SEALING AND LAMINATION OF MICROFLUIDIC DEVICES

by

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Presented to the Faculty of the Graduate School of

The University of Texas at Arlington in Partial Fulfillment

of the Requirements

for the Degree of

MASTER OF SCIENCE IN INDUSTRIAL AND MANUFACTURING SYSTEMS ENGINEERING

THE UNIVERSITY OF TEXAS AT ARLINGTON

August 2008

ACKNOWLEDGEMENTS

I would like to show my appreciation of the encouragement of my wife Lauren, my family and the faculty at the University of Texas at Arlington. In particular I want to acknowledge the leadership of Dr. Billo, the guidance of Dr. Priest and the direction of Dr. Dennis over the past year. I would also like to acknowledge the skills of Howard and Jimmy and the flexibility of Dr. Huff who never turned down a question. Finally, I want to thank the administration Kim, Julie, Dr. Liles and Dr. Imrhan for helping smooth out the waves I like to cause.

July 17, 2008

ABSTRACT

SEALING AND LAMINATION OF MICROFLUIDIC DEVICES

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The University of Texas at Arlington, 2008

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A difficult problem facing microfluidic manufacturing of devices remains that often material requirements force device designers to choose materials that are difficult to bond, seal or enclose. Examples of these materials include the Polyolefin family such as HDPE (High Density Polyethylene), PP (Polypropylene) or high performance polymers such as PEEK (Polyetheretherketone), Acetal or Polyimide. The problem arises from the chemical resistance of the materials making them ideal for processing corrosive chemicals but the same property reduces the effectiveness of laminates, surface bonding, surface coatings and solvents.

To address this problem a methodology for sealing devices made of these materials is explored and results related to laminating similar substrates is presented. This is followed by suggestions for further study and potential improvements.

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CHAPTER 1

INTRODUCTION

1.1 Problem Definition

Mass manufacturing microfluidic devices faces many challenges. One such challenge is material selection. Selecting a suitable material is difficult because process requirements limit the choice of materials to materials that are difficult to bond, seal or enclose. These materials can also be difficult to manufacture with or be cost prohibitive. Examples of these materials include the Polyolefin family such as HDPE (High Density Polyethylene), PP (Polypropylene) or high performance polymers such as PEEK (Polyetheretherketone), Acetal or Polyimide.

The problem arises from the chemical resistance of the materials making them ideal for processing corrosive chemicals but the same property reduces the effectiveness of laminates, surface bonding, surface coatings and solvents for sealing the devices. The need to seal devices arises from the manufacturing process for producing the device.

Microfluidic devices are produced in several ways which are determined by the feature sizes (Holger 2000). There are many technologies and process for producing microfluidic devices. Devices with features smaller than 100 microns are usually produced using etching, laser machining, hot embossing and other traditional MEMS methods. Devices with larger features are usually micro-milled, Hot Pressed or Micro Injection molded. Some process can do both feature types depending on the application. Generally regardless of the method the resulting product looks similar to Fig. 1.1. That is a flat substrate with features (channels, wells, ports) inlayed but the geometry size, layout and feature type may vary.

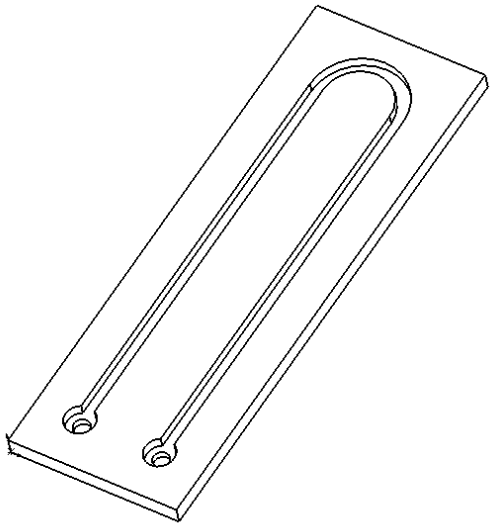

Figure 1.1. Sample microfluidic device: open channels

However these features remain open and must be sealed to create full channel structure for liquids to flow through such as Fig. 1.2 which shows the same device as above but covered and with the channels exposed.

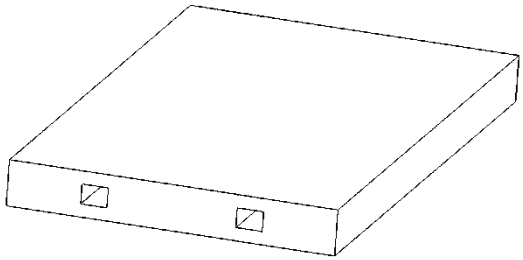

Figure 1.2. Sample microfluidic device: closed channels

1.2 Aim of Thesis

To address this problem a methodology for sealing devices made of these materials is explored and results related to laminating similar substrates is presented. This is followed by suggestions for further study and potential improvements.

1.3 Task Description

The hypothesis behind this study is to confirm that planer lamination of similar substrates made of polymer HDPE microfluidic devices is a useable method for their manufacture. Preliminary experiments and literature suggests that the main factors related to lamination are time, temperature and pressure. The experiments following are to confirm the importance of each factor, find other important factors and optimize the relation of the factors to create a working process. The purpose is to validate the hypothesis or provide some data to contribute to the heuristic knowledge and theory of lamination of similar substrates.

1.4 Experimental and Design Issues

While the experiments should yield information about the physics and properties of lamination there are several impacts on the microfluidic designer and manufacturer that need to be addressed in process development (Neter 2005), namely:

1. Improved process yield
2. Reduced process variability
3. Reduced development time
4. Reduced cost
5. Evaluation and comparison of basic configurations of design
6. Evaluation of various materials
7. Selection of design parameters so that the product will work well under a wide variety of field conditions (or so that the design will be robust)
8. Determination of key product design parameters that affect product performance

The eight impacts will be addressed as information about their relation to laminar design is realized.

1.4.1 Intuitive Advantages and Disadvantages of Lamination Leading the Study

The need for creating a device that is made of a solid piece of a single material type in order to take advantage of the materials chemical resistance and surface characteristics is leading this study. From this a survey of possible methods for creating a sealed device was done and ideas were generated about possible advantages and disadvantages of the proposed method.

Inherent advantages of planar lamination are that the entire surface of the devices is sealed such that there is no longer a distinction from top and bottom; rather it becomes a solid flexible device of uniform material with a delaminating strength greater than the material. Other advantages are that if done in a chemically resistant material such as PP or HDPE any material can be used as a delivery mechanism without the potential for weakening the bond unlike those joined by adhesives, the device can be tested individually easily, joined with other devices, handle high pressures (>0.86 MPa) and have moderate working temperatures in the range of 60C-120C (Depending on the material).

Disadvantages of the process such as the heating of the entire device, potential need for annealing to relieve internal stresses and possible variation between laminated parts also need to be considered and some discussion will be given as to the impact and methods for dealing with these disadvantages.

1.5 Thesis Organization

The discussion of sealing methods is introduced first by looking at background material related to the topic. This is followed by the methods used in the experiments related to sealing microfluidic channels and then results are provided with appropriate discussion. From the results and discussion conclusions are drawn and presented with a summary of the impact of the findings.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

A summary and review of the theory and methods that have already been published will provide a foundation for the discussions to follow. Several methods of sealing micro devices will be discussed briefly.

2.2 Lamination

Traditional lamination using adhesives is a well known process that comes in two flavors. The variation depends on the substrate, the adhesive and the location of the adhesive. Either the adhesive will be located on the substrate as a thin layer that melts when heat is applied. Or the adhesive is a thin paper like layer that is placed in between substrates when heat is applied.

Many technical problems have been tackled by industry such as Teflon coated “pockets” for holding laminating materials that keep the rollers or platens free from debris and provide a non stick surface so that the substrate or excess adhesive do not stick to the pouch. Another example is the contribution of the mechanical design for laminating machines. Specialized platen laminators use a floating plate design which allow the top and bottom plate of the laminator to press across the substrate without sliding and adjust to provide even pressure to various substrate thicknesses and sizes consistently. Traditional office lamination can be used to temporarily seal a device but degrades quickly.

2.3 Chemical Bonding

Chemical Bonding is useful for certain polymers such as PMMA (Polymethyl methacrylate), which can be joined using a solvent that softens the surface of the substrate

allowing a second surface to be joined to it. Other chemical bonding relies on a chemical reaction to form a bond similar to epoxies. The drawback of chemical bonding occurs when the material is very chemically resistant such as HDPE which has no room temperature solvent. Smooth surfaces also have trouble with many adhesives because there are few points for adhesion, scored or roughened surfaces allow the adhesive areas to flow into and grip (Bharat 1997).

2.4 Heat Staking

Heat Staking is a time intensive process that joins two parts together with the use of "stakes". A schematic of Heat Staking is shown in Fig. 2.1.

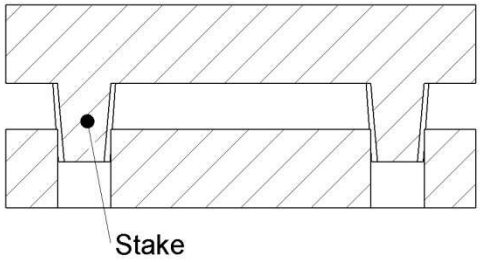

Figure 2.1. Heat staking diagram

One plate will generally have a protrusion which will snap/fill a port in another part. By heating through the stake rather than across the whole part surface thermal deformation is minimized (Eileen 2007). The heated stake will fuse at low temperatures over a long period of time to the part. Constant pressure is applied between both plates to ensure a tight fit.

2.5 U8 and UV Cures

Ultra Violet light curing materials such as U8 are often used both in etching and in adhesion. The principles are similar to gap filling adhesives except the agent is not oxygen but UV light to harden the adhesive (Abgrall 2006). This method also works for smooth surfaces such as silicon or glass by creating an air tight seal and using the pressure differential to make removal difficult. This type of behavior can also be seen in Silicone on glass.

2.6 Microwave Sealing

Microwave sealing of microfluidic devices is an experimental method that shows promise in selectively heating two polymer sheets quickly with little thermal deformation. Work done by Yussaf (2007) has demonstrated the applicability of this method to several polymers often used in Microfluidics such as PMMA and PDMS (Polydimethylsiloxane).

The essentials of the process include applying a mask of a conductive polymer between the lamina and cover plate and applying even pressure across the surface. This layered device is then subjected to microwave radiation. The non conductive polymers allow the waves to pass through with little thermal impact aside from the small amount of water absorbed through the atmosphere while the conductive polymer quickly heats above the melting point of the base polymer allowing a selective amount of material directly around the mask to flow and join with the face plate.

2.7 Bolting/Pressure Sealing

Fasteners are a traditional method for sealing using screws, bolts and rivets to apply a load at a point and distribute it using a gasket, substrate or washer. In micro devices gasketing is difficult without creating space between substrates or blocking channels with displaced gasketing material. This method works well with some variations or in conjunction with lamination. Tight tolerances must be kept in thickness variation of a device or else sealing is difficult and methods will be shown to compensate for pressure distribution variation.

2.8 Laser Welding

Laser welding uses a wide beam of high energy to melt two thin surfaces together. It is often used in the automotive industry and works well with a wide variety of polymers such as PVC but laser working of soft polymers such as HDPE is limited. Another drawback for microfluidic applications is the requirement of fusion points several mm in width as shown in Fig. 2.2.

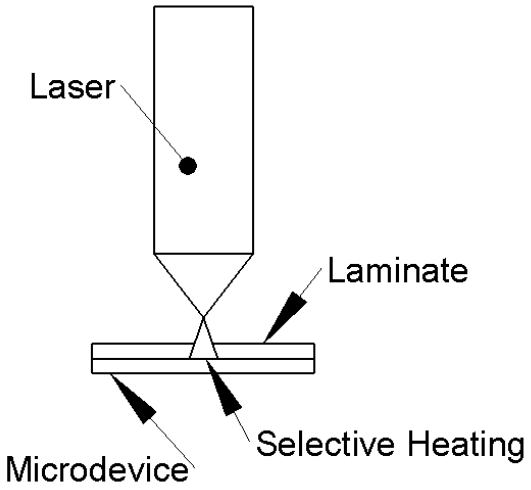

Figure 2.2. Laser welding schematic

2.9 Plastic Welding

Plastic welding is usually done in three varieties hot air welding, fusion welding and vibration welding. Vibration welding applies a horizontal oscillation between two substrates locally bonding and heating the parts at the friction point. Unique part design is required and 2-4 mm joining surfaces are required. The part must be augmented to allow the walls of the micro device to have fusion points so that a limited area of friction meets the second plate. Hot air welding locally melts the plastic with heated air allowing a part to be inserted but is difficult to control.

2.10 Summary of Literature Review

Each method discussed has advantages and disadvantages related to the specified application. The feature size, tight tolerances and material requirements limit highly corrosive micro applications to a limited sub set of methods such as pressure sealing, heat staking, micro sealing and lamination. This study focuses on pressure sealing and lamination because of the relative ease of manufacture without the need for highly specialized machinery but other methods are briefly explored.

CHAPTER 3

METHEDOLOGY

3.1 Introduction

It is apparent there are many options for sealing micro devices together but for HDPE few options make sense. Common methods such as laser welding, vibration, chemical/adhesive, staking and traditional plastic joining methods each have inherent disadvantages in Microfluidics using HDPE. Microwave sealing methods are currently being pursued but bolting and laminating are both simple to process and show promise to meet each of the disadvantages posed by other methods. For this reason these two methods are explored in greater detail. Other experiments were performed to evaluate potential sealing methods and each is discussed. The experimental and analysis methods used are discussed in this section.

