The Development of an Innovative Bonding Method for Microfluidic Applications

By

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ABSTRACT
The field of microfluidics has powerful applications in low-cost healthcare diagnostics, DNA analysis, and fuel cells, among others. As the field moves towards commercialization, the ability to robustly manufacture these devices at low cost is becoming more important. One of the many challenges in microfluidic manufacturing is the reliable sealing of the microfluidic chips once the channels have been generated.

This work was an investigation of innovative ways to robustly heat the substrate-cover plate interface of a microfluidic device for the purpose of bonding and sealing the microfluidic channels. An extensive literature review revealed the benefits of interfacial heating, and both simulations and experimental investigations were used to evaluate a few different methods. Ultimately, a unique method was established that uses light to provide both the bonding energy and the illumination for an in-process vision system for real-time viewing and control of the bonding process. The process results in the generation of a homogenous and optically clear bond, and preliminary tests show that when properly controlled, a bond with minimal microchannel deformation can be created.
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1 Research Motivation

1.1 Importance of Microfluidics

The field of microfluidics has taken off in the past couple of decades as a promising way to create “labs on a chip.” Microfluidic devices require a very small amount of liquid and can be used to conduct powerful analyses in many arenas, including healthcare diagnostics, DNA analysis, drug discovery and fuel cell technologies.

In 1990, Manz et al [1] discussed the miniaturization of a total chemical analysis system to monitor the concentration of a chemical species. This was one of the first microfluidic demonstrations, published even before the term “microfluidic” was coined. This research showed that miniaturizing the total chemical analysis system would enhance its analytical performance. Transport of fluid through the total chemical analysis system is faster when it is miniaturized and less liquid is needed. As a result, chromatographic and electrophoretic separations can occur more quickly and multiple processes could even run in parallel on one chip.

A few years later in 1993, Gravesen et al [2] discussed the advances in the fluid mechanics modeling that described the phenomena behind the microfluidic technology, and also described the advances in micromechanical valves and micropumps to control fluid flow. These elements are at the base design of most microfluidic devices. At the time, the most common devices were flow sensors and there were a few simple microfluidic systems, such as inkjet printer heads, on the market.
Since its infant stages, the field of microfluidics has grown to include a variety of microfluidic elements and applications. Daktari Diagnostics has developed a low-cost blood analyzer to measure glucose levels [3], Caliper Life Sciences developed a chip for electrophoretic DNA separation [4], and Choban et al developed a microfluidic based fuel cell that could lead to the development of efficient room temperature based power sources [5]. These are just a few of the many examples of microfluidic applications under investigation today.

As Whitesides states, “As a technology, microfluidics seems almost too good to be true: it offers so many advantages and so few disadvantages (at least in its major applications in analysis)” [6]. However, microfluidic chips are not yet widely used, for there are still many questions to be answered before it will become a major new technology, making it an intriguing avenue for academic research.

1.2 Manufacturing Microfluidic Devices

As is described by Becker [7], the basic process to develop a microfluidic chip consists of:

1. General design work

2. Microstructuring the substrate. This can include processes such as hot embossing, injection molding, and laser ablation, among others. At the end of this process, there exists a substrate with the desired microchannels in it.

3. Backend processes. Microstructuring generates the desired micro features, but does not produce functional chips. About 80% of the manufacturing costs for microfluidic devices stem from processes such as depositing electrodes, cutting
the device out of a larger master, and encapsulating the channels so that fluids can flow through them.

The specifics of carrying out each of the above steps are dependent on the device’s material. Microfluidic devices have been made out of a variety of materials, including silicon, glass, thermoplastic polymers and thermoset polymers. In general, polymeric materials have lower costs associated with them, in terms of both material costs and manufacturing costs. In applications such as low-cost healthcare diagnostics, it is also desirable to have disposable devices, which is much easier with polymeric materials. Thermoset polymers are often used in the lab environments because they can be cast and cured in small quantities. However, on a larger scale, thermoplastic polymers are more desirable, both for their material properties and their manufacturability. These thermoplastics include polymethylmethacrylate (PMMA), cyclic olefin (co)polymer (COC), polycarbonate (PC), polystyrene (PS), and others.

The MIT Center for Polymer Microfabrication has been running a MicroFactory project, µFac, in which the automation of manufacturing thermoplastic microfluidic devices is under investigation [8]. In this project, the microfluidic mixer shown in Figure 1-1 below is manufactured out of polymethylmethacrylate (PMMA).
In this project, hot microembossing is used to complete the microstructuring step. The blank substrate is heated to some temperature above the plastic's glass transition temperature. At these elevated temperatures, the polymer begins to soften. A tool with a mold of the desired channels is then brought in contact with the top surface of the substrate and pressure is applied for a specified amount of time. After the softened polymer plastically deforms into the mold, it is cooled and pulled off of the tool. The embossed substrate is now complete. Within the MIT lab, Dirckx [9] and Hale [10] have worked on the development of equipment for the hot microembossing process that achieves fast cycle time at low capital cost.

After embossing, the substrate moves to an inspection stage and then onto the bonding stage. The bonding equipment used in this example was developed based on Hale's hot embossing technology. The substrate and cover plate are both heated and then pressure is applied to get the surfaces of the substrate and cover plate to bond together. This is a technique commonly used in industry known as thermal bonding. Its details will be discussed further in the next two chapters. Once bonded, the device can be functionally tested, to ensure that fluid moves through the channels and mixes together as is intended.
One of the biggest challenges in the microfactory project, and in many microfluidic manufacturing environments, is the reliable and high-yield bonding of the microfluidic devices. As described by Ng et al., a good bond is high in strength, is leak-proof under high fluidic pressure, has a homogenous bonding interface free of voids, and has minimal deformation of microstructures [11]. It is also beneficial for the device to have high optical clarity, so that a user can view the fluid flowing through the channels.

An example of a bonded device that was not leak-proof is shown below in Figure 1-2.

![Figure 1-2. Leaks on a microfluidic device due to insufficient bonding. Courtesy of Ali Beyzavi, NTU.](image)

An example of a bonded microfluidic device with blocked channels due to excessive deformation during bonding is shown below in Figure 1-3.
Figure 1-3. Blocked channels on a bonded microfluidic device due to excessive deformation during bonding. Courtesy of Ali, Beyzavi, NTU.

There are many methods of bonding microfluidic devices, including adhesive printing, lamination film bonding, thermal bonding, surface modification, solvent bonding, and localized welding, each of which will be discussed in the next chapter. The optimal bonding method is different for every application. In many cases too, there is a method that works for one microfluidic pattern, but adapting that method to other patterns takes a lot of adjusting and often results in a lot of wasted parts. Especially as microfluidics move further towards commercialization, there is a great need for a high-yield, high-throughput method that is more robust to process changes.

This need has been definitively stated in the literature. In his 2006 article in Nature [6], Whitesides states, “An important aspect of the commercial development of microfluidics – crucial to many of these applications – is the development of the technology for manufacturing microfluidic devices.” He then raises questions about many microfluidic manufacturing issues and ends with, “And what about technologies for sealing and packaging?” Even more recently, Tsao and DeVoe state in their 2009 review of microfluidic bonding technologies, “It is clear that the thermoplastic bonding technique need [to be] further advanced to superior compatibility to meet various microfluidic applications...the need for bonding methods which are compatible
with high throughput manufacturing will increase.” It is this clear need for further
developments in bonding technologies that motivated this research.

In this thesis, an innovative method of bonding is developed. This method uses light to
provide both the bonding energy and the illumination for an in-process vision system for real-
time viewing and control of the bonding process. This research sets the groundwork for a
concept that, with further development, will be able to control the bonding of each chip
individually, ultimately making the process more robust and high-yield.
CHAPTER

2 Current Microfluidic Bonding Methods

2.1 Bonding Thermoplastics

In 2009, Tsao and DeVoe [12] wrote a comprehensive review article of the various methods in existence to bond thermoplastic microfluidic devices. The methods reviewed included adhesive bonding, lamination film bonding, thermal bonding, surface modification, solvent assisted bonding, and localized welding. This chapter will review each of these methods, and will also consider the laser welding of microfluidic devices.

2.2 Adhesive Bonding

In adhesive bonding, glues are used to secure the substrate and cover plate. These glues include liquid adhesives that set when the solvent evaporates, epoxies and acrylics that cure after being mixed with a catalyzing agent, and thin layer high viscosity resins that are UV curable. The latter is the most common. One disadvantage of adhesive bonding is that the channel walls are no longer homogenous, as this resin forms part of the wall and could have an effect on the fluids flowing through the channels, particularly when the fluids are biological reagents. The biggest challenge with adhesive bonding is that the channels can get easily clogged with the glue being used. Careful application of the glue is critical.

An interstitial bonding technique has been developed that allows for the careful placement of the glue only on the regions in between channels [13]. A diagram of this method is shown below in Figure 2-1. In this process, both the chip and cover plate are cleaned and dried. Resin is loaded in the loading reservoir and in a matter of seconds, capillary action draws
the resin into the appropriate interstitial space. The resin is precured for one minute to make it
tacky and then postcured for two hours.

![Resin loading reservoir](image)

**Figure 2-1. Schematic of interstitial bonding technique [13]**

This process had almost 100% yield and was able to successfully bond PMMA to
PMMA, glass, and PC. The thickness of the resin was small compared to the channel height, so
there was negligible altering of the surface properties, though the channel wall was still not
100% homogenous. The resulting bond strengths were as follows: PMMA-PMMA: 9.3kPa,
PMMA-glass: 12.4kPa, Scotch tape: 3.1kPa, thermal bonding at 110°C for 1 hour: 18.6kPa.

In another kind of adhesive bonding [14], PDMS was spin-coated in 10-25 μm
thicknesses and then room temperature cured most of the way for 20 hours. The cover plate
was then bonded to the microchip under pressure at 90°C for 3 hours. In this example, there
was 100% bonding across the desired interface and a 15.7MPa bond strength.

Other types of adhesive application microcontact printing, screen-printing the adhesive
onto a cover plate, and injecting the channels with a resin, applying a UV-curable adhesive, and
then flushing the channels out with nitrogen before curing.
2.3 Lamination Film Bonding

In lamination film bonding, a thin polymer film is used as the cover plate and is laminated onto the substrate surface. In one example of lamination film bonding [15], thermal lamination was accomplished with a polyethylene terephthalate (PET)/polyethylene film in a standard industrial lamination apparatus. Lamination occurred at 125°C with less than 3 seconds of exposure to hot temperatures. The reservoirs that were needed to access the channels were laser ablated after lamination. The technique worked well and because of the low temperatures and rapid sealing times, it was also possible to predeposit sensitive biological reagents. The bonding was strong enough such that 40 μL/min could be externally pumped through the device. No leaks were detected and the profile above the channel showed no deformation of the lamination into the gap, even at 190 μm wide.

In another example [16], the PMMA was annealed at 80°C for at least 90 minutes prior to bonding to avoid stress cracks in the structure. The PMMA structure was then heated at 100°C and a cold lamination foil was manually pressed onto the surface. The film only heated where it was touching the top surface of the substrate because of the low conductivity of air in the channels. The foil adhered to the substrate without the foil dissolving and clogging the channels.

In general, lamination film bonding is advantageous because there is minimal channel deformation and the process occurs quickly. However, the film can embed in the channel and since the film is a different material than the substrate, the channel walls are non-homogenous.

2.4 Thermal Bonding

In thermal bonding, the substrate and cover plate are heated to a temperature slightly greater than the glass transition temperature of one or both of these pieces. Pressure is then
applied to increase the contact forces between the two pieces. This combination of elevated
temperature and pressure results in sufficient flow of the polymer at the interface and
interdiffusion of the polymer chains between the surfaces. With this method, it is feasible to
create an optically clear homogenous bond, which is highly desirable in microfluidic
applications. However, this process can also be very low-yield because the optimal bonding
parameters are different for every application. Even just slight changes to the grade of material
or the microfluidic pattern itself can cause the need for an adjustment to the bonding
parameters. Channel deformation or voids in the bonded area can result if the process
conditions are not optimized.

Nevertheless, the simplicity of this process and the reasonable bond strength that can be
achieved makes it a promising option that is widely studied and implemented. The simplicity is
clear in implementations such as that by Sun et al [17]. The PMMA pieces to be bonded were
placed on a hot plate and an aluminum plate was placed on top of them. Weights on top of the
aluminum plate provided the pressure and the heating was controlled by adjusting the hot plate
temperature. This particular study used high hot plate temperatures, on the order of 165°C, low
pressures, typically 20kPa or less, and long time frames, on the order of 30 minutes.

In another implementation of thermal bonding, Chen et al [18] was successful at
bonding two PMMA pieces at 110°C for 20 minutes. Zhu et al [19] studied bonding
temperatures between 88°C and 100°C and pressures between 1MPa and 3MPa for bonding
PMMA. Huang et al [20] bonded PMMA to PMMA using pressures of 1.25 kPa for 3 minutes,
after heating for 12 minutes (heating temperature unreported).

In just these few examples of thermal bonding, the range of temperatures and pressures
is vast, as is the complexity of the bonding equipment. There was successful bonding in each of
these examples, though, which is why the technique is common. It is just a matter of choosing the right parameters.

2.5 Surface Modification

By modifying the substrate and cover plate surfaces to be bonded, there is an increase in the surface energies, which improves the wettability of the substrate and cover plate, allowing better contact and increasing the interlocking of the polymer chains across the interface. The surface modifications also generate electrostatic interactions and can produce hydrogen or covalent bonds across the interface if the modification is in the form of polar functional groups.

In an example of monomer modification [19], the PMMA was modified with its monomer, methylmethacrylate, before being thermally bonded. This modification decreased the glass transition temperature of the surface, thus allowing the surface of the part to soften more than the bulk when the temperature was raised. The thermal bonding occurred at 95°C and 2MPa for 3 minutes. In this particular study, the bond strength was monitored over time and compared to two pieces that were thermally bonded without surface modification. It was found that the bond strength over time was higher and steadier when the surface was modified prior to thermally bonding it. The maximum bond strength achieved without channel collapse was 1MPa.

It is also possible to modify the surface with ultraviolet light. In one example [21], the surfaces of PC were exposed to UV radiation for 30 minutes. The radiation had a wavelength of 254nm and a UV intensity of 15 mW/cm². This surface modification also lowers the glass transition temperature of the surface. Bonding temperatures were varied between 146°C and 160°C and the bonding time was fixed at 30 minutes. A bonding temperature of 150°C was
chosen in the end because it resulted in sufficient sealing without deforming the microstructures. Fluid was sent through the device and no leaks were detected.

Treating the surfaces with UV/ozone produces energetic surfaces and results in at least 1-2 orders of magnitude improvement in bond strength for PMMA and COC, and allows the bonding to occur at temperatures well below the glass transition temperature with a good degree of control over the hydrophilicity of the surfaces [22]. In this example, the surfaces were exposed to the UV/ozone with a commercial ozone cleaning system. They were then rinsed with IPA and DI water and dehydrated in a vacuum oven for two hours. They were finally inserted into a hot press at 4.8MPa for 10 minutes. Both PMMA and COC were successfully bonded in this way.

Plasma treating the thermoplastic oxidizes the surfaces being bonded. This removes the dust and polishes the part, shortens the polymer chains at the surface, and lowers the surface layer’s melting temperature to be much lower than the temperature of the bulk polymer [16].

While surface modifications have been shown to have a positive effect on the bonding quality of microfluidic devices, the presence of a surface modification is not always ideal. Surface modifications alter the surface chemistry of the interfacial surface and often the channel walls as well. The surface chemistry can have an effect on the fluid flow through the channels, especially in biological applications, and so altering these surfaces is not always an option.

2.6 Solvent Assisted Bonding

Similar to bonding with surface modifications, solvents can also be used to facilitate better bonding. When a thermoplastic surface is solvated, the polymer chains become mobile and can diffuse across the interface better, resulting in high bond strength. At the same time, the softening of the polymer surface may cause even more channel collapse than would have
occurred otherwise. Solvent bonding is very similar to surface modification bonding in that the solvent facilitates better bonding, but alters the surface chemistry of the polymer.

A variety of solvents have been used for this purpose, including methylcyclohexane [23], acetonitrile [24], and ethanol [16]. In some examples, the solvent bonding is also accompanied by the use of phase-changing sacrificial materials in the microfluidic channels. In one case [25], paraffin wax filled the PMMA channels and acetonitrile was used for solvent assisted bonding. The hardened wax in the channels prevented the softened PMMA from collapsing during the bonding process. Once the bonding was complete, the wax was melted out of the channels.

2.7 Localized Welding

In localized welding, a thin layer of the surface of the cover plate and substrate is melted for enough time that the two melted surfaces at the interface will weld together. The process happens quickly enough that the high temperatures only affect the surface layers of the plastic, and the bulk remains rigid and doesn’t deform.

In ultrasonic welding, area contact energy directors are built into the design of the microfluidic part in between channels. The substrate and cover plate are then clamped together and ultrasonic vibration is applied. In one example [26], ultrasonic vibration with an amplitude of 13 µm, a bonding pressure of 0.32MPa, and a bonding time of 8s, was applied and the resulting part was able to sustain a burst pressure of 680-800 kPa and had a bonding strength of 2.25MPa.

In microwave bonding, gold or another metal film is deposited on the surface of the PMMA and the substrate and cover plate are clamped together. The stack is then exposed to
microwaves, selectively melting the film and causing localized melting of the PMMA. In one trial of this technique [27], a bonding strength of 0.7MPa was achieved.

In both of the above examples, the localized welding achieves a good bond, but the implementation of energy directors or the addition of metal films adds complications to the process. In particular, adding metal films can cause problems when electrodes or other metallization is required to make the microfluidic device function.

2.8 Laser Welding of Plastics

Similar to the localized welding of microfluidic devices, laser welding involves generating a weld seam across the interface through the use of laser energy. In many cases, in order to accomplish localized welding at the surface, the laser travels through a transparent cover plate and gets absorbed by an opaque substrate. While the weld seams generated by this method are good, the device by nature will no longer be optically clear because of the opaque substrate. Malek [28] explains in his review of laser bonding techniques, that the opaque-transparent laser bonding is well-established but there is still much room for innovation in transparent-transparent laser bonding. Existing methods of laser bonding that were discussed include using carbon black coatings as a selective absorber [29, 30], using a hybrid laser-IR welding technique, and using reverse conduction welding with an IR-absorbing backplate that absorbs and retransmits the heat through a thin-film cover plate [31]. Only the latter of these methods would result in an optically clear weld.

