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Encapsulation of ionic polymer-metal composite (IPMC) sensors with thick parylene: Fabrication process and characterization results



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ABSTRACT

lonic polymer-metal composites (IPMCs) tend to have inconsistent sensing properties when operating in air under different humidity levels, or after exposure to different ambient media. Motivated by the need to ensure consistent sensing performance of IPMCs under different ambient environments, in this paper we propose thick (up to 25 micrometers) parylene C coating for IPMC sensors, develop effective coating processes, and evaluate the performance of the encapsulated sensors, along with the comparison with the typical naked IPMC sensors. The proposed fabrication process features the water drive-in step which enables the control and adjustment of the hydration level of an encapsulated IPMC. Experiments are conducted to evaluate the sensing consistency of IPMC sensors in a humidity chamber, first under different humidity levels, and then following exposure to solutions with different cations and different organic solvents. Experimental results show that the proposed thick parylene coating can effectively keep the water content inside the IPMC, isolate the IPMC sensors to be used in practical applications. © 2014 Elsevier B.V. All rights reserved.

1. Introduction

Ionic polymer-metal composites (IPMCs), one important class of electroactive polymers (EAPs), have built-in sensing and actuation capabilities [1,2]. In particular, they hold strong promise for versatile sensing applications because of their high sensitivity, inherent polarity, and direct mechanosensory property, which minimize the complexity in both the sensor construction and signal processing. Furthermore, they are bio-compatible and amenable to fabrication [3–6].

An IPMC sample typically consists of a thin ion-exchange membrane (e.g., Nafion), chemically plated with a noble metal (typically platinum) as electrodes on both surfaces [7]. Inside the polymer, anions covalently fixed to polymer chains are balanced by mobile cations. An applied force or deformation on an IPMC beam breaks this charge balance, which results in the redistribution of cations and accompanying solvent molecules, and leads to the generation of a detectable electrical signal (typically open-circuit voltage or short-circuit current) across the electrodes. Studies on modeling IPMC sensors can be found in [8–15]. Recent applications of IPMC sensing capabilities span measurement of force [16], flow [17,18],

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http://dx.doi.org/10.1016/j.sna.2014.05.013 0924-4247/© 2014 Elsevier B.V. All rights reserved. shear loading [19], curvature [20], structural health monitoring [12], and energy harvesting [21–23].

A critical issue in the practical use of IPMC sensors is that IPMCs need ionic hydration to operate. As the most commonly used solvent, water content contained in the polymer varies with the humidity level of the ambient environment, which affects the sensing behavior of an IPMC in air [24,25] and thus results in the difficulty of maintaining consistent sensing properties. If the sensor operates in a liquid medium, the working environment (such as sea water, acid solution, gasoline, etc.) could also affect the consistency of IPMC sensing properties, or even cause the failure of the IPMC sensor. For example, the contact resistance between the platinum electrodes and the attached or soldered conductive wire would increase dramatically due to the galvanic corrosion effect in the water. IPMCs with different cations inside (e.g., Na, Li, H) will display different sensing characteristics [7,26,27]. If the sensor is exposed to the ambient medium, the cations inside IPMC will exchange with those in the medium gradually, which results in uncertainties in the sensing behavior. Direct exposure of the IPMC sensor to organic solvents could also destroy the sensing capability of the sensor. Therefore, in order to use IPMC sensors in practical applications, it is necessary to adopt some measures to ensure their consistent responses over time and under different environments. One potential solution to this problem is to coat the IPMC with some waterproof material to suppress water permeation, and to isolate the IPMC from the ambient environment.

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2 Table 1

Reported water vapor transmission rate (WVTR) for parylene C. The unit is (g mil)/(100 inch² day), where mil and 100 inch² refer to the thickness and the size of the membrane, respectively.

WVTR of parylene C (g mil)/(100 inch ² day)	WVTR at 37 °C, 90% RH	WVTR at 20 $^{\circ}\text{C}$, 90% RH	WVTR at 20 $^\circ\text{C}$, 30% RH
Hubbel et al. [36]	3.3–7.3	N/A	N/A
Loeb et al. (Union Carbide) [37]	0.5	N/A	N/A
Specialty Coating System, Inc.	0.2031	N/A	N/A
P.R. Menon et al. [35]	0.207	0.08675	0.04515

Several encapsulation processes have been reported for IPMC actuators. For example, Shahinpoor et al. [28] and Akle and Leo [29] proposed the encapsulation of IPMCs using Saran plastic membrane. Franklin [30] reported using KaptonTM film to cover a multilayer IPMC actuator. Malone and Lipson [31] proposed the use of a PDMS membrane material for IPMC encapsulation. However, encapsulation with these high-stiffness materials increases the IPMC stiffness and makes it difficult to deflect, considerably affecting the IPMC performance as actuator and sensor.

