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# Electrode reactions in Cu-Pt coated ionic polymer actuators

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#### Abstract

Composite actuators consisting of sheets of the solid polymer electrolyte (similar to Nafion<sup>®</sup>) with  $Cu^{2+}$  counter ions inserted and coated with platinum and copper metal layers (so-called ionomeric polymer–metal composites; IPMCs) have been synthesised and their electromechanical performance upon actuation has been monitored. Resistance measurements on the electrodes show that the electrical conductivity of the membranes metal surface increases on the cathode side during the actuation process, contradictory to the situation when Cu is absent from the metal coating. This phenomenon is explained by the subsequent reduction of  $Cu^{2+}$  ions on the cathode upon actuation; Cu layer growth in this side prevents it from cracking and decreases its electrode resistance. The phenomenon opens up for longer lifetimes for Cu-based IPMCs. However, additional problems with Cu layer oxidation and Cu dendrite growth on the electrodes should be considered. © 2007 Elsevier B.V. All rights reserved.

Keywords: Ionomeric polymer-metal composite; Nafion; Copper-platinum electrode; Electrode resistance; Copper growth; SEM

### 1. Introduction

Electroactive polymers (EAPs) are polymer materials that change their shape or size in response to electrical stimuli. The feature makes this class of materials to a good candidate for using in the field of biomimetic devices, since it mimics the behaviour of biological muscles. EAPs inhibit similar properties to biological materials in terms of force, strain and speed. They are attractive for creating artificial muscles used in biologically inspired robots [1], biomedical devices and security applications [2].

Ion conducting polymers have been used widely as EAP materials. They have the advantage of operating at low electric voltages. Among the most commonly used ionic EAP systems are the ionomeric polymer–metal composites (IPMCs), consisting of a 200–400  $\mu$ m thick ionomer membrane (most commonly Nafion<sup>®</sup> [3]) plated with metal (typically with Pt or Au) and with metal ions coordinating to the ionic groups in the polymer. The material exhibits a bending motion when a potential of 1–5 V is

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0925-4005/\$ - see front matter © 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.snb.2007.11.044 applied over the metal surfaces [4]. The applied electrical field causes the mobile cations in the swollen polymer film to move towards the cathode, which leads to an expansion at this side of the membrane and therefore causes the membrane to bend towards the anode in an actuating motion [5].

One of the problems facing these materials, and their future use as actuator, is that the bending motion gives rise to cracks in the surface metal layers that constitute the electrodes. The cracks lead to leakage of solvent from the ionomer, and thus to lower ion conductivity and a lower degree of bending. The phenomenon also gives rise to a higher electronic resistance in the surface electrode layers, and thus to slower response time and a smaller actuating motion. Therefore, an increase of active lifecycles could be achieved by improving the mechanical and electrical properties of the surface electrode layers [6].

In our study presented here, we report on our efforts to overcome the problem with the metal cracking by using Cu as a composite material together with Pt as electrode, and Cu<sup>2+</sup> as counter ion to the Nafion<sup>®</sup> sulphonate groups. Similar type of IPMC materials are reported first by Uchida and Taya [7–9]. They showed that when a voltage is applied over the membrane, the following electrochemical reactions occur in the material: U. Johanson et al. / Sensors and Actuators B 131 (2008) 340-346

Anode reaction:

$$Cu(s) + 5H_2O \rightarrow Cu^{2+} \cdot 5H_2O + 2e^{-1}$$

Cathode reaction:

 $Cu^{2+} \cdot 5H_2O + 2e^- \rightarrow Cu(s) + 5H_2O$ 

They also observed that when a series of electrical impulses with the same voltage polarity were applied for a long time, copper on the surface of the anode vanished. At the same time a new copper layer was formed on the cathode side. In the meantime,  $Cu^{2+}$  ions in the polymer transported water molecules from the anode to the cathode side. The process is reversible, so that when the polarity of applied impulses to the electrodes change, the water and Cu-ions transport goes in the opposite direction through the membrane.

