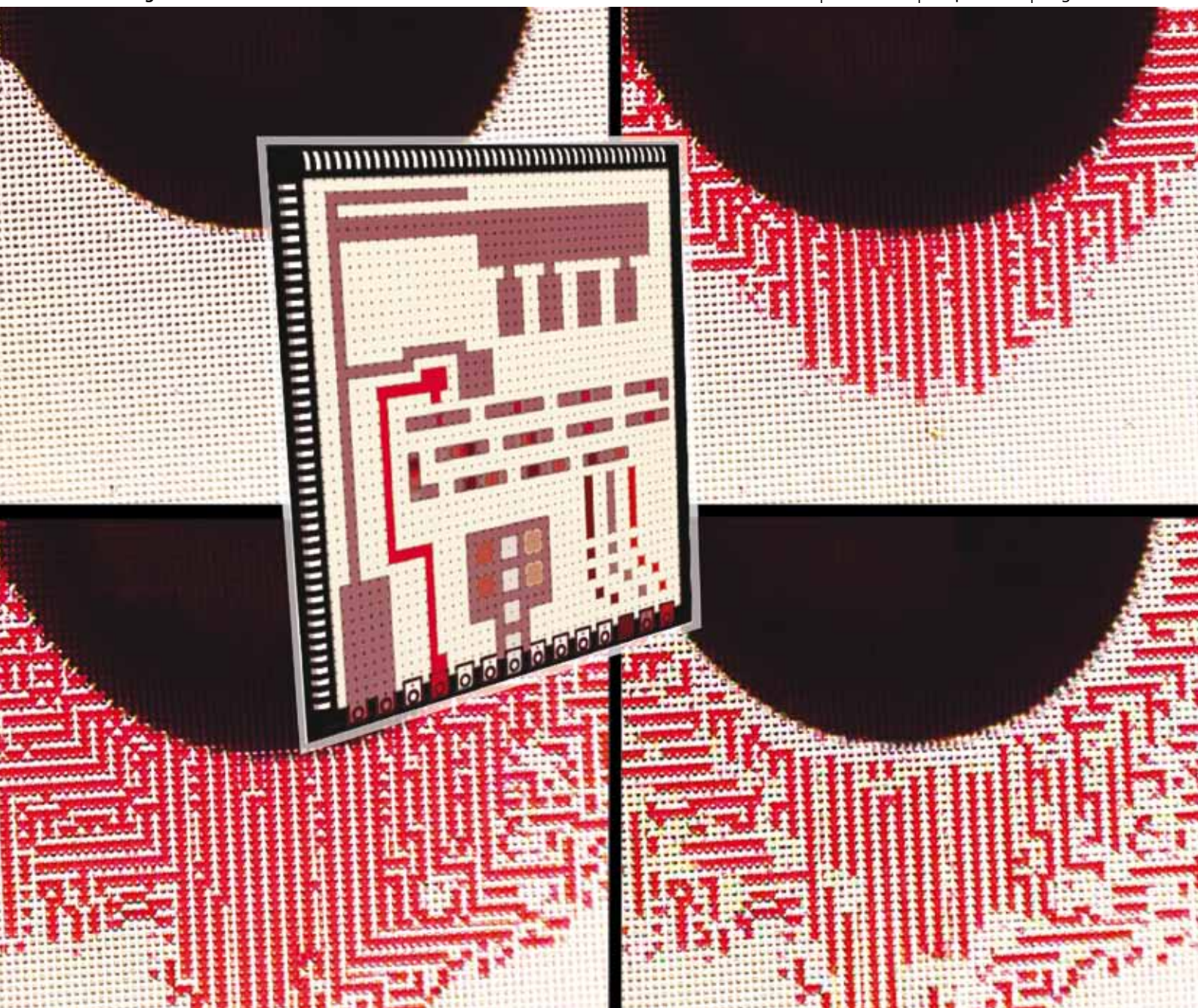


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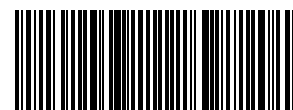
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Virtual electrowetting channels: electronic liquid transport with continuous channel functionality†

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Reported is a new mechanism for electronic transport of liquid in virtual channels. These virtual channels are formed by application of voltage to an array of polymer posts. The posts are coated with a conducting electrode and hydrophobic dielectric, and thereby capable of electrowetting. Directional channel formation, as well as splitting and merging, is also demonstrated using specific arrangements of posts. The channel dimensions are $\sim 20\ \mu\text{m}$ in cross-section, are scalable, and at the threshold for channel formation the minimum transport speed is $\sim 1\ \text{mm s}^{-1}$. The virtual electrowetting channels are further unique as they can retain any channel geometry even in the absence of voltage. With the addition of arrayed voltage controls, the virtual electrowetting channels have the potential to combine the advantages of programmable electrowetting and continuous channel functionality into a single lab-on-chip platform.

