Modeling and Control of a Silicon Substrate Heater for Carbon Nanotube Growth Experiments

by

David Held B.S. recommended by the Department of Mechanical Engineering Massachusetts Institute of Technology, 2005

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MODELING AND CONTROL OF A SILICON SUBSTRATE HEATER FOR CARBON NANOTUBE GROWTH EXPERIMENTS

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DAVID HELD

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ABSTRACT

The precision engineering research group at MIT is working on carbon nanotube growth experiments on silicon substrates and in microfabricated silicon devices, to try to produce improved bulk nanotube growth. For this thesis, a heating control system was designed and implemented for eventual use in CNT growth experiments. The computer program that controls the heater is user-adaptable, so that an experimenter can easily change the desired temperatures at various points of the process. Later, this heating system will become part of a much larger system that also incorporates a controlled flow rate. The goal of the system is to achieve high-bandwidth control of reaction conditions.

In the heating control system designed, a computer controls a power supply attached to a wire-wrapped silicon chip, which is used to heat up the system, and the temperature is measured by a thermocouple. The control algorithm uses proportional gain, and the output is a PWM voltage. For accurate control of the system, a goal was set out to achieve an error of within 10%. For gains above 5, the computer can accurately control the temperature to less than 5.5% of the desired values in steady state, and an error of 0.75% was achieved with a gain of 50. Thus the system meets the desired specification of error. Also, while the error drops dramatically with increasing gain, the overshoot increases much more slowly, making a higher gain desirable.

Also, the system still has only reached temperatures of 650 degrees Celsius, although temperatures of 1000 degrees Celsius are required for nanotube growth. In order to achieve this, further tests will be performed with thicker wire and more voltage. Also, contact resistances within the chromel decrease with increasing temperatures, which reduce the percentage of power dissipated in the chromel compared to the lead wires. If the system is modified to eliminate this effect, by wrapping the wire differently or by using doped silicon, higher temperatures can be achieved. This will also make the system more predictable, leading to a better model and better control. Finally, to improve overall performance, one can experiment with changes to the switching time, using a PI or PID controller, and active cooling.

Thesis Supervisor: Alexander H Slocum

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1. Introduction

Anastasios John Hart, in the precision engineering research group is working on carbon nanotube growth experiments on silicon substrates and in microfabricated silicon devices, to try to improve bulk nanotube growth. One aspect of his work that is not yet well known is how the reaction conditions, such as temperature and flow rate, affect the nanotube growth reaction. The goal of this thesis is to design a control system for a heater for use in CNT growth experiments. The use of this heater will be to support A.J. Hart's research experiments.

This heating system uses resistive heating of a wire-wrapped silicon chip. It is hypothesized that this method will result in much faster heating than with a conventional tube furnace. The heating is created by a PWM voltage applied across a chromel wire. Various system parameters can be varied to improve the system, such as the system setup, the input voltage, and the PWM switching frequency. The control algorithm and the control gains can also be changed. In this thesis, many of these issues are explored.

Although only a heating system was constructed, it will later become part of a much larger system that also incorporates a controlled flow rate. By running many reactions with different temperatures and flow rates, the resulting nanotubes can be compared and a better understand can be achieved of how the reaction conditions affect the nanotube growth reaction.

2. Background

Carbon nanotubes consist of molecular cylinders of graphite. Figure 1 schematically shows a carbon nanotube, which exhibit certain useful properties. For instance, theory suggests that nanotubes can be conducting or semiconducting depending on their structure.

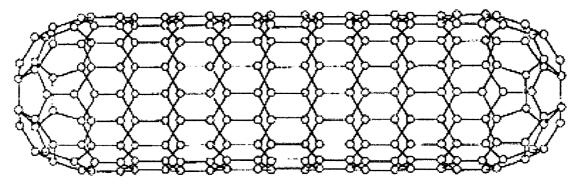


Figure 1: A carbon nanotube capped by one half of a C_60 molecule.

Nanotubes exhibit amazing stiffness, strength, and resilience. Individual carbon nanotubes have been shown to have a strength-to-weight ratio of 10-500 times that of steel¹. Finally, carbon nanotubes have outstanding thermal properties, with a thermal conductivity of 2000-6000 W/m-K.¹

Typically, current methods for growing nanotubes in bulk have created tangled tubes, which limit their commercial possibilities. Attempts have been made to solve this

problem by untangling the nanotubes. However, the result is often still not very well packed or aligned. Additionally, high quality bulk carbon nanotubes can cost from \$10-1000 per gram, which is prohibitively expensive. Other attempts have been made to grow nanotubes from floating catalysts, which can self-organize into loosely-knit fibers; however, the nanotubes resulting from this experiment still lack the properties of individual nanotubes. The temperature control system described in this thesis will help to conduct experiments to grow nanotubes under a variety of conditions, in order to increase the understanding of nanotube growth, leading to better bulk nanotube production.

3. Overall System Experimental Setup

This proposed overall nanotube growth system can be seen schematically in figure 2. This thesis focuses on the temperature control portion of this system.

