Micro ICPF actuators for aqueous sensing and manipulation

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Abstract

This paper presents the results of our ongoing investigation of using Nafion solution to construct micron scale actuators as grippers to operate under aqueous environment. Thus far, we have demonstrated that MEMS-based fabrication of cantilever structures composed of Au/Nafion/Au trilayers on silicon substrates is possible using Nafion solution. The smallest actuators fabricated were 30μm wide, 300μm long and 0.4μm thick. We have shown that 2-finger actuators (each 100μm wide, 1200μm long and 0.4μm thick) can be fully actuated in water at ~7V DC. In addition, with a special sacrificial release process, micro ICPF structures can be made to curl whenever they come in contact with water, and can be de-curled instantaneously when they are immersed in acid, suggesting that they can be used in sensing pH level changes.

Keywords: polymer actuator; micro ICPF; underwater actuator

1. Introduction

Micromanipulation of biological entities is one of the most exciting ongoing research areas in Bio-MEMS. One of its goals is to find an improved technology to replace the current cell positioning and injection method, i.e., the use of pipettes to hold cells during the microinjection of chemicals or DNAs into cells. The major problem of the current technology is that, since the pipette suction force cannot be well controlled by hydrodynamic pressure, excessive suction force may often break the cell membranes. The pipette technology also cannot be used to rotate a cell during the microinjection process, which is a function that is highly desired in that the route of the injection may determine the localization of the injected matters (such as RNA) and subsequent response of cells [1]. Hence, this conventional technique of cell manipulation is rigid, imprecise, and invasive.

The deficiencies of the pipette technology have spawned much interest in using robotic means to grasp and manipulate biological cells. Reported microactuators targeting eventual robotic manipulation of biological cells are diversified. The first demonstration is an overhanging electrostatic gripper made by surface-micromachined polysilicon to grab a dry cell in 1992 by C. J. Kim et al. [2]. However, MEMS electrostatic actuators typically require higher actuation voltage and have small displacements, and hence are not suitable for operations in biological fluids (bubble generation by electrolysis will occur at ~2V potential in water). For a more comprehensive discussion on the limitations of conventional MEMS actuators operating in aqueous environments, [3] can be referenced.

In recent years, novel materials, mostly belonging to the electroactive polymers categories such as Nafion-based Ionic Conducting Polymer Films (ICPF), polypyrrole of conjugated polymers, and PANI of conductive polymers, were investigated by researchers as possible materials for artificial muscles (see [4] for example). As the development of these materials becomes more matured, they offer the potential to break some of the limitations that are set by current MEMS thin film materials such as polysilicon, SiO_2, Si_3N_4, and metals in engineering micro sensors and actuators. Smela et al. (1999) [5] and E. W. H. Jager et al. (2000) [6] have already developed fabrication processes for conjugated polymers and demonstrated micro-robotic appendages capable of manipulating micro objects in aqueous environments. Nevertheless, their actuators have slow response and were limited to operations in electrolyte solutions, which may not be suitable for the survival of many biological entities.

Our ongoing work is to investigate the possibility of using ionic conduction polymer to develop Ionic Conduction Polymer Films (ICPF) micro actuators. These flexible materials convert electrical energy into mechanical energy effectively. Under an electric field, ions move in and out of the conducting polymers, which lead to simultaneous changes in volume as well as variations in physical properties. These polymers are undergoing intense analyses and improvements by the artificial muscles community (for example see [4] and [7]). However, to the best of our knowledge, work reported thus far only focus on meso and macro scale

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actuators, and no existing data are available for Nafion based actuators with dimensions less than 1mm in length except from our group.

In this paper, we will present our work on the fabrication of Nafion-based microactuators using MEMS related processes and some initial experimental results in using them as actuators and sensors in aqueous environment. Our goal is to find how well does the ionic conduction mechanism scale with size, i.e., if the relative large force to input voltage ratio and fast frequency response of ICPF actuation characteristics can be preserved at micro scale, then these actuators will find many new applications in the bio-manipulation area not accessible by existing MEMS actuators.

