

Cite this: DOI: 10.1039/xxxxxxxxxx

## Development of a hot-embossed PMMA microfluidic flow focusing device for the generation of edible fibres

Kelvin Chow, Brady Gallant, Michael Mohan and Siwan Park <sup>a</sup>

Received Date

Accepted Date

DOI: 10.1039/xxxxxxxxxx

www.rsc.org/journalname

The field of Molecular Gastronomy is a rapidly growing area of cuisine, with the generation of scientifically inspired foods yet to exploit the benefits of microfluidic technologies. A wide variety of microfluidic flow focusing geometries can be utilized in conjunction with food grade ingredients for the generation of many interesting edible structures. Here, the design and fabrication of a multi-layer, 16 flow focusing microfluidic device is introduced. The device, constructed of three layers of poly(methyl methacrylate)(PMMA), is hot embossed using a series of epoxy molds in order to transfer channel features, after which, the layers are bonded using a temperature assisted solvent bonding protocol. The devices are then tested by applying different flow rates to observe a variety of edible fibers generated by the ionic cross linking reaction of sodium alginate and calcium chloride. The alginate flow diameter is hydrodynamically controlled by water as the sheathing flow until it exits into the bath of calcium chloride to yield the chemical reaction. Variations in these fibers observed were attributed to distortion from the hot embossing process and slippage during bonding of the layers. Once hot embossing and bonding protocols are further tuned, such a device will be applicable to the field of molecular gastronomy where it will be able to achieve real time generation of a wide variety of edible fibers.

### 1 Introduction

Microfluidics involves the science and technology of systems that have the capability to process and manipulate minute amounts of fluid ( $10^{-9}$  to  $10^{-18}$  liters), utilizing many well-known fluid characteristics in sub-millimeter and even sub-micron channels. These features not only allow for small sample sizes to be used but also offer higher resolution and sensitivity, low cost and footprint, and many of the inherent advantages of low Reynold's Number (laminar) fluid flow<sup>1</sup>.

Microfluidics often draws comparisons to the microelectronics industry, which has revolutionized they way we live and work on a day-to-day basis. Although there is still much research and development required, microfluidics has the potential to revolutionize the laboratory and the way scientific experiments are performed. One key difference between these two industries, however, is the scaling of physical effects. While electronics can be scaled down with virtually no change to the governing physics, fluid mechanics changes drastically as the system approaches microliter to nanoliter scales. An excellent representation of this occurs from non-dimensionalization and can be seen in the Reynold's number, shown in Equation 1.

$$Re = \frac{\rho u L}{\mu} = \frac{\text{Inertial Forces}}{\text{Viscous Forces}} \quad (1)$$

The Reynold's number is a comparison of the inertial and viscous forces in a fluid. In a microfluidic system, inertial forces become extremely small, resulting in small Reynold's numbers which typically are indicative of laminar flow. This is extremely favourable as it makes possible some analytical predictability of fluid behaviour, thus simplifying flow characterization, as the complexity introduced by turbulence is generally not present<sup>2</sup>.

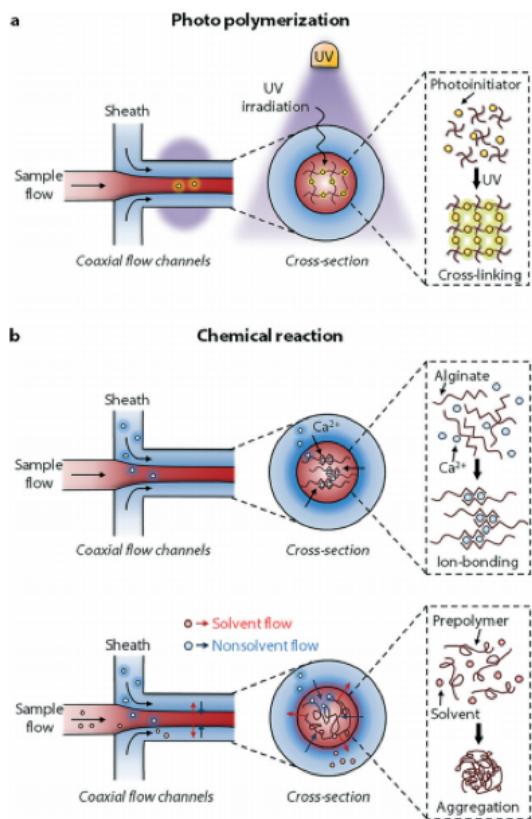
Soft lithography is one of the most common techniques for producing microfluidic devices, especially in academia. Soft lithography involves pouring an elastomeric material, commonly poly(dimethylsiloxane) (PDMS), over a silicon mold coated with a photoresist<sup>3</sup>. This method has many advantages, most notably its relative ease to perform. However, it does not lend itself well to large-scale production and is limited to elastomeric materials. Hot embossing is an alternative process that overcomes these limitations. In this case, thermoplastics are used as device materials. By heating the thermoplastic substrate above its glass transition temperature and applying a compression force, features can be stamped into the substrate<sup>4</sup>. Silicon molds are commonly used to emboss the desired features into the substrate<sup>4</sup>, however copper molds patterned with SU-8 photoresist are also used<sup>5</sup>.