Recently, IPMC encapsulation with much less stiff materials has been reported. Barramba et al. [32] proposed the use of dielectric gel materials as an IPMC encapsulant, which showed a very low stiffness and a high dielectric constant. Kim et al. [33] explored coating isotactic polypropylene, silicone rubber and parylene on the IPMC and found that parylene was the most effective coating material to suppress water leakage from IPMC. They further evaluated the displacements and the maximum forces for the parylene (1 µm) coated IPMC under various actuation voltages, and tested the adhesion between parylene and Pt electrode. Their approaches were primarily focused on extending the lifetime of IPMC actuators in air. Experiments have not yet been conducted to verify how these encapsulants maintain the IPMC sensing performance in air, or in other media than air. In addition, Bennett and Leo proposed the use of ionic liquids as solvents for IPMCs [34]. These liquids have very low vapor pressures, which enable IPMCs to operate in air for a long time. However, IPMCs with ionic fluid solvent typically cannot be used in liquid media.

This paper contains four major contributions to the state of the art in IPMC encapsulation. First, we explored the impact of the parylene thickness and found that the use of thick parylene C (up to 25 µm) would significantly improve the impermeability of the encapsulant. Second, we developed a novel scheme for tuning the water content of an encapsulated IPMC, with which we studied the impact of hydration level on the sensing performance. Third, we examined the corrosion effect between the platinum electrodes and the conductive wires of an IPMC sensor and verified that the parylene encapsulation could effectively prevent the contact points from corrosion. Last, we rigorously conducted extensive performance comparison between a naked IPMC and an encapsulated IPMC, both in air with different humidity levels, and following exposure to different liquid media, and found the proposed approach to be effective in maintaining consistent sensing behavior under various ambient environments.

The remainder of the paper is organized as follows. We first present the proposed fabrication process for the IPMC encapsulation in Section 2. We further evaluate the parylene-encapsulated IPMCs on four aspects: the physical properties are characterized in Section 3; the control of IPMC hydration level and its impact on sensing performance are discussed in Section 4; the anti-corrosion

effect is presented in Section 5; and the sensing consistency in different ambient environments is evaluated in Section 6. Finally, concluding remarks are provided in Section 7.

2. Fabrication process

2.1. Coating material

Parylene C was adopted as the encapsulation material for the following three reasons. First, parylene C is well known for its effectiveness as water barrier because it has very low water vapor transmission rate (WVTR), as shown in Table 1. The reported WVTRs were measured under certain conditions of temperatures, relative humidities (RH), and thicknesses of thin films. Note that the WVTR is not identical for different thicknesses of parylene films. For example, the actual WVTR for 25 µm thick parylene should be much smaller than that measured with $8 \,\mu$ m. The WVTR at $20 \,^{\circ}$ C, 30%RH and WVTR at 20 °C, 90% RH from [35] are used in this paper to estimate the water permeation of parylene encapsulation in air and in water respectively, as shown in Table 2. Second, the Young's modulus of parylene C is only 0.4 MPa, which would minimize the influence of the encapsulation layer on the IPMC sensor stiffness and thus the sensitivity, considering that the Young's modulus of a typical IPMC sample is around 300 MPa [9]. Finally, parylene deposition is conducted in a chemical vapor deposition (CVD) system at room temperature, which enables conformal, uniform and true pinhole-free coating on surfaces with various geometries. Compared with other encapsulation approaches reported in the literature, the CVD system offers the advantage of accurately controlling the deposition thickness and rate. The deposition process is also compatible with standard microfabrication technologies, which makes it possible to integrate high-density IPMC sensor arrays with electronics on a single platform.

2.2. IPMC sensor fabrication

IPMC sensors were fabricated with the traditional impregnation-reduction ion-exchange process [7]. Nafion-1110 (254 μ m) films from Dupont were first roughened with fine sand-papers. The residues on the film were removed with ultrasonic cleaner. The films were boiled in dilute 2 wt% hydrochloric acid for 30 min to remove ions and impurities. Then the films were boiled in deionized (DI) water for another 30 min to remove the acid and swell the films, and immersed in a platinum complex solution ([Pt(NH₃)₄]Cl₂) for more than 12 h to allow Pt ions to completely diffuse into the Nafion films through the ion-exchange process. After that, the films were rinsed with DI water and immersed in a water bath at 40 °C. We then added 2 ml of sodium borohydride solution (5 wt% NaBH₄ aq) as the reducing agent every

Table 2

Estimated water permeation for encapsulated IPMC sensor, where the parylene layer is assumed to be $10 \,\mu$ m (0.394 mil). The size of the IPMC is 0.626 inch \times 0.121 inch and the weight is 32 mg. Note that the encapsulation area is computed as $2 \times \text{length} \times \text{width}$.

Encapsulated IPMC sensor	WVTR (g mil)/(100 inch ² day)	Water permeation rate (mg/day)	Water permeation (wt%)
In air	0.04515	0.1736	0.5425
In water	0.08675	0.3338	1.0431



Fig. 1. Fabrication procedure for IPMC encapsulation.

