

Development of a Novel High Reliable Si-Based Trace Humidity Sensor Array for Aerospace and Process Industry

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Abstract: In this paper we present high reliable and accurate silicon-based trace humidity sensors for use in aerospace and process industry. The sensors have been realized by using simple MEMS technology in this work. One is a single sensor (sensor cell) for monitoring of humidity, and sensor array is able to measure the total trace humidity in the atmosphere. It's very suitable for aerospace and process industry such pharmacy applications.

Keywords: MEMS, Micro sensors, Humidity sensor.

1. Introduction

Pressure, temperature and humidity are the three most measured physical properties. The question of moisture determination and control is an important one in many scientific and industrial disciplines. Trace humidity sensors are needed in many applications, such as Mars mission, semiconductor technologies, pharmaceutical processes.

The coulometric sensor principle [1] is normally used in trace humidity sensing (Fig. 1). This is one of the best techniques available for measuring water content of gases at very low concentration. Many liquids can also be analyzed by this technique after vaporization.

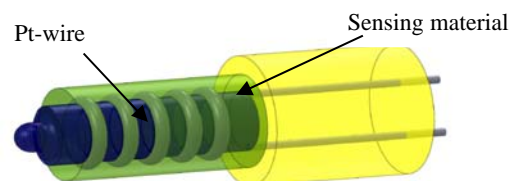


Fig. 1. Schematic view of a sensor on the market on glass carrier with Pt-wire and sensing material.

Other methods are generally unsuccessful when water concentrations to be measured are under a few thousand parts per millions. The common sensor concept is using glass carrier with Pt-wire and sensing material (phosphoric pentoxide P_2O_5). It is based on

the absorption of water vapour from the atmosphere by the highly hygroscopic phosphoric pentoxide (P_2O_5), which is located on the sensor between platinum electrodes. Realized sensors using glass carrier and thick film technology are robust but bulky and have some limitations such as reliability [1]. Another disadvantage of the concept is that – just one part of the water can be absorbed and electrolyzed.

2. Sensor Concept, Design and Technology

The single sensor concept is shown Fig. 2. The sensing Pt-electrode is deposited on a 3D silicon surface etched in silicon substrate in KOH-solution. The idea behind it is to increase the active surface of the sensing electrode in compare to a planar concept. The other advantage of this 3D concept is to use the capillary force to get the sensing material into the silicon trenches. The very smooth trench surface is very suitable for a thin high quality Pt electrode. The Pt layer shows no defects and non-perfections such as in standard sensors. Main disadvantage of this single sensor cell concept is the nonability to have exact information about the total humidity in the air like the concept in Fig. 1. Just one part of the water in the air can be absorbed and converted into charges. Sensors on the market need therefore a calibration. Fig. 3 shows the array concept. The sensor surfaces are exposed to the air flow. By adjusting the sensors voltages the signal of the last sensor can be adjusted toward zero – it means that the humidity in air is fully absorbed by the sensors. So it's very easy to measure the absolute humidity in the air without calibration. Another advantage of the array concept is a better redundancy.

The designed single sensor (sensor cell) has a size of $11.5\text{ mm} \times 20\text{ mm} \times 525\text{ }\mu\text{m}$, and the sensor array has dimensions of $60\text{ mm} \times 20\text{ mm} \times 525\text{ }\mu\text{m}$ [2-3]. The manufacturing technology is very simple using standard silicon MEMS processes. First the substrate is passivated by using double layers SiN and SiO. The thicknesses of SiO and SiN layer are 100 nm and 180 nm respectively. After that the double layer is structured as etching mask for following KOH etching (33 % KOH solution, 80°C). The etching rate is constant at $1\text{ }\mu\text{m}/\text{min}$. After KOH etching the mask is remove. Before Pt deposition by using sputtering a thin SiO was grown by using thermal oxidation. Purpose of this SiO layer is an isolation of Pt layer. Spray coating is used to make a lift-off mask for Pt deposition. After Pt sputtering the resist layer is removed.

