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A Bisphenol-A Based Low Cost Photolithography Method for Fabricating Lab on Chip (LOC) Devices

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Abstract

We propose a low cost and wieldy photolithography method which circumvent the need of expensive fabrication tools like mask aligner and EBL (Electron beam lithography) systems, making it possible to use this protocol in resource constraints laboratories. The proposed protocol involves use of a spinner and an Osram (Ultra Vitalux) lamp along with an in-house formulated low cost photo resist material. The photoresist consist of mixture of epoxy resin and cationic photo initiators. A minimum feature size of 40 microns with the separation of 70 microns is achieved by the proposed protocol. Micro channels fabricated by this method was used for detection of microorganisms.

Keyword- Photolithography, Bisphenol A, negative photoresist, low cost lithography.

I. INTRODUCTION

Microfabrication is one of the most important processes in modern IC industry. This process is used in making devices used in biological study, medical diagnosis, electrical circuits, biosensors and many more ([1], [2]). Photolithography process is commonly used for patterning microstructures. It mainly involves exposing photoresist material to light for formation of micro structures and has been widely used for lab on chip applications. SU8 is the most commonly used negative photoresist material due to various advantages like high aspect ratio, chemical and mechanical stability, planarization, excellent coating ([3], [4]). However, SU8 is very expensive photoresist material which requires high end facilities for fabrication. Thus there is a need for development of low cost photo resist material along with an easy to use cost effective fabrication method that is compatible with new photoresist material. Such system will make exploration and adaption of lab on chip technology at grass root level, like laboratories lacking high end facilities in schools and colleges and help them explore migration of many lab protocols easily and effectively. In past decade countless efforts have been made to make photolithography process low cost and easy to use. Some of the examples include like use of sunlight in place of specialized UV exposure systems to facilitate patterning features of up to 50 microns which can be used for microfluidics applications [5]. However the photoresist material used in experimentation, belongs to SU-8 series which is still an expensive approach. Similar approach have been used for photolithography where in, expensive equipment like mask aligner instrument has been replaced by UV (Ultra Violet) rays equipment which is commonly used in PCB (Printed Circuit Board) industry. This setup circumvent the need of high end clean room facility for fabrication, and it involves use of photomask (photographic transparent sheets) instead of standard chromium mask and Ferric oxide coated mask, but the photoresist material used in the process is expensive and also the high resolution photomask adds to expense [6].Various research groups have tried different approaches to make high resolution and low cost devices ([7], [8]).

Bisphenol-A based epoxy resin have been used as an excellent coating agent in various formulations with dihydrazides, polyphenol, polyamide ([9], [10], [11]). Triarylsulfonium hexafluorophosphate salts have been used actively as catalyst for polymerization reactions since many years ([12], [13]). In this paper, the new photoresist formulation is made by mixture of Bisphenol-A based epoxy resin and triarylsulfonium hexafluorophosphate salts. The formulated photoresist material is patterned on the photo mask as a base substrate in place of silicon or glass substrate, by using an Osram (Ultra Vitalux) lamp with a wavelength range of 300 nm - 1200 nm. The minimum feature size obtained is up to 40 microns. The fabricated channels were used in many microfluidics applications.

II. EXPERIMENTAL

A. *Mask and Substrate Preparation*:

The mask of desired microfluidic channel design was made in Corel Draw (X5 version) and printout on transparency was taken by the process of positive graphics. (Fig 1.a). The transparency itself was used as a base substrate instead of silicon wafer or glass substrate.

B. *Photoresist Formulation:*

Five different Bispheno-A based industrial grade epoxy polymers bought from local hardware store or obtained as samples from manufacturers were used in the study. The polymers used were GY 250 (Araldite), GY257 (Araldite) and Lapox11 (ATUL Resins), DER 330 (Dow Epoxy Resins) and DER 331(Dow Epoxy Resins). The above polymers were selected due to varying viscosity ranging from 500-14000 mPas (one millipascal-second). The epoxy polymers were mixed separately with photo acid named Triarylsulfonium hexafluorophosphate salts (Sigma-Aldrich) in a ratio of 90:10 (by volume) respectively to make photo resist material. The formulated photo resit material was found to be negative photo resist.

C. *Photolithography setup:*

A wooden box measuring 35 cm height x 22.5 cm width x 20.5 cm length was fixed with a day light lamp bulb (Osram 300 W bulb). A slit of 4 cm length X 3 cm width was made on the top side of wooden box to place the sample. The sample was placed at the distance of 12 cm from the source of light. The overall set up looks like in Fig 1.d.

