Micro-Fabricated Ionic Conductive Polymer Film Actuators for Aqueous Micromanipulation

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ABSTRACT

The development of a novel Ionic Conductive Polymer Film (ICPF) micro actuator is presented in this paper. A commercial solution from Dupont Co. (Nafion SE-5012) was used to prepare ~0.2µm thick ionic conductive polymer film. Cantilever structures composed of Au/Nafion/Au film layers were microfabricated on silicon substrate based on photolithographic technique. The structure was released by etching sacrificial aluminum in phosphoric acid at room temperature. The smallest actuators fabricated were 200µm wide, 400µm long and 0.2µm thick. We have proved that these actuators could be fully actuated in water at ~3V DC voltages. In addition, another interesting actuation behavior of the micro actuator was observed during fabrication. The actuators tended to curl whenever contacted with water during sacrificial release process. The process reversed instantaneously with the immersion in acid. We suggest that this phenomenon is due to the different volume expansion of Nafion in different medium. We are currently studying the consistency of the actuation behaviors. In the future, the design of the structures will be modified to realize precise control of actuation behavior and practical applications.

Keywords: ionic conductive polymer film (ICPF), polymer micro actuator, aqueous micromanipulation, cellular gripper

1. INTRODUCTION

Micromanipulation of biological entities is one of the most important research areas in Bio-MEMS. However, 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 the limitation of operation 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 biological entities.

Pioneering work using Ionic Conductive 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]. The large deformation and force output, low power consumption and especially the capability to be both actuator and sensor, demonstrated advantage over other materials such as SMA, ceramics, *etc*. Most of the existing ICPF actuators were developed using commercial membranes with standard thickness around 200µm, which restricted the allowable deflection while scaling down the whole structure. 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 in a strip size of micro-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.

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In our previous work, ICPF actuators made from commercial Nafion 117 membrane were successfully fabricated using Nd:YAG laser system [8]. Actuators with dimensions of w=300μm, l=3000μm, t=200μm 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 the 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. In this paper, we present our results in scaling down the actuators. Micro ICPF actuators that could function in water were successfully fabricated with Nafion solution using lithography-based techniques. 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 and yield

Commercial Nafion solution from Dupont Co. (Nafion SE-5012) was employed to fabricate the ICPF micro actuators. The actuators were composed of Au/Nafion/Au layers with the Nafion film thickness controlled by spin-on process. Several designs of ICPF actuators were batch fabricated on a 4inch silicon wafer using surface micromachining technology. The process flow is shown in Figure 1. The fabrication started with the thermal oxidization of a SiO_2 layer (~0.3 μm) on the silicon substrate, followed by the deposition of an ~1 μm thick aluminum layer. The aluminum layer served as the sacrificial layer for the actuator cantilever structure. The first gold layer (~0.15μm) with a thin chromium layer as an adhesion promoter was sputtered on and patterned by lift off. Afterwards, Nafion solution was spun-on and cured to a thin film. The spun-on Nafion was cured by baking on hotplate at ~70°C for 5~8 minutes and ~150°C for 5~8 minutes according to the recasting process reported by T. Arimura [9]. It was possible to increase the Nafion thickness by repeating the spinning and heat-curing procedures. At most, three spin-on layers could be smoothly piled up without wrinkles or cracks on the plain surface in our experiments. However, wrinkles appeared around the step edges if two spin-on layers were overlapped on a non-planar substrate. They hindered the fabricating procedure which followed. In order to generate a relatively uniform thin film, only one layer (~0.2μm thick) was used to conduct full process flow.

Then, ~0.15μm thick gold layer was sputtered as the top electrodes of the ICPF actuators. Considering the compatibility of Nafion thin films with MEMS-based fabrication chemicals, two different processes were developed to pattern the gold layer: 1) shadow mask technique, and 2) direct wet etching technique. This gold layer also served as the mask of the oxygen plasma employed to etch the Nafion thin film. Although the yield by shadow mask was acceptable, it was difficult to generate smooth pattern edges with uniform thickness due to isotropic coating of the sputtering technique. Combined with the feasible output of the direct wet etching, which was much more promising for integration, we laid the shadow mask technique aside. Finally, the sacrificial aluminum layer was removed by phosphoric acid to release the structure.

Our experimental results showed that the release procedure played a key role in producing the final structure, i.e., the sacrificially released actuators had different released-configurations depending on the etchant used. At the beginning, we used phosphoric acid without dilution and heated it to ~40°C. The average etch rate for aluminum was less than 100Å/min. The sample was taken from the acid solution and immersed into DI water 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 2(a). When DI water was added into the acid, the cantilever curled up as the microscope picture shows in Figure 2(b). If sufficient water was added, it completely curled up instantaneously as shown in Figure 2(c).

