

Electrode filling using capillary action of 3D printed elastomer microchannels

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ABSTRACT

Soft polymer actuators are in increasing demand due to their more fluid like motion and flexibility when actuated than compared with rigid actuators, which makes them valuable in diverse engineering applications. One of the main types of soft polymer actuators is the dielectric elastomer actuator, whose working principle is to apply a voltage potential difference between electrodes to reduce the thickness of the elastomeric material while expanding its area. This paper looks at manufacturing a micro soft polymer dielectric elastomer actuator utilizing two-photon polymerization 3D printing. The actuator contains micro channels that are filled with an electrode by using capillary action. A complex helical geometry is designed, printed, and tested for electrode filling capabilities. Quite a few obstacles are described in this paper including the use of a newly released two-photon polymerization resin which has limited supporting resources, as well as the complex helical geometry having a large compliance that vastly complicates its fabrication, post-processing, handling, electrode filling, electrode integration, and actuation testing. However, these challenges are overcome by using the standard printing recipes currently available for the resins, adding electrode isolation layers, and printing thicker elastomer zones for more structural support. The results found solidify the approach of filling microchannels with electrodes through capillary action and lead to further the focus and creation of multi-functional micro soft actuators.

Keywords: two-photon polymerization, dielectric elastomer, additive manufacturing, capillary action, printing, microstructures

1. INTRODUCTION

Actuators are needed and used every day, but many are stiff and lead to jolting/specific rigid movements. To make movements smoother, more fluid like, and tunable gives rise to soft actuators. These types of actuators have a much better compliance to them and are more dynamic than most rigid actuators, making them perfect for biological interaction and mimicking^{1,2}. However, replicating and manufacturing soft actuators is a challenge due to the complexity of the soft components¹. This complexity is increased when moving into the micro scales. This is especially true of dielectric elastomer actuators (DEAs), which utilizes two or more compliant electrodes with elastomers to convert a voltage potential difference to mechanical work through electrostatic pressure. In order to actuate, the elastomer between the electrodes needs to be very thin. The thicker the elastomer, the higher the voltage is needed to actuate, e.g., thickness doubles, actuation voltage basically quadruples.

Technology now is able to fabricate small microstructures using 3D printing techniques. One such technique is two-photon polymerization (2PP), which utilizes a femtosecond laser to convert red light to ultraviolet (UV) light at a specified focal point, allowing for polymerization of specific UV curable resins in the micro and nano scales. Fabrication of elastomer is possible with this technique while using IP-PDMS 2PP capable resin (Nanoscribe). Being able to fabricate the elastomer with this technique gives extremely small feature sizes, sub millimeter, which would also allow for lower voltages to be used during actuation of the DEAs.

Even though the elastomer is able to be fabricated with small complex geometries, getting the electrode inside is the major challenge and is relevant for actuators and sensors with the need for compliant electrodes that are usually too difficult to handle³. Firstly, removing excess resin inside of the fabricated microchannels is usually a lengthy process waiting hours to days of development time, especially for high viscosity resins^{4,6}. One novel approach to removing excess resin out of thin fragile enclosed microchannels is explored using a low-pressure vacuum chamber during development process. Other

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methods to aid in the removal of liquid resin were taken into consideration before deciding on the aforementioned vacuum method. Sonication is able to assist in removing remaining resin from microchannels very effectively; however, this process is very harsh⁷. Applying sonication causes excess stress on the printed parts and has caused the parts to be removed from the substrates. In the case of soft polymers, this method has either induced or aggravated fractures in the material, as seen in Figure 1. For this reason, sonication was ruled out as a method of removing resin and the next two options were either letting the parts sit in developer at elevated temperatures⁴⁻⁶ or trying to force the resin out and developer into the channels by inducing a vacuum with the printed part inside and outside of developer. The vacuum method was chosen for speeding up development times and enabling quicker cleaning of tested parts for retesting, which all permits quick redesigns if necessary.