D. *Photolithography protocol:*

The photo resist formulation as described in process B was degassed in a desiccator for 30 minutes. The photo resist was drop casted on the mask (Fig. 1.b) and placed on the spin coater (Delta Scientific) (Fig 1.c). Spin speed range used in the experiment was from 1500 rpm to 8000 rpm. The spin coat time was from 20 to 80 seconds. The mask was then exposed in photolithography set up box (Fig: 1.d) for 90 and 120 seconds. It was then developed in acetone for 5 to 10 seconds (Fig 1.e). The template with the pattern was ready to use for microfluidics applications.

E. *SEM and Profilometer analysis:*

For SEM analysis GY 250 and Lpox11 separately were spincoated on transparency with desired pattern at 1500 rpm for 80 seconds. The samples were exposed in Osram lamp for 120 seconds and developed in acetone. As epoxy polymer is non-conducting material to make it conducting gold was sputtered on the surface of features and silver paste was applied at the sides of sample for better image focusing. For profilometer analysis all the samples were prepared as described in protocol D. The film thickness was measured by DektakXT Profilometer. All the measurements were performed four times and an average value was calculated and plotted in the graph.

F. *Soft Lithography:*

A functional microfluidic device was made by casting PDMS-polydimethylsiloxane (Dow Corning) on the template. Standard soft lithography method was used to make PDMS device [14]. The base was mixed with curing agent in proportion of 10:1 (by weight) respectively. The mixture was mixed for several minutes vigorously and was degassed for 30 minutes. After pouring on template, the mixture was again degassed for 15 minutes. Then the mold was baked in conventional oven at 80^0C for 15 minutes. PDMS stamp with desired pattern was separated from the mask and access holes were made at the channel inlets and outlets. A clean glass cover slip (Blue star, No.1 - 0.13 to 0.16 mm thick) was cleaned in acetone for 10 minutes, followed by blow drying for few seconds. The PDMS stamp and cover slip were exposed in plasma oven (Harrick plasma cleaner) for 60 seconds and bonded immediately. The chip was baked in conventional oven at 80^0C for 30 minutes for enhanced bonding (Fig 1.g).

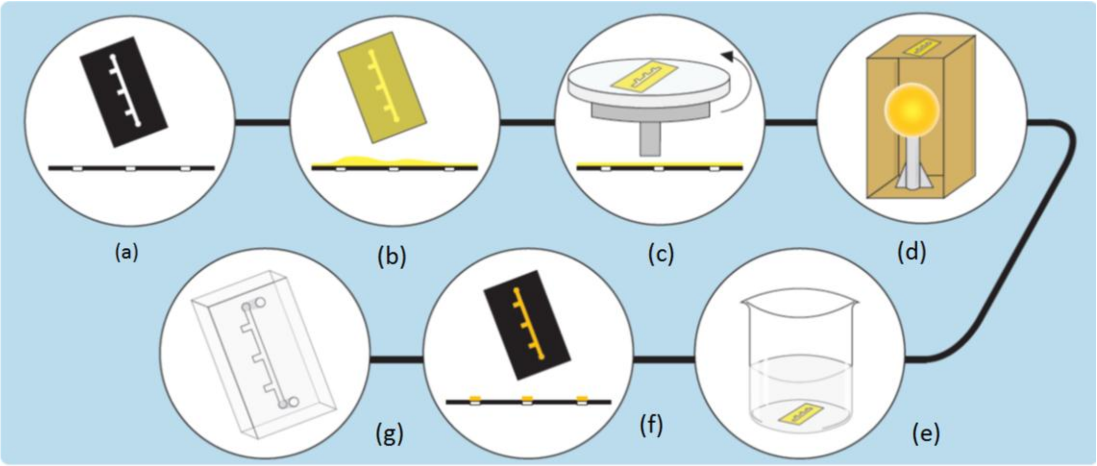

Fig. 1: Photolithography process: (a) mask with pattern, (b) photoresist on mask, (c) spin coat photoresist, (d) day light lamp set up, (e) acetone developer, (e) mask with pattern, (f) PDMS chip.

G. *Microbial analysis:*

The device fabricated by the method described in protocol F was used for analysis of microorganisms. A pure culture of *Escherichia coli* and *Staphylococcus aureus* was passed inside channels. The channels were observed at 100X magnification in EVOS FL AUTO microscope. Morphological analysis of microbes was done by using CEMIAS image analysis software [15].

