Soft, flexible micromanipulators comprising polypyrrole trilayer microactuators

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ABSTRACT

Within the areas of cell biology, biomedicine and minimal invasive surgery, there is a need for soft, flexible and dextrous biocompatible manipulators for handling biological objects, such as single cells and tissues. Present day technologies are based on simple suction using micropipettes for grasping objects. The micropipettes lack the possibility of accurate force control, nor are they soft and compliant and may thus cause damage to the cells or tissue. Other micromanipulators use conventional electric motors however the further miniaturization of electrical motors and their associated gear boxes and/or push/pull wires has reached its limits. Therefore there is an urgent need for new technologies for micromanipulation of soft biological matter.

We are developing soft, flexible micromanipulators such as micro- tweezers for the handling and manipulation of biological species including cells and surgical tools for minimal invasive surgery. Our aim is to produce tools with minimal dimensions of 100 µm to 1 mm in size, which is 1-2 orders of magnitude smaller than existing technology. We present newly developed patterning and microfabrication methods for polymer microactuators as well as the latest results to integrate these microactuators into easy to use manipulation tools. The outcomes of this study contribute to the realisation of low-foot print devices articulated with electroactive polymer actuators for which the physical interface with the power source has been a significant challenge limiting their application. Here, we present a new bottom-up microfabrication process. We show for the first time that such a bottom-up fabricated actuator performs a movement in air. This is a significant step towards widening the application areas of the soft microactuators.

1. INTRODUCTION

1.1 Micromanipulation

The handling and manipulation of small objects under a microscope, micromanipulation, is generally difficult at this scale and needs specialized instruments. Various techniques and technologies for different purposes have been explored [1] and some are even commercialized now [2, 3]. In the medical field micromanipulation is the practice of manipulating cells or small pieces of tissue. A well-known example is the use of micropipettes to manipulate egg and sperm cells in the in vitro fertilization technique Intracytoplasmic sperm injection (ICSI). However, it does not avoid the manually handling of samples and may lead to difficult reproducibility of the manipulation. Moreover, when the objects are fragile or soft it becomes even more cumbersome and it may not be possible to handle the objects anymore. Therefore, new, automated tools are needed to avoid the use of human hand.

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Throughout the years, different concepts to grasp small objects have been presented in the literature. For instance various MEMS grippers have been developed but they are complex to micro-fabricate, brittle, non-compliant and develop small deflections [4-6].Another interesting gripper design uses the Jamming effect of granular materials [7]. This system allows these grippers to grab objects with arbitrary shapes. However, it would be difficult to micro-fabricate this kind of device and it should be difficult to grab soft objects. Actuators such as rotary micromotors [8, 9] cannot be used at this scale either because again they are difficult to microfabricate and would require a transduction mechanism such as a gear box or push/pull wires to operate the gripper.

Thus, there is a need to develop new microtools for the micromanipulation in biology and the medical fields.
Some soft materials based on recent mechanisms are explored in order to create new original micromanipulators which should be easy to microfabricate, have accurate position control and have compliant mechanical properties to avoid damaging the sample and, in the case untethered applications, low energy consumption. They should produce enough forces to displace objects such as a cells and tissue. Different new soft actuators meet these requirements and have shown the possibility to create enough force and displacement for micromanipulation and or microrobotics.

