

THE ART OF MICROCHANNEL MOLDING IN  
MICROSCOPE GLASS SLIDES

by

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## Abstract

# THE ART OF MICROCHANNEL MOLDING IN MICROSCOPE GLASS SLIDES

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This research is an effort to develop a new process by combining engineering principles with the history of glass art molding to develop clear micro-level test features in glass, for bio-medical and drug experiments dealing with cells, tissue, and micro-level organisms. Most of the present research in the bio-medical field involving micro-level cell and brain tissue testing is done using expensive glass or developed using Polydimethylsiloxane (PDMS) as the main material. Disadvantages of PDMS is that, over time the cell or blood tissue reacts with polymers causing a reduction in clarity making it difficult for a research scientist to observe the growth and reactions of the cells and tissue. Glass can be used to overcome many troublesome issues of PDMS due to such properties as: 1) high chemical resistance, 2) good light transmission which allows direct visualization of chemical or cell reactions, 3) high thermal and electrical resistance which allows the application of high voltage for electrophoretic separation and 4) good mechanical stability allowing application of high hydraulic pressure for transporting fluids inside micro-channels.

Research into existing inexpensive low-quality glass, has resulted glass slides which can be used by bio-researchers for many applications. Soda-lime glass slides have been in use by biomedical researchers to conduct experiments for decades. The only constraint was they were used as raw glass slides, without any additional manufacturing being done on the slides due to their machinability restrictions. Fabricating micro features in glass is a challenging task as glass reacts differently to different die materials, micro feature manufacturing processes, and different process temperatures. These issues have kept researchers from using inexpensive low-quality sodalime glass, such as that used for the manufacture of microscope slides.

The aim of this research is to understand the principles and reactions of glass to varying temperature and machining processes, choosing a best machining process and material, optimizing the temperature and machining variations of glass with the selected die material. The research objective was to produce clear micro-level features in glass. Standard glass microscope slides made of soda-lime glass were selected as the material of choice. The results of the study found mold materials, mold machining processes, glass working techniques, and optimal heat cycle for clear glass working of microfeatures.

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## Chapter 1

### Introduction

#### 1.1 Need for Glass Slides with Microchannels

Microfabricated devices, resulting from fabrication of three-dimensional microstructures with well-defined topographies, are generating intense interest from the analytical and engineering community, for many applications in the areas of microfluidics, microoptics, micromechanics and biochemistry, based on their potential to revolutionize analytical measurements [1] [2]. These devices are used in micro analytical systems including capillary electrophoresis for Deoxyribonucleic acid (DNA) and protein separations, as biochemical reactors, sensors, and applications including biology and tissue engineering [1]. Microfluidics can benefit from such three-dimensional (3D) complex structures by affecting the hydrodynamics of fluid flowing inside microchannels which can generate vortices at low Reynolds numbers. Microfluidic devices consist of a microchannel network for transporting fluids to regions where reactions, separation, and analysis can be performed more rapidly with less consumption of reagents. Usually the dimensions of the channels are in the range of 10-100  $\mu\text{m}$ , which result in low Reynolds numbers making the flow laminar. Under laminar conditions, mixing is dominated by diffusion which is very slow process [2]. The substrate used also has an effect on the speed of the process and use of hydrophobic molecules requires robust and stable surface. A glass substrate is considered best for these processes as well as for many biomedical experiments [3] [1].

A microchannel fabrication process often requires multiple sequential operations including lithography, alignment, replication and bonding, making the microfabrication process complex and expensive. Among most materials used for the fabrication of microfluidic systems, glass is a preferred material for biochemical applications owing to such interesting properties as: 1) high chemical resistance, 2) good light transmission which allows direct visualization of chemical or cell reactions, 3) high thermal and electrical resistance which allows the application of high voltage for electrophoretic separation and 4) good mechanical stability allowing application of high hydraulic pressure for transporting fluids inside microchannels. Due to its chemical inertness, the glass surface can be functionalized with biomolecules.

### 1.2 Background of Research and Challenges

The first microfluidic systems were fabricated in glass, using fabrication technology borrowed from the semiconductor industry, such as electron beam lithography, photolithography, and wet and dry etching [1]. All of these operations provide robust and high quality microfluidic systems; however, the processes for fabrication are often time consuming and require sophisticated equipment located in clean rooms that are not always readily accessible. The drive to fabricate complex microfluidic devices where direct visualization of the reaction taking place within the unit is required, led to the development of soft lithography and the use of polydimethylsiloxane (PDMS) as a substrate.

Several methods used for fabrication of 3D microfluidic structures, which mostly consist of fabricating the microchannel directly or to prepare master mold for their

replication in clear polymer materials (e.g., PDMS, PMMA) using time consuming and sophisticated equipment located in clean rooms that are not always readily accessible clean room technologies such as photolithography and etching. Even with limitations, PDMS is preferred as most of these technologies are used for making a master-mold which is a one-time cost. Microchannels in PDMS are easily replicated using the molds, making PDMS more advantageous than glass. One of the main disadvantage to use glass microfluidic systems in comparison with PDMS systems comparing all the existing technologies and processes, is the tedious and expensive methods some of them involving the requirement of special facilities, expensive equipment and experts, in order to produce glass microchannels currently. Leaving enough potential for any new inexpensive processes that can be developed without the requirement of special facilities of complex mechanisms or machines [1].

## Chapter 2

### Literature Review

#### 2.1 History of Glass Working Processes

Glass working is a tradition that developed around 5000 years ago, and while the origin is still debated, historians agree that it was invented in Syria, Asia, or Egypt [4].

Glass has a base formula of silica and flux [5]. The silica originally used, was found in the form of raw sand, while the flux was sodium derived from plant ashes. When combined, these ingredients make up the pre-melted powder mixture, or batch. The batch is then heated in a crucible composed of ceramic material where the batch turns into a liquid. This process is also known as a primary melt, or fritting, because the top layer of the pot is crushed into small chunks and re-melted while the bottom layer is not in a condition to be used. The glass chunks from the top of the primary melt are then re-melted in a secondary melt where it is heated again to 1710°f and then gathered onto a rod to be worked into glass objects [6]. Along with coloring agents, each ingredient in the batch has an effect on the overall visual characteristics of the final glass product [7].

In each of the three areas where historians agree to be the places of glass invention, there is an abundance of sand, which was the original source of silica in the glass batch mixture. It wasn't long though before glassmakers realized that the source of silica had an effect on the quality of glass. Sand from riverbeds and beaches had many mineral impurities, which results in glass with internal cloudiness. For early glass manufacturers the opacity wasn't a problem because they were often trying to duplicate expensive stone objects similar to lapis lazuli or jade [8]. Another side effect resulting

from the use of ordinary sand with varying mineral deposits was inconsistency during the manufacturing process. When sand from beaches and rivers was used each mixture or batch would melt differently. Some melted easily at a relatively normal temperature while others had to be heated to extremely high temperatures for long periods of time using tremendous amounts of resources. This inconsistency showed up in the durability of the final product as well. While some objects, were strong, others were more fragile and brittle [9]. The manufacturing process was so rudimentary that most of the early examples of glass were susceptible to atmospheric humidity, which caused the surface of the glass to lose its luster and become opaque. Opacity is caused by microscopic pits in the surface of the glass, which have deteriorated over time. Over time manufacturers in the Levant began to see the potential for transparency in the material and began to experiment with different sources for silica.

The second ingredient added to the batch was ash from burned plants. Ash act's as a flux, a mixture that combines with the silica particles, allowing them to coagulate and melt at a much lower temperature. Just like the silica over time different qualities became apparent with the addition of different ashes. Initially the glassmakers were unconcerned with the effect that the quality of plant material had on the clarity of glass, but as time went on that also would change [7].

For seven centuries Roman, Sassanian, and Egyptian glass manufacturers continued experimenting with different sands and types of ash to observe the results each had on the glass. In Mesopotamia manufacturers found that pebbles, rocks or sand made of quartz, which is 99.9 percent silica, made magnificent translucent glass when melted.



Sand from the Belus River became a main ingredient in glass making because the water gradually wore down the pure quartz pebbles, leaving a rare naturally occurring, quartz sand bed [9]. The use of quartz sand improved optical transparency, but also increased the melting temperature beyond what could be achieved in ancient furnaces. To attain the transparency through the use of quartz, manufacturers needed a more powerful flux than the plant ashes that were previously used to reduce the melting temperature.

The manufacturers introduced ash that was higher in lime, a source of natron (natron is a naturally occurring mixture of sodium carbonate decahydrate, a kind of soda ash and about 17% sodium bicarbonate along with small quantities of sodium chloride and sodium sulfate) as high-powered flux [6]. The natron lowered the melting temperature and stabilized the surface of the glass, which prevented it from turning opaque. This dramatically improved the optical clarity of the glass [10]. Additionally, manufacturers realized that sand from the Belus was high in mussel shells, which were a rich source of natron. This began the introduction of mussel shells into batch recipes in most Levantine glass manufacturing centers and increased the demand for glass ingots (A block of glass cast into standard shape for shipments) from the area. They managed to find sand and leaves that were high in sodium and low in other impurities, and added extra natron in the form of mussel shells, and achieved a transparent material [7].

By the 9th century glass from Mesopotamia became not only the most transparent material, but also glassmakers in the area made huge contributions to the aesthetics that would be employed for centuries to come. Glassmakers from Islamic lands continued using traditional Egyptian and Roman methods of embellishing the glass that included

marveled trails borrowed from Egypt, as well as luster painting and carving that originated in Rome. Islamic glassmakers began using the sgraffito technique they borrowed from the ceramic artisans of the time. The sgraffito technique uses a sharp object to incise the surface of an glass, which allowed them to create beautiful patterns on glass. The sgraffito vessels of this period always employed a diagonal cross hatching that creates a background, which allows the decorative element to stand out, or a vertical cross hatching to fill small spaces. The detail attained with this technique allowed Islamic glassmakers to use arabesques and other floral and vegetal patterns, which alluded to the garden of paradise. The second aesthetic development by the Islamic glassmakers, which was also borrowed from the ceramics tradition, was the employment of luster painted glass. This technique enabled them to communicate Islamic words and calligraphy as well as another avenue to communicate the promise of the garden of paradise after death through vegetal patterns. The luster painting technique required that the glass painter mix minerals with a sticky substance and then paint this mixture on to a blown form. After it was painted, he would then re-heat the vessel, causing a chemical exchange between the minerals and the glass leaving a smooth permanent surface [11]. The process of re-heating the vessel and doing some final shaping allowed the design to morph with the shape of the vessel. The result was a more organic painterly approach than the sgraffito technique.

By the 12th century, glass artists in Islamic lands had learned and improved many of the traditional shapes, technologies and aesthetics of the Roman and Byzantine artists. The glass from the Islamic lands was the best in the world without a competent rival [12].

Their success did not last, however; economic decline in the Levant caused glassmakers to go out of business and forced glass manufacturers to export their raw glass to Venice, which would become the glass center of the world by the mid-12<sup>th</sup> century. This transition was easy for the Venetians who had been absorbing glassmaking knowledge and skills from Islamic lands for centuries.

Over time, glass recipes became more technical, and manufacturing became more complex. In ancient Egypt the glass manufacturing process was done at the same locations where objects were formed. By the Byzantine era, evidence suggests the manufacturer's melted batch then crushed it into small chunks or ingots, which could then be shipped to the glassmakers who re-melted the ingots and formed the glass into objects [13]. Wreckage found in a Bronze Age shipwreck at Ulu Burun suggests that the manufacturing and forming processes were indeed separated. Among other precious materials such as gold, silver and copper, there were glass beads and clear glass ingots on board the ship that was believed to have been carrying goods from the East to Europe [14].

As the east-west gateway for trade, Venetian glass made the transformation from the previously green tinted material to a transparent glass with a similar recipe as that found in the Levant. This optically clear canvas and examples of enameled imported from the east inspired a young artist named Gregorio to team up with local glassmakers and market a Venetian enameled beaker for export to Europe. Venice was able to control much of the information that traveled from Islamic lands into Europe. As the glass industry became profitable, laws were passed forbidding European export of the raw

silica and ash used to make glass. Venice became a major trade center and a hub of activity, allowing residents, including glassmakers, access to many different cultures, social customs, religions, and artistic styles thereby resulting in a unique and original environment. The developing materialistic culture in Venice meant a greater preoccupation with luxury objects. The absence of a formal court left the nobility and newly rich searching for a way to stand out among their peers. They redirected their spending habits toward the consumption of luxury goods that average citizens could not afford.

#### The Emerging Glass Industry in Venice

In order to preserve this strong economic and cultural climate, the Venetian government began to regulate several industries in Venice, including glassmaking. Despite the stricter guidelines that they were forced to operate within, glassmakers continued to experiment with form, surface decoration and manufacturing technology until they derived the recipe glassmakers all over the world continue to use today. With the Invention of Cristallo, an optically clear glass, they began making glass objects that were in demand all over the world.

One of the first regulations enacted in an attempt to organize the glassmakers was the forced move for all glass workers from under the Rialto Bridge, where they had previously operated, to the island of Murano. It is widely believed that the decision was one based on fire safety for Venetian citizens. However the decision came simultaneously with the emerging economic industries has not gone unnoticed. The decision to consolidate industries by geographic location was not isolated to the glass industry.

In 1224 the oldest devotional society or (scuola) was comprised of workers from many different trades that met for religious practice. They also extended good will and charity to the Venetian community. These workers soon joined together to form trade guilds (arte), called the scuola dell'arte. The purpose of these groups was to ensure that each craft had controls and to attain proper allegiance to the Venetian government. Members of the glass guild had to prove that they were able to work glass and pay a membership fee. This meant that furnace owners paid the government a tax to operate. Unlike guilds elsewhere in Italy, Venetian guilds had no power, and no representation in government until 1275, when the first Atti dei Podesta, or guild appointed representative to the council, took his seat. This person typically had great experience in state affairs and politics. He ensured that the guilds were subordinate to the state, and was in charge of making statutes that created a greater uniformity among all guilds [7].

After the organization of the Scuola dell' arte, the Venetian government enacted a series of capitolare de Fiolariis (Regulations for glassmakers). Some of these laws were beneficial to the industry while others created problems. The capitolare dictated product regulations, who could work in the glass factories, the hours of operation, items to be manufactured and how glassworkers could spend their vacation time. The first capitulary stated that glassmakers were required to stamp glass bottles with a state approved stamp. This stamp guaranteed consumers were getting a product that met government issued standardized size and weight requirements, it also required that all oil and wine be sold in locally produced glassware with blue rims, and the official stamp. This stimulated a local necessity for utilitarian objects made by the glass industry. There were some articles in

the capitolare which demanded that furnaces run 24 hours a day and outlined that hot furnace workers worked 8 hour shifts, and torch workers worked 12 hour shifts. The capitolare specified that the workers work continuously for 7 months out of the year with a 5 month break to re-build equipment and sell off inventory. Workers were given three meals a day and were expected to be loyal to their employer, not leaving them until the annual break. Articles 11 to 14 of the document stated, among other things, that it was forbidden to lure a worker away from another furnace during the working season. During times when the workforce became limited, Articles 6 to 8 permitted foreigners to enter the Venetian factories provided they pay an additional fee to the Doge while this fee made it difficult for foreigners to work in Venice, records show that it was not impossible and there is documentation of foreign workers [7].

For the workers, the articles that dictated the length of the break in the season was established to allow them time to rest and change factories if they chose. For furnace owners, it allowed the furnaces to be re-built, and most importantly it allowed the products to be sold and limited the supply of product. It stated that workers must take an oath that they would not work outside Venice. The penalty for those who did was a 10 denari grosi fine and banishment from the guild. Still many of the workers took jobs outside Venice during the break. The migration of Venetian workers (and glass technology) became a major complaint between the glass workers, the guild and the Venetian state. A declining workforce as a result of banishment from the guild for working outside Venice during the break, left furnace owners asking for a change in the

regulation. In 1315 the capitolare was changed to include a heavier fine and long prison sentence (up to 6 months) for the infraction.

While one could assume that the capitolare were meant to protect the industry and maintain a workforce, it was also an attempt to keep glass technology on the island and away from competing European glass houses. In 1277, a treaty signed between Doge Jocopo Contarini and the prince of Antioch, Bohemond VII, stated that Venetian merchants who intended to trade broken bits of glass called cullet or verre brize must pay a tax on it. The treaty is significant in that it assumes a dependence on the trade of cullet. Historians also believe that this treaty may indicate that the Venetians had been trading without taxes for some time, and that the volume warranted taxation [12].

