A Wireless Chemical and Biological Microsensor Based on Dissolvable Membranes

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Abstract — **We demonstrate a wireless bio/chemical microsensor based on dissolvable membranes. The dissolution of a target specific membrane enables fluid flow into an on-chip microcapacitor, thereby drastically changing its impedance. Here, this fact is used to generate large intrinsic amplification and thus, a large output voltage, which allows the microsensor to directly drive a microcontroller operating at 3.6964 MHz, without complex electronics. The electronic modules, operating at 5 V, have been integrated onto a printed circuit board of dimension 10.2 cm × 10.2 cm, consuming 135 mW of power. An automated procedure to estimate the concentration of the sensed species (here, dithiothreitol) and interface circuitry for wireless transmission at 916.48 MHz with a range of 150 ft in closed premises are realized.**

I. INTRODUCTION

There has been a need for the development of portable, low cost, wireless bio/chemical sensors for applications such as autonomous detection of toxins in food supplies and environmental monitoring. This would necessitate a bio/chemical to electrical signal transduction scheme which is relatively compact, cost effective, low in power consumption, and capable of delivering a large electrical output that greatly simplifies associated signal conditioning circuits. Many schemes have been proposed for the detection of bio/chemical species with high sensitivity, such as nanowire nanosensors [1], zero mode waveguides [2], and porous silicon based detection schemes [3]. However, these methods generally require relatively complex electronics to generate a significant electrical output or special equipment that has portability limitations.

We demonstrate a miniature wireless sensing system applicable to the detection of bio/chemical targets. We previously reported on the sensing mechanism, which indicates the presence or absence of a target species. Fig. 1 shows the configuration of the sensing system. The microsensor has two key components – a dissolvable membrane as the sensing element and a microcapacitor with interdigitated electrodes as the bio/chemical to electrical signal transduction element (Fig. 2). The functioning of the microsensor is based on the selective dissolution of the membrane (here, a poly(acrylamide) (PAAm)-based disulfide-crosslinked hydrogel) in the presence of target species (here, dithiothreitol: DTT) [4]. Upon dissolution of the membrane, the target species flows into the microcapacitor (initially in air) causing a drastic change in its impedance. This is converted to a large electrical output (here, 5 V) using an external resistor.

The state of the microsensor is constantly monitored and transmitted wirelessly to a remote computer using RF communication modules operating at 916.48 MHz providing a range of 150 ft within closed premises. The high bio/chemical to electrical transduction of the microsensor facilitates it to be interfaced directly with the input ports of a microcontroller. The electronics have been integrated on a printed circuit board, which consumes 27 mA of current at 5 V.

This sensing mechanism, based on molecular-level chemical reaction, is highly sensitive and specific, and universal [5]. No DC power is consumed by the microsensor until detection of the target species. This feature is especially desirable for the microsensor to be integrated with portable wireless data communication modules. A single microsensor can serve to sense multiple species by using multiple membranes, each membrane being sensitive to a particular species. Also, electronically controllable valves [6] can enable or disable the sensing of a particular species. A remote computer can individually address these valves using wireless communication modules interfaced to the microsensor and computer. The fabrication process for the microsensor is based on liquid-phase photopolymerization (LP^3) and is compatible with conventional IC fabrication technologies [7, 8].

II. FABRICATION AND WIRELESS SYSTEM SETUP

The fabrication of the microsensor is a merger of MEMS and LP^3. The interdigitated electrodes of the microcapacitor are fabricated using MEMS electroplating. The dissolvable membrane and fluidic channels to direct the flow of target species are fabricated using LP^3. The fabricated microsensor is interfaced to electronic circuitry for wireless transmission.

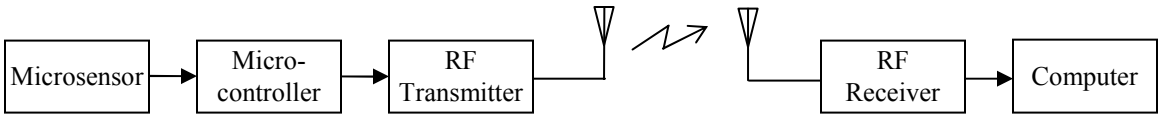

Figure 1. Schematic of the wireless sensing system. A bio/chemical sensor (Fig. 2) providing two output signals is fabricated and interfaced to the data input ports of a microcontroller (AT90S8515, Atmel Corporation) running at 3.6964MHz (CTS MX045HS Clock Oscillator, CTS Communications Components). The dissolution of the membranes in the sensor due to the target species generates signals having a magnitude equal to the sensor supply voltage (here, 5 V). This high output signal is attributable to the *high* bio/chemical to electrical transduction of the sensor. The time difference of the two sensor output signals gives an estimate of the concentration of the target species. The microcontroller polls the state of the sensor constantly. The serial interface of the microcontroller drives the transmit pins of the RF transmitter (916-SC-PA, Linx Technologies) directly. The RF receiver is interfaced to the serial port of a computer and communicates via the RS-232C standard. A 'C' program on the computer computes the time difference between the two sensor signals.