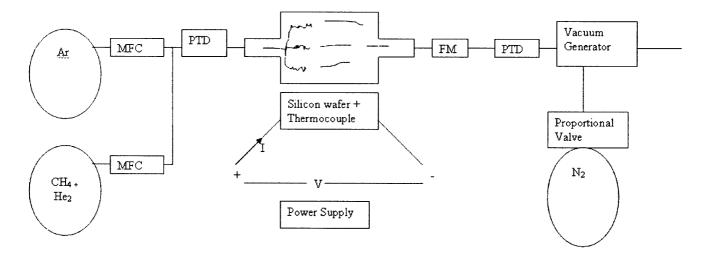


Figure 2: Physical CNT growth system layout

In the center is a furnace with a substrate and a catalyst, from which the nanotubes will be grown. The proposed method for heating and controlling this furnace involves resistive heating of a silicon substrate. The heating system will be described in more detail below. Currently, as designed by A.J. Hart as part of his PhD research at MIT, the input of the tube is a tank of argon, and a tank of methane and hydrogen, and the output is connected to a tank of nitrogen and a venturi. Argon and the methane-hydrogen tanks will each have a mass flow controller to control the flow rate out of these tanks. Pressure transducers will be used to measure the pressure drop across the tube, and a flow meter is used to measure the volumetric flow out of the tube. These sensors will be used both to control the reaction, and to detect the possibility of any leaks. If the flow into the chamber is greater than the flow out, most likely this is indicative of a leak in the chamber, which then needs to be repaired. Finally, a proportional valve will be connected to the nitrogen tank at the output of the tube.

Overall, this system involves six sensors: a thermocouple, two mass flow controllers, a flow meter, and two pressure transducers. The measurements from these sensors can be input into a computer. The computer will compare the actual values with

the desired temperature and flow rates, to control four different actuators. These actuators are the two mass flow controllers at the tank input, the proportional valve at the tank output, and a power supply connected to the resistive substrate. Fans may also be added for active cooling of the substrate. A block diagram of this system is shown in figure 3.

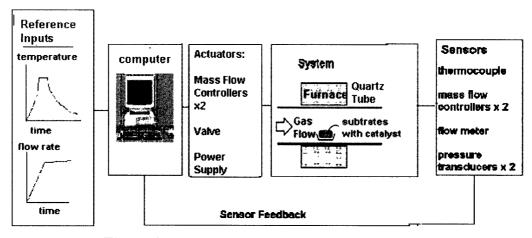


Figure 3: Block diagram of the CNT growth system

4. Heating System Experimental Setup:

In order to test the heating device before using it in the actual carbon nanotube growth system, experiments are performed in a quartz tube 770 mm long and 42 mm in diameter. A tank of nitrogen is connected to this tube, which is used to eliminate oxidation effects. Nitrogen flows through the tube at a rate of 5 cubic feet per hour while trying to flush out the air, and at 1 cubic foot per hour during normal operation. Given the tube dimensions and the initial flow rate, the nitrogen must flow at 5 cubic feet per minute for 28 seconds to flush out the tube. Given a safety factor of 10, in practice it is run for 4.5 minutes. The quartz tube setup is shown in figure 4.



Figure 4: Quartz tube for testing of resistive heating

The method of heating that is being tested is resistive heating of a silicon substrate. To achieve this, a piece of chromel wire is wrapped around a silicon substrate. The chromel has a voltage applied across it, which heats up the wire, and thereby heats the silicon substrate. The apparatus is shown in figure 5.



Figure 5: chromel wire wrapped around a silicon substrate, with a thermocouple

The electronic hardware for the heating system is shown in figure 6.

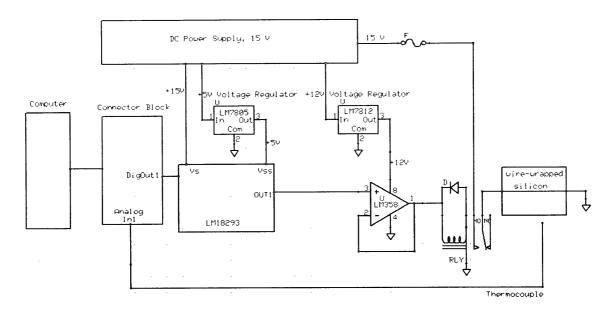


Figure 6: Electronics hardware for the heating system

A computer is used to process the data, and it sends and receives sensory information through the connector block. The connector block sends a digital output signal to the LM18293 push-pull driver. The digital output signal ranges from 0-5 V, but 10 V is needed in order to close the relay. The push-pull driver then converts the 0-5 V signal to a 0-15 V signal. This is buffered by an op-amp, and sent to the inductor of the relay. When a "high" is sent, the relay closes, sending 15 V through the wire-wrapped silicon. When a "low" is sent, the relay is open, sending 0 V through the wire-wrapped silicon. Thus by using a PWM scheme, one can create an approximately variable voltage from 0 to 15 volts. A thermocouple is then used to measure the temperature of the silicon, which is sent back to the computer through the connector block.

