



## Short communication

# Investigation of membrane electrode assembly (MEA) processing parameters on performance for wholly aromatic hydrocarbon-based proton exchange membranes

Abhishek Roy<sup>1</sup>, Michael A. Hickner<sup>2</sup>, Ozma Lane, J.E. McGrath\*

Macromolecules and Interfaces Institute, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061, United States

## ARTICLE INFO

## Article history:

Received 14 December 2008  
 Received in revised form 18 February 2009  
 Accepted 19 February 2009  
 Available online 4 March 2009

## Keywords:

Membrane electrode assembly  
 Proton conductivity  
 Fuel cell performance

## ABSTRACT

The membrane electrode assembly (MEA) is well known to be the critical component of a proton exchange membrane fuel cell (PEMFC). The interface between the membrane and the electrodes plays a significant role in controlling overall performance and durability. Moreover, the processing parameters to produce MEA have a major influence on the interface and allow novel materials to be evaluated in high performance fuel cell devices. In this paper, several parameters influencing processing conditions for MEAs with membranes based on novel wholly aromatic polymers were investigated and optimized processing parameters are suggested. This paper demonstrates that the water content present in the copolymers during MEA fabrication significantly influences the nature of the interface and, consequently, fuel cell performance. The optimized fabrication temperature reflects viscoelastic behavior and appears to be close to the hydrated glass transition or  $\alpha$  relaxation temperature of the copolymer. It is suggested to be a function of both water content, which can plasticize and reduce  $T_g$ , and molecular weight of the copolymer.

© 2009 Elsevier B.V. All rights reserved.

## 1. Introduction

The integral most basic part of a fuel cell is the membrane electrode assembly (MEA). At the most fundamental level, MEA consists of a proton exchange membrane (PEM) bonded to two electrodes, which serve as the anode and cathode. The electrodes are usually platinum black metal or carbon supported platinum particles dispersed in a copolymer matrix [1,2]. The proton exchange membranes are responsible for proton or hydronium ion transport between the anode and the cathode. The MEA manufacturing process typically involves the preparation of a perfluoro sulfonic acid (PSA) copolymer, usually Nafion platinum (Pt) alcohol/water dispersion, which is also referred to as the “ink.” The latter is painted onto decals to obtain 0.2–1.0 mg cm<sup>-2</sup> of Pt loadings. The decals are then hot pressed onto the PEM at selected time and temperatures to form the MEA [3]. Alternative procedures such as reverse roll or spray coating are known, but are not considered here.

Our research group had been engaged over the past several years in synthesizing and characterizing PEMs based on partially

disulfonated poly(arylene ether sulfone) (BPSH-xx) random and multiblock copolymers [1,4–20]. The most studied random copolymer has been a biphenol-based disulfonated poly(arylene ether sulfone) known as BPSH. In BPSH-xx, the BP stands for biphenyl, S for sulfone, H for the acidified form and xx is the degree of disulfonation (Fig. 1). Fundamental copolymer and transport properties evaluated for BPSH-xx copolymers demonstrated comparable results at moderate to high humidities to that with the state of the art material Nafion® [9,10,20]. Preliminary fuel cell testing on BPSH-xx copolymers also showed satisfactory results both in H<sub>2</sub>/air and DMFC systems. On one hand, the fundamental transport properties are a function of copolymer chemical composition and structure. On the other hand, the fuel cell performance is a function of copolymer fundamental properties, electrode composition, and the MEA fabrication process. Optimizing these factors maximizes the fuel cell performance for a novel PEMFC. Substantial research is ongoing in synthesizing new PEM and electrode materials [1,21–23]. In this paper, a summary of recent investigations on the MEA fabrication process will be highlighted.

Perhaps the most important objective during MEA fabrication is to achieve a good interfacial bond between the electrodes and the membrane. Good melt flow properties of a copolymer at some temperature are critical to achieve good bonding and adhesion to the electrodes. Therefore, a thorough understanding of the parameters affecting the viscoelastic flow properties

\* Corresponding author. Tel.: +1 540 231 5976; fax: +1 540 231 8517.