The chemical reaction that takes place is an equilibrium process (Equation (1) and (2)).

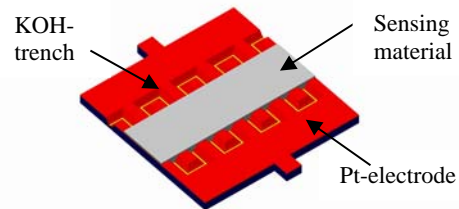
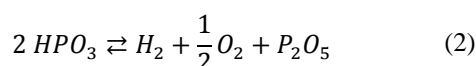


Fig. 2: Schematic view – single silicon-based humidity sensor in this work.

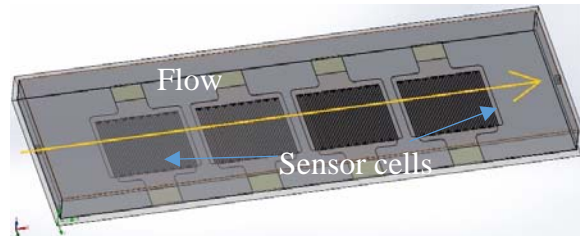


Fig. 3. Schematic design of the sensor array containing 4 single sensor cells for measurement of absolute humidity.

According to Faraday's law of electrolysis, 0.5 g/mole of water (9.01 g) requires 95.500 C of electricity for its electrolytic decomposition. The electrolysis current is proportional to the number of moles of water absorbed per unit time. Thus, measurement of water vapor mass is traced back to an electric current measurement [1]:

$$Q = I\Delta t = Fa = F \frac{m_{water}}{M_{water}} z, \quad (3)$$

where Q is the electric charge, I is the electric current, a is the absolute humidity, z is the number of exchange electrons, F is the Faraday constant 96484 As/mol, m_{water} is the mass of absorbed water vapor, M_{water} is the molar mass of water vapor.

The sensor current (charges) can be calculated by using the simple equation:

$$I = 1.0712104 * a * \dot{V}, \quad (4)$$

where a is the absolute humidity, \dot{V} is the gas flow.

A single measurement cell with a length of several meters would be necessary to achieve complete electrolysis of water in the carrying gas. Realized sensors have a conversion rate of $>30\%$. Normally calibration of these sensors is necessary because less than 100 % of the water vapor present take part in the reaction. In our array concept a calibration is not needed. The total amount of water in the air is the sum of the currents of all single sensor cells. Fig. 2 shows a concept with 4 sensor cells. One easy indicator for total reaction of water is the zero current in the last sensor cell in flow direction.

The sensor cells in this work are realized on a 3D surface etched in silicon substrate by using KOH. Such surface is much larger than 2D sensor surface typically

realized in thick film technology. The capillary effect on such 3D surface is also high which suitable to improve adhesion and contact between Pt- and P_2O_5 coating [2-3].

Another advantage of this concept is the better redundancy. Fail of one or more sensor cells do not affect the whole system function. The wearout of material on the first sensor is normally higher than on the other sensors. According to the absorbed mass of water from the air.

3. Fabrication

The sensor cells are fabricated by using standard silicon technology. First the alignment marks were etched in (100) silicon wafer. In the next step the deep trenches (100 μm) were etched by using KOH. After KOH etching the surface was passivated by a thick SiO_2 layer. For lift-off a spray coating for photo resist was necessary. Pt layer of 100 nm was sputtered. Fig. 4 shows briefly process steps for fabrication of the sensor array in silicon.

