Fast three dimensional ac electro-osmotic pumps with nonphotolithographic electrode patterning

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Three dimensional (3D) stepped electrodes dramatically improve the flow rate and frequency range of ac electro-osmotic pumps, compared to planar electrodes. However, the fabrication of 3D stepped electrodes for ac electro-osmosis (ACEO) pumps usually involves several processing steps. This paper demonstrates results from ACEO pumps produced by a faster and less expensive method to fabricate the 3D electrodes—extending the previous work to disposable devices. The method is based on shadowed evaporation of metal on an insulating substrate that can be injection molded. Flow velocities through the 3D ACEO pump are similar to those seen in the previous work. © 2010 American Institute of Physics.
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I. INTRODUCTION

The miniaturization of fluidic systems requires new techniques for pumping fluids. Pressure-driven flow is acceptable for many applications¹ but causes sample dispersion and requires bulky equipment. Electro-osmosis (EO) is a pumping technique which has been explored to replace the traditional pressure driven flows. In dc EO an electric field is applied across the channel of the microfluidic device using dc voltage. Direct current causes unwanted side effects, such as producing bubbles due to faradic reactions, consuming metal electrodes through electrolysis, and the need for high voltage (>10 V) which can be an obstacle in the miniaturization of the device and electronics.

A. Origin of the three dimensional ac electro-osmotic pump

To avoid the problems and drawbacks of using microfluidic pumps using dc, Ramos *et al.*² proposed the first microfluidic pump based on a nonlinear electro-osmotic flow, which is commonly known as “ac electro-osmosis” (ACEO). The device used symmetric planar electrodes. Brown *et al.*³ proposed asymmetric interdigitated microelectrode arrays which are different in widths and gaps. Then Bazant and Ben⁴ simulated a dramatic improvement of the fluid flow velocity in ACEO pumps by an arrangement of three dimensional (3D) electrodes that they described as a “fluid conveyor belt.” Figure 1 shows how these step electrodes are expected to increase the flow velocity by aligning the flow direction of the pair of counter-rotating vortices that are created at each electrode. Thorsen *et al.*⁵ then demonstrated experimentally a dramatic improvement compared to planar ACEO pumps using electroplated gold electrodes. Recently, Huang *et al.*⁶ introduced an ultrafast ACEO pump to achieve higher velocities (>1 mm/s) and much larger pressure ($>1\%$ atm). They eliminated the costly electroplating process by using three layers of photolithography for etching, metal deposition, and lift-off. Currently, the above ACEO pumps do not work well at physiological salt concentrations for reasons suggested in Ref. 7 and explored further in Ref. 8, but such devices can pump a dilute electrolyte (for example, de-ionized water) to push on biological solutions.⁶

FIG. 1. Schematic of the surface slip velocity (solid line) and resulting pumping streamlines (dashed lines) of (a) asymmetric planar electrodes and (b) optimal 3D electrodes (the fluid conveyor belt proposed in Ref. 4).

B. Low-cost fabrication concept

Even in the improved 3D ACEO pumps, there is still a need for a cheaper and simpler method to fabricate the 3D stepped metal electrode arrays that does not involve multiple photolithography steps. In this paper, a new fabrication technique eliminates the need for microlithographic patterning of each individual device. The new method depends on the shadowing effect caused by the directionality of thin metal film evaporation. We discuss how to produce an insulating stepped substrate (here from SU-8 negative photoresist but a similar structure could be injection molded) for shadowed metal deposition; the only metal patterning required is a large-scale (1 cm) stencil. Similar techniques have previously produced nanoscale interdigitated electrodes.⁹

II. METHOD

A. Creating a two-level insulating substrate

A two-height insulating substrate is needed; the thinner layer will provide the electrode steps and a taller feature at the end of each electrode will create a shadow that interrupts the conducting metal path between adjacent electrodes. Figure 2 illustrates this structure and the resulting shadows. SU-8, an epoxy-based, negative photoresist (Microchem, Inc.) has been used to produce the 3D two layered structure in this work. While SU-8 is a good material for prototyping, these structures have no overhangs and could be replicated by casting or injection molding instead of photolithography.

