Integrated Optical Sensor in a Digital Microfluidic Platform

Lin Luan, Randall D. Evans, Nan M. Jokerst, *Fellow, IEEE*, and Richard B. Fair, *Fellow, IEEE*

***Abstract*—The advent of digital microfluidic lab-on-a-chip (LoC) technology offers a platform for developing diagnostic applications with the advantages of portability, increased automation, low-power consumption, compatibility with mass manufacturing, and high throughput. However, most digital microfluidic platforms incorporate limited optical capabilities (e.g., optical transmission) for integrated sensing, because more complex optical functions are difficult to integrate into the digital microfluidic platform. This follows since the sensor must be compatible with the hydrophobic surfaces on which electrowetting liquid transport occurs. With the emergence of heterogeneous photonic component integration technologies such as those described herein, the opportunity for integrating advanced photonic components has expanded considerably. Many diagnostic applications could benefit from the integration of more advanced miniaturized optical sensing technologies, such as index of refraction sensors (surface plasmon resonance sensors, microresonator sensors, etc.). The advent of these heterogeneous integration technologies, that enable the integration of thin-film semiconductor devices onto arbitrary host substrates, enables more complex optical functions, and in particular, planar optical systems, to be integrated into microfluidic systems. This paper presents an integrated optical sensor based upon the heterogeneous integration of an InGaAs-based thin-film photodetector with a digital microfluidic system. This demonstration of the heterogeneous integration and operation of an active optical thin-film device with a digital microfluidic system is the first step toward the heterogeneous integration of entire planar optical sensing systems on this platform.**

***Index Terms*—Digital microfluidics, electrowetting, lab-on-a-chip (LoC), optical detection.**

I. INTRODUCTION

MUCH OF THE reported work on electrowetting-based lab-on-a-chip (LoC) microfluidic devices has focused on miniaturization of analytical methods and protocols for the purpose of improving performance and throughput. The benefits of miniaturization, such as smaller sample requirements, reduced reagent consumption, decreased analysis time, and higher levels of throughput and automation, have been demonstrated. However, to date little work has been reported on integrating the backend function of optical detection, in part because of the difficult requirement that detector integration must not interfere with electrowetting-based droplet transport, and in part because the heterogeneous integration of thin-film (microns thick) photonic components is an emerging technology that is just beginning to be applied to microfluidic systems.

Currently, almost all microfluidic devices are based on continuous fluid flow in permanent microchannels in glass, plastic, or other polymers. Numerous papers have been published that describe optical sensor integration with continuous flow devices. [1]–[21] One particularly interesting paper that is related to the research herein also underscores the importance of integrating photonic components with microfluidics. [21] This paper describes the heterogeneous integration of a thin-film surface normal LED with a microchannel fluidics system. However, continuous-flow-based microfluidic devices offer very little flexibility in terms of scalability and reconfigurability, and they are usually application specific.[22]–[25] This paper outlines the heterogeneous integration of a thin-film compound semiconductor photodetector with a digital microfluidics system. This integration technology has been used to demonstrate complex photonic systems, for example, that integrate lasers, optical waveguides and photodetectors [43], and thus, the heterogeneous integration of the planar optical component described herein presages the integration of complex photonic systems with digital microfluidics systems to achieve integrated systems with complex optical functions and digital fluidic control.

An alternative to continuous flow devices is to manipulate the liquid as unit-sized discrete microdroplets. Due to the architectural similarities with digital microelectronic systems, this approach is often referred to as "digital" microfluidics. Digital microfluidic systems have several advantages over continuous-flow systems, the most important being reconfigurability and scalability of architecture [26]. Electrowetting [26] and dielectrophoresis [27] are the two most commonly used techniques for microdroplet actuation, although other methods have been demonstrated, such as thermocapillary actuation [28] and surface acoustic wave actuation. [29] Electrowetting is primarily a contact line phenomenon, and refers to electric field-induced interfacial tension changes between a liquid and a solid conductor.

The use of electrowetting for dispensing, transporting, splitting, merging and mixing of aqueous and nonaqueous droplets has been demonstrated previously. [26], [30]–[37] Use of optical transmission in a droplet-based thermocapillary chip was described by Valentino, *et al.* [38] Planar thin-film waveguides were integrated into the droplet transport surface of a thermocapillary chip. The presence of a droplet in the optical beam path created a nonuniform substrate refractive index that caused detectable intensity modulation by reflective losses and attenuation.

The attachment of optical transmission detectors has been shown to be relatively easy on an electrowetting microfluidic

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The authors are with the Department of Electrical and Computer Engineering, Duke University, Durham, NC 27708 USA (e-mail: lin.luan@duke.edu; randall.evans@duke.edu; nan.jokerst@duke.edu; rfair@duke.edu).

