# FABRICATION AND CHARACTERISTICS OF POROUS POLYIMIDE FILM FOR HUMIDITY SENSING

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***Abstract*:** The capacitive humidity sensor was made by thin film technology. A polyimide film by 30 μm thickness was metallised using e-beam evaporation at a bases pressure of $10^{-5}$ torr and deposition rate of 15 Å/s by chromium in 0,5 μm thickness and embedded in 2 blades with 4 apertures. The sensor performed measurements in the range 0-95%RH and according to sensitive characteristic the capacitance increase linear with relative humidity in the studied range. The capacitance at 33% RH, 20 °C, 20 KHz was 90 ± 15 pF, the linearity 1%, the hysteresis in the humidity range, 3% at 50 % RH, the temperature dependence of the capacitance 1 % RH/ °C in the range of the temperature 0-50 °C. The response time was 1 minute for the humidity cycle 0-95 % RH and 5 minutes for cycle 95-0 % RH. Excellent stability was exhibited in various environments in particular the sensor showed values smaller then ± 3 % RH drift after 1000 hours exposure to 50 °C and 95 % RH conditions.
***Keywords***: Relative humidity, capacitive polymer sensor, thin technology, polyimide

# HERSTELLUNG UND CHARAKTERISTIKEN DES PORÖSEN POLYMIDFILMS FÜR DIE FEUCHTIGKEITSBEMERKUNG

***Zusammenfassung*:** Der kapazitive Feuchtigkeitsfühler wurde durch die Technologie der dünnen Schichten hergestellt rin Polymidfilm mit 30 μm dicke wurde in Vakuum metallisiert bei einen Grunddruck von $10^{-5}$ torr, bei einer Absetzungsrate von 15 Å/s mit Chrom von 0,5 μm Dicke und in 2 Lamellen mit 4 Offnungen eingekapselt. Die Fühlermessungen wurden im Bereich 0-95% RF und in Übereinstimmung mit der Empfindlichkeitscharakteristik durch-geführt, die elektrische Leistung wächst linear mit der relativen Feuchtigkeit im studierten Bereich. Die Leistung von 33% RF, 20°C, 20KHz war von 90 ± 15 pF, die Linearität 1%, Hysteresis im Feuchtigkeitsbereich, 3% bei 50% RF, die Abhängigkeit mit der Leistungstemperatur 1% RF/°C im Temperaturbereich 0-50°C. Die Antwotszeit war von 1 Minute für den Feuchtigkeitskreislauf 0-95 % RF und 5 Minuten für den Kreislauf 95-0 % RF.Es wurde eine ausgezeichnete Stabilität in verschiedenen Umgebungen bestimmt; der Fühler zeigt Variatioswerte kleiner als ± 3 % RF nach 1000 Stunden Aussbellung bei 50°C und 95% RF.
***Schluesselworte***: relative Feuchtigkeit, Kapazitive Polymerfühler, Dünne Technologie, Polymid

## Introduction

The reliable measurement of humidity and moisture is gaining great importance in many applications such as industrial, meteorological, food and agriculture production. A large number of sensor principles and techniques have been made commercially available over the last years. Surveys have shown that mostly capacitive polymer thin film humidity sensors are used [1].

Various kinds of polymers have been used to prepare humidity sensors. From the point of view of their basic principles, they are classified into two categories. The first one is based on the change in electrical properties of the material due to the sorption of water vapor and second one is based on the gravimetric change in the material. The latter utilizes a quartz crystal oscillator. The first group is divided into two types, that is, the electrical resisitive type and the capacitive type. For resistive type humidity sensors, hydrophilic polymers are used, while hydrophobic polymers are preferable for capacitive type sensors.

There are several requirements for practical humidity sensors, i.e. high sensitivity, little hysteresis, small temperature coefficient, fast response, long term stability at high umidity, resistance in various gases and organic solvents.[2].

