Development of a Capacitive Humidity Sensor for Physiological Activity Monitoring Applications

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Abstract

MEMS sensors and their circuitry are small and inexpensive to produce. If worn on the clothing, a relative humidity sensor can monitor sweat levels of highly active persons, such as athletes and soldiers, and can provide a real time diagnostic tool for health professionals. Capacitive humidity sensors with collagen-based dielectrics were designed, fabricated, and tested for use in physiological activity monitoring. The devices utilize interdigitated electrode geometry with a dielectric sensing medium composed of a gelatin thin film. Thermal PVD was used to deposit a layer of copper onto a glass substrate. Positive photolithography along with a wet chemical etch were used to develop the electrodes. A liftoff technique was then used to place the gelatin thin film between the electrodes. Fabrication was optimized to support the 20 μm features of the photolithography masks and a 180 nm thick gelatin thin film.

Using a controlled relative humidity chamber, the precision and accuracy of the humidity sensors were tested. Characterization was determined by two tests. A "High-Low" test determined the accuracy of the devices while a "Step Test" was used to gauge consistency. In the High-Low test, the capacitance was measured for twenty minutes at 50% relative humidity, twenty minutes at 90% relative humidity, and again at 50% relative humidity. For the Step Test, the capacitance was measured for five minutes at 50% RH, 70% RH, 90% RH, and again at 70% RH. The devices operate at 36^0 C, and have been tested between 50% and 90% relative humidity. The range of capacitance is between 2 and 5 pF.

1. Introduction

Micro Electromechanical Systems (MEMS) is a branch of engineering that deals with the development of micro scale devices that can be fabricated and packaged onto a small chip to reduce size, power consumption and cost of conventionally produced devices. MEMS devices are everywhere in today's society, from pressure and temperature sensors in cars, to the touchscreen on a smartphone, to the gyroscope and accelerometer in a GPS. Sensors developed using MEMS technologies have been mass produced since the 1970s. Physical sensors, such as

pressure sensors and accelerometers, have been the most commercially successful types of sensors produces. However, there is an increasing demand for gas and chemical sensors[1].

MEMS fabrication techniques were applied to optimize capacitive humidity sensors built specifically for physiological monitoring. If worn on the clothing, a relative humidity sensor can monitor sweat levels of highly active persons, such as athletes and soldiers, and can provide a real time diagnostic tool for health professionals. A relative humidity sensor is an essential component of gas sensors, as it is the mechanism used to determine the concentration of different chemicals. The ideal capacitive humidity sensor would have a continuous linear relationship between capacitance and relative humidity that can repeatedly reach an instantaneous steady-state value for a given humidity. Gelatin was chosen as the sensing material for this application as an alternative to more costly materials because it is inexpensive and readily available[2].

The two most popular sensing mechanisms for MEMS sensors are resistance and capacitance, where a sensing medium alters the electrical property of the device, which can then easily be measured[1]-[3]. MEMS relative humidity (RH) sensors can be divided further into four major groups: capacitive, resistive, optical and mass-sensitive; however capacitive RH sensors are by far the most popular. The signal conditioning circuits required for capacitive sensors are cost effective and simple to implement once the RH sensor has been developed[3]. The basic form of a capacitive RH sensor consists of a dielectric sensing material sandwiched between two conductive plates. The capacitance is given by Equation 1, where C is capacitance, A is the surface area of the plates, d is the distance between the plates, and ε_0 is the electric permittivity of free space. As water is absorbed by the sensing material, the relative permittivity ε_R changes as predicted by Equation 2, where v is the fractional volume of water absorbed in the dielectric, and where ε_w and ε_p are the electric permittivity of water and the sensing material respectively[2]-[3]. Numerous electrode designs have been implemented to increase the performance of RH sensor, but most are derivative of either parallel plate or interdigitated electrode structures. Some modifications to the basic form include: etching the substrate to increase direct electric field lines through the dielectric, thus increasing sensitivity; finding ways to increase the thickness and surface area of the dielectric to improve response time and sensitivity; and the implementation of a heater to improve response time and reduce hysteresis[4]-[6].

