Improving electromechanical output of IPMC by high surface area Pd-Pt electrodes and tailored ionomer membrane thickness

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(Received 9 December 2013; final version received 7 April 2014)

In this study, we attempt to improve the electromechanical performance of ionic polymer–metal composites (IPMCs) by developing high surface area Pd-Pt electrodes and tailoring the ionomer membrane thickness. With proper electroless plating techniques, a high dispersion of palladium particles is achieved deep in the ionomer membrane, thereby increasing notably the interfacial surface area of electrodes. The membrane thickness is increased using 0.5 and 1 mm thick ionomer films. For comparison, IPMCs with the same ionomer membranes, but conventional Pt electrodes, are also prepared and studied. The electromechanical, mechanoelectrical, electrochemical and mechanical properties of different IPMCs are characterized and discussed. Scanning electron microscopy-energy dispersive X-ray (SEM-EDS) is used to investigate the distribution of deposited electrode metals in the cross section of Pd-Pt IPMCs. Our experiments demonstrate that IPMCs assembled with millimeter thick ionomer membranes and newly developed Pd-Pt electrodes are superior in mechanoelectrical transduction, and show significantly higher blocking force compared to conventional type of IPMCs. The blocking forces of more than 0.3 N were measured at 4V DC input, exceeding the force output of typical Nafion® 117-based Pt IPMCs more than two orders of magnitude. The newly designed Pd-Pt IPMCs can be useful in more demanding applications, e.g., in biomimetic underwater robotics, where high stress and drag forces are encountered.

Keywords: ionic polymer–metal composite; IPMC; electroactive polymer; palladium; electrode

1. Introduction

Ionic polymer–metal composites (IPMCs) are a type of electroactive polymers (EAPs) that have recently become increasingly important as soft actuators and sensors [1]. A typical IPMC material consists of a perfluorinated ion-exchange polymer membrane, such as Nafion®, which is plated on both faces with noble metal (Pt or Au) electrode layers [2]. The membrane also contains water as a working solvent and mobile cations such as Li⁺ or Na⁺. When a voltage is applied across the metal electrodes, IPMC exhibits a large bending deformation. Conversely, mechanical bending of the material can also induce the voltage [3]. Other notable features inherent to IPMCs include soft and flexible structure, low driving voltage (<5 V), large bending strain (~1%), and ability to operate in aqueous environment. These properties make IPMCs excellent candidates for underwater robotics,
medical and human affinity applications, and biomimetic robotics as the material can emulate the motion of biological muscles [4,5].

The manufacturing of IPMC materials is traditionally based on the electroless plating (aka impregnation-reduction, chemical deposition) of noble metals onto the surface of ionomer membrane [6]. Platinum and gold are commonly used electrode materials due to their high electrochemical stability and excellent electrical conductivity [2,7]. Platinum electrode, however, after prolonged cyclic deformation tends to develop cracks, leading to a higher surface resistance that in turn can adversely affect the actuation performance of IPMC [8]. Gold is more elastic and offers better mechanical durability [9]; however, the low stability of gold complexes in aqueous solutions makes the electroless plating process rather complicated. Also, several alternative electrode materials and their combinations have been explored. For instance, an electrode composed of platinum and copper has been proposed in which the reversible electrochemical reactions upon the actuation (i.e., dissolution and reduction of Cu^{2+} ions at the inner surface of the electrodes) can maintain electrical connections between the cracks of platinum layer [10]. Also, nickel and silver nanopowder have been used as cost-effective electrode materials [11,12]. However, low electrochemical stability of these materials limits the cycle life of IPMC.

Besides electrical conductivity, an interfacial surface area of electrodes is another important parameter that affects the performance of IPMC. It is becoming clear that the large specific surface area of electrodes leads to a higher electromechanical output [13]. This can be directly related to the accumulated charge near the electrodes. Namely, a study by Wallmersberger et al. [14] shows that in the calculations, large surface area of the electrodes corresponds to large effective dielectric permittivity value in the governing equations. This is illustrated in Figure 1, where the accumulated charge (normalized) is calculated for various dielectric permittivity values. It can be seen that the charge increases with the increase of the dielectric permittivity, or the area of the electrodes. In this regard, some authors have incorporated porous conductive powder materials into the electrodes to create a larger interface for the double-layer charging. These examples

Figure 1. Calculated normalized accumulated charge in the vicinity of cathode for different absolute dielectric permittivity values.
include use of ruthenium dioxide powder [15], carbon nanotubes [16], highly porous carbide-derived carbons [17], and carbon aerogels [18] in IPMC electrodes. However, powder materials require physical assembly by heat pressing and therefore are mainly suited for fabricating the so-called ‘dry type’ actuators based non-volatile electrolytes such as ionic liquids.