In addition to chemical and biological applications, microflu-

<sup>a</sup> University of Toronto, 27 King's College Cir, Toronto ON M5S 1A1, Canada. Tel: (416) 978-1282; E-mail: guenther@mie.utoronto.ca

idic devices have also been used to extrude fibers from a number of materials and for various applications. One of the most prominent applications of these fibers is in tissue engineering. Collagen fibers with a smooth cylindrical shape and high mechanical properties which mimic those found in natural tendon and muscle have been produced by microfluidic devices<sup>6,7</sup>. Microfluidics produced fibers have also found potential applications in drug delivery<sup>8</sup> or as cell carriers<sup>9</sup>. The most common way to produce these fibers is through a flow focusing mechanism. In this setup, the desired material is dissolved in a solution and flows through a sheathing fluid, which induces polymerization and gelation of the fiber material. The sheathing action focuses the fiber solution, reducing the final fiber diameter and often inducing fibrillar alignment of polymer molecules within the produced fiber<sup>6,7</sup>.

One of the most common methods of introducing the sheathing flow and hydrodynamic focusing is through the use of a T-junction, as shown in Figure 1<sup>10</sup>.



**Fig. 1** T-junction flow focusing and fiber polymerization.

In this type of system, the polymer solution is flowed through a central inlet, which meets with sheathing flow at an intersection which forms a “T” shape. The sheathing fluid is allowed to flow through one or two separate inlets. A variety of mechanisms are used to induce polymerization of the fiber, including photopolymerization through UV light or a chemical reaction between the sheath flow and the polymer solution<sup>10</sup>.

In this project, the aforementioned principles will be used to produce fibers for molecular gastronomy. The fibers will be composed of an edible material and will resemble spaghetti. The mi-

crofluidic device design, methods, and materials will be discussed in this report.

## 2 Device Design

The 16 flow focusing (16FF) device was first modelled using Autodesk Fusion 360, due to its ease of use and added ability to generate tool paths for future CNC operations. The designs were then used to micro-mill prototype devices and finally PMMA casings containing these tested geometries for the generation of hot embossing molds. In all cases 1.5mm thick PMMA layers were used due to ease of machining while also maintaining durability. The top layer of the device contained 16 radially distributed channels that were 800μm wide and 400μm deep. The channels extended to the 16 matching 2.0mm ports that were located on the middle layer. The bottom layer then contained a distribution channel system that would allow for the sheathing fluid to reach all flow focusing geometries from a single inlet. The 16 radially located outlets also exit at the device wall to decrease the occurrence of clogging when gelation occurs. Finally, the 3.6mm inlets at the top and bottom layers allow for connection with P10 pipette tips which were in turn connected to tubing and syringe pumps.

### 2.1 Fabrication Methodology

#### 2.1.1 CNC Micro-milling and Laser Cutting

As previously stated, Fusion 360 was used to generate CNC tool paths for all models. This allowed for geometries on the epoxy mold casing to be micro-milled using the Tormach PCNC 770 milling machine via a .TAP file containing G-code, generated from Fusion 360. The device channels were machined with 1/32" (0.79375mm) square, four-flute end mills and the edges of the circular PMMA device were machined using 5/64" (1.98438mm) square, four-flute end mills. The blanks for the final hot embossing as well as the middle layer containing only ports were all laser cut with the Epilog Mini 24 laser cutter. The machine used a carbon dioxide laser and was able to quickly cut out material for multiple device layers in a matter of minutes, using a simple layout generated in AutoCAD.

