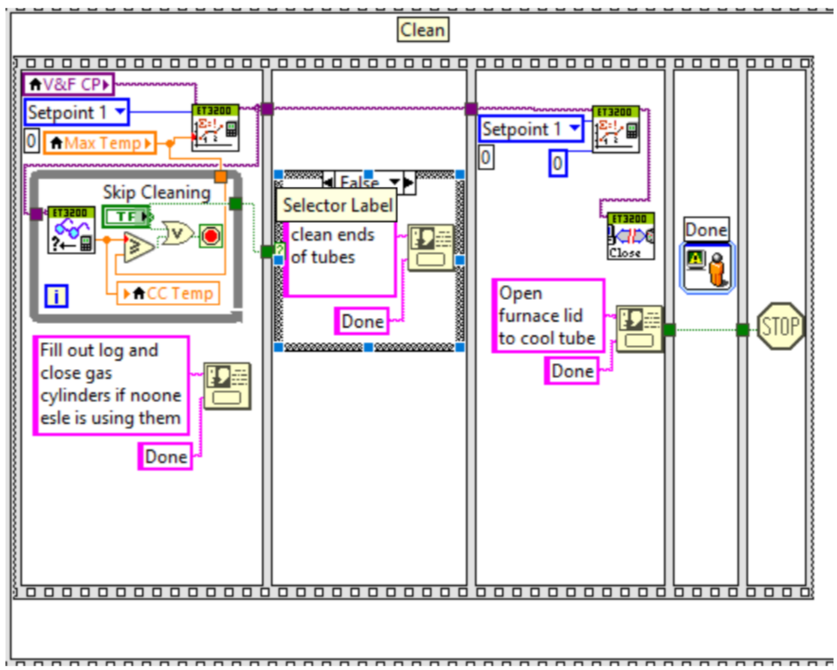

Figure A.15 Sets the furnace Temperature to max temp, the highest temperature run in the recipe process. It then runs a while loop reads the temperature and then moves on once it is at the max temp or the user inputs to skip cleaning. While it is heating up it has the user fill out the log and close the gas cylinders. It prompts the user to move the tube if they don't skip the cleaning process. Sets the temperature to 0 and closes communication with the furnace. It tells the user to open the furnace and then sends a message that it is done and stops the program.

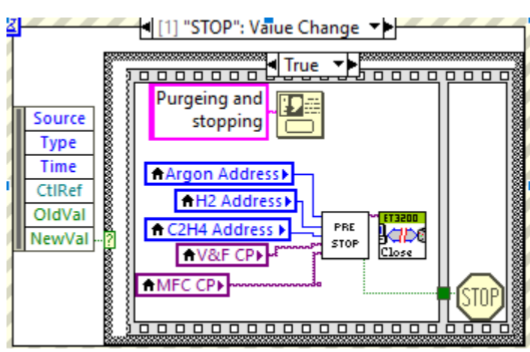

Figure A.16 This is the emergency stop process which turns off all gas other than argon and turns the furnace temp to 0. Then stops the process.

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