Also discussed though, were examples of microfluidic bonding using Clearweld®, an infrared absorbing dye made by Gentex, that was developed for welding optically transparent polymers [32]. This dye absorbs certain wavelengths of NIR and IR radiation and is highly transparent in the visible. The dye can either be incorporated into the bulk, which leads to slight
discoloration, or can be applied as a thin film. This again would require the addition of a foreign material into the bonding area, which might not always be ideal. Lai [33] was able to successfully bond a three-layer microfluidic chip using Clearweld at the interface and a fiber-coupled 808nm diode laser. The laser beam moved around the channels to create a weld seam in the appropriate locations; this relative motion was accomplished with moving stages. In a tensile test, the parts bonded in this way achieved a bonding strength of 58MPa.

2.9 Research Direction

Each of the above methods that have been reviewed has its distinct advantages and disadvantages, and clear areas for possible innovation. One of the main goals of this research was to investigate a method that could be adapted to high-throughput manufacturing processes, such as the MIT μFac project. Of all of the methods mentioned, the thermal bonding method was the simplest in terms of process complexity and had the most examples of scale-up for manufacturing. Furthermore, other than ultrasonic welding, it was the only method that also generated an optically clear homogenous bond. For these reasons, thermal bonding and localized welding were at the forefront of thinking in choosing a research direction.

The next chapter will look at the mechanisms behind thermal bonding in more detail and will also investigate the idea of localized welding/interface heating.
3 Thermal Bonding and Interface Heating

3.1 The Thermal Bonding Process

As was described in chapter 2, during the thermal bonding process, the substrate and cover plate are heated to a temperature greater than the glass transition temperature and pressure is applied to facilitate bonding across the substrate-cover plate interface. A schematic of this process is shown in Figure 3-1 below.

![Figure 3-1. Schematic of a conventional thermal bonding setup. The substrate and cover plate are clamped in between two heaters. Both pieces are brought up to a temperature greater than their glass transition temperature and pressure is applied to facilitate bonding across the interface.](image)

The physics of what is happening at a molecular level during the bonding process is rooted in the reptation model of diffusion. When the material heats up, the polymer chains begin to flow. It is the intertwining of these chains across the interface that causes the bonding to occur. In the reptation model [34], shown in Figure 3-2 below, the molecules are described as sliding, or “reptating,” through a “tube.” The tube contours are defined by the locus of entanglements with neighboring molecules, with the motion of the polymer chains transverse to the contour of the tube severely restricted.
The diffusion process is measured by the diffusion coefficient of the polymer, which is dependent on the temperature and viscosity of the material. The stages of diffusion can be described as is shown in Table 3-1 below.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Time Stage Starts</th>
<th>What’s Happening</th>
<th>Average Displacement of Chain Segments</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>t₀</td>
<td>Chains do not feel constraints of surrounding chains</td>
<td>( \sim t^{1/4} )</td>
</tr>
<tr>
<td>2</td>
<td>t_{entangle}</td>
<td>Perpendicular movement restricted</td>
<td>( \sim t^{1/8} )</td>
</tr>
<tr>
<td>3</td>
<td>t_{Rouse}</td>
<td>Movement of segments of whole chain becomes correlated</td>
<td>( \sim t^{1/4} )</td>
</tr>
<tr>
<td>4</td>
<td>t_{reptation}</td>
<td>Fickian diffusion dominates</td>
<td>( \sim t^{1/2} )</td>
</tr>
</tbody>
</table>

The polymer’s radius of gyration, self-diffusion coefficient, and molecular weight define the \( t_{entangle} \), \( t_{Rouse} \), and \( t_{reptation} \) time constants. Kunz and Stamm [35] used neutron reflectometry to study the interface of protonated and deuterated PMMA films and estimated these constants.
to be 13min, 48min, and 1547min, respectively. The interface widths at these times were shown to be 3.9nm, 3.9-4.7nm, and 5.8-7.2nm respectively.

These times scales are very long compared to the desired time scales for a high-throughput scenario. The \( \mu \)Fac project targets a takt time of 5 minutes. With embossing processes taking on the order of minutes, a 2-hour long bonding process is not ideal. Elevating the temperatures of the polymer can decrease these time constants, which is a promising case for high-temperature thermal bonding. Furthermore, the polymers are placed under pressure for two reasons during the thermal bonding process. The first is to ensure that there is contact between the substrate and cover plate surfaces. The second is to facilitate the flow of the polymeric chains. As such, the combination of elevated temperatures and pressures can allow for faster bonding.

In a conventional thermal bonding setup, the temperature of the entire cover plate and substrate must be brought up to the bonding temperature. Therefore, bonding at high temperatures is not ideal because the entire substrate will soften, resulting in excessive channel deformation. However, in localized welding applications, heat is only applied at the interface, allowing the rest of the substrate to maintain its rigidity during the welding process. Focusing on this concept of interfacial heating can be powerful in a number of ways, which will be described in the next section.

### 3.2 Interfacial Heating

Interfacial heating can be beneficial in both welding and thermal bonding applications.

In welding, a thin layer at the interface is quickly melted and low pressures are applied to weld the surfaces together. The welding process occurs in three phases, shown in Figure 3-3 below [36]. In the first two stages, the interface has no mechanical properties and two distinct
faces still exist. In the last phase, wetting occurs, as intimate contact between the two surfaces has been achieved and potential barriers associated with inhomogeneities at the interface disappear. The molecular chains are now free to move across the interface in reptation. Once the reptation time has elapsed, the chain forgets its original configuration.

Figure 3-3. Phases of welding. a) and b) The interface has no mechanical properties and two distinct faces still exist. c) Wetting – intimate contact between the two surfaces has been achieved and potential barriers associated with inhomogeneities at the interface disappear. The molecular chains are free to move across the interface in reptation. Once the reptation time has elapsed, the chain has forgotten its original configuration. [36]

In interfacial heating for thermal bonding, the interfacial surfaces would be heated to a temperature greater than the glass transition temperature of the polymer. However, instead of the entire part being heated to this temperature, a temperature gradient would form throughout the thickness of the substrate and cover plate. As a result, the surface layer of the substrate would be the softest. When pressure is applied, this surface would experience the most deformation, with less deformation occurring throughout the thickness of the channels. The deformation that occurs is a result of both polymeric diffusion and basic compression of the plastic. There is an operating region right around the glass transition temperature in which the modulus of elasticity of a thermoplastic is highly dependent on the temperature. This relationship is shown below in Figure 3-4 [37]. This model is for shear modulus, but since the
Young’s modulus of a material is proportional to the shear modulus, its general trends are still useful to look at.

![Diagram](image)

Figure 3-4. Model for temperature dependence of shear modulus. The glass transition temperature of 80°C is specified here for PETG. Model can be used for other $T_g$'s as well. The $\Delta \theta$ gap is typically 30°C. [37]

As this model shows, in a region around $T_g$, the modulus is highly dependent on temperature. If the immediate surface were to be bonded at a temperature above $T_g$ but within the $\Delta \theta$ gap, then there would be a temperature gradient throughout the substrate, and also a modulus gradient throughout the substrate, with the modulus at the layers beneath the surface being higher than the modulus of the surface itself. As such, the substrate would be stiffer than the surface, resulting in less deformation of channels and more deformation right at the surface where the bonding occurs. At temperatures beyond this $\Delta \theta$ gap, there is still a small dependence of modulus on temperature, implying that this reasoning would be valid at high temperatures as well, depending on how steep the temperature gradients were. With this reverse temperature gradient, it might be possible to apply higher pressures with less total channel deformation in a pure thermal bonding application.
3.3 Research Focus

Based on the literature review and preliminary thinking on the topic, interfacial heating was chosen as the main bonding technology to focus on. A variety of methods were explored, each of which will be presented in the next chapters. For the analysis and thinking done here, the $\mu$Fac microfluidic mixer from Figure 1-1 was considered. This part is currently made out of PMMA. The thermal properties of the PMMA vary depending on the commercial grade of the material. For the analyses done here, the following thermal properties were assumed: $T_g=90^\circ\text{C}$, $T_{\text{melt}}=160^\circ\text{C}$, and $T_{\text{boil}}=200^\circ\text{C}$.
CHAPTER 4
Three-Stage Non-Contact Surface Heating

4.1 General Concept

The basic concept of three-stage, non-contact surface heating is depicted below in Figure 4-1. In the first step, the cover plate and substrate are held apart a small distance from one another and a heater heats the surfaces of each. In step 2, the heater is removed, and in step 3, pressure is applied to the cover plate-substrate stack in order to promote interfacial diffusion.

![Figure 4-1. The basic concept of three-stage, non-contact surface heating. In step 1, the cover plate and substrate are held apart a small distance from one another and a heater heats the surfaces of each. In step 2, the heater is removed and in step 3, pressure is applied to the cover plate-substrate stack in order to promote interfacial diffusion.](image)

This process is similar to hot tool welding and infrared welding described by Yousefpour et al in their review of fusion bonding and welding of thermoplastic composites [38]. However, this review focused on the welding of large rods of material in which case the preservation of fine features, such as microfluidic channels, was not important. A displacement-controlled hot plate welding system was described by Stokes [39]. In this system, the two pieces being welded were brought into contact with a hot tool and pressure was applied to ensure contact. The pressure was then lessened to allow the melt region to grow, the heater was removed, and the parts were brought together and held under pressure to allow the bond/weld to form. This system was used to bond thinner plates of plastic, on the order of a
few millimeters, as opposed to the long rods. However, in this case too, the weld thicknesses were at least on the order of a few hundred microns, which is thicker than many microfluidic features. Therefore, if this three-stage heating process were to be employed for microfluidic applications, a set of detailed simulations would first need to be conducted to test for feasibility and to find a viable set of heating parameters. The rest of this chapter is dedicated to those simulations.

4.2 Heat Transfer Simulations

4.2.1 Simulation Setup

To test the feasibility of this concept, a series of finite element analyses were conducted using SolidWorks Simulation. These simulations considered two blocks of acrylic, each 1.58mm thick. The cross-sectional area of each block was a 1.58mm x 1.58mm square in order to create an efficient mesh. The mesh was such that the temperature was measured every 100 microns through the thickness of each piece.

Each block started at a given initial temperature. Any initial temperature above room temperature assumed that the entire part had been previously heated in a convection oven or by some other means before undergoing the surface heating process. In the first step, a heat flux was applied to the interfacial surfaces of both the substrate and cover plate for a specified heating time. There was also a convection condition applied to the interfacial surfaces. In the second step, the heat flux was removed, but the convection condition remained the same. The duration of this second step was termed the changeover time. In the third step, the two pieces were brought together for a specified bonding time and there was only heat transfer by conduction. The temperature profile throughout the thickness of the substrate and cover plate was monitored at each time step.
4.2.2 Initial Simulations

An initial simulation was completed to get an idea of the approximate time and heat fluxes required. The parameters used are summarized in Table 4-1 below. Heat fluxes are in units of Watts/chip. This is the amount of power that would be required to heat the 25.4mm x 25.4mm muFac chip.

Table 4-1. Heating parameters for initial three-stage heating simulations.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>67 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>67 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Bonding Time</td>
<td>3 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>17 W/m²·K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>22°C</td>
</tr>
<tr>
<td>Cover Plate Initial Temp</td>
<td>22°C</td>
</tr>
<tr>
<td>Substrate Initial Temp</td>
<td>22°C</td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
</tbody>
</table>

The temperature trends over time at various positions throughout the thickness of the cover plate are shown below in Figure 4-2.
Figure 4.2. Temperature vs. time for various nodes throughout the thickness of the cover plate during simulation of three-stage heating process. Simulation parameters are those from Table 4-1. The z-values represent the distance of the node from the interface surface.

The temperature profiles through the thickness of the cover plate at the beginning and end of the changeover stage are shown below in Figure 4-3.
It is clear from the above graphs, that there is rapid heat loss from the surface of the cover plate during the changeover period. These losses are a combination of convective losses to the surrounding environment and conduction losses to the rest of the cover plate. To minimize convective losses, efforts could be taken to minimize the gap in between the two parts, thus restricting airflow. Furthermore, the entire process could occur inside an oven to lessen the surface-ambient temperature difference. To minimize conduction losses, the entire cover plate and substrate could be pre-heated to a temperature below $T_g$ or a constant temperature heat source can be applied to the back of the cover plate during changeover, such that the surface-bulk temperature difference would be smaller. The simulations in the next sections test the effects of these heating conditions and show that the conduction through the bulk causes a majority of the rapid heat loss from the surface.
The temperature profile throughout the cover plate and substrate when the two are bonded together in stage 3 is shown below in Figure 4-4.

![Figure 4-4](image)

Figure 4-4. The temperature profile throughout the thickness of the substrate and cover plate when the two are bonded together; profiles are from stage 3 of the 3-stage bonding process. The bold profile line is the initial temperature profile after stages 1 and 2, using parameters from Table 4-1. Arrows indicate progression of time from 0 to 3 seconds. Each new profile line represents a time step of 0.1 seconds.

The simulation data shown in Figure 4-4 made it clear that a three-stage heating scheme would not make it possible to thermally bond the parts, but rather that welding would be necessary. As was described in the last chapter, bonding requires sufficient time, at least on the order of minutes, sometimes as high as 20 minutes, to cause the polymer chains to intertwine and a bond to form. In the above simulation it takes a matter of only seconds for the profile to flatten, which is not enough time for a sufficient thermal bond to form. Once the profile is flat, the benefits of surface heating are no longer present. In welding however, once the interface surfaces reach $T_{\text{melt}}$, it takes a matter of seconds for the weld to form. As such, the goal of the
following simulations is to find a series of conditions such that the surface temperatures of the cover plate and substrate are greater than $T_{\text{melt}}$ at the end of the changeover period.

### 4.2.3 Minimizing Convective Losses

Minimizing convective losses during the heating and changeover periods can be accomplished by changing the ambient temperature and/or the convection coefficient of the environment in which the process is taking place.

To test the effect of the ambient temperature, two simulations were run with the parameters shown in Table 4-2. The two ambient temperatures tested were 22°C, which is room temperature, and 90°C, which is the $T_g$ of acrylic.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>18.6 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>18.6 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>17 W/m²K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>22°C or 90°C</td>
</tr>
<tr>
<td>Cover Plate Initial Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Substrate Initial Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
</tbody>
</table>

The surface temperatures were recorded at the end of the heating and changeover stages and the results are shown below in Table 4-3.

<table>
<thead>
<tr>
<th>Ambient Temperature</th>
<th>Temperature After Heating</th>
<th>Temperature After Changeover</th>
<th>$\Delta T$ during changeover</th>
</tr>
</thead>
<tbody>
<tr>
<td>90°C</td>
<td>138.30°C</td>
<td>108.35°C</td>
<td>29.95°C</td>
</tr>
<tr>
<td>22°C</td>
<td>138.36°C</td>
<td>105.47°C</td>
<td>30.89°C</td>
</tr>
</tbody>
</table>

In this example, the heat fluxes were not high enough to raise the surface temperature to $T_{\text{melt}}$. However, the results still show that changing the ambient temperature only accounts for
of the change in temperature during changeover. Thus, altering ambient temperature will not cause a significant difference in the heat loss during the changeover period.

To test the effect of the convection coefficient, two simulations were run with the parameters shown in Table 4-4 and the surface temperatures were recorded at the end of the heating and changeover stages. The two convection coefficients tested were 17 W/m²-K, which is typical for still air, and 0 W/m²-K, which is the extreme case to test the effect of eliminating convection.

Table 4-4. Simulation parameters for convection coefficient study. Two simulations were run, each with different convection coefficients.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>17 W/m²-K or 0 W/m²-K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>160°C</td>
</tr>
<tr>
<td>Cover Plate Initial Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Substrate Initial Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
</tbody>
</table>

The resulting surface temperatures for the two simulations are shown below in Table 4-5.

Table 4-5. Simulated surface temperatures after heating and changeover stages for varying convection conditions.

<table>
<thead>
<tr>
<th>Convection Coefficient</th>
<th>Temperature After Heating</th>
<th>Temperature After Changeover</th>
<th>ΔT during changeover</th>
</tr>
</thead>
<tbody>
<tr>
<td>17 W/m²-K</td>
<td>255.22°C</td>
<td>154.47°C</td>
<td>100.75°C</td>
</tr>
<tr>
<td>0 W/m²-K</td>
<td>261.69°C</td>
<td>157.63°C</td>
<td>104.06°C</td>
</tr>
</tbody>
</table>

This result indicates that the convection coefficient only accounts for ~4% of the temperature drop during the changeover stage. Thus, changing the convection coefficient will not make a significant difference in the temperature drop during changeover. Thus, by combining this result with the ambient temperature study results, it is concluded that the heat losses during changeover are not a result of the convection.
It should also be noted that the temperatures after the heating stages in Table 4-5 are greater than the boiling temperature of acrylic, which is 200°C, and yet the temperature after changeover is still less than acrylic’s melting temperature of 160°C. It is unacceptable for the acrylic to boil; this result raises this as another issue that will have to be considered when choosing operating parameters.

4.2.4 Minimizing conduction losses

To minimize conduction losses, the entire cover plate and substrate could be pre-heated to a temperature below $T_g$ or a constant temperature heat source can be applied to the back of the cover plate during changeover, such that the surface-bulk temperature difference would be smaller.

To test the effect of pre-heating the cover plate, two simulations were run with the parameters shown in Table 4-6 and the surface temperatures were recorded at the end of the heating and changeover stages. The two $T_{\text{initial}}$ tested were 25°C, which is about room temperature, and $T_g$, 90°C.

