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## Sensitivity enhancement of a flexible MEMS strain sensor by a field effect transistor in an all organic approach

D. Thuau<sup>1</sup>, M. Abbas<sup>1</sup>, S. Chambon<sup>1</sup>, P. Tardy<sup>1</sup>, G. Wantz<sup>1</sup>, P. Poulin<sup>2</sup>, L. Hirsch<sup>1</sup>, I. Dufour<sup>1</sup> and C. Ayela<sup>1</sup>

<sup>1</sup>Université de Bordeaux, CNRS UMR 5218, Laboratoire de l'Intégration du Matériau au Système, 16 avenue Pey Berland, Pessac 33607, France

<sup>2</sup>Université de Bordeaux, CNRS, Centre de Recherche Paul Pascal, 115 Avenue Schweitzer, 33600 Pessac, France

We report a low-cost piezoresistive nanocomposite based organic micro electro mechanical system (MEMS) strain sensor that has been combined to an organic field effect transistor (OFET) with the objective of amplifying the sensitivity of the sensor. When the MEMS cantilever is strained by a mechanical deflection, the resulting variation of resistivity influences the gate voltage ( $V_{GS}$ ) of the OFET and, hence, changes the drain current ( $I_{DS}$ ) of the transistor. The present combination allows an enhancement of sensitivity to strain by a factor 3.7, compared to the direct detection of resistance changes of the nanocomposite. As a consequence, a low limit of detection of 24 ppm has been estimated in terms of strain transduction efficiency. Furthermore, the organic microsystem exhibits a short response time and operates reversibly with an excellent robustness.

### I. INTRODUCTION

Continuous demand for flexible electronics technology has been the driving initiator for the development of organic devices such as polymeric MEMS and organic field effect transistors (OFET) [1]. Today, organic MEMS and OFET exhibit performances comparable to costly and brittle traditional amorphous silicon-based electronic devices. In fact, the use of low Young's modulus polymer for cantilever devices instead of conventional silicon-based materials has been reported to improve surface stress sensitivity [2]. Polymer microcantilever based MEMS have been proven to be efficient devices for physical, chemical and biological sensing applications [3-5]. One of the main challenges in the conception of successful organic MEMS concerns the integrated electromechanical transduction which converts physical quantity (i.e. strain) into an electrical measurable quantity (i.e. current). In contrast to external optical detection, integrated electromechanical transduction schemes are often preferred nowadays opening the way for the development of portable electronic devices. Specifically, piezoresistive based nanocomposite readout technique has been shown to be a sensitive and appropriate transduction method for organic MEMS sensors with sensitivities ranging from unit to hundreds [6-9]. Previous studies on piezoresistive responses of particle modified polymers have given valuable information on the parameters (type and shape of

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the fillers and conduction mechanisms) that influence the material's sensitivity [10-12]. In fact, sensitivity enhancement of the sensor as well as lowering both its limit of detection and resolution have gathered a large amount of research interest. Particularly, in the present case of strain sensing, when a force is applied to the cantilever it induces a deflection of the cantilever associated to a longitudinal strain of the material. The level of strain is often on the order of 0.001 to 0.1% making the onset of its detection extremely difficult to achieve. From these observations, our focus has been directed towards the enhancement of sensitivity and limit of detection of piezoresistive organic MEMS devices. The original principle developed in this work consists in using an OFET-based device in order to amplify the output sensitivity (gauge factor) of the piezoresistive organic MEMS sensor. To this purpose, the organic MEMS sensor has been connected to the gate of an OFET with the aim of converting the resistance variations of the nanocomposite strain sensor into a current signal, thus amplifying the sensitivity of the strain sensor utilizing the intrinsic gain of the OFET in an all organic approach.