The fabrication process is illustrated in Fig. 3. It starts with thermal deposition of a 300–400 nm SiO_2 layer on a silicon wafer to create an insulating floor for the pump channel. To improve the adhesion of the SU-8 films, the oxidized wafers were cleaned using acetone and isopropyl alcohol, and then dehydration baked on a hot plate at 200 °C for at least 20 min. A thin layer of SU-8(5) was used to produce the 5 μm thick electrode layer. SU-8 was spread at 500 rpm for 2 s, then spun at 3000 rpm for 10 s. The coated wafer was soft baked on a hot plate for 2 min

FIG. 2. Illustration of shadow evaporation on a two-layer stepped electrode. Left: top view. Right: detail view. The barrier at the end of the electrode creates a shadow that breaks the connection between adjacent electrodes.

at 75 °C followed by 5 min at 105 °C. The first layer (electrode pattern) was then exposed to ultraviolet (UV) in a photoaligner (Karl Suss). Cross-linked features appear after a postexposure bake (PEB) for 1 min at 75 °C and 2 min at 105 °C; this PEB time can be increased for extra 1 or 2 min to increase adhesion if needed. These steps were repeated again with different parameters to obtain a 15 μm second layer. The spin speed was slower (1375 rpm) and the soft bake was longer (4 min at 75 °C and 7 min at 105 °C). The second mask (with the shadowing features) was

FIG. 3. Fabrication process for the three dimensional electrode pump.

FIG. 4. Large-scale stencil to isolate left and right interdigitated electrodes at the ends of the electrode array.

aligned and the UV exposure in our system was 20 s instead of 16 s for the electrode layer. The PEB was 1 min at 75 °C and 3 min at 105 °C. Both layers of SU-8 were then developed in propylene glycol methyl ether acetate for 4 min with gentle agitation, followed by rinsing in isopropanol and drying in nitrogen.

B. Producing interdigitated electrodes by shadow evaporation

To separate the interdigitated electrodes at the ends of the electrode arrays, tape was applied to the wafer across the ends of the arrays, as illustrated in Fig. 4. The alignment is not critical in this step, as long as the tape extends some distance into the electrode array, and the tape is relatively wide (5 mm). This step could therefore be accomplished with a stencil during manufacturing. After masking, the wafer was centered on the stage and tilted at an angle of 50° with respect to the substrate normal in a vacuum electron-beam evaporator (Kurt J. Lesker). A 50 nm titanium adhesion layer was followed by a 150 nm gold layer.

Shadowing by the thick SU-8 feature served to cut the connection between adjacent electrodes, while shadowing by the thinner layer creates the uncoated region on the channel floor between electrodes. Figures 5(a) and 5(b) show the shadowing effect that occurs between the electrodes due to deposition of metals at an angle. Figure 5(c) is a voltage contrast scanning electron micrograph (SEM) image, which shows the electrical isolation of the interdigitated electrode pair. The choice of the deposition angle and the height of the second layer is critical to obtain interdigitation.

C. Making the fluidic and electrical connections

To create fluid inlets and outlets, poly(dimethylsiloxane) (PDMS) elastomer microchannels were aligned and sealed over the electrodes. Standard soft lithography techniques were used with SU-8(50) molds to create channels with a typical height of 80 μm .^{10,11} To make more efficient use of the cleanroom-fabricated SU-8 molds, we cast new PDMS channels on replicated PDMS elastomer molds of opposite “polarity” that were sprayed with Ease Release 200 (Mann Release

FIG. 5. SEM shows (a) disconnection between the electrodes due to shadowing effect. Gold appears light while shadows appear dark. (b) Detail of disconnection area. (c) Voltage contrast electron microscopy shows electrical isolation of the interdigitated electrode pair: grounded electrodes appear white, while positive electrodes appear black. Scale bars are 25 μm .

Technologies). After removal of the second molded PDMS from the PDMS mold, the channels were cleaned in isopropyl alcohol and water to remove the release agent. The PDMS piece was cut to size and holes were punched for fluidic connections. The PDMS channels and the 3D electrode devices were then rinsed in water and dried, exposed to an air plasma at 9 W rf and 100 mTorr pressure for 30 s, aligned under a binocular microscope and pressed together. This sealed the PDMS to metal and oxide surfaces, with two large metal pads extending beyond the PDMS on the sides of the channel, each connected to opposite sides of the interdigitated electrode array. Electrical contact wires were bonded to these pads using silver conductive epoxy (MG Chemicals). The entire device was heated to 60 °C in a gravity convection oven for at least 1 h to improve the bond between the PDMS and substrate and to cure the conductive epoxy.