Table 4-6. Simulation parameters for cover plate pre-heat study. Two simulations were run, each with different $T_{\text{initial}}$.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>0 W/m²-K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>160°C</td>
</tr>
<tr>
<td><strong>Cover Plate/Substrate Initial Temperature</strong></td>
<td><strong>25°C or 90°C</strong></td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
</tbody>
</table>

The resulting surface temperatures for the two simulations are shown below in Table 4-7.
Table 4-7. Simulated surface temperatures after heating and changeover stages for varying initial temperature conditions.

<table>
<thead>
<tr>
<th>Cover plate/substrate initial temperature</th>
<th>Temperature After Heating</th>
<th>Temperature After Changeover</th>
<th>ΔT during changeover</th>
</tr>
</thead>
<tbody>
<tr>
<td>25°C</td>
<td>196.69°C</td>
<td>92.63°C</td>
<td>104.06°C</td>
</tr>
<tr>
<td>90°C</td>
<td>261.69°C</td>
<td>157.63°C</td>
<td>104.06°C</td>
</tr>
</tbody>
</table>

These results prove that the temperature drop during the changeover period cannot be made smaller by preheating the cover plate and substrate. This behavior can be expected because the change in surface temperature is dynamic behavior for which $T_{initial}$ is just a starting point. Preheating does help though in getting $T_{surface}$ closer to $T_{melt}$ after the changeover period. However, the results show that achieving $T_{surface, after changeover} > T_{melt}$ is difficult, especially considering that in this case $T_{after heating} > T_{boil}$.

To determine the effect of adding a constant temperature boundary condition to the back of the cover plate, three simulations were run with the parameters shown in Table 4-6 and the surface temperatures were recorded at the end of the heating and changeover stages. Constant temperature boundary conditions of 25°C, 90°C, and 110°C were used. This temperature condition was only applied to the back of the cover plate and substrate during the changeover stage.

Table 4-8. Process parameters for study of constant temperature boundary conditions applied during changeover to back of cover plate and substrate. Two simulations were run, each with a different $T_{boundary}$.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>65 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>0 W/m²·K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>25°C</td>
</tr>
<tr>
<td>Cover Plate/Substrate Initial Temperature</td>
<td>25°C</td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
<tr>
<td>Constant Temperature Boundary Condition</td>
<td>25°C, 90°C, or 110°C</td>
</tr>
</tbody>
</table>

The resulting surface temperatures for the two simulations are shown below in Table 4-9.
Table 4-9. Simulated surface temperatures after heating and changeover stages for varying constant temperature boundary conditions.

<table>
<thead>
<tr>
<th>Boundary Temperature</th>
<th>Temperature After Heating</th>
<th>Temperature After Changeover</th>
<th>ΔT during changeover</th>
</tr>
</thead>
<tbody>
<tr>
<td>25°C</td>
<td>203.1°C</td>
<td>98.91°C</td>
<td>104.19°C</td>
</tr>
<tr>
<td>90°C</td>
<td>203.1°C</td>
<td>98.98°C</td>
<td>104.12°C</td>
</tr>
<tr>
<td>110°C</td>
<td>203.1°C</td>
<td>98.99°C</td>
<td>104.11°C</td>
</tr>
</tbody>
</table>

These results show that applying a constant temperature boundary condition to the back of the substrate and cover plate has a <1% effect on the temperature drop during changeover.

One last method of potentially decreasing the surface temperature drop is to apply heat over a longer period of time during the heating stage. In this way, the temperature profile throughout the thickness of the substrate and cover plate would move closer to steady state and there would be less opportunity for heat loss across that temperature gradient during the changeover period. To test this, three simulations were run, varying heat flux and heating time. Heating times of 0.4s, 0.9s, and 1.9s were used. To keep the total heat applied to the cover plate and substrate surfaces approximately equal for each of the simulations, heat fluxes of 67W, 34W, and 17W were used respectively. The simulation parameters are shown below in Table 4-10.

Table 4-10. Process parameters for heating time/heat flux study. Two simulations were run, each with a different heating time.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cover Plate Heat Flux</td>
<td>67, 34, or 17 W/chip</td>
</tr>
<tr>
<td>Substrate Heat Flux</td>
<td>67, 34, or 17 W/chip</td>
</tr>
<tr>
<td>Heating Time</td>
<td>0.4, 0.9, or 1.9 seconds</td>
</tr>
<tr>
<td>Changeover Time</td>
<td>1.1 seconds</td>
</tr>
<tr>
<td>Convection Coefficient</td>
<td>17 W/m²·K</td>
</tr>
<tr>
<td>Ambient Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Cover Plate/Substrate Initial Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Simulation Time Step</td>
<td>0.1 seconds</td>
</tr>
</tbody>
</table>

The resulting temperatures for the two simulations are shown below in Table 4-11.

Since the temperatures after heating were so different for the three scenarios, it was important to also look at the percent change in surface temperature during the changeover stage.
Table 4-11. Simulated surface temperatures after heating and changeover stages for varying heating time/heat flux combinations.

<table>
<thead>
<tr>
<th>Heating Time</th>
<th>Temperature After Heating</th>
<th>Temperature After Changeover</th>
<th>( \Delta T ) during changeover</th>
<th>% ( \Delta T_{\text{surface}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4 seconds</td>
<td>143.01°C</td>
<td>59.89°C</td>
<td>83.12°C</td>
<td>58%</td>
</tr>
<tr>
<td>0.9 seconds</td>
<td>115.08°C</td>
<td>60.90°C</td>
<td>54.18°C</td>
<td>47%</td>
</tr>
<tr>
<td>1.9 seconds</td>
<td>90.96°C</td>
<td>58.41°C</td>
<td>32.55°C</td>
<td>36%</td>
</tr>
</tbody>
</table>

Heating the parts quickly allows the surface to reach higher temperatures during the heating stage but this is accompanied by a greater temperature drop during the changeover stage. Thus, at first glance it appears that heating the surface for a longer time at lower flux can minimize the temperature drop during changeover and make this surface heating method more viable. However, these smaller temperature drops are a consequence of the smaller temperature gradients at the surface that come with the longer heating time/smaller heat flux combination. In order for this surface heating for welding option to work, it is critical that the temperature gradient at the surface is no smaller than 1°C/µm so as to minimize how much of the surface is truly melting. The surface temperature gradients for the above scenarios are shown below in Table 4-12. These were measured by taking the temperatures at the surface nodes and at nodes 100µm beneath the surface (the mesh elements were 100µm deep).

Table 4-12. Temperature gradients at surface of part after heating and changeover stages for varying heating time/heat flux combinations.

<table>
<thead>
<tr>
<th>Heating Time</th>
<th>Surface Temperature Gradient After Heating</th>
<th>Surface Temperature Gradient After Changeover</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4 seconds</td>
<td>0.35°C/µm</td>
<td>0.0087°C/µm</td>
</tr>
<tr>
<td>0.9 seconds</td>
<td>0.20°C/µm</td>
<td>0.0074°C/µm</td>
</tr>
<tr>
<td>1.9 seconds</td>
<td>0.10°C/µm</td>
<td>0.0050°C/µm</td>
</tr>
</tbody>
</table>

The surface temperature gradients of the parts after changeover are 2-3 orders of magnitude smaller than the allowable temperature gradient for this application. Even the temperature gradients after the heating stage are an order of magnitude smaller than the requirement, indicating that even eliminating the changeover stage might not make this method viable. If a set of parameters were found that did make it feasible, it is clear from the above
simulations that there would be such a small window of usable parameters, that it would not be a robust process.

In order to round out the simulations and the study of this general concept, a few brief investigations of the bonding stage were conducted.

4.2.5 Bonding/Welding Stage

The actual bonding/welding occurs during the third stage of the non-contact surface heating process described in Figure 4-1. To simulate this stage, the cover plate and substrate were brought into contact with zero contact resistance. The initial temperature conditions were the temperature profiles obtained from the heating stage simulations. The simulations in the previous sections proved the difficulty of achieving $T_{\text{melt}}$ at the surface after changeover. As was mentioned before, this three-stage process would require bonding by welding because the surface cools too quickly after the heat source is removed. However, it is also interesting to note what would happen if the cover plate is preheated to a temperature greater than $T_{\text{melt}}$. Since $T_{\text{boil}}$ of the acrylic being studied is 200°C, this is the highest possible pre-heat temperature for the cover plate, though such a preheat would be very risky. For the purpose of testing extremes, if the cover plate were heated to 200°C and the substrate to $T_g$ at 90°C, then the equilibrium surface temperature would still only be 145°C, which is below $T_{\text{melt}}$ and much greater than $T_g$. Since time and pressure would be required to bond the two surfaces, channel deformation and shape recovery would begin to occur. In order to reach $T_{\text{melt}}$ at the surface with $T_{\text{cover plate}}$ at 200°C, $T_{\text{substrate}}$ would have to equal 120°C after a preheat, which is not realistic.

It is potentially possible to preheat the cover plate to 200°C and the substrate to $T_g$ at 90°C, and then surface heat the substrate such that the resulting surface temperature during the bonding stage is above $T_{\text{melt}}$. However, at this point, the process becomes unwieldy and moves
away from its original purpose, which was to simply and quickly surface heat such that a fast weld could be generated.

The main problem with this method is that the heating and bonding processes are separated. The bonding, or welding, needs to occur while the heat source is being applied to the surface. It is thus interesting to look at other methods of surface heating. As discussed earlier in Chapter 2, there are a variety of methods already being studied, such as ultrasonic bonding, microwave welding, and the application of selectively absorbing coatings. The next two chapters will focus on the exploration of this last idea – the application of selectively absorbing coatings to the interface surfaces.
5. Heat Absorbing Surface Coatings

5.1 Surface Gold Nanoparticles

The use of gold nanoparticles for selective heating was originally inspired by the work of the Hamad-Schifferli group at MIT [40]. This research group uses laser pulses to excite gold nanorods dissolved in a biological solvent. Since the laser pulse is transmitted through the solvent, the solvent only heats up at the surfaces of the gold nanorods. In a similar way, if gold nanoparticles were to be deposited on the surfaces of the substrate and cover plate, a laser pulse of a specific wavelength— one that PMMA is transparent to — can be sent through the microfluidic chip, its energy only being absorbed by the nanoparticles at the interface. In fact, since these particles are made of gold, microwaves can even be used. A solution of these particles would be selectively applied to the surface by a process such as microcontact printing, thus eliminating channel contamination. Since the particles are so small and spread out in their solution, the interface would still exhibit optical clarity, an improvement upon conventional microwave bonding.

There have been a number of studies conducted in the last few years about the thermal excitation of gold nanoparticles [41 – 44]. There are in particular a couple of studies that were especially helpful in evaluating the use of gold nanoparticles as a selectively absorbing coating for microfluidic applications.

Sassaroli et al completed a numerical study in which they investigated the thermal response of gold nanoparticles, or GNP’s, to laser pulses on the order of nanoseconds [45]. The thermal response of the surrounding medium was also studied. In this case, the surrounding
medium was water, with a thermal diffusivity of $1.53 \times 10^{-7} \text{ m}^2/\text{s}$. PMMA has a similar thermal diffusivity, $1.25 \times 10^{-7} \text{ m}^2/\text{s}$, and since the thermal response is mostly dependent upon this property, the results from Sassaroli’s study can be helpful here.

In a simulation to study how the temperature profile in the GNP and its surroundings vary over time, a laser pulse with an intensity of $2.5 \times 10^9 \text{ W/m}^2$ and duration of 1000 seconds was used. In 1000 seconds, the temperature at the surface of the GNP reached 100°C. The temperature gradient in the water immediately adjacent to the nanoparticle was $30 \degree \text{C}/(\text{particle radius})$. As such, if particles with a diameter of 40nm were used on the PMMA, the temperature gradient at the surface would be $1500 \degree \text{C}/\mu\text{m}$, which is substantially greater than the $1 \degree \text{C}/\mu\text{m}$ requirement. The profile obviously flattens at points further from the particle and within 0.6 μm, the material is unaffected by the heating.

Bendix et al [46] generalizes this response with the formula in Equation (5-1) which describes temperature as a function of distance from the GNP.

$$
\Delta T(D) = \frac{R^3 A}{3DK}
$$

where $D$ is the distance from the center of the GNP, $R$ is the radius of the GNP, $A$ is the heating rate per unit volume of the particle, and $K$ is the thermal conductivity of the surrounding material. This study included experimental results in which GNPs were optically trapped on a lipid bilayer and the fluorescence this bilayer was measured to determine the degree of melting.

Such a thermal response is optimal when considering the temperature gradient through the thickness of the part. However, the heating of the surface is also dependent upon the overlapping of temperature profiles from adjacent nanoparticles. Based on Sassaroli’s simulation, in order to get even heating across the surface, particles would need to be carefully positioned within less than a micron from each other. Results from Bendix’s study imply that
this spacing could be as high as a few microns when larger GNPs are used (d=200nm). Spacing this small could likely affect the original desire for optical clarity at the interface. Furthermore, typical solutions of gold nanoparticles [47] are sold in concentrations that would allow for spacing on the order of 15 microns, an order of magnitude larger than the few microns that would be desired for even heating. As such, depositing particles of the desired spacing would require more complex printing.

In the further consideration of using GNPs as a selectively absorbing coating, two other issues became clear. First, the even heating across the interface does not only require a high enough concentration of GNPs, but they must also be precisely positioned even distances away from each other. While microcontact printing would allow for precise positioning of the solution around the microfluidic channels, it would not be possible to precisely position individual nanoparticles. Secondly, once the solution is printed on the substrate and cover plate, as the water or other medium holding the particles in suspension begins to dissolve, the particles will come out of suspension and could escape into the surrounding air or channels. This would be both a contamination and quality control problem.

In conclusion, the use of gold nanoparticles as an interface coating, while innovative, is not feasible from a technical sense. The one benefit from the GNPs was the steep temperature gradient that could be achieved through the thickness of the substrate and cover plate. As such, it is desirable to find a different coating that will allow for a similar temperature gradient without the uniformity issues across the surface. The next chapter will discuss a near-infrared absorbing dye that was experimentally investigated.
5.2 Selectively Absorbing Dyes

5.2.1 Overview

As was described earlier in Chapter 2, there is much research into the use of selectively absorbing coatings to selectively heat the surface of microfluidic devices during the bonding process. In many of these applications, a narrow beam laser light is used as the energy source. From a production and automation point of view, using lasers is less ideal because only a single seam weld can be made during each pass of the laser. Furthermore, in many of these examples, selective heating is achieved by weaving the laser around the existing microfluidic channels. Depending on how complicated the channel patterns are, this process can become very timely and a new laser path will need to be generated for every new device that needs to be bonded.

Leister Laser Corporation has developed and patented a mask welding system [48], shown below in Figure 5-1 that allows for faster laser welding of plastics. In this system, a laser absorbing plastic and a transparent plastic are being joined. The two are placed underneath a mask of the desired micropattern. When a curtain of light passes over it, the light is absorbed by the plastic interface in the desired pattern.

![Figure 5-1. Leister Laser Mask Welding. The parts to be welded are placed under a mask of the desired micropattern. When the curtain of light passes over it, the light is selectively absorbed by the plastic in the pattern of the mask. [48]](image-url)
A similar system could potentially be implemented in the case of using selectively absorbing infrared dyes, such as Clearweld, which was described earlier in Chapter 2. In this case, two pieces of transparent plastic can be joined. As is shown in Figure 5-2, once the dye absorbs the correct amount of light, the interface becomes optically clear, which is a desired property of microfluidic bonds. If the dye were to be selectively printed onto the substrate and cover plate through a process such as microcontact printing, then the light would only be absorbed by those regions that were coated with the dye. Furthermore selective printing would prevent channel contamination.

![Figure 5-2. Transmission curves of PMMA in three cases. [49]](image)

The use of a selectively absorbing dye has both proven examples in industry and many opportunities for innovation, making it an intriguing option for further exploration. A process overview is shown below in Figure 5-3.
For optimal performance, the NIR source must provide sufficient power at the correct wavelengths to be absorbed by the dye. Thus, in order to begin exploring this option, basic heat transfer simulations were conducted to get an idea of the power requirements.

### 5.2.2 Basic Heat Transfer Simulations

The basic system shown in Figure 5-4 below was modeled using finite element analysis.

![Figure 5-4. Basic surface heating model.](image)

A small element size of 8μm was used such that the temperatures and temperature gradients at only 1μm beneath the surface could be extrapolated. Since the last two ideas were focused on welding, this investigation began with a look into welding as well. Varying heat fluxes, represented by $\dot{Q}$ in the figure above, were applied to the surface of a piece of PMMA...
and the temperature profiles were monitored over time. Specific attention was paid to the temperature 1μm below the surface. When the finite element nodes at this position reached $T_{\text{melt}}$, the time was recorded. These times were compiled for different heat fluxes as shown in Figure 5-5.

![Graph](image)

Figure 5-5. Time for 1micron of PMMA surface to reach $T_{\text{melt}}$, 160°C, for varying heat fluxes applied to surface of chip (cover plate or substrate). Determined by finite element simulations.

It is clear from the above results that when the power applied to the surface of a chip is greater than ~50W, fast welds can occur. This defines the region of ideal operating points, for small deviations in power will not greatly impact the time required for welding. It is also important, especially in welding applications, to monitor the temperature gradient at the surface of the part. These temperature gradients were recorded at the moments when the surface temperature reached $T_{\text{melt}}$, and are reported in Figure 5-6 below.
Figure 5-6. Surface temperature gradients (through thickness of part) at moment when the temperature at 1 μm deep reached $T_{\text{met}}$. As determined by finite element analysis for various applied heat fluxes.

These temperature gradients are smaller than the previously stated minimum of $1^\circ\text{C}/\mu\text{m}$, except at powers greater than 150W per chip. These findings indicate that true welding might not be possible, but since the heating occurs while the substrate and cover plate are in contact and under pressure, it will be possible for high temperature thermal bonding to occur. At high temperatures, it takes less pressure and time for the polymer chains to diffuse across the interface and a bond to form. As explained earlier in Chapter 3, there is an extreme temperature dependence of viscoelastic properties at temperatures around $T_g$ and a small temperature dependence at higher temperatures. Because of the reversed temperature gradient at the surface, the presence of even a small temperature gradient will facilitate more robust high temperature thermal bonding.