A. Fabrication of the microsensor

The fabrication process flow is reported in detail in our previous work [4]. The microsensor is fabricated on a glass substrate. To fabricate the microcapacitor, the substrate is sputtered with 0.05 µm Ti, 0.4 µm Cu, and 0.05 µm Ti. The microcapacitor is formed by electroplating nickel (Ni) using Cu as the seed layer. A 6.5 µm electroplating mold is patterned using AZ4620 photoresist. The Ti layer on top of the Cu layer, which prevents the oxidation of Cu, is removed just before electroplating. Ni is electroplated and the mold is removed. The seed metal layers (Ti/Cu/Ti) underneath the mold are removed, but the microcapacitor is not released.

The fluidic channels to direct fluid flow into the microcapacitor are constructed with an isobornyl acrylate-based polymer (poly(IBA)), using LP^3 and a film photomask [7]. Next, the hydrogel membranes which sense the target species are patterned using LP^3. Poly(IBA) and the hydrogel are similar to negative photoresists and are in the liquid state prior to ultraviolet (UV) exposure, and harden when irradiated by UV.

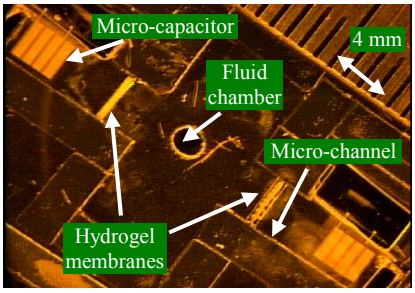

Figure 2. The bio/chemical sensor. A bio/chemical recognition membrane (here, a dissolvable poly(acrylamide)-based hydrogel) separates the sampling microfluidics and an interdigitated micro-capacitor. The capacitor is initially in air. The membrane becomes porous and dissolves in the presence of the target species (here, dithiothreitol or DTT), allowing the fluid to flow to the capacitor, drastically changing its impedance. Two proximal hydrogel membranes of widths 200 µm and 600 µm, having a thickness of 350 µm are used to track the time difference of dissolution, and thus, estimate the concentration of the target species. Fig. 9 illustrates the results.

B. Wireless system setup

Wireless communication between the microsensor and a remote computer is realized by interfacing wireless transceivers to the microsensor and remote computer. Fig. 1 illustrates the setup of the system for wireless transmission. The microsensor (Fig. 2) is interfaced to the data input ports of a microcontroller (AT90S8515, Atmel Corporation, San Jose, CA, USA) running at 3.6964 MHz (CTS MX045HS Clock Oscillator, CTS Communications Components, Bloomingdale, IL, USA). The microcapacitor, which provides an electrical output from the microsensor, is connected to the microcontroller through a probe station and BNC cables, as shown in Fig. 3. The microcontroller polls the state of the microsensor constantly and is programmed using the AVR-ISP in-system flash programmer (Atmel Corporation). The inbuilt Universal Asynchronous Receiver Transmitter (UART) of the microcontroller transfers data serially to a radio frequency (RF) transmitter (916-SC-PA, Linx Technologies, Grants Pass, Oregon, USA) [9]. The transmitter is specified to operate at a frequency of 916.48 MHz, 9600 baud with a bit error rate of less than 10^{-5}, and an output power of -10 dBm and a range of 150 ft in closed premises. A RF receiver is interfaced to the serial port of a remote computer and communicates with the computer via the RS-232C serial communication standard. A 'C' program on the remote computer computes the time difference between the two wirelessly communicated microsensor signals and thus, will give an estimate of the concentration as described in section III. Fig. 4 shows the RF transmitter interfaced to the microsensor using a bread-board based design. Fig. 5 shows the RF receiver interfaced to the remote computer.

