MOS hydrogen sensor array for 2D gas distribution mapping

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ABSTRACT

A MOS capacitor hydrogen sensor array is reported for hydrogen gas distribution mapping. Si(100) was used as substrate for film deposition. After silicon oxide growing on the silicon surface with dry thermal oxidation, palladium film was sputtered on silicon oxide as hydrogen-sensitive gate material in MOS structure. The 3x3 sensor array was patterned on silicon substrate and packaged in one chip. Sensors' response was test with impedance analyzer. The sensors in array were calibrated in the hydrogen concentration range from 10ppm to 10000ppm. The 2D hydrogen concentration distribution was obtained with the calibrated sensor array.

INTRODUCTION

With the emerging fuel cell technology and hydrogen industry, there rises a need for many kinds of new hydrogen sensor technology specifically for fuel cell applications. Hydrogen sensors are required in varied fuel cell applications for safety, monitor and control. Existing hydrogen sensor technology ranges from ambient atmospheric monitoring devices to safety critical space / aeronautic leak detectors. While at one end of the spectrum, the hydrogen sensor technology is relatively low cost, although usually bulky with only fair performance and reliability; at the other end of the spectrum, the hydrogen sensor technology is much more sensitive and reliable, although extremely expensive. In fuel cell and hydrogen industries, there are still strong, urgent needs for many special sensor and sensor applications. For a viable solution, the hydrogen sensor technology needs to become very reliable and economical in all applications.

Among hydrogen sensor studies, the research on semiconductor hydrogen sensor has attracted wide interesting[1-5], because of their unique features such as small size, low cost and reliability. Varies metal oxidation semiconductor (MOS) device sensors were reported by many research groups. In this study, we developed a metal oxidation semiconductor (MOS) senor array for hydrogen gas distribution sensing. The sensor array comprises 3X3 capacitor hydrogen sensors in one chip, which can provide high spatial resolution for gas distribution. Some applications can be expected such as leak detection and localization, gas mapping and gas flow monitoring. More potential applications are expected.

EXPERIMENTAL PROCESS

Fabrication process

The sensor array was fabricated on n-type (100) silicon wafer with resistivity of 1-10 Ω cm. After RCA clean and deionized water rinse, a near 1000nm field oxide layer (FOX) was grown first, with wet oxidation at 1100°C for 3 hours. This FOX layer surrounds sensor area and penetrates 44% below the original silicon surface to separate sensor elements in array. A plasma enhanced CVD process was employed to deposit a protective silicon nitride layer before the FOX grown, to protect the surface of silicon during the FOX growing. After removing the protective layer and cleaning the silicon surface of sensor area, a thin silicon oxidation layer (270 Å SiO_2) as insulator was grown with dry oxidation process at 1000°C for 25 minutes. In this study, pure palladium was employed as the gate material for MOS fabrication on the silicon oxide layer. The palladium layer of 1000 Å in thickness was deposited with DC sputtering (250W*MIN at 0.2A). For better electric connection in wiring and packaging, a thick palladium pad was deposited prior to the gate deposition. Finally, the 3X3 sensor array was attached die and bond wires on a 24-pin DIP package through normal integration process.

Test setup

To test sensor array for characterization, two kinds of gas environments were employed in measurement. For sensor element response test and calibration, a reliable, stable and accurate gas blending system was built (Figure 1). The gas flow was analyzed with simulation. The parameters that may affect sensor test such as temperature, humidity, and electromagnetic interference were carefully considered. The hydrogen concentration ranges from 10ppm to 10000ppm. This provides a reliable calibration for sensors.