$$C = \varepsilon_R \varepsilon_0 \frac{A}{d} \tag{1}$$

$$\varepsilon_R = \left[v\left(\varepsilon_w^{1/3} - \varepsilon_p^{1/3}\right) + \varepsilon_p^{1/3} \right]^3 \tag{2}$$

One apparent challenge in designing a capacitive humidity sensor was overcoming the effects of the dielectric sensing material's volume change as it absorbs water. For traditional parallel plate capacitors this volume change has an adverse effect on the device's capabilities: while the dielectric permittivity of the device increases due to water absorbed, the distance between the electrodes also increases —hence reducing the effectiveness of the device[6]. Therefore, to

optimize the design of the sensor, the dielectric's swelling effects on capacitance must be minimized. Coplanar capacitor designs with interdigitated electrode (IDE) structure are used in this research to maximize the surface area of the device while allowing the dielectric to swell up rather than out. Both comb-shaped electrodes of the IDE capacitors are on the same substrate with the "fingers" separated by a gap of the patterned dielectric. Nine IDE designs with gap and finger widths ranging from 10 µm to 160 µm were printed onto high resolution photomask transparencies. Two devices of each of the 9 unique designs fit onto one 1"x1" sample.

2. Fabrication

The gelatin used as the sensing material was prepared on site using dry, locally purchased food grade gelatin and deionized water. For our devices, the gelatin was to be spun onto the substrate and patterned using a liftoff technique. Therefore, in order to produce a reliable gelatin thin film process, the film thickness as a function of spin speed was characterized for a range of gelatin solution concentrations. The substrate chosen for these devices were 3"x1" glass microscopy slides. The mixture concentrations examined were 1:10, 1:15, 1:20, 1:40 and 1:80 grams gelatin to mL water. Each sample of gelatin was prepared with 30 mL of water and an appropriate amount of gelatin to match. For every sample two trials were tested at 1000, 2000, 3000, and 5000 RPM. Every trial was spun for 20 seconds. The results of the characterization are shown in figure 1.

The substrate was washed with water and acetone and dried using laboratory towels. Scotch tape was used to forego a time-consuming photolithography process to mask the substrate. On a flat surface the substrate was taped down across its shorter edge with approximately 30 mm of scotch tape. The excess tape along the edge that held the substrate to the table was removed using a razor. The gelatin and water were measured for the desired concentration then mixed in a 150 mL beaker. The beaker was then placed on a hot plate set at 110° C. The time required for the gelatin to fully dissolve varied between 10 and 20 minutes, depending on the concentration. Once ready, the hot plate temperature was reduced to keep the solution concentration from increasing. For each trial the solution was stirred for at least 20 seconds before spin coating to ensure a uniform temperature throughout. Between 2.0 and 2.5 mL of gelatin solution was applied to the substrate. Previous research showed that the gelatin had a tendency to mound on the center of the substrate where it was applied[2]. To prevent this from occurring, the solution was applied after the spin processor began accelerating. The gelatin was then allowed twenty minutes to cure. The tape was removed and then the film thickness was measured using a Dektak IIA surface profilometer.