## **MATERIALS AND METHOD**

We used Graphistrength Epoxy Master batch pellets from Arkema which consist in 25 wt% of multi-walled-carbon nanotubes (MWNTs) made via catalytic chemical vapor deposition (CCVD) dispersed in an epoxy type matrix. The nanocomposite has been prepared by mixing the SU-8 epoxy photoresist with the pellets using a high shear mixer, Silverson LART at 5000 rpm for 60 min in an ice bath. The U-shaped piezoresistive organic cantilevers were fabricated in a low-cost, two-step process. The micro-machining process used in the framework of this work does not require the high-cost semiconductor manufacturing equipment used for silicon micro-fabrication and thus, reduces drastically the fabrication cost of the MEMS devices. This process requires only a spin coater to deposit the material and a vinyl cutting machine to pattern the structure. The CNT/SU-8 piezoresistive solution was spin coated on a sheet of 100 mm thick Polyethylene terephthalate (PET) and soft baked at 95°C for 5 min. Then, like SU/8, the CNT-SU/8 thin film was cross-linked by exposure to UV (365 nm) radiation but at a higher dose (250 mJ.cm<sup>-2</sup>) combined with a post-exposure bake step at 65°C for 1 min and 120°C for 10 min followed by final hard baked of 150°C for 15 min. Afterwards, the resonators were patterned simply using a cutting plotter machine (Graphtec Craft ROBO Pro). The final dimensions of the U-shaped structures composed of a PET layer covered with the CNT/SU-8 nanocomposite were 2800 μm long, 400 μm wide and 118 μm thick (inset FIG. 1.a). The piezoresistive sensitivity of the MEMS has been electromechanically characterized. The cantilever was bent by applying a force at the cantilever's tip using a microprobe (MicroBot, Imina Technologies SA) while measuring the resistance changes of the piezoresistor. FIG. 1 shows the evolution of the resistance as a function of applied strain,  $\epsilon$ . The strain experienced by the cantilever due to an applied force at the cantilever tip is defined as:

$$\varepsilon = \frac{3h\omega}{2L^2} \quad (1)$$

With  $h$  and  $L$  being the thickness and length of the cantilever respectively, and  $\omega$  the deflection at the tip of the cantilever. The sensitivity of the MEMS sensor defined by its gauge factor (the slope of the curve) has been calculated to be 8.3 at low strain level ( $\varepsilon < 0.6\%$ ) and is in agreement with reported sensitivity values of hybrid piezoresistive MEMS sensors [9]. Previous studies have attempted to describe the complicated interplay of the different mechanisms behind the piezoresistive behavior of the CNT network [10-12]. It has been noticed that piezoresistive nanocomposites containing a CNT wt% concentration close to the percolation threshold exhibit higher sensitivity suggesting that the piezoresistive effect is essentially governed by changes of the nanotube connectivity. According to a previous study, a 2wt% CNT/SU-8 nanocomposite was chosen for the following measurements [9]. In the present case, we focused our attention on the enhancement of the sensitivity of our sensor at low strain values. At such strain level, ( $\varepsilon < 0.6\%$ ), the changes of resistivity of the hybrid nanocomposite are linear and typically modeled using a linear scaling rule. As expected, the resistance of the CNT/SU-8 thin layer increased linearly as a function of the applied strain. In this sense, we designed an experimental set-up where a piezoresistive organic MEMS has been mounted in a half Wheatstone bridge configuration and connected to the gate of an OFET as shown in FIG. 1.b. In response to applied strain the resistance of the MEMS piezoresistive transducer changed and consequently altered the gate voltage ( $V_{GS}$ ) of the OFET. As a result, the strain applied to the cantilever is correlated to the variations of the drain current ( $I_{DS}$ ) of the OFET.

The p-channel type OFET device was prepared on glass substrate. 100nm thick Al has been evaporated through shadow mask (13mm×1mm) as gate contact. 30 nm  $\text{Al}_2\text{O}_3$  layer has been formed via anodization to act as dielectric layer. 15 nm of Poly(1-vinyl-1,2,4-triazole) (PVT) was spin coated on the oxide surface as passivation layer providing high capacitance and low interface trap density [13,14]. Then, 30 nm of dinaphtho[2,3-b:2',3'-f]thieno[3,2-b]thiophene (DNNT) was thermally vacuum evaporated ( $7.10^{-7}$  mbar) as active layer at a deposition rate of  $0.1 \text{ \AA}\cdot\text{s}^{-1}$ . DNNT was chosen as it has high mobility and air stability [15]. Finally, 100 nm of Au source/drain contacts have been deposited on the active layer through shadow mask forming a channel with length of 50  $\mu\text{m}$  and width of 2.5 mm. Glass cover with UV cross-linked epoxy (NOA 61) was used to seal the device. The device structure is shown in FIG. 1.c.