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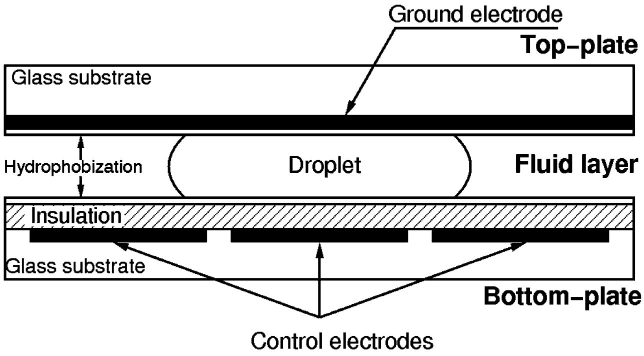

Fig. 1. Side-view of a digital microfluidic platform with a conductive glass top plate. By adding a conductive top plate and adding individually addressed buried electrodes in the bottom plate, the droplet can be actuated from one electrode position to the next by the application of voltages.

platform, since the platform can be made using plates and see-through indium-tin-oxide electrodes. [39] However, optical transmission detection scales poorly with miniaturization, since Beer's law incorporates path-length dependence. [40] For the detector described in [39], the optical path length typically was 100–300 μm, which is 30–100 times smaller than conventional systems (10 mm). This small path length poses serious sensitivity issues. Thus, alternative optical sensing and integration schemes are needed for electrowetting-based microfluidic systems.

The ultimate goal of the work presented in this paper is to integrate an entire optical sensing system that is compatible with an electrowetting-based microfluidic system to create a portable LoC system with optical sensing. The heterogeneous integration of active photonic devices (emitters, detectors) and the integration of sensors and optical interconnections (photodetectors, resonators, waveguides) into chip scale optical systems is currently under development. In this paper, these heterogeneous integration techniques are utilized to integrate a thin-film compound semiconductor optical photodetector sensor with a droplet-based electrowetting platform, without interfering with electrowetting transport. The photodetector is an InGaAs-based inverted metal-semiconductor-metal (I-MSM) device integrated into the eletrowetting microfluidics system, and optical sensing is demonstrated.

A. Electrowetting-Based Chip Technology

Electrowetting is the phenomenon whereby an electric field can modify the wetting behavior of a polarizable and/or conductive liquid droplet in contact with a hydrophobic, insulated electrode [41]. This effect is illustrated in Fig. 1. The application of a voltage to a series of adjacent electrodes that can be turned on or off creates an interfacial tension gradient that can be used to manipulate droplets. Droplets are usually sandwiched between two parallel plates—the bottom being the chip surface, which houses the addressable electrode array, and the top surface being either a continuous ground plate or a passive top plate (the nature of the top plate is determined by chip's characteristics). When an interfacial tension gradient is created by applying a voltage across a droplet partially overlapping an electrode, the droplet will move to the most stable position, which is generally centered on the activated electrode. When droplets are dispensed from reservoirs using electrodes adjacent to the reservoir, the droplets can be made to overlap multiple electrodes, and can be produced with a small variance in volume. Droplets dispensed in such a fashion have been demonstrated to move at speeds as high as 20 cm/s [52] by sequentially applying voltage to adjacent electrodes. This actuation can also be used to split large droplets, as well as merge and rapidly mix droplets. The low-power, reprogrammable, small reagent volume, and scalable technology free of moving parts, which is used in digital microfluidics, causes it to be a very promising avenue of exploration for LoC, with potentially greater versatility than continuous flow microfluidics and unique integration and design challenges.

Co-planar designs have also been developed where both the buried activation electrode and the exposed electrodes that ground the droplet are all located on the bottom surface. [39], [42], [52]. The top plate is not required for droplet actuation on coplanar devices, but its use is advised to contain the oil and the droplets. This architecture frees up the top plate for use by the optical detector, sensing surfaces, etc., with the only requirement being that the top surface that comes in contact with the droplet must be hydrophobic. Also, the top plate can be customized with specific chemistry or structures appropriate for each application.

The electrowetting chip surface is coated with an insulating layer of Paraylene C ($\sim$800 nm), and both the top and bottom surfaces are covered in a Teflon-AF thin-film ($\sim$50 nm) to ensure a continuous hydrophobic platform necessary for smooth droplet actuation. A spacer separates the top and bottom plates, resulting in a fixed gap height. The gap is usually flooded with silicone oil which acts as a filler fluid, thus preventing droplet evaporation and reducing surface contamination. [41] Details of fabrication processes are reviewed in [52].

