

Optical Detection Heterogeneously Integrated With a Coplanar Digital Microfluidic Lab-on-a-Chip Platform

Randall Evans, Lin Luan, Nan Marie Jokerst, Richard B. Fair
Department of Electrical and Computer Engineering
Duke University
Durham, NC
randall.evans@duke.edu

The intimate integration of optical components with microfluidics technology will enable next generation portable LoC systems that may completely contain optical generation, processing, and detection. Toward the chip scale integration of digital microfluidics with active compound semiconductor devices, heterogeneous integration technology is used in this paper to integrate thin film (approx 1 micron thick) compound semiconductor photodetectors with digital microfluidic systems. Each thin film InGaAs photodetector is bonded onto a glass platform, coated with Teflon AF, and integrated into the digital microfluidics system. The detection function is tested using the mixing and digital droplet movement of chemiluminescent compounds, which clearly indicate the functionality of the microfluidics system and the integrated thin film InGaAs photodetector.

I. INTRODUCTION

Lab-on-a-Chip research is focused on automating and miniaturizing analytical procedures, primarily investigating device and procedure miniaturization. However, limited research has been performed on integrating optical detectors with digital microfluidic systems, a necessary step for the development of miniaturized and portable LoC devices. One example would be the development of a malaria detection device for use in third-world countries. This paper discusses the integration of a thin film InGaAs photodetector with a coplanar digital microfluidic chip.

A. Digital Microfluidic Lab-on-a-Chip

Digital microfluidic systems transport fluids by utilizing the principle of electrowetting on a dielectric (EWoD), where a liquid's wetting behavior is modified by an electric field under certain circumstances. A droplet of conductive or polar liquid on a dielectric containing a buried electrode may change its contact angle with the surface when a voltage is applied between the droplet and the buried electrode. The surface interfacial tension between the liquid and the surface is lowered by the electric field, according to the Lippman-Young equation [1]. This When the dielectric is hydrophobic

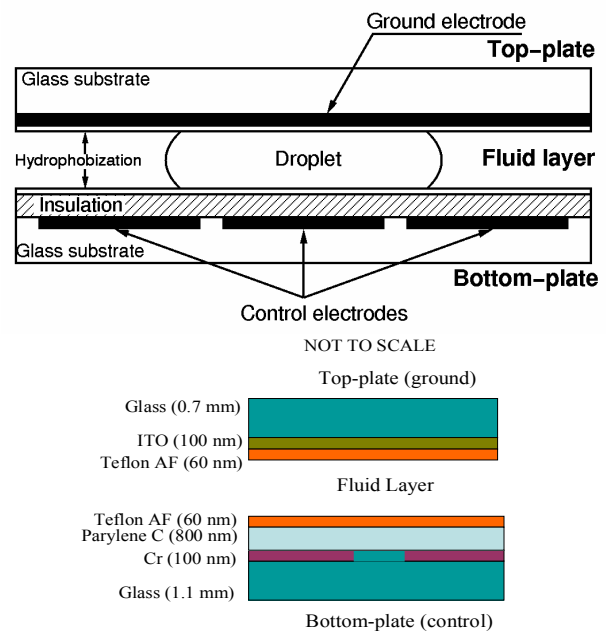


Figure 1. Side-view of digital microfluidic platform with a conductive glass top (top). Materials and construction of the actuator (bottom). By adding a conductive top plate and adding individually addressed buried electrodes in the bottom plate, the droplet can be actuated from one electrode position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

and a sufficient voltage (the actuation voltage) is applied, the voltage causes the droplet to wet the surface. A series of electrodes that may have voltages turned on and off can utilize the EWoD phenomena to manipulate droplets [2]. Standard digital microfluidic devices use a hydrophobic, conductive, and transparent top plate to apply voltage to the droplets, silicone oil to reduce the actuation voltage, eliminate evaporation, and reduce surface contamination [2], a gasket to contain the silicone oil and maintain a specific gap height between the dielectric and the top plate, a hydrophobic dielectric burying the electrodes, and an array of buried, addressable electrodes to manipulate droplets. Fig. 1 demonstrates this setup. Careful design of these devices