Microfluidics has been intensely investigated for nearly two decades, being traditionally explored within fixed geometries of continuous polymer or glass microchannels.¹ Microchannels allow for both pressure driven flow control² and electrokinetic transport,³ which are useful for a wide array of biomedical and analytical applications. There have also been numerous demonstrations of programmable liquid transport, including surface-energy defined virtual channels,⁴ electrowetting droplet transport,^{5–7} and dielectrophoresis.^{8–10} In addition, electrophoresis and programmable droplet transport have been co-located within a single microfluidic system.¹¹ However, none of the prior approaches is capable of on-demand creation of any desired channel geometry, followed by preserving that channel without external stimulus. If such capability was realized, one could also integrate electrically induced channel functions such as electrophoretic separations¹² or electrochemical sensing,¹³ thereby truly bridging the gap between the domains of programmable droplet and continuous flow microfluidics.

Reported herein is a new mechanism for electrical creation of liquid channels with virtual sidewalls (Fig. 1a). These virtual channels are formed by application of voltage to an array of electrowetting posts, and retain their geometry even after removal of voltage. Directional channel formation as well as splitting and merging are also demonstrated at $\sim 1\ \text{mm s}^{-1}$ and a theoretical model of the transport mechanism is presented. With the addition of arrayed voltage controls,¹⁴ the virtual electrowetting channels have the potential to combine the

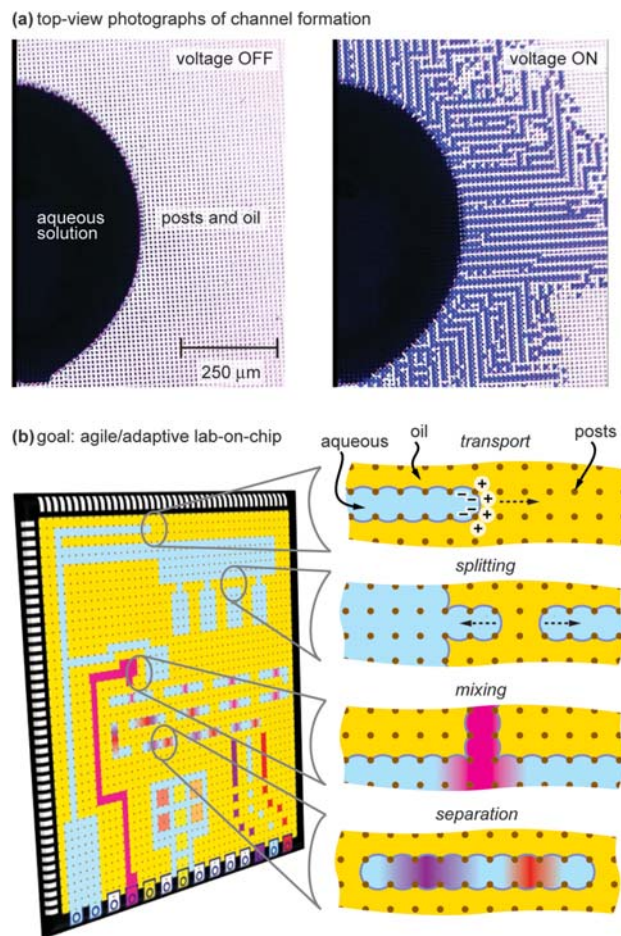


Fig. 1 (a) Photographs of proof of concept for agile formation of virtual electrowetting channels. (b) Diagram depicting true 'laboratory'-on-chip functionality through use of virtual electrowetting channels.

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advantages of programmable electrowetting and continuous channel functionality into a new, highly adaptive, lab-on-chip platform (Fig. 1b). This level of capability might move applied microfluidics beyond ‘instrument’-on-chip and closer to the promise of true ‘laboratory’-on-chip. Furthermore, highly programmable modules may actually improve lab-on-chip affordability by a combination of mass-production and user-defined specialization.