3.2 Initial Sealing Methods Investigated

The methods in this section have been briefly described previously. It was stated that each had inherent advantages and disadvantages that precluded it from being used in the bulk of this study. The methods used to evaluate each sealing mechanism and the advantages and disadvantages found are discussed here.

3.2.1 Laser Sealing

To evaluate laser sealing for this application experiments were done using a 90 Watt CO2 laser with variable power, cutting speed and pulses/inch (ppi). In general laser cutting/welding of any material requires that the wavelength and pulse type of the laser be matched with the materials molecular structure. The CO2 laser had a wavelength approximately equal to 1200 nm which is “longer” in terms of the wavelength spectrum for lasers. The spot size of the laser was approximately 127 microns with a radial shape and an

energy density fall off similar to the curve shown in Fig. 3.1 which is Gaussian. The lens is focused on the top of the part by moving the laser table in the z direction up or down depending on the thickness of your material. The focal length of this lens is 50.8 mm.

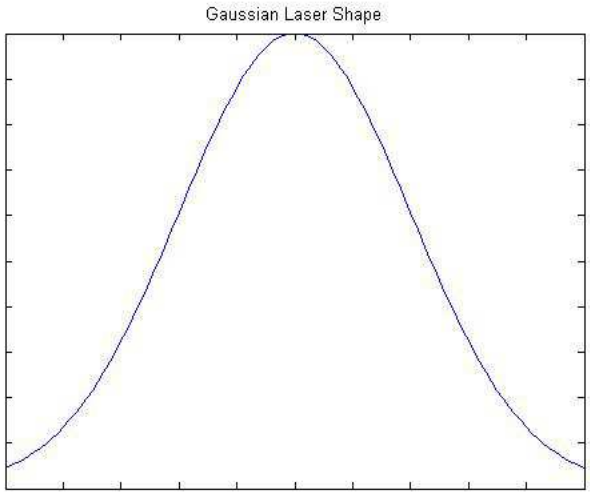

Figure 3.1. Approximate energy distribution of CO2 laser

Soft materials like HDPE have already been shown to not machine well on a 1200 nm laser system but laser welding requires only that the two materials stay in contact and that sufficient heat is transferred through both materials to allow them to fuse by the weight of one substrate on top of another. The design of our micro devices required several constraints. The first was that the substrate was HDPE, the second was that the one layer be 1000 microns thick and the other 4000 microns thick. In addition to material and thickness constraints it is not uncommon for micro device designers to have features that require a hermetic seal within 750 microns of each other.

A rectangular micro device measuring 127 mm by 44.5 mm was used to experiment. A layer of laminate made of HDPE 0.25 mm thick was placed on top of the device and the path around the channels was programmed into the laser using Auto Cad. This was done using the

settings shown in Table 3.1. Each test was then examined for defects, tensile and shear strength.

Table 3.1 Laser experiment settings

Num.	Power (% of 90 Watts)	Speed (% of Max)	PPI (Pulses/Inch)	Focus
1	30	5	300	0
2	30	5	300	+0.01
3	30	5	300	+0.02
4	30	5	300	+0.03
5	30	5	300	+0.04

The parameters power, speed and PPI were held constant and the focus was changed to melt rather than cut the plastic. The basis for the settings given for Power, Speed and PPI were based off a process parameterization done for cutting HDPE which as stated does not cut well on a laser system in general.

3.2.2 Vibration

No vibration experiments were performed. However industry experts did provide information regarding the benefits and disadvantages. These will be included for further study or general information in the results section.

3.2.3 Chemical Bonding

It is well known that HDPE and other polyolefin's are difficult to use with adhesives. HDPE has no known room temperature chemical solvent which means any adhesion will be surface bonding only and will require significant scoring of the material. HDPE can dissolve in certain chemical solvents but only at elevated temperatures above 60 C. One commercially available adhesive was recommended from several sources including Loctite.

An experiment was performed by applying Cyanoacrylate to the laminate substrate with a dropper and using a spin coater to provide a nearly uniform surface coating. The coated laminate was then placed on the micro device to bond and seal it. This was performed three times and the force required to remove the laminate was below the threshold of the measuring device. The direction of the delimitation force is shown in Fig. 3.2.

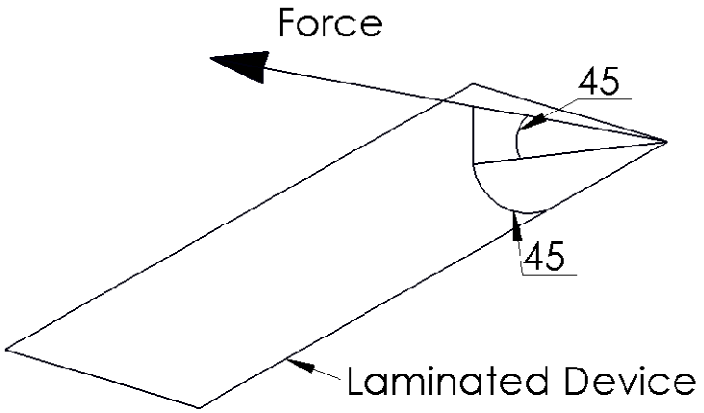

Figure 3.2. Direction of force for delimitation test

The results and discussion of these experiments are provided in the appropriate section. No other experiments were performed using adhesives.

3.3 Pressure Sealing

Pressure sealing of a micro device can take on an infinite number of forms. However two major ideas were explored. The first was the concept of planer force across a device and the second was the user of fasteners through the device. The schematic of the two methods are shown in Fig. 3.3 and Fig. 3.4 respectively. It was decided after initial experimentation and historical review that the important parameters for this methods were the pressure method used, amount of pressure, distance between pressure points, substrate properties, support material properties, tolerance of the micro device (thickness variation), gasketing materials and pressure distribution.

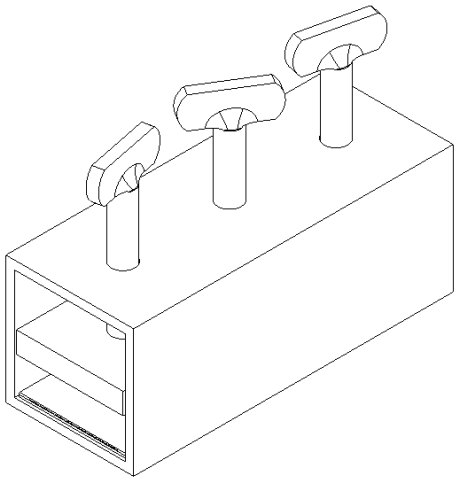

Figure 3.3. Planar sealing schematic

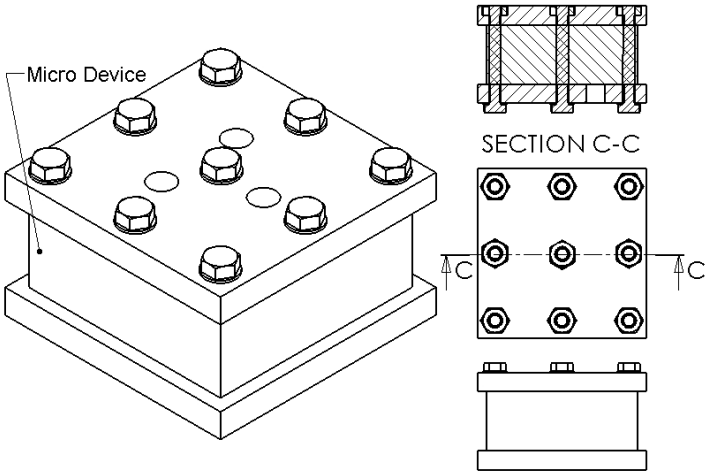

Figure 3.4. Pressure sealed schematic

3.3.1 Discussion of Application Experiment

The first fixture using planar force applied pressure across the entire surface of the part. Ensuring a seal was difficult and so attempts were made to distribute the pressure in an even manner. This was done by identifying several materials that would distribute force. The ideal material would realign itself to the force distribution across the plate with a viscosity like water

(8.90×10^{-4} Pa·s at 25 C) while remaining bound to the plate. Rather intuitively the best idea seemed a gel or grease. However, many materials were attempted as shown in Table 3.2.

Table 3.2 Sealing/Gasketing materials

Material	Thickness (mm)
Viton Rubber	3.175
Lining Wax	3.175
HDPE Foam	3.175
HDPE Film	1.52
Buna-N Foam	6.35
Silicone Gel	3.175
Silicone Rubber	1.52
Neoprene Foam	6.35
Grease w/ petroleum wax filler	0.254

The second fixture developed relied on bolt fasteners rather than planer pressure using screws pressed against a plate. This fixture was made of a bottom and top plate with bolts that passed through the plate and the micro device as shown in Fig. 3.4. Three materials were used to make the plates including polycarbonate, acetal and aluminum. Washers were used to help distribute the pressure evenly. Bolts were first placed 50 mm apart and later 25 mm apart. The results of these experiments will be discussed in the following chapter.

Two major factors determined the appropriateness of this method: leaking and repeatability of assembly measured as pressure at a constant flow rate.

3.4 Lamination Sealing

The majority of experimentation relates to lamination of two similar planer substrates with micro features embossed on one of the devices. The basic experimental process will be to

define the important factors, discuss the apparatus, design of experiments, metrics of success and then provide the results of the experiment and observations.

3.4.1 Definition and Variables

Potential factors in the experiment are shown in Fig. 3.5 by means of an Ishakawa diagram. The diagram demonstrates that there are too many factors to include in a formal study but each factor does need to be considered and a discussion for how that factor was controlled or previous work related to that factor will be give.

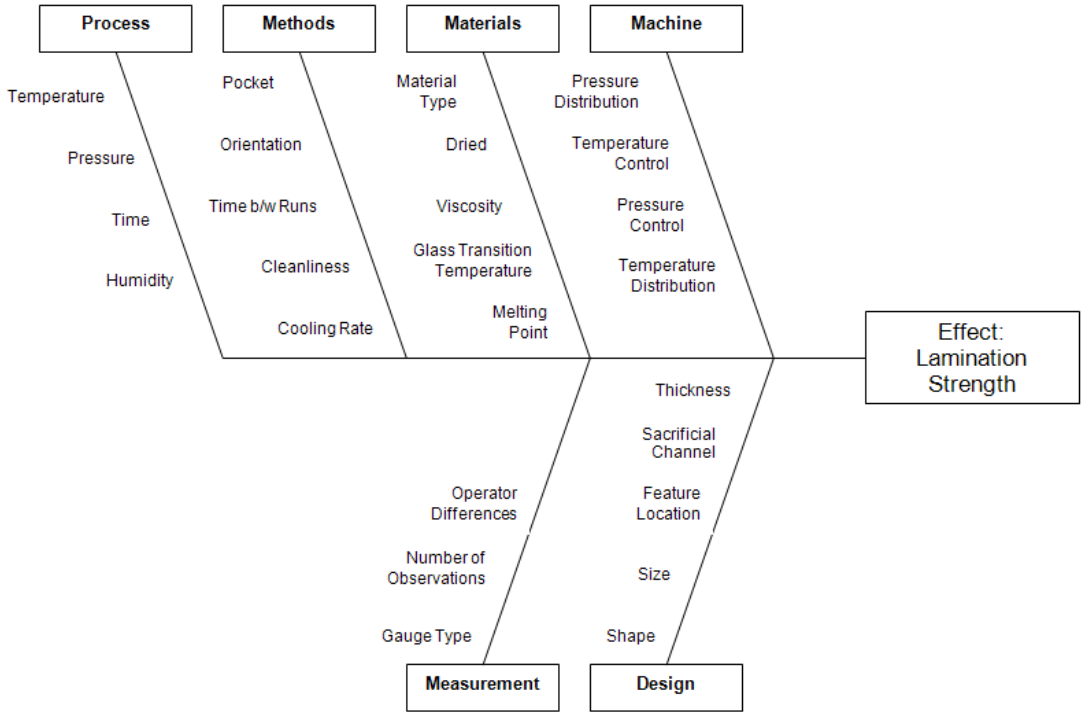

Figure 3.5. Lamination factor analysis

3.4.2 Machine Characterization

As part of the experimental process it is important to understand the machines you are working with. Four machines were used in developing a lamination process. The first machine was the same aluminum fixture used for sealing testing. The second machine was a rolling,

heated laminator by Banner American. The third machine was a Carver 30 ton laboratory press. The final machine was a light platen laminator produced by Bienfang.

Each machine provided one part of the puzzle but the Bienfang laminator was used for the majority of process development. The Carver press simply used to much power but was well instrumented, the aluminum fixture was difficult to instrument and had too much thermal mass and the rolling laminator had inherent disadvantages related to the rolling mechanisms which will be discussed in further detail. While some information is given on each machine a full machine characterization is only done on the Bienfang laminator since the final results were produced on it.

The light laminating press used for this process is a Bienfang platen laminator with a 544 kg maximum force across the surface of the platens which measure 470 mm by 585 mm. The maximum temperature of the press is 180 C. The top plate is free floating and the pressure applied can be adjusted by using two torque screws. Pressure is applied by mechanical force using a lever system. Pressure deviation across the surface attempts to be buffered by a foam pad in the bottom plate. Only one side of the press is heated. Measurement of the press is divided into thermal and pressure characteristics.

