

MEMS-fabricated ICPF microactuators for biological manipulation

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ABSTRACT

Ionic Conducting Polymer Film (ICPF) micro grippers with multi-finger configurations were developed using spin-on Nafion and photolithographic technology. A commercial solution from Dupont Co. (Nafion SE-5012) was used to prepare ~0.2 μ m thick ionic conducting polymer film. Micro cantilever structures were fabricated which composed of Au/Nafion/Au film layers. Grippers with 2-finger and 4-finger configurations were successfully developed. We have proved that the 2-fingered grippers can be actuated in water at ~5V DC voltages. The smallest 4-fingered grippers fabricated were 30 μ m wide, 300 μ m long and 0.4 μ m thick for each finger. In addition, another interesting actuation behavior of the micro actuator was observed during fabrication. The actuators would curl up whenever they came in contact with water during the sacrificial release process. The curling process reversed instantaneously when the actuators are immersed in acid. We suggest that this phenomenon is probably due to the different volume expansions of Nafion in different medium. Besides, the current-voltage property was measured. We are further studying the consistency of their behaviors to modify the design and fabrication process for potential use in biological manipulations.

Keywords: ionic conducting polymer film (ICPF), polymer micro actuator, biological micromanipulation

1. INTRODUCTION

MEMS technology is gradually impacting many areas of science and engineering, including biology. Traditionally, the manipulation of biological entities (e.g. cells) is implemented by using pipette and therefore the performance is not flexible, not precise, and easily damage cell membranes. Thus, micromanipulation of biological entities has gained more interests in the research areas of Bio-MEMS recently. Nearly all existing MEMS actuators are limited to specific or narrow applications due to their limited displacement, force output, and necessary working environment. As stated by S. Shoji, each micro actuation principle, e.g., electrostatic, piezoelectric, electromagnetic and thermal, has its own advantages and disadvantages^[1]. For example, the electrostatic and piezoelectric actuators are limited by their total deflections. Electromagnetic actuators suffer from the lack of integrated micro-electromagnets that can produce large magnetic field. Although thermal actuators can produce large force and deflection, they require large power and may influence the temperature of the surrounding environment. Moreover, most of the existing MEMS actuators are unfavorable to biological applications because of their limitation of operating only in dry-environments. Recently, Jager, *et al.*^[2] and Smela, *et al.*^[3] have demonstrated two micro-robotic appendages capable of manipulating micro objects in aqueous environments with the use of novel conjugated polymers. Nevertheless, their actuators are limited to the operation in electrolyte solutions, which are not suitable for the survival of many biological entities.

Pioneering work using Ionic Conducting Polymer Film (ICPF) as artificial muscles for robotic applications led by R. Kanno, *et al.*^[4], M. Shahinpoor, *et al.*^[5], and Y. Bar-Cohen, *et al.* have demonstrated the feasibility of *macro*-sized (>10mm) actuators that work in both water and air^[6]. Their large deformation, force output, low power consumption, and especially the capability to be both actuator and sensor, demonstrated advantages over other materials such as SMA, ceramics, *etc.* Most of the existing ICPF actuators are developed using commercial membranes with standard

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thickness around 200 μm , which restricted their allowable deflection when the whole structure is scaled down in length. To solve the problem of this restriction in miniaturizing ICPF actuators, the thickness of membrane has to be reduced. K. J. Kim, *et al.*, have developed a fabrication method to scale the thickness up and down (a strip size of micron-to-centimeter thickness) by dissolving the as-received membrane and recasting the resultant solution^[7]. Nevertheless, no miniaturization work on fabrication of a potentially IC-integratable device in micro scale has been reported yet.

In our previous work, ICPF actuators made from commercial Nafion 117 membrane were successfully fabricated using Nd:YAG laser system^[1]. Actuators with dimensions of $w=300\mu\text{m}$, $l=3000\mu\text{m}$, and $t=200\mu\text{m}$ were successfully actuated under water with 15V DC voltage. We have performed parametric experiments to understand the behavior of Nafion actuators with variations of applied voltage and actuator geometries. On the other hand, we have also presented results from experiments using Nafion commercial membranes as sensing elements to measure mechanical forces. The resultant elements could be potentially integrated with a force-feedback controlled interface^[9].

We have recently reported the first effort in scaling down the actuators. Micro ICPF actuators with single cantilever structure that could function in water were preliminarily fabricated with Nafion solution using lithography-based techniques^[10]. In this paper, we present our work in the fabrication of micro Nafion actuators with multi-fingers using a modified process. 2-fingered actuators were successfully actuated under water. The current-voltage property of a single microcantilever was measured as well. We will characterize the capability of the sensing behavior of the fabricated micro-sized actuators and the possibility of force-feedback micromanipulation eventually.