Since the expansion of the cracks in the Pt layer occur on the same side of the membrane as the Cu layer growth (*i.e.* the cathode side), this growth may compensate for some of the cracking, leading to increased electrical conductivity and higher mechanical and chemical stability in the cathode surface layer. We have monitored this by measuring the resistance per unit length in the two surface layers using the non-symmetrical actuation process.

#### 2. Experimental

#### 2.1. IPMC synthesis

Ready-made Pt-coated Na-ions containing MuscleSheet<sup>TM</sup> IPMCs were purchased from BioMimetics Inc. These IPMCs are 0.2-0.4 mm thick, and contain ionic polymer which is similar to Nafion<sup>®</sup> 1110 but not fully fluorinated. Three 3 cm long and 1 cm wide strips were cut out, and their actuator response was tested prior to further synthesis. A scanning electron microscope (SEM) image of the cross-section of an IPMC sample is shown in Fig. 1. Subsequently, the strips were boiled for 20 min in 1 M HCl(aq) to substitute all Na<sup>+</sup> ions with H<sup>+</sup> ions, after which the samples were cleaned from residue of hydrochloric acid by boiling in deionised water. Thereafter, the sheets were put in 2 M CuSO<sub>4</sub>(aq) for 12 h; afterwards the presence of  $Cu^{2+}$ -Nafion<sup>®</sup> was verified by optical microscopy: the cross-section of the membranes had a clear blue colour. The blue colour also indicates that each  $Cu^{2+}$  ion is complexed by 5 molecules of water. Finally, the IPMC sheets were coated with Cu layer by electrochemical plating from CuSO<sub>4</sub> solution. All chemicals used were analytical grade, and were used without further purification.

The quantity of copper deposited during the electrochemical plating was controlled by the amount of charges passing through the system during the copper layer preparation. Calculated from the passed current, the average thickness of the copper layers was 2  $\mu$ m. The formed copper layers were however uneven, which is a familiar effect of the electrical field being stronger at the rim, therefore resulting in a faster metal layer formation there. An X-ray line-scan of the IPMC cross-section (Fig. 2) shows how

2<u>0 µm</u>

Fig. 1. SEM image of the cross-section of a MuscleSheet  $^{TM}$  IPMC before copper deposition.

copper, Cu-ions and platinum are distributed over the IPMC cross-section.

For detection of  $Cu^+$  compound formation, some samples were left overnight in 2 M CuSO<sub>4</sub>(aq) while other samples were left in deionised water prior to X-ray microanalysis.

# 2.2. Surface resistance measurements and X-ray microanalysis

The experimental setup used for the resistance measurements and its benefits in the context of IPMCs have been described in



Fig. 2. X-ray line-scan of the IPMC cross-section after copper layer deposition on the electrodes.



Fig. 3. The experimental setup for measuring resistance on the surface layer of an IPMC.

detail elsewhere [10]. A schematic picture of the experimental setup is shown in Fig. 3. We clamped the IPMC strips in vertical cantilever positions in a container filled with de-ionised water. Four pairs of contacts (gold) were attached to each sample (see Fig. 4). These contacts were made as lightweight as possible, but strong enough to ensure good contact. The measurements were conducted using National Instruments LabView7 control software. The driving voltage was generated by NI PCI-6703 DAQ board and amplified with NS LM675 power op-amp. Voltages on all contacts with respect to the ground were measured with National Instruments PCI-6034 DAQ board.

The measurements of the surface resistance were conducted using a four-probe system. This method has been shown to eliminate inexactnesses caused by the inconsistent current density and resistance from the contacts [11,12]. The actual values of the reference voltage  $U_0$  and the resistor R in Fig. 3 were selected



Fig. 4. A Cu-coated IPMC sample with contacts.



Fig. 5. Driving pulses during one cycle in a resistance measurement.

according to the range of the DAQ performing the measurements, but were all far below the voltage limit for electrolysis. The actual electric current during the measurement of membrane surface metal layer (electrode) resistance was in the range of 1-6 mA.

