Integrated Sample Preparation and Sensing: Microresonator Optical Sensors Embedded in Digital Electrowetting Microfluidics Systems

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Abstract— The intimate integration of digital electrowetting microfluidics and optical microcavity sensors results in an excellent platform for low power consumption, high sensitivity integrated sample preparation and testing, which is particularly useful for medical diagnostics and water quality testing. In this work, a microresonator structure with a buried channel waveguide (resulting in an external Q factor of 28,000 in silicone oil and 17,000 in water) was embedded in the top plate of a digital electrowetting system, enabling sensing with a single droplet of <100 nL volume. The integrated system was used to successfully demonstrate resonant optical wavelength shifts proportional to index of refraction changes of the liquid actuated to the sensor.

I. INTRODUCTION

The next generation of portable, low power medical diagnostic and water quality monitoring systems may have highly integrated sensing and sample preparation, including a high performance sensor array and low power sample preparation in a low cost package [1]. Planar optical microresonators meet the requirements for high performance, integratable sensors, and have the potential for low cost implementation by leveraging conventional planar fabrication techniques. Planar microresonators have been utilized for sensing of a variety of biologically relevant analytes, including DNA [2], streptavidin [2], glucose [3], and other biological targets [1]. Electrowetting-on-dielectric (EWD) microfluidic systems can perform the necessary fluidic operations for sample preparation and sample delivery to a sensor by moving discrete droplets, and have advantages that include small footprint (no external pumps), low power consumption, minimal use of reagents, and rapid fluid transport [4]. An electrowetting device with an integrated microresonator reported by Luan [5] demonstrated that glucose sensing could be performed in a silicone oil filled channel. However, that device structure utilized a diskshaped resonator, and a hole in the top plate of the

microfluidic system, which did not allow droplet motion off of the sensor. Although microdisk resonators tend to have higher quality (Q) factors due to improved confinement of the optical field, ring structures support fewer optical modes and, thus, have fewer overlapping resonant peaks or nulls in their spectra. Shifts of isolated peaks or nulls, which are used to track the shift in the micoresonator's resonant wavelengths due to analyte detection, are much easier to track than shifts of groups of overlapping peaks or nulls that exist in microdisk resonator spectra. This microdisk resonator sensor also needed a large droplet volume for the fluid to make contact with the resonator through the hole. In contrast, the integrated sample preparation and sensing system herein has a microring resonator sensor embedded in the top plate of the microfluidic system (with no hole), with a resonator O about 4 times greater (in water) than that of the microdisk structure reported by Luan et. al.. A single droplet was sufficient to make contact with the microresonator and the droplet could be actuated onto and off of the resonator in the device reported herein.

II. DESIGN AND FABRICATION

A. Digital Microfluidics

Electrowetting-on-dielectric (EWD) microfluidics, also known as digital microfluidics, produces fluid transport by moving droplets of liquid by applying an electric field to an electrode that is separated from the droplet by a dielectric. The applied electric field, when applied to a portion of the droplet, modulates the interfacial surface tension of the liquid droplet non-uniformly, forcing the droplet to move in the direction of higher electrical potential. A droplet can be transported along a series of closely spaced control electrodes by applying a voltage to the electrodes individually in sequence in the desired direction of droplet motion. These systems support a variety of basic fluidic operations including transport, splitting, merging, mixing, and dispensing from a

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Fig. 1. Diagram of the sensor embedded in the digital microfluidic system. (a) Top view of the embedded sensor. (b) Cross section view (not to scale) of the integrated system with embedded microresonator.

reservoir. These basic operations can be combined to perform higher level operations, such as sample preparation and sensing.

An EWD microfluidic system requires a dielectric material between the control electrodes and the droplet, a ground plane, and hydrophobic surfaces. Additionally, a channel medium, such as dodecane or silicone oil, is often used to reduce actuation voltage, prevent droplet evaporation, and prevent surface fouling [4]. EWD systems usually employ both a bottom plate and a top plate with the bottom plate having the control electrodes and the top plate providing a top cover to seal the system. If the ground plane is located on the top plate, a transparent conductor, typically a transparent conductore like indium-tin oxide (ITO), is utilized. A variety of materials have been utilized to for the dielectric film, including Parylene C, Teflon AF, and SiO₂ Increasing the dielectric constant and reducing the [4]. thickness of the dielectric produces lower droplet actuation voltage thresholds. Surfaces are typically rendered hydrophobic by coating the bottom plate and the top plate with a fluoropolymer, such as Teflon AF or Cytop [6].

Two main types of EWD system designs that differ mainly in the placement of the ground plane may be utilized [4]. In the parallel plate design, the ground plane is located on a top plate. In the co-planar design, the ground plane is located on the bottom plate along with the control electrodes. The co-planar system design has an advantage over the parallel plate design in that the ground plane need not be specially patterned to accommodate top plate integrated devices. However, the co-planar system requires a two-level metal structure on the bottom plate, which requires more sophisticated fabrication techniques to produce than does a single-level metal structure. The EWD design utilized in this work was a parallel plate design to simply the integration and fabrication. The bottom plate consists of the layers from Si up through the first Cytop layer and the top plate consists of the fused quartz layer down to the Cytop layer above the droplet, as shown in Fig. 1.