Since thermal bonds do not occur instantly, there are two possible heating schemes that could be implemented. The first is to apply high heat flux (>50W/chip) to the surface, allow the
surface to quickly reach $T_{\text{desired}}$, and then turn the light power down in order to maintain $T_{\text{desired}}$ at the surface while the bonding occurs. The other is to use a lower heat flux and allow the bond to begin forming as the temperature slowly rises. Temperature/heat flux control will then have to be implemented in order to ensure that the temperature does not exceed $T_{\text{melt}}$. Both of these methods were kept in mind while exploring possible heating methods.

In order to evaluate heating methods, the absorption spectrum of both the PMMA that the light needs to pass through, as well as the dye that the light needs to be absorbed by, must be known. The next two sections will discuss these spectra.

5.2.3 Absorption Spectrum of PMMA

PMMA is highly transparent to visible light with 92% of visible light being transmitted. However, it does absorb select wavelengths of near-infrared and infrared light. A general transmission spectrum for a piece of 1/8" thick acrylic is shown in Figure 5-7 below [50].

![Graph showing the absorption spectrum of PMMA](image)

Figure 5-7. The transmission spectrum of 1/8" thick PMMA (acrylic) as reported by Fresnel Technologies, Inc. [50] The solid line (general purpose acrylic) is the material of interest for this application.
This spectrum indicates that almost all light with wavelengths less than 1μm will pass through the PMMA. Thus, it would be most ideal to choose a coating that dominantly absorbs light in this wavelength region.

5.2.4 Coating Selection

The NIR-absorbing coatings on the market are powders that are mixed into a solvent such as water, ethanol, or acetone in varying concentrations. For this study, the powder was purchased from Fabricolor Holdings, llc. [51]. There were four recommended dyes based on optical clarity and thermal requirements. It is critical for the dye to become optically clear after absorbing the necessary light energy. Furthermore, the dye must be stable at temperatures as high as 200°C, which allows for a margin of safety if the surface reaches PMMA’s melting temperature.

Each of these four coatings is soluble in acetone and ethanol. The dyes have much higher solubility in acetone, thus allowing for greater concentration in the solution and greater light absorption in the dried coating. However, PMMA is not chemically resistant to acetone. Acetone is actually frequently used for solvent bonding because of its ability to chemically alter the PMMA surface, and so it is not an ideal solvent for this application, as the goal is to avoid all chemical altering of the PMMA. Ethanol has been used before in order to clean PMMA, making it a potentially better choice for a solvent. In trials, the PMMA was visibly dissolved by the acetone, but appeared to remain unaffected by the ethanol. This would need to be tested further (chemically) in order to promote this general concept as having minimal effect on the surface chemistry. While the ethanol appears to be more compatible with the PMMA, the solubility of the Fabricolor dyes in ethanol is significantly lower than in acetone (3% solubility compared to 12%), resulting in less light absorption for a given volume of coating solution.
The four dye options have peak absorption at wavelengths of 808nm, 848nm, 1002nm, and 1031nm. A sample absorbance profile is shown below in Figure 5-8.

Figure 5-8. Absorbance profile of Fabricolor Dye 8472, peak absorption at 848nm. [51]

It should be noted that while the dye does absorb the most light at 848nm, it does exhibit absorption at other wavelengths between 600 and 900nm. As a result, it is possible that utilizing a light source with emission across all wavelengths in this range will result in even more absorption from this particular coating. Each of the four recommended dyes had absorbance profiles with similar shapes, and thus the same principle could be applied in the case of each coating.

5.2.5 Investigation of Light Sources: IREDs

Considering the idea of implementing a light source that emits light in a range of wavelengths, there were three main options for light sources to be explored: single-wavelength
narrow-beam lasers, IREDs that emit light in a small range of wavelengths, and broadband light sources such as a tungsten-halogen light bulb that emit light in a large range of wavelengths. Single-wavelength narrow-beam lasers are both expensive and already extensively studied and implemented. And so, more effort was placed on these last two options. Since IREDs have light emission spectra that more closely resemble the coatings’ absorption spectra, they were a logical option to begin with.

An IRED is a light emitting diode that emits infrared light. The light is emitted in a Lambertian pattern with a substantial amount of its light falling within a cone defined by the diode’s half power beam angle. As the IRED moves away from a surface, the spot diameter on that surface grows larger but the heat flux incident on the surface decreases as defined by Equation (5-2) below. All of the following equations are based on the IRED Application Notes published by Perkin Elmer [52].

\[ E_{d_2} = E_{d_1} \left( \frac{d_1}{d_2} \right)^2 \]

where \( E_{d_1} \) and \( E_{d_2} \) are the irradiances (measured in W/m\(^2\)) at distances \( d_1 \) and \( d_2 \) from the diode.

For this analysis, the spot size on a surface a distance \( d \) from the diode is defined as in Equation (5-3) below.

\[ d_{\text{spot}} = d \tan(\theta_{1/2}) \]

where \( \theta_{1/2} \) is the diode’s half power beam angle. Any light at this angle is at half of the maximum intensity (maximum intensity occurs at the center of the beam). This is the equivalent of studying the light emitted within a cone of angle \( \Omega \) as is described in Equation (5-4) below.
\[ \Omega = 0.25 \pi \tan^2 \left( \frac{\theta_{1/2}}{2} \right) \]  

(5-4)

where \( \Omega \) is measured in steradians (sr). For a given IRED, the spot sizes and heat fluxes were found at a few distances from the diode using the above equations in conjunction with (5-5) below.

\[ M = \frac{I_e \Omega}{0.25 \pi d_{\text{spot}}^2} \]  

(5-5)

where \( M \) is the heat flux in mW/mm\(^2\), \( I_e \) is the radiant intensity in mW/sr and \( \Omega \) and \( d_{\text{spot}} \) are based on the distance from the diode and are defined in the equations above. Based on the calculation for radiant exitance, the time for the spot on a surface to reach \( T_{\text{melt}} \) is approximated from the graph in Figure 5-5 above. While this application will likely move from welding to high temperature thermal bonding, a measure of the time to reach \( T_{\text{melt}} \) is an approximation of the time to reach a desired temperature for comparative purposes across different diodes and distances. Based on this time, the total time necessary to bond an entire chip could be estimated. This is a function of the number of diodes used and the spot size covered for one pass of the diode across the surface. Two configurations were tested: one in which a line of diodes moved back and forth across the length of the chip and another in which an array of diodes across the whole chip moved small distances, allowing each diode to bond an area equivalent to the area of the diode itself. Since IREDs are extremely inexpensive (<$1 per diode), it is possible to implement many of them across a chip.

The total part bonding times using a line and array scan for Perkin Elmer Part #VTE1163H, a typical IRED, are shown below in Table 5-1. This diode has a center wavelength of 880nm, a radiant intensity of \( I_e=285\text{mW/sr} \), and a half power beam angle of 10°.
Table 5-1. Spot sizes and bonding times for Perkin Elmer VTE1163H IRED.

<table>
<thead>
<tr>
<th>Distance from Diode</th>
<th>Spot Diameter</th>
<th>Spot Time</th>
<th>Line Scan Time</th>
<th>Array Scan Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5mm</td>
<td>0.09mm</td>
<td>0.01 seconds</td>
<td>155 seconds</td>
<td>28 seconds</td>
</tr>
<tr>
<td>1.0mm</td>
<td>0.18mm</td>
<td>0.1 seconds</td>
<td>410 seconds</td>
<td>73 seconds</td>
</tr>
<tr>
<td>1.5mm</td>
<td>0.26mm</td>
<td>0.4 seconds</td>
<td>724 seconds</td>
<td>128 seconds</td>
</tr>
<tr>
<td>3.0mm</td>
<td>0.52mm</td>
<td>4.7 seconds</td>
<td>1910 seconds</td>
<td>338 seconds</td>
</tr>
<tr>
<td>5.0mm</td>
<td>0.88mm</td>
<td>26.6 seconds</td>
<td>3904 seconds</td>
<td>692 seconds</td>
</tr>
<tr>
<td>10.0mm</td>
<td>1.76mm</td>
<td>280.3 seconds</td>
<td>10304 seconds</td>
<td>1825 seconds</td>
</tr>
</tbody>
</table>

Based on the calculated required bonding times reported in Table 5-1 above, the only way that a line scan would be possible with this IRED is if the diode were placed 0.5mm or less away from the coating. However, this thickness will be entirely consumed by the substrate or cover plate. In fact, it would have to be the cover plate because the substrate in this case is already 1.5mm thick, and so a thin cover plate would need to be used. Moving the IRED any further away from the part will cause the line scan time to become even greater than conventional bonding times. It should be noted that the long time scales are the result of the very small spot sizes compared to the size of the IRED itself; the calculations were completed with the IREDs packed as tightly as possible. For such small spot sizes, it would be more worthwhile to just use a single wavelength laser.

When the IRED moves further away, the fluxes become too low to achieve appreciable heating. Even though this analysis considers the time to reach $T_{\text{melt}}$, the time scale is on the right order of magnitude to determine that there is just not enough power when the IREDs are far away from the targeted surface. Array scans will allow for more reasonable heating times at distances up to 1.5mm away. However, 1.5mm spacing is still difficult to achieve and the nature of the array scan could produce uneven welds.

While conventional IREDs do not seem powerful enough for this application, the OSRAM Ostar Observation IR Emitter (SFH 4740) has four times as much power as conventional IREDs, with a radiant intensity of $I_e=1200\text{mW/sr}$ compared to $I_e=285\text{mW/sr}$.  

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Furthermore, it emits a total of 3.6W of power compared to 0.11W with IREDs. A similar time analysis was conducted for this emitter and the results are presented in Table 5-2 below. This emitter has a center wavelength of 850nm and a half power beam angle of 60°.

<table>
<thead>
<tr>
<th>Distance from Diode</th>
<th>Spot Diameter</th>
<th>Spot Time</th>
<th>Line Scan Time</th>
<th>Array Scan Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5mm</td>
<td>0.87mm</td>
<td>0.0009 seconds</td>
<td>0.03 seconds</td>
<td>0.8 seconds</td>
</tr>
<tr>
<td>1.0mm</td>
<td>1.7mm</td>
<td>0.01 seconds</td>
<td>0.14 seconds</td>
<td>2.1 seconds</td>
</tr>
<tr>
<td>3.0mm</td>
<td>5.2mm</td>
<td>0.41 seconds</td>
<td>2.0 seconds</td>
<td>9.7 seconds</td>
</tr>
<tr>
<td>5.0mm</td>
<td>8.7mm</td>
<td>2.3 seconds</td>
<td>6.8 seconds</td>
<td>20 seconds</td>
</tr>
<tr>
<td>10.0mm</td>
<td>17mm</td>
<td>24.4 seconds</td>
<td>35.7 seconds</td>
<td>52 seconds</td>
</tr>
</tbody>
</table>

Because of the higher power capabilities and larger half power beam angle of this emitter compared to IREDs, the required heating times shown in Table 5-2 were much more reasonable, and even potentially an improvement upon conventional bonding. Furthermore, with a spectral emission centered around 850nm, the Fabricolor 8472 dye could be effectively utilized.

5.2.6 Bonding with the OSRAM Ostar Observation IR Emitter

The test setup for the OSRAM IR emitter is shown below in Figure 5-9. Included in the setup is a chiller block to act as a heat sink and a glass stage for the substrate/cover plate stack to sit on. The stage is made of heat-resistant borosilicate glass and is attached to the assembly with magnetic supports on either side of the emitter.
Figure 5-9. Test setup for the OSRAM IR Emitter. The emitter sits on a chiller block for thermal management. A glass stage is attached to the assembly with magnetic supports on either side of the emitter for the substrate/cover plate stack to sit on.

The setup shown in Figure 5-9 above was configured such that the gap between the emitter and the substrate-cover plate interface was 6.3mm. Based on the spot size/bonding time analysis described in Table 5-2 above, this configuration should produce an 11mm-diameter spot in ~5 seconds.

The clamping force required during the bonding process was applied using an Instron machine. The structure shown in Figure 5-9 above was mounted in the Instron as is shown in Figure 5-10 below. It is not obvious in the photo, but there is also a shield on the front of the assembly to block the IR light from the user. Since this emitter only emits light in the NIR region, users will not see the light if they look straight at it. As such, it is important to have a shield up as a reminder to users.
The emitter was driven by a Supertex LED Driver Demo Board (Supertex Part #HV9910BDB1). This board included a potentiometer to regulate the output current, which controls the intensity of the emitter’s radiation. In order to measure the current, a resistor was put in series with the emitter and the voltage across that resistor was monitored. To control the timing, a square wave TTL signal was applied between the PWMD and ground terminals of the demo board. The PWMD pin was driven using an opto-isolator, as is shown in Figure 5-11 below (as recommended by Supertex).

Figure 5-10. IR Emitter assembly as mounted in the Instron machine.

Figure 5-11. Circuit used to isolate driver from AC line [53].
This signal was controlled via a Labview/NI USB-6009 DAQ interface. The electronics box with the LED driver board is shown below in Figure 5-12.

**Figure 5-12.** Electronics box for OSTAR IR emitter assembly. Wires to emitter deliver the regulated current to the emitter itself. There are two sets of wires to the DAQ; one set is used to monitor the current and the other receives the TTL timing signal.

**Preliminary Experiments**

To test this assembly, a solution of the Fabricolor 8472 dye was made in both acetone and ethanol, with the dye concentration equaling the saturation point of the given solvent (12% for acetone, 3% for ethanol). Acetone was used because the dye has a much higher solubility in acetone than in ethanol. However, since acetone evaporates so quickly, it is very difficult to spread an even coat of the acetone solution across the part before the acetone evaporates. Furthermore, since PMMA is not chemically resistant to acetone, it is not an ideal solvent; however, it is helpful to be able to observe how the dye reacts when the coating is of a higher dye concentration.

In each of the preliminary experiments, a few drops of coating were applied to the surface of two blank pieces of PMMA and the solvent was allowed to evaporate, leaving a film
of dried coating on the PMMA. The two pieces were then put into the Instron as is shown in Figure 5-10 above. Pressures between 186 and 620 kPa were applied; higher pressures were shown to break the glass stage. The emitter was turned on at various powers (between 30% and 100% of the maximum power capability) for a specified amount of time, with times ranging from 10 to 90 seconds. After the radiation was turned off, the parts were held under pressure for another 20-30 seconds. Qualitative observations about the resulting parts were made at the end of each experiment.

Overall, it was very difficult to achieve bonding, though some successful bonds were made. An example of one such successful bond is shown below in Figure 5-13. In this case, the parts had been coated with an acetone-8472 dye solution. To get these images, the two PMMA pieces were pulled apart after bonding and looked at under the microscope. The images show matching, complementary contours, implying that a chunk of the PMMA from one surface adhered to the other surface with greater strength than the cohesion of the PMMA itself. This is a preliminary sign of a weld.

Figure 5-13. Evidence of welding in preliminary experiments with 8472 dye and Osram emitter. Dye is dissolved in acetone. After welding occurred, the two PMMA pieces were pulled apart and their surfaces were studied under the microscope. The matching, complementary contours in these images imply that they were welded.
While evidence of a weld did occur, the spot diameter was smaller than the anticipated 11mm and it took closer to 45-60 seconds for the bond to occur, as opposed to the estimated 5 seconds. At this rate and spot diameter, it would take entirely too long for a bond to occur across the entire part, making this method less feasible than originally anticipated.

One possible reason for the discrepancy in the required time is that the model used to determine bonding times assumed perfect absorption of the available light. However, the final absorption in the coating is heavily dependent on the dye concentration, which is limited by the solubility of the dye in acetone and ethanol. While welding was observed for the acetone-based coating, this was infeasible for the ethanol-based coating. It is potentially feasible to coat the PMMA pieces with multiple layers of the dye in order to increase the total amount of dye present on the surface. However, when attempted, the application of more layers just caused the already applied layers to mix in with the newly applied layers, ultimately affecting the uniformity of the coating. Furthermore, because of the dilute nature of the ethanol-based solution, too many layers would be necessary, thus making the final process cumbersome.

Another flaw in the model used to determine spot size and bonding time is the assumption that radiation is being applied across the entire part at once. However, in the actual experiment, heat was only being applied to a finite spot. As a result, the area at the surface directly surrounding the spot would originally be at room temperature, thus establishing a temperature gradient from the center of the spot to the surrounding areas. This temperature gradient would facilitate heat loss from the spot to the surrounding areas, thus affecting the thermal transient behavior and the required bonding times. In the next section, a new series of simulations will be conducted to account for this spot-heating phenomenon.
5.2.7 Heat Transfer Simulations of Spot Heating

In an effort to minimize the discrepancies between the heat transfer simulations and the experimental data, the following simulations are a modification of those described in Section 5.2.5 to account for the spot heating that occurs with the OSRAM IR emitter.

For this next series of simulations, heat fluxes of varying intensities were applied to the surface of a piece of PMMA in four different spot sizes. The simulation setup is shown below in Figure 5-14. Taking advantage of the circular symmetry in this model, only a 1° slice of a circle was studied and the symmetry boundary condition was applied.

![Figure 5-14. PMMA slice used in FEA simulation for spot heating model. A 1° slice was studied with circular symmetry.](image)

Mesh control was applied so there were 2 μm elements at the spot surface and the elements would grow by a factor of 2 as they extended from this surface. The maximum element size was 20 μm. A heat flux of a given intensity was applied at the surface specified by
the spot radius. Simulation time steps varied to ensure that the appropriate transient behavior
was observed for each of the different heating scenarios. The radial and thickness temperature
profiles were measured over time and specific attention was paid to the amount of time it took
for the center of the spot to reach $T_{\text{melt}}$. The times to reach $T_{\text{melt}}$ for the various heating
conditions are reported in Figure 5-15 below. Spot radii of 0.1mm, 0.25mm, and 0.5mm were
tested. A spot rectangle of 5mm x 2mm was also studied, for it corresponds to the shape of the
Ostar emitter tested previously. All of these results were compared to the infinite plate
simulations described in Section 6.2 above.