3.4.2.1 Bienfang Thermal Characteristics

Measurements of the press were taken in order to evaluate its characteristics. The important measurements were deemed to be temperature variation across the surface of the plate, temperature ramp up and cycle, and pressure distribution across the surface of the part. Temperature variation was measured by using thermocouples inserted into an aluminum plate measuring 30.5 cm by 30.5 cm. Fourteen thermocouples were placed inside the aluminum plate at locations shown in Fig. 3.6.

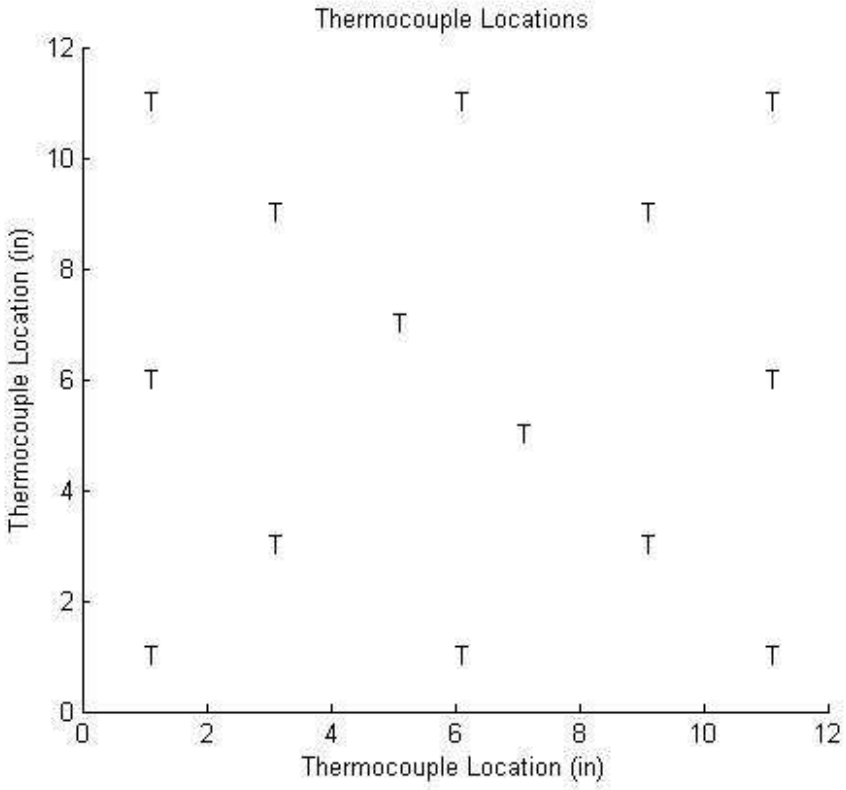

Figure 3.6. Thermocouple locations for heat distribution characterization

Measurements from the thermocouples were measured every 10 seconds in order to evaluate the ramp up and cycle range for temperatures. From experience thermocouple 11 measures several degrees higher than the other thermocouples and must be considered during analysis.

The temperature between thermocouples was interpolated and the measurement plate was placed in the center of the press where lamination occurred. Fig. 3.7 shows the temperature distribution across the platen while closed after the machine was allowed to heat fully and cycle for 180 minutes.

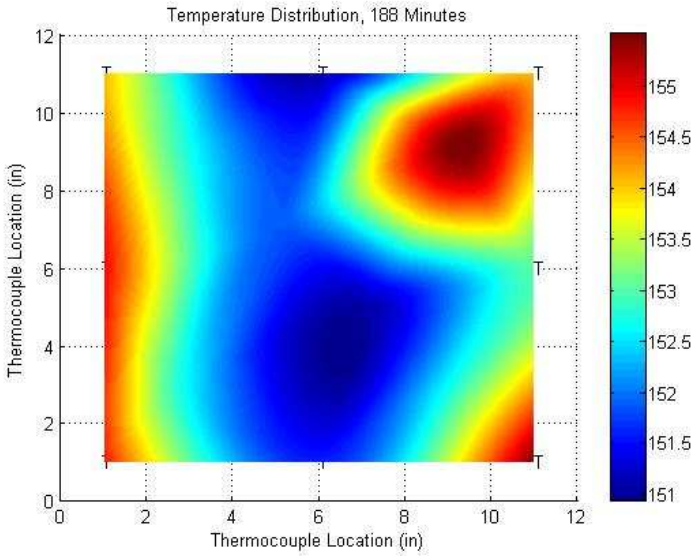

Figure 3.7. Bienfang platen temperature distribution after heating

This data was compiled into a dynamic output using MATLAB so that the temperature variation over time could be visualized as varying heights. A sample of this animation is shown in Fig. 3.8 at a point relaying the same information as Fig. 3.7.

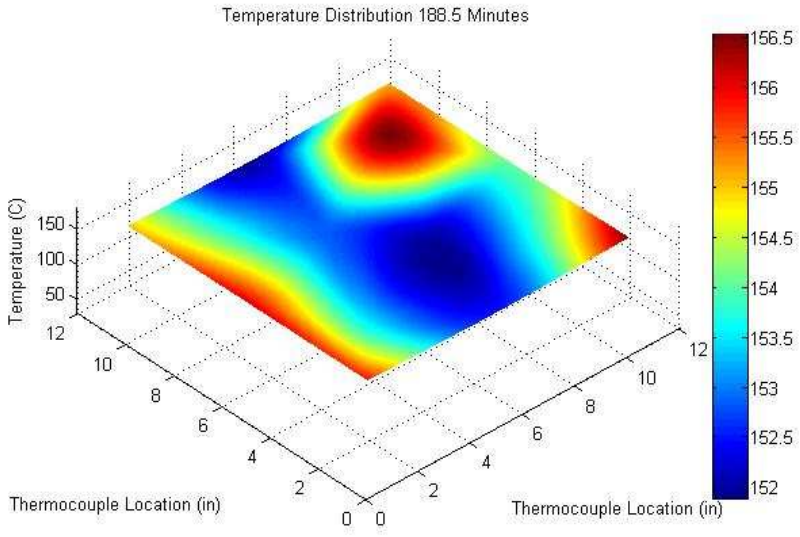

Figure 3.8. Temperature distribution animation over 188.5 minutes

From Fig. 3.9 it can be seen that the machine takes 100 minutes to reach a steady state of between 152 C and 157 C. It can also be seen that the temperature cycle slows down as it reaches temperature rather than over shooting the temperature and having to drop the temperature back to the correct level.

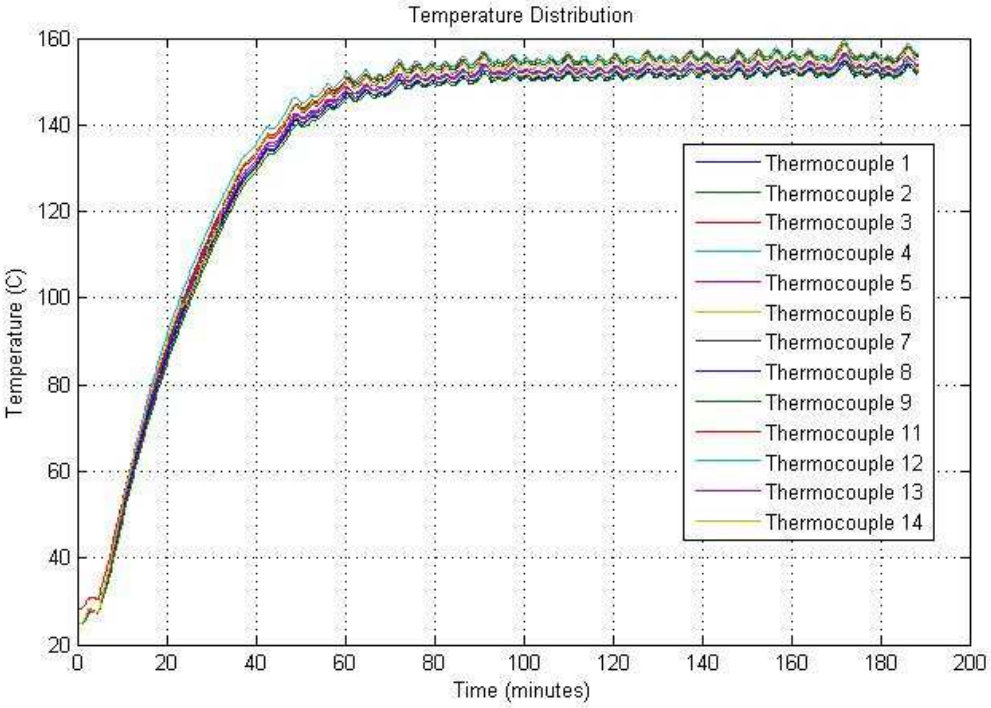

Figure 3.9. Temperature ramp up and cycling

Fig. 3.10 shows that after reaching steady state the machine cycles thermally every 3.5 minutes within a 5 degree trigger. An important inference from this plot is that because of the cycle of heating rather than a constant applied heat the plat never fully settles and the same region on the surface always lags behind a warmer region.

In terms of production the result is that at least 100 minutes should be allowed for heating the machine before initial use. This should reduce the number of defects caused by insufficient heating.

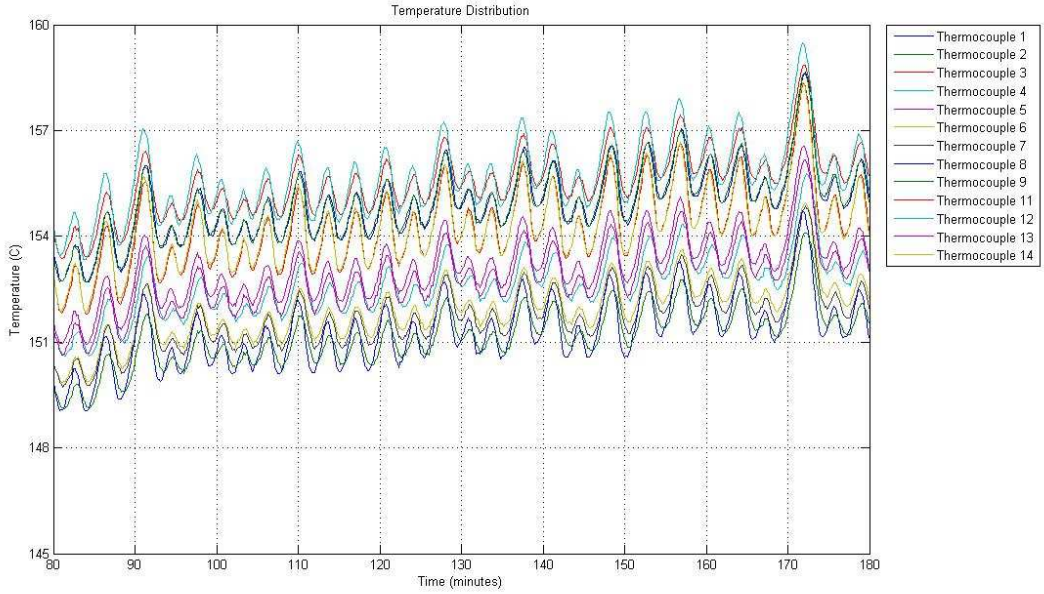

Figure 3.10. Temperature cycling by thermocouple

Fig. 3.11 illustrates the temperature deviation across the platen surface as a function of time. From this diagram it is seen that after reaching steady state the variation across the plate is approximated around 5 C with small perturbations above and below that point. It is also seen that variation is highest during ramp up and reaches steady state at 100 minutes similar to the temperature of the platen.

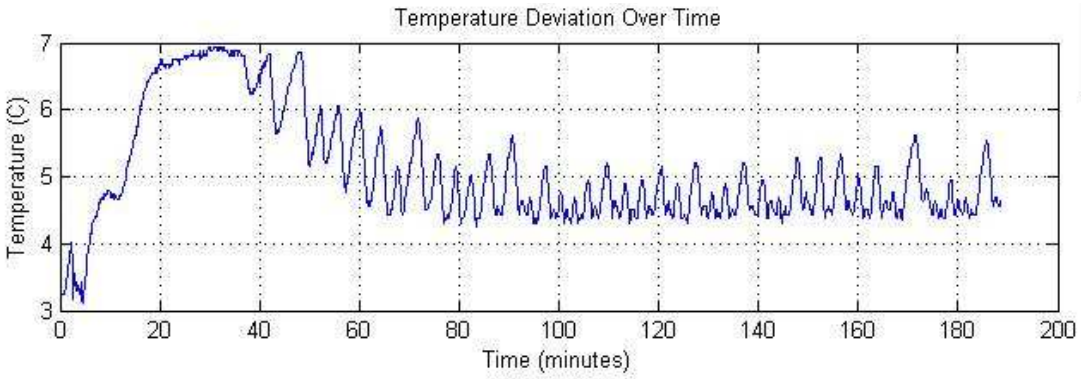

Figure 3.11. Temperature deviation over time

From this a cycle time will try to be achieved of 4 minutes to allow the press to reach nearly the same temperature state in between laminations.

3.4.2.2 Bienfang Pressure Characteristics

A sample part was placed in the press to evaluate the pressure distribution across the part. The result of the press is shown in Fig. 3.12 and show that the edges of the part and edges of features receive the highest level of pressure while feature less flat areas receive less pressure.

