Development and Characterization of Wax Molds for 3D Microfluidic Applications

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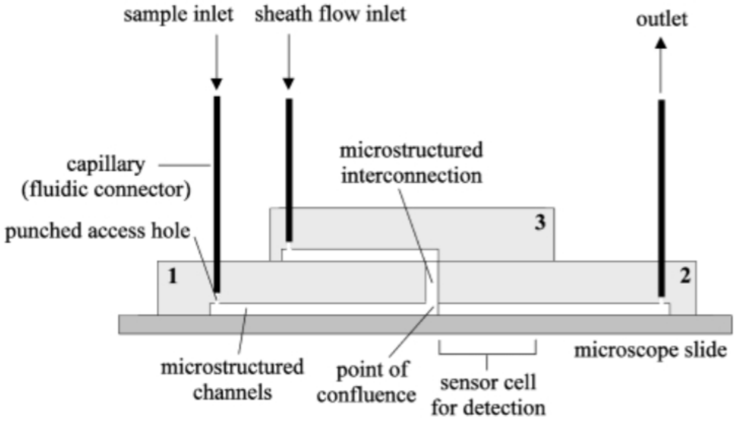
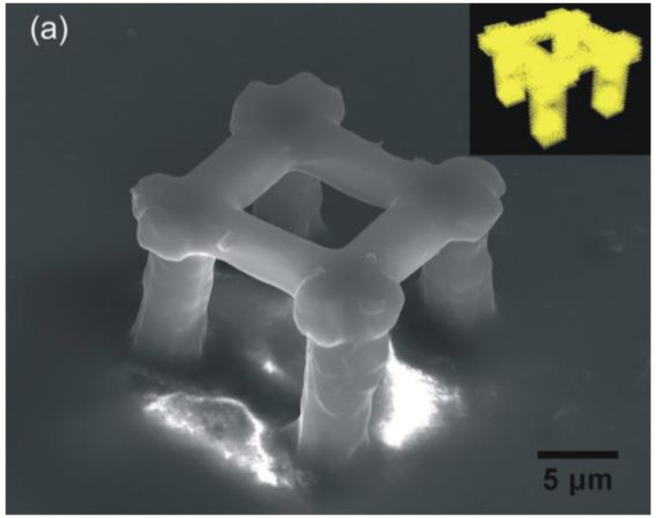
Introduction

- 2D microfluidics can be easily realized with known lithography techniques.
- “2d+” microfluidics: stacking laminate sheets (e.g. Paul Yager, University of Washington), vias between 2d devices
- 3D microfluidics require new techniques
  - Laser-induced curing of PDMS -- 5um resolution, but requires a pulsed femtolaser (Ref: Juodkazis, 2013)
  - **3d printed molds**





Ref: M Juodkazis anz, 2001.

3d Wax Printing

- Printing precise, one-time designs overnight, ”rapid” prototyping
- Printing truly 3d microfluidic molds (e.g. cannot be created without the use of support material during printing)
- Microfluidic applications
  - Precision valves
  - radial sheath flow for better flow focusing

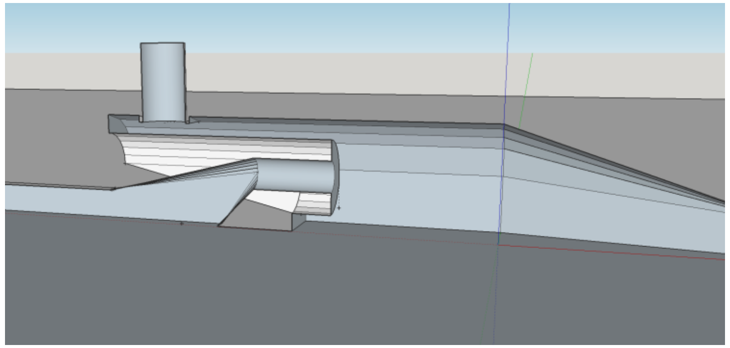
Solidscape Studio Capabilities	Nominal Capabilities	Measured Capabilities
Z-axis layer	~6 um	~14 um
Minimum feature size	250um	200um
Droplet size	76 um	~150um
Surface Roughness	0.9-1.6 um	0.6-1.2 um
X-Y positioning	197 dots/mm (~5um)	
Approximate Print Rate (including setup, auto-calibration)		5 min/mm^3 (very slow)

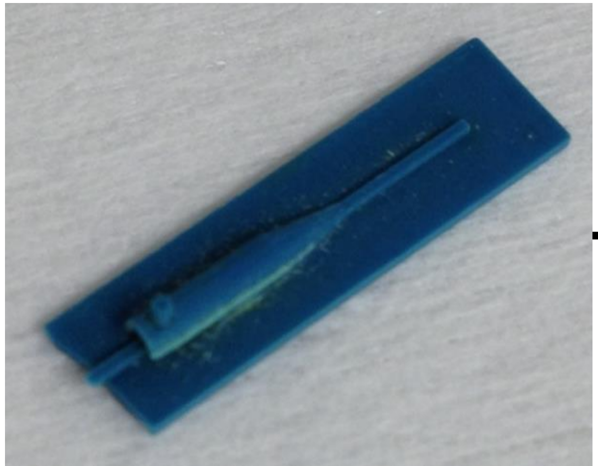
Overall Procedure:
1. CAD 3d model, export .stl
2. Run print overnight
3. Remove support material in print
4. Clean printed model

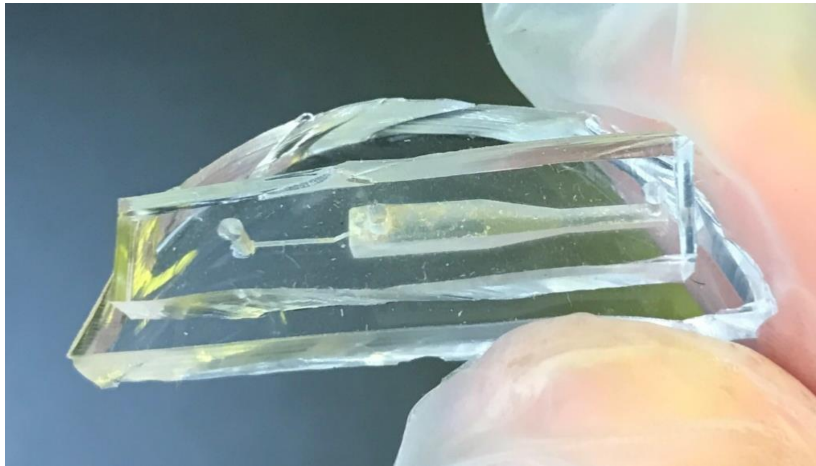
ExFab contains the **SolidScape 3ZStudio 3D Wax Printer.** This printer was originally designed for jewelers to create wax molds for casting.

Our Solution

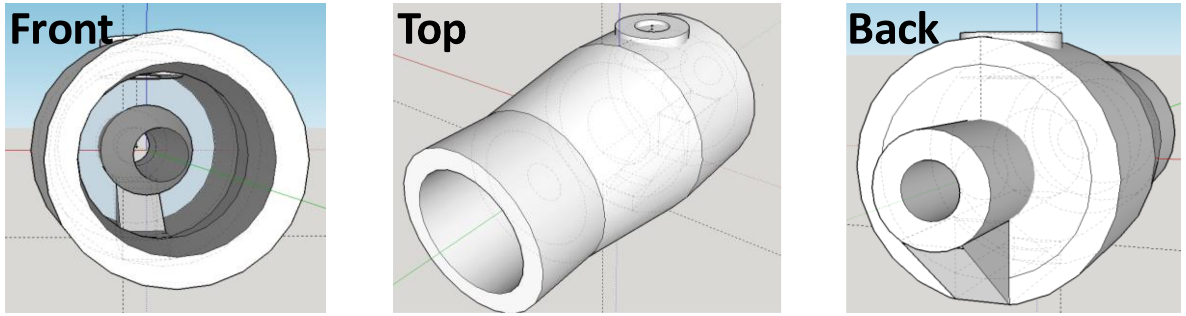
- Use wax printer to create inverted microfluidic mold ("print the flow")
- PDMS or epoxy poured over the mold to define channel
- Melt wax out of channel after casting
- Bond channel to substrate (e.g. glass slide)