Recently, we reported palladium buffer-layered IPMCs [19] in which the palladium was used as a supporting layer on the surface of Naﬁon® 117 membrane (thickness 0.178 mm) underneath the platinum top layer. The Pd-Pt electrodes showed noticeable improvement in the actuation range and better mechanical stability compared to the conventional Pt electrodes. In this study, we attempted to increase the electromechanical performance of IPMC by developing high surface area Pd-Pt electrodes and tailoring the ionomer membrane thickness. First, IPMCs were fabricated on 0.5 and 1 mm thick perﬂuorinated sulfonic acid membranes. Thicker membranes were used in order to increase the maximum force output. For the two actuators both having the same length \( L \), width \( b \), but different thicknesses \( t \) and \( 2t \), the ratio of the stresses for the same tip displacement can be expressed as [20]:

\[
\frac{\sigma_{2t}}{\sigma_t} \approx 2 = \frac{F_{2t}}{2^2 F_t},
\]

where

\[
\sigma_t = \frac{6F_tL}{bt^2}.
\]

Therefore, the maximum blocking force for the thicker actuator is expected to be \( F_{2t} = 8F_t \), i.e., eight times larger than that of the actuator with thickness of \( t \). Second, we have modiﬁed the conditions of palladium electroless plating process and created highly dispersed Pd particles not only at the ionomer surface, but deep in the polymer membrane, thereby increasing notably the interfacial surface area of the electrodes. The latter has given a signiﬁcant increase to the blocking force output of IPMC. This paper reports and discusses the electromechanical, mechanoelectrical, electrochemical and mechanical properties of the newly designed 0.5 and 1 mm thick Pd-Pt IPMCs. Also, using the same ionomer membranes, 0.5 and 1 mm thick IPMCs with conventional Pt electrodes were fabricated and comparatively studied.

2. Experimental details

2.1. Chemicals and materials used

Perﬂuorinated sulfonic acid membranes with thicknesses of 0.5 and 1 mm were purchased from GEFC, Co., Ltd (Beijing, China) and were conditioned as described further in Section 2.2. The following reagents were of analytical grade and were used as received: tetraammineplatinum (II) chloride monohydrate (Pt(NH3)4Cl2·H2O, 98%, Sigma), tetraamminepalladium (II) chloride monohydrate (Pd(NH3)4Cl2·H2O, 98%, Sigma), sodium borohydride (NaBH4, 98%, Sigma), hydroxylamine hydrochloride (H2NOHHCl, 98%, Sigma), hydrazine monohydrate (H2NNH2·H2O, 98%, Sigma), lithium chloride (LiCl, 99%, Sigma), hydrogen peroxide (H2O2, 50%, Sigma), sulfuric acid (98%, Pharmaco-AAPER), ammonium hydroxide (35%, EM Industries, Inc.).
2.2. Fabrication of IPMC materials

First, the ionomer membranes were pretreated by roughening both surfaces with sandpaper (Grit #600) in order to enhance the physical bonding between the polymer and metal electrode. Both the surfaces were polished until the membrane was uniformly flat and non-transparent, after which it was thoroughly rinsed with deionized water. Then, the ionomer membrane was cleaned in 3 wt% hydrogen peroxide (H₂O₂) solution at 80°C for 45 min and then in 1 M sulfuric acid (H₂SO₄) solution at the same temperature and time. Finally, the membrane was boiled in deionized water for 30 min (twice) to remove acid residues.