In order to ensure galvanic insulation of the contacts on the opposite sides of the sample during the measurements, a 8-channel 3-position switch was used. In position A in Fig. 3, the driving voltage from the switch is fed to contacts 1-2 on the sample. In position B, the surface resistance on one side of the sample is measured between contacts 3-5, using the contacts 1-3-5-7. In position C, the surface resistance on the other side of the sample is measured between the contacts 4-6.

The measurements were conducted in the following order:

- 1. The switch was in position A, and the samples were fed with asymmetrical driving pulses: 2 s with one polarity, followed 1 s with the opposite polarity. The samples were treated for 100 cycles before the surface resistance was measured.
- 2. By swiftly switch to positions B and C, the surface resistances of both sides of the samples were measured.

The experiment was repeated by applying reversely asymmetrical driving pulses; the pulse for one cycle is shown in Fig. 5.

To study the growth of Cu dendrites, the muscle sheet actuation was driven at a too high voltage for this kind of material -4.5 V, which made the system short-circuit after 300 cycles. Samples prepared this way were used for X-ray microanalysis measurements and for taking scanning electron microscope images.

The composition of the IPMC actuators was determined with X-ray microanalysis using electron-probe microanalyzer JXA-840 (JEOL, Japan) supplied with energy dispersive spectrometer Voyager (Noran, USA). The  $K_{\alpha}$  lines were used as analytical X-ray lines for the determination of the content of Cu, Cl, S and the  $L_{\alpha}$  line for Pt. Tugtupite and pyrite minerals, covered with thin carbon layer, and tested by chemical analysis, where used as standards for the analysis of Cl and S, respectively. The influence of carbon layer was taken into account with using thin film analysis program Stratagem (SAMx, France). For the analysis of Cu and Pt high purity metal standards were used. The scanning electron micrographs were obtained using the same electron-probe equipment in secondary electron image mode with 10 keV primary electron energy.



Fig. 6. Electrode resistance per unit length at the left and right electrode for MuscleSheet<sup>TM</sup> IPMCs upon mechanical bending. Bending to negative angles corresponds to bending towards the left.

#### 3. Results and discussion

#### 3.1. Pt-coated actuators

The data presented in Fig. 6 shows how the surface resistance changes for the first two strips upon mechanical bending, prior to coating with Cu. The strips are somewhat bended in their relaxed states, which makes the resistance differ at the two sides at 0 degree bending. This is an artefact of that the Pt metal layer is uneven at the two different sides of the membrane.

It is clear here that the strips behave as expected: when bended to the left, the metal surface layer on the right side of the electrode cracks, which results in a severe increase in resistance per unit length. At the same time, the resistance on the left side of the material decreases, due to that the metal particles are being pressed together. This process is reversible, so that when the material is bent in the other direction, to the right, the resistance drops on the right side, while it increases on the left. Similar results have already been shown for the same kind of IPMCs [10].

### 3.2. Cu-Pt coated IPMCs

Table 1 summons the resistance measurements of the three samples after they have been coated with an additional copper layer. Just as for the untreated membranes, there is a large difference in electrode resistance per unit length between the left and right side of the membranes, and between the individual membranes, probably due to cracks and other imperfections in the coating metal layers. All samples were then first bended to the right direction for 100 cycles, where the motion during one cycle was actuated by a 2V pulse of 2 s, followed by a opposite potential step from a -2V pulse during 1 s. It could be observed that the bending angle decreases continuously over the cycles, with the initial bending being at around 45 degree.

Due the ionic conductivity of the membrane, it is impossible to measure the electrode resistance directly at the same time as when the voltage is applied to the sheet and therefore the corresponding measurements were done quickly after switching off the voltage. However, after switching off the outside voltage a very fast relaxation process of the sheet occurs. It relaxes back from its bent state due to water leakage through the electrode cracks and water diffusion towards the anode. This relaxation goes faster for the Cu/Cu<sup>2+</sup>-based IPMCs than for the conventional forms, based on Li<sup>+</sup>- or Na<sup>+</sup>-Nafion<sup>®</sup>. The reason for this is that the water molecules transported to the electrode surface are uncomplexed to any cation after the reduction of  $Cu^{2+}$  to Cu, and therefore are freely mobile. All measurements of the electrode resistance were thus done in non-bent state. After these measurements, the polarity of the driving pulses was switched, and the samples were bent another 100 cycles to the left direction. Finally, they were bent yet another 100 cycles to the right direction.