E-mail address: [jmcgrath@vt.edu](mailto:jmcgrath@vt.edu) (J.E. McGrath).

<sup>1</sup> Current address: The Dow Chemical Company, Edina, MN, United States.

<sup>2</sup> Current address: Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA 16802, United States.

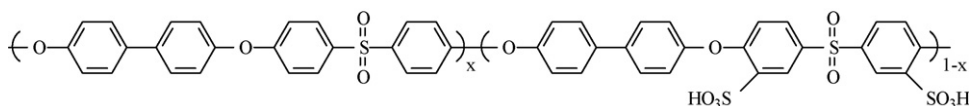


Fig. 1. Chemical structure of BPSH-xx random copolymer.

Table 1

Depression of glass transition temperature under hydrated conditions.

Copolymer	Water uptake (%)	$T_g$ of dried sample ( $^{\circ}\text{C}$ )	$T_g$ or $\alpha$ relaxation of fully hydrated sample ( $^{\circ}\text{C}$ )
BPSH-30	26.7	257	136
BPSH-40	49.5	267	126
BPSH-50	108.7	272	98
Nafion-1135	33.0	160	99

Data obtained from Ref. [19].

can serve to facilitate optimizing processing conditions for MEA fabrication. These parameters include the processing temperature or the MEA bonding temperature, the molecular weight, and the possible plasticization effect of water content during fabrication. The MEA bonding temperature is normally close to the glass transition or the  $\alpha$  relaxation temperature of the copolymer, as illustrated by the modulus vs. temperature plot depicted in Fig. 2 for BPSH-35. In addition to glass transition temperature, control of molecular weight and the water content in the copolymers can help optimize the processing temperature. The critical impact of the molecular weight and water content of a copolymer on MEA processing temperature is discussed below.

### 1.1. Importance of molecular weight of the copolymer on processing temperature

For most copolymers, melt viscosity has a linear relationship with molecular weight up to a particular point, which is known as the entanglement molecular weight. Below the entanglement molecular weight, the flow properties of a copolymer scale approximately linearly with molecular weight. Above the entanglement molecular weight, however, the viscosity of a copolymer is proportional to 3.4 power of the weight average molecular weight [24]. As a result, the processing temperature may need to be shifted to a higher temperature to compensate for the effect of the markedly increased viscosity. Nafion membranes of an apparently constant molar mass have been well studied. These features have not been extensively reported for alternate hydrocarbon membranes such as BPSH [1].

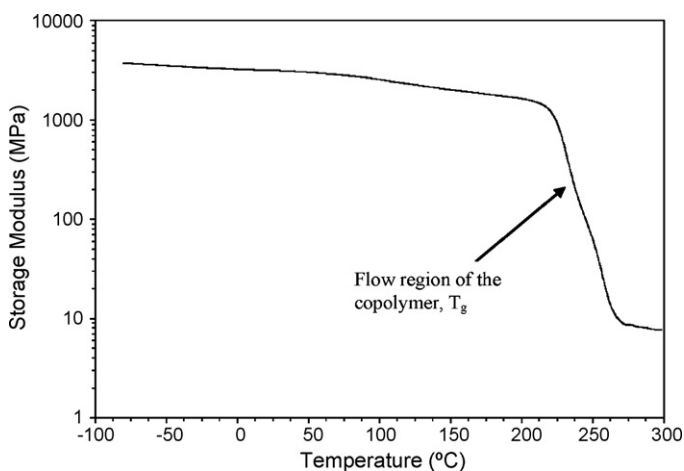


Fig. 2. Storage modulus vs. temperature graph for BPSH-35 random copolymer.