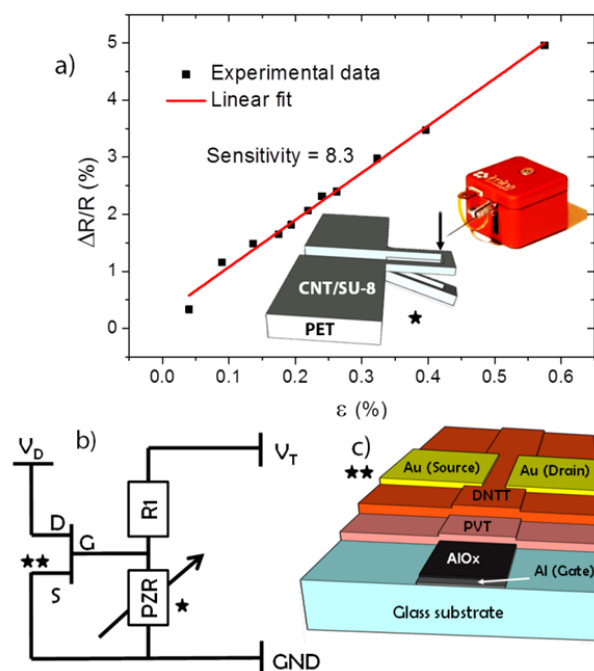


FIG. 1.a) Relative change of resistance of the piezoresistive MEMS cantilever as a function of applied strain; inset: schematic of the MEMS strain sensor deformed by tensile strain using a micro-probe, b) Experimental set-up, and c) schematic of the structure of the DNTT-based p-channel OFET device with  $\text{Al}_2\text{O}_3/\text{PVT}$  as dielectric layer

## RESULTS AND DISCUSSION

The electrical measurements have been performed at room temperature under ambient atmospheric conditions using a Keithley 4200 semiconductor parameter analyzer. It is worth noting that an optimization of the switching characteristics and the mobility of the OFET have been performed in order to get a sufficient gain, and therefore amplify the sensitivity of the MEMS. Initially, Fullerene ( $\text{C}_{60}$ ) was chosen as active layer. Nevertheless the  $\text{C}_{60}$  based devices showed an  $I_{ON/OFF}$  ratio slightly higher than  $10^2$ . Consequently,  $\text{C}_{60}$  was substituted by DNTT as active layer leading to a high current ratio over a small variation of gate voltage. These transfer characteristics make it rather sensitive and lead to an amplification of the strain sensitivity of nearly a factor 4. Transfer ( $I_{DS}$  versus  $V_{GS}$ ) and output ( $I_{DS}$  versus  $V_{DS}$ ) characteristics of the present OFET have been measured as shown in FIG. 2. The sealed device exhibited excellent stability against ambient and repetitive testing conditions. Transfer characteristics were identical after 3 days in air (supplementary information). Almost no hysteresis was observed, a prerequisite for sensing applications. The OFET showed low gate leakage current and good switching characteristics ( $I_{GS} = 1.10^{-11}$  A at  $V_{GS} = -10$  V and  $I_{ON/OFF} = 10^6$ ). The gate leakage was in the range of tens of picoamperes validating the choice of  $\text{Al}_2\text{O}_3/\text{PVT}$  as dielectric layer. The output characteristics of the OFET exhibited a linear increase in

the drain current at low drain bias (inset of FIG. 2.a). At high drain biases, proper saturation of drain current was observed proving the good operating of the device. In linear regime, the carrier mobility at the dielectric/semiconductor interface ( $\mu$ ), can be expressed as:

$$\mu = \frac{L}{C_{diel}WV_{DS}} \frac{\partial I_D}{\partial V_{GS}} \quad (2)$$

Where  $C_{diel}$  is the gate dielectric capacitance per unit area,  $W$  is the channel width, and  $L$  is the channel length of the transistor. Here, the width and length of the transistor channel are 50  $\mu\text{m}$  and 2.5 mm respectively. The drain voltage was set to -2V and the dielectric capacitance was measured at 127 nF/cm<sup>2</sup>. A high mobility of 0.45 cm<sup>2</sup>.V<sup>-1</sup>.s<sup>-1</sup> has been obtained at  $V_{GS} = -10\text{V}$ .

