Design and characterization of a low thermal drift capacitive humidity sensor by inkjet-printing

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Abstract – Small, low-cost and flexible humidity sensors were designed, fabricated by using an inkjet-printing process, and fully characterized. Based on the principles of the capacitor and the ability of a polyimide to absorb humidity, the sensor was fabricated by printing silver interdigitated electrodes on a thin polyimide film of 75 µm of thickness. After modelling, the total area of the printed sensor was optimized to be 11.65 mm^2. A relative humidity sensitivity of 4.5 fF/%RH and a thermal coefficient of -0.4 fF/°C were measured at 100 kHz, whereas the sensitivity and the thermal coefficient were 4.2 fF/%RH and -0.21 fF/°C, respectively, at 1 MHz. This latter result implies that it could not be necessary to include thermal compensation to use this sensor depending on the required accuracy and the chosen frequency. This work shows a reliable, fast, simple and low-cost manufacturing process to make small humidity sensors with low thermal drift and high temporal stability. These sensors could be easily integrated into inkjet-printed RFID tags for monitoring of environmental humidity in diverse applications.

Keywords: Moisture sensor; Interdigitated electrode capacitor; Flexible electronics; RFID tag

1. Introduction

In recent years, printed and flexible electronic devices have become increasingly attractive due to their potential low-cost per surface area, mechanical flexibility and feasibility of large scale processing. The main advantage of printed electronics is a simplified manufacturing process, which results in lower cost processes and shorter cycle time.

On another front, today there is a very strong and growing demand in world trade for humidity sensors. In fact, the field of smart packaging including sensor capabilities opens new challenges in the ~~developing~~ development of flexible and printed humidity sensors compatible with this kind of technologies. An important additional advantage of printed sensors is the possibility of integrating them with printed radio frequency identification (RFID) tags. There is a lot of interest at present in converging RFID tags and sensing capabilities that are able to save and store the acquired information related to both identity and measured parameters, see for example [1-3]. The introduction of RFID and the Electronic Product Code (EPC) standard as a substitute of popular barcodes in packaging has advanced markets in intelligent packaging. It will be possible to read not only many packages at the same time but also environmental parameters extracted from sensors incorporated into the containers. There is a special interest in the capability of tracking the condition of a package through the whole supply chain to certify that products in their packages have not been endangered because of being exposed to wrong environmental conditions.

Great efforts and very valuable advances have been made in the design of flexible and printed humidity [3-6] and other gases sensors [7-9]. Related to the requirement of low energy consumption, the classic transduction mechanism of these humidity sensors is capacitive, specifically through changes in the electrical permittivity of some component of the capacitor and the dielectric thickness. This requires the use of chemicals (usually polymers) whose electrical permittivity changes with the relative humidity of the environment. One of the most frequently used structures for capacitive sensors is based on planar interdigitated electrodes (IDE) due to its compactness, high contact area and relative ease of manufacturing [10-12].

Different fabrication processes have been used to develop this kind of sensors, such as gravure, screen printing and inkjet-printing; and different strategies have been applied to include the sensing capability in the capacitor. The most common approach has been to deposit the sensing layer over the IDE capacitor [13-15]. Some frequently used polymers are cellulose acetate butyrate (CAB), polymethylmethacrylate (PMMA) and polyvinylchoride (PVC), among others. Another possibility is to use the flexible substrate as the sensing element. In this case, polyimide [6] and photographic paper [5] have already been described, saving fabrication steps compared with the former approach. Despite all the previous work, these capacitive sensors have a high thermal drift as one of the main challenges to be overcome in order to obtain an accurate humidity measurement. Differential measurements with reference capacitors (not sensitive to humidity) [2] or including additional temperature sensors [16, 17] are some of the used strategies to reduce the interference due to thermal drift. Both solutions imply the addition of other devices, consuming more area and energy.

In this work, we present the design, fabrication and characterization of a capacitive humidity sensor which uses the flexible substrate as sensitive element. This capacitor has been printed with silver nanoparticles by inkjet-printing on a polyimide thin film. Our aim has been to obtain a very small device with optimized dimensions based on numerical simulation, minimal fabrication steps and a very low thermal drift without additional components, useful in many applications. Furthermore, we have analysed the influence of the number of printed layers ~~in~~ on the sensor performance.

