Integration of Organic Field-Effect Transistor-based strain sensors to soft robotic devices and systems

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***Abstract*— The integration of flexible organic electronics in soft robotic devices is a valuable way to enhance their functionality, towards augmented controllability and performance. Nonetheless, this field is generally unexplored. Here, we report a preliminary study on the integration of soft robotic components with Organic Field-Effect Transistor-based strain sensors. Such sensors will be tested as deformation transducer for bioengineered muscle tissues operating as biohybrid actuators. Moreover, the integration of ultra-flexible devices on catheter-like soft robotic supports is discussed.**

Keywords*— *strain sensors, OFETs, biohybrid actuators, catheters, biohybrid machines.

I. INTRODUCTION

Soft robotics is attracting a huge interest for the development of novel classes of medical devices, which find applications in the biomedical field for surgery [1], drug delivery [2] and many more. Indeed, thanks to their peculiar mechanical properties, soft robots are capable to safely interact with human beings, exploiting actuation strategies based on different approaches that reduce the complexity of their assembly and operation [3]. At the state-of-the-art, several actuation strategies have been proposed, including the employment of pneumatic forces [4], chemically-driven shape variations [5] and even the employment of engineered tissues, optically [6] or electrically driven [7], in those defined biohybrid actuators. At the same time, less has been done in terms of sensor integration in soft robots for medical applications [8]. Aside of analytical and diagnostic purposes, sensorization of soft robots is fundamental to ensure a complete control of their functionality, in particular when optical access is avoided. To date, only one clear example of real-time monitoring of a biohybrid actuator has been reported in literature, based on the integration of a strain gauge sensing platform to monitor optogenetic muscle tissue [6]. Interestingly enough, the employment of organic electronics in this field is still substantially unexplored, although organic materials have great potentialities in the development of sensors by means of large-area, cost-effective techniques and on flexible and stretchable substrates. In particular, the development of strain sensors has been explored by several groups [9], and applied to fields related to soft robotics, such as prosthetic skins [10]. In this paper, we discuss about the feasibility of integration of Organic Field-Effect Transistor (OFET)-based strain sensors to biohybrid actuators and soft-robots, towards the development of innovative instruments capable to be operated inside the human body. The capability of such sensors to transduce the mechanical deformation induced by an electrically-stimulated, 3D-bioengineering muscle tissue in different conditions will be demonstrated. In parallel, the possibility of fabricating such sensors by large-scale processes, compatible with a future industrial-scale up, will be considered. In order to meet flexibility requirements of soft robotic structures, all-organic, printed OFETs will be considered, since organic conductors shows enhanced flexibility than their inorganic counterparts. Moreover, the possibility of all-organic device fabrication over ultra-flexible substrates will be demonstrated, thus ensuring an overall suitable elastic propertiy of the sensor. The integration and preliminary characterization as strain sensor of such devices over a soft-robotic compatible catheter structure will be presented.

II. Materials and Methods

A. Fabrication of OFETs for integration with muscle cells

The OFET structure is based on a bottom gate, bottom contact configuration. A 13 µm-thick PET foil (Goodfellow) was employed as a substrate. Gate is made by aluminum (Sigma-Aldrich) by thermal evaporation and photolithography. The gate dielectric is composed by native aluminum oxide grown by thermal annealing, and a 200 nm-thick layer of Parylene C (Specialty Coating System) deposited by Chemical Vapor Deposition (CVD). Source and drain have an interdigitated layout, obtained by photolithographic patterning of a thermally evaporated gold layer (Sigma-Aldrich). The semiconductor, 6,13-Bis(triisopropylsilylethynyl)pentacene (TIPS pentacene, Sigma-Aldrich) was then drop-casted from a 1wt% solution in anisole anhydrous (Sigma-Aldrich). A 2 µm-thick Parylene C layer was then deposited by CVD, acting as device encapsulation layer. The device was then completed with two polydimethylsiloxane (PDMS) 3D printed notches used to assemble the muscle tissue on the strain sensor, fabricated by means of a Cellink's Inkredible+ 3D bioprinter from a 20:1 solution with curing agent and cross-linked at 65ºC over night.