30 min when the temperature went up to $60 \,^{\circ}\text{C}$ gradually. Once the platinum deposition was complete, the films were cut into beam-shaped samples with proper sizes. Finally, an IPMC sensor was formed by soldering two electric wire connectors to the IPMC platinum electrodes.

2.3. Parylene encapsulation

Parylene encapsulation was conducted with a parylene coater (PDS2035, Specialty Coating System, Inc.), where parylene C was deposited conformally on the IPMC sensors under a low pressure of 30 mTorr. The adhesion between the parylene and the platinum electrodes can be improved by plasma treatment. Please refer to [33] for more details. According to the experimental results reported in [33], IPMC samples with 1 μ m thickness of parylene coating still had complete water swelling when soaked in water for 24 h, showing that 1 μ m thickness of parylene encapsulation failed to isolate the IPMC from outside media effectively. It was reported in [38,39] that 8 μ m might be a threshold value for the thickness of the parylene C film in terms of whether the film would be affected by defects or not. Thus, thick parylene coatings larger than 10 μ m were selected for the proposed encapsulation process.

IPMC sensors need ionic hydration to operate; however, it is challenging to deposit parylene on IPMC while maintaining the moisture inside, because the water molecules will evaporate completely in the deposition chamber under the low pressure. To address this challenge and furthermore, to control the hydration level inside the encapsulated IPMC, we propose a novel fabrication recipe that consists of parylene deposition and water drive-in processes, as shown in Fig. 1. Based on the reported data in Table 1, the WVTR of parylene C is greatly affected by the temperature. Under the room temperature, parylene has excellent water barrier capability; but when the temperature goes up to 37 °C, the WVTR almost doubles. The water drive-in process was realized by taking advantage of this temperature effect: after the parylene deposition, the encapsulated IPMC sensors were soaked in a hot water bath of 80 °C for sufficient time. During this process, the water molecules diffused through the parylene layer with a high WVTR and hydrated the IPMC sensor. The hydration level was controlled by the water bath temperature and the length of soaking time, and thus we can adjust the water content inside the IPMC sensor so that the best sensitivity can be achieved. After the water drive-in process is finished, the diffused water molecules will be sealed inside by the encapsulation layer under the room temperature. Fig. 2 shows the experimental result for the water drive-in process for three parylene-encapsulated IPMCs (with 25 μ m parylene coating) submerged in hot water baths under 60, 70 and 80 °C, respectively. The value of the water content is obtained by weighing the IPMC sample before and after the soaking. It can be seen that the proposed drivein process gives a nearly linear relationship between the soaking time and the increased weight when the hydration level is relatively low, indicating the feasibility of using this method to hydrate and re-hydrate a coated IPMC sensor. From Fig. 2, one can also see that the rate of water drive-in shows a pronounced dependence on the bath temperature, as one would expect.

Evaluation of the parylene-encapsulated IPMCs has been conducted on four aspects: physical properties, control of hydration level and its impact on sensing performance, anti-corrosion effect, and sensing consistency in different media. All the experiments were done with the comparison between the coated and the naked samples, which had the same dimensions (25 mm by 3 mm by 270 μ m and were cut from the same big piece of IPMC (50 mm by 50 mm by 270 μ m), so that the differences of original properties for each sample were minimized.



Fig. 2. Water drive-in process for parylene-encapsulated IPMCs with 25 μm parylene coating (water bath of 60, 70 and 80 $^\circ$ C).



Fig. 3. The SEM images of surface morphology for samples with parylene coating thickness of (a) 1 µm, (b) 10 µm, (c) 25 µm.

3. Characterization of physical properties

3.1. Surface morphology

Scanning electron microscopy (SEM) images of surface morphology were taken to directly investigate the deposition results for different thicknesses of parylene (Fig. 3). Parylene was deposited on the surface of the IPMC samples and these images in Fig. 3 show the roughness of the coated parylene surface. Since parylene is transparent, a thin layer (around 2 nm) of gold has been sputtered onto the surface of the parylene layer to better capture the SEM images. Note that the globules observed in Fig. 3(b) are most likely the gold particles. From these SEM pictures, one can see that the surface of the sample with thick parylene coating was much smoother than that with thin coating. In particular, pinholes through the encapsulation layer can be observed with 1 µm coating (see Fig. 3(a)), indicating that there is significant improvement of the surface condition with thick parylene deposition, which is consistent with the relevant results reported in [39]. When the thickness of the parylene coating increases from 10 µm to 25 µm, there is no significant improvement of the surface condition, as shown in Fig. 3(b) and (c). Ideally, the encapsulation layer always has better water barrier capability with thicker deposition, but considering the increasing stiffness of the whole sensor and the cost of thick parylene deposition, a good balance of the encapsulation thickness would be $10-25\,\mu$ m, which increases the mass of the sample by approximately 1.9-4.8 mg based on the density of the parylene C (1.289 g/cm^3) .