For the capacitive type humidity sensors the polyimides ($PI_s$) have been extensively investigated for use in relative humidity sensors [3-8]. These materials are tough, thermally stable available as films and resins. In addition, the resin forms are used extensively in integrated circuit (IC) fabrication and can be used in integrated RH sensor design. The polyimide is formed by a thermally activated polycondensation reaction, liberating water in the process. Environmental water can break the carbon-nitrogen bond in the imide ring, changing the polarizability and the moisture uptake properties of the $PI_s$. These changes modify the RH sensing characteristics of the film, which in turn will change the characteristics of the sensor. The technology used in the fabrication of the sensor is that of thin film capacitors. The stages of the process consist first in the choice of a hygroscopic dielectric (a polymer) and secondly in the elaboration of a porous electrode.

The dielectric constants of the polyimide is very low (c.3), when compared to that of water (c.80). When a small amount of water is sorbed by the polyimide, the apparent dielectric constants increase, resulting in a linear increase in capacitance with relative humidity. Polyimide was advantages over the other polymers due to its stability against solvents, other volatile chemicals and high resistance of temperature.

The porous electrode is obtained by chromium evaporation under conditions such that the film is tensile stressed. These stresses generate a very large number of cracks in the polymer. These cracks some 1000 Å wide transform the polymer into a lot of small islets, protected by a thick layer of chromium. Thanks to this technology, permeability is achieved without impairing conductivity or mechanical strength and the penetration rate of moisture is increased by several orders of magnitude whatever the thickness of chromium (100 Å to 1μm). [9-11]

In this paper the author presents a capacitive humidity sensor utilizing polyimide as a sensing material, the fabrication process and sensing characteristics.

**Experimental**

A variety of polyimide film by 30 μm thickness was metallised using vacuum e-beam evaporation by chromium in a 0,5 μm thickness. The coat is done through one metal mask in a vacuum evaporation plant at the pressure $2x10^{-5}$ torr and deposition rate of 10 Å/s. Finally the sensor was embedded in 2 blades with 4 apertures. Figure 1 shows the model of the sensor.

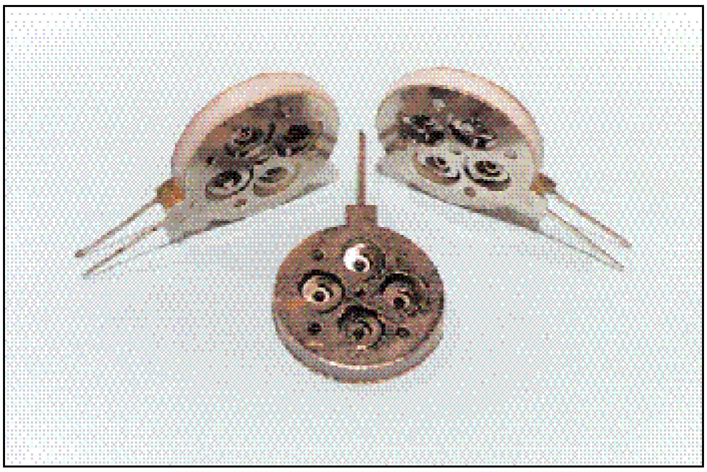

*Figure 1. The model of the capacitive humidity sensor*

**Results and discussion**

The sensor was tested in an apparatus which consists: thermostat test chamber, selector, recipients of saturated salts who generate humidity controlled atmosphere, a Hewlett Packard type LCZ bridge analyser. Calibration of the sensor was accomplished in the saturated salt solutions such: LiCl for 12 %RH, $MgCl_2$ for 33%RH, $K_2CO_3$ for 43%RH, NaBr for 56%RH, NaCl for 75,5%RH, KCl for 85%RH and $K_2SO_4$ for 95%RH. For 0%RH was utilized silica gel and for 100% RH water vapours. Figure 2 shows the characteristic capacitance-relative humidity of the sensor which was measured in the range 0-95%RH, at 20 KHz and 20°C. The stabilizing time between each measurement was about one hour. According to this characteristic, the capacitance of the sensor increase linear with relative humidity in the studied range.