After running these simulations, some inaccuracies in the simulation setup were
discovered. The PMMA slice used as a model had a radius of 1mm. For simulations in which
the heating to $T_{\text{melt}}$ happened in a matter of seconds, the surface temperature 1mm from the
center was unaffected by the heating, so this model was sufficient to describe a spot in the
middle of an infinite plate. However, when the heating happened over a longer period of time, there was more time for the heat to conduct away from the spot area and the region 1mm from the center of the spot was affected by the heating. In these cases, the model was not accurately depicting a spot in the middle of an infinite plate, resulting in an underestimation of the time required to melt the spot surface. While the longer times in the above model are underestimated, the model can still be used to get a better understanding of the spot heating trends.

As can be expected, for a given heat flux, more time is required to heat a smaller spot size because there is less total power and as a result of the smaller circumference:area ratio, there is more chance for the heat to conduct away from the spot along its radius. Thus, the infinite plate model from earlier was underestimating the times required for melting because it was not accounting for this spot effect.

It was also interesting to look at the heat-affected zone of the plastic in each of the above scenarios. Here, the heat-affected zone is considered the thickness through which the temperature of the PMMA is greater than $T_g$. The heat-affected zone as a function of heat flux is shown below in Figure 5-16.
Figure 5-16. Heat-affected zone for various spot heating conditions. Heat-affected zone is defined as the thickness through the part that has a temperature greater than $T_g$. The substrates are 1500 μm thick; such a heat-affected zone implies the entire substrate is affected by the heat.

The heat-affected zones vary in a similar manner to the times to melt. If it takes longer for the PMMA to heat the surface to a desired temperature, there is more time for the heat to dissipate to the rest of the part. A larger heat-affected zone means a greater chance for the substrate, and thus the microfluidic channels, to deform under pressure, which is undesirable.

There is a portion of both the time to melt and heat-affected zone curves that are flat and have small values of their respective quantities (i.e. short time to melt or small heat-affected zone). Operating in this high heat flux region would result in a desirable and robust process. Small changes in heat flux would not cause a significant change in the time required to melt. However, care would have to be taken to ensure that the time of exposure to the heat was not even the slightest bit too long because temperatures as high as $T_{boil}$ could be achieved very quickly.
In order to compare the performance of the OSTAR emitter chip with these new simulations, the light spectrum of the emitter chip was multiplied together with the absorption spectrum of the Fabricolor dye. The three curves are shown below in Figure 5-17.

![Emitter Chip Spectrum](image1)

![Fabricolor Dye Spectrum](image2)

Figure 5-17. Emitter chip and fabricolor dye spectra. Emitter chip spectrum from [54] and Fabricolor dye absorption spectrum from [51].

Multiplying these two spectra together, the spectrum of light that is actually absorbed by the dye from the emitter is found and shown in Figure 5-18.
Integrating this curve, there are 2.63W of power absorbed by the dye if it is behaving perfectly. Assuming a 11mm spot diameter, as per previous calculations, if all of the power were to be spread uniformly over this spot, there would be a heat flux of 0.03W/mm². However, the emitter is a Lambertian source and the heat flux at the center of the spot is greater than at the edges. Even if the heat flux at the center of the spot were 0.06W/mm², the operating point would still be on the brink of long weld times according to the graph in Figure 5-15 above, and this is assuming perfect absorption by the dye. This explains the long bonding times and the Lambertian nature of the light source explain the smaller spot sizes.

This more detailed analysis, combined with the experimental results, indicate that this emitter does not have sufficient power for microfluidic bonding applications. From here, the study can move on to broadband light sources.
5.2.8 Investigating Light Sources: Broadband Light

There are two main advantages to using broadband light sources to be absorbed by the coatings. In general, these sources are available at much higher powers than the IR emitters discussed previously. Furthermore, they emit substantial light at all of the wavelengths that the coatings absorb at, thus maximizing the amount of absorption that can take place with a given amount of coating material.

Basic Broadband Analysis

Broadband light sources are often treated as blackbody radiators with specific color temperatures. The blackbody equations can then be used to calculate the light intensity profile across a range of wavelengths. The output power of a blackbody radiator with color temperature $T$ at wavelength $\lambda$ is defined by Equation (5-6) below.

$$\Phi_\lambda = \frac{\Phi_0 2\pi hc^2}{\sigma T^4} \frac{1}{\lambda^5 (e^{hc/kT\lambda} - 1)}$$

(5-6)

where $\Phi_0$ is the total light power of the source, $h$ is Plank’s constant, $c$ is the speed of light, $k_b$ is Boltzmann’s constant, and $\sigma$ is Stefan’s constant. The light power intensity profile for a typical 50W halogen, 3000K color temperature, light bulb is shown below in Figure 5-19.
To determine the total power that will be absorbed by the Fabricolor dye, this halogen bulb light spectrum is multiplied by the absorbance spectrum of a given dye. The integral of the resulting curve over all available wavelengths is the total power absorbed under ideal conditions. This process was completed for three different dyes: the 8472 dye with peak absorption at 850nm, the 10022 dye with peak absorption at 1002nm, and a 50-50 mix of these two dyes. The total power absorbed by the coating for each dye is shown below in Table 5-3.

Table 5-3. Power absorbed by different selective coatings from 50W and 100W light sources. Light sources have a color temperature of 3000K. Assumes perfect absorption.

<table>
<thead>
<tr>
<th>Dye Type</th>
<th>Power absorbed – 50W light bulb source</th>
<th>Power absorbed – 100W light bulb source</th>
</tr>
</thead>
<tbody>
<tr>
<td>8472 dye</td>
<td>2.67 W</td>
<td>5.34 W</td>
</tr>
<tr>
<td>10022 dye</td>
<td>4.40 W</td>
<td>8.80 W</td>
</tr>
<tr>
<td>8472-10022 mix</td>
<td>3.54 W</td>
<td>7.07 W</td>
</tr>
</tbody>
</table>
Because the 10022 dye has a peak absorption at a wavelength closer to the peak emission wavelength of the 3000K source, the most absorption occurs with this particular dye. It is clear that over three times as much absorbable power is emitted from this broadband source than from the IR emitter discussed in the previous section (8.8W vs. 2.67W).

One potential disadvantage to using a broadband source is that the light will also be emitting wavelengths of light that will be absorbed by the PMMA, causing the substrate to heat up as well. However, the presence of the coating will cause more heating to occur at the surface, thus preserving the surface heating idea. The bulk absorption will just allow everything to come up to temperature more quickly.

As a simple test, two halogen MR16 light bulbs with aluminized reflectors were used to bond PMMA blanks. A sketch of the test setup is shown in Figure 5-20 below. In this configuration, pressure was applied manually.
Figure 5-20. Simple setup to test the bonding with broadband light idea. Light bulbs are MR16 halogen bulbs with aluminized reflectors. Glass is heat-resistant borosilicate. Pressure was applied manually.

In the first tests, PMMA blanks, both coated and uncoated, were successfully bonded together. The required heating took between 30 and 45 seconds, similar to the times required using the OSTAR emitter. However, unlike with the OSTAR emitter, these bonds spanned almost the entire part, making these broadband sources a favorable option. The bonds were not uniform across the entire parts, but that can be attributed to the non-uniform pressure and light sources. The pressure uniformity can easily be dealt with in future iterations by using an Instron machine to apply the pressure. The thermal uniformity is a bit more difficult to achieve
and will be discussed further later. First, temperature measurements would be taken to get a better idea of the absorptive coating performance.

**Temperature Measurements**

In order to evaluate the performance of the absorptive coatings, temperature measurements were taken at the interface during the heating process. Dye was first deposited onto the PMMA blanks. The 8472, 10022, and 8472-10022-mix dyes were used. Each of these was dissolved in ethanol to the point of saturation. A fourth coating was made by dissolving the 8472 dye in acetone, so as to achieve a higher dye concentration. A graduated eyedropper was used to deposit the same amount of dye material on each blank. The blanks were then left out to allow the solvent to evaporate, leaving a thin film coating on the PMMA blanks. This film was as uniform as possible, though there was some natural spreading to the edges and corners during the drying process, implying that the coating was not perfectly uniform.

An Omega SAl-XL surface mount Type T thermocouple was mounted to the coated surface of one piece of PMMA. Another piece with the same coating was placed on top, with the coated surfaces touching at the interface. This PMMA stack was then sandwiched in between two glass stages and two lights. Heat-resistant borosilicate glass was again used. A photo of this simple setup is shown below in Figure 5-21.
The light bulbs, each 50W, were powered by a DC power supply. The thermocouple fed to a National Instruments USB-9211 Thermocouple Amplifier/DAQ that interfaced with LabView.

In a first set of experiments, pure bulk heating was measured in three scenarios. In the first, only one light bulb was used, but the PMMA blanks were still sandwiched between two pieces of glass. In the second, the PMMA blanks were sandwiched between two pieces of glass and both light bulbs. However, only the bottom light bulb was turned on. This particular test showed the effect of adding a reflector to reflect back the IR energy that passes through the PMMA from the bottom light bulb. In this test, there was also less convection off of the top piece of glass. In the last test, both light bulbs were used and turned on. There were no replicates for these preliminary experiments, and the starting temperatures all had to be normalized to 25°C after the data was taken. The results are shown in Figure 5-22 below.
These results show that there is a significant amount of bulk heating in the PMMA when utilizing these light bulbs. Furthermore, the presence of the reflector proved to have an effect on the total heating at the interface.

The next tests investigate the effect of dye type and dye concentration. Three replicates were tested for each dye type, as well as for a set of PMMA blanks. Two light bulbs were used, and both were turned on, in each run. All tests were done in a randomized order. The temperature trends over time for each of the different ethanol-based dyes are show below in Figure 5-23. All of the ethanol-based dyes are saturated solutions. The glass and thermocouples returned to room temperature in between each test, and so temperature trends did not have to be normalized to an initial start temperature.
These temperature trends prove the selective nature of the dye. On the most basic level, the presence of the dye caused the surface of the PMMA to heat up more than when uncoated PMMA blanks were exposed to the light. Furthermore, as is shown in Table 5-3 above, the 10022 dye should absorb the most light. The trends in Figure 5-23 show that the surface temperature increases the most when this dye is used, followed by the 8472-10022 mixed dye and then the 8472 dye, as was expected.

To test the effect that the dye quantity/concentration has on the temperature trends, the 8472-ethanol and 8472-acetone solutions were used. The dye concentration in the 8472-acetone solution was higher than in the ethanol solution. In a third trial, the ethanol-based coating was applied to one piece of PMMA and a blank was placed on top of it. As such, only half the
amount of dye was present. Three replicates of the above procedure were run for each of these three trials. The resulting temperature trends are shown below in Figure 5-24.

![Figure 5-24. Effect of dye quantity/concentration on the temperature trends at the interface during heating by halogen light bulbs. The 8472 dye is used. The acetone solution is at a high concentration. The ethanol solution is saturated at a low concentration. In the 8472 Ethanol + PMMA blank trial, the low concentration ethanol coating is only applied to one piece of PMMA and the other piece is blank in order to achieve the effect of having less dye present. Multiple trendlines of a particular style indicate replicates.]

These temperature trends prove that the dye concentration/quantity has an effect on the total heat absorption at the surface, and thus also on the temperature trends over time at the surface. Thus, these concentrations would also need to be taken into account in the heating simulations to make them accurate, which explains some of the longer experimental heating times compared to the simulated heating times.

Since bulk heating played such a significant role in the heating process, it was interesting to see if some of the extra light being absorbed by the PMMA could be filtered out before it hit the PMMA, thus concentrating more of the heat absorption to the interface where
the coating was. To achieve this, a 600nm long-pass filter and an 1100nm short-pass filter were used in sequence. This created a band-pass region of 600-1100nm, which covered the absorption region of the two dyes under investigation. In this experiment setup, the two light bulbs were actually placed about 2” apart and the PMMA pieces being studied were clamped between two pieces of glass in the center of them. In this way, the extra conduction from the hot light bulb would be eliminated. Furthermore, the light uniformity improves further away from the light bulb. Temperatures were measured at the interface of coated and uncoated parts, and with and without the filters. The results are shown below in Figure 5-25.

![Temperature trends over time for coated and uncoated PMMA pieces, and with and without light filters. Multiple trendlines of a particular style indicate replicates.](image)

Comparing the uncoated temperature trends with and without the filters, it is clear that the presence of the light filters decreases the amount of bulk heating in the PMMA. The coated substrates also show lower temperatures over time when the filters are used versus when they
are not used. Because of the range of wavelengths involved in the band-pass filtering, there should not be any change in the absorption of heat by the coating itself. Thus, this is attributed to a change in the bulk heating conditions. One possible reason for using the filters is to increase the difference in the temperature trends between the coated and uncoated trials. If there is less bulk absorption, but the same amount of absorption by the coating, then the temperature gradient through the part would be greater. However, there does not seem to be any significant change in the difference between the coated and uncoated temperature trends as a result of adding the filters in.

In this last set of tests, the light bulbs were moved further away from one another, thus decreasing the intensity of light hitting the PMMA parts. Figure 5-26 below shows the temperature trends for uncoated PMMA being heated without filters for this setup (low intensity light) and the original setup (high intensity light). As can be expected, the intensity of the light has a significant effect on amount of heating that occurs.
Figure 5-26. Temperature trends over time for blanks being heated by broadband light of different intensities. Intensities are changed by moving the PMMA different distances from the light bulbs. Multiple trendlines of a particular style indicate replicates.

One of the most interesting components of the above temperature measurements is the realization of how much bulk heating is actually contributing to the surface heating. The dyes do achieve some level of selective heating, but without the bulk heating, it is difficult to get the interface up to temperature using the light sources that have been discussed. The halogen light bulbs, however, can get the PMMA up to temperature such that PMMA blanks can be bonded together. This simple system was also used to bond a cover plate to an actual microfluidic chip and the channels seemed to remain intact. As was discussed previously, the bond was not perfectly uniform, but that can be compensated for in later design iterations. This particular example is a case of high temperature, low pressure bonding, and is a promising sign for the possibility of eliminating the coatings and returning to the idea of generating a homogenous bond. This idea will be explored in the next chapter.
CHAPTER

6 Heating with Broadband Light

As was described at the end of the last chapter, broadband light can be utilized to bulk heat the PMMA in order to bond microfluidic devices. This concept will be further investigated in this chapter.

6.1 Achieving an advantageous temperature gradient while heating with broadband light

The basic heating condition is one of constant flux throughout the part, instead of a constant temperature source at the edges as in conventional thermal bonding. When the light passes through the PMMA, it is absorbed through the bulk according to the Beer-Lambert law. The most heat is absorbed at the surface where the light initially hits the PMMA, and there is exponential decay of the light absorption through the part. This decay is described by the absorption coefficient, which is unique to each wavelength of light. If the light is arranged as in the ideal experimental setup shown in Figure 6-1 below, there will still be an advantageous temperature gradient at the substrate surface where the channels are.
Figure 6-1. Ideal setup for bonding with light idea. A uniform light source sits below a stage made of IR transparent glass. The substrate is flipped upside down so that the most light is absorbed at its surface and there is an advantageous temperature gradient through the channels. Furthermore, since the substrate is inverted, the cover plate can be molten and will into droop into the channels by gravity.

In this setup, a uniform light source sits below a stage made of IR transparent glass – either heat-resistant borosilicate glass or extreme temperature pure quartz glass. Uniform pressure is applied with an Instron machine. The substrate is flipped upside down so that the most light is absorbed at its surface and there is an advantageous temperature gradient through the channels. Furthermore, since the substrate is inverted, the cover plate can be molten and will not droop into the channels by gravity, although the pressure from the substrate will cause some flow of the cover plate into the channel. Heat can also be conducted from the cover plate into the substrate. Since all of the bonding would occur during the transient bulk heating process, and not during steady state, the appropriate temperature gradient would be maintained.

It is also possible to intensify this temperature gradient by adding active cooling. In the above setup, a chiller block can be added to the top of the substrate while applying the pressure. The glass stage can also be cooled in order to reverse the temperature gradient in the cover
plate. In a sketch model of this idea, shown below in Figure 6-2, a clamp was made out of polycarbonate with cooling channels in it.

![Image of a sketch model setup to study active cooling at the backs of the cover plate and substrate. Channels for chiller fluid surround area where cover plate and substrate stack will be clamped.](image)

There were two big problems with this setup. First, because polycarbonate has a similar transmission spectrum to acrylic, there was less light available to the acrylic for heating. Furthermore, the chilling mechanism needed to not only chill the back of the cover plate, but it also needed to cool off all of the heat absorbed in the polycarbonate itself. The polycarbonate also has a very low thermal conductivity, and the cooling process took place too slowly. In a more ideal design, the clamp would be made out of glass. Glass will transmit more light than polycarbonate, and thus experience less bulk heating. It also has a higher thermal conductivity, allowing it to cool off more effectively. The glass would have to be extremely resistant to thermal shock, especially if a design similar to the above were to be used. In the above design, a natural gradient will form between the edges where the chilling channels are and the center of the clamp. Such a temperature gradient would put even more thermal stress on the glass, and
the glass will already be under stress from the pressure needed for bonding. For these reasons, this chilling channels idea is not worth pursuing further.

Another possible method of chilling the back of the cover plate would be to blow cold air over the glass and cool by convection. The feasibility of such a setup would be dependent upon the light source and the ability to fit air hoses in the necessary configuration. Since a uniform light source is the most critical component of this design and cooling the back of the cover plate is not absolutely critical, focus would first be turned to choosing a uniform light source.

6.2 Choosing a source of uniform light

6.2.1 Different Light Bulbs

In the previously discussed experimental setup, 50W MR16 halogen light bulbs were used as the broadband light source. These particular bulbs had an aluminized reflector that projected both the visible and the IR radiation out towards the PMMA parts. They also had an open face, and so there was no diffuser available to smooth out the hot spots from the filament. In the next setup, PAR16 bulbs would be used. PAR stands for “parabolic aluminized reflector” and so the IR radiation would still be appropriately projected forwards. Instead of an open face, PAR light bulbs have a diffuser built in, and so the light will be better diffused than with the MR16 bulbs. A 75W PAR16 light bulb was used in the experimental setup shown in Figure 6-3. In this setup, the light bulb is mounted beneath a stage. The PMMA parts sit on a quartz glass window that is secured within the stage for increased stiffness against the pressure being applied. The pressure is applied using the Instron machine.
Figure 6-3. Experimental setup for PAR light bulb. The setup is mounted in an Instron machine so that the Instron can uniformly provide the necessary pressure. The bulb sits below a stage. The PMMA parts sit on a quartz glass window that is secured in the stage for greater stiffness against the Instron pressure.

