

Inkjet Printed Capacitive Transducers on Flexible Plastic Substrates with Increase Stability: Ag on PET

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Abstract

Inkjet printed interdigitated transducers have been produced on flexible substrates and optimized for gas sensing applications by improving the sintering conditions and employing additional passivation procedures, like electroplating with Ni or whole transducer passivation through Parylene-C coating.

The results of the performed investigations showed that the transducers printed with Ag ink can be used, as prepared, in capacitive gas sensing structures up to 70% relative humidity only. Higher humidity levels resulted in irreversible shifts of the nominal capacitance and conductance of the transducers. This drawback could be eliminated by the already addressed passivation procedures or upgraded sintering.

The gas sensor structures based on the passivated printed transducers displayed good stability and sensing performance, demonstrating that fully printed and flexible capacitive gas sensors are feasible.

Key words: inkjet printed Ag electrodes, capacitive gas sensor, influence of humidity, plastic substrate

Introduction

Smart sensing system on foils can be used for goods monitoring, environmental pollution detection and surveillance, smart textiles manufacture, etc. All these application, at least in their mobile versions, ask for low power consumption, low cost materials and production technologies and, if possible, have to be environmentally friendly.

Comparative tests on cheap, commercially available, polymeric foils for electrotechnical use, like poly ethylene therephthalate (PET), poly ethylene naphthalate (PEN) and a polyimide (PI) showed that they are, more or less, suited for the transducer substrates[1].

The low power consumption desiderate can be achieved by using capacitive transducers. According to [2] "A transducer transforms the energy carrying the chemical information about the sample into a useful analytical signal". In the case of ideal capacitive transducers no principle dissipation comes into play.

Previous investigations on flexible integrated systems for temperature and gas sensing showed good stability of the capacitive

transducers from Pt and Au patterned by photolithography on PI [3].

In order to reduce the production costs the manufacturing of the structures has to be based on additive technologies only. Therefore the conducting elements of the new sensing systems devised by our groups have been deposited on cheap PET foils by inkjet printing as drop-on-demand technology.

Preliminary results on the inkjet printed silver electrodes, on their stability and on the influence of the electropassivation on the capacitive transducer performance have been carried out and published before [4], [5]. The performed tests, even though encouraging, revealed the need for deeper investigations on the electrode material and its interaction with the substrate and gaseous ambient.

In consequence, the main aim of the reported investigation was the thorough characterization and optimization of the printed transducers. It has been demonstrated that different levels of electrodes stabilization can be achieved by either the optimization of the sintering conditions of the "as printed" Ag-film or by several transducer passivation procedures.

Humidity, always present in the ambient atmosphere, has been used as main “test gas,” for the study of the electrodes stability.

Experimental

Inkjet printing as drop-on-demand technique has been used to directly print the electrode material (Ag nanoparticle, ANP DGP 40LT-15C) on a common plastic substrate, namely poly ethylene terephthalate (PET, Melinex® ST506). As it will be shown later on, the transducers, sintered under the conditions recommended by the ink supplier (30 min at 150°C) are not stable enough.

For improving the stability of the electrodes two approaches have been followed:

- the optimization of the sintering conditions
- the use of additional passivation steps.

The simplest passivation procedure is the supplementary electroplating of the easy oxidable Ag electrodes with Ni. Another one is the whole transducer passivation by its coating with Parylene-C monomer under high vacuum conditions. The polymer had to be, subsequently, cross-linked.

As last testing step the passivated transducers have been coated with well known gas sensing materials, such as poly ether urethane (PEUT), in order to evaluate their quality in full sensor structures [3].

