HUMIDITY MICROSENSOR WITH POLYIMIDE SENSITIVE LAYER

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A new humidity microsensor has been developed and fabricated by a microelectromechanical system (MEMS) technology and thin-film technology, using Polyimide thin film. The way of sensing is in connection with the adsorption of the humidity in the volume of the sensitive layer, changing the capacitance of the structure. The sensitive layer has been deposited by vacuum deposition of Polyimide material, with following thermal treatment. These devices have been developed, manufactured and tested. The estimation procedure is performed by specially built test camera, giving the possibility to create different humidity levels. The results from the humidity measurements are presented. The capacitances, sensitive to changes of relative humidity (RH), give the possibility to measure this parameter with a sensitivity of 40-percent change of capacitance, corresponding to humidity changes. The capacitance-humidity characteristics for various relative humidity are presented.

Keywords: Humidity Microsensor, Polyimide, MEMS, Humidity measurements

1. INTRODUCTION

In our everyday life, in different areas, for several applications, we use sensing devices, capable to measure and control the water content in air, gases, liquids etc. There are different techniques and types of humidity sensors, depending on the type of sensing material, the method of humidity measurement, the construction of the sensor and its area of application.

First of all, we have to mention different ways of estimation of the water vapor content in the gas. These are Absolute Humidity, Dew Point and Relative Humidity. The Absolute Humidity is the ratio of the mass of water vapor to the volume of air or gas. It is commonly expressed in grams per cubic meter. The Dew Point, expressed in °C or °F, is the temperature and pressure, at which a gas begins to condense into a liquid. Relative Humidity (RH), refers to the ratio (stated as a percent) of the moisture content of air, compared to the saturated moisture level at the same temperature and pressure. Depending on the case, some or several of these parameters are controlled and measured.

According to the method of measurement, we can mention four big groups of hygrometers, which are shown in Table 1, with their working principles, advantages, disadvantages and problems.

Rapid advancements in semiconductor technology, such as thin film deposition, ion sputtering and using of different materials, have made possible highly accurate humidity sensors with resistance to chemicals and physical contaminants, at economical prices. No single sensor, however, can satisfy every application.

Table 1

Hygrometer	Principle	Advantage	Disadvantage and Problem
Hygrometers using hygroscopic materials	Mechanical property change (length, volume, stress)	No power requirement Low sensitivity to temperature Inexpensive Simple	Non-linear output Hysteresis Drift over time
	Electrical property change (resistivity, capacitance, frequency)	Can be mass produced Simple Inexpensive Small Easy to maintain	Hysteresis Sensitive to contamination
Psychrometer	Relative humidity estimation based on dry- and wet-bulb temperature measurements	No requirement of calibration	Requirement of regular replacement of wick and distilled water requirement of air-flow with high flow rate (3 m/sec)
Dew-point hygrometer	Measurements of dew-point temperature by detecting dew formation on a cooler base	High accuracy Wide dynamic range No requirement of calibration	Large size Expensive Large power consumption Regular cleaning of mirror surface
Infrared hygrometer	Selective absorption of distinctive infrared spectrum by water vapor	Can be used with corrosive gases Wide dynamic range	Expensive Possibility of interference with other gas species

Resistive, capacitive, and thermal conductivity sensing technologies each offer distinct advantages. Resistive sensors are interchangeable, usable for remote locations, and cost effective. Capacitive sensors provide wide relative humidity (RH) range and condensation tolerance, and, if laser trimmed, are also interchangeable. Thermal conductivity sensors perform well in corrosive environments and at high temperatures. For most applications, therefore, the environmental conditions dictate the sensor choice.

As sensitive material, different groups could be divided. Organic Polymers include for example Cellulose Acetate, PolyMetilMetacrilat (PMMA), Polyimide etc. The group of Porous Ceramics has some typical representatives as TiO_2-V_2O_2, Al_2O_3, $MgCr_2O_4$-TiO_2 etc. The third is the group of Electrolytes, such as LiCl. The fourth is the group of Porous Silicon. Each of these divisions has some advantages, disadvantages, manufacturability and typical areas of application.

Having in mind this information, a humidity sensor, using vacuum deposited Polyimide film as a sensitive layer, based on capacitive principle of operation, was designed, produced and measured.

