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Thermal management of micro gas chromatographs

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THERMAL MANAGEMENT OF MICRO GAS CHROMATOGRAPHS

A Thesis

Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering

in

The Department of Mechanical Engineering

By
Chetan Ramesh
B.E., Visveswaraiah Technological University, India, 2003
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To my parents:

Sathyavathi Ramesh

and

N. Ramesh
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ABSTRACT

Gas chromatography is an analytical technique for separating and analyzing individual components in a gas mixture. Applications of gas chromatographs (GCs) include chemical analysis, environmental maintenance, greenhouse gases monitoring, and forensic analysis when used in conjunction with mass spectrometry. There has been a tremendous interest in miniaturization of gas chromatograph systems because of significantly reduced size, portability, fast response times, low power consumption and low cost.

Isothermal analysis is a chromatographic analysis at one column temperature. In temperature programming, a linear increase of column temperature with time is used during the course of the analysis. Temperature programming facilitates separation of a wider range of components, when compared to isothermal analysis, in less time. Though a few miniaturized gas chromatograph systems with temperature programming capability have been reported, all reported findings until now demonstrate very slow heating ramp rates (~5°C/sec). To enable faster analysis and to analyze a wider range of chemicals, higher heating rates (of the order of ~40°C/sec) have yet to be achieved.

A heater was designed to implement different temperature programming cycles for a LiGA fabricated nickel micro GC. The thermal behavior of the device was modeled using an energy-based approach to determine the thermal power requirements. Thermal stress analysis was carried out to determine the thermal stresses in the system. The heaters were fabricated by electrodepositing nickel and copper on a polyimide insulating layer on top of the nickel micro GC column. Heating ramp rates from 5°C/sec to 50°C/sec were obtained with the aid of a PID controller. The heaters were also able to maintain the column at different elevated temperatures,
which showed that the heaters could be used to achieve a variety of temperature programming profiles and they offered flexibility in selecting different ramp rates and soak temperatures.
CHAPTER 1. INTRODUCTION

1.1 Micro Gas Chromatography

Gas chromatography is an analytical technique for separating and analyzing individual components in a gas mixture. The central item in the apparatus for gas chromatography is the chromatographic column. The inside walls of the column are coated with an adsorbent called the stationary phase. Sample components are transported through the columns by a mobile phase, usually an inert gas like hydrogen, and are separated based on their boiling points and their relative affinities to the stationary phase. A detector is used at the end of the column to identify the separate components of the mixture as they emerge one by one.

Applications of gas chromatographs (GCs) include chemical analysis, environmental maintenance, greenhouse gas monitoring, forensic analysis when used in conjunction with mass spectrometry, and military applications. Typically GCs are bulky, because they consist of a carrier gas supply, a sample injection system, a separation column, a temperature programmable oven, an output detector and a data processing unit. Although commercial GCs like the HP 6890N GC\(^1\), microFAST GC\(^2\) and Varian CP-4900 Micro-GC\(^3\) have reduced in size considerably over the years, there is scope for further miniaturization, leading to the ultimate goal of a handheld GC.

The miniaturization of analytical instrumentation is receiving considerable attention as a means of reducing instrumentation and analysis costs and for the development of novel applications, which benefit from small instrument size [Lambertus, 2004]. Miniaturized GCs offer capabilities like portability, fast response times, low power consumption and low cost. Since the original idea of fabricating an integrated GC on a silicon wafer was demonstrated at Stanford [Terry, 1979], researchers at Sandia National Laboratories [Hudson, 1998], the Georgia

\(^1\)http://www.chem.agilent.com, \(^2\)http://www.microfastgc.com, \(^3\)http://www.varianinc.com
Institute of Technology [Noh, 2002], the University of Michigan [Aga, 2005] and other groups have worked on making miniaturized GC columns. At Louisiana State University, Bhushan fabricated 50 µm wide, 200-500 µm tall gas chromatographic columns using the LiGA (German acronym for X-ray lithography (X-ray Lithographie), Electroplating (Galvaniformung), and Molding (Abformung)) process [Bhushan, 2001].

1.2 Temperature Control of Micro GC columns

1.2.1 Isothermal Analysis vs. Temperature Programming

Isothermal analysis is a chromatographic analysis at one column temperature. In temperature programming, a linear increase of column temperature with time is used during the course of the analysis. Isothermal operation of a chromatographic column has a number of drawbacks, as illustrated in Figure 1.1 (a). An isothermal run at 150°C prevents the lighter components (<C8) from being totally separated and still takes over 90 minutes to elute the C15 paraffin. If the selected isothermal column temperature is too low, the early-eluting peaks will be closely spaced, while the more strongly retained components will be broad and low-lying (C14 and C15). These strongly retained components can be more quickly eluted by selecting a higher isothermal temperature, which will also improve their detectability. However, in doing so, there is more rapid coelution of components, peaks too closely spaced, and an overall loss of resolution result at the start of the chromatogram. This situation, which prevails in all practical versions of elution chromatography, is often called the “general elution problem” and it can be solved by temperature programming, which is the variation of the column temperature with time during an analysis.

As shown in Figure 1.1 (b), the separation can be significantly improved using programmed temperatures. In this figure, the temperature starts at 50°C less than the isothermal
temperature used in Figure 1.1 (a) and is ramped at 8 degrees per minute up to 250°C, a temperature higher than the isothermal temperature. The programmed run facilitated the separation of the low-boiling paraffins, easily resolving several peaks before the C₈ peak while increasing the number of paraffins detected. The C₁₅ peak eluted much faster, in about 21 minutes and it was not the last peak, because six more hydrocarbons were observed.

![Comparison of (a) isothermal and (b) temperature programmed separations of n-paraffins](McNair, 1998)

**Figure 1.1** Comparison of (a) isothermal and (b) temperature programmed separations of n-paraffins [McNair, 1998]
1.2.2 Current Approaches to Temperature Programming

Commercially available gas chromatographs are usually equipped with ovens to perform temperature programming. The Agilent HP 6890N GC (Agilent Technologies Inc., Palo Alto, CA) can achieve a maximum temperature ramp rate of 75°C/min. From a practical standpoint ovens are bulky, require large amounts of electrical power, and are not easily portable. A typical commercial GC with an oven occupies the space of a personal computer, which makes it impractical for use in the field.

Capillary columns coated with a conductive material, used for resistive heating, have been used for temperature programming. Jain et al. successfully applied a conductive coating to a capillary tube column and obtained a fast heating program with heating rates of up to 20°C/sec [Jain, 1995]. Another concept of resistive heating is to coil a metal heater wire around the capillary tube or run it parallel to it. Ehrmann et al. tested one such collinear at-column heater that enabled heating rates of 20°C/sec [Ehrmann, 1996]. They also tested a coaxial heater but found that it suffered from differences in expansion coefficients of the metal and fused silica, causing damage to the fused silica column.

Patterning of Ti/Pt thin film heaters on GC columns was reported recently but with limited success [Agah, 2005; Potkay, 2006]. They had a big drawback in that columns etched in silicon have to be sealed anodically using glass. Since glass has very low thermal conductivity, any heating system will require relatively large amounts of power to heat the column at any desired ramp rate.

1.2.3 Proposed Solution and Device Requirements

With the miniaturization of gas chromatographs, with the ultimate aim being the development of hand-held GCs, the emphasis is on developing in situ heaters for temperature
programming. The advantages of using LiGA fabricated nickel micro GC columns are discussed in Chapter 2. Heaters were designed to enable fast temperature programming on 0.5m LiGA nickel columns. The heaters were fabricated on top of the columns coated with cured polyimide as the insulating layer between the heater coil and the nickel column are expected to give a uniform temperature distribution over the column. Polyimide has an advantage for integrating the heaters and the columns because the effect of thermal expansion mismatches between the heater coil and the substrate are reduced since the CTEs of nickel and copper, the heating coil materials, are almost the same as that of polyimide. Polyimide also has exceptional thermal stability and can withstand very high temperatures (>450°C) over repeated cycles.

Temperature programming requires more sophisticated instrumentation than an isothermal GC. The ability to control the column temperature while keeping the injection port and detector at a constant temperature is important. An electronic PID controller was needed along with a high heat flux heater to carry out temperature programming of the GC.

1.3 Thesis Outline

The second chapter introduces the principles of gas chromatography, nomenclature, a history of gas chromatography and miniaturization of gas chromatographs. The mathematical models of the thermal system, power consumption calculations and lumped parameter analysis of the heater layout are presented in Chapter Three. Chapter Four details the process of heater design and 3-D thermal finite element analysis of the heater layout. The heater fabrication steps are explained in Chapter Five. Chapter Six features the results obtained following the testing of the heaters. Finally, Chapter Seven concludes the research and addresses recommendations for future work in this field.
CHAPTER 2. BACKGROUND

2.1 Chromatography

During the 19th century works of scientists like F.F. Runge, F. Goppelsroeder, C. Schönbein, and D. T. Day laid the foundation for early methods of chromatography [Grob, 2004]. In 1903, the Russian scientist Mikhail Semyonovich Tswett separated plant pigments by liquid-adsorption column chromatography. He used calcium carbonate as an adsorbent and petrol ether/ethanol mixtures as eluent to separate chlorophylls and carotenoids. Tswett is credited as being the “father of chromatography” because he coined the term chromatography (“Chroma” – color and “Graphein” – write) and scientifically described the process [McNair, 1998; Poole, 2003]. In later years scientists like R. Kuhn, A. Tiselius, J.N. Wilson A. J. P. Martin, R. L. M. Synge, E. Cremer, C. S. G. Phillips, and M. Golay further contributed to the advancement of the field of chromatography. A. J. P. Martin and R. L. M. Synge were awarded the Nobel Prize for Chemistry in 1952 for their invention of partition chromatography.