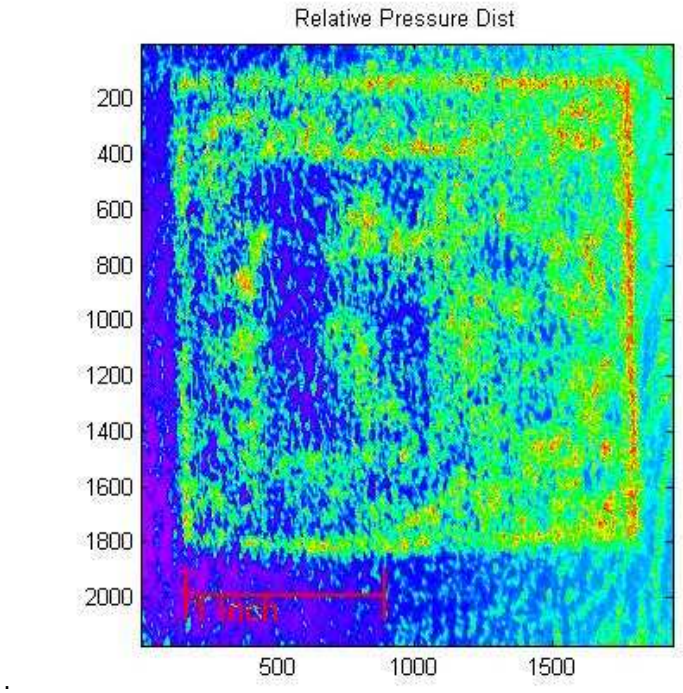

Figure 3.12. Pressure distribution across part surface

While this is the case measurement of lamination across a square part at 9 points each 133 mm from the other showed that the variation although visible is limited. These measurements were taken using a digital point sensor device (ELF) and the pressure variation between was interpolated for ease of evaluation. The results of this are shown in Fig. 3.13.

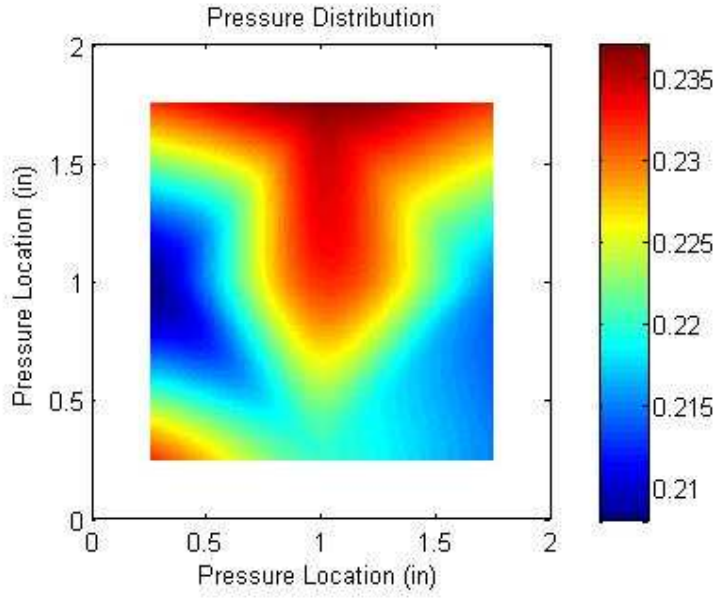

Figure 3.13. Pressure distribution measurement map (N)

Finally the percentage of total pressure applied across the part was measured over time to understand if there were any points during the process in which pressure would peak high or low. This is shown in Fig. 3.14 and ensures that the mechanism used to apply pressure to the part is smooth and responsive.

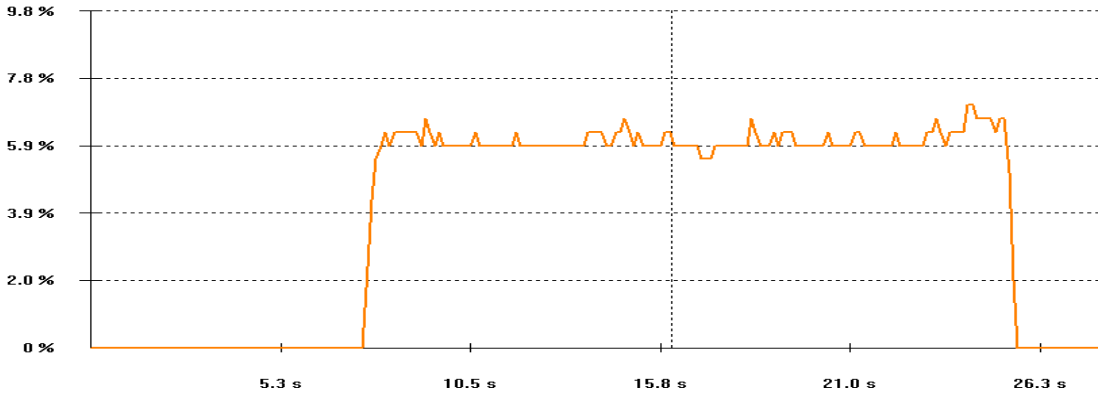

Figure 3.14. Pressure across part over time

There is a small increase in pressure when the force is released but otherwise noise in the sensor readings are the only noticeable pattern. It should also be noted how cleanly pressure is applied and removed.

The interpretation is that the temperature variation between any two points on the plate is 4 C or less and that the variation in pressure across the plate is 0.00011 and the mean is 0.225. From this a CV of the pressure distribution is approximately 0.0466 which can be considered an extremely low variation process. The conclusion drawn is that this machine is suitable in terms of variation of performance to experiment lamination on because the processing performance is within acceptable range.

3.4.3 Discussion of Experimental Design

Several preliminary experiments were performed to develop a sense of what factors were important and narrow the search parameters for processes optimization. The experiments were divided into phases. The first phase examined proof of principle, followed by an extreme value band experimental analysis which resulted in a narrower band of possible candidates. A follow up experiment was performed within this band to settle on the most important factors parameters. After an optimized set of process parameters were chosen, these were held constant and acted as a baseline compared to changes made on individual factors.

3.4.3.1 Proof of Principle

The first method used an aluminum fixture and a hot plate to fuse the material, the second used an industrial press, the third used hot rolling lamination and finally, the fourth used a light industrial press. Each of these will be discussed in turn.

The process of lamination went through several steps. In order to understand the fundamental nature of the process preliminary experiments were performed using an aluminum fixture shown in Fig. 3.15.

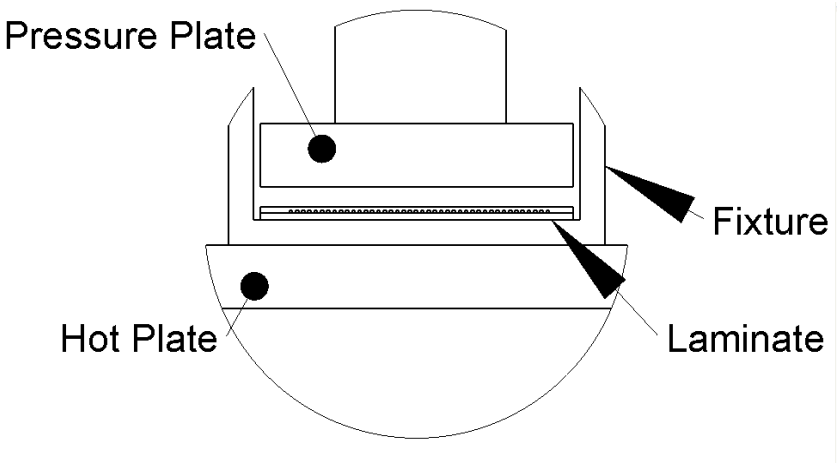

Figure 3.15. Aluminum lamination fixture

This fixture provided three points of vertical force with no feedback across a rectangular device measuring 50.8 mm x 152.4 mm x 1 mm made of HDPE. A laminate sheet of HDPE measuring 50.8 mm x 152.4 mm x 1 mm was placed under the device. Pressure was applied across the surface and the device was positioned so that the edge of the channels could be observed. Heat was introduced on one side using a heating plate starting at 115 C and incrementally increasing the temperature 10 C up to 205C. Observations were taken during each temperature change and pressure was cycled at each temperature increment from 'light' to 'heavy' over 10 minutes. Visual inspection was used to decide if lamination had occurred and the process could be ended. The first experiment was performed across the entire range and a second was performed at an approximation of the correct settings being 'light' pressure, 155 C and 10 minutes. Note that ten minutes was required because of the extra thermal mass of the fixture to allow the heat transfer time to occur and settle.

The results of this experiment were measured in two ways. The first was by manually attempting to delaminate the material, the second was by measuring the cross sectional area using a Confocal microscope. The results of this experiment were used to develop the experimental design of the final experiment.

3.4.3.2 Experiment 2 – Carver Press

A second experiment was performed using the same concept with a Carver bench top press to test extreme conditions / outer limits of pressure. The carver first applied high pressure using 400 pascal for 10 seconds at 160 C. The laminate sheet was 1 mm thick and the part was 5 cm by 15.25 cm by 1 mm thick. Channels were embossed into the surface 500 microns deep and 500 microns wide at intervals of 500 microns. A diagram of the carver lamination method is shown in Fig. 3.16.

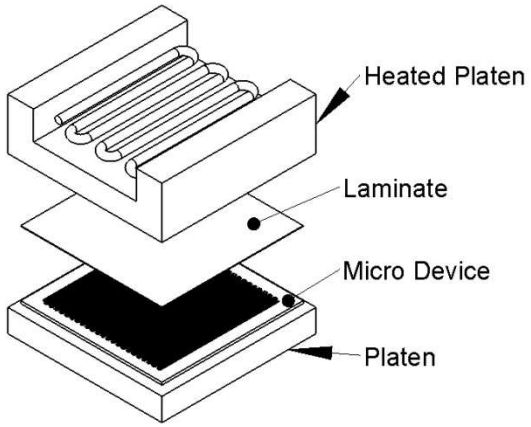

Figure 3.16. Carver lamination schematic

The opposite extreme pressure was tested on the carver press by using no pressure across the part. Heating and time were allowed to be the same as the previous experiment. Results are discussed in the next chapter.

3.4.3.3 Experiment 3 – Rolling Lamination

A lamination machine was purchased with the ability to control rolling speed, temperature and pressure. Pressure was controlled by the use of springs tightened to various levels and the pressure was measured using the ELF pressure sensor. Temperature was recorded using a standard thermocouple and speed was measured as a % of motor power.

Several experiments were performed to evaluate the plausibility of a rolling laminator versus a platen laminator design. The schematic for the rolling laminator is shown in Fig. 3.17.

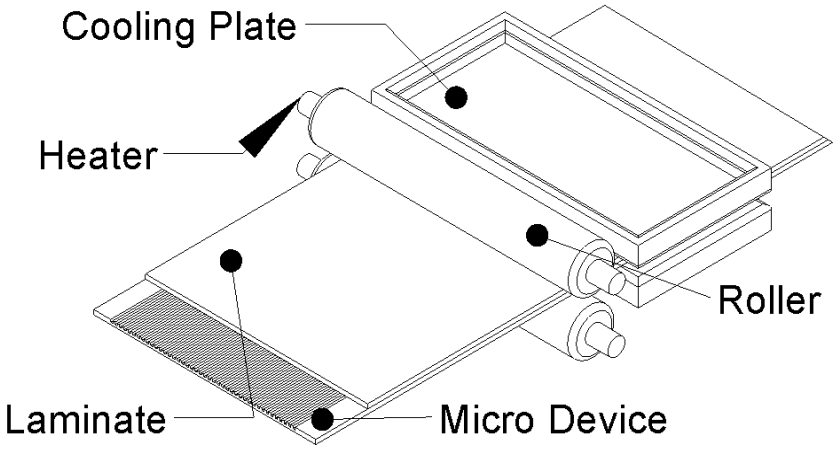

Figure 3.17. Rolling lamination schematic

A pouch was used on both sides of the material to keep liquefied plastic from sticking to the rollers. The rollers were made of a semi hard rubber and heat was applied by a heating element inside of the rollers. Additional pressure was applied during cooling by a sled behind the rollers. Potential benefits of this design are the continuous fashion that lamination could occur and the ability for the device to adjust to various thicknesses.

Laminates used were 15.25 cm by 5 cm by 1 mm and features embossed into the device were 500 microns with an aspect ratio of 1. An array of parameters were tested including the orientation of the device as it was inserted into the laminator (long or short directionally), the thickness of the laminate sheet (0.1 mm, 0.15 mm, 0.25 mm, 0.025 mm) and a removable border to reduce edge effects of thermal cycling.

While the results proved unsatisfactory a great deal was learned and the results are provided in the following chapter. However, since the results were unacceptable in terms of the goal of the process experimentation was performed using the Bienfang laminator.

3.4.3.4 Experiment 4 - Bienfang Platen Lamination

Due to the disadvantage of the rolling laminator platen lamination was investigated solely. Since the major factors were known previously to be temperature, heat and pressure across the surface of the part a fractional factorial design of experiments was developed for this to understand the response of the lamination measures. Other considerations will be performed once an optimal condition for these three parameters is found and their relationship defined.

The design of the experiment is based on (Neter 2005):

1. The set of explanatory factors
2. The set of treatments included in the study
3. The set of experimental units included in the study
4. The procedures by which the treatments are randomized to experimental units
5. The outcome measurements that are made on experimental units

The factors of the experiment are temperature (C), pressure (Pascals) and time (s) related to deformation in the channel, lamination success/failure and if fluid can flow through the channels.

Factor levels have been defined in terms of equidistant quantitative levels such that detailed information about the shape of the response curve can be obtained. Based on the preliminary experiments the range for the factor levels are based on experience of the experimenter. Temperatures range from 125 C (based on the glass transition point/melting point of HDPE) up to 165 C and be changed in 10 C increments. Smaller increments would not yield more information as the temperature change across the plates can vary up to 5 degrees from one point of the laminate to another. Pressure was set to a specified level to provide resistance to thermal forces and ensure contact without pushing material into the channels.