For the past 500 years however glass produced on the small island of Murano has been the most prized and its glass-working tradition has become somewhat legendary. Today artists in glass employ “Traditional Venetian Techniques” and will go to great lengths to study with “Venetian Masters”. For us to understand how such a small community of glassworkers on Murano became leaders of glass working and manufacturing we must look at the development of the material, how the Venetians acquired glass traditions and technology, and how they protected the information from spreading to potential competitors in the marketplace.

## 2.2 Glass Working Processes

Glass Working can be broadly classified into three different types on the basis of the temperature at which that are being worked on.

### 2.2.1 Cold Working

For the Egyptians, glass was ‘Stone of the kind of material that flows’ [15]. In its hot and liquid states, it is a flexible material that can be molded or twisted into canes. Before the discovery of glass blowing about 50 BC, ingenious methods were used to form this remarkable substance.

Cold-working describes a great variety of subtractive processes in which glass is shaped using a variety of abrasives at room temperature, it can also be known as any process that changes glass in its natural or room temperature state. These procedures could include cutting, drilling, engraving, grinding, sanding, polishing or sandblasting.

Cold working can either be a single step by itself or it can also be a preparation or finishing stages of glass fusing process to deliver a product. Preparation or finishing stage involves shaping and cleaning up pieces either before firing or after they have been fused inside the kiln. Getting the pieces of glass to the shape or design is preferred before firing. This could involve cutting figures into the glass to obtain a certain pattern, or engraving a pattern into a dichroic piece. Some of the finishing processes might include grinding sharp edges or misshaped pieces.

Cold working methods:

- 1) Cutting: Cutting glass can be accomplished either by hand or by using a glass saw. Patterns can be drawn or a design can be cut freehand. Scoring glass with a carbide bit or a diamond bit helps in cutting the glass up to a certain thickness and after that a saw must be used. Thick glass cutting can be done using a water cooled ring saw, band saw, wire saw, bridge saw or tile saw. All



these saws have in common, is the use of a diamond coated blade to cut glass with the blade running through water. Tile saws with a special cutting blade are usually used for cutting very thick pieces. Ring and band saws are generally used for shaping and cutting intricate shapes or patterns.

- 2) Drilling: Drilling in glass is performed using a diamond drill bit, with the glass inserted in a water bath which acts as a coolant. Special diamond coated drill bits are available from 1/8" to well over 2". Drilling can be carried out with a hand held electrical drill or in an electric pedestal drill. Drilling can be done before or after glass fusing.
- 3) Engraving: The technique glass engraving also known as copper wheel engraving is an ancient technique. The art form has its origins in Mesopotamia, dating back to 1000 BC, during the Baroque era. Cameo engraving was mainly used by the Romans with only little interest in Intaglio and point engraving. This art almost disappeared with the decline of Rome [16].

At the end of the 16<sup>th</sup> century, Prague witnessed the rebirth of wheel engraving on glass. This is a traditional technique which requires a belt driven lathe carrying a spindle mounted wheel made of copper. Different varieties of wheels varying in width, diameter and profile are used to make different types of cuts in glass. A slurry of carborundum grit, oil and paraffin is applied to the turning wheel, while the glass is held against the wheel to make the cut. Coarse grit is used for rapid and large scale cutting while fine grit is used for

more polished and delicate work. Most artists now use stone and diamond impregnated wheels, which replaces the slurry with water making it easy. Historically lathes were driven by foot and later with emerging technologies, were driven by motors [16] [17].

Engraving is done after fusing to add details to the piece or for signing a piece of art. The piece can also be engraved before fusing only if it is done on a dichroic glass, as clear glass will naturally lose the image during the fusing process. There are a large number of small engraving machines available as well as high-powered units such as dermel or jewelers flex shaft.

- 4) Grinding: Grinding can be done either before fusing, to obtain a certain shape; or after fusing, to clean up any undesired edges. A wide variety of grinding equipment is available which includes water cooled table top grinders, wet belt sanders and flat lap grinders. With a wide range consisting different grades, of grinding bits, belts and grinding discs are available for these machines. These machines are excellent for shaping and edge finishing. Hand held Power grinders are another type of tool used for grinding, being hand held they are quite portable but require a constant source of running water and can be very messy to use.
- 5) Sanding: One of the simplest methods of sanding is the use of different grades of diamond hand pads, generally used for sanding sharp edges of glass. The abrasive surface can be used either wet or dry for working on the glass. Wet and dry hand pads are very handy for cleaning up faulty edges, baked on kiln

wash, as well as edge spikes on frit castings. Sanding using various pads and compounds can be used to produce polished surfaces.

6) Polishing: Use of polishing creams, grinder and polishing pads are common practice to remove any residue from finished pieces. Glass can also be fire polished in a kiln which will be covered in warm glass working as this is done in the kiln at higher temperature.

7) Sandblasting: Sand blasting is like spray painting but using abrasive sand instead of paint. Sandblasting is generally used to impart a finish, design or pattern to fused objects. A resist material is applied to the glass and the desired pattern or design is cut into the resist removing the pattern, to expose the part of the glass to be blasted. Generally done after fusing to ensure the finish, design or pattern is retained. Sometimes used to obtain a pattern on a dichroic glass before fusing, when being fused to a clear glass so as to make sure the pattern is visible, but should not be heated to higher temperatures.

Different sandblasting methods used are

- Surface Etching: it is a single uniform blasting that produces a uniform texture.
- Shadow etching: similar to surface etching, instead of a single uniform etch, the design is shadowed with some parts sandblasted more than others so the texture can vary from, slightly etched to fully etched.
- Multi-stage (deep craving): it is done at higher pressures than regular sandblasting and usually requires especially thick rubber resists. This is

done by removing the stencil in stages. One part is removed and sand blasted, then more resist is removed and sandblasted, then still more removed and sandblasted. The early removed stencil parts will be more deeply etched than later removed.

Sand blasting can also be used for:

- Removing devitrification (discussed in detail below) is easily done by just sandblasting off all the devitrification and then firing the glass in the kiln to fire polish the sandblasted surface.
- Matte finish can be applied to produce an attractive satin finish to contrast with the usual full polish of glass
- Drilling holes can be done very quickly by holding the sandblasting nozzle just a few inches away from the glass. This is handy when one doesn't have the right size drill bit for the hole.
- Removing layers of glass to expose lower layers allows one to experiment with some very unusual and attractive techniques.

### 2.2.2 Warm glass working

There are many different warm working production methods and recipes for making glass, resulting in as many different types of glass, with different uses. Glass has different formulas which are adjusted based on the purpose that the glass is being used. Soda-lime, the substrate of choice, is most often used for warm glass working due to its low softening temperature. Soda-lime glass is obtained by adding soda and other metals

to silica. There are many sub classes of soda-lime glass which are formed into different shapes or types, for different uses [15].

Kiln working is often referred to as warm glass [15]. Much of the kiln working process generally dealing with soda-lime glass requires temperatures between 1200° and 1600° F. The process of heating glass in the kiln to transform the glass to a thermoformed glass is called firing.

The kiln firing process starts with the determination of a graduated schedule, also known as the firing schedule. In this process each step indicates an important segment of the heating and cooling cycle of the glass forming process. Each step is defined by the rate of temperature increase and the hold time required for the glass to attain the desired temperature. The firing schedule is developed taking into account different stages and its effects on glass, the important transformations their impact and the effects as discussed below.

Quartz Inversion: Refers to the temperature range that should be considered when one is determining a firing schedule and actually firing the kiln. This process can be explained as the change of the physical molecular composition of bricks, kiln shelves, kiln furniture or everything in the kiln except for the glass, changing from alpha quartz to beta quartz during the heating cycle and vice versa for the cooling cycle. This change in the physical makeup of quartz causes the heating and cooling process in the kiln to slow down, when temperatures reach from 1000° to 1150° and vice versa [15].

The temperature effects caused by the quartz inversion cannot be avoided but can be used to one's advantage, by holding the kiln at the quartz inversion range for a heat

soak, allowing the transformation to occur naturally, which makes the kiln raise in temperature beyond 1150 faster. Quickly raising the temperature of the contents of the kiln to the fusing temperature helps avoid devitrification, an unsightly, unstable contamination that develops on the surface of the glass.

Devitrification: Is the process of contamination of the glass by the development of crystals that are not glass on the glass surface. The chemical composition of devitrite crystals is not glass, and they have a very different coefficient of expansion that is not compatible with that of glass. Devitrification crystals are contaminants within the glass that migrate to the glass surface or grow from deposits upon the glass surface. These crystals are not attractive and are not compatible with the glass surface which turns cloudy. When the kiln is held for a lengthy amount of time in a specific temperature range (1050 – 1100°F for soda-lime glass) crystals grow. Devitrification problems are not limited to annealing temperatures, but may occur as the glass fires and will not burn away when the kiln is taken to the full fusing or casting temperature, it may also appear when a kiln is cooled down too slowly during the devitrification range.

The undesirable surface caused by devitrification can be sandblasted to remove the devitrification. This dull surface will be redeemed only if the sandblasted area is thoroughly cleaned and dried before glass is re-fired to obtain its original shiny and clear surface. This is done by heating glass past its softening point to its soft surface melt temperature to help attain improved surface quality.

Annealing: A generic term annealing refers to the cooling of glass. Scientifically speaking, annealing refers to the process by which internal stress is removed from the

glass [15]. Glass always has strain within; if not strain were present, the substance we refer to would not be “glass” [15]. When glass is heated it expands, and it contracts as it cools. These states cause stresses within glass, especially between the interior and the glass surface. Unlike most solids the atoms of glass are not arranged in a perfect crystalline lattice, they form an imperfect network arrangement. To relieve these stresses and the internal strain which can lead to excess strain or breakage at room temperatures, it is necessary to cool glass in a very controlled manner, through a predetermined temperature gradient. This controlled process is called annealing [18]. Although glass never melts when subjected to low fire maturation temperatures, the molecules within the glass that are heated past its annealing point temperature will have relaxed enough that they will require the reorganization of the crystals, which will happen during the annealing process [15].

The first phase of the annealing process is the anneal soak, which will help to equalize the temperature throughout the glass and relieve 95% of the stress that is present. The duration of the soak time at this temperature depends on both the thickness of the glass, shape of the glass how it is setup in the kiln, and sometimes on the type of kiln being used. Kilns with firing elements on the walls tend to evenly spread the heat compared to the kiln with the element on its the door, which heats up the portion closer to the door much quickly compared to the further portion.

The second phase is gradually cooling the piece through the rest of the annealing range once the temperature within the glass body is uniform throughout. The rate of the temperature change plays a vital role as it is very important to keep the temperature

difference throughout the body of the glass within 5°F from 900° - 700°F this removes the remaining 5% of the stress allowing glass molecules to ease back into their amorphous cell structure. Conforming to the temperature difference, annealing time drastically varies based on the thickness of the glass, ranging anywhere from an hour to several weeks.

**Glass Transition Range:** Glass is an amorphous material. Its molecules are not arranged in a regular, specific pattern, like those of a crystalline material, but are random imperfect network arrangement. Because of its amorphous molecular configuration, glass reacts to heat differently than other materials. Glass goes through a very gradual transformation from a material that behaves like a solid to a material that behaves like a liquid, unlike other metals which change from solid to liquid at a specific temperature. It is this unique characteristic of glass that allows it to be blown or to be worked in the myriad ways we call kilnforming.

As glass exhibits the molecular structure of a stiff liquid even in its solid form, glass at room temperature is sometimes referred to as super cooled liquid. As it is heated, glass gradually begins to behave more and more like a liquid until at temperatures above 2000°F, it will flow easily with a consistency similar to honey [18]. Addition of heat starts transforming the glass into different states which depend on the viscosity of the glass. Viscosity of the glass increases as the glass cools, while it decreases as the glass becomes hotter and hotter. Various layers of glass composition pass through the glass transition range at various times, which is due to the internal temperature gradient

Strain point, softening point, and annealing point are the terms of specific temperatures that indicate specific viscosities within a kiln firing schedule [15]. These



names of the zones are a description of the viscosity of glass and also designate specific temperature points that the glass passes through. The reason for choosing a range of temperatures rather than a specific point is due to the transformation happening within a range of temperature rather than at an exact predictable temperature.

**Softening point:** The softening point indicates the temperature at which glass begins to transform from what we call a solid state to a liquid state [15]. It is also defined as the temperature at which glass deforms under its own weight. This softening point temperature plays a vital role in the outcome of the project as firing the kiln hotter than the softening point will result in increase in the dimensionality of the glass composition. Considering the effect of gravity glass will actually begin to deform at temperatures slightly lower than the softening point. This actually helps when slumping glass in molds at temperatures very close to the softening point and holding for long periods of time which facilitates the glass's ability to pick up the mold features without imprinting the mold texture onto the surface of the glass that comes in contact with the mold.

**Annealing point:** The annealing point is described by glass engineers as the temperature that allows the cooling glass to be relieved of the largest percentage of internal strain possible within a fifteen minute timeframe [15]. The annealing range is the temperature range in which the cooling glass that has already been formed in the kiln is still viscous enough to allow for the formation of the molecular network within the glass, while still freeing the glass of some strain. The glass is in a contraction mode while passing through the annealing point until the strain point is reached, and this is the range during which glass loses the majority of its strain. This is also called the low anneal. This temperature

range can easily be defined as the temperature that is cooler than the annealing temperature but warmer than the strain point. Additional strain cannot be removed from the glass, once the glass reaches the strain point.

Strain point: The strain point helps to determine the annealing schedule for the glass. The amorphous cell structure that makes up the glass network formation continues to move at a very slow rate, until the glass cools down below the strain point. If the glass is cooled too quickly, it results in a frozen formation of molecules out of formation appearing as a solid. The result of an irregular formation of the atoms filled with stress, rather than a regular fashioned formation with relieved stress. A glass with much internal stress always tends to break or crack over time.

Strain point is also a point of consideration in firing the glass, as rapid heating around this temperature would cause thermal shock, so care must be taken when heating or cooling around this temperature.

### Kiln Forming Glass

Having discussed all the different points of significance has and the role of each, now we will briefly discuss two of the glass forming processes involving the use of a kiln.

Fusing: Fusing is the process of using kiln to join together pieces of glass. If heat is applied to glass it softens. If heated further, the glass will become more fluid and flow together. When two compatible pieces of glass are heated and then cooled properly, the resulting fused glass piece will be solid and unbroken. The glass forming technique that requires the awareness of when the glass begins to soften is called tack fusing. Fusing

allows for a topography of the glass surface happens at temperatures that are just a little hotter than the softening point temperature. Scientifically when glass fuses the atomic ions from the different glass surfaces begin to bond or weld together.

**Slumping:** Slumping glass refers to the transformation of pre-formed glass by heat and gravity. Transformation of glass during the slump firing as the soft glass slumps or sags into a form or over a form. It can take place through an object, as in a drop through mold or between objects within reason. The actual temperature at which glass starts to slump during a slump firing is lower than the fusing temperature, as glass starts to slump slightly once the temperature in the kiln is increased beyond the annealing point.

### 2.2.3 Hot glass working

Hot shaping, glass blowing or lamp working is working of glass with flame and heat, after collecting molten glass onto a pipe or a rod.

Glass blowing is a glass forming technique which was invented by the Phoenicians around 50 BC somewhere along the syro-palestinian coast. The earliest evidence of glass blowing comes from a collection of waste from a glass shop, including fragments of glass tubes, glass rods and tiny blown bottles, which were dumped in a mikvah, a ritual bath in the jewish quarter of the old city of Jerusalem, dated from 37 to 4 BC. Some of the glass tubes recovered are fire-closed at one end and are partially inflated by blowing through the open end while still hot to form a small bottle, thus they are considered as a rudimentary form of blowpipe. Hence, tube blowing not only represents the initial attempts of experimentation by glassworkers at blowing glass, it is also a revolutionary step that induced a change in conception and a deep understanding of glass

## Glass blowing tools

Tools play a vital role in the act of glass blowing: dealing with a material that is rapidly changing heat from 2300° to 900°. As such, the tools are able to work at these varying temperatures. These tools also determine the quality of the product being produced. While many glass blowers have a variety of custom tools, there are some tools which are fundamental and used during most blowing processes. Explained below are the basic tools and their working procedure in the order of their use.

