Semiconductor-Type MEMS Gas Sensor for Real-Time Environmental Monitoring Applications

Seung Eon Moon, Nak-Jin Choi, Hyung-Kun Lee, Jaewoo Lee, and Woo Seok Yang

Low power consuming and highly responsive semiconductor-type microelectromechanical systems (MEMS) gas sensors are fabricated for real-time environmental monitoring applications. This subsystem is developed using a gas sensor module, a Bluetooth module, and a personal digital assistant (PDA) phone. The gas sensor module consists of a NO_2 or CO gas sensor and signal processing chips. The MEMS gas sensor is composed of a microheater, a sensing electrode, and sensing material. Metal oxide nanopowder is drop-coated onto a substrate using a microheater and integrated into the gas sensor module. The change in resistance of the metal oxide nanopowder from exposure to oxidizing or deoxidizing gases is utilized as the principle mechanism of this gas sensor operation. The variation detected in the gas sensor module is transferred to the PDA phone by way of the Bluetooth module.

Keywords: Gas sensor, metal oxide, nanopowder, microheater.

I. Introduction

Wireless sensor networks (WSNs) have many attractive applications, inevitably making them an important and growing market in the near future [1]. Inventory systems, logistics management, healthcare, domestic control, and environmental monitoring are only a few examples of WSN applications. Such sensor networks require the combination of a huge number of inexpensive small sensors with wireless communication and ultra-low power consumption. Communication can be direct with base stations or in multihop networks that are configured ad hoc.

Air quality control is the main aspect in the field of environmental monitoring, as respiratory organ diseases are mainly ascribed to poor air quality. Atmospheric environmental standards are somewhat different in each country, and air quality can be expressed through the concentration of several pollutants, such as carbon monoxide, sulphur dioxide, nitrogen dioxide, and ozone. The threshold values specified for these pollutants by the European Environment Agency (EEA) assure indoor air quality of 10 mg/m^3, 350 $\mu g/m^3$, 40 $\mu g/m^3$, and 120 $\mu g/m^3$, respectively [2]. Among these, the sensing of carbon monoxide and nitrogen dioxide in the atmosphere has been assumed to be of great importance owing to the serious problem of atmospheric air pollution caused by automobile exhaust and other sources.

Various types of gas sensors with different operational methods have been developed in recent years. In spite of their high operating temperature, resistive metal oxide sensors play a significant role because of their low cost and high sensitivity. Having a smaller size than electrochemical and optical gas sensors and lower power consumption than optical gas sensors are the other merits of WSN applications [3]-[12].

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Metal oxide nanopowders for gas sensors present higher sensitivities and shorter recovery times than conventional metal oxide film sensors, owing to the effects of their size reduction [13]-[17]. For these reasons, metal oxide nanopowders are believed to be very promising in the development of a new generation of metal oxide sensors with improved properties.

In this paper, we present the design and experiment results of a semiconductor-type microelectromechanical systems (MEMS) gas sensor utilizing a metal oxide nanopowder gas sensor for the detection of carbon monoxide and nitrogen dioxide for a wireless environmental monitoring subsystem, which is small enough to be directly attached to wireless nodes, such as a Bluetooth head set, and operate using a personal digital assistant (PDA). This system is composed of four major parts: a gas sensor, sensor-interrogating and digital-interfacing printed circuit boards (PCBs), a Bluetooth module, and a real-time data acquisition program executed on a PDA. The actual performances of our sensor chip and wireless environmental monitoring subsystem are evaluated for carbon monoxide and nitrogen dioxide gases. These results are described and discussed below, along with the embodiment processes of the PDA-based wireless environmental monitoring subsystem.

II. Fabrication and Measurements

1. Metal Oxide MEMS Gas Sensor

For rapid gas sensing and desorption, a microheater is necessary for high-temperature operation. In a microheater structure, the two semicircular Pt heaters are connected to the power supply and are embedded in the membrane, which consists of SiO_2 and SiN_x thin films. The resistance of each heater becomes electrically equal, and each heater line is divided in half by the heat-spreading circular line. The generated heat diffuses through the heat spreader to promote thermal uniformity in the center area, which is thermally isolated by air using a membrane structure. The power consumption of the microheater device is simulated using commercial finite element method software. A uniform temperature distribution at the center of the microheater can be confirmed, and a temperature of 400ºC is attained at the expense of 20 mW of power consumption.