Minimum pressure on the Bienfang was used and is a result from using no pressure to laminate on the Carver Press suggested at least resistive pressure was needed. The same was

used on the aluminum fixture in which the downward force resulted in only resistance pressure. The machine was calibrated so that the reading from the ELF sensor was 12 kg.

Time was based on observation of the time required for a volume of material measuring 15.2 cm by 5 cm, .25 mm thick and 0.152 mm thick to melt. The maximum time required to melt was approximately 65 seconds if the platens were already heated and the only material between them was a 1 mill sheet of paper with a Teflon coating. Temperatures tested were 135 C and 175 C. The incremental step chosen for time was 10 seconds.

Nine samples were collected at each of the control points. The complete design used all possible treatment combinations of the described factors above. Time was randomized but pressure was held constant and temperature was blocked because of time constraints and difficulty in recalibrating the press to an exact temperature.

The laminate thickness chosen was 0.152 mm thick and 57.15 mm by 159 mm to allow for slight misalignments without providing a fulcrum point for the press. The thickness was also chosen because it would make the device symmetrical which should be helpful for thermal effects. Each micro device measured 15.2 cm by 5 cm and .76 mm thick. 500 micron channels were placed 500 microns apart from each other.

3.4.3.5 Experiment 5 - Optimization of Design

After optimum parameters were chosen for the lamination process other factors needed to be considered and several are presented. During the process of experimenting it became evident that a rectangular shape had some disadvantages because the cooling rate was not uniform throughout the material and internal stresses from laminating caused the material to try to wrap around its long axis as shown in Fig. 3.18.

Also features near the edge of the device appeared to have more lamination defects than that elsewhere. A cross sectional view of the entire device shows that on many laminations the outermost two or three channels get progressively smaller in area as you approach the edge of the part.

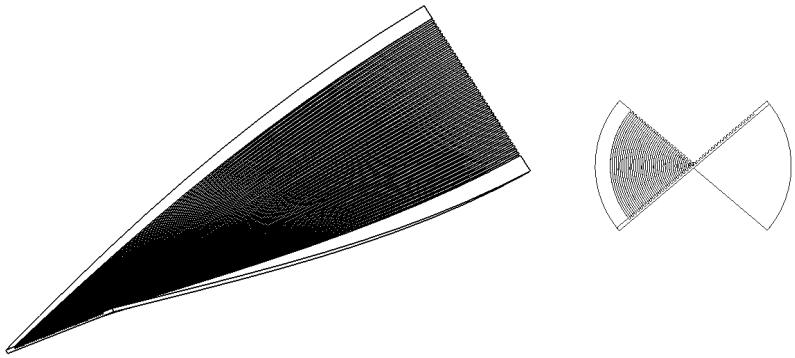

Figure 3.18. Isometric and lateral view of twist

To address these issues several experiments were performed using the optimized parameters of the lamination process in order to improve the performance of the micro device. Each of these experiments used a one factor experimental design approach with 20 replications in order to ensure statistical significance of the results. All conditions from the conclusions of the lamination study were kept constant and one factor was changed at a time.

The first experiment took the optimized lamination parameters and a square device measuring 5 cm by 5 cm using a sacrificial channel around the important features of the device. The concept of a sacrificial channel is shown in Fig. 3.19 in which the channel incorporates the edge effects of thermal cycling. The square shape improves the effects of thermal cycling by allowing the stress to be taken in equally on all sides.

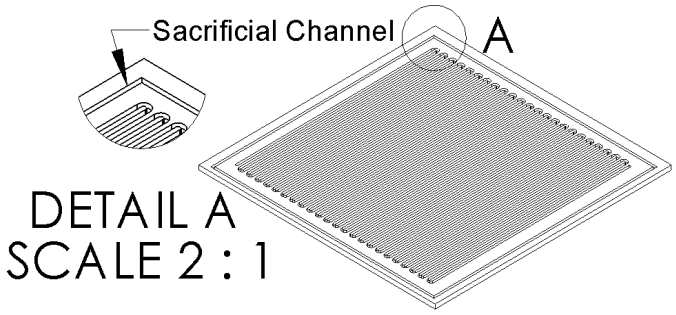

Figure 3.19. Sacrificial channel example

The third experiment tested laminate thickness. Four thicknesses were tested (0.1 mm, 0.15 mm, 0.25 mm, 0.025 mm). The intent of this experiment is to demonstrate two things the first hypothesis is that the thicker material will not sag into the channels as much as thinner materials. The second intent is to demonstrate the effect the additional mass has on the processing parameters which should be minimal.

A final experiment performed was using filling materials in the channels to reduce the tendency to deform. Sodium Bicarbonate was used a filling agent both wetted and un-wetted and the device was tested using the chosen optimum testing parameters.

3.5 Testing Methods

3.5.1 Geometry Comparison Between Devices

While testing of lamination strength is mechanically done in a qualitative way it is more difficult to ensure that the geometry within the sealed channels is uniform and unchanged by the lamination process. It would also help to have a quantitative measurement of “sameness”. In order to test this so that mass manufacturing or multi unit devices can be produced a method had to be developed to indirectly compare the internal geometry of the devices.

To achieve this, the variation in the pressure required to achieve a set flow rate of fluid inside the micro device was used. The inference here is that if two devices have the same internal geometry then the pressure required at a constant flow rate will be the same. This method is also useful for testing the device for leaks, obstruction and other defects that are difficult to detect by sight.

3.5.1.1 Testing Machine Description

The machine designed to test devices works on a simple principle of using a source of fluid, a pump, tubing and an interface that is mechanically joined to the part to measure the pressure inside the channels.

Any constant flow rate device can be used, for this experiment a cole parmer syringe pump was used with a 30 cc plastic Bic syringe.

3.5.1.2 Machine Design

The design of the machine required several stages in order to reduce the error rate to acceptable levels. The first design of the machine used a point pressure DAQ measurement tool ELF. This tool was calibrated using a known KPa (34.4 KPa) 20% greater than the expected KPa to be measured. This was done to reduce the error in the measurement. The ELF sensor shown in Fig. 3.20 was placed between the syringe and pump mechanism as shown in Fig. 3.21. Problems that developed with this design are discussed in the next chapter.

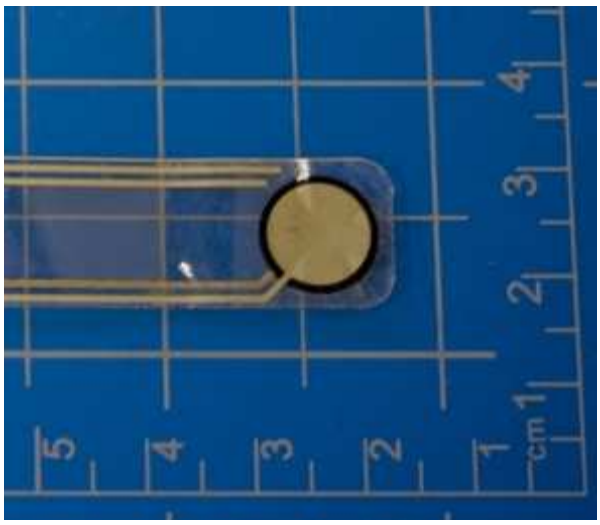

Figure 3.20. ELF low pressure sensor

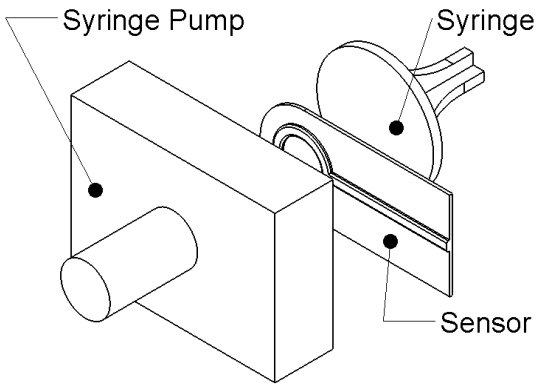

Figure 3.21. ELF pressure sensor placement

As a result of the lack of confidence in the original testing machine a second generation testing machine was designed. The sensor was replaced with a digital pressure gauge that had a range from 0-207 KPa and another gauge with a range from 0-35 KPa. Each gauge had an error rate of +/- 5% across the entire range of the device.

Also the device used to temporarily attach the fluid delivery tube to the laminated micro device was improved. A C-Clamp was altered by attaching a permanent port to it with a small gasket which provided up to 2.06 MPa of pressure on the inlet of the micro device ensuring a clean seal. The schematic and picture for this mechanism are shown in Fig. 3.22.

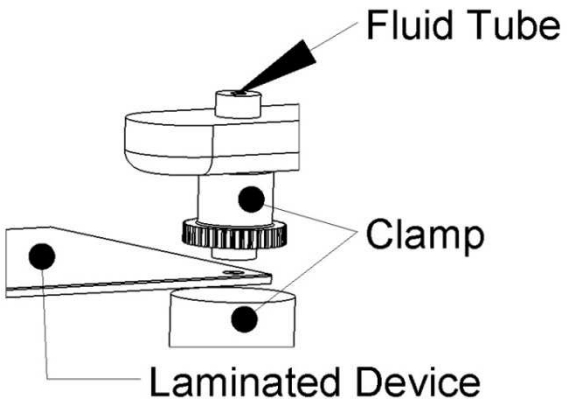

Figure 3.22. Testing machine clamp

Between measurement error rates for this device demonstrated to be less than 35 Pa ensuring that error caused by an operator changing devices was minimized. This was tested by measuring twenty sealed micro devices three times each and recording the pressure data. The error rate for each micro device was below the given maximum of 35 Pa between measurements.

A future design for the measurement device has been designed replacing the manual fixture with an automated air cylinder and using higher quality measuring devices such as a mass flow pressure sensor and a continuous flow HPLC pump. After further study is done to ensure improvements were made details will be published.

3.5.2 Testing Methodology

Each micro device needs to be measured to understand the variation in the lamination process. In order to quantify how much variation the lamination process introduces into the devices the variation in the devices before lamination must be established. This was done by laminating the micro devices using a temporary lamination technique that would not alter the geometry of the device. A heated rolling laminator applied an adhesive coated laminate sheet 1 mil thick made of PMMA to the open side of the micro device. This lamination produces an even, consistent seal that can handle low pressures for limited periods (less than 10 minutes). After removing the laminate sheet from the device it was cleaned with a solvent to remove the adhesive residue.

By measuring a random sample of micro devices that were hot pressed using a Delrin die in HDPE by a Carver laboratory press the basis for the variation between samples was obtained. Twenty micro devices were measured using this method. From this baseline the variation introduced by the lamination procedure can now be quantified.

In order to measure the variation caused by the lamination process 20 micro devices will be randomly selected and tested in the testing apparatus using a constant flow rate of 120 ml/hr and measuring the pressure three times. The distribution fitted to this resulting sample will be used to infer the resulting variation between parts caused by lamination.

3.5.3 Microscope and Results

The second method to measure the results of the lamination process was done by measuring the amount of material flow into the micro channel of the device at three points. A sealed micro device was cut so that a cross sectional area of the channel can be measured using 3D microscopy. The 3d microscopy will be achieved using a Keyence microscope at 100X using 20 micron increments for each slice of the image. An example of the resulting image is shown in Fig. 3.23 and Fig. 3.24 as the cross section of a micro channel with the lamination secured and without lamination respectively.

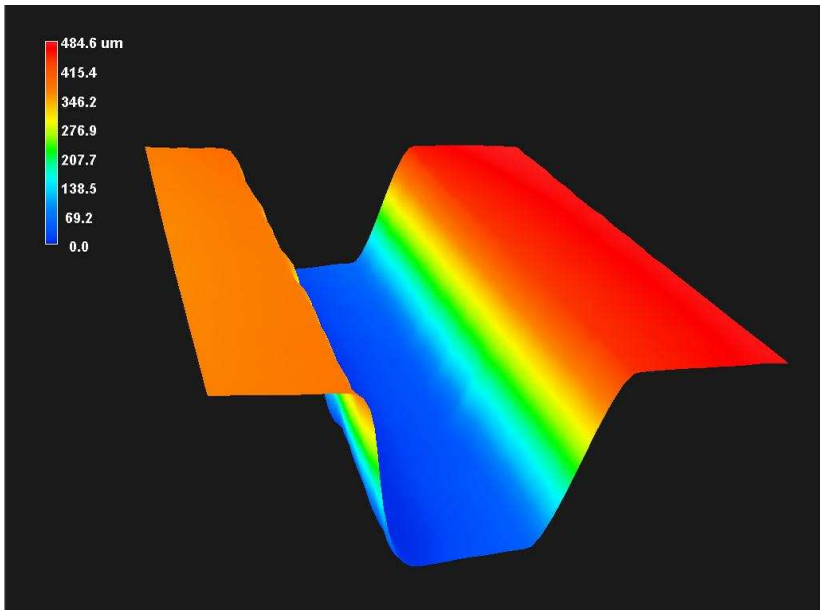

Figure 3.23. 3D Microscopy of open channel

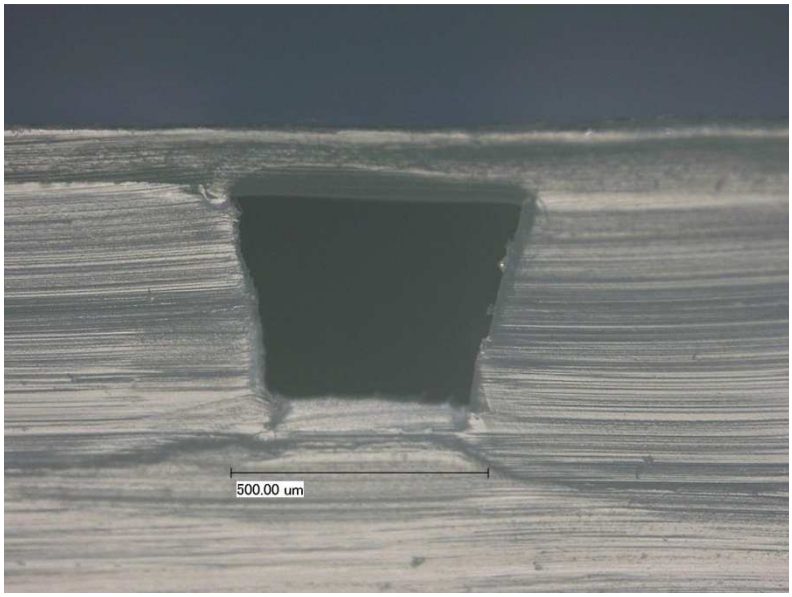

Figure 3.24. Cross sectional view of laminated channel

This method is used to establish the appropriateness of a particular set of process parameters for lamination. A sample from each treatment will be used to evaluate quickly the

damage to the overall dimensions of the micro channel. The ideal lamination would produce a sealed micro channel with the same dimensions as it was originally.