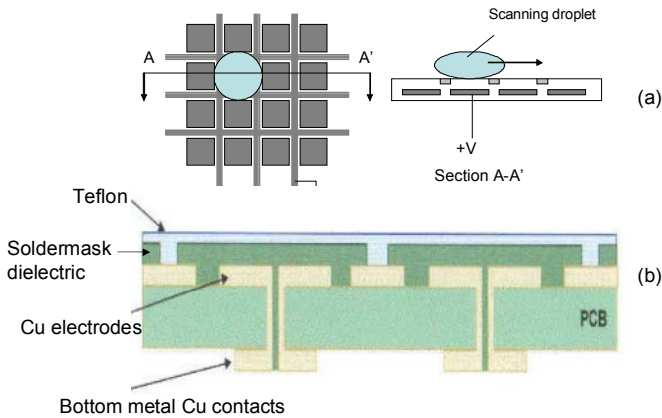


Figure 2. Coplanar actuation array for droplet scanning where electrical contact to the droplet is provided by surface electrodes, obviating the need for a top contact plate. (b) Co-planar construction in printed-circuit board technology [9].

enables the actuation of droplets as small as ~ 3 nL [3], droplet actuation speeds as fast as 25 cm/s [4], the accurate dispensing of droplets of a specific volume from a larger reservoir, and rapid droplet mixing and splitting. Digital microfluidics enables rapid liquid processes, and reduced reagent volumes via low power manipulation of droplets.

A modification of a standard digital microfluidic system resulted in the coplanar digital microfluidic platform [5-8]. This design utilizes a conductor that is coplanar to the dielectric and contacts the droplets, enabling the voltage to be applied to the droplet without the use of a conductive top plate. In fact, this system does not require a top plate at all (Fig. 2), but one is often used to contain the droplets and the silicone oil. Since the top plate need not be conductive, this enables significant modification of the top plate, such as the addition of a photodetector and the corresponding leads and contact pads. Fig. 3 shows a top-down view of a coplanar

digital microfluidic chip manufactured with printed circuit board (PCB) technology by Advanced Liquid Logic (ALL). The square metal pads that form the droplet path are buried electrodes, and the light colored lines surrounding this path are the coplanar gold wires which supply voltage to the droplets. Threshold voltages for these devices are typically larger than 130V, as the soldermask dielectric is thick [9].

B. Thin Film InGaAs Photodetector

Photodetectors are used to sense photons, and, in this work, a compound semiconductor thin film metal-semiconductor-metal photodetector (MSM photodetector) was integrated into an electrowetting microfluidics system so that an optical detection function could be integrated with a fluidic sample processing system. Two common types of optical detection devices are p-i-n photodetectors and MSM photodetectors, of which the highest performance devices are made of compound semiconductor materials. MSM photodetectors have interdigitated gold Schottky contacts on a semiconductor structure that contains a photodetecting absorption layer. MSM photodetectors are attractive photodetectors because they have lower capacitance per unit area than p-i-n detectors, and thus, can be larger, and more alignment tolerant than p-i-n detectors. However, the fingers that form the Schottky contacts on conventional MSMs are of the surface of the detector, and thus shadow the incident optical beams, resulting in low responsivity in comparison to p-i-n photodetectors. Inverted MSMs (I-MSMs) are thin film MSMs with fingers on the bottom of the devices, thus eliminating the finger shadowing effect, which increases the responsivity in contrast to conventional MSM photodetectors, resulting in a comparable responsivity to p-i-n devices, while still retaining the attractive low capacitance per unit area feature of the MSMs [10]. The InGaAs-based MSMs used for this research consist of five epitaxial layers, which were grown by molecular beam epitaxy (MBE) lattice