The procedure for fabricating Pd-Pt electrodes was as follows: First, an impregnation-reduction process was carried out to incorporate palladium particles into the inner surface of the membrane. The pretreated membrane was soaked in palladium salt (Pd(NH₃)₄Cl₂·H₂O) solution for 1.5 h to impregnate the polymer with palladium complex ions. After rinsing thoroughly with deionized water, the membrane was immersed for 4 h in aqueous solution containing reducing agent such as sodium borohydride (NaBH₄) and ammonium hydroxide (NH₄OH) for pH adjustment. During this step, the palladium salt contained in the ionomer was chemically reduced to palladium metal (Pd°) at the inner surface of the membrane. The obtained composite was then cleaned in 1 M sulfuric acid solution for 30 min at 70°C and then in deionized water at the same temperature and time. Next, the platinum particles were deposited at the outer surface of the ionic polymer membrane using a chemical deposition method. The membrane was immersed in an aqueous platinum complex (Pt(NH₃)₄Cl₂·H₂O) solution and while moderately stirring, a mild reducing agents such as hydrazine monohydrate and hydroxylamine hydrochloride were added in every 30 min for 4 h to deposit the platinum on the palladium surface. After typical cleaning procedure in sulfuric acid and deionized water, the chemical deposition step was repeated 1–2 times, depending on the electrode surface conductivity.

IPMCs with Pt electrodes were fabricated using a traditional impregnation-reduction method with platinum complex (Pt(NH₃)₄Cl₂·H₂O) and sodium borohydride (NaBH₄) [6]. The impregnation-reduction cycle was performed twice in order to grow the platinum layer deeper into the polymer surface, thereby improving the reliability of electrodes against mechanical deformations. Additionally, the chemical deposition was carried out to deposit extra platinum particles onto the outer surface of the composite to further increase the electrical conductivity.

Using the aforementioned steps, four different sets of IPMC samples were fabricated – 0.5 and 1 mm thick Pd-Pt-IPMCs, and 0.5 and 1 mm thick Pt-IPMCs. All samples were cut into 10 mm × 50 mm rectangular shape and ion-exchanged to Li⁺-form by soaking in 1 M lithium chloride (LiCl) solution for overnight. Li⁺ ion due to its relatively small ionic radius and strong solvation in water provides optimal deformation performance and actuation speed, and is therefore commonly used for counter ion in IPMC studies.

2.3. Electromechanical and electrochemical characterization

The electromechanical responses such as displacement and blocking force of the prepared IPMCs were measured by a laser displacement sensor (optoNCDT-1401, Micro-Epsilon, Ortenburg, Germany) and a load cell (GSO-30, Transducer techniques) with a sample size of 50 mm × 10 mm in the test setup composed of a signal generator (FG-7002C, EZ digital, Bucheon, South-Korea), a power amplifier (LVC-608, AE Techron, Elkhart, IN, USA), a DC
power supply (Tektronix), and a DAQ (SCB-68, National Instruments, Austin, TX, USA) as illustrated in Figure 2. An IPMC was clamped in a cantilever configuration with a free beam length of 40 mm. The blocking force measurements were performed in air condition at room temperature (22°C) and normal humidity (~30%). The experiments were repeated with three samples from each type and the standard deviation percentages were within 16% from average, which is considered reasonable margin for soft polymeric actuators.

The mechanoelectrical transduction response was measured using an oscilloscope (PicoScope 2203, Pico technology, St. Neots, UK) and a vibration test system composed of a vibration controller (VR-8500, Vibration Research Corp., Jenison, MI, USA), a power amplifier (BAA-120, TIRA, Schalkau, Germany), and an electrodynamic shaker (S-521, TIRA) as shown in Figure 3. An IPMC sample with a size of 50 mm × 10 mm was clamped from both ends with a free beam length of 40 mm. The voltage output was measured at the clamp contacts.

Cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements were performed at ambient temperatures using a Radiometer Analytical PGZ-402 potentiostat (Radiometer Analytical, Lyon, France) in a three-electrode configuration. The two IPMC electrodes served as a working and counter electrode, and saturated calomel electrode (SCE) was used as a reference electrode. All measurements were performed in 0.5 M H₂SO₄ (aq) solution. Cyclic voltammograms of IPMC samples were recorded in a potential range of −0.25 to 1.2 V at a scan rate of 20 mV/s. The
capacitance versus frequency plots were obtained from EIS measurements at a frequency range of 1 kHz to 0.1 Hz using an AC perturbation of 10 mV and DC potential bias of 0.1 V versus SCE.