3.2. Stiffness of the composite beam

Compared with a naked IPMC sensor, the most noticeable change of mechanical characteristics for an encapsulated IPMC sensor is the stiffness. Fig. 4 shows the schematic and photo of the experimental setup for measuring the stiffness of a beam. The base of the beam was fixed on a frame which could be moved up and down, and the beam tip rested on a load cell used to measure the force. A laser displacement sensor was mounted above the beam measuring the tip displacement. Given the measured force, the corresponding tip displacement and the beam dimensions, we can calculate the stiffness of the composite beam.

IPMC samples with different thicknesses of encapsulation were tested to evaluate the influence of the parylene encapsulation on the stiffness of the sensor beam. The uncoated sample has a nominal dimension of 25 mm by 3 mm by 270 µm, while the coated samples have the nominal dimension plus the thickness of encapsulation. The coated samples were tested without hydration while the uncoated sample was tested in the ambient relative humidity of 40%. For each sample, the tip bending and the resulting force at 60 positions were collected, and the stiffness was calculated by linear fitting so that the measurement error was minimized, as illustrated in Fig. 5. Fig. 6 shows the measured stiffness for the IPMC sensors with various thicknesses of parylene coating. It can be seen from Fig. 6 that the stiffness of the encapsulated IPMC sensor increases with the thickness of the parylene layer, which is expected considering the constraint of the encapsulant on the beam bending. Unless noted otherwise, we adopted the IPMC sensor with 25 µm encapsulation for the rest of the testing.

3.3. Impermeability characterization

To evaluate the water permeation of the parylene coating layer, a test was first conducted on the evaporation loss of water from the inside of the IPMC sensors under heating. A naked IPMC sample and coated samples with different parylene thicknesses were heated on a hot plate at $60 \,^{\circ}$ C for $60 \,^{\circ}$ L min. They were weighed every 3 min during the test to measure the water evaporation loss. The test results are shown in Fig. 7. The naked sample lost almost all the water after



Fig. 4. The schematic (a) and photo (b) of the experimental setup for measuring the beam stiffness.



Fig. 5. Experimental data for the stiffness measurement of a sensor with parylene thickness of 25 μm

10 min baking, while the encapsulated ones did not show such a quick water loss. For coated samples with different parylene thicknesses, it can be seen that the water evaporation rate decreases noticeably as the encapsulation layer gets thicker. In particular, the



Fig. 6. Measured stiffness for IPMC sensors with different thicknesses of coating.



Fig. 7. Experimental results of water impermeability test under heating.

sample with 25 μ m coating held its hydration level without significant changes during the one hour baking. Note that the water evaporation through the electrode surface and the parylene layer was greatly accelerated by the heating in this test. When operating under room temperature for most applications, the encapsulated IPMC sensors would have much slower evaporation loss.

Another test was conducted by soaking the parylene-coated IPMC samples in deionized water for up to 10 days to further evaluate the water barrier capability of the proposed encapsulation. The water absorption of IPMC sensors was evaluated by measuring the weight of the samples every 12 h. The sensors were cleaned gently with dry cloth and dried in air for 5 min to eliminate the residual water on the surface. After the weighing process, they were put back into DI water. The experimental results are shown in Fig. 8. For the sample encapsulated with 10 µm thick parylene, the WVTR reported by Menon et al. [35] can be used to estimate the water permeation rate, since they measured WVTR on a film of 8 μ m thickness. During the 10-day test, the 10 μ m sample had water absorption of 7 mg and an average water permeation rate of 0.7 mg/day. Compared with the estimated water permeation rate of 0.3338 mg in Table 2, the measured higher water absorption is reasonable since the estimation was based on 90% RH. Also parylene deposition was conducted under different conditions in [35] in terms of the deposition pressure and rate, which would affect the properties of the parylene layer. For the samples with



Fig. 8. Experimental results of impermeability test under soaking in water.

different parylene thicknesses, the test results in Fig. 8 show that the water permeation rate drops consistently with an increasing coating thickness, and this rate under $25 \,\mu m$ coating is significantly lower than that under $15 \,\mu m$ coating.

4. Control of IPMC hydration level and its impact on sensing performance

It has been discussed in [33] that an IPMC actuator requires particular content of water to operate effectively. For IPMC sensors, it is observed in [24] that while the signal amplitude of the sensor increased with the ambient humidity level, the sensor noise increased as well. Therefore, it is of interest to investigate the impact of hydration level on IPMC sensing performance. In the following experiments, both an uncoated IPMC sensor and an encapsulated one were tested in ambient air under 20 °C and 60% RH. The sensors were tested with different levels of hydration. The uncoated sample was first soaked in deionized water to get saturated hydration and then exposed in air for different amounts of time to obtain different water contents. Testing time was much shorter than the exposure time, so the sample's hydration level stayed nearly constant under each test. For example, in one test the uncoated sample was exposed in air for 5 min after saturation; then it was clamped on the mini-shaker for base-excitation. The sensor output became stable after several seconds and was then collected for 5 s. Immediately after the testing, the sample was weighed to

measure the water content. For the uncoated sample, 0 wt % water content was obtained by heating it on hotplate at $60 \circ \text{C}$ for 20 min (see Fig. 7). For the encapsulated IPMC sample, different levels of hydration were achieved by controlling the soaking time in the water bath during the water drive-in process, as described in Section 2.3, and the hydration level of encapsulated sample before the water drive-in process was assumed to 0 wt% because the water molecules inside the IPMC would evaporate completely during the parylene deposition process.