**Punty Rod:** The term punty rod simply means a solid steel rod, and is mostly used when there is no need to introduce air into a glob of glass. The word punty in the studio refers to the following;

**Punty:** A solid steel rod, and always referred to as punty rod or punty rod irons

**Punty:** A small piece of glass on the end of a punty rod used to remove a piece from the blowpipe.

**Punty:** The act of removing a piece from the blowpipe using a punty on a punty rod.

**Jacks:** The primary tool of the glassblower is a good pair of jacks. Jacks have three working surfaces: the blade edge, the blade side, and the hell. Blades are usually coated with wax which acts as a lubricant and prevents metal to glass contact, which can damage the surface of the glass. There is a certain way that these are supposed to be used and placed on the bench as jacks can, when used incorrectly burn the hands of the user, as a result of recent contact with glass. Not just the heat that is dangerous, it is the hot wax coating that continues to fry the epidermis long after dropping the tool. The jacks occupy

the first position on the tool bench closest to the Gaffer (or glassblower in charge. The blades hang over the edge of the bench pointed towards the back.



Figure 1 Glass blowers bench with tools.

Tweezers: Tweezers are used for grabbing the glass, it is most important that they are always clean and free of wax, as even a little amount of wax would render them slippery on the glass and therefore useless. Glassblowing tweezers are different from the regular tweezers as they are stiff, which is a benefit when we need forcefully grab a piece of glass or when we need the spring of the tweezers to move the glass. As with Jacks, tweezers must be handled properly, they need to be gripped firmly between the thumbs

and forefinger, this position allows to apply all the force of the palm to overcome the inherent stiffness of the tool.

Shears: Glass shears also known as snips are specialized scissors for cutting glass. These shears come in several styles with varying blade lengths and curvatures. If the shears don't cut, it can be either because of improper placement on the bench which has cause wax to accumulate on the blades, or from improper use in trying to cut cold (below 900 degree F) glass causing the blades to dull., Wax accumulation on the tools can be overcome by rubbing them on a molten bit of glass which burns off the wax, but if it is due to the glass being cold it is better to reheat the glass Since they are made of carbon steel they always need to be dry to prevent them from rusting.

Diamond Shears: Diamond shears are specialized shears that are used to cut hot bits of glass and hold pipes. This tool has specific areas which are dedicated to different tasks. The outer curve is not a shear at all, but it is a curved pair of pliers designed to hold a pipe or punty rod, this portion of the diamonds are not the most efficient part of the diamonds for actually cutting the glass. Conversely trying to hold a pipe or punty rod with the part of the diamond shear designed to cut the glass, will end up holding the pipe but will damage the diamond shears cutting edges. Unlike the straight shears, diamond shears are less affected by wax because of the nature of their cutting action, but they should still be kept wax free. Diamonds are damaged easily by using them to cut cold glass. The main problem with diamond shears is they tend to cool the glass as they cut. If we can cut fast, less than a second, the glass will not have time to cool down, and the diamond shears will do their job completely.

Parchoffi: Parchoffi are similar to jacks but the blades are wood, they are specialized tool used mainly for shaping the inside of bowls. Parchoffi are used to shape the outside of pieces, as they tend to scratch the glass less than the metal jacks.

Soffietta: This is a specialty item called by its Italian name soffietta, is a metal cone that helps to inflate a piece that has been removed from the blow pipe, by blowing through the mouth of the object. This is a great tool for thinning hot thick necks on the punty.

Steam Cone: It is a conical piece of wet wood which is similar to a puffer, except for the use of steam instead of lung power, which require them to be wet, and used wet all the time. When the cone is inserted into the lip of a piece of hot glass, the water turns to steam and inflates the piece.

Newspaper pad: Newspaper pad can be made by folding newspaper into a pad, at least the size of a human hand. It is then soaked in water for fifteen minutes, for the damp newsprint paper to protect the bare hand from 2000° glob of glass less than half an inch away from hand. This is due to the leidenfrost effect (explained as the phenomenon in which a liquid, in near contact with a mass significantly hotter than the liquids boiling point produces an insulating vapor layer which keeps that liquid from boiling rapidly). It is common for glassblowers to protect them from the colored inks in the newspaper by removing the pages with colored ink, or wax coatings using just the black and white newsprint. Though it is the most inexpensive tool there is a protocol for its correct use. First it should steam not smoke. If the paper is smoking, it indicates that the paper is burning and it is time to wet the paper. Second, the newspaper should be kept out of the block water, as newspaper ash is an insidious contaminant, should be kept clear of other

tools. Finally, the pad should not be excessively wet, excessive moisture can result in standing pools of water, which can check or crack the glass, along with the possibility of running into the hand in burning rivulets.

**Paddle:** The paddle is a simple piece of pre-charred fruit wood used to shape the glass and protect the artist, which can be used either wet or dry. Wet paddles are typically used to shape the glass as the water forms a steam barrier between the wood and the hot glass, which lubricates the movement. Dry paddles depend on the layer of charred wood mostly very slick carbon which acts as lubricant.

**Blocks:** Blocks are specially shaped wet wooden molds that are used to shape glass immediately after it has been gathered from the furnace. They come in various sizes and are the famous tools of many beginners as they mold the glass into the proper shape, yet they are only good for a few seconds when glass is between very specific temperatures. Blocks are frequently used immediately after the glass has been gathered. Like any other wet wooden tools they need to be kept wet at all times, as allowing it to dry would cause them to crack and deem worthless.

**Blowpipe:** despite the fact of the practice of glass making getting the name from the blowpipe, it is relatively a new tool in the glass studio. Lovely objects of glass were being made much before the metal workers were able to make tubes out of steel. The fact that this activity is now called glassblowing is testimony to the tremendous impact that the blowpipe has had. In simpler terms a blowpipe is an iron or steel tube that can be used to introduce air into the center of a glob of glass. Although they are homogeneous modern blow pipes are actually two pieces of metal, the shaft and the head. The shaft is a simple



tube of relatively thin-walled metal, attached to the end of the shaft is a head, usually an 8 inch piece of steel sporting a hole less than ¼ inch in diameter, which is designed to withstand the heat, the shaft is not, which indicates that heating the blowpipe past the head is a good way of damaging the blowpipe.

**Hand Torch:** A hand torch is a small canister of propane or MAPP gas with a small torch head connected directly to it. It is very hot compared to glass standards and is very inexpensive, and it is one of the few glass working tools that we can buy from any local hardware stores, The hand torch is, great for heating punties, drawing on the glass with colored canes, and spot heating specific areas of a piece.

**Plumbers Torch:** Also called a hand torch, it is a large torch head connected to a standing canister or a gas supply line, through a flexible hose. It shoots flame from several inches to several feet and is especially good for working larger pieces. It is generally used to preheat a portion of the glass prior to going into the glory hole or to build up heat to get a specific area to blow out, or to heat a specific portion so that you introduce temperature differential to thermal shock it. A plumber's torch produces a lot of noise and heat which prevents it from being used in many studios.

**Oxygen Torch:** It is a small torch similar to the hand torch in flame size, with an additional oxygen supply mixed with the gas, making it very hot and good for polishing out punty marks, popping bubbles and super heating spots of a large piece. There is always a caution when working with this torch as it is too hot to damage the glass by yellowing it.

Glass blowing involves three furnaces and a number of tools (discussed in detail after this section) and equipment used. The first is just referred to as a furnace, inside which is a pot, also called a crucible, which is under the process of charging. The furnace is filled with large amounts of batch that melts at temperatures higher than 2000 degrees Fahrenheit. While glassblowing can be done individually, it is so challenging that it is often done by a team, led by a gaffer (lead glassblower). The gaffer uses a blowpipe which is usually made of iron or steel and measures about 4 feet long to collect a gob of molten glass on one end from the hot furnace, after the glass is secured on one end the other end of the pipe is cooled off in a barrel of water.

Once the gaffer is ready, he'll blow through the tube and start to create a growing bubble in the glass. More layers can be gathered and added with a gathering iron, or by dipping the glass attached to the blowpipe back in the furnace.

Glass blowers often use a large flat surface called a marver generally made of iron or steel, to roll and shape the glass. During the blowing process, the partially blown glass is turned around and a bit of glass is often added with the use of a smaller metal punty rod. The punty is also used to add colorants and it can also be laminated on with heat or adhesive, threads and wraps can also be laid in decorative patterns as glass is turned, or shards can be melted in. While pulling and shaping, a metal punty rod is attached to the base of the blown glass to hold it while the mouth end is being shaped. The punty mark left on the bottom of the object is usually ground or polished away later, and sometimes also used as a mark distinguish between machine manufactured and hand blown art.

While the glass is being blown, it often cools to the point where it is unworkable, which is where the reheating furnace “glory hole” comes in. The glory hole is the second furnace in the modern three-furnace setup. It is commonly a round, insulated cylinder. Partially formed glass can be held suspended from the end of the rotating blowpipe, which rests on metal stands called yokes, until it is hot enough to continue working. Although a piece of glass may appear done when the last gob of glass has been melted in, there is still a crucial step that needs to take place.

Once a piece of glass is complete, to help it cool properly, an annealing furnace is used. As the glass starts to cool it may naturally start to crystallize, but that’s not a good thing, as that means the glass has lost the properties that made it so special to begin with. The goal is for the glass to cool and retain its scattered but rigid molecular structure. As this happens it contracts and loses more and more of its viscosity until it becomes solid glass. To ease the newly blown piece of glass through this process, an annealing furnace is used to control the rate of cooling eliminating the chance of thermal shock and the piece becoming unstable. A pyrometer carefully measures the temperature during the annealing process and computer takes these values as input, monitors the cooling schedule, while the glass is brought below the softening point and carefully cooled over an extended period of time. This helps get rid of any internal strain or tension in the blown glass, making it less likely to break down the line.

Glass blowing can be classified into different types [19]:

- 1) Hot shaping: Small objects could be formed by heating small blobs of glass, or sections from glass canes, on a flat surface until the top surface is rounded

naturally. The base could be finished by grinding. Rings and bangles were made with the aid of a central former, such as a wooden or metal rod, which could be used to perforate and then enlarge a hole in a blob of glass. Softened glass was wound around the wooden or metal rod to join the ends.

- 2) Core forming: core forming was an early method of shaping glass vessels and objects, for example making beads in the Bronze age and in the iron age. Softened glass was shaped around a core, which was later broken-up and removed to leave the inside hollow.
- 3) Free Blowing: The regular glass blowing process explained above.
- 4) Mold blowing: vessels and objects could be blown into a reusable mold, transferring the shape and decoration of the mold to the glass partisan when it was inflated within the mold. Molds are known from the roman period, most often made of clay, but sometimes made of stone, wood or metal. Several molds for several pieces were used for vessel forms that could not be removed intact from a single-piece mold, use of multi piece mold resulted in an object with seam lines where the mold pieces joined. The vessel could be finished in a similar way to free blown vessels. In later periods more complex and robust molds were developed, made from iron or copper alloys and hinged. Initially the molds were for shaping the base of post-medieval bottles, but in the 19<sup>th</sup> century glassmakers developed a series of multi-piece molds, used for mass production, which shaped the complete bottle. However, the glass was still inflated by a

glassworker blowing down a blowing iron, the mechanized production of bottles, using compressed air, did not become widely adopted until the 20<sup>th</sup> century.

- 5) Optic blowing: A gather of glass could be partially inflated in a mold to transfer a pattern onto the gather. Stone molds are known but other materials, such as metal, would be suitable. The vessel was completed by free blowing, so that the final pattern had a flowing, distorted appearance. This technique was used in the roman period, and was also used to produce fluting and other designs on early medieval and post-medieval vessels.

Lamp working also known as flameworking or torchworking is a type of glass working that uses a gas fueled torch to melt rods and tubes of clear and colored glass. The reason for calling it lamp working is the use of oil-fueled lamps in the olden times. Lampworking differs from glass blowing in that glassblowing used a blowpipe to inflate a glass blob known as a gob or gather, whereas lampworking manipulates glass either by the use of tools, gravity, or by blowing directly into the end of a glass tube. Early lampworking was done in the flame of an oil lamp, with the artist blowing air into the flame through a pipe. Most artists today use torches that burn either propane or natural gas, or in some countries butane, for the fuel gas, with either air or pure oxygen as the oxidizer.

## Chapter 3

### Present Technologies & Limitations

The proposed research is to review many of the existing technologies, overcome some of the disadvantages in present technologies to develop a simple process for manufacturing micro channels in microscope glass slides. Briefly explained below are several existing technologies being used for microchannel manufacturing on glass slides, and their limitations.

#### 3.1 Powder Blasting

Erosion is the phenomenon that can be observed in a lot of hydrodynamic applications. It generates serious damage to objects in contact with eroding particles present in air or liquid flux. There were some studies performed on the erosion mechanisms of brittle materials like ceramics and glass by the impact of hard and sharp eroding particles. They were found to be very similar to results from quasi-static or scratching Vickers-indentation measurement. When a particle reaches the target surface having a certain velocity and having energy greater than the fracture threshold, it induces a local material deformation in the form of cracks resulting in the removal of some parts from the target material. This process of removing material from a target by bombarding it with particles is called powder blasting [20].

Powder blasting technique allows obtaining complex and controlled shapes of the eroded structure. The erosion rate of the process typically ranges in several hundreds of micrometers per minute, which is much higher than what is obtained by standard wet or dry etching process, making this process very appropriate for fast and complex three-

dimensional micro structuring. The powder blasting method is used to fabricate structures with a 3D topography in glass using elastomeric masks. The relation between the mask opening width and the erosion depth is exploited to fabricate microstructures with varying depth in a single micropatterning step [2].

Powder blasting technology has a series of steps which result in a 3-d microchannel in glass. The first step is the preparation of a mask made of photoresist, photopolymers or metals, which is basically the negative of the microchannel required in the glass. The mask machining process can be anything from a CNC machining to Laser machining to vapor deposition, depending on the material being used for the mask [21].

Photosensitive materials and elastomers are generally used as masks due to their good resistance to powder blasting. Due to their photosensitive nature, photolithography can be used to fabricate the mask directly on the glass substrate, which makes it easy to obtain different features with high resolution. Once the glass is ready with the photoresist it is placed in front of the powder blasting nozzle, at a certain distance. The piece is fixed and the nozzle is allowed to move in different directions in a zigzag path limiting the scanning area to that of the glass slide, once it starts operating, resulting in micro channels in glass [21].

There are different parameters based on which the resultant features will depend on, some of them are [20]

**Powder velocity:** Erosion rate is defined as the ratio of the removed weight to the incoming particle weight, which is strongly dependent on the indenting particle velocity, which makes this parameter an essential one for the characterization of the powder

blasting process. It is also observed that increase in the kinetic energy with pressure tends to saturate at higher pressures.

Geometrical effects on etching rate: It has been observed that etching rate is mostly effected by the geometry as smaller openings due to the difficulty of the particles to enter and leave the mask opening, compared to the particle size which can result in possible particle accumulation inside the structure and eventually decreasing the etch rate. This problem contributes to the improper geometry of the resulting feature on glass.

Angle of the nozzle: Angle of nozzle impacts the walls, as the substrate near the edge of the mask is less exposed to erosion as a consequence of the finite size of the impacting powder particles, resulting in non-uniform erosion rates.

The physics of powder blasting produces a taper angle on through holes, which leaves the entry hole opening to be larger than the exit hole or the bottom of the hole with a different dimension from the hole opening. This is one of the limitation which restricts this process from producing a precise dimensioned micro-channel. The surface finish resulted from this process is rougher compared to most of the processes. These are few major limitation of this process given other process an advantage, as they can create higher aspect ratio straight side wall channels.

### 3.2 Laser Machining

Laser (Light amplification by stimulated emission of radiation) is a device that emits light through optical amplification, resulting in a high degree of spatial and temporal coherence.



Laser micromachining of glass is considered due to its non-mechanical force contact machining feature, significant process flexibility and direct writing by CAD/CAM software. It is also considered a flexible tool for micromachining with simple and inexpensive operations cost except for the capital investment. A variety of lasers can be used for micromachining, from nanosecond lasers to ultrafast lasers or from IR lasers to UV lasers, depending on the material to be fabricated and desired applications. Femtosecond, UV and CO<sub>2</sub> laser are most commonly used to fabricate silicon structures, as silicon is known as a difficult material to fabricate [22] [3].