2. Microfabrication of Nafion actuators

2.1. Fabrication by Laser-micromachining

Most of the reported ICPF actuators were developed using commercial Nafion™ membranes with standard thickness of 200μm. This film thickness restricts the allowable deflection of ICPF actuators when they are scaled down in length and width. In our previous work, ICPF actuators made from commercial Nafion 117 membrane were successfully fabricated using a Nd:YAG laser system [8]. Actuators with dimensions of w=300μm, l=3000μm, t=200μm were actuated under water with 15V DC voltage. Incidentally, an actuator of these dimensions is considered as a "microactuator" by traditional robotics community, but it is considered as a "meso-scale" actuator by MEMS researchers. We performed parametric experiments to understand the behavior of those Nafion actuators with variations of applied voltage and actuator geometries, and found that to achieve grasping motion (i.e., closing the fingers of a gripper) more than 15V of applied voltage was necessary because a large energy was need to overcome the structural rigidity of the Nafion films.

2.2. Fabrication by MEMS-based Process

Commercial Nafion solution from Dupont (SE-5012) was used to fabricate the ICPF micro actuators in the current study. The actuators were made 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 4-inch silicon wafer using surface micromachining technology. The process flow is shown in Fig. 1. The fabrication started with thermal oxidization of a SiO_2 layer (~0.5 μm) on the silicon substrate, followed by the deposition of ~1 μm thick aluminum layer. The aluminum layer served as the sacrificial layer for the actuator cantilever structures. The first gold layer (~0.1μm) which was used as the bottom electrode was sputtered on and patterned by lift off. Afterwards, Nafion solution was spun-on and cured by baking on a hotplate at ~70°C for 5~8 minutes and ~150°C for 5~8 minutes (similar to the recasting process reported by T. Arimura [9]). The relationship of the film thickness and the spinning rate is also shown in Fig. 1. It was possible to increase the Nafion thickness by repeating the spinning and heat-curing procedures. However, as number of layers are increased, residual thermal stress may cause cracking of the Nafion film. In order to generate a relatively uniform thin film, we used ~0.2μm thick Nafion in our process. Then, ~0.1μm thick gold layer was sputtered on top of the Nafion as the top electrode for the ICPF actuators. Finally, the sacrificial aluminum layer was removed by phosphoric acid to release the structures. All the film thicknesses were measured by an Alpha-step profiler.

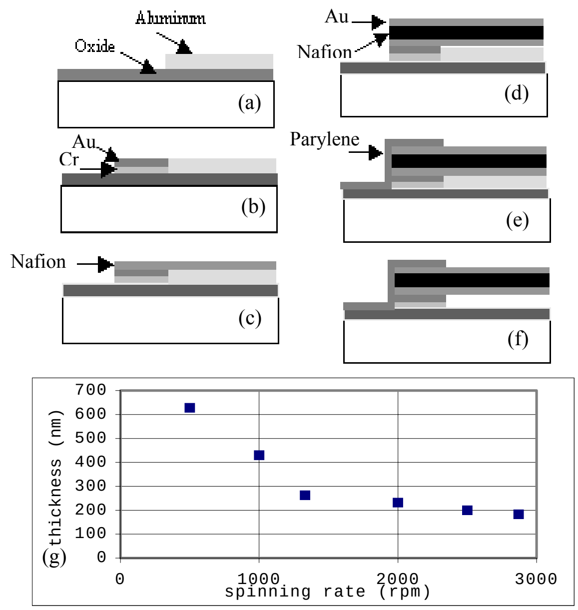

Fig. 1. Nafion actuator fabrication process flow. (a) Deposition and patterning of sacrificial aluminum on oxidized Si. (b) Deposition and etching of adhesive chromium layer. Parylene coated and patterned. (c) Deposition and liftoff of bottom gold layer. (d) Spin-on Nafion. Deposition and etching of top gold layer. Etching of Nafion by plasma. (e) Parylene layer coated and patterned. (f) Removal of sacrificial layer. (g) Relationship between the spin-on Nafion thickness and the spin rate.

