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Review

# A Review of Plasma Chemical Surface Treatment on Parylene for Deposition on Ionic Polymers

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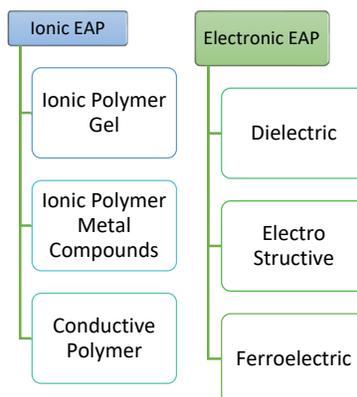
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**Abstract:** The field of smart materials is attaining global industrial importance rapidly. The ionic polymer metal metal composites withheld there importance in this field. They are not only used as biocompatible devices but also in the field of thin and conformal bio coatings. The nafion 117 is unstable at elevated temperatures and its water intake resistance is low, so the nafion 117 is coated with the noble material to increase its thermal stability and water resistant property. The high cost of noble metal make its too costly to use in day to day applications so parylene has been used as substitute for noble metal in order to reduce the cost. The high thermal stability and water intake property if parylene make its suitable for the same. Multiple chemical treatments like plasma ionization and others have been done on the parylene coated nafion to increase its desirable properties considerably. The low adhesion rate between parylene and nafion 117 has been increased upto the certain extend by the use of ionized plasma. This paper reviewed the plasma chemical treatment of parylene coatings for the biocompatible devices and provides an insight knowledge on classification, structure and thermal stability of the parylene coatings

**Keywords:** Nafion117; actuators; parylene; plasma

## Introduction

There are certain polymers that gives respond when stimulated by the inputs such as heat, light, pressure, electric or magnetic field. Those polymers stimulated by the electric field come under the category of electro active polymer. These EAP polymers can be further classified as electronic EAP or Ionic EAP as in Figure 1. Ionic EAP's has the capability to mimic the artificial muscles behavior when applied the stimulated input [1]. EAP's are resilient in nature with high fracture tolerance. These ionic polymers can also be termed as smart material when used under the certain circumstances. They exhibit the properties which are highly desirable in artificial muscles. The EAP's can be classified into ionic and electronic EAP's.



**Figure 1.** Classification of Electroactive Polymers [2].

These EAP's transducer have lesser density, very small driven voltage and high fatigue strength when compared with other materials of same category. The high actuation strain with small driven voltage is the most desirable property of them.

**Table 1.** Comparison between Electroactive Polymers.

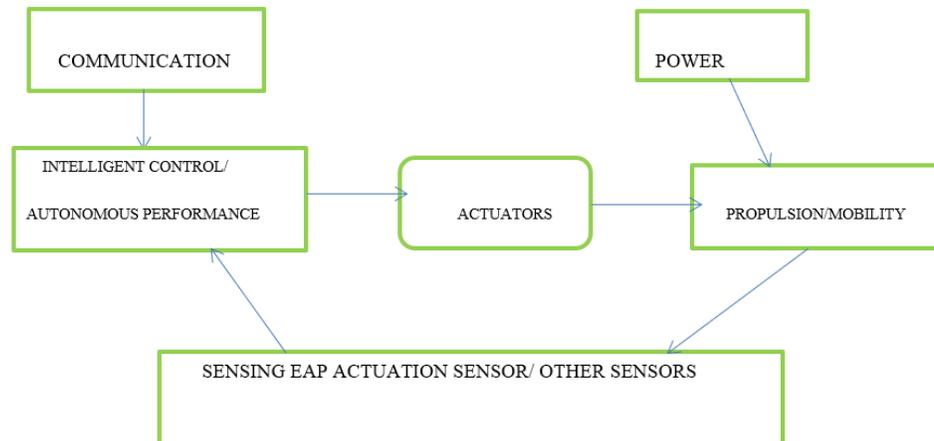
Property	EAP	SMA	EAC	Reference
Force [MPa]	0.1–25	200	30–40	[3]
Actuation strain	Over 300%	<8% [short fatigue life] Typically 0.1–0.3 %	Typically 0.1–0.3 %	[2,3]
Density	1–2.5 g/cc	5–6 g/cc	6–8 g/c	[3]
Consumed power	m-Watts	Watts	Watts	[3]
Reaction speed	μsec to min	msec to min	μsec to sec	[2,3]
Drive voltage	Ionic EAP: 1–7 V 5–800 V Electronic EAP: 10–150 V/μm	5-Volt	50–800 V	[3]
Fracture behavior	Resilient, elastic	Resilient, Fragile elastic	Fragile	[3]