The “official” definition of chromatography given by the International Union of Pure and Applied Chemistry (IUPAC) is:

“Chromatography is a physical method of separation in which the components to be separated are distributed between two phases, one of which is stationary (stationary phase) while the other (the mobile phase) moves in a definite direction” [McNair, 1998].

The stationary phase can be a solid or a liquid and the mobile phase is either a gas or a liquid. The mixture to be separated is dissolved in a solvent mobile phase and passed over an adsorbent stationary phase. Since different compounds adsorb to the stationary phase to differing extents, they migrate through the phase at different rates and are separated as they emerge, elute,
from the end of the chromatographic column [McMurry, 1992]. Since different components of the sample mixture elute at different times, it results in separation and identification.

2.1.1 Gas Chromatography

Gas chromatography is a type of chromatography in which the mobile phase is a carrier gas, usually an inert gas such as helium or nitrogen, and the stationary phase is a layer of solid or liquid on an inert solid support, called a column. If the stationary phase is a solid, the gas chromatography technique is called gas-solid chromatography and if it is a liquid, gas-liquid chromatography.

Figure 2.1 shows the various parts of a typical gas chromatograph. The components, dissolved in a volatile solvent, are injected into the injector (A). In the injector the mixture vaporizes and is carried by the carrier gas (usually He or N\textsubscript{2}) that is supplied by the gas cylinder (E), via a regulator (F), to the column (B). This column is situated in a programmable oven that regulates the temperature. The separation occurs in the column and the separated components elute one by one into the detector (C).

![Figure 2.1 Generalized schematic of a GC system](http://www.iupac.org/didac/Didac%20Eng/Didac05/Content/ST18.htm)
2.1.2 Basic Apparatus for Gas Chromatography

2.1.2.1 Stationary and Mobile Phase

The sample is dissolved in the mobile phase, which may be a gas, a liquid or a supercritical fluid. The sample with the mobile phase is forced through the GC column, where it is adsorbed in the immobile, immiscible stationary phase. The stationary phase in gas-liquid chromatography is a liquid that is immobilized on the surface of the column wall by adsorption or by chemical bonding.

The phases are chosen such that components of the sample have differing solubilities in each phase. In contrast to most other types of chromatography, the mobile phase in gas chromatography does not interact with the molecules of the analytes. A component that is quite soluble in the stationary phase will take longer to travel through it than a component which is not very soluble in the stationary phase but very soluble in the mobile phase. As a result of these differences in mobilities, sample components will become separated from each other as they travel through the column.

2.1.2.2 The Column

The tube containing the retentive medium in which the separation is carried out is called the column. Figure 2.2 illustrates the cross-sections of the three major types of capillary column in current use. In open tubular columns, the stationary phase is coated as an even layer over the inner wall of the tube. This phase can either be a liquid, or it can be a porous layer of solid adsorbent material such as alumina, or a molecular sieve. The latter columns are known as porous layer open tubular (PLOT) columns and make a very important contribution to modern capillary GC. Most open tubular columns are now constructed either from fused silica or stainless steel.
Sometimes a thin film of liquid phase is coated on the inside wall of the fused silica tubing. Such columns are called wall coated open tubular (WCOT) columns. Packed capillary columns have similar tube geometry to open tubular columns except that they are filled with packing material. The packing is accomplished in a variety of ways, but usually the packing density is rather less than in conventional packed columns thus giving higher permeability.

### 2.1.2.3 Detector

The function of the detector is to provide a coherent, instantaneous signal in response to the elution of components from the column. Modern gas chromatography owes much of its current success to the development of high sensitivity ionization detectors, and in particular to the flame ionization detector (FID). This is the most ubiquitous detector and most laboratory gas chromatographs are equipped with one for general purpose applications. In the FID, hydrogen and air are used to produce a flame. A collector electrode with a DC potential is placed above the flame and measures the conductivity of the flame. With pure hydrogen, the conductivity is low.
However, as organic compounds are burned, the conductivity increases and the current increases, which can be correlated with the compounds.

An important feature of modern detection techniques is the availability of detectors which respond selectively to particular species. IUPAC defines a selective detector as one which responds to a related group of sample components in the column effluent, and a specific detector as one which responds to a single sample component or to a limited number of components having similar chemical characteristics. For example, the electron capture detector (ECD) is used in the analysis of trace pesticides and other environmentally important compounds and is regarded as a selective detector. Other widely used examples of detectors are the flame photometric detector (FPD) and the nitrogen phosphorous detector (NPD).

The detector signal is normally amplified before being recorded, possibly after further manipulation by a data system. In a basic system the amplified signal is merely monitored by a recorder to produce a chromatogram which is then used for all calculations. Although modern data systems are now pervasive in many laboratories the chromatogram is still required for visual assessments of separation and the calculation of chromatographic performance such as plate numbers.

2.1.3 The Chromatographic Process

A schematic representation of the chromatographic process is shown in Figure 2.3. The horizontal lines represent the column; each line is like a snapshot of the process at a different time (increasing in time from top to bottom). In the first snapshot, the sample, composed of components A and B, is introduced onto the column in a narrow zone. It is then carried through the column (from left to right) by the mobile phase.
Figure 2.3 Schematic representation of the chromatographic process [McNair, 1998]

Each component is partitioned between the two phases, as shown by the peaks above and below the line. Peaks above the line represent the amount of a particular component in the mobile phase, and peaks below the line the amount in the stationary phase. Component A has a greater distribution in the mobile phase and as a consequence it is carried down the column faster than component B, which spends more of its time in the stationary phase. Separation of A from B occurs as they travel through the column. Eventually the components leave the column and pass through the detector as shown. The output signal of the detector gives rise to a chromatogram shown at the right side of Figure 2.3.
2.1.4 Nomenclature

2.1.4.1 Phase Ratio

A fundamental property of the open tubular column is the phase ratio $\beta$, given by Equation 2.1.

$$\text{Phase ratio (}\beta\text{)} = \frac{\text{vol of carrier gas in column}}{\text{vol of stationary phase in column}} \quad (2.1)$$

From the known geometry of the column, the phase ratio can be calculated using Equation 2.2.

$$\beta = \frac{d_i}{4d_f} \quad (2.2)$$

where, $d_i$ is the internal column diameter, and $d_f$ is the thickness of the stationary phase film in the same units.

2.1.4.2 Partition Coefficient and Retention Factor

The retention properties of compounds depend critically on the phase ratio, and also on the affinity of compounds for the stationary phase, as measured by the partition coefficient $K$, given by Equation 2.3.

$$\kappa = \frac{\text{conc in stationary phase}}{\text{conc in carrier gas}} \quad (2.3)$$

where, the concentrations are measured in terms of mass or volume percent. In practice it is more useful to convert this to the retention factor, $k$, defined as the ratio of the mass of each component in the stationary phase to its mass in the carrier gas at equilibrium. The retention factor is obtained from the partition co-efficient from Equation 2.4.
The retention factor can be calculated from the measured retention times in the chromatogram. The time interval between sample injection and the maximum peak is the total retention time $t_R$ which is the actual time spent by the solute in the stationary phase (Figure 2.4). The hold up time is the retention time of a component which travels through the column at the same velocity as the carrier gas. The dead time describes the hold up time.

**Figure 2.4** Basic chromatographic measurements. Key: $t_0$ = column holdup time; $t_R(A) = $ total retention time for $A$; $t_R(B) = $ total retention time for $B$; $w_{1/2}(A) = $ peak width at half height for $A$; $w_{1/2}(B) = $ peak width at half height for $B$ [Grant, 1996].
2.1.4.3 Theoretical Plate Number

A typical method of characterizing the column of a GC is the theoretical plate number, $N$, also known as the column efficiency, calculated from a chromatogram run under isothermal conditions from Equation 2.5.

$$N = 5.54 \times \left( \frac{t_R}{w_h} \right)^2$$  \hspace{1cm} (2.5)

where $w_h$ is the width of the peak at half height. The column efficiency defines the width of the chromatographic zone inside the column just before it emerges and enters the detector. Columns with high theoretical plate numbers produce a very narrow peak that increases the probability of separating all the components of complex mixtures.

2.1.4.4 Theoretical Plate Height

The theoretical plate height is another meaningful way to characterize a GC column. It is calculated by dividing the length of the column by its number of theoretical plates (Equation 2.6).

$$H = \frac{L}{N}$$  \hspace{1cm} (2.6)

Although the theoretical plate height is a measure of column performance, it does not indicate the resolving power of the column.

2.1.4.5 Separation Factor

Two components having the same partition coefficients will travel along the column at exactly the same velocity and so they will elute unresolved at the same time. In order to assess the ability of the column to resolve any two compounds A and B, the effect of differences in the partition coefficients must be included. The separation factor, $\alpha$, is given by Equation 2.7.
separation factor $\alpha = \frac{k_B}{k_A} \geq 1$  

(2.7)

It is defined as the ratio of two retention factors, calculated using the larger of the two values as the numerator.

### 2.1.4.6 Peak Resolution

The peak resolution, $R_S$, is a measure of the separation of two components. It is defined by Equation 2.8.

$$
R_s = 1.2 \frac{t_{r_b}(B) - t_{r_b}(A)}{w_a(A) + w_a(B)}
$$

(2.8)

Defining resolution in this way quantifies the amount of peak area overlap between two peaks. This is important for quantitative analysis where mutual interference should be minimal. An acceptable overlap for most practical purposes is less than one percent of the respective peak areas which is equivalent to a resolution of less than 1.5.