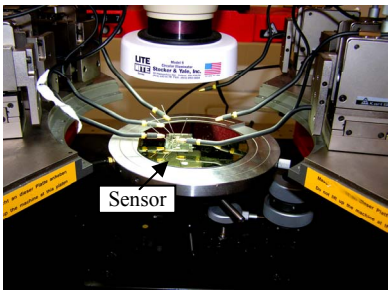

Figure 3. On-wafer testing is accomplished using a Karl Suss PM5 probe station. Probes are used to connect the output of the sensors to the microcontroller.

We have designed a two layered printed circuit board (PCBFabExpress, Sunnyvale, CA) having dimensions of

10.2 cm × 10.2 cm (Fig. 6), incorporating the electronics for wireless transmission. The circuit board consists of the microcontroller, RF module with antenna, and associated passive components. Its operation is as described previously, and is powered by a 9 V battery. A 5 V regulator drives the electronics. The circuit board consumes about 27 mA of current at 5 V (135 mW).

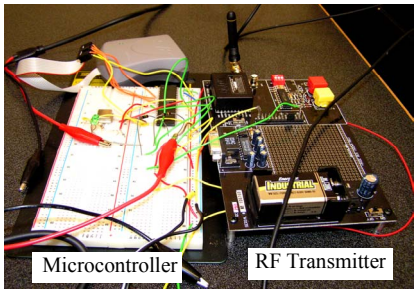

Figure 4. The wireless transmitter using a bread-board based design. The microcontroller and RF transmitter form the wireless transmitter operating at 916.48 MHz and 9600 baud with a bit error rate less than 10^{-5}. The range of transmission is about 150 ft inside a concrete building.

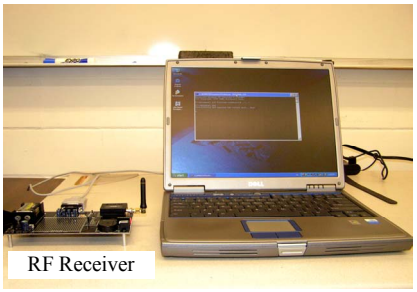

Figure 5. The wireless receiver. The RF receiver is interfaced to the serial port of the computer through the RS-232C standard. The computer employs a 'C' program to determine the time difference of the two received sensor signals.

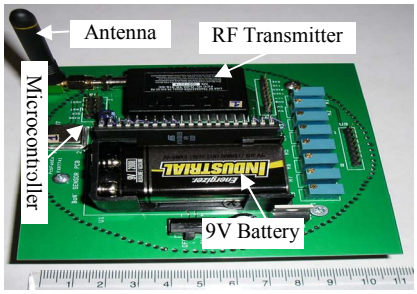

Figure 6. The printed circuit board incorporating the RF transmitter and microcontroller. A 9 V battery supplies power to the board. A 5 V voltage regulator drives the electronics. The board consumes around 27 mA at 5 V (135 mW) and measures 10.2 cm × 10.2 cm. The RF module can deliver up to 1 mW of output power.

III. EXPERIMENTS AND RESULTS

Fig. 7 shows SEM images of the fabricated microcapacitor. The electrodes have a width of 9.4 µm separated by a gap of 3.5 µm. Functional testing on the microcapacitor was done using a Karl Suss PM5 probe station and HP4284A LCR meter. Following the functionality testing, the microcapacitor was connected to a 5 V DC power supply and an external 470 kΩ resistor to form a high pass RC circuit (Fig. 8). The voltage across the external resistor was used to drive the input ports of the microcontroller directly.

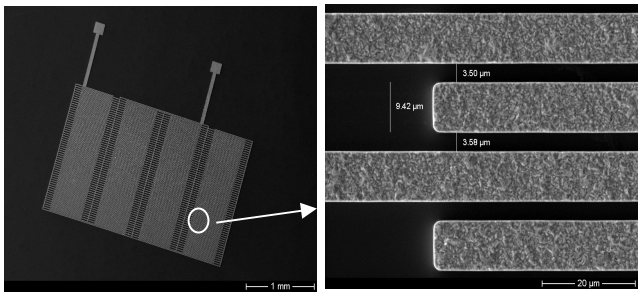

Figure 7. SEM images of the fabricated microcapacitor. **(a)** The entire microcapacitor. **(b)** The interdigitated microcapacitor electrodes. The electrodes have a width of about 9.4 µm separated by a gap of about 3.5 µm.

Upon introduction of the target species into the microsensor, the membrane became porous and dissolved. The slightly conductive target species entered the interdigitated electrodes of the microcapacitor (initially in air) and transformed it into a resistor of value around 1 kΩ. An output voltage equal to 5 V was generated using an external resistor R_o of value 470 kΩ in the transduction circuit of Fig. 8.