B. Fabrication of printed, ultra-flexible OFETs for catheter integration

The ultra-flexible OFET was designed with a bottom-gate bottom-contact structure. A 250-µm-thick polyethylene naphtalate (PEN) substrate was employed as support for the OFETs fabrication. A polyvinyl alcohol (PVA) sacrificial layer was spin coated on PEN substrates, and two 13-µm-thick PET supports were laminated on the PVA film. Then, a curing step over a hot plate (90°C for 10 minutes) was performed on the PEN-PVA-PET support. A 700-nm-thick Parylene C layer, the actual substrate of the sensor, was deposited by CVD. Gate, source and drain electrodes are made of PEDOT:PSS (Clevios™ PJet HC) and patterned by inkjet printing with a SUSS LP50 inkjet printer (PiXDRO) and a 12-nozzles cartridge with a single drop volume of 2.4 pL (Samba®, Fujifilm Dimatix). A 300-nm-thick Parylene C layer was deposited as gate dielectric by CVD. TIPS pentacene (1.5wt% in anisole anhydrous) was drop-casted on each transistor. The OFETs structure was completed with a 2 µm-thick Parylene C encapsulation layer deposited by CVD.

C. 3D Bioengineered Muscle Tissue

C2C12 mouse myoblasts were purchased from ATCC and cultured in T-175 flasks. Once cells reached the 80% of confluency, they were trypsized and mixed with a custom made hydrogel. Then, this cell-laden hydrogel was mold casted in a PDMS-based circular rings and there cultured for 2 days. Afterwards, the tissue was transferred to a Petri dish containing two notches (same design as the ones printed on top of the strain sensor) for differentiation. Muscle tissues were left to differentiate for at least 7 days before performing experiments.

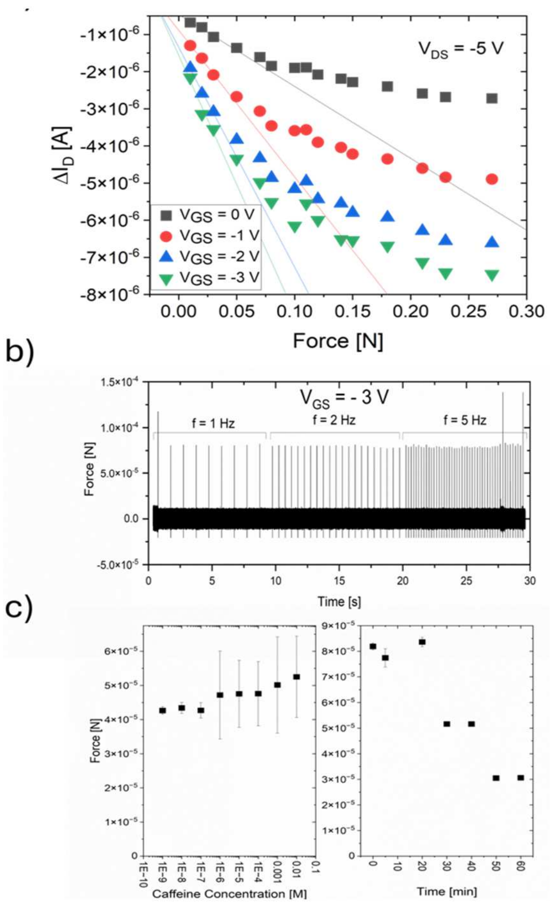

Fig. 1: a) example of sensor calibration with controlled forces at different V_{GS} values; b) force sensing on muscle tissues at different stimulation conditions; c) real-time transduction of chemical modulation of cell forces by addition of increasing concentration of caffeine, and for a fixed concentration of dantrolene (muscle relaxant) in time.

D. Catheter Fabrication

Catheter were fabricated by molding. Firstly, molds were CAD-designed and manufactured with a stereolithography (SLA) (Form 3B+, FormLabs), using the Surgical Guide Resin to improve the surface finish and quality. The molds were designed for a catheter with an internal diameter of 5 mm and a wall thickness of 500 µm. The material used to build the catheter is a mix of polydimethylsiloxane (PDMS, Sylgard 184) and Ecoflex 00-10, used with a ratio of 1:10, excluding the reticulating agent of the PDMS. The catheters were polymerized at 80 °C for 1h and 30 minutes, and left at room temperature for 2h and 30 minutes before their use.

