Miniaturized two-level Controller based on moisture-sensitive Hydrogels

A. Steinke, Th. Frank, A. Cyriax
CiS Forschungsinstitut für Mikrosensorik und Photovoltaik GmbH
D-99099 Erfurt, Germany
asteinke@cismst.de; tfrank@cismst.de; acyriax@cismst.de

C. Bellmann, G. Gerlach
Solid-State Electronics Laboratory
Technische Universität Dresden
D-01069 Dresden, Germany
christian.bellmann@tu-dresden.de; gerald.gerlach@tu-dresden.de

R. Sarwar, H. F. Schlaak
Laboratory of Microtechnology and Electromechanical Systems
Technische Universität Darmstadt
D-64283 Darmstadt, Germany
r.sarwar@emk.tu-darmstadt.de; schlaak@emk.tu-darmstadt.de

***Abstract*— In this paper, we present a novel binary threshold sensor, which is able to use the energy provided directly from the measured relative humidity of the ambient air to mechanically switch an electrical micro contact. This zero-power switch behavior is realized by using the humidity-sensitive volume swelling of a polymer layer as the detection element deflecting a mechanical deformable silicon boss structure, thus closing the electrical contacts of the switch. For the humidity-sensitive sensor switch considered here, a hydrogel blend of 15 wt% poly(vinyl alcohol) (PVA) and 7.5 wt% poly(acryl acid) (PAA) was used. According to first swelling experiments with a prototype the provided deflection of approximately 36 µm of a 20 µm thin silicon flexure plate seems very promising.**

Keywords: binary sensor switch; relative humidity; zero-power; boss structure; hydrogel (PVA / PAAs)

I. Introduction

The demand for improved processes in industry and the personal environment of humans requires better sensors. As a consequence, sensor industry has been growing in average by 8 % per year since almost three decades. Remarkably, 70 % of all sensors in process control and more than 90 % in building automation are sensors switches acting as threshold switches when changes of particular conditions require dedicated actions. For these purposes, sensors are required, which binarily switch between two pre-defined states. Most of the commercial sensors related to this task are based either on a resistive, a capacitive or an optical measurement principle [1]. Their advantages are a low response time, a high accuracy and a continuous measurement. Unfortunately, they need an electrical processing circuitry as well as an external energy supply to monitor the desired parameter continuously (Fig. 1).

This paper describes another approach for the two point control of relative humidity as water vapor concentration in air and the reduction of the system complexity (compare Fig. 1 with Fig. 2).

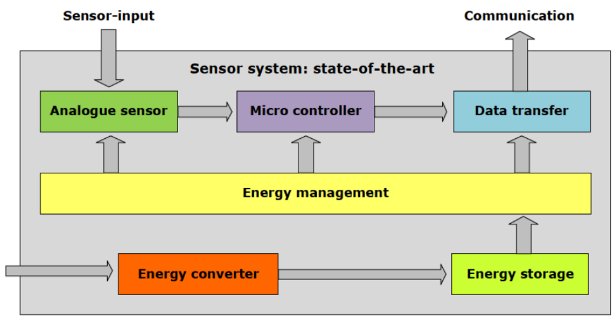


Fig. 1. Signal system of a sensor switch corresponding to the state of the art [7].

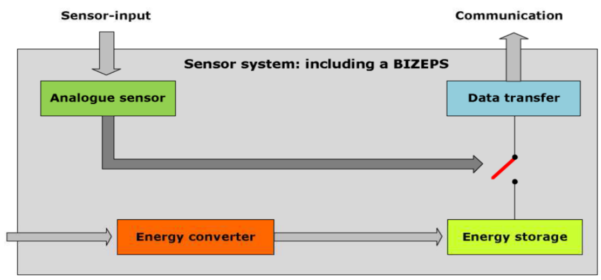


Fig. 2. Signal system of a sensor switch based on the BIZEPS™ platform [7].