III. RESULTS AND DISCUSSION

A. Testing the fabricated pumps

The shadow evaporation method produces a structure with a few differences from the original 3D ACEO pump: Self-shadowing produces a nonconductive wall on the back edge of the step, and there is a conductive pillar at the end of each electrode. For evaluating the performance of this new design, we fabricated a pump with 144 electrode pairs having total length of 5 mm and width of 260 μm . The electrode height was 5 μm , the electrode width was 10 μm , and the spacing between the electrode pairs was 25 μm . These width and spacing, and the 80 μm PDMS channel height were taken from Ref. 12, a detailed study of 3D ACEO pump geometry where the electrode height was varied systematically from 0 to 10 μm . The shadowing pillars at the end of the array were 15 μm tall. Particle imaging velocimetry experiments were then carried out as follows.

Tubes used for loading the fluid were connected to the PDMS cap through the inlet and outlet ports. The fluid used was de-ionized water with red fluorescent polystyrene beads with 2 μm diameter (fluospheres, Invitrogen Inc.). The fluid was loaded using a syringe, and experiments were not started until pressure driven flow reached equilibrium and the particles were no longer moving.

A function generator (BK Precision 4003A) was used to run the ACEO pumps at different ac frequencies (0.5–100 kHz) at 1 $V_{\text{p.p.}}$ [peak-to-peak (p.p.)] and 2 $V_{\text{p.p.}}$. Movies were captured

FIG. 6. Semilogarithmic plot of velocity vs frequency for the pump at 1 and 2 $V_{p.p.}$.

using a charge coupled device camera (ProgRes CF, JenOptik) and noninverted fluorescence microscope (Zeiss Axioskop2 Plus). The centroids of particles in 50 sequential frames were tracked using IMAGEPRO 6 PLUS software. This software provided the accumulative distances of the tracks in pixels, while the ProgRes camera driver provided a file of frame timestamps. The velocity was calculated from the slope by plotting the accumulative distance versus time. The mean and the standard deviation of the velocities of the particles inside the channels were calculated based on velocities measured for 3–27 particles at each voltage/frequency setting. Figure 6 shows velocity versus frequency for 1 and 2 $V_{p.p.}$ driving signals. All velocity measurements were conducted on particles in focus near the pumping surface because there was a recirculating flow observed at the top of the PDMS channel due to buildup of back pressure in the channel and the long tubes used to load the fluid into the device.

B. Comparison to pumps fabricated by other methods

Figure 7 shows the average velocity of the particles inside the channel for this pump along with velocities for three other ACEO pumps extracted from literature, over a frequency range of 0.5–100 kHz at 2 $V_{p.p.}$. The peak velocity at 2 $V_{p.p.}$ is 260 $\mu\text{m/s}$ at 500 Hz and the velocity slightly decreases to 246 $\mu\text{m/s}$ at 1 kHz. After 1 kHz, the velocity decays with frequency, then there is a small increase in velocity (70 $\mu\text{m/s}$) around 8 kHz, then it decays again until it reaches almost zero at 100 kHz. The performance is in line with that of previous 3D ACEO pumps, in the range of hundreds of microns per second, and all generate faster flow velocities than the planar ACEO pump. Slight differences in electrode geometry from one publication to another make direct comparison difficult, but these 3D ACEO pumps are qualitatively similar in performance. In all, the velocity of the flow decreases at higher operating frequencies and a second peak of velocity at a higher frequency is observed. This secondary peak^{6,12} is possibly explained by different frequency responses for positive and negative charging in asymmetric electrolytes at high voltages.⁷ However, the secondary peak velocity in our pump is not as prominent as the ones in the other 3D ACEO pumps.

IV. CONCLUSIONS

In summary, we have demonstrated a simple and inexpensive fabrication process to obtain three dimensional interdigitated stepped electrodes. We studied the behavior of the flow of the fluid inside the microchannel of the ACEO pump device at different frequency/voltage settings and found it consistent with 3D pumps created by other methods.

FIG. 7. Semilogarithmic plot of velocity vs frequency, comparing the low-cost shadow evaporated ACEO pump in this work to three other published pumps at $2 V_{p,p}$. (Refs. 3, 5, and 6).

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