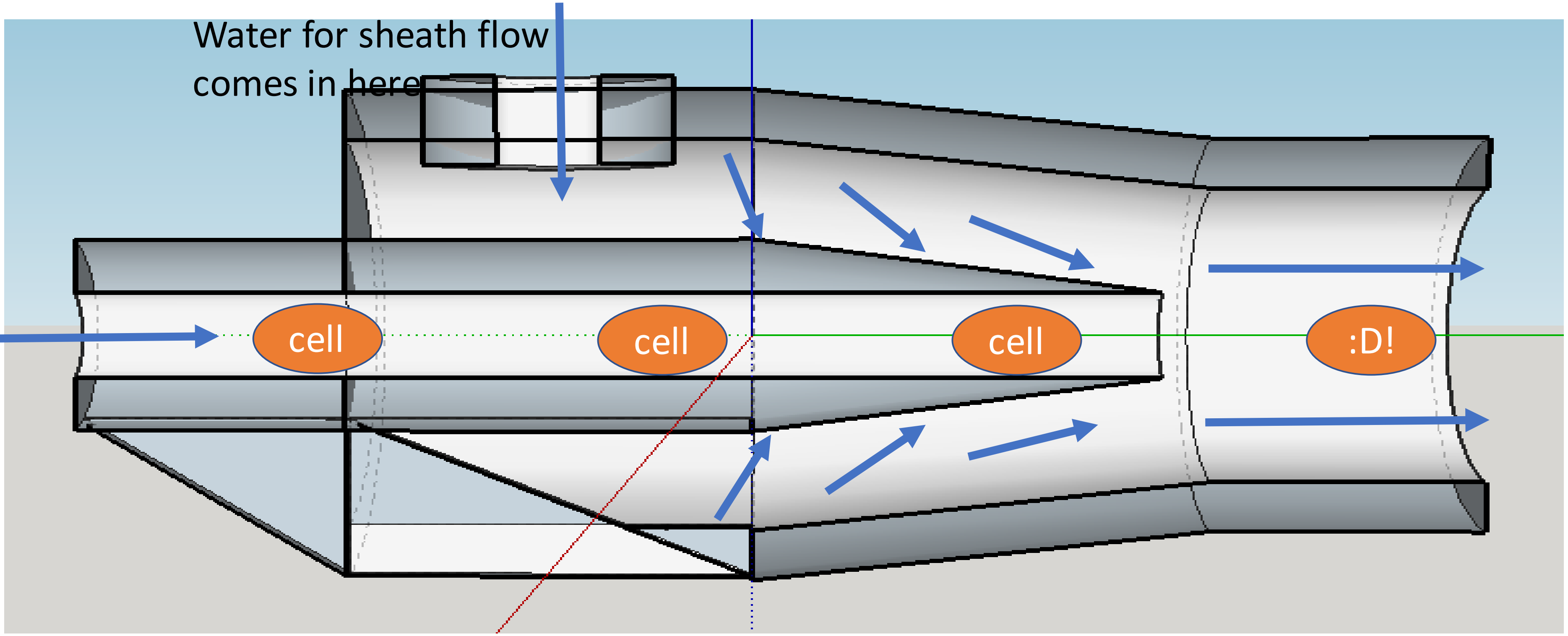




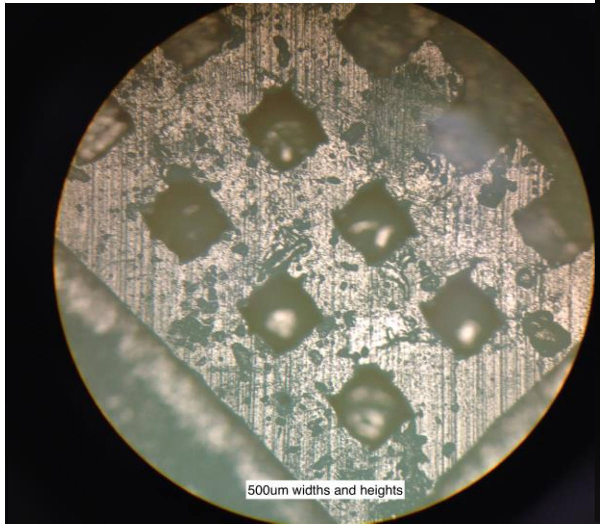
Application: Radial Sheath Flow

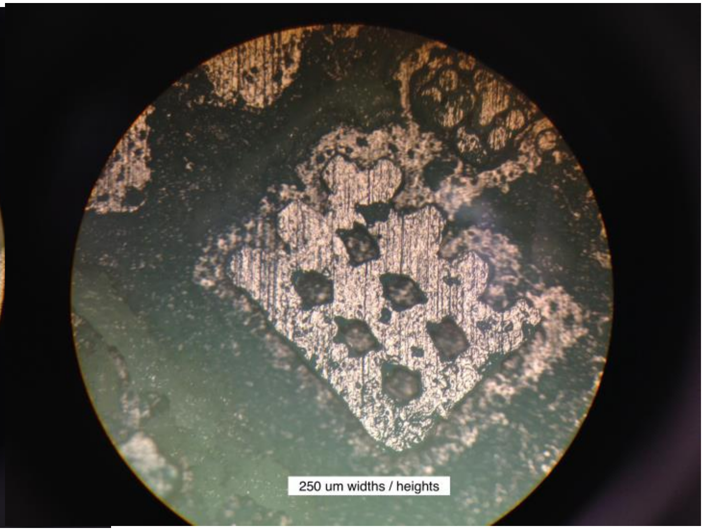


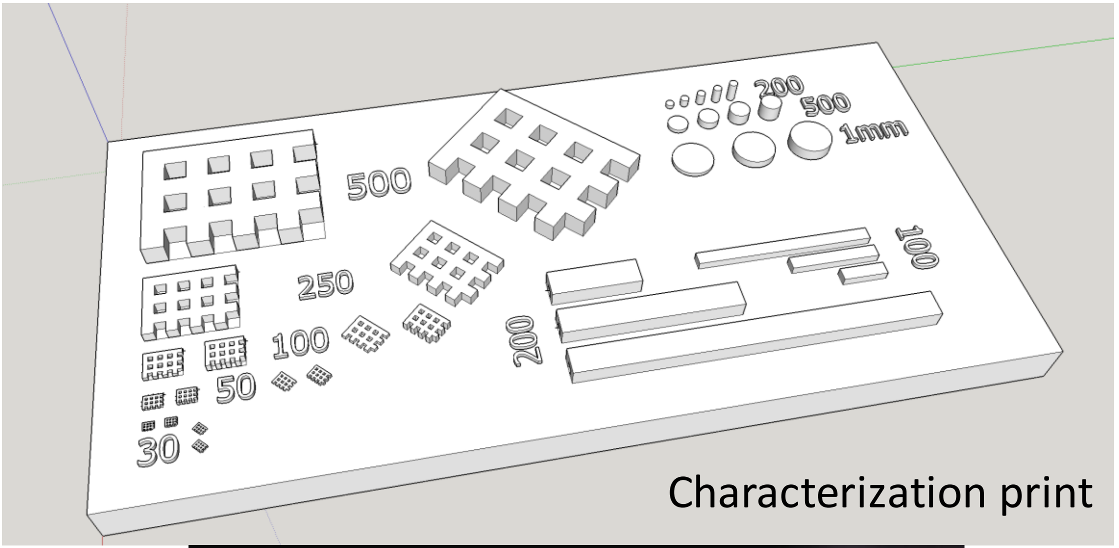
Cells come in through this connection point.