B. Optical Sensor Fabrication and Integration

The optical components integrated with the electrowetting microfluidics system in this paper were fabricated using heterogeneous integration. Heterogeneous integration is a technology through which thin-film semiconductor devices [e.g., lasers, light emitting diodes, photodetectors, high electron mobility transistos (HEMTs), heterojunction bipolar transistors (HBTs), resosant tunneling diodes (RTDs)] can be integrated onto arbitrary host substrates including Si [43], Si CMOS [44], and printed circuit boards [45]. This process selectively removes the growth substrate from the active layers of the device, and the resulting thin-film devices are then bonded to host substrates. Since the semiconductor devices are so thin (typically, 1–5 microns thick), the topography of the host substrate is minimally changed. Thus, the integration of photonics with microfluidics (or Si CMOS integrated circuits, or printed wiring boards) is further enabled because the thick (350–500-micron-thick) substrates on the photonics devices do not need to be accommodated.

Heterogeneous integration also enables the integration of multiple independently optimized semiconductor devices with cost-effective sparse integration of the semiconductor devices onto microfluidics platforms. Since many high-efficiency photonics devices are composed of compound semiconductors, such as GaAs- or InP-based materials, the sparse distribution of these relatively expensive devices onto sites only where they are needed is critical to system cost. A single 4 inch diameter laser growth substrate, for example, can be fabricated into tens of thousands of lasers that can each be individually integrated into a different microfluidics system.

Heterogeneous device integration onto Si and Si CMOS using sparse integration thin-film device transfer diaphragm techniques [44] has been demonstrated for a variety of devices and substrates, including thin-film devices composed of Si [43], GaAs [46], InP [47], and GaN [48] for thin-film transistors, photodetectors [49], and lasers [43], and many of these devices have been integrated onto Si CMOS [47]. The thin-film heterogeneous integration process begins with the device of interest grown on a lattice-matched growth substrate (e.g., GaAs, InP) that has been engineered for selectively etched substrate removal using an etch stop layer. The devices are fabricated on this substrate, and mesa etched down to the etch stop layer. The components are then coated with a handling layer, and the growth substrate is removed through selective etching. The resulting array of devices are then contact bonded to a transparent transfer diaphragm, the handling layer removed, and the thin-film devices are transferred and bonded to the host substrate (in this paper, the photodetector is integrated onto a glass plate that is bonded into the microfluidics system) either selectively from the array for sparse integration, or as an entire array bonded simultaneously for dense integration. The capability to sparsely integrate the thin-film devices enables a "mix and match" of multiple different devices into the same microfluidic system, and is the most cost-effective method of distributing the photonic compound semiconductor components only where necessary.

This paper describes the heterogeneous integration of a photodetector with a digital microfluidics system. The key issue is making sure that the detector is compatible with the hydrophobic surfaces required for electrowetting transport, which is the first step toward integrating more complex photonic sensing systems with the platform. Research into planar lightwave integrated circuits (PLICs), which are planar implementations of optical systems, are modeled on the fabrication and integration technologies used for integrated circuits. Edge emitting laser sources, waveguides, resonant sensors, filters, and photodetectors are also being investigated for the integration of an entire optical system with microfluidics at the chip scale. The thin-film devices are so thin that they can be embedded in waveguides. [43] thin-film devices embedded in polymer waveguides on silicon [50], ceramic [51], and FR-4 printed circuit board [45] substrates have been demonstrated, proving that optics can be integrated onto thermally sensitive, rough (surface texture) FR-4 and Si CMOS ICs using post-processing of the boards or ICs, thus eliminating interference with the board or chip foundry. This is also applicable to microfluidic systems—the photonics can be integrated after the microfluidics systems have been fabricated, or can be inserted into the microfluidic system fabrication process.

II. EXPERIMENTAL TECHNIQUE

To integrate a compound semiconductor photodetector with an electrowetting microfluidics system, a coplanar digital microfluidic chip fabricated by Advanced Liquid Logic in printed circuit board (PCB) technology was used. The gap between the top plate and the surface of the chip was approximately 90 μm (gasket height—see Figs. 1 and 2). The chip was attached to the controller, and electrodes were switched through a computer GUI connected to the controller. Silicone oil ($\sim$2 cSt) was dispensed onto the area of the chip used for this experiment. Electrode voltages of 220 V were applied to the chip.

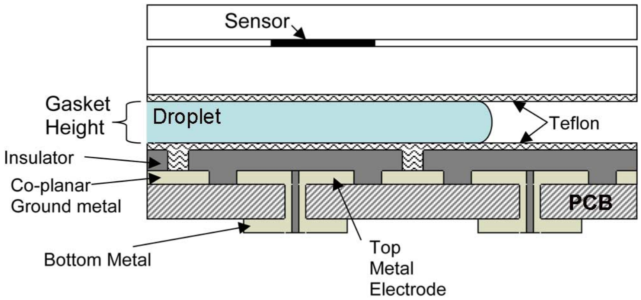

Fig. 2. Side view of a co-planar electrowetting chip made on a printed circuit board. The top plate can be customized to accommodate the optical sensor and its sensing surface.