 ISBN: 978-1-61208-297-4

We propose a binary zero-power sensor (BIZEPS™) (see Fig. 3) where the humidity causes a transducing element (here a humidity-sensitive hydrogel) to swell. This swelling provides, in contrast to existing solutions, the energy to trigger mechanically an electrical contact. As long as a defined threshold is not reached, the electrical micro contact remains open. The advantages of such polymers are their easy processing technology, their low costs, the adequate availability and the possibility to tailor their properties. The effect of polymer swelling is already used in bimorph sensors [2], biosensors [3] and in sensors for reflectometric interference spectroscopy [4].

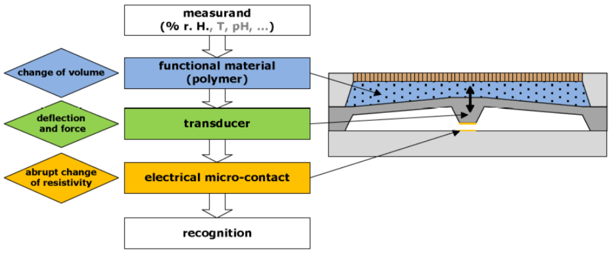


Fig. 3. Operation principle of a binary sensor switch based on the BIZEPS™ platform.

For our intention, two swelling properties can be used. The approach with the largest sensitivity uses the volume increase of the polymer (type B). Due to the absorption of water molecules the polymer swells but remains incompressible. By restricting the free space one can use the resulting high swelling pressure. The second approach uses the bimorph effect, which is based on two connected material layers with different linear expansion coefficients (type A). The expansion of one of the layers due to factors such as temperature or, in our case, humidity, leads to a bending of the bimorph in one direction.

The mechanical part of the sensor switch, which is deflected by the polymer, will be realized with a silicon boss structure as construction part of a thin silicon flexure plate. It offers several advantages:

- The flexure plate shows a suitable compliance with respect to a large enough deflection for the switching movement.
- The boss structure can serve as the switching contact.
- Fast switching needs snap behavior. This can be realized by applying a preload through an oxide layer onto the silicon flexure plate to minimize the effects of the swelling hysteresis.

Due to the expected low contact forces (< 1 mN) provided by the dynamic behavior of the polymer layer, a special contact design has to be applied. Gold nanowires are a promising candidate to minimize the necessary contact force, to decrease the contact resistance and to raise the reliability [5]. They are used in our BIZEPS™ switch as backplate electrode (Fig. 4).

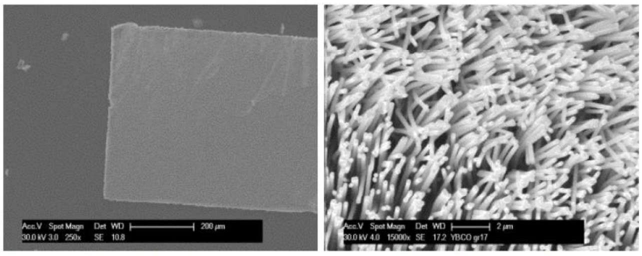


Fig. 4. SEM image (left with low, right with high magnification) of gold nanowires with a density of about 1/μm^2, 200 nm diameter, 10 μm length and an aspect ratio of 90:1 [5].

The gold nanowires are manufactured by optical lithography, galvanic processes and the use of a polycarbonate template which is described more detailed in [6].

II. Experimental

A. *Manufacturing of the humidity sensitive hydrogel*

One key element of the BIZEPS™ platform is the sensitive polymer, which interacts with the parameter to be measured (% r. H., T, pH). Here a hydrogel blend of 15 wt% poly(vinyl alcohol) (PVA) and 7.5 wt% poly(acryl acid) (PAA) in a mass ratio of 4:1 is used. A polymer layer of approximately 20 μm in thickness (measured in dry condition) was created by filling a casting mould of poly(tetrafluorethylen) (PTFE) with the hydrogel solution. PTFE was used because of its very low adhesion force and its hydrophobic behavior. After the evaporation of the solvent, the polymer was annealed at 130 °C for at least 40 min to ensure a high crosslinking. Afterwards, the polymer layer was cut to size.