The experimental test vehicle for demonstrating virtual electrowetting channels is like a conventional microfluidic chip with topside entry ports,⁵ but replaces microchannels with a continuous array of electrowetting posts (Fig. 2a). The first generation devices described herein were formed using conventional microfabrication processes that can be implemented on glass or plastic. It is likely that this process can also be economically implemented in a standard liquid-crystal-display fabrication facility (scalable). A newly available negative-acting dry film photoresist DuPont PerMX-3020 (20 μm thick) was laminated at 80 °C and 40 psi onto a glass wafer using a Western Magnum dry film laminator. The resist was then UV exposed with the post-pattern, puddle developed in propylene glycol monomethyl ether acetate, and hard baked at 150 °C for 30 min. Approximately 150 nm of Cu was sputtered onto the posts to create the bottom electrode. Parylene C dielectric was then conformally deposited onto the Cu using a Speciality Coating Systems PDS2010 system. The post array was then dip coated in Cytonix Fluoropel 1601 V solution, then baked at 120 °C for 20 min to obtain a hydrophobic fluoropolymer layer of ~50 nm.

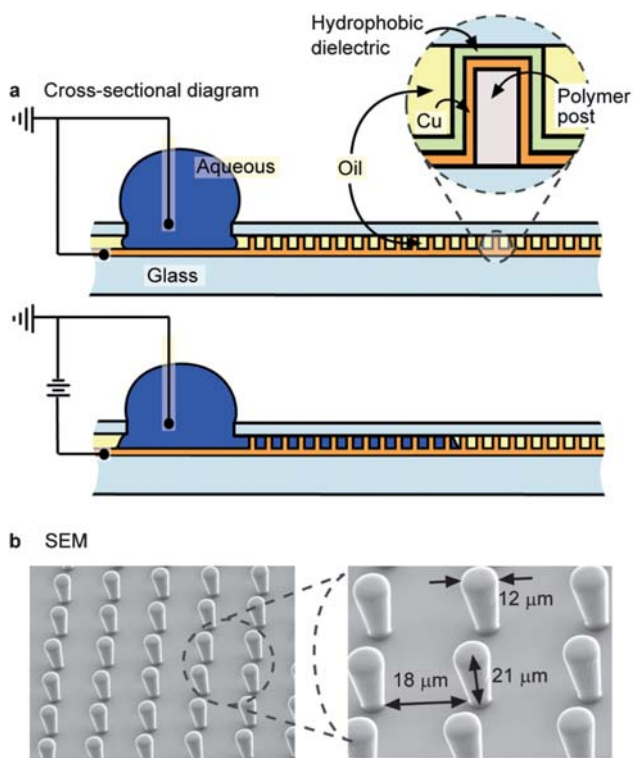


Fig. 2 (a) Diagram of the experimental setup used to generate virtual electrowetting channels in arrays of electrowetting posts, and a magnified diagram of a single electrowetting post. (b) Scanning electron microscope image of the arrayed electrowetting posts.

Before describing the virtual channel formation, a basic understanding of electrowetting is first required. Electrowetting is an electromechanical effect¹⁵ that can modulate an aqueous/oil contact angle on a hydrophobic dielectric. By applying a voltage between the aqueous phase and an electrode beneath the hydrophobic dielectric, the liquid contact angle can be varied from Young’s contact angle ($\theta_Y \approx 160\text{--}180^\circ$) to an electrowetted contact angle ($\theta_V \approx 30\text{--}60^\circ$). Contact angle vs. voltage (V) is predicted¹⁶ by the electrowetting equation $\cos \theta_V = (\gamma_{od} - \gamma_{ad}) / \gamma_{ao} + CV^2/2\gamma_{ao}$, where C is capacitance per unit area of the hydrophobic dielectric and γ is the interfacial surface tension between the aqueous liquid (a), the oil (o), and the dielectric (d). The tested liquids included a 0.1 wt% NaCl aqueous solution that additionally included a self-dispersing pigment for coloration, and tetradecane oil containing Dow Corning Triton X-15 (a surfactant that reduces γ_{ao}). Shown in Fig. 3a are the contact angle (θ_V) vs. voltage responses for the 0.1 wt% NaCl solution on 1 and 2.5 μm thick Parylene C. The data also include tests for both 1 wt% and 5 wt% Triton X-15 in the tetradecane. A surfactant in the oil phase was implemented because an aqueous phase surfactant is often incompatible with biological species in lab-on-chip. The lowest electrowetting voltage was obtained with 1 μm Parylene C and 5 wt% Triton X-15 (Fig. 3a), and was therefore implemented in all the experiments. AC voltages were used herein to prevent electrowetting relaxation resulting from the non-symmetric nature of the Triton X-15 molecules. DC voltages can be utilized if an aqueous phase surfactant is used or if a lower γ_{ao} is achieved by a more polar oil in conjunction with a more polar dielectric.¹⁷