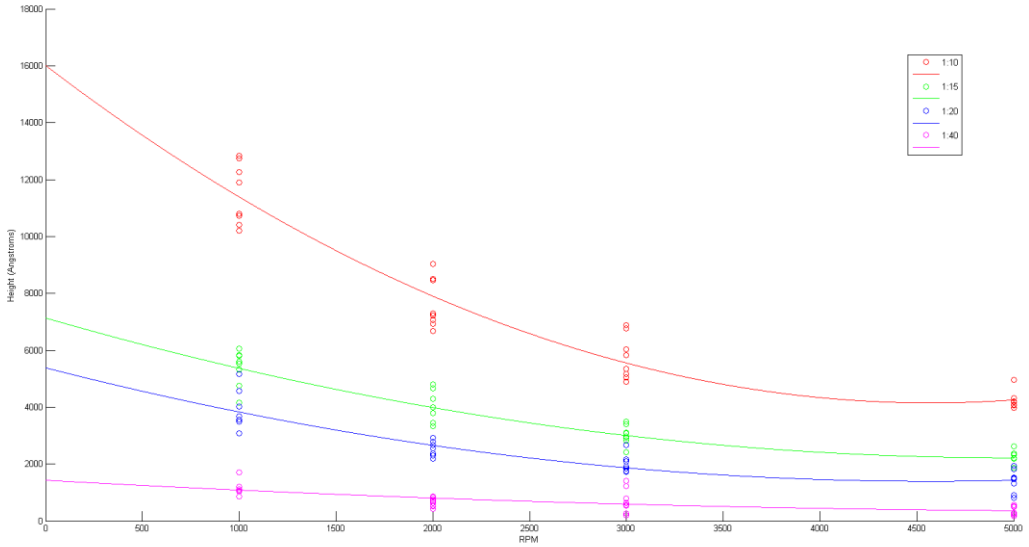

Figure 1. Gelatin thickness characterization as a function of spin speed, with the spin time kept constant at 20 seconds.

Physical vapor deposition (PVD) was used to deposit a thin film of copper onto the glass substrate. Prior to the deposition the slides were rigorously cleaned. They were first boiled in detergent for twenty minutes. They were then boiled in acetone for ten minutes, and then set in a 50% hydrogen peroxide and water solution for 5 minutes. Between each step the substrates were rinsed with DI water and placed in an ultrasonic bath for five minutes. They were then covered to prevent dust from accumulating and allowed to air dry overnight. The 95 nm copper thin film was applied on site using thermal PVD at a rate of 1.75 Å/s. The metallized 3”x1” substrates were cut into 1”x1” squares for further processing.

The metallized 1”x1” substrates were cleaned by rinsing them in DI water, acetone, methanol, then again with DI water. The spin coater was used to process the substrates with photoresist. First, approximately 1 mL of Hexamethyl Disilizane (HMDS) primer was applied to the substrate and spun at 2000 RPM for 20 seconds. After standing for 15 seconds to allow the HMDS to fully dry, S1813 photoresist was applied and spun at 4000 RPM for 50 seconds. The substrate was then moved to a 110° C hot plate and soft baked for 90 seconds. The samples were then exposed for 4.5 seconds using an HTG 3000HR Mask Aligner. After exposure, the samples were soft baked for 210 seconds at 110° C. During this time the gelatin and developer solution were prepared.

The developer solution was made by mixing 5 mL of 25% Tetramethyl Ammoniumhydroxide (TMAH) with 50 mL DI water in a 200 mL plastic beaker. Two 200 mL plastic beakers were filled with DI water and were used as baths to quickly rinse the samples after developing. Each sample was placed in the developer and slightly agitated for approximately 130 seconds,

continually monitored to prevent overdevelopment. Once developed, it was rinsed with DI water then consecutively placed in each bath. The sample was then allowed to air dry for 10 minutes. Excess water was evaporated by placing the samples on the hot plate for less than a minute. An Olympus PME optical microscope was used to quickly examine the edges of the photoresist to ensure the samples were ready for etching.

The etchant was made by mixing 100 mL of DI water with 25 grams of Sodium Persulfate in a 150 mL beaker which was placed on the hot plate and heated to 50° C. The solution was transferred to a glass petri dish which allowed quick retrieval of the samples after they were fully etched. The samples were individually placed in the solution for approximately 10 seconds. Discretion was used to determine when the samples were fully etched. The samples were then rinsed with DI water and placed on a hot plate to dry. The Olympus microscope was used once again to determine the quality of the samples.