Based on the above-mentioned design, a bulk micromachined microheater is fabricated using a CMOS-compatible MEMS process, as shown in Fig. 1. Multilayers are initially deposited onto a 500-μm-thick two-sided polished p-type Si wafer, where the resistivity is approximately 1 Ω·cm to 30 Ω·cm and the direction is (001). After forming the multilayers, the deposited Ti/Pt thin film is patterned for the microheater by using a lift-off process. To maintain electrical insulation between the heater and gas sensing electrode and to protect the sensing electrode from thermal damage, a SiO_2 passivation layer is deposited and patterned. We complete the bulk micromachined MEMS microheater using back side anisotropic etching with KOH (40%wt) at 75ºC [18]-[23], in which etching is automatically stopped at the dielectric layer.

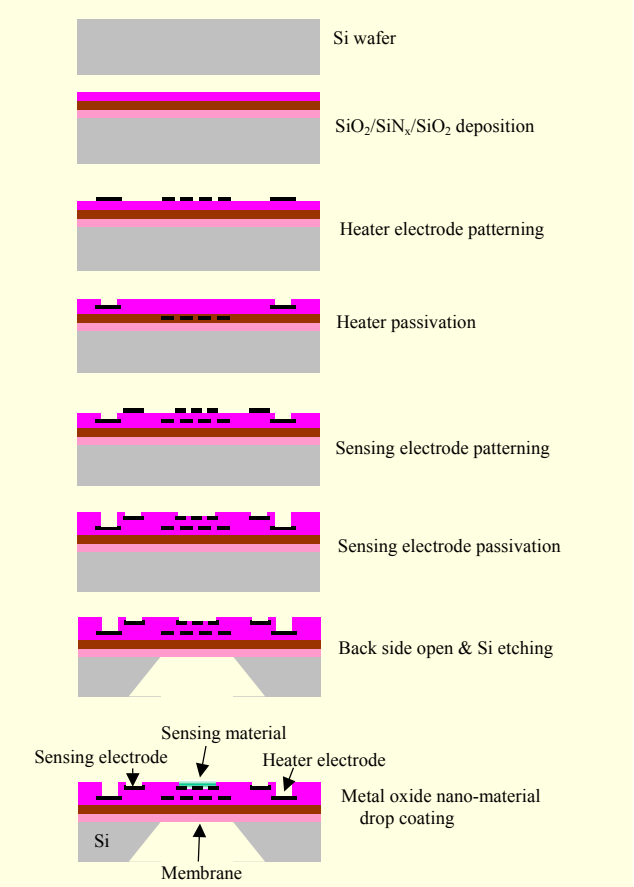

Fig. 1. Fabrication process of micro gas sensor based on CMOS-compatible MEMS process.

Semiconducting SnO_2 nanopowders are synthesized using a coprecipitation (CPT) method. For a homogeneous CPT process, 0.5 mol/L urea solution is added drop-wise to 0.025 mol/L of $SnCl_4 \cdot 5H_2O$ solution, which is stirred over a 10-minute period. The mixture is then heated slowly to 80ºC and allowed to react for four hours. The urea decomposes slowly during the release of ammonia and carbonate ions into the solution. The gradual and uniform rise in pH results in nucleation and growth of uniformly sized and shaped particles of the metal oxy-basic carbonate. The mixture is then cooled in an ice bath for three hours. After discarding the supernatant, the precipitate is washed to remove any traces of chloride ions. It is then dried at 120ºC, hand-ground, and calcined in an alumina crucible at 550ºC for two hours to form tin oxide powders.

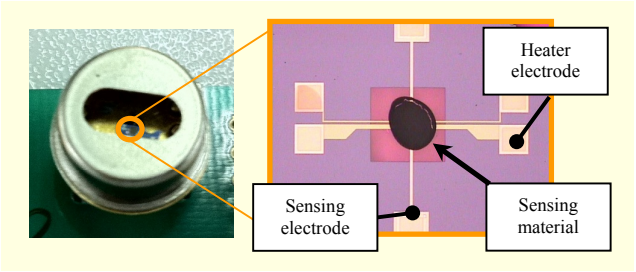

Fig. 2. Photographs of MEMS gas sensor based on SnO_2 nanopowder.