The InGaAs-based photodetectors were grown, fabricated, and each heterogeneously integrated onto a 35 mm × 50 mm glass substrate by metal/metal bonding, and then attached to the electrowetting system. Conventional vertically illustrated photodetectors are divided into two broad categories: p-i-n/avalanche photodetectors and metal-semiconductor-metal (MSM) photodetectors. MSM photodetectors, with interdigitated Schottky finger contacts, are attractive because they have a lower capacitance per unit area than p-i-n detectors. Thus, MSMs are larger than p-i-ns operating at the same speed, reducing the cost associated with alignment and packaging. Inverted MSMs (I-MSM) are thin-film MSMs with fingers on the bottom of the device, thus eliminating the finger shadowing that causes conventional MSMs to have low responsivity. I-MSMs have been demonstrated that have responsivities that are comparable to p-i-n devices for the same operational speeds [49].

The bonding of the thin-film photodetectors to the microfluidics systems in this paper was a vertical metal-to-metal bond, and was a mechanically strong, as well as electrically and thermally conductive bond. A variety of metal bonding formulations have been used, and a Ti/Pt/Au Schottky barrier on the InGaAs-based photodetector bonded to a Au pad on glass has been experimentally shown to produce a strong bond. The bonding is essentially like bump bonding, but the bonded devices/circuits are thin (about 1 micron for the photodetectors in this paper), and the metal bumps are thin (on the order of 2500 Å). The devices are inverted in this process, so the photodetector top contacts and fingers face the host substrate, and, after bonding a stable metal/metal bond is formed with the metal pad deposited onto the glass. Since the thin-film devices are on the order of microns thick, the topography of the glass is not substantially changed by the heterogeneous integration, and, for devices that need further processing (the photodetectors for this paper did not), standard dielectric and metal deposition and photolithography can be used to complete the fabricated system. Critical to the integration of the photonic devices with the microfluidics system demonstrated is that the photodetector could not be integrated as demonstrated if the photodetector growth substrate remained on the device.