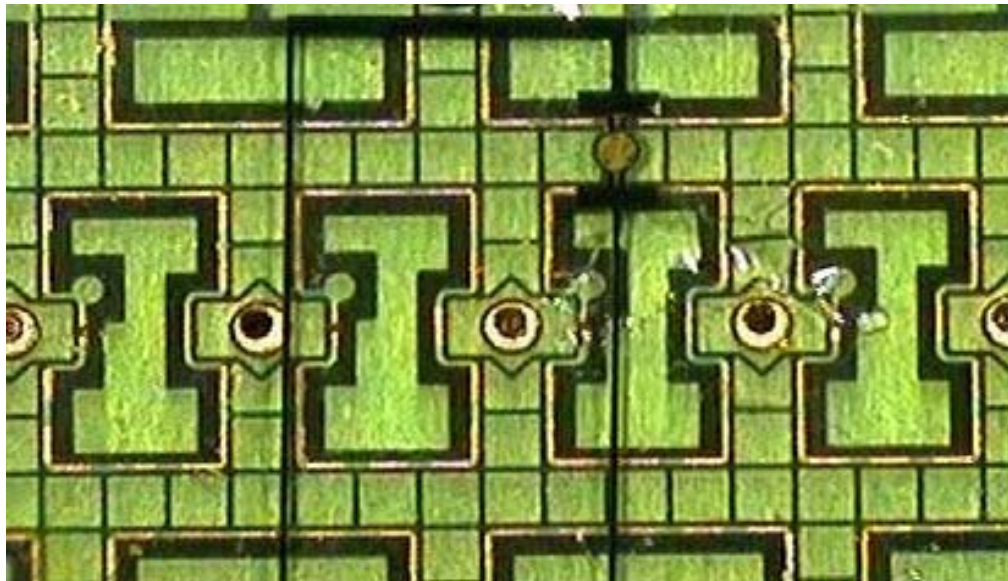


Figure 3. Coplanar Digital Microfluidic chip with integrated photodetector above a square electrode path.

matched to an InP substrate. The five layers, as grown, from top to bottom were 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). After metal fingers (Pt-Ti-Au) were deposited and patterned on top of the wafer to form the Schottky contact, a mesa etching process was used to separate individual MSM devices from each other, and then, the sample was coated with Apiezon W (black wax) and attached to a carrier. The InP substrate was then removed by selective wet etching of the InP substrate. The substrate removal etch stopped on the InGaAs layer, which was then removed with a second selective etch. Next, the thin film MSM photodetectors (total thickness of 1.24 μ m), were bonded to a transparent Mylar transfer diaphragm that was supported by a Si ring. The carrier and Apiezon W were then removed from the thin film devices, which remained on the mylar transfer diaphragm. The transfer diaphragm was then inverted and aligned with respect to metallized pads on the glass host integration substrate. The thin film photodetectors were then heterogeneously integrated onto the glass substrate by transferring the devices from the diaphragm to the metal pad on the glass substrate. This metal/metal bonding thus integrated the photodetector onto the top plate of the microfluidics system (a 35 mm \times 50 mm glass substrate).

To complete the integration of the inverted-MSM PD into the microfluidics system, an approximately 1 μ m thick Teflon AF layer was spin coated on the surface of the photodetector to create a hydrophobic layer for contact with the droplet. The glass slide with the inverted-MSM PD was mounted as the top plate in the microfluidics system, with the photodetector facing downward. It was aligned with the linear array of electrodes on the lower part of the chip.

A computer controlled Keithley source measurement unit (SMU), was connected to the contact pads of the photodetector via two DC probes to apply voltage bias and measure photocurrent. A calibrated pigtailed laser diode emitting at a wavelength of 660 nm was normally incident on the surface of the photodetector to measure the responsivity of the I-MSM. The measured responsivity for surface normal illumination of the inverted I-MSM was 0.39 A/W.

II. METHODS

In order to perform these experiments, a coplanar digital microfluidic chip and voltage controller were used to manipulate the droplets, a hydrophobic top-plate with an integrated photodetector performed optical detection, and reagents for a suitable chemiluminescent reaction provided the signal to detect. The coplanar digital microfluidic chip was manufactured by ALL via printed circuit board (PCB) technology. The microfluidic voltage controller and software were also purchased from ALL were used. The coplanar digital microfluidic chip enables nearly any hydrophobic top plate to be used. The controller and software enable the programming or manual control of the voltages and timing of

each individual input of the digital microfluidic chip. All images were taken by removing stills from a video recorded using a Panasonic GP-KR222 CCD with a magnifying lens.