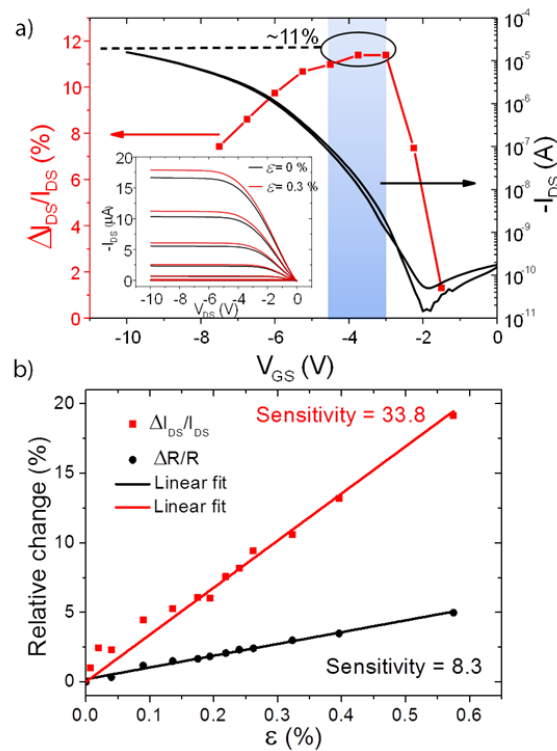


FIG. 2. a) Transfer characteristic of the OFET (black curve) and relative change of  $I_{DS}$  as a function of  $V_{GS}$  for a strain of  $\epsilon = 0.3\%$  (red curve); inset: output characteristic of the OFET between initial position and under 0.3% of applied strain at different  $V_{GS}$  ranging from -1.5V to -10V by steps of -0.75V, b) Amplification of the sensitivity of the piezoresistive MEMS using an OFET

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Similarly to the characterization of the intrinsic piezoresistive MEMS device, 0.3% of tensile strain was applied to the cantilever connected to the OFET in a half Wheatstone bridge configuration (FIG. 1.b) where the resistance  $R_I$  was chosen to be identical to the piezoresistor value (PZR) at rest, namely 6138 k $\Omega$ . This aimed to obtain thereafter the largest variations of  $I_{DS}$  of the OFET and hence the highest sensitivity. Subsequently, variations of  $I_{DS}$  for different  $V_{GS}$  were recorded in order to find the best operating point. Each relative change of drain current between the cantilever at rest and under 0.3% of applied strain have been recorded for the nine operating points and their sensitivities plotted as a function of gate voltage as shown by FIG. 2.a. The highest sensitivity has been found for gate voltage in the range -3 to -4.5V where the slope of the transfer characteristics curve is steeper. As a result, a low gate voltage of -4V was chosen for all the following measurements. Under this optimized condition, an increase by a factor 3.7 (gauge factor jumped from 8.3 to 33.8) has been observed for the sensitivity of the integrated transduction of mechanical strain, compared to the direct detection of changes of resistance in the nanocomposite, as shown in FIG. 2.b. Furthermore, the linearity of the response of the piezoresistive strain sensor has not been altered by adding the OFET. Also, a low limit of detection (LOD) of 24 ppm has been estimated in terms of strain transduction efficiency. This LOD of the strain measurement made with the MEMS coupled with the OFET is twice better than the LOD of the resistance changes of the MEMS itself. The LOD of the fabricated MEMS combined with an OFET has been estimated by calculating the standard deviation of the drain current for 1000 points at rest position. The LOD could then be determined by taking three times the standard deviation and dividing it by the sensor' sensitivity.

While in situ strain sensing capability of carbon nanotubes based composite strain sensors has been widely reported, here we present real-time measurements that are required for a large number of applications. We develop a real time sensor which can track instantaneously the force applied either by steps (FIG. 3.a) or at a constant velocity (FIG. 3.b) at any time on a sample. From an experimental point of view, this real-time sensor mounted on micro probes could be used to determine precisely the force applied to materials or devices studied during measurements.