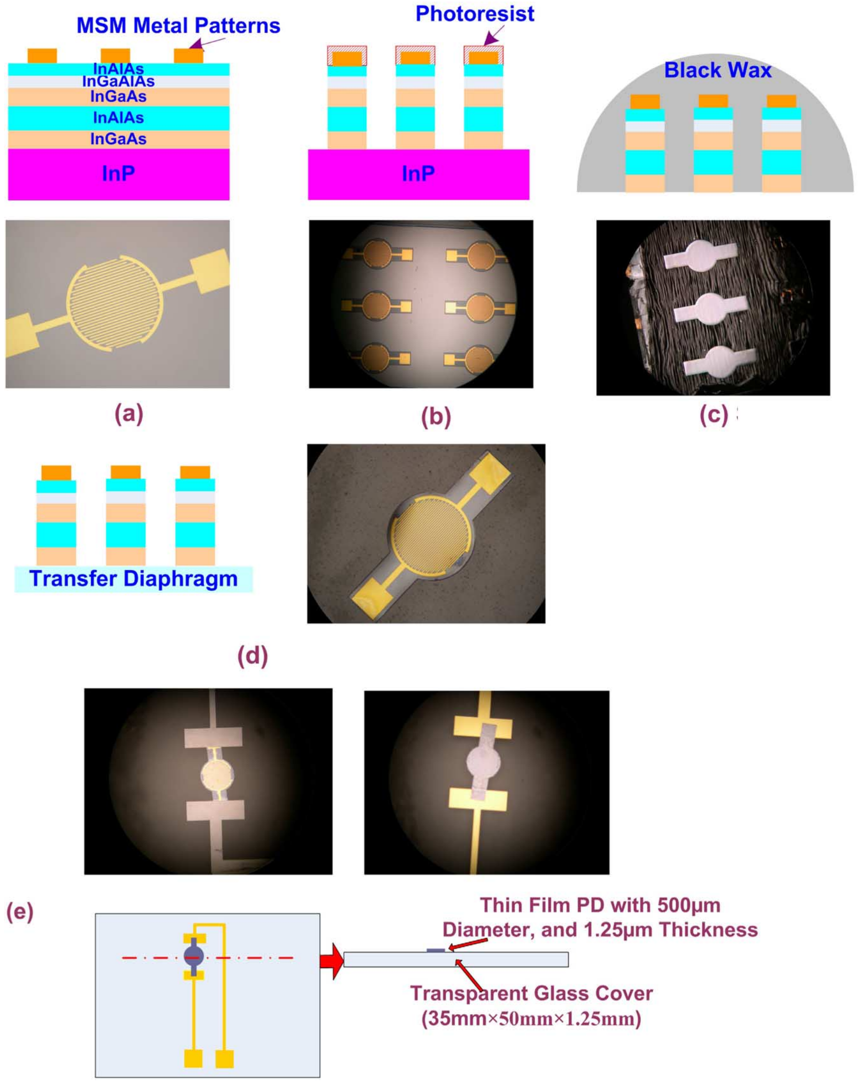

Fig. 3. (a) Top view of MSM photodetector metal fingers on substrate. (b) Top view of MSM photodetector metal fingers after mesas etching. (c) MSM thin-film devices after substrate removal (from the backside): these thin-film devices are 1 micron thick, and the metallized finger/pad surface shown in (a) is on the other side of the devices. (d) Thin-film MSM photodetector on a transfer diaphragm. (e) The top views of the I-MSM photodetector after bonding to metal pads on the glass substrate, and the top view and cross section of the photodetector integrated on the glass substrate.

The material used to fabricate the I-MSM photodetectors used in this work was grown by molecular beam epitaxy on an InP substrate. The grown layers consisted of 40 nm InAlAs (cap layer), 20 nm InGaAlAs (superlattice graded layer), 800 nm InGaAs (absorbing layer), 200 nm InAlAs (cladding window layer), 200 nm InGaAs (selective etch stop layer), with all of the layers nominally undoped. Metal fingers (Ti/30 nm-Pt/40 nm-Au/200 nm) were defined after standard photolithography and metal evaporation, as shown in the photomicrograph in Fig. 3(a), followed by a H_2O_2/citric acid mesa etch. The MSM mesas were then coated with Apiezon W and immersed in HCl, which selectively removed the InP substrate [Fig. 3(b)]. Next, the I-MSMs were bonded to a transparent transfer diaphragm, and the Apiezon W was slowly removed with TCE [shown in Fig. 3(c)]. The devices were then transferred (and, in that transfer, inverted) to the host substrate metal pads (Ti/Au, which were deposited onto the transparent glass) substrate and metal/metal bonded, as shown in Fig. 3(d) and (e).

The photodetector was mounted on one side of a glass substrate. The microfluidic chip must present hydrophobic surfaces to the droplets contained therein, also as shown in Fig. 2. So, a second 1-mm-thick acrylic plate coated with Teflon AF was placed between the photodetector and the droplets, as shown in Fig. 2. The photodetector (and the glass plate it was integrated onto) were mounted on the upper surface of the top plate, with the photodetector facing downward, and it was aligned with the linear array of electrodes on the lower part of the chip. All biases and data from the photodetector were applied and measured, respectively, using a Keithley source measurement unit (SMU), which was connected to the photodetector via two probes connected to the contact pads that protruded from the microfluidic device. The responsivity of the I-MSM was measured using a calibrated pigtailed laser diode at 660 nm, and an optical power meter. The measured responsivity for surface normal illumination of the I-MSM (on the side without fingers) was 0.39 A/W.

In order to test the integrated photodetector, a signal must be generated. Chemiluminescence by mixing two droplets was chosen as the method of optical signal generation. In order to generate a substantial signal in an aqueous medium, the oxi-