#### 2.1.2 Hot Embossing

##### a. Epoxy Procedure

A high temperature, flexible epoxy was used to create the hot embossing stamp. The epoxy used was Duralco 4538 and was purchased through McMaster Carr. As per the Duralco data-sheet specifications, the epoxy could withstand a maximum temperature of 450°F (223°C), had a thermal expansion coefficient of  $3.4 \times 10^{-5}^{\circ}\text{C}^{-1}$ , and a hardness of 70 Shore A.

First, the epoxy resin and hardener were mixed at a mass ratio of 1:1.2 until uniform in colour. During the mixing process air is frequently incorporated into the mixture, and thus degassing was necessary for 30 minutes to 1 hour. The working time before the epoxy began to cure was 30 minutes, so degassing for intervals beyond this would cause problems with the epoxy mold. After degassing, the epoxy mixture

was poured into the milled PMMA casing. The epoxy was then placed in an oven set at 80°C. This step was used to reduce the viscosity of the epoxy, allowing the epoxy to completely fill all channels and produce a reasonably flat back face. The epoxy was then left in the oven for at least 2 hours. Once cured, the epoxy was carefully extracted from the milled PMMA casing. The flexible nature of this epoxy allowed for easy removal of the acrylic without the need for mold removal agents or non-stick coatings. After the epoxy stamp was removed, the back face of the epoxy stamp was manually filed to further improve the flatness, which was essential for good pattern transfer during hot embossing.

#### b. Hot Embossing

Hot embossing of the final PMMA device layers was carried out at the Center for Microfluidic Systems (CMS) clean room at the University of Toronto. The PMMA layers were placed on the fixed platen of the EVG 520HE hot embosser, protected with Teflon sheets on both sides to prevent temporary adhesion to the platens. Then, the upper platen was lowered on to the sample. A force of 1500N at 210°C was applied for 6 minutes and the sample was then subsequently cooled to a final temperature of 80°C. After pressing, the sample was removed from the platen and the PMMA layer was carefully detached from the epoxy mold. To prevent the PMMA layers from warping, the mold was left untouched for several minutes at ambient temperature before removal of the PMMA layer.

#### 2.1.3 Device Assembly

##### a. Solvent Bonding

The PMMA layers were bonded with 80% acetone diluted in de-ionized (DI) water. The sample was first cleaned with pressurized air, after which the acetone was applied between each layer using a pipette. The layers were then first placed between two thin sheets of rubber and two flat steel plates which allowed for protection of the device as well as equalization of the pressure distribution. A total weight of 70 pounds was then placed on top of the stacks to apply a pressure of 15 pounds-per-square-inch (103kPa). The sample and the weights were placed on a hot plate to maintain a temperature of 40°C for 20 minutes for each two-layer bonding cycle. Each process was limited to bonding only two layers to avoid the dislocation of the matching inlets and outlets. After bonding, any remaining solvent was removed by pressurized air.

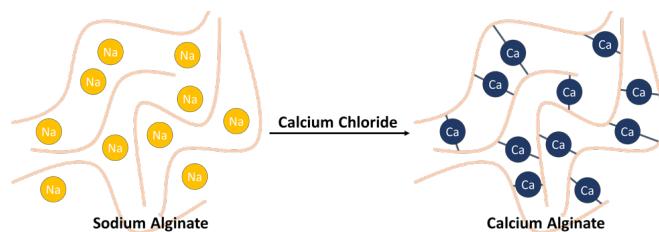
##### b. Tube Connections

The two main inlets for the device were connected with rubber tubes and bonded with epoxy steel (LePage). The bond possessed sufficient adhesion force to prevent leakage due to the inlet pressures of both flows during fiber formation.