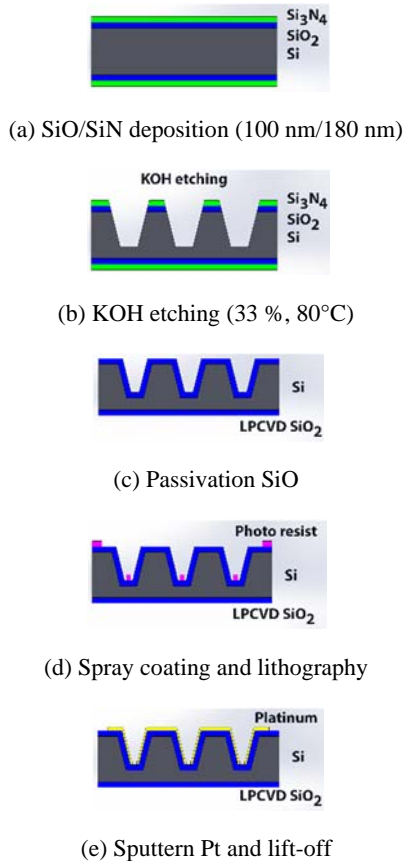


Fig. 4. Main process steps to fabricate the sensor array [3].

4. Characterization

Fig. 5 shows the top view of a realized sensor array with KOH-trenches and Pt-electrodes on top.

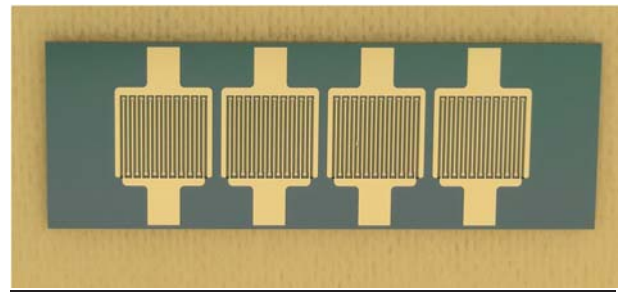


Fig. 5. Top view of the realized silicon-based sensor array without sensing material on top (60 mm \times 20 mm \times 525 μm).

Tests have been done to characterize the realized sensors. A special package has been realized to meet the requirements for monitoring of humidity [2]. In figure below the design of the package is shown. The design has totally four level. The first and second levels are the base plate and substrate to provide the stability and the sealing for the sensors. The third layer is a sealing membrane to create a hermetic in the flow channel over the sensor surface. The top level is a massive part over the sensor with the integrated channel. This part contains the connectors for supply gas flow and the pins for electrical contacts/interface. The design allows a laminar flow directly over the sensor surface. The Au pins are sealed and provide an easy electrical contact for sensor signal. The connector pins are equipped with springs to ensure an electrical connection at all times without damaging the sensors. Due to requirement for chemical resistance, the materials have to be chemically stable. The gas connection is created via stainless steel ports on the lateral edges of the structured plate. The structured plate is made of Polyethylene, the base substrate is made of FR-4 glass epoxy and the sealing layer is made of Viton. The realized system can be seen in Fig. 6. The system has a total size of 96 \times 50 \times 25 mm³ [2].

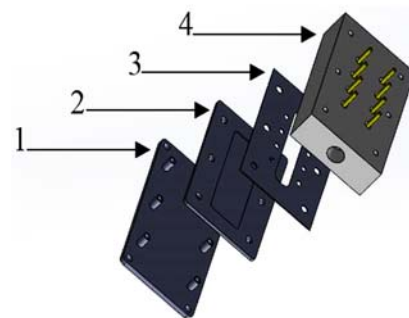


Fig. 6. Exploded view of the realized sensor package: (1) base plate; (2) base substrate; (3) sealing layer, and (4) plate with integrated flow path.

The realized sensor system is shown in Fig. 7.

The principle measurement set up in this work can be seen in Fig. 8.

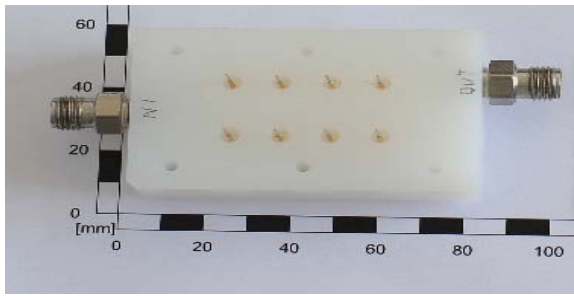


Fig. 7. Package for the sensor array.

