

A miniaturized low-power wireless remote environmental monitoring system based on electrochemical analysis

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Abstract

In this paper, we report a miniaturized low-power wireless remote environmental monitoring system. This system has been developed for on-site monitoring of water pollution by heavy-metal ions. The system is composed of three parts: an electrochemical sensor module using microfabricated electrodes for detecting heavy-metal contamination in sample water; a custom potentiostat module including readout circuitry, analog-to-digital converter and microcontroller; and a radio frequency (RF) module for sending detected signals to a base station through wireless communication. The electrochemical sensor module is implemented using microfabricated mercury working electrodes (WEs), solid-state reference electrode (SSRE), and platinum counter electrode (CE). For the low-power operation, direct frequency-shift keying (FSK) modulation and simple binary FSK demodulation methods are used for RF module which is realized using 0.18 μm CMOS technology. All the modules are hybrid integrated in a printed circuit board (PCB) and low-power consumption below 1 mW has been achieved.

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1. Introduction

As the contamination of groundwater by heavy-metal ions is critically harmful to many organisms, including humans, there have been various researches and reports on in situ and on-site detection of heavy-metal contaminants using electrochemical detection methods [1–5]. Electrochemical detection can be easily realized in a simple and cheap implementation platform with high sensitivity and its implementation is quite compatible with conventional semiconductor processing technologies. The previous approaches have used electrochemical sensor integrated with a custom designed potentiostat for simple on-site analysis system. However, they have used a laptop computer for data acquisition, signal processing, and system control, and this makes it difficult to achieve periodic on-site environmental monitoring in broad field areas.

In this paper, we have proposed and implemented the miniaturized wireless remote environmental monitoring system. This system monitors water pollution of heavy-metal ions by using electrochemical detection and wirelessly transmits the detected signal to a base station. As for wireless communications, we have adopted a low-power distributed wireless network scheme [6–9]. The proposed environmental monitoring system includes sensing electrodes, potentiostat, and radio frequency (RF) communication module with an antenna and can be located in broad open fields such as river, coast, etc. When the analysis is requested by the base station, heavy-metal ions in the water are analyzed by the electrochemical sensor with potentiostat and the detected signals are transferred to the base station through the RF module.

There are some requirements for the proposed wireless monitoring system. In order to realize a miniaturized system, all the important components for the proposed sensor system should be integrated in a small form factor. Also, as a distributed portable system, the entire components must be optimally designed to reduce the power consumption. In

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order to fulfill these requirements, the proposed sensor system has been implemented using electrochemical sensor fabricated on silicon substrate by microelectromechanical system (MEMS) technology and a custom RF communication device optimally designed for low-power consumption.

2. System description for wireless environmental monitoring

The block diagram of the proposed wireless environmental monitoring system is shown in Fig. 1. The base station

generates and transmits instruction signals for the operation of distributed sensor systems. The distributed sensor system receives instruction signals from the base station and performs the monitoring of water pollution and transmits the sensing information to the base station again.

For the distributed sensor system, we have developed three basic modules: sensing electrodes module for electrochemical detection; custom potentiostat module for sensor read-out, signal processing and control; and RF communication module for wireless communication. Fig. 2 shows the detailed block diagram of the proposed distributed sensor. In this work, we have fabricated the mercury working electrodes (WEs) and solid-state Ag/AgCl reference electrode (RE) on the same silicon substrate using MEMS technology and assembled the rest of modules in a hybrid platform by integrating the custom potentiostat and the custom RF module chip fabricated using 0.18 μm CMOS technology.

Instruction signals will be wirelessly transmitted from the base station at a close distance and received at the sensor system through an antenna. The targeted minimum communication distance is about 50 m. In the RF module, the received RF signals are demodulated and converted into digital signals which will be the series of input control signals to the MCU. The MCU generates sensor driving signals which will be applied to the sensing electrodes through digital-to-analog converter (DAC) and amplifiers. Electrochemical analysis is performed at the sensing electrodes according to the received control signals. Next, the detection signal from the sensor module is transferred into the MCU and finally transmitted to the base station after modulation in the RF module.