This PAR bulb turned out to be powerful and capable of bonding the PMMA pieces together, but it was not as diffuse as needed. There was an obvious hot spot in the middle of the PMMA part. While the light was being diffused, the Lambertian nature of the light source still caused a natural hot spot at the center of the bulb. In the previous setup, the two MR16 light bulbs were positioned manually. As a result, the light bulbs were definitely not perfectly lined up and any small offset of the two MR16 filaments effectively “spread out” the hot spot across a larger portion of the PMMA parts being bonded. In this case however, only one, higher-powered bulb was used, thus emphasizing the hot spot in the middle of the part. In an effort to further diffuse the light, a fused silica ground glass diffuser was placed beneath the quartz window. While this did help with light diffusion, it did not generate a uniform light source across the entire part.
It is also possible to use just the small bulb that contains the filament instead of the entire MR or PAR bulb. Many of these small bulbs can be put in an array behind a ground glass diffuser with hopes of having the various spots blend together. While this did work to a small extent, the lack of directionality to the light was a problem. The aluminized reflector is necessary to project all of the light forward.

In addition to the ground glass diffuser, a series of lenses were also considered. Condenser and Fresnel lenses collect all of the light being emitted at an angle from the light bulbs and redirect it forward. The problem with these lens systems is that while the light will be collected into a smaller region, the basic light pattern will not be adjusted, as was seen with the ground glass diffuser.

In another trial, the MR16 bulbs were used and the parts were moved far away from the bulb’s face. The light is more uniform further away from the bulb, but it is also more diffuse. In these trials, there was just not enough heat flux to get any appreciable heating of the PMMA. In further attempts, a tall aluminum foil tube was placed around the bulb to try and contain all of the light within that tube. However there were still too many losses along the tube and there was not significant heating of the PMMA.

After considering these various options, it was clear that a solution more complex than just using a simple light bulb would be necessary. Fiber optic cables and integrating spheres were the next to be explored.

6.2.2 Fiber Optics

Broadband light sources, such as the Tungsten-Halogen light sources from Spectral Products, Avantes, and Infinon Research [55 – 57], contain typical tungsten-halogen light bulbs at a color temperature of 3000K, as well as the necessary light shaping equipment to collimate
and focus the light to be sent to a fiber optic cable. The collimated light is what is needed in this application, but these sources are really made for the fiber optic coupling.

The largest available fiber optic cable is 1mm in diameter. Liquid light guides are available in sizes up to 5mm in diameter and can handle more power than typical fiber optic cables. With fiber optic sources there will be losses to attenuation down the cable as well as coupling losses from coupling the cable to the broadband source. The light exiting the cable also has an angle to it and might need to be collimated or refocused depending on its angle of divergence. While the broadband source coupled to a fiber optic cable would make the light more uniform, the small cable sizes and the many opportunities for power loss do not make this a favorable option.

6.2.3 Integrating Spheres

An example of an integrating sphere is shown below in Figure 6-4. Light enters one port of the sphere, all of the light is integrated and re-averaged, and uniform light exits another port. There is a baffle between the input and output ports, to prevent any direct viewing of the incoming light at the exit port. The light exiting the sphere can be as uniform as +/-5% and the exit ports on commercially available spheres are as big as 1.5” in diameter, making these an attractive option to explore more in depth.
Figure 6-4. Picture of an integrating sphere. Light enters through one port, all of the light is integrated and re-averaged, and uniform light exits another port.

The interior of the sphere is made of a diffuse reflective surface of high reflectivity in the range of wavelengths needed for a given application. The fraction of light received by any differential area element in the sphere is defined by Equation (6-1) below. The following analyses and equations (6-1) - (6-6) are based on/from a technical document published by LabSphere, the manufacturer of the integrating spheres [58].

\[
dF = \frac{dA}{4\pi R^2}
\]

(6-1)

where \(dF\) is the fraction of total light received by the differential element, \(dA\) is the area of the differential element, and \(R\) is the radius of the sphere. This fraction is not determined by the position of the element being considered, the angle of this element from the light source, or the distance of this element from the light source. As such, the fraction of light received by a differential element is the same for any radiating point on the sphere. The radiating points on the sphere include any internal sphere surface area that is not taken up by an entrance or exit port. The surface area of the sphere dedicated to entrance and exit ports is defined by the sphere’s port fraction, shown in Equation (6-2) below.
\[ f = \frac{A_i + A_e}{A_s} \]  

(6-2)

where \( f \) is the port fraction, \( A_i \) is the area of the input port, \( A_e \) is the area of the exit port, and \( A_s \) is the total surface area of the sphere.

When the light enters the sphere, it undergoes a series of reflections within the sphere before exiting the exit port. With each reflection, the amount of flux incident on the sphere surface is dependent on this port fraction, as well as the internal reflectance, \( \rho \), of the sphere. This dependence is shown below in Equation (6-3). \( P \) is the light power incident on the sphere’s surface and \( \Phi_i \) is the input power.

\[ P = \Phi_i \rho (1 - f) \]  

(6-3)

In a correctly designed sphere, the number of reflections inside the sphere approaches infinity, and multiplies the total surface radiance by a factor, \( M \), defined in (6-4) below.

\[ M = \frac{\rho}{1 - \rho (1 - f)} \]  

(6-4)

Combining the above equations, the total sphere radiance, in W/m\(^2\)/sr, can be defined by \( L_s \) in (6-5).

\[ L_s = \frac{\Phi_i}{\pi A_s} \frac{\rho}{1 - \rho (1 - f)} \]  

(6-5)

In general, the port fraction should be no greater than 0.05 and larger sphere:port ratios generate more uniform light. A sphere:port diameter ratio of 3:1 will generate light that is uniform to +/-1% and a 4:1 ratio will generate light uniform to +/-0.5% [59]. However, larger spheres also decrease the total light throughput, as is shown by the inverse relationship between \( L_s \) and \( A_s \) in (6-5) above. A balance needs to be found between these quantities. The light
uniformity is also dependent upon the distance that the PMMA part is from the sphere’s exit. The light emitted from the sphere is Lambertian in nature, similar to the light being emitted from the previous light bulbs. However, there are no major hot or dark spots associated with the sphere, the Lambertian profiles are much flatter than those of conventional light bulbs, and the nature of the profile is much more reliable. Furthermore, when the parts are against the exit port of the sphere, the Lambertian nature of the profile is virtually eliminated. Thus, with creative design, the part can be positioned in this region, maintaining the benefits of the uniform light source.

While it is enticing to custom fabricate a sphere, the interior of the sphere must be a diffuse reflector of high reflectivity. Aluminum has high reflectivity in the NIR and IR regions, but it is a specular reflector; light that hits aluminum at a particular angle is reflected at that same angle. In a diffuse reflector, the light that hits a surface is then scattered in a Lambertian pattern, which is what makes the light averaging phenomenon possible and eliminates hot spots. The spheres that are commercially available are generally made of Spectraflect or Spectralon, which are proprietary materials made by the sphere manufacturer, Labsphere. The reflectance curves for these materials are shown below in Figure 6-5.
Figure 6-5. Reflectance curves for Spectralon and Spectraflect, as published by Labsphere [58].

Spectralon has much higher reflectivity in the NIR/IR range, making it the obvious choice for this application. While Spectralon is available for purchase to machine, at this phase it made more sense to investigate the feasibility of a commercially available sphere. The Spectralon-coated spheres from LabSphere come in two styles— one is made for collimated light inputs and the other for diverging light inputs. Since conventional halogen light bulbs will be used, the latter style was chosen. These spheres come with three ports, two of which are baffled from one another. One of these ports will be used as the input port and the other as the output port. The third port will be plugged with a Spectralon-coated port plug.

There were four available sphere sizes, shown below in Table 6-1. This table reports the port sizes and the thermal coating limits of each sphere. This thermal limit is the maximum amount of power that the sphere can absorb before overheating. The sphere is made of Spectralon and then spin coated with aluminum. The Spectralon is thermally stable up to 350°C.
Table 6-1. Commercially available integrating spheres from LabSphere.

<table>
<thead>
<tr>
<th>Sphere Diameter</th>
<th>Input Port Diameter</th>
<th>Exit Port Diameter</th>
<th>Coating thermal limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1&quot;</td>
<td>0.25&quot;</td>
<td>0.25&quot;</td>
<td>Not specified</td>
</tr>
<tr>
<td>2&quot;</td>
<td>0.5&quot;</td>
<td>1&quot;</td>
<td>25W</td>
</tr>
<tr>
<td>3.3&quot;</td>
<td>1&quot;</td>
<td>1.5&quot;</td>
<td>100W</td>
</tr>
<tr>
<td>5.3&quot;</td>
<td>1&quot;</td>
<td>2.5&quot;</td>
<td>225W</td>
</tr>
</tbody>
</table>

Of the four available spheres, only the two larger ones are practical options. The exit ports for the smaller spheres are not large enough to illuminate an entire microfluidic chip. Furthermore, in order to get the PMMA as close to the exit plane of the sphere as possible, the piece will likely need to be submerged inside of the sphere, making a scanning approach infeasible.

These larger two spheres are compared in Table 6-2 below. The sphere:exit port diameter ratio can be used as a measure of how uniform the light will be. Since the PMMA primarily absorbs NIR/IR radiation, the power calculations were completed for only this portion of the spectrum. P_{PMMA} in the below table is a measure of the total NIR/IR power that would hit a 1"x1" PMMA part at the exit port of the sphere if two 50W halogen light bulbs are used at the input. The P_{PMMA} was calculated using (6-6) below where P_{PMMA} is the NIR/IR power available to be absorbed by the PMMA pieces, A_{PMMA} is the area of the PMMA pieces and Ω is the projected solid angle, equal to π here.

\[ P_{PMMA} = L_{A_{PMMA}}Ω \]

(6-6)

Table 6-2. Comparison of two integrating spheres on basis of light uniformity and power requirements.

<table>
<thead>
<tr>
<th>Sphere Diameter</th>
<th>Input Port Diameter</th>
<th>Exit Port Diameter</th>
<th>Sphere : Exit Port Diameter Ratio</th>
<th>Coating thermal limit</th>
<th>P_{PMMA}</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.3&quot;</td>
<td>1&quot;</td>
<td>1.5&quot;</td>
<td>2.2</td>
<td>100W</td>
<td>37W</td>
</tr>
<tr>
<td>5.3&quot;</td>
<td>1&quot;</td>
<td>2.5&quot;</td>
<td>2.12</td>
<td>225W</td>
<td>16W</td>
</tr>
</tbody>
</table>

As is expected, the smaller sphere has a greater power output for a given power input. The 5.3" sphere can handle 2.25 times the thermal absorption as the 3.3" sphere. If it can
handle more input power, it could potentially result in a greater output power as well. However, the $P_{\text{PMMA}}$ for the 3.3" sphere is more than 2.25 times the $P_{\text{PMMA}}$ for the 5.3" sphere, so using the bigger sphere with greater input power would not necessarily result in greater output power. It would just be less energy efficient, and in this case also slightly less uniform because the sphere:port exit diameter ratio is lower for the 5.3" sphere than it is for the 3.3" sphere. The only advantage of the 5.3" sphere is the 2.5" exit port of the 5.3" sphere would be advantageous, but the larger power output:power input ratio and better uniformity of the 3.3" sphere makes it the better choice.

As a source of comparison for the powers involved, in the simple 2-light bulb setup in Figure 5-21 above, about half of the power of each of the 2 light bulbs was hitting the PMMA parts. Thus, the equivalent of one light bulb’s worth of NIR/IR power was being utilized. Integrating over the blackbody radiation curves for all $\lambda>1000\text{nm}$ yields an estimated NIR/IR power requirement of 36W. However, since light is only hitting the PMMA parts from one side when the sphere is used versus when the two light bulbs were used, the transient heating conditions will be slightly different and more power will likely be needed. The 36W is a good minimum requirement though.

As can be seen in Table 6-2 above, the 3.3" sphere could achieve this minimal power requirement when powered by two 50W light bulbs. Since the 36W requirement is a threshold, it is possible that three bulbs would be required. The coating thermal limit for this sphere is 100W. The thermal absorption is dependent on how much heat gets absorbed by the Spectralon and how much heat the sphere is able to dissipate, and the latter can be increased by using a fan to blow air over the sphere during operation. The Spectralon has a reflectance of $>95\%$, closer to 97-98\%, over all wavelengths. Assuming that the rest of the heat is absorbed, it can be conservatively estimated that 5\% of the input power to the sphere is absorbed by the Spectralon
and converted to heat. Thus, if 150W of light are used at the entrance port, an estimated 7.5W will be absorbed, which is significantly lower than the thermal limit of the sphere. Even if the thermal limits are not reached, the sphere will still get hot, especially over time, and so a fan can still prove to be useful.

Based on this analysis, integrating spheres seem to be the best choice for a source of uniform light. The 3.3” Spectralon sphere was purchased and a fixture was made for testing, as will be described in the next section.

6.3 Implementing the Integrating Sphere

6.3.1 Initial Testing

The setup for the initial phase of testing with the integrating sphere is shown below in Figure 6-6. The sphere sits in a foam base that is carefully carved out to fit the sphere’s contour. Three 50W light bulbs are secured in ceramic holders and carefully placed at the input port so that the bulbs are inside of the sphere. The bulbs are powered by a DC power supply. A custom glass and part holder slips into the exit port, the base of which is flush with the interior of the sphere. A piece of glass sits on the lip at the bottom of the holder and the PMMA pieces rest on the glass. Grooves in the glass/part holder allow a 1”x1” square to fit inside of it.
Once the glass and PMMA pieces were placed inside of the glass/part holder, weights were placed on top of the part in order to apply the required bonding pressure. In this iteration, the sphere was supporting the load, and so only a few pounds of force were used, but a more formal structure is introduced below.

In initial testing, the bonding of the microfluidic chips was successful. The bonds had some voids owing to the low and uneven bonding pressures, but bonds did form across the width of the parts. Three 50W light bulbs were used and bonding took on the order of a few minutes. Since this concept seemed promising, a more in-depth study of the uniformity of the light exiting the sphere was conducted. This study will be discussed in the next section.

6.3.2 Measuring the Uniformity of Light Exiting the Sphere

To measure the uniformity of the light emitted from the integrating sphere, a digital camera was used to take images of the light at different planes and MATLAB was used to process the image files. The setup for these measurements is shown below in Figure 6-7.
The light was projected onto a piece of paper and the position of the paper determined the plane of light being imaged. Planes both inside and outside of the sphere were analyzed. To measure the light outside of the sphere, the paper was secured to the supporting structure and the sphere was positioned such that the output port of the sphere was a given distance from the paper plane, as is shown in Figure 6-8 below.

Figure 6-7. Setup for light uniformity measurements. The integrating sphere sits within a supporting structure and the light emitted from the sphere shines through a hole in the supporting structure, where it can be imaged by the Nikon D90 camera.

Figure 6-8. Setup for measuring planes outside of the sphere. A) Paper, used as an imaging surface, is secured to the supporting structure. B) The sphere-paper gap is varied in each measurement to generate multiple data points.
To measure the light inside the sphere port, a piece of paper was secured to the bottom of the custom glass holder that had been fabricated previously, as is shown in Figure 6-9 below. Using shims, the position of the holder within the sphere's output port was varied.

![Figure 6-9. Setup for measuring planes inside of the sphere. Paper is secured to the back of the previously fabricated glass holder. The position of the glass holder is varied using shims.](image)

The output port is configured as shown in Figure 6-10 below. The light exiting the port first travels through the thickness of the Spectralon and then through a short aluminum tube before truly exiting the sphere. In this analysis, the planes were measured to be a given distance, 'd', from the interior of the Spectralon.
To take the photographs, a Nikon D90 digital camera was used. Straight edges were used to position and align the camera with the sphere’s output port. The flash was turned off and the images were focused manually. The exposure compensation was set to -1.3EV to avoid image saturation. To further avoid saturation, only 9W of light power was used to illuminate the sphere. This was accomplished by only running one bulb at 2.15V instead of 2-3 bulbs each at 12V.

Images were saved as RAW files. The desired portion (i.e. the illuminated circular region) of each of these files was then selected and saved as a separate 16-bit TIFF file. These TIFF files would then be used for processing in MATLAB.

Each TIFF file was imported into MATLAB as a 3-level matrix. Each level corresponded to the appropriate RGB color values where the 16-bit color values were reported as a number between 0 and 65,535. To make the analysis less cumbersome, the relative color values were analyzed; each reported color value was divided by 65,535 and this fraction was used as a measure of color value. The uniformity of a given RGB color value across the illuminated area can be used as a measure of the uniformity of the light’s intensity over the same area. In this way, the color values were used as a proxy for intensity values; i.e. the higher
the color value, the more intense the light. To study the trends of the light intensity, the color
dvalues were plotted across the centerline of the illuminated area that is indicated in Figure 6-11
below.

![Figure 6-11. Centerline across the illuminated area. The color values across this centerline were used to study the trends in light intensity.](image)

The plot of color values/intensities across the centerline shown in Figure 6-12 below
indicates that, as can be expected for this system, each of the RGB color values follows the
same trend, but at different intensities. Thus, only one color value needs to be analyzed. Since
the intensity values are the strongest for the red component, this component will be used from
now on. The high frequency ‘noise’ in the trends of Figure 6-12 can be attributed to the
granular nature of the paper being used.
Figure 6-12. Plot of color values/intensities across the centerline for each of the RGB color components.

The light emitted from the sphere is specified to be Lambertian in nature with varying degrees of uniformity depending on how far the light plane is from the outlet of the sphere. The Lambertian intensity profile can be readily seen in the profile shown in Figure 6-12 above.