III. RESULTS AND DISCUSSION

A. *Photolithography Setup:*

Unlike in standard photolithography systems which involve use of glass or silicon wafer as a base substrate to make patterns, here the mask/transparency itself was used as a base substrate for photolithography [16]. As depicted in Fig 1.(d) a wooden box mounted with an Osram lamp was used for photolithography. The distance from the lamp to the sample was kept constant while varying exposure time. The photoresist material was exposed for 90 seconds and 120 seconds of which 120 seconds was optimum time recorded.

B. *Film thickness and spin coat curve:*

The film thickness of fabricated device plays very important role in MEMS. The aspect ratio of the fabricated structure was studied along with the spin coat curve. The film thickness and spin speed data of all the formulated photo resist is displayed in Table I and Fig.2. During this experiment spin speed was varied from 1500 rpm to 8000 rpm while the time of spinning was kept constant at 80 seconds. The data provides information to select appropriate photo resist with respect to film thickness. Maximum film thickness of approximate 25 microns was achieved by using photo resist formulated with GY 250 epoxy resin which is highly viscous formulation while a minimum film thickness of approximate 2 microns was achieved by using photo resist formulated with GY 257 epoxy resin which is least viscous formulation.

The photo resist can be selected for future experimentation depending on application. The Film thickness and spin time data of all formulated photo resist is displaced in Table 2 and Fig. 3. Film thickness increased with decreased spin time. The spin speed in this experiment was kept constant at 8000 rpm while spin time was varied from 20 to 80 seconds. Maximum film thickness of approximate 7 microns and minimum film thickness of 1.5 microns was achieved by using photo resist formulated with GY 250 and GY 257 respectively.

C. *Photolithography and Lab on chip device:*

As shown in Fig.4(a) and Fig.4(b), SEM image was captured after developing pattern on transparency mask (Fig.1(f)). The minimum feature size achieved by the proposed method was 40 microns while the separation gap was observed as 70 microns. The minimum feature size is restricted to 40 microns due to printing resolution limitation of positive graphics technology. A feature size upto 2 microns was achieved by using standard mask aligner system by using in house formulated photo resist (Data not shown). To illustrate application of developed fabrication protocol 2D (2 dimensions) channels for image analysis of microorganisms and 3D channels were demonstrated.

A PDMS lab on chip device was fabricated as described in protocol 2.3.As shown in Fig. 5 (a) a 3D microfluidic device was fabricated. The channel in the focus is control channel while the channel in the background perpendicular to control channel is the flow channel. In Fig 5(b) flow channel is in focus. Round channels surface was observed which can be used as added advantage for fabricating 3D microfluidics channels. As there is problem of fluid leakage from the side of the walls in square channels[16].

Table I. Film Thickness and Spin Speed Data of Epoxy Resin

(*Approximate Values)

Name of Epoxy	Viscosity mPas*	Spin Speed (rpm)	Thickness (μm)
GY 257	500 - 600	1500	14.7
		3000	5.26
		4500	4.15
		6000	3.54
		8000	2.09
DER 330	7000 – 10,000	1500	20.5
		3000	9.63
		4500	6.56
		6000	4.85
		8000	2.71
GY 250	10,000 – 12,000	1500	24.12
		3000	14.8
		4500	10.96
		6000	6.4
		8000	3
Lapox 11	10,000 – 12,000	1500	19.82
		3000	10
		4500	7.67
		6000	4.45
		8000	3.39
DER 331	11,000 – 14,000	1500	15.23
		3000	6.91
		4500	4.76
		6000	4.43
		8000	3.63

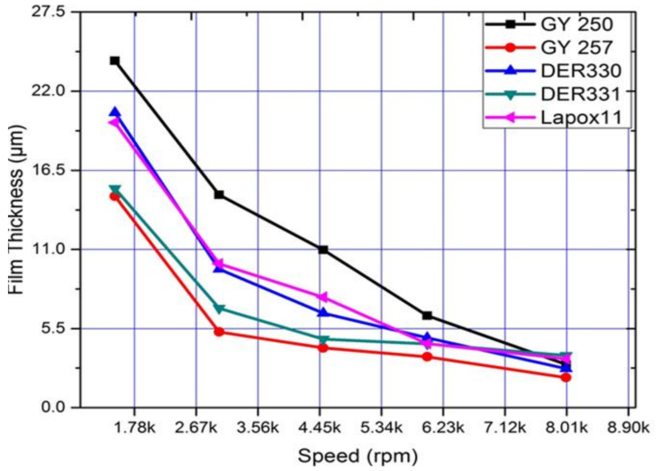

Fig. 2. Spin speed Vs Thickness curve of formulated photo resist

Table II. Film Thickness And Spin Time Data Of Epoxy Resin (*APPROXIMATE VALUES)