Safety precautions were taken to ensure that no expensive equipment was damaged. First, a shielded connector block is used, with resistors and other circuitry to attempt to avoid being damaged. Next, an op-amp is used to buffer the output of the LM18293. Finally, a 10 A fuse is inserted by the DC power supply to make sure that no more than 10 A is sent through the relay, which has a limit of 12 A. Also, in order to limit the number of power supplies needed, two voltage regulators were used. A 5V regulator is used to provide power to the LM18293 chip, and a 12 V regulator is used for the op-amp.

5. Control System

The block diagram for the overall control system is shown in figure 7.

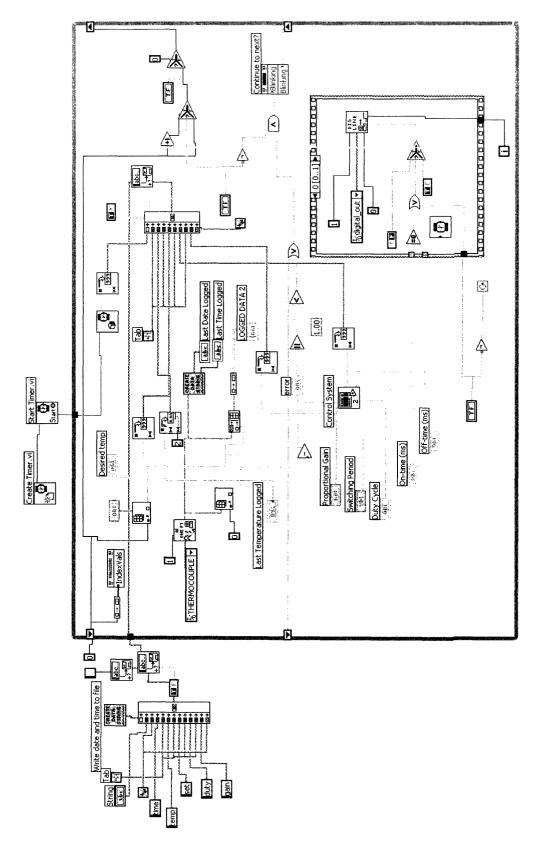


Figure 7: Block diagram of the overall control system

When the user first runs the program, a file is opened for recording the data. A header records the date and time, a description of the experiment from the user, and the types of data being recorded. The first temperature setpoint is chosen from an array, and the current temperature is read from the thermocouple. The error is calculated, which is used to determine the duty cycle of the waveform, using the control system in figure 8. The program enters a loop, in which it sets the output digital waveform to be high for a certain amount of time, and then low for a certain amount of time, depending on the duty cycle and the switching period. Also, if the temperature is within one degree of the setpoint, a button starts blinking, inviting the user to choose the next setpoint in the array. Finally, a file is written with data about the time, current temperature, desired temperature, duty cycle, and proportional gain.

The actual control algorithm is compressed into one block in figure 7 called "Control System." This sub-system is expanded and shown in figure 8.

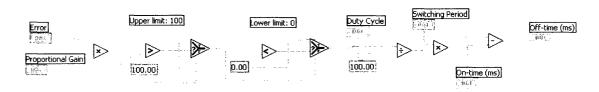


Figure 8: Block diagram of the control algorithm

The basic algorithm is proportional control: the error is multiplied by a proportional gain. The output is limited to be no more than 100 or less than 0, since a duty cycle can only range from 0-100%. The fractional duty cycle is then multiplied by the switching period to determine the on-time of the waveform, and this time is subtracted from the switching period to find the off-time.

The on-time and off-time of the waveform are handled in separate case structures. Figure 9 shows the case structure for the on-time, and figure 10 shows the case structure for the off-time.

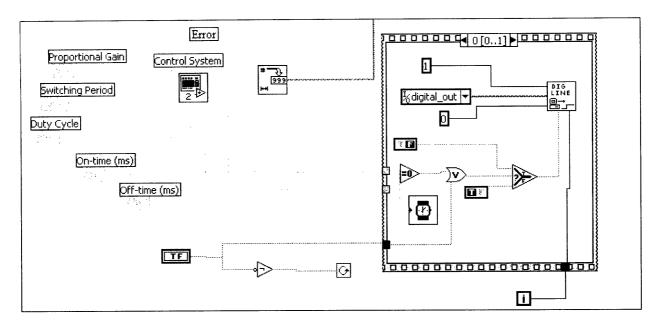


Figure 9: On-time case for the block diagram

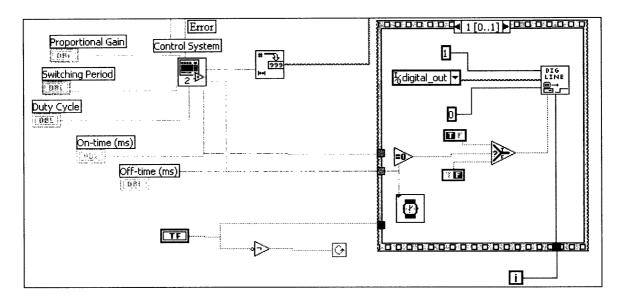


Figure 10: Off-time case for the block diagram

Figure 11 shows the front-panel, with which the user interacts. On the top left, the user can write a description of the experiment on the first line, which appears in the file as a header line. Next, the user can see a graph of the temperature over time. The current temperature appears in blue, and the desired temperatures appear as a thin green line. Below the graph, the user can see information about the last data point that was written to a file.