### 2.2 Miniaturization of Gas Chromatographs

Use of MEMS technology to develop chemical analyzers is a promising approach to the realization of chemical instruments with lower cost, smaller size, lower power consumption, faster analysis, and greatly increased portability for in-field use, which can open new applications [Overton, 1996]. If a rapid, sensitive and selective hand-held GC system can be realized, it would have a wide range of applications. For example, this system could be used as a detection system for chemical and biological warfare agents and explosives [Noh, 2002].

#### 2.2.1 Gas Chromatography Systems on Silicon Wafer and the Parylene Gas Chromatograph

In the 1970s Terry et al. at Stanford developed the idea of fabricating an integrated micro GC on a silicon wafer [Terry, 1979]. This GC system had an injection valve, a long column, and
a thermal conductivity detector. The silicon wafer was bulk micromachined to realize 200 µm wide, 30 µm deep and 1.5 to 3 m long columns. The columns were sealed using Pyrex glass by anodic bonding. A sample mixture consisting of nitrogen, pentane and hexane vapors was injected into the column. Nitrogen, pentane and hexane eluted at 2.6, 3.6 and 4.4 seconds respectively.

Over the years, researchers at other institutions have also worked on improving and developing the microfabricated GC and have reported their findings. At Sandia National Laboratories Hudson et al. used the Bosch DRIE process to fabricate 40 µm wide, 250 µm deep, 1 m long spiral columns and studied transport and surface interaction processes for an optimized design of the GC column [Hudson, 1998].

A 300 µm wide, 10 µm deep and 0.9 m long rectangular column GC system on silicon was studied by Kolesar and Reston [Kolesar, 1998]. A 10 µl sample volume of ammonia and nitrogen dioxide, injected at a pressure of 40 psi, was separated in less than 30 minutes at operating temperatures between 55°C - 80°C.

At the University of Michigan, Lambertus et al. fabricated a 150 µm wide, 240 µm deep, 3 m long rectangular cross section channels using the DRIE technique [Lambertus, 2004]. Twenty compounds were separated isothermally in 4 minutes with hydrogen as the carrier gas. The separation took 10 minutes with air as the carrier.

Potkay et al. fabricated 0.9 m and 3 m long columns using the DRIE process [Potkay, 2003]. The channels were 150 µm wide and 240 µm deep and were sealed with a glass cap. The column was able to separate 16 gaseous components in 75 seconds.

A Parylene gas chromatographic column was developed at the Georgia Institute of Technology by Noh et al. [Noh, 2002; Noh, 2004]. The column made of Parylene was 100 µm
wide, 300 µm deep and 1 m long. The main advantage of this column was its lower heat capacity compared to the micromachined silicon column. This was also the first micro GC with an embedded heating element.

### 2.2.2 LIGA Fabricated MicroGC at LSU

Bhushan et al. at Louisiana State University fabricated 50 µm wide, 600 µm deep, 2 m long nickel columns using the LiGA process [Bhushan, 2001]. The columns were electrodeposited and then sealed with nickel cover plates which were also electrodeposited. External connections were made by attaching stainless steel capillaries (Small Parts, Miami Lakes, FL) with outer diameter of 0.016” and inner diameter of 0.010” to the openings in the sealed GC. Hydrogen was used as the carrier gas at flow rates of up to 20 ml/min and a flame ionization detector (FID) was used for detection. A mixture of four hydrocarbons hexane, octane, decane, and dodecane was separated in a coated column in less than 2 seconds at 70°C [Bhushan, 2006].

### 2.2.2.1 Advantages of LIGA Fabricated Micro GCs

Rectangular cross-section columns with high aspect ratios have better separation performance than equivalent circular cross-sectional areas because they have lower ‘height equivalent to a theoretical plate’ (HETP) [Spangler, 1998]. Such columns can be easily microfabricated using the LiGA process. LiGA fabricated columns have a much higher aspect ratio than the silicon micromachined columns, which are limited to low aspect ratios, typically 1-4. Another advantage of the LiGA process is the ability to fabricate structures having good quality vertical side walls. Sloped sidewalls cause non-uniform absorption of the effluent in the stationary phase, leading to peak broadening.
2.3 Temperature Programming in Miniaturized Gas Chromatographs

Though the concept of temperature programming has been known for quite a few years, the idea of temperature programming for micro gas chromatographs was developed only recently. Pasupuleti at Louisiana State University modeled a temperature programming cycle for the LSU micro GC [Pasupuleti, 2005]. Two types of heaters were designed, one to provide uniform heating over the micro GC column and the other to provide a linear temperature gradient along the length of the micro GC. The heaters were fabricated on silicon substrates by electrodepositing Ni-Cr (97.5 2.5) alloy. The heaters were integrated with a test micro GC, a PID controller and a fan to direct the temperature programming of the micro GC. Heating and cooling rates of up to 2.46°C/sec were demonstrated.

Agah et al. at the University of Michigan fabricated silicon-glass micro-gas chromatography (GC) columns having integrated heaters and temperature sensors for temperature programming [Agah, 2005]. The columns were etched in silicon using DRIE process and sealed with Pyrex glass using anodic bonding. To form the thin film heaters and temperature sensors, 250/500 Å Ti/Pt were evaporated and patterned on the back of the wafer using liftoff. The columns were able to separate about 20 components in 6 minutes. Ramp rates of 5°C/sec were achieved for temperature programming.

Potkay et al. fabricated a 1 m column in an oxynitride dielectric layer by a DRIE process [Potkay, 2006]. A 200/400Å Ti/Pt metal layer was deposited and patterned to form the heaters and temperature sensors. The device was placed in a custom vacuum chamber at a pressure of 10µTorr. To raise the local column temperature by 100°C, 6 mW of power was required. The column was able to separate 10 alkanes in 52 seconds and four chemical warfare agent stimulants and an explosive stimulant in 60s. The separations utilized a temperature ramp from
of 30 to 100°C at 50 °C/min. Though efforts are ongoing to perform temperature programming on micro GCs, all reported findings until now demonstrate very slow heating ramp rates (<5°C/sec). To enable faster analysis and to analyze a wider range of chemicals, higher heating rates (of the order of ~40°C/sec) have to be achieved.
CHAPTER 3. THERMAL MODELING

3.1 Introduction

System models were developed to estimate the thermal power requirements for heating a 0.5 m long nickel micro GC column. The thermal system needed fast response times, with desired heating rates between 5°C/sec - 50°C/sec. Dynamic response of the model was used to define the characteristics of the heat source for heating the GC column.

Figure 3.1 shows a schematic of the micro GC system. It consisted of the column, the top and bottom cover plates and the insulating layer. The heater was placed on top of the insulating layer. In the heat distribution model, the performance of the silicon and polyimide insulating layers between the heater coil and the GC column were compared with respect to heating rates for a given power input. The lumped parameter analysis of the heater layout helped in understanding of the Joule heating process and determination the best heater coil materials for fast heating rates.

![Schematic of the micro GC system](image)

**Figure 3.1** Schematic of the micro GC system, with the column, top and bottom plates, and the insulating layer.
3.2 Thermal System Modeling

3.2.1 Heat Distribution Model

The dynamic thermal response of the gas chromatograph was modeled using lumped mechanical elements. The lumped capacitance method assumes that the temperature of the solid is uniform at any instant during the transient process. This implies that the temperature gradients within the solid are negligible.

The column and the top and bottom cover plates were considered as one lump. The insulating layer was the second lump. The dimensions of the lumps are given in Table 3.1.

Each of these lumps was capable of storing energy. The capacity of these lumps to store energy was calculated using Equation 3.2.

\[ C = mc_p \] (3.2)

where \( c_p \) is the heat capacity of the material and \( m \) is the mass of the lump.

There are three thermal resistances in the system: the resistances for conductive heat transfer, convective heat transfer, and heat transfer by radiation. These are given by Equations 3.3 - 3.5.

<table>
<thead>
<tr>
<th>Lumps</th>
<th>Length (cm)</th>
<th>Width (cm)</th>
<th>Height (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lump 1 (Insulating layer)</td>
<td>2</td>
<td>1</td>
<td>10/600</td>
</tr>
<tr>
<td>Lump 2 (Column + Top Plate + Bottom Plate)</td>
<td>2</td>
<td>1</td>
<td>1500</td>
</tr>
</tbody>
</table>
Conductive heat transfer resistance: \( R_{\text{cond}} = \frac{L}{kA} \) \hspace{1cm} (3.3)

Convective heat transfer resistance: \( R_{\text{conv}} = \frac{1}{hA} \) \hspace{1cm} (3.4)

Radiative heat transfer resistance: \( R_{\text{rad}} = \frac{1}{\varepsilon \sigma A} \) \hspace{1cm} (3.5)

where, \( L \) is the length of the block, \( A \) is the area of cross-section, \( k \) is the thermal conductivity of the material, \( h \) is the convective heat transfer coefficient, \( \varepsilon \) is the emissivity of the material, and \( \sigma \) is the Stefan-Boltzmann constant.

Figure 3.2 shows the schematic of the two lump heat distribution model of the system. The corresponding pseudo bond graph is shown in Figure 3.3. The power input was from the top side of the GC column and was modeled as a constant heat input (flow source). The ambient temperature, \( T_\infty \), was constant and represented by an effort source. Since the ratio of the thickness to the length and width of the device was very low, one dimensional heat transfer was assumed.