Name of Epoxy	Viscosity mPas*	Spin time (seconds)	Thickness (µm)
GY 257	500 - 600	20	3.74
		40	2.1
		60	1.84
		80	1.68
DER 330	7000 – 10,000	20	4.92
		40	3.69
		60	2.94
		80	2.52
GY 250	10,000 – 12,000	20	7.73
		40	5.7
		60	4.62
		80	3.4
Lapox 11	10,000 – 12,000	20	6.81
		40	5.4
		60	4.85
		80	3.32
DER 331	11,000 – 14,000	20	6.93
		40	4.02
		60	3.7
		80	3.06

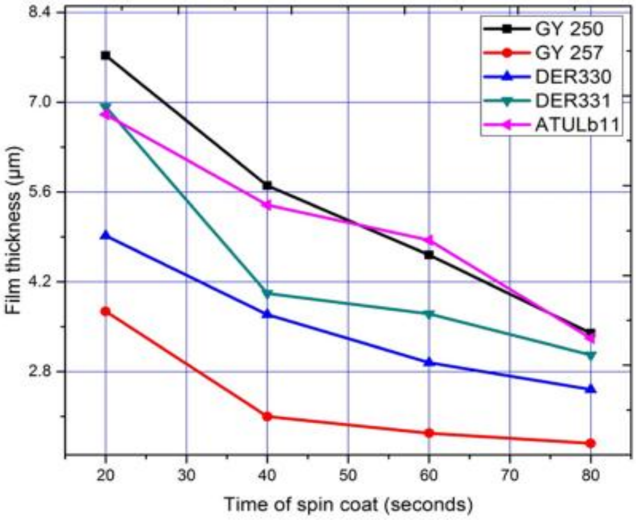

Fig. 3. Spin time Vs Thickness curve of formulated photo resist.

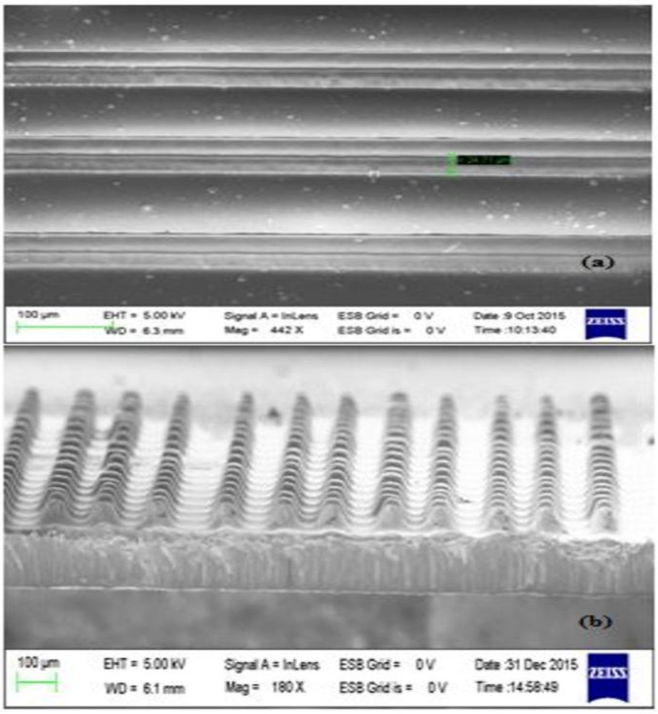

Fig. 4. SEM Image of device fabricated by photolithography. (a) Micro channels fabricated by described photolithography protocol by using photo resist formulated with GY 250 Epoxy resin (scale =100 µm). (b) Micro pillars fabricated by using photo resist formulated with Lapox 11 (scale=100 µm).