From the calcined SnO_2 nanopowders, to increase the response for CO gas, other metal oxide nanopowders and rare metals are added. Based on the SnO_2 nanopowders, 10%wt WO_3 nanopowders and a 0.5%wt Pt solution are mixed and ball milled with zirconia balls and ethanol for 48 hours. After ball milling, the sensing nanopowders based on SnO_2 nanopowders are obtained by drying for 12 hours at 120ºC. The structural properties of the SnO_2 nanopowders are characterized using X-ray diffraction (XRD) and a scanning electron microscope (SEM) analysis.

SnO_2 nanopowders are drop-coated in slurry form, which is a mixture of SnO_2 nanopowders, binder, solutions, and so on. After SnO_2 coating, to obtain a rigid contact between the sensing electrode and nanopowders and to obtain a stable gas detection operation, rapid thermal annealing is performed using an embedded microheater.

A photograph of the metal oxide nanopowder gas sensor is shown in Fig. 2. The photograph is of the packaged gas sensor based on SnO_2 nanopowders. Because the selective metal oxide nanopowder coating process is the final step, the device fabrication process is CMOS compatible.

The current-voltage characteristics are measured using an Agilent 4156C semiconductor precision analyzer. For all metal oxide nanopowder devices, to obtain a rigid contact between the sensing electrode and nanomaterial, rapid thermal annealing is performed using an embedded microheater below 500ºC for less than five minutes. The measured current characteristics between the sensing electrodes are sometimes unstable, and the current magnitude is about several pA at 1 V DC bias before annealing. After annealing, the measured current characteristics are always stable and several hundreds of pA at 1 V DC bias at room temperature, as shown in Fig. 3. Therefore, post-annealing nanopowder devices are used for measuring the electrical and gas sensing properties.

The gas sensing properties are measured using a computer-controlled characterization system and PDA based on a Bluetooth technique. The test gases are mixed with air to achieve the desired concentration, and the flow rate is maintained constantly using mass flow controllers. The response is given by the relative resistance, $R = \Delta R/R_0 = |R_g - R_0|/R_0$, where R_g and R_0 are the resistances in the test gas and air, respectively.

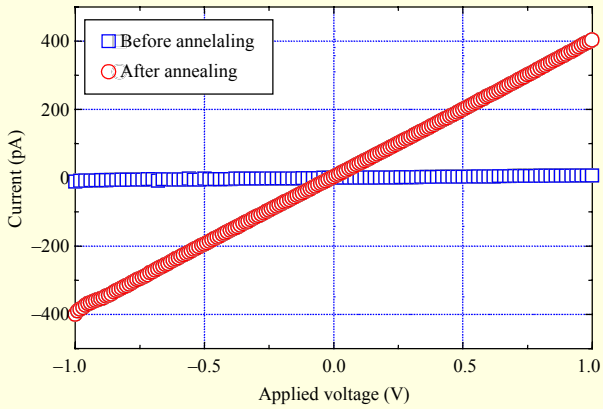

Fig. 3. Measured current-voltage characteristics of gas sensor at room temperature before and after annealing.

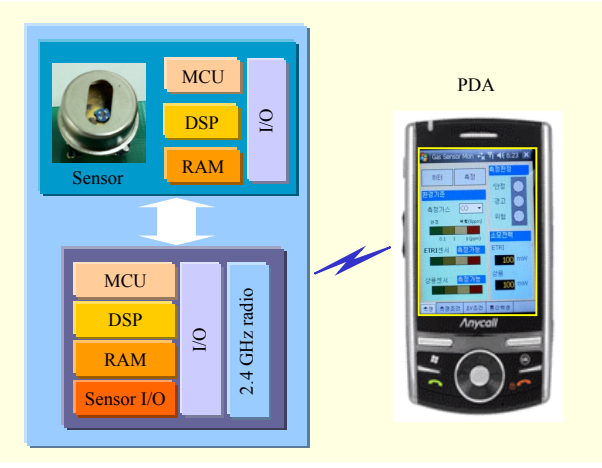

Fig. 4. A schematic diagram of real-time wireless environmental monitoring system including a gas sensor module, Bluetooth module, PDA, and processing program.