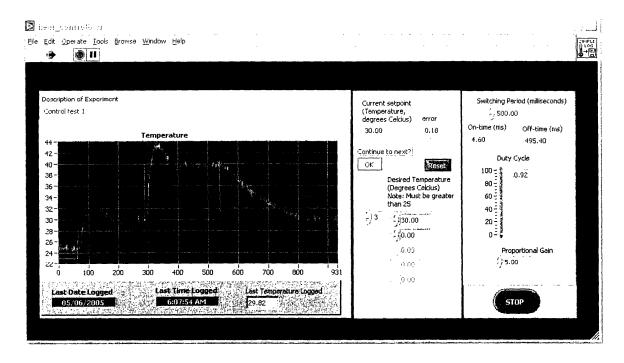


Figure 11: Front Panel, where the user interacts with the system

In the second panel, one can read off the current temperature setpoint and the error. The user can input an array of setpoints, of any duration. At any time, the user can press the yellow button to advance to the next setpoint, or press the red reset button to reset to the first setpoint. After the temperature first comes within one degree of the setpoint, the yellow button flashes, prompting the user to continue to the next setpoint. The button keeps flashing even if the temperature exceeds one degree of the setpoint, such as during overshoot, as seen in the above picture.

In the third panel, the user can set the switching period and the proportional gain. He can observe the on-time and off-time of the digital waveform. He can also observe the duty cycle, both numerically and graphically. Finally, if the user wants to end the experiment, he can press the stop-button in the bottom right.

6. Modeling of Physical System:

The heating system is achieved by applying a voltage across a piece of chromel wire. The chromel wire acts as a resistor and dissipates heat. The wire is also wrapped around a piece of silicon, which conducts heat from the wire and thus acts as the heating element. The silicon and wire is modeled as a lumped thermal mass. Thus the system can be modeled by the equation

$$mc\frac{dT}{dt} = heating - convection - radiation$$
. (1)

where m is the effective mass and c is the effective specific heat. In this experiment, the dimensions of the silicon wafer are 31 mm x 20 mm x 0.75 mm, and the density is about 2330 kg/m³. The specific heat capacity of silicon is 700 J/kg K, so the specific heat of the silicon wafer is 0.76 J/K. The chromel wire is wrapped around the silicon 56 times. Each winding has a length given by length = $2W + \pi(t+D_{wire})$, where W is the width of the silicon of 20 mm, t is the thickness of the silicon of 0.75 mm, and D_{wire} is the diameter of the wire, or $10^{\circ}-4$ m. Thus the total length of the wire must be 2.39 meters. The area of the wire is $7.85*10^{\circ}-9$ m², and thus the total volume of the chromel wire is $1.88*10^{\circ}-8$ m³. The density of chromel is about 8900 kg/m³. The specific heat capacity of chromel is 448 J/kg C, so the specific heat of chromel is 0.075 J/K. The total specific heat of the entire system of the chromel wire and the silicon wafer is then 0.835 J/K.

The convection loss is given by

$$convection = h \cdot A \cdot (T - T_{inf}), \qquad (2)$$

where h is the heat transfer coefficient, A is the face area, T is the surface temperature, and T_{inf} is the temperature of the surroundings. The heat transfer coefficient is calculated for forced convection of air, because the experiments were performed inside a tube with nitrogen gas flowing at $7.87*10^{\circ}-6$ cubic meters per second. The tube area is $1.39*10^{\circ}-3$ square meters, giving a speed of $5.67*10^{\circ}-3$ m/s. The convection coefficient can be calculated by the equation $h = k*Nu_L/L$. In this equation, k is the fluid's thermal conductivity of 0.025 W / m K, and L is the length of the plate, or 31 mm. Nu_L is the Nusselt number and is given by

$$Nu_L = 0.664 \cdot (Pr)^{1/3} \cdot \sqrt{Re_L} \tag{3}$$

where Pr is the Prandtl Number and Re_L is the Reynolds number². This equation is valid for Prandtl numbers above 0.5 and for Reynolds numbers less than 10^5. The Prandtl Number is given by $c_p\mu$ / k, where c_p is the fluid specific heat of 1005 J / kg K, μ is the fluid viscosity of 1.7 *10^-5 kg / m s, and k is the fluid conductivity of 0.025 W / m K. This gives a Prandtl number of 0.683. The Reynolds number for the plate is given by $\rho u_{\infty} L/\mu$, where ρ is the fluid density of 1.29 kg/m³, u_{∞} is the fluid velocity of 5.67*10^-3 m/s, L is the plate length of 31 mm, and μ is the fluid viscosity as before. This gives a Reynolds number of 13. Thus the Nusselt number is found to be 2.11. The heat transfer coefficient can then be found to be 1.70 W / m² K. The face area can be found to be 12.4 * 10^-4 square meters for both sides, and the nitrogen temperature is assumed to be 25 degrees Celsius.