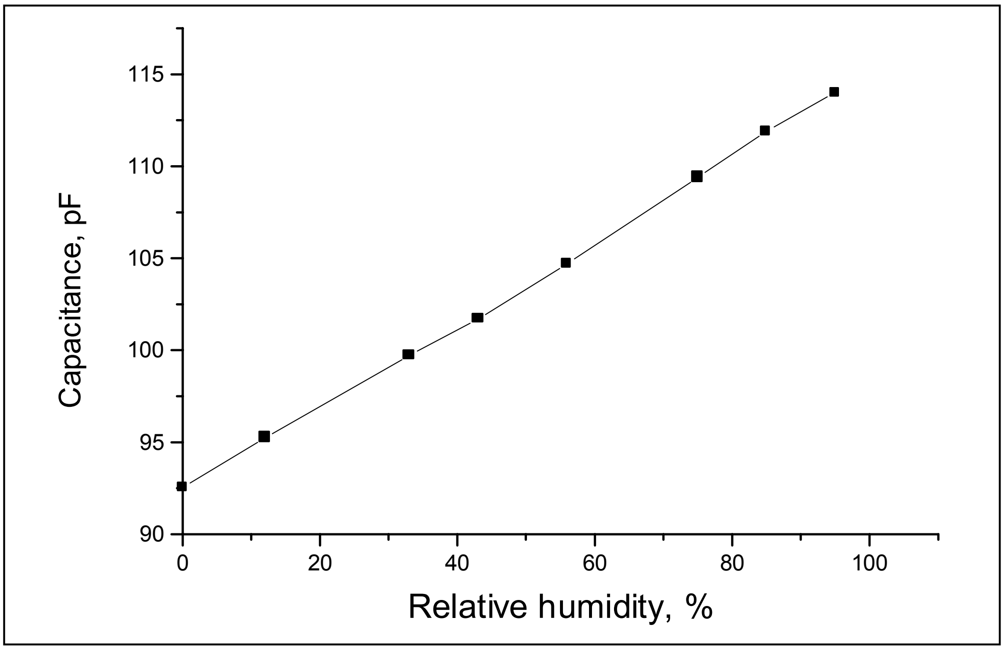


*Figure 2 The characteristic capacitance-relative humidity of the sensor*

Table 1 shows the other characteristics of the sensor

*Table 1 Summary with the performances of the sensor*

| | |
|---|---|
| The capacitance at 33%RH, 20°C, 20KHz | 90±15 pF |
| Sensitivity | 0,2 pF/%RH |
| Hysterezis at 50%RH, 20°C, 20KHz | 3% |
| Temperature dependence of the capacitance in the range of the temperature 0-50°C | 1%RH/°C |
| Response time for the:<br>Humidity cycle 0-95%RH<br>Humidity cycle 95-0%RH | <br>1 minute<br>5 minutes |

Long term stability of the sensor was evaluated under various ostile environments. The sensor was exposed to the testing conditions over a humidity cycle of 0-95% RH at –20°C ÷ +80°C for 1000 hours. Some of the test results are summarized in table 2. The test results shown in this table are the maximum value, mostly observed at 95%RH. Excellent stability was exhibited in the various environments. In particular, the sensor showed ± 3% RH drift after 1000 hours exposure to 50°C, 95% RH conditions. This stability under hot and humidity conditions is very important for a wide variety of applications for this sensor. Other environmental test results proved to be 2-3 % RH output shift, reproducibility showing an overall good performance of this sensor.

*Table 2 Long term stability of the polyimide sensor*

| Test condition | Sensor output shift ( % RH ) |
|---|---|
| Low temperature, -20°C, 1000 h | ±2,5 |
| High temperature, +80°C, 1000h | ±3 |
| Temperature cycle –20°C, +80°C, 100 cycles | ±3 |
| Volatile solvents | ±2 |
| 5 cycle 50°C, 95% RH, 1000 h | ±3 |

**Conclusions**

Was fabricated a humidity capacitive sensor utilizing thin film technology by generating cracks in the film of polyimide.The test results show that the sensor has a capacitance at 33% RH, 20 $^{o}$C, 20 KHz by 90 ± 15 pF, the linearity 1%, the hysteresis in the humidity range 3% at 50 % RH, the temperature dependence of the capacitance 1 % RH/ $^{o}$C in the range of the temperature 0-50 $^{o}$C. The response time was 1 minute for the humidity cycle 0-95 % RH and 5 minutes for cycle 95-0 % RH. Excellent stability was exhibited in various environments in particular the sensor showed values smaller then ± 3 % RH drift after 1000 hours exposure to 50 $^{o}$C and 95 % RH conditions.

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