2. Wireless System Design and Sensor Integration

A schematic diagram of the real-time environmental monitoring system consists of a sensing unit, communication module, and display module, as shown in Fig. 4. The microprocessor-based sensing unit interfaces with sensors and is capable of acquisition, digitization, and wireless signal transmission using the Bluetooth standard. Due to the necessity for communication of the acquired environmental monitoring results, a PDA phone is adequate for display and communication.

The sensor can be operated using a simple voltage divider in the PDA-based gas sensing measurements. A schematic circuit of the sensor module is shown in Fig. 5.

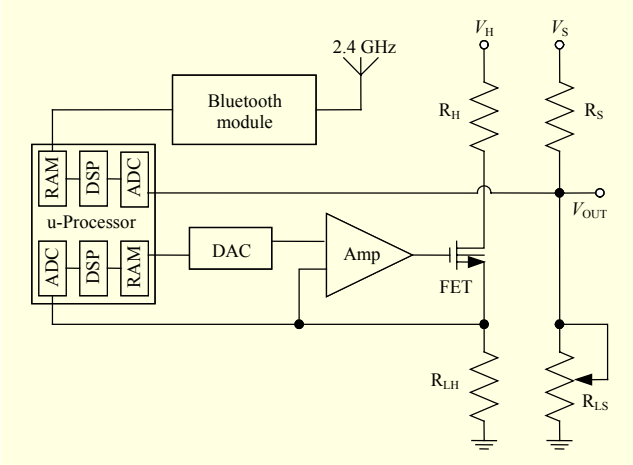

Fig. 5. A schematic circuit for sensor module used to measure gas sensing performance and control operating temperature of developed gas sensor. R_H, R_S, R_{LH}, and R_{LS} are microheater resistance, gas sensor resistance, resistance used to measure current flowing in R_H, and resistance used to measure V_{out}, respectively.

This requires two voltage supplies: microheater voltage, V_H, and circuit voltage, V_S, where V_H is applied to the microheater to maintain a constant, elevated temperature for optimum sensing, and V_S is applied to allow for a measurement of the output voltage, V_{out}, across load resistor R_{LS}.

The change in resistance of the metal oxide nanopowder gas sensor is converted into a change in voltage using signal-conditioning circuits connected to the microprocessor unit. The operating temperature of the developed gas sensor is feedback-controlled using a database that shows the given power-dependent microheater temperature, digital/analog converter, field-effect transistor, and microprocessor. The power consumption can be calculated using the data listed above. In addition, the power consumption of the commercial gas sensor embedded in the wireless environmental monitoring module shown in Fig. 6 can be calculated using the voltage and current recommended in a commercial brochure.

To obtain the sensor responses for detecting a gas exposure, sensor-interrogating circuits are made on a small PCB with a size of about 4 cm × 4 cm, photographs of which are given in Fig. 6, in which the front and back sides of the wireless environmental monitoring subsystem using the metal oxide nanopowder gas sensors are shown.

The program interfaces with a serial Bluetooth device at the host controller interface and universal asynchronous receiver/transmitter level. The software is developed on a Micorsoft.net platform using Visual C#. It also includes a visual interface to plot the real-time data from different sensor channels on the wireless device. It can also log and save the data in memory for further analysis and comparison.