Our experimental results showed that the release procedure played a key role in producing the final structures, i.e., the sacrificially released actuators had different released-configurations depending on the release process. For instance, we used phosphoric acid without dilution and heated it to ~40°C to sacrificially etch Al (etch rate ~100Å/min). The samples were taken from the acid solution and immersed into DI water to inspect the progress of the release occasionally. After about 2 hours with several immersions into DI water, the cantilevers laid horizontally in acid as shown in Fig. 2 (a). When DI water was added into the acid, the cantilevers curled up as the microscope picture shows in Fig. 2 (b). If sufficient water was added, it completely curled up instantaneously as shown in Fig. 2 (c). However, this phenomenon did not occur when the samples were released in phosphoric acid for several hours without occasional submersion in water at room temperature. In this case, the resultant cantilevers remained horizontal as shown in Fig. 2 (a). These cantilevers were used to do the electrical actuation test described in the Experimental Results section. The smallest actuators fabricated were 30μm wide, 300μm long and 0.4μm thick (including the metal electrodes). The exact mechanism of the curling behavior is being studied but possible causes were suggested in [3]. The above behavior suggests that with proper process steps, Nafion structures can be used as a pH sensor.

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 during the fabrication process (see Fig. 3).

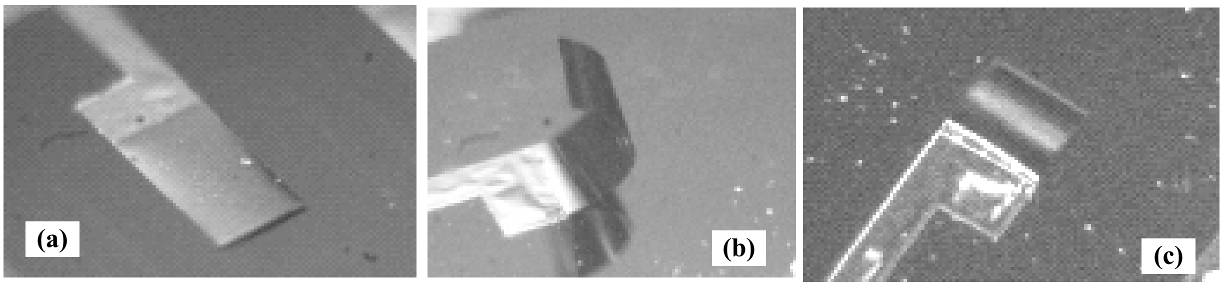

Fig. 2. (a) A released actuator in phosphoric acid. (b) When DI water was added to acid, the actuator begun to curl. (c) If sufficient DI water was added, the actuator completely curled up. The actuators were released in phosphoric acid with occasional immersion in water at room temperature. The dimensions of the Nafion layer in the actuator shown above are *w*=200 μm, *l*=500 μm, *t*=0.2μm.

3. Experimental results

The testing of the polymer actuators was carried out using a micro probe station. The samples were placed in a Petri dish filled with DI water. Voltage was applied to the samples through two micro probes. The resultant deflection was captured through a microscope with a CCD camera linked to a computer terminal.

A sequence of motion of a 2-finger actuator actuated in DI water is shown in Fig. 4 (2-D top-view under microscope). The actuator (each finger *w*=100 μm, *l*=1200 μm, *t*=0.4μm) started to deflect at ~5V. It reached full deflection, i.e., 90˚ change of tip direction when the voltage was ~7V. Gas bubbles due to electrolysis were generated from both electrodes. After the voltage was removed, the beams returned to their original positions. 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 Fig. 5 with the current measured at linearly increased DC voltage (data from a single finger structure with *w*=300 μm, *l*=1200 μm, and *t*=0.4μm). The maximum power consumption of the actuator was estimated to be 0.054W 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 calibration is ongoing and will be reported later.