### 1.2. The impact of water content in BPSH copolymer on the processing temperature during MEA fabrication

Water is known to sorb into hydrophilic copolymers in at least three different states, which can be categorized according to thermal and relaxation properties [19]. *Non-freezing bound water* is strongly associated with the copolymer and shows no  $T_m$  (melting point) by differential scanning calorimetry, but will depress the  $T_g$  of the copolymer as a function of the volume fraction. *Freezable bound water* may be weakly bound to the copolymer (or weakly bound to the non-freezing water), and displays a broad melting behavior around  $0^{\circ}\text{C}$ . Finally *free water* shows a sharp melting point at  $0^{\circ}\text{C}$ . The tightly bound water acts as a plasticizer and, hence, can influence melt the flow temperature of a copolymer. Table 1 shows the depression of the glass transition temperature of BPSH and Nafion copolymers under fully hydrated conditions [19]. The water content in the membrane when it undergoes MEA fabrication will clearly influence the glass transition temperature and, hence, the processing temperature.

The main objective of this paper was to investigate the influence of water content and hot pressing temperature on the BPSH-MEA fabrication process, as well as to determine how these variables influences fundamental transport properties such as proton conductivity. This paper will also describe the apparent preferred operating processing conditions for fabricating membrane electrode assemblies. For this experimental study, disulfonated poly(arylene ether sulfone) random copolymers were chosen [8]. However, the fundamental findings obtained in this research are proposed to be applicable to Nafion<sup>®</sup> and other alternate membrane systems.

## 2. Experimental

### 2.1. Materials

Highly purified 4,4'-dichlorodiphenyl sulfone (DCDPS) was provided by Solvay Advanced Polymers. 4,4'-Biphenol (BP) was obtained from Eastman Chemical. These monomers were well dried under vacuum before copolymerization. Disulfonated derivatized comonomer 3,3'-disulfonate, 4,4'-dichloro diphenyl sulfone (SDCDPS) was synthesized according to modified literature methods [25], analyzed and dried under *vacuo* at  $160^{\circ}\text{C}$  before copolymerization. The solvents *N,N*-dimethylacetamide (DMAC, Fisher) or *N*-methyl-2-pyrrolidinone (NMP; Fisher) were vacuum-distilled from calcium hydride onto molecular sieves. Potassium carbonate (Aldrich) was dried *in vacuo* before copolymerization. Toluene, sodium chloride, 30% fuming sulfuric acid and methanol were obtained from Aldrich and used as-received. The fuming sulfuric acid was further analyzed for active  $\text{SO}_3$  concentration [25].

## 2.2. Copolymers studied

The synthesis of 4,4'-biphenol-based disulfonated poly(arylene ether sulfone) copolymers (BPSH-35; 35 mol% disulfonated monomers) was successfully carried out via nucleophilic aromatic substitution as described earlier [8,9]. The number average molecular weight of the copolymer was  $80,000 \text{ g mol}^{-1}$ . The structure is given Fig. 1, where  $[(1-x)=35\%]$ .

## 2.3. Differential scanning calorimetry (DSC), thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA)

The DSC studies were performed in a TA DSC instrument using liquid nitrogen as a cooling medium for the sub-ambient experiments. Samples were equilibrated in a relative humidity oven (ESPEC SH 240) to achieve the desired hydration levels, then placed in thermally sealed pans capable of withstanding pressures up to 100 atm. Samples were cooled to  $-70^\circ\text{C}$  and then heated at a rate of  $5^\circ\text{C per min}$  under a  $\text{N}_2$  atmosphere.

The TGA studies were conducted on the samples equilibrated at different RHs to achieve the desired hydration level. Just before the experiment, the samples were removed from RH chamber, and placed in a TGA sample pan. The samples were heated at  $10^\circ\text{C min}^{-1}$  under air atmosphere.

Dynamic mechanical analysis was performed on a TA DMA 2980 using a thin film tension clamp in order to characterize the thermal properties of the copolymer. After heating to  $220^\circ\text{C}$  to evaporate any remaining solvent, the samples were then equilibrated for 10 min at  $0^\circ\text{C}$  under nitrogen and heated at a rate of  $3^\circ\text{C min}^{-1}$  to  $450^\circ\text{C}$ , using an oscillation of 1 Hz.