However, this phenomenon did not occur when the sample was statically released in phosphoric acid for several hours without submersion in water at room temperature. In this case, the resultant cantilevers remained horizontally as shown in Figure 2 (a). These cantilevers were used to do electrical actuation test described in the following section. The smallest actuators fabricated were 200μm wide, 400μm long and 200nm thick. In addition, our experiments showed that the fabricated structure were vulnerable to vibration if developer was used to etch the aluminum.

The exact mechanism of the curling behavior is being studied. 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. When water was added to the acid, water molecules could not permeate into Nafion immediately. Therefore, the curling-up phenomenon did not happen. 2) With water bath, H^+ level in Nafion film was slightly changed. The beam was softer. When water was added, the osmolar pressure gradient and the surface tension with respect to the viscosity of solution probably curl the beam. Therefore, this fabricated actuator is suitable for an ionic sensing actuator as well. We are currently studying the consistency of this actuation behavior.

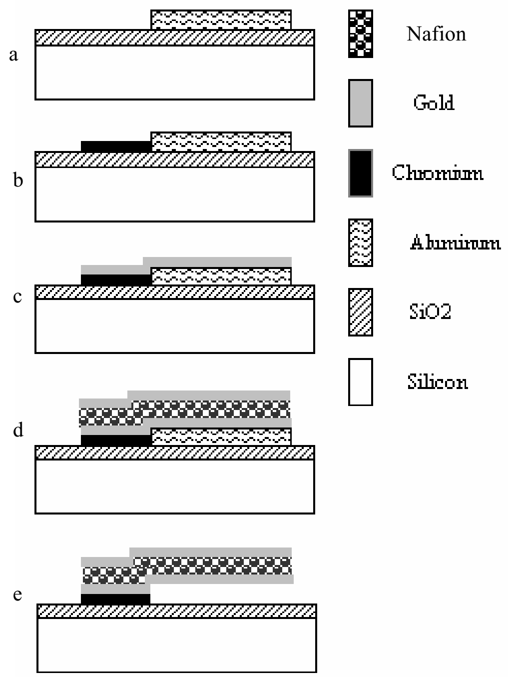

Figure 1: A schematic drawing of the process steps to fabricate the micro Nafion actuator where the top gold electrode is wet etched. (a) Deposition and etching of sacrificial aluminum on oxidized Si. (b) Deposition and etching of adhesive chromium layer. (c) Deposition and liftoff of bottom gold layer. (d) Deposition of Nafion by spin-on. Deposition and etching of top gold layer. Etching of Nafion by plasma. (e) Removal of sacrificial layer. For the shadow mask procedure, step d) is replaced and gold is patterned during deposition.

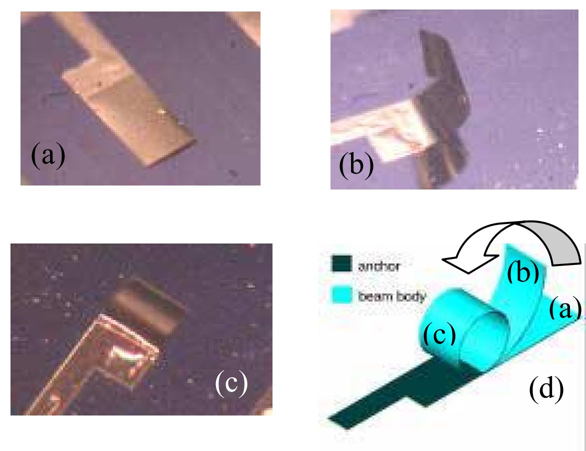

Figure 2: (a) A released actuator in phosphoric acid. (b) When DI water is added to acid, the actuator begins to curl. (c) If sufficient DI water is added, the actuator completely curls up. (d) A schematic drawing showing the procedure to curl up from state (a), (b) to (c). The actuators are released in phosphoric acid with immersion of water at room temperature. The dimensions of the Nafion layer in the actuator shown above are w=200 µm, l=500 µm, t=0.2µm.

In addition, the thickness difference between the top and bottom electrodes also determined 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. We are seeking a way to quantify the effect of thickness difference – guidance for the modification in the design of the structures to realize batch yield and post precise control of the actuation behavior.

2.2 Actuation test

The testing of the polymer actuators was carried out using a micro probe station. The setup of the experiment is shown in Figure 3. The sample was located in a petri dish filled with DI water. DC voltage was applied 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 actuator under water is shown in Figure 4. Gas bubbles due to electrolysis were generated from both electrodes when the voltage applied was ~2V or higher. The actuator (w=300 µm, l=1200 µm, t=0.2µm) started to deflect at ~3V. It reached full deflection, i.e., 90° change of tip direction when the voltage was 5V. The smallest actuators (w=200µm, l=400µm, t=200nm) could be fully actuated in water at ~3V.