To pattern the gelatin, the photoresist (PR) layer from the copper etching process was kept on the samples. Photoresist is soluble in acetone whereas gelatin is not. Therefore the PR pulls the gelatin off the copper electrodes while the PR dissolves, leaving walls of gelatin between the copper electrodes. From the previous gelatin characterization it was determined that a 280 nm film of gelatin could be applied to our samples using a 1:20 concentration spun at 2000 RPM for 20 seconds. Using these process parameters the gelatin thin film was applied. The samples were then placed on the lab bench until completely dry. The liftoff was accomplished by filling a 150 mL beaker with approximately 50 mL of acetone. The samples were placed in the beaker, and the beaker was placed in an ultrasonic bath for 40 seconds. The samples were then rinsed again with acetone to ensure gelatin fragments did not stick to the devices.

3. Preliminary Testing

The devices were given a two minute preliminary inspection prior to rigorously testing them in the humidity chamber. The devices were tested for room temperature capacitance (RTC) and instantaneous response (IR). The RTC was measured with a digital multimeter to quickly determine whether the devices were successfully fabricated. If the multimeter read a resistance rather than capacitance then the device was clearly a failure. The instantaneous response was measured by breathing onto the sample while measuring its capacitance. This allowed us to determine which devices responded well to humidity changes. The results were compared to an air sample, a sample that was not processed with gelatin, to see which devices performed best. This two minute inspection allowed us to determine the yield of our fabrication process per device type. The two minute inspection also was the basis for determining which devices to test further. Generally, if the IR was an order of magnitude greater than the RTC, the device was further tested for consistency and accuracy. This reduced the list of over 100 working devices down to 10 devices that were worth testing.

3.1 Testing.

Two tests were used to examine the functionality of gelatin as a sensing material for capacitive humidity sensors. Both tests were completed using a Blue M Vapor-Temp Controlled Relative Humidity Chamber with the temperature set to 36° C. The humidity was recorded using a Nomad Omega OM-73 Temperature/Humidity Data Logger[7] which was placed alongside the sample in the chamber. The devices were prepared for testing by using conductive silver paint to connect wires to the electrodes which were then connected to the GLK Model 3000 Digital Capacitance Meter[8] using alligator clips. The capacitance meter was then connected via USB to a computer which recorded the capacitance at a rate of 4 Hz. The commercial sensor's onboard recording was then uploaded to the computer after the test to compare the relative humidity (RH) with the sample's capacitance.

The first test was designed to measure if the devices can generate a unique capacitance for a given humidity level. This test is referred to as the "High-Low" test. The chamber humidity was set to 50% RH for the first twenty minutes, 90%RH for the next twenty minutes, and then 50%RH again for the final twenty minutes. The extended cycle times ensure that the device can settle on a capacitance for that given humidity.

The second test measured how consistent the device is at generating a capacitance for a certain humidity level after changing from lower or higher humidity levels. This is referred to as the "Step Test." The test is broken into three repeating twenty minute cycles. Each cycle consists of four, five-minute steps of humidity. The first step is 50%RH, the second is 70%RH, the third is 90%RH, and the fourth is 70%RH. The repeated humidity levels allow us to determine whether or not gelatin based humidity sensors can display the same range of capacitance as previous measurements taken at the same humidity.

Results & Conclusion.

Figure 2a shows an example of a High-Low test performed on a device with 80 µm finger width, 20µm gap width, and 2000 µm finger length with patterned gelatin 280 nm thick. The capacitance generally follows the chamber's humidity shifts. The relationship is nonlinear, and although the capacitance level during the second 50% RH cycle settles to a limited range of values, it inconsistent with the first 50% RH cycle. This is evident Figure 2b, where capacitance is plotted as a function of humidity: there are two definite lines fit between 58%RH and 67%RH.

