Formation and characterization of Pt-Cu-IPMC with high specific surface area and dendritic electrode

Aifen Tian\textsuperscript{a}, Jiahua Li\textsuperscript{a*}, Xixi Wang\textsuperscript{a**}, Yue Sun\textsuperscript{a}, Xinrong Zhang\textsuperscript{b}, Shanshan Yao\textsuperscript{c} and Huiling Du\textsuperscript{a}

\textsuperscript{a}School of Materials Science and Engineering, Xi'an University of Science and Technology, Xi'an China; \textsuperscript{b}Key Laboratory of Expressway Construction Machinery of Shaanxi Province, Chang'an University, Xi'an China; \textsuperscript{c}Department of Mechanical Engineering, 161 Light Engineering, Stony Brook University, Stony Brook, USA

\textbf{ABSTRACT}

Ionic polymer–metal composite (IPMC) is a kind of electroactive intelligent material. The dendritically interfacial electrodes (DIEs) is very important to the performance of IPMC. Herein, the copper–platinum composite electrode (Pt-Cu-IPMC) with a high specific area DIEs was prepared by combining impregnation-reduction plating platinum (IRP-Pt), autocatalytic plating platinum (ACP-Pt) and chemical reduction plating copper (CRP-Cu). The effects of DIEs and the cycles of ACP-Pt have been systematically investigated on the electrode's thickness, micro-morphology and electromechanical response characteristics of all samples. Importantly, when the cycles of ACP-Pt increases, the platinum and copper particles in the ionomer membrane are highly dispersed. For example, when the cycles of ACP-Pt is 4, the penetration depth of the DIEs reaches 31.98 \( \mu \)m. Thus, copper and platinum dendrites were clearly observed in the samples. Combined with the analysis of the strain energy density, it showed that the presence of DIEs has some impact on the driving performance of the IPMC, the tip displacement, and blocking force of 67.27 mm and 27.21 mN have been achieved in the Pt-Cu-IPMC after three cycles of ACP-Pt.

In this paper, five IPMCs with copper and platinum composite electrodes were prepared by seven processes: pretreatment, ion adsorption (Pt\((\text{NH}_3)_2\text{Cl}_2\) solution), impregnation-reduction plating platinum (IRP-Pt), autocatalytic plating platinum (ACP-Pt), ion adsorption (AgNO\(_3\) solution), chemical reduction plating copper (CRP-Cu) and ion exchange. In the process of studying its morphology, we found that dendritically interfacial electrodes will be generated due to the existence of composite electrode, which is mainly composed of platinum and copper. With the increase of the cycle of ACP-Pt, the DIEs will extend deeper and wider in the film, which is the characteristic of our IPMC. Therefore, we tested the water uptake, resistivity and output performance of IPMC, and analyzed the influence of DIEs on the performance of IPMC. It shows that the presence of DIEs can improve the overall electrical actuation performance of IPMC.

\textbf{CONTACT} Xinrong Zhang \texttt{zxrong@chd.edu.cn} Xi’an University of Science and Technology, Xi’an 710054, China

*These authors contributed equally to the work.

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1. Introduction

Electroactive Polymer (EAP) is one of the electro-induced materials transforming electrical energy into mechanical energy [1,2]. Among the EAP, ionic polymer–metal composite are attractive candidates for low driving voltage, large displacement and deformation, fast response speed and good biological compatibility [3–6]. Traditional IPMC with a typical sandwich structure is composed of polymer film with ion exchange ability and noble metal electrode layer on both sides [7–9]. When applying electrical signals on both ends of IPMC, an electric field is formed within the basement membrane. The migration of hydrated cation in the basement membrane resulted in the increase of water molecule expansion at the cathode side, which finally lead to the curved shape at the macro-level of IPMC [10–12]. On the contrary, when IPMC is deformed by external force, the movement of ions will be generated in the IPMC to generate current, which can be used as a sensor. Due to its special performance, IPMC can be applied to underwater robots, biomimetic sensors, linear drivers, medical devices and other fields [13–15]. But at present, there are some problems in the research of IPMC, such as the short service life, cracks appearing in the metal electrode layer easily and so on.