The radiation loss is given by

$$radiation = \varepsilon \cdot A \cdot \sigma \cdot (T^4 - T_{\text{inf}}^4), \tag{4}$$

where ϵ is the total hemispherical emissivity of chromel, A is the face area, σ is the steffan-boltzmann constant of 5.67 * 10^-8 W / m^2 K^4, T is the surface temperature, and T_{inf} is the temperature of the surroundings. The emissivity of chromel could not be found; however, chromel is made of 90% nickel, so the emissivity of nickel is used, which is 0.37. The surface area is given by half of the surface area of the wire, since only half of the wire is visible from the outside. The surface area of the wire is 7.51*10^-4 m^2, so the outer surface area when wrapped around a piece of silicon is 3.75*10^-4 m^2. This gives a coefficient of 7.87*10^-12. The silicon protrudes slightly from the chromel wire, with a protruding area of 1.2*10^-4 m^2. The emissivity of the silicon cannot be found, because it depends on many factors that are unknown³. However, we know that the value must be between 0 and 1, so if we arbitrarily choose a value of 0.5, then the silicon contributes to the radiation by 3.40*10^-12, giving a total radiation coefficient of 1.13*10^-11. The radiation coefficient can change from this value by 30% depending on the emissivity of silicon, so in the future this value should be researched further.

The last element to this model is heating. Heating occurs because the chromel wire acts as a resistor, which dissipates heat. The heat lost is equal to V^2/R , where V is the voltage and R is the resistance. The voltage is a variable that can be adjusted. The resistance depends on the shape of the wire, and is equal to $R = p*L_W/A$, where p is the resistivity, L_W is the length of the wire and A is the area of the wire. The resistivity of the wire varies with temperature, and is equal to $p = p_{ref}*(1+a*(T-T_{ref}))$, where p_{ref} is the resistivity at a reference temperature T_{ref} , T is the current temperature, and a is the temperature coefficient of resistance. At 25 degrees Celsius, chromel has a resistivity of $70.6*10^{\circ}-8$ ohm-meters, and a temperature coefficient of resistance of $4*10^{\circ}-4$ ohms per degree Celsius.

The above equations can be combined to form the following equation:

$$0.835 \frac{dT}{dt} = \frac{V^2}{270[1 + 0.0004(T - 298K)]} - 2.11 \cdot 10^{-3} (T - 298K) - 1.13 \cdot 10^{-11} (T^4 - 298K^4)$$
(5).

This equation can also be written as:

$$\frac{dT}{dt} = \frac{V^2}{225[1 + 0.0004(T - 298K)]} - 2.52 \cdot 10^{-3}(T - 298K) - 1.35 \cdot 10^{-11}(T^4 - 298K^4).$$
(6)

7. Model Verification

Attempts to model the resistance of the chromel have not been successful. Figure 12 shows the results from four experiments. In each experiment, the current and voltage are recorded at various points, from which the resistance is calculated, using R = V/I. The temperature is also recorded at each of these points, so the resistance can be seen as a function of temperature..

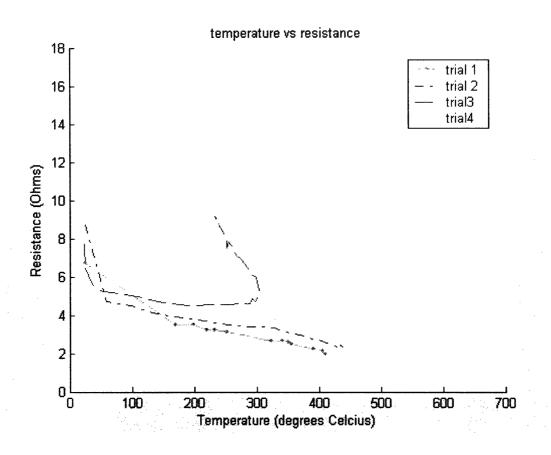


Figure 12: Resistance as a function of temperature

Also, figure 13 shows two experiments in which a 9 V step is applied, and the temperature is recorded.