The sensing outputs of both uncoated and encapsulated IPMC sensors under base-excitation were collected with the same experimental setup, as shown in Fig. 9. The IPMC samples were excited at their clamping ends by a mini-shaker (Type 4810, Brüel & Kjær) with a frequency of 10Hz and a base-excitation amplitude of 1 mm. The short-circuit sensing current was conditioned through an amplifying circuit and collected by a dSPACE data acquisition system (RTI 1104, dSPACE). The amplitudes of the IPMC sensor outputs at 10Hz were extracted as the signals through fast Fourier transform. The noise levels were obtained by calculating the root mean square (RMS) of the amplitudes at other frequencies. The signal-to-noise ratio (SNR) was computed for each case to evaluate the sensor performance at different hydration levels.

The experiment results are shown in Figs. 10 and 11. For the uncoated IPMC sample, under the condition of 0 wt% water content, it failed to generate any noticeable signal as expected. When the water content went up, both the signal and the noise showed an increasing trend initially, but the SNR of the sensor did not show the same trend. Beyond 3.5 wt% water content, both the signal and the noise started to drop as the water content kept increasing. When the water content was higher than 8 wt%, it was difficult to collect accurate sensor outputs experimentally since the water evaporated too fast. Note that this experiment was conducted under the relative humidity of 60%; it is conceivable that the sensor outputs will be different under other relative humidities, given the fact that the sensing properties of a naked IPMC are affected by the ambient humidity level [25]. However, we expect the general trend observed in Fig. 10 to hold for other humidity levels.

The performance of the parylene-encapsulated IPMC sensor at different hydration levels is shown in Fig. 11. Unlike the case for the naked IPMC sensor, the impact of water evaporation and the ambient humidity level could be ignored for the coated sample due to the parylene encapsulation, thus a larger range of water content had been tested. Compared with the performance of the naked IPMC sensor in Fig. 10, the coated sensor had similar patterns for both the signal and the noise amplitudes when the water content went up, while the SNR was slightly more consistent. In both



Fig. 9. The schematic (a) and photo (b) of the base-excitation experimental setup for characterizing IPMC sensing performance.



Fig. 10. Performance of the uncoated IPMC sensor at different hydration levels: (a) signal magnitude, (b) noise magnitude, (c) signal-to-noise ratio.



Fig. 11. Performance of the coated IPMC sensor at different hydration levels: (a) signal magnitude, (b) noise magnitude, (c) signal-to-noise ratio.

Figs. 10 and 11, peak points can be found for the amplitude of the signal. At the peak point, it is believed that the excitation frequency matched the sensor resonance frequency, which varies with the change of IPMC beam mass at different hydration levels. Therefore, adjustment of the resonance frequency can be done for the parylene-encapsulated IPMC sensors to achieve larger sensing outputs in different applications, while it is not feasible for the naked IPMC sensors. Note that the peak point in Fig. 11 (around 8 wt%) is different from that in Fig. 10 (around 3.5 wt%), and at each peak point, the signal amplitude of the coated sample is twice larger than that of the naked sample. This difference between the naked sample and the coated sample is attributed to their respective mechanical properties which are different due to the added mass and constraint of the parylene layer.

5. Evaluation of anti-corrosion effect

For a typical IPMC sensor, conductive wires need to be connected to the surface metal electrodes in some appropriate ways to form the contact points for practical applications. The latter can be done through soldering, clamping, taping, or pasting with conductive ink. In any case the contact between different metals leads to nonnegligible galvanic corrosion effect on the more active metal when the IPMC sensor is working in a solution, as shown in the Fig. 12 for the soldering case. The corrosion effect can result in an increment of the contact resistance between the surface electrode and the wire, which will reduce the sensing output of the IPMC sensor and result in inconsistent sensing behavior. If the corrosion process lasts too long, the sensor will eventually fail to function due to the large contact resistance.

In this study, the parylene-encapsulated IPMC sensor was evaluated in terms of its anti-corrosion performance, and compared with some other approaches used to form the contact points. Specifically, the following schemes were compared:

- Solder: copper wires were soldered with tin to the platinum electrodes; the galvanic corrosion would mainly occur between platinum and solder tin.
- Silver conductive epoxy (MG chemicals): copper wires were pasted to the electrodes by the silver epoxy; the corrosion would mainly occur between platinum and silver.
- Solder and epoxy sealing: copper wires were first soldered to the electrodes, and then the whole end of sensor where the soldering tin existed were covered with epoxy, leaving the rest of the sensor





Fig. 13. Experimental results of contact point corrosion in tap water.

naked; the corrosion would occur between platinum and solder tin.