B. *Sensor prototypes*

1) *Type A – Investigation of the bimorph effect*

The investigation of the swelling pressure caused by the bimorph effect and the resulting deflection was done with the sensor configuration shown in Fig. 5.

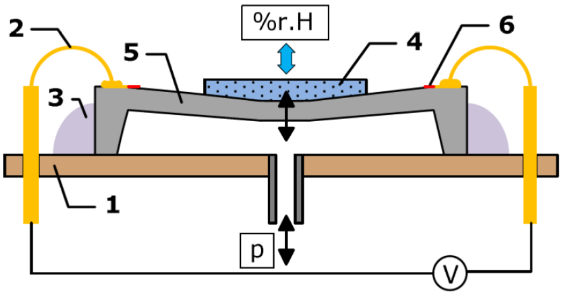


Fig. 5. BIZEPS™ sensor switch based on the bimorph effect (type A): 1. TO8-socket, 2. gold-wire bond, 3. glue, 4. PVA/PAA layer, 5. Si-chip, 6. resistance strain gauge.

A pressure sensor chip with a silicon flexure plate of 3800 x 3800 μm^2 and a thickness of 20 μm was used as basic transducer. A 3 μl drop of the hydrogel blend PVA/PAA was coated directly onto the flexure plate and annealed. It showed a circular shape with a diameter of 2.5 ± 0.8 mm. The

 ISBN: 978-1-61208-297-4

resulting thickness of the polymer layer varied between 5.5 ± 1.2 µm in the middle and 8.8 ± 1.5 µm at the edges. Afterwards, the Si-chip was glued onto a TO8-socket with a small hole which allows varying the pressure to the flexure plate. The electrical contacts between the socket and the resistance strain gauges were done by gold-wire bonding.

The sensor was placed into the conditioning cabinet (Heraeus-Vötsch HC0020) to maintain a constant temperature of about 19.8 °C for 24 hours. During this time the relative humidity φ was reduced in steps from 85 to 10 % r. H. and the voltage output $U_a(\varphi)$ of the resistance strain gauges, which were connected to a Wheatstone bridge, was measured. In the next step, the relative humidity φ was maintained at 10 % and a pressure p between 0 and 40 kPa was applied with a pressure controller (Druck DPI 510) while the resulting output voltage $U_a(p)$ was monitored.

By correlating the characteristic curves of $U_a(\varphi)$ and $U_a(p)$, the dependence $p(\varphi)$ between the pressure and the relative humidity as an approximation of the swelling pressure is obtained. The deflection dependence $w(p)$ of the silicon flexure plate was investigated with a two-beam vibrometer system consisting of a Polytec OFV3001 vibrometer controller and a Polytec OFV502 fiber interferometer. One beam was focused on the edge of the silicon chip while the other one was adjusted to the middle of the flexure plate onto a reflex strip on top of the polymer. This measurement was performed at 10 % r. H..

2) Type B – Investigation of the volume swelling

The influence of the volume swelling to a silicon flexure plate was investigated with the configuration according to Fig. 6.

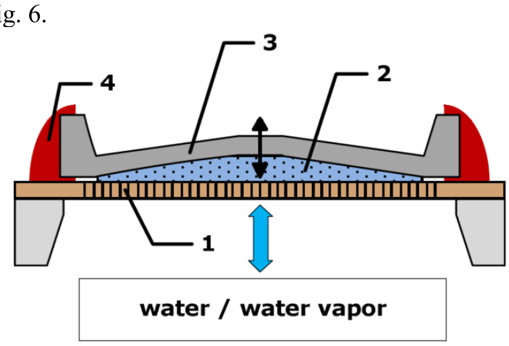


Fig. 6. BIZEPS™ sensor switch based on volume swelling (type B): 1. porous Al_2O_3 membran, 2. crosslinked PVA/PAA layer, 3. silicon chip with a 20 µm silicon flexure plate, 4. silicone glue Scrintec® 901.

