Automated Digital Magnetofluidics

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Abstract. Drops can be moved in complex patterns on superhydrophobic surfaces using a reconfigured computer-controlled x-y metrology stage with a high degree of accuracy, flexibility, and reconfigurability. The stage employs a DMC-4030 controller which has a RISC-based, clock multiplying processor with DSP functions, accepting encoder inputs up to 22 MHz, provides servo update rates as high as 32 kHz, and processes commands at rates as fast as 40 milliseconds. A 6.35 mm diameter cylindrical NdFeB magnet is translated by the stage causing water drops to move by the action of induced magnetization of coated iron microspheres that remain in the drop and are attracted to the rare earth magnet through digital magnetofluidics. Water drops are easily moved in complex patterns in automated digital magnetofluidics at an average speed of 2.8 cm/s over a superhydrophobic polyethylene surface created by solvent casting. With additional components, some potential uses for this automated microfluidic system include characterization of superhydrophobic surfaces, water quality analysis, and medical diagnostics.

1. Introduction

In the last decade there have been many research articles on superhydrophobicity and superhydrophobic surfaces, even though this has been an area of study since at least the 1940s, spearheaded at that time by the textile industry’s desire for increased water repellency [1-3]. It is also important to note that an understanding of how liquids interact with rough surfaces origins is seen in the literature since the 1930s due to the pioneering work of Cassie and Wenzel in the 1930s and 1940s [4-6].

Many different types of superhydrophobic surfaces have been fabricated [7-25], and surface characterization is typically done by using a goniometer to measure the contact angle of a drop on the surface. Advancing, receding, and/or sessile [11] contact angles are usually used to characterize the hydrophobicity and overall ability of the surface to repel water. However, this does not provide a full view of the quality of superhydrophobic surfaces in drop microfluidics systems. In digital Magnetofluidics, a superhydrophobic surface that has very high water contact angles also needs to foster drop movement, manipulation, and analysis in order for it to be effective as a platform to manipulate and analyze the components of a drop.

Prior work by our group [25-28] has demonstrated the concept of digital microfluidics using manual manipulation of magnetic fields to control the movement of drops on a superhydrophobic surface. This work has established that digital magnetofluidics is a flexible system for analysis with unique capabilities that may prove useful in water analysis and biological fluids diagnostics. The automated device described in this paper is one means of automating digital magnetofluidics,
permitting focused, quantitative research, and an important step towards designing a commercially viable device.

The needs of our work parallel the needs described by others working in digital microfluidics, particularly as it relates to developing a flexible testing system based upon individual drops. A flexible system would allow reconfiguration of a platform or the ability to potentially run several tests simultaneously as a point of care application. Flexibility and adaptability will be important to hold down costs. This need for flexibility will become even more important in the future with the advent of personalized medicine. [29, 30].

Digital microfluidics architecture differs in many ways from continuous flow microfluidic systems such as MEMS. In digital microfluidic architecture, the basic liquid unit volume is fixed by the geometry of the system (fluid quantization), and volumetric flow rate is determined by the droplet transport rate and the number of droplets transported. Transport occurs in multiples of the minimum unit volume (fluid packetization) [31], and digital microfluidics [31] has been performed using several different driving forces including electrowetting [32-34], dielectrophoresis [35], thermocapillary transport [36], surface acoustic wave transport [37], and magnetic techniques [38-42].

Most microfluidic devices to date have been purpose-built devices (e.g., MEMS) populated with custom made fluidic components such as pumps, valves, detectors, sensors, etc. More importantly, such devices are application-specific, and a similar device for a related application must be designed largely from scratch. In other words, because these technologies are not digital, they are not readily reconfigurable, in the same way that an analog electronic computer cannot be as easily rewired for a different application as a digital computer can be reprogrammed. In contrast, digital microfluidic devices may achieve a true lab-on-a-chip device, in that it can be easily reconfigurable to a new application with the same ease as a chemistry laboratory bench top since they can be reconfigured just by software changes [31].

The type of digital microfluidics described in this paper, namely digital magnetofluidics, uses the motion of magnetic fields to manipulate discrete liquid drops on a superhydrophobic surface. Drops can be mixed, split, coalesced, and moved. Magnetizable particles are introduced into the liquid. Under the influence of a moving magnetic field, these particles move and so entrain the liquid drop as well. The combination of a relatively strong driving force and a superhydrophobic surface allows few requirements placed on the solution, and even solutions several hundred times more viscous than pure water can be moved [26] which is important since biological fluids can have much higher viscosities than water due to the presence of soluble long chain biopolymers [43, 44]. In order to translate these capabilities into a useful device, we identified the automation of drop movement which can be altered through software control as an important first step.