#### 2.1.4 Fiber Formation

The sample flow containing 2% sodium alginate was allowed to enter the upper inlet of the device at a flow rate of  $5\text{mL min}^{-1}$

while the sheathing flow of water was allowed to enter the lower inlet at three times this rate ( $15\text{mL min}^{-1}$ ) in order to have a flow focusing effect on the sample flow. The reaction for creating a gel-like structure was then carried out by introducing sodium alginate solution into the calcium chloride bath. The sodium ion ( $\text{Na}^+$ ) would then bond with the oxygen strand to produce a flexible structure easily dissolved in water. When introduced into the calcium chloride bath, the sodium ion would be replaced by the calcium ions. Since calcium ions have two positive charges, two bonds between one calcium ion and two oxygen ions are formed, reducing the bond angle and increasing the rigidity of the strand, thus forming gel-like structures or, in this, case fiber structures. The chemical reaction is shown below along with an illustration of the reaction in Figure 2.



**Fig. 2** Illustration of ionic cross linking reaction between sodium alginate and calcium chloride to result in calcium alginate.

## 3 Experimentation and Quantitative Results

### 3.1 Device Characterization

In order to quantify the accuracy of the feature replication during the hot embossing process, an optical profilometer was used to image the embossed PMMA pieces to determine the amount of deviation from nominal feature dimensions. Consistency of the embossed features in the radial direction was also measured. Four measurements were taken for both the bottom and top layers, each at a different location. On average, it was found that the channel depth was  $350\mu\text{m}$  near the outer edge of both layers, and  $300\mu\text{m}$  near the center, resulting in a  $50\mu\text{m}$  variance in depth along the length of the channel. This is in comparison to a desired depth of  $400\mu\text{m}$  as indicated by the design schematics in Figure 6.

### 3.2 Fiber Characterization

Several different flow rate regimes and ratios of fiber to sheathing flow rates were investigated for their effect on the final fiber diameters. These different flow regimes and resulting fibers are shown in Figure 3. The resultant fiber diameters for each extrusion condition (numbered 1-4 as shown in Figure 3) were measured and shown in Figure 4.

Data sets were unavailable for condition one as the low flow rates did not consistently produce fibers. It was clear that condition four produced the most consistent fibers, having the smallest variation in diameter, both from fiber to fiber as well as at different locations along the fiber. Despite this, there was still some variability present which was suspected to be the result of



**Fig. 3** Comparison of flow rate regimes and resulting fiber quality.

defects during the PMMA hot embossing and bonding processes. Misalignment during hot embossing and bonding seemed to have caused some variation in the cross sectional areas of channels. Additionally, a consistent bond was not always achieved along the outer edge of the devices. This resulted in some outlets having larger effective diameters than others, thus producing fibers of varying diameters. In future work, it is recommended that hot embossing and bonding protocols be further optimized to reduce misalignment and achieve a consistent bond along the outer perimeter, thus producing more consistent fibers.

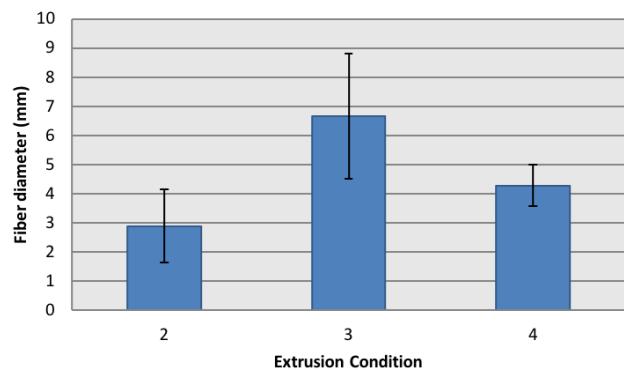
#### 4 Assessment of Scalability

One of the major advantages of microfluidics is the capability to scale designs to run parallel operations. The device design presented in this report shows 16 flow focusing units that are theoretically identical. The flow rates running through the device were as high as  $5\text{mL min}^{-1}$  for alginate and  $15\text{mL min}^{-1}$  for the sheathing fluid. These flow rate ratios correspond to a fiber diameter of  $300\mu\text{m}$  according to the following equation<sup>10</sup>:

$$R_s = R \left[ 1 - \left( \frac{Q_{sh}}{Q_s + Q_{sh}} \right)^{1/2} \right]^{1/2} \quad (3)$$

where  $R_s$  is the diameter of the sheathed fiber,  $R$  is the equivalent radius of the channel and  $Q_s$  and  $Q_{sh}$  are the sample and sheathing fluid flow rates respectively.

