ABSTRACT

Ionic polymer-metal composites (IPMCs) have intrinsic sensing and actuation capabilities. However, IPMCs require ionic hydration to operate. As the most commonly used solvent, water content contained in the polymer changes with the humidity level of the ambient environment, which affects the sensing behavior of an IPMC in air. Motivated by the need to ensure consistent sensing performance of IPMCs under different ambient environments, in this paper we propose thick (up to 10 micrometers) parylene C coating for IPMC sensors, develop effective coating processes, and evaluate the stability of the encapsulated sensors in air. During the process of parylene coating, water molecules would evaporate inside the deposition chamber, resulting in the encapsulated IPMCs’ losing sensing capability. To address this challenge and control the hydration level of an encapsulated IPMC, the proposed fabrication process comprises major steps of parylene deposition, water absorption, and SU-8 seal. The influence of hydration level controlled by the water absorption step is studied to improve the sensitivity of the IPMC sensor. The water impermeability of the proposed encapsulation technique is tested in different media. Experiments have also been conducted to evaluate the performance of the encapsulated IPMC sensor. The sensing consistency and the lifetime of an encapsulated sensor in air are studied in an environment with changing humidity, along with the comparison with an uncoated IPMC sensor. Experimental results show that the proposed thick parylene coating can effectively maintain the water content inside the IPMC and reduce the interference due to the ambient humidity change, which allows IPMC sensors to be used in many practical applications.

INTRODUCTION

Ionic polymer-metal composites (IPMCs), one important class of electroactive polymers (EAPs), have built-in sensing and actuation capabilities [1, 2]. They hold strong promise for versatile applications such as sensors because of their high sensitivity, inherent polarity and direct mechanosensory property,
who minimize the complexity in both the sensor construction and signal processing. Furthermore, they are also biocompatible and amenable to microfabrication [3, 4].

An IPMC sample typically consists of a thin ion-exchange membrane (e.g., Nafion), chemically plated with a noble metal as electrodes on both surfaces [5]. 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, as illustrated in Fig. 1. Studies on modeling IPMC sensors can be found in [6–9]. Recent applications of IPMC sensing capability span measurement of force, pressure, displacement and shear loading, structural health monitoring, and energy harvesting [10–19].

A critical issue in the practical use of IPMC sensors is that IPMCs need ionic hydration to operate for both sensing and actuation. As illustrated in Fig. 1, the sensing mechanism of IPMC is believed to be the charge redistribution induced by mechanical deformation. Ideally, IPMC sensor should respond only to mechanical stimuli. However, 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 [20] and thus results in the difficulty of maintaining consistent sensing properties of the IPMC sensor. Therefore, an encapsulation procedure is necessary for an IPMC sensor for practical applications to have consistent response over time. One possible solution to address this challenge is to coat the IPMC with some waterproof materials to suppress the water permeation.

A few encapsulation processes have been reported for IPMC actuators. For example, Shahinpoor et al. [21] and Akle and Leo [22] proposed the encapsulation of IPMCs using Saran plastic membrane. Franklin [23] reported using Kapton™ film to cover a multilayer IPMC actuator. Malone and Lipson [24] proposed the use of a PDMS membrane material for IPMC encapsulation. However, encapsulation with these high stiffness materials has increased the IPMC Young’s modulus and reduced the IPMC free deflection amplitude, considerably affecting the IPMC performance as actuator.

Recently, IPMC encapsulations with much less stiff materials have been reported. Barramba et al. [25] 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. [26] 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. However, their approaches were primarily focused on IPMC actuators. Experiments have not yet been conducted to verify how these encapsulants maintain the IPMC sensing performance in air.

In this paper we propose the use of thick parylene C (up to 10 µm) as an encapsulant for IPMC sensors, which is shown to effectively suppress the water permeation. The proposed fabrication process enables us to control and maintain the hydration level of an encapsulated IPMC sensor to achieve large signal-to-noise ratio (SNR). Experiments have been conducted to study the water content control, and evaluate the water barrier capability and sensing consistency of the encapsulated IPMC sensor in air.