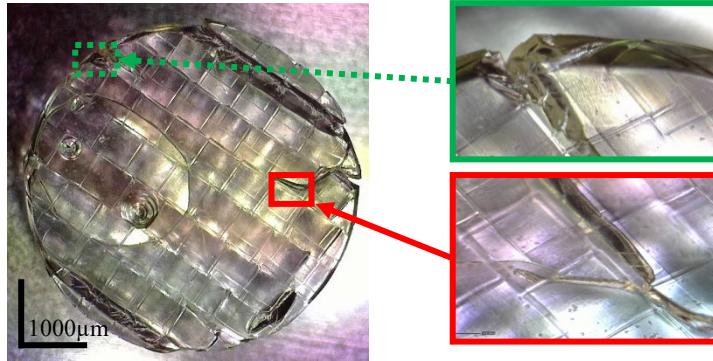


Figure 1. IP-PDMS resin printed cylinder with block stitching and removed from substrate using sonication with zoomed in defects caused/exacerbated by sonication.

liquid, and surface tension between the channels and the electrode material using 2PP technology, with a goal of producing soft polymer microchannels usable for DEAs and tests the capillary filling technique using liquid electrode material.

2. METHODS

Multiple fabrication experiments performed with IP-S (Nanoscribe) and IP-PDMS (Nanoscribe) resins using 2PP capable Nanoscribe Photonic Professional GT2 printer. The parameters used for printing 2PP prints on indium tin oxide (ITO) coated glass slides using the 25x lens with a 0.8 numerical aperture (NA) can be seen in Table 1. Thin microchannel bridges along with microchannel walls were printed with IP-S photo resin and double helix microchannels were printed with IP-PDMS photo resin. The pre-cleaning of the ITO glass substrates was performed first by a heated sonication bath in deionized water for 2-5 minutes, then moved into a heated sonication bath in 99.5% acetone for 1-3 minutes and then into a heated sonication bath in 99.9% isopropanol for 2-8 minutes. Afterwards, the substrates are blow dried and put into an oxygen plasma chamber with the ITO side face up to activate the surface. For the IP-PDMS printing, after the oxygen plasma activation, the ITO is placed into a small dish of 10 mL of acetone and 50 μ L of 3-(trimethoxysilyl)propyl methacrylate for 1-2 hours after which it is rinsed with acetone and deionized water and blow dried.

Table 1. Parameters used for 2PP printing on ITO glass with 25x objective.

Resin	IP-S	IP-PDMS
Slicing, μ m	1	0.3
Hatching, μ m	0.5	0.3
Base Count	6	
Contour Count		20
Core Power, mW	50	40
Base Power, mW	40	
Contour Power, mW		30
Core Writing Speed, μ m/s	100,000	100,000
Base Writing Speed, μ m/s	100,000	
Contour Writing Speed, μ m/s		20,000

Once the channels have been cleaned out, they need to be filled with liquid electrodes. There are a few different filling techniques enquired, such as vacuum filling/pressure filling⁸ or utilizing capillary effect⁹⁻¹¹. Each technique has their advantages and disadvantages. Filling in electrodes using vacuum filling is difficult when the electrode material evaporates and could cause the thin channels to collapse or fracture, whereas pressure filling is also difficult since soft polymers can tear easily under too much pressure especially if it causes large deformations along with cracks¹². This leaves with filling using capillary action, which can be used for long heights and long channels, but is limited to the smoothness of the channels, viscosity of the material⁹. This paper explores the fabrication of microchannels using 2PP technology, with a goal of producing soft polymer microchannels usable for DEAs and tests the capillary filling technique using liquid electrode material.