1.2 Soft actuators

Piezo polymers have been developed for microrobotic purposes, [10] however they produce small displacements. Conjugated polymer (CP) actuators, on the other hand, are very promising and they already have shown an example of well-controlled micromanipulation. A 670 µm long microrobot arm [11] was able to successfully grasp and displace a 100µm glass bead. The drawback of that microrobotic arm was that it could only operate in water.
Recently, a so-called trilayer geometry has been used to allow CPs actuators working in air [12, 13]. Two slightly different concepts are used to fabricate this trilayer structure. The first one uses a commercial polyvinylidene difluoride (PVdF) membrane which is coated with a sputtered Au layer on both sides followed by an electrochemically synthesized Polypyrrole (PPy) layer on either side[12]. The other uses an interpenetrating network (IPN) as the electrolytic membrane into which poly3,4ethylenedioxythipopheneis chemically synthesized on both sides [13]. The PVdF or IPN membrane, onto which conjugated polymer is deposited, is used as an ion reservoir containing the ions necessary for the oxidation-reduction process thus allowing the device to operate in air. When a voltage is applied between the two faces of the device, one conjugated polymer layer is oxidized and the other is reduced, inducing opposite volume change created by ions of the two active layers causing the bending of the material (Figure 1).

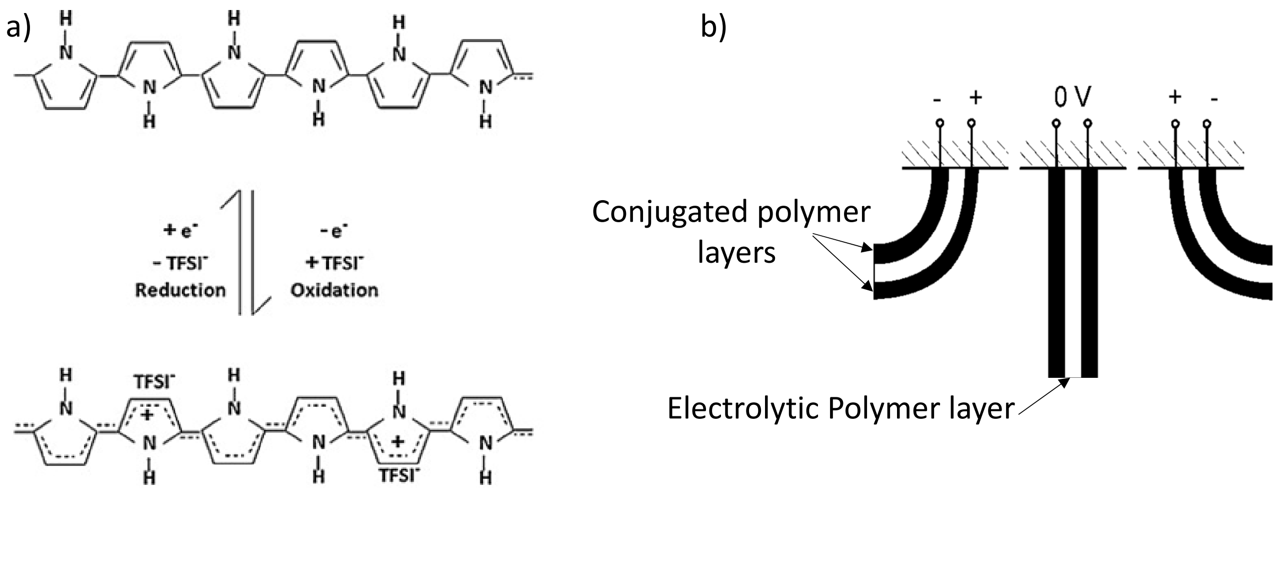

Figure 1: a) Oxidation-reduction process of the conjugated polymer and illustration of the bending of a trilayer conjugated polymer actuator

Recently, these conjugated polymer trilayer actuators have been microfabricated using different techniques such as laser ablation[14] or photolithography combined with reactive ion etching [15]. These early microfabricated trilayer CP actuators employed relatively thick membranes, which limited the achieved deflection. To increase the deflection the thickness of the actuator structure has to be reduced. Thin CP microactuators have been obtained using spin-coating of a

PVdF solution with porogens combined with a heating step at 200 °C to achieve a porous electrolytic layer, followed by a similar sputter deposition of gold electrodes on both faces of the polymer film, electrodeposition of the conjugated polymer and patterning by laser ablation as previous [16-18].