The basis for virtual electrowetting channel formation is now presented. For the simple model presented herein, it is assumed that the advancing meniscus edge has sufficient time to relax to well-defined contact angles, even though it continuously advances. This is because the spacing between the posts is small (~10’s of μm, fast meniscus damping) and because as will be shown later, the liquid advances at least an order of magnitude slower than the expected contact line velocity ($>1 \text{ cm s}^{-1}$). Therefore static contact angles can be used as convenient way to describe channel propagation. Both the posts and the bottom substrate are capable of electrowetting modulation. As shown in Fig. 3b (top plate not shown) for the case of $V = 0 \text{ V}$, θ_Y imparts a highly convex aqueous meniscus. A convex meniscus creates a positive Young–Laplace pressure^{7,18} calculated from $\Delta p = \gamma_{ao}(1/R_1 + 1/R_2)$, which includes the principle radii of curvature for the aqueous meniscus (R_1 and R_2). A voltage is then applied, and the aqueous contact angle is reduced by electrowetting on the side-surfaces of the posts and on the bottom substrate. With a positive Δp the aqueous liquid (blue lines) will clearly not propagate forward. However, because the top substrate is not electrowetting, no matter what voltage is applied Δp cannot be inverted (if θ_Y on the top plate is $\sim 170^\circ$, then θ_V on the bottom would need to be an impossibly small 10° or less). This is where the posts play a critical role. With posts, the system has a threshold potential (V_{th}) for liquid transport when the electrowetted contact angle θ_V causes the aqueous contact line to touch the base of the next set of posts. Once the aqueous contact line touches the next set of posts, the contact angle is then transferred from the horizontal substrate to the vertical post surface (a 90° shift). The resulting wetting geometry then inverts