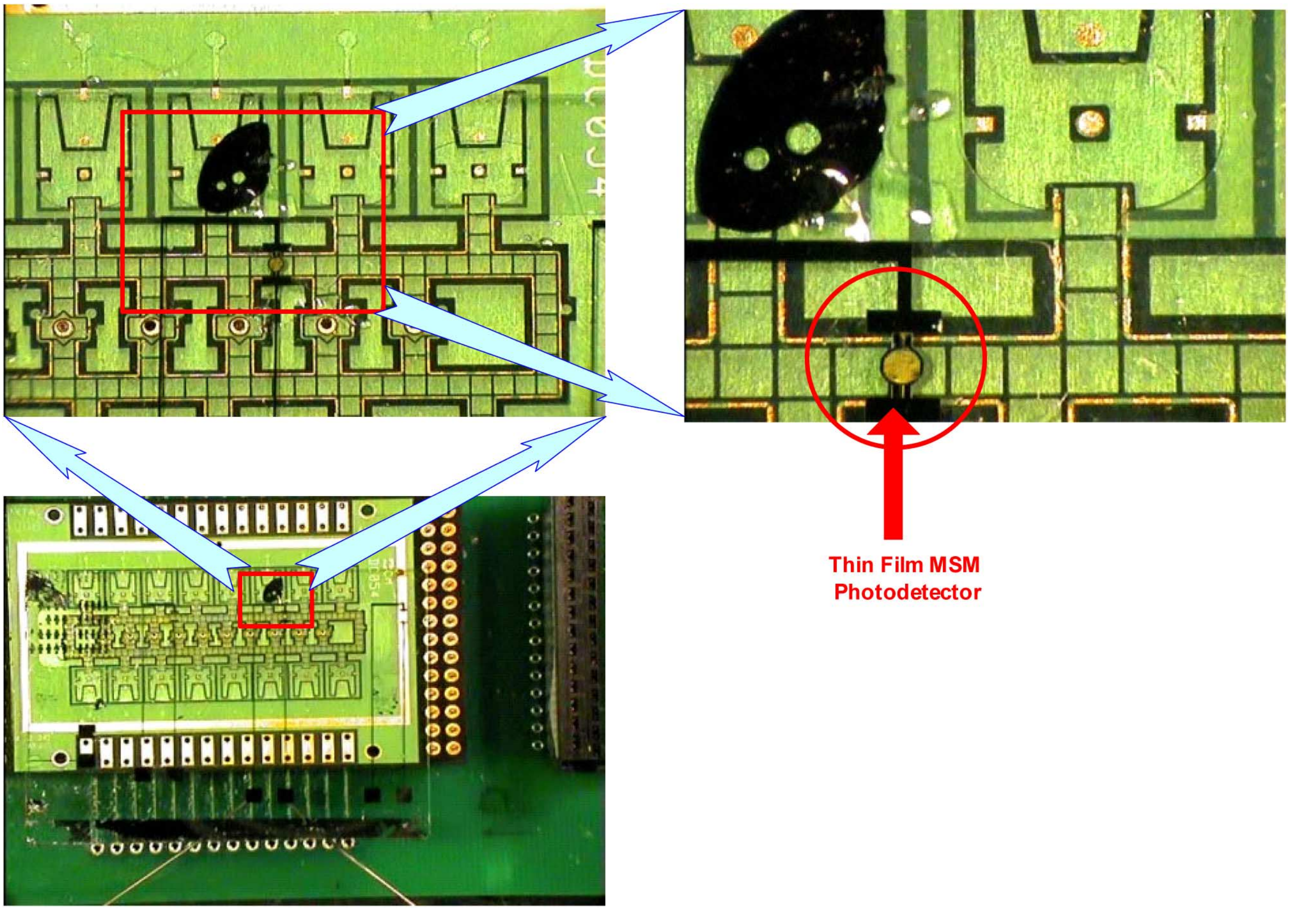

Fig. 4. Top views of the electrowetting chip with a thin-film MSM photodetector showing filled reservoirs and linear arrays of buried electrodes. The photodetector was aligned over the array near the 30% H_2O_2 reservoir.

dation of pyrogallol (1, 2, 3-trihydroxibenzene) in an alkaline solution was used (the Trautz-Schorigin reaction). First, equal parts of 0.8 M pyrogallol and 9 M NaOH were mixed, and then another equal part of 38% formaldehyde was added and mixed. When two parts of the previous solution are mixed with one part of 30% H_2O_2, it generates a short-lived, bright orange light if the solution is fresh, or a longer lasting, lower intensity light if the solution is not fresh (has been given time to cool). Both solutions are immiscible in silicone oil. The pyrogallol solution has a contact angle of 100° with Teflon AF and the 30% H_2O_2 has a contact angle of 115° at room temperature, which demonstrates the hydrophobicity that is necessary for electrowetting actuation. Chemicals used in the experiments were dispensed from on-chip reservoirs, as shown in Fig. 4, and the dispensed droplets were actuated together underneath the sensor. When the droplets mixed underneath the sensor, the chemiluminescent reaction began to generate light.

III. RESULTS

Two measurements were performed to demonstrate the detection of an optical signal from the mixing of two fluids that produce chemiluminescence. First, two droplets were mixed on a Teflon AF-coated glass cover slip, 0.13–0.17 mm thick (not the acrylic substrate onto which the photodetector was integrated), that was placed over the photodetector. The chemiluminescent optical signal was measured by the integrated thin-film I-MSM photodetector, by the side that had no metal fingers (which is the side that faced the chemiluminescent droplet in the electrowetting system in the next experiment). The mixture on the coverslip was 2.5 μl of H_2O_2 mixed into 5 μl of pyrogallol solution, and data was taken for about 210 s. An additional measurement was performed by using the integrated photodetector and microfluidic system. A droplet of the H_2O_2 was actuated into a larger droplet of the pyrogallol solution, which was previously actuated to a location underneath the photodetector. The resulting photocurrents from the thin-film I-MSM with 1 V MSM bias voltage were measured with a Keithley 236 SMU, and are displayed in Fig. 5.