2. DESIGN OF HUMIDITY SENSOR

The design procedure starts with choosing of the type of measured parameter. After checking the possible areas of application, the measurement of Relative Humidity was chosen. We need to have a sensitive, small, simple and inexpensive humidity sensor, which is not contamination sensitive and could be produced, using technological processes and sequences, known by Microelectronics and Microsystems (MEMS) Technologies. The choice was on the group of Hygrometers using hygroscopic materials, using principals of Electrical property change (resistivity, capacitance, frequency). In the device should be used some humidity sensitive material, such as Polyimide. The humidity evaluation will be carried out by measurement of capacitance between metal electrodes.

The construction of the capacitance sensor includes a substrate carrier, on top of which, a sensitive Polyimide layer is deposited. There, there are at least two metal electrodes, between which, the structure capacitance is measured. These electrodes could be created beneath and over the sensitive layer (sandwich structure), or on the same side of the Polyimide film, in the form of interdigitated electrodes.

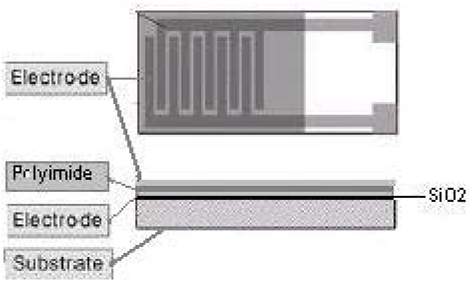

Figure 1. Experimental structure

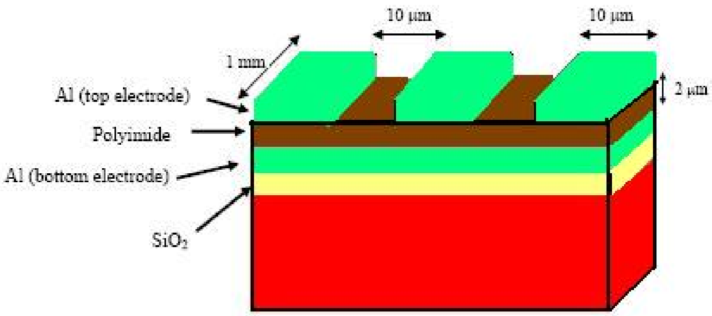

Figure 2. Cross section of the real structure

In the case of "sandwich" structure, the capacitance of interest will be that of the volume of the layer, between electrodes. In this case, the upper electrode should be partly permeable (transparent) for the water vapors. In order to increase the reaction volume, the surface electrode should be as thin and narrow as possible. As a result, the quality factor of the structure will be law.

In the second case, the sensitive layer will participate in humidity evaluation by its surface area. Created two comb-shape electrodes on top of the structure, we let the most of the surface to be used as a sensitive area.

In Fig. 1, the view (horizontal geometry) and the cross section (vertical geometry) of such structure are shown. It is clear that, beneath the Polyimide layer, there is the bottom electrode of the microsensor. On top of the sensitive coating, there are two interdigitated electrodes. This structure gives the possibility to measure and estimate both the volume and surface capacitances. A cross section of the structure, with the dimensions of the layers and electrodes, is given in Fig. 2. Two different structures were manufactured, one with 10 μm fingers and spaces ("Big"), and the other – with 7 μm fingers and spaces ("Small").

3. POLYIMIDE DEPOSITION

As it was already mentioned, as a sensitive material, a Polyimide layer was used. It was deposited from gas phase and has high dielectric constant, good adhesion, uniform thickness and density, alongside the whole sample.

The Polyimide deposition technology starts by using of the following precursors:

- ODA – oxydianiline - $C_{12}H_{12}N_2O$;
- PMDA – pyromellitic dianhidride - $C_{10}H_2O_2$.