2. Methods and materials

2.1. Superhydrophobic surface

The genesis of the protocol for solvent casting large superhydrophobic sheets began with the work of Erbil et al [23] and Lu et al [24]. Erbil dissolved polypropylene in xylene, and experimented with methyl ethyl ketone (MEK), cyclohexanone, and isopropyl alcohol as nonsolvents. Nonsolvents promote precipitation by increasing the extent of polymer phase separation (between solvent + nonsolvent, and polymer + solvent); and by increasing the nucleation rate. Using these methods, he was able to achieve contact angles of up to 160° by applying this solution to small, rigid substrates such as microscope slides.

Lu dissolved low density polyethylene (LDPE) in xylene, using cyclohexanone for the nonsolvent). These experiments were aimed at controlling crystallization behavior by adjusting crystallization time and nucleation rate. Using small silicon wafers as the substrate, contact angles of up to 170° were achieved.

Because of its inherent hydrophobicity, low cost, and flexibility in manufacturing large sheets, we decided to use LDPE as the substrate to develop the automated system. Ninety-six combinations of
nonsolvent (cyclohexanone, methyl ethyl ketone [MEK], and 50:50 v/v cyclohexanone/MEK); LDPE concentration in xylene (10, 20, 30, 40 mg/ml); and solvent-nonsolvent ratio (0.75:0.25, 0.65:0.35, 0.55:0.45, 0.45:0.55 v/v) were tested. The surface made with 30 mg/ml LDPE and 0.55 xylene: 0.45 MEK was chosen for all future research, as this surface displayed the highest contact angle with the highest reproducibility of contact angle. Subjectively, this surface also displayed the best appearance (homogeneity, opacity indicating good coating thickness, lack of cracks, and lowest sliding angle for water drops).

Commercial-grade LDPE sheet, 1.59 mm (1/16 inch) thick was purchased from McMaster-Carr (Los Angeles, CA). The materials to grow the LDPE crystals comprised LDPE pellets (Sigma-Aldrich, St. Louis, MO) with a melt index of 2.50 g/min at 190ºC, xylene (isomers plus ethylbenzene, reagent grade, Sigma-Aldrich), and methyl ethyl ketone (MEK) (reagent grade, J. T. Baker, Phillipsburg, NJ). The LDPE sheet was cut into 61 x 99 mm (2.40 x 3.90 in) rectangles, lightly abraded, and cleaned with acetone. The LDPE pellets and chemicals were used without additional preparation.

Preliminary experiments had shown that there were losses (spillage of the solution off the edges of the sheet) and insufficiently even coating thickness (primarily due to slight curvature of the LDPE sheets as supplied). To give greater coating uniformity, fixtures were constructed for the solvent casting process. These fixtures, consisting of a base and a cover, were machined from 6061 aluminum. When the plastic sheet was clamped between the two pieces, a seal was formed at the edge of the LDPE sheet substrate, in effect forming a “bathtub” into which the solution could be pipetted without loss. Maintaining a constant depth of solution over the area of the substrate gave a more uniform deposition of LDPE crystals.

The same preliminary tests had shown a tendency for the sample to bow in the middle, even when held in the solvent casting fixture. Presumably this was caused by swelling of the LDPE in hot xylene. For better coating uniformity, some means of mechanical restraint were needed to hold the LDPE sheet flat during drying. It was found that the substrate could be heat-softened in an oven and bonded under pressure to a perforated metal sheet (26 Ga. [0.4 mm] brass, 1.15 mm hole diameter, 1.68 mm hole spacing center-to-center, 37% open area, McMaster-Carr, Los Angeles, CA), and this sandwich clamped in the solvent casting fixture. After the solvent had evaporated and the sample removed from the fixture, the substrate could be carefully removed from the perforated metal sheet. To prevent instant crystallization when the hot solution contacted the cold LDPE sheet, and to slow the crystallization rate, the fixture and LDPE/brass sandwich were preheated to 65oC before use.