The next measurement used the integrated electrowetting/photodetector system shown in Figs. 2 and 4. Two droplets were mixed to generate a chemiluminescent signal, the resultant mixed droplet was then moved relative to the photodetector, and the photodetector output current sensed the changing optical signal with the changing droplet position. The integrated thin-film photodetector was placed facing downward with the fingerless photodetector face toward the microfluidic channel. A 1.2-mm-thick Teflon AF-coated acrylic top plate was placed between the photodetector and the droplets. The H_2O_2 and pyrogallol solutions were dispensed from their reservoirs separately, and were actuated to mix together. The droplet was

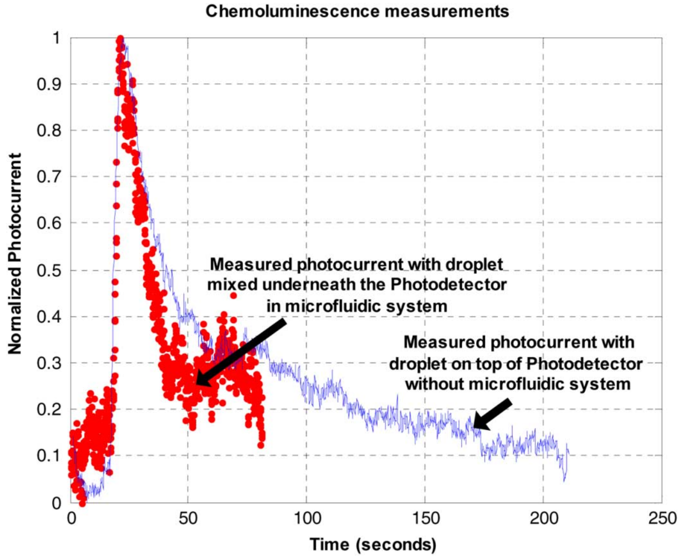

Fig. 5. Comparisons of measured photocurrent by photodetector when droplet mixed with/without microfluidic system.

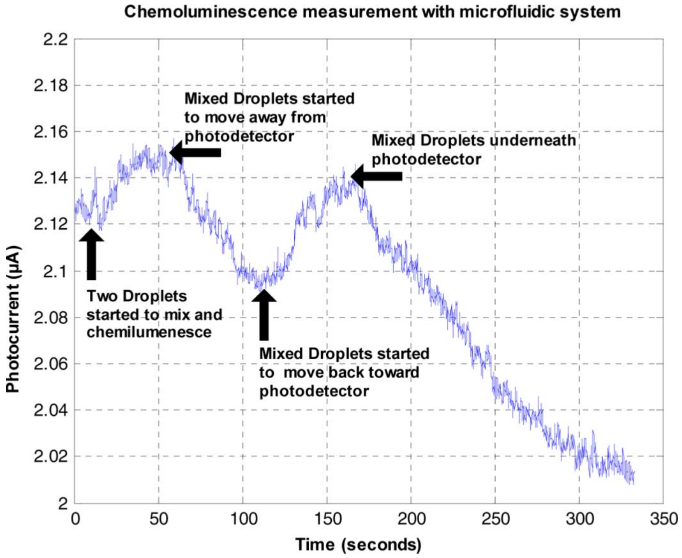

Fig. 6. Chemiluminescent optical signal sensed by the photodetector integrated with the electrowetting system. Two droplets were first dispensed from their reservoirs, mixed together, then moved under the photodetector. The chemiluminescent droplet was next moved away from the photodetector, moved back under the photodetector again, and then, finally, moved away from the photodetector. The photodetector output current reflects this optically chemiluminescent droplet movement.

mixed near the photodetector, then moved under the photodetector, then out from under the photodetector, then moved under the photodetector and away from the photodetector again, using actuation voltages. Fig. 6 shows the measured current from the photodetector for this optically emitting droplet actuation. After being moved under the photodetector twice, the droplet was then moved far away from the photodetector, and this is reflected in the response of the photodetector, as shown in Fig. 6. These measurements were extremely difficult primarily due to the temperature dependence of the pyrogallol solution (both intensity and surface tension) and the microfluidic system's uncharacterized heat transfer properties.