Characterization: Screening Test



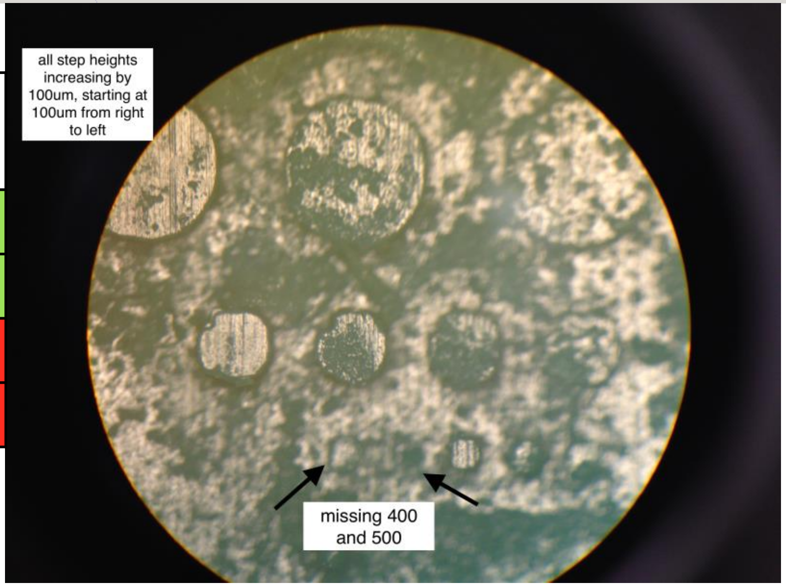




Characterization print

Grid Size (um)	Approximate Line Roughness (um)
500	30
250	37

Cylinder Aspect Ratio	Survived Post-Processing?
1	yes
1.5	yes
2	no
2.5	no

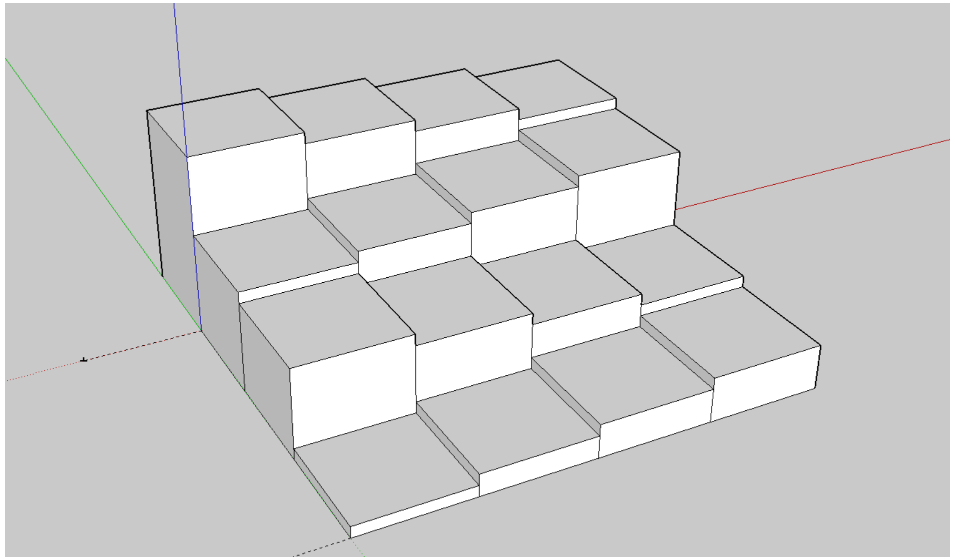


- **Agitation causes structures with aspect ratio > 2 to break**
- Angle of grids has little effect on performance.

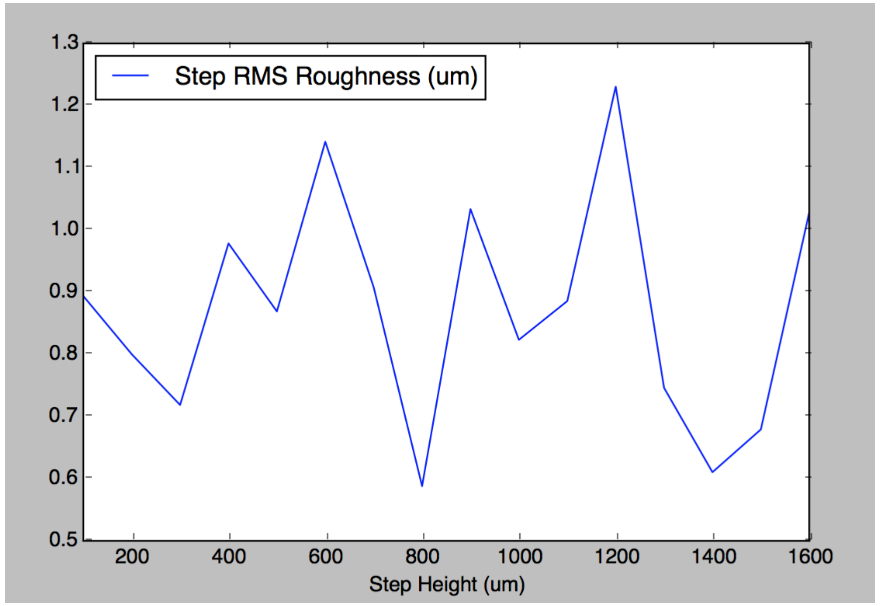
Characterization: Surface Roughness

Optical images show what appears to be surface roughness dependent on surface height (possibly due to error in vertical rastering with respect to ideal cad model) -> after testing, this theory proved wrong.

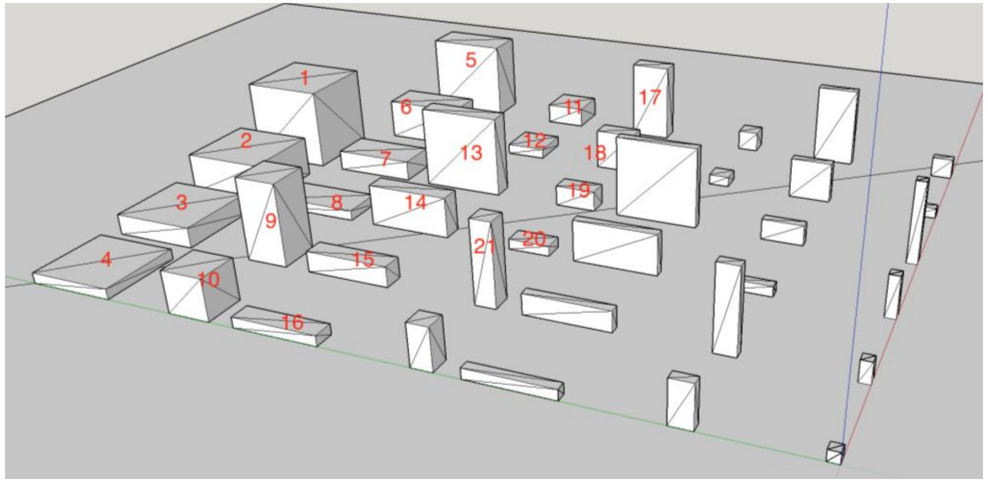
Surface roughness at various step heights -> no correlation