Table 1

Electrode resistance per unit length at the left and right electrode for Cu-coated MuscleSheet<sup>TM</sup> IPMCs after bending with cycles containing asymmetrical steps [see Fig. 5] in one direction (first, +2 V for 2 s and -2 V for 1 s after that) or in another direction (first, -2 V for 2 s and +2 V for 1 s)

Total no. of bending cycles	Bending direction during next 100 cycles	Left electrode resistance ( $\Omega$ /cm)	Right electrode resistance ( $\Omega$ /cm)
Sample 1			
0		9.5	57
100	Right	2.4	>400
200	Left	5.7	0.95
300	Right	0.62	1.7
Sample 2			
0		4.1	26
100	Right	1.5	>400
200	Left	2.1	3.2
300	Right	0.58	21
Sample 3			
0		2.0	13
100	Right	1.2	200
200	Left	2.0	0.65
300	Right	0.60	100

It is clear from the data presented in Table 1 that the Cucoated membranes display an opposite phenomenon compared with those only coated by Pt. The resistance per unit length increases at the side to which the material is bended (the anode side) and it decreases at the other (cathode) side. Therefore we assume (in accordance with the discussion in the Section 1) that a new layer of Cu is formed between Pt-particles in the electrode layer and also in the Nafion<sup>®</sup> matrix close to the Pt-electrode on the cathode side. At the same time, Cu is depleted from the anode side during the creation of  $Cu^{2+}$  ions. Both these electrode reactions occur while the Cu<sup>2+</sup> ions migrate towards the cathode in the polymer matrix, and thus the process assists to pump water in that direction, causing the actuating motion. This continuous production of Cu<sup>2+</sup> also represents an elimination of another limitation to the use of IPMCs, since back-diffusion of cations towards the anode will be much less of a problem. Yet another positive factor to take into account is that the number of possible mobile cations increases with several orders of magnitude compared to IPMCs without electrochemical electrode reaction. In IPMCs the number of mobile ions is usually limited by cations initially paired to the polymer-SO<sub>3</sub><sup>-</sup> groups, while here new  $Cu^{2+}$  ions are created at the anode during the actuation process. Therefore the water quantity which can be transported in this system should be significantly higher. This, however, increases the importance of solvent leakage protection at the electrodes.

Although the variations in resistance per unit length is large, and fluctuates severely upon switching the polarity for 100 cycles, it is also clear from the data presented in Table 1 that the final resistance values of the electrodes are *lower* than the initial values prior to cycling. The only exception to this rule is the right side electrode of sample 3, which however displayed highly fluctuating values in resistance, possibly due to an imperfect Pt coating. In fact, the data in Table 1 suggests that the electrode conductivity is continuously improving upon cycling. These results indicate that the Cu layers in the samples are displaced over the cycling process, getting more evenly distributed over the surface and in better contact with the Pt-particles. From the X-ray line-scan of a sample membrane, it can also be seen that the Cu layer re-locates during cycling: from its position on the outer surface of the platinum electrodes directly after electroplating (Fig. 2), it is transported to the electrodes inner surface (Fig. 7).

The results in Table 1 are promising in the context of developing IPMCs, since they show that the problems of cracking here have been overcome, which opens up for longer lifetimes. The problem with this type of IPMCs rather seems to be the Cu depletion on the anode side, which possibly is the reason for the decreasing bending angle upon cycling. This problem should however be possible to solve by improving methods of preparation of copper on the electrodes, or any other electronically conductive additive to the membrane surfaces. The artificial muscle fibres also actuate at a low voltage, which avoids the problems of electrolysis – common for other IPMCs – and generates bending as far as 90° already at 2 V.