For the low-power operation, the proposed distributed sensor network has been designed to have two operation

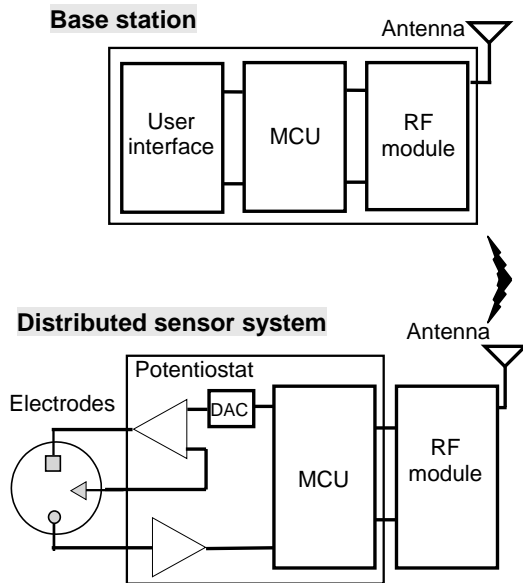


Fig. 1. Block diagram of the proposed wireless environmental monitoring system.

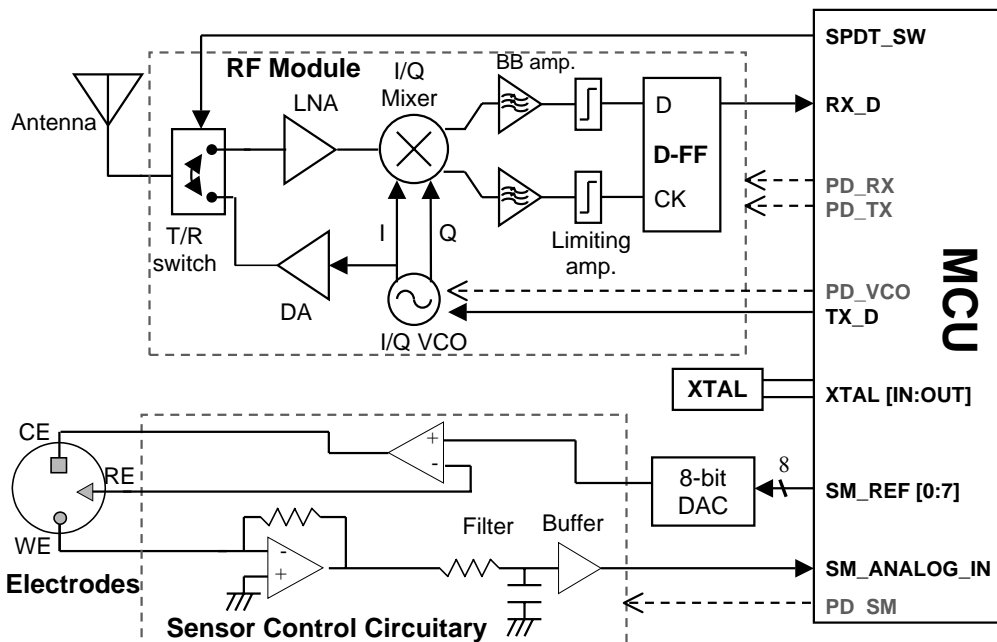


Fig. 2. Block diagram of the proposed distributed sensor system including sensor module, potentiostat, and RF module with antenna.

modes—sleep mode and wake-up mode. During the sleep mode, only the MCU operates in non-active state consuming very small fraction of power, while the rest of the other modules are turned off. During the wake-up mode, the MCU takes an active state and scans the RF input signal. If the instruction signals are detected during the wake-up mode, the MCU activates the system and starts to supply power to the other modules. To minimize the power consumption in this wake-up mode, the MCU gradually enables the system as needed and properly disables the unnecessary parts of the system. For example, during the receiving-mode, the sensor module and drive amplifier (DA) in RF module are not activated. On the other hand, during the transmitting-mode, the sensor module and LNA, mixer, and baseband circuits are not activated. The sensing module is only activated during the sensing-mode according to power scheduling by the MCU.

3. Electrochemical sensor

Previously, we reported our research results on electrochemical detection of heavy-metal ions using three electrodes (working, counter, and reference electrodes) separately microfabricated on individual silicon substrate [10]. In this paper, we have integrated all three electrodes on single silicon substrate for the miniaturized electrochemical sensor of the proposed monitoring system and utilized the same detection mechanism as in the previous report.