2.4. Scanning electron microscopy (SEM), energy dispersive X-ray (EDS) analysis, and flexural modulus characterizations

Scanning electron micrographs of IPMC cross sections were obtained using an Hitachi S-4200 microscope (Hitachi, Japan) in secondary electron image mode with a 20 kV accelerating voltage. EDS detector was used to perform chemical line-scan analysis in order to investigate the distribution of deposited electrode metals in the cross section of Pd-Pt IPMCs. The flexural (bending) modulus of the samples was determined from stress–strain measurements in a three-point bending mode using an Instron universal testing machine (5565, Norwood, MA, USA) (Figure 4). The three-point bending tests were performed in air condition at room temperature (22°C) and normal humidity (~30%).

Figure 3. Schematic of experimental setup used for mechanoelectrical characterization of IPMC (a) and corresponding photograph (b).

Figure 4. The three-point bending test setup used for determining the flexural modulus of IPMC.
3. Results and discussion

The cross sections of Pd-Pt IPMCs were examined using SEM-EDS to investigate the distribution of deposited electrode metals in the composite (Figure 5). The EDS line-scan analysis shows clearly that the palladium particles have penetrated deep into the polymer membrane, ranging more than 100 microns from the surface of 0.5 mm thick sample and more than 200 microns from the surface of 1 mm thick IPMC sample.

The widely dispersed palladium particles near the polymer surface provide a large interfacial area and the platinum layer at the outer surface of the composite serves as a highly conductive current collector. A higher content of palladium in case of 1 mm thick sample is probably due to the fact that the thicker membrane is able to absorb more palladium complex during the ion-exchange process, thus resulting in more Pd particles in the polymer after the reduction step. As can be seen, the middle region of the cross section is relatively void of Pd particles, indicating that the electrodes are still electrically insulated from each other. The low intensity for Pt or Pd in this region is mainly due to the noise signal. Electrolessly plated polymer membrane can have trace amounts of metal particles in the polymer, which can be also a reason for low-level detection of Pt or Pd through cross section. The thickness of the platinum layer at the membrane surface is around 7–10 microns, which is typical for Pt-electroded IPMCs and also for the Pt IPMCs fabricated in this work.

![Figure 5](image-url)

Figure 5. SEM micrographs of the cross sections of 0.5 mm (a) and 1 mm (b) thick Pd-Pt IPMCs, and corresponding EDS line-scan profiles for elements of Pt and Pd (c and d).
The CV was employed to investigate the electrochemical properties of the Pt and Pd-Pt IPMCs. Figure 6 shows the cyclic voltammograms measured in 0.5 M H₂SO₄ (aq) solution in a potential range of −0.25 to 1.2 V at a scan rate of 20 mV/s. An approximately exponential increase in the current density corresponds to the cathodic hydrogen evolution process and the flat wide maximums characterize the ionization of adsorbed hydrogen at the Pt and porous Pd-Pt electrodes. A large potential difference between the overpotential of cathodic evolution and ionization process refers to a slow mass transport in the porous Pd-Pt system. The region between 0.2 and 0.5 V corresponds to ideal polarizability, where the electrical double-layer formation occurs. It can be seen that the Pd-Pt samples have the highest peaks in that potential range, especially the 1 mm thick sample. This is in agreement with SEM-EDS analysis (Figure 5) that also shows higher content of palladium for 1 mm Pd-Pt IPMC. Starting from 0.6 V, the adsorption of oxygen occurs at the Pt electrodes and more intensely at the Pd-Pt electrodes. The wide peaks at the reduction curve at 0.3 V characterize the electroreduction of adsorbed oxygen at the Pd-Pt and Pt electrode surface. A large difference between the peak potentials in oxidation and reduction curves indicates the irreversible nature of oxygen adsorption. All these processes occur more intensely at the Pd-Pt electrodes, indicating their large interface.