The experimental layers were created by vacuum deposition of the precursors – PMDA and ODA with following thermal treatment. The Polyimide coatings were prepared by using of planetary movement of the substrates 30 r.p.m./min, in vacuum system UVN, at vacuum level around 10^{-6} Torr. The precursors were evaporated from two independent sources with resistive heating. As a result of the experiments, the temperature ranges of evaporation were defined – 100 - 110^{o}C for ODA and 120 - 145^{o}C for PMDA. At such conditions, deposition speeds of 0,2 – 2 Å/sec were obtained. At higher evaporation speeds, the number of the defects is increased. The latter is carefully controlled by quartz oscillators. Thus, the optimal ratio in the flux of ODA: PMDA = 1 : 1 vapors was ensured. The co-deposited precursors were transformed into Polyimide layer by a two – steps thermal treatment (1 hour at 170^{o}C + 1 hour at 250^{o}C) by inducing polycondensation reaction at strictly controlled temperatures. Using the described procedure, layers with thickness of 630 nm and 1000 nm were produced.

4. TECHNOLOGICAL SEQUENCE

The technological sequence includes the following processes: semiconductor (silicon) substrate was covered with layer of SiO_2 with thickness of 0,3-0,4 μm by thermal oxidation. On top of this structure, an Aluminum layer, 0,5-0,6 μm thick, was applied by thermal evaporation in vacuum. The Polyimide coating was deposited, using the described procedure. Finally, again with thermal evaporation in vacuum, the upper Aluminum layer, 0,5-0,6 μm thick, was put. The next technological processes are photolithography and chemical etching of Aluminum layer. The structures were mounted on substrate carriers and bonded by metal wires.

The measurements were performed by using of specially designed and made measuring chamber, supplied with vacuum pump, bubbler for the water vapors, dehydration filter and witness humidity sensor.

5. MEASUREMENTS AND EVALUATION

The structures were measured by test camera. First, the **humidity response** was estimated. Starting the measurement procedure, dry air was introduced and the structure was measured. After that, through the bubbler, different quantity of moisture was introduced. In this way, the humidity response, from 0% to 100% RH was obtained. This procedure was called **Calibration/Sensitivity**. The reverse process was performed by introducing some quantity of dry air, through dehydration filter, starting from 100% RH and decreasing the values to 0% RH. In this way, so called **Hysteresis** of the structure was obtained.

The **recovery time** of the structures was measured by introducing of dry air inside of the camera, through dehydration filter, starting from 100% RH and decreasing the values to 50% RH.

Three different cases of connection were tested. The capacitance was measured between one upper and bottom electrodes, between two shorted upper and bottom electrodes and between two upper electrodes. The tests were done for "Big" and for "Small" structures. Some results are shown in Fig. 3.