Based on the flow rates and the fiber diameter, the theoretical velocity of the alginate solution would be  $75\text{mm s}^{-1}$  in each channel. Thus, in order to produce one serving of Falooda, ( $\sim 1\text{m}$ ), it would take the microfluidic device approximately 1.2 seconds to



**Fig. 4** Plot of fiber diameter against extrusion condition number.

produce this amount under these operating conditions.

Since the device inlets were able to withstand pressures at this flow rate (and in excess), there is the possibility for further scaling by potentially adding more flow focusing units and increasing the flow rates of the solutions. Alternatively, the flow rates can be increased while maintaining the same ratios to also result in higher throughput due to the increasing velocities of the fluids.

Along with the scalability of the device design, the fabrication process used to create the device also has the potential for scalability. In comparison with other fabrication methods such as additive manufacturing and soft lithography, hot embossing allows for the shortest turnaround time for the creation of a single device. At the end of the project, a total of 10 working microfluidic devices were produced from the hot embossing process. Once a set of epoxy stamps were satisfactorily obtained, transferring the pattern on to a PMMA blank took approximately 40 minutes. Bonding then took approximately 30 minutes, and finally attaching inlets, another 10 minutes. Thus, a new device could be fabricated in under 2 hours if the manufacturing was performed as a serial process.

#### 5 Molecular Gastronomy Recipe

Falooda Kulfi is a popular dessert hailing from the subcontinent of south Asia. Kulfi is very similar to ice cream and is often served alongside Falooda, a sweet, paste-like delicacy. The microfluidic device was used to successfully form Falooda-like fibers from the ionic crosslinking of sodium alginate using calcium chloride. Figure 5 below shows side-by-side, commercially available Falooda Kulfi and a sample of Falooda generated from the device served with ice cream for demonstration.

The details of this reaction are discussed in previous sections. The water-based alginate solution was altered slightly to produce Falooda using microfluidics. The base recipe was as follows:

- 1% (wt) Sodium alginate powder
- 33% (wt) Granulated sugar
- 66% (wt) Water

All percentages were mass-based. The three ingredients were blended until all clumps were completely removed. The mixture



(a) Commercial image of Kulfi dessert with Falooda.



(b) Ice-cream pop served with device generated alginate strands closely resembling Falooda.

**Fig. 5** The chosen molecular gastronomy recipe - microfluidic falooda.

was then left to sit for a few minutes to remove bubbles created during blending. Finally, the sugar content was determined through an excruciating, highly qualitative tasting protocol to perfect the sweetness of the Falooda.

## 6 Conclusion

For the duration of the term, the group was able to successfully design and fabricate a working fiber generating device via the process of hot embossing. This involved first designing and validating the layout of each of the three PMMA layers, creating molds, hot embossing and finally bonding the devices before testing. While the generation of alginate fibers in a bath of calcium chloride was successful, the variations in fiber diameters across the 16 flow focusing geometries were determined to be due to distortion from the hot embossing process and slippage during bonding. Thus a future recommendation would be to further tune the protocols for hot embossing and bonding, in order to achieve a more uniform distribution of flow in all channels and thus by extension, the production of uniform fibers with controlled diameters. This device thus presents an initial step for the field of microfluidics into the area of molecular gastronomy for the production of a wide variety of edible structures.

## Acknowledgements

The completion of this undertaking could not have been possible without the assistance and resources provided by university facilities as well as professors and fellow classmates. The group would therefore firstly like to express their gratitude for the guidance and insight provided by Axel Guenther, the course instructor, regarding the design of the devices and the pitfalls associated with various fabrication techniques. Edmond Young, a professor of micro-engineered technologies was also able to provide advice on various bonding and mold making techniques as well as graciously allowing the use of IBMT Lab facilities for machining and fabrication. For this, the group is very grateful. Much of the final device creation would not of been possible without the use of hot embossing technologies provided by the Center for Microfluidic Systems and the invaluable contributions and motivation provided by Dan Voicu, the CMS manager. The group thanks him for his assistance with troubleshooting and for his excitement that kept the group pushing forward. Finally, the group would like to thank all fellow classmates of the course for their input, support and camaraderie throughout the entire process.