Figure 7 presents the capacitance of different IPMC samples as a function of frequency, determined from EIS measurements. The data indicates that the widely dispersed palladium in the electrodes increases significantly the capacitance of IPMC at the lower frequencies (<10 Hz). The capacitances at 0.1 Hz are up to 110 and 70 mF/cm² for 1 and 0.5 mm Pd-Pt IPMCs, and 43 and 10 mF/cm² for 1 and 0.5 mm Pt IPMCs. These results are consistent with the CV measurements that also indicate higher capacitance for the Pd-Pt samples (Figure 6). It should be noted that while the CV results are in good agreement with our previous paper, it is difficult to compare the EIS data due to very different measurement conditions used [19]. Namely, the EIS measurements in our previous study were performed at very high DC bias potentials – at 2 V (as opposed to
0.1 V used in this work). AC voltage perturbation at high bias potentials can prevent electrolyte ions from adsorbing effectively at the electrodes with porous interface. The faradic processes (i.e., water electrolysis) occurring at this voltage can cause a blocking effect of the pores by adsorbed gaseous products (H₂ and O₂), leading to lower value of capacitance [21]. This explains the lower EIS capacitance observed for Pd-Pt electrodes in our previous study compared to Pt electrodes, while the CV measurements (at 20 mV/s) clearly indicated the opposite and are consistent with this study.

Figure 8 shows the flexural (bending) moduli of Pt and Pd-Pt IPMCs with different thicknesses determined from the three-point bending test. It should be noted that the IPMC’s flexural rigidity can have a great impact on its electromechanical performance. Generally, a stiffer sample exhibits less displacement, but on the other hand, is capable of
generating higher blocking force, since the rigid structure of material is able to sustain more stress. The measurements show that IPMCs with Pd-Pt electrodes have noticeably higher stiffness compared to Pt IPMCs. The respective flexural modulus values for Pd-Pt samples are 225 MPa (0.5 mm) and 262 MPa (1 mm), whereas the flexural modulus for Pt samples are 198 MPa (0.5 mm) and 218 MPa (1 mm). This result can be expected since the ionomer membranes in case of Pd-Pt IPMCs contain palladium that gives rise to the stiffness.

The displacement measurements were carried out at different actuation frequencies from 0.1 to 1 Hz at ±3 V square-wave input. Figure 9 shows the measured actuation in terms of displacement (mm) and Figure 10 in terms of bending strain (%). The bending strain ($\varepsilon$) was obtained from the measured displacement ($\delta$) using the following relation [2]:

$$\varepsilon = \frac{2\delta t}{L^2 + \delta^2},$$  \hspace{1cm} (3)

where $L$ is the free beam length and $t$ is the cross-sectional thickness of IPMC. Due to a large variation of thicknesses of the samples, the data in terms of displacement provides better perspective of the actuation range. The results show that the displacements of the samples are relatively minor at 1 Hz and increase noticeably as the actuation frequency is decreased to 0.1 Hz. It should be noted that these IPMCs have slower actuation response compared to typical Nafion 117 membrane-based Pt IPMCs that are capable of generating adequate displacement also at 1 Hz input signal [3]. This can be related to the much larger thickness of the samples, requiring longer time for electrolyte migration and diffusion between the electrodes. As observed in Figure 9, the 0.5 mm thick samples show noticeably larger deflection at lower frequency (0.1 Hz) compared to 1 mm samples.

![Figure 9. Displacement performance of different IPMCs versus frequency at ±3 V square-wave input.](image-url)
Figure 11 shows the strain rates (actuation speed) of different samples at 0.1 Hz square-wave input, calculated by taking the time derivative of the bending strain. It can be seen that 0.5 mm thick samples are capable of faster actuations with much higher peak strain rates compared to 1 mm thick IPMCs. The 1 mm thick Pd-Pt IPMC shows the lowest strain rate among the samples.