There other transducer materials used are Electrostatic silicone elastomer [4], Polymer Electrostrictor [5], Single Crystal Electrostrictor [6] and Single Crystal Magnetostrictor [7]. The comparison between various transducer materials is presented in the table:

**Table 2.** Comparison between various transducer materials.

Property	Electrostatic silicone elastomer	Polymer Electrostrictor	Single Crystal Electrostrictor	Single Crystal Magnetostrictor	Reference
Actuation strain	100%	4%	1.7%	2%	[4,8]
Blocking area	0.2 MPa	0.8 MPa	65 MPa	100 MPa	[5,6]
Reaction speed	msec	μsec	μsec	μsec	[8]
Density	1.5 g/cc	3 g/cc	7.5 g/cc	9.2 g/cc	[8]
Drive field	144 V/μm	150 V/μm	2 V/μm	2500 Oe	[6]
Fracture toughness	Large	Large	Low	Large	[7]

EAP actuated devices can perform multiple function when connected to the power sources. These devices can work both in wet as well as in dry environment. Their unique property enables them to use as sensors as well as mobility devices. The energy storage property of transducer materials make them suitable to use as a fuel cell and other energy storage devices.



**Figure 2.** Elements of EAP Structure [9].

### 1.1. Ionic Electroactive Polymers

The one form of electroactive polymers is ionic polymer metal composites. They have the advantages of large bending displacements and bilateral actuation as per voltage polarity. These ionic polymers are used in the form of ionic gel, ionic polymer metal composites, conductive polymers and carbon nanotubes. These material also exhibit certain disadvantages like slow response, low electromechanical efficiency and when acting in reverse they induce a low actuation force [10].

### 1.2. Ionic EAP Characterization

A comparative performance matrix has been developed to compare the EAP actuators with other materials including piezoelectric materials, shape memory alloys and simple motor also [11]. Key parameters will be identified and various methods have been developed to review its performance. The motion of cations at microscopic level will define their performance and can be visualized by high resolution cameras. The complexity of this mechanism will be due to water wavier rate, moisture content and hysteretic characteristics. According to their properties and this characterization the ionic EAPs are classified as follows:

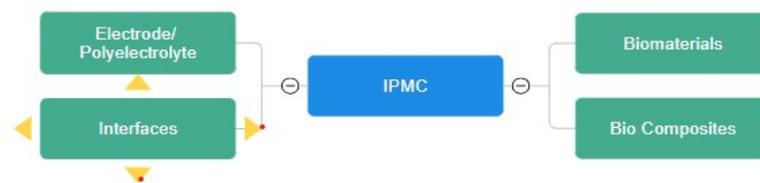
**Table 3.** Comparison between various Ionic EAP materials.

Actuator	Working Principle	Advantages	Disadvantages	Example
Electrorheological fluids	In the presence of electric field their viscosity changes, inducing dipole moments.	Induce haptic mechanism	Requires high voltage	polymer particles in fluorosilicone base oil
Conductive Polymers	When voltage is applied due to oxidation or reduction reaction there is a flow of ion depending on the cell polarity.	With low voltage induce large force. Can be used as biocompatible device.	Under the fatigue loading a cyclic deformation is shown.	Polypyrrole, Polyethylenedioxythiophene, Poly[p-phenylene vinylene], Polyaniline, and Polythiophenes.
Ionic Gels	On applying a voltage movement of	Low voltage required for operation.	Very thin film required for operation.	Poly[vinyl alcohol] gel with dimethyl sulfoxide

	hydrogen ions is there which simulates with chemical reaction according to acid or base.	High compatibility for biological muscles.		
IPMC	Movement of positive ions within fixed surface.	Low voltage. Provide all kind of bending on application of force.	Low frequency response. Permanent displacement due to flow of DC current.	Nafion® [perfluorosulfonate made by DuPont]. Flemion® [perfluorocoboxylate, made by Asahi Glass, Japan]