## 2.4. Proton conductivity

Proton conductivity at  $30^\circ\text{C}$  at full hydration (in liquid water) was determined in a window cell geometry [26] using a Solartron 1252 coupled to a 1287 Impedance/Gain-Phase Analyzer over the frequency range of 10 Hz to 1 MHz following the procedure reported in the literature [27]. The measurement was in-plane conductivity.

## 2.5. MEA preparation and fuel cell characterization

Electrodes were fabricated by preparing an ink from glycerol, Nafion dispersion (5 wt%), and a platinum on carbon black catalyst (20 wt% Pt). The ink was painted onto a Teflon substrate to produce a decal with a platinum loading of  $0.2 \text{ mg cm}^{-2}$ . MEAs were prepared by hot pressing two of the electrode decals onto the acid form BPSH-35 membrane as per the experimental design. A fuel cell test stand (Fuel Cell Technologies) was used to measure high frequency resistance. The MEA was loaded into the fuel cell. The fuel cell was operated with  $\text{H}_2/\text{air}$  at  $90^\circ\text{C}$  and 100% relative humidity. The hydrogen was fed into the anode at 200 sccm with a humidified temperature of  $105^\circ\text{C}$  and the air flow into the cathode was 500 sccm at  $90^\circ\text{C}$ . Both cathode and anode had a back pressure of 20 psig.

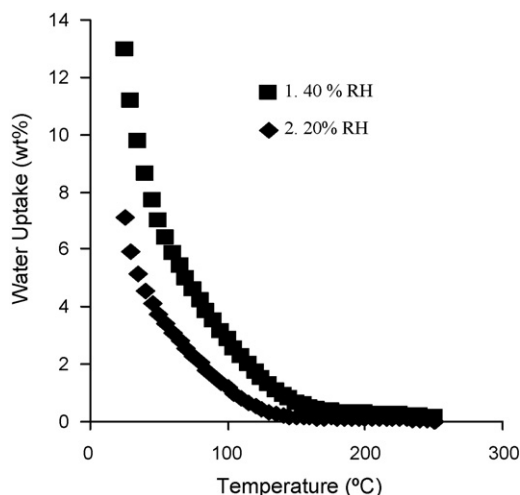
## 2.6. Experimental design

The membrane was hot pressed both with (MEA) and without (blank) electrodes over a matrix of experimental conditions, varying in fabrication temperatures and the RH at which the membranes were equilibrated before the MEA fabrication process. The experimental matrix is given in Table 2, which shows, for example, that Sample 1 was equilibrated at 40% RH to achieve a particular water uptake and subsequently hot pressed at a temperature of  $165^\circ\text{C}$ . The pressure was maintained at 2500 psi for 8 min and was kept same for all the samples. For the MEAs, a similar set of experi-

**Table 2**  
Experimental design.

Sample BPSH-35 <sup>a</sup>	Temperature ( $^\circ\text{C}$ )	RH (%)
1	165	40
2	210	40
3	165	20
4	210	20
5	165	0
6	210	0

<sup>a</sup>  $M_n$  of all samples was  $80,000 \text{ g mol}^{-1}$ .



**Fig. 3.** Retention of water as a function of temperature of the BPSH-35 random copolymer equilibrated at different relative humidities.

ments was performed, wherein the Pt loading was maintained at  $0.2 \text{ mg cm}^{-2}$ .

## 3. Results and discussion

### 3.1. Water uptake

Fig. 3 depicts the retention of water in the presence of water vapor for samples equilibrated at 40 and 20% RH. Samples equilibrated at 40% RH showed higher water uptake than the 20% RH samples over a range of temperatures as expected. Correspondingly, a depression in the glass transition or  $\alpha$  relaxation temperature (Table 3) was observed with increasing water uptake, via DSC experiments.