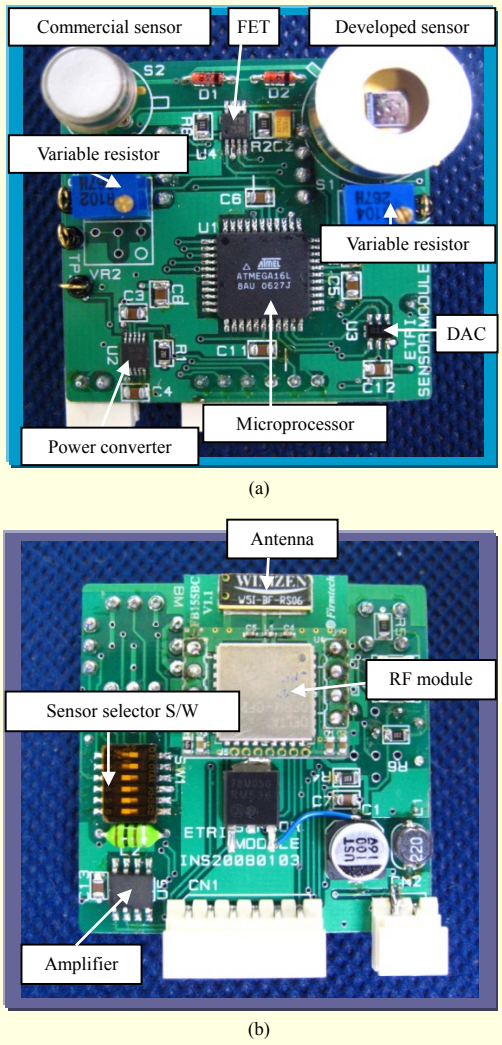

Fig. 6. Photographs of (a) front and (b) back sides of wireless environmental monitoring subsystem using MEMS gas sensor.

III. Results and Discussion

1. Gas Sensor Evaluation

Various gases, such as NO_2 and CO, have been used in chemical sensing studies. The monitoring of these gases in the environment is of paramount importance. As known, chemisorbed gas molecules on a metal oxide surface withdraw or donate electrons to the conduction channel, giving rise to a conductance change.

The test gas is balanced with dry air, and nitrogen dioxide and carbon monoxide gas concentrations are varied from 0.1 ppm to 5 ppm and from 1 ppm to 50 ppm, respectively, with a fixed carrier gas volume. The above metal oxide material (generally n-type semiconductor) devices exposed to oxidizing and deoxidizing gases show decreasing and

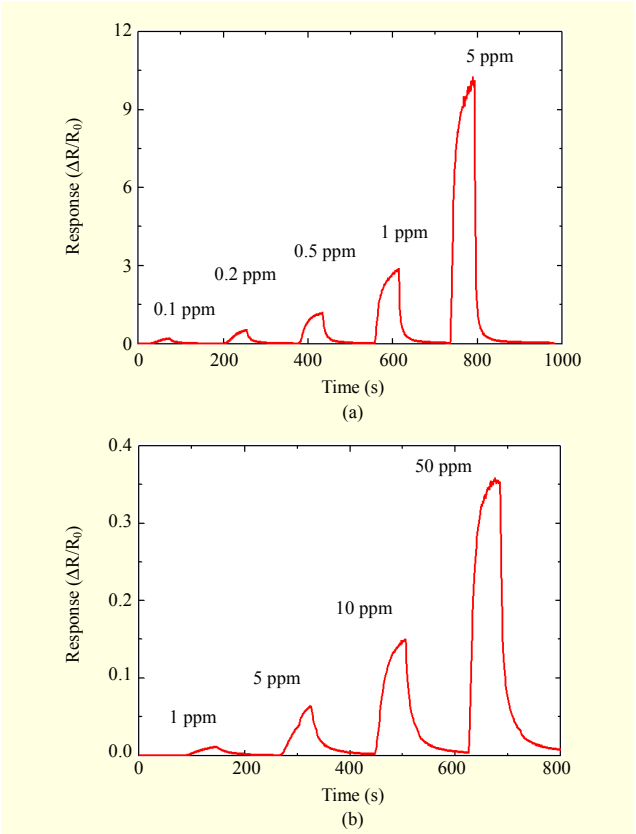

Fig. 7. Gas sensing characteristics of (a) NO_2 gas sensor and (b) CO gas sensor.

