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Fabrication and Characterization of Thin Film Humidity Sensor Using Hydrogenated Carbon Containing Oxygen

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This paper presents the fabrication process and the characteristics of a capacitive humidity sensor using hydrogenated carbon containing oxygen. The sensing film of the sensor has been deposited by chemical vapor deposition. The response time of the sensors due to humidity change is within 40 seconds, and also the width of hysteresis ±1.5%RH at maximum. The sensor works reproducibly against humidity and temperature cycles (0%RH@90%RH, 30°C@60°C). Durability of the sensor in other various environments is also investigated.

Introduction

The development of a humidity sensor which works reproducibly in various circumstances is desired. Humidity sensors are classified into several types by detecting method as follows; (1)Electrical property change(1,2) (2)Mass change(3)

(3) Infrared absorption change (4) (4)Mechanical property change (5). Especially the humidity sensor using the change of electrical properties due to absorption and desorption of water molecules is useful for humidity controlling in industry.

The humidity sensor using inorganic material such as $Al_{20_3}^{(1,2)}$ works even in a high temperature region, but it shows relatively large hysteresis in electrical signal to relative humidity(RH). The sensor using organic material shows good linearity in electrical signal to RH, but the characteristics change and degenerate irreversibly after keeping in a high humidity region.

This paper presents the fabrication process and the characteristics of a capacitive humidity sensor using hydrogenated carbon film containing oxygen.

The characteristics of a hygrometer using the humidity sensor fabricated in this experiment is also described briefly later.

Structure of The Sensor

The structure of the sensor fab-

ricated is shown in Fig.1 schematically. Two lower electrodes (electrode A and B) and the upper electrode (electrode C) make two capacitors, and these capacitors are connected in series. The lead wires are connected to the electrode A and B.

All electrodes are made of chromium, and the electrode C has permeability to water molecule. The hydrogenated carbon film is deposited over the electrode A and B.



Fig.1 Structure of the sensor.

Moisture Sensing Part of The Sensor

Characteristics required for the humidity sensor are as follows;

- (1) quick response
- (2) hysteresis free
- (3) good durability.

To realize the quick response, it is necessary to absorb and desorb water

molecules quickly into/from whole of the sensing part. To reduce the hysteresis in electrical signal to RH, it is important to avoid condensation of water molecules. To satisfy the request it is necessary to control the balance of hydrophobic radicals and hydrophilic radicals. And for good durability, it is necessary to use the material which is durable chemically and mechanically⁽⁶⁾.

From these considerations we select a hydrogenated amorphous carbon film containing oxygen for a sensing part of a humidity sensor. Hydrogenated carbon films are de-

Hydrogenated carbon films are deposited by radio frequency plasma enhanced chemical vapor deposition on a sintered alumina substrate where the lower electrode A and B are previously

formed.

The experimental apparatus for deposition of the hydrogenated carbon film is shown in Fig.2 schematically. The facing electrodes for discharge are set in the center of the vacuum chamber, and a substrate $70X70 \text{ mm}^2$ in size is fixed on the upper electrode. After the substrate is exposed to ar-gon plasma for 30 minutes at 10^{-2} Torr, the chamber is evacuated to 10^{-6} Torr. Then amyl alcohol is introduced, and RF power is supplied to start glow The ratio of hydrophobic discharge. radicals to hydrophilic radicals in the film can be controlled by changing the RF power and the gas flow rate. And the film thickness also can be controlled by deposition time. Typical experimental condition for the film growth is listed in Table 1.

Measurements

The measurements of capacitance (C)-relative humidity (RH) and output voltage of the hygrometer (V_{out})-RH have been done. The measurements have been carried out in the temperature range from 30°C to 60°C and also in the range from 0%RH to 95%RH using a divided flow humidity generator (SHINEI SRH-1R135). For the measurement of capacitance at 1MHz and 1V, an LCR meter (HP MODEL 4275A) is used.