2.1 Development version 1 – same orientation of capillary bridge design

Structures printed from IP-S were developed in 99.5% propylene glycol monomethyl ether acetate (PGMEA) for 25 minutes on a hotplate set to 75°C, afterwards, the prints were switched into 99.9+% isopropanol (IPA) on a hotplate set to 75°C for 15 minutes. The parts were blow dried with an airball and put into a vacuum chamber set to between -700 mmHg and -760 mmHg for 1-5 minutes to assist in evaporation of the remaining IPA. The parts were then moved back into IPA with the channels as perpendicular as possible and all put back into the vacuum chamber. A vacuum was slowly pulled until remaining air bubbles were pulled through the channels, aiding in the removal of the liquid resin still remaining in the channels, while also flushing them with more IPA. This process was repeated multiple times for the capillary bridge design shown in Figure 2.

2.2 Development version 2 – flipped orientation of capillary bridge design

To check the effect of orientation during the vacuum development process, samples of capillary bridges were also developed with the same method as in Section 2.1, but the orientation of the printed part is flipped between vacuum development cycles. The new process is visualized in Figure 3, where (e) and (f) show the flipped orientation of the capillary bridge.

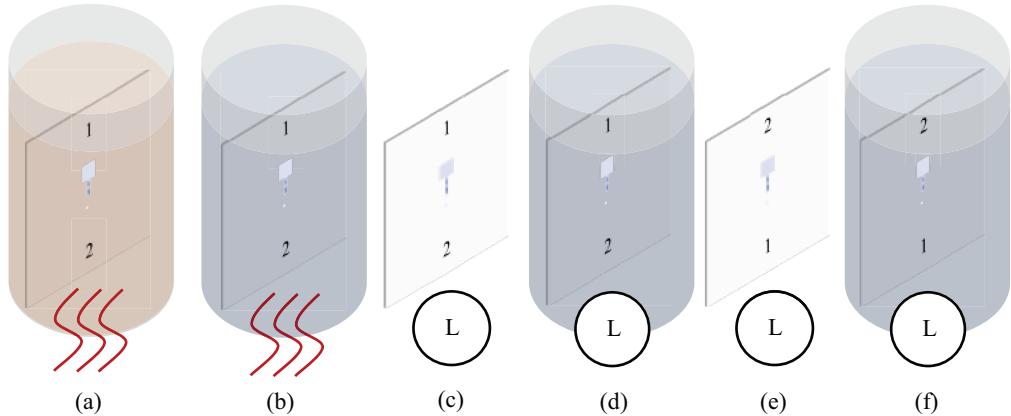


Figure 3. Development process of IP-S microchannel parts. (a) Development in PGMEA at elevated temperature and then into (b) IPA bath at elevated temperature followed by (c) blow dry and placement into vacuum chamber after which the part is then (d) submerged again into IPA and in placed into vacuum chamber. Part is (e) taken out and placed into vacuum chamber with orientation flipped, after which (f) the part is then placed back into IPA bath and into vacuum chamber. Steps (c)-(f) are then repeated multiple times.

2.3 Printing and development of capillary wall

A capillary wall was designed and modeled in CATIA and run through DeScribe software (Nanoscribe) to change the block stitching and get the print files. Block stitching has to occur when a printed part is out of range of the field of view of the lens being used for 2PP fabrication when using galvo scanning. For this paper, the lens being used is strictly the 25x objective with a 0.8 numerical aperture (NA) which has a field of view of 400 μm diameter for the galvo setting. Since all printed parts for this paper are over 400 μm in base dimensions, block splitting was necessary. Figure 4a displays the model in DeScribe and shows how the part is broken into different hexagonal pieces, with Figure 4b displaying the IP-S printed structure.

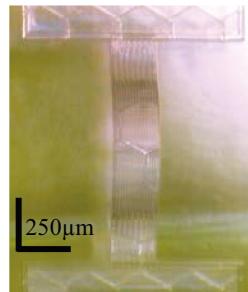


Figure 2. Fabricated IP-S printed capillary bridge with electrode pads on each side after development cycle.