4. Discussion

Full characterization on the performance of the micro Nafion actuators is underway in our lab. The estimated actuation force and considerations on the actuation voltage of these underwater actuators are discussed below.

4.1 Estimation of Actuation Force

The estimated actuation force of the underwater actuators is discussed in this section. Calibration of gripping force and lifting force of these polymer actuators is difficult by conventional techniques since these actuators have large deflections and work under water. Eventually, different dimensions of silicon blocks will be fabricated to calibrate the force output of the actuators. However, the minimum force output of an actuator must exceed the sum of the fluid drag, gravitational and spring-restoring forces in order for it to actuate and grip a cell (Fig. 6). Note that in a liquid medium, capillary force between the actuator and the substrate is not a concern because of the absence of air-water interface as in the case for actuation in air.

Fluid Drag Force - The Reynolds Number estimated for actuators of this dimension moving in water is <1 (refer to [10] for more elaborate analysis), so the flow around a moving actuator is laminar, and hence we can assume the skin friction is negligible, and the only source of drag force is pressure drag. Then, assuming the worst case scenario where a 1D uniform free-stream flow impinging on a stationary plate, the drag force F_p can be calculated using Bernoulli's Equation:

$$F_P = \rho U^2 A/2 \qquad (1)$$

where $A=wl$, (w is the width and l the length of the actuator) which is the area impinging on the flow, ρ is density of water, and U is the average actuator velocity. To estimate the velocity of the actuator we assumed that for a 1.2mm x 100µm x 0.4µm actuator (same as the one shown in Fig. 4), the tip reached 30° within 1 sec (from experiment). Then, referring to Fig. 6, the average velocity U is ~0.5mm/sec. Hence, F_p ~0.015nN.

Weight of the actuator - The weight of the actuator body F_m is mainly that of the gold film (0.2µm). Hence, F_m~ρwlt=4.6nN, where ρ is the density of gold (19.3g/cm^3).

Force to bend the actuator - Using linear bending beam theory, the actuation force required to deform a beam with a spring constant $k=3EI/l^3$ by a distance y is approximated by $F_k=ky$. So, F_k is ~ 32nN when y is ~0.5mm at tip deflection of 30° and F_k is ~ 0.6nN when y is ~10µm (EI is calculated based on a tri-layer structure).

Therefore, the total minimum force output for a 1.2mm x 100µm x 0.4µm actuator is $F_m+F_p+F_k$ and is ~36.6nN to reach tip deflection of 30° based on the above analyses. The main energy consumption is to overcome the spring-restoring force of the actuator.

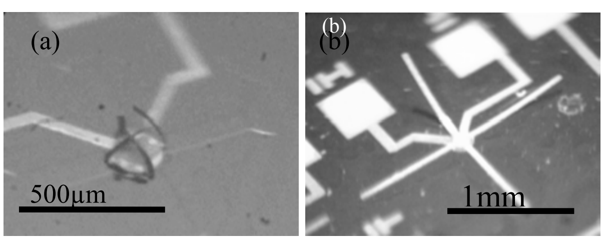

Fig. 3. Micro 4-finger actuators with different configurations in different batches after the release process. The dimensions of Nafion layer in each finger of the actuators are w=30µm, l=300µm, and t=0.2µm in (a), and w=30µm, l=1000µm, and t=0.2µm in (b).