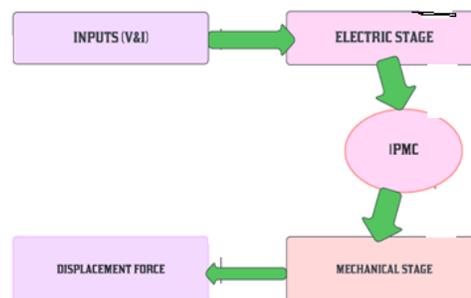
#### 1.4. Actuation of Ionic Polymer Metal Composites

IPMC are mostly soft sensors actuator materials. The actuation response of IPMC is due to chemical structure of IPMC and morphology of its surface properties, water level of cations and its surface properties [12]. The bidirectional coupling in IPMC is created by the potential difference across the cathode. The flow of water ions are responsible for the flow of charge. The IPMC are coated with noble metal in order to hindrance the flow of water ions [13].



**Figure 3.** Applications and Working of IPMC [14].

The electro mechanical response of IPMC has been noted to obtain optimized +actuation response. By using different combination of cations the different patterns of IPMC action can be obtained. The change alternating and direct current can also lead to the change of different bending patterns. In the case of nafion based IPMC its initial motion towards anode is minimized by introducing the slow voltage in opposite direction [15].



**Figure 4.** Flow Diagram of IPMC Working [16].

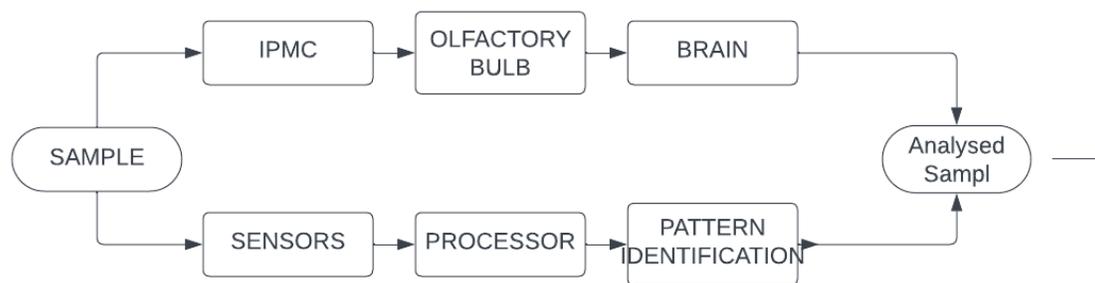
## 2. Electrode and Ionomer Morphology

The actuation of IPMC depends on the transportation of water ions. Therefore, an optimized form of ionomer with good ion exchange quality is recommended [17]. A large surface area with good capacitance and good bending stiffness is required to meet the high actuation rate of IPMC.

## 3. Electromechanical Analysis of Parylene Coated IPMC Electrode

The analysis was done to test the capacity of IPMC working as capacitors to eliminate the battery from the desired systems [18]. The interrelation between capacitor and IPMC is also analyzed. On the application of voltage the energy stores in the form of electric energy which can be converted to mechanical output. The example of such appliances is electronic nose and electronic tongue [19]. TS-5000Z & SA402B are the example of electronic nose & tongue respectively. An interdisciplinary approach is applied along with the use of complex chemicals. Both quantitative & qualitative analysis is done by the same [20].

In order to evaluate the electromechanical characteristics of IPMC the spectroscopy is conducted in which a unique dielectric properties have been shown under the tensile stress. The distance between electrodes influences the capacitance properties of the IPMC. It is inversely proportional to the distance between electrodes [21]. The dielectric phenomenon has been studied by two processes dielectric polarization & dielectric relaxation. On the lower frequencies the polarization takes place while at high frequencies relaxation occurs in the IPMC. In order to analyzed the application of IPMC in field of robots and other practical appliances. The frequency decrease along with the increasing the weight. The permittivity also decreases as the frequency decreases but the dielectric constant increases [22]. The most important characteristics of IPMC is the relation between applied electric field and various loading condition. Due to heterogenous structure most polymers display interfacial polymerization characteristics.