The new process is visualized in Figure 3,

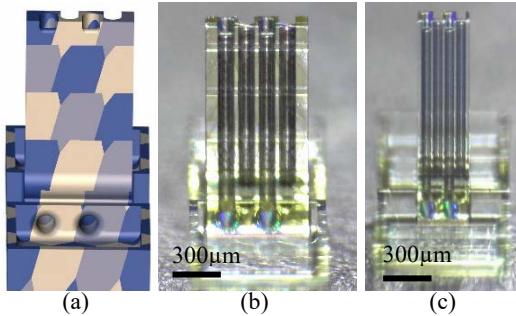


Figure 4. (a) Hexagonal block splitting of microchannel wall design with two electrode pads for DEA fabrication along with the (b) fabricated IP-S part and the (c) fabricated second design with no block stitching along the channel height.

A second wall design was created so the only block stitching occurred on the electrode bases and the main entrances to the microchannels. The part was created in CATIA and split into three separate files to be processed and coded together in DeScribe software. This was done in order to make the center portion of the wall print with no stitching effects except those that attach the center portion to both base pieces. All pieces had an overlap of 2 μm and a stitching angle of 15° as is used in the standard recipe provided by Nanoscribe. The fabricated IP-S part of the second wall design can be seen in Figure 4c.

Both wall designs were developed similarly to the approach depicted in Figure 3 with a difference being that the part was also turned upright during vacuum development. The objective to developing the parts upright was to cause air that might have been trapped in the bottom inlets upward through the capillary channels to help flush the resin out faster.

2.4 Printing and development of helix design with IP-PDMS

A double helix model was produced in CATIA and split into three separate files as was done in Section 2.3. The block stitching on the electrode pads along with the connection to the center portion was set to an angle of 21° to help aid in the attachment of one block to another. The center portion of the model can be seen in Figure 5, depicting that each helical electrode capillary channel makes two rotations which is in a height of 450 μm . The channels are rectangular with circular ends to help aid in the propagation of fluid using the capillary effect while keeping the structures relatively simple⁹. The helical part was developed similarly to the process in Figure 3 with a few minor differences. Due to the fragile nature of IP-PDMS, room temperature IPA for 10 minutes upside down and then placed into fresh room temperature IPA for 2 minutes still upside down to help drain any remaining liquid resin. The part then continues in the vacuum development process in Section 2.2 but by being oriented upright and upside so that the air pockets have a way to easily escape instead of being trapped inside of the helix and add to extra stress and internal pressure as would happen if oriented sideways.

2.5 Testing and electrode filling

Testing was done with a variety of materials including deionized water with blue dye added, PEDOT:PSS 1.3% solution in water by weight (Sigma Aldrich), and PEDOT:PSS 5% screen printing ink (Sigma Aldrich) mixed with deionized water to reduce viscosity. Testing was done by filling 10 mL syringes (Becton, Dickinson and Company) with 1-5 mL of solution. The syringe was attached to a micromanipulator (World Precision Instruments – M325) and attached with 32-gauge or 34-gauge rounded Luer-Lok™ tips and lowered near the fabricated resin pools where the liquid was dispensed into. Figure 6 displays a visual of the needles being positioned for dispensing. Pictures and videos were recorded using a Dino-Lite Edge Digital Microscope with a 10x-140x zoom with a 3cm focus. For dispensing into the IP-PDMS print, oxygen plasma was used for 5-10 seconds to help reduce surface tension and in turn reduce the contact angle of the PEDOT:PSS on the IP-PDMS surface, aiding in flow into the inlet of the microchannels, which was necessary for the naturally hydrophobic surface of IP-PDMS with a surface contact angle $\sim 110^\circ$ ¹³.

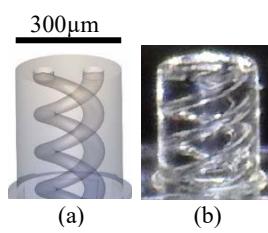


Figure 5. (a) Center portion of double helix CAD design with hollow capillary channels along with (b) 2PP printed IP-PDMS fabricated part.