3.1. Electrodes configuration

One of the most useful electrochemical techniques for detection of heavy-metal ions is anodic stripping voltammetry (ASV), which can achieve a very low detection limit by employing a pre-concentration step [11]. In this work, we have used square wave anodic stripping voltammetry (SWASV), a special form of ASV, where a square waveform is applied to the electrode for a higher sensitivity [12].

In most electrochemical measurement systems, a three-electrode configuration is widely used instead of a two-electrode configuration due to its superior electrochemical stability and suppressed errors [13]. In this work, we have utilized a three-electrode configuration composed of mercury working electrode, solid-state Ag/AgCl reference electrode and platinum counter electrode (CE).

It is well known that the current passing through working electrodes consists of two components. One is *Faradaic current*, which is the actual electrochemical signal that we would like to monitor, and the other is *charging current*, which is the non-Faradaic parasitic current component caused by double-layer capacitance formed at electrode–liquid interface. Using small electrode sizes in micrometer scale, charging current becomes negligible compared to Faradaic current and we can obtain a high signal-to-noise ratio. Previously, we have examined the size

effect of working electrode and have demonstrated that superior sensitivity down to ppb levels is possible by reducing electrode size [10]. In this work, we have used the array of microelectrodes with a diameter of 10 μm . Also, the working electrode has been monolithically integrated with counter electrode as well as solid-state reference electrode which is made of Ag/AgCl thin films coated by a thin polymer layer containing chloride ions for stable potential.

3.2. Fabrication

All the electrodes have been fabricated on single silicon substrate. First, the platinum is deposited and patterned to the thickness of 200 nm for working and counter electrodes by using lift-off method with conventional photolithography followed by patterning of silver layer with thickness of 400 nm for reference electrode. For contact pads, the gold layer is also patterned by using same lift-off technique. After the formation of metal layers, the plasma enhanced chemical vapor deposition (PECVD) silicon nitride is deposited and patterned by photolithography to define the electrodes and contact pads. To form a solid-state reference electrode, the silver layer is converted into Ag/AgCl layer by dipping in FeCl_3 solution for 2 min. Then the Ag/AgCl layer is dipped in 12 ml tetrahydrofuran (THF) solution containing 0.4 g polyvinyl chloride (PVC) and saturated NaCl. After drying for 48 h, Nafion is coated by dipping in 5% Nafion solution [10,14]. During the dip-coating of PVC/Nafion layer, only the Ag/AgCl layer is exposed to solution by covering the other area on the chip with polydimethylsiloxane (PDMS) block which is detached after the coating process. Detailed process flows can be found in the previous publication [10].

Finally, the mercury working electrodes are formed on platinum surface by applying -150 mV with respect to the SSRE in the solution containing 10 mM Hg^{2+} ions. Fig. 3 shows the photograph of the fabricated electrodes including an array of mercury working electrodes.

3.3. Experiments

The fabricated electrodes have been tested using a commercial potentiostat (BAS100b). Fig. 4 shows the potential variation of the fabricated SSRE versus a commercial Ag/AgCl reference electrode with respect to various Cl^- concentrations. The pure Ag/AgCl electrode has a slope of about -53 mV/dec, which agrees well with a theoretical value of -58.5 mV/dec. However, the PVC/Nafion coated electrodes have a much smaller variation with respect to Cl^- concentration changes. The potential variations are about 40–55 mV under 0.1 M Cl^- concentration and this margin may be sufficient to distinguish metal ions from one another for in situ heavy-metal ion measurement where the reduction potentials of interesting ions are separated over about 170 mV.