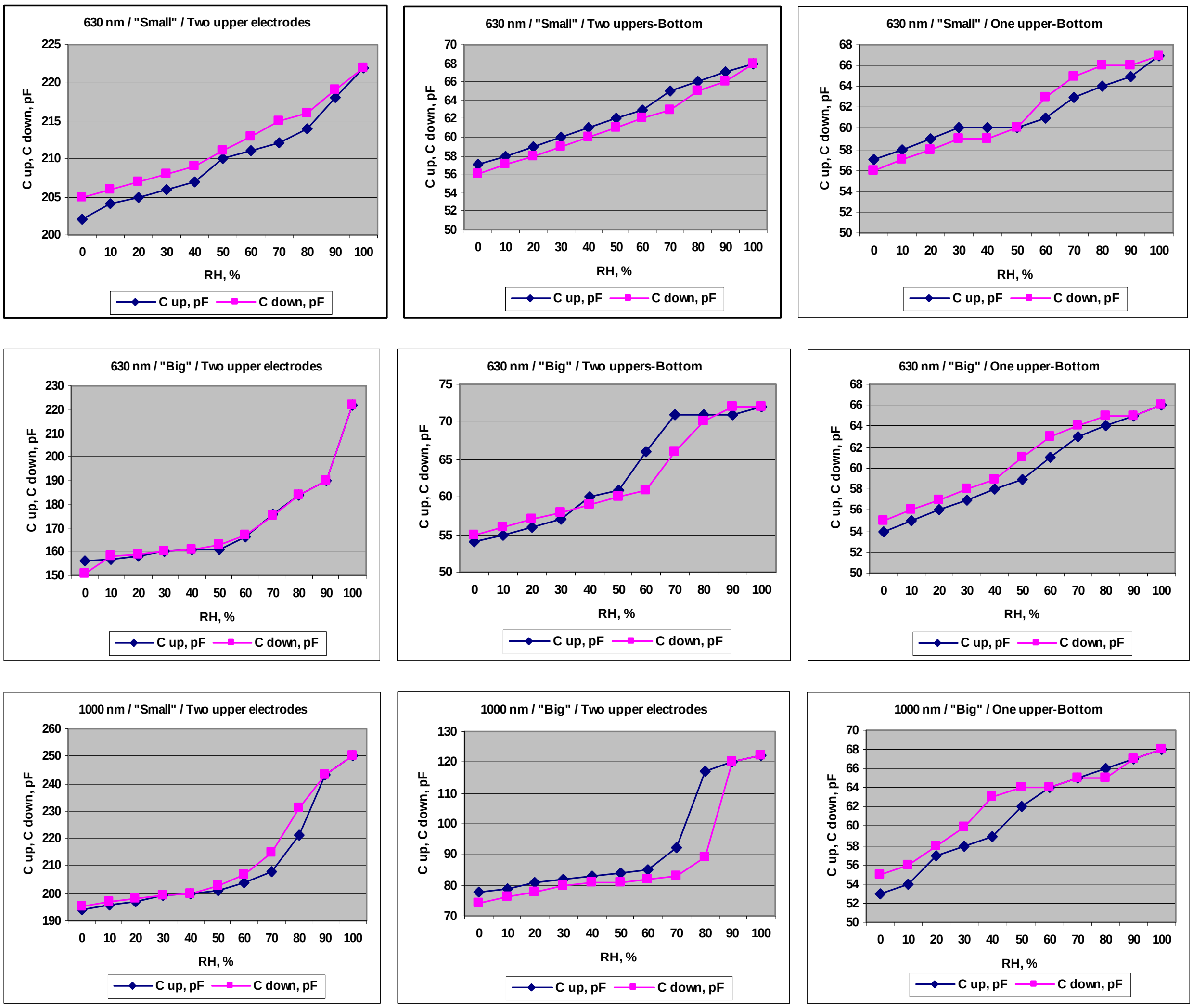

Figure 3. Sensor capacitance vs. RH for different structures

It is clear that the "horizontal" capacitance (between upper electrodes) is higher, comparing to "volume" capacitance (between upper(s) and bottom electrodes). The dependence, between the capacitance and RH, in the case of the "volume" structures, is quiet linear, in opposite to the "horizontal" structures. In general, the "volume" capacitance is in the range between 50 and 70 pF, i.e. the capacitance change is around 40%, comparing the initial point. Increasing the thickness of Polyimide layer, the capacitance of the "horizontal" structures is higher, which is due to deeper penetration of electrical lines in the dielectric material. The "volume" structures capacitance is negligibly changed, because of the small thickness of the layer.

The estimation of the hysteresis shows that it is very difficult to formulate some strong dependence. In both directions of humidity change, the values of the capacitance are similar and near, which shows about the absence of real hysteresis.

The recovery time of the structures is between 9 and 12 sec. It is smaller in the cases of thin Polyimide layer, which is logical, because of smaller moisture adsorbing volume.

The electrodes dimension shows that, in case of measurement between upper electrodes, the smaller electrodes have higher capacitance, because of shorter distances between them.

6. CONCLUSIONS

Humidity sensors were designed, fabricated and tested. As a sensitive material, a Polyimide layer was used. The way of measurement is based on capacitance control. Some conclusions were done, in connection with the thickness of the sensitive material, the shape and configuration of the measuring electrodes, the way of using of sensitive layer ("surface" or "volume") etc. The obtained results give useful information for future developments of micromechanical humidity sensors for different applications. Together with the improvements of sensor parameters, some new innovations could be done. For example, one of them could be implementation of thermal sensor for device calibration and higher sensitivity and accuracy.

7. ACKNOWLEDGEMENTS

The research, described in this paper, was carried out within the framework of the contract № BY – TH 115 / 2005.

The financial support of the National Fund of the Ministry of Education and Science, Bulgaria – contracts: VU-TH – 964/06 and VUH – 09/05 is gratefully acknowledged

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