Thin conducting IPNs have also been recently fabricated [19]. The process starts with the fabrication of the thin electrolytic polymer film using spin coating or hot press. The conjugated polymer is then chemically formed inside the polymer membrane. The multilayer material is then deposited on a Si substrate using a polyvinyl alcohol (PVA) sacrificial layer to allow the following photolithography and reactive ion etching steps. These actuators have shown the capability to operate at frequencies higher than 1000 Hz.

In order to operate the CP actuator, contacts should be placed on both sides of the trilayer structure to link the actuator with an electrical power supply. For this purpose, the actuator should be released from its substrate and the contact is usually realized at a macroscopic scale with clamps or kelvin clips. Using clamps or kelvin clips potentially leads to short-circuiting due to the high clamping force and also is cumbersome to achieve individual control of multiple actuators. Therefore, a new interfacing method, specially designed for thin CP actuators, has been also developed that allows individual control of three CP actuators integrated in a single unit [20].

Patterning is needed to downscale these types of actuators and accurately fabricate complex devices. With a bottom-up microfabrication method, it will be possible to conceive devices for micromanipulation producing both simultaneous and individual controlled motion of each CP actuator of the manipulator [20] .

These recent advances in microfabricated trilayer actuators that have been realized require removing the device from a carrier substrate and flipping it in order to process the opposing side. In order to rationally and reproducibly microfabricate patterned CP microactuators that operate in air, a bottom-up fabrication method is needed without the need of releasing, flipping and reattaching to carrier substrate. In addition, it would be desirable to be able to create a device that can be interfaced without the need to release the device from the substrate and that could be automatically interfaced with the electrical contacts that apply the driving potential in order to create new type of micromanipulator [21]. All these current CP microactuators are realized without their contacts and a rigid “clip” should be used to provide versatile interfacing with different devices. Bottom-up fabrication, for instance on a Si wafer, will also allow easy contacting and individual control of the active materials and interfacing it with other electronic components.

The aim of our research is to develop new tools to achieve robust micromanipulation of fragile objects in the biomedical field. For instance, the use of these new tools could lead to new possibilities in research and more accuracy in medical treatments or diagnosis.

2. MATERIALS AND METHODS

2.1. Materials

PPy was obtained from Sigma–Aldrich, was distilled and stored at −18 °C prior to usage. Propylene carbonate (PC) and bis(trifluoromethane)sulfonimide lithium salt (LiTFSI), Polyvinylidenefluoride (PVdF, powder, 534000g.mol^{-1}) , and dimethylformamide (DMF) (Sigma−Aldrich) were used as received. I_2, KI were acquired from Merck and used as received.

Photoresist S1818 and corresponding developer microposit 351 (to dilute) were acquired from Microchem corporation.

4’’ Si 100 wafer were obtained from semiconductor wafer, Inc.

2.2 Measurements:

Profilometry measurments (Dektak 6M) were used to get the thicknesses of the different materials.

The actuators were electrically addressed using an Ivium compactstat or IviumStat (Eindhoven, The Netherlands).

Crocodile clip are used on the contact pad, external to the actuators, and the displacement is followed by a laser displacement sensor, optoNCDT 1700-50 from Microepsilon (Ortenburg, Germany).