**Figure 5.** Comparison between bio organs with electronic nose.

## 4. Electromechanical Non Linear Deformation of IPMC

### 4.1. Black Box Model

The IPMC shows the nonlinear or cantilever deformation under the DC excitation voltage. A approach known as BLACK BOX is used to study the nonlinear behavior of IPMC [23]. Black Box is neural network model developed based on human brain. Neural connections between the elements will be developed based on artificial intelligence [24]. The coupling behavior of IPMC for electrical. Mechanical and chemical propertied have also been analyzed through the same model. In the black box model the relationship between inputs and outputs of IPMC have been established through statistical equations. The final displacement is studied for its linear & nonlinear displacement [25]. The dynamics of black box model helps to predict the behavior of IPMC according to the feedback or transfer characteristics. The camera based measurement system is installed for output analysis [26]. This model relates the IPMC behavior of IPMC with temperature & frequency [10 to 100 Hz] [27].

#### 4.2. Grey Box Model

This approach is done to investigate the electromechanical transducer characteristics of IPMC. These models deal with the system physics. This model compensates two types of models both white and black models [28]. The mathematical representation of model is written as:

$$[v[\omega]f[\omega]] = [Z_{11}Z_{21}Z_{12}Z_{22}] \cdot [i[\omega]u'[\omega]] \quad [29]$$

where,  $v$  &  $I$  stands for voltage and current of the system.  $f$  &  $u$  are external loading force & tip displacement velocity.  $Z_{11}$  shows the electrical impedance of the immobile IPMC,  $Z_{22}$  defines mechanical impedance of the electrically disconnected IPMC, while  $Z_{12}$  and  $Z_{21}$  are the electrical ↔ mechanical coupling terms [30].

#### 4.3. White Box Model

These models are used for macroscopic level analysis of the system to understand the sub processes taking place. In 1998 these models are used for analysis of Ionic Polymer Gels actuation phenomenon in mechanical, electrical & chemical field [31]. In 2000, Nemat-Nasser studied the bending stresses and suggested that bending occur due to the thickness gradient in the direction of applied voltage. For thermodynamic bending this model has been studied under irreversible static conditions. White box model proposed that bending occur due to the shifting of cations & anions which carry the mobile charge within the material [32]. The cluster of charge is shift to one direction results in formation of electrical dipole. Net electric field is set up due to dipole formation inside the membrane. Multiple mathematical equation were used to describe the sensing & bending phenomenon of IPMC. The equations for the polymers were as below:

$$\mathbf{E} = \mathbf{D} / \kappa \epsilon = \nabla \phi,$$

$$\nabla \cdot \mathbf{D} = \rho = F [C^+ - C^-]$$

$$\partial C^+ / \partial t + \nabla \cdot \mathbf{J} = 0, \quad [33]$$

where,  $E$  is the electric field,  $D$  is the electric displacement,  $\phi$  is the electric charge, charge density  $\rho$ , ion concentrations  $C^{++}$  and  $C^{-}$ , and ion flux  $J$  [34].

### 5. Coating Methods for IPMC

#### 5.1. Chemical Vapor Deposition

This is sublimation process where deposition takes place from solid to gaseous state. In this process a parylene precursor is used to start the reaction and deposition takes place in a vacuum chamber. The deposition chamber is heated to a suitable temperature and pressure [35]. The CVD consists of the following processes sublimation, pyrolysis, deposition and vacuum deposition in a cold trap. The parylene dimer is loaded in a sublimation chamber at 150°C-175°C. This dimer is converted & transferred to the pyrolysis chamber at approx. 650°C. At such a high temperature the parylene is converted to the vapor form directly from the solid form [36]. The deposition of parylene at nafion-117 takes place at room temperature at 25°C in the presence of vacuum created by the liquid nitrogen [37].

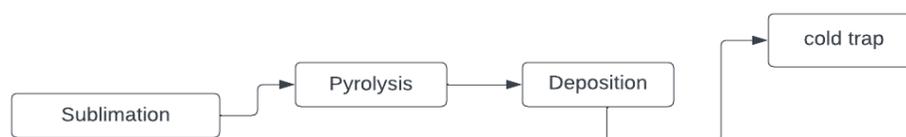


Figure 6. CVD of Parylene Deposition [38].

The advantages of CVD lies in the flexibility of using variety of materials like glass, ceramics and polymeric substances. They can withstand the large variation of temperature ranging from extremely low to extremely high [10C-600C] without the occurrence of failure. The CVD coatings have high resistance to corrosion and extreme wear resistance [39]. The only disadvantage of CVD is limited film thickness due to the coating stress [40]. The other method for parylene deposition are PVD and PEVCD.