Step roughness characterization model



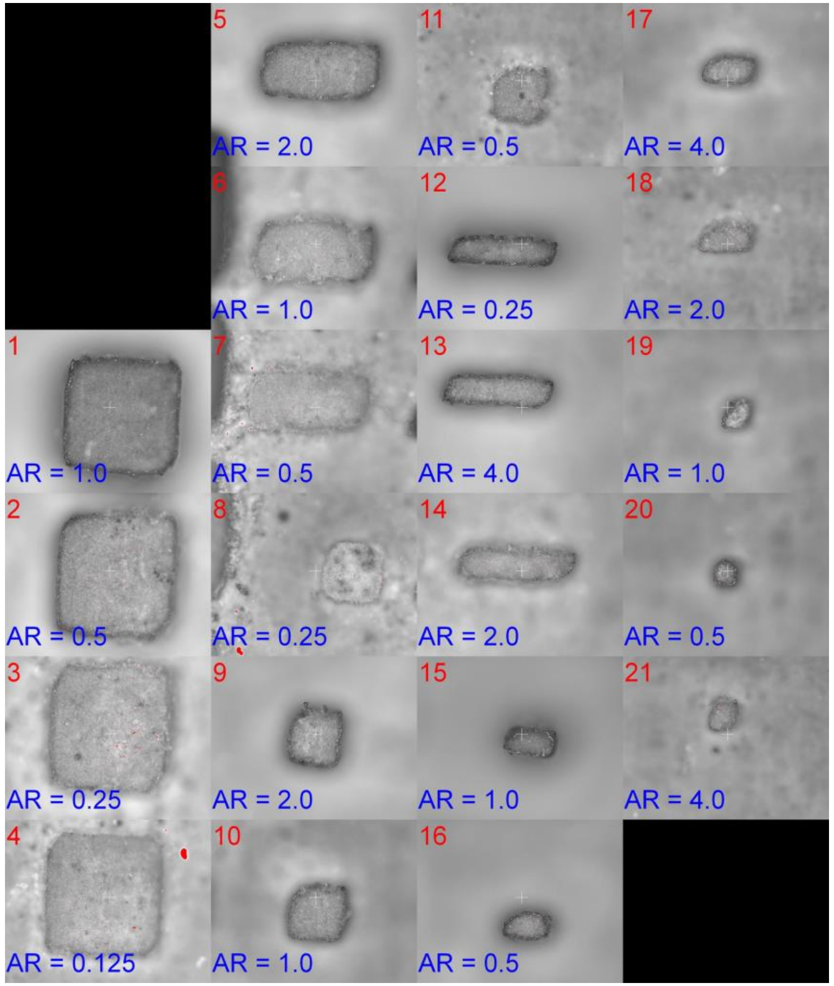
Characterization: Aspect-Ratio Test



Characterization print: various prisms

	5 0.4, 0.8, 0.8	11 0.4, 0.4, 0.2	17 0.2, 0.4, 0.8
	6 0.4, 0.8, 0.4	12 0.4, 0.4, 0.1	18 0.2, 0.4, 0.4
1 0.8, 0.8, 0.8	7 0.4, 0.8, 0.2	13 0.2, 0.8, 0.8	19 0.2, 0.4, 0.2
2 0.8, 0.8, 0.4	8 0.4, 0.8, 0.1	14 0.2, 0.8, 0.4	20 0.2, 0.4, 0.1
3 0.8, 0.8, 0.2	9 0.4, 0.4, 0.8	15 0.2, 0.8, 0.2	21 0.2, 0.2, 0.8
4 0.8, 0.8, 0.1	10 0.4, 0.4, 0.4	16 0.2, 0.8, 0.1	

Structure Incides, Dimensions (l,w,h) (mm)



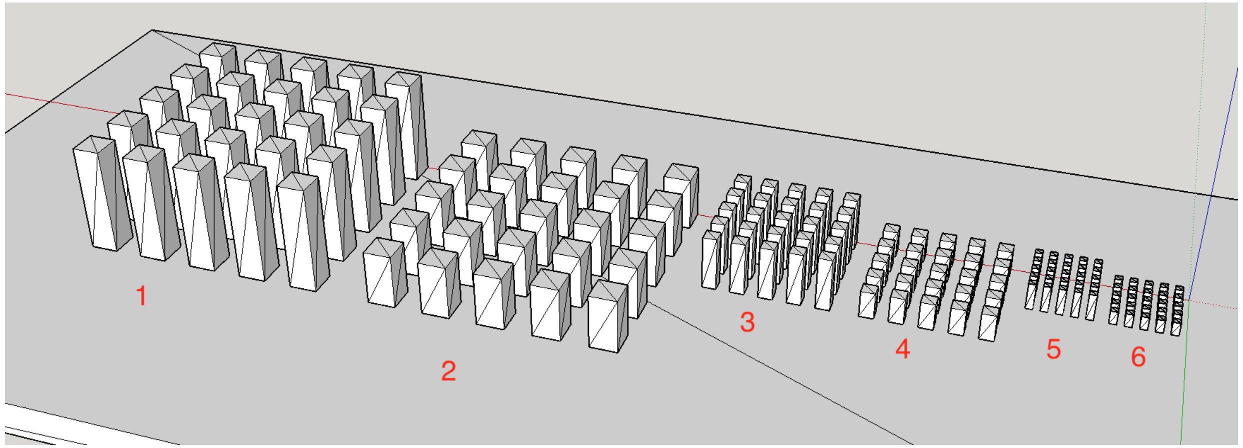
Optical images of top surfaces.
All images same scale, vertical aspect ratios given.

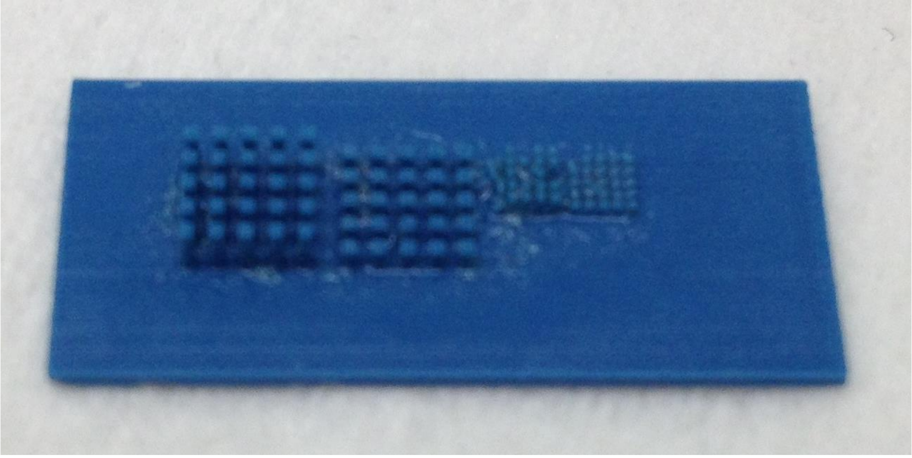
Printer can handle aspect ratio of 4, as long as minimum feature is >=200um.

As in the case of grids, squares lose their structure at smaller dimensions.

The remaining (<=100um) structures did not successfully print.

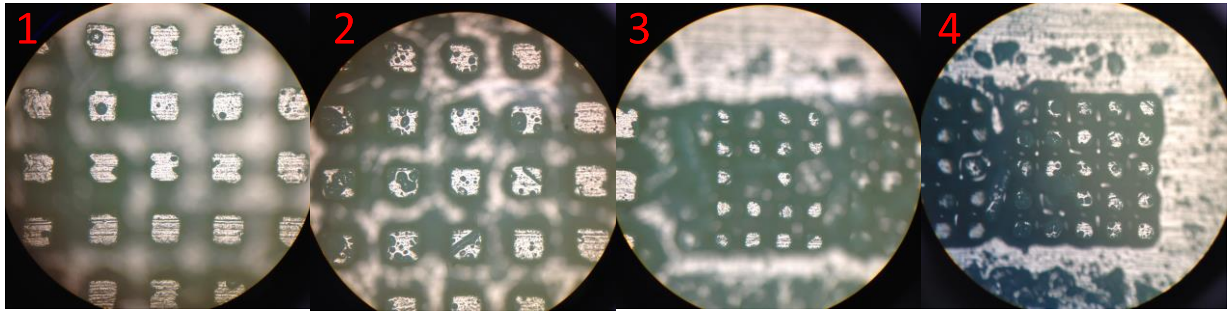
Characterization: Aspect-Ratio Test 2



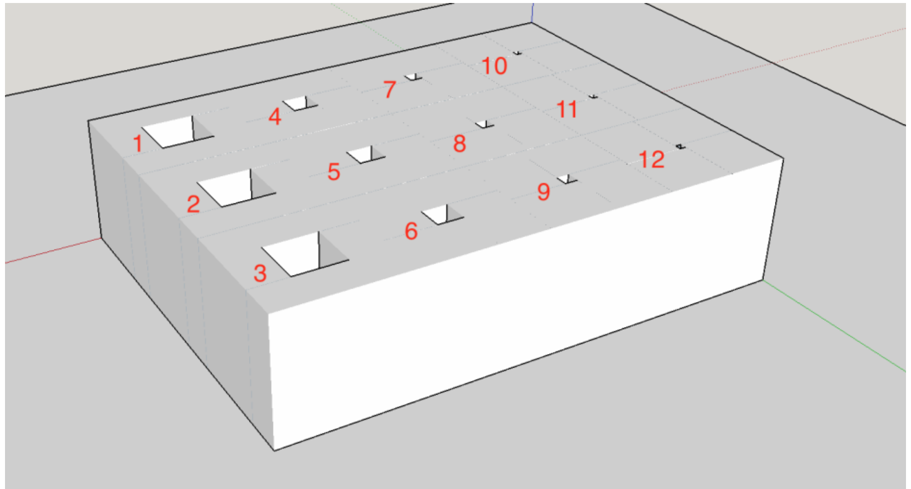


Structure Index	Pillar Dimensions (w, h) (mm)	Aspect Ratio	Yield
1	0.4, 1.6	4.0	100%
2	0.4, 0.8	2.0	100%
3	0.2, 0.8	4.0	68%
4	0.2, 0.4	2.0	72%
5	0.1, 0.4	4.0	0%
6	0.1, 0.2	2.0	0%