3.6 Summary of Methods

Methods for experimenting with four lamination techniques, adhesive sealing, laser sealing and pressure sealing have been discussed. Machine characterization for lamination sealing was given in detail and methods were shown for measuring sealing performance. The next section will review the results from experiments discussed here.

CHAPTER 4

RESULTS

4.1 Introduction

This chapter discusses the results of the experimental analysis performed to develop and optimize the sealing methodology to enclose microfluidic devices. It is presented in similar order to previous sections by first discussing methods that failed and moving towards successes ending with planar lamination.

4.2 Laser Results and Discussion

4.2.1 Definition of Success/Hypothesis

The success of the laser sealing method depended on being able to selectively melt two layers of material together to create essentially a hermetic seal with ports attached. The seal needed to be repeatable and cause little thermal damage.

4.2.2 Results

While laser machining may be an option, it is not an option using the system that we had available. A different wave length may prove to be sufficient and some design considerations would need to be taken into account.

The HDPE experiments that were performed were unsuccessful because the heat transferred by the laser caused thermal deformation along the part in particular because so much area of the part required joining. Similarly, even with a fixture because of the design being planer and light there was no way to ensure contact between the laminate and the part while the laser was welding.

If future experiments were to be done the wavelength would need to be matched and the design should incorporate some features such as those in Fig. 4.1.

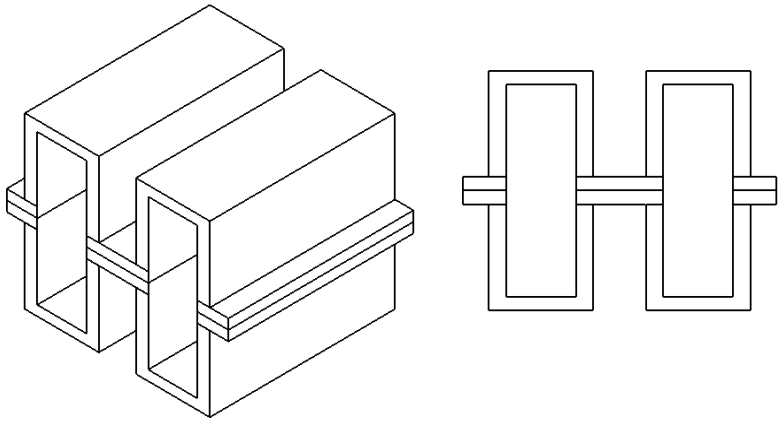

Figure 4.1. Laser joining part design

This method may provide both the weight and space to make a clean joint. Another aspect of this difficult problem is that devices made of HDPE have inherent temperature instability.

4. 3 Chemical Bonding Results and Discussion

4.3.1 Definition of Success/Hypothesis

Success of the chemical/adhesive bonding experiment would have required any directional delimitation force to be stronger than that of the material.

4.3.2 Results

The results of the experiment using Cyanoacrylate provided good tensile adhesion but it cannot withstand any angular shearing force. Each attempt was able to be unsealed with forces that were below our measurement capability.

Two other problems with this method presented itself as the adhesive in such thin layers reacted so quickly with the oxygen in the air there was no more than 3 seconds to apply

the coated substrate. Perhaps it could be performed in a different environment if the facilities were present. Costs comparisons would have to be performed to demonstrate a need for the additional complexity to further study this process. Another difficulty is that even though the coating was only about 50 microns thick it would try to clog smaller micro channels by capillary action. Finally the spin coating method does not provide a perfectly uniform coating as the edges of the material have slightly more coating than the center as shown exaggerated in Fig. 4.2.

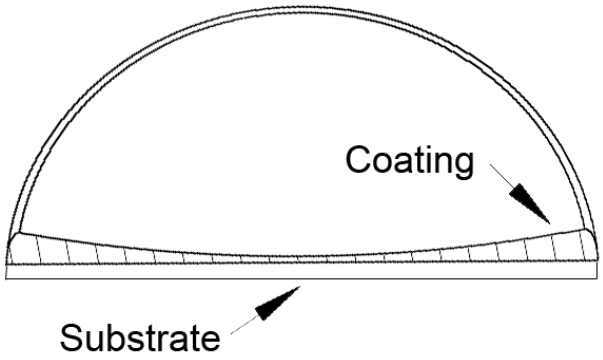

Figure 4.2. Example of curvature of coating in spin coating process

This required the coated area to be significantly larger to attempt to compensate for the variation. The variation has to be minimized so when the laminate is applied the edges, which have a thicker adhesive, will not be pushed further into the channels than the center creating a gradient of channel sizes across the device.

4. 4 Vibration Sealing Results and Discussion

Vibration joining was examined but not experimented so conclusions are drawn from information already available from industry and academic sources.

The benefits are that localized heating occurs rather than heating the whole parts and sealing time is a few seconds. A major drawback is that the minimum feature size that can

laminate is approximately 2mm. It also adds some slight complexity by requiring one of two feature types for joining the substrates. The feature design requirements are shown in Fig. 4.3.

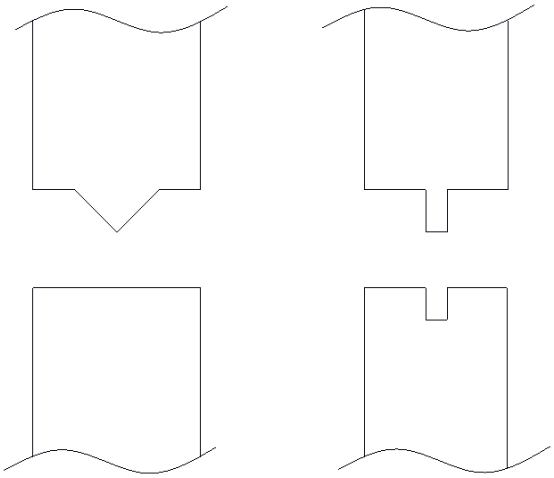

Figure 4.3. Vibration sealing feature design

If these constraints are met then this may be a method a designer may wish to explore. One potential draw back if multiple devices are being used that require very close tolerances (< 10 microns) could occur if the features are not laminated the same way. The potential resulting feature discrepancy is shown in Fig. 4.4.

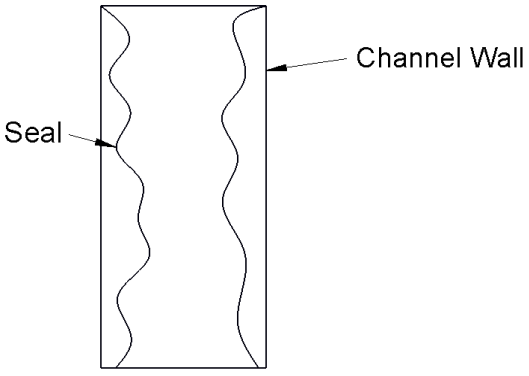

Figure 4.4. Vibration feature issue

Again these results are heuristics given from freely available sources rather than new findings.

4.4 Pressure Sealing

4.4.1 Definition of Success/Hypothesis

Success of the pressure sealing method was determined by the ability to seal without leaking at 35 KPa and to provide consistent pressure readings when assembled in succession.

4.4.2 Results

The first fixture using the aluminum planar force distribution resulted in no appreciable results. While many materials were tested as gaskets the pressure across the plate simply was not uniform and leaks occurred. If a seal was made, the gasket material would also have found a way to block the channels from flowing.

From the second experiment the device would seal but by running a dye through the sealed device it was clear that the center of the part was not sealing. Since the outside bolts were each placed 25.4 mm apart but from edge to edge they were placed 50.8 mm apart it was apparent a central bolt was needed. After this bolt was added and the third experiment was performed the device did seal and with the help of a thin piece of gasket material on the bottom plate it distributed the load well.

A sample of the pressure distribution across the part can be seen in Fig. 4.5. This shows that there are a great number of high pressure areas (green, app. 1000 psi) but some lower pressure spots (purple, app. 300 psi). The majority of the low pressure area as expected is across the open channel area but there are some undesirable low pressure areas in the top half of the device. This may be due to inconsistency in sealing the device on the operator's part and a standard torque tool may improve the distribution.

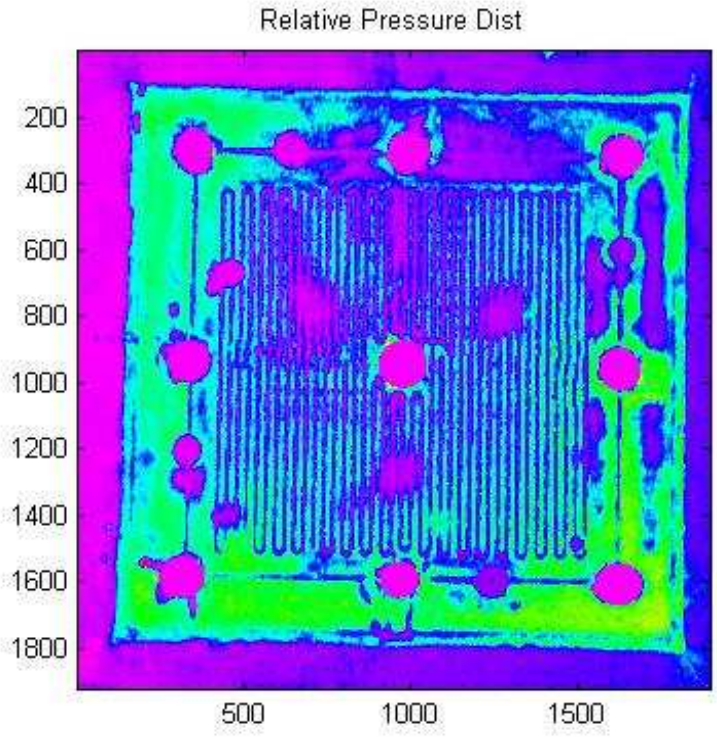

Figure 4.5. Relative pressure distribution across part in pressure fixture

One drawback from the results was seen after thermally cycling the device. After the first thermal cycle each bolt needed to be retightened. This phase of breaking in the device is only required for the first thermal cycle up to 80C, but the use of Belleville disc springs rated for the appropriate load may reduce the need to “charge” the device. This was left for a later research project.

The device did seal and use of a die showed that at 35 KPa no leakage between channels or outside the device occurred. The result is that for multiple devices or single devices pressure sealing is a good method if some rules are followed but creep in the device caused by pressure variation needs to be studied further.

4.5 Lamination

4.5.1 Definition of Success/Hypothesis

Lamination success is dependent on several factors. The first factor is that the method provide a complete seal, this seal also needs to be repeatable to some extent. The second factor is that the channel deformation from the process is minimized and consistent.

4.5.2 Results

4.5.2.1 Experiment 1 – Aluminum Fixture

The result of experimenting with the aluminum fixture is that the only pressure applied across the 152 cm by 50 cm device was applied by a 6.4 mm thick aluminum plate that rested on top of it. The thumb screws which were used to adjust the pressure of the aluminum plate on the device were positioned so they had minimal contact with the aluminum pressure plate.

Any additional pressure at 165 C and the laminate sheet would press into the channels. A second result was the visualization of the lamination process in particular noticing that while flow of the lamination sheet into the channels was expected the device also becomes partially molten and the channel walls try to flow into the channel as well resulting in channels that have slight draft angles of about 10 degrees which increase as temperature, time or pressure push beyond the limits.

Time was measured to be about 10 minutes after applying heat which was attributed to the thermal mass of the fixture. Later experiments on the Bienfang showed that as expected the material would get to temperature much quicker without the aluminum fixture around it. These results were used in preparing for the Bienfang experiment.

A visualization of the channel after lamination was taken to measure and describe the change in the part. This is shown in Fig. 4.6 and shows that the channels in general deformed and grew smaller.

