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Modification and Characterisation of Material Hydrophobicity for Surface Acoustic Wave Driven Microfluidics

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***Abstract*—Surface acoustic waves (SAW) generated in a piezoelectric substrate may be used to manipulate micro-scale droplets of liquid in a digital microfluidic system for lab-on-a-chip applications. The wettability of the surface over which a droplet is driven determines the ease and speed with which the droplet is propelled. This provides the opportunity to achieve fine control of SAW driven droplets simply by patterning of the surface into areas with different levels of wettability. This paper evaluates a number of different materials and surface preparation techniques and assesses their manufacturability and efficacy for this application. Test structures have been designed and developed to help optimise a fabrication process using the biocompatible polymer Parylene. Early results obtained using airflow as a driving force show that it is possible to manipulate droplets through direction changes of up to 60°. Additional work has been done using surface acoustic waves as the driving force to determine the extent to which droplets can be guided to desired locations.**

I. INTRODUCTION

The manipulation of discrete droplets of liquid with µl or nl volumes is often referred to as digital microfluidics. This technology can have many advantages over more traditional lab-on-a-chip (LOC) systems using microchannels, not least in the reduction in the volume of expensive reagents required. Surface acoustic waves (SAW) are often used in the manipulation and sensing of droplets in a digital microfluidic system but it would require relatively complex control systems to achieve precise control over the positioning of droplets with SAW.

One possible method to address this issue is to pattern the surface of a microfluidic chip with areas of high and low wettability. A droplet of aqueous fluid placed on a boundary between regions of differing wettability will tend to favour the region which is more hydrophilic. This effect has been used in the past to control the transport of droplets in a passive system [1]. In this work, the capability of an active actuation system, which uses a combination of SAW and wettability control to guide the movement of droplets, is examined.

If this is to be successful, it will be important to understand the materials and processes used to fabricate surfaces with different characteristics. For example, although a large difference in hydrophobicity is useful in guiding a droplet it is difficult to actually move the droplet if it is on a strongly hydrophilic surface. In addition, some of the processes used to alter the wettability may create a surface with unstable surface properties that change over time. Understanding these issues is the purpose of this study, which investigates two candidate materials and a range of processes for altering their surface properties.

II. TEST STRUCTURES

Test structures have been designed to determine the extent to which a droplet moving under the influence of an external driving force can be manipulated. Specifically, the structures test how much of a directional change the droplet can be forced through when it experiences the boundary between two surfaces of different wettability. The test structure mask designed for a 3" (75mm) diameter wafer is shown in figure 1. Each design is of a Y-shaped structure, in which the more hydrophilic regions are shown in black and the more hydrophobic areas are shown in white. There are a total of twelve chips on the test mask, comprising four different layouts, each repeated three times. The angle of slope of the more hydrophobic area structures has been varied to give a range between 10° and 45°. Larger angles can be achieved by rotating the mask by 90° (to obtain 45° to 80°) if required. The concept for the microfluidic control requires that a droplet be placed on a more hydrophilic surface upstream from a sloped boundary to a more hydrophobic area, as shown schematically in figure 2. The SAW energy is then applied so that the resultant wave travels from left to right on the chip. When the droplet reaches the boundary between hydrophilic and hydrophobic surfaces, it will either be directed along the boundary edge or forced over the boundary. The direction the droplet takes will be dependent on the angle of the slope and the SAW energy applied, and it is these parameters that will be used to characterise the technology.