For this, the prepared polymer layer was fixed between the sensor chip and a customized porous Al_2O_3 ceramic filter with a thickness of 630 µm using a silicone glue (Scrintec 901). The pores within the ceramic filter was perforated with a laser beam. The average distance between the pores is about 200 µm, their diameter amounts to 50 µm. The filter serves as mechanical restriction for the polymer in the lower direction to lead the volume swelling in the upper direction only deflecting the silicon membrane. On the other side, the porous filter allows the diffusion of the water vapor (and water) into the hydrogel to ensure the sensing function of the polymer layer.

After fabrication, several drops of water were brought into contact with the porous ceramics and the dry polymer. As a result, the water diffused as expected through the holes, and it lead to the volume swelling of the polymer layer. Due to the mechanical stiffness of the ceramic filter the silicon membrane was deflected upward, which was measured by laserprofilometry (µScan, Nanofocus) and a chromatic sensor.

III. Results and Discussion

1) Sensor type A – Investigation of the bimorph effect

The influence of the relative humidity to the hydrogel PVA/PAA coated onto a silicon flexure plate has been studied. According to the results presented in Fig. 7 the silicon flexure plate shows nearly linear deflection up to 23 µm in the range of 10 to 85 % r. H. while the approximated swelling pressure is increasing up to 38 kPa.

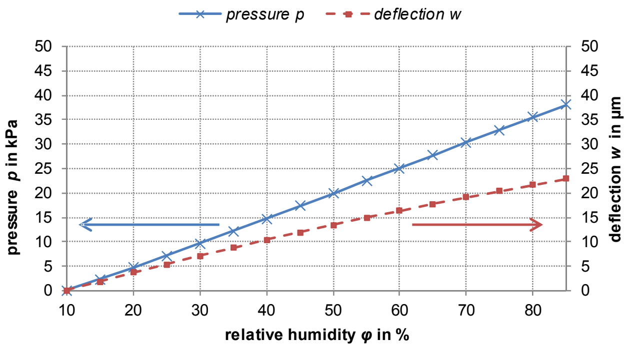


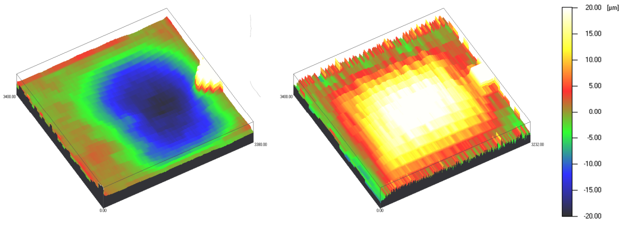
Fig. 7. Influence of the relative humidity on the deflection of a 20 µm silicon flexure plate coated with a PVA/PAA hydrogel blend for the BIZEPS™ sensor of type A based on the bimorph effect.

Because the polymer was not restricted in space during swelling this deflection is mainly caused by the bimorph effect. In detail, by raising the relative humidity, the absorbed water molecules lead to a reduction of the young's modulus and a swelling of the hydrogel fixed to the silicon flexure plate. Because of the geometrical dimensions the polymer expands more in plane than out of plane leading to the deflection of the silicon membrane.

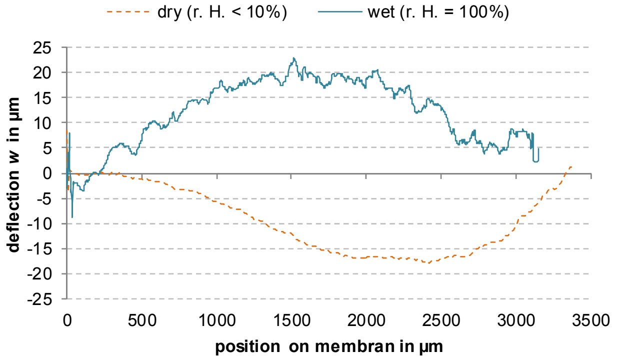
2) Sensor type B – Investigation of the volume swelling