The optical detection function of the integrated photonic and microfluidic system was tested by mixing two droplets that participate in a chemiluminescent reaction. This chemiluminescent reaction, the oxidation of pyrogallol (1, 2, 3-trihydroxybenzene) in the presence of formaldehyde, in an alkaline solution, was chosen for its luminescent intensity and its peak wavelength. This reaction also occurs in an aqueous medium, which is a requirement for EWoD. These two solutions mix underneath the photodetector; equal parts of 0.8M pyrogallol and 9M NaOH were mixed, and an additional equal part of 38% formaldehyde (HCHO) was added. When two parts of this pyrogallol solution were mixed with one part of 30% H₂O₂, a short-lived, intense orange light was generated. We observed that intensity of the light decreased and the duration of the light emission increased with decreasing pyrogallol solution temperature prior to mixing. The pyrogallol solution and the hydrogen peroxide had contact angles with Teflon AF of 100° and 115° respectively, which are suitable for EWoD.

III. EXPERIMENTS AND RESULTS

Three measurements were performed. First, the response of the photodetector to a static microfluidic reaction was measured, followed by two tests where the reacting droplet was moved toward and away from the photodetection site. The first test was performed by connecting the coplanar digital microfluidic device to its controller, followed by the pipetting of two droplets (1.2 μ l for the pyrogallol solution, and 0.6 μ l for the H₂O₂ solution) onto appropriate electrodes for actuation towards each other. Subsequently, the hydrophobically coated photodetector was placed photodetector-side down onto spacers (required due to the large droplet size), carefully positioning the photodetector over the reaction site. The top plate was then fixed in place, and probes connected to the gold pads on the top plate (which were connected to the photodetector) to the SMUs for measurements. The ambient light was dimmed significantly, and 5V was applied to the photodetector through the SMU. The photodetector response to the mixing of the two droplets without any actuation (i.e. a static reaction) is shown in Fig. 4. This first data set indicates that the chemiluminescent reaction was detectable by the photodetector integrated with the top plate.

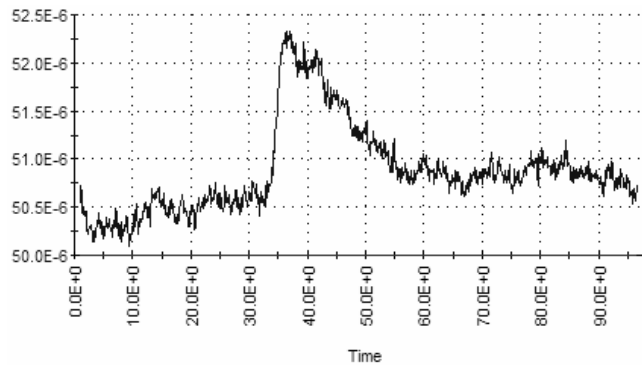


Figure 4. Chemiluminescence underneath the TeflonAF coated photodetector

The second and third tests performed were very similar to the first. The setup prior to actuation is the same; however the droplets were mixed by actuating them together through the use of the controller software, which caused the controller to apply 280V to the appropriate electrodes, causing the droplets to mix together underneath the photodetector. The resulting reacting (photon emitting) droplet was then slowly moved away from the photodetector, then toward the photodetector, and finally away from the photodetector. The result of a reacting droplet mixing underneath the photodetector and moving back and forth is found in Fig. 5. The difference in detectable signal duration and intensity between Fig. 4 and Fig. 5 may be explained by the cooling of the pyrogallol solution prior to the second experiment. This cooling resulted in a longer reaction time but reduced peak intensity. The second result demonstrates that the microfluidic system can move the reacting droplet, and that the heterogeneously integrated photodetector is capable of detecting the movement of the reacting fluid. This is a first step in demonstrating the heterogeneous integration of digital microfluidics and optical components. This result paves the way for future work that will involve the integration of more complex optical sensing devices and methods with digital microfluidics.