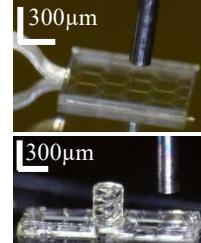


Figure 6. Positioning of micro needles for filling electrode pools.

3. DISCUSSION

After testing the microchannel development procedures from Section 2.1 and Section 2.2 against each other, it was determined that changing the orientation during vacuum development is necessary for fully cleaning out the channels. Keeping the orientation of the microchannel bridges the same during the development in Section 2.1 led to only one side filling with electrodes, as seen in Figure 7a-b, whereas changing the orientation led to a more even filling of both sides as Figure 7c depicts.

After trying increased temperature technique along with inducing a vacuum for filling and then for drying, the channels were able to be cleaned out slowly; however, increased use and cleanings led to small fractures in the IP-S printed parts allowing for fluid seepage out of the microchannel bridges as seen in Figure 8.

As noticed in Figure 8, it appears that multiple microchannels stop around where the block stitching is occurring. This phenomenon was readdressed with the stitched capillary wall from Section 2.2. This print contained stitching with larger sized channels to help visually determine if capillary effect was hindered by stitching of blocks during printing. Four tests were done on the same printed part with cleaning in IPA and deionized water while utilizing the vacuum chamber for air removal/channel flushing and for aid in liquid removal/evaporation. The results of the four tests can be seen in Figure 9, which shows that if the microchannels are not filled all the way, they happen to be stopped right where block splitting occurs. The block stitching regions appear to be causing a hydrophobic affect in the channels that is very minimal since retesting the same structure gives different heights of liquid each time. Further tests were done to determine if the block stitching is causing too big of an obstacle for capillary effect to overcome or if it is related to the print material or other particles stuck in the channels.

A second microchannel wall was created with no stitching in the channel, as shown in Figure 10. Figure 10 (b)-(d), only two microchannels were filled and the other two stopped at the stitching areas where the center portion connects to the base. Upon cleaning again, the channels were able to be filled. Refilling was able to be done with PEDOT:PSS 1.3% solution in water by weight.

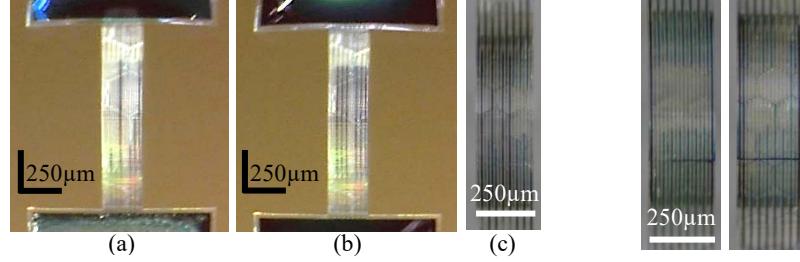


Figure 7. (a) Microchannel bridge after cleaning in IPA and vacuum a total of 4 times in same orientation. (b) After cleaning in IPA and vacuum a total of 8 times in same orientation. (c) After cleaning in IPA and vacuum a total of 4 times in each orientation.

Figure 8. Microchannel seepage after multiple cleanings for repeat trials.

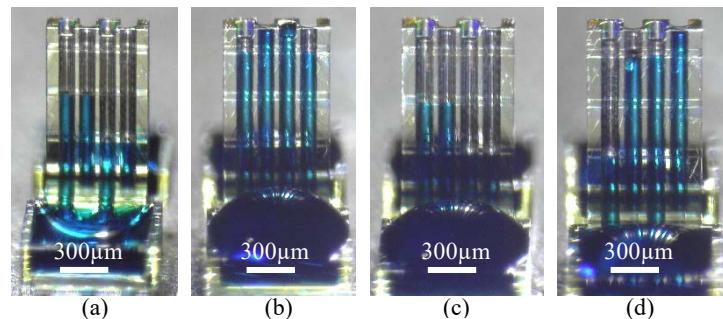


Figure 9. (a) Test one of filling both electrode pools for capillary channel filling, followed by cleaning for (b) test two, cleaning for (c) test three, and cleaning for (d) test four.