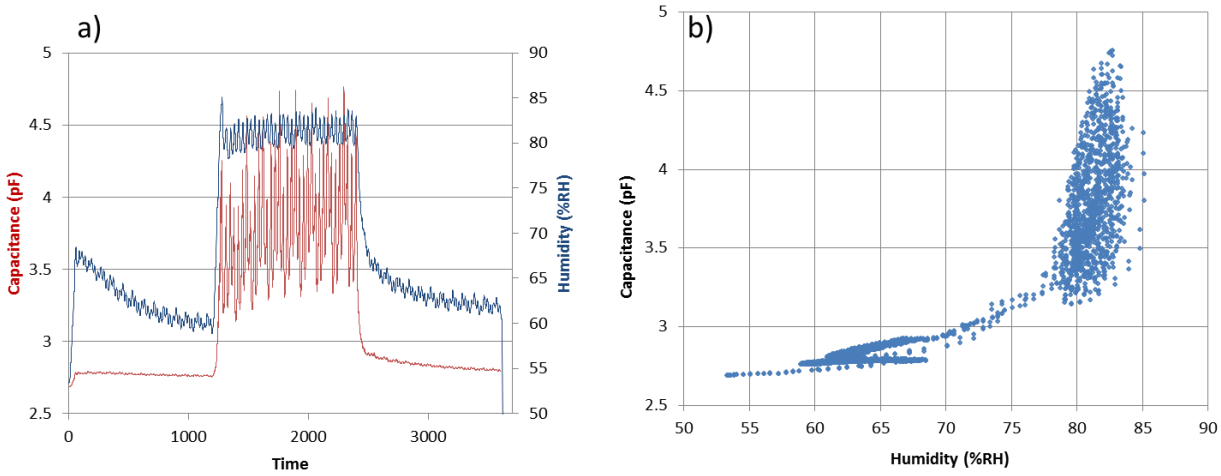

Figure 2. High-low test plotted (a) with capacitance and humidity as a function of time and (b) capacitance as a function of humidity.