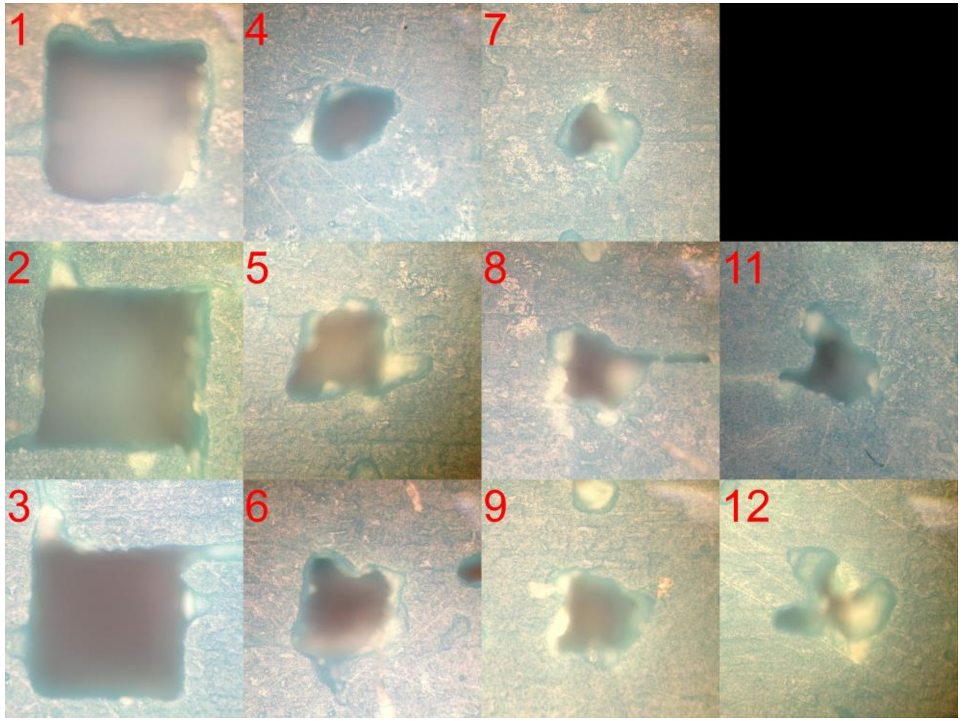
- **Last two structures did not print (100um width pillars)**
- **~70% yield for 200um pillars**
- Difficult to remove mineral oil from within pillars



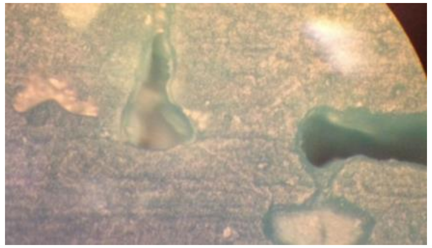
Characterization: Well Test



Structure Index	CAD width (mm)	CAD depth (mm)	Actual depth (mm), %depth
1	0.4	0.2	0.2 (100%)
2	0.4	0.4	0.4 (100%)
3	0.4	0.8	0.8 (100%)
4	0.2	0.2	0.2 (100%)
5	0.2	0.4	0.4 (100%)
6	0.2	0.8	0.4 (50%)
7	0.1	0.2	0.2 (100%)
8	0.1	0.4	0.4 (100%)
9	0.1	0.8	0.25 (31%)
10	0.05	0.2	DID NOT PRINT
11	0.05	0.4	0.24 (60%)
12	0.05	0.8	0.2 (25%)



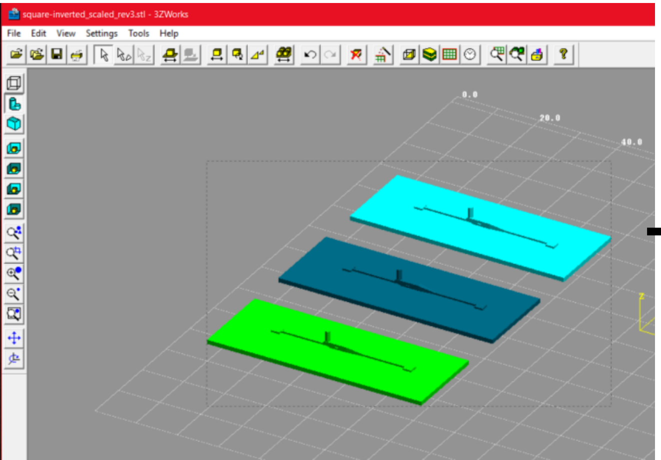
- **All support wax dissolved after light ultrasound, but >4 aspect ratio failed**
- Large pitting (~50um diameter) occurred in build material after ultrasound.

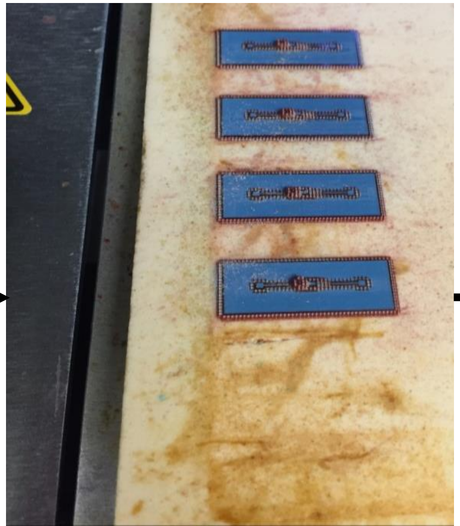


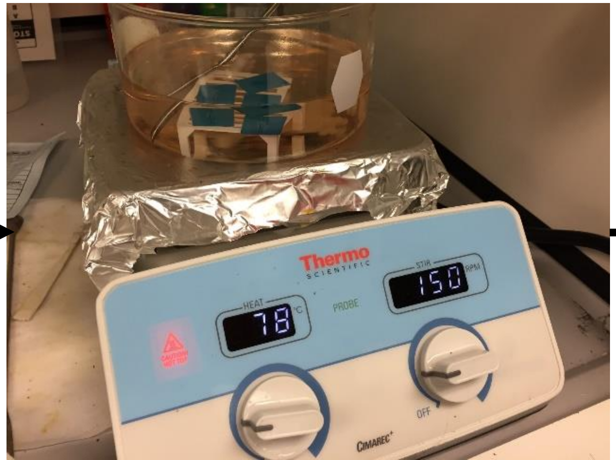
pitting

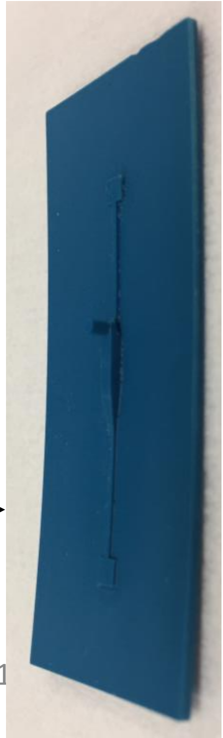
Procedure

- Desired STL files are loaded into 3ZWorks and the .3zs file is generated.
- .3zs file is loaded via USB Drive to the printer. Job starts.
- Once job completes, molds need to be scraped off of the build plate.
- Red support material removed via a mineral oil bath