IV. CONCLUSIONS

In this paper, a thin film compound semiconductor photodetector has been integrated with a digital microfluidics system. The InGaAs-based thin film photodetector was bonded to the top glass plate of the digital microfluidics system, and was successfully used to detect the emission from two droplets that were mixed by actuation of the digital microfluidics system, and produced a chemiluminescent

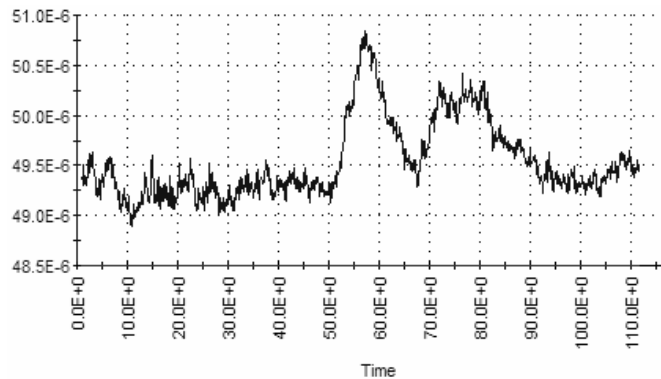


Figure 5. Chemoluminescing droplet moving back and forth underneath the photodetector twice

optical output when mixed. This heterogeneous integration of an optical detection component with microfluidics technology is a first step toward next generation portable LoC systems that can contain optical functions.

REFERENCES

- [1] F. Mugele and J. C. Baret, "Electrowetting: from basics to applications." *J. Phys: Condens. Matter*, 2005, vol. 17, pp R705-R774.
- [2] M. G. Pollack, A. D. Shenderov, R. B. Fair, "Electrowetting-based actuation of liquid droplets for microfluidic applications." *Appl. Phys. Lett.*, 2000, vol. 77, pp 1725-1727.
- [3] M. G. Pollack, A. D. Shenderov, R. B. Fair, "Electrowetting-based actuation of droplets for integrated microfluidics," *Lab on a Chip*, 2002, vol. 2, pp 96-101.
- [4] Sung Kwon Cho, Shih-Kang Fan, Hyejin Moon, CJ Kim, "Towards Digital Microfluidic Circuits: Creating, Transporting, Cutting and Merging Liquid Droplets by Electrowetting-based Actuation," *MEMS, 2002, The Fifteenth IEEE International Conference on*, pp 32-35.
- [5] M. Gong, CJ Kim, "Two-dimensional Digital Microfluidic System by Multilayer Printed Circuit Board," *MEMS, 2002, The Eighteenth IEEE International Conference on*, pp 726-729.
- [6] V. K. Pamula, M. G. Pollack, P. Y. Paik, H. Ren, and R. B. Fair, "Apparatus for manipulating droplets by electrowetting-based techniques." *U.S. Patent 6,911,132*, June 28, 2005.
- [7] C. G. Cooney, C-Y. Chen, M. R. Emerling, A. Nadim, J. D. Sterling, "Electrowetting droplet microfluidics on a single planar surface." *Microfluid Nanofluid*, 2006, vol. 2, pp 435-446.
- [8] U-C. Yi and CJ Kim "Characterization of electrowetting actuation on addressable single-side coplanar electrodes." *J. Micromech. and Microeng.*, 2006, vol. 16, pp 2053-2059.
- [9] P. Y. Paik, "Adaptive hot-spot cooling of integrated circuits using digital microfluidics." *Ph.D. thesis, Duke University*, 2006.
- [10] S.-Y. Cho, Sang-Woo Seo, "High-Speed Large-Area Inverted InGaAs Thin-Film Metal-Semiconductor-Metal Photodetectors," *IEEE Journal of Selected Topics in Quantum Electronics*, vol. 10, 2004.