The remainder of the paper is organized as follows. We first introduce the proposed fabrication process for the IPMC encapsulation. Experimental results are then discussed, followed by concluding remarks.

FABRICATION PROCESS
Coating material

Parylene C is chosen as the coating material for three main reasons. Firstly, parylene C is well-known for the water barrier capability because of its low water vapor transmission rate (WVTR), as shown in Tab. 1. The reported WVTR is measured under certain conditions with various temperatures, relative humidity (RH), and thicknesses of thin films. In this paper, we adopt the data reported by Menon et al. [27] for calculation because they measured WVTR on a film of 8 µm thickness, which is very close to the coating thickness of 10 µm in our experiments. Note that the WVTR is not identical for different thicknesses of parylene films. The actual WVTR for 10 µm thick parylene should be smaller than that measured with 8 µm. The WVTR at 20 °C, 30% RH and WVTR at 20 °C, 90% RH from [27] are used to estimate the water permeation of encapsu-
lated IPMC sensor in air and in water respectively, as shown in Tab. 2. Secondly, as discussed in the previous section, the sensing mechanism of the IPMC sensor is believed to be the charge redistribution induced by mechanical deformation. Therefore, the stiffness of the coating material is another essential concern for the selection of coating material. As a sensor, IPMC typically has a beam shape and needs to bend or vibrate freely in order to generate an effective signal. If the coating material is too stiff or too thick, the sensitivity of the IPMC sensor will be greatly reduced. The Young’s modulus of parylene C is only 0.4 MPa. It can be ignored compared with the Young’s modulus of a typical IPMC sample, which is around 300 MPa [7]. Finally, parylene deposition is conducted in a chemical vapor deposition (CVD) system at room temperature, which enables conformal and true pin-hole-free coating on surfaces with various geometries. This process is compatible with standard microfabrication technologies, which makes it possible to integrate high-density IPMC sensor arrays with electronics on a single platform.

**IPMC sensor fabrication**

IPMC samples were fabricated by traditional impregnation-reduction ion-exchange process [5]. Nafion-1110 (254 μm) membrane from DuPont was first roughened with fine sandpaper. The residues were removed by washing the membrane with ultrasonic cleaner. Boil the membrane in dilute 2 wt% hydrochloric acid for 30 minutes to remove impurities and ions in the membrane. Then boil the membrane in deionized water for 30 minutes to remove acid and to swell the membrane. Immerse the membrane in a platinum complex solution ([Pt(NH$_3$)$_4$Cl$_2$]) for more than 12 hours to allow Pt ions to diffuse into Nafion through the ion-exchange process. After rinsing the membrane with deionized water, place the membrane in a water bath at 40 °C and add 2 ml of sodium borohydride solution (5 wt% NaBH$_4$ aq) every 30 min as the temperature increases up to 60 °C gradually. Once the platinum was fully deposited on the surface of the membrane, the film was cut into beam shape with proper sizes. Beam shape is the most common structure for an IPMC sensor. After soldering two electric wire connectors at the platinum electrodes on two surfaces of the IPMC beam, an IPMC sensor was formed.

**parylene encapsulation**

The parylene encapsulation process was conducted with a parylene coater (PDS2010, Specialty Coating System, Inc.), where parylene C was deposited conformally on the IPMC sensors under a pressure of 26 mTorr. IPMC sensor usually requires hydration to operate; however, it is challenging to coat parylene on a piece of IPMC while maintaining the water inside, since water molecules will evaporate in the deposition chamber under low pressure. To address this challenge and further control the water content inside the encapsulation, we propose one feasible fabrication process which consists of parylene deposition, water absorption, and SU-8 seal (Fig. 2):

1. Deposit parylene on IPMC sensors with thicknesses up to 10 μm;
2. Strip off a small piece of parylene on one tip of the IPMC beam to form an opening;
3. Dip the open tip into deionized water for sufficient time to refill the IPMC with water;
4. Seal the open tip with SU-8.