Gaining control over the surface electrode and metal/polymer interface has attracted much attention in IPMC. The chemically stable and noncorrosive metal is usually preferred as a flexible electrode, such as platinum, palladium and gold, due to their high electrochemical stability and excellent electrical conductivity [16–19]. Because of their high price, there are lots of difficulties in the practical processing of IPMC materials. Therefore, many scholars are devoted to the study of IPMC with cost-effective electrode structure. For example, copper, nanometre nickel and silver powder have been used as cost-effective electrode materials, but the low electrochemical stability of the materials limits their life cycle [20–22]. Therefore, many efforts have been committed to choose the appropriate metal material as the surface electrode layer of IPMC to generate high surface area, so as to obtain low cost, high bonding strength, large contact area and high performance [23,24]. For example, some researchers reported on their efforts to overcome the problem with the metal cracking by using Cu and Pt as the composite electrode materials and Cu$^{2+}$ as counter ion to the Naftion* sulphonate groups [25]. Some researchers described the fabrication of ionic polymer–metal composites (IPMCs) containing Pd/Pt electrode as an electrode material, and this new IPMC showed increased displacement and blocking force compared to that of conventional IPMC containing Pt electrode and ionic liquid [26]. Wang Yanjie found a dendritic interfacial electrode (DIE) structure by preparing IPMC with palladium electrode, which can increase the electrode thickness of IPMC and improve the driving performance of IPMC [27]. In this paper, Pt with good chemical stability and Cu with low resistivity were selected as the electrode materials for IPMC. The combination of the two electrode materials reduced the surface resistance of the sample, improved the braking performance and reduced the generation of surface cracks.

Herein, a new method of chemical plating was adopted to form a DIE [27] microstructure of a copper–platinum composite electrode ionic polymer–metal composite (Pt-Cu-IPMC) with a high specific area, which aims to explore the relationship among the specific surface area of the electrode and the electrode interface microstructure and the driving deformation mechanism of IPMC samples under different electrode growth.
mechanisms. The DIE increases the specific surface area of the electrode layer and reduces the distance between the electrode layers on both sides of the Nafion membrane, thus enhancing the driving performance of the IPMC and increasing the working time. The new method was to deposit platinum on both sides of Nafion membrane and then continue to deposit copper on the Pt coating. The optimal IPMC samples with a high specific area were prepared according to the controlled times of autocatalytic plating platinum (ACP-Pt). Moreover, the understanding of relationship between the micro-electrode morphology (surface electrode thickness and penetration depth of dendritic electrode) and the number of times of plating was discussed and highlighted the importance of the number of times of plating on the driving performance of IPMC samples. The surface resistance of Pt-Cu-IPMC samples was observed and tested.

2. Experiment

2.1 Materials

Hydrochloric acid (HCl), ammonia water (NH\textsubscript{3}•H\textsubscript{2}O), platinum ammonia complex ([Pt(NH\textsubscript{3})\textsubscript{4}]Cl\textsubscript{2}), sodium borohydride (NaBH\textsubscript{4}), hydrazine hydrate (N\textsubscript{2}H\textsubscript{4}•H\textsubscript{2}O), hydroxylamine hydrochloride (NH\textsubscript{3}OHCl), lithium chloride monohydrate ([LiCl]•H\textsubscript{2}O), ethylenediamine tetracetic acid disodium salt (C\textsubscript{10}H\textsubscript{14}N\textsubscript{2}O\textsubscript{6}Na\textsubscript{2}•2H\textsubscript{2}O), potassium tartrate (C\textsubscript{4}H\textsubscript{4}O\textsubscript{6}K\textsubscript{2}•2\textsuperscript{-1}H\textsubscript{2}O), sodium hydroxide (NaOH), trisodium citrate dehydrate (C\textsubscript{6}H\textsubscript{7}Na\textsubscript{3}O\textsubscript{5}•2H\textsubscript{2}O), silver nitrate (AgNO\textsubscript{3}), copper sulfate pentahydrate (CuSO\textsubscript{4}•5H\textsubscript{2}O), formaldehyde (CH\textsubscript{2}O), potassium ferricyanide trihydrate (K\textsubscript{4}[Fe(CN)\textsubscript{6}]•3H\textsubscript{2}O) and 2,2-dipyridine (C\textsubscript{10}H\textsubscript{8}N\textsubscript{2}) were obtained from Tianjin Chemical Reagent Corp (Tianjin, China). Nafion 117 films with the thickness of 0.183 mm were obtained from DuPon (Shanghai, China).