There were six principal components in the system: the conductive resistance field (\( R_3 \)) representing the conductive heat transfer from lump 1 (insulating layer) to lump 2 (column), given by Equation 3.3; the convective resistance fields (\( R_1 \) and \( R_4 \)) which accounted for convective heat transfer from the system to the ambient, given by Equation 3.4; the radiative resistance fields (\( R_2 \) and \( R_5 \)) which represented radiation heat transfer from the system into the atmosphere, given by Equation 3.5; the thermal capacitances (\( C_1 \) and \( C_2 \)), given by Equation 3.2; the input heat flux modeled by a flow source (\( S_f \)); and a heat sink (ambient temperature) equivalent to the effort source (\( S_e \)).
The heat flux input into a node equals the sum of heat fluxes used to heat the lump, the heat flux transmitted and the heat flux lost due to convection and radiation. Balancing the heat flux at the two zero junctions, equations at the two nodes can be written as shown in Equation 3.6:

\[ S_f = C_1 \frac{dT_1}{dt} + \frac{1}{R_3}(T_1 - T_2) + \frac{1}{R_4}(T_1 - T_\infty) + \frac{1}{R_5}(T_1^4 - T_\infty^4) \]

\[ \frac{1}{R_3}(T_1 - T_2) = C_2 \frac{dT_2}{dt} + \frac{1}{R_4}(T_2 - T_\infty) + \frac{1}{R_5}(T_2^4 - T_\infty^4) \]

Typical values for convective heat transfer coefficients for free convection of gases at a boundary range between 2 – 25 W/m²-K (Incropera, 2002). The coefficient depends on the conditions at the boundary layer which are influenced by surface geometry and the nature of the fluid flow. A value of 15 W/m²-K was taken as a representative value for the simulation.

Figure 3.2 Heat distribution model of the micro GC system
Simulations were carried out with MATLAB 7.0 (The Mathworks, Inc., Natick, MA). An ordinary differential equation solver, ode15s, was used to solve Equations 3.6 for a range of heat flux inputs. A range (5W – 100W) of step inputs was applied to the system to observe the temperature response. With an input power of 25W a ramp rate of 20°C/sec was achieved as shown in Figure 3.4 (a). The system reached 350°C in 6 seconds with a power input of 70W, which corresponded to a ramp rate of 55°C/sec as shown in Figure 3.4 (b).

The polyimide insulating layer provided a faster heating rate than silicon. Though the polyimide had lower thermal conductivity than silicon, the thickness of the polyimide insulating layer was only 10 µm compared to 600 µm for the silicon substrate. Thus heat was transferred at a much faster rate through polyimide.

Varying the convection coefficient did not make much of a difference in the heating rate, but it affected the steady state temperature significantly. Increasing the convection coefficient by
a value of 5, to 10 W/m²-K, decreased the steady state temperature by 25°C. Correspondingly when it was reduced by 5, to 10 W/m²-K, the steady state temperature increased by about 25°C.

This heat distribution model gave only an estimate as to how much power was required to achieve a given ramp rate for a given GC column. It was assumed that the heat flux was constant.
throughout. This model was only a first step in the design of the heaters. It was a good starting point to describe the characteristics of the heat source and determining the resistance for the heater coil. For the physical system, as the temperature increases, the resistance increases, hence the heat flux is not constant. A feedback control loop system would be needed to maintain a constant current, thereby providing a constant heat flux.

3.2.2 Lumped Parameter Analysis of the Heater Layout

A lumped parameter, voltage input model was used to determine the time dependence of the temperature distribution within the heater layout during transient Joule heating of the integrated heater-GC system. Again, the lumped capacitance method was used with the same assumptions which were stated in the previous section.

Figure 3.5 shows the basic unit of a heater layout model. It has four vertical strips (Nos. 1, 3, 5, 7) which were connected by three small connectors (Nos. 2, 4, 6). This heater coil was separated from the nickel GC column by an insulating layer. Any element in the heating coil, when considered in isolation, had its own power inputs, heat capacity and heat losses. The power inputs were the Joule heating plus the conduction from adjacent heating elements. The amount of heat stored in an element depended on the mass of that element and the heat capacity of the material. The heat losses included conduction from the heating element into the insulating layer, conduction into adjacent elements and convection and radiation losses. This is illustrated for an isolated element number 3 from the basic heater unit in Figure 3.6.

Using the conservation of energy principle for element number 3 in Figure 3.6 the Equation 3.7 resulted.

\[
S_f + \frac{kA}{l}(T_2 - T_3) + \frac{kA}{l}(T_4 - T_3) = mC_p \frac{dT_3}{dt} + \frac{kA}{l}(T_3 - T_2) + \frac{kA}{l}(T_3 - T_4) + \frac{kA}{l}(T_3 - T_8) + hA_{conv}(T_3 - T_w) + \varepsilon A_{rad} T_3^4
\]  

(3.7)
where, $S_f$ is the Joule heating of the element, $k$ is the thermal conductivity of the material, $l$ is the length of the element for conduction, $A$ is the area of cross-section for conduction, $C_p$ is the heat capacity of the material, $h$ is the convective heat transfer coefficient, $A_{\text{conv}}$ is the surface area for convective heat losses, $A_{\text{rad}}$ is the surface area for radiation losses, $\varepsilon$ is the emissivity of the material and $\sigma$ is the Stefan-Boltzmann constant.

Extrapolating the conservation of energy principle to all of the elements of the heater layout, Equations 3.8 were developed for the basic heater unit:
\[ (1) \quad S_{f1} + \frac{k_{air} A_{31}}{l} (T_3 - T_1) + \frac{k_{A12}}{l} (T_2 - T_1) \]
\[ = mC_p \frac{dT_1}{dt} + \frac{k_{A12}}{l} (T_1 - T_2) + \frac{k_{A18}}{l} (T_1 - T_3) + \frac{k_{air} A_{47}}{l} (T_1 - T_3) + hA_{conv} (T_1 - T_e) + \epsilon A_{rad} T_1^4 \]

\[ (2) \quad S_{f2} + \frac{k_{A12}}{l} (T_1 - T_2) + \frac{k_{A32}}{l} (T_3 - T_2) \]
\[ = mC_p \frac{dT_2}{dt} + \frac{k_{A23}}{l} (T_2 - T_3) + \frac{k_{A12}}{l} (T_2 - T_1) + \frac{k_{A28}}{l} (T_2 - T_8) + hA_{conv} (T_2 - T_e) + \epsilon A_{rad} T_2^4 \]

\[ (3) \quad S_{f3} + \frac{k_{air} A_{31}}{l} (T_3 - T_1) + \frac{k_{air} A_{53}}{l} (T_3 - T_3) + \frac{k_{A23}}{l} (T_2 - T_3) + \frac{k_{A43}}{l} (T_4 - T_3) \]
\[ = mC_p \frac{dT_3}{dt} + \frac{k_{A32}}{l} (T_3 - T_2) + \frac{k_{A14}}{l} (T_3 - T_4) + \frac{k_{A28}}{l} (T_3 - T_8) + \frac{k_{air} A_{13}}{l} (T_3 - T_1) + \frac{k_{air} A_{35}}{l} (T_3 - T_5) \]
\[ + hA_{conv} (T_2 - T_e) + \epsilon A_{rad} T_3^4 \]

\[ (4) \quad S_{f4} + \frac{k_{A34}}{l} (T_3 - T_4) + \frac{k_{A54}}{l} (T_5 - T_4) \]
\[ = mC_p \frac{dT_4}{dt} + \frac{k_{A13}}{l} (T_4 - T_3) + \frac{k_{A45}}{l} (T_4 - T_5) + \frac{k_{A48}}{l} (T_4 - T_8) + hA_{conv} (T_2 - T_e) + \epsilon A_{rad} T_4^4 \]

\[ (5) \quad S_{f5} + \frac{k_{air} A_{35}}{l} (T_3 - T_5) + \frac{k_{air} A_{75}}{l} (T_7 - T_5) + \frac{k_{A45}}{l} (T_4 - T_5) + \frac{k_{A65}}{l} (T_6 - T_3) \]
\[ = mC_p \frac{dT_5}{dt} + \frac{k_{A54}}{l} (T_5 - T_4) + \frac{k_{A56}}{l} (T_5 - T_6) + \frac{k_{A28}}{l} (T_5 - T_8) + \frac{k_{air} A_{35}}{l} (T_5 - T_3) + \frac{k_{air} A_{52}}{l} (T_5 - T_7) \]
\[ + hA_{conv} (T_5 - T_e) + \epsilon A_{rad} T_5^4 \]

\[ (6) \quad S_{f6} + \frac{k_{A56}}{l} (T_5 - T_6) + \frac{k_{A76}}{l} (T_7 - T_6) \]
\[ = mC_p \frac{dT_6}{dt} + \frac{k_{A65}}{l} (T_6 - T_5) + \frac{k_{A67}}{l} (T_6 - T_7) + \frac{k_{A68}}{l} (T_6 - T_8) + hA_{conv} (T_6 - T_e) + \epsilon A_{rad} T_6^4 \]
These equations were solved in MATLAB using ode15s for a range of input voltages (3V-12V). Simulations were run with nickel, copper and NiCr as the heater materials. The temperature profiles for 6V and 12V inputs are plotted in Figures 3.7 (a) and 3.7 (b). As seen from the plots, nickel and copper had a faster heating rate for the same power input compared to nickel chrome which makes them better heater coil materials.

### 3.3 Conclusions

The heat distribution model was used to estimate that for a 2 cm X 1 cm X 1.5 mm half meter long column chip, heated at the rate of ~50°C/sec, the power required was 70W for a polyimide insulating layer. For a silicon insulating layer, the power required increased to 80W to heat the chip at the same rate because of the increased thickness of the silicon substrate (600µm) compared to polyimide (10µm) even though the thermal conductivity of silicon was higher than that of polyimide.

