



## Scaling and mechanism of droplet array formation on a laser-ablated superhydrophobic grid



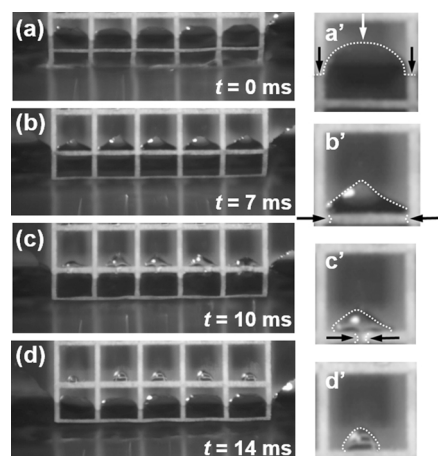
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### GRAPHICAL ABSTRACT



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### ABSTRACT

Superhydrophobic grid patterns were ablated on a hydrophobic polydimethylsiloxane (PDMS) substrate using a nanosecond pulsed laser. The substrate with a wettability contrast (superhydrophobic-hydrophobic) was vertically immersed in a pool of water and withdrawn at different speeds to form droplet arrays spontaneously. The size of droplets was tuned by controlling the withdrawal speed ( $S_w$ ), which resulted in different contact line speeds on non-irradiated cell (NIC) and laser-irradiated grid (LIG) areas with different surface properties of no-slip and partial-slip conditions, respectively. The ratio of the contact line speeds on NIC and LIG areas ( $S_{NIC}/S_{LIG}$ ) decreased exponentially with the withdrawal speed, leading to formation of larger droplets on NIC. A geometric scaling model predicted the size of droplets formed on NIC arrays over a range of contact line speeds on NIC and LIG, the ratio of the contact line speeds, the withdrawal speed, and the size of NIC.

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## 1. Introduction

Formation of droplet arrays is one of the crucial preliminary processes for biomedical and chemical applications such as bioassays, tissue engineering, and material synthesis [1–5]. Various techniques have been employed to generate arrays of droplets using contact [6,7] or non-contact dispensing [8,9] and applying fluid to surfaces with controlled wettability contrast [10,11]. In those techniques, wettability of a solid surface plays a role in the controllable formation of droplet arrays. The wettability of surfaces can be categorized into four distinct regimes, determined by the water contact angle: superhydrophilic, hydrophilic, hydrophobic, and superhydrophobic surfaces [12–15]. Water spreads and covers a large area of superhydrophilic surfaces on which the contact angle is less than  $5^\circ$ . Surfaces with contact angles larger than  $5^\circ$  and smaller than  $90^\circ$  are considered as hydrophilic. On the other hand, a surface is hydrophobic when a water droplet exhibits a contact angle larger than  $90^\circ$  and smaller than  $150^\circ$  and considered as superhydrophobic when the contact angle is above  $150^\circ$ . Patterning those surfaces on a substrate side by side achieves a heterogeneous surface with various wettability contrasts. Since the wetting of a water droplet is significantly inhibited on a superhydrophobic surface, when a water droplet is applied on a substrate with controlled wettability contrast, such as patterned superhydrophilic-superhydrophobic (high wettability contrast), hydrophilic-superhydrophobic (medium wettability contrast), or hydrophobic-superhydrophobic (low wettability contrast) surfaces, the water droplet tends to wet selectively the superhydrophilic, hydrophilic, or hydrophobic areas (Wenzel state) [16] without wetting the superhydrophobic area. The superhydrophobic area will remain dry by the trapped air between the water and the surface structure in those areas (Cassie state) [17]. Therefore, the fluid can be passively dispensed only on the desired area using controlled wettability contrasts without any manual pipetting or liquid handling devices.

Previously, formation of droplet arrays on surfaces with various wettability properties has been achieved using various methods such as dip-coating (immersion and withdrawal) [18–20] or rolling [19] and sliding [21–23] the droplet across the surface. Hjort et al. used a gravity-induced sliding method for parallel deposition of multiple different liquids on a patterned hydrophilic-superhydrophobic surface [22]. Levkin et al. fabricated a patterned superhydrophilic-superhydrophobic surface for high-throughput screening of live cells [19]. Arrays of thousands of individually separated microdroplets were generated on the patterned surface by dipping the substrate into water or rolling a droplet across the surface. Song et al. deposited arrays of droplets by sliding a droplet on the patterned superhydrophilic-superhydrophobic substrate to form arrays of isolated single cells [21]. The size of the droplets was controlled by adjusting parameters such as the size of the hydrophilic area, the contact force, and the relative sliding speed of the droplets on the patterned substrate. Wang et al. generated arrays of cell-encapsulated hydrogels with semi-egg-like architecture on a surface consisting of hydrophobic pads with controlled contact angle hysteresis and a superhydrophobic background with ultra-low contact angle hysteresis via dip-coating [18]. It was reported that the contact angle hysteresis of the hydrophobic pads and the viscosity of the liquid are critical parameters to determine the droplet size. However, the effect of the withdrawal speed ( $S_w$ ) was not systematically studied and the mechanism of droplet array formation was not discussed. Krumpfer et al. also demonstrated the deposition of small water droplets on top of high aspect ratio staggered micro-rhombus posts coated with silane molecules of superhydrophobic properties by manual dip-coating. The mechanism of capillary bridge formation between the top of the posts and the bulk of fluid and the subsequent rupture of the bridge during withdrawal was proposed to describe droplet formation on top of the microposts [20]. Dufour et al. also reported deposition of small amounts of liquid by sliding a droplet on top of a super-omniphobic surface made up of an array of overhung microposts [23]. Similarly,