The responses of the evaluated samples against gases or vapors have been recorded with suitable instrumentation (HP4263A and HP4285A precision impedance bridges) for different excitation frequencies (100 Hz to 10 MHz). Humidity (delivered in 10% incremental and decremental pulses and steps from 0% to 90% and back) has been considered the main analyte, because, on one

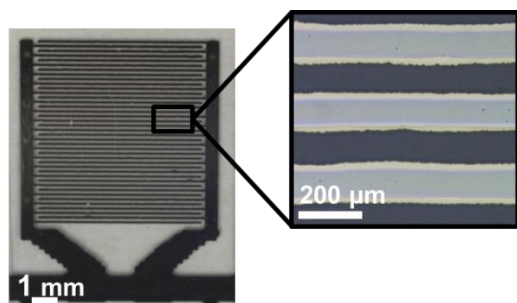


Fig.1. Optical picture of a silver inkjet-printed IDE capacitor, and magnified views of its electrodes. The width of the electrodes and the gap in between them are $95 \pm 3 \mu\text{m}$ and $105 \pm 3 \mu\text{m}$ respectively[4].

hand (as accounted in the introduction), it is always present in the ambient at high

concentration levels in respect with the other possible target gases, and, on the other hand its action on the substrate and deposited conducting films is very complex (can modify the glassy point of the polymer, favoring the corrosion of metals, etc.)

Results and Discussion

Inkjet printed transducers, sintered as proposed by the ink supplier, can be used in capacitive gas sensing applications up to 70 % relative humidity (r.h.). Higher humidity levels result in an irreversible shift of the nominal capacitance and conductance (see Fig.2) of the specimens.

Stability improvements achieved by the optimization of the sintering conditions

The Ag layer stability could be improved by increasing the dwell time in the oven. Instead of sintering for 30 min at 150°C, as recommended by the ink supplier, the samples were kept for 2 h in the oven at the same temperature (150°C).

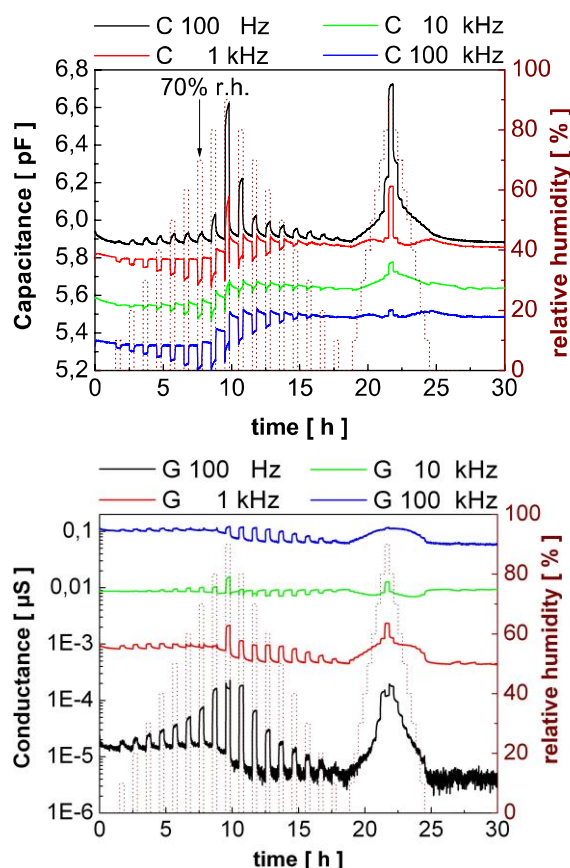


Fig.2 The capacitive (upper panel) and conductive (lower panel) responses of inkjet printed transducers (Ag only) to humidity (dot line). At 70% r.h. a shift of the nominal capacitance occurs.

After this prolonged sintering step the Ag-film quality significantly increased. Even if exposed to higher humidity (up to 90 % r.h.) the shift encountered before in the nominal capacitance

and conductance did not occur anymore (see Fig.3).

Although the irreversible shift in the nominal capacitance could be eliminated in this way, a remanent drift of the nominal capacitance and nominal conductance with the time was still present (see Fig.4). This drift has an exponential behavior indicating a possible stabilization after several weeks of operation.