Xylene and LDPE pellets were placed in a flask and immersed in a water bath at 92ºC. After the LDPE had fully dissolved (approximately 35 min), the MEK was added to the flask. The addition of the nonsolvent to the solvent-plastic solution was shown by Erbil et al [23] to increase surface roughness and aqueous drop contact angle for solvent-cast polypropylene. After the cloudiness caused by the MEK addition cleared, the bath was reduced to 85ºC. This heat soak technique gave better surfaces, presumably by promoting greater crystal formation while still in solution. After 60 min, 5.25 ml of solution were pipetted onto the surface in the solvent-casting fixtures. This volume gave a density of approximately 0.1 ml/cm². The fixtures were placed under overturned deep crystallizing dishes (as slower evaporation promoted better crystal growth) and left in a fume hood to dry at room temperature. When dry, this resulted in a density of crystalline LDPE of 1.65 mg/cm².

Contact angles at three representative areas of each surface were tested using a contact angle goniometer (NRL Contact Angle Goniometer, model 100-00, Rame-Hart, Mountain Lakes, NJ). Drops of deionized were dispensed using a micrometer syringe (Gilmont Instruments, Cole-Parmer, Vernon Hills, IL). Both advancing and receding contact angles were measured.
2.2. Automated stage

The x-y-z stage hardware of the automated testing system was originally designed and built for metrology and inspection in the semiconductor industry. The stage was fabricated by Molecular Devices (Sunnyvale, CA), with major parts machined by Mecpro (Santa Clara, CA). The device consists of a thick aluminum base (38 x 60 x 1.2 cm), stress-relieved and ground flat to metrology standards. As received, there were four sets (one in the x-direction, two in the y-direction, and one in the z-direction) of high-precision linear bearings (SEBS 12A, NB Corporation, San Jose, CA) installed. These bearings have basic load rating of 2.60 kN dynamic and 3.20 kN static per block, and so were well within their ratings for our application. As specified by the factory, motion accuracy (running parallelism) is within 12.5 um over the 47 cm length of the x-direction guide rail, and within 8.5 um over the 25 cm length of the y-direction guide rails. The x- and y-direction bearings were rebuilt by the factory; the z-direction bearing was removed for this research.

The lower carriage rides upon a total of four bearing blocks on the two y-direction linear bearings, with a total travel of 11 cm. The smaller upper carriage rides upon the single x-direction linear bearing (with two bearing blocks) mounted on the lower carriage, with a total travel of 37 cm. By simultaneous movement along the x- and y-axes, complex motion of the upper carriage can be achieved.

The carriages are driven by leadscrews powered by brushless 24 VDC servomotors with optical encoders and hall sensors (ELCOM SL, p/n 4443S013, Pittman, Harleysville, PA). The lead screws have a quadruple lead and a pitch of 0.39 threads/mm (10 threads/in), giving a lead of 10.16 mm/rev (0.4 in/rev). Each lead screw drives a close-tolerance nut assembly. Each nut assembly consists of two delrin nuts in a spring-loaded arrangement so as to minimize backlash. The assembly is in turn hard-mounted to the carriage structure. At 24 VDC, the servomotors have a maximum speed of 5800 rpm, corresponding to a potential maximum linear speed of the carriage of 0.98 m/sec.

The three-channel optical incremental encoders are p/n HEDS-9140 (Avago Technologies, formerly Agilent, San Jose, CA), with two channel quadrature outputs plus a third channel index output, and with a resolution of 500 counts per revolution. The maximum worst-case error (e.g. per factory specifications) of the encoder is 40 min of arc over a full revolution of the encoder code wheel, with a “typical” worst-case error of 10 min of arc. At the maximum worst-case encoder error (40 min of arc = 1/900 rev = 0.00111 rev), this corresponds to a linear error of position of 11.3 micrometers [(10160 um/rev)*(0.00111 rev)].

An acrylic holder was machined to house the neodymium-iron-boron (NdFeB) magnet used to manipulate liquid drops. The N40-grade (moderate strength) magnet used (p/n NSN0619-N40, Magcraft, Falls Church, VA) is 6.35 mm diameter x 25.4 mm long (0.250 in x 1.000 in) with a tensile pull force of 17.1 N (corresponding to approximately 6 N in shear), a surface field of 4910 G and a magnetic energy product of 40 megagauss-oersteds (MGOe), or 318 kT/m3.
A motor controller, amplifier, power supply, and connectors completed the automated stage. The 3-axis controller (DMC-4030, Galil Motion Control, Rocklin, CA) also incorporates the onboard 500W 4-axis servo amplifier (AMP-43040, Galil) and interconnect module (ICM-42000, Galil). An offboard regulated DC power supply (CPS-12-24, Galil) powers the controller. The power supply is rated for 24 VDC at 12 A continuous, and includes a shunt regulator and blocking diode.