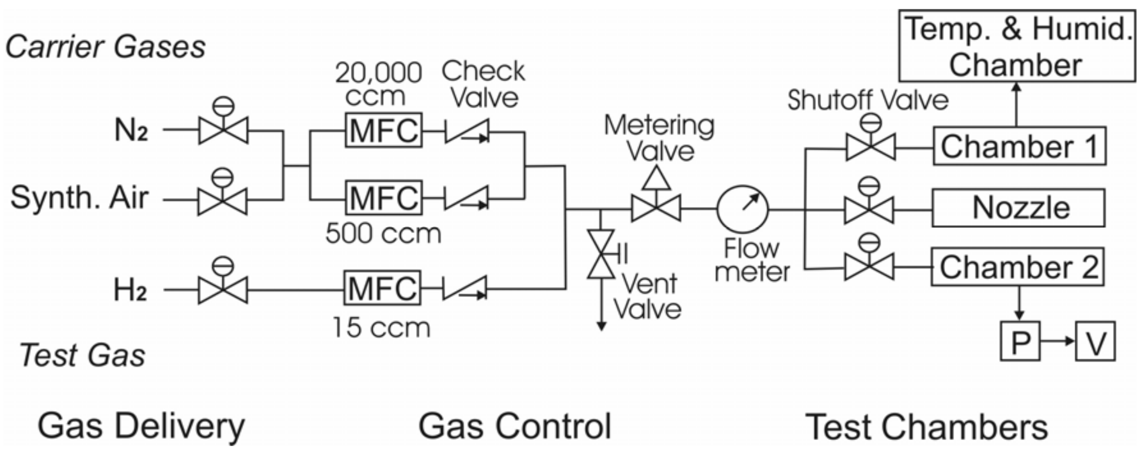

Figure 1 – Schematic diagram illustrating the setup of the gas blending system

As an application for gas distribution mapping, a gas leak environment was established (Figure 2). Pure hydrogen gas flowed from a pinhole and defused freely to open space. To avoid the air convection interference, the test area was blocked in a large scale and remained for more than one hour before test. Sensor array was remote controlled to move smoothly in gas environment without disturbing gas distribution significantly.

A custom multi-channel data acquisition system has been designed and implemented in order to process multi-sensor response data. The system consists of two main parts: a sensor interface including a multiplexer and capacitance-frequency conversion circuitry, and a NI AT-MIO-16E-1 multifunction DAQ module.

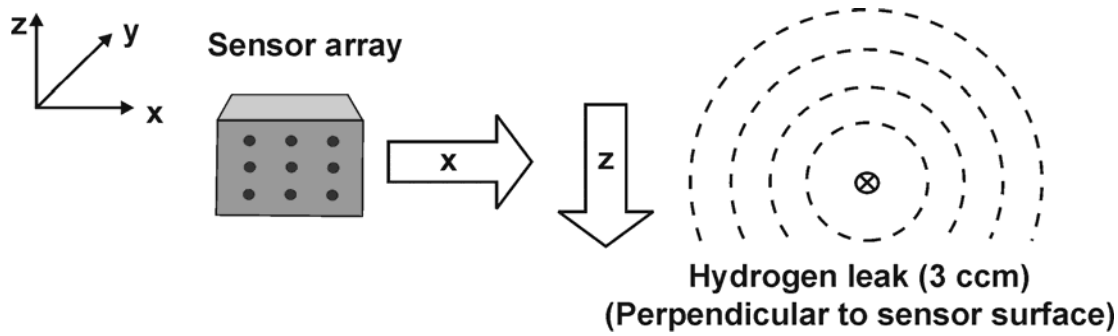

Figure 2 – Illustration of sensor array test in hydrogen gas environment.