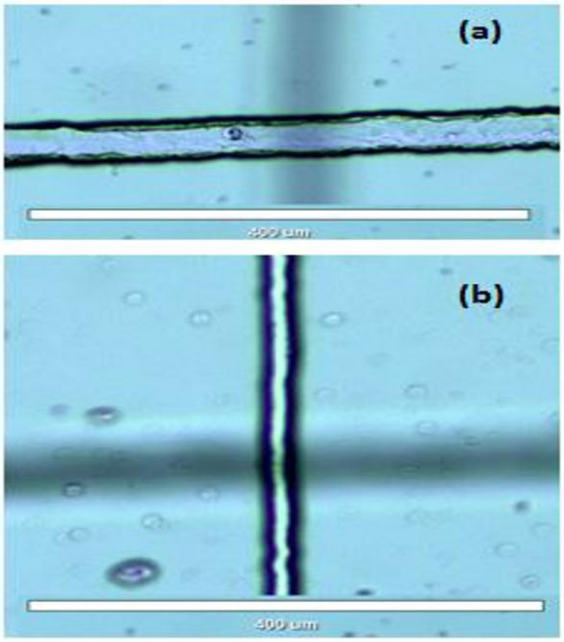

Fig. 5. PDMS lab on chip device. (a) 3D microfluidic channel at 40 X magnification with (a) control and (b) flow channel in focus .(scale =400 microns).

Fig. 6 shows chamber of PDMS microfluidic channel used for pure culture analysis of Staphylococcus aureus. Single cell tracking of microbes is possible in microfluidic channels. The channel was fabricated by using photo resist formulation of GY 257 and the channel height was approximate 2 microns which is ideal for microbial analysis.

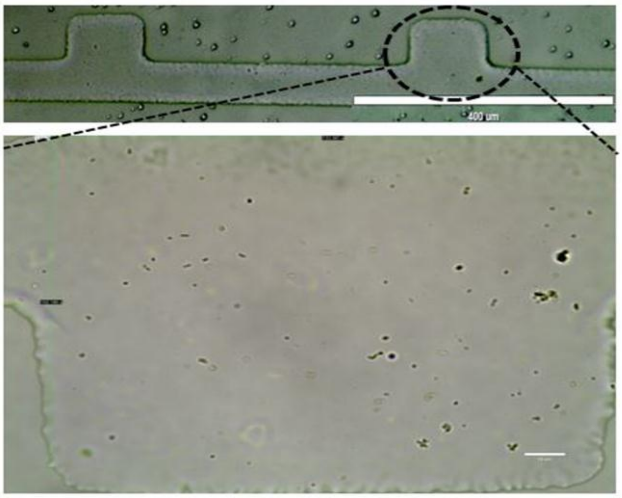

Fig. 6. A Microfluidic PDMS channel at 10 X (Two boxes with the encircled box) and at 100X magnification (single box) with pure culture of *Staphylococcus aureus* (bar size = 400 microns and 10 microns)

Fig. 7 shows a PDMS straight channel with fluorescent stain *Escherichia coli* cells. Each dot represents single cell. This fabrication protocol has been effectively used for fluorescent staining application.

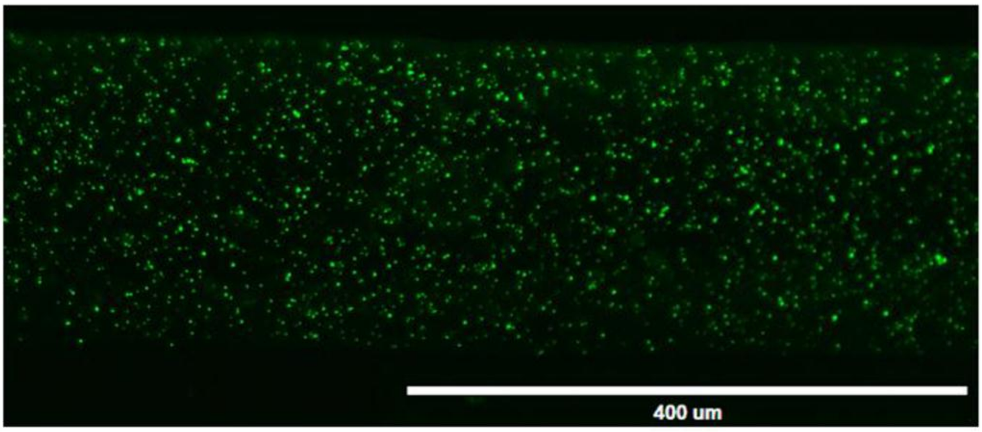

Fig. 7. PDMS straight channel with fluorescent stain Escherichia coli cells at 10X magnification (bar size = 400 microns).