Femtosecond laser source has been extensively used for laser assisted micro fabrication due to its versatility, namely with respect to direct 3D-writing of structures in solid materials. In fact, the nonlinear multi-photon absorption process arising from the irradiation by intense femtosecond pulses ensures that the effective interaction zone inside the bulk of transparent medium is located in a confined neighborhood of the focus. Such unique property of the femtosecond pulses enable one to precisely micromachine inside various transparent materials. In particular 3D microfluidic structures in a single glass chip have been demonstrated by femtosecond laser direct writing for biochemical analysis. Normally a bulk transparent sample is first irradiated by femtosecond laser pulses. After irradiation, microchannels are produced by selective chemical etching of the irradiated sample with diluted hydrofluoric acid. Alternatively, microchannels have also been fabricated by femtosecond laser with a water-assisted technique developed by Lee et al, and microchannels with constant diameter could be easily achieved with their technique. However, femtosecond lasers have relatively low productivity compared with

their related costs, although they improve the quality and resolution significantly, which are very important for multi-generation bio-mimetic channels [22] [23].

The nanosecond laser micromachining of silicon is a substantial challenge since the roughness of the laser ablated surface is caused by strong thermal reaction of irradiated silicon. Explosive boiling for each pulse and overlap of the pulses are not only two main processes in nanosecond laser ablation but also two main reasons for roughness problem. Solidified molten material and splashed debris in the craters created by laser pulses initiates the surface roughness. In addition, the overlapping of the pulses aggravates the surface roughness.

Laser machining of brittle materials such as glass to create channels with smooth surface and free of micro-cracks has been a challenge as laser-induced thermal stress often lead to random micro-cracks and edge chips. Studies have been performed by researchers with selected laser sources to minimize the thermal-induced cracking issue. Experimental observations and analysis of CO<sub>2</sub> laser-induced micro-cracks in glass substrate with both transversely excited atmospheric pressure CO<sub>2</sub> and modulated continuous-wave CO<sub>2</sub> lasers have been reported in a laser marking process. The laser produced marks were formed by a combination of surface cracking and material removal. The residual surface stress following rapid laser heating was identified as the most likely cause for inducing micro-cracks.

There was a new method investigated by H Y Zheng and T Lee [22]. This process was to develop a microchannel by peeling off continuous glass strips with a CO<sub>2</sub> laser beam. This process was different from the conventional melt and blow method, where the

material is heated by the laser beam, and the molten materials are subsequently blown away by an assist gas, the glass peeling mechanism is dealt with the material expansion and contraction produced by a laser-induced localized heating-cooling cycle. This process worked but only to selective glass substrates with high thermal shock resistance. They have stated that the strip formation is only possible for the borosilicate glass but not the soda-lime glass due to its high thermal shock resistance.

Thermal Shock Resistance (TSR)

$$TSR = \frac{\sigma k}{E \alpha}$$

Where  $\sigma$  is the fracture strength,  $k$  is the thermal conductivity,  $\alpha$  is the coefficient of expansion,  $E$  is young's modulus. Due to the high alkali content, the soda-lime glass has relatively high thermal expansion coefficient and low viscosity at temperature due to the low  $Al_2O_3$  content the TSR is low, micro-cracks are not readily formed for glass of high thermal shock resistance [22].

Limitations associated with different laser systems taking into consideration soda-lime glass is a big list compared to different other glass due to its low machinable properties. Summing up all the disadvantages from all the systems, laser machining creates subsurface isolated micro-cracks, continuous cracking belts, glass stripping, melting and also creates a HAZ (Heat Affected Zone) which results in a kerfs or damaged area at the top surface of the machined object. Since it is a thermal process, laser machining can crack or break thin glass pieces. It is difficult to create blind holes or remove material with a fixed depth across a large area with a laser, since it creates an uneven etching as it progresses across the part. Coming to the cost perspective, the capital

investment for this process can be significant since the laser system must be combined with the positioning equipment (motion controller for the laser system). The tolerances of the features are dependent on the quality of the equipment.

### 3.3 Etching

It is the process of using acid or mordant to cut into unprotected parts of the surface covered by a mask. There are different types of etching processes in use to etch glass, as glass is constituted mostly of silicon.

#### 3.3.1 Wet Chemical Etching

Wet etching is a material removal process using liquid chemicals or etchants to remove materials, with the specific pattern defined by a mask. The material that is not protected by the mask is removed by the liquid chemical or etchant. Silicon etching techniques, both wet and dry, have been studied extensively as they are widely used in IC and MEMS fabrication. Glass is a much less investigated process. Glass etching is more difficult because the glass wafers are not pure silicon oxide, and other components of this material have different etch rate.

The wet etching process involves multiple chemical reactions, which results from consuming original reactants and producing new reactants. The wet etch process can be described by three basic steps. 1) Diffusion of the liquid etchant to the structure that is to be removed. 2) The reaction between the liquid etchant and the material being etched away. A reduction-oxidation reaction usually occurs. This reaction entails the oxidation of the material then dissolving

the oxidized materials. 3) Diffusion of the byproducts in the reaction from the reacted surface. Wet etching can further be classified into two types. 1) Anisotropic wet etching: Liquid etchants etch crystalline materials at different rates depending upon which crystal face is exposed to the etchant. There is a large difference in the etch rate depending on the silicon crystalline plane. In materials such as silicon, this effect results in very high anisotropy. Some of the anisotropic wet etching agents are potassium hydroxide (KOH), ethylenediamine pyrocatechol(EDP), tertamethyammonium hydroxide (TMAH), etc.

2) Isotropic wet etching: For wet isotropic etching, aqueous acidic solutions containing HF are necessary. The composition of the solution determines the final shape of the channel. Most common etchant solvents are composed of hydrofluoric acid, nitric acid and acetic acid. The etch rate is determined by the concentration of the etchant, and as the reaction takes place the material is removed laterally at a rate similar to the speed of etching downward.

Wet chemical etching is generally isotropic, even in the presence of the mask, as the liquid etchant can penetrate underneath the mask easily.

### 3.3.2 Dry Etching

Dry Etching: Removal of substrate material using plasmas or etchant gasses is dry etching. The reaction that takes place can be done utilizing high kinetic energy of particle beams, chemical reaction or a combination of both. Dry etching can be classified further into 4 types.

#### 1) Physical dry etching

Physical dry etching requires high kinetic energy (ion, electron, or photon) beams to etch off the substrate atoms. When the high energy particles knock out the atoms from the substrate surface, the material evaporates after leaving the substrate. There is no chemical reaction taking place and therefore only the material that is unmasked will be removed.

## 2) Chemical dry etching

Chemical dry etching also called vapor phase etching, does not use liquid chemicals or etchants. This process involves a chemical reaction between etchant gasses to attack the substrate. The chemical dry etching process is usually isotropic and exhibits high selectivity. Anisotropic dry etching has the ability to etch with finer resolution and higher aspect ratio than isotropic etching. Due to the directional nature of dry etching, undercutting can be avoided. Some of the ions that are used in chemical dry etching are tetrafluoromethane ( $\text{CF}_4$ ), sulfur hexafluoride ( $\text{SF}_6$ ), nitrogen trifluoride ( $\text{NF}_3$ ), chlorine gas ( $\text{Cl}_2$ ), or fluorine ( $\text{F}_2$ ).

## 3) Reactive ion etching

Reactive ion etching (RIE) uses both physical and chemical mechanisms to achieve high levels of resolution. The process is one of the most diverse and most widely used processes in industry and research. Since the process combines both physical and chemical interactions, the process is much faster. The high energy collision from the ionization helps to dislocate the etchant molecules into more reactive species. In the RIE process, cations are produced

from reactive gases which are accelerated with high energy to the substrate and chemically react with the substrate.

Disadvantages of RIE etching are that expensive equipment must be used, and the etching recipes are machine-dependent and also dependent on the etching history of the machine. The walls of the etched structures are also quite rough compared to wet chemically etched structures.

#### 4) Deep reactive ion etching (DRIE)

Reactive ion etching is a process where chemical etching is accompanied by ionic bombardment, where the ionic bombardment opens areas for reactions. This process is a bit advantageous as it prevents undercuts as side walls are not exposed, it also generates increased etch rate, but it causes structural degradation.

DRIE uses electron cyclotron resonance (ECR) source to supplement RIE system, where in microwave power at 245 GHz is coupled into ECR, and a magnetic field is used to enhance transfer of microwave energy to resonating electrons. Even though this process has advantages of getting structures and better selectivity, it has disadvantages which include the use of high plasma power, specialized handling and an expensive setup and operation with outputs being piece flow which makes it a time consuming and costly process.

### 3.4 Laser Machining Followed by Wet Chemical Etching

CO<sub>2</sub> laser which is being considered as a very attractive and efficient method for rapid prototyping of PDMS microfluidics due to its inexpensive price and laser process flexibility has a disadvantage of the machined surface of the microchannel being rough.

The roughness value usually is in a range of a few microns. Initially much attention was paid to the rapid machining technology development with regard to CO<sub>2</sub> laser parameter effect on the microchannel dimensions and laser-PDMS interaction theories rather than the surface roughness. With the progress in practical applications of microfluidics, it has been realized that the rough surface of the microchannels significantly influences the liquid medium flowing behavior, cell adhesion, protein unselective adsorption, particle aggregation, bubble generation, optical signal detection such as optical-based fluorescence microscopy, etc. Hence improving the surface smoothness of microchannels has become an important aspect.

Wet chemical etching was found to be one of the methods to make the channel smoother after they have been laser machined. The microchannel samples fabricated from CO<sub>2</sub> laser ablation are pre washed with deionized water and are dried with nitrogen in order to remove all possible particles on the channels. The samples are then immersed in a chemical etching solution such as acetone-ethanol solution, diluted HF or Hcl, which are subsequently submerged into an ultrasonic bath at room temperature for a period of time, to get a smooth surface.

The effect of concentration of the etchant solution has an impact on the surface smoothness obtained. Much research needs to be done for each design changed and each difference in the height of the channel.

### 3.5 Electro Chemical Discharge Machining

Electro chemical discharge machining (ECDM) is an emerging hybrid machining process used in precision machining of hard and brittle non-conducting materials.



Electro-chemical discharge machining (ECDM), a hybrid machining process of electric discharge machining (EDM) and electro-chemical machining (ECM) process and are mainly used for micro-machining and scribing hard and brittle non-conductive materials such as glass, ceramic, refractory bricks, quartz and composite materials. It is a complex physical-chemical system, where material from the work piece is removed by an anodic dissolution of the material and also by electrical sparks that occur between the working surfaces of the electrode tool and the electrode piece. The electrical discharges and the energy from the sparks produced assure a chain of micro explosions on the surface raising the local spot temperature to a very high value sufficient for melting and vaporizing the material, accompanied by partial chemical etching result in removal of material in micro quantities [24] [25].

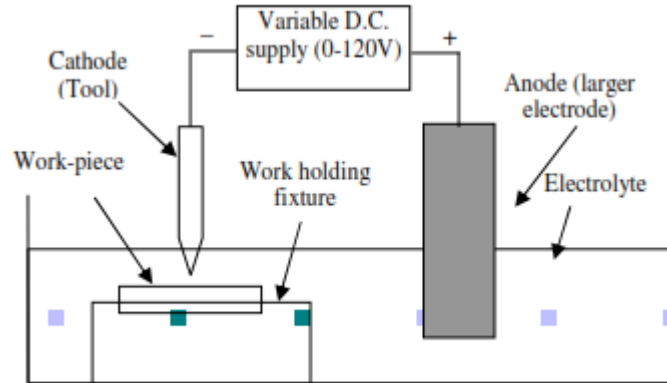


Figure 2 Schematic of electrochemical discharge machining (ECDM) setup [24].

ECDM is also defined to be a high temperature etching process which depends on the type of the electrolyte used. It is also found that the machining rate is greatly affected by the porosity of the sample being used, and the material removal occurs by attacking the grain boundary, caused by etching process. It is also reported that the craters formed in

the ECDM process are almost the same as those formed in the EDM process along with some re-cast effects, which are mainly due to the sparking action.

Much of the work in ECDM has been concentrated on glass (Pyrex and Borosilicate), which has useful properties and applications in industrial, defense, medical and electronic industries. Quartz, alumina-glass ceramic and composites are other materials that have been attempted earlier.

The main challenge is in controlling gas-film in the machining zone, its stability and dynamics, as it is reported that material removal rate and tool wear rate increase with applied DC voltage and electrolyte temperature, depending on the tool and work-piece material. Most commonly used electrolytes are NaOH, NaCl, NaNO<sub>3</sub>, KOH, HCL, H<sub>2</sub>SO<sub>4</sub>, NaF and so forth. [24].

The limitation of this process is dealing with the resulting geometry as there is always an overcut, and an error of about 50 μm in some machining, which makes it not the best process for machining microchannels for biomedical research applications.

### 3.6 Micro Ultrasonic Abrasive Machining

Micro ultrasonic machining is one of the efficient material removal processes suitable for micromachining of hard and brittle material. The principle of ultrasonic abrasive machining involves a series of steps which includes placing the workpiece on the workpiece table which vibrates at ultrasonic frequency. An abrasive slurry (mixture of abrasive particles and water/oil) is then injected on to the top of the work piece there is a rotating tool which hits the abrasive particles in the slurry which, in-turn, hit the workpiece and chip away, the material from the workpiece. Vibration helps in refreshing

the slurry to allow fresh abrasive particles to come into contact with the workpiece and to remove the debris from the tool work piece gap [26].

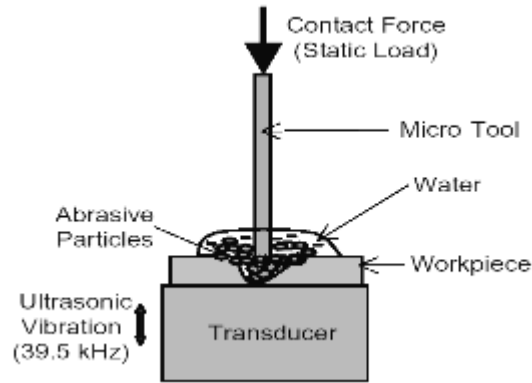


Figure 3 Principle of ultrasonic machining [26].

The water/oil mixed with the abrasive particles plays a vital role by acting as a lubricating agent as well as coolant in reducing the fractional heat generated due to the movement of the abrasive particles on the work piece, the heat generated by the vibration due to the transducer and in removing the debris from the machined area.

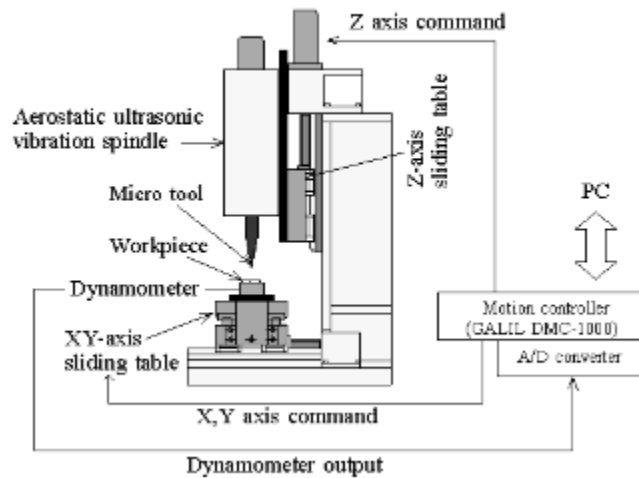


Figure 4 Sample micro ultrasonic machining system [27]

There are many factors which affect the outcome of the machining which are:

Contact force between tool and workpiece: micro tools are sensitive to elastic bending, vibration and breakage, which require special care to limit the contact force between tool and workpiece to a certain level during machining to prevent any of the above problems from happening.

Abrasive particle size: Particle size affects different characteristics like material removal rate and surface roughness. Using fine grain size particles, material removal rate increases with the increase in the abrasive particles, as this increases the number of particles involved in the machining process. More number of fine grains with constant cutting edges hitting the workpiece repeatedly, increases the frictional heat making the surface rougher increasing the surface roughness

Using medium grain sized particles, material removal rate increases with the increase in particles in the slurry as there is an increase in the cutting edges for a given volume of slurry. With the increase in the concentration the particle size decreases in the process of machining, which indicates there is more surface area of the particles to absorb the heat generated during the machining process which reduces surface roughness.