### 5.1.1. Parylene

Parylene is an element of paraxylene group [41]. It provide a superior quality of barrier properties and provides corrosion free and chemically inert pin hole free coatings. The parylene coatings have high dielectric strength and act as a very powerful insulator compared to other materials [42]. The low coefficient of friction as compared to Teflon gives it better resistance and make these coating clean also. The three forms of parylene are parylene N, parylene C & parylene D. Parylene C has good barrier properties along with high dielectric strength but has its high processing cost. It has one chlorine atom. The parylene C exhibit very low permeability hence provide better moisture resistance and pin hole free coatings. Parylene N is a base material for parylene which shows high dielectric strength while Parylene D shows high temperature resistance and better resistance from UV rays.

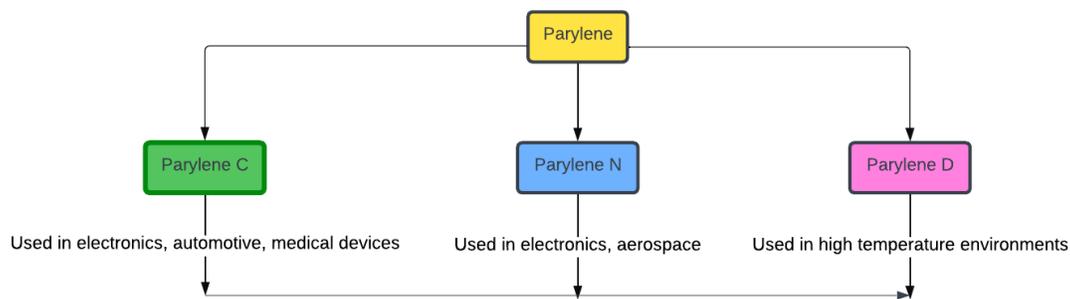


Figure 7. The Parylene & its Types.

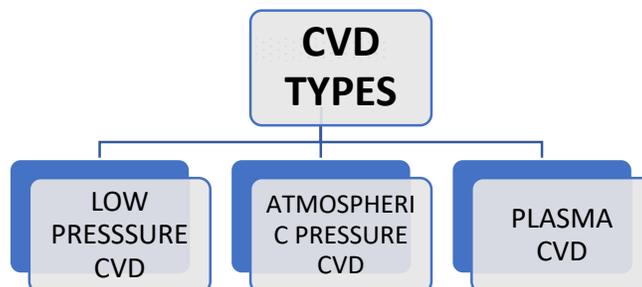
Table 4. Comparison between Types of Parylene.

	Parylene C	Parylene D	Parylene N	Parylene HT	Reference
Structure	Completely linear, high crystalline material, modified by a substitution of chlorine atom for one of the aromatic hydrogen's.	Completely linear, high crystalline material, modified by a substitution of chlorine atom for two of the aromatic hydrogen's.	Completely linear, high crystalline material.	Completely linear, high crystalline material and replaces the alpha hydrogen atom of parylene N with fluorine.	[43]
Aromatic rings					[42,43]
Properties	Useful combination of physical and electrical properties, low permeability to moisture and corrosive gases.	Useful combination of physical and electrical properties, low permeability to moisture and corrosive gases, withstand slightly higher Temperatures.	Primary dielectric, low dissipation factor, high dielectric strength, low dielectric constant invariant with frequency.	Low coefficient friction, dielectric constant; withstand high temperature, long term UV stability and highest penetrating ability of the four variants.	[43]

### 5.2. Plasma Enhanced Chemical Vapor Deposition Process

PECVD is plasma enhanced process for organic & inorganic deposition of doped films. In the PECVD process there is a cross linking between the thin films of polymer. The use of plasma gas helps in ionization of large group of atoms. A vacuum is created at approx. pressure of less than 0.1 torr. The temperature variation is from room temperature to 360°C. The low range of temperature in PECVD enables to control the thermal stresses therefore increase the bond strength of thin films [44].

The primary advantage of PECVD over CVD is the lesser range of temperature which is 600°C to 800°C in CVD while room temperature to 350°C in the PECVD which enables the coating of temperature sensitive devices at lower temperature [45].