### 3.2. Effect of hot pressing conditions on proton conductivity of the blanks

The membranes were equilibrated at different relative humidities and hot pressed at different temperatures with (MEA) and without (blanks) electrodes. After the hot pressing the blanks were equilibrated in liquid water, the protonic conductivity was determined using a Solartron impedance analyzer at  $30^\circ\text{C}$ . Table 4 shows the conductivity results run on the blank membranes. The conductivity value for the unprocessed sample is a control and represents the value before hot pressing.

**Table 3**  
Depression of glass transition temperature of BPSH-35 with water uptake.

Relative humidity (%)	Water content (%)	$T_g$ ( $^\circ\text{C}$ ) <sup>a</sup>
0	0	260
20	7	188
40	13	165

<sup>a</sup> From DSC,  $\text{N}_2$ ,  $5^\circ\text{C min}^{-1}$ .

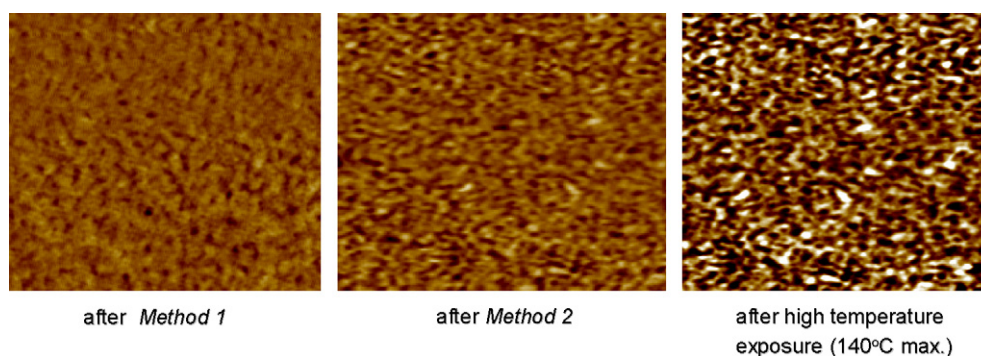


Fig. 4. AFM images of the exposed BPSH-35 at different conditions. Reprinted from Ref. [10], with permission from Elsevier.

Table 4

Protonic conductivity of the hot pressed samples in liquid water.

Sample BPSH-35	Temperature (°C)	RH (%)	Proton conductivity <sup>a</sup> (mS cm <sup>-1</sup> )
1	165	40	50
2	210	40	26
3	165	20	80
4	210	20	35
5	165	0	65
6	210	0	70
Control	–	–	85

<sup>a</sup> Liquid water, 30 °C, ±5%.

Significant changes in the proton conductivity were observed as a function of the hot pressing conditions, namely the temperature and the relative humidity at which the membranes were equilibrated was varied. Particularly for the sample which was equilibrated at 40% RH and then hot pressed at 210 °C (Sample 2), almost a three fold reduction in proton conductivity was observed compared to the control. Even when the equilibration RH was 20% and hot pressing temperature was 210 °C, a decrease in proton conductivity was observed. The hydrated glass transition temperature (Table 3) at 40% RH is 165 °C and at 20% RH is 188 °C, shifting the flow temperature for the copolymer to a lower value. However, the hot pressing temperature (210 °C) is much above the hydrated  $T_g$ . This may have resulted in morphological changes or relaxations within the copolymer. However, when the sample was vacuum dried and then hot pressed, no significant reduction in proton conductivity was observed. In this case, the hot pressing temperature (210 °C) was lower than the  $T_g$  (260 °C) of the copolymer.

As the hot pressing temperature was decreased to 165 °C, the sample (Sample 1) which was equilibrated at 40% RH prior to hot pressing showed a reduction in proton conductivity. The hot pressing temperature was still close to the hydrated glass transition temperature. However, as the sample was equilibrated at lower RHs (Samples 3 and 5), they displayed lower water content, and the hydrated  $T_g$  increased well above the processing temperature. Hence the conductivity remained unaffected with hot pressing.