III. Results and Discussion

A. Sensing of muscle cell contraction by means of OFET-based strain sensors

Fig. 1 shows a representative test of the OFET-based strain sensor integrated with a 3D-bioengineered muscle cell tissue. Fig. 1(a) show a calibration curve, obtained by imposing controlled device deformation by means of a dynamometer that record the applied force. Calibration curves have been obtained at different V_{GS} voltages: the more the device is switched on (V_{GS} becoming more negative), the larger the sensitivity, reported as the slope of the linear fitting obtained at low forces. For V_{GS} = -3 V, a sensitivity of $(-3.3\pm0.2)\cdot10^{-5}$

A/N was obtained over a set of 24 devices. As soon as the sensitivity was derived, the current value recorded in real time during muscle tissue operation can be directly converted into a force. Fig. 1b) report a typical sensing experiment, carried out at fixed operating conditions (V_{GS} = -3 V, V_{DS} = -5 V), and for different muscle stimulation conditions. In particular, voltage pulses with an amplitude of 30 V and duration of 2 ms were applied at the measurement environment by means of graphite-based electrodes. It is noteworthy that the sensor is capable to follow fast muscle contractions, with a repetition frequency of 1 Hz, 2 Hz and 5 Hz. To further demonstrate the functionality of the device, a chemical modulation of the muscle force was monitored, while sensor operating point and electrical stimulation were kept constant. Fig. 1c) reports an example of force modulation upon addition of increasing concentration of caffeine, which stimulate the force to increase, and upon the introduction of a fixed concentration of a myorelaxant (dantrolene, 100 µM) in time. The introduction of caffeine with a rising concentration induces the muscle cell to enhance contraction, resulting in a larger transduced force, and to start showing spontaneous contraction with variable forces (denoted by the increasing error bars). On the contrary, a high dantrolene concentration produces a continuous decrease of the contraction capability of cells, resulting in a decrease of the recorded force.

B. Electrical characterization of printed OFETs and transferring on catheters

Electrical characterization of ultra-flexible printed OFETs was carried out immediately after fabrication, i.e. for devices still on supports, after the peel-off from the support (free standing OFETs), and finally after embedding transistor on the catheter. Fig. 2 reports representative transfer characteristics of OFETs in the three different steps. It is possible to observe that only minimal variations on device performances are noticed during the peel-off and lamination process on catheters. In particular, average VTH of (0.8±0.1) V, mobility of (0.27±0.04) cm^2/Vs, and on/off current ratio of $(1.9\pm0.7)\cdot10^2$ were extrapolated for OFETs on supports (green curve). Average V_{TH} of (1.2±0.4) V, mobility of (0.12±0.03) cm^2/Vs, and on/off current ratio of $(1.2\pm0.6)\cdot10^3$ were obtained for OFETs completely transferred on catheters (blue curve).

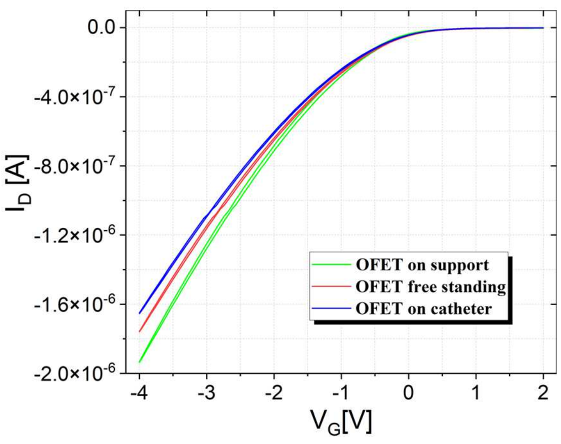

Fig. 2: Transfer characteristics of ultra-flexible printed OFET on support (green), after peel-off (red) and after integration on catheters (blue).