2.2 Fabrication process

In order to investigate the effect of the number of times in cycles of ACP-Pt on the electrode characteristics and the electromechanical properties of IPMC, five samples were prepared and named 0-Pt-Cu, 1-Pt-Cu, 2-Pt-Cu, 3-Pt-Cu and 4-Pt-Cu in this paper according to the process wherein the cycle of IRP-Pt is 1, the cycle of CRP-Cu is 1 and the cycles of ACP-Pt are 0, 1, 2, 3 and 4, respectively. In this experiment, Nafion-117 was used as the base membrane. The fabrication process mainly includes seven processes: pretreatment (including roughing, cleaning and boiling), ion adsorption (Pt([NH\textsubscript{3})\textsubscript{4}]Cl\textsubscript{2} solution), impregnation-reduction plating platinum (IRP-Pt), autocatalytic plating platinum (ACP-Pt), ion adsorption (AgNO\textsubscript{3} solution), chemical reduction plating copper (CRP-Cu) and ion exchange (LiCl). The process flow diagram is shown in Figure 1.

The first step is to roughen the surface of Nafion membranes with 800 mesh and 1000 mesh sandpaper, where it will serve as an effective electrode. Then the membranes were rinsed in ultrasonic cleaning machine for 30 min, and then boiled in 2 mol/L HCl solution for 40 min before boiling in deionized water for 30 min to remove impurities. In the second step, [Pt(NH\textsubscript{3})\textsubscript{4}]Cl\textsubscript{2} solution was used to allow Pt([NH\textsubscript{3})\textsubscript{4}]\textsuperscript{2+} to incorporate the ion adsorption. As for the third step of IRP-Pt, the ionomer membranes with pre-
exchanged Pt\([\text{NH}_3]_4^{2+}\) were subsequently immersed in the reducing solution of NaBH\(_4\), and the reduction reaction will then happen near the inner surfaces of the membrane where the metal atoms will generate granular growth and agglomeration along the thickness direction. The fourth step is ACP-Pt. The membranes were placed in the magnetic heating agitator under the reducing solution of NH\(_3\)•H\(_2\)O, N\(_2\)H\(_4\)•H\(_2\)O and NH\(_3\)OHCl. The copper plating is performed after this step. The fifth step is to immerse the membranes in AgNO\(_3\) solution to allow Ag\(^+\) to diffuse. The sixth step is CRP-Cu. This process is essential to further increase the thickness of the surface electrode and decrease the surface resistivity. After being rinsed in deionized (DI) water, the membranes were immersed in plating solution (40\(^\circ\)C) for 35 min. The final step is ion exchange in which the IPMC samples were placed in the 2 mol/L LiCl solution in turn. After full exchange for 12 h, the IPMC was removed and put into deionized water for storage.

2.3 Testing

SEM and EDS (JSM-6390A) were used to observe the micromorphology of the IPMC (the cross-sections of all samples were obtained by low-temperature cracking in nitrogen) and to analyze the distribution of platinum and copper element in the IPMC. The surface resistivity of samples was tested by double electric four probes tester (RTS-9).

As shown in Figure 2, the actuation process of IPMC was videotaped by video fixed-point method using DC power generator and coordinate paper. The tip displacement relative to the starting point at each time point was observed. The blocking force was calculated according to the formula in Figure 2, where m (A in Figure 2) was measured by an electronic balance (GL124-1SCN). At air temperature, one end of the IPMC sample with a size of 30 mm × 10 mm was clamped, and the free beam length is 25 mm.
3. Results and discussion

3.1 Micromorphology analysis

Figure 3 shows the surface images of five samples. As for Figure 3(a) - (e), most of the particles were evenly distributed on the surface of the membrane, and the concave and convex feeling of Cu coating was observed while Pt particles were barely visible. As shown in Figure 3(a), the smaller copper particles were deposited in grooves that were formed after surface roughening. Figure 3(b) shows that the distribution of copper was...
uniform. The relatively dense surface was formed as the reduction proceeded, filling up empty space due to continuous heterogeneous nucleation and grain growth [21,28]. With the increase of ACP-Pt cycles, the surface electrode where agglomeration occurred became dense and uneven to a large extent, as shown in Figure 3(c). There were many agglomerated Cu particles and gaps between Cu particles in Figure 3(d) and (e). The discontinuity of the local electrode had a certain effect on the service life and performance of the sample, which is related to the Nafion surface that acted as heterogeneous nucleation sites for the deposition of copper in aqueous copper ion solutions during the initial stage of the reduction. With an increased reduction times, the surface of Nafion was observed to be further covered with continuously heterogeneous nucleation and growth of copper. These phenomena indicate that there are some reactions between copper and platinum in the polarization process, including the possible side reactions of copper itself under high pressure and the possible catalytic reactions of platinum. It can reflect the existence of copper electrode layer, which can fill the cracks of Pt electrode layer in the process of polarization [29].