• Parylene encapsulation: the copper wires were first soldered to the electrodes and the whole sensor was encapsulated with thick parylene. The corrosion, if any, would take place between platinum and soldering tin. This is the proposed method.

All of the four samples were soaked in tap water for four days, and their contact resistances between the wires and the electrodes were measured for comparison. The measurement was conducted in air immediately after the sensor was taken out of water and wiped dry. The experimental results are shown in Fig. 13. Note that the first point for each case was measured under dry condition and the second point was measured 5 min after soaking the samples in the tap water. The immediate increase of contact resistance at the second point is believed to be the result of the surface electrode expansion when the IPMC samples went into the tap water from the dry condition. For the parylene coated sample, there were only two points available: one was at the beginning of the experiment and the other in the end, because it was not feasible to directly measure the contact resistance during the experiment, given that the whole sample was encapsulated with parylene. One can see from the figure that the contact resistances of the three naked samples kept increasing as the soaking time went up, while the encapsulated sample did not show any noticeable change after four days of soaking in tap water. The sample with soldered wires and no epoxy sealing had the worst performance, due to the fact that its

contact points were completely exposed to the water. The silver conductive epoxy was much better than the bare soldering mainly because silver is more stable than tin. When the soldered sample was covered with epoxy around the soldering portion, the contact points were protected by the cured epoxy from the direct water penetration, which significantly lowered the rate of electrochemical reaction between the soldering tin and the platinum electrodes. However, the epoxy sealing could not completely block the water attack, since water will still reach the soldering portion through the IPMC itself, given that the polymer is highly permeable to water. From Fig. 13, with parylene encapsulation, the IPMC was isolated from the ambient medium almost completely, thus preventing the galvanic corrosion on the contact points.

6. Evaluation of sensing consistency in different ambient environments

As mentioned in previous sections, IPMC sensors could operate with different levels of water content and in different environments. But it is highly desirable for practical applications that IPMC sensors have consistent sensing properties; otherwise, when the ambient environment changes, the sensors either need to be calibrated every time or even fail to work. In this section we present the evaluation results on the consistency of encapsulated IPMC sensing behavior obtained from two sets of experiments. In the first set of experiments, the sensor was placed in a humidity chamber and its sensing output was obtained under different humidity levels. In the second set of experiments, the sensor was first exposed to a series of different media and its sensing output was then obtained in a base-excitation mode in the same humidity chamber. In all experiments the comparison between an encapsulated sensor and a naked sensor was conducted. For the encapsulated sensor, the hydration level was set to be 8 wt.

6.1. Sensing consistency test under different humidity levels

To evaluate the consistency of sensing behavior in air, one naked IPMC sample and one encapsulated sample were tested experimentally under different humidity levels. Fig. 14(a) and (b) shows the schematic and the picture of the experimental setup, respectively, including a custom-built humidity chamber made of acrylic panels. Inside the chamber, a humidity sensor (HIH-4030, Honeywell) was used to measure the relative humidity (RH) level, which was then acquired by the dSPACE data acquisition system (RTI 1104, dSPACE). A temperature sensor is also used for monitoring purpose



Fig. 14. The schematic (a) and photo (b) of the experimental setup for evaluating IPMC sensor performance in a custom-built humidity chamber.

[15]. A control signal would then be sent to the feedback control circuit inside the chamber, to turn on/off the humidifier (EMS-200, Stadler Form) or the dehumidifier (EDV1100, Eva-Dry) accordingly to achieve a given RH value. Limited by the capability of the dehumidifier and the sealing constraints, this humidity chamber could only achieve 40% or higher. Both the naked IPMC sample and the encapsulated one were clamped at the base on a rigid bar which penetrated the chamber wall and was connected to the output shaft of a mini-shaker (Type 4810, Brüel & Kjær) so that both samples received the same mechanical excitation. Outside the humidity chamber, the shaker generated vibration stimulus (up and down) with a frequency of 10 Hz and some fixed amplitude. A 3D-printed flexible shaft cover allows the rigid bar to vibrate freely while maintaining good sealing at the humidity chamber wall. In order to monitor the mechanical motion of the two samples, two laser displacement sensors (OADM 20I6441/S14F, Baumer Electric) were mounted above to measure the tip displacement of each sensor separately. The mounting frame for the laser sensors was isolated from the table where the mini-shaker was mounted. A two-tier two-channel amplification circuit was used to measure the shortcircuit current generated by the IPMC samples. All the signals were collected half an hour after the RH level became stable at the preset value, so that the naked IPMC sensor got sufficient time to reach the water vapor transmission balance with the ambient humidity. The weights of two samples are not measured during the test since it is infeasible to weigh the sensors within the humidity chamber. Control signal generation, sensing data acquisition, and processing were all performed through the dSPACE system. Fast Fourier transform was used to extract the amplitudes of the IPMC sensor outputs and the tip displacements at 10 Hz, which were used to evaluate the sensor performances under different humidity levels.