2. Materials and Methods

A. Sensor Design

The devices analysed in this study are planar IDE capacitors which allow more direct interaction between the sensor and the surrounding environment compared to other structures [13]. The usual approach for providing humidity (or other gases) sensitivity is to deposit a sensing layer on~~to~~ this structure with some humidity-dependent electrical property. The variation of this property with the humidity produces changes in the capacitance of the whole device. But here, we have skipped this deposition step and directly used the flexible substrate made of polyimide as the sensing element to simplify the fabrication process (Fig. 1). The polyimide is a well-known chemical whose electrical behaviour shows a high sensitivity to the relative humidity. Specifically, the relationship between ~~its~~ the electrical permittivity of this polyimide and the relative humidity ~~of this polyimide~~ has already made it interesting to ~~be~~ test~~ed~~ it as a humidity sensor [6]. This relationship is given by:

$$\varepsilon_r = \varepsilon_{r0} + \alpha \cdot RH(\%) \qquad (1)$$

where RH(%) is the relative humidity in percentage and ε_{r0} and α are material dependent parameters. This relative permittivity also depends on frequency and temperature among other parameters which could interfere with the measurement of the humidity. These dependences ~~have also to~~ must be also analysed in order to obtain a complete overview of the sensor behaviour and to try facing them to improve the sensor performance.

The optimization of the sensor dimensions may potentially introduce more sensors into the devices, saving manufacturing materials and area. For this purpose, we used COMSOL Multiphysics 4.2a (www.comsol.com, COMSOL, Inc. USA) to optimize the design. This is a powerful interactive environment for solving problems based on partial differential equations with the finite element method. This software has previously been used to calculate distributions of potential field in this ~~kind~~ type of structures [18, 19].

The total capacitance is determined by the integral of the electrostatic energy density, W_e, through the ~~relation~~ equation:

$$C = \frac{2}{{V_{port}}^2} \int_{\Omega} W_e d\Omega \qquad (2)$$

where V_{port} is the value of the applied voltage in the port of the sensor. The other electrode is connected to ground. The electrical parameters of the substrate given by the manufacturer and the printed and cured conductive silver ink according to our characterization were included in the numerical simulator [20].

Several parametric analyses were performed varying the fundamental geometrical parameters of the IDE such as the number of fingers, the gap width between two consecutive fingers and their dimensions (width, length and thickness of each finger). In order to optimize the area, we fixed the finger width to the minimum diameter landed drop (in our case 50 µm) and the gap between fingers also to 50 µm. This gap could be reduced below the drop diameter value to increase the capacitance value but this reduction will lead to a strong possibility of short-circuit between electrodes due to printing errors. The parametrical simulations showed that the thickness of the fingers hardly affects the capacitance value in this structure. In this work, we have also tested structures with one and two printed layers. As shown below, their thicknesses are under 1 µm in both cases ~~which implies~~ implying extremely long simulation times because of meshing issues. Due to this fact, we set the thickness of the IDE to 5 µm for all the simulations to drastically reduce the computational time. Then, we extrapolated the value of the capacitance for the thickness of 1 layer and 2 layers according to Fig. 2 where the slope of the curve is 0.019 pF/µm.

According to previous considerations, we manufactured the sensor following the specifications from Table I for a targeted nominal capacitance (for one printed layer) of 2 pF since they presented the best compromise between capacitance and area. Finally, the capacitance predicted by COMSOL Multiphysics for this structure was 1.949 pF with only one printed layer in a dry atmosphere.

The designed IDE area was 11.65 mm^2 (L = 1.85 mm x W = 6.3 mm) composed of 63 fingers (32 fingers for one electrode and 31 for the other one) with 50 µm width and inter-spacing (see Fig. 1). This area is significantly smaller than other comparable printed humidity sensors presented in the literature [13-15, 21]. In order to easily test the capacitor ~~properly~~, we added two long terminals to ~~easily~~ couple the sensor to the measurement set-up.

Obviously, the greater the structure ~~is~~, the bigger the capacitance obtained. Our interest in such a small structure was not only to reduce cost in terms of materials and time of fabrication but also to integrate more different sensors into the same area.