The bottom plate was fabricated on a 2 inch Si wafer. Two micron thick SiO_2 was deposited onto the Si by plasmaenhanced chemical vapor deposition (PECVD). Chrome control electrodes (100 nm thick) were fabricated in a lift-off process. A 60 μ m thick film of SU-8 3035 negative resist was spin-coated at 1000 rpm and, after performing standard baking and developing procedures, hard baked to produce the gasket. Parylene C (800 nm thick) was vapor deposited in a Specialty Coating Systems LABCOTER 2, conformally coating the SU-8 gasket. Finally, 5:1 diluted Cytop was spin-coated on the device and baked overnight at 60 °C to produce a roughly 50-70 nm fluoropolymer film coating to render the channel surfaces hydrophobic.

B. Microresonator Sensor

Planar microresonator sensors have been demonstrated for sensing a variety of biologically relevant targets. Because planar microresonator structures can be fabricated with standard CMOS processes, they are well-suited for low-cost, portable diagnostic applications. A microresonator requires bus waveguides to transmit light from the light source to the resonator and from the resonator to a photodetector. In a laterally-coupled microresonator design, the bus waveguides are coplanar with and adjacent to the microresontor. In a vertically-coupled design, the bus waveguides are located above or below the microresonator, separated by a transparent dielectric layer. In the lateral coupling configuration, the bus waveguides are exposed to solutions in contact with the sensor and to the gasket material used for microfluidic channels that might be combined with the microresonator device to control fluid transport to the sensor. The entire device can be covered with a coating material, such as Teflon AF [2], to mitigate interaction of light in the bus waveguides with the solutions and with gasket materal. However, the coating process can potentially be a problem for surface sensing protocols in which the microresonator is functionalized with DNA, antibodies, or some other specific capture molecule for biosensing, because interactions with other materials could significantly reduce the density of surface functional groups (i.e. epoxy groups on the SU-8 surface) used for bonding capture molecules to the sensor's surface. Moreover, a vertically-coupled structure in which the waveguides are located below the microresonator

inherently provides a buffer layer between the bus waveguides and other materials present on the device surface. For these reasons, a vertically-coupled microresonator structure was chosen for this work.

One of the most important parameters to consider for the vertically-coupled configuration is the size of the gap between the bus waveguide and the resonator. Varying the thickness of the interlayer dielectric between the microresonator and the bus waveguides simultaneously varies the external Q factor and coupling efficiency of the microresonator as well as the optical confinement of the bus As the interlayer dielectric thickness is waveguides. increased, the external Q factor increases, coupling efficiency (and, thus, transmitted power) decreases, and optical confinement in the bus waveguides increases. A high O factor is important because the limit of detection of the sensor is inversely proportional to the Q factor [7]. For the system herein, an interlayer dielectric thickness of $\sim 1.2 \ \mu m$ of SiO₂ balanced these trade-offs.

The embedded waveguides in the top plate of the microfluidics system were fabricated by first etching channels into a 2 μ m thick layer of PECVD deposited SiO₂ reactive ion etching (RIE) with O₂ and CHF₃ followed by a buffered oxide etch. SU-8 2002 was then spin-coated on top of waveguide channels, exposed, cured, hard-baked, and then etched back using RIE with O₂ and SF₆. PECVD SiO₂ was then used to deposit the interlayer dielectric. Indium tin oxide (ITO, 95% InO₂/5% SnO₂) was sputtered globally and then etched locally away from the microresonator sensing surface. Next, the input and drop waveguide facets were formed by dicing the substrate, and Cytop was activated with a light surface ashing in oxygen plasma, then SU-8 2002 was spin-coated and photopatterned to form the microresonator.

III. METHODS

The bottom plate was covered with 20 cSt silicone oil and 300 nL each of de-ionized (DI) H₂O and 2% D-glucose solution were loaded into the EWD system reservoirs prior to placement of the top plate. The top plate was held in alignment with the bottom plate using a V-mount clamp. After attaching the ground connection to the top plate and clamping a contact metal pin array to the control electrode contact pads (to electrically address the device), a single mode fiber was aligned to the input waveguide and a multimode fiber was aligned to the output waveguide of the microresonator sensor. The integrated system was then placed under a microscope with a computer interfaced Basler CCD video camera to observe the movement of the droplets. The electrodes were controlled using a relay with 32 independent channels interfaced to computer control software. An AC voltage of 40-80 V peak-to-peak at 1 kHz was supplied to the relay for droplet actuation. The optical input and output were an 81618A tunable laser module and a 81623A detector head, respectively, controlled by an HP8164A Lightwave Measurement System. The HP8164A was controlled through a computer interface with Labview.

A half-wave plate was used to select TE or TM resonant modes. Spectra were measured approximately every 12 seconds using Labview for instrument control and data acquisition.