Comparing the magnitude of drift in the nominal capacitance displayed in Fig.4 with the substrates sensitivity S to humidity (see Table 1, equation 1), it is evident that there is

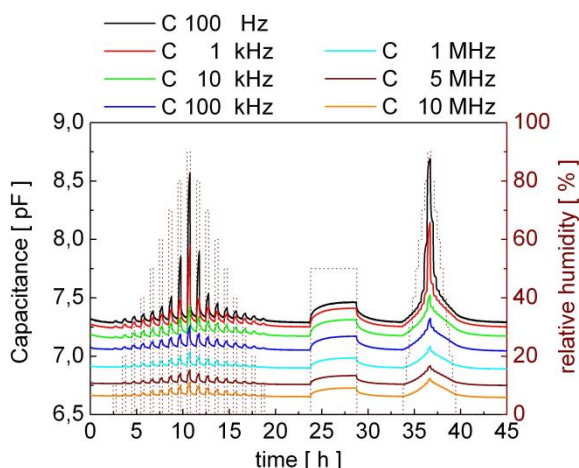


Fig.3 Response of inkjet printed transducers (Ag only) to humidity (dot line). The electrodes of the samples undergone improved sintering procedure. Nominal capacity value remains unchanged up to more than 90% r.h.

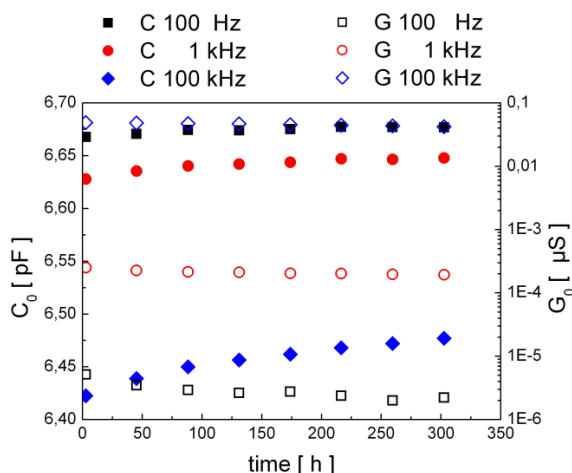


Fig.4 Drift of the nominal Capacitance C_0 (closed symbols) and nominal conductance G_0 (open symbols) for the frequencies 100 Hz, 1 kHz and 100 kHz. The experimental points are only graphic examples; the measurement was continuous.

still need for further improvement in the sintering conditions.

$$S = \frac{dC}{dc} \quad (1)$$

Table 1 Frequency dependent sensitivity S ($\Delta C / r.h.$) obtained in range of constant change in sensor response with change in analyte concentration.

Frequency	S [10^{-3} pF / % r.h.]
100 Hz	3,15
1 kHz	2,92
100 kHz	2,03

Stability improvements achieved by passivation procedures.

The second way approach for electrodes stabilization was the passivation or whole transducer passivation, as already partially reported in [4].

a. Whole transducer passivation

The performed evaluation under humidity exposure showed that whole transducer passivation with Parylene-C stabilizes the transducer impedance for ambient humidity up to 90% r.h. but increases the response and recovery times of the transducers (see Fig. 5).

b. Ag electrode passivation with Ni

Electrodes passivation with Ni stabilizes both the nominal capacitance and the transducers response (see Fig.6).

The transducer responses towards 50 % relative humidity recorded over a period of more than 4 months display good signals reproducibility which has to be ascribed to increased electrodes stability (see Fig.7).

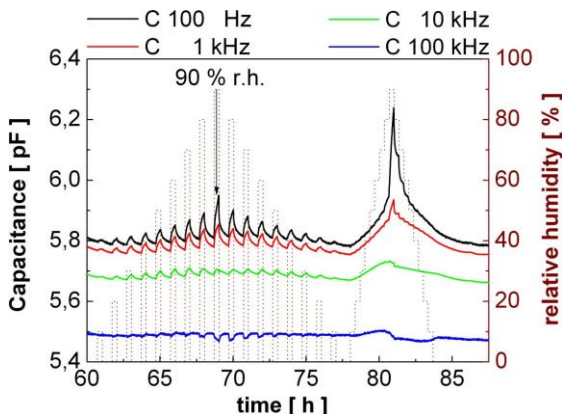


Fig. 5 The capacitive response towards humidity of the transducers passivated by Parylene C.