RESULTS AND DISCUSSION

C-V measurement (Impedance analysis)

Each sensor in sensor array was characterized with impedance analyzer. Under a series of stable conditions, the sensors' C-V measurement was carried out for calibration. A Solartron SI 1260 Impedance Analyser and a SI 1287 Electrochemical Interface were utilized to make the C-V analysis. This equipment applied AC signal and DC bias voltage to the sensor, swept the bias voltage in a given range (-2V to 2V), and plot out capacitance versus voltage (C-V) curves. These curves show clearly the flat-band voltage change ΔV of the MOS sensor while exposed to hydrogen gas. By selecting the constant bias voltage, the corresponding capacitance change ΔC could be acquired as well. With sweeping time mode, the real-time capacitance (C-T) curves at constant bias were also obtained. Figure 3 shows the responses of a sensor element to hydrogen gas at leak rate of 0ccm, 0.2ccm, and 0.5 ccm, respectively. The 0.2 ccm (cubic centimetre pre minute) is the lowest flow rate that the hydrogen flow meter in present market can achieve. The sensor shows sensitive response at such a low concentration. This high sensitivity provides a high spatial resolution of sensor array.

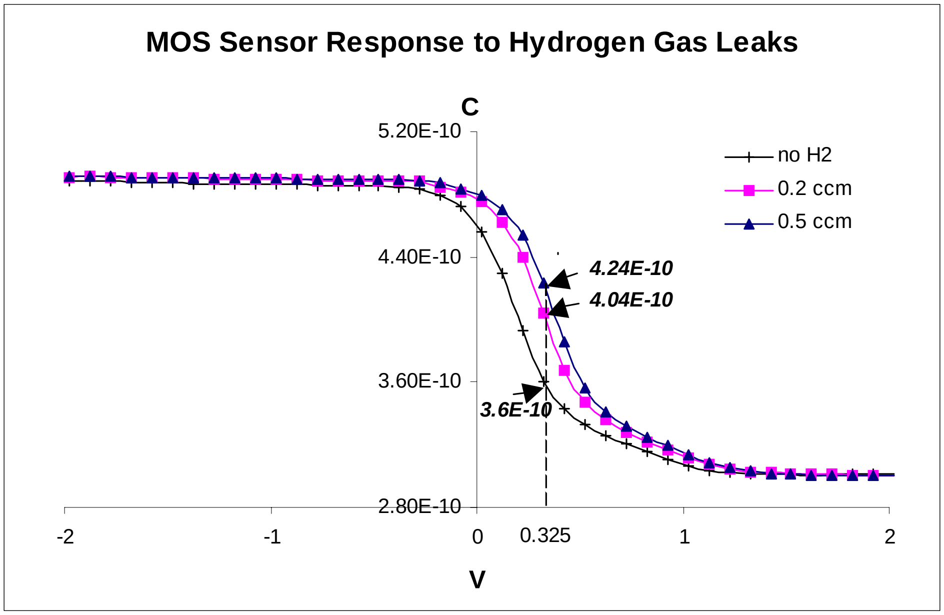

Figure 3 – C-V curve illustrating MOS sensor response to hydrogen gas leakage

Calibration

Because of the variation between sensor elements in array, the calibration is necessary to ensure all sensors in array have identical response in same conditions. In this study, we achieved the calibration in a series steps: first, to set a series of test conditions for all sensor test. Since sensor is very sensitive to many environment parameters such as temperature, humidity and pressure, it should be much careful to keep all sensors test in same conditions. Second, based on the sensor response data, to induce a suitable mathematic equation to figure out sensor's response. Theoretically, infinite complex solution can be obtained with varied curve fitting methods. We undertook the study with several considerations: 1. The identity for all sensors; 2. The concordance both in mathematics and physics. The mathematical equation should have apparent physical explanation. 3. The accuracy in the whole test range. Finally, with the obtained sensor response equation, all sensors' response could be calibrated identically and linearly. As a result, we finally obtained a first-order logarithmic function for sensor response.

$$f\,(x_n,\, a_{n)} = \; a_0 + a_1 \ln\,(x + 1)$$

where a_0 and a_1 are the intrinsic constant depending on sensor. Figure 4 shows a curve fitting result for a sensor element. The good concordance ensure us the calibration should be reliable.