3. RESULTS

3.1. Actuator fabrication

We have developed a novel bottom-up microfabrication process that allows construction of a patterned trilayer actuator including the addressing lines and contact pads, all integrated in one single device. The process flow of the actuator microfabrication including its contact pads is described in the figure 2. The bottom-up fabrication approach starts by 500 Å gold evaporation on a Si wafer. Then S1818 photoresist used to pattern the gold layer. S 1818 is spin coated at 4000rpm/4000rpm.s^{-1} during 30s and baked at 100ºC during 20 minutes. The photoresist is then exposed during 10 s to an UV source at 365 nm (Power:10 mJ.cm^{-2}) using a Karl Suss MJB-3 mask aligner using a printed photomask from Acreo AB, Norrköping, Sweden. After dissolving the exposed part of photoresist in the developer, the exposed gold surface is wet chemically etched in KI/I_2 aqueous solution (4 g KI, 2 g I_2 in 100 ml H_2O). A first patterned electrode containing the contacts pad is formed. Then the first PPy layer is electrodeposited at 0.1 mA.cm^{-2} using an Ivium compactstat or IviumStat on the patterned gold electrodes from a 0.1 M Pyrrole, 0,1 M LiTFSI and 1 % water carbonate propylene solution during 2 hours at -18 ºC. A two electrode set-up was used for the electrosynthesis: the patterned Au electrode was connected to the working electrode lead of the potentiostat and a stainless steel mesh was used as the counter electrode. The reference electrode lead was connected to the counter electrode. The obtained thickness of the PPy layer is ≈ 8µm. A 200 g.L^{-1} PVdF solution in DMF is then spin-coated at 500rpm/500rpm.s-1 during 30s. The thickness of the PVdF layer after drying for 30 min at 50 ºC is 14 µm. The second gold layer was sputtered using a Vacutec PlasmaSystems sputter system at a pressure of 1.8 mTorr, 26.0 cm^3/min Ar flow during 5 minutes resulting in a layer of 400Å. The patterning of the second electrode is realized in the same way as the first. The second PPy electrode is then grown in the same conditions as the first one. To finish the actuator fabrication, the wafer is finally immersed in the electrolytic solution of 0.1 M Li TFSI in propylene carbonate a room temperature. The swelling effect (stress created at interfaces) of the membrane allows it to release the patterned multilayered system. The released, finished device is depicted in the figure 3. After releasing the material, the samples were manually cut out in the desired final shape in order to test their actuating capabilities.