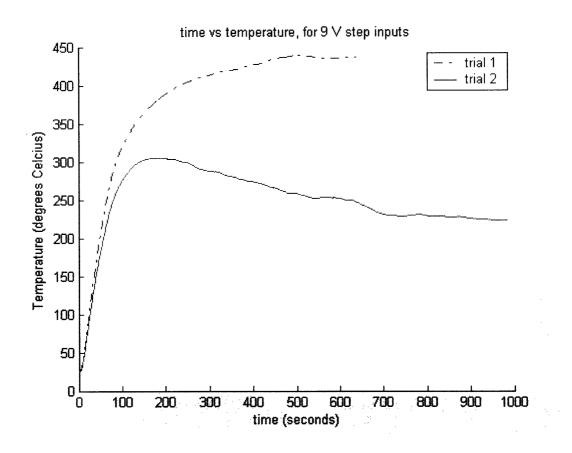


Figure 13: temperature as a function of time, for a 9 V step input

Both of these graphs indicate the lack of repeatability of the experiments. In the above figures, trial 3 in figure 12 corresponds with trial 2 of figure 13. In trial 3 of figure 12, the resistance decreases until it reaches about 4.5 ohms, and then it starts to increase. Because the resistance is increasing, the temperature also drops, as shown in figure 13. This is most likely due to changes in the contact resistance. The chromel wire is attached to wires from the power supply with two alligator clips. Further, the chromel wire is wrapped very tightly and is making contact with itself at many points. As the temperature increases, the contact resistances change in unpredictable ways, causing the unrepeatability observed. However, for a few trials the system does appear to be repeatable. On figure 12, for the first 2 trials and half of the third, the graph appears to be approximately following the same curve. Halfway through trial 3, the graph switches to a different curve, and the same curve is followed on trial 4.

Unfortunately, not enough time was available to change the system after these realizations were made. Instead, we focus on simply modeling the cooling of the given system. Figure 14 shows the results of three cooling experiments. The trails 1 and 2 involved cooling from 420 degrees Celsius, whereas trial 3 involved cooling from 591.5 degrees Celsius.

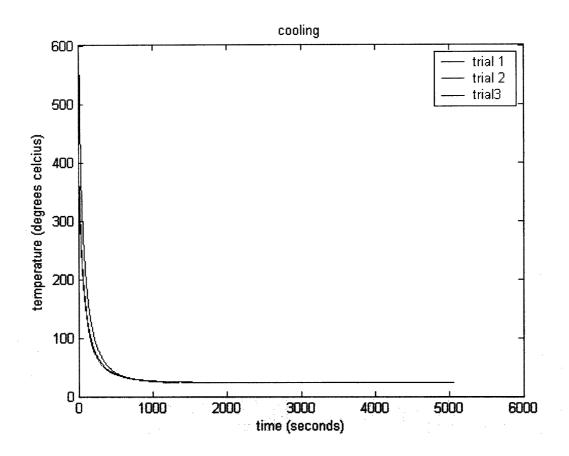


Figure 14: Three cooling experiments, very repeatable

Clearly, these results are much more repeatable than the heating data. Next, a least squares method was used to fit a cooling function to the data from each of these trials. For trial 1, the resulting best-fit function was found to be:

$$\frac{dT}{dt} = -1.035 \cdot 10^{-3} (T - 297.263K) - 3.5810 \cdot 10^{-11} (T^4 - 297.263K^4)$$
 (7)

The fit of this data is shown in figure 15.

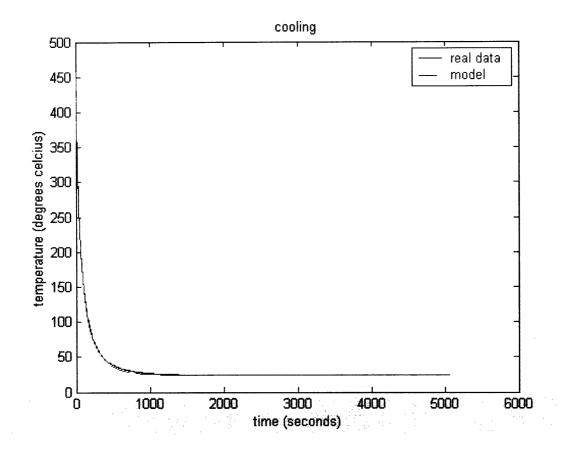


Figure 15: First cooling experiment, and model

For trial 2, the resulting best-fit function was found to be:

$$\frac{dT}{dt} = -8.49 \cdot 10^{-4} (T - 297.563K) - 3.9124 \cdot 10^{-11} (T^4 - 297.563K^4)$$
 (8)

For trial 3, the resulting best-fit function was found to be:

$$\frac{dT}{dt} = -4.870 \cdot 10^{-3} (T - 298.158K) - 1.4932 \cdot 10^{-11} (T^4 - 298.158K^4) \tag{9}$$

The fit of these curves is also very accurate.

Finally, a graph of steady state temperature as a function of power is shown in figure 16. The highest steady state temperature reached was 600 degrees Celsius, and about 140 watts were needed.

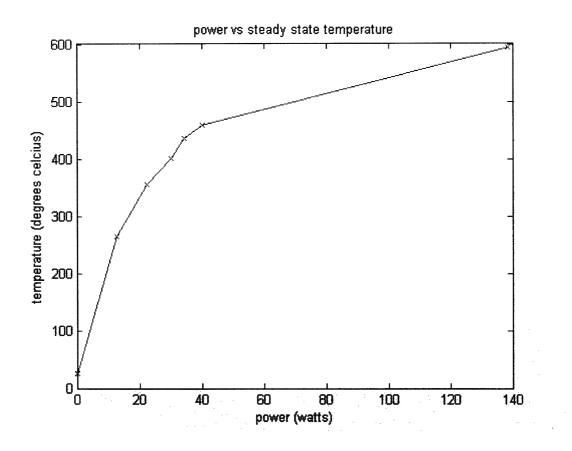


Figure 16: Temperature as a function of power

8. Testing with the Control System:

Figure 17 shows the results of attempting to control the system with a gain of 1. It can be clearly seen that there is no overshoot, but there are large steady-state errors, of about 36.5%.