After sensor preparation a first swelling/deswelling cycle was performed. Fig. 8 shows the resulting silicon flexure plate deflection. As can be seen a deflection change of about 36 µm occurs in the range of 10 to 100 % r. H. The initial deflection at 10 % r. H. seems to be caused by a superposition of the bimorph effect resulting from the initial swelling cycle. Nonetheless, the amount of deflection seems to be sufficient for switching devices. The porous ceramic filter operated as expected both as stiff mechanical limitation and element to allow the humidity to penetrate into the hydrogel. However, for applications in fluids, alternative materials like zeolite should be used.

 ISBN: 978-1-61208-297-4



a. b.



c.

Fig. 8. Overview of the deflection of the silicon membrane in contact with the dry polymer layer (a.) and one line-profile dry (c.) and after contact with drops of water (b.) and on line-profile wet (c.).

3) *Suitability of hydrogels for humidity sensor switches*

According to the presented experimental results the hydrogel PVA/PAA is a very promising material to provide the required forces to deflect a silicon flexure plate as needed to close an electrical contact. This could be demonstrated both for the volume swelling and for the bimorph effect. However, commercial applications need a more comprehensive investigation of the properties. In particular, several polymer-specific problems have to be considered.

First of all, the fabrication of thin polymer layers which are not coated directly onto the final surface is problematic. This regards the handling, the homogeneity of the layer thickness, the shrinking due to the evaporation of the solvent, the initial swelling and the cross-linking conditions which have to be taken into account. Spin-coating seems to be the more favored technique but did not lead to sufficient results yet, because the layer could not be removed from the used silicon wafer. So far the dipping method with the PTFE casting mould was applied. For an industrial mass production this process shows limited reproducibilty especially when the solvent content in the hydrogel blend is unknown or changingover time.

The second challenge regards the diffusion of water molecules into the hydrogel. The favored all-sided volume swelling is a very slow process.

Another problem is the temperature influence on the maximum saturation vapor pressure and, hence, on the relative humidity as well as the swelling behavior of the polymer.

Swelling and deswelling processes in hydrogels show different time constants which leads to hysteretic behavior during humidity cycles. This hysteresis can be used for the switching behavior of sensor switches but is challenging with respect to the design parameters of the silicon sensor chip and the hydrogel.

Studies on test chips with structured hydrogel of approximately 15 µm revealed that the complete drying (deswelling) is substantially longer than the wetting (swelling). The time until the switch can be switched from the line profile in figures 9 and 10 are not directly derived.

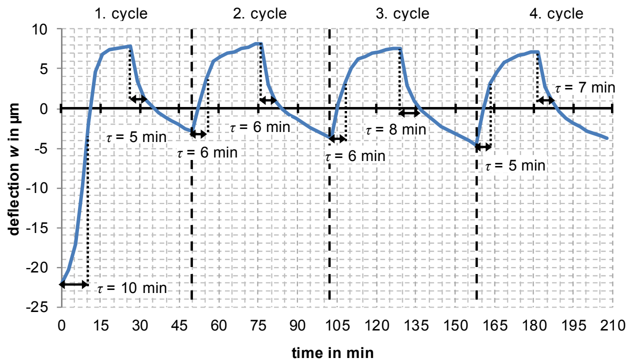


Fig. 9. Line-profil of the deflection of the polymer-coated flexural plate under cyclic wetting / dehumidification (6% ↔ 90% r. H.). Between moisture changes each time 26.3 min.