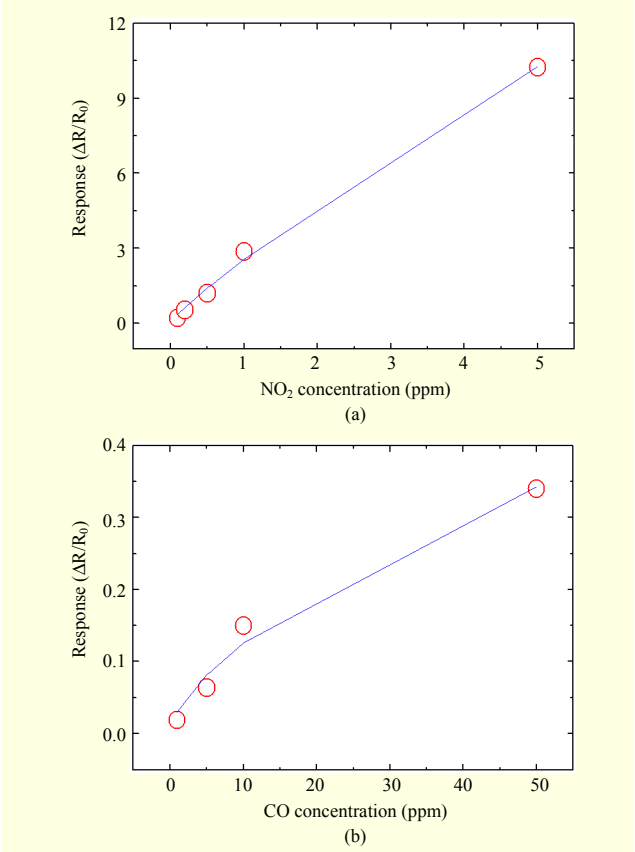

Fig. 8. Gas concentration dependent gas sensing properties for (a) 0.1-ppm to 5-ppm NO_2 gas and (b) 1-ppm to 50-ppm CO gas.

increasing conductance, respectively, as the gas concentrations increase. An operating temperature of about 400ºC is chosen for the gas sensor. The measured nitrogen dioxide and carbon monoxide gas sensing properties are shown in Figs. 7(a) and 7(b), respectively. An increasing curve with respect to the measured time corresponding to the bottom of the graph represents a change in response. For both cases, the gas sensing response increases under a higher concentration. The sensor device shows responses of about 2.9 and 0.15 for 1-ppm NO2 and 10-ppm CO, respectively. In addition, the response and recovery times for a 90% response rate are 38 s and 20 s, respectively, for 1-ppm NO_2, and 39 s and 44 s, respectively, for 10-ppm CO gas. The performance of the above micro gas sensor fulfills the limits for NO_2 and CO pollutants set by the National Ambient Air Quality Standards of the Korean Ministry of Environment, which are 0.1 ppm and 25 ppm [24], respectively.

Despite a variation in base resistance R_0, several forms of empirical phenomenological formulae describe the dependence of the responses of SnO_2-based gas sensors on the concentration of various gases [25]. Under a flat band condition, it is known that the formulae assumes the power law form of $R = K p_g^{\alpha}$, where $R = \Delta R/R_0$, K is a reaction constant, p_g is the gas concentration or partial pressure, and α is an exponent between 0 and 1 [26]. As our sensor, which consists of nanopowders with a thickness of approximately λ_D, may satisfy the flat band condition, it is very likely that the response can be described in the equation above. Figures 8(a) and 8(b) show the dependence of the response on the NO_2 and CO concentrations of the sensor. The data is fitted to the above equation, with $K = 2.6$ and $K = 0.03$ and $\alpha = 0.86$ and $\alpha = 0.62$, respectively, where the value of exponent α differs somewhat according to the nature of the acceptor center [27], [28]. At this high temperature, the gas sensing and desorption are facilitated with an ultra-low power consumption of about 15 mW, which is a benefit of microheater technology. With further optimization efforts, the response and recovery times of our gas sensor can be reduced to much lower levels.

2. Evaluation of PDA-Based Environmental Monitoring Subsystem

The operational performance of the PDA-based environmental monitoring subsystem is evaluated through a response test for CO and NO_2 gases. In the environmental

monitoring subsystem, a commercial metal oxide gas sensor is also integrated, and the performances of the gas sensors, including our own, can therefore be tested simultaneously. For example, the microheater power consumption, minimum gas detection limit, gas response time, and so on can be confirmed through the data display on a PDA panel.

To test the operation of the environmental monitoring subsystem, it is placed in a stainless steel chamber through which the test gas based on air gas with the desired concentration will flow. The response detected in the gas sensor is then converted and delivered to the PDA using the Bluetooth module. All measurements of the gas responses are accomplished twice per second successively by saving the response-time profile data, which is composed of the initial stabilized voltages for several seconds, the transformed voltage variation of the given time exposure to each gas, and a recovery profile for the air gas. The measured voltage variations are converted and displayed as gas concentration in the PDA panel through a comparison with the database stored in the PDA, which shows the relation of voltage variations to the detected gas concentration.