The initial test was planned with air blown droplets with the plan to move on to SAW devices with hydrophobic surface treatments. In order to minimise the demand for relatively expensive SAW substrates, it was decided to use a single

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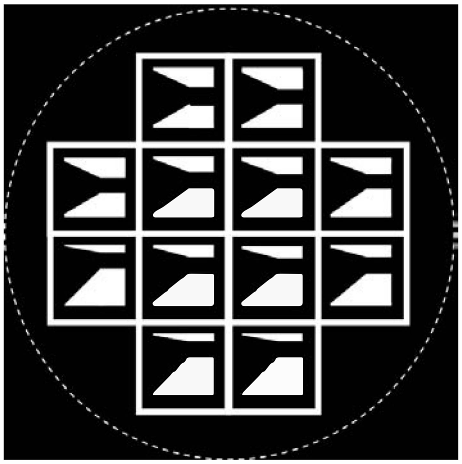


Fig. 1. Test mask for SAW actuated droplet manipulation

SAW device and to fabricate separate "superstrate" chips with the test structure patterned surfaces. The SAW energy is transferred to the superstrate from the underlying piezoelectric substrate through a liquid coupling layer as described in [2]. The key advantage of this method is that the superstrate can be quickly and cheaply replaced with an alternative design if so required; however, a disadvantage is the loss of acoustic energy when it is transmitted into the superstrate.

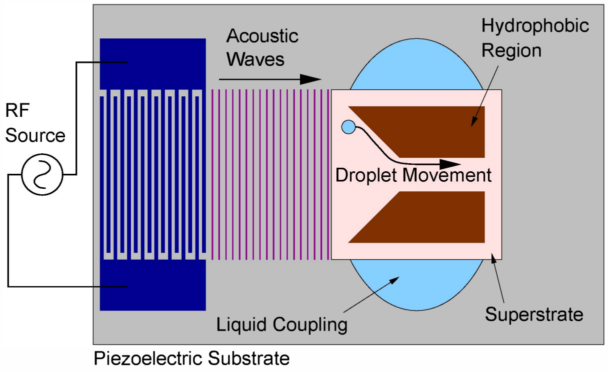


Fig. 2. Schematic diagram of a digital microfluidic system using SAW energy transmitted to a superstrate chip via a liquid coupling layer

III. MATERIALS AND FABRICATION

The principal surface materials under investigation in this study are Parylene-C and CYTOP. These have differing levels of wettability and could be used to create the surface boundary necessary to guide a droplet.

Parylene or poly(para-xylene), is commonly used to protect printed circuit boards from moisture and contaminant attack or as a biocompatible coating for medical implants such as stents. It has also been used as a dielectric layer in electrowetting on dielectric (EWOD) devices [3] and also for confining aqueous solutions in reservoirs [4]. It is deposited using a room-temperature, vapour deposition process to create conformal, pinhole free coatings. Although it provides a reasonably hydrophobic surface as-deposited, the surface can be altered to make it more hydrophobic with a CF_4 plasma treatment [5] or super-hydrophilic by exposure to an oxygen plasma [6]. In this study, Parylene was deposited using a PDS 2010 Labcoter from Speciality Coating Systems.

CYTOP is a fluoropolymer, developed by AGC Chemicals, which is routinely used to provide a hydrophobic coating in EWOD systems. It is deposited in a thin layer (∼20nm) by spin coating a substrate with a dilute solution of CYTOP followed by baking in an oven to remove the fluorinated solvent. It can be patterned using standard photolithographic processing with a high viscosity photoresist and subsequent oxygen plasma etching.

The requirement for the proposed application is for a surface patterned with regions of differing wettability. This presents the option of using two different materials or selectively modifying the surface of a single material. Parylene is less hydrophobic than CYTOP and so the SAW superstrate chips could have an initial coating of Parylene with a patterned CYTOP layer on top. The alternative is to use Parylene alone, but to pattern it through selective exposure to CF_4 or O_2 plasma in a reactive ion etch (RIE) tool to alter its surface properties.

IV. PROCESS DEVELOPMENT

In order to develop an optimised process, it is necessary to characterise the wettability of these materials before and after exposure to various processes, and to monitor any change in wettability over time. This was achieved by measuring the contact angle (CA) of a de-ionised water droplet on the surface under test using a goniometer system. Further details of the measurement system can be found in a previous publication [7]. A CA>90° defines a material as hydrophobic while a CA<90° means that the material is hydrophilic. Initial figures for the two materials deposited on glass substrates were ∼89° for freshly deposited Parylene C and ∼115° for CYTOP. Samples of these materials have undergone a variety of processes in order to alter their surface properties. Due to inconsistencies in the results for CYTOP surfaces, the focus in this abstract will be on the use of Parylene. The Parylene surfaces were subjected to the following processes: O_2, CF_4 and Argon plasmas, and photoresist (Shipley SPR350) coating followed by an acetone rinse. The results are summarised in table I.