Figure 11. Strain rates (%/s) of Pt and Pd-Pt IPMCs actuation frequency at ±3 V square-wave input.
The electromechanical performance of Pt and Pd-Pt IPMCs was also evaluated in terms of blocking force. Figure 12 presents the measured blocking force response in time at 3 and 4 V DC input. It can be seen that IPMCs with Pd-Pt electrodes show significantly higher blocking force output compared to Pt IPMCs with respective thicknesses. At 3 V DC input, the peak blocking forces for 1 and 0.5 mm Pd-Pt IPMCs are nearly 200 and 60 mN, whereas the forces for 1 and 0.5 mm Pt samples are 50 and 9 mN. Moreover, at 4 V DC input, the blocking forces more than 0.3 and 0.1 N were recorded for 1 and 0.5 mm Pd-Pt samples. The peak forces for Pt samples were 0.1 and 0.03 N. The higher performance of Pd-Pt electrodes is related to highly capacitive interface of dispersed Pd particles near the Pt surface, leading to higher electrolyte transport and adsorption at the electrodes, as observed from EIS capacitance measurements (Figure 7). A higher performance of 1 mm samples over their 0.5 mm thick versions can be due to a combination of several effects: twice larger thickness, higher interfacial surface area of electrodes, and higher stiffness. It should be noted that the 1 mm Pd-Pt IPMC has noticeably higher capacitance and stiffness compared to other samples, which can contribute to a higher force output. However, the palladium in the polymer matrix can inhibit the electrolyte diffusion, lowering the rate of double-layer charging, which is reflected in the slower force response in the case of 1 mm Pd-Pt IPMC. For comparison, the blocking forces of the previously reported Pt and Pd-Pt IPMCs made on conventional Nafion 117 membranes were 0.3 and 1.1 mN, respectively [19]. Thus, by incorporating thicker ionomer membranes and high surface area Pd-Pt electrodes, the blocking force performance of IPMC was significantly increased.

Besides functioning as an electromechanical actuator, IPMC can also operate in a reverse mode, as a mechanoelectrical transducer, i.e., generate voltage upon mechanical deformation. The mechanoelectrical transduction of Pt and Pd-Pt IPMCs was monitored using an electrodynamic shaker assembly, which was programmed to apply a constant sinusoidal displacement to the IPMC tip in a sweeping frequency mode. Since the shaker had limits regarding the displacement range at higher frequencies, the measurements were performed at two different displacement and frequency settings: (a) at 4–40 Hz with 5 mm displacement, and (b) at 4–20 Hz with 10 mm displacement. Figure 13 shows the induced voltage amplitude as a function of imposed actuation frequency at these testing regimes. It
can be seen that IPMCs with Pd-Pt electrodes generate considerably higher output voltage compared to Pt IPMCs, especially at the higher frequencies. This can be related to the larger interface of Pd-Pt electrodes, allowing more effective surface charging. It is interesting to note that the output voltage generated from Pd-Pt electrodes increases drastically with the increase of actuation frequency, while in case of Pt electrodes the overall change in the signal amplitude is rather minor. It can be also noticed that the voltage produced at 10-mm displacement is almost double the voltage amplitude at 5-mm displacement, indicating that the mechanoelectrical output of IPMC is strongly dependent on the amount of applied deformation.

4. Conclusions
In this work, IPMCs with high surface area Pd-Pt electrodes and thick ionomer membranes (0.5 and 1 mm) were developed. Also, using the same ionomer membranes, IPMCs with conventional Pt electrodes were fabricated and comparatively investigated. The electromechanical, mechanoelectrical, electrochemical and mechanical properties of different samples were evaluated and analyzed. The results demonstrate that the newly designed Pd-Pt IPMCs are superior in mechanoelectrical transduction and exhibit significantly higher blocking force compared to the IPMCs made with conventional Pt electrodes. The peak blocking forces more than 0.3 N were measured for Pd-Pt IPMCs and more than 0.1 N for the Pt-IPMCs at 4 V DC input, exceeding the force output of typical Nafion 117-based Pt IPMCs more than two orders of magnitude. The higher performance of Pd-Pt electrodes is related to highly capacitive interface of dispersed Pd particles near the Pt surface, leading to higher electrolyte transport and adsorption at the electrodes – responsible for the actuation process of IPMC. Both types of IPMCs showed adequate displacement performance, however, the actuation response was slower compared to typical Nafion 117-based Pt IPMCs. The SEM-EDS cross-sectional analysis indicated that the palladium particles were widely dispersed in the ionomer membrane, thereby increasing the interfacial surface area of electrodes and the capacitance of IPMC. The measured capacitances were in good correlation with the blocking force data of the
samples. Thus, by incorporating millimeter thick ionic polymer membranes and high surface area Pd-Pt electrodes, the IPMC performance was successfully improved.

Acknowledgments
This work was supported in part by the Office of Naval Research under Grant N000140910218 and N000141310274.

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