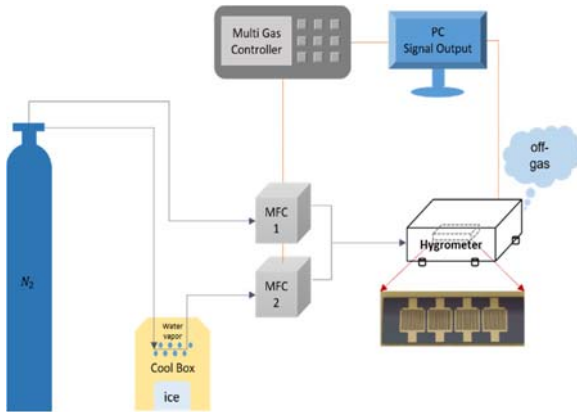


Fig. 8. Principle setup for characterization of the realized sensor arrays.

For the characterization nitrogen gas (N_2) with 99,999 % purity is used as gas supply based on its ideal behavior which follows Dalton's law of partial pressure at standard atmosphere. The dry nitrogen gas flow through Teflon tubing in two directions separately. One of them flow through the cool box with ice, which keep the condition of low humidity approx. 1000 ppm. In this case the humidity concentration can be calculated according to DIN 4108 [4]. The reference flow is mixed with dry air to create an air steam with specific humidity concentration for the characterization. The Fig. 9 shows the used set up.

The principle of generation of dew point in cool box is described in DIN 4108. For an ideal gas ($pV = nRT$) in thermal equilibrium is it possible to generate a reference gas steam with a dew point of about $t \sim -20^\circ C$ and a humidity concentration of 1000 ppm, as shown in Equation (5).

$$P_s [Pa] = 4.689 [Pa] \cdot \left(1.486 + \frac{t}{100^\circ C} \right)^{12.30} \quad (5)$$

$$(-20^\circ C < t < 0^\circ C, s. t. p.) \quad (6)$$

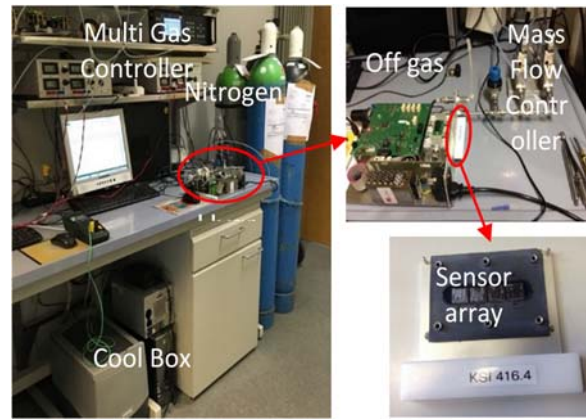


Fig. 9. Measurement setup for the sensor array.

Based on the Dalton's law of partial pressures shown in Equation (7), the reference gas flow is then mixed with dry air to create a gas flow with a specific humidity concentration. It is well known that the total pressure of a mixed gas is the sum of partial pressure of the individual gases.

$$P_{total} = P_1 + P_2 + \dots + P_n \quad (7)$$

If the mixture of gases containing water vapor, the total pressure is equal to the sum of the partial pressure of the dry gas and the partial pressure of the water vapor. The Equation (7) can be rewritten as:

$$P_{total} = P_d + P_w \quad (8)$$

The relationship between the total pressure (P_t) and the partial pressure of water vapor (P_w) can be expressed as follows [1]:

$$P_{t1}/P_{t2} = P_{w1}/P_{w2} \quad (9)$$

The quantity of any gas in a mixture can be expressed as a pressure. Controlling the gas pressure and temperature (cool box) is an easy way to control the water content. With the help of compressed air, the existing dew point can be converted to the dew point under atmospheric pressure – according to dew point diagram below (Fig. 10). It can be clearly seen in Fig. 10, for a temperature (cool box) of $-2^\circ C$ and a pressure of 4,2 bar a dew point of $-20^\circ C$ is achieved.