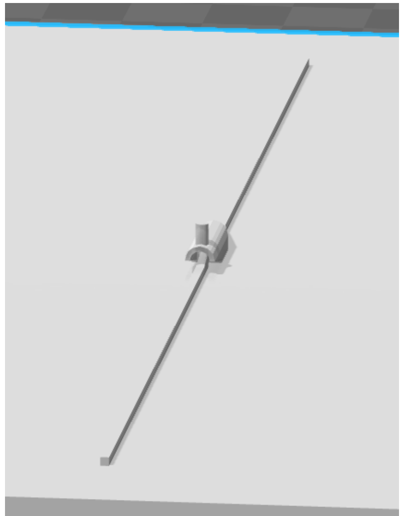


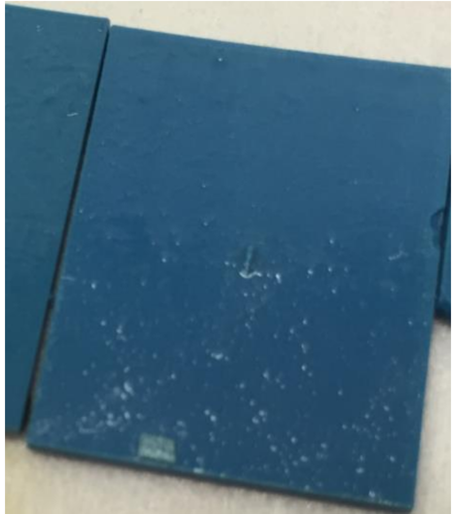


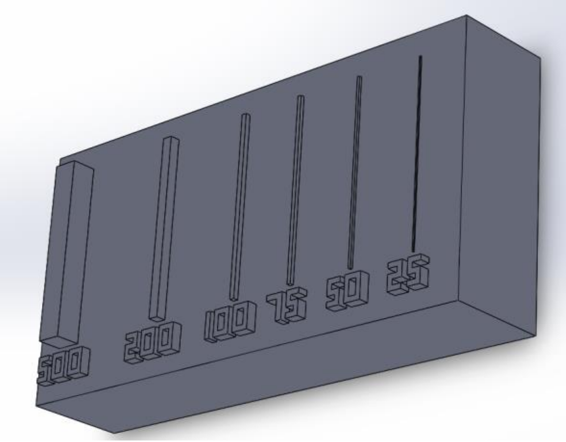


Challenges: Channel Thickness

- Although the printer advertises 6 um resolution, it was impossible to manufacture channels below 100 um in thickness.
- Previous characterizations of the wax printer did not print features below 250 um thickness.
- The 6 um resolution most likely is responsible for the smooth edges seen on round features.

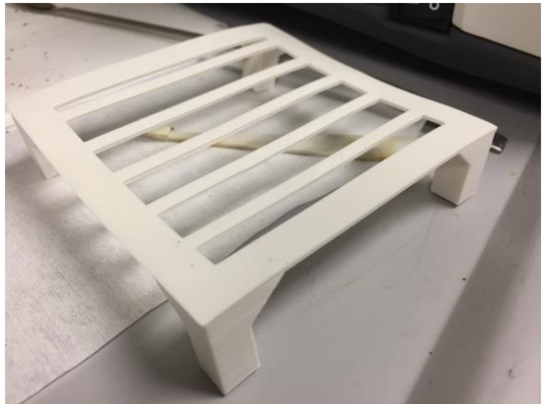






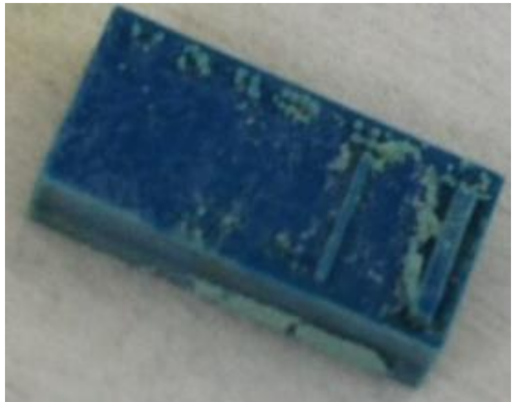
Challenges: Warping

- Caused by uneven temperature gradients when the molds are being submerged in the heated bath.
- Once warping occurs, it is irreversible, and the mold is rendered useless.
- Solutions:
  - Be sure not to leave molds in the bath no longer than an 1.5 hours.
  - Place molds on an elevated surface so that they do not touch the glass bottom.

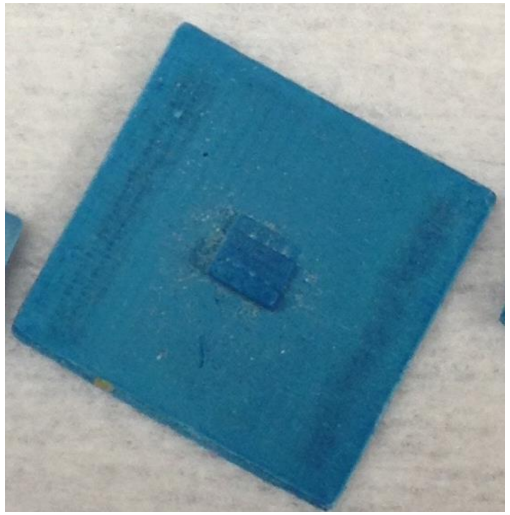


Challenges: Residue

- There are three main residues we encountered during this project that proved troublesome: brown thermal residue, blue build residue, leftover support material.
- Thermal residue results if molds are left too long in too cold a bath.
- Build residue results if vacuum bag on printer isn't replaced enough.
- Support material may take longer to remove in larger molds. Second dip is necessary.