IV. Discussion

The photodetector sensed the chemiluminescent signal from the mixed droplets, as shown in the data from Figs. 5 and 6. As expected, in both cases, when mixing pyrogallol solution with H_2O_2, the initial optical output intensity is large, and then decays as a function of time. The photocurrent from the mixed droplets in the electrowetting system, shown in Fig. 6, was slightly lower in magnitude and produced an optical signal over a longer time period than the photocurrent from the droplets mixed above the photodetector, as shown in Fig. 6. The movement velocity of the mixed droplet was slow due to the high viscosity of the droplet, and due to the large droplet size. The optical photocurrent decreases as a function of time (after approximately 175 s) due to both the decrease in optical output intensity of the chemiluminescence and because the droplet was being transported away from the photodetector.

V. Conclusion

This paper reports on the demonstration of an integrated optical sensor integrated with an electrowetting-based digital microfluidics system. The InGaAs-based thin-film photodetector was heterogeneously integrated onto a glass substrate, which was then integrated with the digital microfluidic system. The integrated thin-film I-MSM photodetector detected the chemiluminescent optical output signal generated by a pyrogallol solution mixed with H_2O_2, both external to, and internal to the electrowetting system. This demonstration of the heterogeneous integration of an active thin-film compound semiconductor optical device with a digital microfluidics platform is the first step toward a fully integrated chip scale optical sensing system integrated with digital microfluidics. Applications for these systems include biological and chemical sensing for medical and environmental sensing and monitoring.

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Lin Luan received the B.S. degree in electrical engineering from Shandong University, Shandong, China, in 2001, and the M.E. degree in communication and information system from Peking University, Peking, China, in 2004. She is currently working towards the Ph.D. degree in electrical and computer engineering at Duke University, Durham, NC.

Her research interest includes photonic biochemical microresonator sensors integrated with digital microfluidic systems.

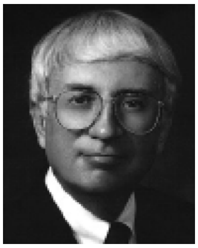
Randall D. Evans received the B.S. degree in electrical and computer engineering from the University of California at Riverside, Riverside. He is currently working towards the Ph.D. degree at Duke University, Durham, NC, where he works as a Research Assistant in the Digital Microfluidics Laboratory.

His research interests include the high-resolution printing of hydrogel scaffolds for engineered tissues, and the integration of capillary electrophoresis with an electrowetting microfluidic chip.

Nan M. Jokerst (F'03) received the Ph.D. degree from the University of Southern California, Los Angeles, in 1989.

She is the J. A. Jones Professor of Electrical and Computer Engineering at Duke University, Durham, NC. She has published more than 250 papers in refereed journals and conference proceedings, three book chapters, and has four patents awarded in the areas of chip-scale integrated systems, sensors, integrated photonics, optical interconnections, and metamaterials.

Dr. Jokerst received the IEEE Education Society/Hewlett Packard Harriet B. Rigas Medal (2002) and the IEEE Millennium Medal (2000). She is a Fellow of the Optical Society of America (2000). She is a DuPont Young Faculty Awardee and a National Science Foundation Presidential Young Investigator, and has won a Newport Research Award, three teaching awards, and was a Hewlett Packard Fellow. She has organized and served on numerous conference committees, and served on the Board of Directors of the Optical Society of America as the Chair of the Engineering Council, also on the IEEE Lasers and Electro-Optic Society (LEOS) Board of Governors as an elected member, and as the Vice President of Conferences and as the Vice President of Technical Affairs. She has also served as the elected Chair, Vice Chair, Secretary, and Treasurer of the Atlanta IEEE Section.



Richard B. Fair (S'63–M'75–SM'75–F'90) received the Ph.D. degree from Duke University, Durham, NC, in 1969.

He spent 12 years at Bell Laboratory working on semiconductor devices and integrated-circuit technology. He returned to North Carolina in 1981 and spent 13 years as a Vice President of Microelectronics Center of North Carolina (MCNC), having responsibilities in chip design, computer-aided design, packaging, process technology, and microelectromechanical system (MEMS). He is currently a Professor of Electrical and Computer Engineering at Duke University. He has published over 150 papers in refereed journals and conference proceedings, written ten book chapters, edited nine books or conference proceedings, and given over 120 invited talks, mostly in the area of semiconductor devices or the fabrication thereof. His current research interests include bio-MEMS and digital microfluidic chips. His research group first demonstrated microdroplet transport over electrode arrays based on electrowetting actuation.

Dr. Fair is a Fellow of the Electrochemical Society, past Editor-in-Chief of the PROCEEDINGS OF THE IEEE, and he has served as Associate Editor of the IEEE TRANSACTIONS ON ELECTRON DEVICES. He is a recipient of the IEEE Third Millennium Medal (2000) and the 2003 Solid State Science and Technology Prize and Medal from the Electrochemical Society, which was presented in Paris, France.