2. EXPERIMENTS AND RESULTS

2.1 Fabrication

Based on our first effort of microfabrication and experimental study of the resultant Nafion microactuators^[10], two main considerations have been included in the modified process. Firstly, the step height difference of the beam body and anchor in the device is decreased by adding a parylene coating which can serve as a good electric insulator. Secondly, before the sacrificial release of aluminum, the whole surface except the beam body and the bonding pad is covered with another parylene coating. Consequently, the aqueous medium will be impeded from directly penetrating into the Nafion under gold wire, which will prevent this Nafion from volumetric expansion. Besides, multi-fingered structures were included in order to realize gripping action with full closure of the appendages.

We also used the commercial Nafion solution from Dupont Co. (Nafion SE-5012) in this newest process. The appendages are composed of Au/Nafion/Au layers with the Nafion film thickness controlled by spin-on process. Figure 1 shows the process flow. The fabrication started with the thermal oxidization of a SiO_2 layer ($\sim 0.5\mu\text{m}$) on the silicon substrate, followed by the deposition of an $\sim 1\mu\text{m}$ thick aluminum layer. The aluminum layer served as the sacrificial layer for the actuator cantilever structure. A thin chromium layer ($<0.05\mu\text{m}$) as an adhesion promoter was evaporated and patterned. A parylene layer ($\sim 1\mu\text{m}$) was coated and patterned. The first gold layer ($\sim 0.1\mu\text{m}$) was sputtered on and patterned. Afterwards, Nafion solution was spun-on and cured to a thin film. The relationship between the thickness and the spin-rate is shown in Figure 2. The Nafion film in the process was $\sim 0.2\mu\text{m}$ thick.

Then, $\sim 0.1\mu\text{m}$ thick gold layer was sputtered on and patterned by direct wet etching technique. This gold layer served as the top electrodes of the ICPF actuators as well as the mask of the oxygen plasma employed to etch the Nafion thin film. Afterwards, another parylene coating was added and patterned to protect the connecting wires. Finally, the sacrificial aluminum layer was removed by phosphoric acid to release the structures. The smallest actuators fabricated were $30\mu\text{m}$ wide, $300\mu\text{m}$ long, and $0.4\mu\text{m}$ thick.

Grippers with 2-finger and 4-finger structures were fabricated. As shown in Figure 3, the fabrication-induced thickness difference between the top and bottom electrodes determines the shape of the structure after release - an indication that it is very important to keep the residual stresses in both the Nafion and metal films under control in the fabrication process. The resultant structure may point downwards to the substrate after release if the top gold layer was much thicker than the bottom gold layer. Moreover, the released configuration is also related to the layout-designed geometric size of the beam body as Figure 3 (a) and (b) reveals.

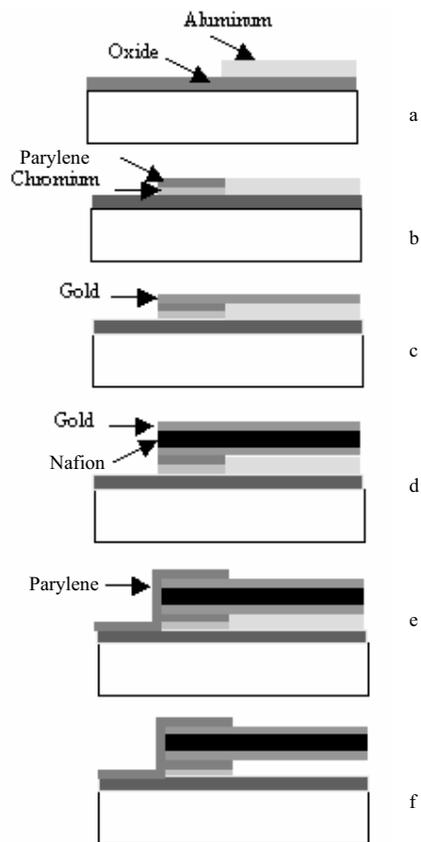


Figure 1: A schematic drawing of the process steps to fabricate the micro Nafion actuator. (a) Deposition and etching of sacrificial aluminum on oxidized Si. (b) Deposition and etching of adhesive chromium layer. Parylene coated and patterned. (c) Deposition and liftoff of bottom gold layer. (d) Deposition of Nafion by spin-on. Deposition and etching of top gold layer. Etching of Nafion by plasma. (e) Waterproof parylene layer coated and patterned. (f) Removal of sacrificial layer.