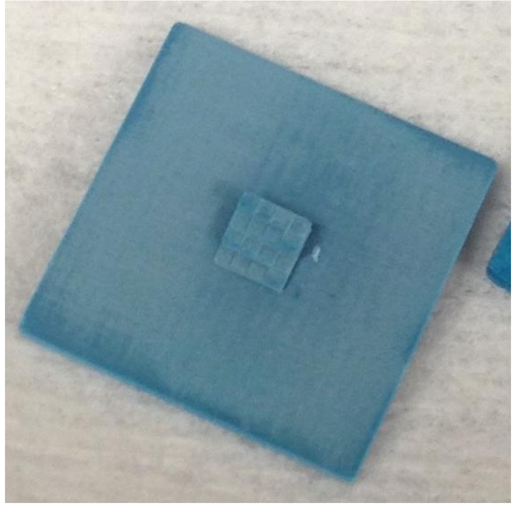


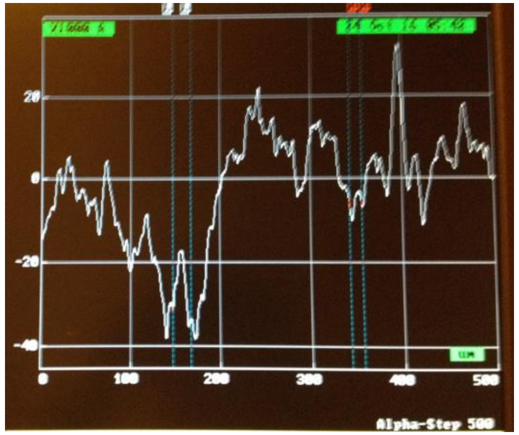
Mold Cleanliness: Two Outcomes

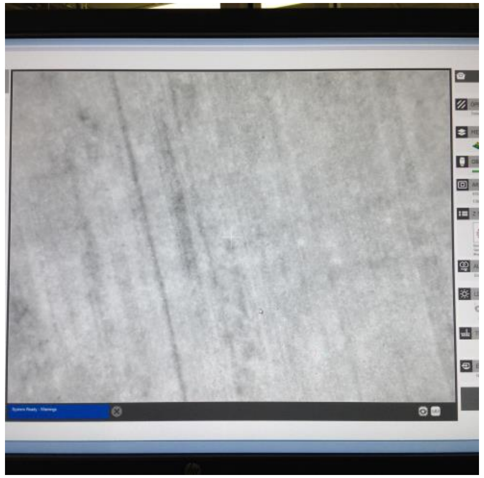


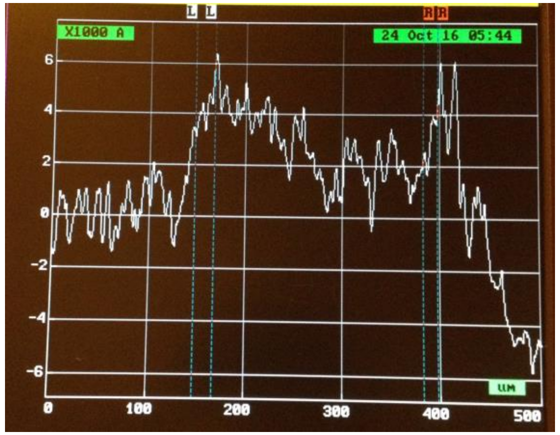
- **BAD**: Darker blue on surface - mineral oil absorbed by build material power generated during print
- Appears grainy under microscope
- ~20um roughness

- **GOOD**: Lighter blue - no powder
- Streaks under microscope show printer rastering
- **~4um roughness** (more cleaning needed to get 1 um)





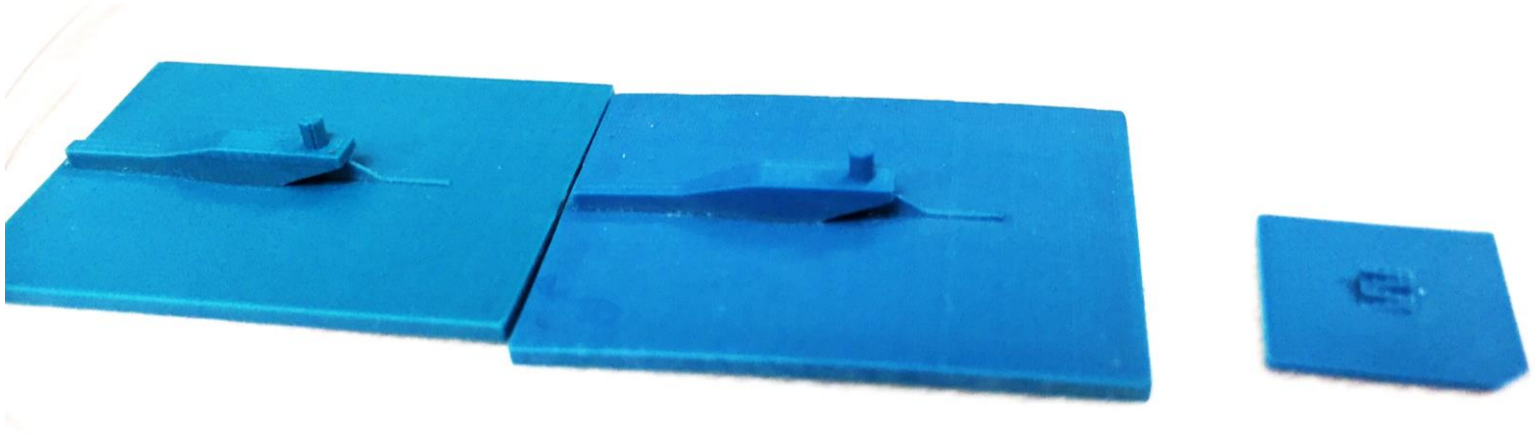




Results: Successful Molds!

I have a well paying job!

- Molds without warping, residue, or broken features were generated.
- A step-by-step guide for generating perfect molds has been created and will be added to the lab wiki.
- These molds may now be used to generate microfluidic structures.

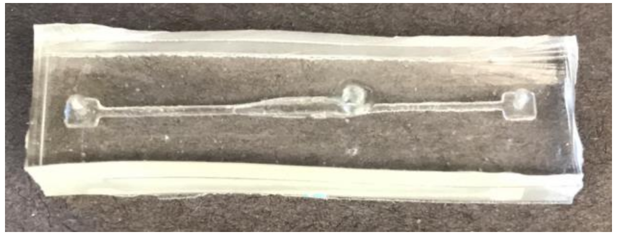


Fabrication of microfluidic channels

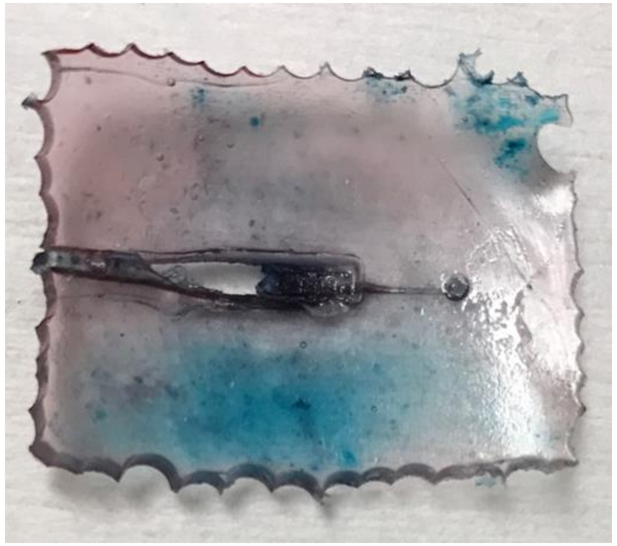
Materials	Description	Vendor	Uncured mixed viscosity (cps)	Tensile strength (MPa)	**Young's** modulus	Working time	Curing time and condition	Color
PDMS	Polydimethyl siloxane, prototyping polymer	Dow Corning	5550	2.2	360-870 kPa	NA	~2 hrs (@65ºC)	Clear
Conapoxy FR-1080	High temperature epoxy	Cytec Industries	2500	29	2.7 GPa	>2 hrs	4-16 hrs (@120ºC)	Light amber
5-Minute Epoxy	Rapid-curing epoxy	Devcon	10000	13	1.2 GPa	3-6 mins	0.75-1 hrs	
2-Ton Epoxy		Devcon	8000	16	1.5 GPa	30-35 mins	~2 hrs	
SU-8 3005	Epoxy-based negative photoresist	MicroChem	70	60-75	2.0 GPa	NA	100-350 mJ/cm2 UV radiation (exposure energy depends on thickness)	
SU-8 2025			5485					
SU-8 2075			27192					
SU-8 5			338					
SU-8 100			60000					

Fabrication of microfluidic channels

Material	Pros	Cons	Comments
PDMS	Soft Clear Easy to bond Thermostability	Too flexible for some 3D structures	Widely used for the fabrication and prototyping of microfluidic chips
Rapid-curing epoxy	Strong 3D structure Short curing time	Hard Fragile Bubble Wax removal problem Distorted when heating to 100ºC Hard to bond	Adding organic solvent, such as ethanol, can decrease stiffness and increase curing time for bubble removal. But most organic solvents can dissolve wax mold.
SU-8	Strong 3D structure Photocuable (radiation curing) Thermostability	Expensive (relatively) Bubble Hard to bond	There are a series of SU-8 with some different performances. But as a epoxy-based material, SU-8 has a lot of similar properties with normal epoxy.