C. Electromechanical characterization of ultra-flexible OFETs on support and catheter prototypes

The electromechanical characterization of ultra-flexible devices has been preliminary carried out on PEN supports, since their thickness resembles the one of the catheter walls. Typical calibration curves are shown in Fig. 3a). The plots report the relative current variation $\Delta I/I_F = (I - I_F)/I_F$ with respect to the applied strain (where I_F is the current in the flat state). The deformation was applied to the OFET support in such a way that ensures the semiconductor component of the OFET is subjected to strain. The strain is ε=d/(2R), being d the substrate thickness and R the radius of curvature imposed on the substrate. The resulting strain values corresponded to measurements conducted under five different radii of 4.5 cm, 3 cm, 2 cm, 1.5 cm and 1 cm. These radii corresponded to a percentage strain range from 0.3% to 1.25%, designated as S1 to S5. Each bending radius was applied three times, following a standardized procedure: first the device was bent to the desired radius, and then the device was returned to its flat condition. Each experimental point is the average of these measurements. When an increasing strain was applied, the current decreased due to strain-induced alterations in the semiconductor lattice structure, which influenced the charge transport direction[11]. Before characterization, some devices were preconditioned, i.e. bent at the minimum bending radius. As a result of this procedure, higher sensitivity and enhanced linearity was obtained. This was likely due to the formation of microcracks in the semiconductor film, which influenced the current variation in the device during bending[12]. Ultimately, the preconditioning process enhances the device's response to strain, resulting in more pronounced changes in current during subsequent bending tests.

After this preliminary characterization, the devices were peeled off the supports and laminated onto catheters using liquid PDMS to ensure adhesion to the catheter surface. The devices were then left to dry in an oven at 65°C for one hour. Fig. 3b) reports a typical example of real-time measurements on sensorized catheters during the application of deformation. It is evident that, upon deformation, the current decreases as observed in the preliminary characterization. This demonstrates that the working mechanism of OFET-based strain sensors is effectively transferred to the catheter functionality.

IV. Conclusions

In this paper, a preliminary demonstration of the integration of flexible organic electronics with soft robotic devices for medical applications is discussed. OFET-based strain sensors were integrated with biohybrid actuators composed by 3D-bioengineered muscle tissues, showing the capability of the device to follow rapid contractions and correctly transduce force modulation obtained by exposing the cells to chemicals. Moreover, the capability of effectively integrate ultra-thin OFET strain sensors on soft robotic-compatible catheter structures is reported, showing device robustness upon transfer, and that sensitivity to deformation applied to the catheter is obtained. These results represent a step forward towards the development of biohybrid machines with augmented controllability for surgical and drug-delivery applications.

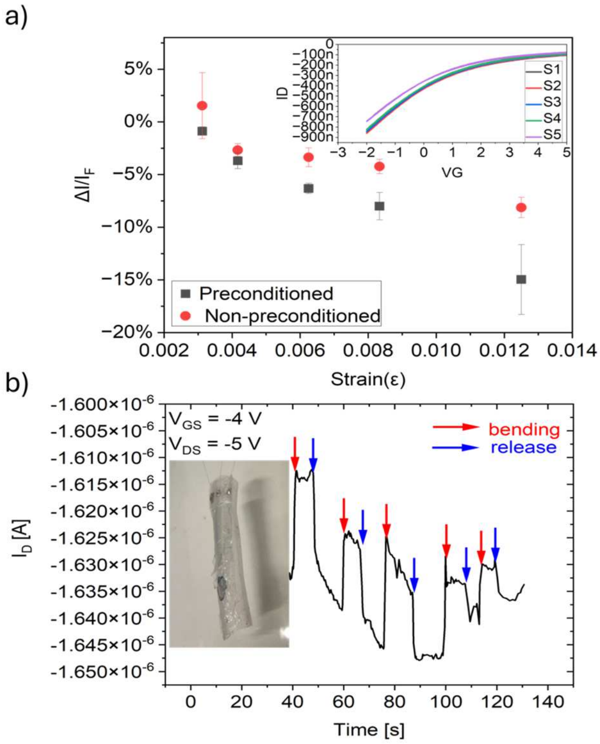

Fig. 3: Electromechanical characterization of ultra-flexible OFETs in soft-robotic applications: a) calibration curve on plastic supports vs increasing strain (inset: transfer characteristic curves for different strain values); b) real-time characterization of catheters hosting OFET strain sensors.

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