The SWASV results using the fabricated three electrodes and commercial BAS potentiostat is shown in Fig. 5. The

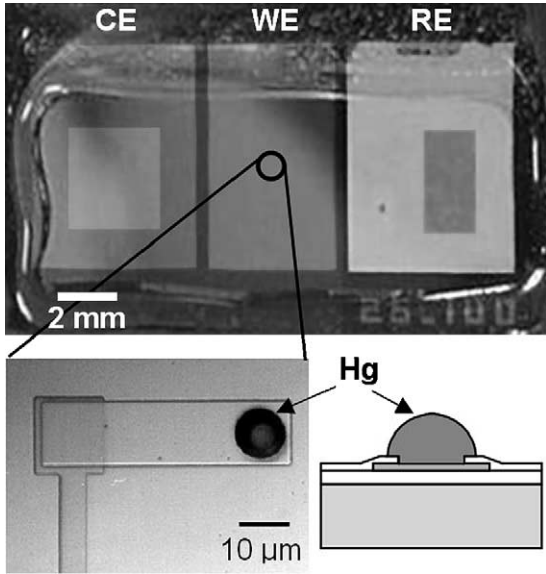


Fig. 3. Three electrodes microfabricated on the same silicon substrate. The magnified view of mercury working electrode is shown with its cross-section.

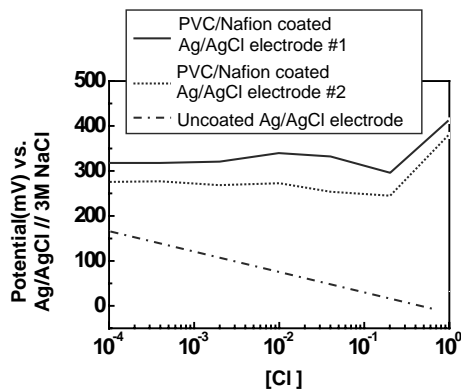


Fig. 4. Potential deviation of the fabricated solid-state reference electrodes compared with commercial Ag/AgCl standard reference electrode for various Cl⁻ concentrations.

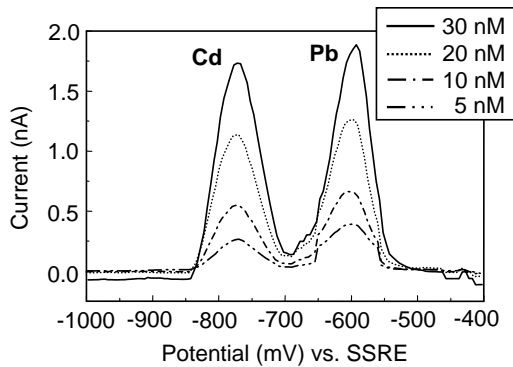


Fig. 5. Measured current from SWASV in the fabricated electrodes using commercial potentiostat.

experiment was performed for successive additions of Cd and Pb ions to distilled water without buffer solution. The preconcentration was carried out for 60 s and the frequency of square wave was 120 Hz. This experiment demonstrates that distinctive detection peaks for two heavy-metal ions can be monitored with a minimum detectable limits equivalent to 1 ppb for Pb and 0.5 ppb for Cd ion contaminations, respectively.

4. Potentiostat

A potentiostat is an electronic circuit that controls an electrochemical reaction by maintaining a voltage between a solution and a sensing electrode. In this work, a custom potentiostat has been designed and implemented using hybrid integration of commercial low-noise op-amps (AD8551, Analog Devices Co.), MCU (MSP430F112, Texas Instruments Co.), DAC, and some passive components on a printed circuit board (PCB).

In this work, eight-bits digital signals generated from the MCU are converted into square wave signals at DAC, which is used to define the voltage between the working electrode and the reference electrode to induce electrochemical reaction on the surface of the working electrode. The resultant reaction current passing through the working electrode is amplified in the current-to-voltage amplifier stage and transferred into the MCU through high-pass filter and buffer stage. Then, this analog signal is converted into a digital signal by the ADC. Finally, the acquired signal is processed in MCU and transferred into the RF module where the signal is modulated and transmitted to the base station.

5. RF communication module

This section will describe the design and operation of RF communication module and briefly explain some key components of the proposed communication system. The more details can be found in other references [15,16].

5.1. Architecture and modulation scheme

As shown in Fig. 6, a direct conversion architecture has been chosen in the proposed RF communication

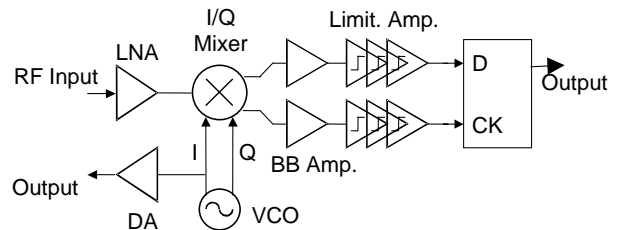


Fig. 6. Direct-conversion transceiver for binary FSK modulation.