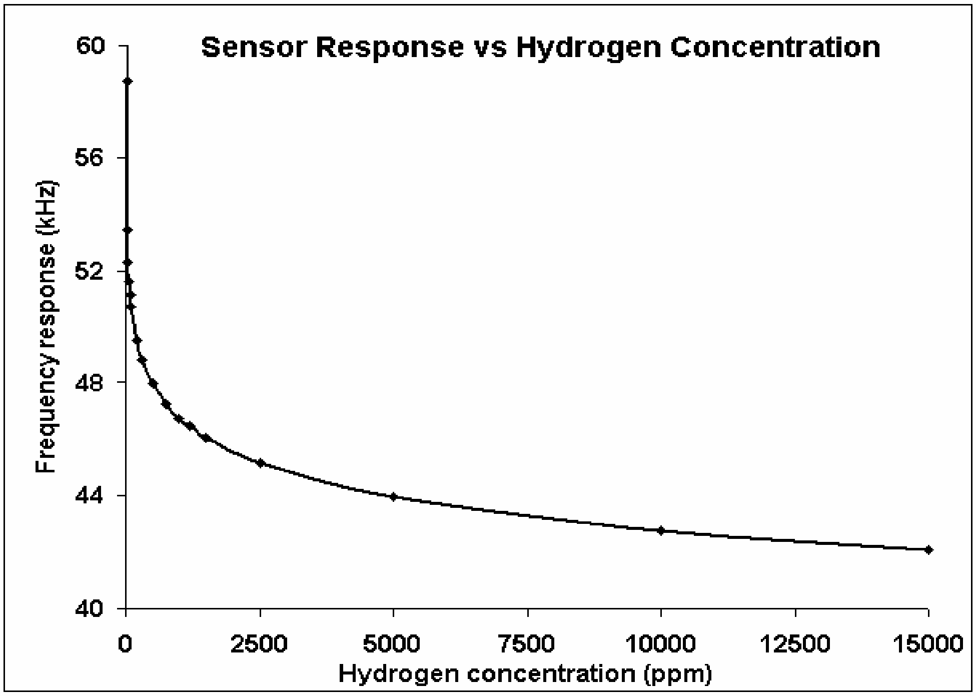

Figure 4 – Sensor response curve using a linear scale

Gas mapping

As an application of hydrogen sensor array, a hydrogen distribution was mapped by move sensor array scanning the whole test area. Pure hydrogen was flow out from a pinhole at 3ccm rate, and diffused in open space freely, as show in Figure 2. The sensor array was remote controlled to move step by step across the test area in horizontal and vertical directions, respectively. Each step obtained 9 points (3X3) of gas concentration. The gas distribution was mapped by conjoining all those images together.

Measurements have been taken at each step. Figure 5 illustrates the gas distribution contour (top view). In total, 225 (15 × 15) sensor response data are recorded. It is observed that the peak hydrogen concentration (hydrogen concentration between 1600-2000 ppm) is near the centre of the monitoring area, which is corresponding to where the leak point is located, and that the concentrations decrease at approximately same slope in each direction.

The spatial resolution of gas mapping depends on the density of sensor in array. Technically, sensors can be fabricated in a distance less than 1 mm in array without difficulty. Our observation also have the record of 1mm spatial resolution for gas distribution. However, it also depends on the accuracy of sensor response and calibration.

As an application of gas mapping, the sensor array can be used for leak location search. As shown in Fig 6, the local gas distribution gradient can be analysed after the mapping. Then the direction of higher concentration can be acquired. By moving the sensor array along the analysed direction step by step (as shown the step 1, 2, … 5), the location of gas leak (which has the highest concentration read) can be approached efficiently.