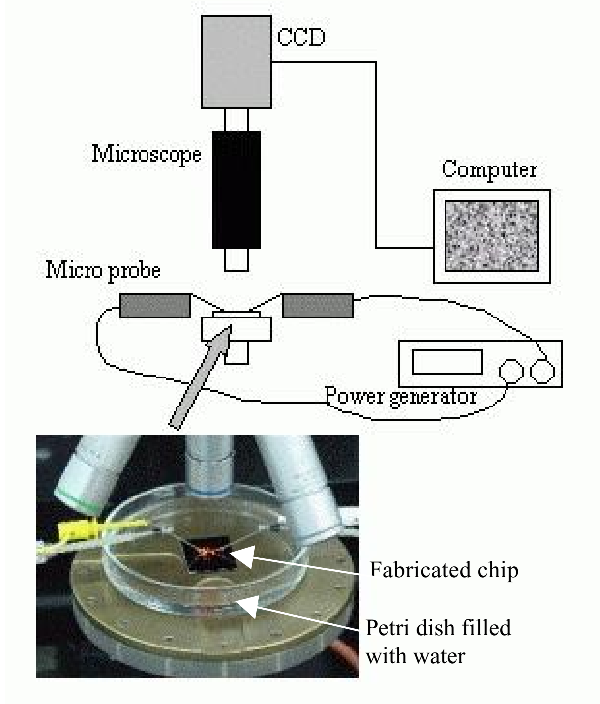

Figure 3: Experiment setup.

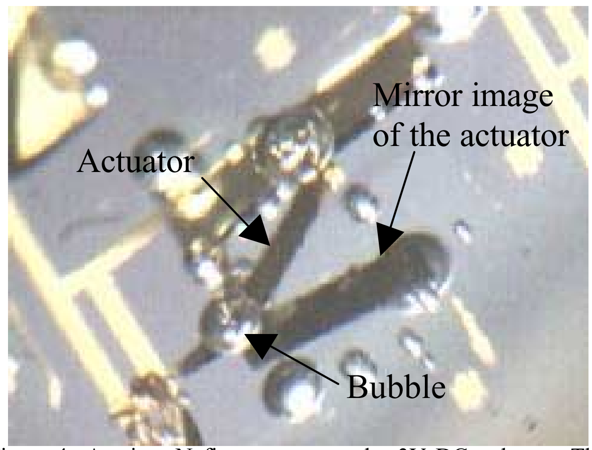

Figure 4: A micro Nafion actuator under 3V DC voltage. The bubbles are caused by the electrolysis of the aqueous medium. The mirror image of the actuator is also visible due to the Si substrate surface. The dimensions of the actuator are w=300 µm, l=1200 µm, t=0.2µm.

After actuation, the beam did not return to the original position. There existed a residual angle between the beam body and the substrate. It was observed that the link step from the anchor to the beam body had deformed. Thus the strength of the link was not enough. Moreover, the residual angle increased after the second actuation. Therefore the strength of the link step has to be increased in the future design. If the top gold electrode was too thin (e.g., ~700Å), it would easily peel off due to the electrolysis during actuation. Moreover, after photolithographic patterning the top gold of such a thickness, wrinkles appeared around the gold pattern. We suggest that the gold etchant penetrated into the Nafion layer underneath through the relative thinner gold layer. The penetration was probably due to the strong ionic exchange ability of ICPF supplied by SE-5012.

2.3 On going work

We are currently fabricating structures with multi-fingers in order to realize gripping action with full closure of the appendages. A four-finger structure fabricated before sacrificial release is shown in Figure 5. Based on the experimental study carried out, two main considerations have been included in the modified design:

1. The step height difference will be decreased by adding a parylene coating which can serve as a good electric insulator.
2. Before the sacrificial release of aluminum, the whole surface except the beam body and the bonding pad will be covered with another parylene coating. Consequently, the aqueous medium will be impeded from penetrating into the Nafion under gold wire, which will prevent this Nafion from volumetric expansion.

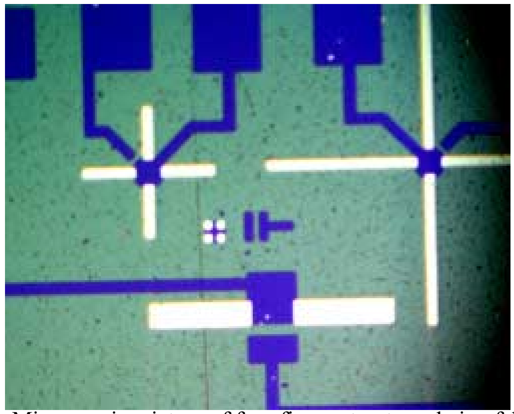

Figure 5: Microscopic picture of four finger structures being fabricated.

3. SUMMARY

The micro fabrication process of a novel ICPF micro actuator is presented in this paper. We have demonstrated that this actuator can operate in aqueous environment. The actuation voltage required is ~5V. The actuators can be potentially used for manipulation of micro objects under aqueous solution. However, electrolysis is observed during actuation in water. In our future work, the design of the structures will be modified to realize precise control of actuation behavior. The successful development of micro ICPF actuators will enable effective and fast control of underwater micro objects and pave the way for ICPF actuators to eventually manipulate biological elements.

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