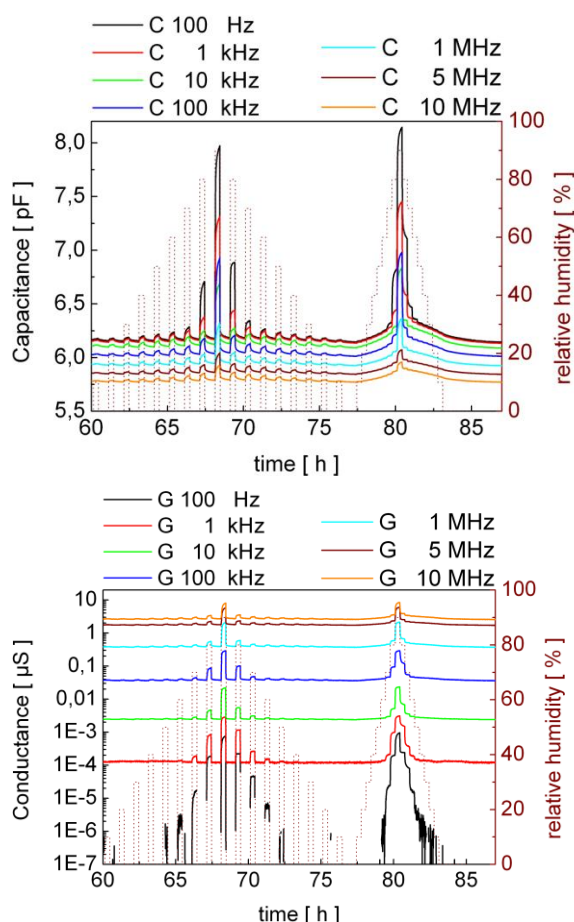


Fig.6 The capacitive (upper panel) and conductive (lower panel) responses towards humidity of the transducers passivated by electrodeposition (Ni, 1 μm).

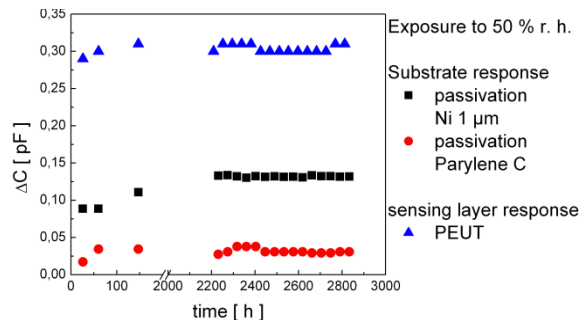


Fig.7. Middle term (more than 4 month) stability of differently passivated test specimens. Middle and lower curve describe passivated transducers alone (with Ni and respectively Parylene C). The upper curve represents the differential response of a PEUT/Ni/Ag/PET structure. It has been obtained by subtracting the response of bare Ni/Ag/PET transducer from the response of the same transducer coated with PEUT (PEUT/Ni/Ag/PET). Repetitive (at the beginning) and continuous (at the end) sample exposures to pulses of 50 % r.h.

Transducer potential for application in gas sensing systems

The investigated interdigitated transducers have been devised for capacitive gas sensors

integrated on plastic foils. It makes therefore sense to evaluate the stability of the responses provided by a standard sensing layer which was deposited on such transducers. For instance we address the case of spray coated PEUT. The spray coating (the large area "old brother" of the ink jet printing) has been chosen for its effectiveness on larger areas / low resolution films, as the sensing ones are.

The PEUT example is illustrated in Fig.7 upper curve. The displayed differential response of the capacitive sensor (PEUT/Ni/Ag/PET) has been obtained by subtracting the response of bare Ni/Ag/PET transducer from the total response of the sensor (PEUT/Ni/Ag/PET) [3].

$$C_{\text{Difference}} = C_{\text{PEUT + substrate}} - C_{\text{substrate}} \quad (2)$$

Conclusion

Capacitive transducer exclusively based on Ag inkjet printing on PET foils have been manufactured and their stability extensively investigated. The evaluations performed over more than four month demonstrated good device functionality and stability. The upgraded sintering procedures or the use of additional passivation layers enabled the increase of the humidity operation range from 70% r.h. to 90% r.h. Whole research results demonstrate that fully printed and flexible capacitive transducers for gas sensors are feasible.

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