The ratio of dry gas and moist gas is well-controlled by using multi gas controller and mass flow controller. The mixture of gases flow over the sensor array, which was installed in hygrometer, to participate in chemical reaction. Therefore, the performance of sensor array with implementation from quantity of water vapor can be tested and determined.

The Table 1 shows the possible parameters set in this work.

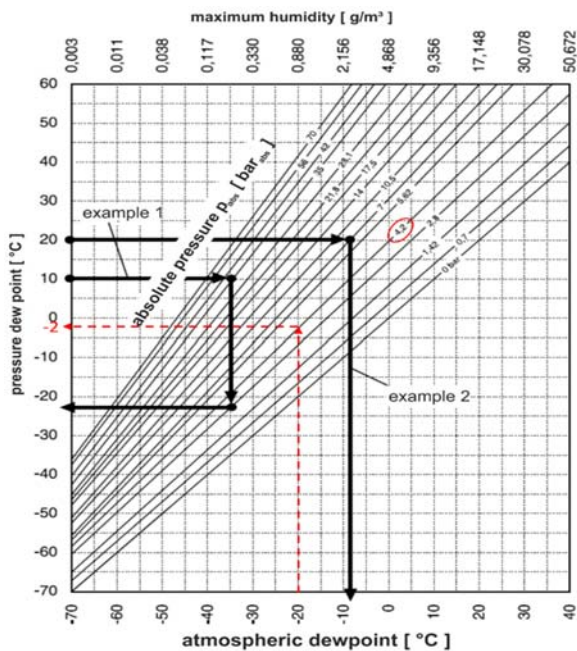


Fig. 10. Conversion diagram of dew point [1, 4].

Table 1. Parameters used in the characterization of the realized sensor arrays.

Parameter	Value
Humidity measuring range	10-1000 ppm _v
Dew point range	-60 ~ -20 °C
Ambient pressure	Approx. 1000 mbar
Ambient temperature	24-29 °C
Flow rate	20-150 ml/min
Mixture of gas	99,999 % N ₂ H ₂ O < 2 ppm/mol
Cool box temperature	ca. -2 °C
Operating voltage	40 V

Coating of Sensor Material

The coating solution used in this work was phosphoric acid H₃PO₄ with 85 % concentration. For the characterization an amount of 0.5 μL phosphoric acid was dosed on each sensor cell. The sensor cells with coating were then cured at 50 °C for 30 min. Fig. 11 shows sensor array with coating after curing. In order to get the sensing materials better into the trenches a wetting step in O₂-plasma (5 min) was conducted prio coating.

For the characterization a flow rate of 50 ml/min was used. The electrical voltage was 40 V. Theory and measurement results are shown in Fig. 12. The electrolytic current behavior was a linear function of absolute humidity. At low absolute humidity till approx. 100 ppm, the agreement between theory and test results was excellent. Increasing the absolute humidity increases the difference of experimental current from theoretical current, which in accord with faraday's law. However, the range of deviation is acceptable. Discrepancy can be explained in the non-perfect setup.

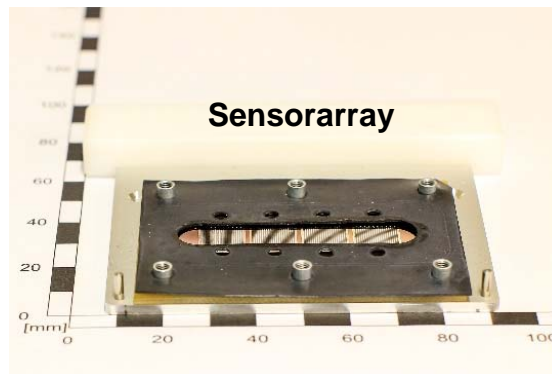


Fig. 11. Sensor array with coating ready for measurement.