The AMP-43040 servo amplifier contains four transconductance, PWM amplifiers for driving brushless or brush-type servo motors at up to 7 A continuous (10 A peak) at 20-80 VDC. The PID (proportional-integral-derivative) gain settings of the amplifier are user-programmable. The switching frequency is 60 kHz.

The DMC-4030 controller employs a RISC-based, clock multiplying processor with DSP functions, accepts encoder inputs up to 22 MHz, provides servo update rates as high as 32 kHz, and processes commands as fast as 40 milliseconds. The controller is able to operate in stand-alone mode, although for our research it is usually controlled by a laptop via a 10/100BASE-T Ethernet. As currently configured, it provides a trapezoidal commutation for the brushless motors. Power connections are via Molex connectors, and signal connections (encoder and Hall sensor) are via D-sub connectors.

Programming is accomplished with the Galil-supplied BASIC-like instruction set, although LabVIEW (National Instruments, Austin, TX) and other programming languages can also be used. Standard programming features include PID (proportional-integral-derivative) compensation with velocity and acceleration feedforward, dual-loop control for backlash compensation, velocity smoothing to minimize jerk, multitasking, and I/O processing commands for synchronizing motion with external events. Modes of motion include point-to-point positioning, position tracking, jogging, linear and circular interpolation, contouring, and electronic gearing and electronic cam.

Figure 2. Approximate isometric view of automated stage, showing x-axis servomotor, y-axis servomotor, controller, lower stage, upper stage with magnet and holder, and y-axis linear bearing.
3. Results

3.1. Superhydrophobic surface
Goniometer measurements of these superhydrophobic surfaces showed that advancing contact angles of 160±2º and receding contact angles of 155±2º could be achieved with high inter- and intra-sample uniformity; these measurements and tolerances were usually applicable to all five samples made in one batch. Over 100 of these samples have been made, and there is no measurable deterioration even in the oldest samples, which have been stored for over a year in typical laboratory conditions.

(a)          (b)                (c)

Figure 3. SEM images of solvent cast LDPE superhydrophobic sheet: (a) 50X; (b) 600X; (c) 1500X. The similar appearance of details at different magnifications is indicative of roughness at more than one length scale (e.g. fractal roughness).

3.2. Automated stage
A drop motion profile was programmed and run. This profile demonstrates some of the capabilities of the automated stage, and consisting of axial, diagonal, circular, and sine wave movements, all with a linear acceleration ramp. A 20 microliter aqueous drop (with 2 wt% polyxiloxane-coated paramagnetic carbonyl iron microparticles) starts at the upper right corner of the LDPE sample. The drop starts at the upper right corner, and moves about the perimeter of the sample in a CCW direction. When returning to its starting point it then travels on a diagonal to the far corner, moves vertically up to the other corner, and then diagonally to the other opposite corner, then vertically halfway to the starting point. Here it travels horizontally to the left one-fourth of the distance, and then completes two full revolutions CCW, as circumscribed by the top and bottom of the sample. After this is completed, it travels the rest of the way to the left edge, and then beginning traveling upwards it makes one full sine wave along the horizontal axis. At the rightmost edge it continues traveling upwards and completes a sine wave to the left, then retraces its original sine wave to the right, ending back at the starting point. This is a distance traveled of over 1 m (1116 mm) over a total area of only 27.4 cm². Total time of travel was approximately 40 s, for an average speed of 2.8 cm/s.

This speed was limited by the servomotor acceleration profile, not any decoupling of the drop from the driving force. With further tweaking of servomotor parameters it will be possible to increase the speed over the course. It should also be noted that more than 20 full cycles (total traversed length >20 m) of this motion profile have been run continuously, with no intervention required after sample loading. There was no tendency to instability, and at the end of the cycle the drop returned to its starting point.
Figure 4. Sequential still images of drops on automated motion profile: (a) static, in starting position; (b) moving vertically downward after axial move in –x direction and nearing end of move in –y direction; (c) moving vertically upwards after axial move in +x direction and beginning axial move in +y direction; (d) moving toward lower left during diagonal move from upper right corner to lower left corner.