PDMS channel



Epoxy channel

Channel test

- Experimental Setup
  - Inner flow: 0.05-0.2 mL/hr, 200-400 µm
  - Sheath flow: 1-2 mL/hr, 1-2 mm

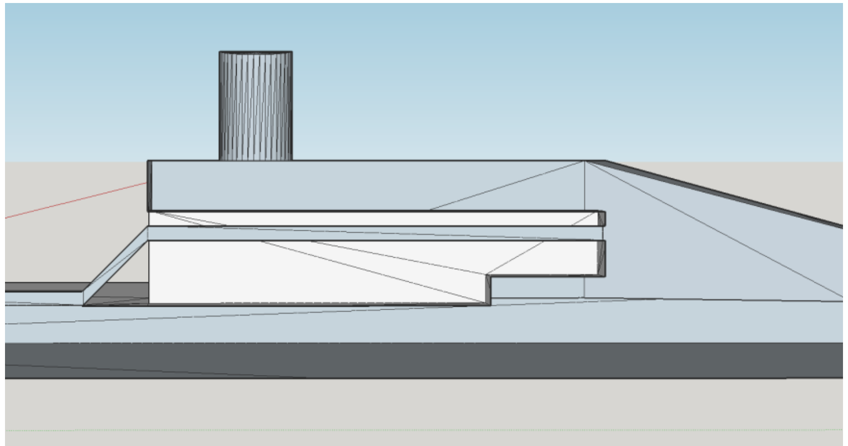
- Calculation and estimation
  - Reynolds number
    - Maximum Re ≈ 0.8 << 2000
    - Laminar flow
  - Péclet number
    - Pe ≈ 64000
    - Fluid remains largely unmixed

$$\mathrm{Re} = \frac{\rho v L}{\mu}$$

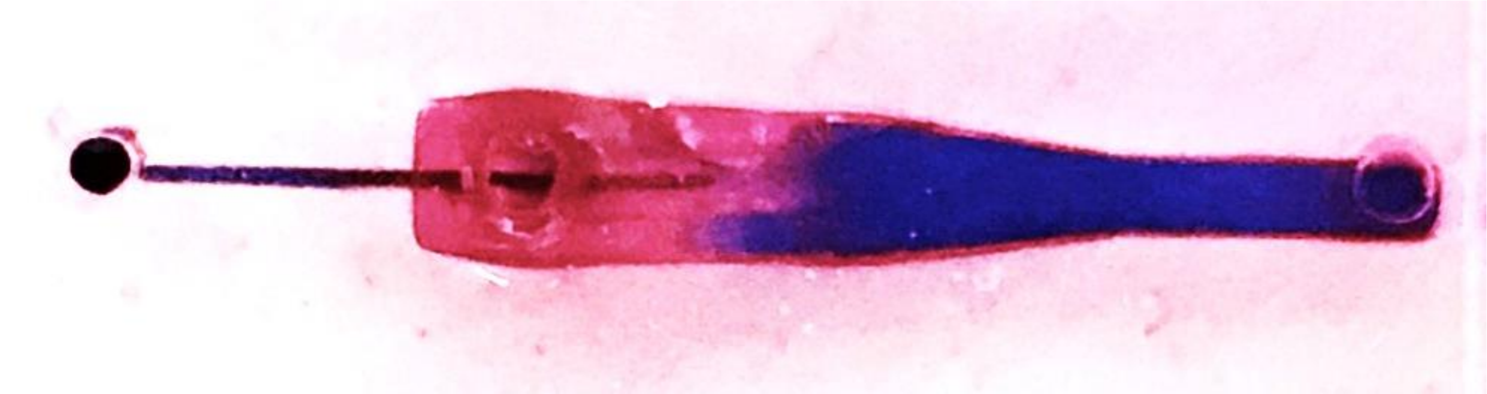
$$\mathrm{Pe} = \frac{\text{advective transport rate}}{\text{diffusive transport rate}}$$

$$\mathrm{Pe}_L = \frac{Lu}{D} = \mathrm{Re}_L\,\mathrm{Sc}$$

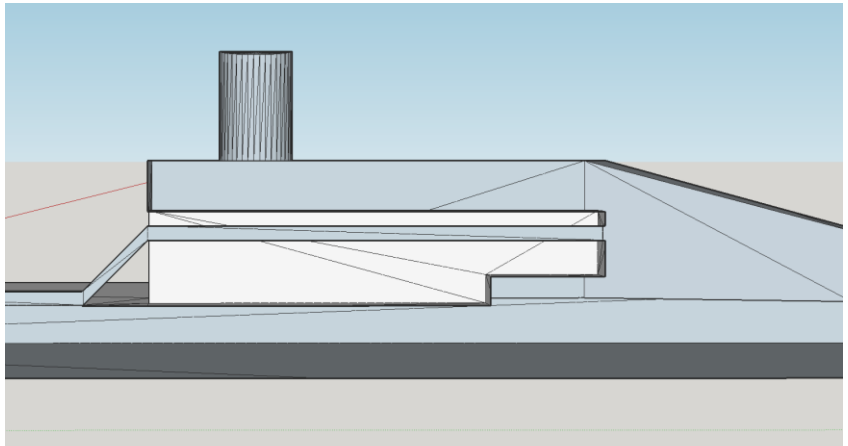
Channel test



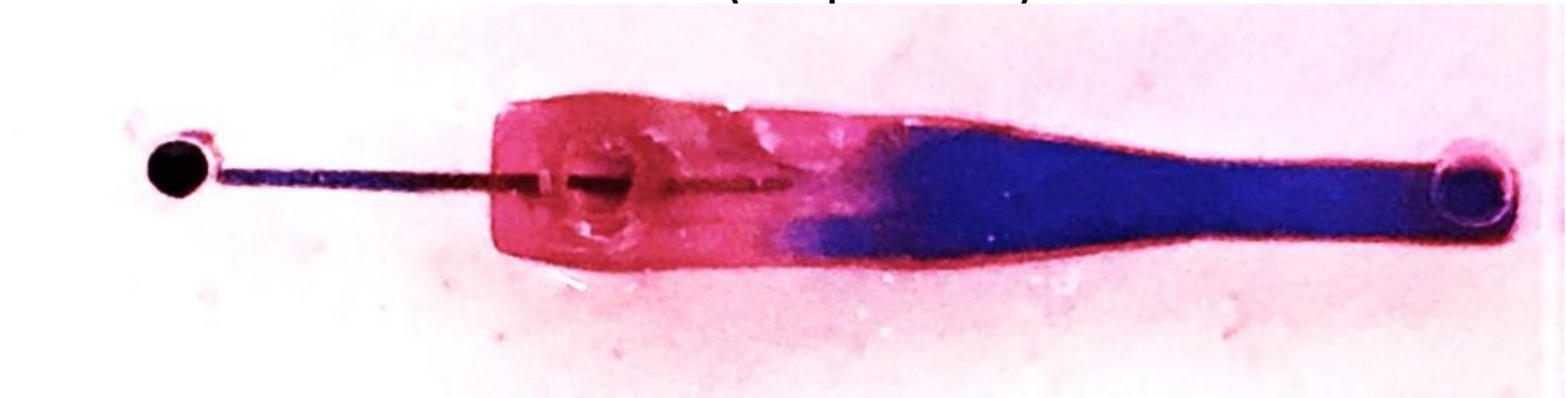
- Result
  - Laminar flow
  - Sheath flow in vertical direction



Channel test



- Problem
  - No radial sheath flow
  - Big diameter of sheath flow channel
  - Flexible inner flow channel (suspended)



Channel test

Traditional microfluidic channel fabrication	Our method using 3D wax printer
• Time-consuming (~1 week) • 2D (stacking for “2D+”) • High resolution (~5 μm)	• Rapid (~1 day) • 3D • Low resolution (~200 μm)

- Conclusion
  - Wax printer is a great tool for rapid prototype at large scale.
  - 3D microfluidic device can be fabricated using wax printer.
- Future plan
  - Add more symmetric support for the suspended channel
  - Decrease the diameter and flow rate of the sheath flow
  - Iterations for channel design

Thank you!
Q&A

- Reference

    - Hofmann, Oliver, Philippe Niedermann, and Andreas Manz. “Modular Approach to Fabrication of Three-Dimensional Microchannel Systems in PDMS—application to Sheath Flow Microchips.” *Lab Chip* 1, no. 2 (2001): 108–14. doi:10.1039/B105110P.
    - Rekštytė, Sima, Mangirdas Malinauskas, and Saulius Juodkazis. “Three-Dimensional Laser Micro-Sculpturing of Silicone: Towards Bio-Compatible Scaffolds.” *Optics Express* 21, no. 14 (July 15, 2013): 17028. doi:10.1364/OE.21.017028.