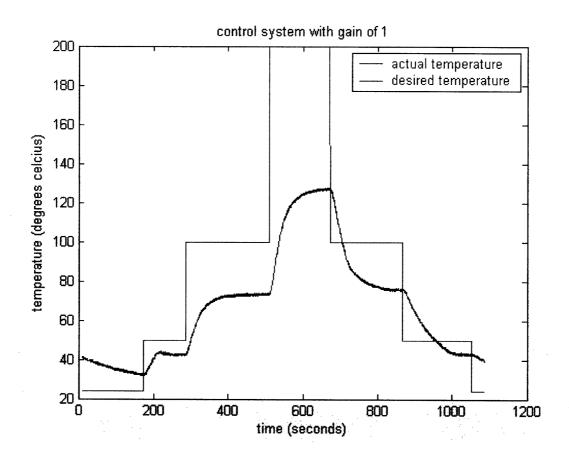


Figure 17: Control system with a gain of 1

Next, a control system with a gain of 5 is used, as shown in figure 18. There is an overshoot of about 8%, but steady state errors appear to be very small.

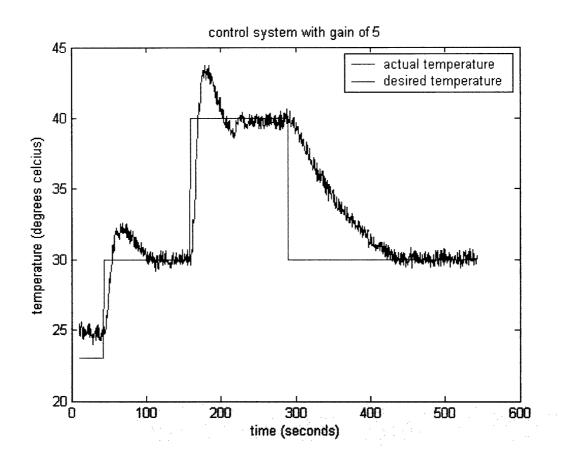


Figure 18: Control System with a gain of 5, small temperatures

The same control system is then used with higher temperatures, as shown in figure 19. At these larger temperatures, the steady-state error becomes apparent, and appears to be about 5.5%.

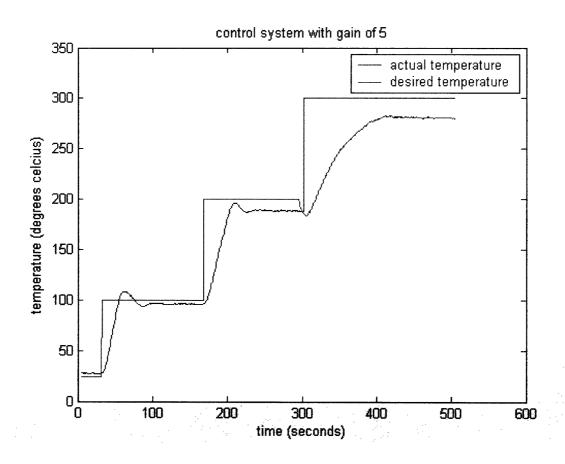


Figure 19: Control system with a gain of 5, larger temperatures

Next, a gain of 15 is used, as shown in figure 20. The overshoot has increased to 9.6% for 100 degrees Celsius, but the steady-state error has decreased to about 2.3%.

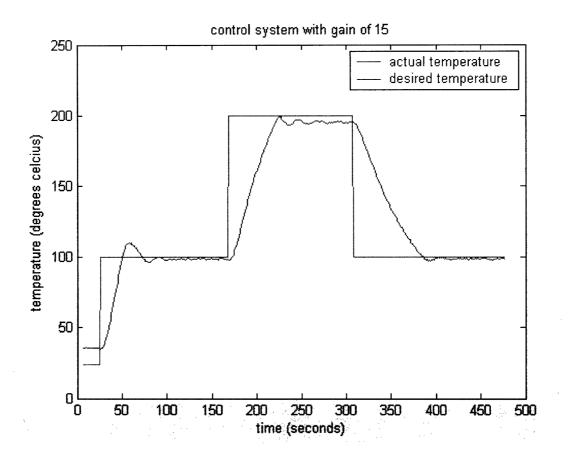


Figure 20: Control system with a gain of 15

Finally, a gain of 50 is used, as shown in figure 21. The overshoot is now about 10% for 100 degrees Celsius, but the steady state error is only 0.75%.