Water uptake and DSC results indicated a depression in glass transition temperature with increasing water uptake (Table 3). This trend shifted the flow temperature of the copolymer to a lower value. The shift was more pronounced at higher water uptake levels. Processing the membranes at a temperature significantly

higher than the flow temperature likely results in morphological changes, which is influenced by relaxation within the copolymer. For example, in case of Nafion and other PEMs, conductivity initially increases with an increase in temperature, but then decreases once a particular temperature has been reached. This specific temperature has been found to be close to the hydrated  $\alpha$  relaxation temperature [9,28]. Above the  $\alpha$  relaxation temperature the copolymer morphology changes, thereby disturbing proton transport and hence, conductivity decreases. Similar morphological changes are also observed for BPSH-35 copolymer at higher temperatures. Fig. 4 represents the AFM images of BPSH-35 after exposure to different temperatures [10].

Methods 1 and 2 represent two different types of acidification treatments. In Method 1 the membrane was acidified at 30 °C in water, and in Method 2 the sample was exposed to 100 °C, also in water. It is evident from the AFM images that at 140 °C the sample experienced some morphological changes as the hydrated  $T_g$  for BPSH-35 copolymer under fully hydrated conditions was found occur at approximately that temperature [19]. Hence, hot pressing the membranes at higher water contents and at higher temperatures may have caused some morphological changes that resulted in a drop in conductivity values. To further investigate the effect of morphology on conductivity, the hot pressed Samples (blanks) 2 and 4 were treated with boiling DI water for 2 h, which is known as a hydrothermal treatment. Table 5 represents the enhanced conductivity values of the hot pressed samples after hydrothermal treatment.

For both Samples 2 and 4, conductivity values increased after hydrothermal treatment. In other words, the hydrothermal treatment produced a different morphology that facilitated the transport process as compared to the samples' pre-treatment morphology. Although it is difficult for now to conclusively comment on the reestablishment of the morphology, it is evident that the decrease in conductivity was due to a morphology effect rather than an irreversible chemical or mechanical degradation effect.

### 3.3. Effect of hot pressing conditions on fuel cell performance of the MEAs

Representative fuel cell performance for the MEAs equilibrated at different RHs and hot pressed at different temperatures is shown in Fig. 5. In case of the blanks, Sample 6 (vacuum dried and then

Table 5

Proton conductivity values after hydrothermal treatment for the hot pressed samples.

Sample	Temperature (°C)	RH (%)	MW (1000 g mol <sup>-1</sup> )	Initial <sup>a</sup> conductivity (mS cm <sup>-1</sup> )	Final <sup>b</sup> conductivity (mS cm <sup>-1</sup> )
4	210	20	80	35	80
2	210	40	80	25	65

<sup>a</sup> Liquid water, 30 °C, ±5%.

<sup>b</sup> Liquid water, 30 °C, ±5% after boiling water treatment.

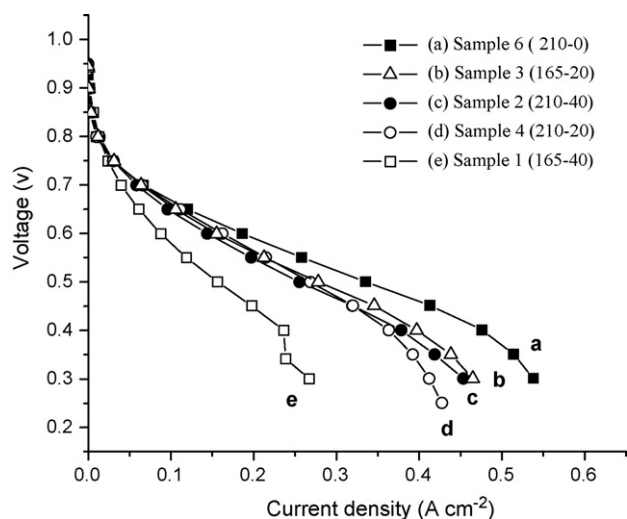


Fig. 5. (a–e) Influence of processing conditions on fuel cell performances for the MEAs.