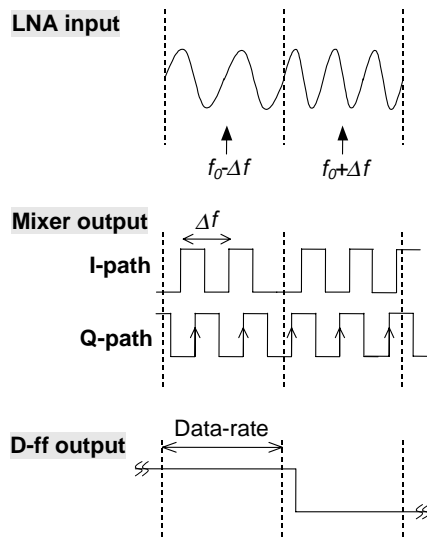


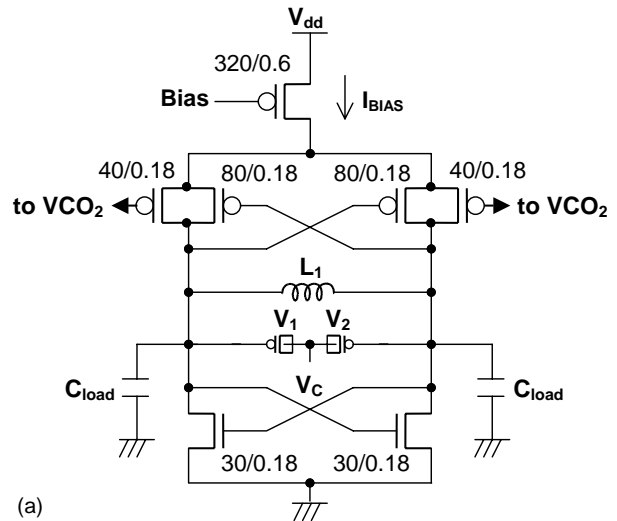
Fig. 7. Waveforms in the receiver path of binary FSK demodulation module.

network. This RF module requires only minimum RF circuit blocks including a low-noise amplifier (LNA), a mixer, a voltage-controlled oscillator (VCO), and a driver amplifier. Since most subsequent amplification and filtering can be processed in the base band module at low frequencies, the power dissipation in the RF section is very small compared to other architecture options [17].

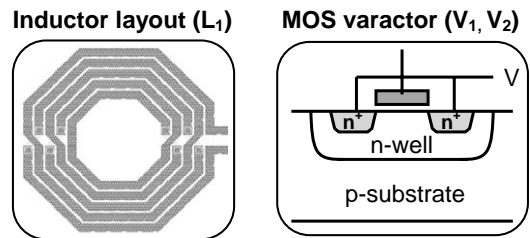
The received signal through antenna is demodulated by using a binary frequency-shift keying (FSK) demodulation method. For transmitting signals, the signal output from the MCU is modulated using a direct FSK modulation scheme and transmitted to the base station through antenna. The carrier frequency is 2.4 GHz and the FSK offset frequency (Δf) is 5 MHz. Fig. 7 shows the waveforms in the receiver path. Incoming RF signals are frequency-converted into two paths by an I/Q mixer driven by quadrature phases from a local oscillator (LO). Consequently, the mixer outputs are phase-shifted by 90° with respect to one another. After limiting the magnitude of the mixer output signals, one is applied to the data-input, and the other to the clock-input. With the phase difference in the I - and Q -paths, a D flip-flop detector can make instantaneous decisions. As shown in Fig. 7, increase in the FSK offset frequency, Δf , results in the reduction of decision errors from the LO frequency drift, which is desirable in our implementation.

5.2. Voltage-controlled oscillator and transmitter

The schematic diagram of quadrature VCO is shown in Fig. 8. Two cross-coupled NMOS and PMOS pairs in positive feedback compensate for LC tank loss. This structure has a better phase-noise performance for a given bias current due to higher oscillation amplitude and waveform symmetry [18]. To make quadrature waveforms, two LC VCOs are directly cross-connected by using additional PMOS' [16].