4. Discussion

This automated stage has demonstrated the ability to run a complex drop motion profile with no human intervention beyond drop loading. Although this experiment was performed with a drop of water, this is not a limitation of the system. As earlier research [26] by our group achieved facile movement of 20 microliter drops of Dextran 428 (20% wt/v, 125 mPa-s viscosity) using manual manipulation of a NdFeB magnet, it is likely that drops of similar viscosity could be moved using this automated system. This is a much higher viscosity than that reported with other digital microfluidic techniques (electrowetting-on-dielectric achieved movement of 10mg/ml BSA solutions [45, 46] which corresponds to a viscosity of approximately 3 mPa-s [47]).

The drop speed achieved with this automated system, 2.8 cm/s, is moderate compared to electrowetting-on-dielectric, which has reported speeds as high as 25 cm/s for small (<0.5 ul) drops [31]. However, the speed of this device is significantly faster than that of other magnetically driven methods [39, 42] which report speeds of 2.5 mm/s or less. As the speed of the automated device in this experiment was limited by the servomotor control parameters and not by anything inherent in the
drop or the magnetic particles or the surface, it is likely that higher drop movement speeds can be achieved in the future with tailoring of the motion profile parameters.

During our testing of many superhydrophobic surfaces made by LDPE or silicon nanowires, we have found that contact angle measurements are not reliable indicators of its utility for moving drops using digital magnetofluidics. Dettre and Johnson discussed contact angle hysteresis as an important parameter to characterize surfaces [48, 49] and more recently, McCarthy and colleagues discussed how roughness at two or more length scales is an important consideration in characterizing surface hydrophobicity [9, 50, 51]. Other researchers such as Yang and colleagues have expanded this concept to note that fractal roughness is important [52]. As shown above in figure 3, LDPE surfaces prepared for this research possess fractal roughness. More importantly, roughness on two or more length scales is not readily quantifiable, nor is there any guarantee that other important characteristics have still not been identified to quantify superhydrophobic surface quality.

What is needed, then, is a quick method to quantitatively rate superhydrophobic surfaces. We propose that an automated system could accomplish that task. Once programmed with the desired motion profile, the system could run without intervention for as long as needed to measure the ease of drop movement, surface erosion, surface degradation, etc. Optical or other sensors could easily be added (this controller has 56 additional I/O ports for just that purpose) to extract detailed information on the maximum velocities than can be achieved under conditions with drop size and magnetic microparticle concentration.

Microfluidic technologies have been used for biomedical applications such as electrophoretic separations [53], DNA analysis [54, 55], protein/enzyme analysis [56], immuno- and bioassays [57-60], and pathogen detection [61, 62]. Clearly, there is a potential for microfluidic techniques to supplant existing diagnostic tests. Besides the capability for automation which has been demonstrated here, there are other features of digital magnetofluidics which may be advantageous over other digital microfluidic systems in some testing applications. Because the driving force is spatially removed from the superhydrophobic surface, unlike technologies such as electrowetting-on-dielectric, a low-cost substrate can be used without the need for complex manufacturing of microcircuits. Another potential advantage of this technology is the absence of an oil bath, unlike other digital microfluidic technologies, which avoids the potential concerns of protein adsorption and entrapment (emulsification) at the water-oil interface [63], and liquid-liquid phase transfer [64]. Some digital microfluidic technologies also have difficulty moving solutions with higher concentrations of proteins (>2 micromoles per liter in water) [64], in addition to the viscosity limits mentioned above. The high driving force of this device, coupled with the spatial separation of this force from the drop, means that there are fewer limits imposed on the sample.

5. Conclusions
Microcrystalline superhydrophobic LDPE surfaces useful for microfluidic movement using magnetic fields, and with minimal inter- and intra-sample variation can be easily manufactured. These substrates are integrated into an automated system to move droplets along a complex drop course at an average speed of 2.8 cm/s for over 1 meter. More than 20 full cycles (total traversed length >20 m) of this motion profile have been run continuously, with no intervention required after sample loading.

Along with prior research demonstrating manual drop manipulation of rare earth magnets, digital magnetofluidics shows promise as an automated device with programmable, accurate, and complex movement with potential applications in water quality analysis and medical diagnostics. In addition, the repeatability of the automated system may lead to a quick and inexpensive way to gather data that would help compare superhydrophobic surfaces.

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