TABLE I
CONTACT ANGLE MEASUREMENT RESULTS FOR PARYLENE

Treatment Process	Contact Angle
As deposited	89°
Argon plasma (1 min)	58°
Oxygen plasma (1 min)	18°
CF_4 plasma (10 seconds)	88°
SPR350 + Acetone soak	87°

The results for the CF_4 plasma were unexpected as no increase in the contact angle was observed for a short exposure to the plasma. A similar process described in [5] reported an increase in the CA for Parylene to 123°. The experiment was repeated with increasing CF_4 exposure times and the results are presented in figure 3.

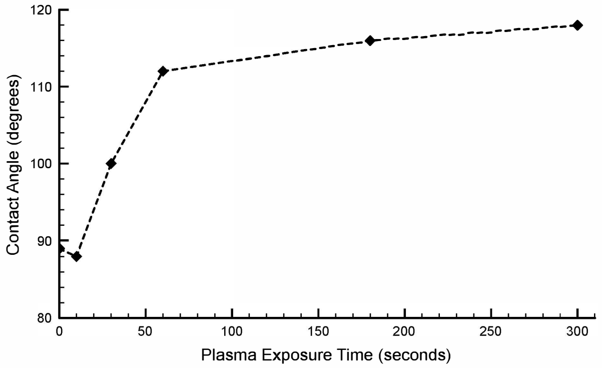


Fig. 3. Graph of the contact angle measured on Parylene C treated with different exposure times to a CF_4 plasma

The contact angle change after 5 minutes to ∼118° is sufficient to fabricate the required "hydrophobic step" on a superstrate with a patterned Parylene surface to demonstrate droplet guiding. Using this method, the concept has been successfully demonstrated with air actuation to guide a droplet of water along a boundary between untreated and treated Parylene when the actuation force is at an angle to the boundary (see figure 4).

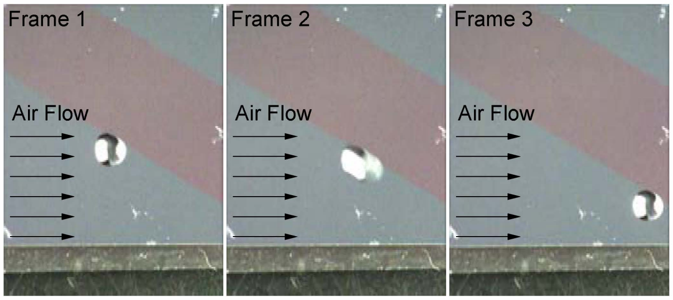


Fig. 4. Three frames of video showing a water droplet pushed by a jet of compressed air along a wettability boundary at an angle of 30 degrees

Figure 5 shows the contact angle difference across a boundary between CF_4 plasma treated Parylene and untreated Parylene. The contact angle value of the droplet sitting completely on the CF_4 treated area is around 115°. When the droplet is on the untreated side but touching the boundary, it shows a contact angle of 85° on the Parylene side and 95° where it intersects the boundary.

V. SAW DRIVEN MICROFLUIDIC TESTS

Following on from the airflow tests, SAW driven test devices have also been investigated. Figure 6 shows the definition of the "boundary angle", α, which is the angle between the line separating the treated and untreated Parylene regions, and the direction of the SAW propagation. This is also the angle of the directional change applied to a droplet, assuming it is guided by the boundary without crossing it. A larger angle indicates that a greater change in direction is required relative to the direction of the SAW propagation.