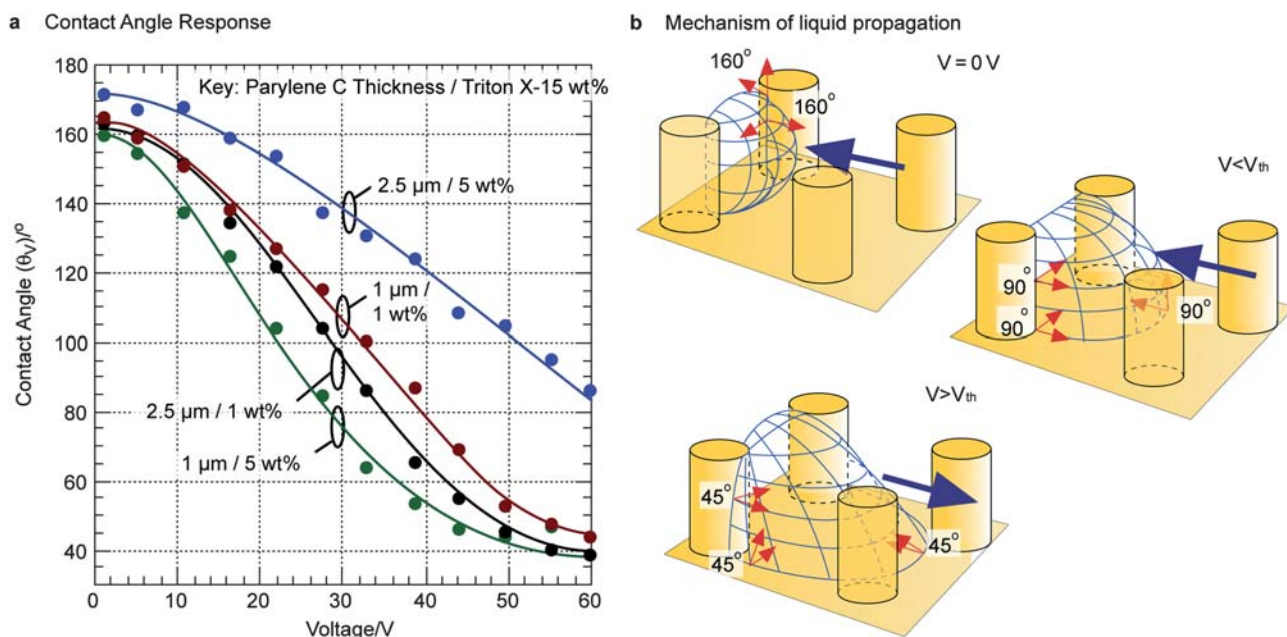


Fig. 3 (a) Electrowetting contact angle response for two different Parylene C thicknesses and two different Triton X-15 concentrations in tetradecane. (b) Diagram of the evolution of the aqueous/oil meniscus up to the threshold of virtual electrowetting channel formation (top plate not shown).

Δp and the aqueous liquid propagates forward. This ratcheting forward of the aqueous liquid will continue indefinitely, unless the applied voltage is reduced below V_{th} .

In the first set of experiments, the posts were arranged in a uniform array, which included a single large area Cu electrode. Although this testing setup cannot provide programmable channel formation, it was used to validate the theoretical model described for Fig. 3b. As shown in Fig. 4, for posts with $\sim 18 \mu\text{m}$ spacing the threshold voltage was $\sim 55 \text{ V}$ ($\theta_v < 45^\circ$, Fig. 3a). As shown in Fig. 1a and 4, a highly complex and random arrangement of channels can be formed. The reader is encouraged to view the video of this complex channel formation, which is provided in the ESI†. Fig. 4 reveals a clear increase in channel length with increasing voltage. This voltage dependence is due to finite conductivity effects like that reported by Baret *et al.* in rectangular trenches.¹⁹

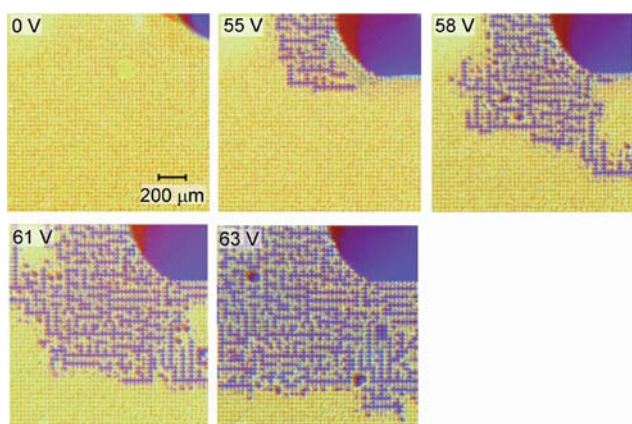


Fig. 4 Top view photographs of formation of a complex network of aqueous channels for several applied voltages.

At first glance one might expect the system to achieve continuous wetting in all directions (no distinct channels). However, distinct channels are dominant in the experimental results. Long channels form quite easily with only a few volts application above the threshold potential. Therefore the direction of liquid propagation through the posts should be highly sensitive to any local perturbations in electrical and microfluidic parameters. It is speculated that the formation of distinct channels is directly related to the electromechanical pressure that drives the electrowetting effect. Two adjacent channels might partially screen each other's electric field and slightly reduce the related electromechanical pressure.²⁰ Such behavior might be predicted using theory similar to the fractal nature of electrical tree formation in dielectrics.²¹ This discussion is of scientific interest, but practically the channel formation should not be random for lab-on-chip applications. Further investigation of distinct channel formation is reserved for future work. As will be discussed in a later section, with patterned posts, segmented electrodes, or programmable electrode arrays, the channel formation can be made predictable. Additional arrayed post experiments were performed with constant post height but with a varied array pitch between 5 and 25 μm . V_{th} was observed to be highly dependent on the spacing between posts. Some examples of experimentally validated pitches and V_{th} were $\sim 5 \mu\text{m}/\sim 60 \text{ V}$, $\sim 15 \mu\text{m}/\sim 30 \text{ V}$, and $\sim 25 \mu\text{m}/70 \text{ V}$. Generally, samples with posts pitch below $< 15 \mu\text{m}$ required higher V_{th} (large contact angle modulation) because of the increasing influence of the horizontal radius of meniscus curvature between posts. If the post spacing was too large ($> 20 \mu\text{m}$) the V_{th} also increased as the aqueous contact line on the bottom substrate has further to travel in reaching the base of the next set of post pairs. Alternate post array patterns were not explored, but patterns such as hexagonal arrays could be of further scientific and practical interest. Next, directional channel formation was explored in order to