IV. EXPERIMENT AND RESULTS

Prior to characterizing the embedded sensor, the electrowetting system was characterized. Electrowetting systems were tested both with air and with silicone oil in the channels. Additionally, electrowetting systems with both interdigitated and non-interdigitated electrodes were tested. Without the silicone oil medium, a DC voltage of 100-120 V was required to dispense DI H₂O out of a reservoir and splitting of a droplet from the reservoir could not be achieved. Moreover, the electrodes failed after several cycles under these high voltage conditions. Using silicone oil as a channel medium allowed droplets to be dispensed from the reservoir using an AC voltage of only 60 to 80 $V_{\text{p-p}}. \label{eq:Vp-p}$ For the system with interdigitated electrodes, the threshold voltage for movement of a droplet from one electrode to an adjacent electrode was about 40 $V_{p\mbox{-}p\mbox{-}}$. For the system with non-interdigitated electrodes, the threshold voltage was about 25 V_{p-p} . Although the non-interdigitated electrodes had a lower threshold voltage, the dispensing function was simpler with interdigitated electrodes, because the reservoir liquid readily wetted the first channel electrode adjacent to the reservoir electrode. In dispensing with the non-interdigitated electrodes, the reservoir fluid often had to be shuttled backand-forth several times by turning the reservoir electrode on and off before the reservoir fluid would wet the adjacent electrode.

Two experiments were performed to characterize the microresontor sensor embedded in the fluidic system top plate. The first experiment measured baseline stability by measuring the resonant wavelength shift due to the merging of DI H₂O droplets on the sensor. The second experiment measured the sensor response to $\sim 1\%$ D-glucose by merging a droplet of 2% D-glucose solution with a droplet of DI H₂O. For both experiments, the first droplet of DI H₂O was dispensed from the DI H₂O reservoir. Next, it was transported through the channel by turning off the electrode on which the droplet was sitting and turning on the next electrode in the path until the droplet was in contact with the sensor. Once the droplet was at its destination, the electrode on which it stopped was turned off. Measurement of spectra with Labview was started immediately after the droplet was put into contact with the sensor.

Sometimes the dispensed droplet would be twice the normal size sufficient to cover a single electrode. In that case, the 'double droplet' was split into two droplets by first applying a voltage to the two electrodes that it covered. Next, an electrode adjacent to the double droplet would be turned on. Once the droplet was wetted to the third electrode, the center electrode was turned off, forcing the droplet to split into two smaller droplets. One droplet could then be moved to the desired location and the other could be moved back into the reservoir.



Fig. 3. Image sequences illustrating the movement of the droplet onto the microresonator sensor and the subsequent merging of a glucose containing droplet. (a) Initial DI H₂O Droplet Transport. (b) 2% D-glucose droplet merging with the DI H₂O droplet.

After the first droplet was dispensed, the second droplet was dispensed from the appropriate reservoir. The second droplet was then transported to an electrode which was located two electrodes away from the first droplet. To merge the two droplets, a voltage very near the threshold voltage for droplet motion was applied to the electrode between the two droplets. This low voltage caused the second droplet to move toward the first droplet and to make contact and merge with the first droplet with minimal disturbance to the first droplet. A droplet merging operation is shown in Fig. 2.

For the baseline stability measurement, negligible shifts in the resonant wavelengths were observed. For the glucose measurement, a resonant wavelength shift of 17 pm was estimated from the difference in the maxima between the resonant peaks before and shortly after the merging of a glucose droplet with the droplet in contact with the sensor, as shown in Fig. 3. Using a reasonable assumption of equal volumes for both droplets, the measured glucose concentration changes, and the factor 1.4*10⁻³ RIU/% for glucose [8], a sensor sensitivity estimate of 12 nm/RIU was calculated. The microresonator Q factor in silicone oil was 28,000. The microresonator measured sensitivity is higher in air than in the silicone oil medium, due to the presence of a thin film of silicone oil on the microresonator surface. Thus, there is a trade-off between sensitivity and actuation voltage.

V. CONCLUSION

Microresonator sensors embedded into digital microfluidics systems are excellent platforms for low power consumption, high sensitivity integrated sample preparation and testing, which is useful for medical diagnostics and water quality testing. Addressing of the sensor with a single droplet and sensing refractive index changes in that droplet with an embedded sensor were demonstrated in the current system, which integrated a microresonator sensor and input/ouput waveguides into the top plate of an EWD microfluidics system. Dispensing of droplets, mixing, droplet separation, and presentation of droplets to the sensor were all



Fig. 2. Spectra measured before (blue) and after (red) the glucose droplet was merged. (Inset) Close-up of the resonant wavelength peak shift in picometers.

demonstrated in this system. The embedded microresonator exhibited a Q factor of 28,000 in silicone oil, which was not affected by integration with the electrowetting system. Thus, the integration and test of a microresonator sensor embedded in the top plate of a digital EWD microfluidics system has been successfully demonstrated.

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