4.2 Actuation voltage

Several models used to characterize the actuation behavior of commercial ICPF actuators have been presented by De Gennes *et al* [11], Shahinpoor *et al* [12], Kanno and Todokoro [13], Nemat-Nasser and Li [14]. Among them, the electromechanical model proposed by Nemat-Nasser and Li revealed a relationship between the microstructural composition of the Nafion polymer, including the cluster radius and spacing inside the membrane, and the response of ICPF as actuator and sensor, in that it was theoretically supported and experimentally observed that Nafion polymer tends to aggregate to form tightly packed regions referred to as clusters. Their linearized results implied a direct relationship between the performance and dimensions of an actuator. Here we compare Nemat-Nasser and Li's model to our gripper actuation data in order to see if the dimensional scaling effect is valid. The voltage required for an actuator to reach full deflection is therefore considered. To achieve a static deflection, the equivalent moment M_e induced by voltage is balanced by the bending moment M_k calculated according to the

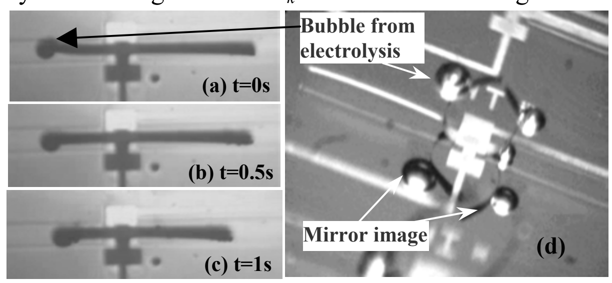

Fig. 4. A micro Nafion actuator under 5V and 7V DC voltage input. (a), (b),(c) are 2-D top views under 5V. (d) is a 3-D picture of actuator under 7V to reach full closure of gripper. The bubbles were caused by electrolysis of the aqueous medium.

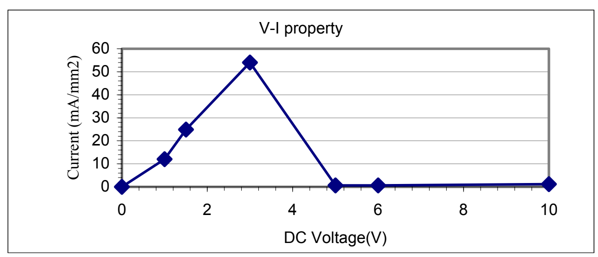

Fig. 5. I-V curve of a single finger actuator (w=300 µm, l=1200 µm, and t=0.4µm). Current was measured when DC voltage was linearly increased.

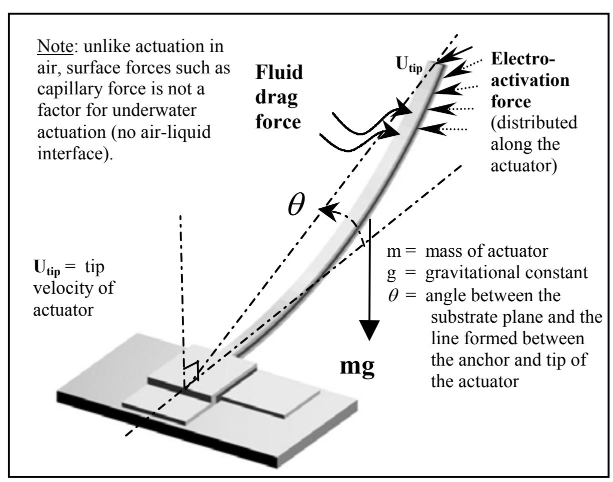

Fig. 6. Illustration showing various forces acting on an actuator.

conventional linear bending theory, i.e., $M_e=M_k$. In this model, effects from the ion type, cluster size, water transportation, electric field, and elastic deformation were all considered. The induced moment in an actuator can be simplified as

$$M_e = k_0 \kappa_e \phi_0 atw \qquad (2)$$

where t is the thickness of Nafion film, κ_e is effective dielectric constant of the polymer, ϕ_0 is the voltage applied, and $1/a$ is the length of the boundary layer [15]:

$$a^2 = C^- F^2 / \kappa_e RT \qquad (3)$$

where C^- is the negative ion density (mol/m^3), F is Faraday's constant, R is the gas constant, T is the absolute temperature, and k_0 is a parameter to be calculated as (unit: J/C [14]):

$$k_0 = (e / 4\pi\kappa_e r_c^{\,2})(r_d^{\,3} / \pi r_e^{\,2}) \qquad (4)$$

where e is the elementary charge, r_c is the cluster radius, r_d is the mean distance between clusters, r_e is the effective radius of the polymer chain.