From the lumped parameter analysis of the heater layout, for an applied potential difference between the ends of the continuous heating element, based on Joule heating and heat
losses to the ambient through convection and radiation, the individual temperatures of the heating coil, the insulator and the heat sink were determined. Since copper and nickel have lower
electrical resistivities than nickel-chrome, for a given power input, the heating rate for copper and nickel was higher than that for nickel chrome.
CHAPTER 4. DESIGN OF THE HEATERS

4.1 Introduction

Isothermal operation of gas chromatographs limits the range of solutes that can be eluted within a reasonable time. In order to broaden the scope of separable compounds, chromatographers resort to changing the column temperature during the elution process, which is called temperature programming.

Commercial GCs are equipped with ovens to perform temperature programming. The disadvantages of using these bulky ovens have already been discussed in Section 1.2.2 of Chapter 1. A resistive heater, integrated with a GC column, has advantages including reduced power requirements, faster heating rates, and facilitation of temperature control for thermal focusing, multi-dimensional gas chromatography, and the further miniaturization of GCs [Jain, 1995].

4.2 Device Requirements

The heater used to heat the 0.5m LiGA nickel column needed to be able to ramp the temperature of the column at a rate of up to 50°C/sec. The power required to achieve this would be about 70W over 2 cm² area as shown in Figure 3.4 (b). The heater should also be stable, with no material failure, at high temperatures (~350°C) and should be able to withstand repeated heating and cooling cycles (~10000).

4.3 Design Methodology

4.3.1 Basic Unit of a Heater Layout

The basic unit of a heater layout is illustrated in Figure 4.1. It consisted of four vertical strips joined by three connectors. Any heater will be a multiple of this basic unit. From Figure
4.1, the number of strips ($n$) and the gap ($g$) between each strip can be evaluated using Equations 4.1-4.2:

\[ n = \frac{l - q}{p - w} \]  \hspace{1cm} (4.1)

\[ g = \frac{pq - lw}{l - q - p + w} \]  \hspace{1cm} (4.2)

where,

$n = \text{number of vertical strips},$

$g = \text{gap between each strip (m)},$

$l = \text{total length of the heater coil from end to end (m)},$

$p = \text{total length of heater layout (m)},$

$q = \text{total width of heater layout (m)},$ and

$w = \text{width of each strip (m)}.$

Figure 4.1 Basic unit and dimensions of the heater layout.
4.3.2 Calculation of the Resistance of the Heater Coil

The resistance of the heater was based on power required to heat the column at a desired ramp rate and the voltage supply available. From Equations 3.6 and Figure 3.4 (b), the power required to ramp up the 0.5 m GC column was estimated as 70W. For a given voltage supply, the resistance of the heater coil was calculated using Equation 4.3.

\[ R = \frac{V^2}{P} \]  

(4.3)

where,

\( V \) is the voltage,

\( P \) is the power, and

\( R \) is the resistance.

For a voltage input of 12 volts and the required power of 70 watts, the required resistance of the heater coil was 2 ohms.

4.3.3 Calculation of Final Dimensions for the Heater Layout

The resistance of the heater coil is given by Equation 4.4.

\[ R = \frac{\rho l}{wt} \]  

(4.4)

where,

\( \rho \) is the resistivity of the material,

\( l \) is the length of the coil,

\( w \) is the width of each strip, and

\( t \) is the thickness of the coil.
Table 4.1 Final dimensions of the heater layout

<table>
<thead>
<tr>
<th>Item</th>
<th>Symbol</th>
<th>Number/Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of vertical strips</td>
<td>$n$</td>
<td>10</td>
</tr>
<tr>
<td>Width of each strip</td>
<td>$w$</td>
<td>500 $\mu$m</td>
</tr>
<tr>
<td>Gap between strips</td>
<td>$g$</td>
<td>333.33 $\mu$m</td>
</tr>
<tr>
<td>Thickness of strips</td>
<td>$t$</td>
<td>10 $\mu$m</td>
</tr>
<tr>
<td>Length of the heater coil</td>
<td>$l$</td>
<td>14.3 cm</td>
</tr>
</tbody>
</table>

By trial and error, the width of the strip was selected as 500$\mu$m with 10$\mu$m thickness to fit the 2 cm X 1 cm layout for a 2 ohm resistance. The final dimensions of the heater coil were calculated and are shown in Table 4.1. The heater design is illustrated in Figure 4.2.

![Figure 4.2 Final heater layout design](image)
4.4 Finite Element Analysis of the Heaters

4.4.1 Introduction

Finite element analysis of the heater was carried out to simulate the temperature distribution and estimate the thermal stresses developed in the heaters. ANSYS (version 9.0, Canonsburg, PA) was the finite element software that was used for this analysis. The model consisted of the GC column, the polyimide insulating layer and the heater coil (Figure 4.3). The nickel GC column was approximated by a nickel block with the same overall dimensions. This was done because the University Advanced version of ANSYS only supports 128,000 elements and incorporating the actual GC column into the model was not possible, as it exceeded this element number limit while meshing.

Figure 4.3 Finite element model of the heater, the insulating layer and the GC column.
Table 4.2 Dimensions of the sections used in the finite element model

<table>
<thead>
<tr>
<th>Item</th>
<th>Length</th>
<th>Breadth</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel GC column</td>
<td>2 cm</td>
<td>1 cm</td>
<td>1.5 mm</td>
</tr>
<tr>
<td>Polyimide layer</td>
<td>2 cm</td>
<td>1 cm</td>
<td>10 µm</td>
</tr>
<tr>
<td>Heater coil</td>
<td></td>
<td></td>
<td>Dimensions as shown in Figure 4.2</td>
</tr>
</tbody>
</table>

4.4.2 Geometry

The finite element model of the heater and the GC system had three sections. They were the nickel GC column (block), polyimide insulating layer, and the heater coil. The dimensions of these sections are given in Table 4.2. The different volumes of the model were joined using the VGLUE command. Use of this command generated new volumes by “gluing” input volumes. The VGLUE command results in the merging of areas, lines, and keypoints at the common volume boundaries. The glue operation redefines the input volumes so that they share areas along their common boundaries. The new volumes encompass the same geometry as the original volumes.

4.4.3. Material Properties

Two different materials were used in the model. Nickel was used as the material for the GC column and as heater material. Polyimide (PI 5878G, HD MicroSystems, Parlin, NJ) was selected for the insulating material. The properties of these materials are given in Table 4.3.

4.4.4 Meshing

Tetrahedral elements were used to mesh the model because the geometry was too complicated to use quadrilateral elements. In an ideal scenario all sections of the model would be meshed using the smallest element size. Since the University Advanced version of ANSYS supports only 128,000 elements, different sections of the model were meshed using different
element sizes (Table 4.4). To test the effect of using different mesh sizes and their impact on the results, mesh refinement was performed and the results were tested for convergence which is explained in Section 4.4.6.

4.4.5 Boundary Conditions and Loads

On all of the surface areas, convection and radiation boundary conditions were applied. An average convection coefficient of 15 W/m².K (Incropera, 2001) was used. The results varied less than 1% for a change of ±5 W/m².K of the convection coefficient. Heat generation was applied to the heating coil corresponding to the voltage difference between the ends of the coil.

4.4.6 Results and Test for Convergence

Simulations were run on the FEM model shown in Figure 4.3. A heat generation of 70 watts, corresponding to a 12 volt input to the heating coil and the temperature profile was obtained. The transient temperature distribution at the end of 10 seconds, in isometric and bottom views are shown in Figures 4.4 and 4.5, respectively. There is a variation in temperature of about 5°C/sec over the system.

Table 4.3 Material properties used in the finite element model

<table>
<thead>
<tr>
<th>Model section</th>
<th>Material used</th>
<th>Density, $\rho$ (kg/m³)</th>
<th>Specific heat, $C_p$ (kJ/kg.K)</th>
<th>Thermal Conductivity, $k$ (W/m.K)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>300°K 400°K 600°K 800°K</td>
<td>300°K 400°K 600°K 800°K</td>
<td>300°K 400°K 600°K 800°K</td>
</tr>
<tr>
<td>GC column</td>
<td>Nickel</td>
<td>8900 8900 8900 8900</td>
<td>444 485 592 530</td>
<td>90.7 80.2 65.6 67.6</td>
</tr>
<tr>
<td>Insulating layer</td>
<td>Polyimide</td>
<td>1420 1420 1420 1420</td>
<td>1090 1090 1090 1090</td>
<td>0.16 0.16 0.16 0.16</td>
</tr>
<tr>
<td>Heater coil</td>
<td>Nickel</td>
<td>8900 8900 8900 8900</td>
<td>444 485 592 530</td>
<td>90.7 80.2 65.6 67.6</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Model section</th>
<th>Material used</th>
<th>Elastic Modulus (GPa)</th>
<th>CTE ($10^{-6}$/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC column</td>
<td>Nickel</td>
<td>200</td>
<td>13.3</td>
</tr>
<tr>
<td>Insulating layer</td>
<td>Polyimide</td>
<td>2.5</td>
<td>17</td>
</tr>
<tr>
<td>Heater coil</td>
<td>Nickel</td>
<td>200</td>
<td>13.3</td>
</tr>
</tbody>
</table>
Table 4.4 Mesh sizes used in the FEM model

<table>
<thead>
<tr>
<th>Model section</th>
<th>Element edge size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC column</td>
<td>500</td>
</tr>
<tr>
<td>Insulating layer</td>
<td>500</td>
</tr>
<tr>
<td>Heater coil (Coarse)</td>
<td>500</td>
</tr>
<tr>
<td>Heater coil (Medium)</td>
<td>250</td>
</tr>
<tr>
<td>Heater coil (Fine)</td>
<td>125</td>
</tr>
</tbody>
</table>

In order to test the effect of the mesh sizes on the results obtained, three different mesh sizes were used (coarse, medium and fine). The edge length of the medium element was taken to be half that of the coarse element size and the edge length of the fine element was chosen to be half that of medium element size. The sizes used for the coarse, medium and fine were 500 µm, 250 µm and 150 µm, respectively. This mesh refinement was carried out only on the heater coil section; the mesh sizes of the bulk insulating layer and the GC column remained constant throughout the analysis because of the restriction in the number of elements (128,000) in the University Advanced version of ANSYS used.