Results and Discussions

C-RH characteristics of the sensor at 30°C and 60°C are shown in Fig. 3. The capacitance increases almost linearly with increasing RH in the range from 5%RH to 95%RH. It is observed that the capacitance depends on temperature, and the hysteresis is about ± 1.5 %RH. It is found that the hysteresis is smaller than that of the sensor using inorganic material. From these results it is considered that water molecules absorbed into the sensing part are scarcely condensed, and some part of the water molecules are ionized⁽⁷⁾.



Fig.2 Experimental apparatus preparing the hydrogenated carbon film containing oxygen.

Table 1 Typical experimental	condi-
tion for hydrogenated carbon	film
containing oxygen growth.	

Reaction time	4hours
R.F. power	8014
Gas flow rate	45ccm
Pressure	4 10 ⁻¹ Torr
Reaction gas	C5H11OH



Fig.3 C-RH characteristics of the humidity sensor.

Typical time responses of the sensor at 30°C for increasing (30%RH) 90%RH) and decreasing (90%RH)30%RH) humidity at each 10000cc/min and 2000 cc/min ventilation rate are shown in Fig.4. Full response is indicated by the 100% line for increasing and decreasing humidity.

In the case of 10000cc/min ventilation rate, it takes 6 minutes and 7.5 minutes for 95% response in increasing and decreasing humidity, respectively. Decreasing the ventilation rate to 2000cc/min the response duration time increases about 2.5 times compared to that of 10000cc/min. It is found that the response duration time is influenced by ventilation rate, and the response duration time in decreasing humidity is about 1.4 times longer than that in increasing humidity. If the sensor in an evacuated chamber (0%RH) is quickly taken out to a humid circumstance (70% RH), duration time for full response is about 30 seconds. It is considered that duration time for full response for step humidity decreasing is about 40 seconds.

The capacitances of the sensors



time (min.) Fig.4 Time response of the sensor at 30°C for increasing and

decreasing humidity at 2000cc/min. and 10000cc/min. ventilations.





Fig.5 Long-term stability of the sensor against humidity (0RH-95RH) and temperature cycles (30°C-60°C).

condition of the test	difference of RH-V _{out} (at 30°C)
atmosphere of aqueous solution of toluene, xylene and benzene at 20 °C. (10:3:4:3, 80 hours)	not observed
atmosphere of aqueous solution of C ₂ H ₅ OH at 20 °C. (20wt%, 80 hours)	increasing of V _{out} corresponds to 2~3%RH is observed.
saturated water vapor at 95°C. (20 hours)	decreasing of V _{out} corresponds to 1~2%RH is observed.
saturated water vapor at -18°C. (20 hours)	not observed
dry air at 100°C. (100 hours)	not observed
dry air at -18°C. (20 hours)	not observed
atmosphere of NaOH solution at 20°C. (10wt%, 20 hours)	not observed
atmosphere of H_2SO_4 solution at 20 $^{\circ}C$. (10 \pm t%, 10 hours)	increasing of V _{out} corresponds to 1~3%RH is observed.

Table 2 Experimental conditions and results for durability test of the humidity sensor.

due to humidity changes at 30° C are plotted against time in Fig.5. In these measurements, the sensor experiences a humidity cycle (0%RH Θ 90%RH) at 60° C between each measurement at 30° C. A small decrease of capacitance corresponds to about 3%RH is observed at 90%RH line within first 50 days. But at any other region the sensor works reproducibly in ±1 %RH accuracy. It is found that the sensors work in stable state against humidity and temperature cycles.

The durability of the sensor in various environments are tested. Before and after the durability tests V_{out} -RH characteristics at 30°C are measured and compared. Table 2. shows the test conditions and the results. It is found that the humidity sensor works in \pm 3% accuracy after it has been kept in various environments.

Conclusion

 Full response duration time of the capacitance change against step humidity change is about 40 seconds.
Hysteresis in capacitance to RH is estimated to be ±1.5%RH.
The humidity sensor using hydrogenated carbon film containing oxygen works reproducibly against humidity

(0%RH\$95%RH) and temperature (30°C 60°C) cycles for 150 days.

4 The sensor does not show any degeneration in characteristics even after experiencing various durability tests. References

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