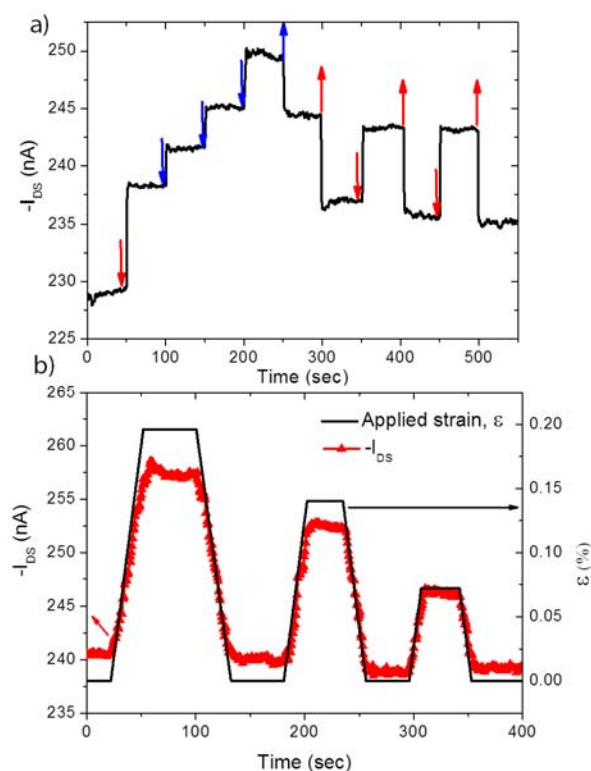


FIG. 3. Real time responses of  $I_{DS}$  as a function of time under applied strain by a) steps of 900 and 1800 ppm for blue and red arrows respectively and b) at constant speed (trapezoidal shape)

FIG. 3.a shows the drain current ( $I_{DS}$ ) of the OFET versus elapsed time for low strain level cycles. The blue and red arrows represent tensile strain of 1800 and 900 ppm while arrows pointing up or down show when tensile deformation steps have been applied to or taken away from the cantilever, respectively. The OFET has been found to display significant changes of  $I_{DS}$  that correlated well with strain level applied. When a force is applied on the cantilever, the top surface of the cantilever (where the piezoresistive transducer is located) experiences tensile strain resulting in a rapid increase of  $I_{DS}$ . Conversely, when the force is taken away from the cantilever, rapid strain relaxation occurs and thus  $I_{DS}$  decreases to reach its initial value.

The electrical current responses for the cyclic tensile strain deformation have consistent shape, where  $I_{DS}$  closely follows the steps of tensile strain applied as shown in FIG. 3.b. This result demonstrates the reversibility and repeatability of the OFET+MEMS strain sensor. It is worth noting that excellent response time (estimated below 500ms) and the absence of



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hysteresis after several cycles of tensile strain applied to the organic MEMS proves the powerful combination of these organic electronics devices as sensitive strain sensors.

## CONCLUSION

The concept of using an OFET to enhance the sensing performance of an organic piezoresistive MEMS sensor has been demonstrated. The OFET has been used to convert the resistance change signal into an amplified current signal consequently improving the sensitivity and the limit of detection of the sensor. High performance OFETs were realized with  $I_{ON/OFF}$  around  $10^6$  and mobility of  $0.45 \text{ cm}^2/\text{Vs}$  at  $V_{GS} = -10 \text{ V}$ . Cycling experiments were performed and proved the high sensitivity, reversibility and reproducibility of strain sensing. At low deformation, the drain current ( $-I_{DS}$ ) changes linearly with applied strain to the sensors which allows quantitative detection. The sensitivity of the coupled sensor and OFET was improved by a factor 3.7 compared to the direct measurement of the change in resistance (gauge factor jumped from 8.3 to 33.8) and a limit of detection as low as 24 ppm was determined for the strain. In addition, the ability to sense strain in real time with no hysteresis is very promising for biological and chemical sensing applications. The coupling of polymeric MEMS and OFET paves the way for the development of further integrated fully organic sensors.

## SUPPLEMENTARY INFORMATION

See supplemental material namely the stability of the transfer characteristics of the OFET as well as a video of the device measuring strain in real-time can be viewed.

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