The bending moment M_k can be related to the bending curvature R_c as: $M_k = EI/R_c$. For full deflection of a gripper, i.e., for it to reach a half circle of deformation as shown in figure 4 (d), $R_c = l/\pi$.

To simplify our analysis, the pressed Nafion film and spin-on Nafion film are supposed to have the same microstructural composition and constructive parameters as the commercial Nafion membrane. From equation (2), the induced moment is proportional to the voltage, thickness and width of the actuators, i.e., $M_e \propto \phi_0 tw$. Here we introduce an electromechanical coefficient m_e, where $m_e = k_0 \kappa_e a$ (unit: Nm/Vm^2), which can be physically related to the moment induced by unit cross-sectional area of a Nafion film by unit voltage when ideally the electrical energy is completely converted into mechanical energy. Hence, from equations (3) and (4):

$$m_e = (e / 4\pi r_c^{\,2})(r_d^{\,3} / \pi r_e^{\,2})(C^- F^2 / \kappa_e RT)^{1/2} \qquad (5)$$

That is to say, m_e is also a parameter which is determined by the microstructural composition of the material. M_e can thus be written as $M_e = m_e \phi_0 tw$. Then the required actuation voltage for full deflection can be expressed as:

$$\phi_0 = \pi EI / (m_e tl / w) \qquad (6)$$

To verify equation (6), we used m_e determined by our experimental results as described in the following analysis.

In our laser-micromachined Nafion actuators (15mm x 1mm x 200μm), the actuation voltage for full deflection was ~4.5V [8]. The mechanical moment needed to fully bend them is M_k= 27.9μNm (The effective Young's modulus of Nafion membrane was experimentally determined as 0.2 GPa). Thus, the electrical moment generated by unit voltage $m_e tw$ is 27.9/4.5= 6.2μNm/V to balance the mechanical moment, or m_e= 31Nm/Vm^2.