The intensity plots across the centerline were of two basic forms. The plot in Figure 6-12 above is a typical plot for the planes of light outside of the sphere outlet, as measured by the setup shown in Figure 6-8 above. This illuminated area was 1.5", the same as the diameter of the sphere outlet. The R-intensity plots for four of the planes measured in this way are shown below in Figure 6-13.
Figure 6-13. Intensity plots across the centerline for the four planes measured outside of the sphere. Centerline is 1.5" long.

The planes of light measured inside of the sphere only had a diameter of 1.15" because of the presence of the glass holder being used for plane positioning. The intensity plots across the centerline for three of these planes are shown in Figure 6-14 below.
Figure 6-14. Intensity plots across the centerline for three planes measured inside of the sphere. Centerline is 1.15" long.

It is clear from Figure 6-13 and Figure 6-14 that as the plane of interest moves further from the sphere outlet, the light becomes more Lambertian and less uniform. Furthermore, the light also decreases in intensity, as can be expected. In order to better characterize these changes as a function of distance from the sphere, the means and standard deviations of the relative intensity values was found for each centerline distribution. In order to create a fair comparison, only a 1.15" diameter circle was analyzed for the planes outside of the sphere.

Figure xx below is a plot of the average intensity across the centerline compared to the distance of the light plane from the interior of the sphere outlet.
In Figure 6-15, it is clear that the light uniformity is nearly constant until the light exits the Spectralon portion of the sphere. Once beyond the Spectralon, the light intensity starts to decrease and the variation in the light intensity across the centerline increases. To better illustrate this increase in variation, Figure 6-16 below shows the standard deviations as a function of the distance from the interior of the sphere outlet.
Figure 6-16. Standard deviation of light intensity as a function of the distance from the interior of the sphere outlet. This is the distance, ‘d’, from Figure 6-10 above.

The trend in decreasing uniformity is quite clear from Figure 6-16. It should be noted that the decrease in uniformity is not a result of the change in measurement techniques, for the uniformity decrease began even with the “in sphere” measurements. It is thus the result of exiting the Spectralon portion of the sphere that results in a decrease in uniformity, as can be expected.

Labsphere, the manufacturer of the sphere specifies the uniformity profile as a function of distance from the outlet of the Spectralon portion of the sphere as is shown in Figure 6-17 below, where x/D is the ratio of the distance from the outlet of the sphere compared to the diameter of the sphere’s output port. The d/D ratio is the ratio of the viewing diameter compared to the actual diameter of the output port. The Ee/Eo uniformity ratio is the ratio of intensity at the edge of the illuminated area to the intensity at the center.
Figure 6-17. Uniformity profile as a function of the distance from the outlet of the Spectralon portion of the sphere. $x/D$ is the ratio of the distance from the outlet of the sphere compared to the diameter of the sphere's output port. The $d/D$ ratio is the ratio of the viewing diameter compared to the actual diameter of the output port. The $E_e/E_o$ uniformity ratio is the ratio of intensity at the edge of the illuminated area to the intensity at the center. [58]

Since this analysis is using color values as a proxy for actual intensity measurements, an exact comparison of the LabSphere uniformity profile and the profile found from these measurements cannot be made. However, the standard deviation measurements are a good measure of the light variation, which is the inverse of uniformity. The variation profile, shown in Figure 6-18 below, has the same general profile shape as the inverse of the above uniformity profiles. This variation profile can be compared to the uniformity trend for $d/D=0.7$ in Figure 6-17 since the standard deviations were taken across a 1.15” diameter and the sphere port diameter is 1.5”. The $x/D$ value for which there is maximum variation is the same in both the reported and measured profiles.
Figure 6-18. Intensity variation versus x/D where x/D is the ratio of the distance from the outlet of the sphere compared to the diameter of the sphere’s output port. The sphere’s output port is 1.5” in this case. This trend can be compared to the uniformity trends for d/D = 0.9 in Figure 6-17 above.

As one last measure, it is interesting to compare the intensity profile of the sphere’s light output to the light profile of a typical MR16 light bulb at the face of the bulb, which is shown in Figure 6-19 below. There are two obvious hot spots and the standard deviation of the relative intensity values across the face of the bulb is 0.0428, which more than double the greatest variation experienced using the sphere, confirming that the sphere is a more optimal light source than the light bulbs.
This series of light uniformity measurements were a good indicator of sphere output performance from both a light intensity and light uniformity point of view and can be used as a tool for future design work. They also confirmed the benefits of using an integrating sphere as a uniform light source. In the next step, the temperature profiles across the microfluidic parts being bonded will be monitored over time.

6.3.3 Monitoring Temperature Profiles Through Thickness of Part During Bonding

The goal of this section is to get a better idea of how the temperature profile throughout the microfluidic part varies over time. A piece of glass and two pieces of PMMA were placed in the custom glass holder described above in Figure 6-6, and the holder was placed in a fixture that allowed the bottom surface of the holder to be flush with the interior surface of the sphere. Three types of glass were used: 1/16”-thick quartz, 1/8”-thick quartz, and 1/8”-thick

Figure 6-19. Light profile of a typical MR16 light bulb at the face of the bulb.
A borosilicate glass plug was placed on top of the PMMA pieces and uniform pressure was applied to the top of this plug. A cross-section of the glass holder setup is shown in Figure 6-20 below.

![Diagram](image_url)

**Figure 6-20. Cross-section of the glass holder setup.**

Measurements were taken at the glass – PMMA, PMMA – PMMA, and PMMA – plug interfaces. To take the measurements, a SA1XL-T surface mount thermocouple was secured at the interface of interest. Only one interface was investigated at a time to ensure that the presence of the thermocouple wasn’t interfering with the transmission of light to that particular interface. Each interface was studied multiple times. During all measurements, the fan to cool the outside of the sphere was left on and three 50W halogen light bulbs were turned on at the sphere input. The measurements were collected using a National Instruments USB-9211 Thermocouple Amplifier/DAQ that interfaced with LabView. There was a slight variation in the starting temperatures for each run due to the presence of residual heating effects. The initial temperature for each run was subtracted from every data point in that run, so that the change in temperature from the initial temperature could be studied. Thus, all of the data was normalized.
and could be compared across runs. The temperature trends over time for the three interfaces when a piece of 1/8”-thick quartz glass is used as the base is shown below in Figure 6-21.

![Graph showing temperature trends over time for the three interfaces](image)

Figure 6-21. Temperature trends over time for the three interfaces when 1/8”-thick quartz is used as the base glass. Multiple trendlines of a particular style indicate replicates.

The temperature trends above show a basic first order response over time. As was described earlier, direct heating is occurring throughout the glass and both pieces of PMMA. There is an exponential decay in the amount of heat of absorbed as the radiation passes through the part, as specified by the Beer-Lambert Law. It is expected that the interfaces further away from the light source will heat up more slowly than the interfaces closest to the light source. However, that is not the case for the measurements above. The glass-PMMA interface, which is closest to the light source, is consistently cooler than the PMMA interface. This can be attributed to the high transmittance of the quartz glass. Since it doesn’t absorb as much radiation as the PMMA, it doesn’t get as high in temperature. The glass-PMMA interface
temperature is effectively an average of the instantaneous surface temperatures that result from the bulk heating of the glass and the PMMA. As such, the surface of the PMMA that touches the quartz glass is “cooled” by the glass. There is a slightly different response when the 1/8”-thick borosilicate glass is used as the base, as is shown in Figure 6-22 below.

![Figure 6-22. Temperature trends over time for the three interfaces when 1/8”-thick borosilicate is used as the base glass. Multiple trendlines of a particular style indicate replicates.](image)

In this case, the PMMA-PMMA interface starts out hotter than the glass-PMMA interface, but after about 90 seconds, the gradient flips and the glass-PMMA interface is hotter. This phenomenon is also an effect of the differing absorptive characteristics of the PMMA and borosilicate glass. The borosilicate glass still absorbs less radiation than the PMMA, which is why the PMMA-PMMA interface is initially hotter than the glass-PMMA interface. However, the borosilicate absorbs more radiation than the quartz glass across all wavelengths, allowing the borosilicate-PMMA interface to eventually get warmer than the PMMA-PMMA interface.
While there is some conduction occurring between the PMMA and the glass, this is all local conduction. Since PMMA and glass have low conductivities, the heat transfer that occurs by conduction takes place much slower than the bulk heating from the light, which happens instantaneously. As such, the behavior at the glass-PMMA and PMMA-PMMA interfaces can be separated. This is confirmed by looking at how the temperature trends at the PMMA-PMMA interface compare when different base glasses are used, shown in Figure 6-23 below.

\[ (T - T_{bulk}) \text{ (°C)} \]
\[ \text{Time (s)} \]

**Figure 6-23.** Temperature trends at the PMMA-PMMA interface for different base glass. Multiple trendlines of a particular style indicate replicates.

Despite the differing behavior at the glass-PMMA interface when borosilicate glass is used as the base glass versus when quartz glass is used, the PMMA-PMMA interface temperature trends are effectively the same when the same thickness glass is used. This is because the temperature at this interface is primarily attributed to the amount of bulk heating that occurs from the absorption of light. As is shown in Figure 6-15 above, the average light
intensity is largely dependent on how far the targeted surface is from the exit of the Spectralon. The 1/8"-thick borosilicate and quartz glass discs both place the PMMA pieces the same distance away from the Spectralon exit, and as a result, the light available for absorption by the PMMA is comparable. When 1/16"-thick glass is used as the base, the PMMA is closer to the exit of the Spectralon and so the light intensity is higher. As a result, the temperature at the PMMA-PMMA interface is higher at a given point in time when this thinner glass is used.

It is also interesting to observe how the temperature at the PMMA-plug interface changes over time when different pieces of glass are used, shown below in Figure 6-24.

![Figure 6-24. Temperature trends at PMMA-plug interface for different base glass. Multiple trendlines of a particular style indicate replicates.](image)

At this interface, the temperature trends over time are not at all dependent on the type or thickness of glass used. As the light travels through the PMMA, there is an exponential decay of the amount of light absorbed. It appears that by the time the light reaches this top surface of
the PMMA, the exponential decay has flattened out such that changing the glass type or thickness has no significant effect on the absorption.

In order to summarize all of this information, the temperature difference between the interfaces is plotted over time in Figure 6-25 below.

![Temperature difference between interfaces for different base glass.](image)

Figure 6-25. Temperature difference between interfaces for different base glass.

Ultimately, these temperature measurements provided information about how the PMMA heats up over time. Furthermore, they proved that by choosing the base glass carefully, there is control over the temperature boundary condition at the glass-PMMA interface.

In the next section, an in-process vision system will be developed to allow the user to observe the bonding in real time.
6.4 In-Process Vision System for Bonding with Light

A schematic of the cross-section of the bonding setup used above is shown in Figure 6-26 below. Light enters the sphere through an input port and a custom glass holder sits in the exit port and holds the glass base and the PMMA parts to be bonded. A plug on top of the parts allows uniform pressure to be applied from a source external to the sphere.

Figure 6-26. Schematic of the bonding setup in its current state. Light enters the sphere through an input port. A custom glass holder sits in the exit port and holds the glass base and the PMMA parts to be bonded. A plug on top of the parts allows uniform pressure to be applied from a source external to the sphere. In its current state, the light exiting the sphere passes through the glass and the PMMA parts and certain wavelengths get absorbed. The plug absorbs the remaining light that is transmitted.

In its current state, the light exiting the sphere passes through the glass and the PMMA parts and certain wavelengths get absorbed. The plug absorbs the remaining light that is transmitted by the glass and PMMA. But what if this light could instead be viewed external to
the sphere to allow for real-time viewing of the part being bonded? By making the plug out of glass and then putting a beamsplitter on top of the plug, visible light can be redirected to an external video camera. A schematic of this setup is shown below in Figure 6-27.

![Figure 6-27. Schematic of bonding setup with real-time video control. Light exiting the sphere first passes through the glass and PMMA, and some of it gets absorbed. The light that is transmitted travels through a glass plug and beamsplitter. At the diagonal midplane of the beamsplitter, a certain percentage of the light is redirected to the video camera, where a real-time image of the part being bonded will appear. Uniform pressure can still be applied.](image)

This system was fabricated, and a picture of the setup is shown below in Figure 6-28. In this setup, there is an aluminum support structure surrounding the sphere. There is a hole in the top plate of this structure that lines up with the exit port of the sphere. The lip of the custom glass holder can then rest on the support structure instead of on the sphere so that the sphere does not have to bear any load. As a result, the glass holder has to be taller and the plug thicker.
The glass plug is made of two 0.75" thick heat-resistant borosilicate glass discs. The two borosilicate discs and the beamsplitter are secured together with a UV-cured optical glue. A piece of rubber is placed on top of the beamsplitter to effectively spread the pressure from the Instron. A Dino-Lite camera is mounted on the top plate of the support structure, in line with the beamsplitter. It is mounted such that its position and focus can be easily adjusted in real-time. It also feeds into a computer to allow for real-time viewing of the bonding process by the user. A 100 cfm fan sits next to the setup to cool off the sphere either during the bonding process or intermittently throughout testing when the sphere gets hot.

Figure 6-28. Integrating sphere experimental setup.
Figure 6-29 shows an image of the input port where the light enters the sphere. The light bulbs are held by ceramic bulb holders, which have wires that extend to the power supply. To keep the light bulbs in place, a piece of aluminum slid in place over the bulb holders and clamped down to the top plate of the sphere structure.

To test this setup, a cover plate-substrate stack was put into the experimental setup with a piece of 1/16" thick quartz glass being used as the base. A pressure of 155 kPa was applied. All three 50W light bulbs were turned on, and the bonding was observed with the Dino-Lite camera/computer interface. Since all three light bulbs are shining and most of the visible light is transmitted through the PMMA, if all of the light is processed by the camera, the image will
saturate. To prevent this, the beamsplitter is such that 70% of the light is transmitted and 30% of the light is reflected towards the camera. Furthermore, the Dino-Lite auto-adjusts for varying light levels, eliminating the need for neutral density filters. Images from the bonding video at three time points are shown in Figure 6-30 below. The lighter region is the area that has been bonded. This region is clearly spreading as time goes on, allowing the bonded region to be monitored over time.

![Figure 6-30. Images from bonding video at three different time points. The lighter region is the area that has been bonded and it spreads out over time. This bonding is done with 1/16” quartz as the base glass, 0.155MPa of pressure, and three 50W halogen bulbs at the input.](image)

The optical transparency of the microfluidic part changes when the pieces are bonded together versus when they are just sitting on top of one another under pressure. When the two PMMA pieces are touching but not bonded together, there is a natural air gap at the interface. When the light passes through this air gap, some of it refracts and reflects, reducing the total amount of light transmitted. However, once the bond forms, all of the light can be transmitted, thus increasing the optical clarity of the PMMA stack [60]. The increased optical clarity results in a lighter color appearing wherever a bond has formed.

In this particular setup, there are actually two interfaces where bonding can occur. The first, and most obvious, is at the substrate-cover plate PMMA interface. The second is at the PMMA cover plate – glass base interface. While permanent bonding will not occur between the
glass and the plastic, under the elevated temperature and pressure conditions during bonding, the PMMA will temporarily adhere to the glass. When this occurs, the interface’s air gap disappears, increasing the optical transparency of the stack. PMMA-glass bonding can technically also occur between the top of the substrate and the glass-beamsplitter stack. However, the temperature at this interface never gets hot enough to cause such bonding to occur.

Due to the presence of two bonding interfaces, the change in optical transparency actually occurs in two stages, as can be seen in Figure 6-31 below. In this figure, there are three regions labeled, each a different shade of gray, indicating a different optical transparency. The darkest, or least optically transparent, region is the area on the chip that has not yet experienced any bonding. The brightest region is the area where bonding has occurred at both interfaces, thus allowing the most total light through the part. The region of middle intensity is the area where only one of the two interfaces has been bonded. The interface to bond first depends on the glass and plastic involved. In trials, when borosilicate glass was used as the base glass, the PMMA-glass interface bonded first. This is because the PMMA-glass interface is hotter than the PMMA-PMMA interface, as was shown by the temperature measurements in the previous section. When quartz glass was used as the base glass, the PMMA-PMMA interface bonded first because the PMMA-PMMA interface is hotter.
Figure 6-31. Three bonding regions on a microfluidic chip. Region 3 is the area on the chip that has not yet experienced any bonding. Region 1 is the area where bonding has occurred at both the PMMA-glass and PMMA-PMMA interfaces. Region 2 is the area on the chip where bonding has only occurred at one of these two interfaces. The brightness and contrast have been adjusted to make the different shades more visible.

As one last proof of this concept, a series of parts were bonded and instead of waiting for the entire part to be bonded, the process was stopped prematurely. The Instron pressure was immediately released and the contour of the bonded region on the part was compared to the last frame of the corresponding bonding video to see how the two matched up. The matching contours prove that this method of monitoring bonding is accurate. An example of these matching contours are shown in Figure 6-32 below.
If the lights were turned off but the pressure was not released immediately, the bonded region on the part was actually slightly larger than the bonded region in the last frame of the video. The video ends when the lights are turned off, but the parts are still hot even though the lights are turned off and if they are still under pressure, there is an opportunity for the bond to continue spreading. An example of this is shown in Figure 6-33 below. This spreading was allowed to occur because the interface had not yet cooled down, but it was still under pressure.
This real-time monitoring system made it possible to observe the bonding process for many substrate-cover plate combinations in order to gather more information about how bonds form using this process. These observations will be discussed in the next section.

6.5 In-Process Bonding Measurements

In a first set of tests, a series of microfluidic substrates were bonded to cover plates and the process was recorded with the in-process vision system developed above. In each of these videos, the bond began as a small circle that then grew in size over time. The exact location of the initiation point varied from part to part. In order to judge the bond’s trajectory as it spread, the size of this bonded region was plotted over time. The diameter of the brightest bonded spot (greatest optical clarity, both PMMA-glass and PMMA-PMMA interfaces are bonded) was measured along two axes and these two measurements were averaged. During the bonding
process, three 50W halogen bulbs were turned on at the sphere’s input, 1/16”-thick quartz glass was used as the base glass, and the fan used to cool the sphere’s exterior was kept on. There were eight microfluidic chips, four each from two different embossing runs. Each of the four parts from an embossing run was bonded at one of the following pressures: 77.5kPa, 155kPa, 310kPa and 465kPa. The diameter measurements over time are shown in Figure 6-34 below.