Using coarser grain size particles, coarser particle grain boundaries try to interlock resulting in a reduced number of cutting edges, thereby reducing the material removal rate. With the increase in the concentration there are more number of coarser particles hitting the work surface making the surface rougher, increasing the surface roughness. From all the above we can conclude that good surface finish is given by machining with finer particles and poor surface finish is obtained from using coarser grains [26].

Coolant used in the slurry: Even the Coolant used in the slurry affect different characteristics like material removal rate and surface roughness. Water based slurry is suitable only for finer particle sizes with higher concentration or medium particle sizes with medium concentration or coarser particle sizes with lower particles, whereas machining with oil based slurry is always suitable for all particle sizes with low concentration.

Limitations of this process of manufacturing micro-channels are:

- Unlike vertical side walls produced by ultrasonic machining, abrasive blasting machined features have tapered sidewalls ranging from 18 to 26°, depending on several factors
- Maximum aspect ratio to 3:1 (Thickness : Diameter)

## Chapter 4

### Research Procedure

Limitations of the existing processes and focusing on the complex processes involved in the microchannel manufacturing, result in high cost and special facilities requirements for many processes. We targeted reducing process complexity and manufacturing cost for producing microchannels in glass.

#### 4.1 New Process Development

This research included glass selection that served as a substrate for microchannel feature development, and development of a robust manufacturing process for fabrication of microchannel geometries in glass substrate. The research involved a series of steps to finalize a process that included identification of process parameters needed to produce a high quality product.

##### 4.1.1 Glass Selection

We broadly classified glass into three types studying their material, physical, optical, chemical and thermal properties to determine which type of glass would best serve for microchannel fabrication. Table 1&

The procurement cost for these glass types increases sharply from soda-lime to borosilicate to fused silica/quartz glass. Ultimately, soda-lime glass was selected for our research based on its thermal properties, chemical composition and its very inexpensive cost compared to the other glass materials. Cost played a main role in our material selection decision as soda lime glass is 75 times less than the next type of glass: borosilicate. In fact, a typical microscope glass slide produced from soda lime costs only about 30 cents, compared to a borosilicate slide at a cost of \$ 25. However, manufacturability of soda lime glass for micro features is limited and is fraught with challenges including its poor machinability and chemical resistivity.

Table 2 Different Types of Glass Thermal and Material Properties

below list these various types of glass that were studied, and their associated properties.

Table 1 Different Types of Glass Physical and optical properties [28] [29] [30]

Physical Properties	Borosilicate glass	Soda-lime Glass	Fused Silica
Density g/cm <sup>3</sup>	2.23	2.48	2.2
CTE (20°C - 300°C) K <sup>-1</sup>	3.3* 10 <sup>-6</sup>	9.06 * 10 <sup>-6</sup>	.55* 10 <sup>-6</sup> °C
Thermal Conductivity W/m°C	1.14	0.937 W/mK	1.38
Specific Heat j/Kg °C	750		740
Dielectric constant (1MHz, 20°C)	4.6	7.75	3.82
Poisson's Ration (25°C - 400°C)	0.2	0.2	0.17
Optical Information			
Refractive Index (Sodium D line)	1.474	1.523	nD( 589 nm) - 1.458
Visible light transmission, 2mm	92%	92%	92%

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Table 2 Different Types of Glass Thermal and Material Properties [28] [29] [30]

Critical Temperature's			
Annealing Temp °C	565	545	1215
Softening Point °C	820	720	1683
Stress Point °C	510	n/a	1000
Strain °C	n/a	494	1120
Chemical Composition			
	SiO <sub>2</sub> : 80.6%	SiO <sub>2</sub> : 72.20%	SiO <sub>2</sub> : 99.99%
	B <sub>2</sub> O <sub>3</sub> : 13.0%	Na <sub>2</sub> O : 14.30%	
	Na <sub>2</sub> O : 4.0%	CaO : 6.40%	
	Al <sub>2</sub> O <sub>3</sub> : 2.3%	MgO : 4.30%	
		Al <sub>2</sub> O <sub>3</sub> : 1.20%	
		K <sub>2</sub> O : 1.20%	
		Fe <sub>2</sub> O <sub>3</sub> : .30%	



#### 4.1.2 Manufacturing process selection

Having finalized soda-lime to be the glass material, primary process needed to be developed that could overcome its first major limitation: lack of robust machinability of this material. For soda lime glass, soda and many other chemicals are added to silica to obtain an inexpensive glass with lower processing temperatures. Unfortunately negative features that accompany lower processing temperature are a reduction in thermal stability, fracture strength, thermal shock resistance, thermal conductivity and an increase in its CTE. These properties restrict many current machining and fabrication processes from being used for soda-lime glass.

We reviewed all of the glass working processes from the domain of art in an attempt to find the best glass working process that could be adopted that would satisfy our requirement of producing microchannels and be easily scalable for mass production. Cold working of glass eliminated as it involves the use of tools and machines that directly machine the glass itself. These processes tend to result in defects in the microchannels such as poor surface quality, lack of clarity and dimensional control restrictions.

Hot working of glass involves working with glass in its liquid state while it is highly viscous. The hot liquid is collected and modified to the desired shape or form. This process is not easily scalable for mass production and involves an experienced glass artist working in front of a high temperature furnace which is typically above 2300°F.

Warm glass working makes use of a kiln typically heated to 1600 °F. The kiln itself is small (typically less than 3 feet square) which makes it ideal for working with small parts, and inexpensive to produce (\$700). Two main processes of warm glass

working are fusing and slumping. Fusing would not be of help to us for manufacturing microchannels, as this is a process that bonds or joins two or more pieces of glass together. However, we made use of this process at a later stage to bond glass slides with channels for producing a stack of glass slides with channels in between the slides.

Slumping is the process of placing glass atop of any mold and allowing the glass to gradually take on the shape of the mold. Slumping was tested to determine whether this process was feasible for producing micro-level features or if the process only works for larger features. The first tests made use of Thinfire (kiln shelf paper) to produce microchannels in the soda lime glass. Thinfire strands created microchannels by allowing melted glass to slump over the strands. As can be seen in Figure 5, although this process showed slumping to prove useful for making microchannels, the resulting features were in no way precise.

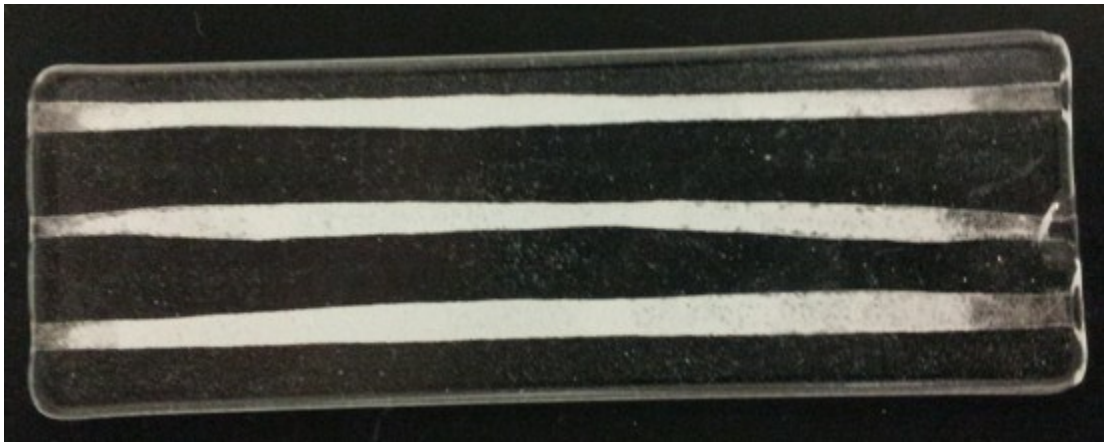


Figure 5 Glass slides with channels formed by slumping on Thinfire

#### 4.1.3 Mold Material Selection

Mold materials used throughout the history of glass and till present day are typically clay or of a plaster/silica mixture. The mold is prepared in the glass studio

without the involvement of any expensive equipment or processes, which is quick and meets the requirements of the glass artist. The main advantages of both clay and plaster are that they will not expand or contract differentially resulting in a broken piece of glass. These materials however, cannot be used for our process as obtaining micro-level precision using them is a tedious or often impossible task. This required us to look for a different mold material. We started looking into all the characteristics that a mold material would need to obtain the precision and the clarity we need for our process. Research found that the main characteristics needed to be concentrated on included the coefficient of thermal expansion (CTE), thermal conductivity (TC), melting point/working temperature, machinability and cost.

Coefficient of thermal expansion: CTE plays a vital role in deciding the resulting product quality from the process. CTE of the mold material should be very close to that of the glass we are using. Both the glass and the mold will be heated and cooled at the same rate. If this heating/cooling rate produces differential expansion or cooling then the result is often a shattered piece of glass rather than a single piece of glass with the features we desire. The mold material that we choose should have a CTE which is very close to that of glass.

Melting point/working temperature: This is another characteristic which directly affects the product quality from the kiln. If the melting point of the mold material is less than or close to the softening point of the glass, then the mold material will distort or melt resulting in a damaged product. The melted mold material and glass become inseparable from each other and from the kiln shelf. The melting point of the mold material should be

1.5 times or more to that of the softening point of the glass. For example if the softening point of the glass substrate is 720°C the melting point of mold material should be at least 1100°C.

**Thermal conductivity:** TC plays a vital role in understanding the glass response to temperatures, reducing additional steps involved in determining the temperature difference between the kiln atmosphere and the glass surface temperature (glass enclosed in the mold). The greater the thermal conductivity the better it is for controlling the process.

**Machinability:** If the die material satisfies all the other conditions but is not easily machinable, it deviates from the basic objective of the process. If machining the mold requires a complicated process then avoiding that material would be best.

**Cost:** There are a limited numbers of mold materials satisfying the requirements for glass molding as described above. Available mold materials vary widely in cost, from inexpensive graphite to expensive platinum.

Filtering all the materials based on the search criteria cited above resulted in very few materials that could actually be used as mold materials for glass working. Listed below are mold materials that were reviewed for the research.

**Metals:** platinum, rhodium, ruthenium, vanadium and tungsten alloys [31].

**Graphite:** GLASSMATE, Biomedical, Semiconductor, EDM grades [32].

**Ceramics:** Traditional (Based primarily on natural raw materials of clay and silicates), Advanced (Includes artificial raw materials that exhibit specialized properties, require more sophisticated processing) [33].

From the above list of materials, graphite was initially considered due to its low cost and easy machinability. Technically there were a lot of varieties of graphite which all satisfied the requirements, leaving experimentation as the only option to find the best out of all the available graphite varieties. We started our experiments using GLASSMATE™ grade graphite. It was machinable and worked very well as a mold when used in a hot vacuum press, however, this particular graphite oxidized when used in the kiln. On explaining the situation and the requirement with the manufacturer of the graphite, their technical staff suggested GLASSMATE-HT™ graphite as it has a higher oxidation threshold. Machining it was smooth as the previous graphite; however, the glass from the kiln was full of shiny spots which turned out to be zinc deposits. Zinc was the oxidation resistant material that was added to the graphite to increase its oxidation threshold, unfortunately leaving residue on the glass.

We next tested a semiconductor grade SFG™ graphite which resulted in a satisfactory solution for obtaining clear channels comparable to the previous two graphite materials (GLASSMATE™ & GLASSMATE-HT™). However, a major problem arose when we attempted to machine features in the range of 10-50 microns. Cracking of the graphite mold was observed during machining and also during the slumping process. As the graphite particle size was 5 microns, it easily chipped during machining. A material capable of withstanding the machining process without disintegrating, and later withstanding the weight of the glass sample during the slumping process was required. As a stronger material was required, we next tried a semiconductor grade SFG-2™

graphite. Apart from particle size, SFG-2<sup>TM</sup> is comparable to SFG<sup>TM</sup> in all respects. SFG-2<sup>TM</sup> was finalized as our graphite mold material.

SFG-2<sup>TM</sup> graphite mold material delivered glass slides with clear channels which had provided a scope and opportunity for further improvement of the process. Although acceptable for the current research, the graphite mold material had two major drawbacks: 1) the graphite mold can be used only once, as it oxidizes at higher temperatures under atmospheric conditions. 2) Absorption of tiny graphite particles during the slumping process (were visible under high resolution microscope).

Titanium was considered as a mold material as it satisfactorily met the desired characteristics of a mold material for glass working. Experimentation with titanium as mold produced a dull black residue on the glass surface. At high temperatures during slumping, the residue mixed with the glass surface. Even an acid wash did not remove the residue from the glass surface. After conducting multiple tests, it was concluded that graphite was much better than titanium as mold material. Optimizing the kiln firing schedule was sought to eliminate particle problems.

Initially, ceramics were not considered for mold materials due to their machinability restrictions. Ceramic materials are hard and require sophisticated equipment for machining. MACOR<sup>TM</sup> (machinable glass ceramic) [34] developed by Corning<sup>TM</sup> was considered a satisfactory mold material for future research. Machining MACOR<sup>TM</sup> was simple compared to other ceramics, as the needed protrusions for molding the microchannels were easily machined. Few slumping experiments using glass slide in MACOR<sup>TM</sup> mold produced microchannels in glass slides. The consistency of the

channels was not tested as it was a preliminary stage, and this mold material is proposed for future research. If the results produced are consistent over several experiments, this mold material would reduce the cost of this microchannel manufacturing process drastically.

#### 4.2 Machine Selection

Having determined the manufacturing processes and the mold material, deciding upon the machines and the machining processes utilized for mold machining and slumping were then required.

Immediate machining capabilities included:

Oxford Lasers<sup>TM</sup> micro-machining center (Diode-pumped solid-state UV nanosecond laser): The laser micro-machining center is based on a 355 nanometer wavelength diode-pumped solid state nanosecond laser system. The center is designed as a general purpose laser micromachining tool for cutting, drilling, marking and 2.5D milling, capable of machining most types of engineering materials. The system is built on a granite gantry platform, supported by a special base with vibration isolation legs. Motion control is achieved using a 2-axis CNC stage consisting of suction table and jig attachments. Focus control optics move along the third axis.

Laser specifications:

Laser: pulsed fully diode-pumped solid state

Wavelength: 355 nm

Output: 5W@10kHz

Pulse length: 15ns(FWHM) nominal

Axis: 4 axis (X, Y, Tip & Tilt)

Focal length: 66mm with 15 mm clear aperture

Spot size: < 10 micron



Figure 6 Oxford lasers™ micro-center [35].

ToolCrafter™ CNC High speed micro mill: a 4-axis high speed 1/8<sup>th</sup> inch machining center capable of spindle rotation speed of 60,000 revolutions per minute. Has 6”x4” work area capable of machining micro and meso-scale features. The machine has a built in misting system to cool the work piece and extend the life of the endmill.





Figure 7 Tool crafter™ micro milling machine.

AVS™ Vacuum hot press: It is a mini 5 ton hot press furnace designed and developed by AVS™ especially for research and development (R&D), for hot pressing, diffusion bonding, thermal forging and powder densification, with a maximum operating temperature of 1650°F, and welded vacuum chamber allowing high vacuum range up to  $10^{-6}$  torr, with a leak rate of  $<5\mu$ /hour. Vacuum furnace system sub systems and their specifications are:

Vacuum pumping system: This system consists of two sub systems which are

Rough pump system: it is provided with Kinny Model KDH-150 single stage mechanical pumping system, which is capable of evacuating the vacuum furnace down to  $10^{-6}$  torr.

High vacuum system: This consists of a Varian HS-2 diffusion pump, a foreline valve and a high vacuum valve.



Figure 8 AVS vacuum hot press furnace [36].

Hot press system: The hot press system consists of a hydraulic pump, a controlling servo valve and a hydraulic cylinder with a 1 3/8" diameter rod, 3 1/4" bore and a 6" stroke capable of applying up to 5 metric tons of force.

The whole system requires a 460 volt ac, 3-phase, 60 Hz electrical service, supporting a full load current of 80 amps. The system also requires water supply at a rate of 15 gallons minute with an operating pressure in the range maxing at 50 psi for proper vacuum furnace operation, the system uses inlet water temperatures up to 104°F with sufficient water flow to keep the outlet below 104°F.