In [13], we have described a self-sensing actuator by measuring the changes of the surface resistance with Pt-plated IPMCs. The disadvantage of the Cu-plated actuator in this context is that



Fig. 7. X-ray line-scan of the Cu-IPMC cross-section after cycling 300 cycles with voltage pulses of 2 V magnitude.

the electrode resistance changes during cycling, and therefore not only depend on the bending angle of the actuator.

#### 3.3. Unwanted side-reactions

Although the resistance data for the Cu-coated IPMCs look promising, we observed unwanted side-reactions during our measurements. These side-reactions in turn cause new problems for the IPMCs. First, if the sheet actuation was driven at too high voltage for a such type of material -4.5 V, the system clearly short-circuit after 300 cycles. This indicates the growth of Cu dendrites, which is known to occur in many other systems for example lithium batteries [14], and was confirmed by optical microscopy and SEM. An SEM line-scan of a cross-section of a copper plated actuator (Fig. 8) shows that there is a substantial amount of unevenly distributed copper inside the IPMC membrane. More importantly, the SEM image also shows that the actuator contains a lot of holes in polymer matrix (Fig. 9). The only obvious explanation for these holes is that they are results of micro-explosions of detonating gas (a 2:1 mixture of hydrogen and oxygen), which melts the surrounding polymer. Both gases are produced on the copper dendrites (which in turn are connected with electrodes) during the alternating polarity:

Cathode:

 $2H^+ + 2e^- \rightarrow H_2(gas)$ 

Anode:

$$2OH^- - 2e^- \rightarrow 1/2O_2(gas) + H_2O$$



Fig. 8. X-ray line-scan of the Cu-IPMC cross-section after cycling 300 cycles with voltage pulses of 4.5 V magnitude.

For the gas explosion to occur, however, a catalyst (platinum) is needed at the room temperature. Nevertheless, an actuator with platinum electrodes, and which is prepared using impregnation–reduction methodology, always contains small amount of platinum inside of the polymer matrix (these can also be seen on the X-ray line-scan; Fig. 2).

Also interesting is the distribution of holes inside the membrane—at some points in the samples, they are located only close to the electrodes, indicating that there are no long dendrites inside the polymer matrix in these areas. In other cases, measured closer to the contacts, these holes are also found quite far from electrodes and deep inside the polymer matrix, thereby indicating the existence long dendrites. X-ray maps also show



Fig. 9. SEM image of destroyed Cu-IPMC cross-section after cycling at 4.5 V.

that the total amount of copper inside the polymer matrix is higher in this case, and that these higher concentrations can be found deeper inside the polymer matrix.

Another observation is the appearance of non-conductive insoluble  $Cu^+$  compounds on the electrodes.  $Cu^+$  ions are created through the spontaneous redox reaction between Cu atoms in the metal electrode layer and  $Cu^{2+}$  ions in the membrane:

$$Cu(s) + Cu^{2+} \rightarrow 2Cu^{+}$$

 $Cu^+$  compounds are quite stable in these conditions (often insoluble in water), which means that the salt formation depletes the Cu(s) layers, and thus raises the resistance again on the electrode surfaces; the salts are also electronically isolating. The formation of Cu<sup>+</sup> salts severely decreases the lifetime of the IPMCs, and it was therefore necessary to conduct our resistance measurements directly after the Cu-coating. If the samples were left overnight in 2 M solution of CuSO<sub>4</sub>, the Cu layer had transformed almost completely into a Cu<sup>+</sup> salt. In addition, initially blue coloured ionic polymer layers turned colourless after long time (several days) due to the reduction of blue coloured Cu<sup>2+</sup> to colourless Cu<sup>+</sup> compounds.