Convergence meant that the difference in results using successive mesh sizes was decreasing. The following equation was used for testing for convergence (Sinclair, 2005):

\[
| T_{\text{cor}}^{\text{max}} - T_{\text{med}}^{\text{max}} | > | T_{\text{med}}^{\text{max}} - T_{\text{fin}}^{\text{max}} |
\]

where, \( T_{\text{cor}}^{\text{max}} \), \( T_{\text{med}}^{\text{max}} \) and \( T_{\text{fin}}^{\text{max}} \) are maximum temperatures obtained from the coarse, medium and fine mesh sizes, respectively. Table 4.5 summarizes the results obtained from the three mesh sizes. The results were found to be converging which makes them independent of element sizes used in meshing the model.
**Figure 4.4** Isometric view of temperature distribution at the end of 10 seconds for a 70 watt power input

**Figure 4.5** Bottom view of temperature distribution at the end of 10 seconds for a 70 watt power input
Table 4.5 Convergence testing data

<table>
<thead>
<tr>
<th>Variable</th>
<th>Convergence values</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T^\text{max}_{\text{cor}}$</td>
<td>391.525 °K</td>
</tr>
<tr>
<td>$T^\text{max}_{\text{med}}$</td>
<td>392.556 °K</td>
</tr>
<tr>
<td>$T^\text{max}_{\text{fin}}$</td>
<td>392.547 °K</td>
</tr>
<tr>
<td>$</td>
<td>T^\text{max}<em>{\text{cor}} - T^\text{max}</em>{\text{med}}</td>
</tr>
<tr>
<td>$</td>
<td>T^\text{max}<em>{\text{med}} - T^\text{max}</em>{\text{fin}}</td>
</tr>
</tbody>
</table>

4.4.7 Thermal Stresses in Heaters

4.4.7.1 Introduction

Thermal expansion mismatches between the heater material and the substrate on which it was fabricated were a very important aspect of the heater design that needed to be investigated in order for the heaters to be reliable. Typical values of the coefficient of thermal expansion (CTE) for silicon and nickel are 2.49 µm/m-°C and 13.1 µm/m-°C (at 25°C). When nickel heaters are electrodeposited on silicon, there is significant thermal stress at elevated temperatures because of the large difference in the CTE values of the two materials. However, polyimide, which was used as the substrate for the heater and the insulating layer in the current project, had a CTE of 17 µm/m-°C (for 10 µm thickness) which is very close to that of nickel. As a result the thermal stresses developed in a nickel on polyimide heater were significantly less than the thermal stresses developed in a nickel on silicon heater.
4.4.7.2 Thermal Stress Analysis Using ANSYS

The coupled field analysis method in ANSYS was used for investigating the thermal stresses in the heaters. In this method, transient thermal analysis was followed by structural analysis. The temperatures obtained from the thermal analysis were applied as body loads on each node and the model was analyzed structurally. The von Mises stress distribution from the coupled field thermal-structural analysis of the heater layout for a power input of 70 watts is shown in Figure 4.6. The maximum stress obtained from the finite element analysis was found to be 60 MPa.

Figure 4.6 Thermal stresses in the heater layout and column
4.4.7.3 Results

The maximum stress obtained from the thermal-structural analysis was 60 MPa. The yield stress of electroplated nickel in a similar temperature range is 180 MPa [Fritz, 2002; Cho, 2002]. Comparison of the two values showed that the maximum stress developed was less than the yield strength with a factor of safety of 3.0.

4.5 Summary

The design concepts used in the heater design and the finite element analysis of the heater layout were described. Temperature profiles were obtained for power inputs corresponding to 6 volts and 12 volts. Thermal stress analysis of the heaters showed that the stresses developed were less than the yield strength of electroplated nickel, which made the heater design safe.
CHAPTER 5. FABRICATION

5.1 Process Overview

The fabrication of the heaters was done using the UV lithography technique. Figure 5.1 gives an overview of the steps involved in the microfabrication process.

Fabrication began with the spinning of liquid polyimide (PI 5878G, HD MicroSystems, Parlin, NJ) on the nickel GC column. The polyimide was then cured in a vacuum oven. The next step was to electron beam (e-beam) evaporate a 0.01 \(\mu\)m titanium adhesion layer and a 0.05 \(\mu\)m copper plating base to facilitate electrodeposition in future steps. A 14 \(\mu\)m layer of SPR 220-7 (Rohm and Haas, Philadelphia, PA) positive photo resist, a masking layer, was spin coated and baked. A UV exposure was performed, the resist was developed, and nickel was deposited into the micropatterned recess to form the heater coil. The remaining resist was completely removed, and the copper and titanium seed layers were etched away in the exposed areas to leave free standing heater coils on a polyimide insulating layer on top of the GC column.

5.2 Optical Lithography

Optical lithography transfers a pattern from an optical mask into a layer of photoresist. Optical mask blanks are readily available in standard sizes, with chrome and photoresist in place. For ultra violet (UV) light based lithography, a 300 Å layer of chromium typically forms the radiation absorbing region. Based on the device design, the chrome layer is patterned on the mask. Dark regions on the mask are covered with chrome and block the UV light. Clear field regions allow the UV light to pass through and modify the photo resist. Depending on the chemical composition of the photoresist, the UV light changes the properties on exposure. The light either polymerizes a negative resist into longer chains or causes molecular scissions in a positive resist. The masks where absorber defines the pattern are called dark field masks, while
those where the clear areas define the pattern are called clear field masks. Dark field and clear field masks for the same pattern have chrome on complimentary areas and are in “opposite tone” to each other.

5.3 Optical Mask Design

The heater patterns were laid out in AutoCAD 2004 (Autodesk Inc., San Rafael, CA) and converted to .dxf file format. This was then converted to the .gds file format using DXF2GDS® conversion software (DFX, Artwork Conversion Software Inc., Santa Cruz, CA) and viewed using the GDSVUE® software from the same vendor. After doing the conversions, all features on the mask were assessed for errors. Industry standard chrome-on-soda-lime glass optical mask blank, 5” X 5” X 0.09” in dimension, was purchased from Advance Reproductions, North
Andover, MA. Figure 5.2 shows the important dimensions of the heater pattern, which are listed in Table 5.1

5.4 Substrate Preparation

The nickel GC columns were polished on a Polimet Polisher polishing wheel (Buehler Ltd., Evanston IL) to make the surface flat and smooth for subsequent processing (Figure 5.3). The polishing was done using silicon carbide grinding paper (Buehler Ltd., Lake Bluff, IL) of grit number 60. The polishing step was subsequently repeated with grinding papers of grit numbers 120 and 400 to get a mirror-like final finish on top of the nickel GC column. The columns were then soaked in 15% sulfuric acid for 30 seconds to remove any oxides from the surface.

Figure 5.2 Characteristic dimensions of heaters
Table 5.1 Characteristic dimensions of heaters

<table>
<thead>
<tr>
<th>Item</th>
<th>Number/Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of vertical strips</td>
<td>10</td>
</tr>
<tr>
<td>Number of turns</td>
<td>9</td>
</tr>
<tr>
<td>Width of each strip</td>
<td>500 µm</td>
</tr>
<tr>
<td>Gap between strips</td>
<td>333.33 µm</td>
</tr>
<tr>
<td>Length of the heater coil</td>
<td>14.3 cm</td>
</tr>
</tbody>
</table>

Figure 5.3 Polishing wheel for substrate preparation

5.5 Spin Coating and Curing of Polyimide

The next step in the fabrication process was the spin coating and curing of the insulating layer. The nickel GC column was placed on the vacuum chuck in a light duty photo resist spinner.
(PWM101, Headway Research Inc., Garland, TX). It was held in place by a two-sided Scotch tape (3M, St.Paul, MN). Approximately 5 ml of PI-5878G (HD MicroSystems, Parlin, NJ) liquid polyimide was placed on the substrate and spun at 2000 rpm for 30 seconds to obtain a uniform coat of 10 µm thick polyimide. The polyimide was cured in a vacuum oven (VT5042EKP500, Heraeus Holding GmbH, Hanau, Germany). The cure heating cycle drives out the remaining carrier solvent and converts the polyimide precursor (or polyamic acid) into an insoluble polyimide film. As the process continues, water is released as a by-product of the imidization reaction. The curing profile is shown in Figure 5.4.

![Figure 5.4 Polyimide cure temperatures and times](http://www.hdmicrosystems.com/tech/techinfo.html)

(PWM101, Headway Research Inc., Garland, TX). It was held in place by a two-sided Scotch tape (3M, St.Paul, MN). Approximately 5 ml of PI-5878G (HD MicroSystems, Parlin, NJ) liquid polyimide was placed on the substrate and spun at 2000 rpm for 30 seconds to obtain a uniform coat of 10 µm thick polyimide. The polyimide was cured in a vacuum oven (VT5042EKP500, Heraeus Holding GmbH, Hanau, Germany). The cure heating cycle drives out the remaining carrier solvent and converts the polyimide precursor (or polyamic acid) into an insoluble polyimide film. As the process continues, water is released as a by-product of the imidization reaction. The curing profile is shown in Figure 5.4.