To achieve simplicity in the process, it needed to be developed using machines available in the Micro Fabrication laboratory which are less expensive as compared to typical semiconductor fabrication clean room machine tools. The CNC micro mill was

the first machine to be considered for machining due to its high speed capability. The micromill had an inbuilt mist sprayer which delivers coolant to the machining surface, which also removes debris. Unlike metals, graphite is a powder based material with carbon particles bonded together. Using coolant would cause problems as the machined particles tend to bond together again, with the coolant acting as a binding agent. This process of re-bonding resulted in an uneven machined surface with higher surface roughness, and excessive tool wear. Rather than using the mist sprayer, the vacuum was used to overcome the problem of debris removal. However, the problem of tool wear and overheating now had to be addressed. This challenge was solved by making use of Aluminum-Titanium-Nickel (ALTiN) coated tools. These tools have higher wear and temperature resistance. Having overcome initial hurdles with machining graphite, we were able to machine a graphite mold to the desired initial requirement to test for channels being produced.

Using the high speed micromill to produce the mold, work was then undertaken to develop molds that would ultimately produce microchannels in the 10-100 micron range.

An attempt was made to machine the mold for the channels with the required dimensions, the resultant mold was not satisfactorily close to the required dimensions. The problem was identified as a large endmill diameter compared to the small required channel width dimensions. Based on the channel, a micro endmill with diameter less than 30 micrometers was required. It was difficult to find endmills and drillbits that satisfy the requirement for machining the mold with required dimensions as well as being

compatible with the micro milling machine. Under the given conditions an alternative machining solution was sought.

The laser micro machining center was considered as an alternative to CNC micro milling machine for mold machining. Machining the complete mold using laser is time consuming which might take days to complete based on our surface roughness requirement. This would make this process expensive compared to many of the existing microchannel manufacturing processes. To overcome this problem, an alternative of using both the CNC micro mill and the laser micro machining center was considered. The mold machining process was divided into two steps, with the major part of the machining first to be done by the micromill. The second step makes use of the laser to machine the microchannel features in the range of 10 to 60 microns.

The division of the machining operation into steps is only for the bottom part of the mold Figure 26. The top part [Figure 25] does not need any micro-features machined. Therefore the top part of the mold is machined only by the micromill.

The final step in the process for microchannel manufacturing in glass slides is to slump the glass slide in the mold, as it approaches the softening temperature of the glass. Early attempts at molding made use of the vacuum hot press without any external load. However, slump molding of glass in vacuum causes outgassed (see Figure 14). Under vacuum, particles within the glass during its semi liquid state are absorbed. This absorption creates bubbles and disturbs and disperses the amorphous structure of glass. Therefore, the vacuum hot press was abandoned for an artist's kiln to perform the slump molding operation

The kiln purchased was Paragon™ TNF663 from Paragon™. A clear glass with channels was produced from the kiln.

Glass kiln TNF663: It is a 7-sided digital glass kiln with 3” firebrick walls, with a heavy galvanized steel base that covers the reversible brick bottom completely and also covered by stainless steel on all the sides which in-turn strengthens the firing chamber. This is a small kiln ideal for small dolls, jewelry, yet still large enough for most ceramic projects [37].

It is built in with a reliable user friendly solid-state sentry controller which continually monitors the firing, relieving the glass artist from sticking to the kiln till the end of the project.

#### Features of the TNF-663

- 3” firebrick walls
- Sentry 12-key digital controller made by Orton ceramic foundation
- Full-formed, galvanized steel base
- Heavy gauge stainless steel case
- Fall-away two stage prop-r-vent
- Two wide-view tapered peepholes



Figure 9 Paragon™ glass kiln TNF 663 [37].

- Ventilated, louvered switchbox
- Reversible brick bottom
- Dropped, recessed brick grooves to eliminate element pins

The kiln added value to the process as, it was the main machine that delivered clear glass slides with channels, and also reduced the manufacturing cost drastically making the manufacturing cost much lesser than many other microchannel manufacturing processes.

#### 4.3 Design Selection

With satisfactory channels being produced in glass slides by using the process developed, we approached researchers from the Biomedical department at UTA for their

suggestions and product requirements. Microchannels in glass would be of research interest for the Biomedical department. Interested in such capability and its contributions to his research in nerve regeneration Dr. Romero Ortega provided us with a design. The design provided by Dr. Romero Ortega would serve as a scaffold for nerve regeneration and growth. It had two adjacent cylindrical wells connected by microchannels, at the bottom of both wells. The process developed by our laboratory produced open channels on the glass slide surface, whereas the design needed by Dr Ortega's laboratory required closed channels. To make our process work for their requirement, the design provided by them was split into two parts. In the modified design, the top part would consist of all the channels and wells, and the bottom part would consist of a glass slide "cap" for the device. The cap had two cylindrical holes that aligned with each end of the microchannel array. The top and bottom would be joined by glass fusing, such that the channels would be between the two glass slides. Since the channels on one glass slide are bounded by the second slide, they become closed channels. The device currently being used by Dr Romero Ortega's research team up to this point for nerve re-generation studies was PDMS, which had the disadvantages previously described.

Model diagrams:

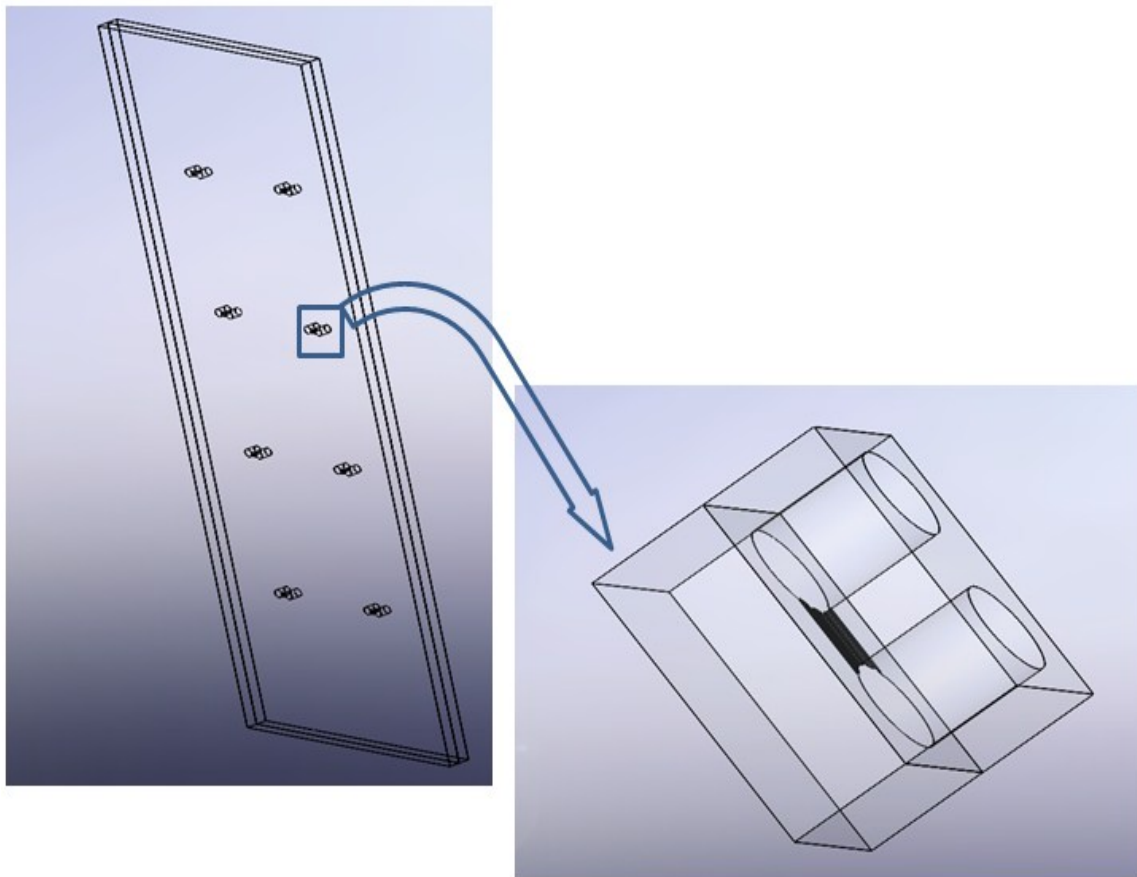


Figure 10 Full design of devices in glass slides & full single device



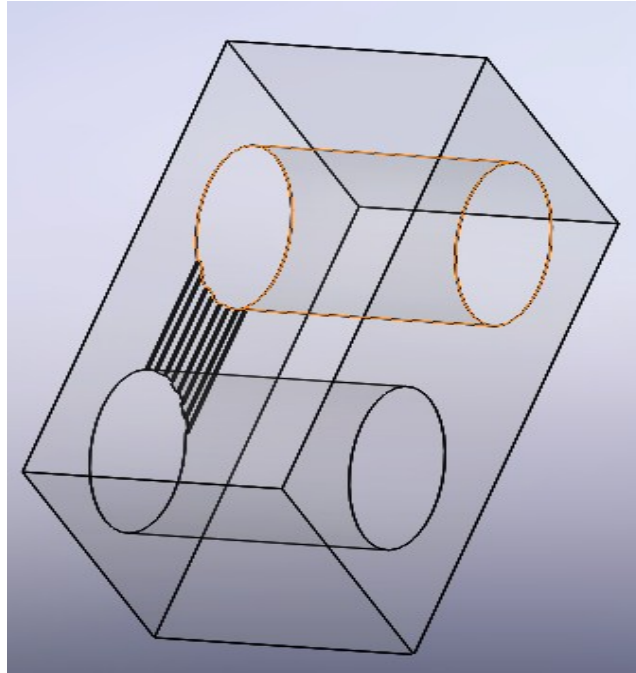


Figure 11 Device top part with channels

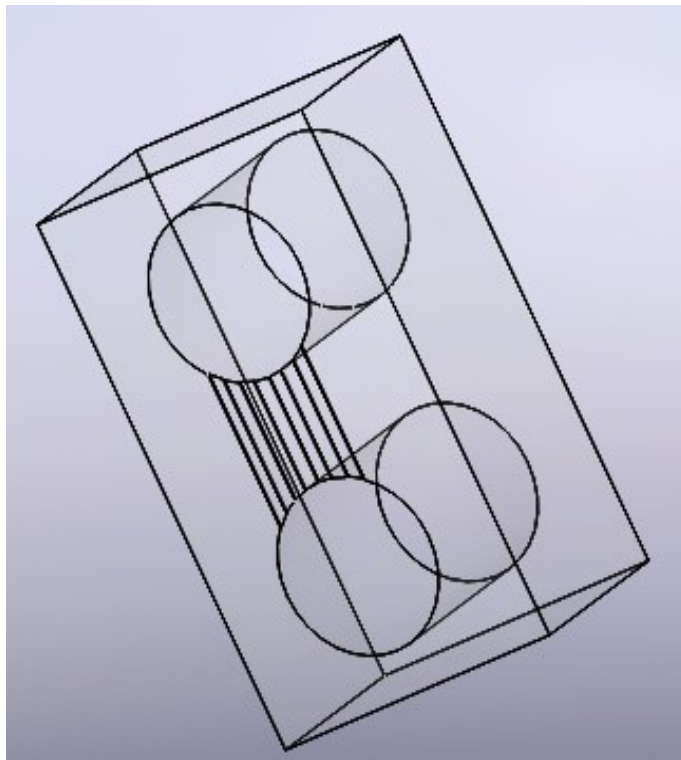


Figure 12 Device top part

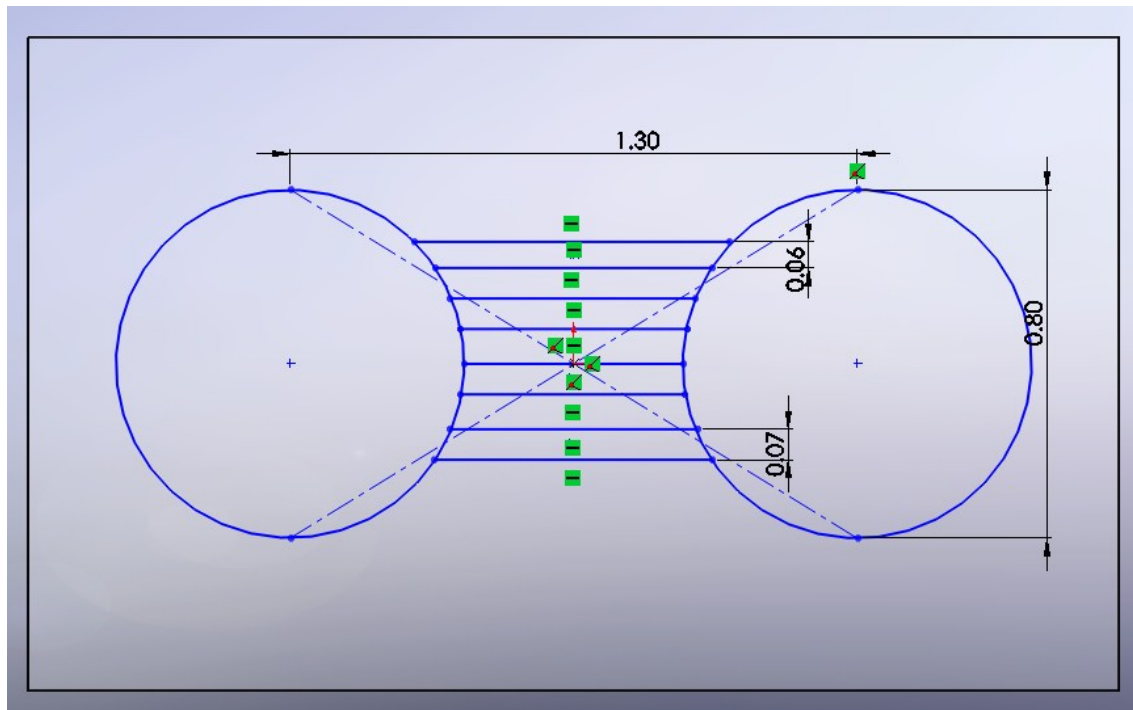


Figure 13 Wells and channels with dimensions

#### 4.4 Machine Operating Parameter Selection

Having finalized the process and the sequence of steps involved for producing microchannels in soda-lime glass slides, parameterization was considered to optimize the process. Optimizing the parameters on all the machines used, helps in establishing a constant repeatable process in delivering microchannels with consistent quality. A traditional trial and error method was chosen to perform parameter optimization rather than the new design of experiments approach. The trial and error method was selected due to time and raw material constraints. Design of experiments was not appropriate due to constantly changing design. The main process parameters and their importance, is explained below.

CNC Micro milling machine

The main parameters that affect the result being produced by this machine, and affect the mold material being used are speed of the spindle, feed rates, and the endmills being used. This section will be discussing only about spindle speeds and feed rates as endmills selection was discussed in the previous section.

Spindle speed is the rotational frequency of the spindle measured in revolutions per minute (RPM), also defined as the rate at which material moves past the cutting edge of the tool. Spindle speed depends on the hardness of the material and the material of the tool being used. This plays a vital role in determining the quality of the product being produced by the micro mill. This parameter has a major impact on the material removal rate and the wear of the endmill. Slower spindle speeds cause excessive debris deposition on the tool, which results in excessive wear, higher surface roughness and irregular machining. Higher spindle speeds cause excessive tool wear, tool breakage or work piece shatter which could lead to dangerous conditions. Graphite's Shore Sclerescope Hardness (SSH) is 88. This is equivalent to Vickers hardness number 789, which is equal to that of tool steel. Since graphite is hard, with an absence of coolant during machining, the speed needs to be low at the same time not too slow to damage the machining surface and the tool.

Feed rate is the velocity at which the tool advances against the workpiece, which also depends on the spindle speed and the flute number. Feed rate determines the nature of material removal from the work piece. If the feed rate is too high with less number of flutes, the material is removed by the moving tool rather than the cutting edges, which is destructive and does not result in desired features. Slower feed rates with higher flute

number would result in the tool rubbing against the machining surface rather than the cutting edge removing material, which result in tool marks on the surface which are not desired. Considering all the working conditions, this parameter should be tested for optimal as this plays a major role in the surface roughness of the product from the micro milling machine.