Figure 4 shows the cross-section images of samples. With the increase of ACP-Pt cycles, DIEs became denser and larger, and the interfacial area also became larger and larger. As shown in Figure 4(a) and (b), the electrode was thin and uneven, and DIEs began to appear where the metal electrodes are keeping well touch with the membrane. With the increase of ACP-Pt cycles, the thickness of electrode layer was increased (which were showed in Figure 6(a)). As the compactness and smoothness were enhanced, which can be seen from Figure 4(c), the interfacial area got bigger along with deeply growing dendrites. When it was plated with platinum, with the process of electroless plating, Pt ions in the solution are reduced and accumulated in the surface electrode. During each ACP-Pt, Pt ions continue to deposit on the Pt metal layer that was reduced last time.

Figure 4. Cross-section images of samples dependent on different cycles of ACP-Pt: ((a) 0-Pt-Cu, (b) 1-Pt-Cu, (c) 2-Pt-Cu, (d) 3-Pt-Cu and (e) 4-Pt-Cu).
Copper ions were then deposited on the reduced platinum layer during copper plating. Each reduction led to an increase in metal particle density on the electrode surface. From Figure 4(b) to (c), it shows that the DIEs become obviously dense; from Figure 4(c) to (d), the DIEs are deeper into the Nafion membrane. In the Figure 4(e), the maximum and deepest DIEs are clearly distinguished with maximum interfacial area.

In order to deeply study the DIEs structure, we have performed EDS, and the corresponding EDS profiles are shown in Figure 5. From the cross-section images of samples, we observed that the dendritic distribution displays a perfect DIE structure together with interfacial electrode of Pt and Cu. In Figure 5(a) to (e), the content of Pt and Cu starts from the red point on the surface of IPMC. With the increased distance, the content of Cu was the highest on the electrode surface, decreased suddenly at the interface and then tended to be stable in the inner Nafion film. The content of Pt was less on the surface of the electrode,
increased at the interface and then decreased in the inner Nafion film. Furthermore, the platinum and copper contents gradually declined from the surface to the inside of the membrane, and the DIEs emerged inside all samples, extending from the surface of the Nafion basement membrane to the middle. The branch-like electrodes deepened with the increase of ACP-Pt cycles. The platinum and copper were widely and deeply distributed over the Nafion basement membrane, and this wide distribution provided a large interfacial area, which was beneficial for the driving performance of IPMC.

Figure 6(a) shows the thickness of samples’ section electrode of the different cycle times of ACP-Pt. It shows a gradually increasing trend with the increased cycles of ACP-Pt. The thicknesses of the five samples are 10.94, 17.5, 18.44, 20.63 and 31.98 µm, respectively. It can be seen that when the cycle number of ACP-Pt is greater than 3, the electrode thickness of IPMC will suddenly increase. The reason for this phenomenon may be that when the number of ACP-Pt is greater than 3, the content of Pt will increase, the possibility of metal particle agglomeration will be greater and the possibility of DIEs emergence will become greater and the volume will be larger, so the electrode thickness will be thicker.

3.2 Surface resistivity

Figure 6(b) shows the surface resistivity of samples. It shows that the surface resistivity first decreased and then increased slightly with the increased cycles of ACP-Pt, and 3-Pt-Cu showed the minimum value. Combined with the microscopic morphology of the sample, it indicates that although the surface electrode of the sample is thickened with the increased cycles of ACP-Pt, the density and flatness of the electrode show a trend of decline, which means the surface resistivity does not show a trend of constant decrease. In addition, the surface resistivity is closely related to the driving performance of IPMC, and the sample with lower surface resistivity is usually stronger in bending deformation. The results of strain energy density analysis (as shown in Figure 8) indicate that the sample 3-Pt-Cu has a higher strain energy density and better driving performance, which is consistent with the changing trend of surface resistivity.
Figure 7. Displacement and blocking force characteristics of samples at different DC voltages.