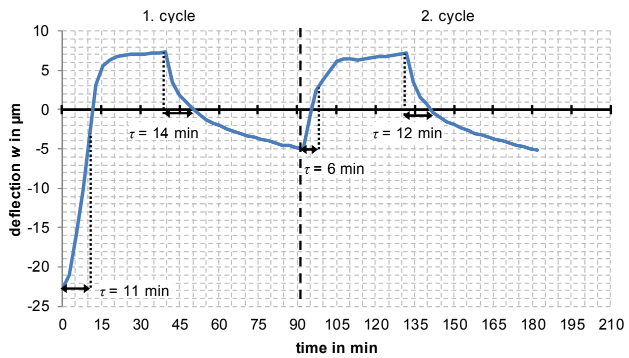


Fig. 10. Line-profil of the deflection of the polymer-coated flexural plate under cyclic wetting / dehumidification (6% ↔ 90% r. H.). Time between changes in humidity respectively 39.5 min to 52.7 min.

For this, the electrode spacing and thus the distance to be covered must be defined first. The time constant τ corresponds to the time when 63% of the final value is reached. According to the measured values of the reversibility within the displayed range is given.

IV. Conclusion

The concept of a novel binary zero-power sensor for the monitoring of the relative humidity as water vapor concentration in air was presented. The main advantage of this concept is that the continuous monitoring does not need any external energy supply. Instead, the humidity itself causes the humidity-sensitive hydrogel as basic transducing element to swell and to deflect a silicon flexure plate. Two

 ISBN: 978-1-61208-297-4

swelling-related effects were studied: (type A) the direct volume increase of the hydrogel and (type B) the bimorph effect using the swelling of a hydrogel layer deposited on aflexure plate. The experimental results showed that both effects can provide sufficient deflections of the silicon membrane up to 23 µm with the bimorph effect and 36 µm by volume swelling. A nearly linear correlation between the relative humidity and the deflection was found for the bimorph effect. The estimated swelling pressure amounted to 38 kPa in the range of 10 to 85 % r. H.

ACKNOWLEDGMENT

Financial support from Bundesministerium für Bildung und Forschung (BMBF) (SEMIS, FKZ: 16SV5506K) is gratefully acknowledged.

REFERENCES

[1] Z. M. Rittersma, “Recent achievements in miniaturised humidity sensors – a review of transduction techniques”, Senors and Actuators A 96, 2002, pp. 196-210

[2] L.-T. Chen, C.-Y. Lee, W.-H. Cheng, “MEMSbased humidity sensor with integrated temperature compensation mechanism”, Sensors and Actuators A 147, Elsevier, 2008, pp. 522-528.

[3] T. Miyata, T. Uragami, K. Nakamae, “Biomoleculesensitive hydrogel”s, Advanced Drug Delivery Reviews 54, Elsevier, 2002, pp. 79-98.

[4] D. Reichl, „Construction, characterization and optimization of an optical sensor system for the reflectometric interference spectroscopy with multicolored LEDs“ – „Aufbau, Charakterisierung und Optimierung eines optischen Sensor-systems zur reflektometrischen Interferenzspektroskopie mit mehrfarbigen Leuchtdioden“, Dissertation, Eberhard- Karls-Universität Tübingen, 2000.

[5] S. S Baek, R. S. Fearing, “Reducing contact resistance using compliant nickel nanowire arrays”, IEEE Transactions on Components and Packaging Technologies, Vol. 31, No. 4, 12.2008.

[6] S. Quednau, H. F. Schlaak, “In-situ generation of arrays of metallic micro-and nanowires in silicon microsystems” – “In-Situ Erzeugung von Arrays aus metallischen Mikro- und Nanodrähten in Silizium-Mikrosystemenen”, Mikro-Nano-Integration (GMM-FB 68) – 3. GMM-Workshop, Stuttgart, ISBN: 978-3-8007-3334-7, 03.2011.

[7] Th. Frank, G. Gerlach, A. Steinke, “Binary Zero-Power Sensors: an alternative solution for power-free energy-autonomous sensor systems”, Microsyst Technol (2012) 18:1225–1231 DOI 10.1007/s00542-012-1547-4 18.04.2012 - 20.04.2012.