In the experiment reported in [26], IPMC sample with 1 μm parylene coating still had complete water swelling when immersed in water for 24 h, indicating that 1 μm thickness of parylene encapsulation cannot effectively isolate the IPMC from outside media. It has been reported in [30] and [27] that 8 μm is a threshold value for parylene C film thickness in terms of whether the film is affected by defects or not. Thus, the 10 μm thick parylene coating is selected for the proposed encapsulation process. By controlling the length of absorption time in the dipping step, we can adjust the water content inside the IPMC sensor so that the best sensitivity can be achieved. SU-8 is used here to seal the open tip. Compared with other sealants, SU-8 can cross-link quickly under UV light exposure without heating, which minimize the loss of the water content during the polymer curing process. Another advantage is that cross-linked SU-8 does not absorb water or swell in water.

**EXPERIMENTAL RESULTS AND DISCUSSION**

In this paper, the sensing signals (short-circuit current) of both unencapsulated and encapsulated IPMC sensors were all collected with the same experimental setup of base-vibration for
### TABLE 1. REPORTED WATER VAPOR TRANSMISSION RATE (WVTR) FOR PARYLENE C.

<table>
<thead>
<tr>
<th>WVTR of parylene C (g − mil/100in² − day)</th>
<th>WVTR at 37 °C, 90% RH</th>
<th>WVTR at 20 °C, 90% RH</th>
<th>WVTR at 20 °C, 30% RH</th>
<th>Year of publication</th>
<th>Data source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hubbel et al.</td>
<td>3.3-7.3</td>
<td>None</td>
<td>None</td>
<td>1975 [28]</td>
<td>Measured by authors</td>
</tr>
<tr>
<td>Loeb et al. (Union Carbide)</td>
<td>0.5</td>
<td>None</td>
<td>None</td>
<td>1977 [29]</td>
<td>Measured by company</td>
</tr>
<tr>
<td>Specialty Coating System, Inc.</td>
<td>0.2031</td>
<td>None</td>
<td>None</td>
<td>2007</td>
<td>Measured by company (SCS)</td>
</tr>
<tr>
<td>P.R. Menon et al.</td>
<td>0.207</td>
<td>0.08675</td>
<td>0.04515</td>
<td>2007 [27]</td>
<td>Measured by authors</td>
</tr>
</tbody>
</table>

### TABLE 2. ESTIMATED WATER PERMEATION FOR ENCAPSULATED IPMC SENSOR.

<table>
<thead>
<tr>
<th>Encapsulated IPMC sensor</th>
<th>Length (inch)</th>
<th>Width (inch)</th>
<th>Thickness (mil)</th>
<th>Weight (mg)</th>
<th>WVTR (g − mil/100in² − day)</th>
<th>Water permeation rate (mg/day)</th>
<th>Water permeation (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>in air</td>
<td>0.626</td>
<td>0.121</td>
<td>0.394</td>
<td>32.0</td>
<td>0.04515</td>
<td>0.1736</td>
<td>0.5425</td>
</tr>
<tr>
<td>in water</td>
<td>0.626</td>
<td>0.121</td>
<td>0.394</td>
<td>32.0</td>
<td>0.08675</td>
<td>0.3338</td>
<td>1.0431</td>
</tr>
</tbody>
</table>

### FIGURE 3. BASE-VIBRATION EXPERIMENTAL SETUP FOR COLLECTING SENSING SIGNAL.

![Figure 3](image)

### Impermeability test of encapsulated IPMC

To estimate the water permeation of the parylene coating layer and SU-8 sealant, a test was first conducted on the water evaporation loss from the inside of IPMC sensor under heating. Uncoated and encapsulated sensor samples were heated on a hot plate at 50 °C for 20 min and weighed every 2 min during the test. The test results are displayed in Fig. 4. The uncoated IPMC lost 21.45 wt% of the water after 20 min baking, while the encapsulated one did not show any noticeable water loss, which indicates that the combination of parylene coating and SU-8 sealing prevents the water loss from IPMC under heating.