(a)



(b)

Fig. 8. Circuit schematic of CMOS VCO. Insets show the layout of the symmetric inductor and the cross section of an accumulation-mode MOS varactor, respectively.

One symmetric octagonal inductor has been used for an LC oscillator because it gives good signal balancing and consumes much less silicon area compared to a VCO circuit schematic using two independent inductors in series. The measured inductance and quality factor are 7.4 nH and 9.2 at 2.4 GHz, respectively. High- Q ($Q > 60$) varactors are designed for frequency tuning using an accumulation-mode MOS transistor with n^+ poly gate and n^+ diffusion source/drain in n-well [16]. The fabricated high- Q quadrature VCO consumes only 1.45 mA at supply voltage of 1.8 V. Also, this VCO directly drives the mixer and driver amplifier to save power consumption at the output buffer.

5.3. Performance summary of RF communication module

Fig. 9 shows the chip photograph of the fabricated CMOS RF communication chip. Except for matching circuits of the LNA input and DA output, all circuit blocks are integrated in a single chip. Die size is $1.35 \times 1.4 \text{ mm}^2$ including bonding pads. Measurement has been performed at 1.8 V supply voltage. The LNA and mixer conversion gain has been measured as 23.9 dB, input-referred 1 dB compression point (P1dB) as -23 dBm , and input third-order intercept point (IIP3) as -14 dBm , respectively. The tuning range of the fabricated VCO is from 2.335 to 2.445 GHz, corresponding to 4.6% of its center frequency. The measured phase

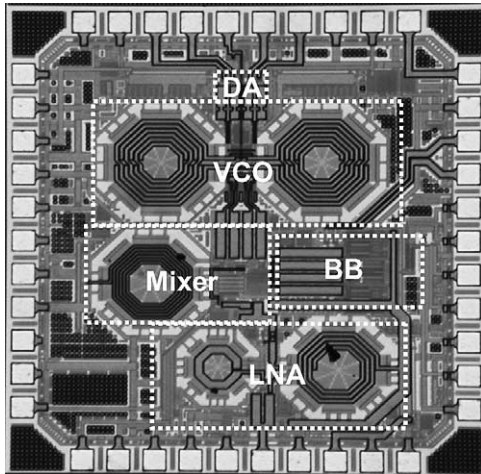


Fig. 9. Fabricated custom CMOS RF chip.

noise is -117.94 dBc/Hz at 1 MHz offset frequency. In the receiving-mode, the LNA, the mixer, BB circuits, and the VCO are all activated, and the total power consumption has been measured as 7.0 mW. In the transmitting-mode, only the VCO and the driver amplifier are turned on and 4.4 mW has been consumed.

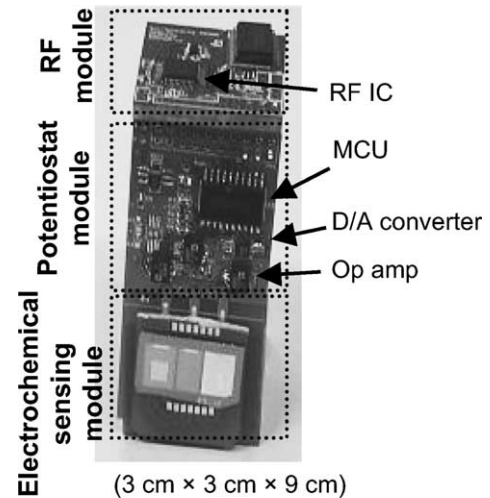
6. Antenna

Implementing a small and cheap antenna for 2.4 GHz transceivers is a challenge in low data rate wireless applications. In general, a patch antenna should have the size of at least half wave length ($\lambda/2$) because mirroring image appears at the center around one side edge. In the case of an inverted- F patch antenna, one side edge is electrically open and the other is short; therefore, it gives a fundamental resonance at quarter wave length ($\lambda/4$). An inverted- F patch antenna can reduce the antenna size by half. In this work, an inverted- F patch antenna [19] has been implemented in a PCB. It size is 4.5 cm² and it provides a high gain (3 dBi) and a large bandwidth of 230 MHz at 2.4 GHz.