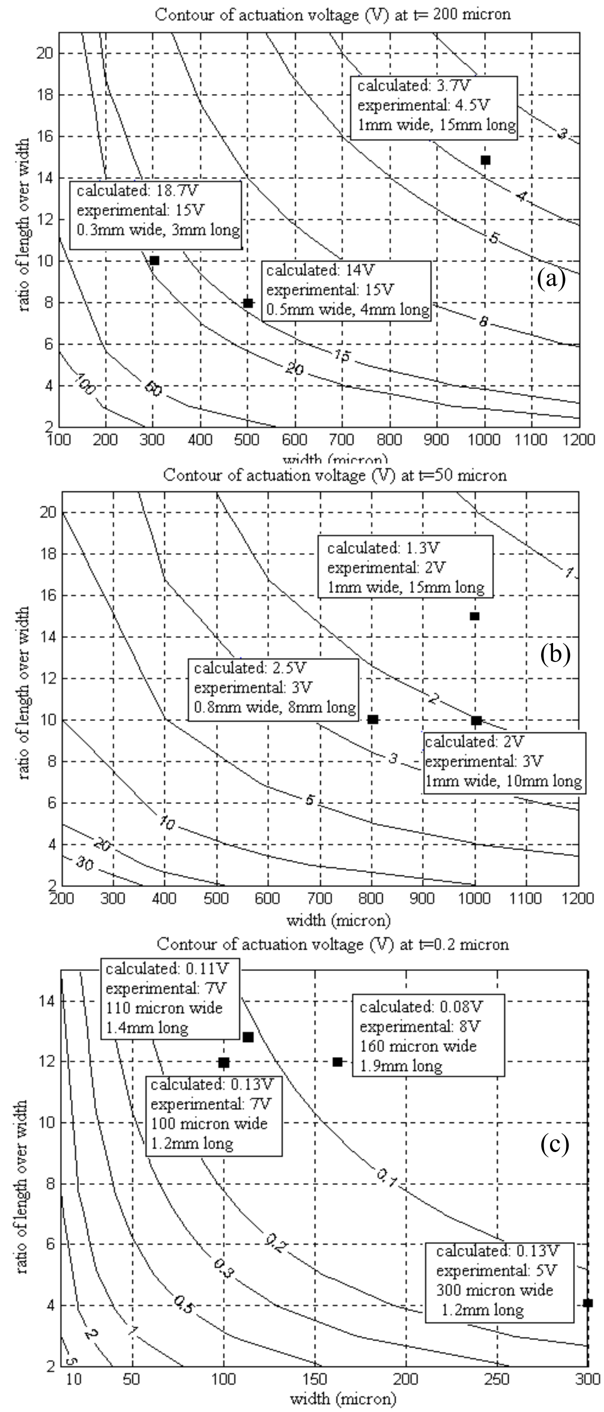

Fig. 7. Contour plots showing isolines of estimated actuation voltages for Nafion actuators with various dimensions constructed by Nafion 117 membrane, pressed Nafion membrane and spin-on Nafion film, respectively. The isolines are constructed using equation (8) with m_e=37.5Nm/Vm^2. The black dots show the dimensions with experimental actuation data. (a) Nafion 117 membrane, t=200 μm. (b) Pressed Nafion membrane, t=50μm. (c) Spin-on Nafion film, t=0.2μm.

Our experimental average m_e is 37.5 Nm/Vm^2, i.e., taking account that the actual voltage for actuators of 4mm x 0.5mm x 200μm and 3mm x 0.3mm x 200μm was both 15V. Using m_e= 37.5Nm/Vm^2, the relationship of the actuation voltage required to fully actuate the structures formed by the commercial Nafion 117 membrane versus their geometric dimensions is ploted in Fig. 7 (a). As shown, the estimated results using equation (6) and experimental values are consistent.

For comparison, combining Nemat-Nasser and Li's theoretical results of $\phi_0 R_c$ and experimental results from Shahinpoor, m_e is derived to be ~24 Nm/Vm^2 for platinum composed IPMC strips in Li+ form with the radius of cluster r_c=5nm by using the relationship $m_e = Et^2/(12\phi_0 R_c)$ (where $\phi_0 R_c$ =0.09, E=0.65 GPa [14]). It is a little less than our experimental results but in the same order of magnitude. One possible reason is that full deflection was considered in our analysis while their work only considered small tip deflections. Other reasons may be the difference of the microstructural composition of their polymer films and the corresponding Young's modulus of their actuators.

Now, consider the thickness of Nafion film scaled down to 50 μm and 0.2μm. The theoretical voltage-dimension relationships for these microscale actuators are shown in Fig. 7 (b) and (c). As shown, the required voltage will be generally decreased when the thickness is scaled down while keeping the width and length constant. However, the voltage will increase when the width of the structures are also reduced. This gives a guide to the Nafion actuator structural design. For example, a family of geometric configurations on the contours can be found when an actuator is expected to achieve a full grasping motion with a given actuation voltage. In addition, for a given thickness, the contour is also a boundary for dimension selection. Most importantly, these plots show that the Electro-Mechano-Chemical coupling effects is also very important for designing micro Nafion actuators, i.e., to achieve full closure of an actuator under 2V, the width of the actuator should not be less than 40 μm, with l/w ratio of 2 (or 10μm width with l/w ratio of 8). Hence, base on our thick Nafion film experimental results, the minimum diameter of cell that can be enclosed by a Nafion actuator should be ~50μm if the film can be reduced to 0.2μm.