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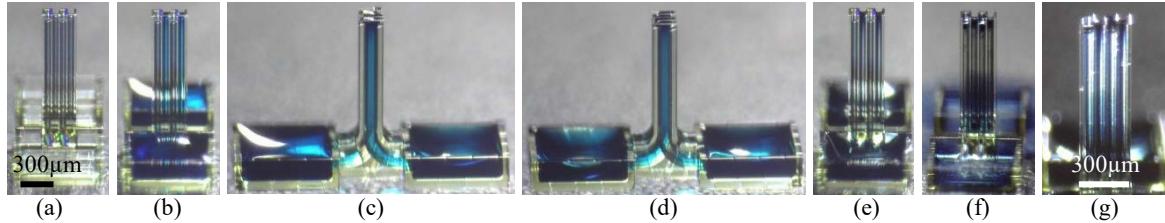


Figure 10. (a) Printed structure of the thinner channeled wall with no vertical stitching. (b) Front view of channel filling along with the (c) left side view and (d) right side view. (e) Filling with 1.3% PEDOT:PSS conductive electrode and then (f) once the PEDOT:PSS solution has dried. (g) Filling with 5% PEDOT:PSS screen printing ink mixed with water.

Once it was determined that the 1.3% PEDOT:PSS solution in water was able to fill the channels, seen in Figure 10 (e)-(f), it was found that as the solution dries out, it pulls the material up the capillary channels and builds up PEDOT:PSS at the top, seen in Figure 10(f). Although this appears to be a possible issue, if the electrode pool can be constantly topped off until PEDOT:PSS fully fills the channels after evaporation, then this would suffice; however, an exact amount of PEDOT:PSS solution needed to do this is unknown and there is also the chance that a buildup around the inlets could cause a seal in the channels. To make a more sustainable electrode whose volume does not decrease much from evaporation over time, a 5% PEDOT:PSS screen ink (Sigma Aldrich) with thickening agent was used. Since this material has the consistency of a solid paste, it was mixed with deionized water until it flowed more easily while still maintaining a light gel consistency. Figure 10 (g) shows the results of the thicker PEDOT:PSS solution and that it was able to fully fill all microchannels and was able to maintain stable inside of the microchannels.

In addition, a new geometry was designed and printed with IP-PDMS for proof of using capillary effect to fill a complex helical geometry with liquid electrode. The IP-PDMS part was printed using the parameters in Table 1 for fabrication. Figure 11 shows the CAD model of the helical geometry inside of the elastomer, the fabricated elastomer with empty channels and the result of filling the elastomer with PEDOT:PSS solution using capillary effect.

4. CONCLUSION

Though fabrication of microchannels is easily doable with technology such as 2PP, cleaning out the microchannels and turning thin microchannel structures into electrode channels is exceptionally complicated when the parts themselves are extremely fragile. A novel approach to cleaning out microchannels through vacuum assist during development was executed and proven to be a viable option for cleaning out fragile microchannels. Included in this approach, microchannels were fabricated using 2PP technology, testing the viability of microchannel fabrication and their ability to utilize the capillary effect for electrode filling. It was determined that block stitching has an ill effect on microchannel filling using capillary action and stitching effects should be reduced or avoided if at all feasible. In retrospect, it is entirely plausible to fill 2PP microchannels with electrodes through capillary action and was accomplished with IP-PDMS resin and PEDOT:PSS solution with thickening agent, creating a specific type of DEA, a helical dielectric elastomer actuator (HDEA), proving fabrication of DEAs using 2PP is accomplishable. Further research and experimentation is needed to test usability of DEAs fabricated with the techniques used in this paper.