2.2 Potential pH sensing ability

The fabricated Nafion structures were vulnerable to photoresist developer solution when the developer was used to etch the aluminum in our experiments. The microstructures would peel off the substrates if their anchors were not large enough. We turned to use of the phosphoric acid (H_3PO_4) to perform the release process. An interesting actuation behavior of the micro actuator was observed during the phosphoric acid release process. The H_3PO_4 solution (85%) was heated to $\sim 40^\circ C$. The average etch rate for aluminum was less than $100 \text{ \AA}/\text{min}$. The sample was taken from the acid solution and immersed into DI water occasionally to inspect the progress of the release. After about 2 hours with several immersions into DI water, the cantilever lay horizontally in acid as shown in Figure 4 (a). When DI water was added into the acid, the cantilever curled up as the microscope picture showed in Figure 4 (b). If sufficient water was added, it completely curled up instantaneously as shown in Figure 4 (c).

However, this phenomenon did not occur when the sample was statically released in phosphoric acid for several hours without occasional submersion in water at room temperature. In this case, the resultant cantilevers remained

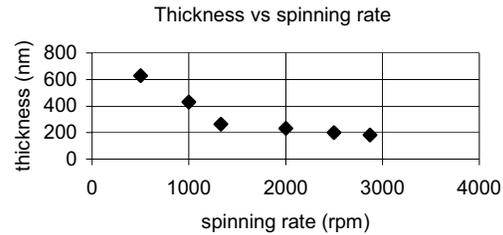


Figure 2: Relationship between the thickness of prepared Nafion film and the spinning rate. (Nafion SE-5012 from Dupont Co.)

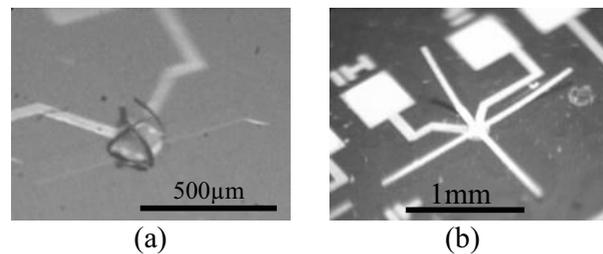


Figure 3. Micro 4-fingered actuators with different configurations in different batches after the release process. The thickness difference between top and bottom electrodes determined the shape of the structure after release. The dimensions of Nafion layer in each finger of the actuators are $w=30\mu m$, $l=300\mu m$, and $t=0.2\mu m$ in (a), $w=30\mu m$, $l=1000\mu m$, and $t=0.2\mu m$ in (b).

horizontally as shown in Figure 4 (a). These cantilevers fabricated by the latter release process were used to do electrical actuation test described in the next section. Thus, our experimental results showed that the release procedure played an important role in producing the final structure, i.e., the sacrificially released actuators had different released-configurations depending on the etchant and procedure used.

The exact mechanism of the curling behavior is currently under investigation. We suggest that Nafion has different volume expansion in different medium. 1) Without water bath during the release of aluminum sacrificial layer in phosphoric acid, Al^{3+} in the solution might exchange with some H^+ in the Nafion during a relatively stable long period. Thus, the Nafion film probably became harder. Therefore, the curling-up phenomenon did not happen. 2) With water bath, H^+ concentration in Nafion film was slightly changed. The beams became softer. When water was added, the osmolar pressure gradient and the surface tension with respect to the viscosity of solution probably curled the beam. Therefore, this fabricated actuator is potentially suitable for an ionic level sensor. We are currently studying the consistency of this actuation behavior.

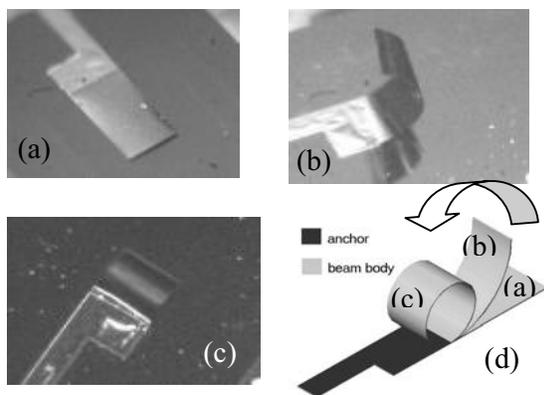


Figure 4: (a) A released actuator in phosphoric acid. (b) When DI water is added to acid, the actuator begins to curl. (c) If sufficient DI water is added, the actuator completely curls up. (d) A schematic drawing shows the procedure to curl up from state (a), (b) to (c). The actuators were released in phosphoric acid with immersion of water at room temperature. The dimensions of the Nafion layer in the actuator shown above are $w=200 \mu\text{m}$, $l=500 \mu\text{m}$, and $t=0.2\mu\text{m}$.