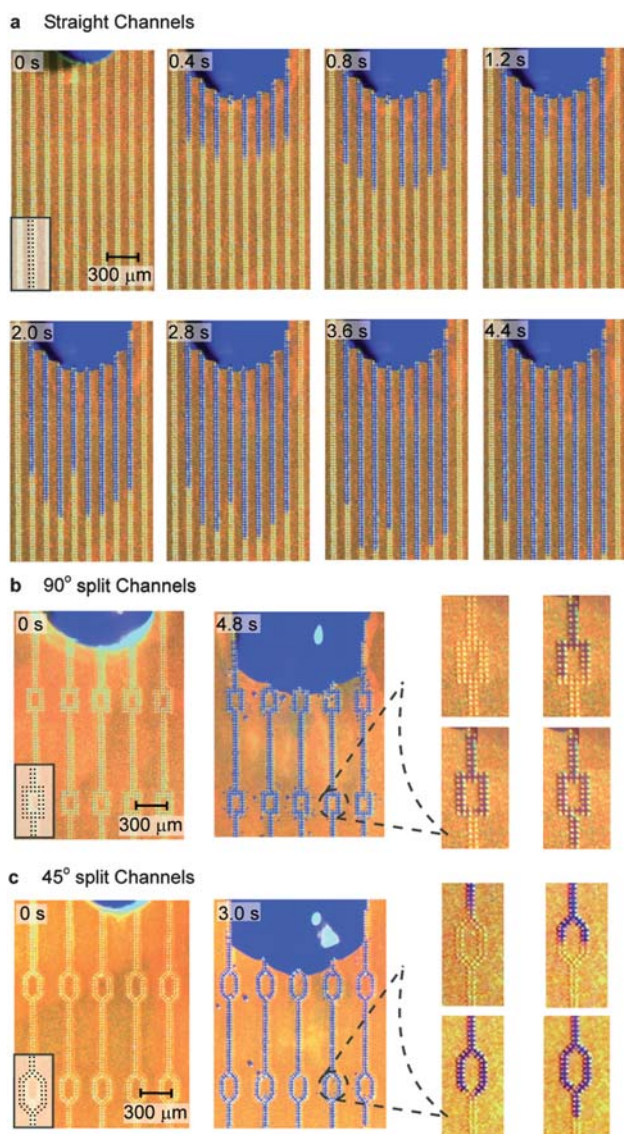


Fig. 5 Directional channel formation for preliminary demonstration of functionality for lab-on-chip: time-lapse photographs of (a) straight, (b) 90° split, and (c) 45° split channels.

demonstrate preliminary functionality for lab-on-chip. In this first report for virtual electrowetting channels, patterned electrodes were not explored because the ability to electrically control channel direction is obvious. Rather, a simpler experiment was implemented wherein the posts were arranged in particular patterns. Several linear rows of paired posts were fabricated and tested at 55 V, and time-lapse images are shown in Fig. 5a. Similarly, two sets of channel splits were fabricated and tested. The first set splits at 90°, (Fig. 5b) and the other at 45°, (Fig. 5c). These experiments demonstrate the preliminary basis for directional flow and channel splitting. Experiments were also performed (not shown) with differently colored aqueous solutions at opposite ends of virtual electrowetting channels and liquid mixing was confirmed.

Speed of fluid transport is an important factor for applications such as lab-on-chip, therefore the channel propagation velocity was measured for both the linear and splitting channels of Fig. 5.