#### CNC Oxford laser<sup>TM</sup>

Laser machining has many parameters which determine the resultant product being produced, increasing the number of parameters to be optimized. Parameters to be considered are frequency, attenuation, cut path spacing and table speed.

Frequency is one of the main parameters that determine the amount of material being removed. To start with the optimization process, frequency was set to an automatic mode where the machine determines the frequency based on table speed and cut path spacing. Having obtained few results of the estimated frequency range, a frequency that matched the material removal depth needed was selected to be our frequency. Since we are trying to produce micro channels in the range of 1-25 microns, the frequency selected is very low (0.1 to 0.6 Hz) compared to other machining processes. Higher frequencies are used for materials that are much harder than graphite such as silicon.

Attenuation is the process of reducing the intensity of the laser beam. Varying the attenuation has a direct impact on the material removal rate. Both the frequency and attenuation can be alternately adjusted. Different sets of parameter values for frequency and attenuation can result in similar material removal results. Frequency was optimized first and based on the frequency value, attenuation was then optimized.

Cut path spacing is the spacing between two consecutive parallel cutting paths. This determines the smoothness of the resultant surface. Considering spacing greater than the laser spot diameter, would result in a thin strip of uncut material in the final product. Considering spacing lesser than the laser spot diameter, would remove excess material resulting in a deeper feature than designed for. With the average size of the laser spot available being 1-10 microns, an optimal spacing would be within this range. Multiple passes and increase in frequency can be compensated for, by reduction in the cut path spacing making it less than the least spot diameter.

Table speed is the rate at which the work piece is moving along the X and Y axis with respect to a constant laser source. Table speed plays a vital role in the resultant product. Choosing a higher table speed would result in lesser laser workpiece contact time, eventually resulting in uneven material removal or an undercut. In this case it would increase the number of passes needed to obtain the required depth. Choosing a lower table speed would result in excess laser workpiece contact time, eventually resulting in excess material removal. In this case the resulting damage due to excess material removal cannot be compensated. The selected table speed should be an optimal value which is neither too high nor too low, either of which would result in a waste of resources.

#### Glass Kiln

Glass reacts differently to different temperatures, temperature gradients and hold times at those temperatures. A kiln operates based on a firing cycle or firing schedule, which consists of multiple steps from the start to the end of the process. Each step in the firing cycle consists of a temperature gradient ( $^{\circ}\text{F}/\text{Hr}$ ), the final temperature ( $^{\circ}\text{F}$ ), and

hold time at that temperature (Min). The main parameters that play a vital role on the resultant product from the kiln are temperature gradient, slumping temperature, slumping hold time, and annealing hold time.

Temperature gradient is the rate at which glass is heated or cooled within the kiln. This depends on the dimensions of the glass and the type of the kiln in use. The dimension is important in determining the temperature gradient as the temperature difference across the object should never exceed 5°-10° range. If the temperature difference across the object is greater than the specified range, the glass object would result in cracking due to stress. Type of kiln plays a role as it directly affects the temperature difference across glass. Glass closer to the filament heats faster than the glass farther, creating a temperature difference. Glass kilns with coils on the walls are preferred as they tend to heat the object equally in all directions. Multiple test were conducted close to the values provided by the glass artist.

Slumping temperature refers to a temperature at which pre-formed glass transforms by heat and gravity. Slumping occurs over a range of temperatures based on the requirement of the end result. Slumping depend on the viscosity of the glass which is directly related to the temperature of glass. Hard slump occurs at higher temperatures greater than softening point, where glass is more viscous and require capturing very deep and complicated details from the mold. Soft slump occurs at lower temperature lesser than the softening point, where glass is enough viscous to sag into the form. Soft slump is preferable used where there is a chance of particles being absorbed into the glass. This

temperature is obtained by experimenting various values lesser than the softening point but still staying close to the softening point.

Slumping hold time is the amount of time for which the kiln is held at the chosen slumping temperature. This should be equivalent to the time required to sag into on to the form, just enough to obtaining the desired shape without staying in there long to absorb the particles on the surface. Less time would result in an incomplete transformation of glass, and more time would result in absorbing particles from the surface. Either would result in a lower quality result. Multiple hold times were experimented for each of the temperature chosen. Slumping temperature and hold time can be varied alternately, resulting in a different set of values resulting in a same output product.

Annealing hold time is the amount of time for which the kiln is held at the annealing temperature of glass. Annealing is one step that is present in any kiln working process. This is not the most important step, yet without successfully passing through this stage the resultant glass would not be relieved of the internal stress. The glass should be annealed for decent amount of time to completely remove the internal stress completely, failing which there is a chance for the glass to crack later on during its use. The annealing hold time depends on the dimensions of the glass. The thicker the glass more time it is to be held in the annealing stage, which usually range anywhere from few hours to few weeks or even months.

The next two process steps are discussed briefly as they are not the actual part of the dissertation however, were necessary for completely produce the device for testing.

Glass Drilling

Glass drilling was used to produce wells (reservoirs) in the device for holding the media and cells. Micro mill is used for the drilling of glass and the drillbits used were diamond micro drillbits TDR346<sup>TM</sup> manufactured by Craurence<sup>TM</sup>.

#### Glass Fusing

After the glass slides with wells and channels are delivered from the above processes, fusing is performed to close the channels and wells. Slide with channels and channels are fused to a new glass slide using the kiln. The fusing temperature is equivalent to the slumping temperature with an additional mold placed on top of the setup, to act as a weight.

#### 4.5 Step Wise Process Improvement

Process development started with testing, to fuse two glass slides using vacuum furnace press. The result from the first few tests resulted in a blob of glass (glass filled with air bubbles) [Figure 14].

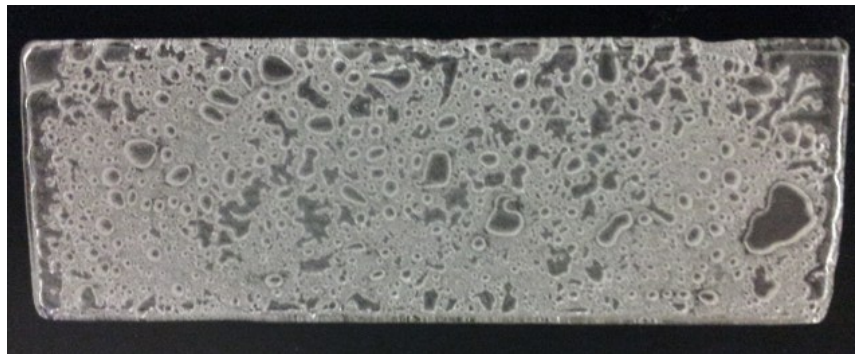


Figure 14 Glass filled with bubbles

With the motive of resolving the problem of bubbles, consulted the glass artists from UTA glass department. Explained the objective, previous experimental results and requested them for suggestions. Different glass working processes and parameters that



glass working depends on are explained. From the glass working processes slumping was chosen for reasons explained in the previous section. To start slumping experiments choose Thinfire™ paper. The paper was cut into the dimensions of the channels required and placed in-between the two glass slides. The setup of the paper in between the glass slides was experimented for slumping based on the estimated parameters provided by the glass artist. Multiple experiments with varying parameters resulted in different improving results in-terms of slumping, but it did not solve the problem of bubbles. Step wise improvements attained with the bubbles still present in glass are shown below [Figure 15][Figure 16][Figure 17].

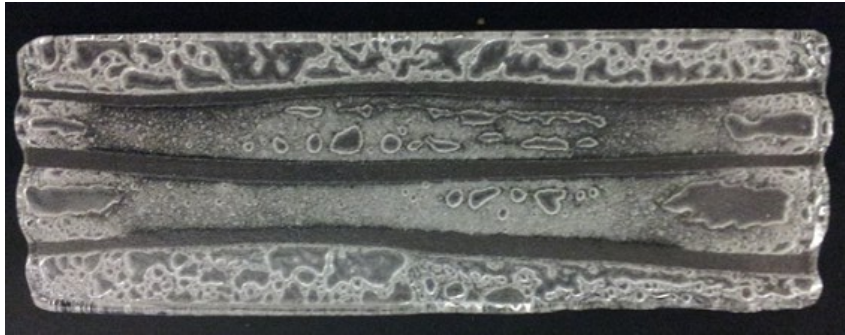


Figure 15 Improvement step 1 using Vacuum Furnace Press

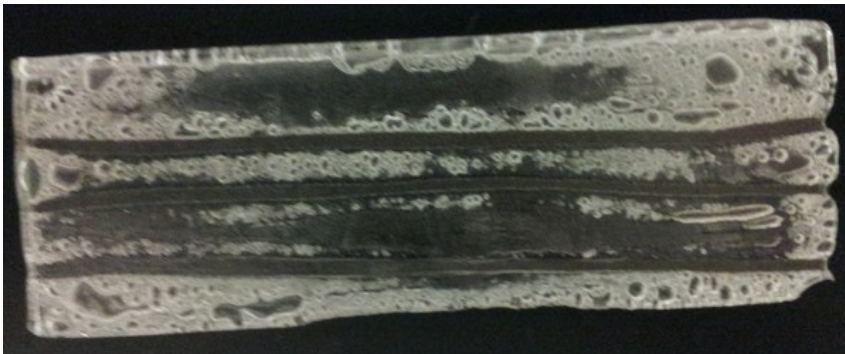


Figure 16 Improvement step 2 using Vacuum Furnace Press

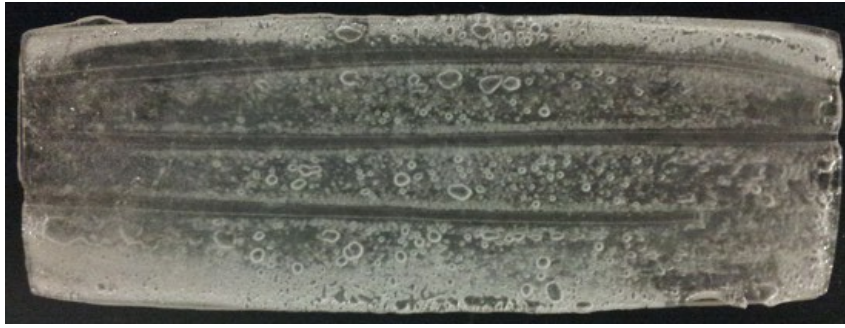


Figure 17 Improvement step 3 using Vacuum Furnace Press

These improvements were obtained by gradually reducing hold time and the slump temperature from above the softening point to near softening point. After several experiments with varied parameters we were unable to remove bubbles from the glass slides. Consulted the glass artist again, requesting for suggestions on obtaining clear surface by removing the air bubbles from glass slides. The glass artist provided us with a glass piece and parameters for the firing cycle to be used. Upon testing the glass for slumping with the provided parameters, the resultant product was similar to foam of glass completely filled with air bubbles. With an unexpected result from the previous experiment, slumping of the glass slides in the kiln using vacuum furnace press parameters was considered. This test resulted in a product without any air bubbles between the glass slides [Figure 18].



Figure 18 Resultant from slumping in glass studio kiln

Further experimenting with varying parameters resulted in clear glass slides with channels obtained using Thinfire™ [Figure 5].

Having obtained clear channels with thinfire, we started experimenting with graphite as mold material to produce channels. After testing with multiple graphite materials, using which the machined structures used to shatter under the weight of glass, a stronger graphite material GLASSMATE – HT™ with higher oxidation threshold was considered. Experiments with this as mold material resulted in channels along with a thick residue on the glass slide [Figure 19]. This was due to the presence of zinc, which was added to increase the oxidation threshold.



Figure 19 Glass slide resulting from using GLASSMATE-HT™ mold

A different graphite material SFG™ was chosen as mold material. Experimentation was necessary ever time there was a change in mold as each graphite had different properties. Porosity, heat dissipation and thermal conductivity are several properties which change from one graphite to another. Change in these properties also changed the slumping parameters required for new graphite. Clear glass slides with channels were being delivered using SFG™, with the only problem being intermittent channels. The cause for intermittent channels was the cracking of the machined channels

due to particle size. Improvement steps in the clarity of glass with varying parameters using SFG<sup>TM</sup> mold are below [Figure 20][Figure 21][Figure 22].



Figure 20 Improvement step 1 using SFG<sup>TM</sup> mold in a kiln

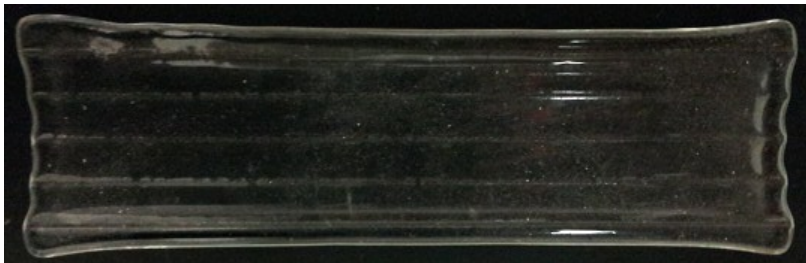


Figure 21 Improvement step 2 using SFG<sup>TM</sup> mold in a kiln



Figure 22 Improvement step 3 using SFG<sup>TM</sup> mold in a kiln

SFG-2<sup>TM</sup> was used to solve the problem of intermittent channels. SFG-2<sup>TM</sup> is comparable to SFG<sup>TM</sup> in all properties except for the particle size. Particle size of this graphite is 1 micron, which resulted in smooth edges of machined channels. SFG-2<sup>TM</sup> is the final graphite mold material chosen for this process as clear glass slides with channels

were being produced consistently. Optimizing the slumping parameters for SFG-2™ did not require extensive experimentation, as both of the materials were similar. Few variations in parameters resulted in clear slides with straight channels.



Figure 23 Improvement step 1 using SFG-2™ mold in a kiln

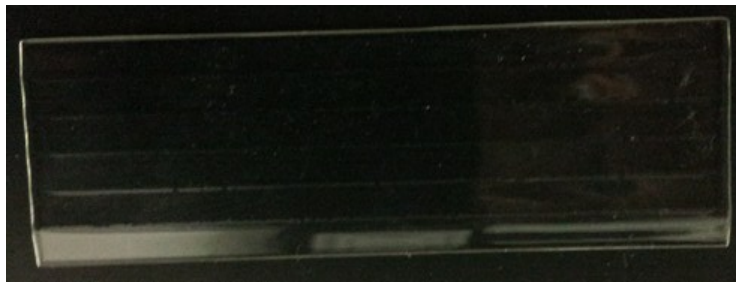


Figure 24 Improvement step 2 using SFG-2™ mold in a kiln

SFG-2™ is the final graphite mold material for the developed microchannel manufacturing process.

## Chapter 5

### Research Results & Validation

#### 5.1 Research Results

In this chapter are discussed all the final parameters of the machines used in the process and the final process developed for microchannel manufacturing in glass. The process developed is a series of steps to be performed on different machines, starting with the CNC micro mill ending with the artist glass kiln.

Step1: The first step involves the manufacturing a major portion of the bottom part and the top part on the mold using CNC micro milling machine. Machining parameters used for both the top and bottom part are the same.