![Diameter of bonded region over time for parts from two different hot embossing runs at four different bonding pressures. During the bonding process, three 50W halogen bulbs were turned on at the sphere’s input, 1/16”-thick quartz glass was used as the base glass, and the fan used to cool the sphere’s exterior was kept on.](image)

Each of the trend lines in Figure 6-34 has the same general shape, but a different starting point. That is, the bond initiated at a different point in time, but once it started, it spread in a similar fashion for all of the parts. The diameter measurements over time with all starting points normalized to time zero are shown below in Figure 6-35. In almost all cases, a higher bonding pressure caused the bond to start forming sooner, as can be expected. Furthermore,
except for the parts bonded at 77.5kPa, the parts from the first embossing run started bonding sooner than the parts under the same bonding pressure from the second embossing run. This is a result of differences in surface asperities and substrate flatness between embossing runs, and between parts in general.

![Graph showing diameter of bonded region over time for different bonding pressures.](image)

**Figure 6-35.** Diameter of bonded region over time for parts from two different hot embossing runs at four different bonding pressures. Starting points all normalized to time zero.

One of the major challenges of bonding evenly across the substrate-cover plate interface is overcoming these differences in surface asperities and substrate flatness from part to part. Such variation contributes to the low-yield nature of certain bonding processes. By implementing this in-process vision system, each individual part can be monitored as it is being bonded. Instead of bonding each part for a specified amount of time, a few points on the part can be monitored for changes in optical clarity. Once the optical clarity at all of the required
points has changed, then the bonding process can be stopped. This process will help to ensure that all parts are given a chance to bond completely.

Furthermore, by actively viewing the way in which the bond spreads, the user will be able to identify any areas on the microfluidic chip that seem to cause problems in the bonding process. For example, in one set of trials, a small portion of the microfluidic chips had not embossed completely and the surface along one of the edges was uneven. In most previous cases, such as in all of the parts described in Figure 6-34 above, the bond initiated somewhere in the center of the part. However, these later parts always started bonding at the edge of the part with the uneven surface and it took longer for the bond to spread for the rest of the part because this difference in surface height needed to be overcome. As a result, the required bonding time was longer than necessary and the channels were heating up under pressure for longer than was actually required, resulting in unnecessary channel deformation. In cases such as this one, the in-process vision system could be used to observe these problem areas and then make subsequent changes to the embossing process in order to mitigate the problem. This particular case also made it clear how sensitive the bonding process is to surface asperities and substrate flatness, heightening the importance of having an in-process vision system for user feedback.

In another instance, the sphere and glass holder had moved out of alignment, resulting in a change in the light pattern across the PMMA parts. Because the glass was at an angle, the light at one edge of the glass was more intense than the light at the other edge. As a result, the bonds were always initiating at the edge with more intense light and the bond spreading slowed down at the edge with lower light intensity. After observing a few parts in different configurations bond in this manner, the alignment issue was discovered and rectified. In this way, the in-process vision system gave the user feedback about how the system itself was performing. Furthermore, it made it apparent to the user how slightly varying the power effects
the rate at which bonding happens. In this case, the bond spreads from the edges. In other cases, when the parts are flatter and there aren’t any alignment issues or uneven surfaces, the parts begin to bond in the middle of the part. As was mentioned earlier, though the light is quite uniform, there is still a Lambertian profile to the light exiting the sphere. Since the parts being bonded are very similar in size to the output port of the sphere, there is a definite light intensity gradient across the part, with maximum power near the center of the part. As a result, the channels at the center have the most opportunity for deformation and shape recovery. In the next section, this channel deformation will be measured.

6.6 Measurement of Channel Deformation

The key to successful bonding is achieving sufficient bond strength without compromising the underlying channels. Accordingly, a series of tests were performed to measure both bond strength and the post-bonding shape of the substrate channels.

To measure channel deformation under various bonding conditions, a series of microfluidic substrates were embossed out of 1.5mm thick extruded PMMA and a series of cover plates were made with 1.5mm thick cast PMMA. When a cast PMMA substrate is bonded to a cast PMMA cover plate, the channels will deform and obliterate faster than when the substrate is made of extruded PMMA. This can be attributed to the slightly differing thermal properties of the two grades of PMMA that affects the shape recovery and deformation of the polymer during bonding. Accordingly, the extruded-cast combination is used. In an effort to create a definable end point for the bond, the area of the cover plate was smaller than the area of the entire microfluidic part, as is shown below in Figure 6-36.

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1 The embossed pattern was that of the µFac microfluidic mixer from Figure 1-1. The parts were embossed with the µFac hot embossing machine developed by Melinda Hale. The embossing parameters were as follows: Forming temperature - 140°C; Forming force - 500N; Heating time without any force - 5 seconds; Forming time - 30 seconds; Cooling time - 95 seconds; Demolding temperature - 60°C
Nine parts were bonded, all with different light input and bonding pressure conditions. The 1/16''-thick quartz disc was used as the base glass. The fan to cool the exterior of the sphere was turned off. The pressure was first applied and once it reached steady state, the lights were turned on. Once the entire cover plate was seen to be bonded to the substrate, the lights were turned off and the pressure was released. The bond was then broken and the post-bonding channel heights were measured. The channel height was defined as the height of the existing channel on the cover plate minus the height of the corresponding protrusion on the cover plate. The heights were measured for channels 1, 5, and 10 of the serpentine, as indicated in Figure 6-37 below.
Figure 6-37. Channels 1, 5, and 10 of the serpentine pattern on the microfluidic chip. These are the channels that are to be measured.

The average post-bonding channel heights for these channels are shown in Figure 6-38 below. These are the averages across nine bonded parts, each of which was bonded with different input powers and bonding pressures. The error bars are the standard deviations of the post-bonding heights. Smaller post-bonding heights indicate more deformation. Since this plot is an average of parts bonded with many different bonding parameters, its purpose is to show the clear trend in degree of deformation across the channels, not to study the variability of this deformation.
Figure 6-38. Average post-bonding channel heights for channels 1, 5, and 10, as shown in Figure 6-37 above. This is the average across nine bonded parts, each of which was bonded with different input powers and bonding pressures. The error bars are the standard deviations of the post-bonding heights. Smaller post-bonding heights indicate more deformation.

It is clear from this chart that channel 5, which is the center channel, consistently experiences the most deformation. This trend also follows when looking at the individual measurements from every part. This behavior can likely be attributed to two factors. The first is that in this particular run, the center of the part tended to bond first. As was discussed earlier, initiating the bond involves both getting that particular region up to temperature and ensuring that the two surfaces are in contact. Once this contact is ensured, there will be more opportunity for channel deformation because the energy in that region will go into bonding and not into overcoming surface asperities or part warping. The channels will also be right at the interface, instead of separated by an air gap, and so they will be immediately affected by the bonding pressure. While this factor was shown to have some effect on the degree of deformation experienced by a particular portion of the chip, a more significant factor is the intensity of the light hitting a particular region of the interface.
As was discussed earlier, there is a Lambertian profile to the light exiting the sphere. While this profile is fairly flat, it is still present, and so the region at the center of the part has the most light available to be absorbed. As a result, the polymer is the softest in this region and more susceptible to deformation. Furthermore, because it gets hotter, it is also more susceptible to shape recovery.

This light profile also contributes to the fact that the bonds are starting in the middle of the part. In this case, the substrates are fairly flat and so surface asperities don’t take over the bonding pattern. As a result, the bonding begins in the center of the part because this is where it is the hottest. In a more ideal configuration, the light would be collimated and the entire part would reach the same temperature at the same time. Broadband light sources, such as the tungsten-halogen source from Spectral Products [55], have options for collimated or fiber optic outputs. Based on these observations, the collimated output configuration of this product could potentially be a better option than the integrating sphere.

It was observed that when the bond begins by forming in the middle of the part, the bond begins spreading fairly quickly, but then slows down as it reaches the edges of the part. This observation can be described by the graph in Figure 6-34 above, where the change in the diameter of the bonded region in a given period of time decreases as time goes by. This same phenomenon was observed for smaller cover plates, indicating that it is an edge effect at the rims of the cover plate and not just a result of the Lambertian nature of the light. If heat escapes from the cover plate at the edges, it takes longer for the edges to heat up and bond. As such, dealing with both the Lambertian nature of the light and the edge effects is important in future developments of this idea.

Another major observation about the average post-bonding heights in Figure 6-38 above was the extreme levels of deformation. Each of those channels was originally 39-40 microns
deep and after bonding they had shrunk to as few as 3 microns. The average channel heights, as well as the required bonding times, for individual parts are shown in Figure 6-39 below as a function of bonding parameters.

<table>
<thead>
<tr>
<th>Bonding Pressure (kPa)</th>
<th>Channel Height (μm)</th>
<th>Bonding Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>7.9 μm</td>
<td>153 s</td>
</tr>
<tr>
<td></td>
<td>11.0 μm</td>
<td>169 s</td>
</tr>
<tr>
<td></td>
<td>7.6 μm</td>
<td>207 s</td>
</tr>
<tr>
<td>131</td>
<td>11.0 μm</td>
<td>188 s</td>
</tr>
<tr>
<td></td>
<td>11.8 μm</td>
<td>205 s</td>
</tr>
<tr>
<td>112</td>
<td>9.0 μm</td>
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<tr>
<td></td>
<td>11.0 μm</td>
<td>325 s</td>
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<td></td>
<td>3.9 μm</td>
<td>235 s</td>
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<td>9.0 μm</td>
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<td>3.9 μm</td>
<td>235 s</td>
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<tr>
<td>75</td>
<td>188</td>
<td>263</td>
</tr>
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<td>564</td>
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</tbody>
</table>

Figure 6-39. Average post-bonding channel heights and required bonding times for individual parts bonded with different parameters. Cover plate is that shown in Figure 6-36 above. The post-bonding channel height is the average of the heights of channels 1, 5, and 10, as measured by the Zygo Profilometer. Channel heights are in microns.

Based on these measurements, it is clear that the correct balance of bonding pressure and light input power is required. At high pressures and input powers, there is excessive deformation because of shape recovery and because of the softening of the polymer under high pressure. At low pressures and temperatures, it takes so long for the bond to form, that there is more time for the polymer to flow and the channels to deform. It is in this regime that shape recovery is likely dominant. The time required to bond the entire cover plate to the substrate has a clear dependence on the bonding parameters. As can be expected, bonding with more power and/or at higher pressures results in faster bonding, though with more channel
deformation. The channel deformation can be attributed to the bonds taking too long to spread. The area with the channels is often bonded before the edges bond and then the channels just sit under heat and pressure for longer than they need to while the rest of the cover plate bonds to the substrate. This leads to excessive deformation of the channels that is not actually necessary for a bond to form.

In order to investigate the effect of excessive bonding times, a smaller cover plate was used so that a smaller area would need to be bonded and a shorter bonding time would result. An image of the substrate-cover plate stack is shown in Figure 6-40 below.

![Substrate-cover plate stack; smaller cover plate. Cover plate is 0.3 in\(^2\) in area.](image)

The same process was used for bonding and the resulting average post-bonding channel heights and required bonding times for the different bonding parameters are reported in Figure 6-41 below.
There is a clear improvement in the post-bonding channel heights when the smaller cover plate is used and the bonding times are shorter at a given pressure-input power operating region, indicating that the slow nature of the bond spreading is an issue. However, in general, the times are not drastically shorter than those reported in Figure 6-39 above. It was observed that the bond spreading still seemed to slow down as it approached the edges of the cover plate, an observation that is interesting to keep in mind as an avenue for future work. Design changes can be made to mitigate this issue, but it is first critical to prove that channels can be bonded with minimal deformation.

To check the hypothesis that shorter bonding times would lead to lesser deformation, a series of experiments were performed where the process was stopped after a substantial portion of the channels were bonded, but before the entire cover plate was completely bonded to the substrate. In this way, the channel area was only exposed to heat and pressure for the amount of
time necessary to bond the critical region, but not more than that. The post-bonding channel heights were measured as before, except this time instead of measuring channels 1, 5, and 10, a few channels from whatever region was bonded were chosen. The average post-bonding channel heights and times of exposure are reported in Table 6-3 below.

Table 6-3. Average post-bonding channel heights after bonding for different amounts of time under various bonding conditions. Bonding was stopped after at least a few channels were bonded. These were the channels that were measured and averaged. Cover plates of various sizes were used. All parts were from the same embossing run except the starred part, which was from a previous μFac run. Bold numbers indicate acceptable post-bonding channel heights.

<table>
<thead>
<tr>
<th>Sphere Input Power (W)</th>
<th>Bonding Pressure (kPa)</th>
<th>Time Spent Bonding (s)</th>
<th>Number Channels Bonded</th>
<th>Average Post-Bonding Channel Height (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>78</td>
<td>176</td>
<td>6</td>
<td>26.5</td>
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<td>28.6</td>
<td></td>
</tr>
<tr>
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<td>930</td>
<td>187</td>
<td>8</td>
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<td>28.7</td>
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<td>104</td>
<td>10</td>
<td>27.2</td>
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All of the above parts showed tremendous improvement in their post-bonding heights compared to the previous runs. While not all of these parts had acceptable post-bonding heights, there were three parts that had less than 5 microns of channel deformation, proving that it is in fact possible to bond microfluidic devices with minimal channel deformation using this heating method. In order for this minimal deformation bonding to occur, the bonding process just needs to be stopped at the right time to prevent excess deformation after the bond forms. In two of the minimal deformation parts above, the cover plate actually showed no signs of
protrusions, indicating that there wasn’t enough time for the softened cover plate to squeeze into the channels, but rather just enough time to cause the interfacial bonding, which is an optimal situation. In a possible future implementation of this technology, once this initial bonding occurs, the input power and/or pressure can be decreased to prevent excess deformation while still allowing the interfacial polymer chains to reptate and a stronger bond to form. This avenue for further development will be discussed further in the next section.

Interestingly, the bonding times are not necessarily significantly lower for the acceptable parts than for parts with more deformation. Based on all of the previous observations, it is clear that the optimal bonding time is different for every part and since this bonding process involves such high temperatures, it will be critical that the bonding process is stopped at the right time. This is a perfect example of how the in-process vision system will come in handy, as will be described further in the following future developments section.

6.7 Future Developments

The data acquired using the integrating sphere setup thus far proves that using light absorption as the heating mechanism for bonding can accomplish bonding of microfluidic channels with minimal channel deformation. It is just critical that the bonding process is stopped at the right time. Furthermore, this particular time is different for every part. It is a function of bonding pressure and input power, but also affected by substrate warping and surface asperities. This is where the in-process vision system that has been created is so helpful. This in-process vision system is currently very useful for visualizing bonding and identifying problem areas on a particular chip. However, in future developments, the system can be implemented to provide real-time control of the bonding process. It is also important that in any future systems, bond strength is also considered.
In the current system, it is possible to tell if a channel is obliterating, and to a small extent, it is also possible to tell when the channel is beginning to deform. However, if a higher resolution camera with better focusing capabilities were used, then it would be possible to observe the distinct channels and to see when they begin deforming. It is also possible to use a stack of beamsplitters and a few cameras in order to observe multiple locations on the chip. If the illumination source were made of an array of independently controlled uniform lights, the area of the chip above each individual light source could be monitored separately. Once that region had achieved bonding, the intensity of the light source below it could be attenuated or shut off entirely. Furthermore, if the channels in a particular region were beginning to deform or collapse, the light in that region could be attenuated. Since it is light being controlled, and not the temperature of an adjacent hot plate, the adjustment will be immediate because there will not be any capacitive cooling of the heaters involved. In an even more complex system, the pressure can be dynamically controlled as well. This method would allow the bonding of each part to be independently controlled in real-time.

Even if an array of independently controlled lights were not used, switching to collimated light from this Lambertian light source and viewing the part at higher resolution would still allow for effective control of the bonding process. Signs of bonding or channel collapse can trigger light attenuation. In such a process, the parts would be heated up quickly in the beginning, but then just kept warm during the bonding process, likely at a temperature just above the glass transition temperature of the polymer. Then once the vision system detected that the entire part was bonded, the process could be stopped. Such a system would make it possible to generate robust bonds with a unique set of bonding parameters for each part. As a result, the challenge of finding a process that is robust to process changes and part-to-part variation would be solved.
Chapter 7

Conclusions

This work was an investigation of innovative ways to robustly heat the substrate-cover plate interface of a microfluidic device for the purpose of bonding and sealing the microfluidic channels. An extensive literature review revealed the benefits of interfacial heating, and both simulations and experimental investigations were used to evaluate a few different methods. Ultimately, a unique method was established that uses light to provide both the bonding energy and the illumination for an in-process vision system for real-time viewing and control of the bonding process. The process results in the generation of a homogenous and optically clear bond.

The equipment configuration in this new system is such that the substrate surface is heated more intensely than the bulk of the substrate, thus allowing a beneficial temperature gradient to develop in the region of the microchannels. Furthermore, since the bonding process occurs during the transient heating stage, this temperature gradient is maintained over time. The use of radiation for bulk heating creates the opportunity for fast control of the heating conditions. An immediate attenuation or intensification of the input light power will result in an immediate change in the absorption and heating conditions throughout the substrate. In conventional thermal bonding systems, however, lowering the external heater temperatures will not immediately affect the heating at the interface, for the heater itself needs to cool down, as do the substrate and cover plate materials. Preliminary tests show that when properly controlled, a bond with minimal deformation of the microchannels can be created.
An even greater benefit of using light for the heating process is that the excess light that is transmitted through the microfluidic chip can be redirected by a beamsplitter and used for real-time viewing and control of the bonding process. This vision system has already revealed useful information about the ways in which bonds form, and has confirmed the sensitivity of the bonding process to variations in the embossed substrates being bonded. In future developments, this in-process vision system can be used to tailor the bonding parameters of each bonding cycle to the specific embossed part being bonded. Such control over the bonding process is incredibly powerful and can lead to higher yield and more robust bonding of microfluidic devices.
CHAPTER

8

References