pressed at 210 °C) had shown reasonable in-plane proton conductivity. The MEA-Sample 6 displayed the best fuel cell performance among all the samples studied. The higher hot pressing temperature ensured good bonding of the membrane with the electrodes. On the other hand, the fabrication temperature was approximately near the  $T_g$ , and no significant morphological relaxation taking place within the membrane. Sample 3 which showed the highest proton conductivity among the blanks, when used as MEA displayed a lower fuel cell performance than Sample 6. A lower bonding temperature (165 °C) used for this sample may have resulted insufficient bonding between the electrodes and the membrane in compare to the Sample 6 MEA.

As the hot pressing temperature decreased and RH at which the membrane was equilibrated increased, the performance decreased. The MEA from Sample 1, equilibrated at the highest RH and processed at the lowest temperature among the series had the lowest performance. It is suggested that the processing conditions shifted the flow temperature below the actual processing temperature resulting in a morphological relaxation within the copolymer and hence a loss in performance.

#### 4. Conclusions

BPSH-35 random copolymers with a molecular weight ( $M_n$ ) of 80,000 g mol<sup>-1</sup> were synthesized. Cast membranes were hot pressed both with (MEA) and without (blanks) electrodes over a matrix of experimental conditions, which were systematically varied in water content and temperature. Proton conductivity values determined for the blanks decreased with both increasing water content and the temperature of the fabrication process. The glass transition temperature or the flow temperature of the copolymer decreased with increasing water uptake, which impacted the ideal bonding temperature of the MEA. Processing the membranes at a temperature much higher than the flow temperature, may result in morphological changes and/or relaxation within the copolymer. Thus, MEAs should be fabricated at a temperature close to the hydrated glass transition temperature or  $\alpha$  relaxation of the

copolymer, which is a function of both water content and molecular weight.

Recommended further suggestions for fabricating MEAs can be summarized as follows:

- The MEA should be fabricated at a temperature close to the hydrated glass transition temperature of the copolymer, which is a function of both water content and molecular weight. Dynamic mechanical behavior might provide some guidance.
- If the processing temperature is too far below the hydrated  $T_g$ , flow will retard and the electrodes will not bind properly to the membrane, thus reducing performance.
- If the processing temperature is well above the hydrated  $T_g$ , the copolymer may undergo a morphological change that may lower its proton conductivity.
- Treatment of the membrane in boiling water has the potential to substantially restore proton conductivity.

#### Acknowledgements

The authors would like to thank UTC Fuel Cells (#PO3561) and Department of Energy (DOE) for their support.