The measured multivariate data proceeds with the methods described above, extracting the detection response for each sensor, one made by us and the other by a commercial product. The photograph in Fig. 9 shows the display contents in the PDA when the sensor is exposed to nitrogen dioxide and carbon monoxide. The type of gas, the power consumption, the concentration of the detected gas, and so on are displayed for both our gas sensor and a commercial gas sensor. Figures 9(a) and 9(b) show the sensing response of the developed NO_2 and CO gas sensors for 0.1-ppm NO_2 gas and 50-ppm CO gas, respectively. In both cases, the gas sensing response of our gas sensor is similar to that of the commercial gas sensor, but the power consumption is lower than that of the commercial one.

The subsystem described above can be applied to real-time environmental monitoring around a user having a ubiquitous terminal companion, a ubiquitous sensor network, a smart home, an intelligent automobile, and so on. As an example, a Bluetooth headset with the developed wireless environmental monitoring subsystem having a metal oxide nanopowder gas sensor is shown in Fig. 10. The detected environmental information, such as the nitrogen dioxide or carbon monoxide gas concentration, is displayed on the PDA panel or delivered to the user.

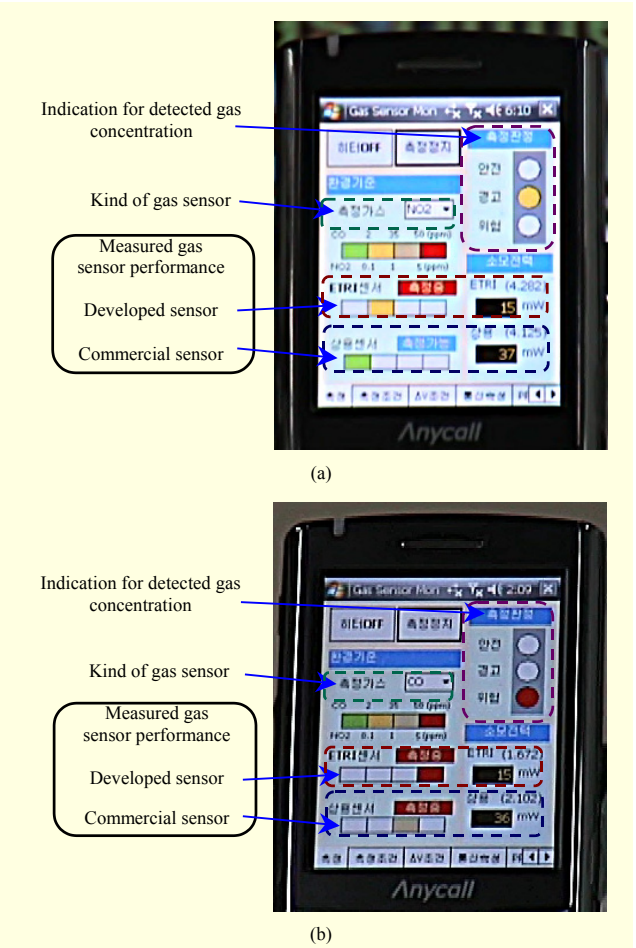

Fig. 9. Photograph of display contents in PDA when sensor is exposed to (a) 0.1-ppm NO_2 and (b) 50-ppm CO, respectively.

Fig. 10. Photograph of Bluetooth headset with wireless environmental monitoring subsystem having MEMS gas sensor.

IV. Conclusion

Metal oxide nanopowder gas sensors for a compact wireless environmental monitoring subsystem were developed and their experiment results evaluated. The responses of a

semiconductor-type MEMS gas sensor for gases nitrogen dioxide and carbon monoxide were about 2.9 and 0.15 for 1-ppm NO_2 and 10-ppm CO, respectively, with a 15-mW power consumption. The subsystem presented in this paper was developed using a gas sensor module, a Bluetooth module, and a PDA phone. The application of this technology can be extended toward the development of accurate and reliable wireless gas sensors for air quality monitors, automobile exhaust monitors, gas leak detectors, and home food spoilage monitors, among others.

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