![Figure 5.4 Polyimide cure temperatures and times](http://www.hdmicrosystems.com/tech/techinfo.html)

5.6 E-beam Evaporation of Adhesion and Seed Layers

The cured polyimide was covered with an e-beam deposited 0.01 µm thick titanium adhesion layer and a 0.05 µm thick layer of copper, which served as the plating base for the heater coil. The e-beam evaporation was done using the Temescal BJD-1800 E-Beam Deposition System (Temescal, Fairfield, CA).

5.7 Spin Coating and Pre-Bake of Resist

The nickel GC column with the seed layer was placed on the vacuum chuck in the light duty spinner and held in place by the double sided scotch tape. About 5 ml of SPR 220-7 (Rohm and Haas, Philadelphia, PA) positive photo resist was placed on the column and then spun at 1100 rpm for 30 seconds to get a 14 µm thick resist layer. About 0.5 mm of the four edges of the GC column was covered with scotch tape to aid in smoothing the flow of the resist and to prevent edge beading. The photoresist was let sit on a flat surface for about 5 minutes to relieve stress and then pre-baked on a hot plate at 105°C for 3 minutes to drive off carrier solvents in the coating.

5.8 UV Exposure and Development of Resist

The SPR 220-7 resist coated nickel GC columns were exposed under UV light through the optical mask for 45 seconds corresponding to incident energy of 360 mJ/cm². After exposure the resist was developed in a 50-50 mixture of MF 321 and MF 322 Developer (Rohm and Haas, Marlborough, MA) for about 3 minutes while intermittently rinsing it in DI water. After the resist was completely developed it was blow dried with compressed air. Figure 5.5 shows the connecting pad area and the portion of the heater coil after development.

7 Rohm and Haas, Philadelphia, PA
5.9 Electrodeposition

5.9.1 Introduction

The results from the lumped parameter analysis (section 3.3.3) of the heater layout suggested using nickel and copper as the heater coil elements. For a given voltage input, nickel and copper have a higher heating rate than nickel chrome. For the nickel plating, a standard nickel sulfamate bath was used, and for the copper plating, a copper sulfate – sulfuric acid recipe was used.
5.9.2 Experimental Apparatus

The electroplating was done at the Center for Advanced Microstructures and Devices\textsuperscript{8} (CAMD), Baton Rouge, LA. The electroplating apparatus is shown in Figure 5.6. It consists of a three baths, two for nickel plating and one for copper plating. The electrolytes were circulated using a filter pump agitation system. The filter used was an Intank Model #5 (Technic Inc., Pawtucket, RI) and the pump was Model # P-1/15 A (Filter Pump Industries, Sun Valley, CA). A Shenzhen Mastech HY 3003D-3 DC Power supply (Precision Mastech Enterprises Co., Kowloon, Hong Kong) was used as the current source for electrodeposition.

Figure 5.6 Electroplating apparatus.

\textsuperscript{8}http://camd.lsu.edu
The copper seed layer inside the recess formed after UV exposure and development of the photo resist was the cathode for electrodeposition. The size of the cathode area exposed for deposition of one heater was 1.0426 cm$^2$. The back side of the nickel GC column was insulated with a non-conducting tape (Model #MD4000 M Q250/Roll, Harman Corp., Rochester, MI) and the side walls were insulated using liquid electrical tape (Star Brite, Ft. Lauderdale, FL) as shown in Figure 5.7. Sulfurized nickel pellets (Inco Limited, United Kingdom) and phosphorized copper pellets (Technic Inc., Pawtuket, RI) were used as the anodes for the nickel and copper electrodeposition, respectively.

Figure 5.7 Bottom and side wall insulation of the nickel GC column for electroplating
(a) Full cathode view; (b) Close up of column with pattern

5.9.3 Electrodeposition of Nickel

5.9.3.1 Electrolytes Used

A standard nickel sulfamate bath was used for the nickel plating. The chemicals used in this bath are shown in Table 5.1.
Table 5.1 Recipe for nickel plating

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>g/liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel sulfamate ( ((H_2NSO_3)_2*4H_2O) )</td>
<td>450</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>37.5</td>
</tr>
<tr>
<td>Saccharin</td>
<td>4</td>
</tr>
<tr>
<td>Coumarin</td>
<td>0.3</td>
</tr>
<tr>
<td>Sodium Lauryl Sulfate</td>
<td>1</td>
</tr>
</tbody>
</table>

### 5.9.3.2 Plating Parameters

An area of 1.0426 cm$^2$ was electroplated with nickel for the heater coil. The plating was carried out at a current density of 10 mA/cm$^2$, corresponding to a current of 10.426 mA. For a thickness of 10µm, the plating time was 48.76 minutes. The pH of the plating solution was 4.00 and the temperature of the plating bath was 40°C.

### 5.9.4 Electrodeposition of Copper

#### 5.9.4.1 Electrolytes Used

For the copper electroplating, a copper sulfate - sulfuric acid bath was used. The electrolytes used in this bath are shown in Table 5.2.

#### 5.9.4.2 Plating Parameters

Copper plating was done at a current density of 10 mA/cm$^2$. For a heater coil area of 1.0426 cm$^2$, this corresponded to a current of 10.426 mA. The plating time was 45.32 minutes for a plating thickness of 10 µm.

### 5.9.5 Results

The thickness of the coils from the electroplating matched with the desired thickness. Pictures of the nickel and copper heaters are shown in Figures 5.8 and 5.9, respectively.
Table 5.2 Recipe for copper plating

<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>g/liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuSO$_4$.5H$_2$O</td>
<td>187.25</td>
</tr>
<tr>
<td>H$_2$SO$_4$</td>
<td>47.9</td>
</tr>
<tr>
<td>HCl</td>
<td>0.11</td>
</tr>
</tbody>
</table>

Figure 5.8 Nickel heater after plating

Figure 5.9 Copper heater after plating
After electroplating the heater coil, the remaining photoresist was removed using acetone. The top copper plating base was etched in the open areas using APS Copper Etchant 100 (Transene Company Inc., Danvers, MA). The etch rate was about 50 Å/sec and the etching took about 10 seconds. The titanium adhesion seed layer was etched away using a 5% Hydrofluoric Acid (HF). The etching was carried out for about 3 seconds with an etch rate of about 30 Å/sec. No significant undercut was observed in either case.

The heater profiles were checked in different places and the plating was uniform across the heater area. The heater profilometry is shown in Figure 5.10.
5.11 Summary

Nickel and copper heaters were microfabricated on a polyimide insulating layer on top of the nickel GC columns. Polyimide was a novel material for the insulation layer because of its resistance to downstream processing. After curing, the polyimide was unaffected by developers or acetone, which made it an ideal material for this application. The testing results of these heaters are presented in the next chapter.
CHAPTER 6. DEVICE TESTING AND RESULTS

6.1 Introduction

Device testing will enable to test the performance of the heaters fabricated on a polyimide insulating layer on top of the nickel GC columns. For the first part of the testing the heaters will try to ramp the GC column at typical heating rates for temperature programming from 5°C/sec – 50°C/sec with the aid of a PID controller. The second part of the testing is the thermal cycling experiment. This experiment will determine if the heaters fail after repeated heating and cooling cycles due to fatigue.

6.2 Feedback Control System

The control system plays an important part in temperature programming the gas chromatograph column. Figure 6.1 shows the closed loop control system for testing the in situ heaters on the nickel columns. In a closed loop system, also called a feedback control system, the output is continuously compared with the input (reference) and the difference between the two signals is used as the input to the controller. Here the reference is the temperature profile input into the controller and the output is the temperature reached by the GC column. This temperature was measured by the thermocouple and the measured output was fed back into the system. The difference between the measured output and the desired input is the error. Since the controller is actuated by the error signal, it will tend to drive the process to produce an output that will result in a smaller error.

6.3 Experimental Apparatus

The experimental apparatus is shown in Figure 6.2. It consisted of a PID profiling controller (Series SD - SD6R-HCCE-AARG, Watlow, Winona, MN), a DC power supply (SP-320-24, Mean Well, Taiwan), a solid state relay (SSR-100-20A-DC1, Watlow, Winona, MN)
and the heater. A K-type thermocouple (SA1-K-SC, Omega Engineering Inc., Stamford, CT) was attached to the bottom of the nickel GC column and was connected to the controller and also to a temperature data-logger (OMEGAETTE™, HH306, Omega Engineering Inc., Stamford, CT). The data from the data-logger was stored on a computer using Thermolog® computer-linking software (Omega Engineering Inc., Stamford, CT).

**6.3.1 The Controller**

The controller used for testing the heaters was a Series SD (SD6R-HCCE-AARG) PID profiling controller made by Watlow. The controller was capable of controlling ramp rates of up to 166.65°C/sec and also has an autotune function. The autotuning feature allowed the controller to measure the system response to determine effective settings for the PID control gains. When autotuning was initiated, the controller reverted to on-off control. The temperature must cross the autotune set point four times to complete the autotuning process (Figure 6.3). Once complete, the controller controls at the normal set point, using the new parameters⁹.