Using X-ray microprobe elemental analysis, we found that this salt only contains the elements copper and chlorine. It gives us reason to suppose that it is CuCl, since there exists no other insoluble compound containing only these two elements. When putting fresh copper plated IPMC samples in deionised water, *i.e.* without boiling it in hydrochloric acid first, no essential changes could be observed. We therefore reached the conclusion that small remains of  $Cl^-$  ions after the cleaning in hydrochloric acid caused the CuCl (s) formation.

# 4. Conclusions

We have here presented a quantitative study of the variations in electrode resistance per unit length for Cu-coated Nafion<sup>®</sup>-based IPMCs upon actuation cycling. We showed that the depletion of Cu(s) at the anode side, and the formation of a new Cu(s) layer at the cathode side, give raise to an opposite resistance per unit length profile than for ordinary Pt-coated IPMCs. This, in turn, represents an excellent possibility to overcome the problem of resistivity increase due to the electrode cracking on the cathode side upon actuation. Copper layer relocation during the actuation cycling is confirmed by Cu-IPMC actuator cross-section X-ray analysis. However, formation of Cu dendrites and insoluble Cu<sup>+</sup> compounds cause severe limitations for these IPMCs, and these problems must be treated further. It is also necessary to overcome the water leakage through the electrode cracks.

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## Biographies

**Urmas Johanson** received his PhD in chemistry in the field of electrochemistry of organic conductive polymers at University of Tartu, Estonia, in 2004. Since 2005, he is a researcher in Tartu University Institute of Technology Intelligent Materials and Systems Laboratory. His current field of interest is preparation and study of new materials for electroactive polymer based actuators and sensors.

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Prof. Väino Sammelselg was born in Tallinn, Estonia, in 1949. He studied applied inorganic chemistry in Chemistry High School Kohtla-Järve (1964-1968) and solid-state physics at the Tartu University (1968-1973), which he finished with diploma work (equated to MSc) on XRD of ammonium halides solid solutions. He received his PhD degree from the Institute of Physics (IP), Academy of Sciences of Estonia in 1989 on electron-probe investigations of AlGaAsSb solid solutions and heterostructures. Over 25 years, he worked in the IP (belonging to UT since 1988) starting as an engineer and reaching senior researcher, but also worked many short periods as visiting scientist in Finland, Sweden, and Russia. Since September 2003, he is working as professor of inorganic chemistry at Tartu University and since 2007, as vice head of department of materials science in IP. His current research is focused on the oxide and polymer (ultra) thin film preparation and characterization, and on the solid surface modification. He is the author or co-author of 100 original scientific publications. Twice, in 1982 and 2007, he has been awarded with the Science and Technology Prize of Estonia.

**Daniel Brandell** took his PhD in chemistry in the field of lithium ion polymer electrolytes at Uppsala University, Sweden, in 2005. He was then active at the Tartu University, Estonia, during 2006, and is currently doing post-doc studies at Virginia Polytechnic and State University, USA. His research interests concern batteries, fuel cells and electroactive polymers.

Andres Punning graduated from the Department of Physics of Tartu University in 1984. From 1984 to 2002, he has worked on various positions in the field of information technology and telecommunications. Since 2002, he is employed as a researcher in Tartu University Institute of Technology Intelligent Materials and Systems Laboratory. In 2007, he received PhD degree in Tartu University. His PhD work was about modelling of the ionic polymer-metal composites. His current field of interest is development of electroactive polymer actuators and sensors.

**Maarja Kruusmaa** received her PhD degree in computer engineering from Chalmers University of Technology, Gothenburg, Sweden. Since 2003, she is a senior researcher in Tartu University Institute of Technology, Estonia, and a co-founder of Intelligent Materials and Systems Laboratory. Since 2007, she is a professor of biorobotics in Tallinn University of Technology and a head of Center for Biorobotics. Her research interests include new materials in robotics, biomimetic robots, underwater robots and robot learning.

**Alvo Aabloo** graduated from the Department of Physics of Tartu University in 1989 and PhD in 1994. His PhD work was about computer modelling of celluloses. In 1995–1996, he was a postdoc at Uppsala University. His research interests are ion conducting polymers, electroactive polymers and their applications, computer modelling of polymer materials.