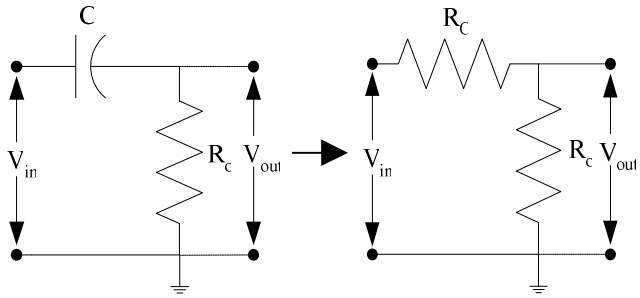

Figure 8. The transduction circuit. The microcapacitor with interdigitated electrodes is connected to an external resistor to form a high pass RC circuit. Upon dissolution of the membrane, the slightly conductive target species enters the microcapacitor and transforms it into a resistor. Thus, an output voltage results following membrane dissolution. This voltage is sufficient to drive the input ports of a microcontroller directly.

The dissolution time of the membrane depends upon the membrane width and concentration of the target species, as shown in Fig. 9 [10, 11]. Hence, for a known membrane width, the concentration of the target species can be estimated based on the dissolution time of the membrane. For autonomous sensing, the instant of entry of the target species into the microsensor is unknown. Due to this fact, it is not possible to establish the dissolution time with a single membrane. Here, we employ two proximal membranes of widths 200 µm and 600 µm and a microcapacitor for each of

these membranes. A signal from the microcapacitor associated with the 200 µm wide membrane indicates the presence of the target species. A subsequent signal from the other microcapacitor gives the time difference of dissolution between the two membranes. This information is used to estimate the concentration of the target species. The signals from the microcapacitors were transmitted to a remote computer which computed the dissolution time difference.

Fig. 9 demonstrates an example of measuring the target concentration. Individual dissolution times of 1320 s and 3214 s were obtained corresponding to two membrane widths of 200 µm and 600 µm respectively. Based on the dissolution time difference of 1894 s of the two membranes, the estimated concentration was approximately 0.25 M.

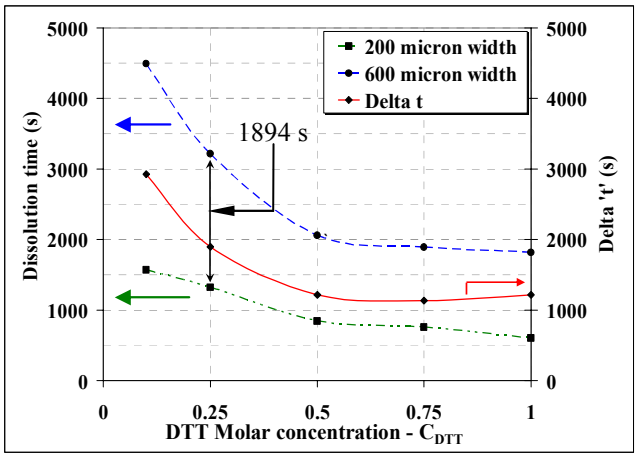

Figure 9. A mechanism to estimate the concentration (C_{DTT}) of the target species from the dissolution time of the bio/chemical recognition membranes. The dissolution time increases when the concentration of the target species (DTT) decreases *or* when the membrane (acrylamide-based hydrogel) width increases. The time difference of dissolution ('Delta t') of two proximal membranes of different widths is used to estimate the concentration of the species. For example, if the time difference of dissolution is 1894 s (i.e., when two electric signals generated due to membrane dissolution are separated in time by 1894 s), the estimated target species concentration is 0.25 M. Concentrations up to 0.5 M can be estimated, beyond which the curve becomes saturated [4].

IV. CONCLUSION

We have demonstrated a wireless bio/chemical microsensor based on dissolvable membranes. The approach is universal since a range of species can be sensed by defining membranes specific to the target species. This bio/chemical recognition process, based on molecular-level chemical reaction can be highly specific and sensitive. A large electrical output can be obtained using a microcapacitor with interdigitated electrodes as a bio/chemical to electrical signal transduction element. Based on this sensing scheme, two proximal membranes with individual dissolution characteristics can be used to estimate the concentration of the sensed species. The state of the microsensor can be monitored remotely using wireless communication modules interfaced to the microsensor. The power consumption of the complete system can be further reduced by using a 3 V supply, at the expense of reduced wireless transmission range. Also, the size of the printed circuit board can be further reduced by using surface mount components instead of through-hole components used in this design.

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