#### References

- [1] M.A. Hickner, H. Ghassemi, Y.S. Kim, B.R. Einsla, J.E. McGrath, *Chem. Rev.* 104 (2004) 4587–4611.
- [2] M. Winter, R.J. Brodd, *Chem. Rev.* 105 (2005) 1021–11021.
- [3] L. Carrette, K.A. Friedrich, U. Stimming, *Chemphyschem* 1 (2000) 162–193.
- [4] Y. Li, A. Roy, A.S. Badami, M. Hill, J. Yang, S. Dunn, J.E. McGrath, *J. Power Sources* 172 (2007) 30–38.
- [5] H.-S. Lee, A. Roy, A.S. Badami, J.E. McGrath, *Macromol. Res.* 15 (2007) 160–166.
- [6] H.-S. Lee, A.S. Badami, A. Roy, J.E. McGrath, *J. Polym. Sci. A: Polym. Chem.* 45 (2007) 4879–4890.
- [7] A. Roy, M.A. Hickner, X. Yu, Y. Li, T.E. Glass, J.E. McGrath, *J. Polym. Sci. B: Polym. Phys.* 44 (2006) 2226–2239.
- [8] F. Wang, M. Hickner, Y.S. Kim, T.A. Zawodzinski, J.E. McGrath, *J. Membr. Sci.* 197 (2002) 231–242.
- [9] Y.S. Kim, F. Wang, M. Hickner, S. McCartney, Y.T. Hong, W. Harrison, T.A. Zawodzinski, J.E. McGrath, *J. Polym. Sci. B: Polym. Phys.* 41 (2003) 2816–2828.
- [10] Y.S. Kim, L.M. Dong, M.A. Hickner, B.S. Pivovar, J.E. McGrath, *Polymer* 44 (2003) 5729–5736.
- [11] Y. Li, F. Wang, J. Yang, D. Liu, A. Roy, S. Case, J. Lesko, J.E. McGrath, *Polymer* 47 (2006) 4210–4217.
- [12] H. Ghassemi, J.E. McGrath, J.T.A. Zawodzinski, *Polymer* 47 (2006) 4132–4139.
- [13] W.L. Harrison, M.A. Hickner, Y.S. Kim, J.E. McGrath, *Fuel Cells* 5 (2005) 201–212.
- [14] M.J. Sumner, W.L. Harrison, R.M. Weyers, Y.S. Kim, J.E. McGrath, J.S. Riffle, A. Brink, M.H. Brink, *J. Membr. Sci.* 239 (2004) 199–211.
- [15] Y.S. Kim, M.J. Sumner, W.L. Harrison, J.S. Riffle, J.E. McGrath, B.S. Pivovar, *J. Electrochem. Soc.* 151 (2004) A2150–A2156.
- [16] H. Ghassemi, G. Ndip, J.E. McGrath, *Polymer* 45 (2004) 5855–5862.
- [17] H. Ghassemi, J.E. McGrath, *Polymer* 45 (2004) 5847–5854.
- [18] B.R. Einsla, Y.T. Hong, Y.S. Kim, F. Wang, N. Gunduz, J.E. McGrath, *J. Polym. Sci. A: Polym. Chem.* 42 (2004) 862–874.
- [19] Y.S. Kim, L.M. Dong, M.A. Hickner, T.E. Glass, V. Webb, J.E. McGrath, *Macromolecules* 36 (2003) 6281–6285.
- [20] W.L. Harrison, F. Wang, J.B. Mechem, V.A. Bhanu, M. Hill, Y.S. Kim, J.E. McGrath, *J. Polym. Sci. A: Polym. Chem.* 41 (2003) 2264–2276.
- [21] G.J.K. Acres, *J. Power Sources* 100 (2001) 60–66.
- [22] N.P. Brandon, S. Skinner, B.C.H. Steele, *Annu. Rev. Mater. Res.* 33 (2003) 183–213.
- [23] J. Roziere, D.J. Jones, *Annu. Rev. Mater. Res.* 33 (2003) 503–555.
- [24] P.C. Hiemenz, T.P. Lodge, *Polymer Chemistry*, Marcel Dekker, New York, 2007.
- [25] M. Sankir, V.A. Bhanu, W.L. Harrison, H. Ghassemi, K.B. Wiles, T.E. Glass, A.E. Brink, M.H. Brink, J.E. McGrath, *J. Appl. Polym. Sci.* 100 (2006) 4595–4602.
- [26] T.A. Zawodzinski, M. Neeman, L.O. Sillerud, S. Gottesfeld, *J. Phys. Chem.* 95 (1991) 6040–6044.
- [27] T.E. Springer, T.A. Zawodzinski, M.S. Wilson, S. Gottesfeld, *J. Electrochem. Soc.* 143 (1996) 587–599.
- [28] K.A. Mauritz, R.B. Moore, *Chem. Rev. (Washington, DC, United States)* 104 (2004) 4535–4585.