For our experiments of Au/Nafion/Au actuators (15mm long, 1mm wide) composed of hydraulically pressed Nafion film with thickness of ~50μm, the voltage for full actuation is about 2V. It is very close to the expected value of 1.62V calculated by our above analysis based on Nemat-Nasser and Li's model. However, for the approximate thickness of 0.2μm of the spin-on Nafion film for our microfabricated grippers, our experimental result as reported in preceding section shows the actual voltage for actuators 100 μm wide and 1.2mm long to reach full deflection is ~7V, which is much higher than the estimated value 0.16V. According to equation (5), we suggest the possible reason is the difference of the microstructural composition between the spin-on Nafion film and Nafion 117, i.e., the cluster radius, the distance between clusters, and the radius of the polymer chain. The difference of the property like effective dielectric permittivity may also play a role in this large discrepancy. As shown in Fig. 7 (c), our experimental results for the 0.2μm Nafion ICPF actuators consistently showed higher actuation voltage than the theoretical prediction. Moreover, the divergence remains when the theoretical calculation includes as much as 50% variation of thickness in each layer which may be induced by the fabrication processing of the sandwich structure in the worst case. It has to be mentioned that extra energy was consumed by the ionic motions in the other Nafion film under the top electrodes outside the released actuators. This might increase a little of the voltage value in the actuation excitation. Nevertheless, we can still conclude that thickness scaling has a significant effect on Nafion-based actuators. The comparison analysis between the experimental data and the model infers that the existing theories are not adequate in modeling this effect. Or, the microstructural composition of spin-on Nafion films is very different from the commercial membrane, because the ion formation in the clusters of the MEMS-fabricated Nafion actuator is more complicated than commercial membrane based ICPF actuators using other reported techniques. We are investigating the feasibility to control the ions in the ICPF film in our new MEMS-fabrication process.

5. Conclusions

A novel Ionic Conduction Polymer Film micro actuator was successfully fabricated on silicon substrate. We have demonstrated that this actuator can operate in aqueous environment by using ~7V to actuate full closure. The actuators can be potentially used for manipulation of micro objects under aqueous solution if electrolysis can be eliminated. By Nemat-Nasser and Li's electromechanical model to analyze our gripper actuation, we have shown that the thickness scaling effect is only valid within a certain Nafion thickness. More effort should be contributed to the characterization of the spin-on Nafion film so as to control its microstructural composition, if MEMS ICPF actuators are to be built and actuated under 2V.

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Biographies

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Wen J. Li received his B.S. and M.S. degrees in Aerospace Engineering from USC in 1987 and 1989, respectively. His industrial experience includes the Aerospace Corporation, Silicon Microstructures Inc., and the NASA/Cal Tech Jet Propulsion Laboratory. He obtained his Ph.D. degree from UCLA in 1997 specializing in MEMS. Prof. Li joined the Departmentof Automation and Computer-Aided Engineering of the The ChineseUniversity of Hong Kong (CUHK) in 1997. He has since then been active in MEMS and nanotechnology research. In the past five years he has published more than 90 papers in professional journals and conference proceedings on MEMS and nanotechnology related work. He gave a workshop on Microsensors and Microactuators for Robotics Applications at the IEEE/RJS IROS in November of 2000 Takamatsu, Japan) and a workshop on Manipulation in the Micro and Nano Domains: New Materials and Technologies at the IEEE ICRA in May of 2002 (Washington DC, USA). He has also helped organized several international conferences related to MEMS, robotics, and nanotechnology. He was the Chair of Organization Committee of the International Symposium on Smart Structures and Microsystems 2000, and was the General Co-Chair for the International Conference on Micro and Nano Systems (August 2002). Dr. Li is currently the Guest Editor for the Focused Section on Micro and Nano Manipulations of the IEEE/ASME Transactions of Mechatronics, and also as a member of the Technical Committee on Nanorobotics and Nanomanufacturing of the IEEE Nanotechnology Council. Prof. Li is now serving as the Director of the Center for Micro and Nano Systems at CUHK. His research interest is to develop micro and nano systems for micro/nano sensing and manipulation.