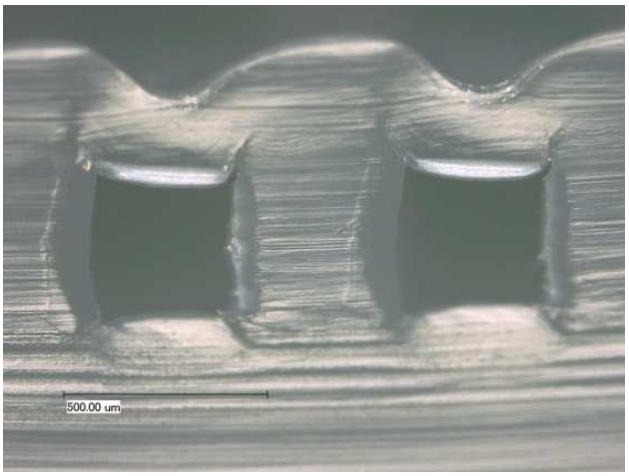

Figure 4.6. Aluminum fixture laminate 100x

4.5.2.2 Experiment 2 – Carver Press

The results of the first experiment on the Carver Press involving 318 kg of pressure across 30.5 cm^2 for 10 seconds at 165 C and 135 C showed that without a doubt any high pressure method would not work in a 'reasonable' period of time. The resulting device had lost nearly all identifiable features but was completely fused with the laminate sheet.

On the other hand, when heat was applied but no pressure the thermal deformation in the HDPE from the temperature gradient across the platens and internal structure of the material caused it to push the laminate sheet off of the device. This result was also expected and confirmed that a token amount of pressure was needed to satisfy lamination requirements.

4.5.2.3 Experiment 3 – Rolling Laminator

Lamination by this method actually proved very useful. "Good" laminations were achievable but with slight defects. Rather complete lamination would occur but features would be distorted.

The disadvantage discovered by this method of lamination was that curved features tended to elongate near the end of the lamination cycle. An example of this is shown in Fig. 4.7.

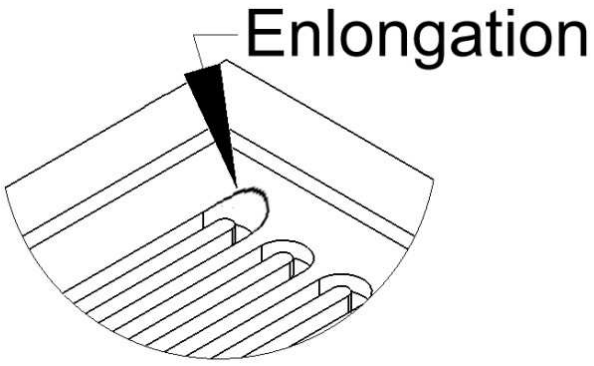

Figure 4.7. Elongation caused by rolling force

It should be noted carefully controlled pressures may reduce the disadvantage shown here. Also equilibrium of the time between cycles and temperature needs to be discovered for continuous lamination as each device acted as a heat draw and pulled heat from the rollers.

4.5.2.4 Experiment 4 – Bienfang Press

The results from the analysis of the Bienfang lamination show several interesting results. The first demonstrates the region of successful lamination as a function of temperature and time shown in Fig. 4.8 as a quadratic polynomial relationship. This relationship is shown in eq. 2.

$$t = 226.11 - 1.33(T) + 0.05(T - 151.66)^2 \quad (2)$$

The variables are defined such that t is defined as time in seconds and T is defined as temperature in C so that for a given temperature we can predict the associated time to laminate successfully but only in the limited temperature range between 145 C and 165 C. This model could be used to develop further experiments with a better suited design of experiments by providing hints as to where the likely candidate solutions would be.

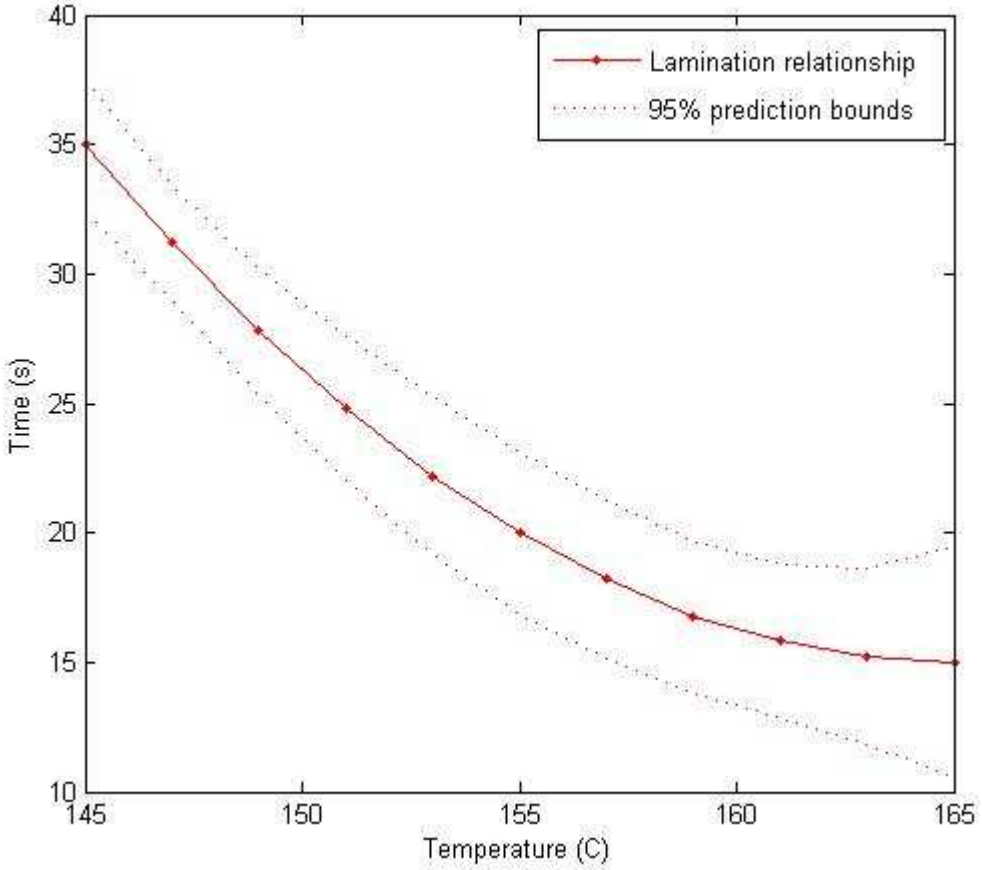

Figure 4.8. Time vs Temperature showing successful lamination relationship

For our purposes additional information about the best performing point on the curve in relation to cross sectional area of the channel after lamination is important. Using a Gaussian process to develop a response surface of the sample space a maximum expected cross sectional area for a successful lamination can be found. The resulting surface response matrix is shown in Fig. 4.9. The surface plot tends to exaggerate the effect of distance between two results which accounts for the negative areas shown. Using the data above a second study could be performed that may reduce the complexity of this surface by sampling from smaller intervals in the independent variables.

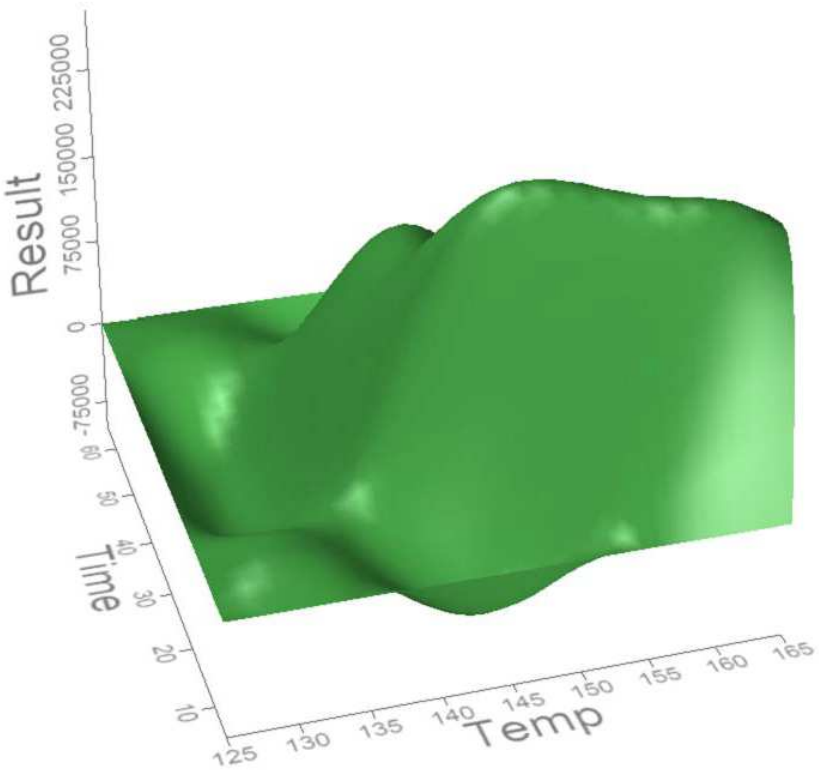

Figure 4.9. Response surface of channel cross section to temp and time

A more visually satisfying representation of the results is provided by a contour profile of the model at the optimum point. This is shown in Figure 4.10, in particular we get an idea of the feasible solution area over which to set our machine parameters to maximize the cross sectional area and minimize deformation of the channels in the device. While several contours are shown the largest area provides the most robust action to machine variation.

This shows that both 155C and 165C have “good” solutions in terms of maximizing cross sectional area while being fully laminated. One other possible extension to the study would be to examine higher temperatures at even lower times and lower temperatures at much higher times.

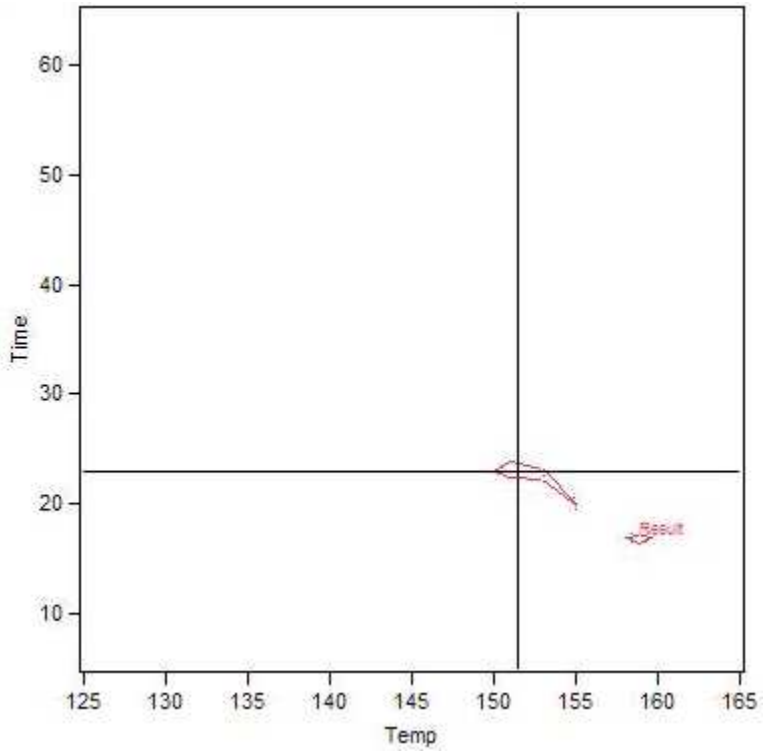

Figure 4.10. Contour optimum of model results

Table 4.1 shows the resulting settings for time and temperature and the expected cross sectional area with error. These results will be used to process the test for the optimization experiments.

Table 4.1 Expected results of lamination

Time (s)	Temperature (C)	Force (kg)	Channel area (μ^2)	Channel std. deviation (μ^2)
23	152	12	232712	$\pm$33153

A sample of non laminated devices show a cross sectional area of similar proportion with a percent change of less than 3%; while theoretical cross sectional area is 250000μ^2 the percent change is then less than 7%. It will be up to the application if this is acceptable, and the results can be improved using the method described in the optimization experiment.

Other considerations need to be given such as surface modifications caused by a heating cycle but in many cases this should not cause an issue.

4.5.2.5 Experiment 5 – Optimization Experiments

The part design was changed such that it was square and included a sacrificial channel. The data from this was compared using ANOVA to pressure data of the square design with a sacrificial channel to the rectangular design with no sacrificial channel. The results are provided as psi in the channels at 120ml/hr. The results of the comparison are shown in Fig. 4.11. The interpretation is that the configuration change significantly shifts the mean from 10 psi to 3.6 psi and reduced the standard deviation 3.17 psi. It is obvious that this design is superior.

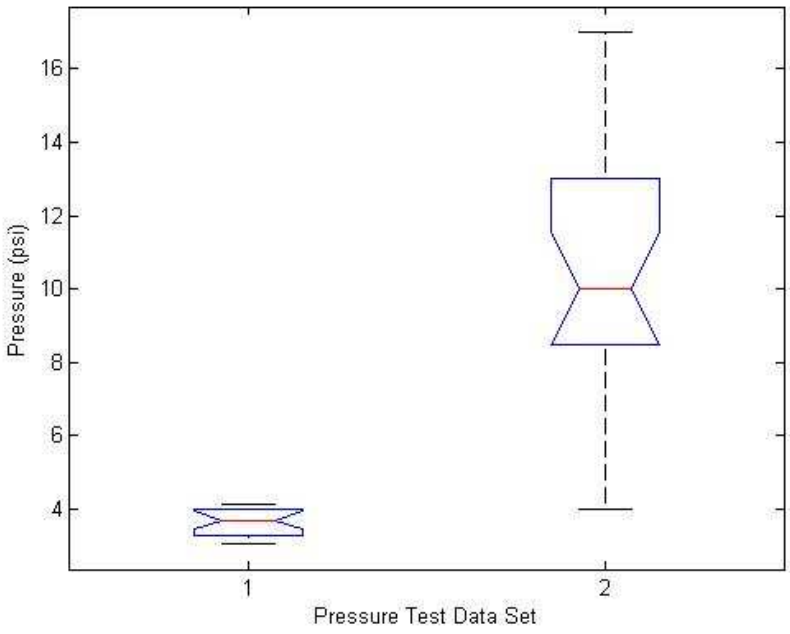

Figure 4.11. ANOVA comparison pressure dist 1(square/channel) 2(rectangle)

However a comparison of the data to its expected performance also was needed. The data from this was compared using ANOVA to baseline data. The results are provided as psi in the channels at 120ml/hr. The results of the comparison are shown in Fig. 4.12. The laminated

square data shows a slight increase in standard deviation of 0.24 psi but the two means of the samples do not vary in a significant way from the data collected.