B. Fabrication Process

The DMP-2831™ Dimatix printer (Fujifilm Dimatix Inc, Santa Clara, USA) was used for inkjet printing. The selected materials were an ink of silver nanoparticles (U5603 SunTronic Technology, San Diego, USA) on a polyimide substrate (Kapton® HN with 75 µm of thickness, Dupont™). Table I shows the main properties of the used ink and substrate, respectively. According to the manufacturer of the substrate, the relationship between the relative permittivity and the relative humidity is given by:

$$\varepsilon_r = 3.05 + 0.008 \cdot RH(\%) \quad at\ 1\ kHz, 23℃ \qquad (3)$$

The first step before printing was to prepare the substrate to ensure the best quality and to avoid failed printings with a cleaning process. First, we immersed the substrate in acetone ~~solution during~~ for 2 min to remove dust on the surface, then we submerged it in propanol about 2 min to remove ~~eliminate~~ the acetone. After that, we washed the sample ~~did a washing~~ with purified water to eliminate the propanol and finally a drying at 120ºC during 5 min.

This treatment was done to remove all traces of particles that could affect the printing process. The substrate temperature was fixed at 40ºC while printing. A drop space of 25 μm was settled in the printer for 50 μm landed diameter drops followed by a sintering step at 120ºC for 60 min. According to the model presented in [13, 20] the amount of used ink is 22.68 nl and 45.36 nl for one and two printed layers, respectively. With that printing and curing conditions, the resistivity of the conductive electrodes were 23 ± 2 μΩ·cm for both one and two printed layers [20]. The fabrication time is much lower than in the case of other sensors because no other sensing layer was needed [13-15]. The fabrication process is also simplified because it only required printing one/two layer/s on one side of the substrate. A matrix of twenty eight capacitors –one half of them with one layer, the other half ~~of~~ with two layers- has been fabricated in order to ~~know~~ test the reproducibility of the process.

C. C*haracterization*

The physical characterization, the roughness of printed patterns as well as the thickness of the patterns has been done using a Wyko NT1100 Optical Profiling System (VEECO, Tucson, AZ, USA). The AC electrical characterization for the different fabricated capacitors has been performed by measuring their capacitance and dissipation factor, using the four-wires measurement technique with a precision Impedance Analyzer 4294A and an impedance probe kit (4294A1) (Agilent Tech., Santa Clara, CA, USA). The excitation voltage applied in all measurements was V_{DC} = 0 and V_{AC} = 500mV. The frequency sweep of analysis was from 100 kHz to 10 MHz. We have considered this frequency range for its compatibility with a wide variety of readout electronic circuits in real applications.

As it has been mentioned above, one of the end sides of the backbones has been enlarged to facilitate its connection to any analyser. A SMA (SubMiniature version A) male connector has been glued to these ends points using silver-filled epoxy EPO-TEK® H20E (Epoxy Technology, Inc., Billerica, USA) (see Fig. 3).

It was necessary to calibrate up to the SMA connectors including the mentioned extensions of the backbones to rigorously characterize ~~rigorously~~. For this purpose, we measured several commercial capacitances placed in the same configuration as shown in Fig. 3a, with the 16034G SMD Test Fixture (Agilent Tech., Santa Clara, CA, USA). After processing all data, the total added parasitic capacitance was 2.70 ± 0.02 pF in the whole spectrum of the impedance analyser. The data acquisition and analysis have been automated using Labview 2012 software (National Instruments Corporation, Texas, USA).

The stationary humidity and temperature responses of this sensor have been measured in a climatic chamber VCL 4006 (Vötsch Industrietechnik GmbH, Germany). The humidity range varied from 10% RH to 98% RH in a temperature range of + 10 °C to + 95 °C. The humidity deviation in time was ± 1% to ± 3%, whereas the temperature deviation in time was ± 0.3 °C to ± 0.5 °C. Due to the slow response of the mentioned climatic chamber, the dynamic response has been measured in a customized humidity measurements set-up

(9 x 3 cm^2), which automatically controls wet and dry airflow inside a small gas cell at room temperature. Two LCR-meters (HP 4284L and Agilent E4980A, Agilent Tech., Santa Clara, CA, USA) have been used to measure the corresponding capacitance values at a frequency of 100 kHz every 5 s. Capacitance and time measurements of the printed sensors have been controlled and recorded using the software Labview 2012. RH and temperature measurements have also been ~~also~~ registered by a commercial sensor (SHT15, Sensirion AG, Switzerland) in order to verify the data given by the chambers' displays. In all cases, the IDE capacitors have been placed in the middle of the climatic chambers allowing the atmosphere interaction in both faces of the sensors, printed and non-printed.