Another test was conducted by immersing the sensor samples in deionized water for 24 h to further evaluate the water barrier capability of proposed encapsulation. The water swelling of IPMC sensors was evaluated by measuring the weight of samples before and after the immersion. According to the experiment results, the amount of water swelling for the uncoated sensor, whose dimensions are listed in Tab. 2, is 6.2 mg (21.5 wt%); while the encapsulated sample of the same dimensions has a water swelling of 0.6 mg. Compared with the estimated water swelling of 0.3338 mg in Tab. 2, it is reasonable to get a larger water swelling than estimation since the estimation is calculated under 90% RH.

### Control of IPMC hydration level

It is verified in [26] that IPMC actuator requires optimal water content to move effectively, which means that the water sat-
uration in the IPMC is not the best condition for actuation. For the IPMC sensor, it is discussed in [20] that when the amplitude of sensing signals increase with the environment humidity level, the sensor noise increases as well. Therefore it is reasonable to deduce that there might be an optimal hydration level for IPMC sensor to achieve the best sensitivity. To verify this hypothesis, an unencapsulated IPMC sensor was tested with different water contents under 20 °C and 60% RH. The amplitudes of sensing signals at 10 Hz were extracted through fast Fourier transform. The noise levels were obtained by computing the root mean square (RMS) of the amplitudes at other frequencies. The signal-to-noise ratio (SNR) was calculated for each case to evaluate the sensor performance at different hydration levels.

The experiment results are shown in Fig. 5 and 6. For the uncoated IPMC, 0 wt% water content was obtained by heating the sample on hotplate at 60 °C for 20 min. For other cases, this uncoated sample was first soaked in deionized water and then exposed in air for different time to get different water content. Under the condition of 0 wt% water content, the IPMC sensor failed to generate any signal as expected. As the water content increased, both the signal and noise increased correspondingly, while the SNR of the sensor did not follow the same trend. Beyond the point of the 2.5 wt% water content, the SNR continuously decreased, indicating that the noise is growing faster than the signal. When the water content was higher than 4 wt%, it was difficult to collect accurate data experimentally because the water evaporates too fast. In fact, the actual water content in an uncoated IPMC, which has been in the water vapor transmission balance between the polymer and open air with some level of

**FIGURE 4.** EXPERIMENTAL RESULTS OF WATER IMPERMEABILITY TESTS UNDER HEATING.

**FIGURE 5.** PERFORMANCE OF UNCOATED IPMC SENSOR AT DIFFERENT HYDRATION LEVELS.

**FIGURE 6.** PERFORMANCE OF ENCAPSULATED IPMC SENSOR AT DIFFERENT HYDRATION LEVELS.
humidity, could not reach the point as high as that. According to the results in Fig. 7, 1.5 wt% is estimated to be the maximal hydration level for these samples within water vapor transmission balance. These results suggest that an apparent optimal hydration level does not exist for this IPMC sensor, but the water content of around 2.5 wt% would give a good balance between signal amplitude and SNR.

According to the experiment results in Fig. 5, it is necessary to adjust the hydration level for encapsulated IPMC sensors to obtain the best sensitivity. This can be achieved simply by controlling the time of the water absorption step. Figure 6 shows the experiment results for fifteen encapsulated IPMC sensors with different water absorption durations from 0 minute to 3 hours. For the convenience of comparison, these encapsulated IPMC sensors had the same dimensions as the sample used in Fig. 5. The analysis of sensing signal and noise was also the same as what is described for the uncoated sample. Note that for the sample with 0 min absorption time, it was fully covered with parylene without opening. The sample showed nonzero SNR, which is different from the SNR of the uncoated sample with 0 wt% water content. This can be attributed to a tiny amount of residual water inside the IPMC after the parylene deposition.