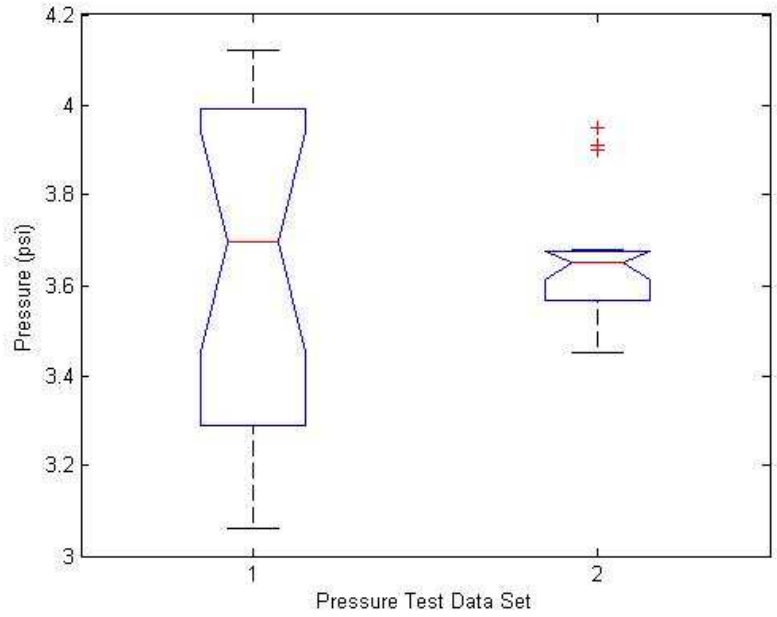

Figure 4.12. ANOVA comparison of 1(laminated) and 2(un-laminated) dist.

The result of the multiple laminate thickness showed that a 0.254 mm thick sheet would provide the best support for devices above while still laminating at the same specifications. This result is a good heuristic for devices that are about 1 mm thick but other device types may need to be retested.

Filler materials would not allow the central area of the microfluidic device to laminate. The material was very carefully put into the channels and the top surface cleaned but no successful laminations occurred. Possible reasons include out gassing or not allowing air flow. Air flow issues were seen when devices did not have through holes. The edges would laminate but the center area wouldn't. Filler materials exhibited a similar trait perhaps because they blocked the air outlets. Cleaned units although un-laminated did appear to have less deformation and so different filler materials may provide better results.

4.6 Testing Machine Results

The results of the testing machine in its original form using the ELF sensor sandwiched between the syringe pump and syringe did not provide reasonable measurements.

While this provided quantitative measurements the system required 2 minutes to stop oscillating at which point creep in the sensor began to effect the measurement. Similarly because the sensor was not permanently attached to the device the error rate between measurements was too high to be useful.

The second version of the machine did provide good results for measuring and comparing micro devices. The pressure gauge provided an accurate quick reading of the average pressure inside the channels with little error. This device was also simple to assemble and had a high degree of repeatability.

The average standard deviation between readings of the same device was 0.015 psi. The result of this is that the measurement process was very reliable and to a high degree of accuracy and precision allowed us to measure the internal geometry variation of the devices.

CHAPTER 5

CONCLUSIONS

5.1 Introduction

The methods for investigating a process for sealing and laminating micro devices and the results of this method have been presented. Conclusions and further recommendations for study will be given and final details related to the production process of microfluidic devices.

5.2 Conclusion on Alternative Sealing Methods

Every sealing method has a particular material set and application set that it is well suited for. Deriving an understanding of that set is useful in developing design knowledge for Microfluidics. Laser machining provides a difficult road for sealing HDPE devices and potentially cold ablation or large devices could be used in this method but this is not well suited for most applications. On the other hand laser micromachining for manufacturing features in micro devices is very applicable and well studied.

For devices made of HDPE Adhesive/Chemical bonding methods provide very little solution but research is being done to see if this trend can be broken. However, some scientists feel that there are physical reasons why a solvent or adhesive for HDPE cannot be developed requiring a detailed explanation of the chemical properties.

5.3 Conclusions on Pressure Sealing

When sealing using a pressure method several conclusions were drawn. The tolerance limits for thickness variation across apart are approximately 25.4 mm between any two points 50.8 mm apart. A bolted design is superior to a planar force design and bolts should be placed fairly close together, approximately 25 mm. Also, part sizes should correspond to the size of the top and bottom plates. Otherwise the plate may begin to bow. A small pressure distribution

device may be needed on the bottom plate to make up for small deviations in thickness and pressure distribution.

Overall pressure sealing is a very good method for sealing devices. It is unable to withstand the same pressures as laminated devices but introduces less variation between parts because the device does not have to go through as many steps.

5.4 Conclusion on Lamination Sealing

The net result of experimenting with lamination is that a suitable method for sealing micro devices is available. The resulting devices made of HDPE are highly chemically resistant and very durable able to withstand thousands of KPa.

The most probable setting would be a resistance force of 12 kg if calibrated on a Bienfang, a time of 23 seconds and a temperature of 152 C. Laminate sheeting of .25 mm thickness for 500 micron channels provided a good balance between sealing and dipping into the channel. Smaller channels could probably use thinner laminate. Appropriate design such as square parts and sacrificial channels reduce variability between parts and reduce defects. However, the number of defects if this process was intended to be commercialized may be prohibitive but bad products can be recycled back into the process if needed.

Features that are not encouraged are filler materials. These experiments tended to produce gases that got trapped inside the laminated device. Other filler materials could be tested but the process is difficult and appears unlikely to yield results.

Variation between parts can be satisfactorily small using the settings and methods outlined. The acceptable internal geometry variation between parts can be calculated and translated into an acceptable KPa variation when measured on the pressure testing machine using constant flow rate through the device. By this method an evaluation of whether or not lamination would be appropriate for an application can be made.

5.5 Conclusion on Design of Parts

In general, recommendations to design square parts, with tight tolerances in thickness provide flexibility in sealing methods. Similarly, a sacrificial channel and distancing features several mm from the edge of the part appear to elongate its useful life, especially if the device goes through thermal cycling.

Potential alternative materials to HDPE are PP, UHMWPE (Ultra High Molecular Weight Polyethylene), Acetal and PEEK for highly corrosive applications. All options offer better thermal stability than HDPE and can hold tighter tolerances. However, HDPE has the most robust chemical resistance and has a low melting point if hot pressing is a desired manufacturing method. No lamination experiments were performed on these materials and may provide an avenue for further study.

5.6 Further Study

One potential process for further study realized during this research was the potential to make a continuous process for manufacturing micro devices using a heated rolling platen that polymer sheet stock could be fed into resulting in sheets of micro devices in minutes.

Improvements in the testing machine are also of great interest particularly as the need for comparing the quality between micro devices increases. Most current applications do not require more than one or two devices but some research projects are requiring thousands and the potential for commercialization is higher with the ability to measure the variation in the manufacturing process.

Other considerations may be the micro device thickness, feature location, cooling method, annealing needs, the time between runs, laminate orientation (rectangular parts), cleanliness, dryness of the material and reduction of operator implications.

5.7 Summary

Many methods for sealing microfluidic devices were examined which was prompted by the need to seal a device made of HDPE. A literature review provided a number of potential

solutions but an alternative with little research was chosen by laminating the devices against a polymer of the same material a closed solid flexible device was formed.

Experimentation was performed in pressure sealing, laminating and evaluation experiments were performed in other areas. The results were provided and conclusions drawn such that bolted pressure sealing and lamination under appropriate conditions provide a good basis for sealing micro devices made of HDPE under different applications.

Variation is introduced into the part but continued efforts to design a lamination machine that controls temperature, pressure and time to tighter tolerances would improve the process error. If variation between parts is within acceptable range then this method would be sufficient for manufacturing microfluidic devices and that decision would be left to the application designer.

In terms of the eight impacts to be discussed, the process yield was improved and variability reduced. The cycle time is quick taking only 15-23 seconds and heuristics for development were provided. Various configurations for design were compared and conclusions provided. Alternative materials were suggested but only one was reviewed for its chemical resistance and price. The extent of robustness of the process was visualized by providing a feasible region. Finally many key characteristics that effect performance were discussed such as temp, pressure and time but also many other issues were shown. With this information the goals of this study were met.

APPENDIX A

MICRO FLUIDIC DEVICE MANUFACTURING

A.1 Introduction

Appendix A provides detailed information on the process for producing the microfluidic devices used in testing the lamination process as well as variation on the production of these devices that may be used for future development.

A.2 Process Overview

The process for making micro fluidic devices depends first on the scale of production as well as the number of simultaneous devices that make a unit. This study considered devices in the micro scale such that feature sizes ranged from 125-750 microns. In order to produce devices on this scale either a one, two or three stage process was used.

The one stage process used a 4 axis micro mill in order to rapidly prototype devices for testing. The two stage process required the production of a die on the micro mill and used a heated press to hot emboss or hot press the die into a thermoplastic. The three stage process used a part made on the mill to press a die used to press a part.

In each case the process for production can be broken down into device design, milling parts, milling dies, hot embossing, hot pressing, material considerations, cooling, measuring and miscellaneous information.

The device design was provided using off the shelf CAD software and the machining operations were done using off the shelf CAM software. While design considerations are impacted by individual use of the micro device some methods need to be addressed to be processed well by the following production method. Following some of these best practices can reduce the cycle time for production of a new device.

1. Allow for at least 1 mm between internal and external features.
2. Stress caused by flow during pressing will be along the line of the features.

3. Square parts can improve thermal reaction of the part during cooling and heating, and improve the ability to seal evenly.

The micro mill is a 4 axis CNC machine that accepts 1/8” tooling. The controller for the micro mill was capable of reading standard FANUC g-code.

The tooling for the mill can come in several varieties, our tooling was purchased from Cutting Edge Technologies and in general the carbide coating (a ceramic coating on a steel tool to improve wear) with a flat end and two flutes was used. Ball ends can be used if geometry of the part is not important.

The dies for pressing HDPE parts were milled in black Acetal 1.5 mm thick. In order to produce smooth edges large amounts of coolant was used and the feed rate was 30.5 cm/min. Replacing the cutting tool with each part improved the surface finish to a level usable for Microfluidics. The Delrin die was cleaned using compressed air (861 KPa) for 5 minutes. Any material that was not removed when cleaned was removed during the first hot pressing operation with that die.

These dies were characterized using a Confocal microscope and an optical microscope to develop 3D images and measurements of the features. An example image is shown in Fig. A1.

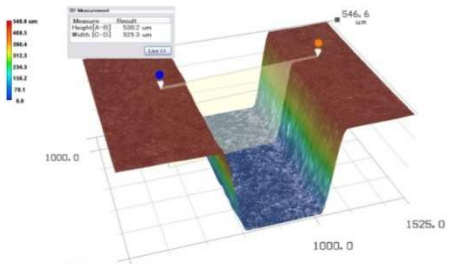

Figure A.1. 3D Microscopy example

After the die was verified to have the correct dimensions it was used to hot emboss/ hot press HDPE parts.

The physical concept of hot pressing is to both heat a thermoplastic material to a temperature above its glass transition temperature or its melting point and then apply even pressure into the material to press a die with micro features into it (Becker 2000). After the features have reached the appropriate depth the material is cooled and the molecules relock into place.

This process was done by using a Carver 30 ton bench top press (shown in Fig. A.2) with a 19 Lpm chiller. The press was set to hot press molten HDPE (135 C) for 120 seconds at 2.9 MPa. Then the part was cooled to 60 C and removed from the press. It was important to remove flash in the wells before lamination or else air would get trapped in the device and lamination would not occur properly. The finished Hot pressed part would then be laminated as described by this paper.

Figure A.2. Carver bench top press

Many other materials were used as a die including graphite, polycarbonate, polysulfone, PEEK, PMMA and aluminum. Various materials were also used as stock material for pressing including PP, PMMA, polysulfone, UHMWPE and Acetal. Most of these processes are public and available through literature review.

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BIOGRAPHICAL INFORMATION

Ryan Oliver attended the University of Texas at Arlington for both his undergraduate and graduate work in Industrial Engineering and graduated summa cum laude as an undergraduate. He has served as the president of the UTA student chapter of the Institute of Industrial Engineers and as an officer of Tau Beta Pi and Alpha Pi Mu. His research interests include signal analysis such as those in financial markets, micro and nano device manufacturing and design, robotics and the application of process improvement to nonprofit organizations. He has worked for DFW Airport and Coca Cola Bottling Company and acted as a consult for The Marriage Center of Texas. His graduate research focused on producing bio fuels using micro fluidic devices. He plans to continue his graduate work to earn a PhD in Industrial Engineering.