3. Results and Discussion

In this section, we first show the physical characterization of the printed IDE capacitors and the fabrication yield. This yield was calculated from low frequency capacitance measurements of ~~the~~ several replicas of IDE capacitors. After that, the capacitance response to the relative humidity was characterized taking into account ~~also~~ the measurement frequency ~~of measurement~~ and the temperature dependences. Regarding all these experimental data, we analysed the ratio of humidity sensitivity and thermal drift to find the frequency range where humidity can be accurately measured without compensation of ~~temperature~~ thermal effects.

3.1. Physical characterization

We have manufactured fourteen samples with one printed layer and other fourteen samples with two layers. According to a previous~~ly~~ developed physical model of layer thickness by inkjet printing [13, 20], the estimated thicknesses are 430 nm and 860 nm, respectively. The real dimensions of the structures are given in Table III.

As can be observed in Fig. 4, we used a profiling system to study the real physical characteristics of the printed sensors. Table III also shows the differences between the estimated dimensions and the measured ones for sensors of one and two printed layers. Uncertainties were calculated as ~~one~~ the standard deviation of the experimental data.

R_q is the root mean square (RMS) and R_a the arithmetic average of the absolute values, both of the surface roughness heights. In general, modelled dimensions are very close to experimental ones, showing the greatest difference in the finger width. This can be explained by the spread of the ink drop when it is deposited on the substrate [22].

There is a very good agreement between the measured and the simulated values of the capacitances of the replicated IDE capacitors; this confirms that the proposed extrapolation procedure works properly for the given electrode thicknesses. The measurements were taken in ambient conditions (30% RH and 25ºC) at 1 kHz. The employed frequency was not too high to hinder polarization. During these tests, a preliminary (the total number of fabricated samples was 28) manufacturing yield of 90% was found, that is, one sample ~~of~~ out of ten was totally or partially broken. As expected by the numerical simulation (see Fig. 2), capacitors with two printed layers showed only a 0.0~~57~~36 pF bigger capacitance than those with one layer, due to the low influence of the finger thickness on this electrical magnitude. Moreover, the standard deviation is reduced in almost 3 times in case of two printed layers because more similar structures are obtained with the second layer. This improvement is due to the fact that the second layer covers the irregularities of the first printed layer, smoothing the structure surface.

According to the simulated capacitance, the measured values are about 5% higher in both cases (Table IV). The discrepancy between these results might be caused because the real dimensions of the sensor are not exactly the same as the simulated ones. As described by

[22], printing electronics techniques present undesirable effects. Those effects lead to an inaccuracy ~~on~~ in the printed structure. For example, drops tend to spread out when they are deposited on many substrates. This behaviour results in wider fingers and narrower inter-spacing as shown in Table III**¡Error! No se encuentra el origen de la referencia.**. In addition to this, the frequency dependence is not taken into account in the simulations. As this sensor is quite small, any dimensional difference can affect the results. Therefore, these results confirm that the approximation taken in the numerical simulations for the thickness can be done without inducing substantial errors.

3.2. Humidity and temperature responses

After the physical characterization, hereafter the sensor capacitance has been analysed as a function of humidity and temperature in a wide frequency range ~~has been analysed~~. Let's remember that a humidity response with minimal thermal drift is our goal for the developed IDE capacitor. For five IDE capacitors with one printed layer, we have measured the capacitance as a function of the relative humidity and temperature in the frequency range from 100 kHz to 10 MHz. Furthermore, the measurements have been carried out in both directions for both humidity and temperature sweeps, that is to say, increasing and decreasing the relative humidity at fixed temperature for obtaining the sensor hysteresis in RH and vice versa for temperature.