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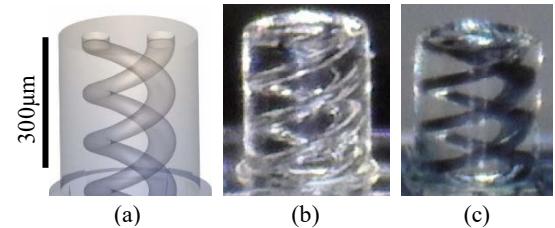


Figure 11. (a) CAD model of printed double helix capillary channel design inside of an elastomer. (b) Fabricated IP-PDMS elastomer empty and (c) filled with PEDOT:PSS solution in microchannels.

REFERENCES

- [1] M. Li, A. Pal, A. Aghakhani, A. Pena-Francesch and M. Sitti, "Soft actuators for real-world applications," *Nature Reviews Materials*, vol. 7, p. 235–249, November 2021.
- [2] K. Zheng, E. Gao, B. Tian, J. Liang, Q. Liu, E. Xue, Q. Shao and W. Wu, "Modularized Paper Actuator Based on Shape Memory Alloy, Printed Heater, and Origami," *Advanced Intelligent Systems*, vol. 4, p. 2200194, October 2022.
- [3] Srinivasaraghavan Govindarajan, R., Stark, T., Sikulskyi, S., Madiyar, F., & Kim, D. (2022, April). Piezoelectric strain sensor through reverse replication based on two-photon polymerization. In Proc. of SPIE Vol (Vol. 12046, pp. 1204611-1).
- [4] Y. Liu, O. Stein, J. H. Campbell, L. Jiang, N. Petta and Y. Lu, "Three-dimensional printing and deformation behavior of low-density target structures by two-photon polymerization," in *Nanoengineering: Fabrication, Properties, Optics, and Devices XIV*, 2017.
- [5] E. Montinaro, M. Grisi, M. C. Letizia, L. Pethö, M. A. M. Gijs, R. Guidetti, J. Michler, J. Brugger and G. Boero, "3D printed microchannels for sub-nL NMR spectroscopy," *PLOS ONE*, vol. 13, p. e0192780, May 2018.
- [6] J. Y. Han, S. Warshawsky and D. L. DeVoe, "In situ photografting during direct laser writing in thermoplastic microchannels," *Scientific Reports*, vol. 11, May 2021.
- [7] F. J. Fuchs, "Ultrasonic cleaning and washing of surfaces," in *Power Ultrasonics*, Elsevier, 2015, p. 577–609.
- [8] S. Chatzimichail, P. Supramaniam, O. Ces and A. Salehi-Reyhani, "Micropatterning of planar metal electrodes by vacuum filling microfluidic channel geometries," *Scientific Reports*, vol. 8, September 2018.
- [9] P. Kolliopoulos and S. Kumar, "Capillary flow of liquids in open microchannels: overview and recent advances," *npj Microgravity*, vol. 7, December 2021.
- [10] L. Hines, K. Petersen, G. Z. Lum and M. Sitti, "Soft Actuators for Small-Scale Robotics," *Advanced Materials*, vol. 29, p. 1603483, December 2016.
- [11] S. Chakraborty, Ed., *Mechanics Over Micro and Nano Scales*, Springer New York, 2011.
- [12] A. Spagnoli, M. Terzano, R. Brighenti, F. Artoni and A. Carpinteri, "How Soft Polymers Cope with Cracks and Notches," *Applied Sciences*, vol. 9, p. 1086, March 2019.
- [13] N. Szczotka, J. Navne, M. V. Adelmark, A.-I. Bunea and R. Taboryski, "Mapping the surface wettability of IP-PDMS micro-hoodoo arrays fabricated by two-photon polymerization direct laser writing," in *48th International conference on micro and nanoengineering*, 2022.