The speeds obtained were at ~ 55 V (just above threshold). As shown in Fig. 6 the rate of liquid flow is initially ~ 800 to $1500 \mu\text{m s}^{-1}$ for all channels, and decreases to $\sim 200 \mu\text{m s}^{-1}$ after reaching $\sim 1000 \mu\text{m}$ in length. During the first 2–2.5 s the front of this electrowetting film advances as $t^{1/3}$, which is also in agreement with the work of Yeo and Chang.¹⁰ The initial $\sim 1000 \mu\text{m s}^{-1}$ speed is fast by lab-on-chip standards,²² but is still far from the 10 cm s^{-1} that can be achieved for conventional droplet-based electrowetting transport.⁶ Two factors are at play, the first being the height/length (d/L) scaling factor.²³ Because the channels demonstrated in this first report are much shorter in height (d) and longer in length (L) than the channels used in conventional electrowetting lab-on-chip, the transport speed will certainly be slower. The post height and pitch can be optimized in future work for higher speeds. The other factor limiting speed is connection of the channels to the large source droplet. The source droplet imparts a very small Δp because of its large radius of curvature. Splitting the aqueous channel from the source droplet, and then moving it, should therefore result in increased speed as the receding end would provide a large Δp to help drive the channel forward. Furthermore, a long moving liquid channel could be split into several smaller channel segments, the smaller channel segments moved in unison, and d/L ratios achieved that are similar to conventional electrowetting lab-on-chip.²³ A second and slower regime for the speed of channel formation can be seen in the plots of Fig. 6. It remains to be determined whether the second regime is dominated by increasing L in the d/L ratio²² or increasing electrical resistance¹⁸ between the voltage probe and the leading edge of the virtual electrowetting channel (Fig. 2a). This electrical resistance effect can be completely eliminated if the top substrate is provided with a transparent

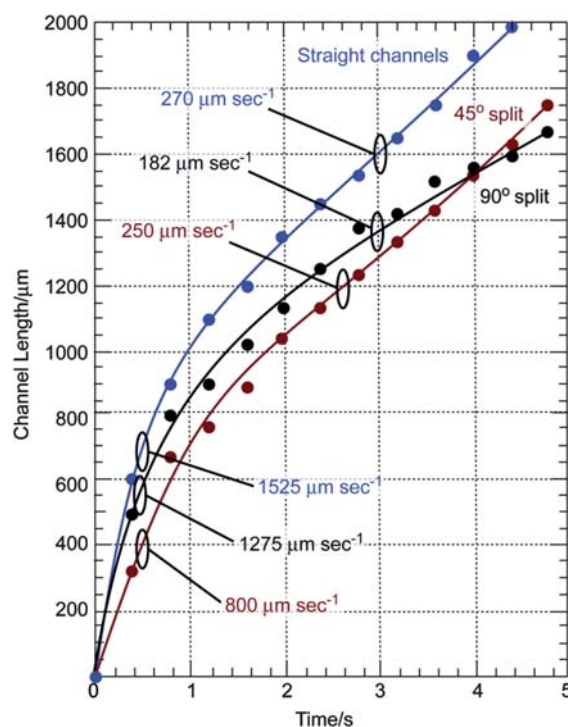


Fig. 6 Channel length vs. time for straight, 90° split, and 45° split channels at 55 V.

sheet electrode ($\text{In}_2\text{O}_3 : \text{SnO}_2$) and a thin non-insulating fluoropolymer coating. Adding topside electrode access is also beneficial for electrochemical sensing or electrophoretic separations.

With the experimental results now reviewed, potential advantages and applications are discussed for virtual electrowetting channels. First, as illustrated in Fig. 2a, this new approach allows for introduction of aqueous liquid into an electrowetting lab-on-chip module without need for syringe-pump pressure. Therefore the world-chip interface might be simplified as compared to traditional electrowetting lab-on-chip.

Second, and most important, one can potentially create any channel geometry if the posts are provided with patterned electrodes. It is fully expected that patterned electrodes could be controlled by an underlying array of thin-film transistors,¹⁴ so-called 'active-matrix' addressing. Active-matrix transistor control is particularly attractive considering arrayed transistor backplanes are widely available on glass, are inexpensive (used in most liquid-crystal displays), and can be controlled through a simple computer video-card. Thirdly, unlike traditional electrowetting devices, Young–Laplace pressure is imparted locally between posts, so long as the aqueous liquid is separated from the liquid at the inlet port, a variety of channel geometries can be preserved in the absence of voltage. As a result, for sensing or separations, additional electrical access can be applied through topside electrodes (Fig. 1b) while voltages on the bottom substrate are turned off (no interference). Channel stability is also attractive because many lab-on-chip functions require substantial time, and in some cases continuous electrowetting operation can cause eventual dielectric failure (electrolysis).²⁴