2.3 Actuation test

The testing of the polymer actuators was carried out using a micro probe station. DC voltage was loaded to the sample through two micro probes. The resultant deflection was captured through a microscope with a CCD camera linked to a computer terminal.

An actuated 2-finger gripper under water is shown in Figure 5. Each finger is $w=100 \mu\text{m}$, $l=1200 \mu\text{m}$, and $t=0.4\mu\text{m}$. Gas bubbles due to electrolysis were generated from both electrodes when the voltage applied was $\sim 2V$ or higher. The fingers started to deflect at $\sim 5V$ and the cantilever body curled to partial closure. The gripper reached full closure, i.e., 90° change of each finger tip direction when the voltage was $\sim 7V$. It is higher than the voltage to fully actuate the single cantilever structure we reported in our first effort of microfabrication^[10]. This increase of the actuation voltage is possibly due to the power consumption needed by the increased ionic motion under longer wire connectors (which have Nafion below) compared with our previous single cantilever structures. We are investigating a new process to eliminate this Nafion layer underneath the wire lines. After actuation, the beam could approximately return to the original

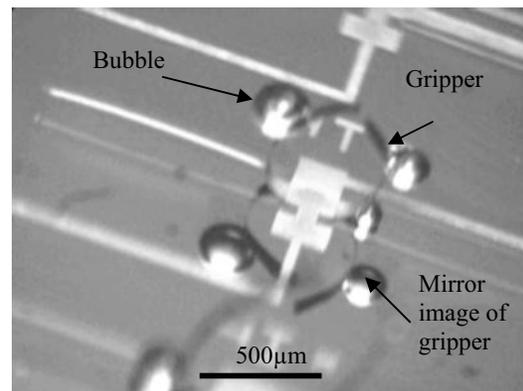


Figure 5: Hirox 3D Microscope picture of a micro Nafion gripper at the moment just after actuation under 5V DC. The bubbles are due to electrolysis of the aqueous medium. The mirror image of the actuator is also visible due to the reflective Si substrate surface. The labeled microactuator have 2 fingers. The Nafion layer in each finger is $w=100\mu\text{m}$, $l=1200\mu\text{m}$, and $t=0.2\mu\text{m}$.

position. Thus, the actuator was stronger structurally than the ones reported in [10], i.e. these actuators have lower link step between the beam body and the anchor (see Figure 6) .

2.4 Current-voltage Property

In order to understand the electromechanical property of the ICPF microactuators, the current-voltage property of some actuators were measured. I-V curve of a typical actuator is plotted in Figure 7 with the current measured at linearly increased DC voltage (data from a single finger structure with $w=300\ \mu\text{m}$, $l=1200\ \mu\text{m}$, and $t=0.4\ \mu\text{m}$). The maximum power consumption of the actuator is estimated to be 0.054mW from the plot. When the voltage goes higher than ~5V, the current almost disappears, which seems to indicate an ionic deficiency in the sandwich structure. Detail characterization is ongoing and will be reported later.

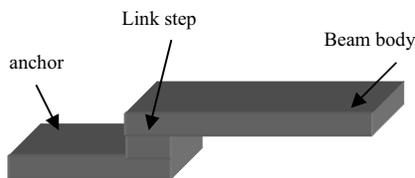


Figure 6. A schematic drawing shows the link step between the beam body and the anchor.

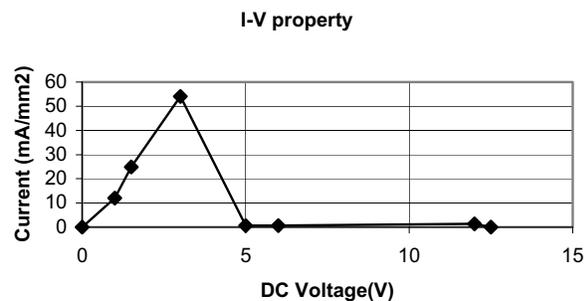


Figure 7. I-V curve of a single finger actuator ($w=300\ \mu\text{m}$, $l=1200\ \mu\text{m}$, and $t=0.4\ \mu\text{m}$). Current was measured when DC voltage was linearly increased.

3. SUMMARY

The modified micro fabrication process of a potentially IC-integratable ICPF micro actuator is presented in this paper. We have demonstrated that 2-fingeres configured actuator as a gripper can operate in aqueous environment. The actuators can be potentially used for manipulation of biological entities in aqueous solutions if electrolysis can be eliminated. The actuators were also demonstrated to sense pH change in the fluidic environment as well. Further study on design, fabrication and characterization of the micro ICPF actuators in terms of gripping force, power consumption, and frequency response is ongoing in our group.

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