The experimental results for the sensing currents of each IPMC sample under different humidity levels are shown in Fig. 15. For the convenience of comparison, all the data points were normalized with respect to the sensing current amplitude of the encapsulated IPMC sample under 42% RH. First of all, the sensing current amplitude of the encapsulated IPMC sensor was larger than that of the naked one during the full range of the tested humidity levels. This was because the water contained in the coated IPMC sample was more than that absorbed by the naked sample from the ambient environment, even under almost 100% RH. For the uncoated sample, one can also see that there was no peak for the sensing current amplitude as the ambient RH rose up to almost 100%. In view of the result in Fig. 10(a), the latter indicates that the water content in the naked IPMC sensor, even under almost 100% RH, had not reached the optimal hydration level observed in Fig. 10(a).

From Fig. 15, one can see that the sensing current of the uncoated sensor varied significantly as the environmental humidity changes, while the encapsulated sensor maintained excellent



Fig. 15. Sensing responses of IPMC sensors under different humidity levels.



Fig. 16. Tip displacements of IPMC sensors under different humidity levels.

sensing consistency. The experimental results for the tip placements of each IPMC sample under different humidity levels are shown in Fig. 16. Similarly, the results were normalized with respect to the initial point under 42% RH for the encapsulated IPMC sample. It can be seen that the tip displacement of the naked IPMC sample kept going up as the ambient RH increased, while the coated sample maintained a stable mechanical property.

6.2. Sensing consistency test following exposure to liquid media with different cations

For a naked IPMC sensor working in a liquid medium, it is expected that the sensing properties will be affected by both the solute and the solvent in the ambient fluid. For example, if the solution where the IPMC sensor operates contains sufficient amount of other cations which are different from the cations within the IPMC (typically Li), those cations tend to exchange with each other given the intrinsic ion-exchange property of Nafion. With different cations, IPMCs have different performances in both actuation and sensing, as discussed in [26,27]. Therefore, it is desirable to protect the IPMC sensor from the outside media.

To evaluate the sensing consistency of the IPMC sensors in the solutions, one naked IPMC sample and one parylene-coated sample were immersed in solutions with different cations, including Li⁺, Na⁺, K⁺, Ca²⁺, Mg²⁺, H⁺. The concentrations of those solutions were all 1 mol/L and the anions were all chloride. The naked and coated IPMC samples were first soaked in one solution together for 12 h; then they were taken out, dried and put one by one into the same experimental setup with humidity chamber shown in Fig. 14. The mini-shaker provided 10 Hz excitation on the base, and the circuit and the laser sensor collected the sensing currents and the tip displacements of the IPMC samples, respectively. To make sure the consistency of the testing conditions, the base-vibration had fixed amplitude and the humidity level inside the humidity chamber was always set to 40% RH. After the testing in the humidity chamber, the same naked and coated samples were soaked in another solution together for 12 h. Considering the metal corrosion problem mentioned in Section 5, the naked IPMC sample was submerged in the solutions without wires soldered on its electrodes; it was clamped with wires attached onto the surface electrodes when tested in the humidity chamber.

The experimental results for the sensing current amplitude of the naked IPMC sample after its soaking in different solutions is shown in Fig. 17. On the *x*-axis, the label order from left to right is the same order of the solutions that the naked IPMC sample was soaked in. All the data points were normalized with respect to the first point on the left for the case of Li⁺. It should be noticed that the naked sample had been soaked in each solution with different cations and tested in humidity chamber for twice. The reason for repeating the experiments is to accommodate the influence of the

Table 3	
Uncoated IPMC expansion in organic solvent	

Naked IPMC sensor	Length (mm)	Width (mm)	Thickness (µm)	Volume (mm ³)
Before ethanol	25	3	270	20.25
After ethanol	32.8	4.5	460	67.90
Before gasoline	25	3	270	20.25
After gasoline	26.8	3.6	300	28.93



Fig. 17. Sensing current of naked IPMC sensor in solutions with different cations.

soaking order, since all the experiments were done on the same IPMC sample. From the figure one can see that the naked IPMC sensor showed strong dependence on the cations, indicating that there was significant exchange of ions with the ambient fluid within the 12-hour soaking. Unlike the case for the IPMC actuation where Li⁺ shows the best performance in terms of force generation [26], in this experiment H⁺ has shown much better sensitivity than other cations tested (in terms of the sensing current amplitude), which echoes the results reported in [27] to some degree. Note that the sensing currents for all the other cations (except H⁺) have increased greatly following the second round of soaking. This is believed to be caused by that some residual H⁺ ions staying in the IPMC sample after it was soaked in H⁺ solution for the first time, which contributed to the increased sensing output during the repeated experiments for other ions.