![Closed loop control system for heater testing.](https://www.watlow.com/literature/prodtechinfo/files/controllers/sd%20rev%20f%205-31-06.pdf)

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Figure 6.2 Experimental apparatus for device testing

Figure 6.3 The autotuning process in the PID controller

6.3.2 Temperature Sensor and Data Acquisition System

The temperature sensor used to record the temperature was a K-type thermocouple. The thermocouple was attached at the center of the bottom side of the nickel GC column. It was connected to the controller for feedback and also connected to a data-logger. The purpose of the data acquisition system was to record temperature profiles from the experiments for evaluation. The data-logger came with a computer linking program that sampled voltage every second.

6.3.3 Solid State Relay

The solid state relay acted as the interface between the controller, the power supply and the heater. The SSR-100-20A-DC1 solid state relay switched the dc current from the power supply ‘on’ or ‘off’ continuously based on the command from the controller. The wiring diagram for the experimental setup is shown in Figure 6.4.

![Figure 6.4 Wiring diagram for device testing.](image-url)
6.4 Experimental Results

6.4.1 Temperature Programs

With the proper wiring and connections in place, the controller was set to autotune and the power was turned on. Different ramp rates from 5°C/sec to 50°C/sec were selected with a soak (dwell) time of 30 seconds at the elevated temperature, for different runs. At the end of the dwell period, the power was turned off and the heater cooled to room temperature by natural convection.

The system response is plotted in Figure 6.5. The heater was able to heat the GC column at different rates - 5°C/sec, 15°C/sec, 30°C/sec, 40°C/sec and 50°C/sec. The heater was also able to maintain the column for 30 seconds at different elevated temperatures – 125°C, 160°C, 180°C, 220°C and 240°C. This showed that the heater could be used to achieve a variety of temperature programming profiles and it offered flexibility in selecting different ramp rates and soak temperatures.

![Figure 6.5](image)

**Figure 6.5** System response for different ramp rates
6.4.2 Thermal Cycling

The thermal cycling test was carried out on the heaters to see if they would undergo any visible material changes during repeated heating and cooling cycles. When the temperature reached a set point (~240°C), the power to the heater was turned off. When the column cooled down to about ~60°C, the power was turned back on again. This cycle was repeated 9 times. The temperature response is plotted in Figure 6.6. There was no visible damage to the heaters after the thermal cycling.

A test was also carried out to see if the heaters are able to withstand temperatures of up to 350°C. The heater was connected to the DC power supply directly, and the column temperature was monitored. The power supply was turned off after the temperature reached 350°C. This test was run two times. The temperature profile is plotted in Figure 6.7. The heater was able to reach 350°C without losing adhesion to the polyimide insulating layer. Steady state testing need to be carried out to see if the heaters can hold the column at 350°C for prolonged periods.

![Figure 6.6](image)

**Figure 6.6** Thermal cycling of the heater
6.5 Conclusions

The device testing enabled the evaluation of the performance of the microfabricated heaters. The heaters were able to heat the GC column at different rates (5°C/sec to 50°C/sec) with the aid of a PID controller. The heaters were also able to maintain the column for 30 seconds at different elevated temperatures. This showed that the heaters could be used to achieve a variety of temperature programming profiles and they offered flexibility in selecting different ramp rates and soak temperatures. The heaters and the polyimide layer showed no visible damage due to fatigue from thermal cycling. The heaters were also able to attain temperatures of up to 350°C without peeling off of the polyimide insulating layer. More thermal cycling tests need to be conducted to give confidence in their reliability.

Figure 6.7 Heaters able to withstand temperatures up to 350°C.
CHAPTER 7. CONCLUSIONS AND FUTURE WORK

7.1 Thermal Modeling and Stress Analysis

7.1.1 Conclusions

The goal of the thermal modeling was to estimate the power required to heat the 0.5m nickel micro GC at ~50°C/sec. Results showed that to achieve this, 70W of power was required with a polyimide insulating layer between the heater coil and the column. For a silicon insulating layer, the power required increased to 80W because of the increased thickness of the silicon substrate (600µm) compared to polyimide (10µm).

Lumped parameter analysis of the heater layout was carried out to understand the Joule heating process and to determine ideal materials for the heater coil. Since copper and nickel had lower electrical resistivities than nickel chrome, for a given power input the heating rate for copper and nickel was higher than that for nickel chrome.

Thermal stress analysis was carried out on the heater and the GC system using ANSYS. Thermal expansion mismatches were an important aspect of heater design. The CTEs of nickel (13.1 µm/m-°C) and silicon (2.49 µm/m-°C) have a large difference whereas the CTEs of nickel and polyimide (17 µm/m-°C) do not differ by very much. Polyimide turned out to be a better heater insulation material compared to silicon. Results from the stress analysis showed that the maximum stress induced in the heater coil (60 MPa), laid out on a polyimide layer, was less than the yield strength of nickel (180 MPa) at that elevated temperature thereby making the heater design fairly safe.

7.1.2 Future Work

For the thermal stress analysis, simple mesh refinement was done to give confidence in the results. The mesh refinement was carried out only in the heater coil section of the model. To
increase confidence in the results, mesh refinement should be performed over the entire system including the insulating layer and the micro GC column. The micro GC column can also be modeled with actual dimensions instead of approximating it to a metal block.

7.2 Fabrication

7.2.1 Conclusions

Nickel and copper heaters were microfabricated on a polyimide insulating layer on top of the nickel GC columns. After curing, the polyimide resists etching by developers or acetone which makes is an ideal material for this application, since downstream chemical processing will not affect it.

7.2.2 Future Work

The heaters were fabricated individually on the micro GCs. It was possible to make multiple heaters on multiple GCs simultaneously by making heater masks corresponding to the GC column masks and aligning them during UV exposure. The heaters can then be electroplated together in the plating bath. Heaters can also be patterned on both sides of the column to obtain faster heating rates.

7.3 Device Testing

7.3.1 Conclusions

The device testing enabled the evaluation of the performance of the microfabricated heaters. The heaters were able to heat the GC column at different rates (5°C/sec to 50°C/sec) with the aid of a PID controller. The heaters were also able to maintain the column for 30 seconds at different elevated temperatures. This showed that the heaters could be used to achieve a variety of temperature programming profiles and they offered flexibility in selecting different ramp rates and soak temperatures. Thermal cycling experiments, where the heater was repeatedly
heated to ~240°C and cooled down to ~60°C over nine cycles, showed no visible fatigue damage on the heaters or the polyimide. The heaters were also able to attain temperatures of up to 350°C without peeling off of the polyimide insulating layer.

### 7.3.2 Future Work

The nickel micro GC columns have to be polished from their present thickness to the minimum possible to achieve least weight. This is important because the thermal capacitance is an important factor in the ramp rates that can be achieved for a given power input. With lower thermal capacitance, the power requirement comes down.

More thermal cycling experiments have to be conducted on the heaters to give more confidence in their reliability.

The GC column with the heater patterned on top of it can be linked to the carrier gas input, flow controller, flame ionization detector and a pulse width modulating controller to check temperature programmed separations.
REFERENCES


APPENDIX A: MESH REFINEMENT IN THE FE MODEL

Figure A.1 Coarse Mesh

Figure A.2 Medium Mesh
Table A.1 Mesh sizes for mesh refinement

<table>
<thead>
<tr>
<th>Model section</th>
<th>Element edge size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GC column</td>
<td>500</td>
</tr>
<tr>
<td>Insulating layer</td>
<td>500</td>
</tr>
<tr>
<td>Heater coil (Coarse)</td>
<td>500</td>
</tr>
<tr>
<td>Heater coil (Medium)</td>
<td>250</td>
</tr>
<tr>
<td>Heater coil (Fine)</td>
<td>125</td>
</tr>
</tbody>
</table>

Figure A.3 Fine Mesh
APPENDIX B: FE MODELS FOR THE INLET CHANNEL HEATING CASE

Figure B.1 Temperature distribution at the end of 2 seconds for a power input of 60 W at the inlet channel

Figure B.2 Temperature distribution at the end of 10 seconds for a power input of 60 W at the inlet channel
Figure B.2 Temperature distribution at the end of 15 seconds for a power input of 60 W at the inlet channel

Figure B.2 Temperature distribution at the end of 20 seconds for a power input of 60 W at the inlet channel
APPENDIX C: PHOTOMASK LAYOUT

Figure B.1 Optical mask layout for the heaters

Table B.1 Characteristic dimensions of heaters

<table>
<thead>
<tr>
<th>Item</th>
<th>Heater Pattern 1 Number/Dimension</th>
<th>Heater Pattern 2 Number/Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of vertical strips</td>
<td>10</td>
<td>8</td>
</tr>
<tr>
<td>Width of each strip</td>
<td>500 µm</td>
<td>700 µm</td>
</tr>
<tr>
<td>Gap between strips</td>
<td>333.33 µm</td>
<td>342.86 µm</td>
</tr>
<tr>
<td>Length of the heater coil</td>
<td>14.3 cm</td>
<td>14.3 cm</td>
</tr>
</tbody>
</table>
APPENDIX D: HEATERS ON SILICON AND KAPTON SUBSTRATES

Figure D.1 Heaters on a silicon wafer

Figure D.2 Heaters on a Kapton film
VITA

Chetan Ramesh was born in Bangalore, India on October 14th 1981. He received his Bachelor of Engineering Degree in Mechanical Engineering from B.M.S. College of Engineering, Bangalore, India, in June of 2003. He joined the graduate program in Mechanical Engineering at Louisiana State University, Baton Rouge, LA in the Fall of 2003. Since then he has been working as a member of the Micro Systems Engineering Team (µSET) Lab. He expects to receive the degree of Master of Science in Mechanical Engineering to be awarded during the LSU commencement ceremonies in December 2006. He has received a job offer from Schlumberger, and will be moving to Houston to being work from January 2007.