Figure 8. Strain energy density of samples at different DC voltages.

### 3.3 Actuation characteristics

Figure 7(a) shows the displacement characteristics of samples at different DC voltages. The maximum displacements of 0-Pt-Cu, 1-Pt-Cu, 2-Pt-Cu, 3-Pt-Cu and 4-Pt-Cu were reached at 6, 6, 5.5, 4.5 and 5 V, respectively. Among them, the 3-Pt-Cu sample shows the maximum displacement of 67.27 mm at 4.5 V while the minimum displacement of 19.7 mm at 5 V for the 4-Pt-Cu sample. The minimum displacement of sample is about 1.5
times larger than that prepared by electroplating Cu [25]. The amplitude of displacement changes gently with the different voltages of all samples, while the displacement change greatly for the samples of 1-Pt-Cu, 2-Pt-Cu and 3-Pt-Cu. The tendency of displacement for all the samples was increased first and then decreased with the increase of voltage. With the increase of voltage, the number of hydrated cation moving to cathode increases, which means that the displacement will also increase. However, when the voltage continues to increase, the water electrolysis reaction will be intensified and the migration of hydrate cations will reach saturation state, so the displacement will not increase any more. In addition, when the voltage increases, copper itself may also have some side reactions, affecting its own activity, which affects the driving performance of IPMC [30,31].

Figure 7(b) shows the blocking force characteristics of samples at different DC voltages. The maximum blocking force of five samples was reached at 4.5, 4, 5.5, 4.5 and 6 V for 0-Pt-Cu, 1-Pt-Cu, 2-Pt-Cu, 3-Pt-Cu and 4-Pt-Cu, respectively. The maximum blocking force of sample is about 3 times larger than that prepared by electroplating Cu [25]. The sample 3-Pt-Cu has the maximum blocking force of 27.21 mN at 4.5 V while the minimum blocking force of 13.7 mN at 4.5 V for the sample 1-Pt-Cu. With the increase of voltage, the blocking force of IPMC was first increased and then decreased. The high blocking force of samples is related to high interfacial area of dispersed Pt and Cu particles near the ionic membrane surface, leading to high transport and adsorption at the electrodes.

3.4 Strain energy density

According to Figure 7, for all samples, when the blocking force reaches the maximum, the corresponding displacement would not reach the maximum. Therefore, in order to describe the electro actuation effect of IPMC more accurately, the concept of ‘strain energy density’ is specially introduced in this paper. According to the bending strain and the Euler Bernoulli beam theory, it can be described as follows [32]:

$$\rho_e = \frac{Flb}{ah(d^2 + b^2)}$$  (1)

Among them, $\rho_e$ is the strain energy density of IPMC, $F$ is the blocking force, $b$ is the displacement of the measurement point of the IPMC sample, $l$ is the length of IPMC sample, $a$ is the width and $h$ is the thickness.

The strain energy density was calculated according to formula 1, and Figure 8 shows that the strain energy density of the five samples increased first and then decreased. Among them, the strain energy density value of 3-Pt-Cu is the largest, which is 5.87 kJ/m³. Combining with the microscopic morphology and driving performance, it shows that the presence of DIEs increases the strain energy density of IPMC, thereby improving the overall electrical actuation performance of IPMC.

4. Conclusion

In conclusion, we have successfully prepared the copper–platinum composite electrode IPMC with a high specific area by electroless plating. The SEM images show that the surface electrode where agglomeration occurs become dense and uneven to a large
extent, and platinum and copper are widely and deeply distributed over the Nafion basement membrane with the increase of ACP-Pt cycles. In addition, the DIEs emerge inside all IPMC samples. Combining with the microscopic morphology and driving performance, it shows that the presence of DIEs can improve the overall electrical actuation performance of IPMC. Among the five Pt-Cu-IPMC, the 3-Pt-Cu-IPMC has obtained the maximum displacement of 67.27 mm and blocking force of 27.21 mN with strain energy density of 5.87 kJ/m$^3$ at 5.5 V, which indicate that the cycles of ACP-Pt also impact on the related properties of IPMC. Our study provides some experimental data for the application of IPMC and provides an optimized process for better performance. However, copper electrode is very easy to corrode, which greatly reduces the recycling service life of the sample. Therefore, the research on packaging technology is the focus of our research group in the following work.

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**Disclosure statement**

No potential conflict of interest was reported by the author(s).

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