Endmill: ALTiN coated 1/8 SE 4FL

Spindle speed: 4000 RPM

Approach feed: 20 mm/min

Engage feed: 20 mm/min

Cutting feed: 300 mm/min

Retract feed: 50 mm/min



Figure 25 Top part machined by micro mill

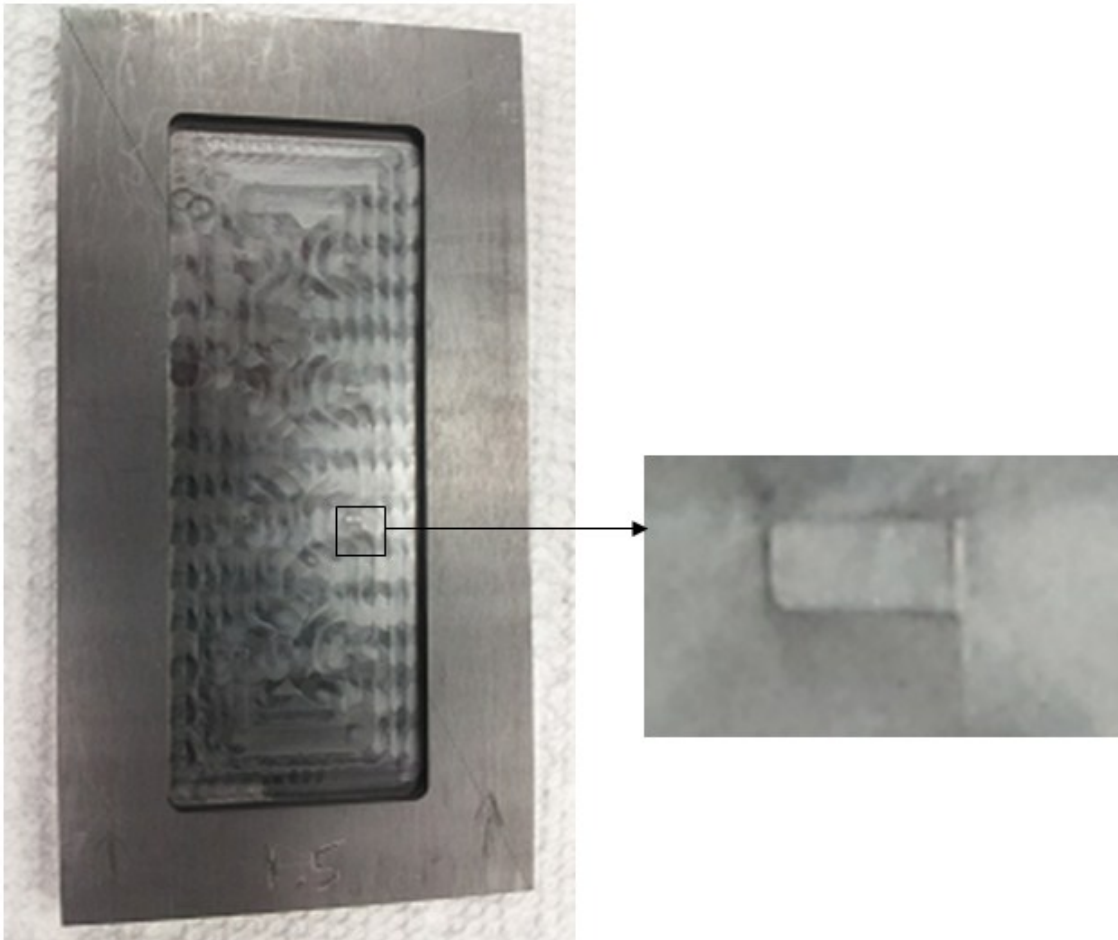


Figure 26 Partially machined mold using micro mill

Step 2: Taking the half machined bottom part of the mold produced by the CNC micro mill. Microchannels to be transferred onto the glass are machined using a CNC laser micromachining system. The two images below clearly show the difference between the resultant surface before and after brushing and blowing away tiny graphite particles. The final process parameters that we used for our machining process are:

Frequency: 0.6 Hz

Attenuation: 35%

Cut path spacing: .001mm

Table speed: .25 mm/min

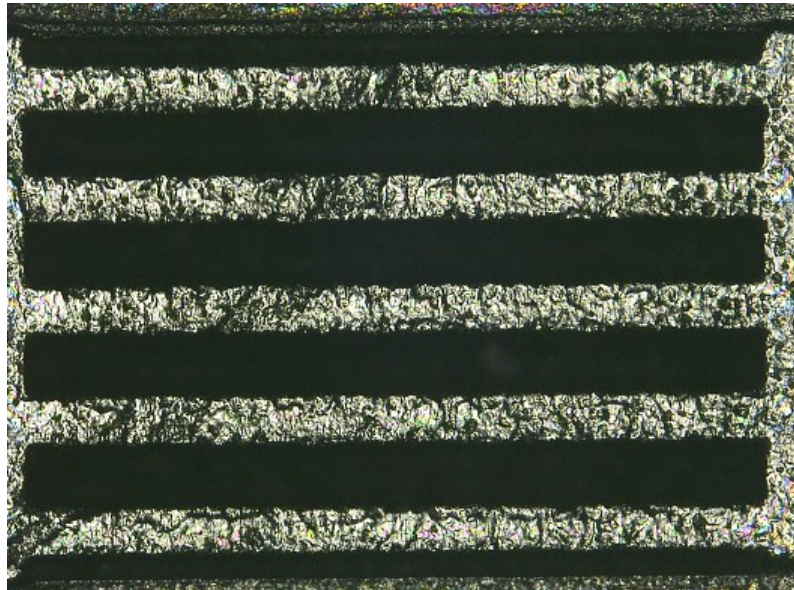


Figure 27 Microchannels machined on the mold using laser (after cleaning)





Figure 28 Microchannels machined on the mold using laser (before cleaning)

Step 3: The final step is the slumping of glass in the mold, for the glass to pick up the laser machined inverse feature, without picking up the graphite particles. The heat in the kiln is controlled by the firing schedule we enter.

Our firing schedule consists of three steps. 1) Stepwise heating with multiple heating rates to the slumping temperature, stepwise heating aimed to heat the glass slide evenly throughout. 2) Maintaining the kiln at the chosen slumping temperature for the chosen time to get the full feature in the glass without letting the graphite particles into soft glass. 3) Annealing the glass in order to get clear and stress relieved glass. The final process parameters and the firing schedule is:

Slumping temp: 1250°F +/- 10°F

Slumping time: 15 Min +/- 7 Min

Annealing temp: 921°F

Firing schedule:

Table 3 Firing Schedule

Step	Heat/Cool rate °F/hour	Target Temperature °F	Hold time (min)
1	356	1000	0
2	572	1200	60
3	1112	1250	15
4	9999	1013	60
5	140	921	1
6			



Figure 29 Resultant glass slide with microchannels

### 5.2 Validation

The process developed is used to produce microchannels in glass slides. The device was validated by assessing its usefulness as a scaffold for nerve regeneration, in support of the research of Dr Romero-Ortega of the University of Texas Bioengineering

Department. Two different approaches were used to validate the process for the production of the channels.

#### 5.2.1 Scanning Electron Microscope (SEM) Imaging

Images of the microchannels produced on soda lime glass slides were captured using SEM. The captured images clearly show the difference between the surface of the channels and the walls, validating the presence of channels. Images obtained from SEM are shown below [Figure 30Figure 31Figure 32]. Although the microchannels geometries fall in the specified range as outlined by the initial design [depth: 10 microns, width: 50-70 microns], surface roughness was similar to that of the mold material. However, comparing images of channels from [Figure 30] to that of mold [Figure 27] indicates the process is working, although it is evident that the glass surface formed is close to the surface roughness of mold.

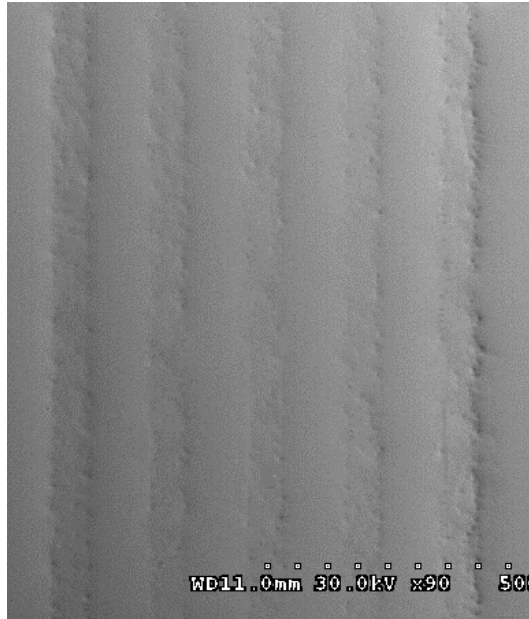


Figure 30 SEM image 1

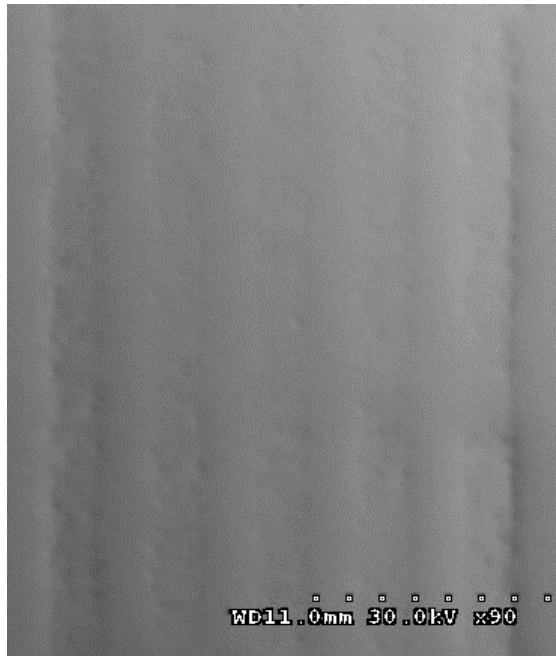


Figure 31 SEM image 2

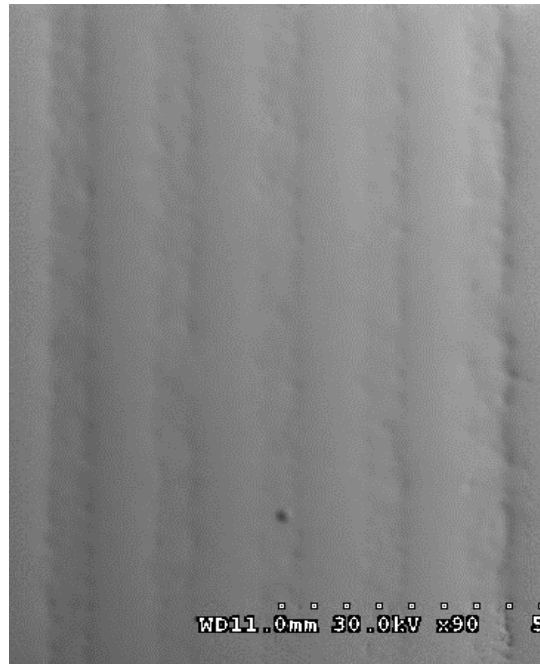


Figure 32 SEM image 3

### 5.2.2 Culturing primary cortical cells

Primary cortical cells from the brain of mouse embryos aged 15 to 18 days, were cultured in the wells (reservoirs) of the device developed. The intent was for the axons to grow through the microchannels to the other well. Added below are images of the device, microchannels and growing axons.

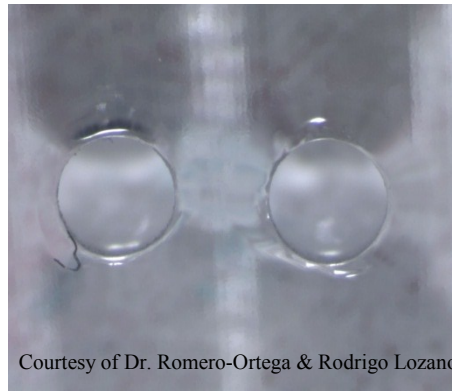


Figure 33 Device (two wells joined by microchannels)

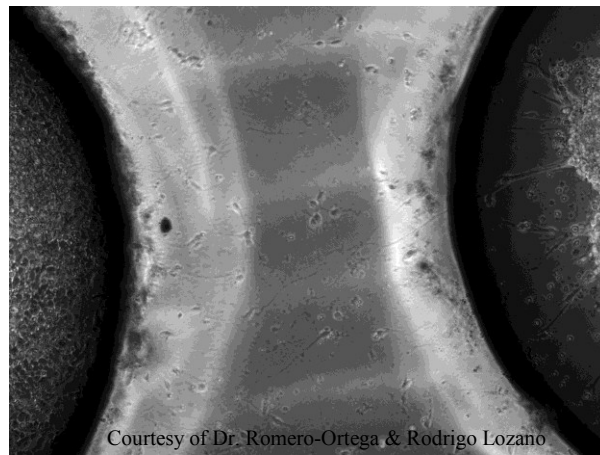


Figure 34 Channels between two wells (magnified)

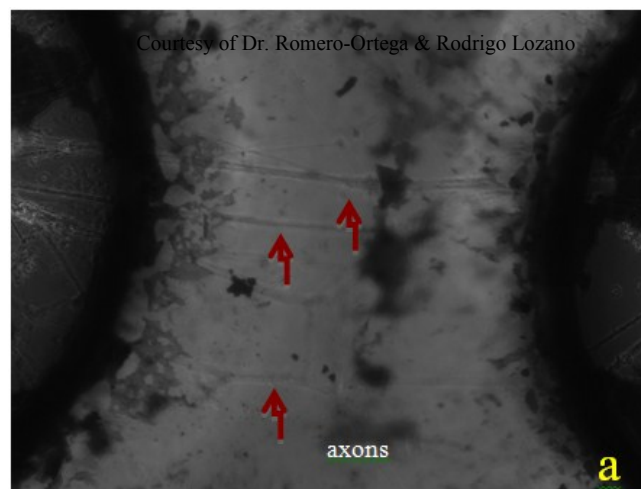


Figure 35 Axons growing through channels

Figure 33 Device (two wells joined by microchannels)Figure 33 is the device that is developed to the requirement provided by the research of Dr Romero-Ortega. It consists of two wells (reservoirs) connected by closed microchannels. Figure 34 is the magnified image of showing the microchannels connecting the two wells. The device was stained with a blue dye to help illustrate its functionality, in supporting axon growth. The dye was injected into the well on one side and the dye flowed through the channels and entered the second well. This was done to ensure there was no leakage between microchannels. The device was then cleaned and autoclaved before using to test the axon growth. In Figure 35 the growth of axons from one well to the other can be observed. The thick dark particles in the image are dye residue that adhered to the glass substrate. Since it has been autoclaved the axons grew without any disturbance. It can be observed that there are many other neurons growing randomly, which is due to the improper sealing of the channels leaving space between the two glass slides. It can also be observed that out of the four channels the axons were growing through three channels.

From the resultant images above we validated that there are channels being produced and axons and cells grow through them.

## Chapter 6

### Conclusions & Future Research

#### 6.1 Research Summary

This research started with the intent to develop a simple yet efficient inexpensive process for microchannel manufacturing in soda-lime glass slides. Microchannels in glass have wide scope in the field of biomedical research, replacing PDMS devices which come with multiple disadvantages. Soda-lime microscope glass slides, which are widely used for bio-related experimentation was considered as raw material.

Traditional glass working processes were studied with the intent to integrate them with engineering in the new process development. The glass slumping process from warm glass working processes was considered as a viable option in developing microchannels. A mold was developed, which serves as the negative of the channels to be formed in glass. Of the few compatible mold materials for glass, graphite was chosen as the mold material for our process. Stepwise mold machining was performed on two different machines, based on the channel dimensional requirement. Finally glass slides are slumped into the mold in a glass kiln.

Parameters for all the machines used in the process were finalized, based on a traditional trial and error method rather than a sophisticated design of experiments due to limited resources. A successful attempt was made to produce a product for biomedical research using the developed process. The process accompanies few limitations, which will be discussed in detail in next section.



## 6.2 Future Research

The new process developed for microchannel manufacturing required different raw materials, machines and machining processes. Involvement of different raw materials, machines and machining processes, always leave a wide scope for improvement. These are discussed below:

Mold material: SFG-2<sup>TM</sup> graphite currently being used as a mold material is working to satisfy the basic requirement. Yet there is opportunity to move to newer mold materials such as ceramics, which were briefly introduced in this work. A compatibility test was performed using a machinable ceramic as mold material, which was satisfactory and left for future research.

Parameter optimization: Using design of experiments to optimize the mold machining parameters and glass slumping parameters. Improving these parameters would increase the quality and consistency of results further.

Glass fusing: Fusing of glass slides is used to produce closed channels. Using the current parameters resulted in fusing, with air gaps between the slides and often improper sealing of channels causing inter-channel leaks. A new process of fusing was researched into, during the last phase which can be of help in improving the fusing process. Optical contacting is a technology used for producing clear optical instruments, which can be of help to improve the process.

New designs: The current process is successful in producing microchannels only up to 50 microns in width. Many biological applications require channels with

microchannels with width as small as 10 microns. The process can be further improved to develop microchannels of such smaller dimensions.

Well machining: Most of the microfluidics applications of bioengineering use wells to hold the medium. Incorporating well manufacturing into the process is a prospective future opportunity for research. Wells are currently produced by drilling with micro drillbits result in rougher walls. Rough wall surface tend to alter the characteristics of the medium being held in wells.

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