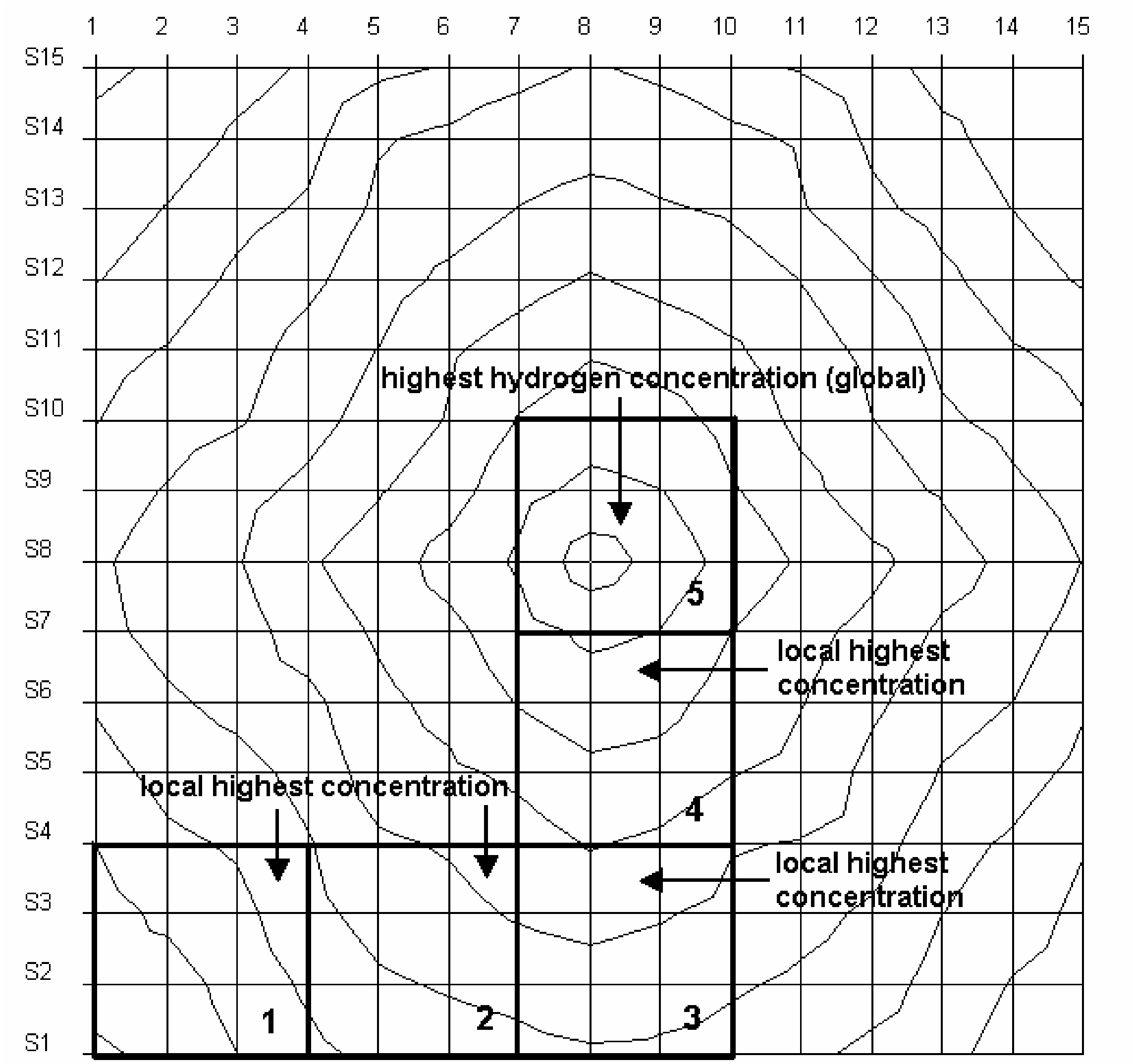

Figure 5 – Hydrogen gas distribution mapped by sensor array

CONCLUSIONS

Based on the semiconductor technology, a MOS capacitor hydrogen sensor array has been developed. Each sensor in array was characterized with impedance analysis and calibrated. The sensor array shows good concentration response and high spatial resolution for concentration mapping. As an application of sensor array, a leak location search method was illustrated with the concentration map.

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