Virtual electrowetting channels may have several distinct advantages, but the platform is not without its challenges. Some challenges are similar to that for conventional electrowetting lab-on-chip, such as surface fouling, which can be reduced by use of a silicone oil¹⁷ where $(\gamma_{\text{od}} - \gamma_{\text{ad}})/\gamma_{\text{ao}} \leq -1$. The post-guided platform also includes several unique challenges. Firstly, the channel geometries demonstrated herein ($\sim 20 \mu\text{m}$) are quite small, and scale up is required for some applications. However, there is nothing inherent to the fabrication process or operation physics that would prevent channel heights in the range of $\sim 5 \mu\text{m}$ to $\sim 500 \mu\text{m}$. A second challenge is operating voltage. Although the demonstrated operation at ~ 30 – 70 V is readily compatible with conventional electrowetting lab-on-chip drivers, a long term goal might be compatibility with the $<15 \text{ V}$ operation of a typical active-matrix transistor backplane. Improved dielectric capacitance may alone resolve that challenge. Not reported herein, but under development, is a second generation platform that can

provide $<15 \text{ V}$ operation without need to decrease the dielectric thickness. This new platform is substantially unique in geometrical design, further simplifies fabrication and operation, and will be reported in a future publication.

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References

- 1 P. Dittrich and A. Manz, *Nat. Rev. Drug Discovery*, 2006, **5**, 210–218.
- 2 G. Whitesides, *Nature*, 2006, **442**, 368–373.
- 3 M. H. Oddy, J. G. Santiago and J. C. Mikkelsen, *Anal. Chem.*, 2001, **73**, 5822–5832.
- 4 B. Zhao, J. S. Moore and D. J. Beebe, *Science*, 2001, **291**, 1023–1026.
- 5 R. Fair, *Microfluid. Nanofluid.*, 2007, **3**, 245–281.
- 6 J. Lee, H. Moon, J. Fowler, T. Schoellhammer and C. J. Kim, *Sens. Actuators, A*, 2002, **59**, 259–268.
- 7 J. Berthier, *Microdrops and Digital Microfluidics*, William Andrew Publishing, NY, 2008.
- 8 P. Y. Chiou, A. T. Ohta and M. C. Wu, *Nature*, 2005, **436**, 370–372.
- 9 T. B. Jones, M. Gunji, M. Washizu and M. J. Feldman, *J. Appl. Phys.*, 2001, **89**(2), 1441–1448.
- 10 L. Y. Yeo and H. C. Chang, *Phys. Rev. E: Stat. Phys., Plasmas, Fluids, Relat. Interdiscip. Top.*, 2006, **73**, 011605-1–011605-16.
- 11 M. Abdelgawad, M. Watson and A. Wheeler, *Lab Chip*, 2009, **9**, 1046–1051.
- 12 G. Carle, M. Frank and M. Olson, *Science*, 1986, **232**, 65–68.
- 13 W. Satoh, H. Hosono and H. Suzuki, *Anal. Chem.*, 2005, **77**, 6857–6863.
- 14 J. Park, S. Kwon, S. Jun, T. Mcknight, A. Melechko, M. Simpson, M. Dhindsa, J. Heikenfeld and P. D. Rack, *IEEE Electron Device Lett.*, 2009, **30**, 254–257.
- 15 T. Jones, *J. Micromech. Microeng.*, 2005, **15**, 1184–1187.
- 16 F. Mugele and J. Baret, *J. Phys.: Condens. Matter*, 2005, **17**, R705–R774.
- 17 M. Maillard, J. Legrand and B. Berge, *Langmuir*, 2009, **25**, 6162–6167.
- 18 J. Heikenfeld, K. Zhou, E. Kreit, B. Raj, S. Yang and B. Sun, *Nat. Photonics*, 2009, **3**, 292–296.
- 19 J. Baret, M. Decre, S. Herminghaus and R. Seemann, *Langmuir*, 2005, **21**, 12218–12221.
- 20 T. Jones, J. Fowler, Y. Chang and C. Kim, *Langmuir*, 2003, **19**, 7646–7651.
- 21 S. Dodd, *J. Phys. D: Appl. Phys.*, 2003, **36**, 129–141.
- 22 M. Prins, W. Welters and J. Weekamp, *Science*, 2001, **291**, 277–280.
- 23 J. Song, R. Evans, Y. Lin, B. Hsu and R. Fair, *Microfluid. Nanofluid.*, 2009, **7**, 75–89.
- 24 B. Raj, M. Dhindsa, N. R. Smith, B. Laughlin and J. Heikenfeld, *Langmuir*, 2009, **25**, 12387–12392.