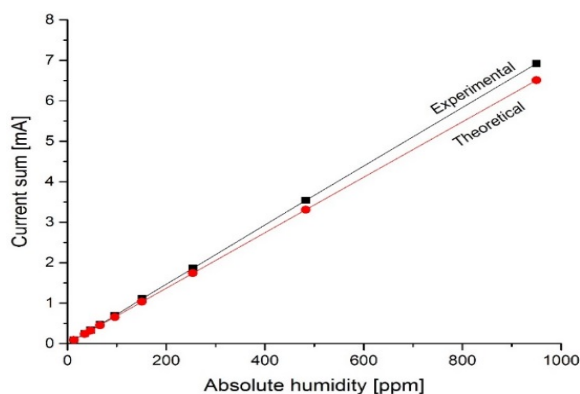


Fig. 12. Comparison of results of test and theory.

Fig. 13 shows the individual currents of individual sensor cells as functions of humidity. The current of the first sensor cell is the highest due to the higher absorbed humidity. The current of the last sensor cell is low indicating a very low humidity in the air flow above the last sensor cell. It shows that a complete electrolysis of water in the air is possible with the sensor array. Same effect could be achieved by adjusting the voltages for the individual sensor cells.

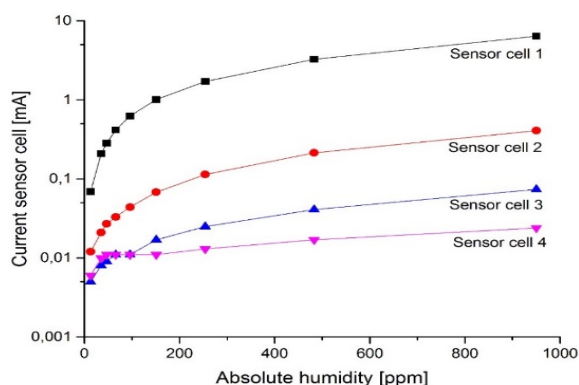


Fig. 13. Signals of individual sensor cells.

Many sensor arrays have been characterized in order to test the repeatability and robustness of the

technology. The Fig. 14 shows the output signals of three sensors as function of humidity. It shows a good repeatability.

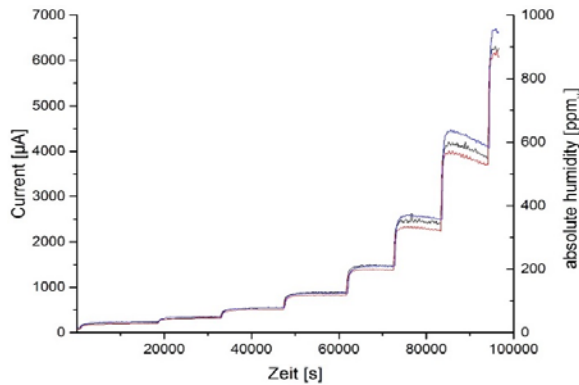


Fig. 14. Output signals of three sensors as function of humidity concentration up to 1000 ppm.

4. Conclusions

We present a novel reliable silicon-based sensor solutions for trace humidity. The single sensor cell can be used to monitor trace humidity as well as the sensor array. The sensor array can be used to monitor absolute water content in the air without calibration. The measurement results are verified and show quite good agreement with theory. The sensor package has been optimized by using simulation. The realized

prototype has a geometry of $96 \times 50 \times 25 \text{ cm}^3$. The simulation results show some limitation within the package. Improvements have been made to get better flow behavior over first sensor cell by modifying the flow path. The long flow path could be integrated in the package and can avoid backlash from air surrounded.

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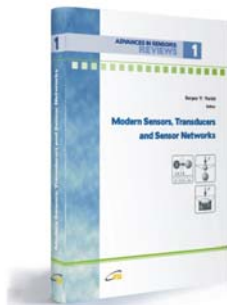


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