Fig.5 shows the measured capacitance of the sensor for different values of RH at constant temperature. As can be observed, the sensor presents very similar response in frequency for each of the tested RH. Weremczuk *et al.* [14] found comparable capacitance curves for an IDE structure with a deposited layer of Nafion as sensing material. The displacement between curves due to RH variations is constant up to 70%. From 80% RH, the displacement between lines slightly increases; this tendency can be explained by the condensation of water on the sensor surface at high humidity levels which could connect the electrodes and modify the global impedance [23]. For our results, the general decreasing trend with the frequency can be explained by the electrical permittivity decrease found in the Kapton HN substrate [24].

As mentioned, the temperature is the most important interfering factor in the response of the developed capacitive sensors. To test this dependence, Fig. 6 displays the capacitance as a function of frequency for several temperatures at 60% RH. These curves are practically overlapped up to 40°C, and then a displacement is observed. The range of frequencies, where the least difference between lines happens, is from 1 MHz to 10 MHz.

To obtain a better insight of the whole sensor response, we have defined the partial sensitivities of the capacitance with the relative humidity, S_{RH} (f), and the temperature, S_T (f) as a function of the measurement frequency, f, ~~of measurement~~ as:

$$S_{RH}(f) \equiv \frac{\partial C_{T=cte}\,(RH)}{\partial\, RH} \qquad (4)$$

$$S_T(f) \equiv \frac{\partial C_{RH=cte}\,(T)}{\partial\, T} \qquad (5)$$

Both sensitivities are shown in Fig. 7. Regarding humidity sensitivity, it decreases from around (4.5 ± 0.2) fF/%RH and ~~tending~~ tends to (4.0 ± 0.2) fF/%RH at the highest analysed frequencies. This tendency is a direct consequence of Fig. 5 where the separation between consecutive experimental curves decreases at higher frequencies. This range of sensitivities, about 2200 ppm/%RH, is a typical value in comparable previous works [13, 21, 25]. With respect to the temperature behaviour of the sensor in the whole spectrum, the thermal dependence decreases at higher frequencies, from about (-0.4 ± 0.2) fF/°C at 100 kHz to less than (-0.2 ± 0.2) fF/°C at 1 MHz. The ~~sensitivities~~ errors in sensitivities have been estimated by linear propagation of experimental errors in the calculated linear regression. Comparing thermal and humidity sensitivities (Fig. 7), this sensor has shows a humidity sensitivity between 11 and 22 times higher than the thermal drift in the analysed frequency range. Considering the central frequency of the analysed span, 1 MHz, we have measured a sensitivities sensitivity ratio of 21. This means that our sensor would show a maximum error of 2% in RH without thermal compensation, within a temperature range of 40ºC. This inaccuracy could be acceptable in many low-cost applications such as those related to RFID tags with sensing capabilities.

3.3. Calibration curves

Now, the calibration curves with the relative humidity and its hysteresis will be presented. The behaviour response of the sensor to changes in the relative humidity is directly extracted from the curves obtained with the impedance analyser. In Fig. 8, we show a graph of these curves at the chosen frequency to compare and contrast the response of the sensor to variations in the RH at different temperatures.

We have calculated the calibration curves of the experimental data shown in Fig. 8**¡Error! No se encuentra el origen de la referencia.**. Equations 6 and 7 show these calibration curves. The coefficient of linearity is bigger than 0.98 in all cases.

$$C(pF) = 0.0041 \cdot RH(\%) + 1.693.\ \mathrm{R}^2 =\ 0.983.\ \text{Increasing RH} \quad (6)$$

$$C(pF) = 0.0043 \cdot RH(\%) + 1.702.\ \mathrm{R}^2 =\ 0.996.\ \text{Descending Decreasing RH} \quad (7)$$

The maximum relative error due to the hysteresis between eq. 6 and 7 and experimental data is less than 2% up to 70% RH and below 4% at higher RH values.

An important design aspect in of sensors design is the hysteresis of the device. The maximum shift between curves with increasing RH steps (represented as “UP” in Fig. 8**¡Error! No se encuentra el origen de la referencia.**) and with decreasing RH steps (representing as “DOWN” in Fig. 8) is less than 7 fF at 1 MHz up to 70% RH and less than 10 fF from 80% RH. Therefore, the maximum absolute error associated to the hysteresis is around 2% RH.