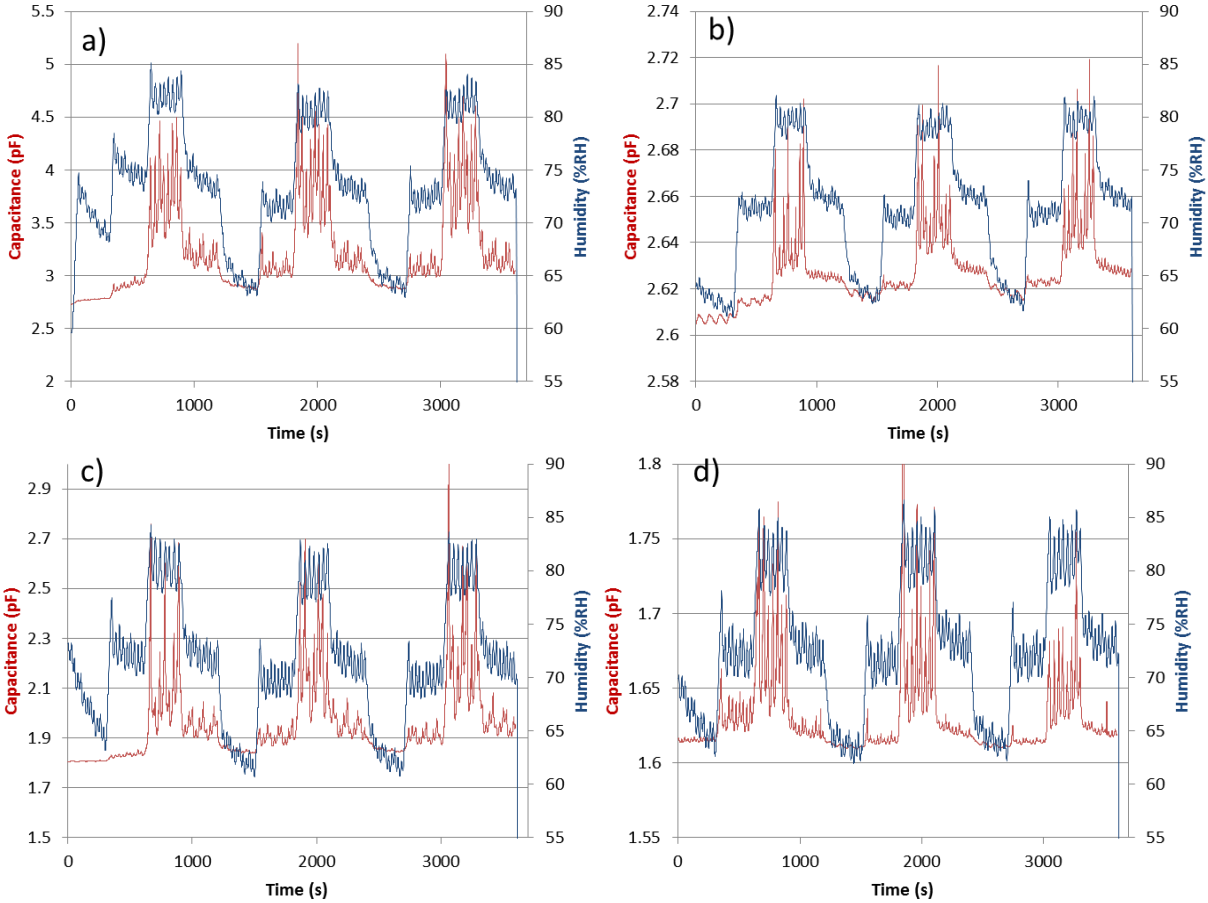

Figure 3. Step tests performed on the same device with (a) and without (b) a gelatin sensing film. The step test was performed on another IDE device with (c) and without (d) the gelatin film.

The results of the step test are shown in Figure 3. The same devices were tested with and without gelatin. This was done by simply washing the sensing layer off of the devices with warm water. The range of capacitance and shape of the plots are two notable features. Figure 3a depicts the results from the device with gelatin and Figure 3b shows the results from the device without gelatin. Superimposed on one another, (a) and (b) appear to have the same general form: a larger variance in capacitance at higher humidity levels, higher average capacitance values for higher humidity levels, and the same response time to humidity changes. The only noticeable difference between (a) and (b) is the range of capacitance. Figure 3a has a 2.73pF minimum capacitance and 5.14pF maximum capacitance, with an average 50%RH step value of 2.9pF, average 70%RH step value of 3.1pF, and average 90%RH step value of 3.8pF. Figure 3b has a 2.60pF minimum capacitance and 2.72pF maximum capacitance, with an average 50%RH step value of 2.619pF, 70%RH step value of 2.623pF, and 90%RH step value of 2.65pF. The procedure was repeated for a device with different IDE dimensions and the results are shown in Figure 3c—with gelatin, and (d)—without gelatin. Again both graphs exhibit the same response to the chamber's humidity changes except that the device with gelatin shows a much greater range of capacitance values, indicating a much greater sensitivity to humidity changes.

The ideal capacitive humidity sensor would have a continuous linear relationship between capacitance and relative humidity. The results from our testing have shown that the response of gelatin based capacitive humidity sensors to shifts in humidity mirrors the response of a commercial sensor. The sensitivity to relative humidity changes is much greater to the sensitivity of an identical device without the gelatin thin film. The High-Low tests show that while gelatin can respond quickly to environment changes, the devices do not consistently approach the same capacitance for a given humidity. The inconsistency in precision is also evident in identical devices without the sensing film. Therefore, the next step of the development of gelatin as a sensing material for capacitive humidity sensors would be to explore different electrode designs in order to increase the device's precision.

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