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## Supplementary Information

### Team Member Contributions

Each team member was able to apply a unique skill set to different aspects of the project, which resulted in a device that was efficiently designed and fabricated. The following is a detailed account of tasks performed by group members, listed in logical order of operations:

**Michael** Responsible for computer modelling of preliminary designs as well as CNC machining of initial prototypes and stamp mold casings. In collaboration with all other group members, also responsible for hot embossing of the final device and document handling.

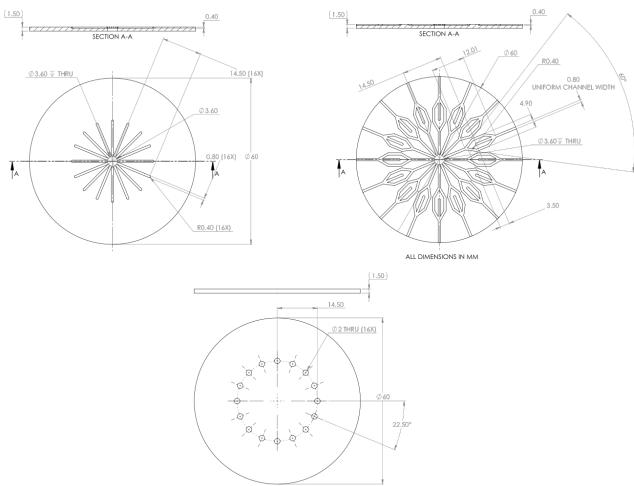
**Kelvin** Responsible for generation of stamps by carefully developing epoxy mixtures and performing curing, degassing and levelling procedures prior to hot embossing. Also responsible for leading all group testing activities during the fiber generation phase. Also collaboratively participated in hot embossing and document handling.

**Brady** Responsible for laser cutting of blank PMMA pieces in preparation for hot embossing as well as booking equipment and leading initial hot embossing recipe tuning. Also responsible for obtaining measurement data for diagnostics and collaboratively handling documentation.

**Siwan** Responsible for perfecting bonding techniques to reduce device leakage as well as assisting with preliminary device milling. Also collaboratively responsible for device testing and hot embossing as well as document handling.

## Detailed Device Design

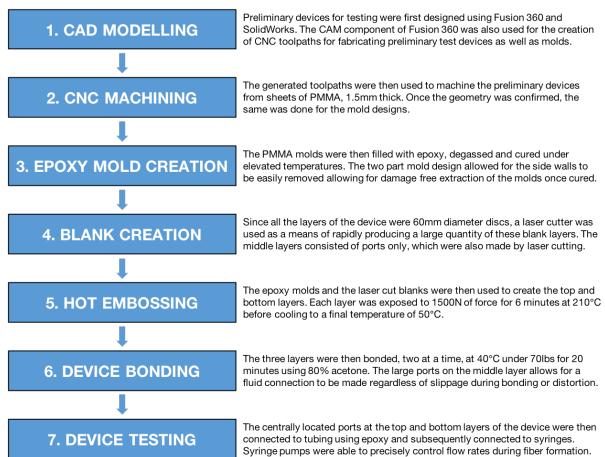
Figure 6 below shows the dimensions and layout of the 16 flow focusing device geometries on three layers of poly(methyl methacrylate) (PMMA).



**Fig. 6** Detailed schematics of the three layers of the device.

## Process Flow

Figure 7 provides a breakdown of the steps taken to produce the final device. Initially, all designs were modelled using Fusion 360 and SolidWorks. This allowed for easy manipulation of the geometry as well as the generation of tool paths for CNC machining at later stages. Once this step was finalized, some preliminary milled devices were made and tested, thus allowing for the molds to also be milled with these tested geometries. The molds were then made using epoxy and hot embossed against blank layers of PMMA that were laser cut from stock material. Finally these layers were all bonded using acetone and then tested at various flow rates to observe the quality and consistency of the fiber formation using sodium alginate and calcium chloride.



**Fig. 7** Details of the process flow throughout the project, starting from computer aided design and ending with the testing of the final device.

## Schematic of Experimental Setup

Figure 8 below shows the experimental setup for device testing. Syringe pumps are situated to the left, with tubes connected to the device, submerged in calcium chloride solution and contained within a beaker which sits on the microscope stage.



**Fig. 8** The experimental setup for testing the quality of fibers generated at various flow rates.