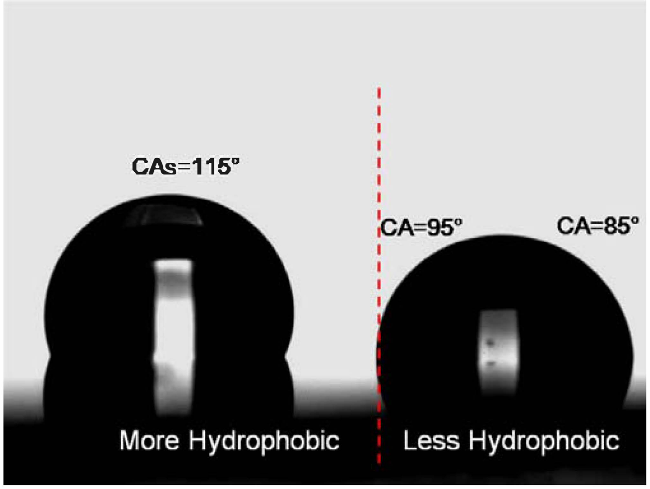


Fig. 5. Photograph showing the contact angles of two water droplets on untreated and CF_4 treated Parylene surfaces

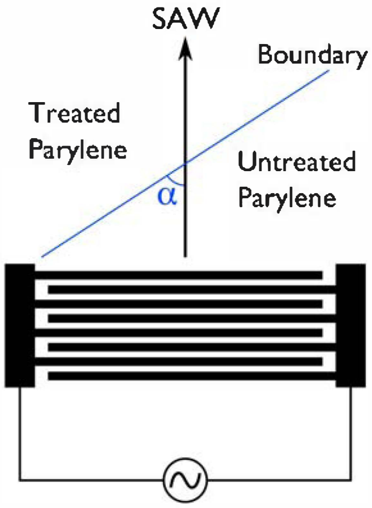
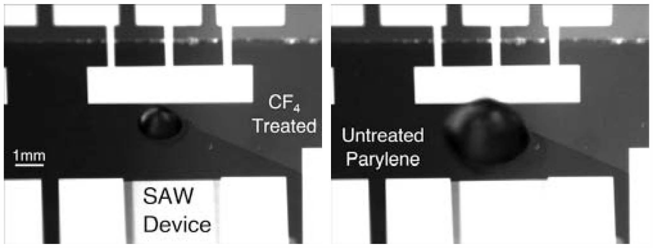


Fig. 6. Definition of the hydrophobic boundary angle α.

A. Hydrophobic boundaries patterned directly on SAW substrates

Initial tests of SAW driven droplet guiding have used hydrophobic boundary test structures fabricated directly onto piezoelectric substrates. Parylene was deposited on 128° Y-cut $LiNbO_3$ wafers with SAW IDTs (interdigitated transducers) and then patterned with CF_4 treated areas. The effects of droplet size, input RF power and boundary angle on the ability to guide droplets with a hydrophobic step have been characterised to determine the limitations of this approach.

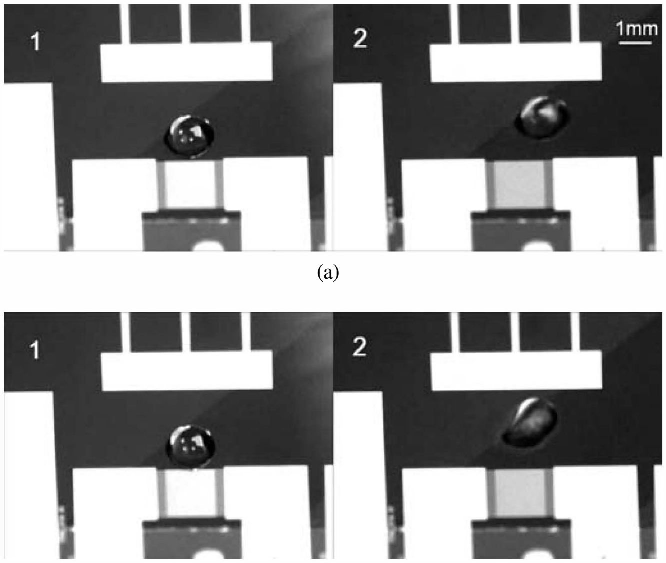
1) Effect of droplet size: Different droplet volumes were used to determine the effect on SAW driven movement and guiding. Figure 7 shows that both 1µl and 4µl droplets behave in a similar manner, following the 60° boundary without running over it. However, the aperture size of the SAW device, together with the boundary angle will decide the maximum drivable droplet size and how far the droplet can travel with certain sizes.