From the figure, it can be seen that in the first 100 min the noise increased very slowly and with oscillation while the signal amplitude increased consistently. However, beyond that point the noise stopped increasing and the SNR was improved, which is opposite to the performance of the uncoated sensor. One possibility is that the water vapor transmission between air and the uncoated IPMC partially contributes to the noise, while for the encapsulated IPMC this transmission has been blocked by the parylene layer no matter how much water it contains. Therefore, based on the data after 100 min in Fig. 6, it can be predicted that the SNR will continuously grow as long as the water absorption time keeps increasing. In this sense, the encapsulation has a higher limit of water content, which cannot be reached by the uncoated IPMC because of water evaporation.

However, the encapsulated IPMC cannot absorb as much water as we want. During the water absorption process, if the soaking time is too long, the interface between parylene and platinum electrodes will be attacked by water, given the generally known fact that parylene adhesion to platinum is poor [31]. Consequently, the parylene layer will delaminate from IPMC surface, which can cause problems for SU-8 seal and waterproof properties. Improvement of the adhesion between parylene and Pt electrodes by plasma treatment [26] would help extend water absorption time and thus enable higher water content.

Evaluation of sensing consistency of encapsulated IPMC sensor

As discussed in previous sections, IPMC sensor is able to operate with different levels of water content. Yet it is highly desirable to guarantee consistent sensing properties of IPMC sensor for practical applications; otherwise the sensor has to be calibrated every time when the humidity changes. To evaluate the uniformity of output signal of an encapsulated IPMC sensor in air, one uncoated sample and one encapsulated sample were placed together in open air for sufficient time with changing humidities. The sensing signals generated in aforementioned experimental setup (Fig. 3) were recorded and compared in terms of signal amplitude.

The experiment results are shown in Fig. 7. The amplitude of sensing current of the encapsulated IPMC sensor is much larger than that of the uncoated one. This is because the former contains more water than the latter. As shown in the Fig. 5, amplitude of less than 0.01 μA corresponds to a water content of less than 1.5 wt% for the uncoated sample, which indicates the limit of hydration level for the uncoated IPMC sensor that has been in the water vapor transmission balance with the ambient humidity. However, the encapsulated sensor is not constrained by this limit. During the period of five days, the sensing current of uncoated sensor varies largely as the environment humidity changes, while the encapsulated sensor proves to be able to maintain good sensing consistency. To better compare their performances, the normalized sensing currents with respect to each sensor’s initial signal are given in the Fig. 7. For the encapsulated sensor, its sensing current has been decreasing gradually but very slowly, which can hardly be noticed in the figure. This is because the encapsulation layer can effectively suppress the water evaporation although it cannot completely stop it. Additionally, the water vapor concentration inside the encapsulated IPMC is always larger than that in the air, resulting in its continuous water loss.

Therefore, it can be seen from the testing results that the encapsulated IPMC sensor has maintained very good sensing consistency over a period of five days with a standard deviation of the normalized signal amplitudes of 0.024, while the signal of the uncoated IPMC sensor varied largely depending on the change of ambient relative humidity with a standard deviation of 0.442.

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 adjustable water absorption. The water impermeability of the parylene encapsulated sample was tested under heating condition, and compared with the uncoated sample. The control of hydration level by water absorption step is 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. The sensing consistency has been evaluated for both the uncoated and encapsulated IPM-
C sensors over a period of five days. The experimental results demonstrate that, compared with the sensing current of the uncoated sensor that changes with the ambient humidity, the parylene encapsulation helps the sensor maintain much better stability of sensing signal.

For the future work, we will further improve the water impermeability of the encapsulation layer and investigate the feasibility of applying such encapsulated IPMC sensor in other media like gasoline.

ACKNOWLEDGMENT

This work was supported in part by National Science Foundation (ECCS 0547131) and the Office of Naval Research (N000140810640, N000141210149).

REFERENCES


[17] Brufau-Penella, J., Puig-Vidal, M., Giannone, P., Graziani,