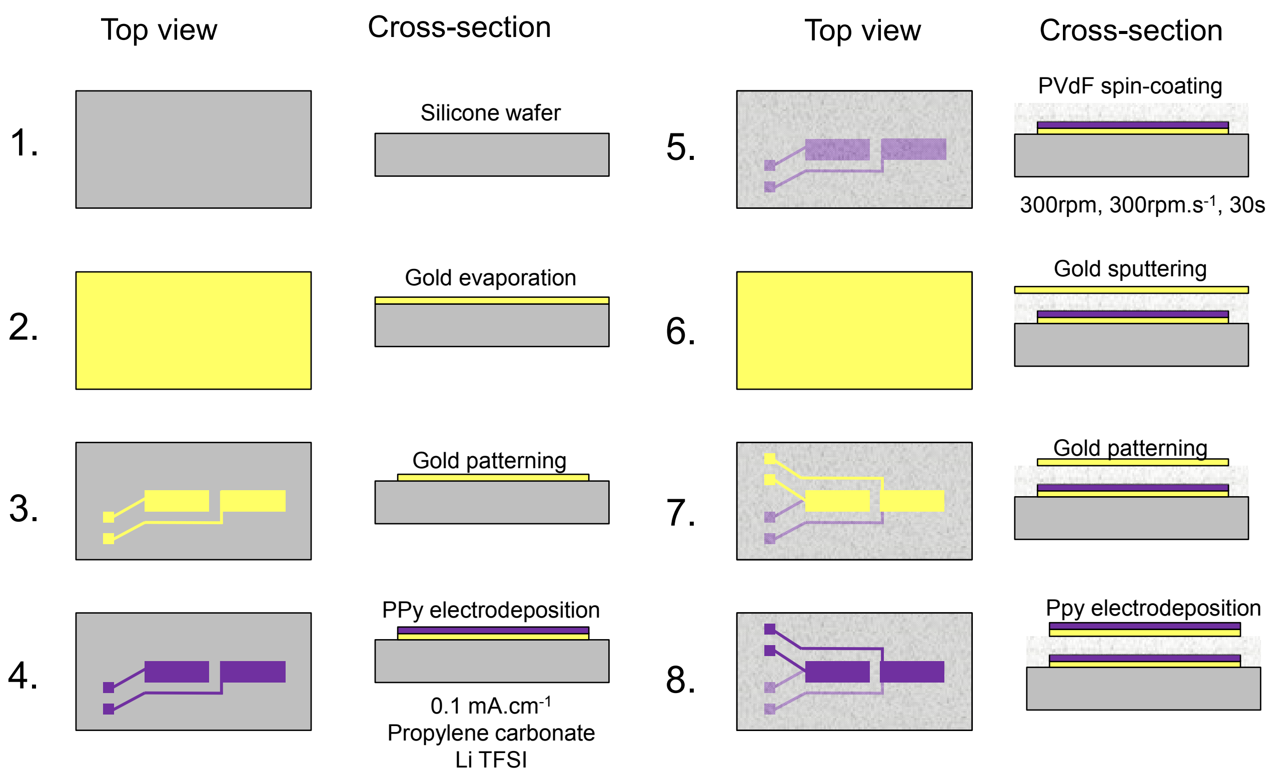

Fig 2: Process flow of the bottom-up microfabrication of tri-layer actuators.

In figure 3, the pattern of bottom-up fabricated trilayer actuators is also clearly visible. It is possible to pattern all contacts pads and electrical leads as wells as two patterned trilayer microactuators of $5*10mm^2$ each with excellent accuracy. Although microfabricated the lateral dimensions are not yet micro-sized. In the next generation the size of the device will be decreased, while keeping a relatively large size for the contact pads to allow for easy integration of the contacts for microactuators for different applications. The current device design, presented here allows to individually control the two different actuators mimicking the articulation of one finger. The bottom-up approach fabrication is a very effective method to fabricate new complex systems.

Fig 3. A photo graph of the finished trilayer microactuator device mimicking a finger that can be articulated. The device comprises two actuators that are $5*10mm^2$ each.

The top actuator has been connected to a potentiostat, and the tip displacement, measured using a laser displacement sensor, is presented in figure 4. The applied potential was ±1V at 0.05 Hz. The actuator's tip displacement is 100 µm. For the actuator having a thickness of 35 µm and a length of 10 mm, the resulting strain is 7×10^{-5} % [22].

This movement is very small. We attribute this low performance to the low ionic conductivity ($\approx 10^{-6}$ $S.cm^{-1}$ for maximum electrolyte swelling ratio) of the crystalline PVdF membranes, however even the stiffness of PVdF membrane might also limit the deflection performance. We are currently working on improving the ionic conductivity of the PVdF membrane as well as to replace the PVdF with another polymeric material that has good ionic conductivity and good mechanical properties.

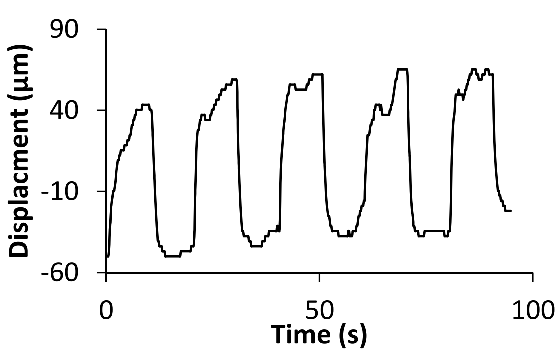

Fig 4 Tip displacement of the top actuator under an application of ±1V at 0.05 Hz.

4. CONCLUSION

A bottom up approach fabrication of individually controlled trilayer actuators working in air is successfully demonstrated in this study. A soft, trilayer actuator device comprising 2 individually addressable actuators with integrated leads and contact pads was made. It has been shown that the device produces a small displacement under low voltage application. However, this displacement is too small in order to be exploited in real, complex micromanipulators capable of grasping and displacing small fragile objects. Other electrolytic polymer membranes will be used in order to improve the performance of this trilayer conjugated polymer actuator and be able to demonstrate micromanipulation of soft, biological objects.

5. ACKNOWLEDGMENTS

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