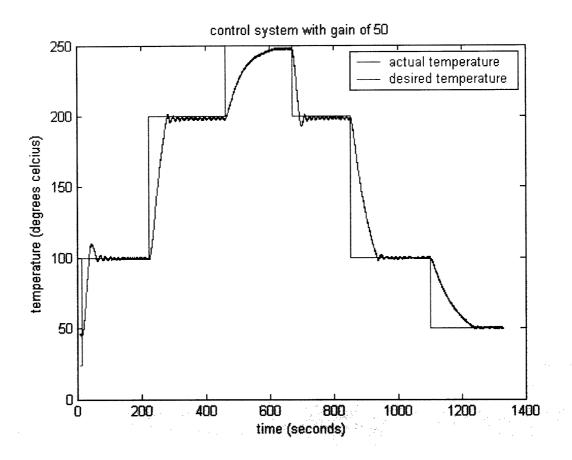


Figure 21: Control system with a gain of 50

9. Results:

First, the system was modeled. Although heating of the system could not be modeled accurately, cooling could be modeled very accurately. A model was used of the form:

$$\frac{dT}{dt} = A(T - B) + C(T^4 - B^4) \tag{10}$$

The following chart shows the attempts to determine the parameters A, B, and C

	A	В	C	% Difference from Model
Model	-2.52*10^-3	298	-1.35*10^-11	
Experiment 1	-1.04*10^-3	297.263	-3.58*10^-11	59%, 0.25%, 165%
Experiment 2	-8.49*10^-4	297.563	-3.91*10^-11	66%, 0.15%, 1.90%
Experiment3	-4.87*10^-3	298.158	-1.49*10^-11	93%, 0.053%, 10%

Next, the control system was implemented and tested. The following are the approximate results:

Gain	Overshoot at 100 degrees C	Steady State Error
1	0	36.5%
5	8%	5.5%
15	9.6%	2.3%
50	10%	0.75%

These results can be plotted, as seen in figure 22.

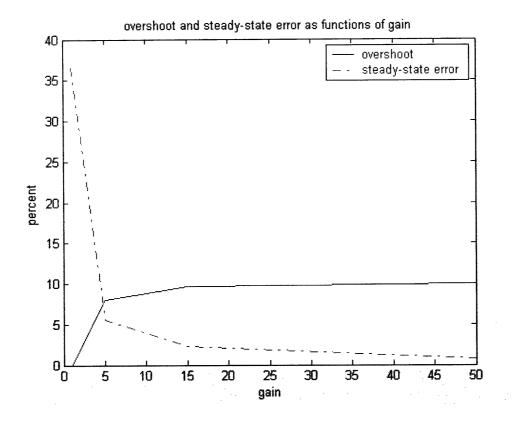


Figure 22: Overshoot and steady-state error as functions of gain

To make the steady-state error and overshoot equal, a gain of 4.74 can be used. This results in an overshoot and steady-state error both of 7.49%.

10. Conclusions and Recommendations:

In order to control a system accurately, it is important to have a good model for the system. A model enables one to understand the system and the factors that cause it to behave as it does. Although the cooling tests were only performed from 591.5 degrees Celsius, the model can be used to predict heat loss for 1000 degrees Celsius, which must be reached for the nanotube growth reaction. Also, a model can be used to better control the system. With a model, one can better calculate what system input should be applied based on the current error. Last, one can use the model in a feed-forward control scheme to better control the system.

Although a model for cooling has been reasonably determined, a model for heating has not. The contact resistances in the system produce a randomness that makes the system unpredictable. A new, similar system must be constructed which does not have these contact resistance issues.

The most important change that must be made is to rewrap the chromel wire in a way so that it does not touch itself. This may prove to be difficult to do by hand; if it is, one can try to construct a piece of silicon with notches for the chromel wire to fit into. Alternatively, one can used doped silicon without a chromel wire. The doped silicon would be conducting, so the chromel wire would not be needed.

Additionally, active cooling can be implemented to make the system cool faster. Currently, the system cools by convection and radiation, which is relatively slow. For example, with a 15 volt input, the system can be heated from 100 to 200 degrees Celsius in 57 seconds, whereas cooling takes 84 seconds. If variable-speed fans are used and controlled, cooling can occur at a much faster rate.

Also, a tradeoff must be made between overshoot and steady-state error. All the values for gain above 5 have a steady state error less than 10%, which is what was required for the system. A gain of 4.74 would make the overshoot and the steady state error equal, at 7.49%. However, as the gain changes from 5 to 50, the overshoot increases by a factor of 1.25, whereas the steady-state error decreases by a factor of 7.3. Because the high gain has a much greater positive effect on the steady-state error than a negative effect on the overshoot, a gain higher than 4.74 should be used. To improve overall system performance, one can also experiment with varying the switching time (the period of the PWM waveform). Additionally, to reduce the steady-state error and improve performance, a PI or PID controller can be used.

Finally, the system still has only reached temperatures of 650 degrees Celsius, although temperatures of 1000 degrees Celsius are required for nanotube growth. This might be achieved with the current setup but with thicker wire and more voltage. Also, the current decreases in resistance with higher temperatures cause a higher percentage of the power to be dissipated in the lead wires than in the chromel around the silicon. By reducing this decrease in contact resistance as described above, more power will be dissipated in the wire wrapped around the silicon, resulting in higher silicon temperatures. Further experiments will be performed, implementing these changes to try to achieve these higher temperatures.

11. References:

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