**Figure 8.** CVD & its Types.

### 5.3. Comparison between PECVD and CVD

**Table 5.** Comparison between CVD & PECVD [46].

Property	CVD	PECVD
Coating Gas	Chemical reaction of precursor gas for deposition process.	The precursor gas is introduced in the deposition chamber for deposition purpose the ionized plasma gas is used.
Coating Direction	Multidirectional Deposition process.	Coatings occurs in line site process
Coating Adhesion	Good	Excellent
Layer thickness	Thicker [1-10 $\mu\text{m}$ ].	Thinner [0.1-2 $\mu\text{m}$ ]
Application	Cutting tools, wear parts & jewelry options.	Cutting parts & medical implants.
Coating Properties	Hard, water resistant & corrosion resistant.	Hard, water resistant & low friction.
Temperature	Higher deposition temperature.	Lower deposition temperature.

### 5.4. PECVD Working & Equipment

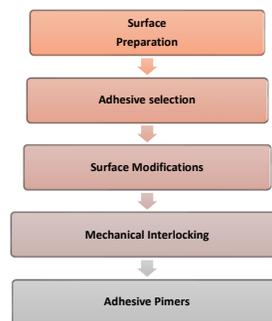
Two parallel electrodes were used for deposition process and substrate is placed inside the deposition chamber. The heating range for substrate is between 250°C-350°C. The common precursor gasses used are silane and ammonia along with the mixture of inert gas [nitrogen or argon] [47]. Shower head is fixed upon the chamber which helps to spread gas along the substrate. For uniform mixing of gas the multiple orifice like outlet is provided. The ignition of plasma is done by placement of two parallel electrodes with an electric voltage between 100eV-300eV. Due to the presence of precursor gas highly ionized plasma gas collides with the energized electron. The desorption of extra chemicals will be done and final deposition will take place [48].

### 5.5. Plasma Treatment for Adhesion Property

The adhesion of parylene with the nafion 117 can be enhanced by the chemical treatment on parylene and on substrate material. The surface of base material is roughened than the adhesion promoter in the form of primer will be introduced in the top layer of roughened surface [49]. The roughening of surface is done by the sand blast or by the plasma argon blast.

### 5.6. Plasma Treatment of Surface

Due to the low surface energies of polymers the wettability and adhesion properties of parylene are not good. The formation of oxide layer increases the surface energy hence resulting in better adhesion. The term for the above treatment is corona method. In this method a high voltage is applied at very high frequencies [50]. The plastic part is placed within the space between the electrodes. The electrical discharge converts the surrounding air into plasma. The plasma particles clean and oxidize the substrate surface increasing its surface energy.



**Figure 9.** Steps in Adhesion Enhancement of Parylene [51].

### 5.7. Thermal Stability of Parylene Coated Nafion 117 Films

The low thermal stability of parylene films is main reason behind its limit use in various devices and services. The literature suggests copolymerization of parylene C and parylene F films for the increased thermal stability as well as increased adhesion of parylene films [52]. The adhesion property is 10.4 times increased and it is confirmed by the FTIR [53]. The higher thermal stability of 301.8 °C to 338.8 °C in a N<sub>2</sub> atmosphere and from 232.2 °C to 273.3 °C in an O<sub>2</sub> atmosphere hence enhancing the use of parylene in the field of bio MEMS devices [54].

**Table 6.** Temperature stability and melting points of the parylenes based on industry literature [55].

Parylene Types	Long-Term Temp [°C] Duration = ~10+ Years	Short Term Temperature Duration = ~1 Month	Melting Point Temperature	References
Parylene C	80	115	290	[55]
Parylene N	60	95	420	[55,56]
Parylene D	100	135	380	[55]

## 6. Conclusion

The chemically treated and highly ionized plasma parylene is best suited for adhesion enhancement and is thermally stable at high temperatures in the presence of nitrogen and oxygen. The increase in the surface energy of parylene yields the best optimized results for the adhesion and thermal stability. The water intake property of parylene coated nafion 117 is also increased

sufficiently. The high water absorption rate gives more free cations hence resulting in better conductivity therefore producing more energy. The better thermal stability makes the parylene compactible to the withstand high temperature and thermal stress. This also extends the application of parylene coated nafion in underwater robots and in other extreme climatic conditions.

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