IV. COST ANALYSIS

The cost of Bisphenol A based epoxy resins used in the formulated photo resist is around \$3.0 - \$4.0 per litre. The photo initiator cost is around \$69.61 for 25 ml. In one 10 mm X 10 mm transparency mask (\$0.08) around 500 microliter of photo resist is used which brings down cost of approximately to \$0.2 per chip. In photolithography setup the Osram lamp costs around \$50 and a wooden box is around \$50. The overall photolithography setup costs around \$100 which can be very low cost and convenient solution for resource poor laboratories like schools and colleges.

V. CONCLUSION

A cost effective photolithography method along with a new low cost photo resist material was formulated. This protocol can be adapted by resource poor labs. This easy to use fabrication technology can be implemented to fabricate lab on chip and MEMS devices.

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REFERENCES

[1] R. Luttge, Microfabrication for industrial applications. William Andrew, 2011.

[2] J. Voldman, M. L. Gray, and M. A. Schmidt, "Microfabrication in biology and medicine," Annual review of biomedical engineering, vol. 1, no. 1, pp. 401–425, 1999.

[3] J. M. Shaw, J. D. Gelorme, N. C. LaBianca, W. E. Conley, and S. J. Holmes, "Negative photoresists for optical lithography," IBM Journal of Research and Development, vol. 41, no. 1.2, pp. 81–94, 1997.

[4] A. del Campo and C. Greiner, "Su-8: a photoresist for high-aspectratio and 3d submicron lithography," Journal of Micromechanics and Microengineering, vol. 17, no. 6, p. R81, 2007.

[5] J. Ma, L. Jiang, X. Pan, H. Ma, B. Lin, and J. Qin, "A simple photolithography method for microfluidic device fabrication using sunlight as uv source," Microfluidics and nanofluidics, vol. 9, no. 6, pp. 1247– 1252, 2010.

[6] V. C. Pinto, P. J. Sousa, V. F. Cardoso, and G. Minas, "Optimized su-8 processing for low-cost microstructures fabrication without cleanroom facilities," Micromachines, vol. 5, no. 3, pp. 738–755, 2014.

[7] Y. He, X. Xiao, Y. Wu, and J.-z. Fu, "A facile and low-cost micro fabrication material: flash foam," Scientific reports, vol. 5, 2015.

[8] J. H. Bruning, "Optical lithography: 40 years and holding," in Advanced Lithography. International Society for Optics and Photonics, 2007, pp. 652 004–652 004.

[9] A. Catalani and M. G. Bonicelli, "Kinetics of the curing reaction of a diglycidyl ether of bisphenol a with a modified polyamine," Thermochimica acta, vol. 438, no. 1, pp. 126–129, 2005.

[10] J. Cheng, J. Chen, and W. T. Yang, "Synthesis and characterization of novel multifunctional epoxy resin," Chinese Chemical Letters, vol. 18, no. 4, pp. 469–472, 2007.

[11] A. M. Tomuta, X. Ramis, F. Ferrando, and A. Serra, "The use of dihydrazides as latent curing agents in diglycidyl ether of bisphenol a coatings," Progress in Organic Coatings, vol. 74, no. 1, pp. 59–66, 2012.

[12] I. A. Barker and A. P. Dove, "Triarylsulfonium hexafluorophosphate salts as photoactivated acidic catalysts for ring-opening polymerisation," Chemical Communications, vol. 49, no. 12, pp. 1205–1207, 2013.

[13] J. L. Dektar and N. P. Hacker, "Photochemistry of triarylsulfonium salts," Journal of the American Chemical Society, vol. 112, no. 16, pp. 6004–6015, 1990.

[14] Y. Xia and G. M. Whitesides, “Soft lithography,” Annual review of materials science, vol. 28, no. 1, pp. 153–184, 1998.

[15] J. Liu, F. B. Dazzo, O. Glagoleva, B. Yu, and A. K. Jain, “Cmeias: a computer-aided system for the image analysis of bacterial morphotypes in microbial communities,” Microbial Ecology, vol. 41, no. 3, pp. 173–194, 2001.

[16] M. A. Unger, H.-P. Chou, T. Thorsen, A. Scherer, and S. R. Quake, “Monolithic microfabricated valves and pumps by multilayer soft lithography,” Science, vol. 288, no. 5463, pp. 113–116, 2000.