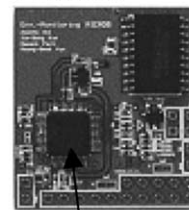
7. System integration of prototype

Fig. 10 shows the entire wireless environmental monitoring system hybrid integrated in a PCB, including sensing electrodes, potentiostat, and RF module. The patch antenna has been formed on the backside of the PCB to reduce a form factor. The total system size is about 3 cm \times 9 cm \times 0.5 cm.

The implemented system has a receiver sensitivity of about -62 dBm and transmitter output power of about -8 dBm. The maximum distance for safe signal transfer of the prototype system has been estimated to be about 20 m. If we increase the performance of base station, which is free from the system restriction such as power consumption and system area, to output power of 10 dBm and receiver sensi-

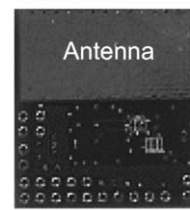


Front side



RF chip

Back side



Antenna

Fig. 10. Completed wireless environmental monitoring system composed of electrochemical sensing module, signal processing unit including potentiostat circuits and RF communication module.

tivity of -80 dBm, then the target communication distance of 50 m can be obtained.

As was explained in the previous sections, the system has been designed to operate in two modes for low-power operation—sleep mode and wake-up mode. The power consumption for sleep mode is measured below 10 μ W. During wake-up mode, the peak power consumption of the system has been monitored as 7, 4.4 and about 8 mW for receiving-mode, transmitting-mode, and sensing-mode, respectively. In this work, the timing ratio between sleep and wake-up modes is scheduled in a way that the total average power consumption is maintained below 1 mW.

8. Experimental results

The MCU/DAC unit in the monitoring system generates a signal which will be applied on working electrode for SWASV. First, the potential is held at a fixed value for a few seconds. During this time, heavy-metal ions dissolved in water are being accumulated into mercury electrodes (pre-concentration). Next, the potential is swept in a square waveform during which the heavy-metals are stripped out into solution to generate a current at respective reduction/oxidation potentials (stripping). The detection current is sampled at the end points of each square waves and

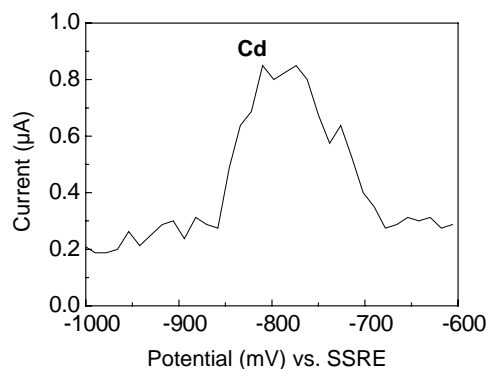


Fig. 11. Measured SWASV result using the implemented monitoring system.

processed in MCU after digitalization and finally transferred to base station. Fig. 11 shows the measured detection signal using the fabricated monitoring system. In this case, we have used a water sample with 1 mM cadmium contamination in 10 mM acetate buffer and are able to observe the reduction peak for cadmium ion. The detection signal shows some noise and signal broadening of about 50 mV compared to the results using commercial potentiostat. This signal distortion, which limits the detectable concentration level of traces, presumably comes from imperfect generation of square waves and inaccurate sampling point of detection current. With the design optimization of signal control/process module, we are expecting improved in situ environmental monitoring system which can detect mixtures of heavy-metal ions with acceptable sensitivity.

9. Conclusions

We have implemented a wireless remote environmental monitoring system. The system consists of three modules: electrochemical sensor module for the detection of heavy-metal ion contamination in water; custom potentiostat module (including readout circuitry and signal processor); and RF module for wireless communication with a base station. The electrochemical sensor electrodes have been fabricated on single silicon wafer. Other circuit components have been hybrid integrated with the sensor. The total system has been implemented for two-way wireless communication with the base station with a receiver sensitivity of -62 dBm, transmitter power of -8 dBm, and average power consumption of below 1 mW. Using the implemented prototype system, we have successfully demonstrated the detection of cadmium contamination in water with low-power consumption.

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