(a) (b)

Fig. 7. Photographs of (a) 1µl and (b) 4µl droplets being driven by SAW along a 60 degree boundary.

2) RF input power effect: Figure 8 shows that when the RF power is increased to 1W, a 1µl droplet will begin to cross a 45° boundary, rather than being guided. However, this value will also depend on the design of the SAW IDT electrodes and the use of different fabrication processes. Higher powers are also likely to be required when using a superstrate. A comparative study of these effects is planned in the future.



(a)

(b)

Fig. 8. Video frames in sequence showing SAW droplet driven using (a) 630mW input RF power and (b) 1W input RF power. The droplet size here is 1µl and boundary angle is 45°

3) Effects of boundary angle: SAW driven droplet guidance has been successfully demonstrated with boundary angles as large as 60°. However, a counterintuitive result was also obtained where a droplet crossed the boundary when the same power was used on a device with a boundary angle of 30°. The issue appears to be related to the alignment of the boundary, coupled with the narrow aperture of the SAW IDT, which affects the effective angle of the force applied to the droplet, pushing it against the boundary rather than along it. Further investigation with careful alignment of the hydrophobic step is required to confirm this.

B. Hydrophobic boundary patterns on superstrates

Following the characterisation work on the SAW devices themselves, hydrophobic boundary patterns have been fabricated on small superstrate chips. Initial tests using 380 µm thick silicon superstrates were unsuccessful. It appears that these are too thick to successfully propagate the acoustic wave from the piezoelectric substrate. Subsequently, 150 µm thick glass cover slips were coated with Parylene and patterned with CF$_4$ plasma to create a hydrophobic step. Figure 9 shows that droplet movement and guidance has been demonstrated using a glass superstrate with 45° boundary angle. Further work is now required to optimise the use of these superstrates and to repeat measurements using different boundary angles and SAW input power.

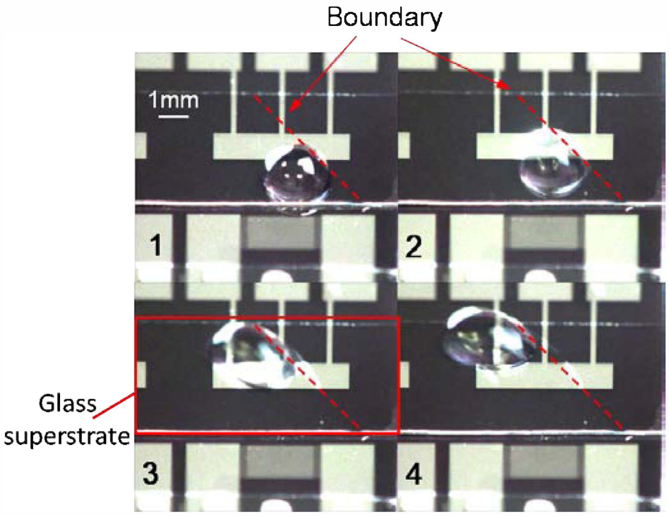


Fig. 9. Video frames showing droplet steering on a thin glass superstrate with a hydrophobic boundary

VI. CONCLUSIONS

Initial results have been presented from a study of materials and processes for the active manipulation of droplets through the engineering of material surfaces. By selectively changing the level of hydrophobicity of a surface it is possible to guide droplets of aqueous fluid, which prefer to stay on regions that are more hydrophilic. Processes for the treatment of Parylene to pattern it into areas of higher and lower wettability have been described along with the design and application of test structures to characterise the technology. Future work will use test structures and SAW to examine the relationship between applied power and angle of approach to a surface boundary and determine the extent to which droplets can be manipulated using material hydrophobicity modification.

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