deposition of a small amount of liquid on top of each post was attributed to formation, stretching, and break-up of the capillary bridges between the post tops and the bulk liquid.

Most of the methods for droplet array formation utilized heterogeneous surfaces with wettability contrasts, which require multiple sophisticated fabrication steps with limited substrate materials [18,19,21,22]. Levkin et al. fabricated a superhydrophilic-superhydrophobic patterned surface by preparing a superhydrophilic thin film of nanoporous polymer through photopolymerization of a pre-polymer mixture. Then, superhydrophobic grid-like patterns were fabricated on the superhydrophilic surface by UV-initiated photografting [19]. In the work by Song et al., a silicon substrate was electrochemically etched with  $\text{HF}/\text{AgNO}_3$  solution, followed by chemical vapor deposition of silane molecules to have the superhydrophobic thin film on the substrate [21]. Superhydrophilic patterns were then created on the prepared superhydrophobic surface by photolithography. In contrast, laser treatment allows repeatable, precise, and controllable surface modifications of a variety of materials including polymers [24–26]. The desired patterns can be directly written on the surface via laser irradiation by using a computer-controlled system. Moreover, the wetting properties of various materials can be changed without any chemical coating step [27]. In a previous work [28], we discussed that laser treatment can be used for the patterning of polydimethylsiloxane (PDMS) substrate with a moderate wettability contrast consisting of hydrophobic areas surrounded by superhydrophobic grids. We also demonstrated that droplet arrays can be formed by simply dipping and withdrawing the laser-patterned PDMS substrate, and the size of formed droplets can be tuned by controlling the pitch size of the superhydrophobic grid and the withdrawal speed. However, the mechanism of droplet array formation on the laser-patterned surface and the correlation between parameters affecting the droplet size were not fully elucidated.

In this study, a laser-ablated platform with moderate wettability contrast (hydrophobic/superhydrophobic) was fabricated in a single step by writing superhydrophobic grid patterns on an intrinsically hydrophobic PDMS without any further chemical treatment. As the laser-ablated PDMS was withdrawn from water at different withdrawal speeds ( $S_w$ ), the dynamics of three-phase contact lines on the laser-irradiated grid (LIG) and non-irradiated cell (NIC) areas were investigated to identify the mechanism of droplet array formation. The size of droplets produced on the grid can be controlled by varying  $S_w$ . A simple scaling model was developed to predict the size of droplets over a range of contact line speeds on superhydrophobic LIG ( $S_{\text{LIG}}$ ) and hydrophobic NIC ( $S_{\text{NIC}}$ ) surfaces. We showed the accuracy of the scaling model experimentally by measuring the droplet size as a function of  $S_w$ ,  $S_{\text{LIG}}$ ,  $S_{\text{NIC}}$ , and the geometrical dimension of the grid. The array of the droplets whose sizes are controlled can potentially be used as templates for the patterning of particles, chemicals, and biomolecules in aqueous solutions.

## 2. Experimental

PDMS substrates were prepared by mixing Sylgard® 184 silicon rubber bases and curing agent (Dow Corning, Midland, MI) at a weight ratio of 10:1. After removing air bubbles by placing the mixture in a vacuum desiccator for 20 min, the liquid PDMS mixture was poured into a glass petri dish up to a height of 3 mm, vacuumed again, and cured at  $65^\circ\text{C}$  for 4 h in a convection oven. The cured PDMS was then peeled off from the glass petri dish and cut into  $3\text{ cm} \times 8\text{ cm}$  rectangular pieces using a razor blade. A laser-patterning system and laser processing parameters described in [28] were employed in this experiment. A superhydrophobic grid pattern with a pitch size of 2 mm and a grid width of  $274 \pm 9\ \mu\text{m}$  was written on the PDMS surface. The static contact and sliding angles of  $5\ \mu\text{L}$  distilled water droplets were measured using a contact angle analyzer (Falcon, First Ten Angstroms, Portsmouth, VA) and a manual goniometer (GN05, Thorlabs Inc.,

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