3.4 Response time

Another important property of any sensor is how fast the sensor output (the capacitance, in our case) changes when there is a variation in the input, which in our case is the relative humidity.

The dynamic response of the sensor is depicted in Fig. 9 and shows a high stability along different measurement cycles and over time. The output of the printed sensor is presented together with that of a commercial sensor and their results are comparable. Defining the response time as $t = \tau$, it would correspond to the 63% of the maximum value of capacitance (reached at equilibrium) for every increasing step of RH presented in Fig. 9. Although the sensor does not reach the steady state in these cycles, their behaviors can be adjusted by exponential curves; we estimated the response time from the adjusted curves. This time is equal to 356 ± 3 s. Additionally, the response time for desorption, τ', can be defined as the time associated to the 37% of the maximum value of capacitance for every decreasing step of RH; this value is 367 ± 4 s. These long response times can be explained in terms of different diffusion rate in our solid substrate compared to the deposited layers in other IDE structures [13, 14]. The printed sensors can hardly compete in terms of response time with commercial CMOS-based humidity sensors (SHT15, Sensirion AG, Switzerland) with times smaller than 10 s. This lower performance in response time does not hinder the use of these printed humidity sensors due to the fact that humidity is an environmental property, which often changes gradually.

In any case, the response time is also directly related to the square value of the thickness of the sensitive layer [26]. Then, this time can be reduced by using a thinner substrate. Anyway, this sensor as it is presented here could be useful for environmental humidity monitoring, where changes are usually gradual and slow. Harrey et al. (2002) [27] developed a parallel-plate capacitive sensors capacitor sensor structures with a number of humidity sensitive polymers including polyimide (Kapton HN) and polyethersulphone (PES). Furthermore, they showed the improvement in time response by using thinner sensitive layers. In that study, the times achieved varied from 5 min to 10.5 min for Kapton HN depending on the thickness of the film.

3.5. Time Stability

The sensors have been measured once a week for more than 6 months and data show a small maximum variation of ± 9.3 fF of the average value. In order to estimate the aging drift, we did a stability test 5 months after its fabrication. After the fifth month, the humidity sensor was tested for 10 days at fixed relative humidity (30%) and controlled temperature (30 °C) every 6 hours. The aging drift was less than 3% RH, which is within the time drift specification of the used climatic chamber.

4. Conclusion

In this paper, we have designed, modelled, manufactured by inkjet printing, and characterized a small and low-cost humidity sensor that can be easily and quickly fabricated and integrated into RFID tags. The sensing element of this sensor is the substrate whose electrical permittivity is directly related to the relative humidity in the environment. Furthermore, the fabrication process is reduced not only because no extra sensing layer is needed, but also because only one printed layer is required to define the interdigitated capacitor.

This sensor only requires less than 12 mm^2 of Kapton HN and one layer of SunTronic U5603 ink (about 23 nl for one silver layer). Experimental data are in good agreement with results extracted from COMSOL Multiphysics, validating the extrapolation procedure for very thin printed layers. The proposed sensor shows a stable humidity response from 100 kHz to 10 MHz; its sensitivity is (4.5 ± 0.2) fF/%RH at 100 kHz and (4.2 ± 0.2) fF/%RH at 1 MHz.

Furthermore, a very low thermal drift has been obtained in a wide frequency range. The capacitance shows a thermal coefficient of around (-0.2 ± 0.2) fF/ºC at 1 MHz whereas this coefficient has a value of (-0.4 ± 0.2) fF/ºC at a frequency of 100 kHz. The relative humidity sensitivity is more than 11 times greater than thermal drift at 100 kHz and 21 times at 1 MHz. This result means that the compensation of temperature can be avoided, if the frequency of work is properly chosen. The time response of the sensor is about 6 min but this value could be improved by reducing the thickness of the substrate. Finally, the sensor response has hardly changed as a consequence of aging effect. This manufacturing process not only requires a small amount of materials but also a short time of fabrication with a high rate of success.

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