Fig. 18 shows the experimental results for the sensing current of the coated IPMC sample. Similarly, the results were normalized with respect to the first point on the left for the case of Li⁺. It can be seen that the encapsulated IPMC sensor maintained excellent sensing consistency after being soaked in solutions with different cations, indicating that the parylene encapsulation effectively prevented ion exchange with the ambient media. Unlike the case for the uncoated IPMC sample in Fig. 17, the coated sample had only been soaked in each solution for once, since it was already enough to draw a solid conclusion based on the experimental results shown



Fig. 18. Sensing current of coated IPMC sensor in solutions with different cations.

in Fig. 18. The tip displacements of the naked IPMC sample and the coated one were also collected during the experiments, which did not show any noticeable dependence on the types of the cations, suggesting that the cations have much more influence on the electromechanical properties of IPMC rather than the pure mechanical properties.

6.3. Sensing consistency test following exposure to organic solvent

IPMC sensors also have potential applications in some organic fluid media, such as detecting the gasoline flow in automotive engines [17], where the sensor properties could be influenced by the ambient organic solvent. Therefore, similar experiments have been conducted to evaluate the sensing performance of the IPMC sensors in organic fluid media. Both the naked IPMC sensors and the parylene-coated sensor were soaked in ethanol and gasoline under room temperature for 12 h, respectively; then they were taken out and tested immediately in the same humidity chamber mentioned above. Note that ethanol is highly soluble to water while gasoline is not. Two naked IPMC samples were tested in ethanol and gasoline separately, while the same-coated IPMC sample was used in ethanol and gasoline. The experimental results first showed that the naked IPMC sample expanded significantly in volume after being soaked in these two organic solvents, as shown in Table 3. Especially in ethanol, the expanded volume was more than twice larger, which was mainly due to the high water-solubility of ethanol. However, the parylene-coated IPMC sample did not show any noticeable change in volume after soaked in both of these solvents, indicating the excellent impermeability of the parylene layer.

The experimental results for the sensing currents and tip displacements of the tested sensors are shown in Figs. 19 and 20. All the data points were normalized with respect to the corresponding initial states. Note that in Fig. 19(b) the tip displacement for the naked IPMC sample increased a little bit after the gasoline soaking, since the sample expanded by 28% in size and still held sufficient stiffness; for the ethanol soaking, the naked sample became so soft that it could not hold the shape of a cantilever beam and the tip dropped down, so the laser sensor could not detect the displacement of the beam tip, and it had no tip displacement in Fig. 19(b). For the naked IPMC sensors, the sensing outputs had greatly dropped after they were submerged in ethanol and gasoline for sufficient time. It is believed that for the case of ethanol, the water contained in the IPMC sensor had mixed quickly with ambient ethanol given that they are mutually soluble, and for the solvent of gasoline, the water diffused gradually. Therefore, it can be seen that there exists one essential problem for the naked IPMC sensor working in organic fluid media, which is the significantly reduced sensor outputs, in other words, very weak sensitivity. Even if the organic media is insoluble to water (for example, the gasoline), the water contained in the IPMC sensor will not stay long to generate consistent output, but diffuse or be washed away gradually into the ambient media, resulting in the continuous loss of sensitivity and thus the sensing inconsistency. However, as indicated by Fig. 20, the parylene-encapsulated IPMC sensor did not show any clear sign of such problems; in particular, the coated IPMC sample had maintained very good mechanical property and



Fig. 19. Performance of the uncoated IPMC sensor after soaking in organic solvent: (a) sensing current; (b) tip displacement.



Fig. 20. Performance of the coated IPMC sensor after soaking in organic solvent: (a) sensing current; (b) tip displacement.

sensing consistency after being soaked in both ethanol and gasoline for 12 h.

All in all, with comparison to the naked IPMC sensor, these experiments have demonstrated that the parylene-encapsulation does not only improve the sensing consistency of the IPMC sensor in different ionic solutions, it also extends its potential application to other organic fluid media by protecting the IPMC from geometric expansion, mechanical change and water loss caused by the solvent pollution.

7. Conclusion

In this paper we have investigated the performance of encapsulated IPMC sensor based on thick parylene C coating. To solve the problem of water evaporation inside the parylene deposition chamber and control the hydration level of IPMC sensor, the proposed fabrication process features thick parylene coating and tunable water drive-in process. The stiffness for the IPMC sensor before and after the encapsulation was measured to investigate the impact of the encapsulation on the sensor's mechanical property. The water impermeability of the parylene encapsulated sample was tested under heating and soaking condition, respectively, and compared with the uncoated sample. The control of hydration level by the water drive-in step was proven to help improve the sensitivity of the encapsulated IPMC sensor and improve the water content of IPMC to a very high level which cannot be achieved by the uncoated sensor in air. The galvanic metal corrosion on IPMC sensors in water was evaluated by experiments in terms of the contact resistance, and the experiments show that the proposed encapsulation scheme offers excellent anti-corrosion performance. Experiments were conducted to evaluate the performance of the coated IPMC sensor in a humidity chamber under different humidity conditions, following exposure to liquid media with different cations, and following exposure to two organic solvents. Experimental results show that the proposed thick parylene encapsulation can effectively keep the water content inside the IPMC, isolate the IPMC sensor from various ambient environment and maintain the sensing consistency, which allows IPMC sensors to be used in more practical applications.

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